

Supporting Information

Cu-Based Ternary Deep Eutectic Solvents for Homo- and Cross-Coupling Reactions of Terminal Alkynes

Weixu Lu,^a Ming Bao^a and Xiaoqiang Yu^{*a}

^a State Key Laboratory of Fine Chemicals, Dalian University of Technology, Dalian, Liaoning 116023, China.

E-mail: yuxiaoqiang@dlut.edu.cn

Table of contents

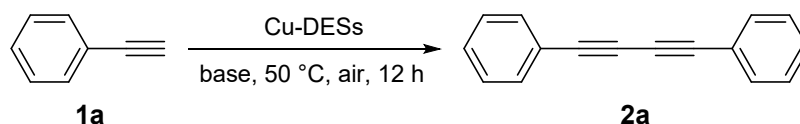
1. General information	2
2. Procedure for optimization studies	2
3. Experimental procedures	3
4. Characterization of products.....	5
5. References	10
6. NMR Spectra	12

1. General information

All reactions were carried out using oven-dried schlenk tube (25 mL) and magnetic stirring (the speed is 1000 rpm) under air unless otherwise stated. All commercially available compounds were purchased from J&K, Alfa, Energy, TCI or Aladdin. TLC was carried out on SiO₂ (silica gel 60 F254, Merck), and the spots were located with UV light (254 nm). Flash chromatography was carried out on SiO₂ (silica gel 60, 200-300 mesh). ¹H and ¹³C NMR spectra were recorded on a Bruker Avance II-400 spectrometer (400 MHz for ¹H, 101 MHz for ¹³C). CDCl₃ and TMS were used as a solvent and an internal standard, respectively. The chemical shifts were reported in ppm downfield (δ) from TMS, the coupling constants *J* are given in Hz. The peak patterns were indicated as follows: s, singlet; d, doublet; t, triplet; m, multiplet; q, quartet. IR spectra were recorded on a NEXUS FT-IR spectrometer. Raman spectra were recorded on a DXR Raman Microscope spectrometer.

2. Procedure for optimization studies

Table S1 Optimization for the homo-coupling reaction^a.



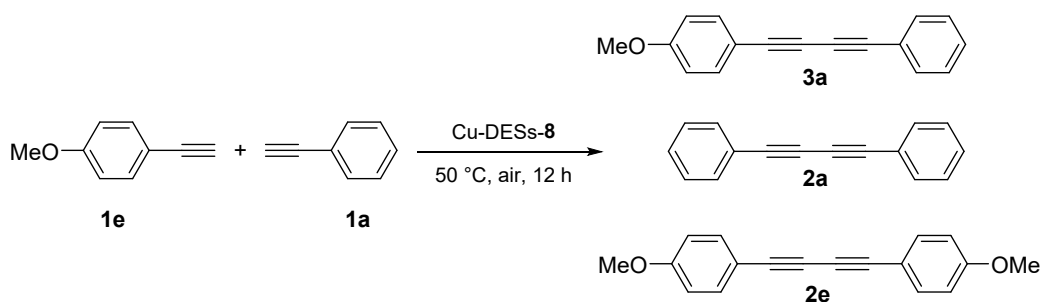
Entry	DESs name	Base (equiv.)	Yield (%) ^b
1	Cu-DESs-1	-	3
2	Cu-DESs-2	-	81
3	Cu-DESs-3	-	99
4	Cu-DESs-4	-	Trace
5	Cu-DESs-5	-	40
6	Cu-DESs-6	-	53
7	Cu-DESs-7	-	Trace
8	Cu-DESs-3	DBU (2)	39
9	Cu-DESs-3	Na ₂ CO ₃ (2)	11
10	Cu-DESs-3	K ₂ CO ₃ (2)	46
11	Cu-DESs-8	-	99
12	Cu-DESs-9	-	84

13 ^c	Cu-DESS-8	-	90
14 ^d	Cu-DESS-8	-	79
15 ^e	Cu-DESS-8	-	99
16 ^f	Cu-DESS-8	-	82
17 ^g	Cu-DESS-8	-	86
18 ^h	Cu-DESS-8	-	6
19 ⁱ	-	-	43

^a The reaction was carried out using **1a** (0.3 mmol) in Cu-DESSs (0.6 mL) at 50 °C for 12 h in air. ^b Isolated yields. ^c Temperature is 40 °C. ^d Temperature is 30 °C. ^e Reaction time is 8 h.

^f Reaction time is 5 h. ^g Under O₂ atmosphere, reaction time is 5 h. ^h Under N₂ atmosphere, reaction time is 8 h. ⁱ The reaction was carried out using 0.6 mL EG, 0.15 mmol Cu(OAc)₂, and 0.3 mmol ChCl for 8 h.

Table S2 Optimization for the cross-coupling reaction^a.



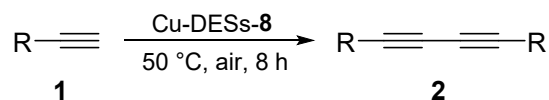
Entry	1e (mmol)	1a (mmol)	3a Yield (%) ^b	2a Yield (%) ^c	2e Yield (%) ^d
1	0.1	0.1	41	53	43
2	0.1	0.2	54	65	36
3	0.1	0.5	66	74	28
4 ^e	0.1	0.5	82	81	13

^a The reaction was carried out using Cu-DESS-8 (0.6 mL) at 50 °C for 12 h in air; isolated yield.

^b Yields based on **1e**. ^c Yields based on **1a**. ^d Yields based on **1e**. ^e Temperature is 70 °C.

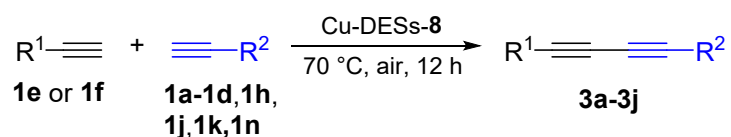
3. Experimental procedures

3.1 .General procedure for the synthesis of symmetric 1,3-diynes



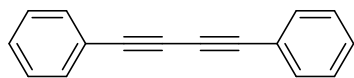
A dry schlenk tube (25 mL) with a magnetic rotor was charged with **1** (0.3 mmol) and Cu-DESS-**8** (0.6 mL). The mixture was stirred at 50 °C for 8 h in air. After the reaction completed, the reaction mixture was extracted with petroleum ether (3×5 mL). The combined organic layer was dried over Na₂SO₄, concentrated under reduced pressure to afford the desired products **2**. [**2i** and **2j** were purified by flash column chromatography on silica gel (petroleum ether/methylene dichloride =30:1)].

3.2 General procedure for the synthesis of unsymmetrical 1,3-diynes.



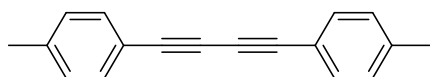
A dry schlenk tube (25 mL) with a magnetic rotor was charged with **1e/1f** (0.1 mmol), **1a-1d/1h/1j/1k/1n** (0.5 mmol) and Cu-DESS-**8** (0.6 mL). The mixture was stirred at 70 °C for 12 h in air. After the reaction completed, the reaction mixture was extracted with petroleum ether (3×5 mL). The combined organic layer was dried over Na₂SO₄, concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (petroleum ether/methylene dichloride =40:1)] to afford **3a-3j**.

4. Characterization of products



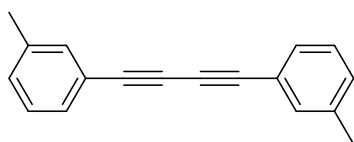
2a

1,4-diphenyl buta-1,3-diyne (2a)¹: White solid; mp 85-86 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.59-7.56 (m, 4 H), 7.41-7.35 (m, 6 H); ¹³C NMR (101 MHz, CDCl₃): δ 132.5, 129.2, 128.4, 121.8, 81.6, 74.0.



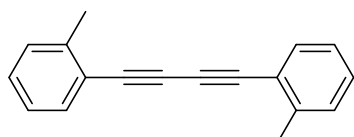
2b

1,4-bis(p-methylphenyl)buta-1,3-diyne (2b)¹: White solid; mp 178-180 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.43 (d, *J* = 8.4 Hz, 4 H), 7.16 (d, *J* = 8.0 Hz, 4 H), 2.37 (s, 6 H); ¹³C NMR (101 MHz, CDCl₃): δ 139.5, 132.4, 129.2, 118.8, 81.6, 73.5, 21.6.



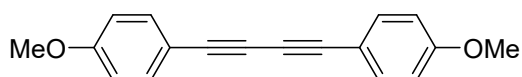
2c

1,4-bis(m-methylphenyl)buta-1,3-diyne (2c)¹: White solid; mp 70-71 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.33-7.31 (m, 4 H), 7.22-7.15 (m, 4 H), 2.32 (s, 6 H); ¹³C NMR (101 MHz, CDCl₃): δ 138.2, 133.0, 130.2, 129.7, 128.4, 121.7, 81.7, 73.8, 21.2.



2d

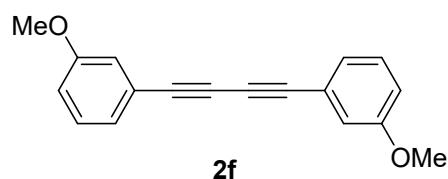
1,4-bis(o-methylphenyl)buta-1,3-diyne (2d)²: White solid; mp 72-73 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.50 (d, *J* = 7.6 Hz, 2 H), 7.27-7.20 (m, 4 H), 7.15 (t, *J* = 7.4 Hz, 2 H), 2.49 (s, 6 H); ¹³C NMR (101 MHz, CDCl₃): δ 141.7, 132.9, 129.6, 129.1, 125.7, 121.8, 81.2, 77.6, 20.8.



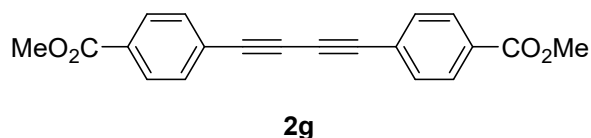
2e

1,4-bis(p-methoxyphenyl)buta-1,3-diyne (2e)¹: White solid; mp 139-140 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.46 (d, *J* = 8.8 Hz, 4 H), 6.85 (d, *J* = 8.8 Hz, 4 H), 3.82

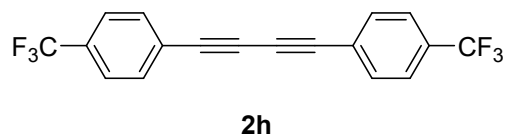
(s, 6 H); ^{13}C NMR (101 MHz, CDCl_3): δ 160.3, 134.1, 114.2, 114.0, 81.3, 73.0, 55.3.



1,4-bis(m-methoxyphenyl)buta-1,3-diyne (2f)³: White solid; mp 96-97 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.24 (d, $J = 8.0$ Hz, 2 H), 7.12 (d, $J = 8.0$ Hz, 2 H), 7.04 (s, 2 H), 6.92 (d, $J = 8.0$ Hz, 2 H), 3.79 (s, 6 H); ^{13}C NMR (101 MHz, CDCl_3): δ 159.4, 129.6, 125.1, 122.7, 117.2, 116.1, 81.6, 73.7, 55.3.



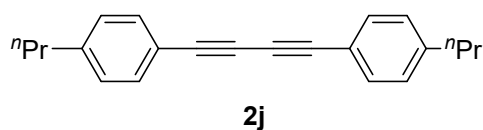
1,4-bis(p-carbomethoxyphenyl)buta-1,3-diyne (2g)⁴: White solid; mp 189-191 °C; ^1H NMR (400 MHz, CDCl_3): δ 8.02 (d, $J = 8.4$ Hz, 4 H), 7.59 (d, $J = 8.4$ Hz, 4 H), 3.93 (s, 6 H); ^{13}C NMR (101 MHz, CDCl_3): δ 166.3, 132.5, 130.6, 129.6, 126.1, 81.9, 76.3, 52.4.



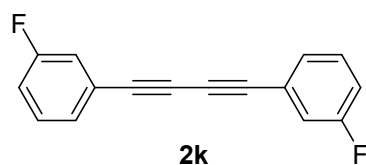
1,4-bis(p-trifluoromethyl)buta-1,3-diyne (2h)²: Yellow solid; mp 169-170 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.66-7.60 (m, 8 H); ^{13}C NMR (101 MHz, CDCl_3): δ 132.8, 131.1 (q, $J = 32.3$ Hz), 125.5 (q, $J = 4.0$ Hz), 125.3, 123.7 (q, $J = 272.7$ Hz), 81.0, 75.6.



1,4-bis(4-n-butylphenyl)buta-1,3-diyne (2i)⁵: Yellow solid; mp 111-113 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.44 (d, $J = 8.0$ Hz, 4 H), 7.15 (d, $J = 8.0$ Hz, 4 H), 2.62 (t, $J = 7.6$ Hz, 4 H), 1.63-1.56 (m, 4 H), 1.40-1.31 (m, 4 H), 0.93 (t, $J = 7.4$ Hz, 6 H); ^{13}C NMR (101 MHz, CDCl_3): δ 144.5, 132.4, 128.6, 119.0, 81.6, 73.5, 35.7, 33.3, 22.3, 13.9.



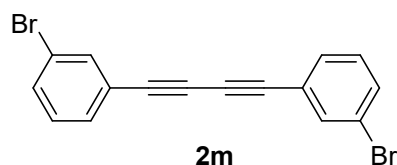
1,4-bis(4-n-propylphenyl)buta-1,3-diyne (2j)¹: Yellow solid; mp 105-107 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.44 (d, *J* = 8.0 Hz, 4 H), 7.14 (d, *J* = 8.0 Hz, 4 H), 2.59 (t, *J* = 7.6 Hz, 4 H), 1.67-1.61 (m, 4 H), 0.94 (t, *J* = 7.4 Hz, 4 H); ¹³C NMR (101 MHz, CDCl₃): δ 144.3, 132.4, 128.6, 119.1, 81.6, 73.5, 38.1, 24.3, 13.8.



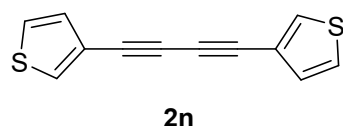
1,4-bis(3-fluorophenyl)buta-1,3-diyne (2k)⁶: White solid; mp 118-120 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.31-7.29 (m, 4 H), 7.21 (d, *J* = 7.2 Hz, 2 H), 7.11-7.06 (m, 2 H); ¹³C NMR (101 MHz, CDCl₃): δ 162.3 (d, *J* = 242.4 Hz), 130.1 (d, *J* = 9.1 Hz), 128.5 (d, *J* = 4.0 Hz), 123.4 (d, *J* = 10.1 Hz), 119.2 (d, *J* = 23.2 Hz), 116.9 (d, *J* = 21.2 Hz), 80.7 (d, *J* = 3.0 Hz), 74.5.



1,4-bis(3-chlorophenyl)buta-1,3-diyne (2l)⁶: White solid; mp 74-76 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.50-7.49 (m, 2 H), 7.41-7.39 (m, 2 H), 7.35 (ddd, *J* = 8.0, 2.4, 1.2 Hz, 2 H), 7.29-7.25 (m, 2 H); ¹³C NMR (101 MHz, CDCl₃): δ 134.4, 132.3, 130.7, 129.7, 123.3, 80.6, 74.7.

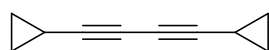


1,4-bis(3-bromophenyl)buta-1,3-diyne (2m)⁷: White solid; mp 95-97 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.67-7.66 (m, 2 H), 7.51 (ddd, *J* = 8.0, 2.0, 1.2 Hz, 2 H), 7.46-7.44 (m, 2 H), 7.23-7.20 (m, 2 H); ¹³C NMR (101 MHz, CDCl₃): δ 135.2, 132.6, 131.1, 129.9, 123.6, 122.3, 80.5, 74.8.



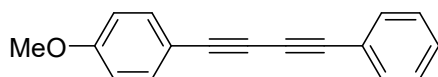
1,4-bis(3-thienyl)buta-1,3-diyne (2n)⁵: White solid; mp 110-112 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.59 (dd, *J* = 3.2, 1.2 Hz, 2 H), 7.28 (dd, *J* = 5.2, 3.0 Hz, 2 H), 7.17 (dd, *J* = 4.8, 1.2 Hz, 2 H); ¹³C NMR (101 MHz, CDCl₃): δ 131.2, 130.2, 125.6, 120.9,

76.6, 73.6.



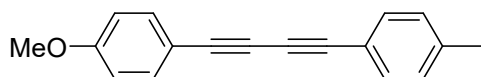
2o

1,4-dicyclopropylbuta-1,3-diyne (2o)²: Colorless oil; ¹H NMR (400 MHz, CDCl₃): δ 1.31-1.25 (m, 2 H), 0.79-0.71 (m, 8 H); ¹³C NMR (101 MHz, CDCl₃): δ 80.1, 60.8, 8.7, 0.1.



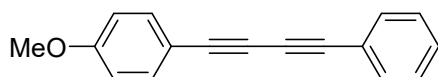
3a

1-methoxy-4-(phenylbuta-1,3-diyne-1-yl) benzene (3a)¹: White solid; mp 97-98 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.52 (d, J = 7.6 Hz, 2 H), 7.47 (d, J = 8.8 Hz, 2 H), 7.37-7.31 (m, 3 H), 6.86 (d, J = 8.4 Hz, 2 H), 3.83 (s, 3 H); ¹³C NMR (101 MHz, CDCl₃): δ 160.4, 134.2, 132.5, 129.0, 128.4, 122.0, 114.2, 113.7, 81.8, 81.0, 74.2, 72.8, 55.4.



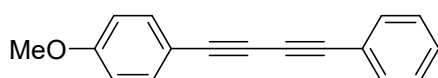
3b

1-((4-methoxyphenyl)buta-1,3-diyne-1-yl)-4-methylbenzene (3b)¹: White solid; mp 141-143 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.47 (d, J = 8.8 Hz, 2 H), 7.41 (d, J = 8.4 Hz, 2 H), 7.14 (d, J = 8.4 Hz, 2 H), 6.86 (d, J = 8.8 Hz, 2 H), 3.82 (s, 3 H), 2.37 (s, 3 H); ¹³C NMR (101 MHz, CDCl₃): δ 160.3, 139.4, 134.1, 132.4, 129.2, 118.9, 114.2, 113.9, 81.5, 81.3, 73.6, 72.9, 55.4, 21.6.



3c

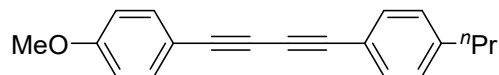
1-(4-methoxyphenyl)-4-(*m*-toluenyl)buta-1,3-diyne (3c)¹: White solid; mp 63-64 °C; ¹H NMR (400 MHz, CDCl₃): δ 7.47 (d, J = 8.8 Hz, 2 H), 7.34-7.31 (m, 2 H), 7.24-7.16 (m, 2 H), 6.86 (d, J = 8.8 Hz, 2 H), 3.82 (s, 3 H), 2.33 (s, 3 H); ¹³C NMR (101 MHz, CDCl₃): δ 160.4, 138.2, 134.1, 133.0, 130.0, 129.6, 128.3, 121.8, 114.2, 113.8, 81.6, 81.3, 73.8, 72.8, 55.4, 21.2.



3d

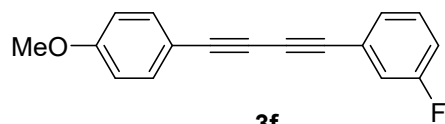
1-(4-methoxyphenyl)-4-(*o*-toluenyl)buta-1,3-diyne (3d)⁸: White solid; mp 62-64 °C;

$^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.48 (d, $J = 8.0$ Hz, 3 H), 7.27-7.13 (m, 3 H), 6.86 (d, $J = 8.0$ Hz, 2 H), 3.83 (s, 3 H), 2.49 (s, 3 H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ 160.3, 141.6, 134.1, 132.9, 129.6, 129.0, 125.7, 121.8, 114.2, 113.8, 82.3, 80.1, 77.7, 72.8, 55.4, 20.8.



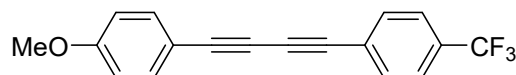
3e

1-Propyl-4-((4-methoxyphenyl)buta-1,3-diynyl)benzene (3e)¹: Yellow solid; mp 90-92 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.48-7.42 (m, 4 H), 7.14 (d, $J = 7.6$ Hz, 2 H), 6.86 (d, $J = 8.4$ Hz, 2 H), 3.83 (s, 3 H), 2.59 (t, $J = 7.6$ Hz, 2 H), 1.66-1.61 (m, 2 H), 0.94 (t, $J = 7.6$ Hz, 3 H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ 160.3, 144.2, 134.1, 132.4, 128.6, 119.1, 114.2, 113.9, 81.5, 81.4, 73.5, 72.9, 55.4, 38.1, 24.3, 13.8.



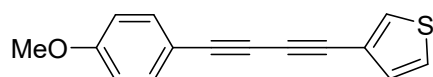
3f

1-fluoro-3-((4-methoxyphenyl)buta-1,3-diyn-1-yl)benzene (3f)⁹: Yellow solid; mp 61-62 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.48 (d, $J = 8.8$ Hz, 2 H), 7.31-7.30 (m, 2 H), 7.20 (d, $J = 8.8$ Hz, 1 H), 7.09-7.04 (m, 1 H), 6.87 (d, $J = 8.4$ Hz, 2 H), 3.83 (s, 3 H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ 162.3 (d, $J = 245.8$ Hz) 160.5, 134.2, 130.1 (d, $J = 8.1$ Hz), 128.4 (d, $J = 3.0$ Hz), 123.9 (d, $J = 9.1$ Hz), 119.2, 119.0, 116.4, 114.2, 113.4, 82.5, 79.6, 75.1, 72.4, 55.4.



3g

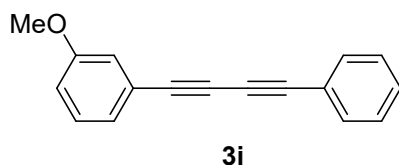
1-(4-methoxyphenyl)-4-(4-trifluorophenyl)buta-1,3-diyne (3g)¹: Yellow solid; mp 142-143 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.63-7.57 (m, 4 H), 7.49 (d, $J = 8.8$ Hz, 2 H), 6.87 (d, $J = 8.8$ Hz, 2 H), 3.83 (s, 3 H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3): δ 160.7, 134.3, 132.6, 130.6 (q, $J = 33.3$ Hz), 126.0, 125.4 (q, $J = 4.0$ Hz), 123.8 (q, $J = 272.7$ Hz), 114.3, 113.3, 83.2, 79.3, 76.6, 72.3, 55.4.



3h

3-((4-methoxyphenyl)buta-1,3-diyn-1-yl)thiophene (3h)⁸: Yellow solid; mp 84-86 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.57 (dd, $J = 3.2, 1.2$ Hz, 1 H), 7.46 (d, $J = 9.2$ Hz,

2 H), 7.27 (dd, $J = 4.8, 2.8$ Hz, 1 H), 7.17 (dd, $J = 5.2, 1.2$ Hz, 1 H), 6.85 (d, $J = 9.2$ Hz, 2 H), 3.82 (s, 3 H); ^{13}C NMR (101 MHz, CDCl_3): δ 160.4, 134.1, 131.0, 130.2, 125.5, 121.1, 114.2, 113.7, 81.6, 76.2, 73.8, 72.7, 55.4.



1-methoxy-3-(phenylbuta-1,3-diyne-1-yl)benzene (3i)⁹: White solid; mp 61-62 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.54-7.52 (m, 2 H), 7.38-7.31 (m, 3 H), 7.26-7.22 (m, 1 H), 7.13 (d, $J = 7.6$ Hz, 1 H), 7.05 (s, 1 H), 6.94-6.92 (m, 1 H); 3.81 (s, 3 H); ^{13}C NMR (101 MHz, CDCl_3): δ 159.3, 132.5, 129.6, 129.2, 128.5, 125.1, 122.8, 121.8, 117.1, 116.1, 81.6, 81.5, 73.9, 73.7, 55.3.



1-methoxy-3-(p-tolylbuta-1,3-diyne-1-yl)benzene (3j)⁹: White solid; mp 76-78 °C; ^1H NMR (400 MHz, CDCl_3): δ 7.42 (d, $J = 8.0$ Hz, 2 H), 7.23 (d, $J = 8.0$ Hz, 1 H), 7.15-7.11 (m, 3 H), 7.04 (s, 1 H), 6.92 (ddd, $J = 8.0, 2.4, 1.2$ Hz, 1 H), 3.80 (s, 3 H), 2.37 (s, 3 H); ^{13}C NMR (101 MHz, CDCl_3): δ 159.3, 139.7, 132.5, 129.5, 129.3, 125.1, 122.9, 118.7, 117.1, 116.0, 81.9, 81.2, 73.9, 73.3, 55.3, 21.6.

5. References

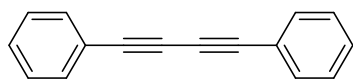
1. H. Xu, K. Wu, J. Tian, L. Zhu and X. Yao, *Green Chem.*, **2018**, *20*, 793–797.
2. S. Tang, L. Li, X. Ren, J. Li, G. Yang, H. Li and B. Yuan, *Green Chem.*, **2019**, *21*, 2899–2904.
3. L. Feng, T. Hu, S. Zhang, H.-Y. Xiong and G. Zhang, *Org. Lett.*, **2019**, *21*, 9487–9492.
4. H. Xu, L. Wu, J. Tian, J. Wang, P. Wang, X. Niu and X. Yao, *Eur. J. Org. Chem.*, **2019**, *39*, 6690–6696.
5. W.-B. Sheng, T.-Q. Chen, M.-Z. Zhang, M. Tian, G.-F. Jiang and C.-C. Guo, *Tetrahedron Lett.*, **2016**, *57*, 1641–1643.
6. X. Ye, P. Zhao and X. Shi, *Angew. Chem.*, **2019**, *58*, 17226–17230.
7. K. Li, K. Wu, Y. Lu, J. Guo, P. Hu and C. Su, *Angew. Chem. Int. Ed.*, **2022**, *61*, art. no. E202114070.
8. M. Yu, D. Pan, W. Jia, W. Chen and N. Jiao, *Tetrahedron Lett.*, **2010**, *51*, 1287–1290.

9. Y. Liu, P. Liu, N. Gu, J. Xie, Y. Liu and B. Dai, *Chinese Journal of Chemistry*, **2016**, *34*, 895 - 900.

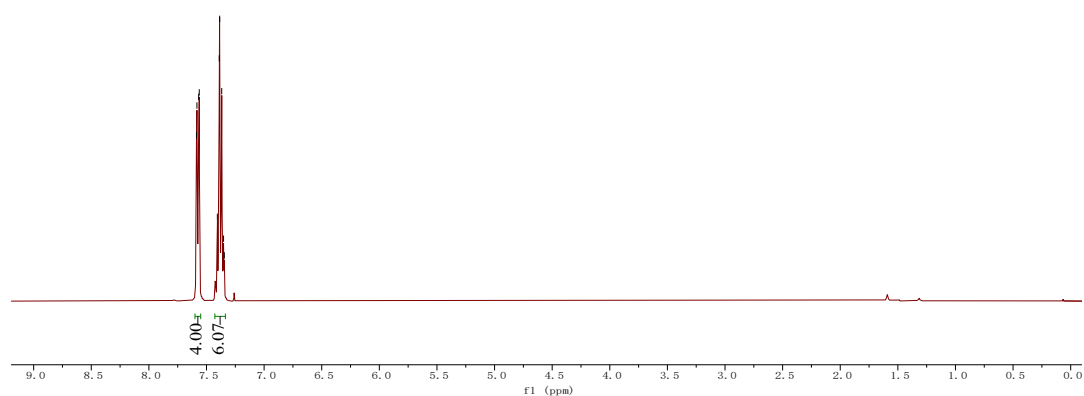
6. NMR Spectra

^1H NMR, 400 MHz, CDCl_3

7.587
7.583
7.568
7.563
7.406
7.389
7.386
7.368
7.352
7.345



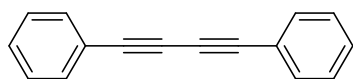
2a



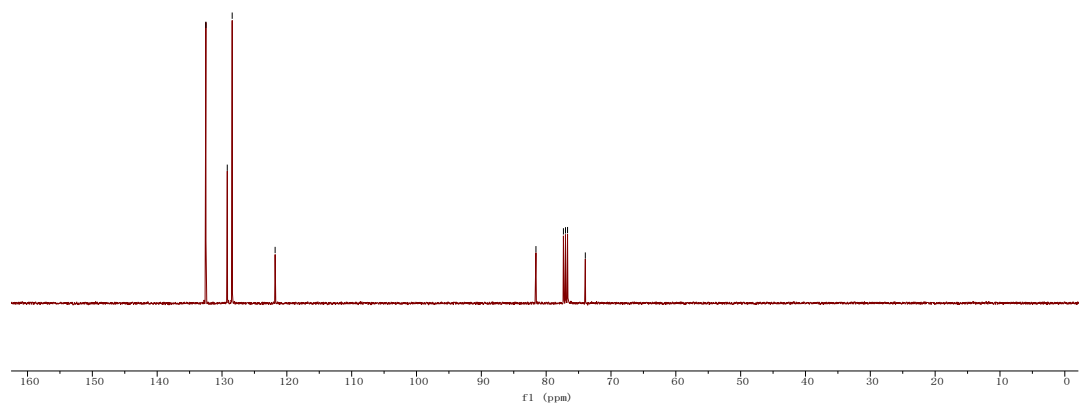
^{13}C NMR, 101 MHz, CDCl_3

132.49
129.19
128.43
121.81

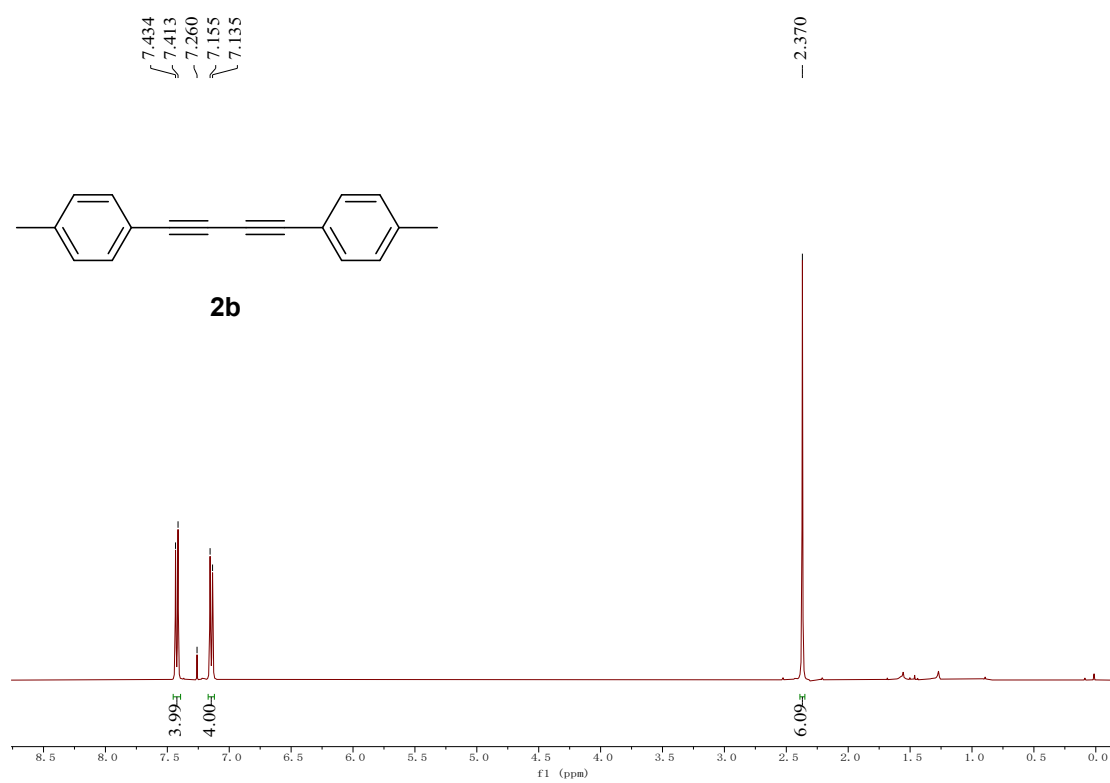
81.58
77.34
77.02
76.70
73.96



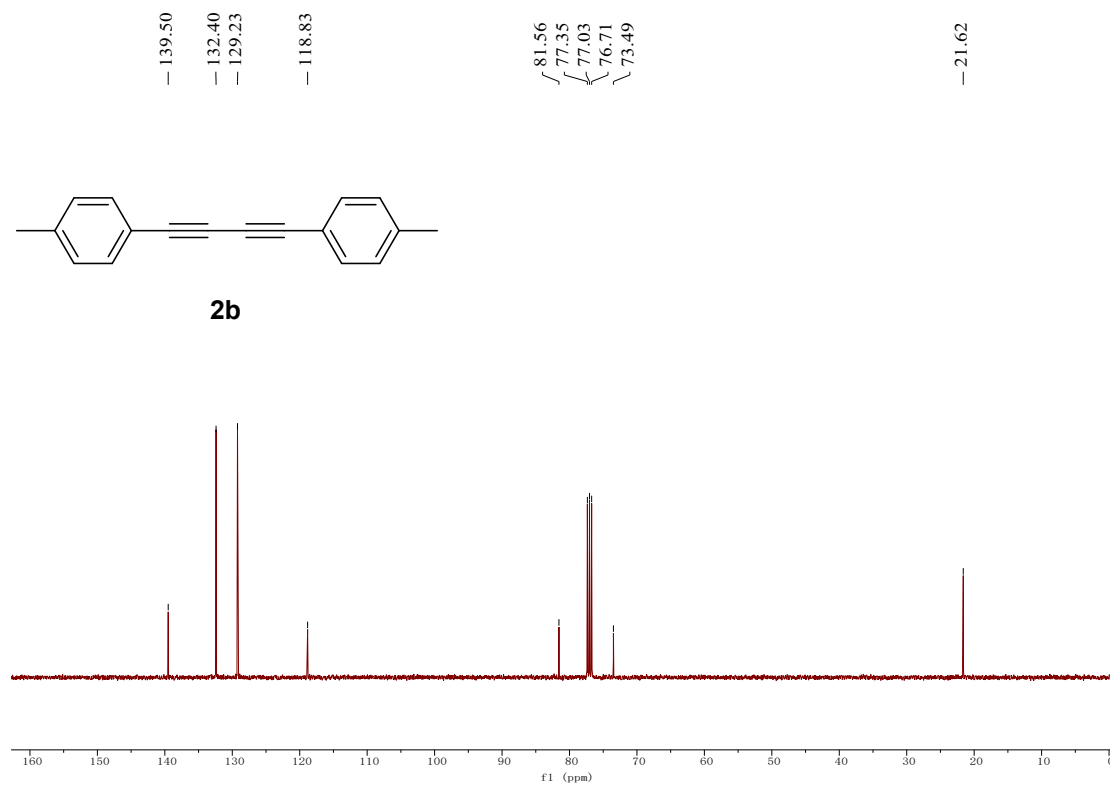
2a



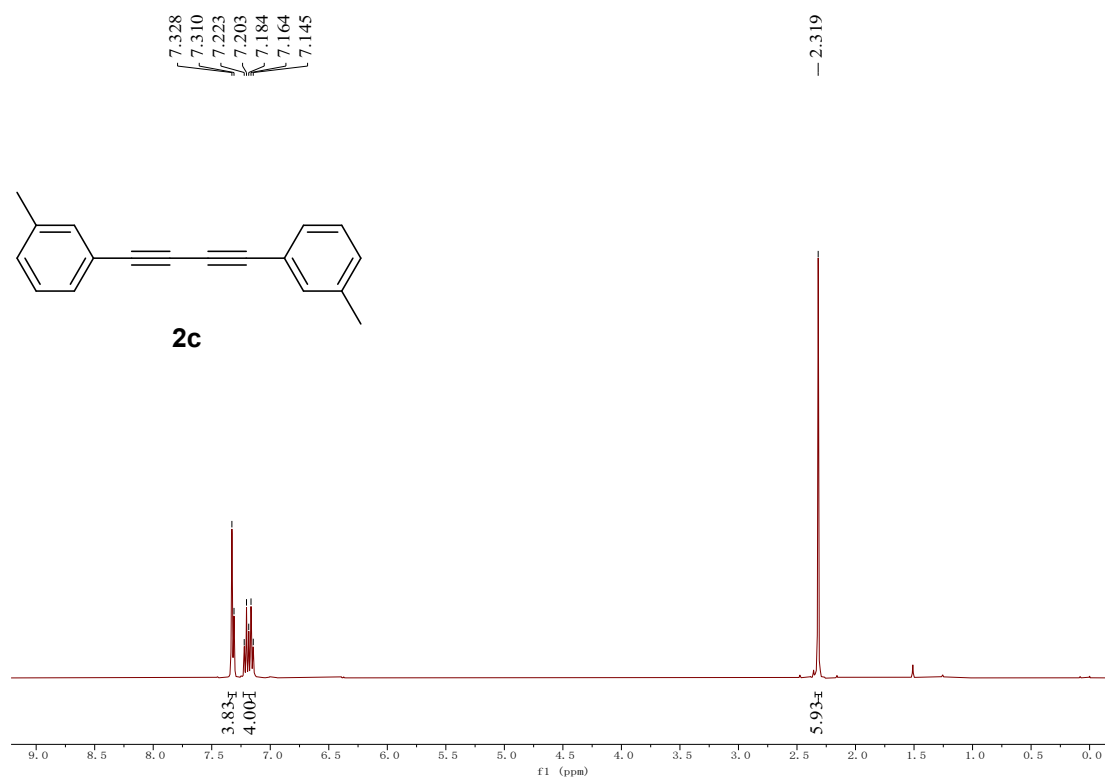
^1H NMR, 400 MHz, CDCl_3



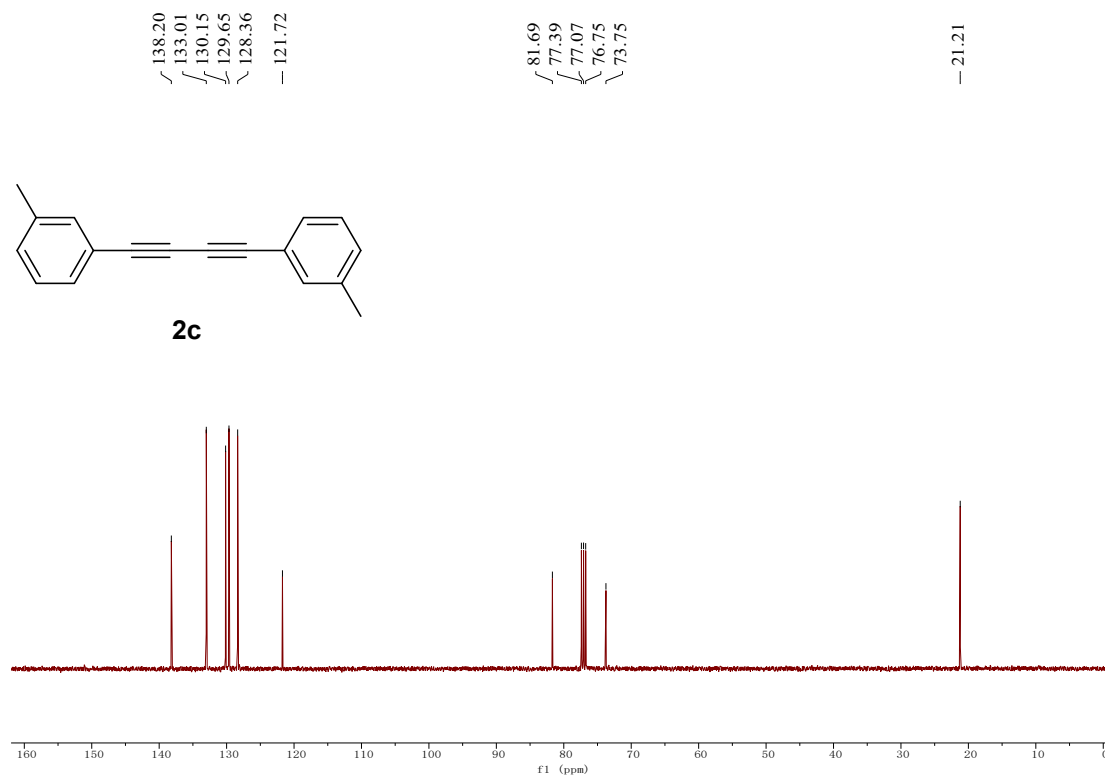
^{13}C NMR, 101 MHz, CDCl_3



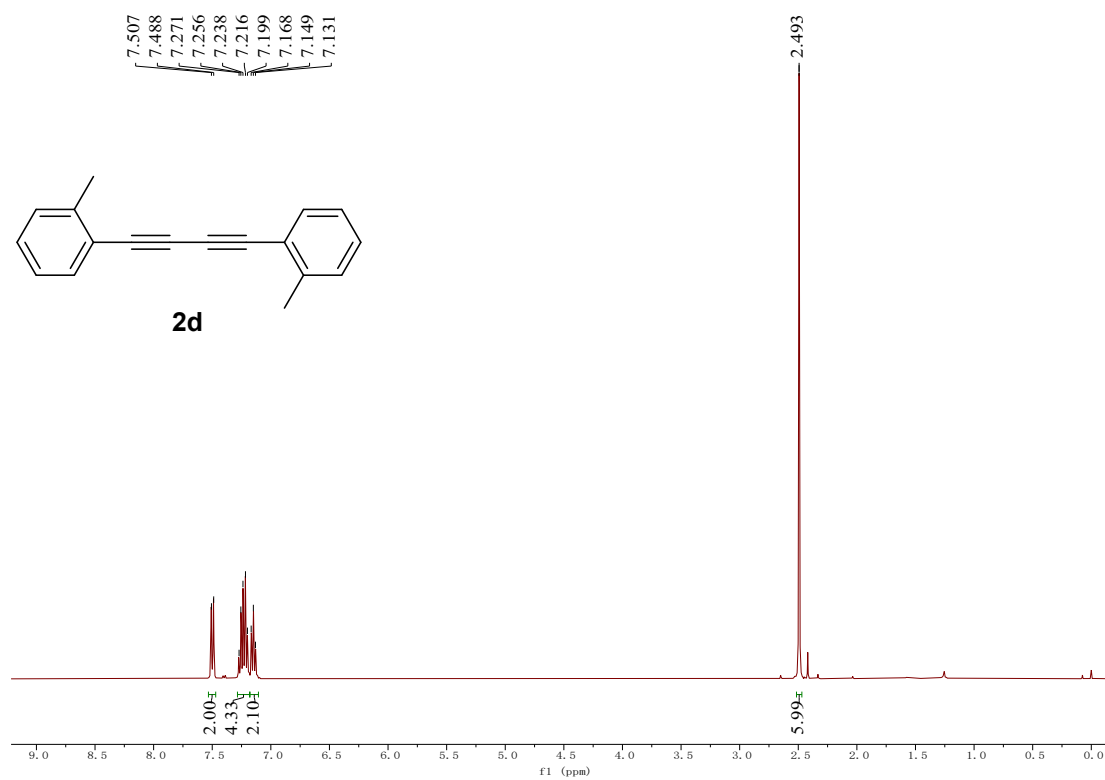
^1H NMR, 400 MHz, CDCl_3



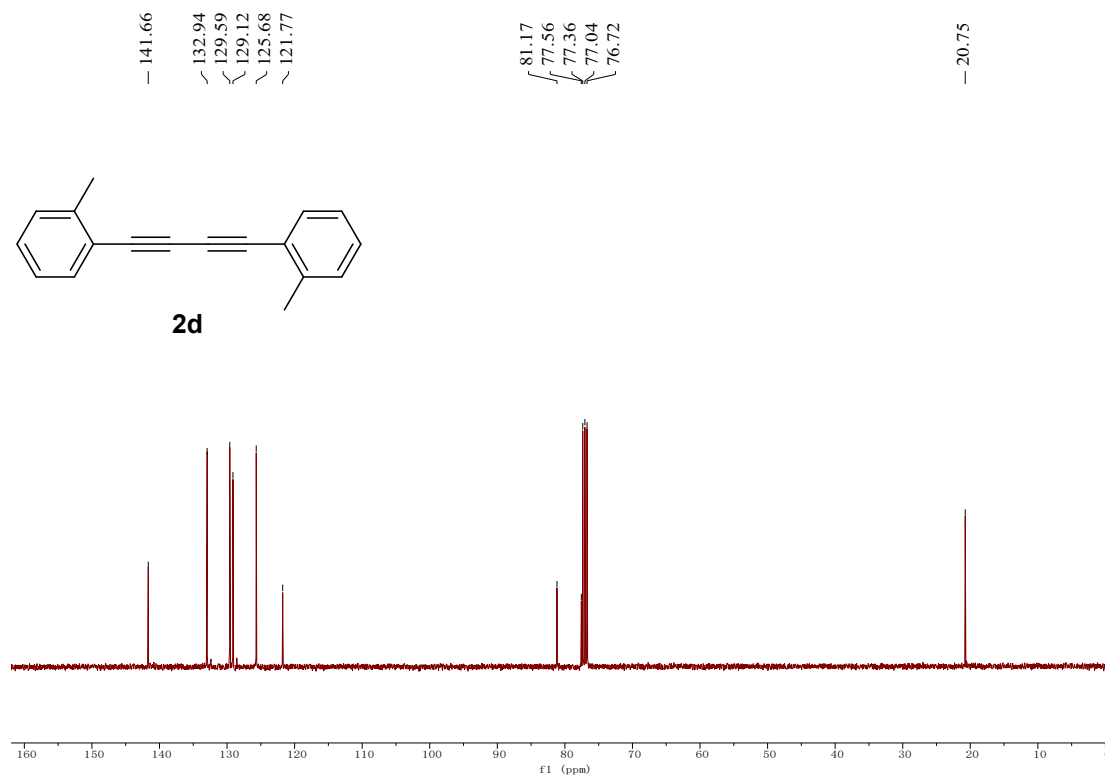
^{13}C NMR, 101 MHz, CDCl_3



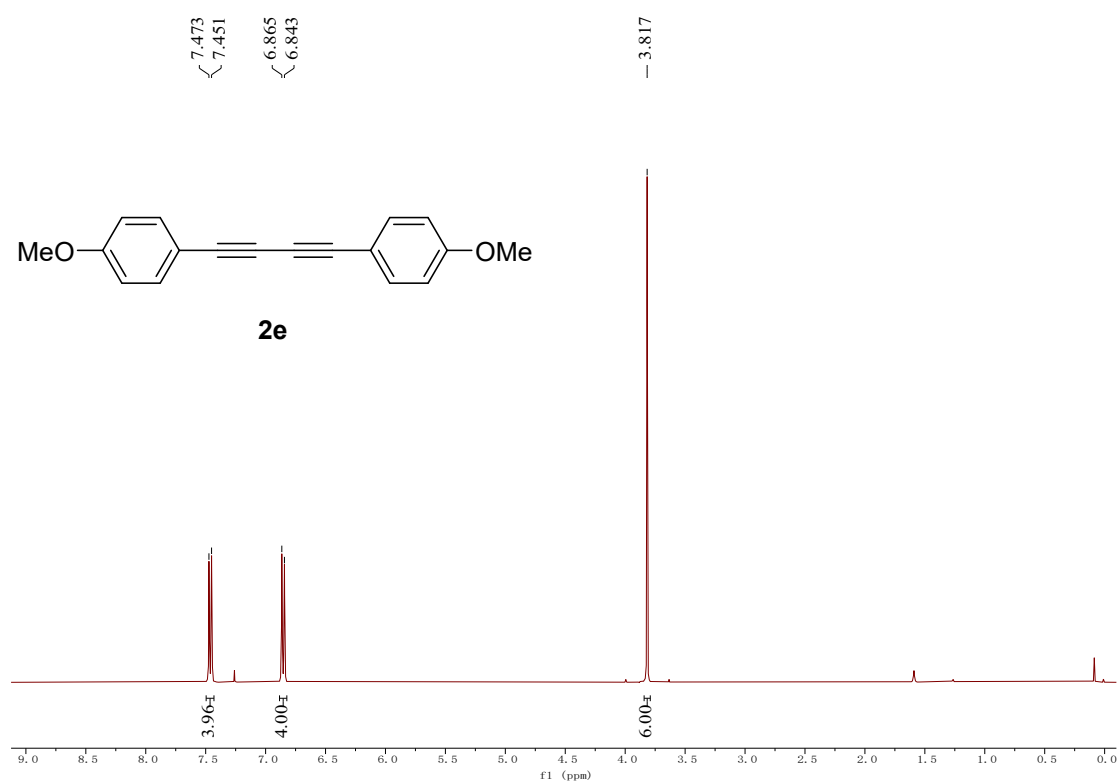
^1H NMR, 400 MHz, CDCl_3



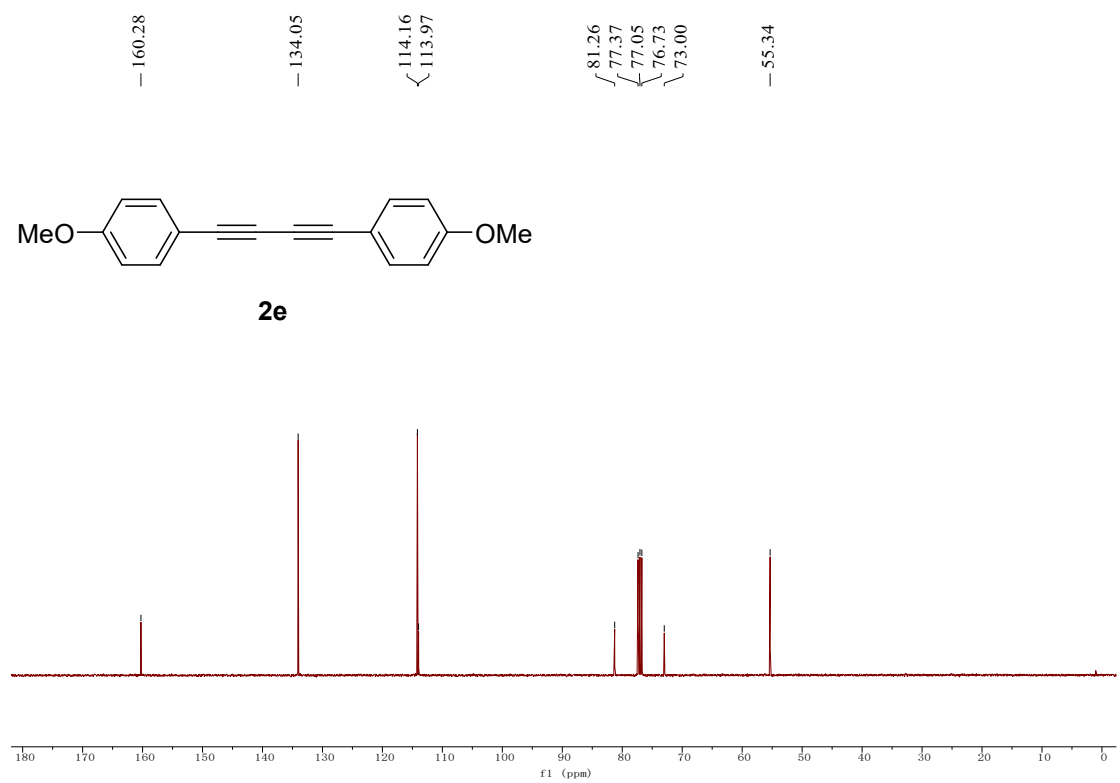
^{13}C NMR, 101 MHz, CDCl_3



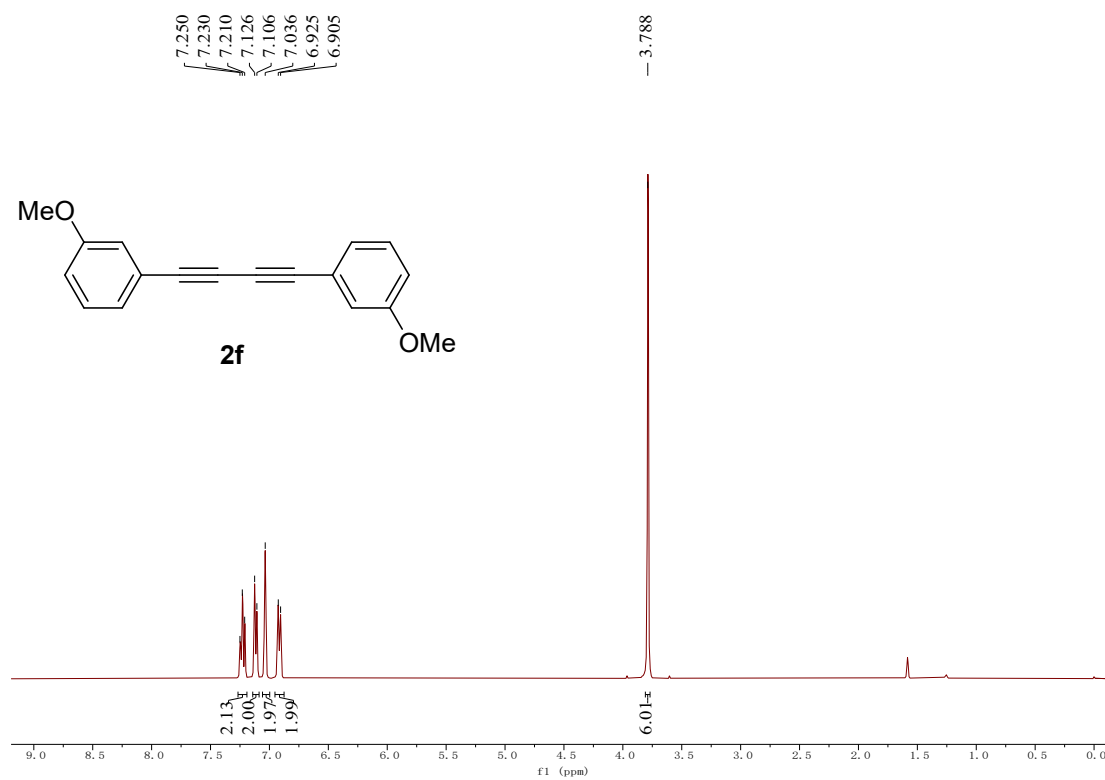
^1H NMR, 400 MHz, CDCl_3



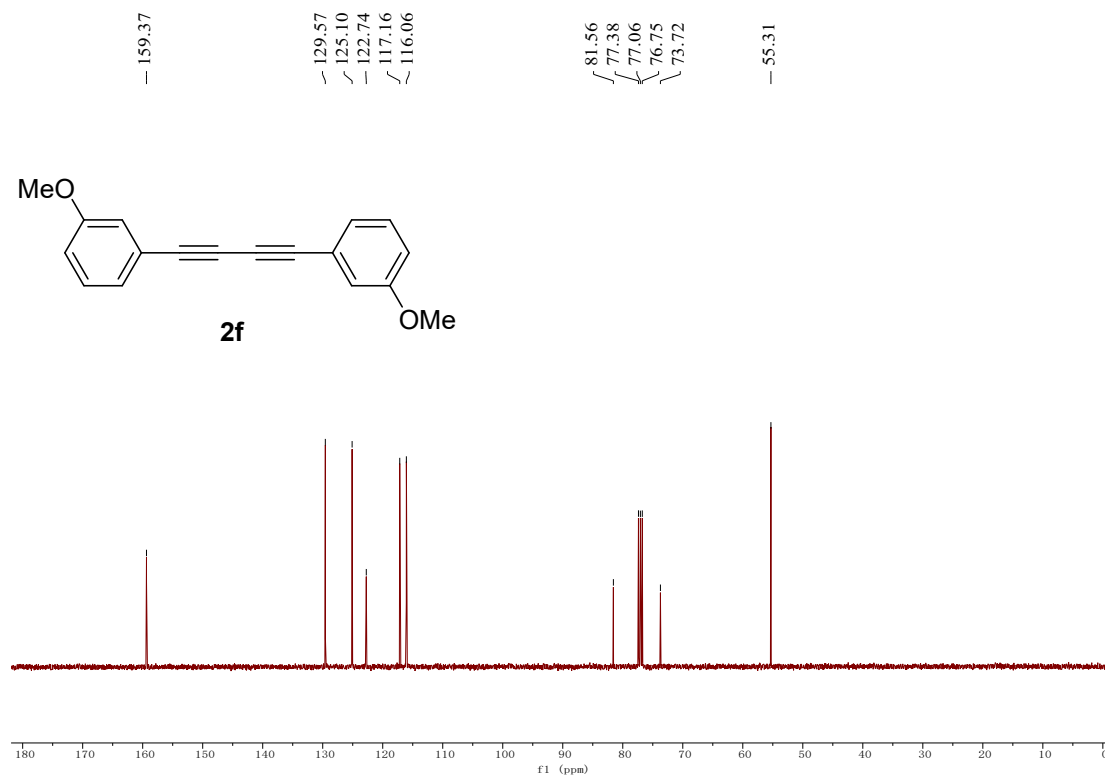
^{13}C NMR, 101 MHz, CDCl_3



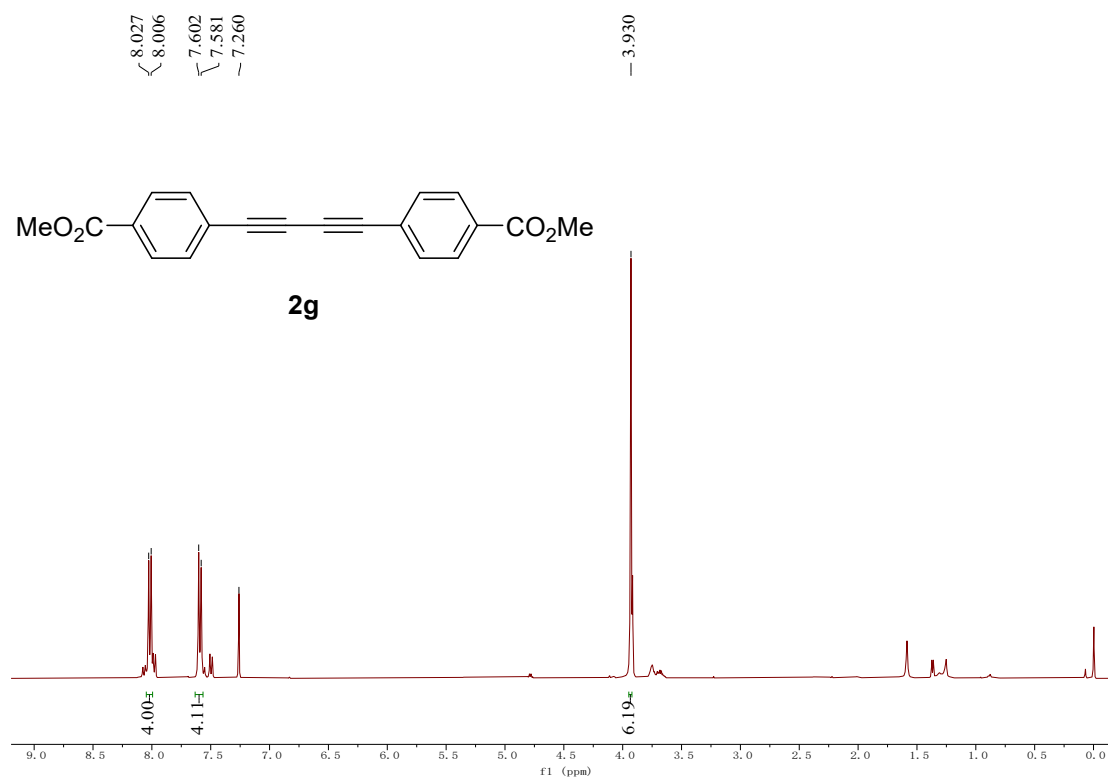
^1H NMR, 400 MHz, CDCl_3



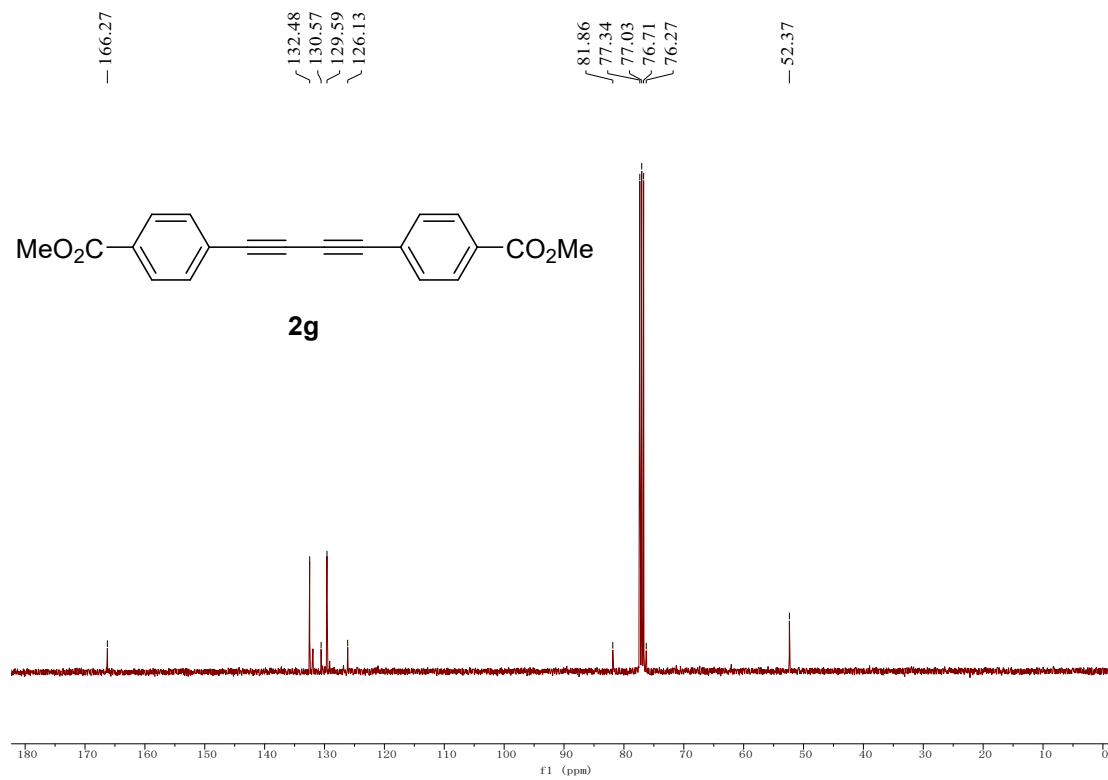
^{13}C NMR, 101 MHz, CDCl_3



^1H NMR, 400 MHz, CDCl_3

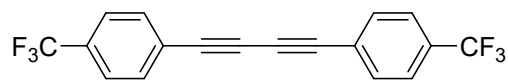


^{13}C NMR, 101 MHz, CDCl_3

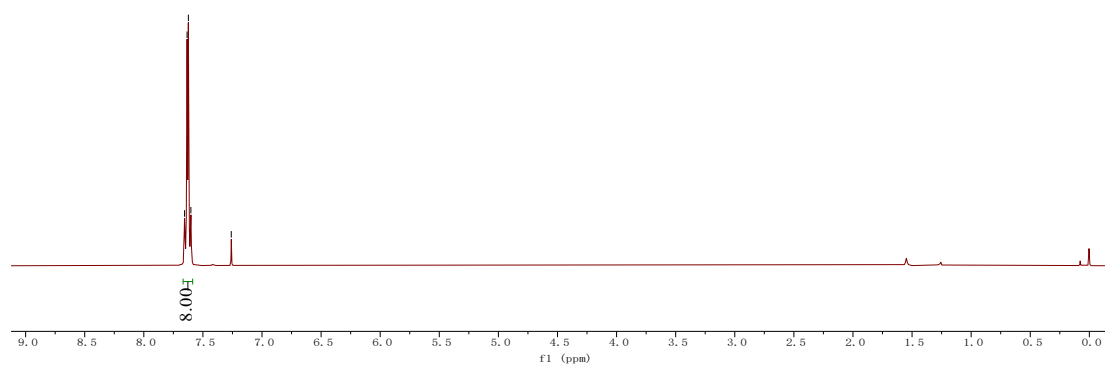


^1H NMR, 400 MHz, CDCl_3

7.655
7.634
7.623
7.602
7.260

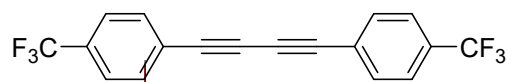


2h

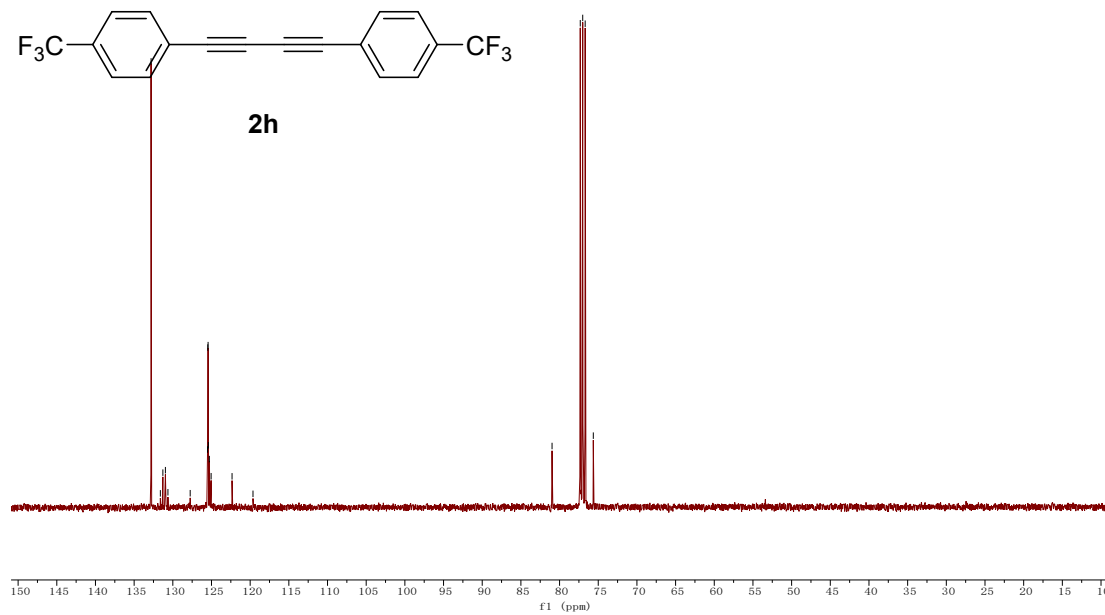


^{13}C NMR, 101 MHz, CDCl_3

132.81
131.61
131.29
130.96
130.63
127.75
125.52
125.48
125.44
125.40
125.28
125.05
122.34
119.64
80.97
77.32
77.01
76.69
75.64



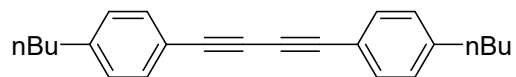
2h



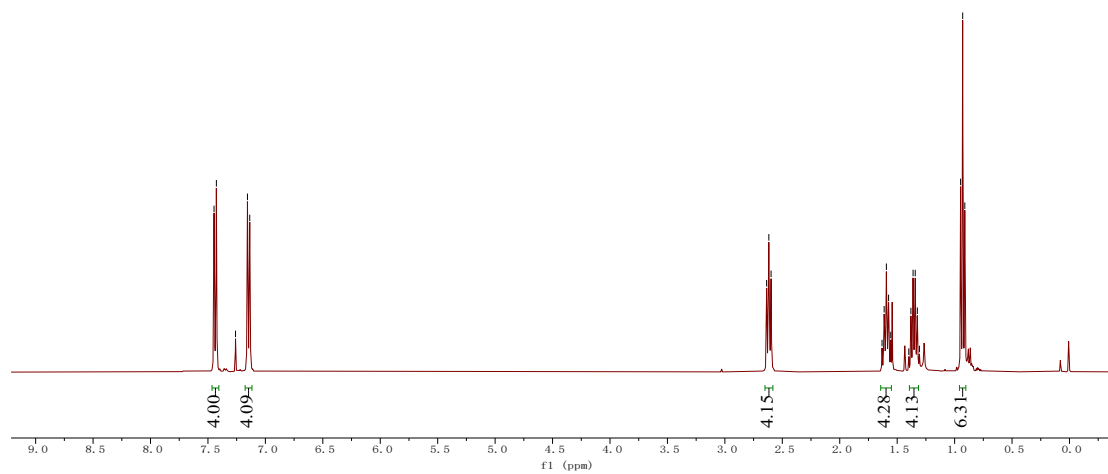
^1H NMR, 400 MHz, CDCl_3

7.447
7.427
7.260
7.156
7.136

2.636
2.617
2.598
1.632
1.613
1.594
1.575
1.556
1.399
1.380
1.362
1.343
1.324
1.306
0.948
0.930
0.911



2i

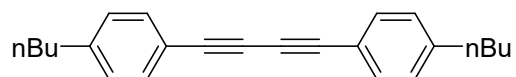


^{13}C NMR, 101 MHz, CDCl_3

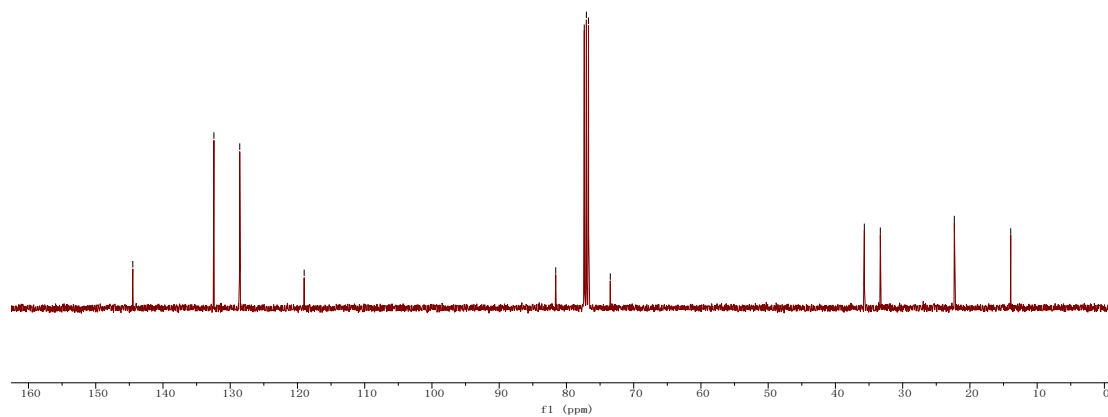
144.48
132.42
128.58
119.00

81.59
77.35
77.03
76.71
73.48

35.70
33.32
22.32
13.93



2i



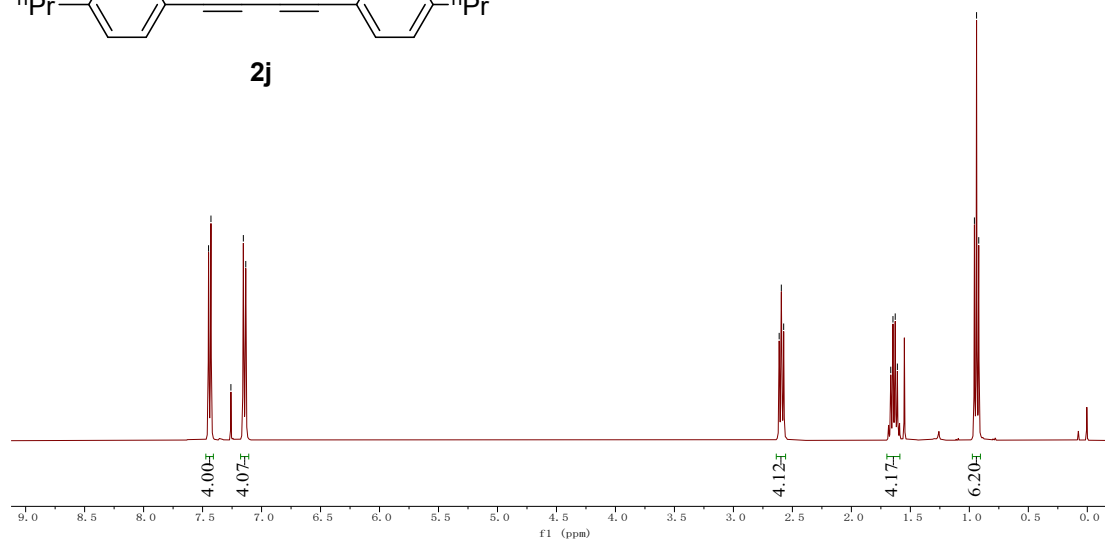
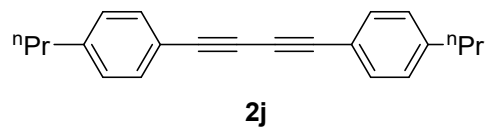
^1H NMR, 400 MHz, CDCl_3

7.449
7.429
7.260
7.154
7.134

2.612
2.594
2.574

1.666
1.648
1.628
1.610

0.957
0.939
0.920



^{13}C NMR, 101 MHz, CDCl_3

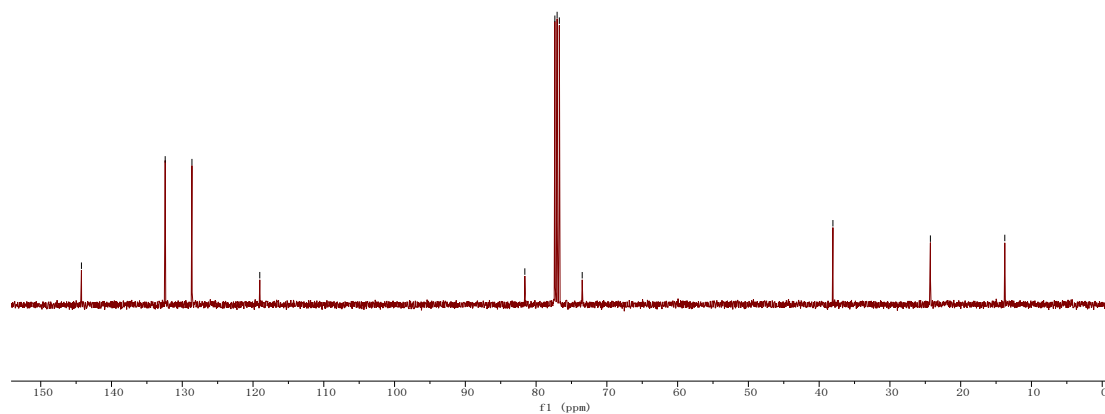
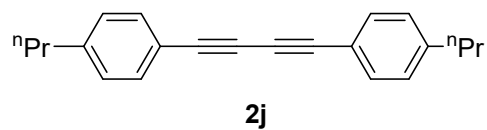
144.25
132.41
128.63
119.05

81.59
77.35
77.03
76.71
73.48

38.06

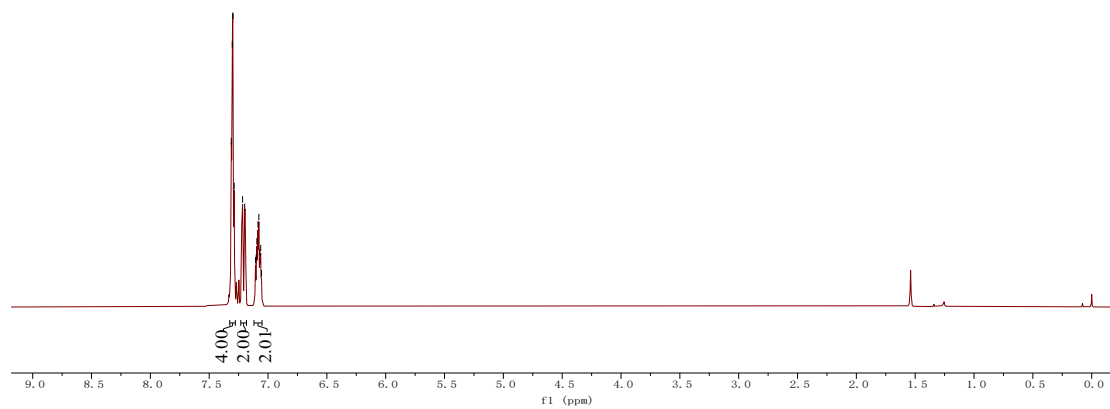
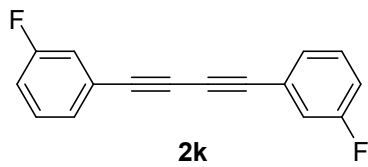
24.28

13.77



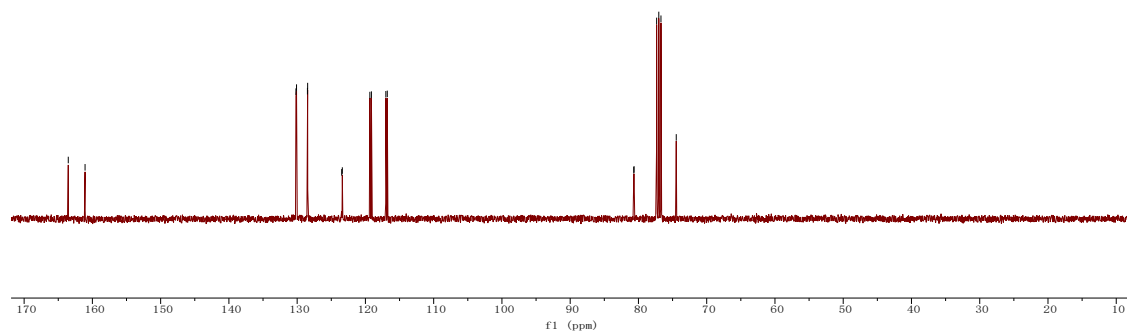
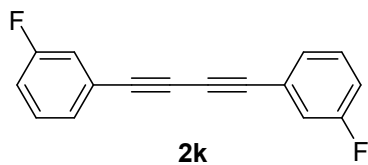
^1H NMR, 400 MHz, CDCl_3

7.313
7.304
7.299
7.288
7.216
7.198
7.107
7.100
7.095
7.085
7.078
7.070
7.062
7.055



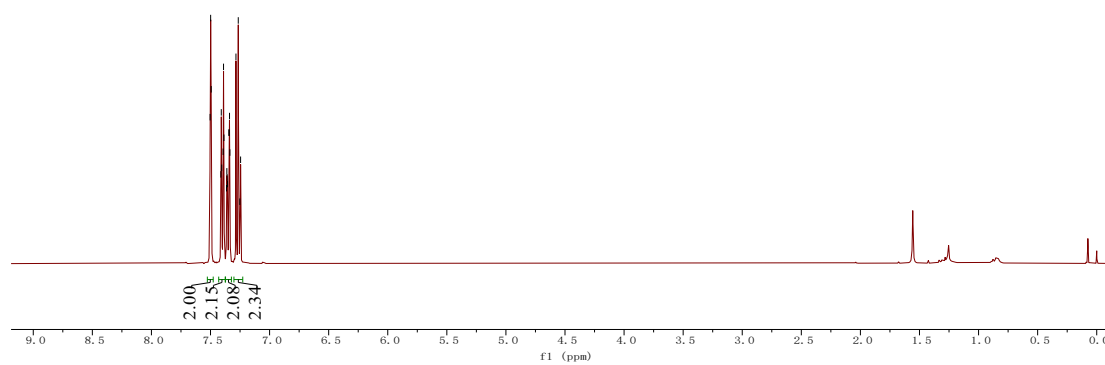
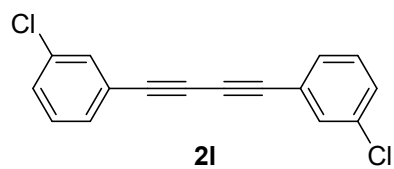
^{13}C NMR, 101 MHz, CDCl_3

163.53
161.07
130.18
130.09
128.50
128.46
123.48
123.38
119.35
119.12
117.01
116.80
80.68
80.65
77.34
77.02
76.70
74.46



¹H NMR, 400 MHz, CDCl₃

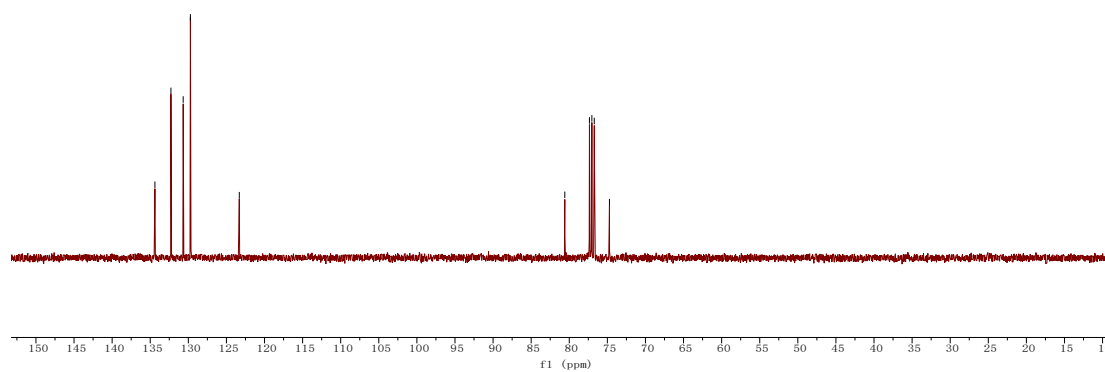
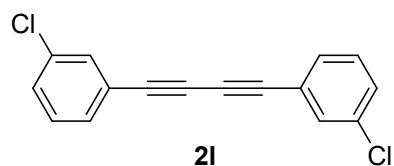
7.503
7.499
7.494
7.412
7.409
7.405
7.393
7.390
7.386
7.365
7.362
7.359
7.356
7.344
7.342
7.339
7.336
7.285
7.265
7.252
7.245



¹³C NMR, 101 MHz, CDCl₃

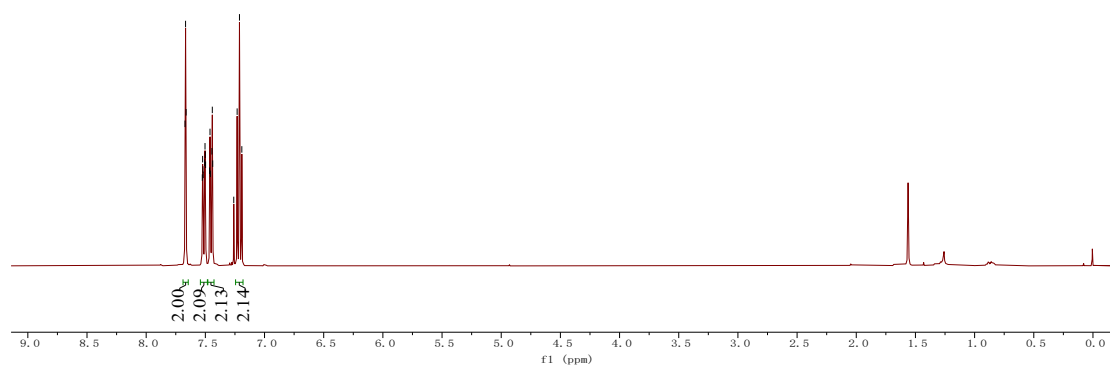
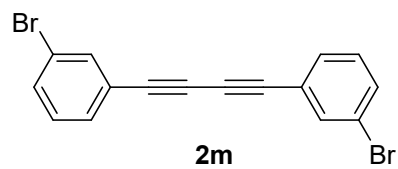
134.39
132.28
130.67
129.73
123.31

80.59
77.35
77.03
76.72
74.73



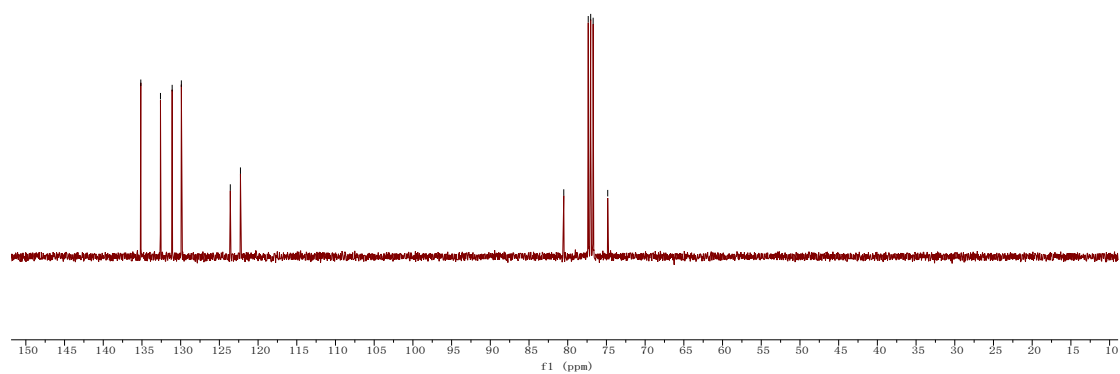
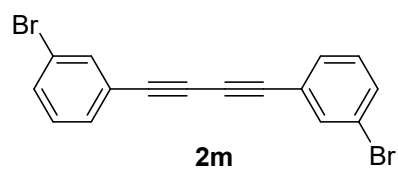
^1H NMR, 400 MHz, CDCl_3

7.672
7.667
7.663
7.526
7.523
7.521
7.518
7.506
7.503
7.501
7.498
7.464
7.461
7.457
7.445
7.441
7.438
7.260
7.231
7.212
7.192



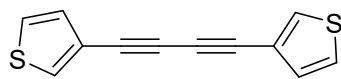
^{13}C NMR, 101 MHz, CDCl_3

135.15
132.61
131.10
129.91
123.59
122.28
80.51
77.35
77.03
76.72
74.83

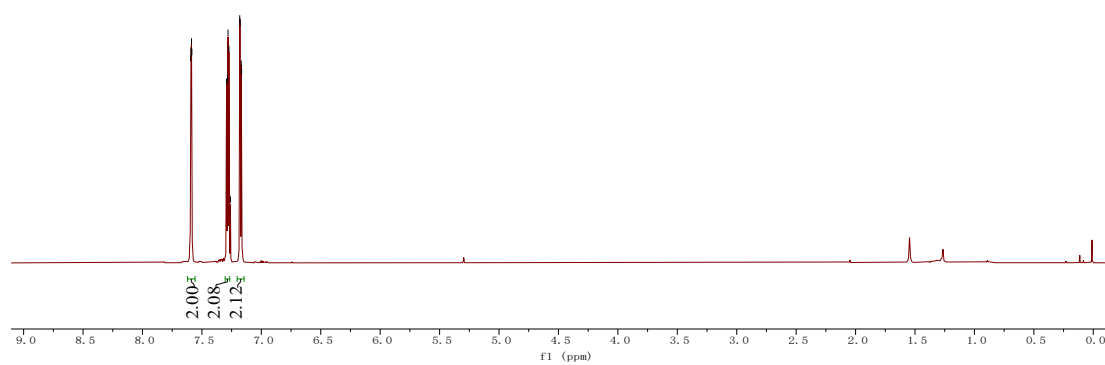


^1H NMR, 400 MHz, CDCl_3

7.595
7.592
7.587
7.584
7.293
7.285
7.280
7.273
7.260
7.181
7.178
7.169
7.166



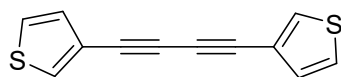
2n



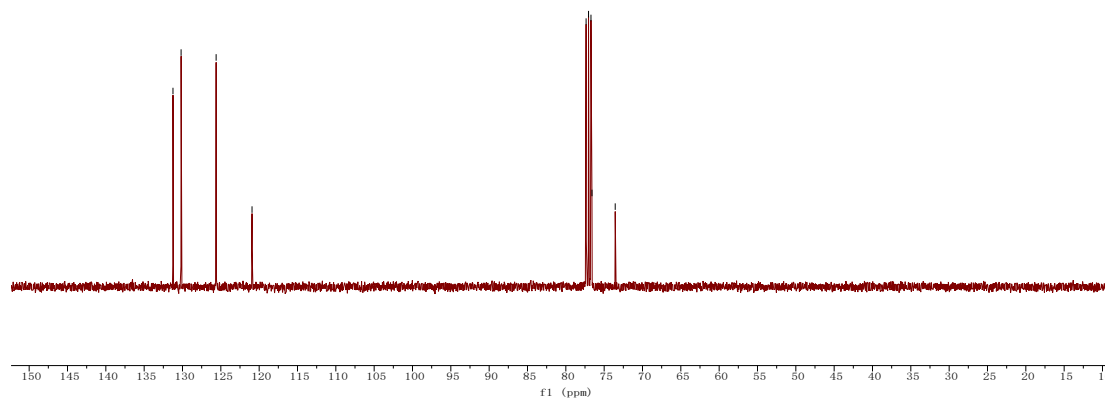
^{13}C NMR, 101 MHz, CDCl_3

131.24
130.18
125.61
120.93

77.36
77.04
76.72
76.59
73.56

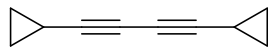


2n

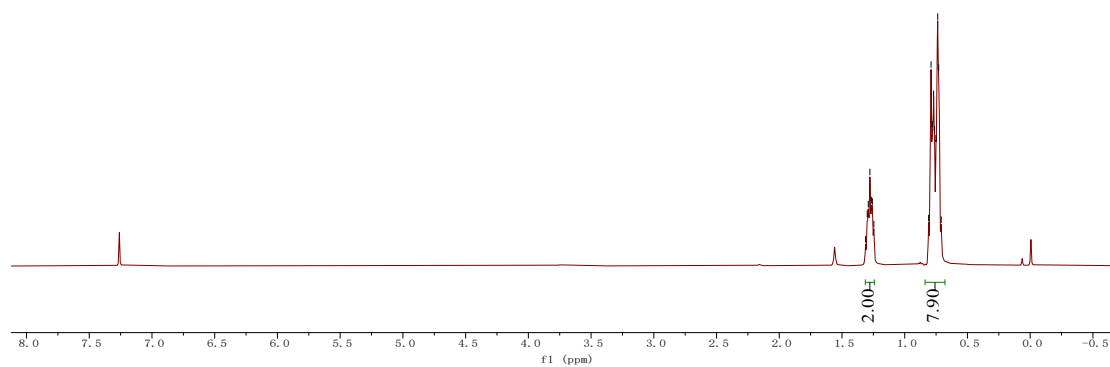


^1H NMR, 400 MHz, CDCl_3

1.312
1.299
1.290
1.277
1.266
1.258
1.246
0.809
0.790
0.780
0.770
0.762
0.751
0.737
0.729
0.709



2o



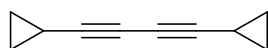
^{13}C NMR, 101 MHz, CDCl_3

80.05
77.35
77.03
76.71

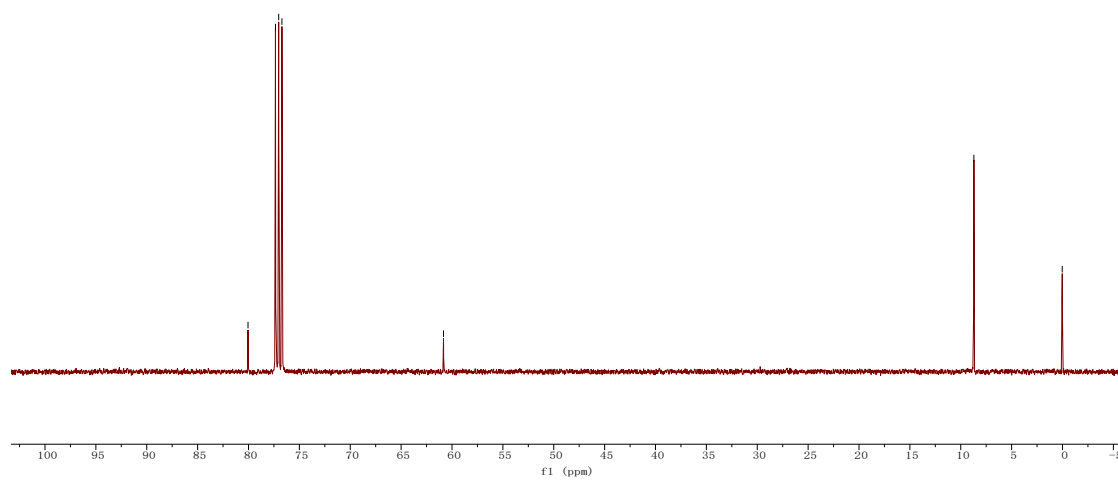
60.83

8.71

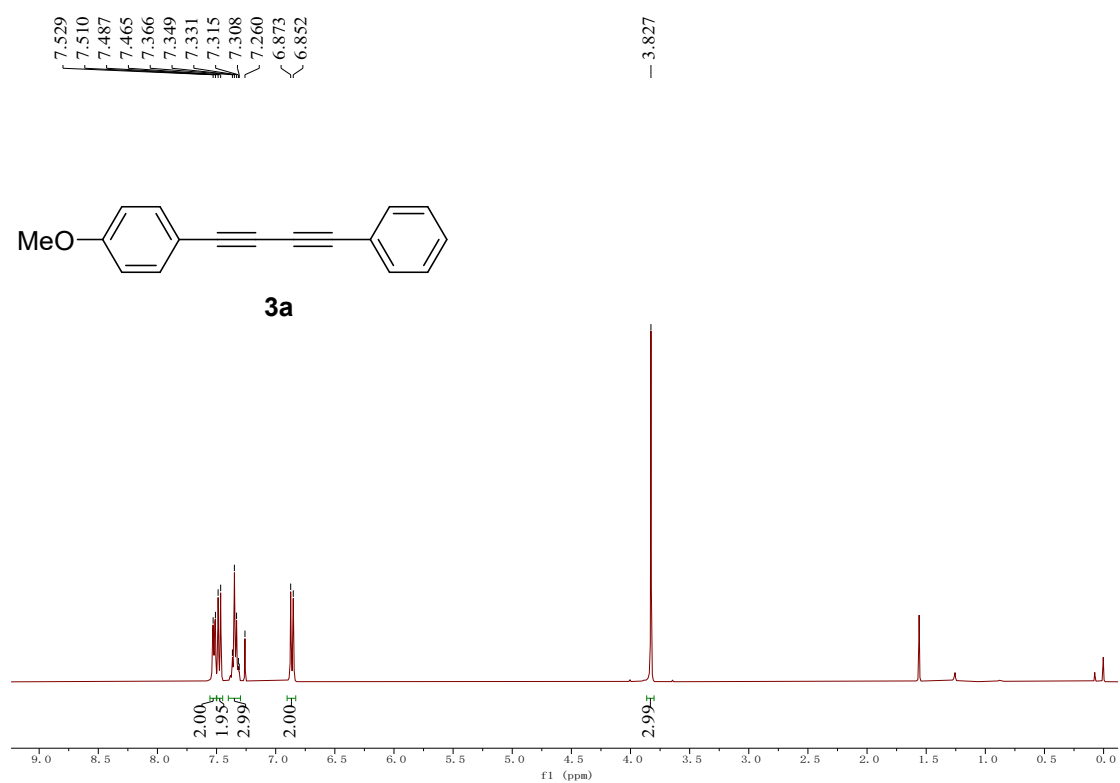
0.03



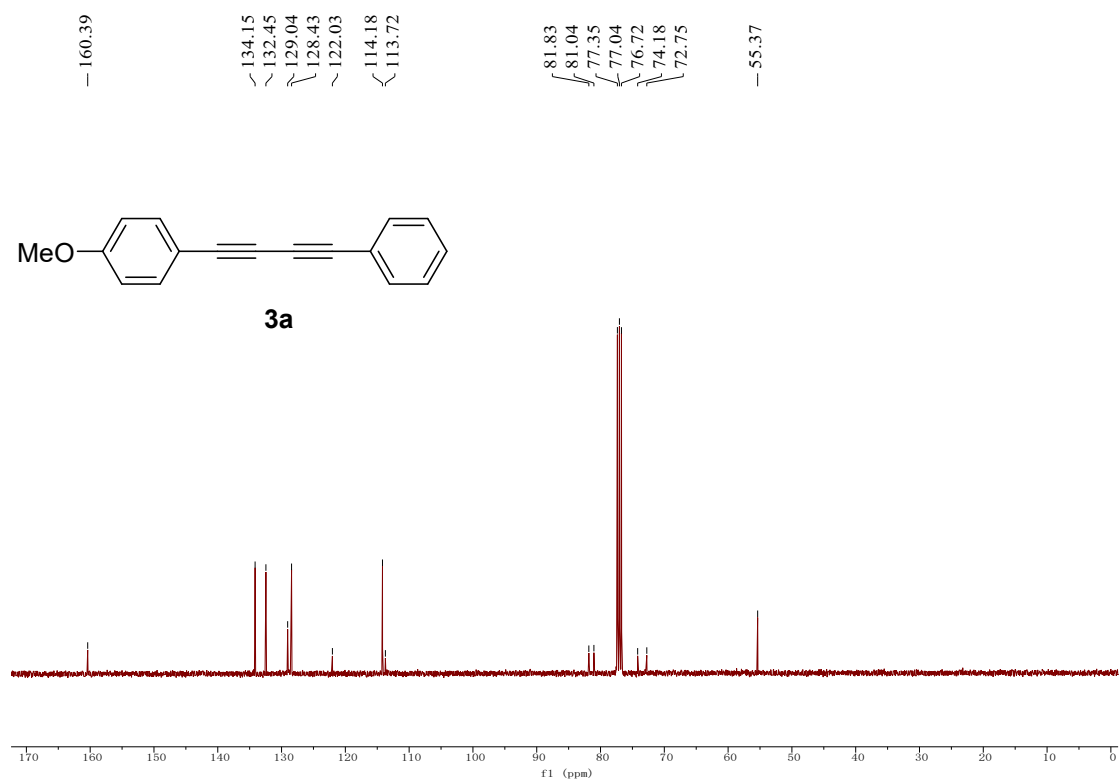
2o



^1H NMR, 400 MHz, CDCl_3



^{13}C NMR, 101 MHz, CDCl_3

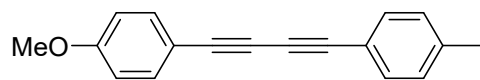


^1H NMR, 400 MHz, CDCl_3

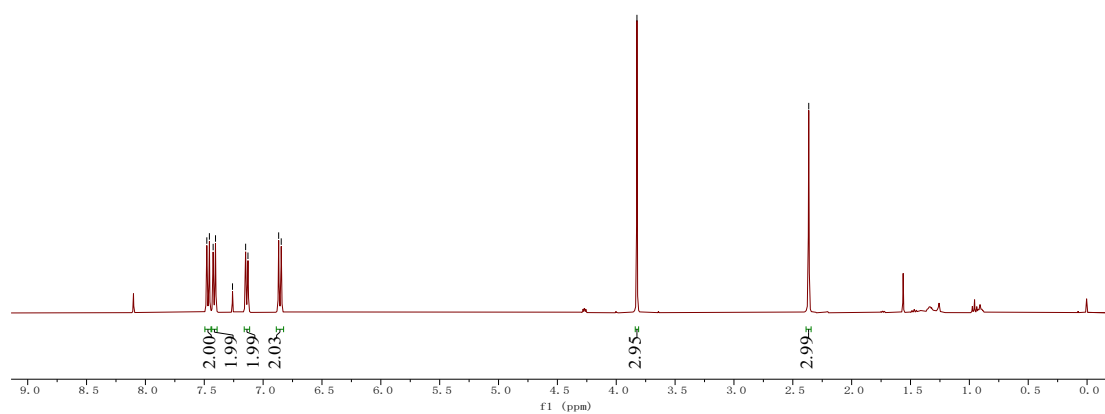
7.478
7.456
7.425
7.404
7.260
7.149
7.129
6.868
6.846

3.824

2.365



3b



^{13}C NMR, 101 MHz, CDCl_3

160.31

139.42

134.10

132.37

129.22

118.89

114.16

113.85

81.48

81.34

77.36

77.04

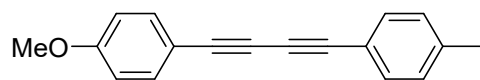
76.72

73.55

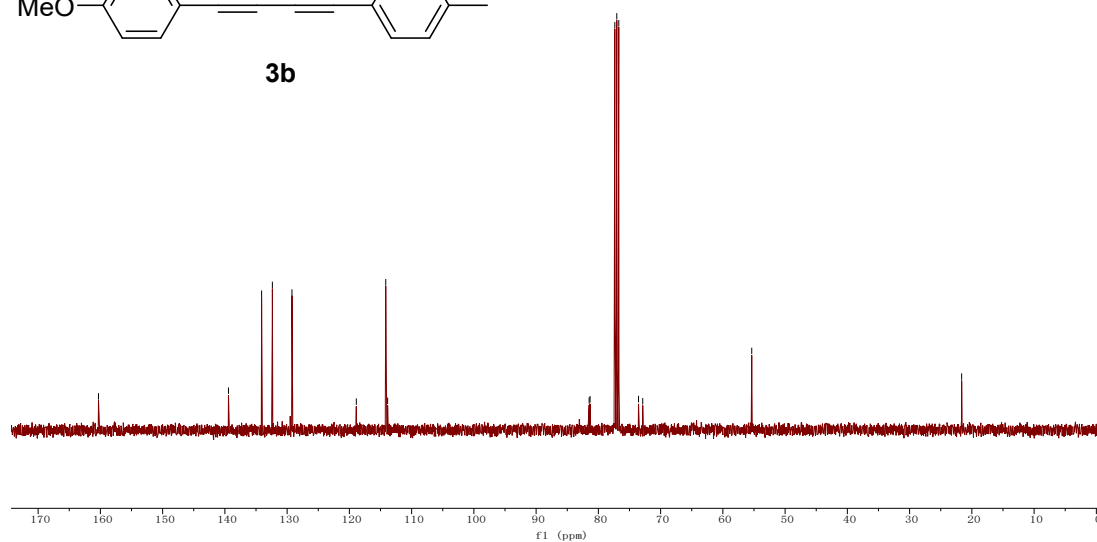
72.87

55.36

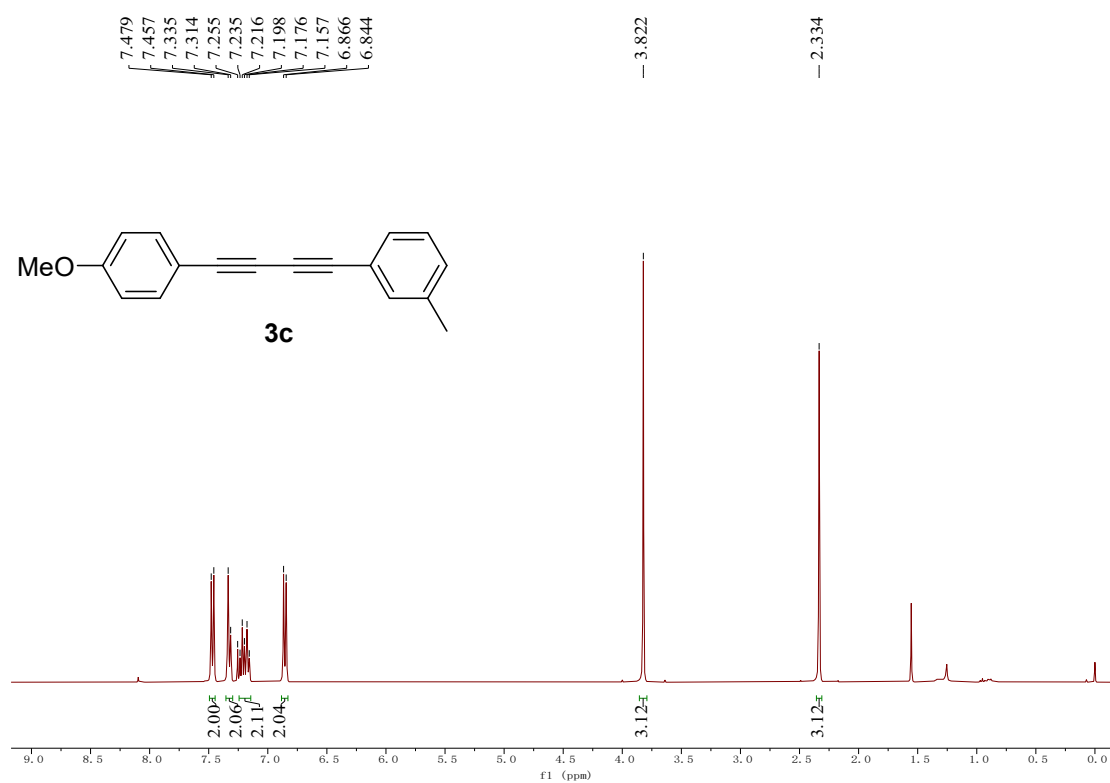
21.64



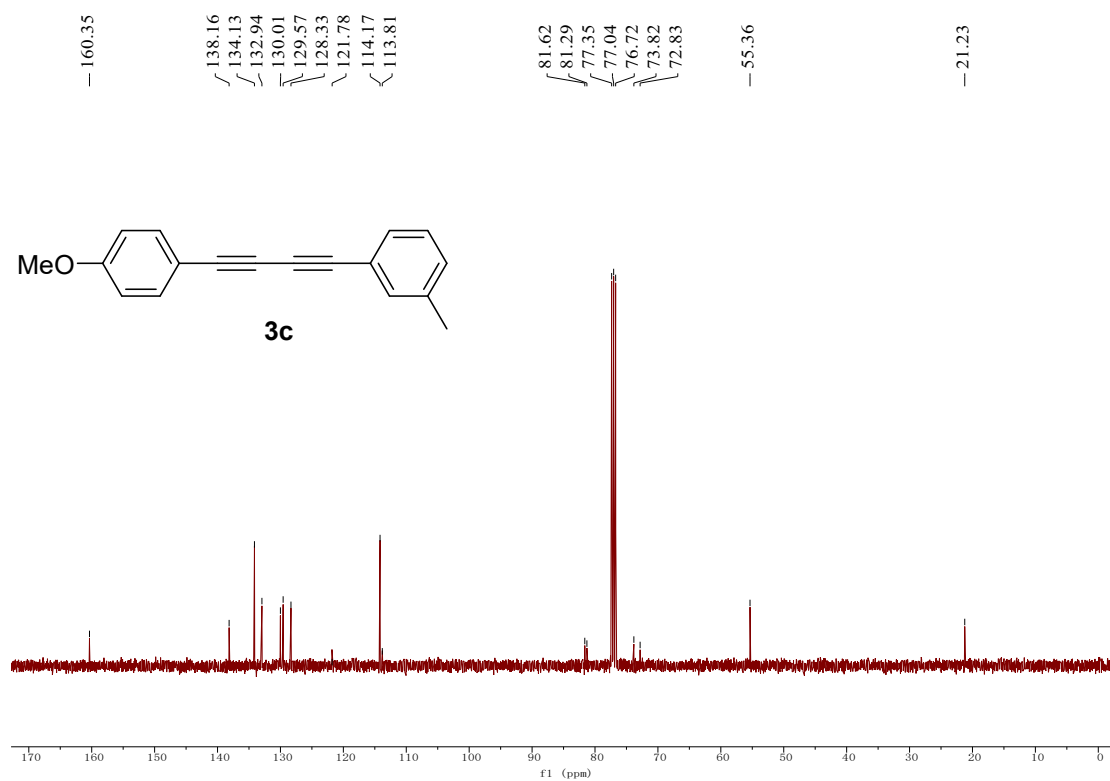
3b



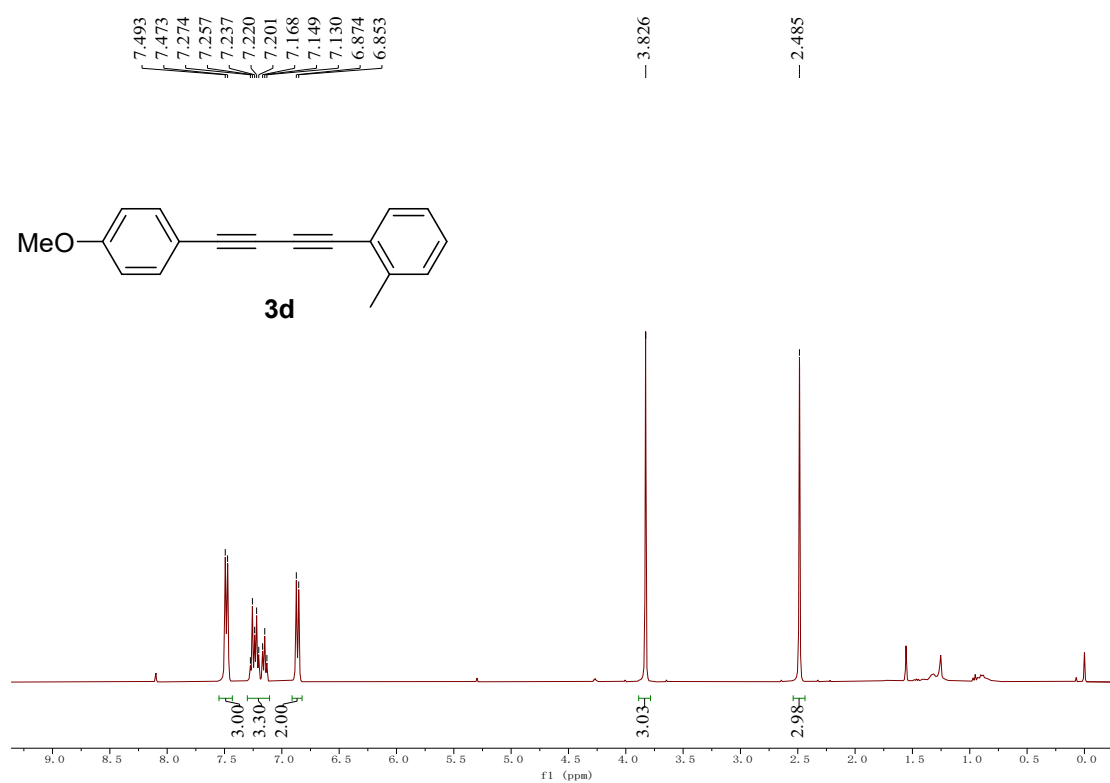
^1H NMR, 400 MHz, CDCl_3



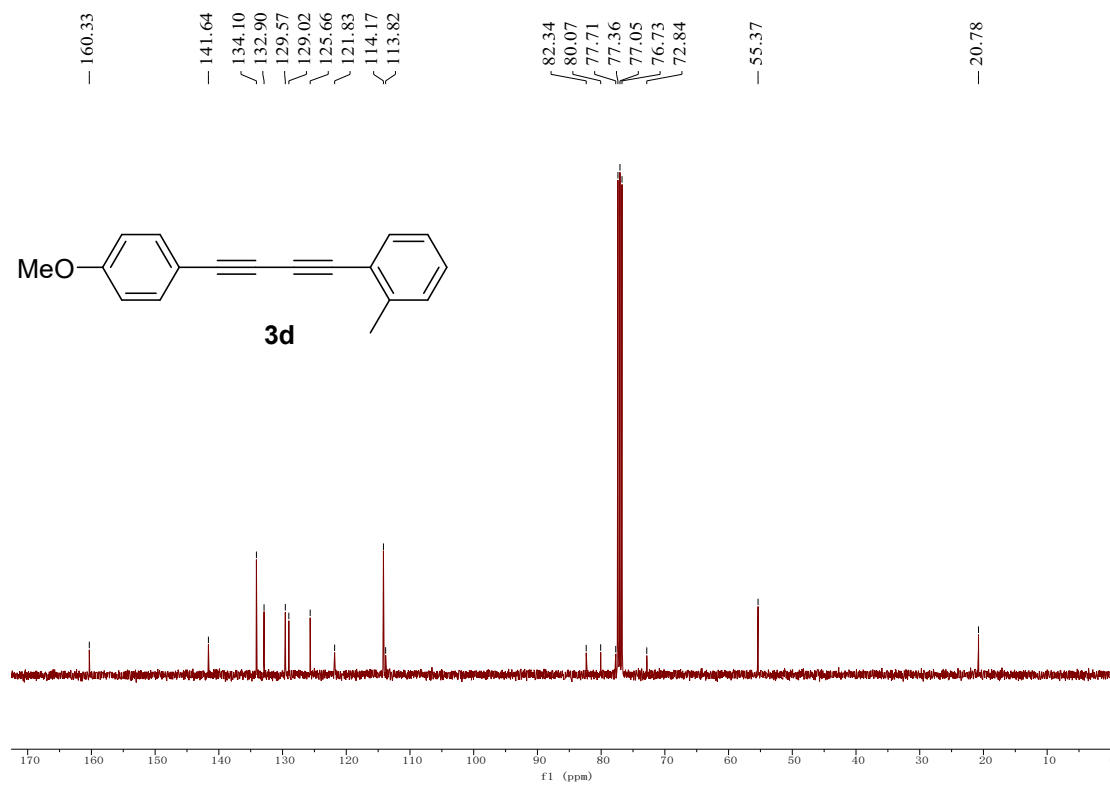
^{13}C NMR, 101 MHz, CDCl_3



^1H NMR, 400 MHz, CDCl_3



^{13}C NMR, 101 MHz, CDCl_3



^1H NMR, 400 MHz, CDCl_3

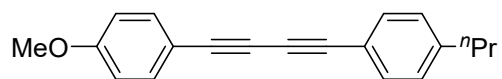
7.480
7.458
7.444
7.424
7.260
7.151
7.132
6.868
6.847

3.825

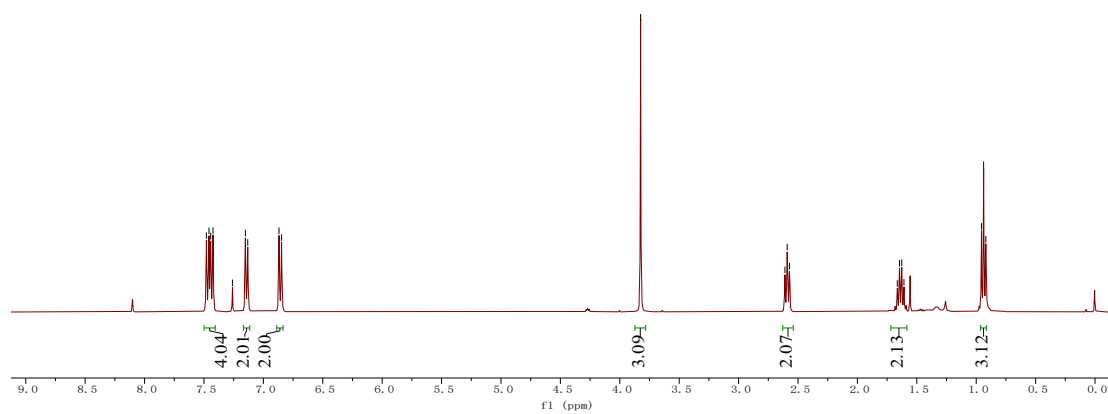
2.610
2.591
2.572

1.664
1.646
1.627
1.608

0.956
0.938
0.919



3e



^{13}C NMR, 101 MHz, CDCl_3

160.30
144.17
134.10
132.38
128.63
119.12
114.16
113.87

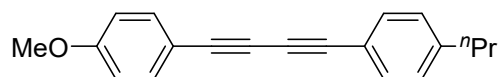
81.46
81.39
77.35
77.04
76.72
73.54
72.91

55.36

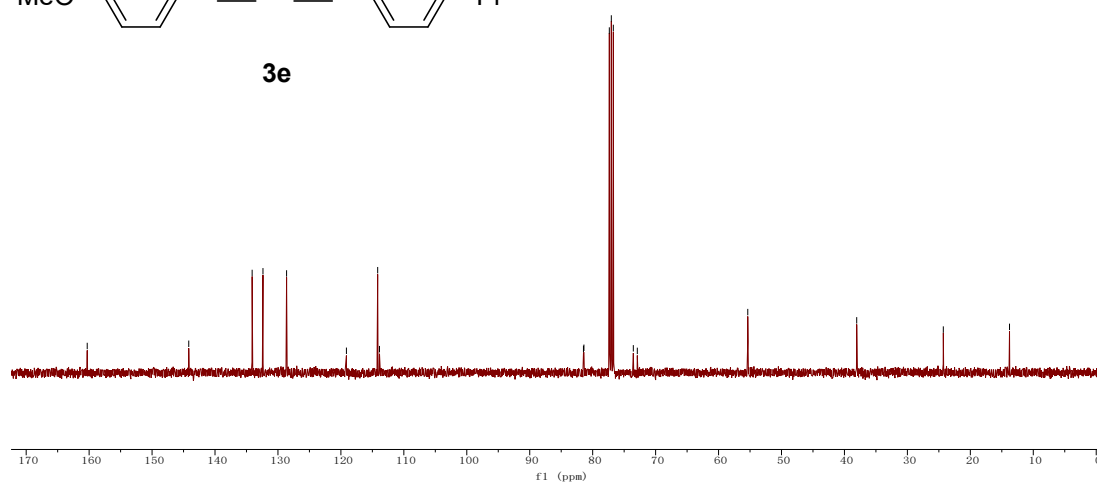
38.05

24.29

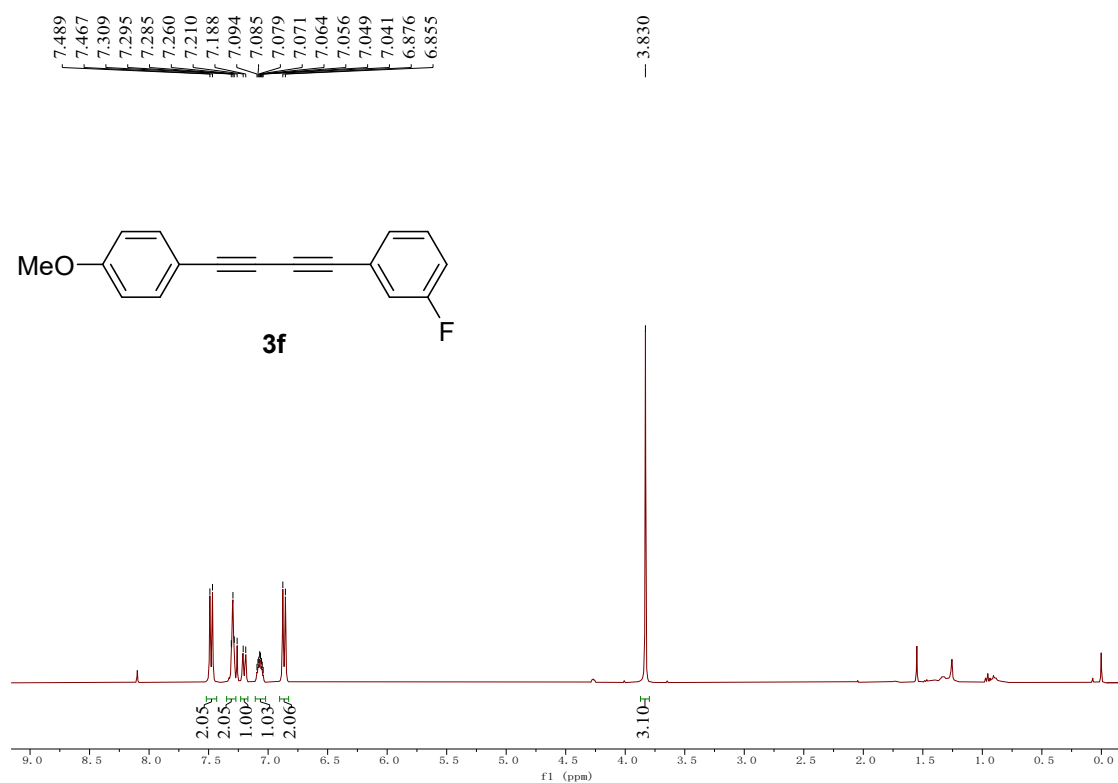
13.78



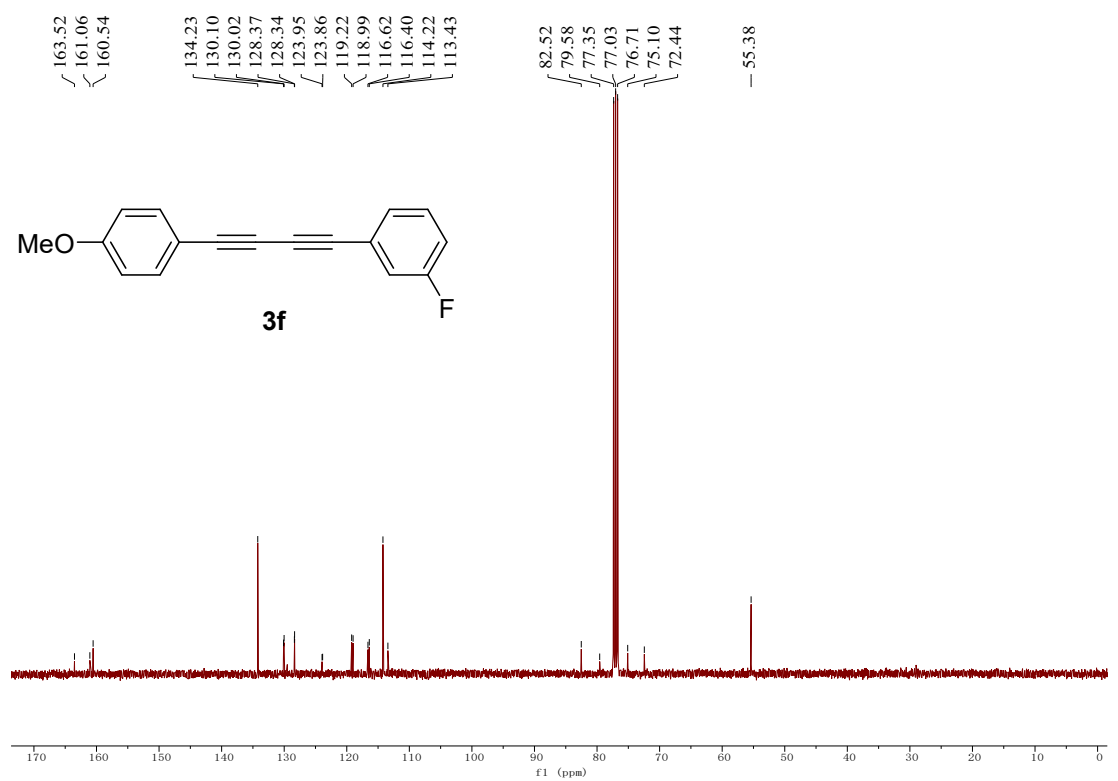
3e



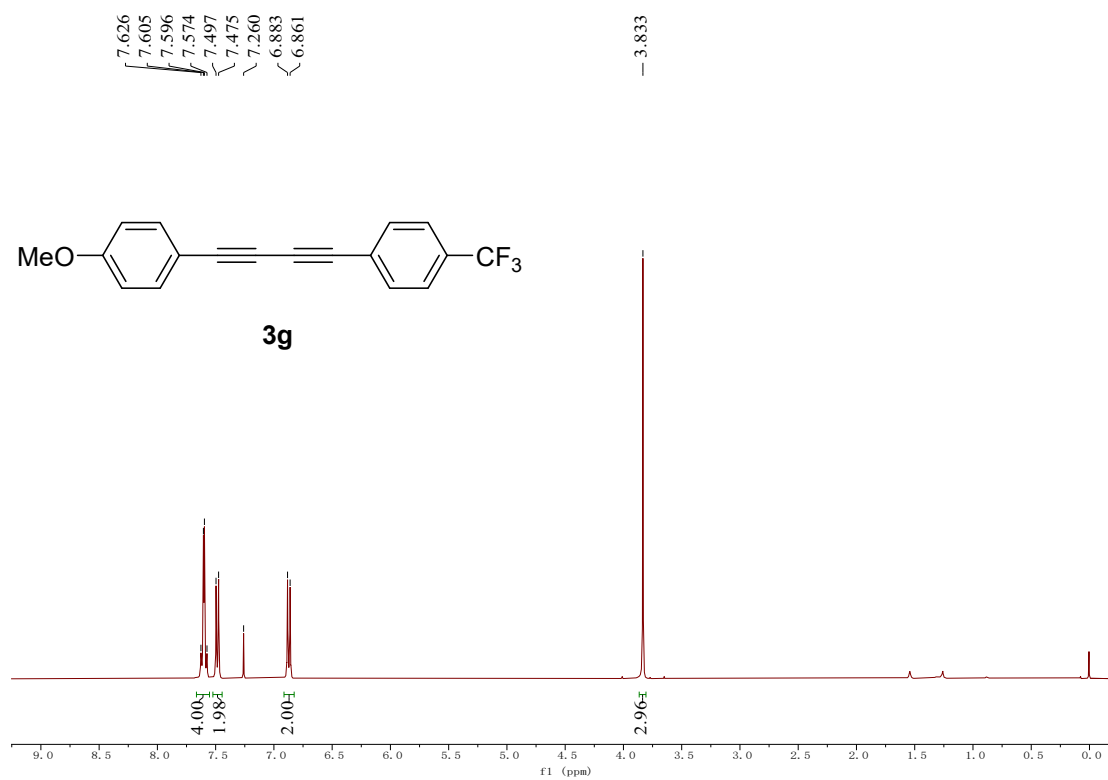
¹H NMR, 400 MHz, CDCl₃



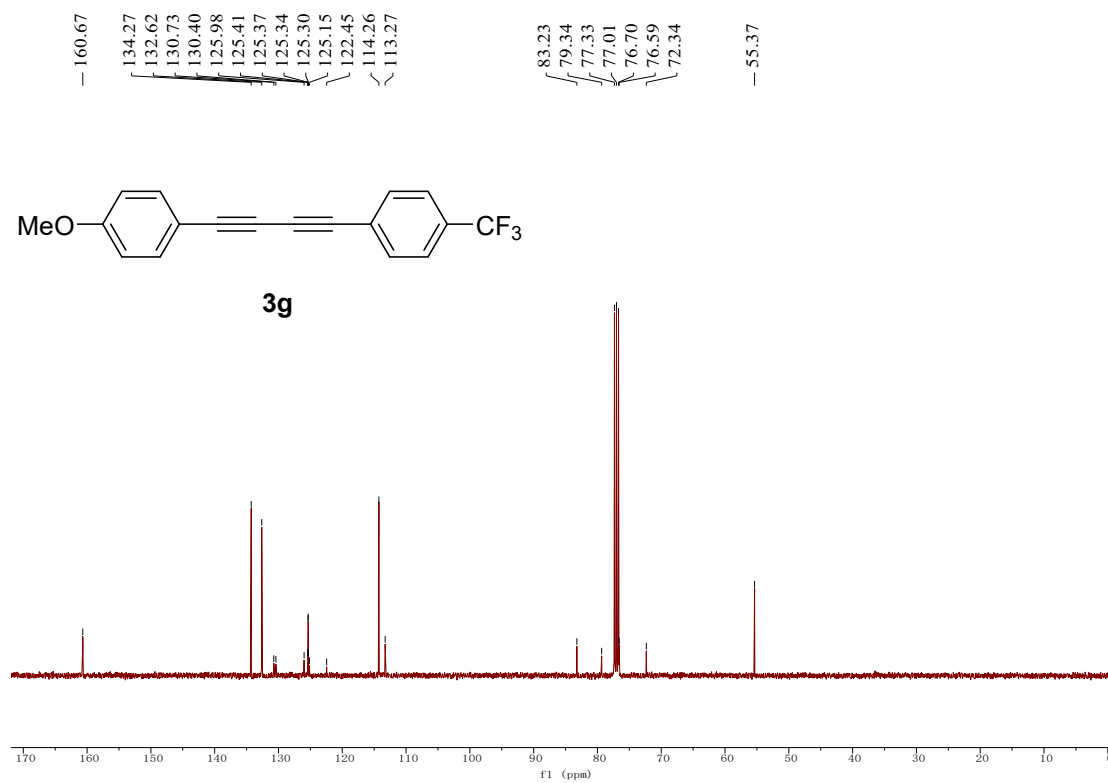
¹³C NMR, 101 MHz, CDCl₃



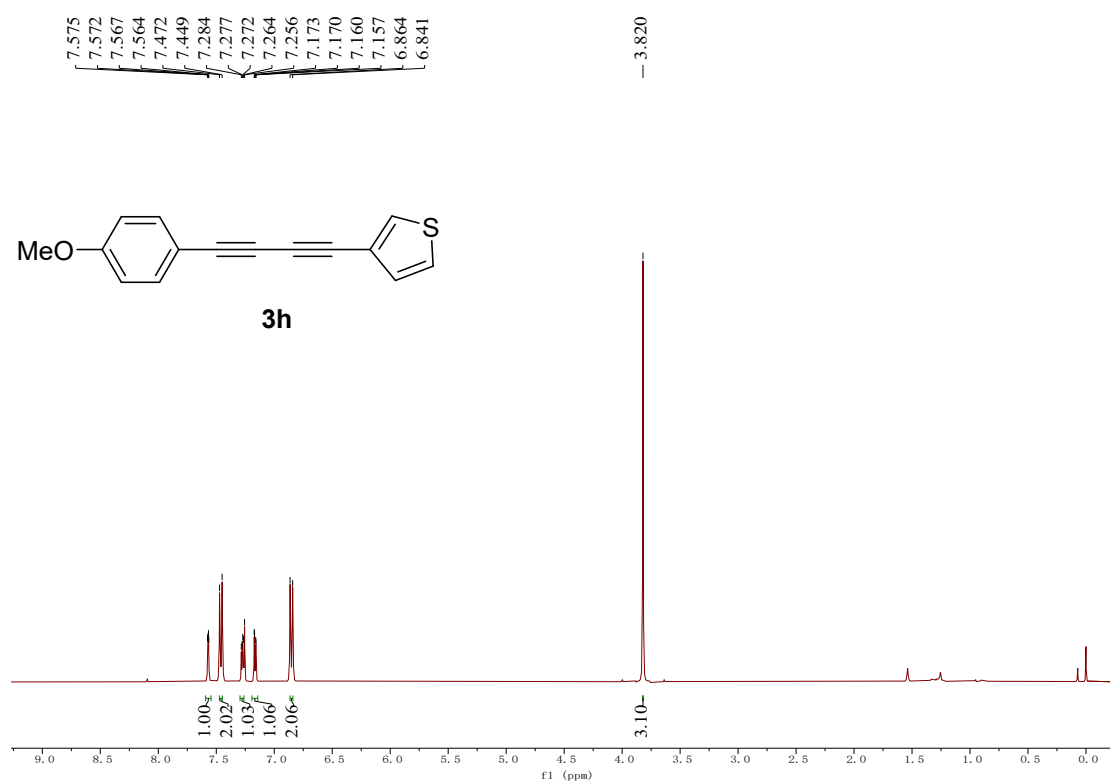
^1H NMR, 400 MHz, CDCl_3



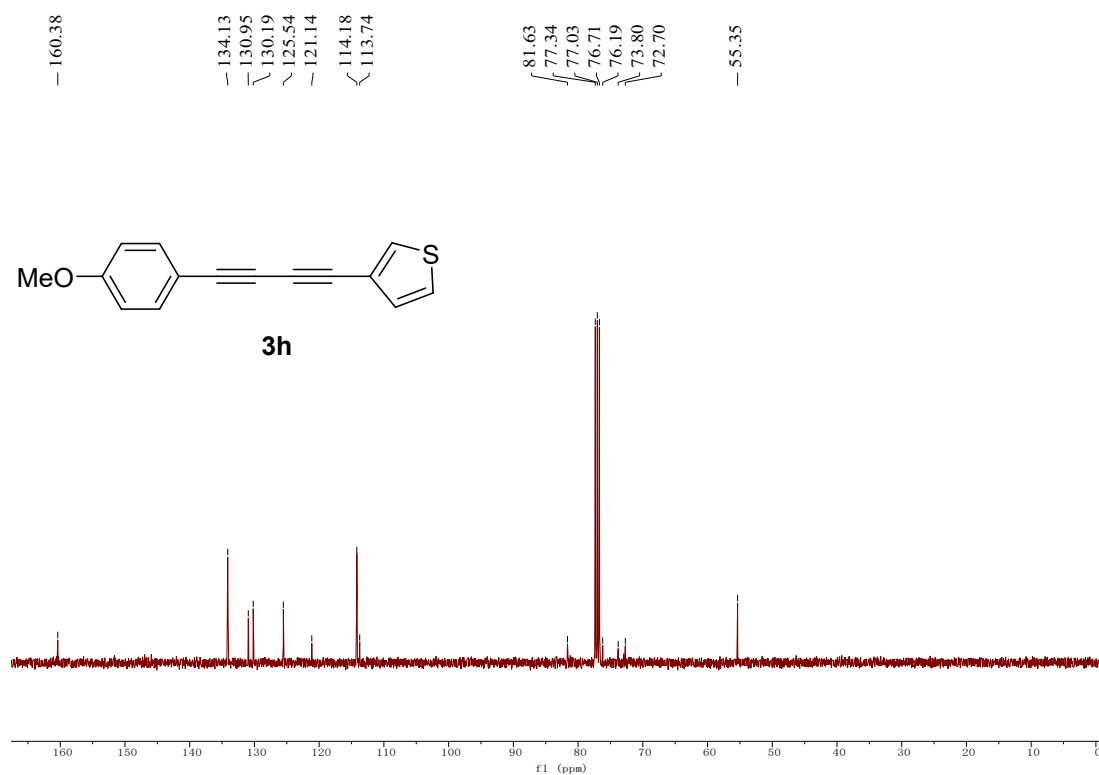
^{13}C NMR, 101 MHz, CDCl_3



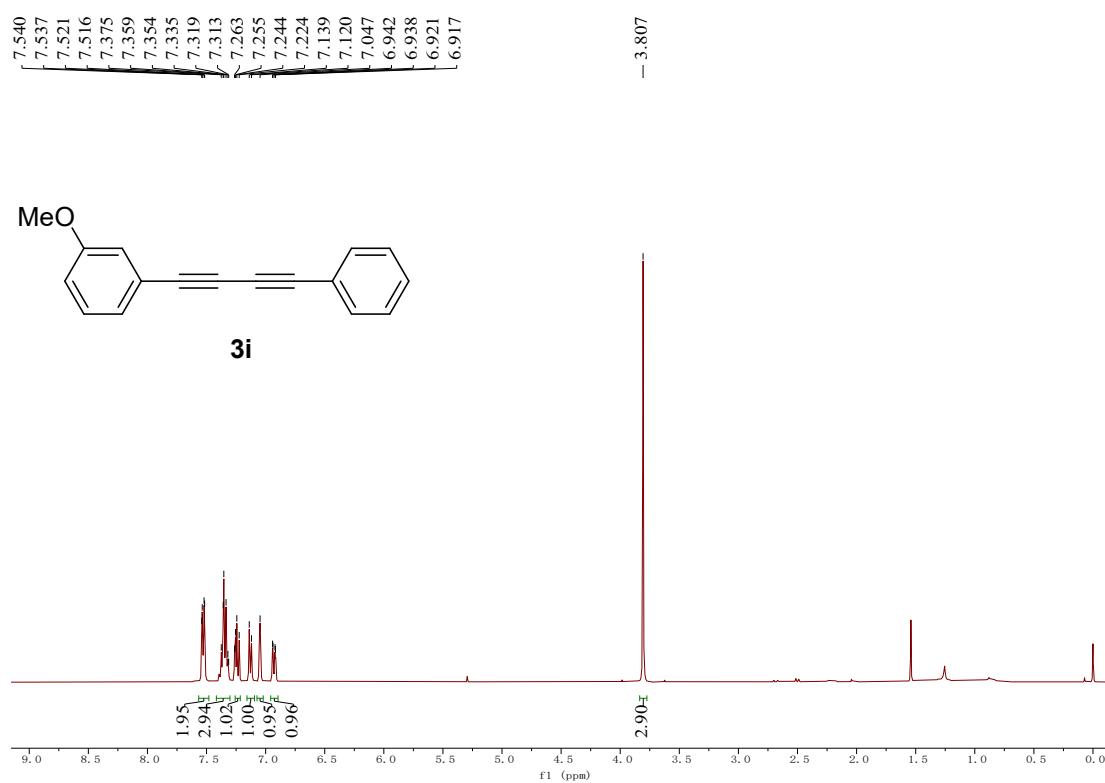
^1H NMR, 400 MHz, CDCl_3



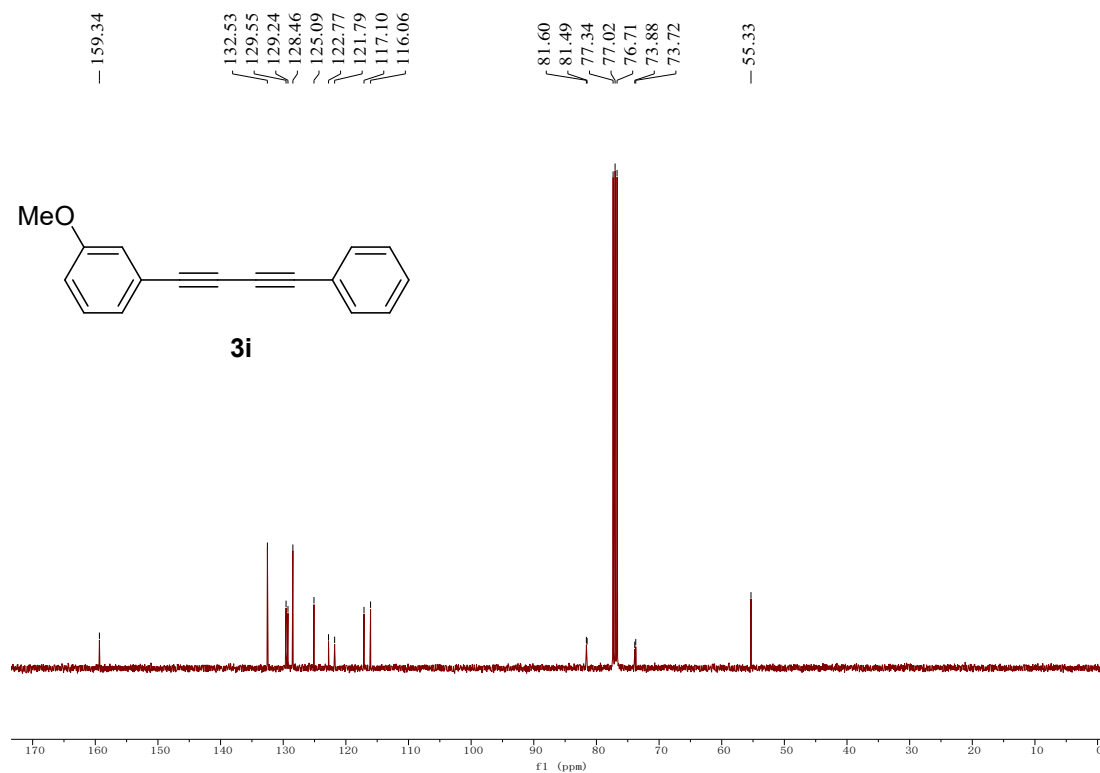
^{13}C NMR, 101 MHz, CDCl_3



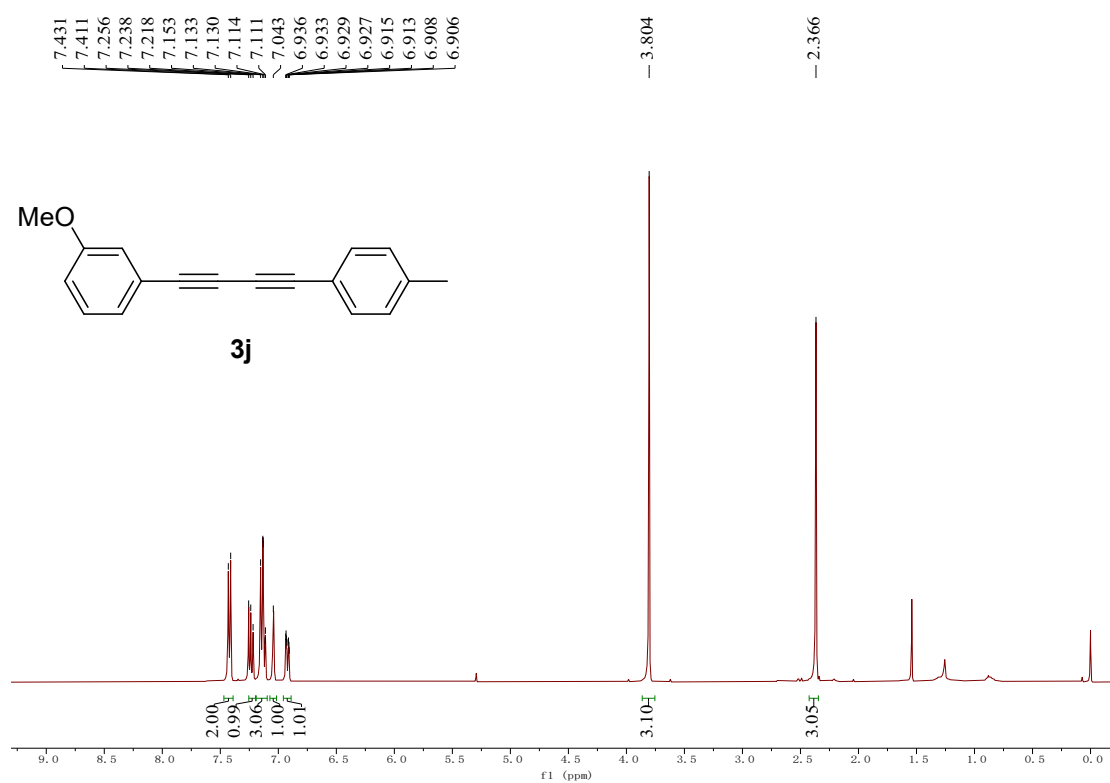
^1H NMR, 400 MHz, CDCl_3



^{13}C NMR, 101 MHz, CDCl_3



¹H NMR, 400 MHz, CDCl₃



¹³C NMR, 101 MHz, CDCl₃

