

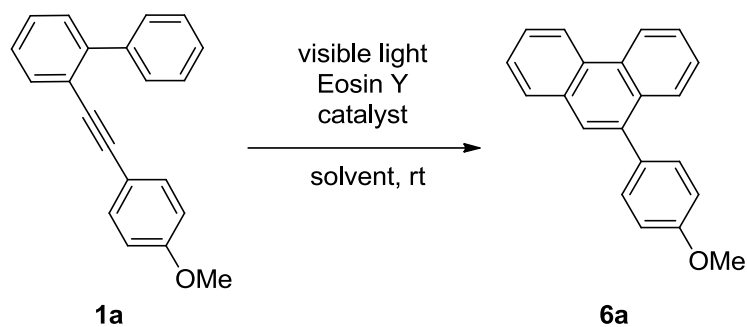
**Merging Photoredox Catalysis with Lewis Acid Catalysis: Activation of
Carbon-Carbon Triple Bond**

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Electronic Supplementary Information

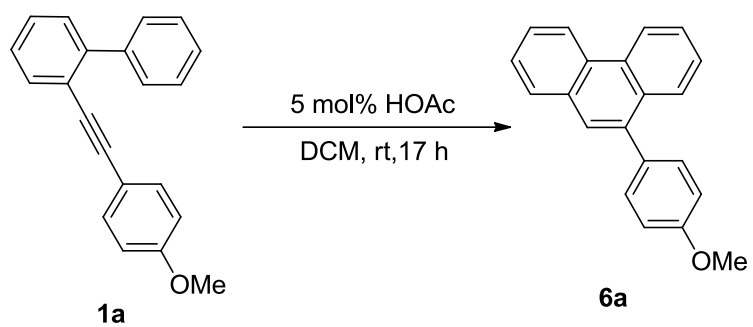
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Table S1. Optimization of reaction conditions.^a

Entry	Solvent	Eosin Y (mol%)	Cat. (mol%)	Time (h)	NMR yield (%) ^b
1	acetone	100	Cu(OTf) ₂ (100)	9	NR (>99) ^c
2	methanol	100	Cu(OTf) ₂ (100)	9	NR (99) ^c
3	DMF	100	Cu(OTf) ₂ (100)	9	NR (>99) ^c
4	acetonitrile	100	Cu(OTf) ₂ (100)	10	93
5	DCM	100	Cu(OTf) ₂ (100)	9	>99
6	DCM	20	Cu(OTf) ₂ (100)	6	>99
7	DCM	3	Cu(OTf) ₂ (100)	6	>99
8	DCM	0	Cu(OTf) ₂ (100)	6	5 (91) ^c
9	DCM	3	Cu(OTf) ₂ (20)	6	>99
10	DCM	3	Cu(OTf) ₂ (5)	11	>99 (96) ^d
11	DCM	3	CuOTf (5)	11	NR (>99) ^c
12	DCM	3	Cu(OAc) ₂ (5)	11	NR (98) ^c
13	DCM	3	CuF ₂ (5)	11	NR (>99) ^c
14	DCM	3	Fe(OTf) ₂ (5)	11	NR (>99) ^c
15	DCM	3	Zn(OTf) ₂ (5)	11	NR (96) ^c
16	DCM	3	ZnBr ₂ (5)	11	NR (>99) ^c
17	DCM	3	Cu(OTf) ₂ (0)	6	NR (99) ^c
18	DCM	0	Cu(OTf) ₂ (5)	6	NR (>99) ^c
19 ^e	DCM	3	Cu(OTf) ₂ (5)	24	NR (96) ^c
20 ^f	DCM	0	Cu(OTf) ₂ (3)	0.5	97 (96) ^d
21 ^f	DCM	0	Cu(OTf) ₂ (0)	0.5	NR (>99) ^c

22 ^g	DCM	3	Cu(OTf) ₂ (5)	4	>99
23 ^g	DCM	0	Cu(OTf) ₂ (5)	4	NR (>99) ^c

^a All reactions were carried out using **1a** (0.2 mmol), Cu(OTf)₂, and Eosin Y in anhydrous solvent (5 mL) in a thin glass tube irradiated by a 23 W household lamp at rt under argon atmosphere. ^b Yield determined by ¹H NMR (400 MHz) analysis employing CH₂Br₂ (0.2 mmol) as internal standard. ^c Recovered yield of **1a**. ^d Isolated yield of **6a**. ^e Reaction was performed without light. ^f The reaction was irradiated by 254 nm UV light. ^g The reaction was irradiated by Green LED lamp.



NR, 99% of **1a** was recovered

Scheme S1. Cyclization of 1a in the presence of HOAc.

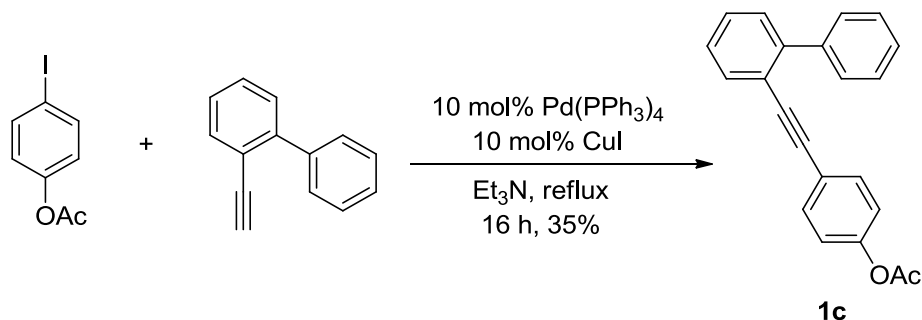
Experimental Section

General information

^1H (400 MHz), ^{13}C (100 MHz), and ^{19}F (376 MHz) NMR spectra of samples in CDCl_3 were recorded on an AVANCE III 400 spectrometer. IR spectra were recorded on an Avatar 360 FT-IR spectrometer. HRMS (EI, 70 eV) determinations were carried out on a Water GCT CA176 spectrometer. HRMS (ESI) determinations were carried out on a Bruker Daltonics APEXIIITM ESI-FTICRMS spectrometer. Melting points were determined on a WRS-2 apparatus. X-ray crystal was carried out on a Bruker SMART CCD. Anhydrous DCM and MeCN were distilled with CaH_2 . **1a**,¹**1b**,¹**1d**,¹**1e**,¹**1f**,¹**1g**,¹**1i**,¹**1j**,¹**1k**,¹**1l**,¹**1m**,¹**1n**,¹**1o**,²**1r**,¹**1s**,²**1t**,¹**1u**,²**1v**,²**1w**,²**1x**,¹ and **1y**¹ were synthesized according to literature procedures.

Typical Procedure I for the synthesis of arene-alkynes.

Synthesis of 2-((4-acetoxyphenyl)ethynyl)-1,1'-biphenyl (**1c**)

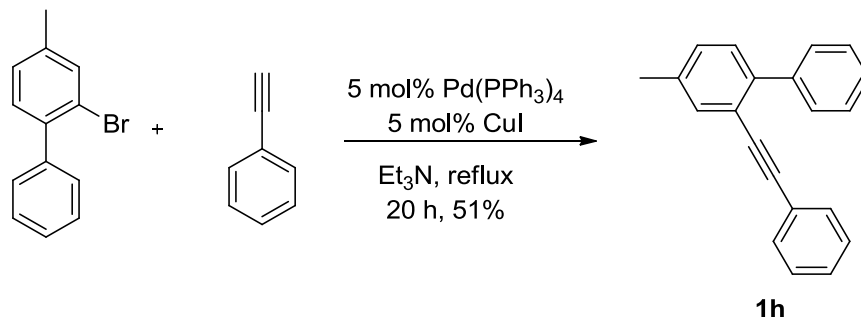


1-Acetoxy-4-iodobenzene (2.613 g, 10.0 mmol), 2-ethynyl-1,1'-biphenyl (1.881 g, 10.6 mmol), $\text{Pd}(\text{PPh}_3)_4$ (1.215 g, 0.98 mmol), CuI (191 mg, 1.00 mmol), and Et_3N (125 mL) were added subsequently into a 250 mL of dry three-necked flask. The resulting mixture was refluxed. The reaction was completed after 16 hours as monitored by TLC (eluent: petroleum ether). Then it was cooled to room temperature. The solvent was removed and the residue was purified by flash chromatography on silica gel (eluent: petroleum ether) to afford **1c** as a liquid (1.082 g, 35%). ^1H NMR (400 MHz, CDCl_3) δ 7.67-7.59 (m, 3 H), 7.47-7.28 (m, 8 H), 7.03-6.97 (m, 2 H), 2.26 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3) δ 169.0, 150.3, 143.9, 140.5, 132.7, 132.4, 129.4, 129.3, 128.5, 127.8, 127.4, 127.0, 121.6, 121.4, 121.1, 91.4, 89.4, 21.0; IR (neat) 1772, 1760, 1738, 1692, 1604, 1482, 1452, 1430, 1366 cm^{-1} ; HRMS (EI, 70

eV) calcd for C₂₂H₁₆O₂ 312.1150, found 312.1146.

The following compound was prepared according to Typical Procedure I.

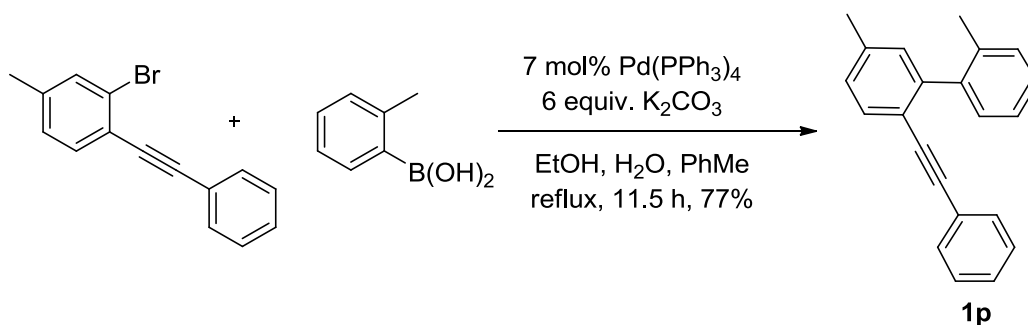
1) 4-Methyl-2-(phenylethynyl)-1,1'-biphenyl (**1h**)



The reaction of 2-bromo-4-methyl-1,1'-biphenyl (5.603 g, 22.9 mmol), phenylacetylene (3 mL, 27.3 mmol), Pd(PPh₃)₄ (1.319 g, 1.14 mmol), CuI (217 mg, 1.14 mmol), and Et₃N (150 mL) afforded **1h** as a liquid (3.126 g, 51%). ¹H NMR (400 MHz, CDCl₃) δ 7.64 (d, *J* = 8.4 Hz, 2 H), 7.51-7.20 (m, 10 H), 7.19-7.11 (m, 1 H), 2.35 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 141.1, 140.5, 136.7, 133.3, 131.3, 129.5, 129.3, 128.2, 128.0, 127.8, 127.2, 123.5, 121.2, 91.8, 89.6, 20.8; IR (neat) 1598, 1509, 1489, 1478, 1439 cm⁻¹; HRMS (EI, 70 eV) calcd for C₂₁H₁₆ 268.1252, found 268.1250.

Typical Procedure II for the synthesis of arene-alkynes.

Synthesis of 2',5-dimethyl-2-(phenylethynyl)-1,1'-biphenyl (**1p**)

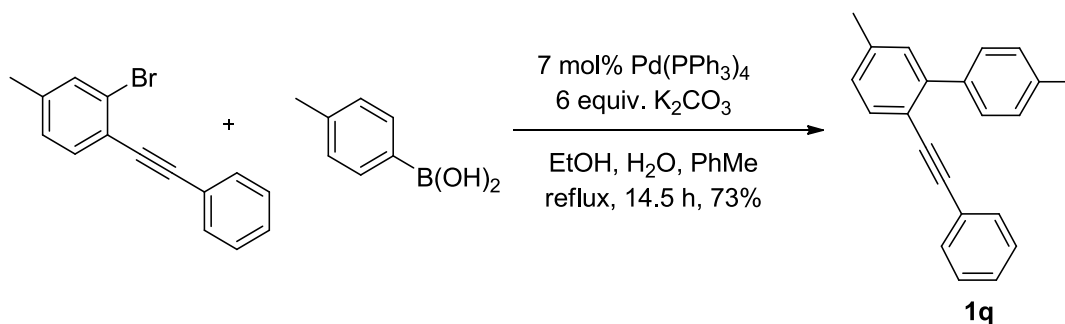


2-Bromo-4-methyl-1-(phenylethynyl)benzene (2.716 g, 10.1 mmol), *o*-tolylboronic acid (1.401 g, 10.3 mmol), Pd(PPh₃)₄ (882 mg, 0.74 mmol), K₂CO₃ (8.311 g, 60.1 mmol), EtOH (50 mL), H₂O (50 mL), and PhMe (200 mL) were added subsequently into a 500 mL of dry three-necked flask. The resulting mixture was refluxed. The reaction was completed after 11.5 hours as monitored by TLC (eluent: petroleum

ether). Then it was cooled to room temperature. The solvent was removed and the residue was purified by flash chromatography on silica gel (eluent: petroleum ether) to afford **1p** as a liquid (2.176 g, 77%). ¹H NMR (400 MHz, CDCl₃) δ 7.49 (d, *J* = 7.6, 1 H), 7.32-7.15 (m, 7 H), 7.14-7.01 (m, 4 H), 2.38 (s, 3 H), 2.21 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 144.6, 140.9, 138.2, 136.3, 131.5, 131.2, 130.2, 129.8, 129.6, 128.1, 127.78, 127.75, 127.4, 125.2, 123.5, 119.9, 91.5, 89.1, 21.5, 20.0; IR (neat) 1595, 1571, 1500, 1488, 1439 cm⁻¹; HRMS (EI, 70 eV) calcd for C₂₂H₁₈ 282.1409, found 282.1407.

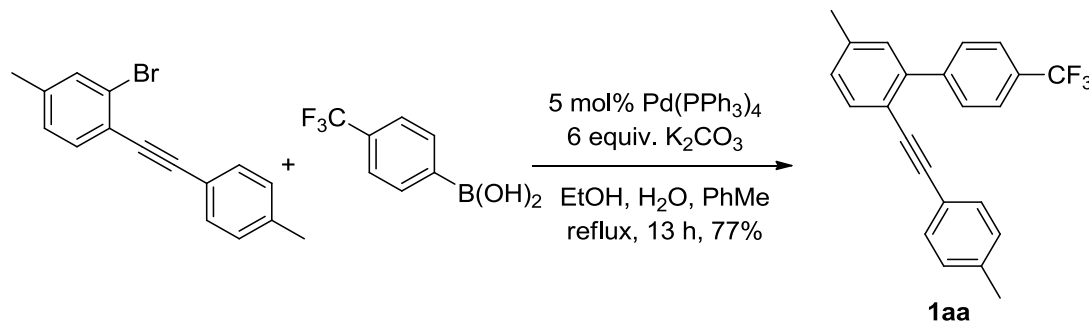
The following compounds were prepared according to Typical Procedure II.

1) 4',5-Dimethyl-2-(phenylethynyl)-1,1'-biphenyl (**1q**)



The reaction of 2-bromo-4-methyl-1-(phenylethynyl)benzene (2.713 g, 10.0 mmol), *p*-tolylboronic acid (1.370 g, 10.1 mmol), Pd(PPh₃)₄ (892 mg, 0.77 mmol), K₂CO₃ (8.312 g, 60.1 mmol), EtOH (50 mL), H₂O (50 mL), and PhMe (200 mL) afforded **1q**³ as a liquid (2.048 g, 73%). ¹H NMR (400 MHz, CDCl₃) δ 7.59-7.46 (m, 3 H), 7.39-7.19 (m, 8 H), 7.12 (d, *J* = 8.0, 1 H), 2.42 (s, 3 H), 2.40 (s, 3 H).

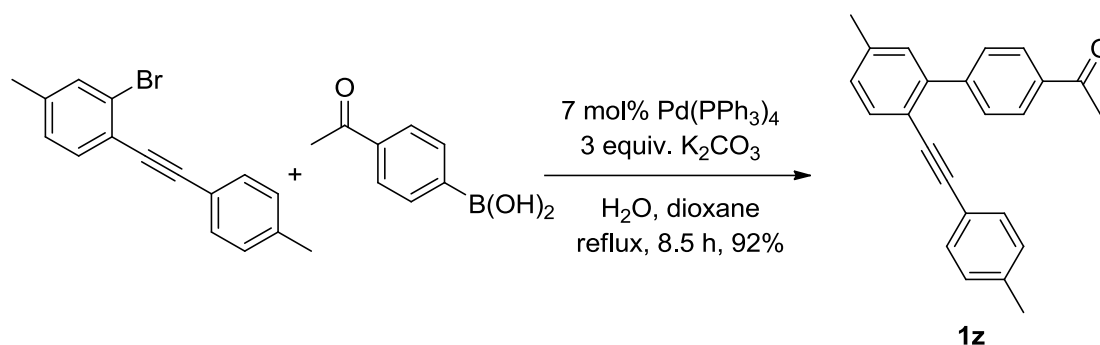
2) 5-Methyl-4'-trifluoromethyl-2-((4-methylphenyl)ethynyl)-1,1'-biphenyl (**1aa**)



The reaction of 2-bromo-4-methyl-1-(*p*-tolylethynyl)benzene (4.270 g, 15.0 mmol), (4-(trifluoromethyl)phenyl)boronic acid (2.900 g, 15.3 mmol), Pd(PPh₃)₄ (877 mg, 0.76 mmol), K₂CO₃ (12.536 g, 90.7 mmol), EtOH (20 mL), H₂O (20 mL), and PhMe

(75 mL) afforded **1aa** as a solid (4.046 g, 77%); mp 111.9-112.7 °C (ethyl acetate/petroleum ether). ¹H NMR (400 MHz, CDCl₃) δ 7.75 (d, *J* = 8.0 Hz, 2 H), 7.68 (d, *J* = 8.4 Hz, 2 H), 7.54 (d, *J* = 8.0 Hz, 1 H), 7.24-7.14 (m, 4 H), 7.09 (d, *J* = 8.0 Hz, 2 H), 2.41 (s, 3 H), 2.32 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 144.3, 142.1, 138.6, 138.3, 132.8, 131.1, 130.1, 129.7, 129.1, 128.6, 124.8, 124.7, 120.2, 118.9, 92.3, 88.2, 21.4; ¹⁹F NMR (376 MHz, CDCl₃) δ -62.3; IR (neat) 1613, 1558, 1513, 1482 cm⁻¹; HRMS (EI, 70 eV) calcd for C₂₃H₁₇F₃ 350.1282, found: 350.1275.

Synthesis of 5-methyl-4'-acetyl-2-((4-methylphenyl)ethynyl)-1,1'-biphenyl (**1z**)

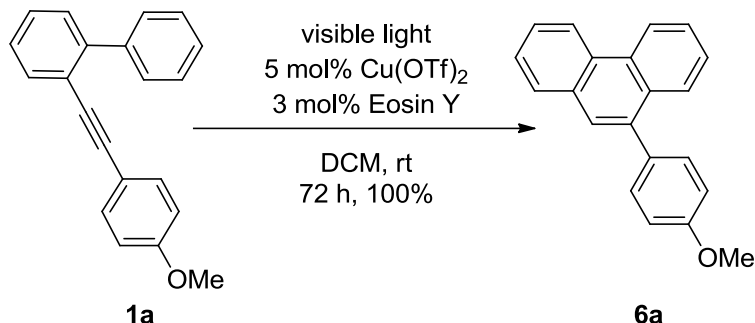


2-Bromo-4-methyl-1-(*p*-tolylethynyl)benzene (853 mg, 3.0 mmol), (4-acetylphenyl)boronic acid (492 mg, 3 mmol), Pd(PPh₃)₄ (243 mg, 0.21 mmol), K₂CO₃ (954 mg, 9 mmol), H₂O (15 mL), and dioxane (50 mL) were added subsequently into a 100 mL of dry three-necked flask. The resulting mixture was refluxed. The reaction was completed after 8.5 hours as monitored by TLC (eluent: petroleum ether:ethyl acetate = 30:1). Then it was cooled to room temperature, and extracted with ethyl acetate (20 mL x 3). The combined organic layer was dried over MgSO₄. After filtration and concentration, the residue was purified by flash column chromatography on silica gel (eluent: petroleum ether:ethyl acetate = 50:1) to afford **1z** as a solid (894 mg, 92%); mp 99.6-100.2 °C (ethyl acetate/petroleum ether). ¹H NMR (400 MHz, CDCl₃) δ 8.04 (d, *J* = 8.4 Hz, 2 H), 7.77 (d, *J* = 8.4 Hz, 2 H), 7.55 (d, *J* = 7.6 Hz, 1 H), 7.25-7.15 (m, 4 H), 7.09 (d, *J* = 8.0 Hz, 2 H), 2.66 (s, 3 H), 2.43 (s, 3 H), 2.34 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 198.0, 145.6, 142.2, 138.6, 138.3, 135.8, 132.9, 131.1, 130.1, 129.5, 129.0, 128.6, 127.9, 120.2, 118.8, 92.2, 88.2, 26.7, 21.4; IR (neat) 1683, 1604, 1558, 1513, 1482, 1446, 1412 cm⁻¹; HRMS (ESI)

calcd for C₂₄H₂₁O (M + H⁺) 325.1587, found 325.1599.

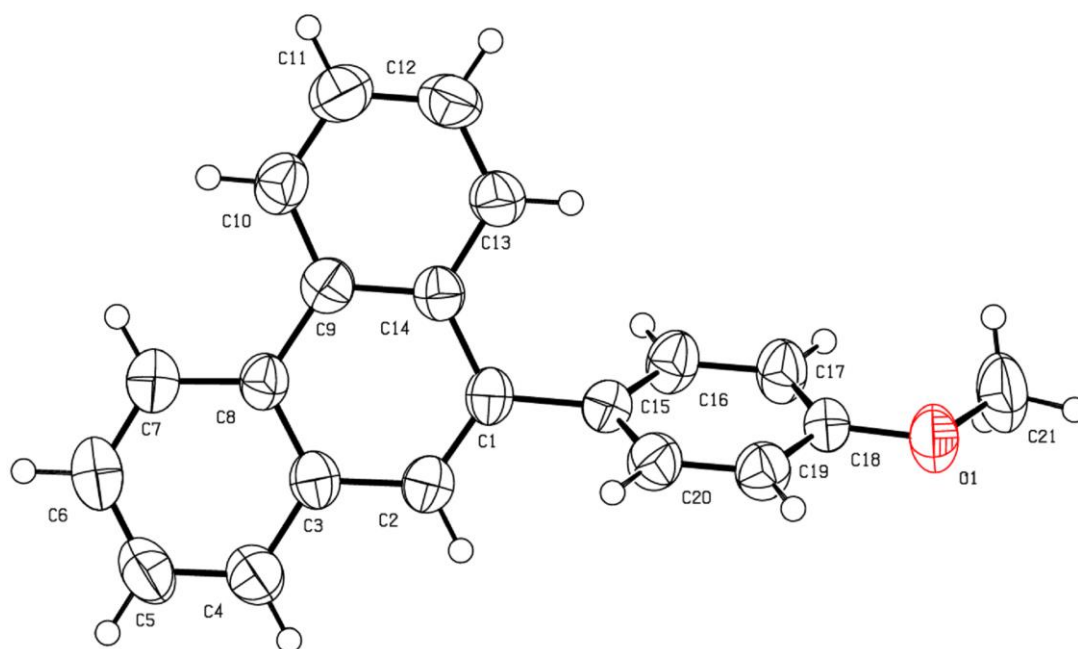
Typical Procedure III for the photoreaction.

Synthesis of 9-(4-methoxyphenyl)phenanthrene (6a)



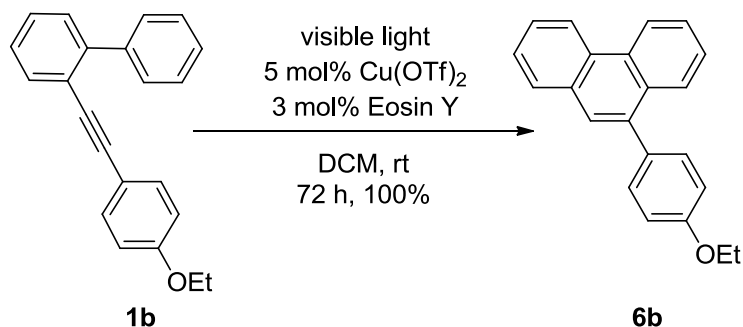
1a (57 mg, 0.20 mmol), Cu(OTf)₂ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous DCM (5 mL) were added subsequently into a 25 mL of dry pyrex sealed tube. The reaction mixture was degassed with three freeze-pump-thaw cycles. Then, the reaction mixture was irradiated by a 23 W household lamp at rt under argon atmosphere. The photoreaction was completed after 72 h as monitored by TLC (eluent: petroleum ether). The solvent was removed and the residue was purified by flash chromatography on silica gel (eluent: petroleum ether) to afford **6a**² as a liquid (58 mg, 100%). ¹H NMR (400 MHz, CDCl₃) δ 8.82 (d, *J* = 8.4, 1 H), 8.77 (d, *J* = 8.4, 1 H), 8.04 (d, *J* = 8.4, 1 H), 7.94 (d, *J* = 8.0, 1 H), 7.78-7.48 (m, 7 H), 7.11 (d, *J* = 8.8, 2 H), 3.94 (s, 3 H).

Crystal data for **6a**: C₂₁H₁₆O, MW = 284.34, Monoclinic, space group P 21/c, a51119b, final R indices [I>2σ(I)], R1 = 0.0476, wR2 = 0.0826, a = 13.33(5) Å, b = 15.13(5) Å, c = 7.59(3) Å, α = 90°, β = 100.77(5)°, γ = 90°, V = 1503(9) Å³, T = 296(2) K, Z = 4, reflections collected / unique: 5793, [R(int) = 0.0977], parameters 200. Supplementary crystallographic data have been deposited at the Cambridge Crystallographic Data Center. CCDC: 1442356.



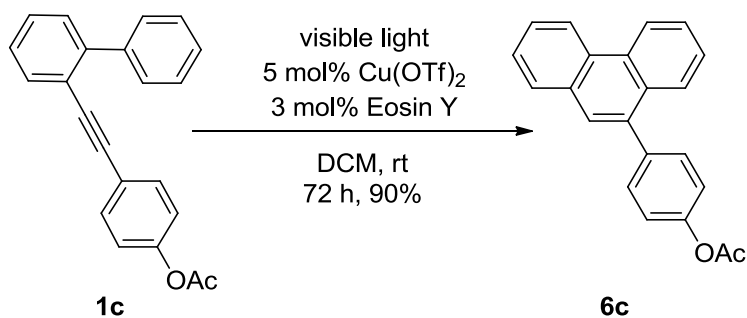
The following compounds were prepared according to Typical Procedure III.

1) 9-(4-Ethoxyphenyl)phenanthrene (**6b**)



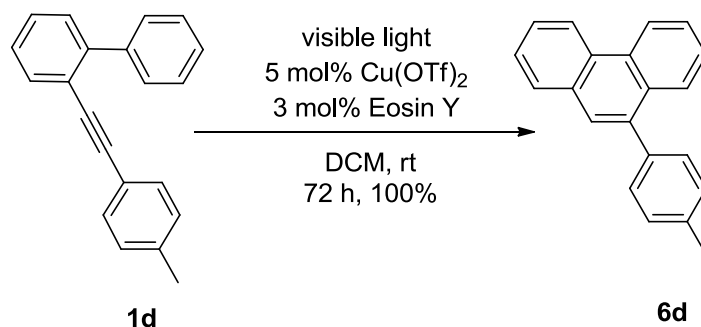
The reaction of **1b** (62 mg, 0.21 mmol), Cu(OTf)₂ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous DCM (5 mL) afforded **6b**² as a solid (63 mg, 100%). ¹H NMR (400 MHz, CDCl₃) δ 8.77 (d, *J* = 8.4 Hz, 1 H), 8.72 (d, *J* = 8.0 Hz, 1 H), 7.95 (d, *J* = 8.4 Hz, 1 H), 7.88 (d, *J* = 8.0 Hz, 1 H), 7.73-7.40 (m, 7 H), 7.04 (d, *J* = 8.8 Hz, 1 H), 4.14 (q, *J* = 7.2 Hz, 2 H), 1.49 (t, *J* = 7.2 Hz, 3 H).

2) 9-(4-Acetoxyphenyl)-phenanthrene (**6c**)



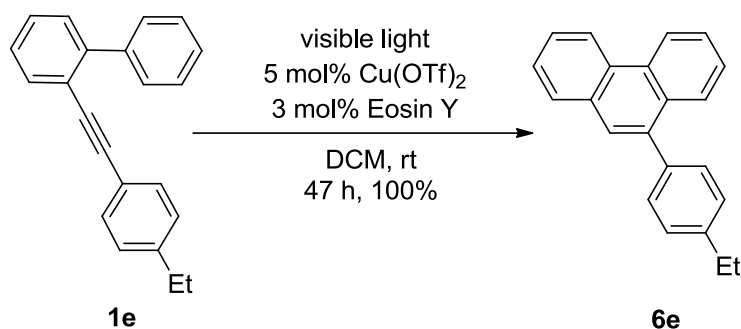
The reaction of **1c** (62 mg, 0.20 mmol), Cu(OTf)₂ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous DCM (5 mL) afforded **6c** as a liquid (56 mg, 90%). ¹H NMR (400 MHz, CDCl₃) δ 8.78 (d, *J* = 8.4 Hz, 1 H), 8.73 (d, *J* = 8.4 Hz, 1 H), 7.91 (t, *J* = 8.4 Hz, 2 H), 7.71-7.49 (m, 7 H), 7.24 (d, *J* = 8.8 Hz, 2 H), 2.37 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 169.5, 150.1, 138.4, 137.8, 131.5, 131.1, 130.6, 130.0, 128.7, 127.7, 126.9, 126.8, 126.7, 126.6, 126.5, 122.9, 122.5, 121.4, 21.2; IR (neat) 1766, 1601, 1528, 1503, 1488, 1452, 1427 cm⁻¹; HRMS (ESI) calcd for C₂₂H₁₇O₂ (M + H⁺) 313.1223, found 313.1222.

3) 9-(*p*-Tolyl)phenanthrene (**6d**)



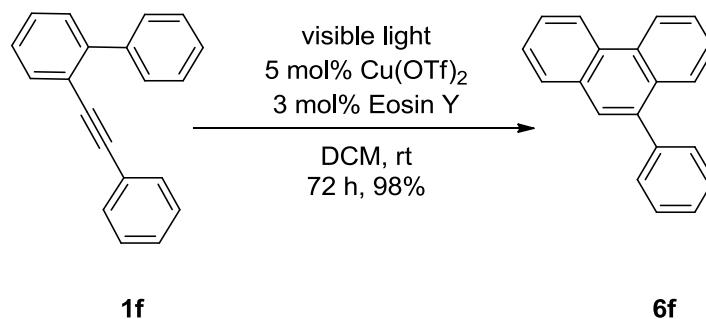
The reaction of **1d** (54 mg, 0.20 mmol), Cu(OTf)₂ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous DCM (5 mL) afforded **6d**² as a solid (54 mg, 100%). ¹H NMR (400 MHz, CDCl₃) δ 8.75 (d, *J* = 8.4 Hz, 1 H), 8.70 (d, *J* = 8.4 Hz, 1 H), 7.93 (d, *J* = 8.0 Hz, 1 H), 7.86 (d, *J* = 8.4 Hz, 1 H), 7.69-7.38 (m, 7 H), 7.31 (d, *J* = 7.6 Hz, 2 H), 2.46 (s, 3 H).

4) 9-(4-Ethylphenyl)phenanthrene (**6e**)



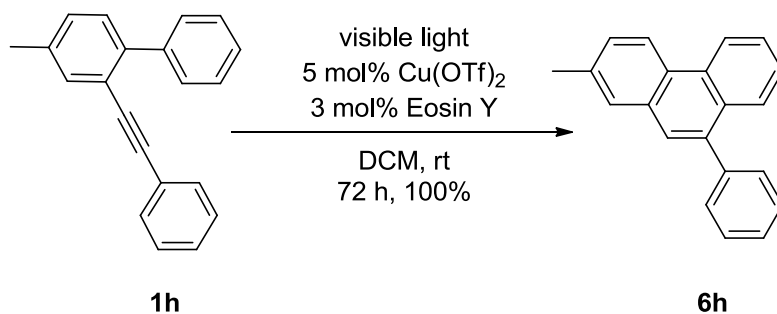
The reaction of **1e** (57 mg, 0.20 mmol), Cu(OTf)₂ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous DCM (5 mL) afforded **6e** as a solid (58 mg, 100%); mp 83.6-85.5 °C (ethyl acetate/petroleum ether). ¹H NMR (400 MHz, CDCl₃) δ 8.77 (d, *J* = 8.0 Hz, 1 H), 8.72 (d, *J* = 8.0 Hz, 1 H), 7.96 (d, *J* = 8.0 Hz, 1 H), 7.89 (d, *J* = 7.2 Hz, 1 H), 7.72-7.43 (m, 7 H), 7.35 (d, *J* = 7.6 Hz, 2 H), 2.78 (q, *J* = 7.6 Hz, 2 H), 1.35 (t, *J* = 7.6 Hz, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 143.4, 138.8, 138.0, 131.6, 131.2, 130.6, 130.0, 129.9, 128.6, 127.8, 127.4, 127.0, 126.8, 126.44, 126.41, 126.36, 122.8, 122.5, 28.7, 15.6; IR (neat) 1525, 1507, 1488, 1449, 1430, 1418, 1409 cm⁻¹; HRMS (EI, 70 eV) calcd for C₂₂H₁₈ 282.1409, found 282.1412.

5) 9-Phenylphenanthrene (**6f**)



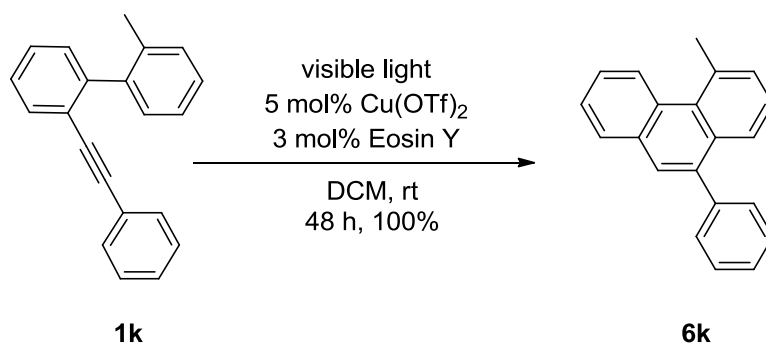
The reaction of **1f** (51 mg, 0.20 mmol), Cu(OTf)₂ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous DCM (5 mL) afforded **6f**² as a solid (50 mg, 98%). ¹H NMR (400 MHz, CDCl₃) δ 8.78 (d, *J* = 8.0 Hz, 1 H), 8.73 (d, *J* = 8.0 Hz, 1 H), 7.95-7.85 (m, 2 H), 7.73-7.41 (m, 10 H).

6) 2-Methyl-9-phenylphenanthrene (**6h**)



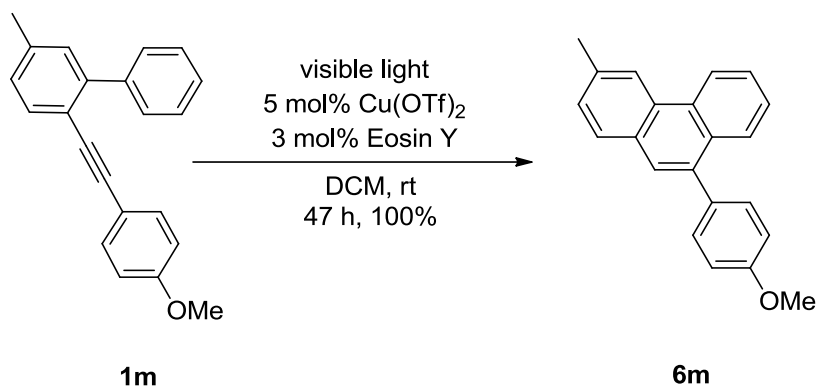
The reaction of **1h** (54 mg, 0.20 mmol), Cu(OTf)₂ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous DCM (5 mL) afforded **6h** as a liquid (54 mg, 100%). ¹H NMR (400 MHz, CDCl₃) δ 8.73 (d, *J* = 8.0 Hz, 1 H), 8.61 (d, *J* = 8.8 Hz, 1 H), 7.89 (d, *J* = 8.4 Hz, 1 H), 7.70-7.59 (m, 3 H), 7.58-7.41 (m, 7 H), 2.57 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 140.9, 138.7, 136.6, 131.7, 130.8, 130.6, 130.1, 128.4, 128.3, 128.2, 127.8, 127.3, 126.9, 126.4, 126.0, 122.7, 122.4, 21.5; IR (neat) 1622, 1595, 1525, 1485, 1449, 1427 cm⁻¹; HRMS (EI, 70 eV) calcd for C₂₁H₁₆ 268.1252, found 268.1250.

7) 4-Methyl-10-phenylphenanthrene (**6k**)



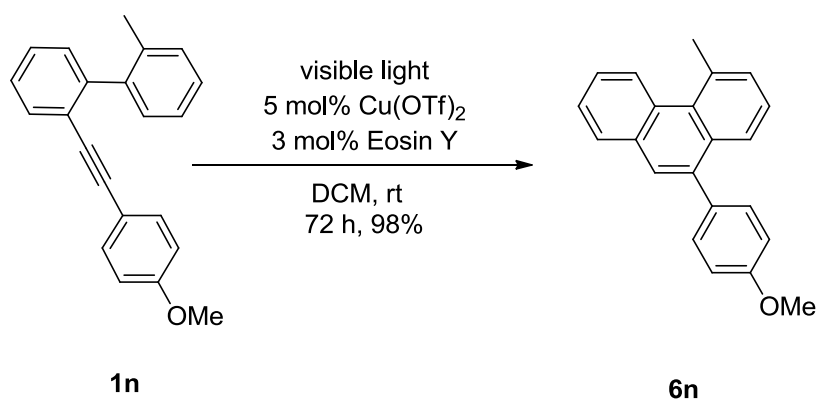
The reaction of **1k** (54 mg, 0.20 mmol), Cu(OTf)₂ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous DCM (5 mL) afforded **6k**² as a liquid (54 mg, 100%). ¹H NMR (400 MHz, CDCl₃) δ 8.89 (d, *J* = 8.0 Hz, 1 H), 7.90 (d, *J* = 6.4 Hz, 1 H), 7.77 (d, *J* = 8.0 Hz, 1 H), 7.69-7.35 (m, 10 H), 3.18 (s, 3 H).

8) 9-(4-Methoxyphenyl)-3-methylphenanthrene (**6m**)



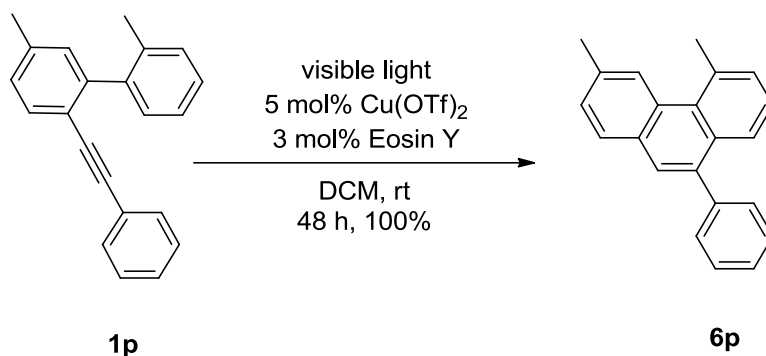
The reaction of **1m** (60 mg, 0.20 mmol), Cu(OTf)₂ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous DCM (5 mL) afforded **6m**⁴ as a solid (60 mg, 100%). ¹H NMR (400 MHz, CDCl₃) δ 8.72 (d, *J* = 8.0 Hz, 1 H), 8.47 (s, 1 H), 7.91 (d, *J* = 8.0 Hz, 1 H), 7.73 (d, *J* = 8.0 Hz, 1 H), 7.63-7.51 (m, 2 H), 7.50-7.34 (m, 4 H), 7.01 (d, *J* = 8.4 Hz, 2 H), 3.85 (s, 3 H), 2.60 (s, 3 H).

9) 10-(4-Methoxyphenyl)-4-methylphenanthrene (6n)



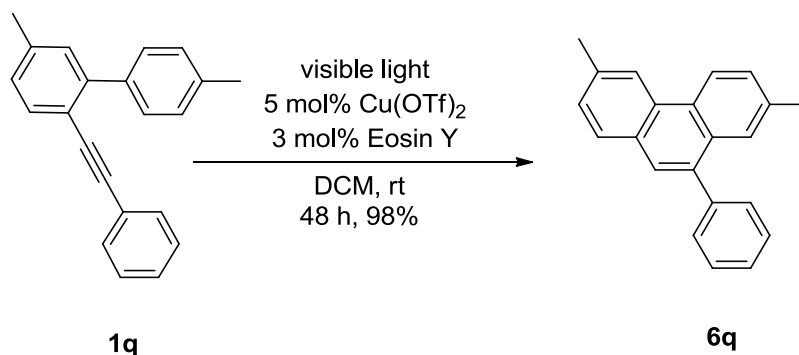
The reaction of **1n** (60 mg, 0.20 mmol), Cu(OTf)₂ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous DCM (5 mL) afforded **6n**² as a solid (59 mg, 98%). ¹H NMR (400 MHz, CDCl₃) δ 8.89 (d, *J* = 8.8 Hz, 1 H), 7.90 (d, *J* = 9.6 Hz, 1 H), 7.81 (d, *J* = 8.0 Hz, 1 H), 7.69-7.55 (m, 3 H), 7.54-7.48 (m, 1 H), 7.46-7.38 (m, 3 H), 7.04 (d, *J* = 8.8 Hz, 2 H), 3.91 (s, 3 H), 3.19 (s, 3 H).

10) 3,5-Dimethyl-9-phenylphenanthrene (6p)



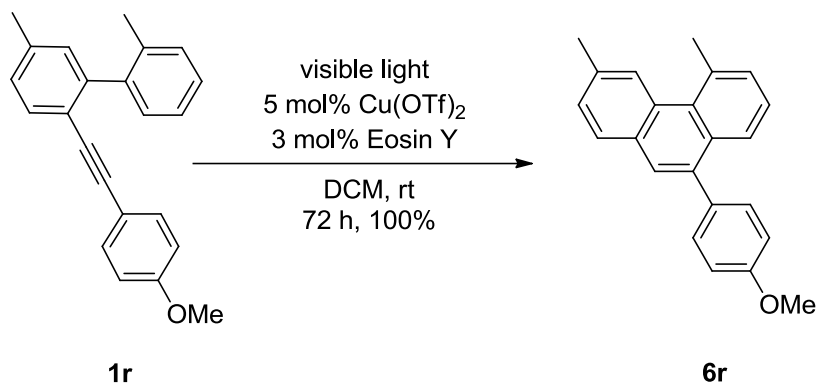
The reaction of **1p** (57 mg, 0.20 mmol), Cu(OTf)₂ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous DCM (5 mL) afforded **6p** as a liquid (57 mg, 100%). ¹H NMR (400 MHz, CDCl₃) δ 8.68 (s, 1 H), 7.79 (d, *J* = 8.4 Hz, 1 H), 7.75 (d, *J* = 8.0 Hz, 1 H), 7.60 (s, 1 H), 7.55-7.35 (m, 8 H), 3.18 (s, 3 H), 2.63 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 141.6, 138.3, 135.6, 135.0, 133.0, 131.2, 130.9, 130.8, 130.4, 130.1, 128.5, 128.2, 127.73, 127.68, 127.5, 127.1, 125.50, 125.48, 27.6, 22.5; IR (neat) 1610, 1598, 1577, 1519, 1503, 1488, 1439 cm⁻¹; HRMS (EI, 70 eV) calcd for C₂₂H₁₈ 282.1409, found 282.1403.

11) 2,6-Dimethyl-10-phenylphenanthrene (**6q**)



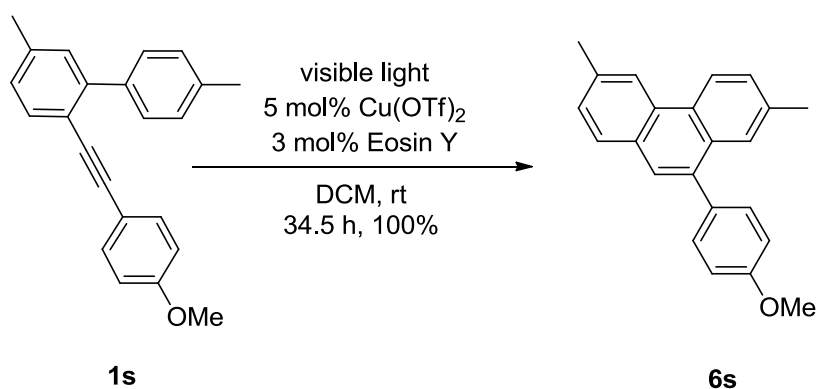
The reaction of **1q** (57 mg, 0.20 mmol), Cu(OTf)₂ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous DCM (5 mL) afforded **6q**⁵ as a liquid (56 mg, 98%). ¹H NMR (400 MHz, CDCl₃) δ 8.64 (d, *J* = 8.4 Hz, 1 H), 8.46 (s, 1 H), 7.75 (d, *J* = 8.0 Hz, 1 H), 7.66 (s, 1 H), 7.60 (s, 1 H), 7.59-7.34 (m, 7 H), 2.62 (s, 3 H), 2.46 (s, 3 H).

12) 10-(4-Ethylphenyl)-2,6-dimethylphenanthrene (**6r**)



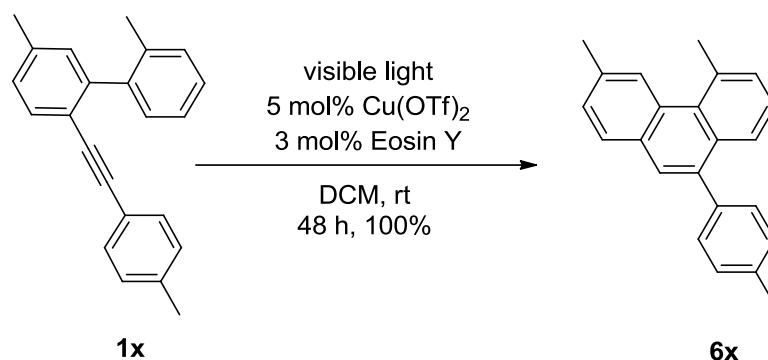
The reaction of **1r** (65 mg, 0.21 mmol), Cu(OTf)₂ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous DCM (5 mL) afforded **6r**² as a solid (65 mg, 100%). ¹H NMR (400 MHz, CDCl₃) δ 8.68 (s, 1 H), 7.79 (d, *J* = 8.4 Hz, 2 H), 7.59 (s, 1 H), 7.52-7.36 (m, 5 H), 7.03 (d, *J* = 8.4 Hz, 2 H), 3.90 (s, 3 H), 3.18 (s, 3 H), 2.63 (s, 3 H).

13) 10-(4-Methoxyphenyl)-2,6-dimethylphenanthrene (**6s**)



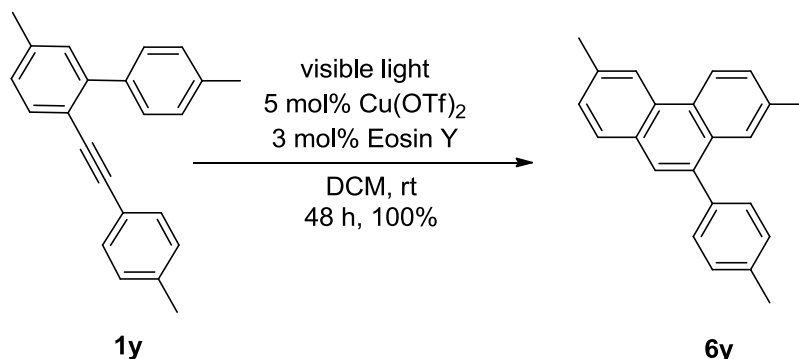
The reaction of **1s** (63 mg, 0.20 mmol), Cu(OTf)₂ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous DCM (5 mL) afforded **6s**² as a solid (63 mg, 100%). ¹H NMR (400 MHz, CDCl₃) δ 8.63 (d, *J* = 8.8 Hz, 1 H), 8.45 (s, 1 H), 7.74 (d, *J* = 8.0 Hz, 1 H), 7.68 (s, 1 H), 7.58 (s, 1 H), 7.53-7.34 (m, 4 H), 7.04 (d, *J* = 8.8 Hz, 2 H), 3.90 (s, 3 H), 2.62 (s, 3 H), 2.46 (s, 3 H).

14) 3,5-Dimethyl-9-(*p*-tolyl)phenanthrene (**6x**)



The reaction of **1x** (59 mg, 0.20 mmol), Cu(OTf)₂ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous DCM (5 mL) afforded **6x** as a liquid (59 mg, 100%). ¹H NMR (400 MHz, CDCl₃) δ 8.68 (s, 1 H), 7.79 (d, *J* = 8.0 Hz, 2 H), 7.59 (s, 1 H), 7.48 (d, *J* = 6.8 Hz, 1 H), 7.45-7.32 (m, 4 H), 7.30 (d, *J* = 7.6 Hz, 2 H), 3.18 (s, 3 H), 2.63 (s, 3 H), 2.46 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 138.7, 138.3, 136.8, 135.5, 134.9, 133.2, 131.2, 130.8, 130.4, 130.0, 129.2, 128.9, 128.5, 127.7, 127.5, 125.6, 125.4, 27.6, 22.5, 21.3; IR (neat) 1614, 1594, 1509, 1497, 1462, 1441 cm⁻¹; HRMS (EI, 70 eV) calcd for C₂₃H₂₀ 296.1565, found 296.1560.

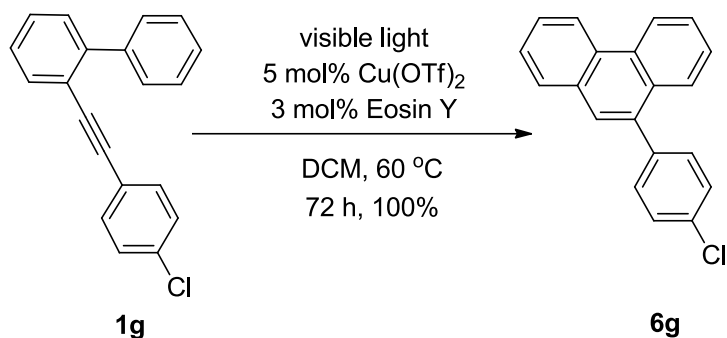
15) 2,6-Dimethyl-10-(*p*-tolyl)phenanthrene (**6y**)



The reaction of **1y** (59 mg, 0.20 mmol), Cu(OTf)₂ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous DCM (5 mL) afforded **6y** as a liquid (59 mg, 100%). ¹H NMR (400 MHz, CDCl₃) δ 8.64 (d, *J* = 8.4 Hz, 1 H), 8.45 (s, 1 H), 7.75 (d, *J* = 8.0 Hz, 1 H), 7.69 (s, 1 H), 7.59 (s, 1 H), 7.50-7.35 (m, 4 H), 7.32 (d, *J* = 7.6 Hz, 1 H), 2.62 (s, 3 H), 2.48 (s, 3 H), 2.46 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 138.2, 137.5, 136.8, 136.1, 136.0, 131.4, 130.0, 129.9, 129.2, 129.0, 128.4, 128.1, 127.9, 127.4, 126.4, 122.8, 122.0, 22.2, 21.7, 21.3; IR (neat) 1619, 1598, 1558, 1522, 1507, 1494, 1439 cm⁻¹; HRMS (EI, 70 eV) calcd for C₂₃H₂₀ 296.1565, found 296.1568.

Typical Procedure IV for the photoreaction.

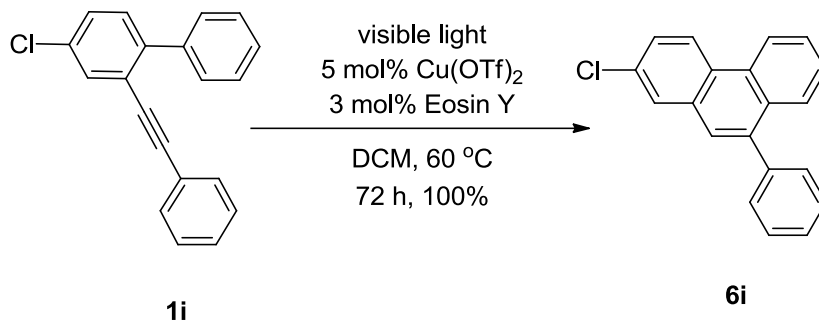
Synthesis of 9-(4-chlorophenyl)phenanthrene (**6g**)



1g (58 mg, 0.20 mmol), Cu(OTf)₂ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous DCM (5 mL) were added subsequently into a 25 mL of dry pyrex sealed tube. The reaction mixture was degassed with three freeze-pump-thaw cycles. Then, the reaction mixture was irradiated by a 23 W household lamp at 60 °C under argon atmosphere. The photoreaction was completed after 72 h as monitored by TLC (eluent: petroleum ether). The solvent was removed and the residue was purified by flash chromatography on silica gel (eluent: petroleum ether) to afford **6g**⁵ as a solid (59 mg, 100%). ¹H NMR (400 MHz, CDCl₃) δ 8.78 (d, *J* = 8.0 Hz, 1 H), 8.72 (d, *J* = 8.0 Hz, 1 H), 7.89 (d, *J* = 8.0 Hz, 1 H), 7.85 (d, *J* = 8.4 Hz, 1 H), 7.75-7.58 (m, 4 H), 7.56-7.43 (m, 5 H).

The following compound was prepared according to Typical Procedure IV.

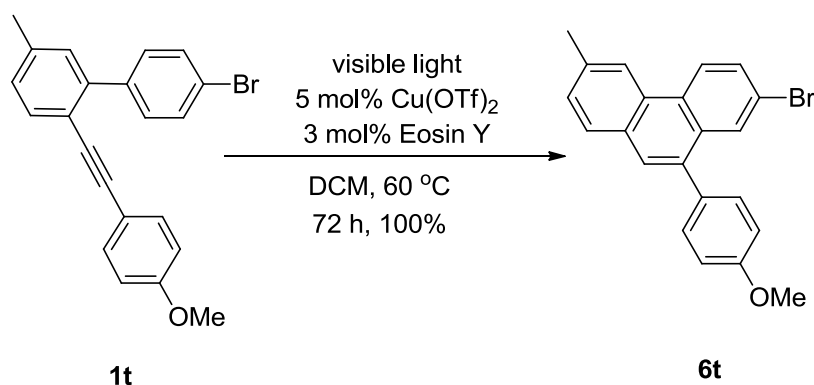
1) 2-Chloro-9-phenylphenanthrene (**6i**)



The reaction of **1i** (58 mg, 0.20 mmol), Cu(OTf)₂ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous DCM (5 mL) afforded **6i** as a liquid (58 mg, 100%). ¹H NMR (400 MHz, CDCl₃) δ 8.69 (d, *J* = 8.4 Hz, 1 H), 8.62 (d, *J* = 8.8 Hz, 1 H), 7.90 (d, *J* = 8.4 Hz, 1 H), 7.85 (s, 1 H), 7.67 (t, *J* = 8.0 Hz, 1 H), 7.63-7.42 (m, 8 H); ¹³C NMR (100 MHz, CDCl₃) δ 140.3, 140.1, 132.6, 131.0, 130.2, 129.9, 128.4, 128.3, 127.6,

127.5, 127.1, 127.0, 126.9, 126.8, 126.4, 124.2, 122.8; IR (neat) 1607, 1567, 1519, 1482, 1449, 1430, 1406 cm^{-1} ; HRMS (EI, 70 eV) calcd for $\text{C}_{21}\text{H}_{13}\text{Cl}$ 288.0706, found 288.0701.

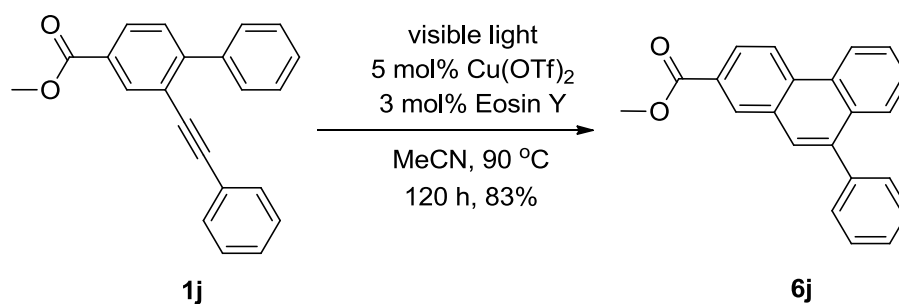
2) 2-Bromo-10-(4-methoxyphenyl)-6-methylphenanthrene (6t)



The reaction of **1t** (78 mg, 0.21 mmol), $\text{Cu}(\text{OTf})_2$ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous DCM (5 mL) afforded **6t**² as a solid (78 mg, 100%). ¹H NMR (400 MHz, CDCl_3) δ 8.59 (d, $J = 9.2$ Hz, 1 H), 8.42 (s, 1 H), 8.04 (s, 1 H), 7.76 (d, $J = 8.4$ Hz, 1 H), 7.70 (d, $J = 6.8$ Hz, 1 H), 7.63 (s, 1 H), 7.50-7.38 (m, 3 H), 7.05 (d, $J = 8.8$ Hz, 2 H), 3.91 (s, 3 H), 2.63 (s, 3 H).

Typical Procedure V for the photoreaction.

Synthesis of 2-methoxycarbonyl-9-phenyl-phenanthrene (6j)

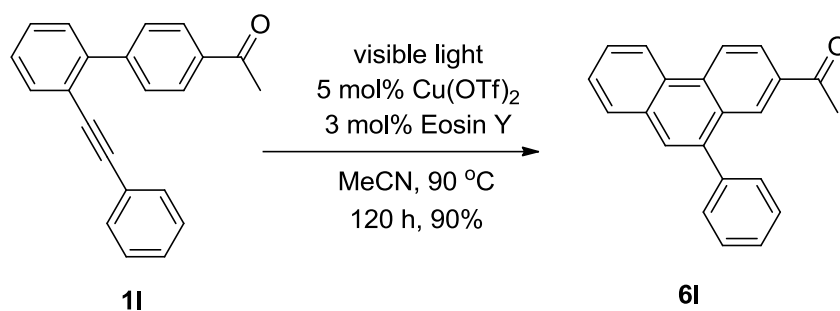


1j (63 mg, 0.20 mmol), $\text{Cu}(\text{OTf})_2$ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous MeCN (5 mL) were added subsequently into a 25 mL of dry pyrex sealed tube. The reaction mixture was degassed with three freeze-pump-thaw cycles. Then, the reaction mixture was irradiated by a 23 W household lamp at 90 °C under argon atmosphere. The photoreaction was completed after 72 h as monitored by TLC (eluent: petroleum ether:ethyl acetate = 50:1). The solvent was removed and the residue was purified by flash chromatography on silica gel (eluent: petroleum

ether:ethyl acetate = 50:1) to afford **6j** as a liquid (52 mg, 83%). ^1H NMR (400 MHz, CDCl_3) δ 8.76 (d, $J = 8.4$ Hz, 1 H), 8.72 (d, $J = 8.8$ Hz, 1 H), 8.59 (s, 1 H), 8.24 (d, $J = 8.8$ Hz, 1 H), 7.92 (d, $J = 8.0$ Hz, 1 H), 7.76-7.63 (m, 2 H), 7.62-7.41 (m, 6 H), 3.99 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3) δ 167.1, 140.3, 139.6, 132.9, 132.0, 130.9, 130.0, 129.9, 128.4, 128.1, 127.62, 127.55, 127.1, 126.8, 126.4, 123.5, 122.8, 52.2; IR (neat) 1723, 1619, 1592, 1571, 1522, 1479, 1433, 1412 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{22}\text{H}_{17}\text{O}_2$ ($\text{M} + \text{H}^+$) 313.1223, found 313.1222.

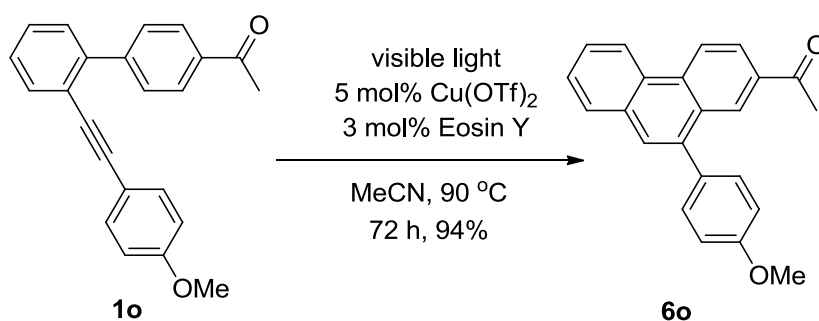
The following compound was prepared according to Typical Procedure V.

1) 10-Phenyl-2-acetylphenanthrene (**6l**)



The reaction of **1l** (59 mg, 0.20 mmol), $\text{Cu}(\text{OTf})_2$ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous MeCN (5 mL) afforded **6l** as a liquid (53 mg, 90%). ^1H NMR (400 MHz, CDCl_3) δ 8.83 (d, $J = 8.8$ Hz, 1 H), 8.74 (d, $J = 7.6$ Hz, 1 H), 8.55 (s, 1 H), 8.23 (d, $J = 8.8$ Hz, 1 H), 7.93 (d, $J = 7.2$ Hz, 1 H), 7.79-7.63 (m, 3 H), 7.58-7.44 (m, 5 H), 2.59 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3) δ 198.1, 140.0, 139.2, 134.8, 133.8, 132.5, 130.6, 130.0, 129.3, 128.8, 128.5, 128.4, 128.3, 128.0, 127.8, 127.0, 124.9, 123.4, 123.2, 26.6; IR (neat) 1680, 1598, 1571, 1525, 1494, 1446, 1409; HRMS (ESI) calcd for $\text{C}_{22}\text{H}_{17}\text{O}$ ($\text{M} + \text{H}^+$) 297.1274, found 297.1276.

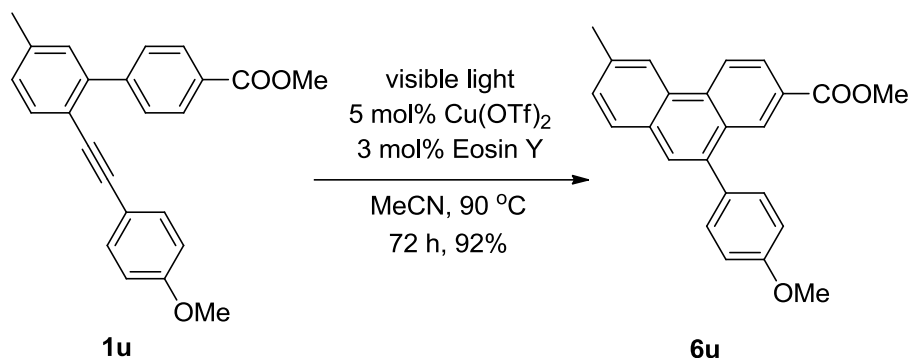
2) 10-(4-Methoxyphenyl)-2-acetylphenanthrene (**6o**)



The reaction of **1o** (65 mg, 0.20 mmol), $\text{Cu}(\text{OTf})_2$ (4 mg, 0.01 mmol), Eosin Y (4 mg,

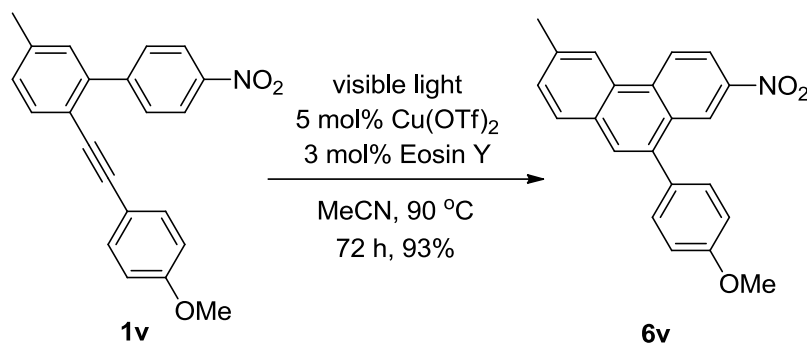
0.006 mmol), and anhydrous MeCN (5 mL) afforded **6o**² as a solid (61 mg, 94%). ¹H NMR (400 MHz, CDCl₃) δ 8.81 (d, *J* = 8.8 Hz, 1 H), 8.73 (d, *J* = 7.2 Hz, 1 H), 8.58 (s, 1 H), 8.22 (d, *J* = 10.0 Hz, 1 H), 7.91 (d, *J* = 8.8 Hz, 1 H), 7.77-7.61 (m, 3 H), 7.48 (d, *J* = 8.4 Hz, 2 H), 7.09 (d, *J* = 8.8 Hz, 2 H), 3.93 (s, 3 H), 2.61 (s, 3 H).

3) 10-(4-Methoxyphenyl)-6-methyl-2-methoxycarbonylphenanthrene (**6u**)



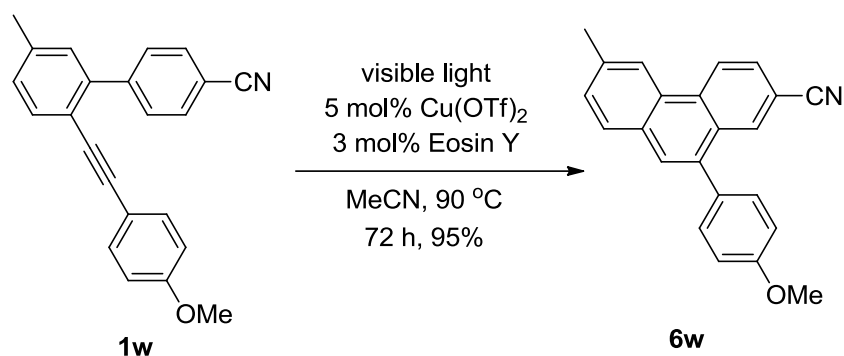
The reaction of **1u** (71 mg, 0.20 mmol), Cu(OTf)₂ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous MeCN (5 mL) afforded **6u**² as a solid (65 mg, 92%). ¹H NMR (400 MHz, CDCl₃) δ 8.79 (d, *J* = 8.8 Hz, 1 H), 8.66 (s, 1 H), 8.51 (s, 1 H), 8.24 (d, *J* = 8.0 Hz, 1 H), 7.79 (d, *J* = 8.0 Hz, 1 H), 7.68 (s, 1 H), 7.52-7.43 (m, 3 H), 7.07 (d, *J* = 8.0 Hz, 2 H), 3.92 (s, 6 H), 2.65 (s, 3 H).

4) 10-(4-Methoxyphenyl)-6-methyl-2-nitrophenanthrene (**6v**)



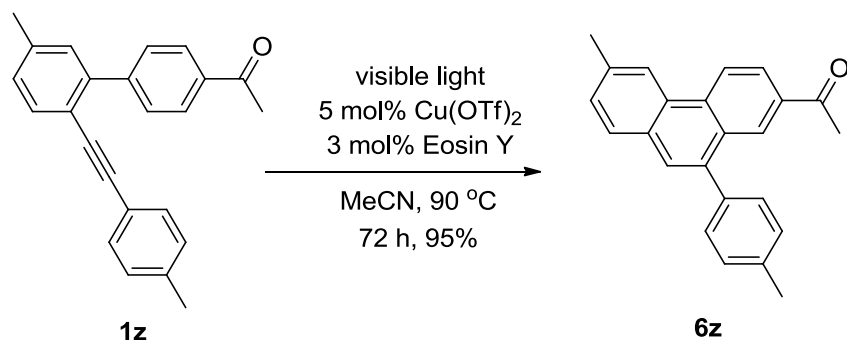
The reaction of **1v** (69 mg, 0.20 mmol), Cu(OTf)₂ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous MeCN (5 mL) afforded **6v**² as a solid (64 mg, 93%). ¹H NMR (400 MHz, CDCl₃) δ 8.90-8.82 (m, 2 H), 8.51 (s, 1 H), 8.41 (dd, *J* = 9.2, 2.4 Hz, 1 H), 7.84 (d, *J* = 8.4 Hz, 1 H), 7.77 (s, 1 H), 7.56 (d, *J* = 8.0 Hz, 1 H), 7.46 (d, *J* = 8.8 Hz, 2 H), 7.09 (d, *J* = 8.8 Hz, 2 H), 3.94 (s, 3 H), 2.67 (s, 3 H).

5) 10-(4-Methoxyphenyl)-6-methyl-2-cyanophenanthrene (**6w**)



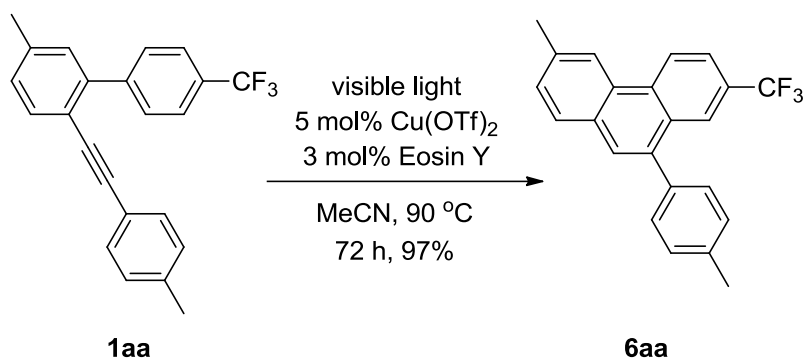
The reaction of **1w** (65 mg, 0.20 mmol), Cu(OTf)₂ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous MeCN (5 mL) afforded **6w**² as a solid (62 mg, 95%). ¹H NMR (400 MHz, CDCl₃) δ 8.81 (d, *J* = 8.4 Hz, 1 H), 8.48 (s, 1 H), 8.27 (s, 1 H), 7.88-7.75 (m, 2 H), 7.73 (s, 1 H), 7.54 (d, *J* = 7.2 Hz, 1 H), 7.42 (d, *J* = 8.8 Hz, 2 H), 7.08 (d, *J* = 8.4 Hz, 2 H), 3.93 (s, 3 H), 2.66 (s, 3 H).

6) 10-(4-Methylphenyl)-6-methyl-2-acetylphenanthrene (6z)



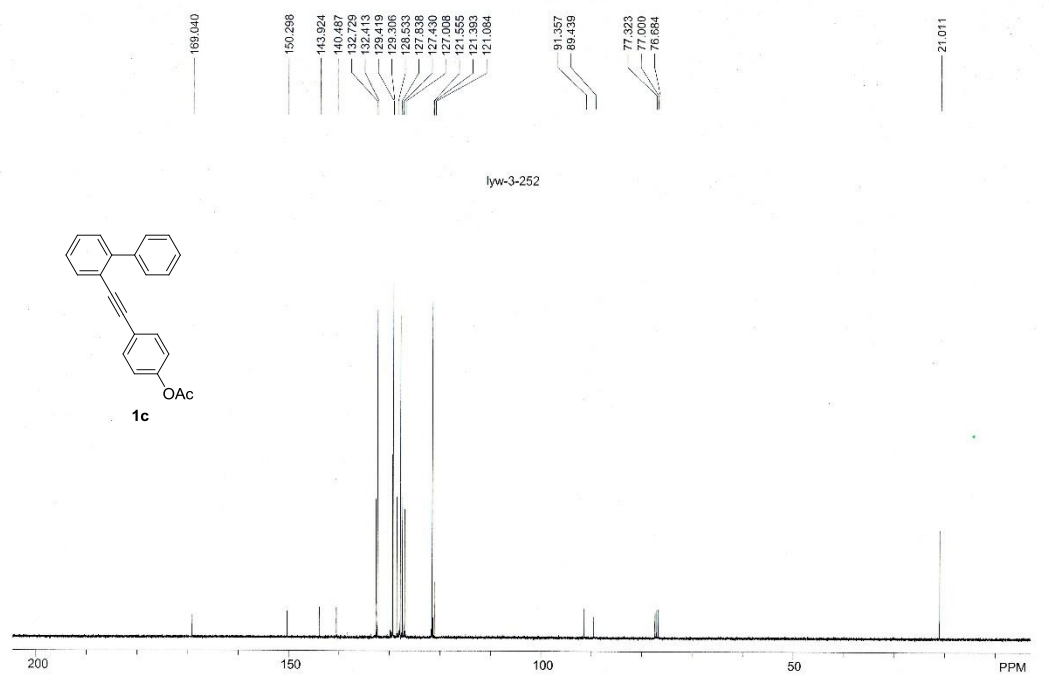
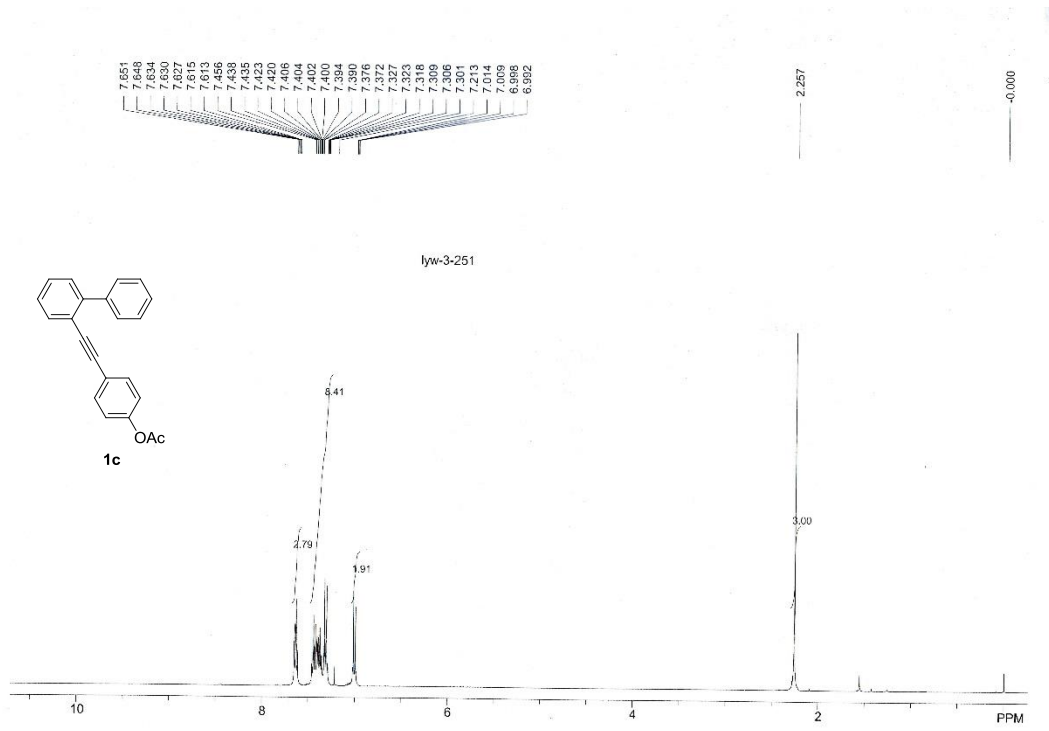
The reaction of **1z** (65 mg, 0.20 mmol), Cu(OTf)₂ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous MeCN (5 mL) afforded **6z** as a solid (62 mg, 95%); mp 133.6-134.5 °C (ethyl acetate/petroleum ether). ¹H NMR (400 MHz, CDCl₃) δ 8.80 (d, *J* = 8.8 Hz, 1 H), 8.56 (s, 1 H), 8.51 (s, 1 H), 8.19 (d, *J* = 8.8 Hz, 1 H), 7.80 (d, *J* = 8.0 Hz, 1 H), 7.70 (s, 1 H), 7.50 (d, *J* = 8.0 Hz, 1 H), 7.44 (d, *J* = 8.0 Hz, 2 H), 7.35 (d, *J* = 7.6 Hz, 2 H), 2.65 (s, 3 H), 2.60 (s, 3 H), 2.49 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 198.2, 138.2, 137.3, 137.2, 136.7, 134.7, 133.5, 130.8, 130.6, 129.9, 129.8, 129.3, 129.2, 128.6, 128.4, 128.2, 124.6, 123.3, 122.9, 26.7, 22.2, 21.3; IR (neat) 1680, 1607, 1592, 1571, 1522, 1503, 1494, 1446, 1421; HRMS (ESI) calcd for C₂₄H₂₁O (M + H⁺) 325.1587, found 325.1593.

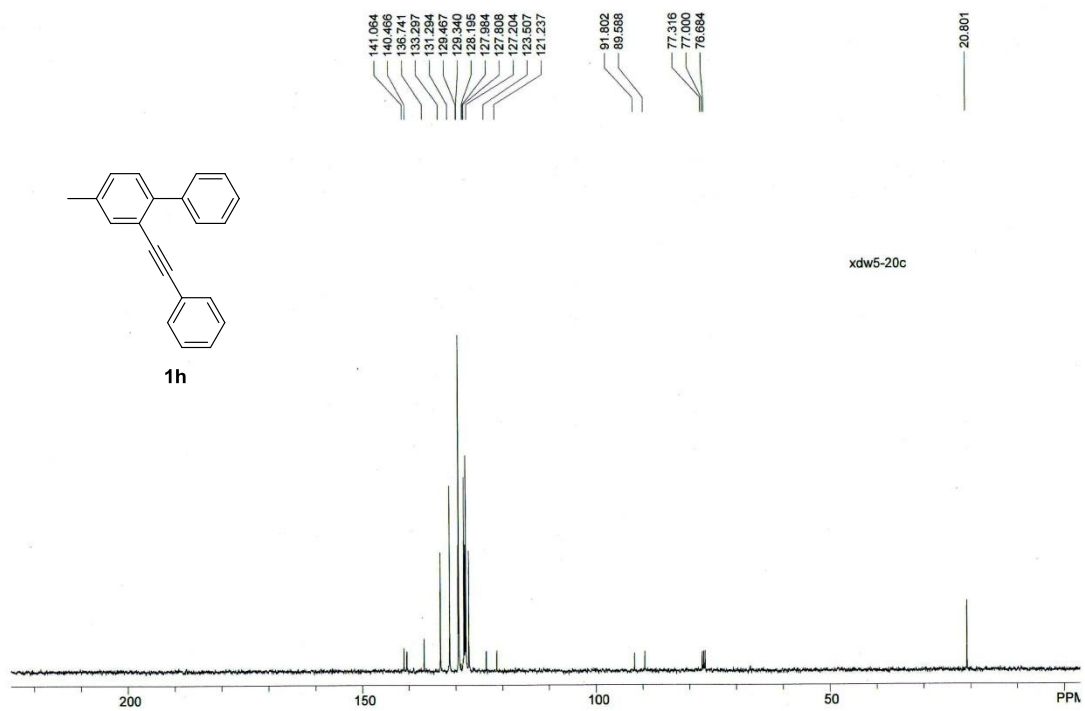
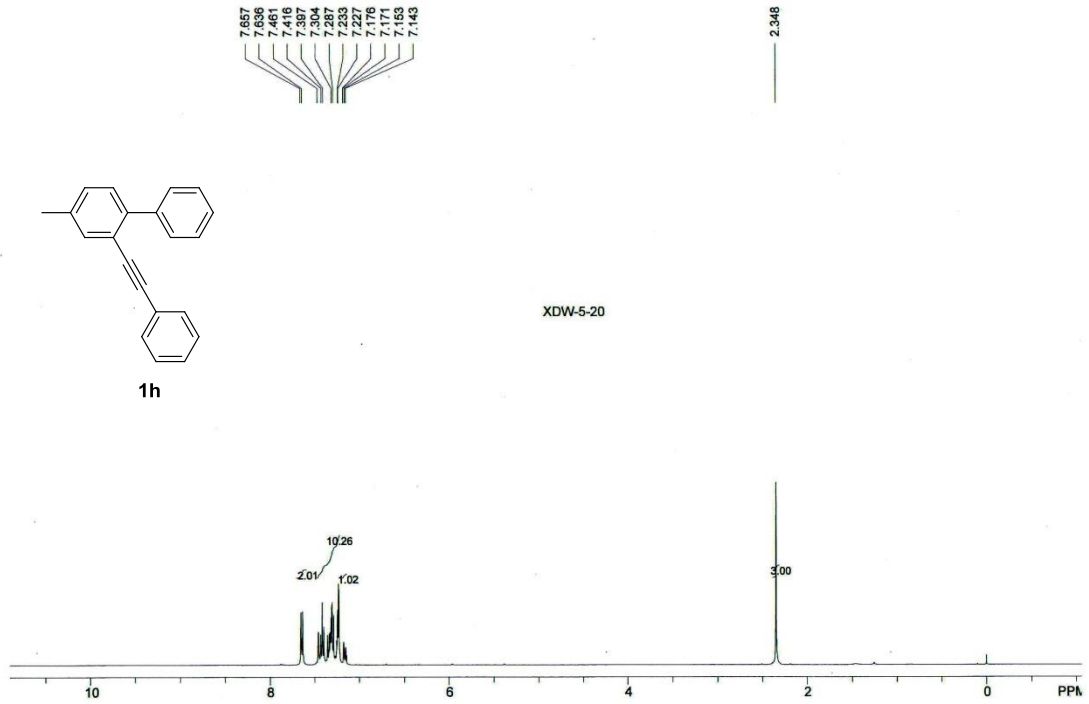
7) 10-(4-Methylphenyl)-6-methyl-2-trifluoromethylphenanthrene (6aa)

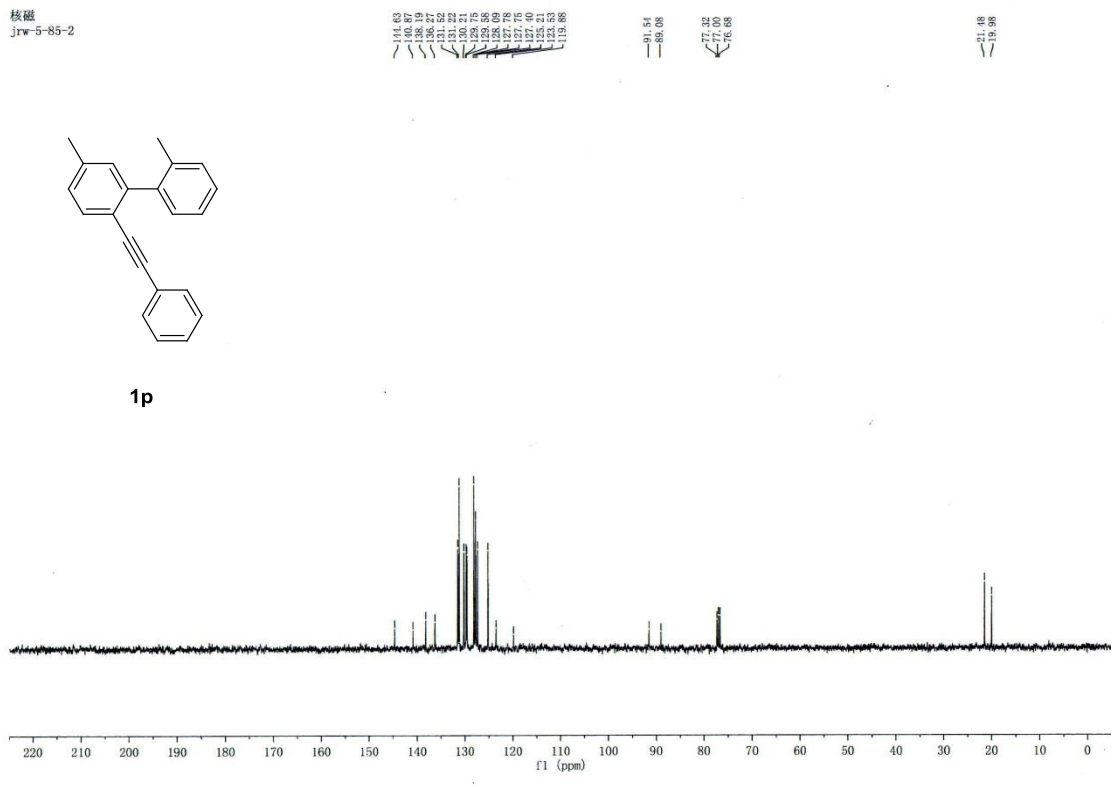
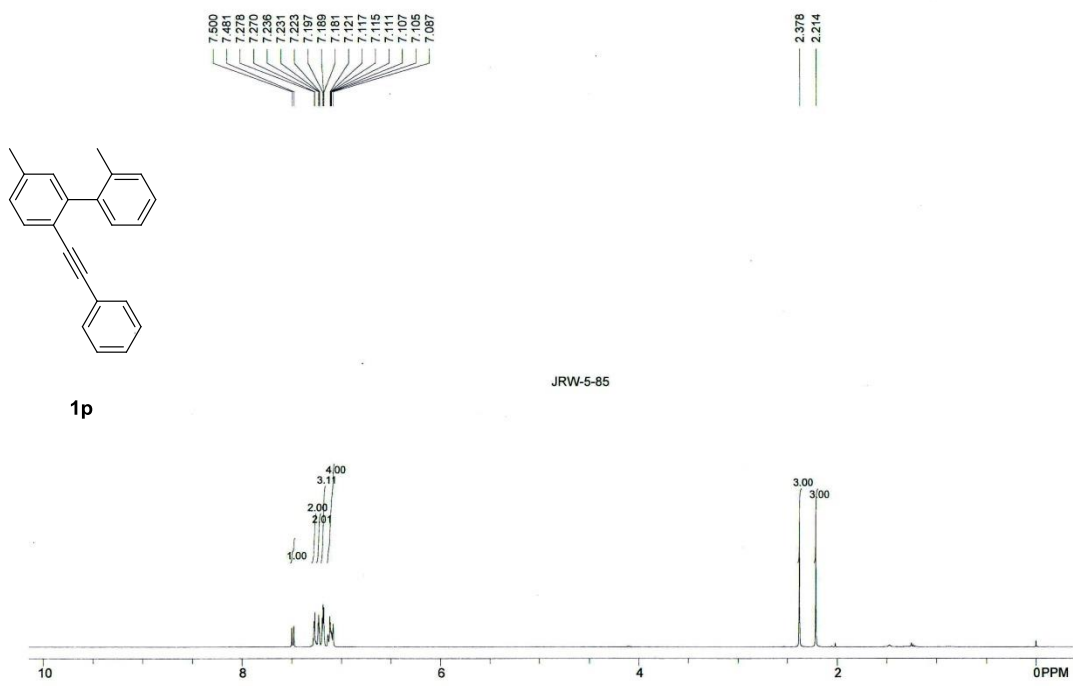


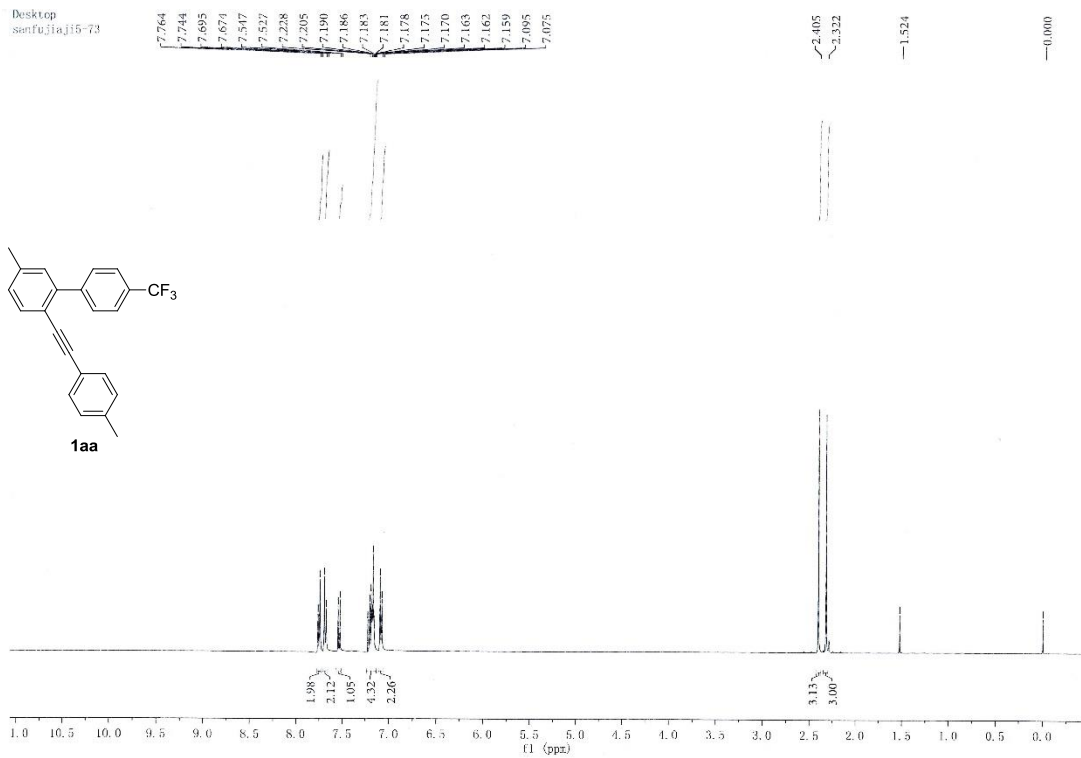
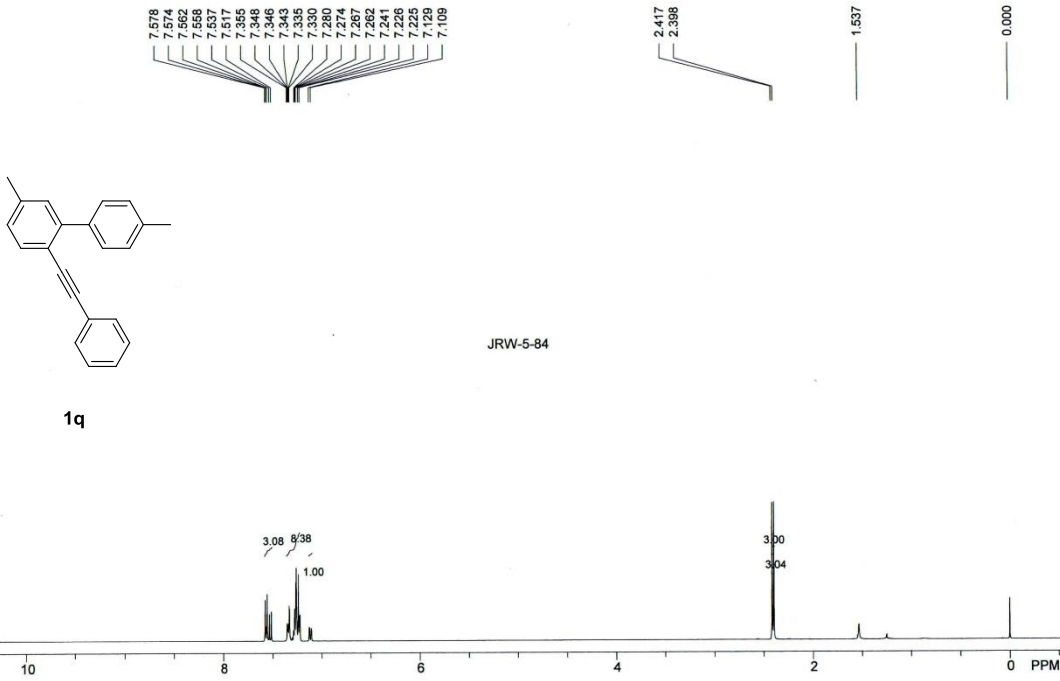
The reaction of **1aa** (70 mg, 0.20 mmol), Cu(OTf)₂ (4 mg, 0.01 mmol), Eosin Y (4 mg, 0.006 mmol), and anhydrous MeCN (5 mL) afforded **6aa** as a solid (68 mg, 97%); mp 100.6-101.3 °C (ethyl acetate/petroleum ether). ¹H NMR (400 MHz, CDCl₃) δ 8.83 (d, *J* = 8.8 Hz, 1 H), 8.49 (s, 1 H), 8.23 (s, 1 H), 7.84-7.76 (m, 2 H), 7.71 (s, 1 H), 7.49 (dd, *J* = 8.0, 0.8 Hz, 1 H), 7.41 (d, *J* = 7.6 Hz, 2 H), 7.34 (d, *J* = 8.0 Hz, 2 H), 2.64 (s, 3 H), 2.48 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 137.7, 137.4, 137.0, 136.8, 132.4, 130.8, 130.2, 129.8, 129.6, 129.3, 129.2, 128.6, 128.2, 127.9, 124.2 (q, *J* = 4.2 Hz), 123.7, 122.6, 122.0 (q, *J* = 3.3 Hz), 22.2, 21.3; ¹⁹F NMR (376 MHz, CDCl₃) δ -61.8; IR (neat) 1625, 1607, 1558, 1503, 1497, 1458, 1452, 1436, 1418; HRMS (EI, 70 eV) calcd for C₂₃H₁₇F₃ 350.1282, found 350.1280.

NMR Spectra







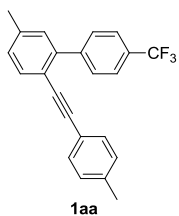
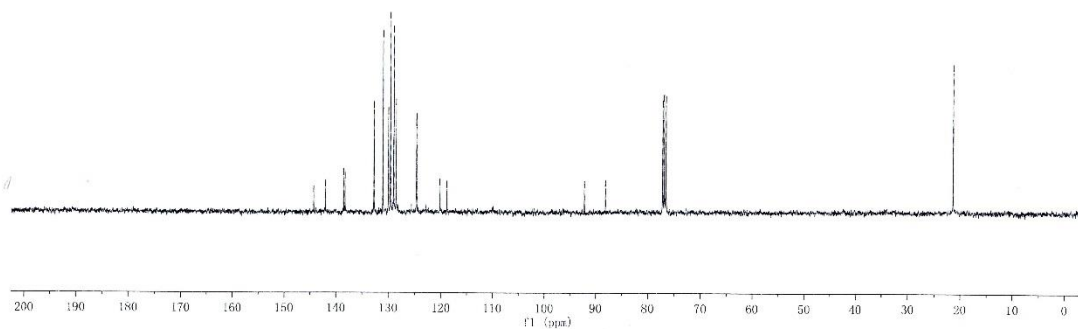
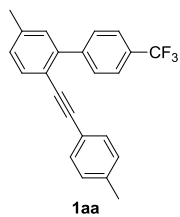


Desktop
sanjujiaji5 73 C

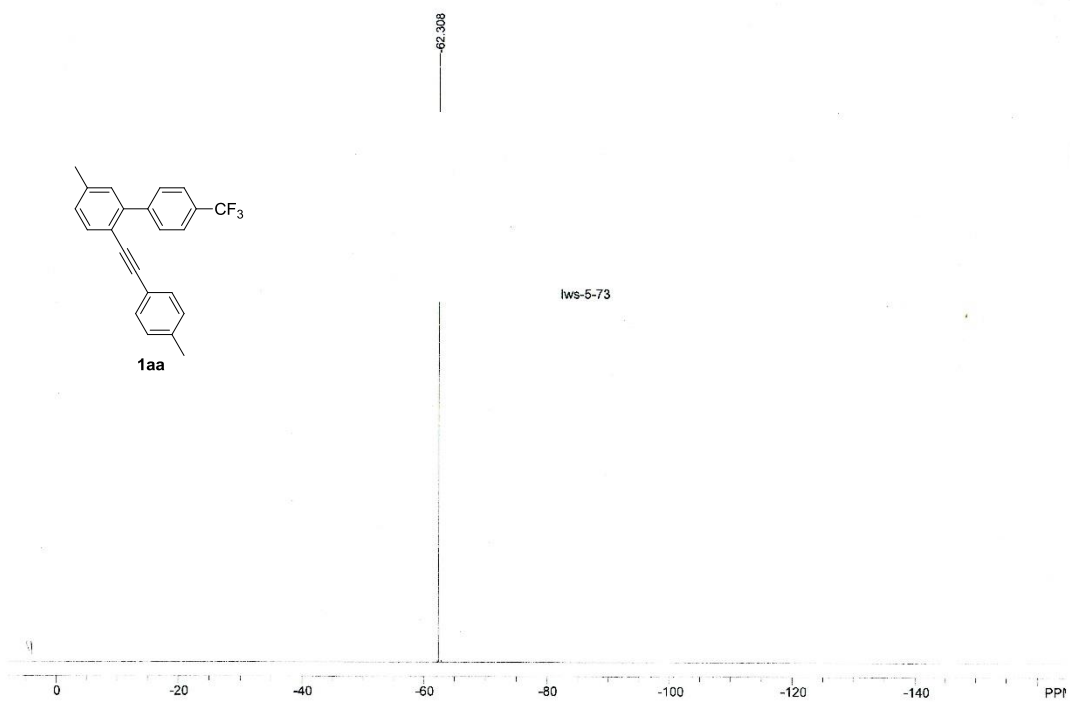
144.334
142.067
138.630
138.338
132.829
131.112
130.073
129.694
129.078
128.629
124.756
124.722
120.179
118.872

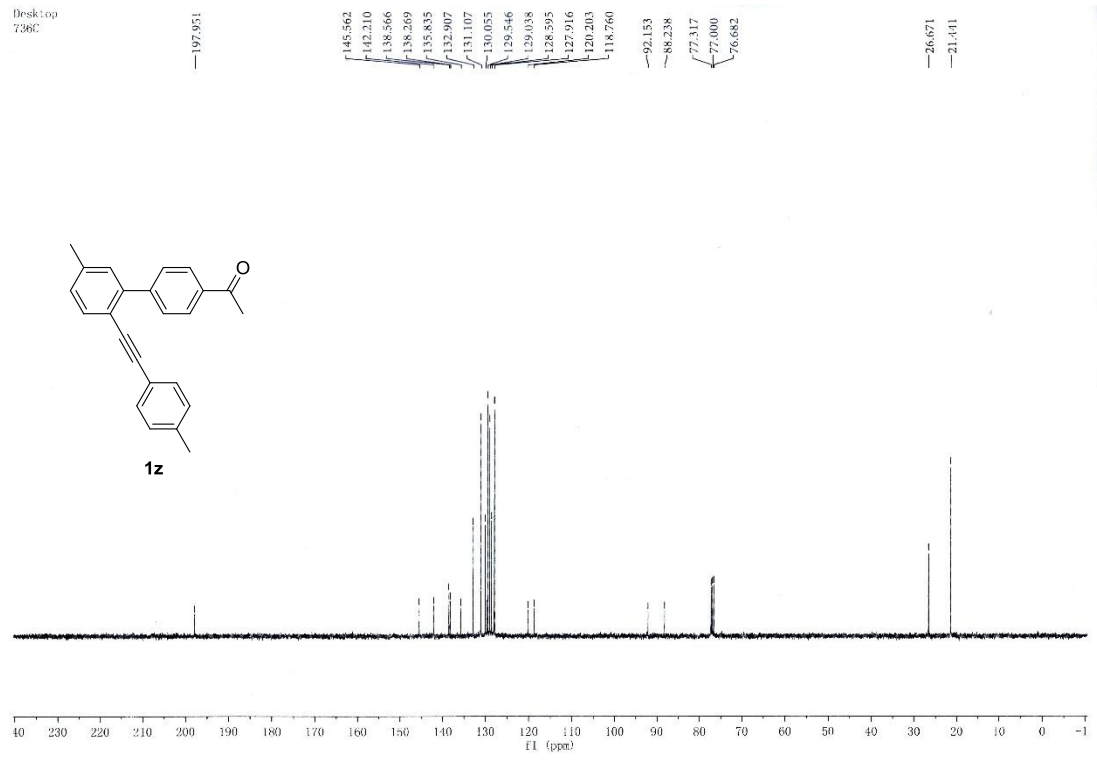
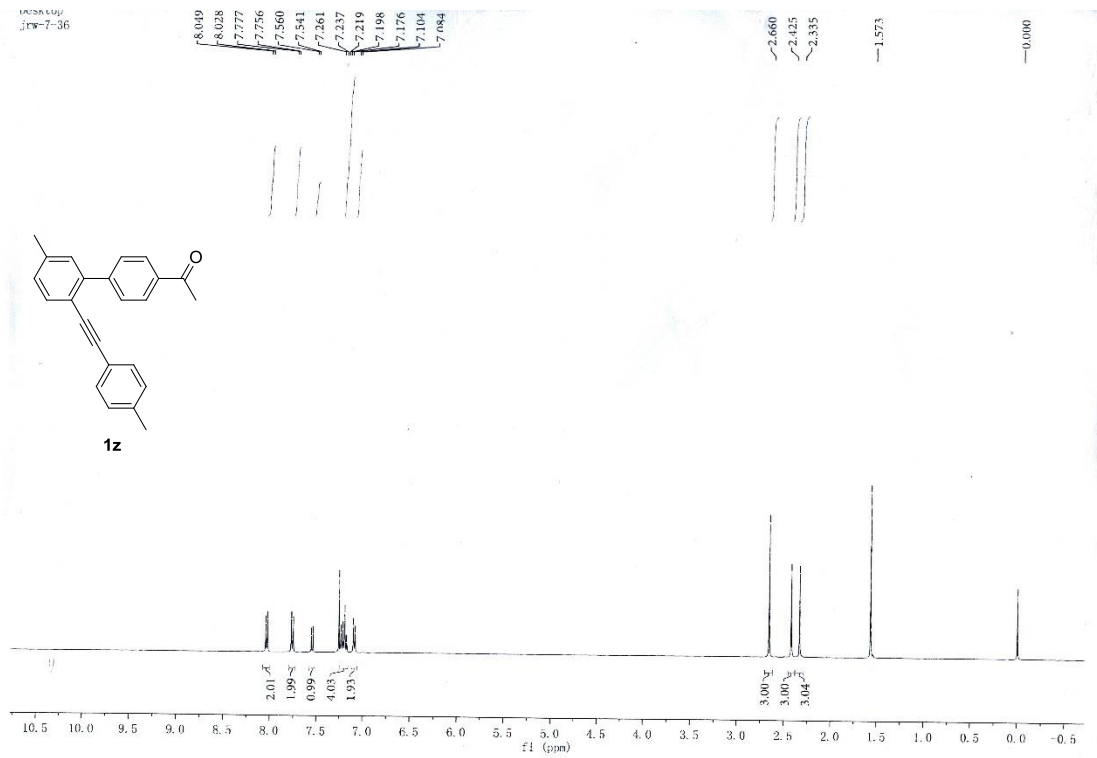
92.263
88.168
77.317
77.000
76.681

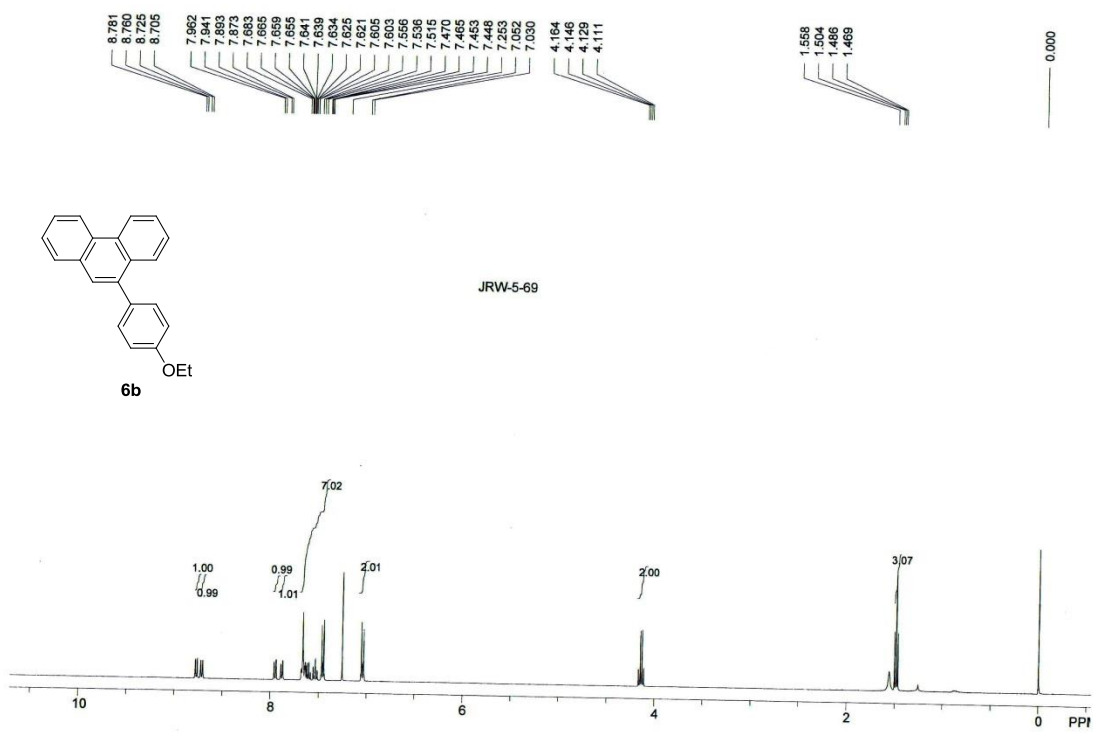
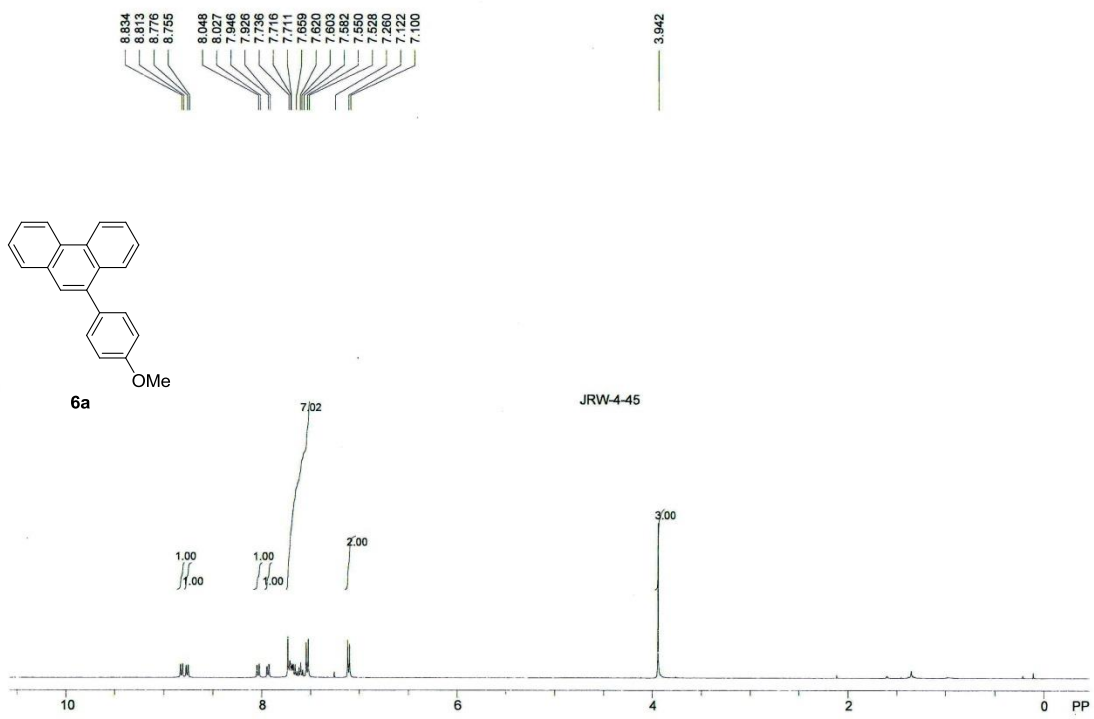
21.444

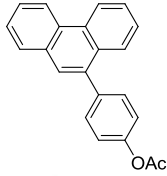
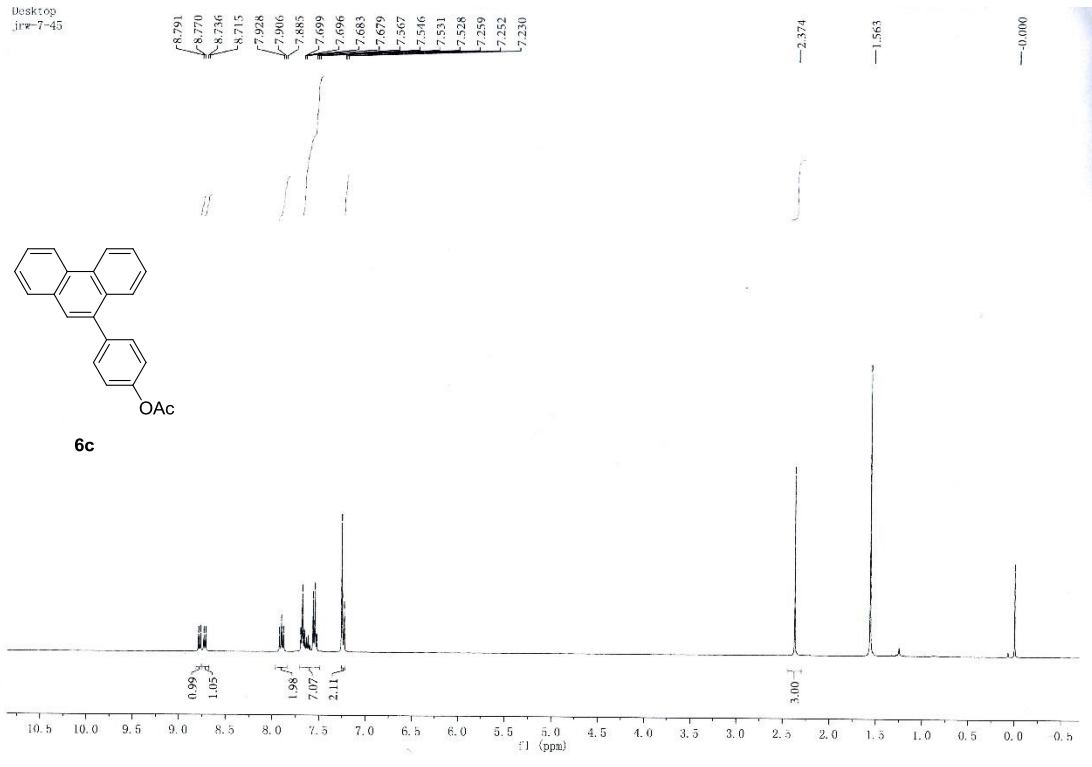


lws-5-73

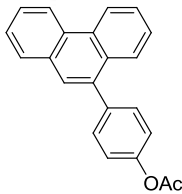
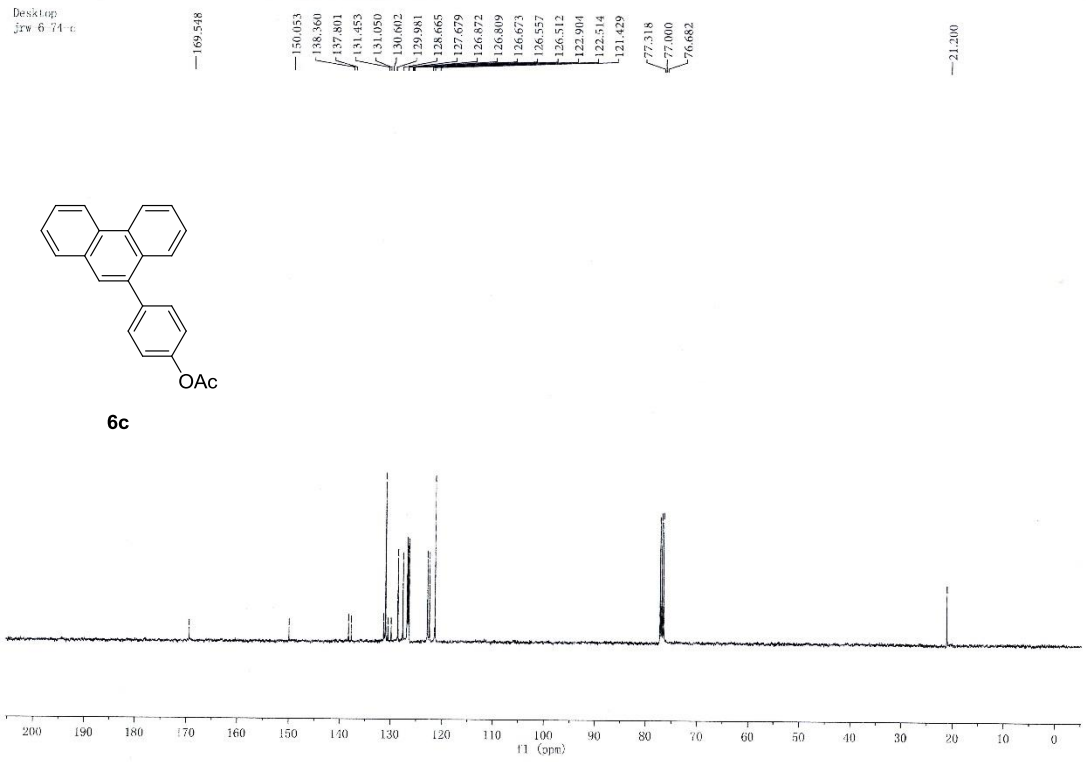




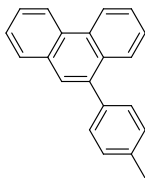
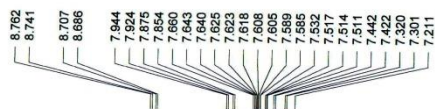




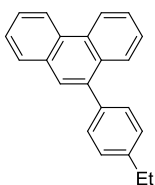
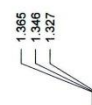
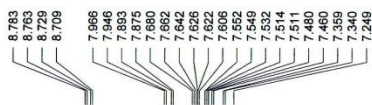
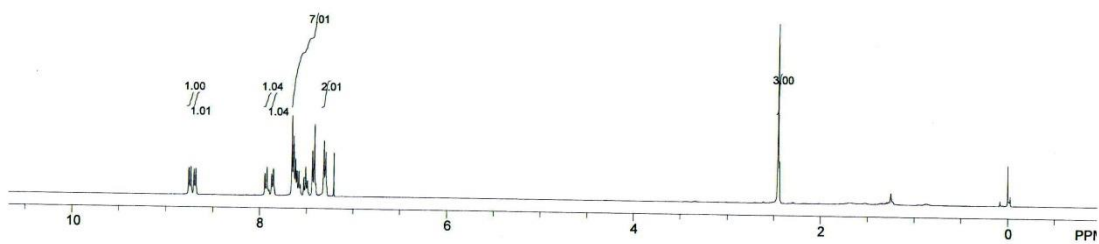
6c



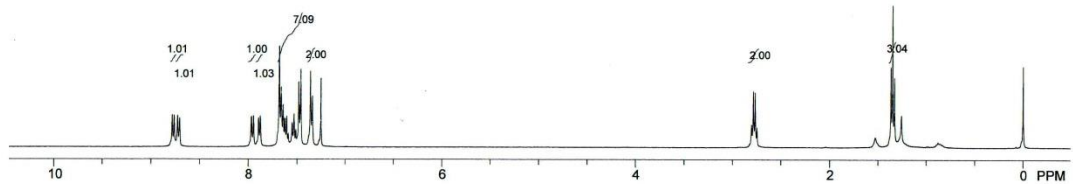
6c



JRW-5-21



JRW-5-47



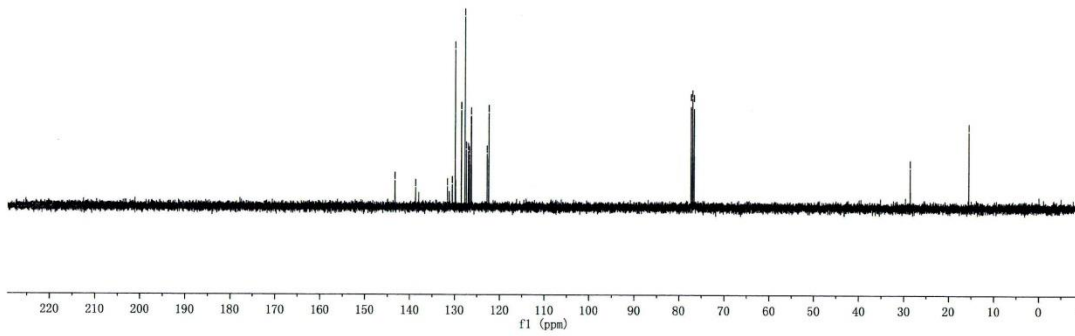
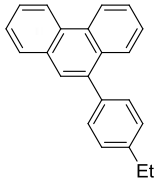
JRW-5-47-c

143.37
138.77
137.99
131.61
131.22
129.07
128.86
128.78
127.79
127.43
126.79
126.44
126.11
125.36
122.84
122.19

77.32
77.00
76.68

28.67

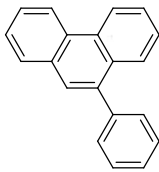
15.61



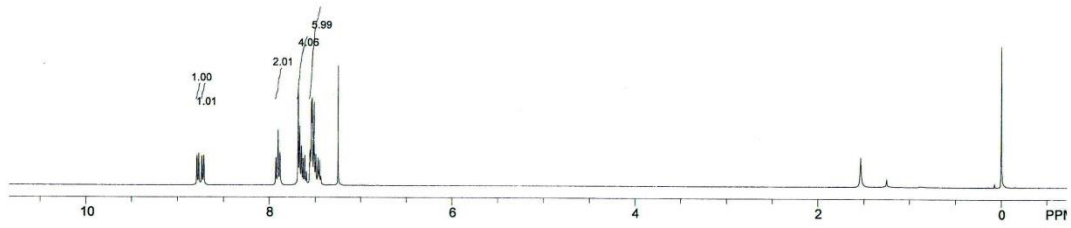
8.769
8.769
8.736
8.716
7.925
7.904
7.885
7.687
7.673
7.669
7.653
7.634
7.631
7.554
7.552
7.514
7.472
7.452
7.457

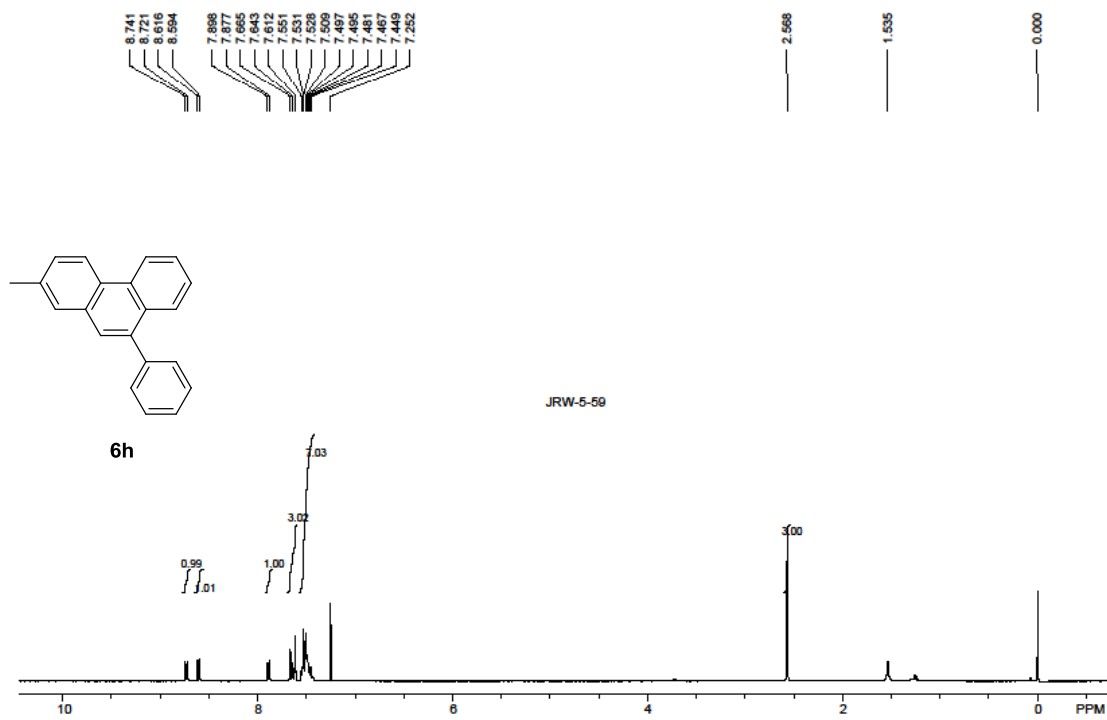
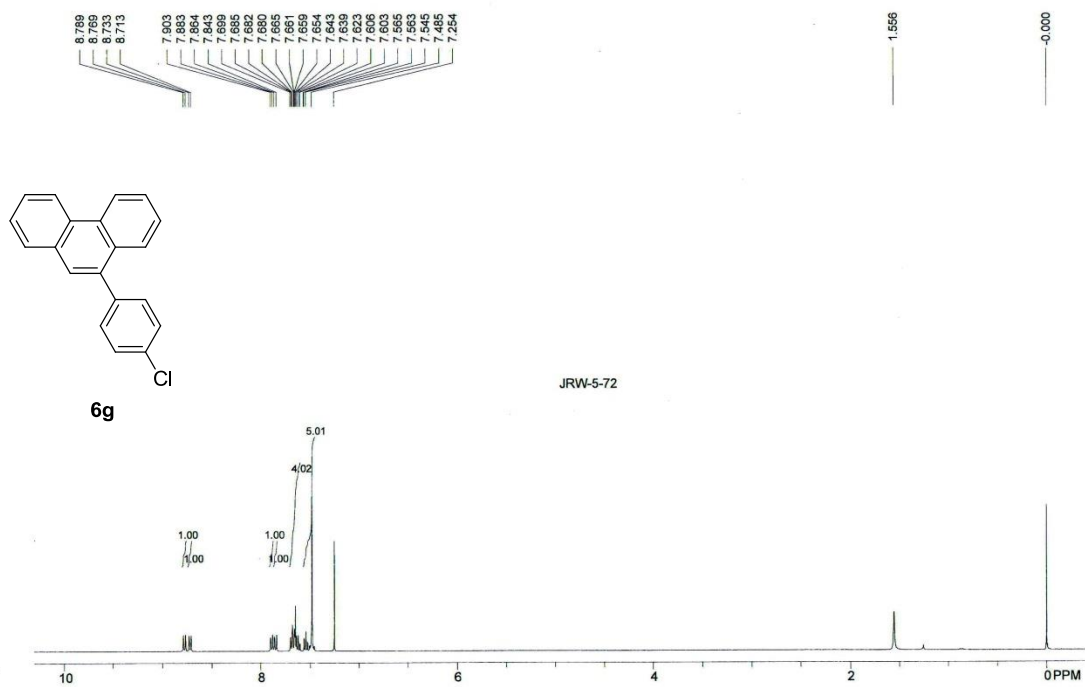
1.538

-0.000



JRW-5-23



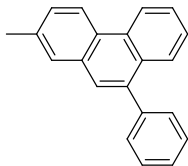


JRW-5-59-c

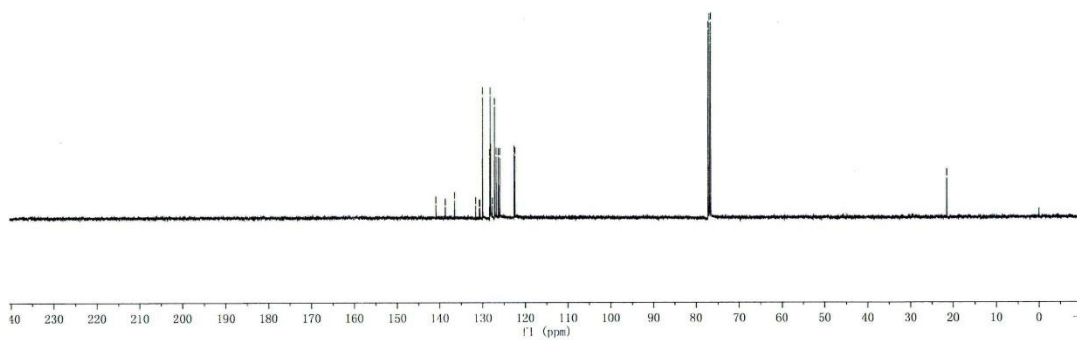
140.92
138.71
136.60
135.62
130.76
130.61
128.38
128.29
127.81
127.28
126.35
125.03
122.13

77.02
76.98

21.48



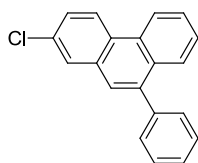
6h



8.703
8.682
8.634
8.612
7.911
7.890
7.847
7.889
7.671
7.650
7.603
7.578
7.557
7.520
7.537
7.523
7.513
7.475
7.464
7.246

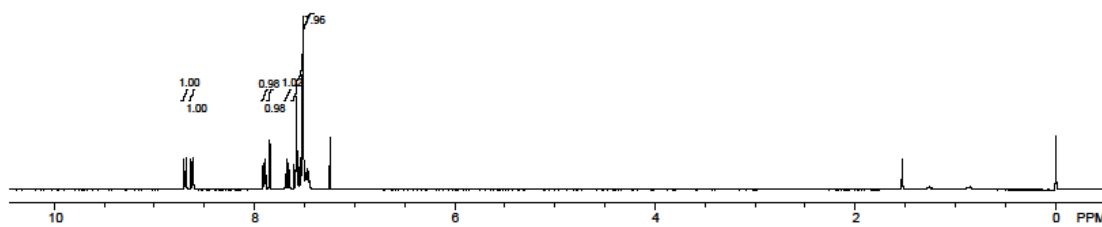
1.534

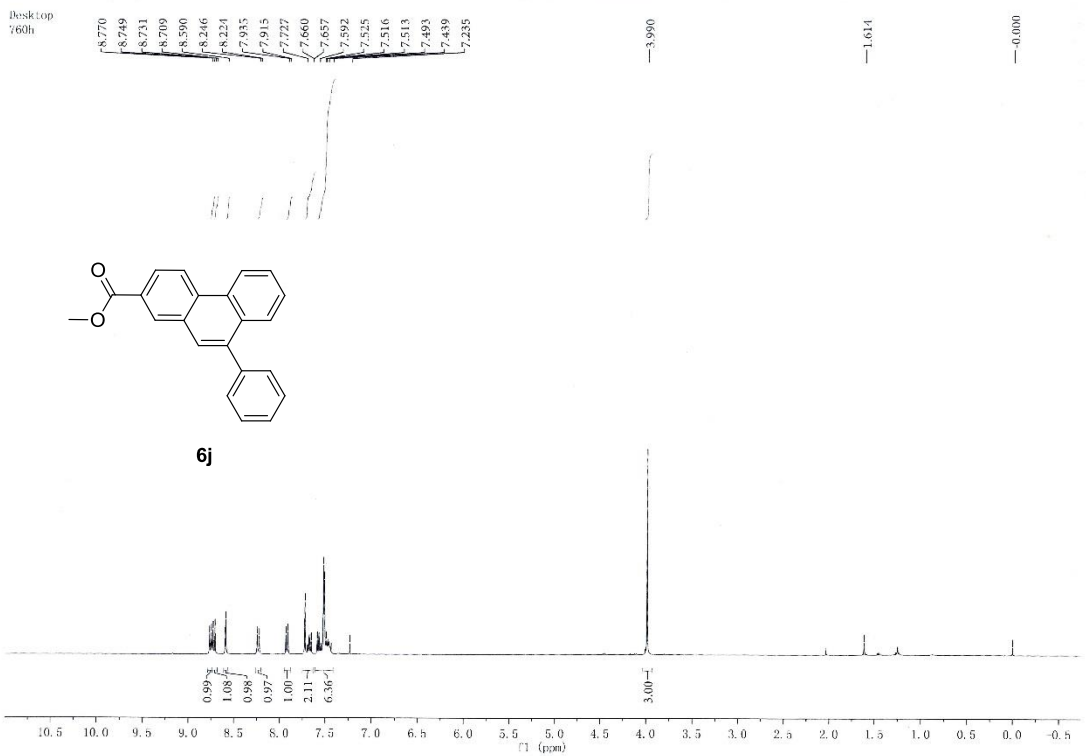
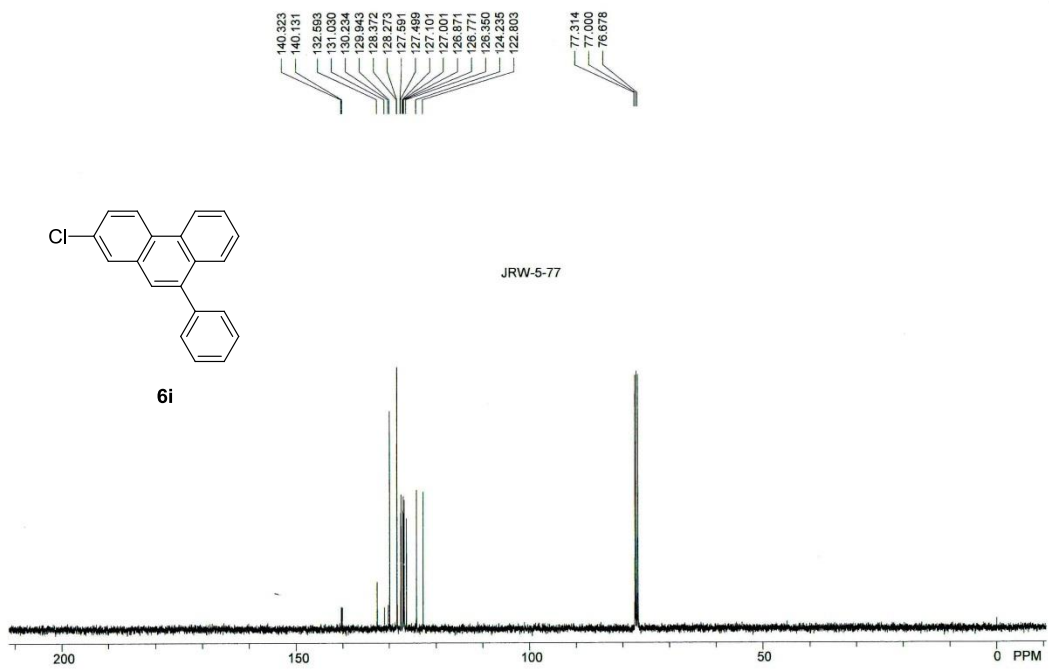
0.000



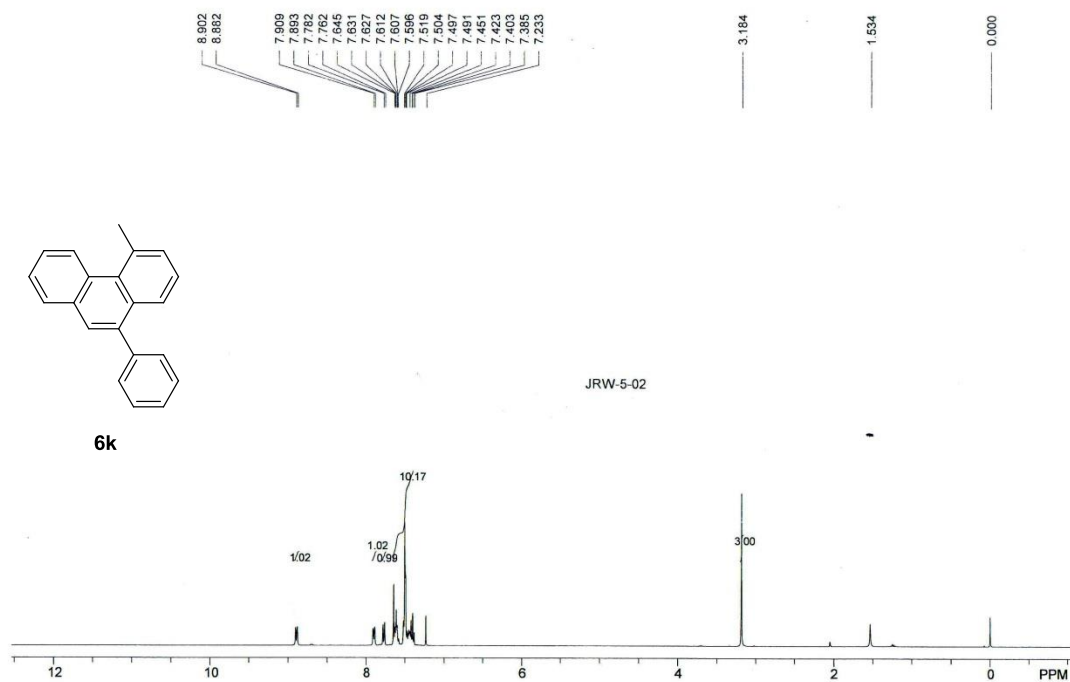
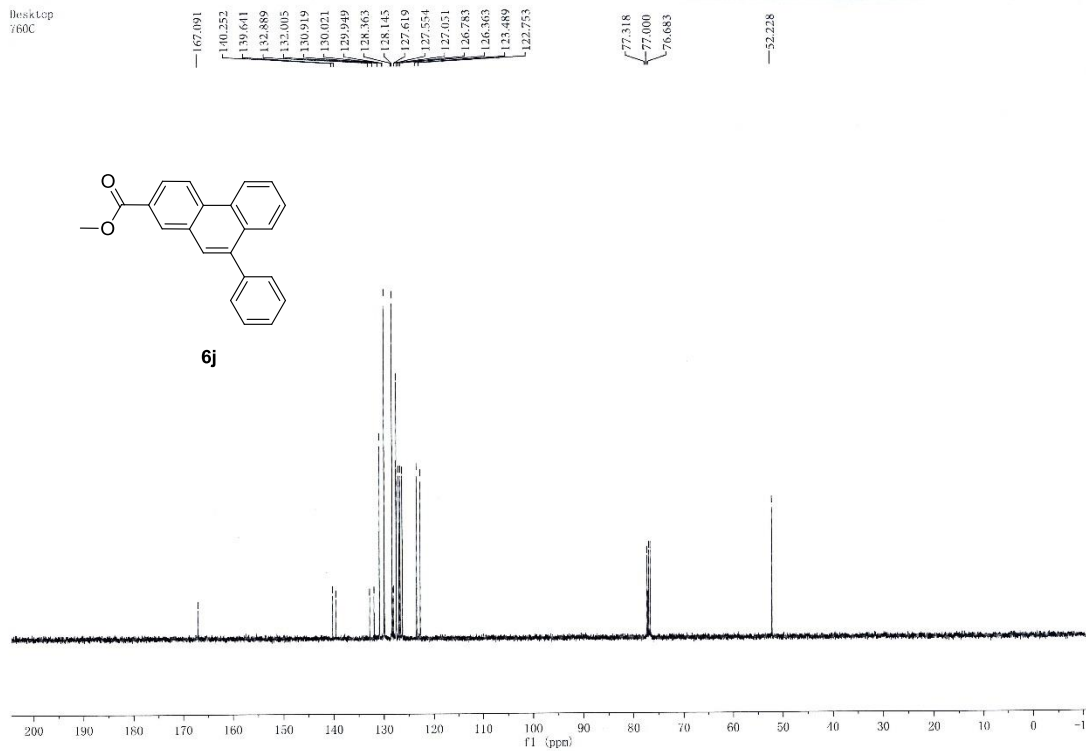
6i

JRW-5-77





BeskiTop
760C



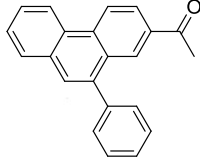
Desktop
765h

8.838
8.816
8.752
8.733
8.549
8.239
8.217
7.936
7.918
7.759
7.698
7.680
7.666
7.563
7.554
7.552
7.484
7.473
7.255

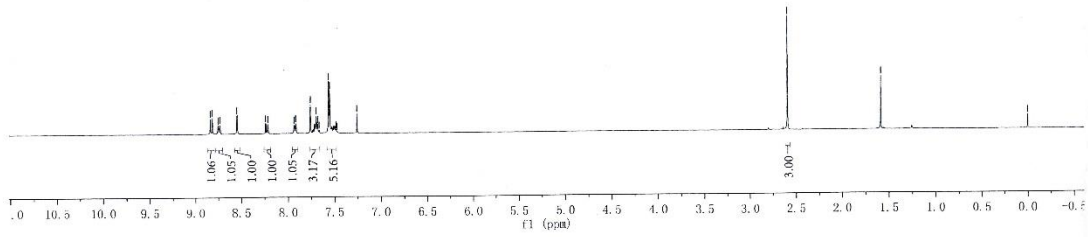
2.593

1.584

0.000



61



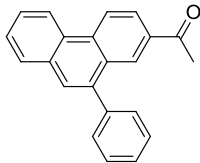
Desktop
765h

198.136

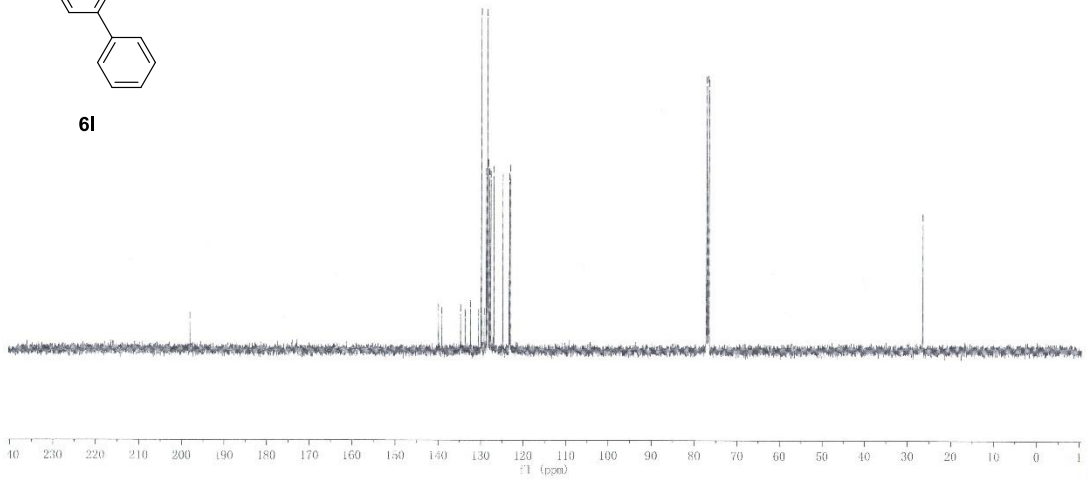
140.017
139.208
134.845
133.783
132.515
130.595
129.994
129.306
128.796
128.535
128.428
128.329
128.041
127.768
127.008
124.927
123.615
123.203

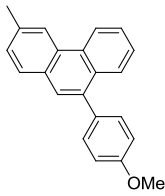
77.317
77.000
76.682

26.632



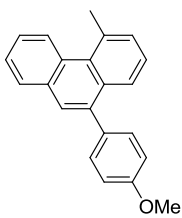
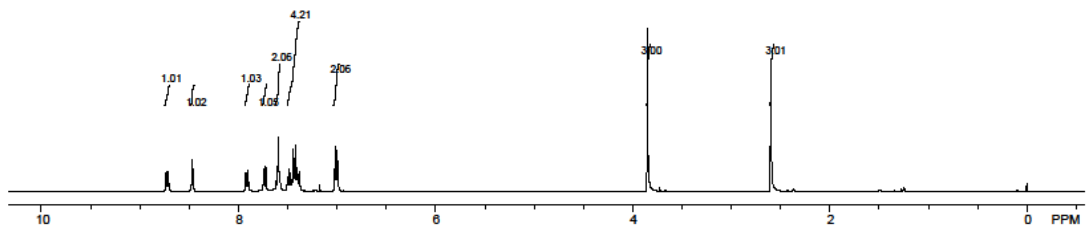
61





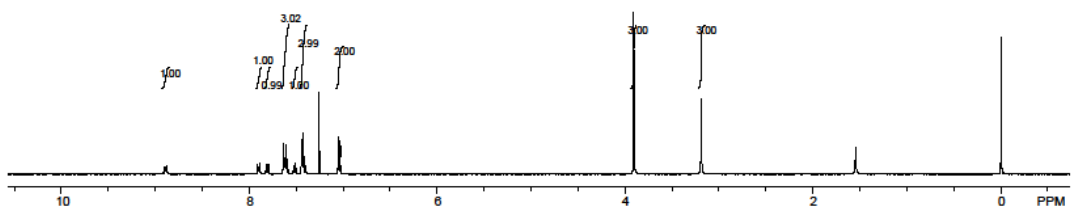
6m

JRW-5-49

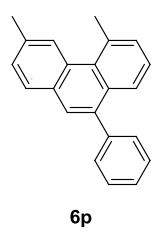
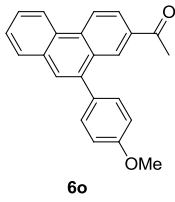
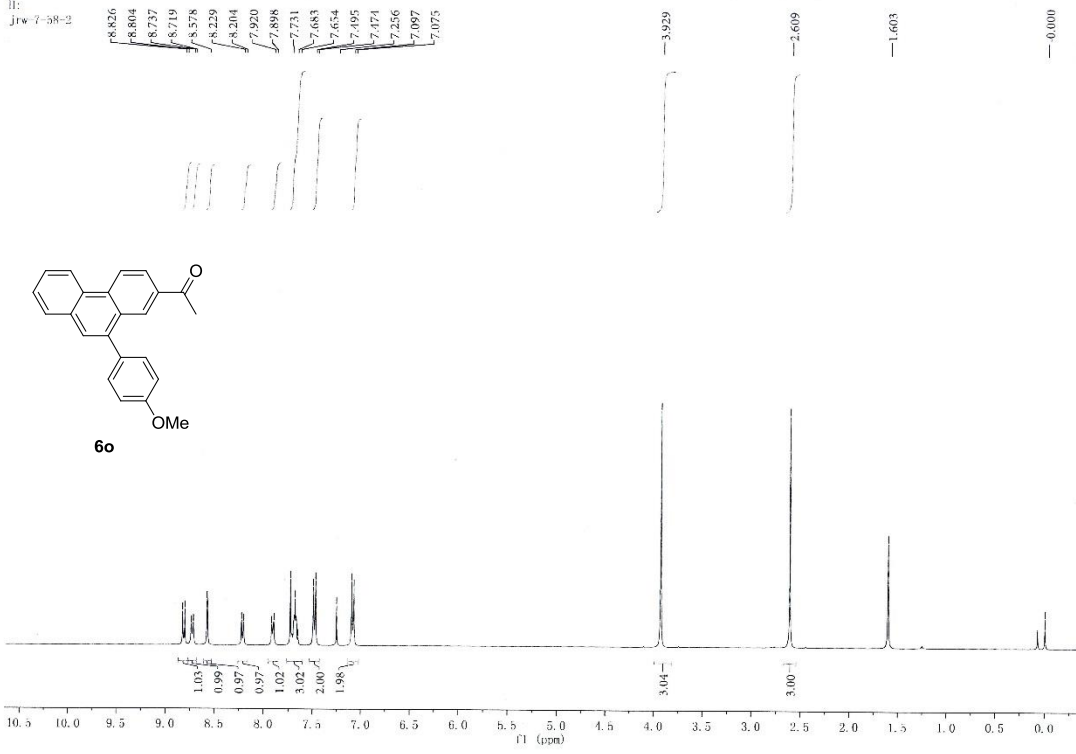


6n

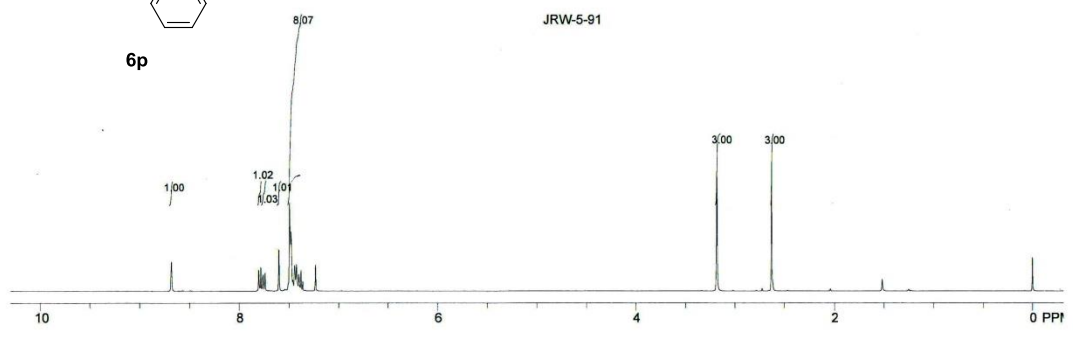
JRW-5-18

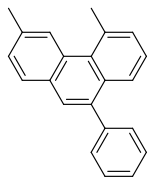


H:
jr w 7-58-2



JRW-5-31





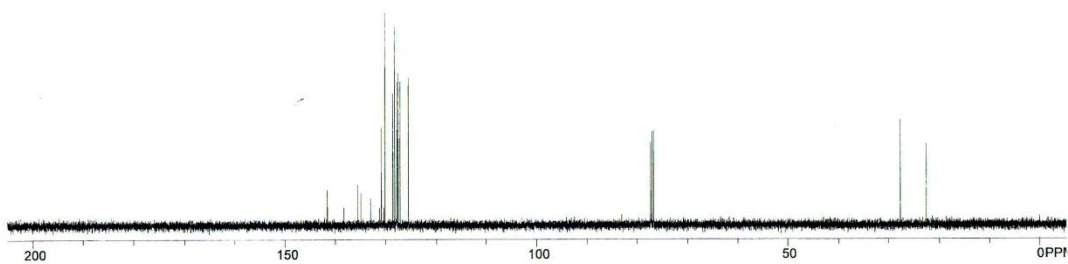
6p

141.603
138.278
135.555
134.967
132.977
131.675
131.275
130.753
130.366
130.119
128.544
128.194
127.727
127.576
127.122
125.496
125.481

77.314
77.000
76.679

27.600
22.474

JRW-5-91



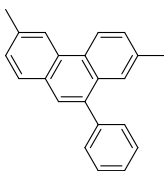
8.651
8.630
8.458
7.762
7.742
7.693
7.690
7.543
7.527
7.523
7.504
7.500
7.485
7.478
7.474
7.469
7.464
7.442
7.412
7.409
7.392
7.389
7.235

2.624
2.455

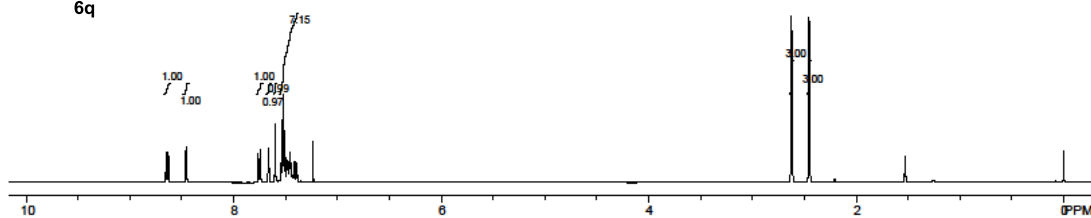
1.529

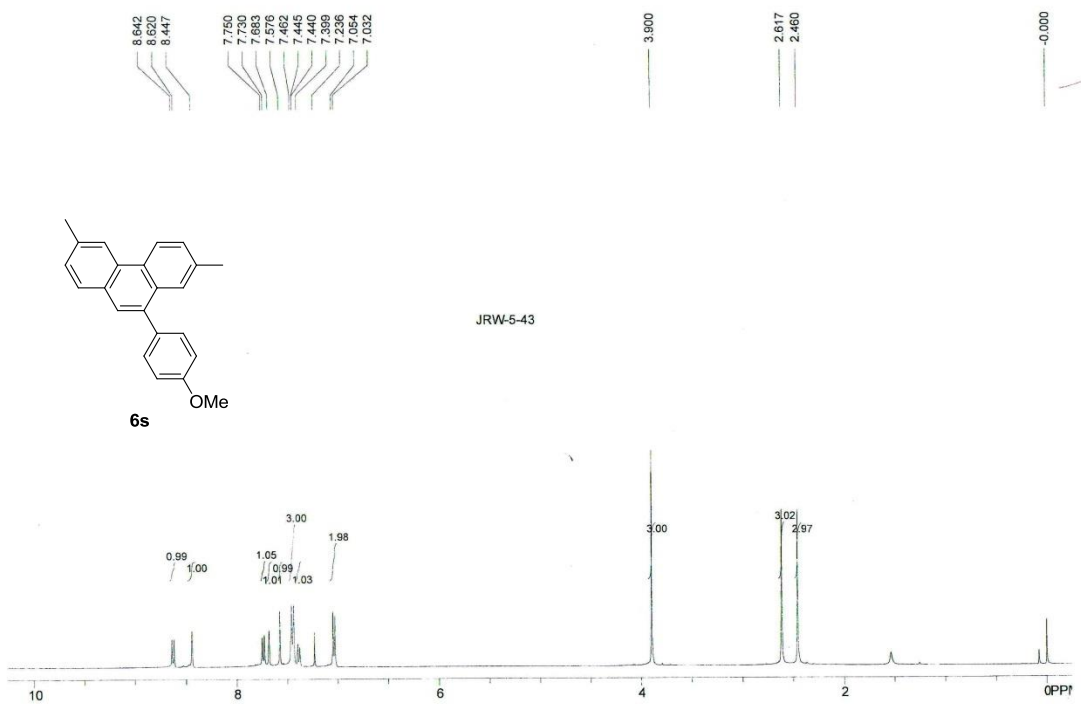
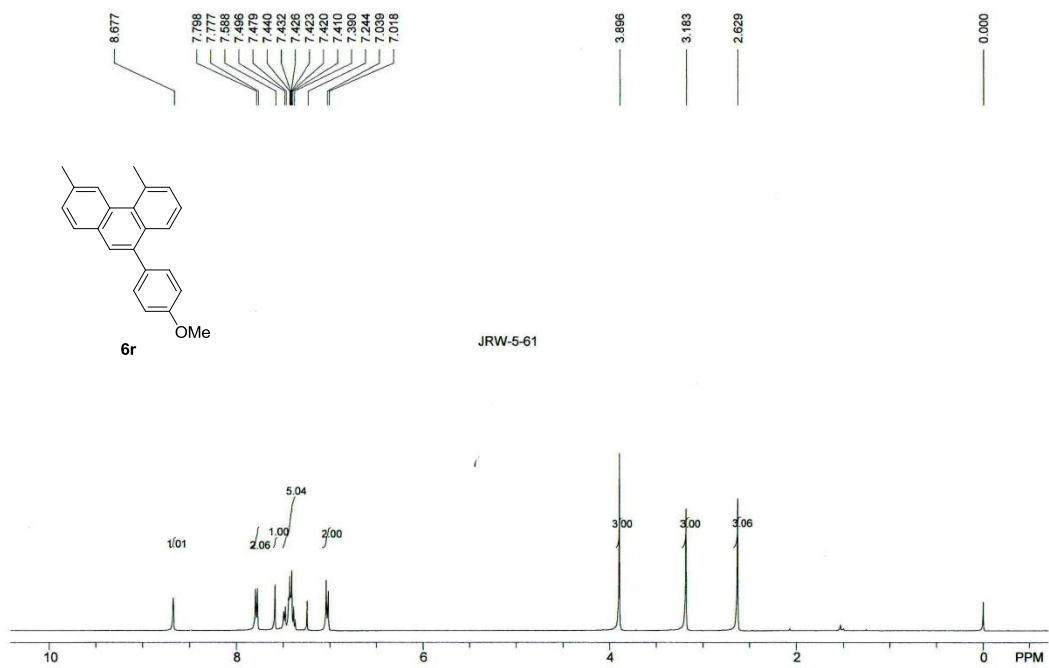
0.000

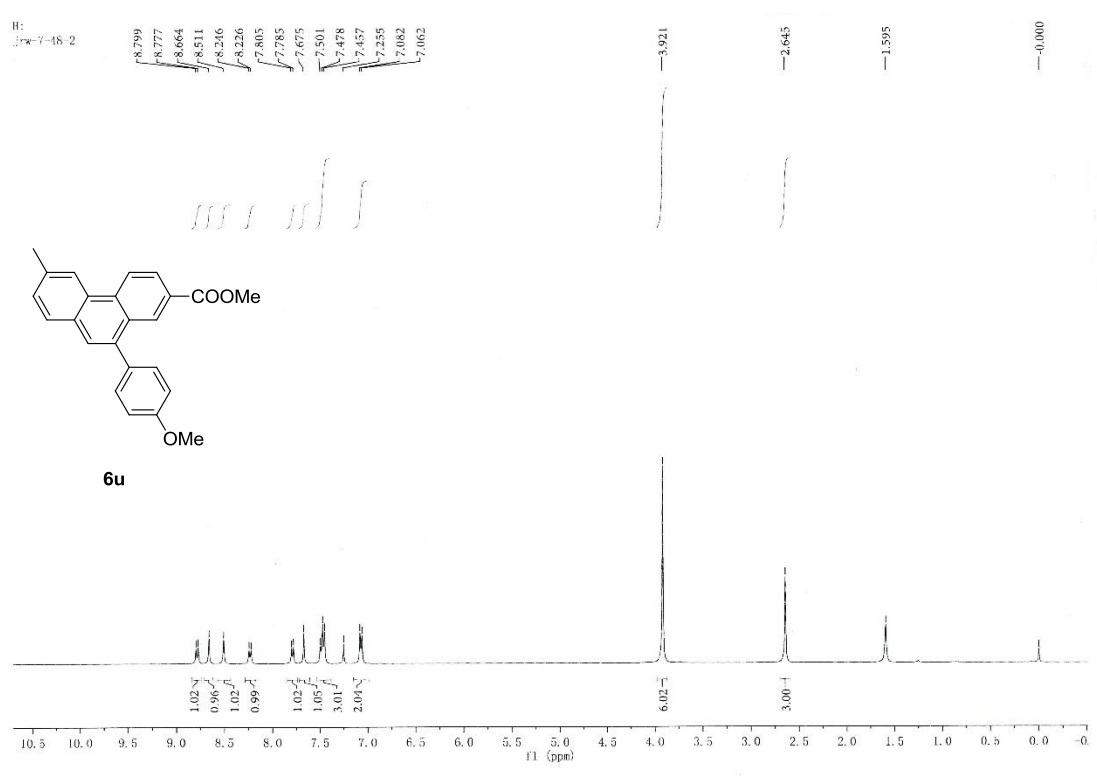
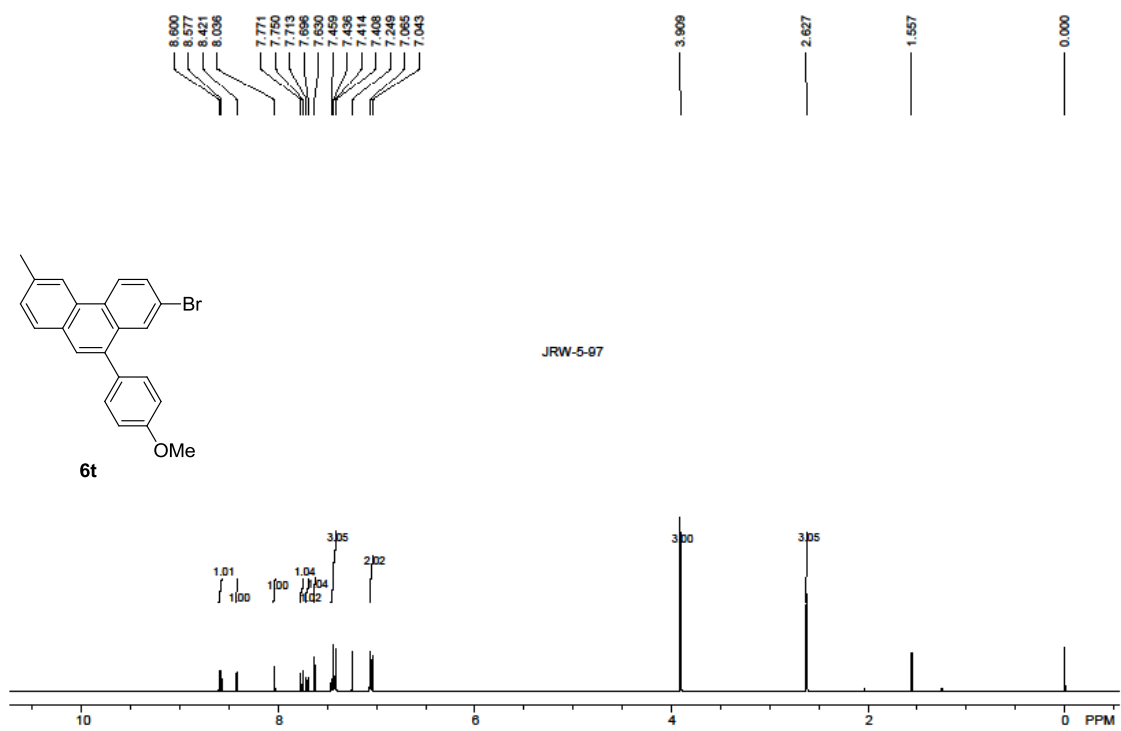
JRW-5-93

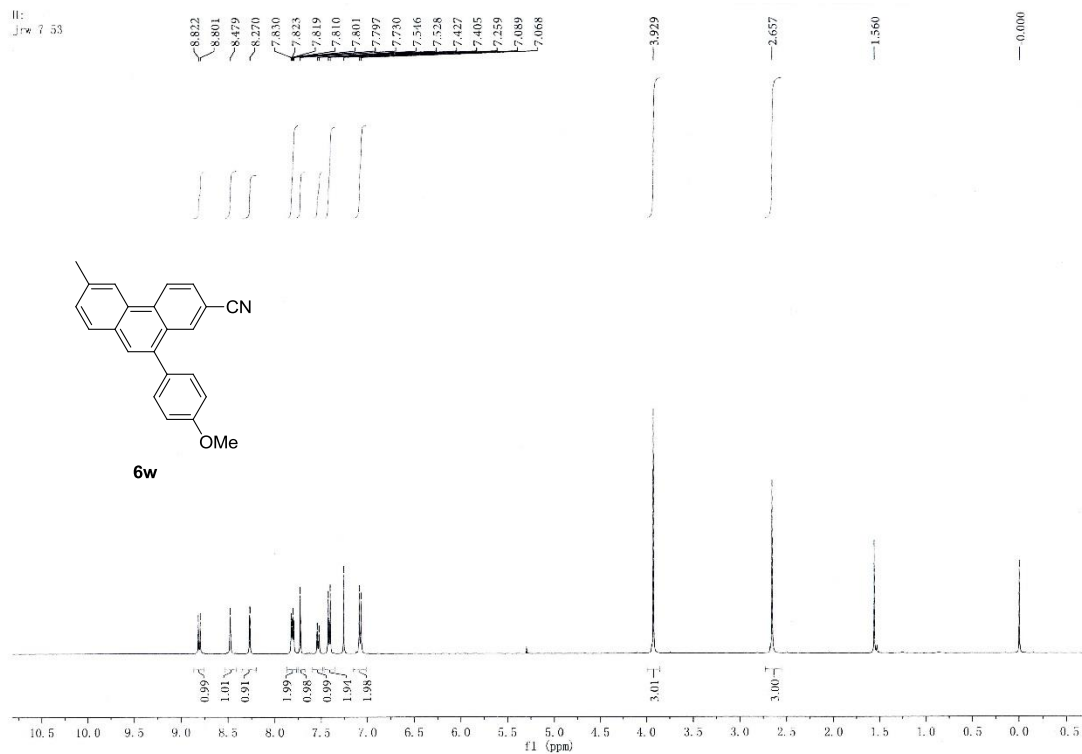
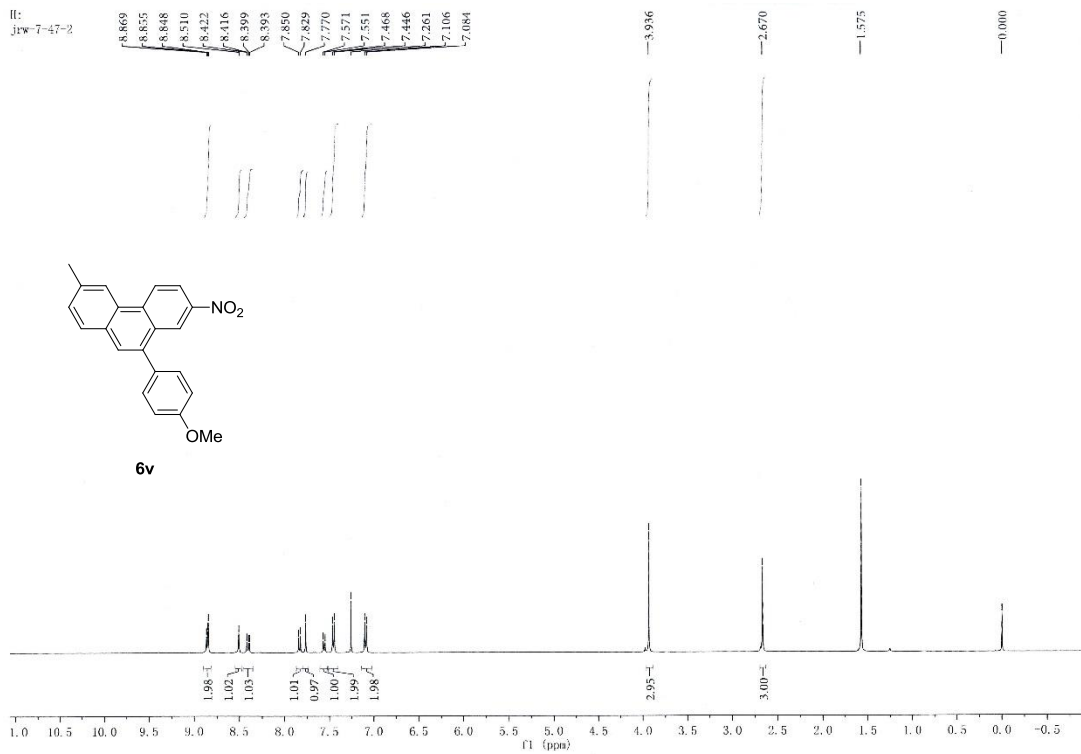


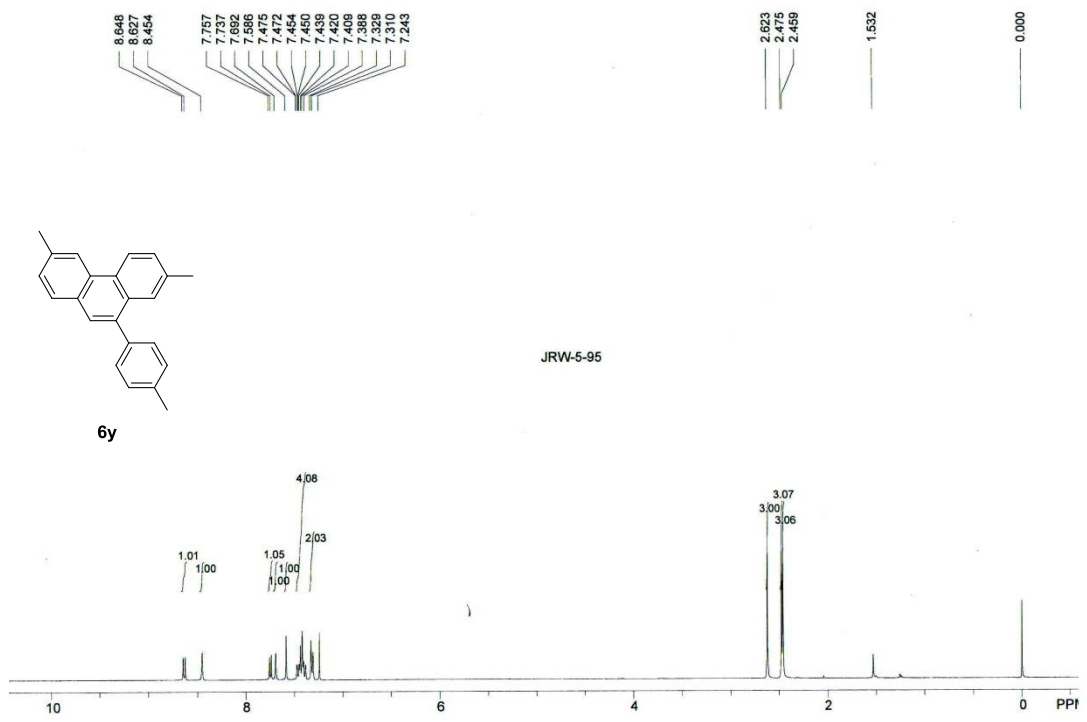
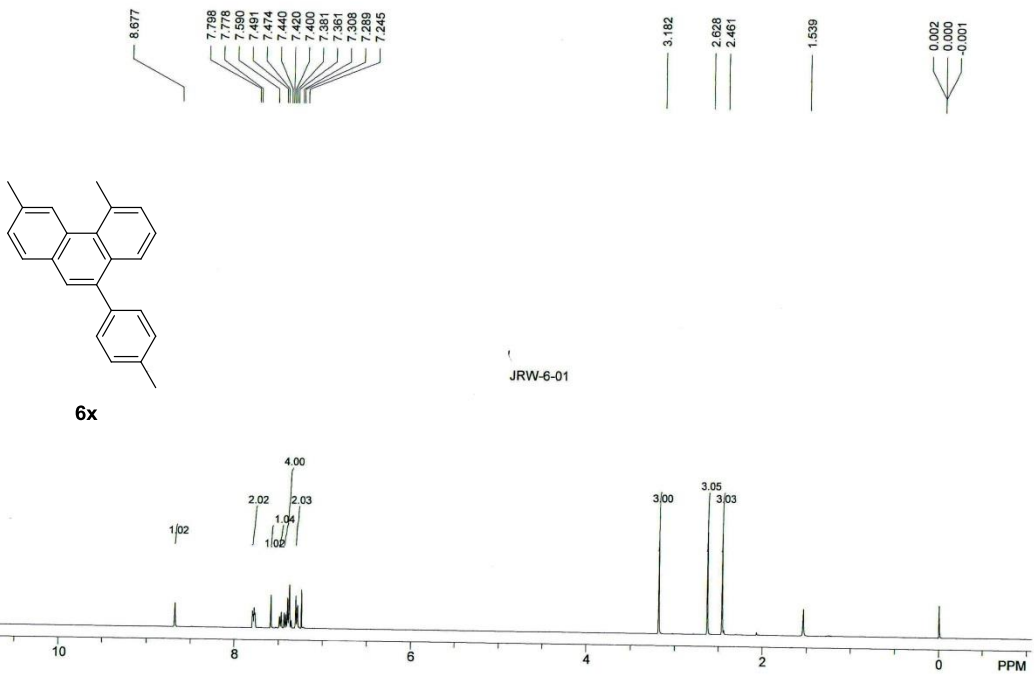
6q



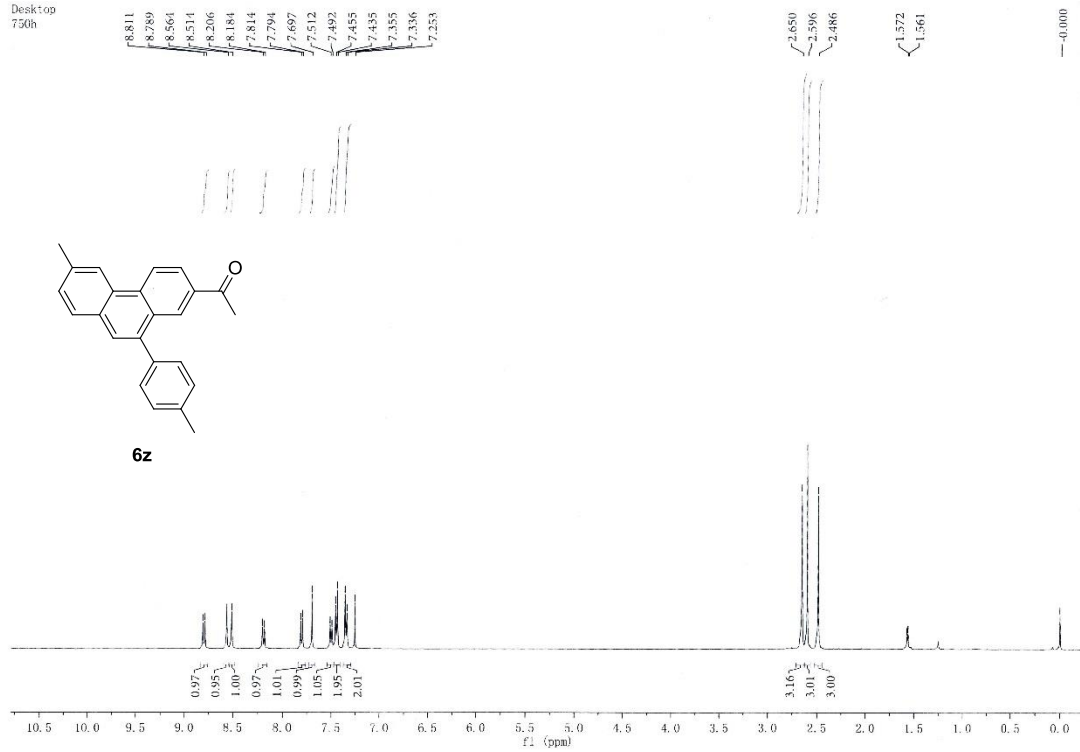




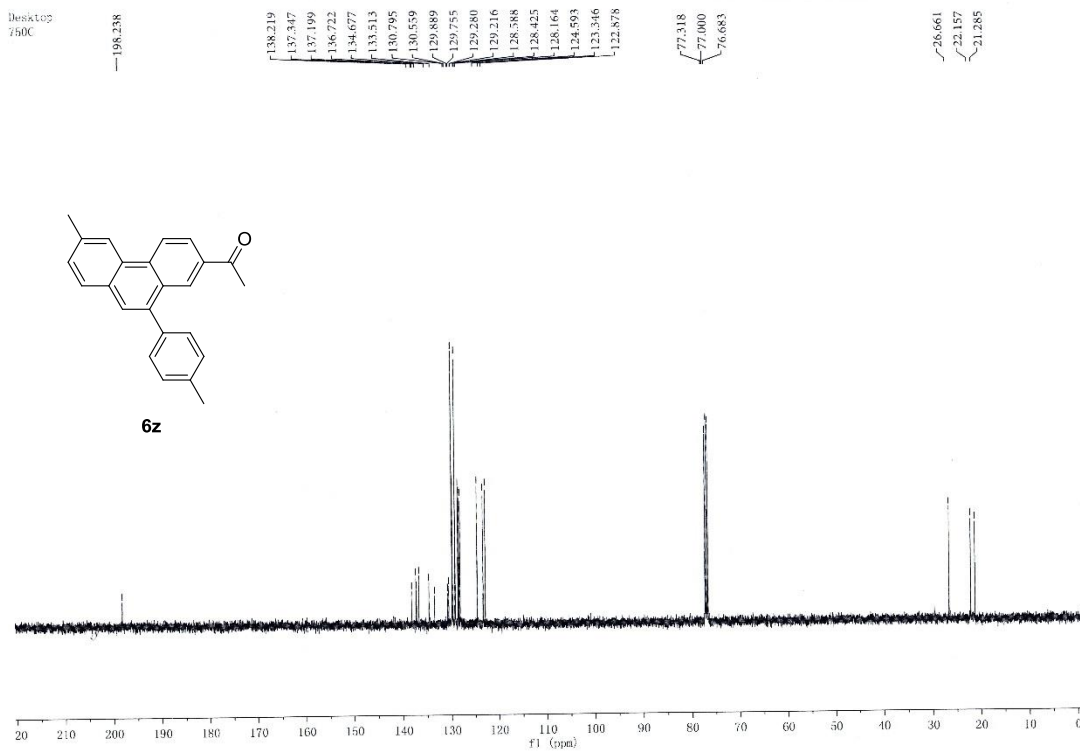


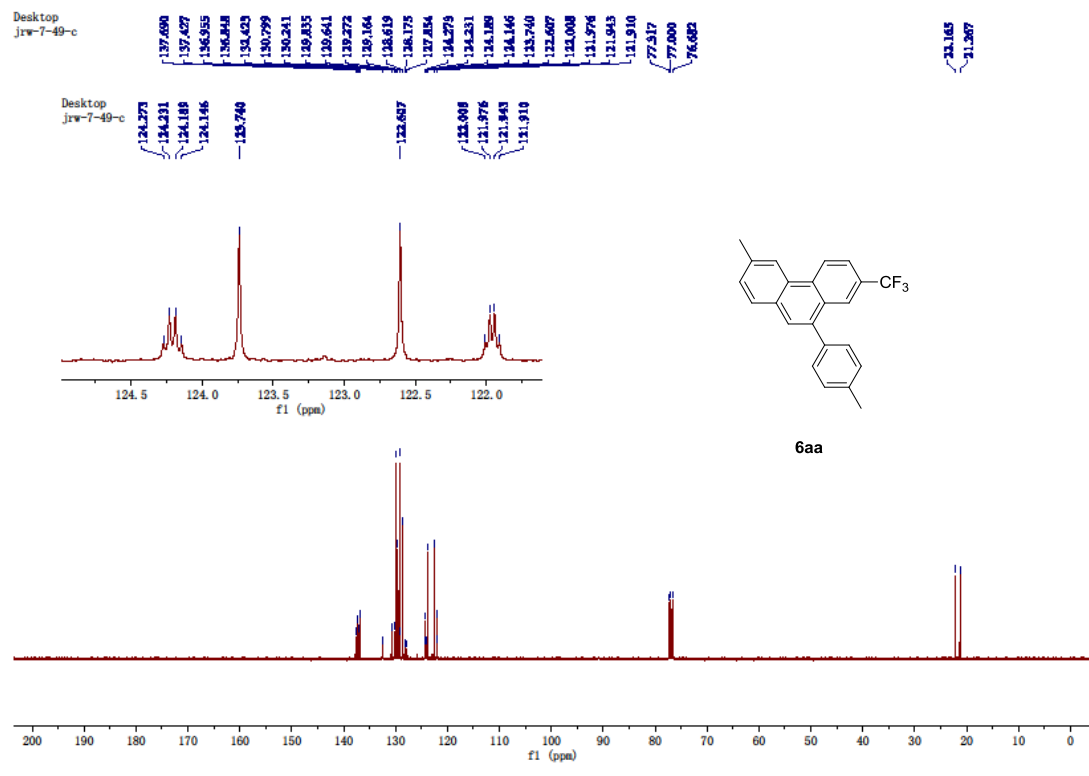
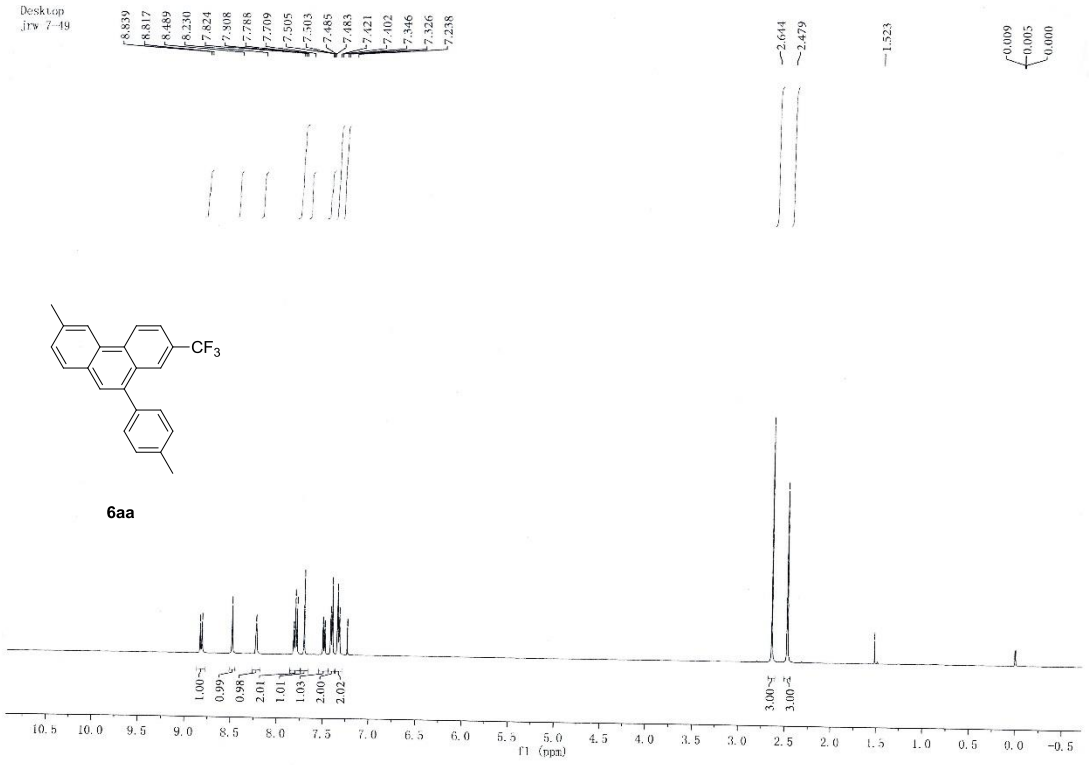


Desktop
750h



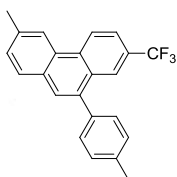
Desktop
750C



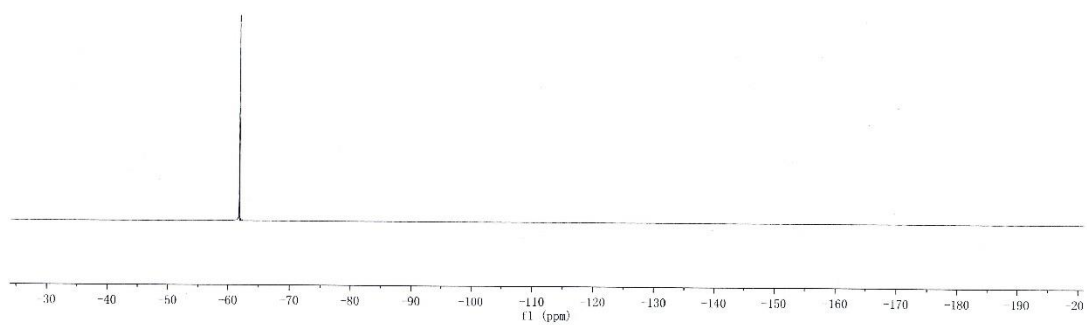


DeskTop
749F

61.801



6aa



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- (1) W. Liu, J. Chen, R. Jin, D. Xu, Y. Li, F. Ba, G. Gu, Y. Kuang and H. Guo, *Org. Chem. Front.*, 2016, **3**, 852-855.
- (2) D. Xu, R. Jin, W. Liu, F. Ba, Y. Li, A. Ding and H. Guo, *Tetrahedron Lett.*, 2016, **57**, 3235-3238.
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