Supporting Information

# Visible-light-activated Copper(I) catalyzed Oxidative $C_{sp}$-$C_{sp}$ Cross-Coupling Reaction: Efficient Synthesis of Unsymmetrical Conjugated Diynes without Ligands and Base

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**Table of contents**

## Experimental section

*General:* All reactions were conducted under 1 atm. of an oxygen atmosphere and oven-dried glass wares were used. All reactions were conducted using a blue light-emitting diode (LED) as the visible-light source (30 lamps, power density: 40 mW/cm$^2$ at 460 nm). All solvents were dried according to the known methods, and distilled prior to use. Starting materials (including starting materials for synthesis of epoxide hydrolase inhibitors) were commercially available (Sigma-Aldrich, Alfa-Aesar or TCI-chemicals) and used as received. NMR spectra were recorded $^1$H NMR at 400 MHz and $^{13}$C NMR at 100 MHz using deuterated $CDCl_3$ as a solvent. Chemical shifts (δ) were reported as parts per million (ppm). The following abbreviations were used to identify the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, b = broad and all combinations thereof can be explained by their integral parts. Unless otherwise specified, the proton/carbon signal of 2 residual solvent (at δ 7.24 and δ 77.00 ppm, respectively) was used as the internal reference.

***General procedure:***

A dry test tube (20 mL) with rubber septum and magnetic stirrer bar was charged with 1.2 -1.4 mmol of **1a** (0.6-0.7 M) terminal alkynes, 0.7 mmol of **2a (**0.35 M), and 5 mol% (relative to **1a**) CuCl is combined in $CH_3CN$ (2 mL). The mixture was irradiated with blue LEDs under an oxygen (balloon) atmosphere for hours until completion of heterocoupling reaction (it was determined by thin layer chromatography). The reaction mixture was diluted with 40 % ethyl acetate in hexane and stirred in for 10 min. The mixture was filtered through celite and silica gel pads, and washed with ethyl acetate. The filtrate was concentrated and the residue was purified by flash column chromatography on silica gel to afford the desired heterocoupling products.

**Evaluation of Green metrics of the process;**

Atom economy defined as “how much of the reactants remain in the final desired product"

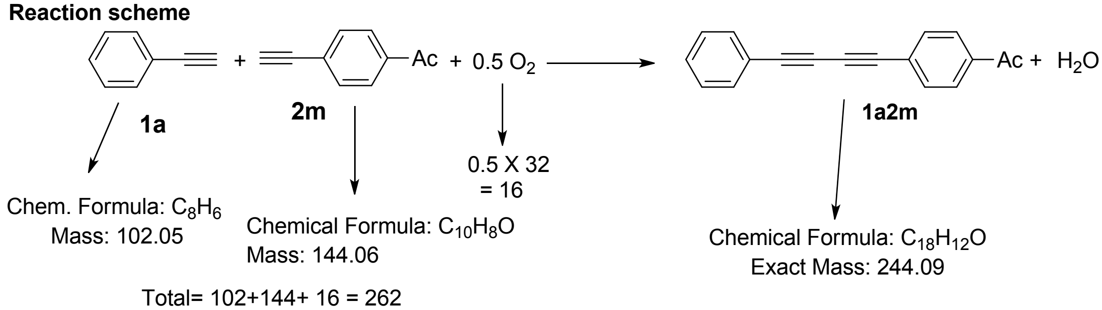
$$\text{Atom economy (AE)} = \frac{\text{Molecular mass of desired product}}{\text{Molecular mass of all reactants}} \times 100$$

Reaction mass efficiency (RME) defined as “the percentage of the mass of the reactants that remain in the product”

$$\text{Reaction mass efficiency (RME)} = \frac{\text{mass of desired product}}{\text{mass of all reactants}} \times 100$$

## a) Evaluation of Green metrics of the current photochemical process

**Reaction scheme**



**Evaluation of Green metrics of the current photochemical process**

| | | | | |
|---|---|---|---|---|
| Reactant A | phenylacetylene (**1a**) | 0.95g | 0.0093 mol | FW 102.05 |
| Reactant B | 1-(4-ethynylphenyl)ethanone (**2m**) | 0.75g | 0.0052 mol | FW 144.06 |
| Solvent | ACN | 11.79g | --- | --- |
| Auxiliary | --- | --- | --- | --- |
| Product | 1,3-diynes (**1a2m**) | 1.06g | 0.0043 mol | FW 244.09 |

**Product yield= 84%**

E-factor = $\frac{0.95\text{g} + 0.75\text{g} + 11.79\text{ g} - 1.06\text{g}}{1.06\text{ g}}$ = 11.7 kg waste/ 1 kg product

Atom economy = $\frac{244}{262} \times 100$ = 93%

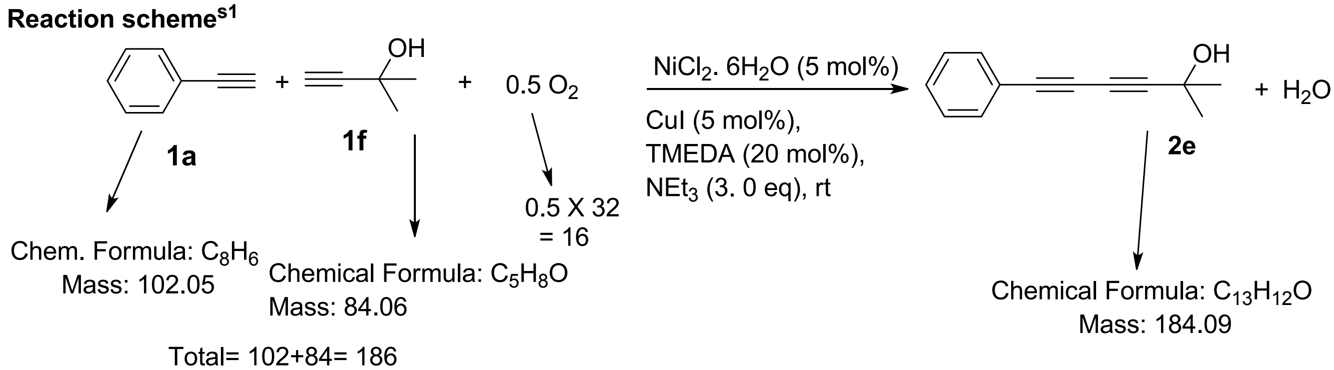
Atom efficiency = 84 % X 93% / 100 = 78%

Carbon efficiency = $\frac{18}{8 + 10} \times 100$ = 100%

Reaction mass efficiency = $\frac{1.06\text{ g}}{0.95\text{g} + 0.75\text{g}} \times 100$ = 63%

## b) Evaluation of Green metrics of the reported process [s1]

**Reaction scheme[s1]**



**Evaluation of Green metrics of the reported process**

| | | | | |
|---|---|---|---|---|
| Reactant A | phenylacetylene (**1a**) | 0.5g | 0.005mol | FW 102.05 |
| Reactant B | 2-methylbut-3-yn-2-ol (**2m**) | 0.084g | 0.001mol | FW 84.06 |
| Solvent | THF | 3.55g | --- | --- |
| Auxiliary | TMEDA | 0.023g | 0.0002 | 116.21 |
| Base | $NEt_3$ | 0.303g | 0.003 | 101.19 |
| Product | 1,3-diynes (**2e**) | 0.158g | 0.00086 mol | FW 184.09 |

**Product yield= 86%** TMEDA: *Tetramethylethylenediamine*

E-factor = $\frac{0.5g + 0.084g + 3.55g + 0.023g + 0.303g - 0.158g}{0.158\ g}$ = 27kg waste/ 1 kg product

Atom economy = $\frac{184}{186 + 101 + 23.2 + 16}$ X 100 = 56%

Atom efficiency = 86 % X 56% / 100 = 48%

Carbon efficiency = $\frac{13}{8 + 5}$ X 100 = 100%

Reaction mass efficiency = $\frac{0.158\ g}{0.5g + 0.084g}$ X 100 = 27%

***EPR measurements**:* EPR spectra were recorded at room temperature on a Bruker ESP-300E(X band, 9.8 GHz) with parameters setting as shown below: receiver gain= 30 n; receiver phase= 0 deg; receiver harmonic= 1; field modulation frequency= 100000 Hz; microwave frequency [Hz]= 9.660469 e+09; field modulation amplitude [T]= 0.00016; receiver time constant [S] = 0.32768; microwave power= 0.015 W; receiver offset [%FS]= 0; DMPO ( 5-,5-dimethyl-1-pyrroline N-oxide) was employed as a radical trap for trapping of the superoxide radical anion.

The reaction under an standard condition (**1a,** CuCl, 1 atm. $O_2$) in $CH_3CN$ was irradiated with blue LEDs for 30 min in the presence of DMPO in an EPR chamber while recording the EPR spectra. The EPR signals shown in Figure S1 is corresponding to DMPO-OO(H). This result indicates that superoxide anion radical was formed in the reaction solution. No superoxide EPR signal was observed from the reaction solution under standard condition in the absence of CuCl (Fig. S2). These results indicate that copper(I) phenylacetylide undergoes single electron transfer to $O_2$, and generate superoxide free radical upon blue LEDs irradiation.

**EPR spectra of the reaction mixture after blue LEDs irradiation**

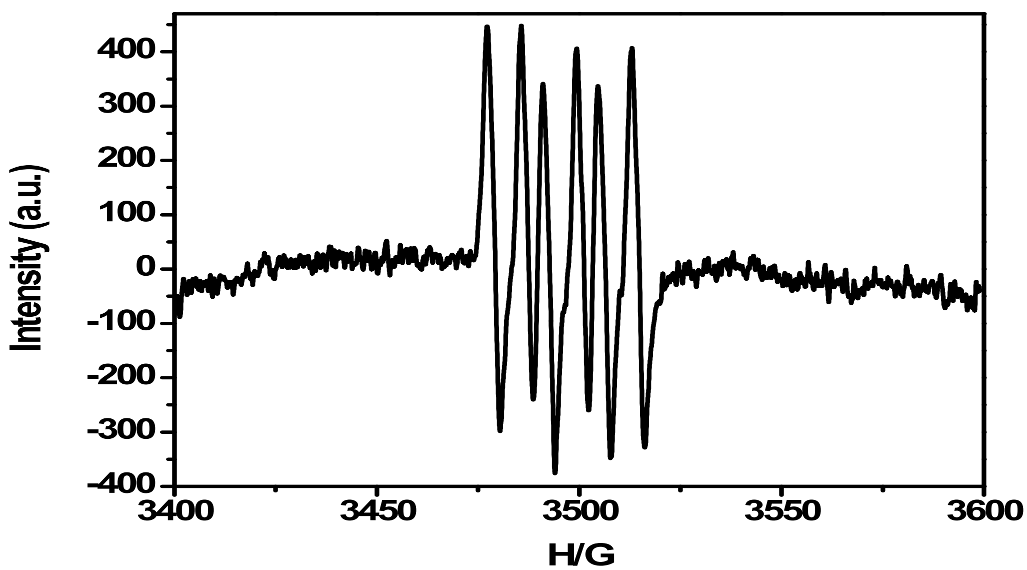



**Figure S1:** EPR spectra of the reaction mixture: phenylacetylene(**1a**) (0.6 M), and 5 mol% of CuCl in $CH_3CN$, 0.5 ml of this reaction solution was taken out into a small vial, followed by the addition of 0.01 ml of DMPO (5 x $10^{-2}$ M). The mixture was irradiated with blue LEDs at room temperature under an oxygen atmosphere (1 atm) for 30 minutes. The reaction mixture was then analysed by EPR spectra. There are classical 6 peaks, the signals corresponding to (DMPO-OO(H)).

**EPR spectra of the reaction mixture in the absence of CuCl**

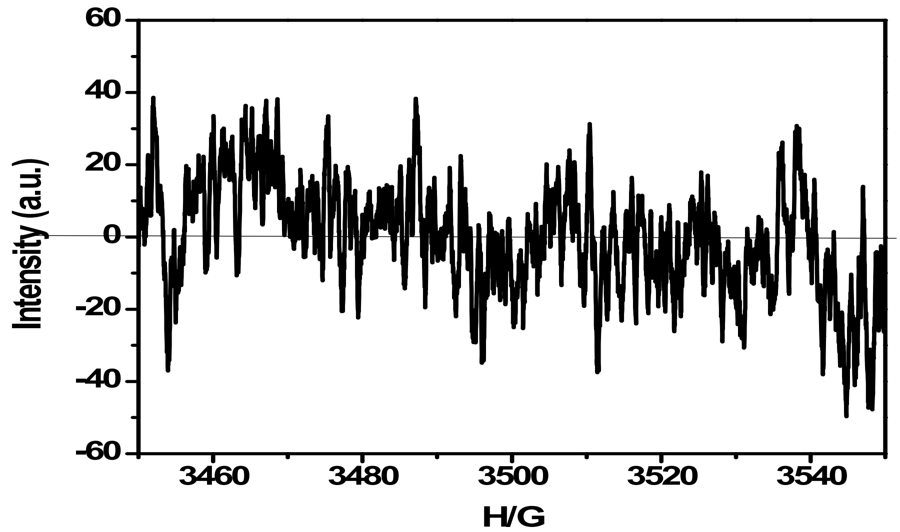



**Figure S2:** EPR spectra of the reaction mixture: phenylacetylene(**1a**) (0.6 M), 0.5 mL of this reaction solution was taken out into a small vial, followed by the addition of 0.01 mL of DMPO (5 x $10^{-2}$ M). The mixture was irradiated with blue LEDs at room temperature under an oxygen atmosphere (1 atm) for 30 minutes (in the absence of CuCl). The reaction mixtures was analysed by EPR spectra. No signals were detected.

**Estimation of association constant using isothermal titration calorimetry (ITC):**

**Experiment 1:** Isothermal titration caloriemetry experiments were carried out at 25 $^{o}$ C on a high precision ITC-200 (MicroCal, LLC, and Northampton, MA). The solution of copper(I) phenylacetylide (Cu(I)-**1a**, 1 mM) and (1-(4-ethynylphenyl) ethanone, **2m**) (2 mM) were prepared by using $CH_3CN$ as a solvent. Before the measurements, samples were degassed for at least 7 minutes. The calorimeter was initially calibrated using water-water titration, in which the reference power of 5 µcal/s was applied. As a control experiment, solvent-to-solvent titration was also performed. Then, the Cu(I)-**1a** solution was loaded into the cell and **2m**-acetonitrile solution was taken in the syringe. 20 injections were performed with an each titration volume of 2 µL. The reference power of 5 µcal/s was applied while the sample contents were stirred at 1000 rpm (rotations per minute).

The binding curve is obtained from a plot of the heat change from each injection against the molar ratio of **2m** (in syringe) and binding partner Cu(I)-phenylacetylide (Cu(I)-**1a**) in the cell (Fig. S1). The binding curve is analyzed with an appropriate binding model to determine the value of K (binding affinity). The isothermal titration reveals the association constant, $K_a$~ 199 $\mu M^{-1}$ and this affinity value suggests a moderate extent of interaction.

Likewise, a **1a**-acetonitrile solution was titrated into a Cu(I)-**1a** acetonitrile solution. No clear complex formation was observed from the ITC measurements in this case (see, results shown in supplementary Figure S4).

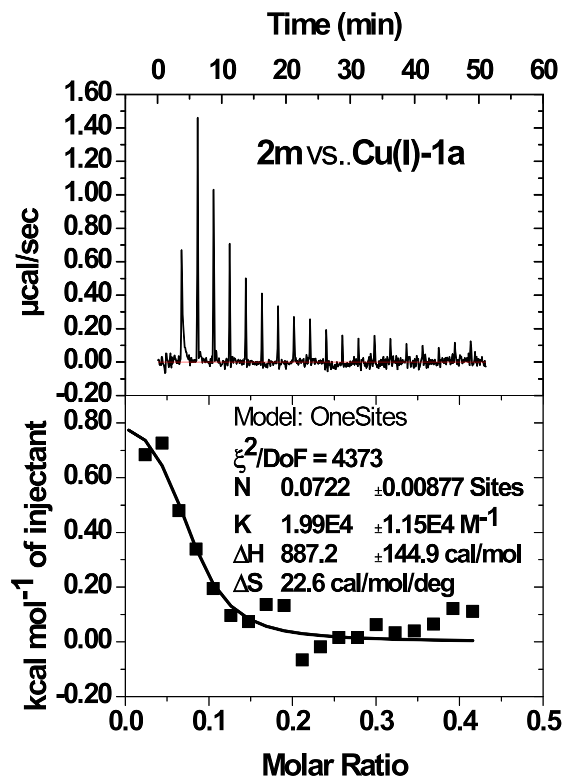



**Figure S3**. Isothermal titration calorimetry (ITC) data for the determination of the association constant values for combination Cu(I)-phenylacetylide (Cu(I)-**1a**) and **2m** (1-(4-ethynylphenyl) ethanone). The inset of the bottom panel indicates the peak fitting results of one set of binding sites obtained from the inbuilt Origin Pro software of the Mircrocal ITC-200.

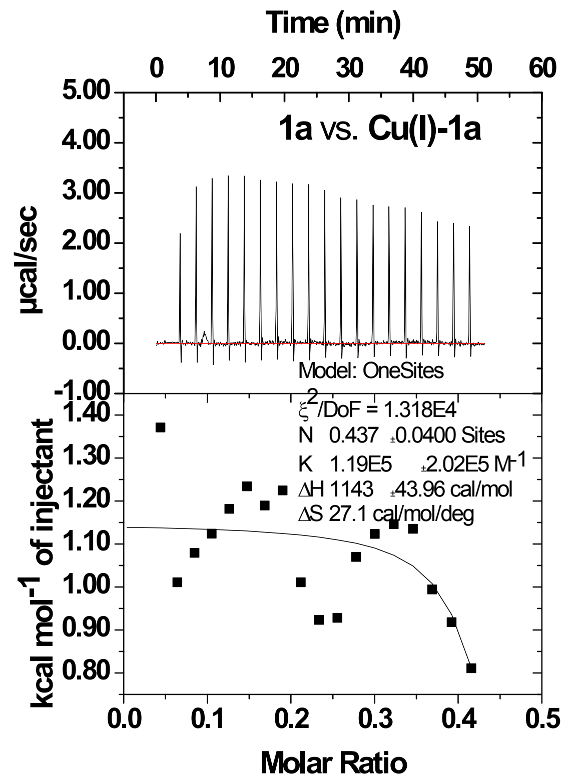
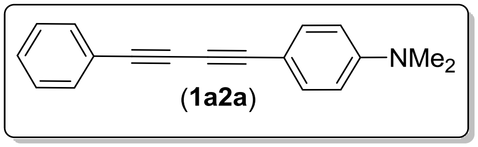



**Figure S4**. Isothermal titration calorimetry (ITC) data for the determination of the association constant values for combination copper(I) phenylacetylide (Cu(I)-**1a**) and phenylacetylene (**1a)**. The inset of the bottom panel indicates the peak fitting results of one set of binding sites obtained from the inbuilt Origin Pro software of the Mircrocal ITC-200

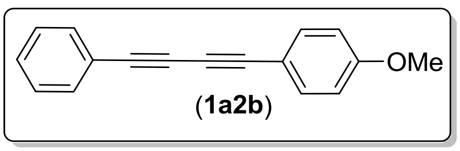
**Spectroscopic data**

**N, N-dimethyl-4-(phenylbuta-1, 3-diyn-1-yl) aniline (1a2a)**[s2]



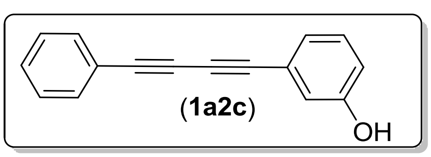
Pale yellow solid; m.p.= 110-114˚C; $^{1}$**H NMR** (400 MHz, $CDCl_3$): δ 7.50-7.48 (m, 2 H), 7.40-7.38 (m, 2 H), 7.32-7.30 (m, 3 H), 6.60 (d, *J*= 4.0 Hz, 2 H), 2.98 (s, 6 H); $^{13}$**C NMR** (100 MHz, $CDCl_3$): δ 150.5, 133.8, 132.2, 128.6, 128.3, 122.3, 111.6, 107.8, 83.4, 80.7, 74.7, 72.0, 40.0; **HRMS** calcd for $C_{18}H_{15}N$: 245.1204, found: 245.1207.

**1-methoxy-4-(phenylbuta-1, 3-diyn-1-yl) benzene (1a2b)**[s3]



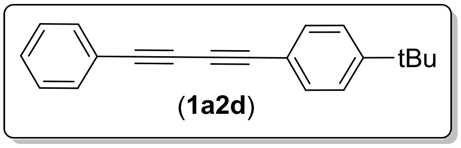
Colorless solid; m.p.= 90-94 ˚C; $^{1}$**H NMR** (400 MHz, $CDCl_3$): δ 7.51-7.49 (m, 2 H), 7.47-7.44 (m, 2 H), 7.33-7.31 (m, 3 H), 6.85-6.83 (m, 2 H), 3.80 (s, 3 H); $^{13}$**C NMR** (100 MHz, $CDCl_3$): δ 160.3, 134.1, 132.4, 129.0, 128.3, 121.9, 114.1, 113.6, 81.8, 81.0, 74.1, 72.7, 55.3; **HRMS** calcd for $C_{17}H_{12}O$: 232.0888, found: 232.0890.

**3-(phenylbuta-1,3-diyn-1-yl)phenol (1a2c)**[s4]



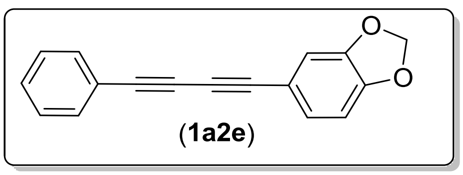
Brown solid; m.p.=119-123˚C; $^{1}$**H NMR** (600 MHz, $CDCl_3$): δ 7.52-7.50 (m, 2 H), 7.32-7.35 (m, 3H), 7.19 (t, *J*= 7.8 Hz 1H), 7.10-7.09 (m, 1H), 6.97-6.96 (m, 1H), 6.85-6.83 (m, 1H), 5.28 (bs, 1 H); $^{13}$**C NMR** (150 MHz, $CDCl_3$): δ 155.3, 132.4, 129.7, 129.2, 128.4, 125.2, 122.9, 121.6, 118.9, 116.8, 81.6, 81.1, 73.87 and 73.81; **HRMS** calcd for $C_{16}H_{10}O$: 218.0732, found: 218.0726.

**1-(tert-butyl)-4-(phenylbuta-1, 3-diyn-1-yl) benzene (1a2d)**



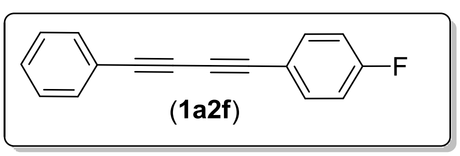

Colorless solid; m.p=78-82˚C, **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.54-7.52 (m, 2 H), 7.48-7.46 (m, 1 H), 7.37-7.33 (m, 6 H), 1.31 (s, 9 H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 152.6, 132.4, 132.2, 129.1, 128.4, 125.4, 121.7, 118.6, 81.8, 81.1, 74.1, 73.2, 34.8, 31.0; **HRMS** calcd for $C_{20}H_{18}$: 258.1409, found: 258.1412.

**5-(phenylbuta-1,3-diyn-1-yl)benzo[d][1,3]dioxole (1a2e)[s3]**



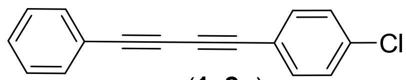

Colorless solid; m.p= 110-114˚C, 1H **NMR** (600 MHz, $CDCl_3$): δ 7.50-7.49 (m, 2 H), 7.34-7.30 (m, 3 H), 7.07-7.05 (m, 1 H), 6.94-6.93 (m, 1 H), 6.75 (d, J= 8.1, 1 H); **$^{13}$C NMR** (150 MHz, $CDCl_3$): δ 148.8, 147.4, 132.4, 129.1, 128.4, 127.7, 121.9, 114.9, 112.1, 108.6, 101.5, 81.6, 81.1, 74.0, 72.4; **HRMS** calcd for $C_{17}H_{10}O_2$: 246.0681, found: 246.0677.

**1-fluoro-4-(phenylbuta-1, 3-diyn-1-yl) benzene (1a2f)[s6]**




Colorless solid; m.p. 121-125 $^{o}$C; **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.52-7.47 (m, 4 H), 7.36-7.30 (m, 3 H), 7.04-6.99 (m, 2 H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 164.3, 164.2, 161.8, 161.7, 134.5, 134.48, 134.47, 132.4, 129.1, 128.44, 128.43, 121.7, 121.6, 116.0, 115.9, 115.79, 115.77, 81.5, 80.4, 73.9, 73.7; **HRMS** calcd for $C_{16}H_9F$: 220.0688, found: 220.0689.

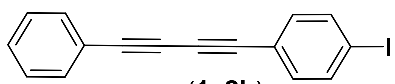
**1-chloro-4-(phenylbuta-1,3-diyn-1-yl)benzene (1a2g)**[s7]




(**1a2g**)

Yellow oil; m.p.= 131-135˚C; **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.51-7.50 (m, 2 H), 7.44-7.42 (m, 2 H), 7.36-7.29 (m, 5H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 135.3, 133.6, 132.5, 129.3, 128.8, 128.4, 121.5, 120.3, 82.1, 80.2, 74.8, 73.6; **HRMS** calcd for $C_{16}H_9Cl$: 236.0393, found: 236.0401.

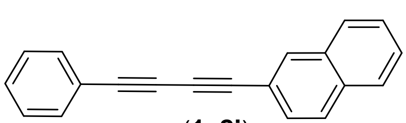
**1-iodo-4-(phenylbuta-1, 3-diyn-1-yl) benzene (1a2h)**[s7]




(**1a2h**)

Yellow oil; m.p.= 138-142˚C; **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.67-7.65 (m, 2 H), 7.52-7.50 (m, 2 H), 7.36-7.30 (m, 3 H), 7.22 (d, *J*= 8.0 Hz, 2 H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 137.6, 133.7, 132.4, 129.3, 128.4, 121.5, 121.2, 95.4, 82.2, 80.4, 75.2, 73.6; **HRMS** calcd for $C_{16}H_9I$: 327.9749, found: 327.9782.

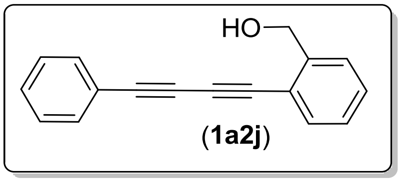
**2-(phenylbuta-1,3-diyn-1-yl)naphthalene (1a2i)**[s7]



(**1a2i**)

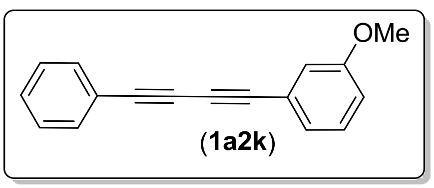
Yellow solid; m.p.= 97-101˚C; **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.04-7.75 (m, 5H), 7.54-7.46 (m, 3 H) 7.36-7.30 (m, 4H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 133.1, 133.0, 132.7, 132.5, 132.4, 129.2, 128.4, 128.1, 127.8, 127.2, 126.7, 121.7, 118.9, 82.0, 81.7, 74.2; **HRMS** calcd for $C_{20}H_{12}$: 252.0939, found: 252.0951.

**(2-(phenylbuta-1, 3-diyn-1-yl) phenyl) methanol (1a2j)**



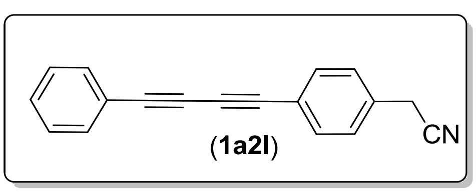

Yellow oil; m.p.= 82-86˚C; **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.54-7.50 (m, 4 H), 7.48-7.33 (m, 4 H ), 7.29-7.25 (m, 1H ), 4.87 (s, 2 H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 143.9, 133.2, 132.4, 129.5, 129.3, 128.4, 127.4, 127.2, 121.5, 119.7, 82.7, 78.8, 78.2, 73.6, 63.5; **HRMS** calcd for $C_{17}H_{12}O$: 232.0888, found: 232.0890.

**1-methoxy-3-(phenylbuta-1, 3-diyn-1-yl) benzene (1a2k)**[s8]



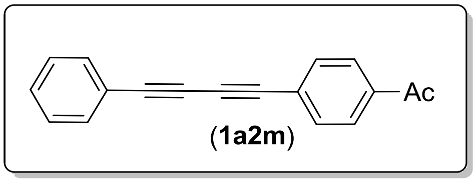

White solid; m.p.= 50-54 ˚C; **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.55-7.52 (m, 2 H), 7.38-7.34 (m, 3 H) 7.27-7.05 (m, 3 H), 6.95-6.92 (m, 1 H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 159.2, 132.4, 129.5, 129.2, 128.4, 125.0, 122.6, 121.6, 117.0, 115.9, 81.5, 81.4, 73.8, 73.6, 55.2; **HRMS** calcd for $C_{17}H_{12}O$: 232.0888, found: 232.0891.

**1-(4-(phenylbuta-1,3-diyn-1-yl)phenyl)ethanone (1a2l)**



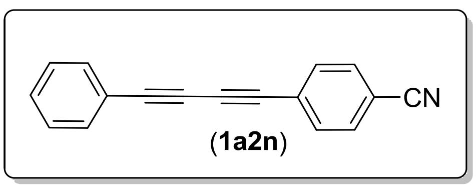

Pale white solid; m.p.= 77-81˚C; **$^1$H NMR** (600 MHz, $CDCl_3$): δ 7.52-7.50 (m, 4 H), 7.36-7.28 (m, 5 H), 3.75 (s, 2 H); **$^{13}$C NMR** (150 MHz, $CDCl_3$): δ 133.1, 132.5, 130.8, 129.3, 128.4, 128.0, 121.8, 121.5, 117.1, 82.0, 80.4, 74.7, 73.6 23.6; **HRMS** calcd for $C_{18}H_{11}N$: 241.0891, found: 241.0893.

**1-(4-(phenylbuta-1, 3-diyn-1-yl) phenyl)ethanone (1a2m)**



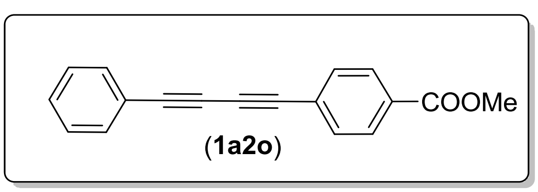
Pale white solid; m.p.= 111-115 ˚C; **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.90 (d, *J*= 4.0 Hz, 2 H), 7.58 (d, *J*= 4.0 Hz, 2 H) 7.52 (d, *J*= 4.0 Hz, 2 H), 7.38-7.33 (m, 3 H), 2.58 (s, 3 H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 197.0, 136.6, 132.5, 132.4, 129.4, 128.4, 128.1, 126.4, 121.2, 83.1, 80.3, 76.6, 73.4, 26.5; **HRMS** calcd for $C_{18}H_{12}O$: 244.0888, found: 244.0893.

**4-(phenylbuta-1, 3-diyn-1-yl)benzonitrile (1a2n)**[s9]



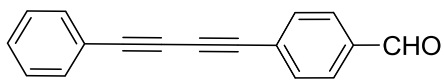
Yellow solid; m.p.= 131-135 ˚C; **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.62-7.56 (m, 4 H), 7.52 (d, *J*= 4.0 Hz, 2 H), 7.39-7.31(m, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 133.8, 132.5, 132.0, 129.7, 128.5, 126.7, 121.1, 118.2, 112.3, 83.9, 79.2, 78.1, 73.2; **HRMS** calcd for $C_{17}H_9N$: 227.0735, found: 227.0738.

**Methyl 4-(phenylbuta-1,3-diyn-1-yl)benzoate (1a2o)**[s10]



Yellow solid; m.p.= 105-109 ˚C; **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.98 (d, *J*= 4.0 Hz, 2 H), 7.57-7.50 (m, 4 H), 7.39-7.30 (m, 3 H) 3.90 (s, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 166.2, 132.5, 132.3, 130.2, 129.5, 129.4, 128.4, 126.4, 121.3, 82.9, 80.4, 76.6, 73.5, 52.3; **HRMS** calcd for $C_{18}H_{12}O_2$: 260.0837, found: 260.0840.

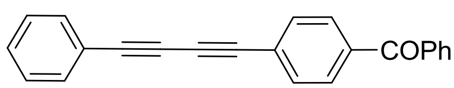
**4-(phenylbuta-1,3-diyn-1-yl)benzaldehyde (1a2p)**




(**1a2p**)

Pale white solid; m.p.= 97-101 ˚C; **$^1$H NMR** (400 MHz, $CDCl_3$): δ 9.90 (s, 1 H), 7.83 (d, *J*= 8.0 Hz, 2 H), 7.64 (d, *J*= 8.0 Hz, 2 H), 7.53 (d, *J*= 8.0 Hz, 2 H), 7.39-7.31 (m, 3 H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 191.2, 135.9, 132.9, 132.5, 129.6, 129.5, 128.5, 128.0, 121.2, 83.6, 80.2, 77.6, 73.4; **HRMS** calcd for $C_{17}H_{10}O$: 230.0732, found: 230.0728.

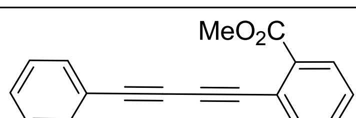
**phenyl (4-(phenylbuta-1,3-diyn-1-yl)phenyl)methanone (1a2q)**




(**1a2q**)

Pale white solid; m.p.= 129-133˚C; **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.76 (d, *J*= 4.0 Hz, 4 H), 7.59 (d, *J*= 8.0 Hz, 2 H), 7.54-7.45 (m, 4 H), 7.37-7.31 (m, 4 H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 195.6, 137.5, 137.1, 132.6, 132.5, 132.4, 132.2, 129.9, 129.4, 129.1, 128.4, 125.9, 121.4, 83.0, 80.4, 76.8, 73.5; **HRMS** calcd for $C_{23}H_{14}O$: 306.1045, found: 306.1044.

**methyl 2-(phenylbuta-1,3-diyn-1-yl)benzoate (1a2r)[s3]**

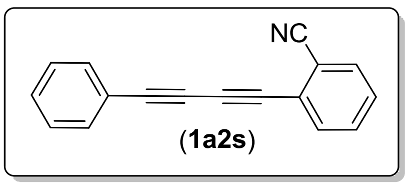



(**1a2r**)

Pale white solid; m.p.= 91-93˚C **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.95 (d, J= 6.3, 1 H), 7.64 (d, J= 6.3, 1 H), 7.52-7.51 (m, 2 H), 7.48-7.46 (m, 1 H), 7.40-7.37 (m, 1 H), 7.35-7.30 (m, 3 H); **$^{13}$C**

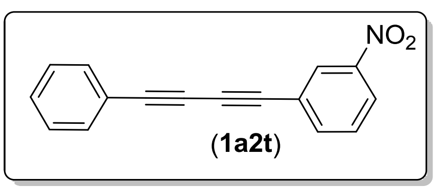
**NMR** (100 MHz, $CDCl_3$): δ 166.0, 135.1, 132.4, 131.7, 130.5, 129.2, 128.6, 128.3, 122.5, 121.7, 83.2, 79.7, 78.7, 74.0, 52.3; **HRMS** calcd for $C_{18}H_{12}O_2$: 260.0837, found: 260.0831.

**2-(phenylbuta-1,3-diyn-1-yl)benzonitrile (1a2s)**



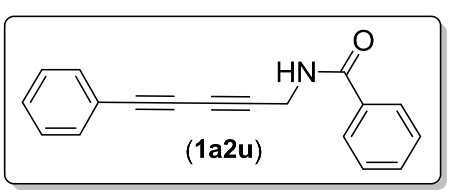

Pale white solid; m.p.= 124-129 ˚C; **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.65-7.60 (m, 2 H), 7.57-7.52 (m, 3 H), 7.45-7.32 (m, 4 H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 133.3, 132.8, 132.6, 132.4, 129.7, 129.0, 128.4, 125.7, 121.0, 117.1, 115.8, 84.3, 79.9, 76.6, 73.2; **HRMS** calcd for $C_{17}H_9N$: 227.0735, found: 227.0732.

**1-nitro-3-(phenylbuta-1,3-diyn-1-yl)benzene (1a2t)[s6]**



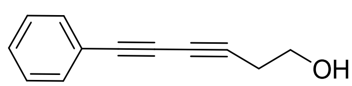

Pale white solid; m.p.= 147-151˚C; **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.32-7.77 (m, 3 H), 7.53-7.48 (m, 3 H), 7.40-7.31 (m, 3 H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 148.3, 138.2, 132.8, 129.9, 129.8, 128.7, 127.4, 124.0, 121.3, 83.3, 78.8, 77.6, 73.4; **HRMS** calcd for $C_{16}H_9NO$: 247.0633, found: 247.0635.

**N-(5-phenylpenta-2,4-diyn-1-yl)benzamide (1a2u)[s11]**



Pale white solid; m.p.= 106-110°C; **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.81-7.79 (m, 2 H), 7.49-7.25 (m, 8 H), 6.98 (s, 1 H), 4.38 (s, 2 H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 167.3, 133.4, 132.5, 131.7, 129.2, 128.5, 128.2, 127.0, 121.2, 78.5, 77.3, 73.3, 68.0, 30.5; **HRMS** calcd for $C_{18}H_{13}NO$: 259.0997, found: 259.1000.

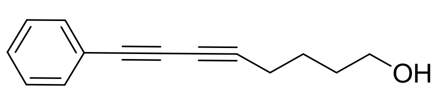
**6-phenylhexa-3, 5-diyn-1-ol (1a2v)** [s12]




(**1a2v**)

Colorless oil; **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.45 (d, *J*= 12.0 Hz, 2 H), 7.32-7.28 (m, 3 H), 3.77 (t, *J*= 12.0 Hz, 2 H), 2.62 (t, *J*= 12.0 Hz, 3 H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 132.5, 129.0, 128.3, 121.7, 80.9, 75.3, 73.9, 66.8, 60.7, 23.9; IR (KBr, cm$^{-1}$) 3054, 2365, 1776, 1604, 1474, 979; **HRMS** calcd for $C_{12}H_{10}O$: 170.0732, found: 170.0729.

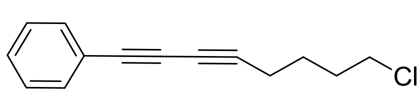
**8-phenylocta-5,7-diyn-1-ol (1a2w)**[s12]




(**1a2w**)

Colorless oil; **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.45-7.43 (m, 2 H), 7.31-7.25 (m, 3 H), 3.65 (t, *J*= 4.0 Hz, 2 H), 2.38 (t, *J*= 8.0 Hz, 3 H) 1.69-1.65(m, 3 H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 132.4, 128.8, 128.2, 121.9, 84.2, 74.8, 74.1, 65.3, 62.1, 31.6, 24.4, 19.3; **HRMS** calcd for $C_{14}H_{14}O$: 198.1045, found: 198.1039.

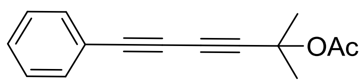
**(8-chloroocta-1,3-diyn-1-yl)benzene (1a2x)**




(**1a2x**)

Colorless oil; **¹H NMR** (400 MHz, $CDCl_3$): δ 7.46-7.44 (m, 2 H), 7.32-7.26 (m, 3 H), 3.56 (t, *J*= 8.0 Hz, 2 H), 2.40 (t, *J*= 8.0 Hz, 2 H) 1.95-1.88 (m, 2 H), 1.76-1.68 (m, 2 H), ; **¹³C NMR** (100 MHz, $CDCl_3$): δ 132.4, 128.9, 128.3, 121.9, 83.5, 75.0, 74.1, 65.7, 44.3, 31.4, 25.3, 18.8; **HRMS** calcd for $C_{14}H_{13}Cl$: 216.0706, found: 216.0800.

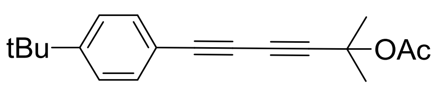
**2-methyl-6-phenylhexa-3,5-diyn-2-yl acetate (1a2y)**




(**1a2y**)

Pale white solid; m.p.= 55-59 ˚C; **¹H NMR** (400 MHz, $CDCl_3$): δ 7.45-7.43 (m, 2 H), 7.33-7.26 (m, 3 H), 2.02 (s, 3H), 1.68 (s, 6 H); **¹³C NMR** (100 MHz, $CDCl_3$): δ 169.2, 132.4, 129.1, 128.3, 121.5, 82.7, 79.2, 73.2, 71.8, 68.9, 28.6, 21.7; **HRMS** calcd for $C_{15}H_{14}O_2$: 226.0994, found: 226.0998.

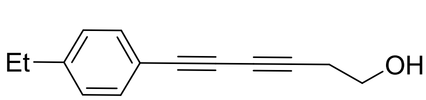
**6-(4-(tert-butyl)phenyl)-2-methylhexa-3,5-diyn-2-yl acetate (2d2y)**




(**2d2y**)

Pale white solid; m.p.= 77-81 ˚C; **¹H NMR** (400 MHz, $CDCl_3$): δ 7.39 (d, *J*= 12.0 Hz, 2H), 7. 30 (d, *J*= 8.0 Hz, 2 H), 2.02 (s, 3 H), 1.68 (s, 6 H), 1.27 (s, 9 H); **¹³C NMR** (100 MHz, $CDCl_3$): δ 169.2, 152.5, 132.2, 125.3, 118.3, 82.2, 79.4, 72.5, 71.8, 69.1, 34.8, 31.0, 28.6, 21.7,; **HRMS** calcd for $C_{19}H_{22}O_2$: 282.1620, found: 282.1619.

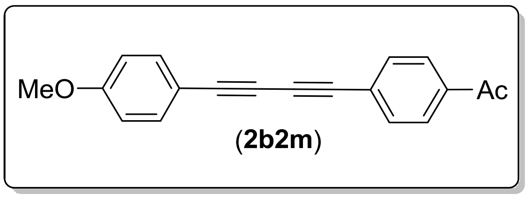
**6-(4-ethylphenyl)hexa-3,5-diyn-1-ol (2z2v)**




(**2z2v**)

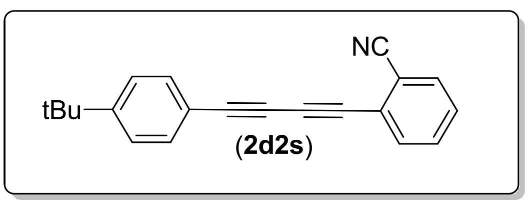
Colorless oil; **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.37 (d, J= 8.0 Hz, 2 H), 7.11 (d, J= 8.0 Hz, 2 H), 3.77 (t, *J*= 4.0 Hz, 2 H), 2.61 (q, *J*= 12.0 Hz, 2H), 2.01 (s, 1 H), 1.19 (t, *J*= 12.0 Hz, 3 H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 145.6, 132.5, 127.9, 118.7, 80.5, 75.6, 73.2, 66.9, 60.7, 28.8, 23.9, 15.1; **HRMS** calcd for $C_{14}H_{14}O$: 198.1045, found: 198.1039.

**1-(4-((4-methoxyphenyl)buta-1,3-diyn-1-yl)phenyl)ethanone (2b2m)**



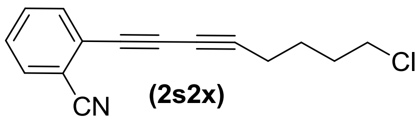

Pale white solid; m.p.= 134-138 ˚C; **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.88 (d, *J*= 8.0 Hz, 2 H), 7.55 (d, *J*= 8.0 Hz, 2 H), 7.44 (d, *J*= 8.0 Hz, 2 H), 6.83 (d, *J*= 8.0 Hz, 2 H), 3.79 (s, 3 H), 2.56 (s, 3 H), ; **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 197.1, 160.5, 136.5, 134.1, 132.4, 128.1, 126.8, 114.1, 113.1, 83.5, 79.9, 77.3, 72.4, 55.3, 26.5; **HRMS** calcd for $C_{19}H_{14}O_2$: 274.0994, found: 274.0992.

**2-((4-(tert-butyl)phenyl)buta-1,3-diyn-1-yl)benzonitrile (2d2s)**




Pale white solid; m.p.= 140-144 ˚C; **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.64-7.52 (m, 3 H), 7.47-7.42 (m, 3 H), 7.36-7.34 (m, 2 H), 1.30 (s, 9 H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 153.2, 133.3, 132.7, 132.4, 132.3, 128.9, 125.9, 125.5, 117.9, 117.1, 115.7, 84.7, 80.2, 76.4, 72.6, 34.9, 31.0; **HRMS** calcd for $C_{21}H_{17}N$: 283.1361, found: 283.1363.

**2-(8-chloroocta-1,3-diyn-1-yl)benzonitrile (2s2x)**



(2s2x)

Pale white solid; m.p.= 120-124 ˚C **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.61 (d, *J*= 8.0 Hz, 1 H), 7.57-7.49 (m, 2 H), 7.40 (d, *J*= 8.0 Hz, 1 H), 3.56 (t, *J*= 8.0 Hz, 2 H), 2.43 (t, *J*= 4.0 Hz, 2 H), 1.93-1.89 (m, 2 H), 1.77-1.71 (m, 2 H) ; **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 133.4, 132.7, 132.3, 128.8, 126.0, 117.1, 116.0, 86.8, 80.4, 70.5, 65.2, 44.2, 31.3, 25.1, 18.9; **HRMS** calcd for $C_{15}H_{12}CN$: 241.0658, found: 247.0656.

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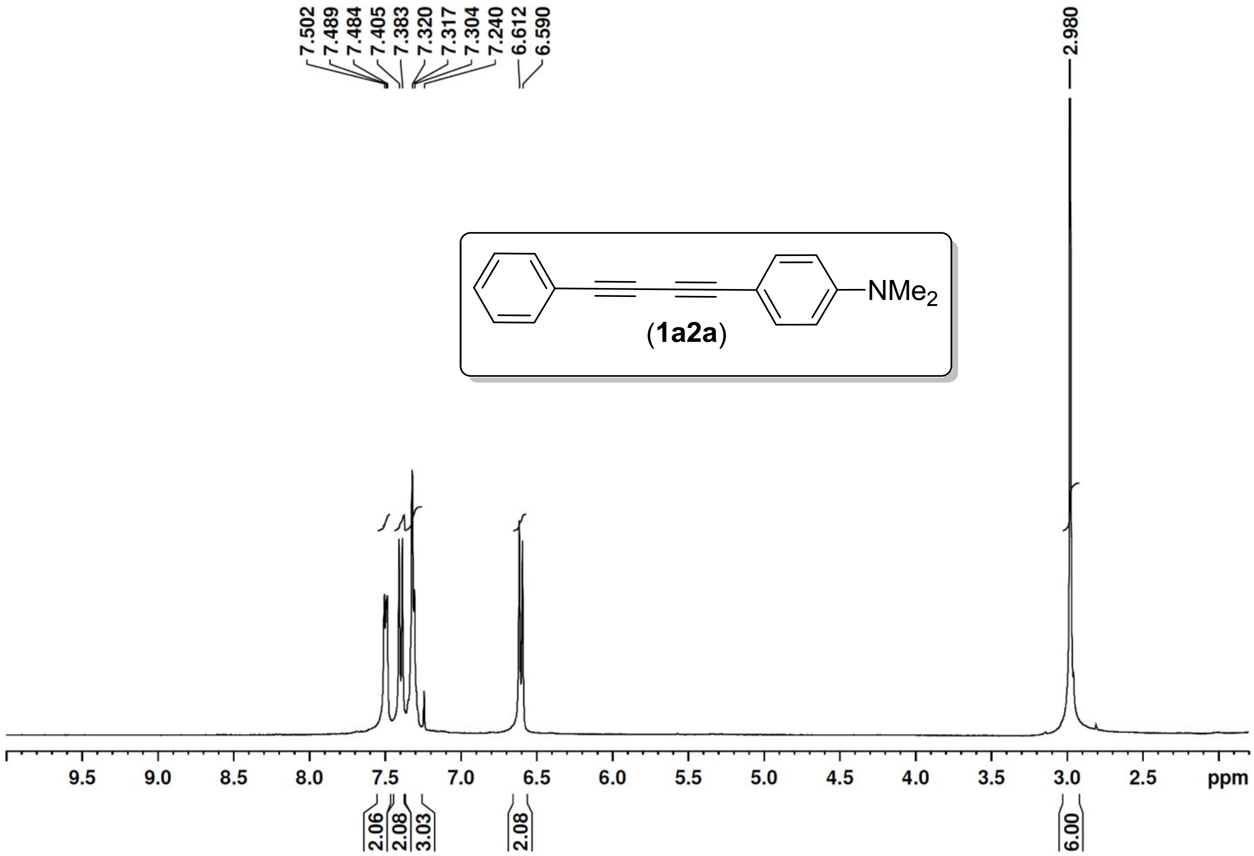
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7.317
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9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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4.0
3.5
3.0
2.5
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2.06
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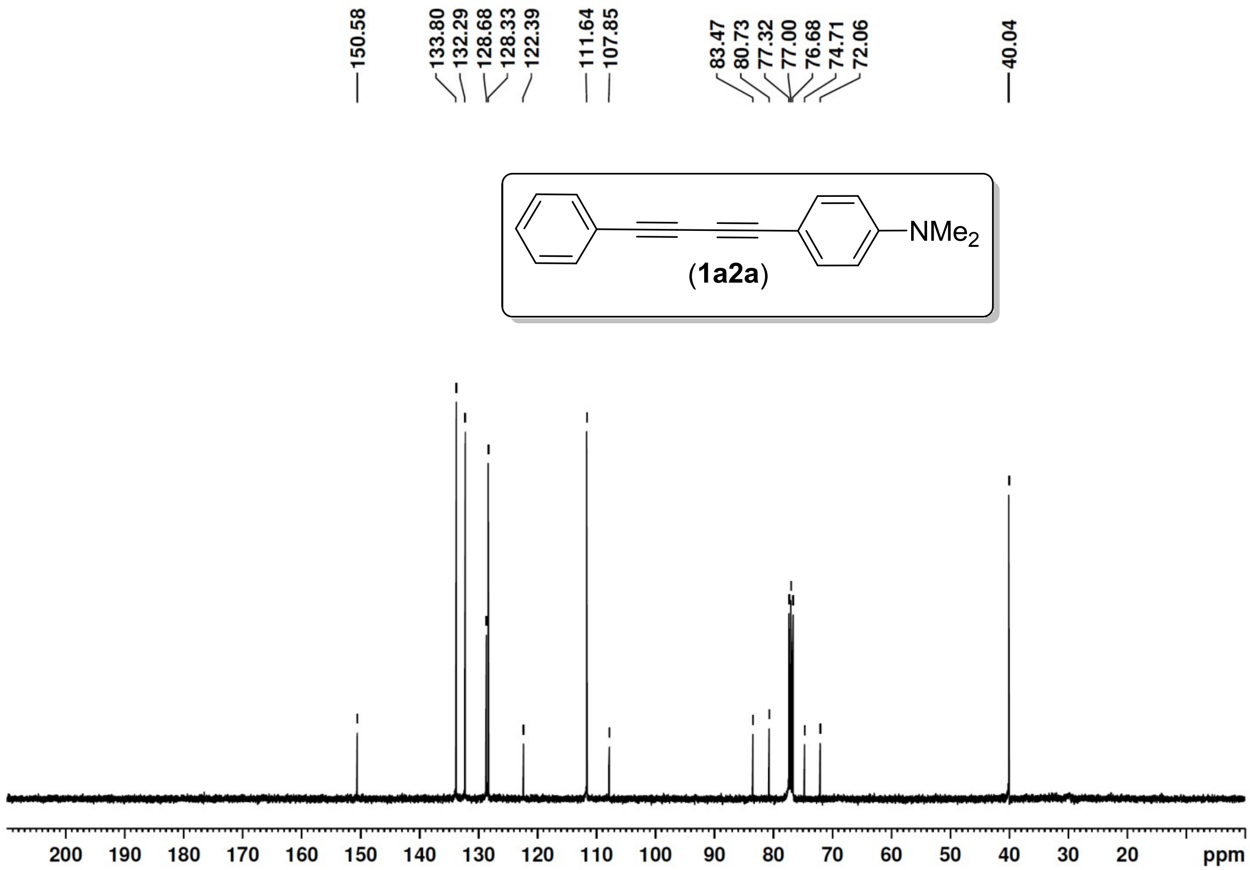
3.03
2.08
6.00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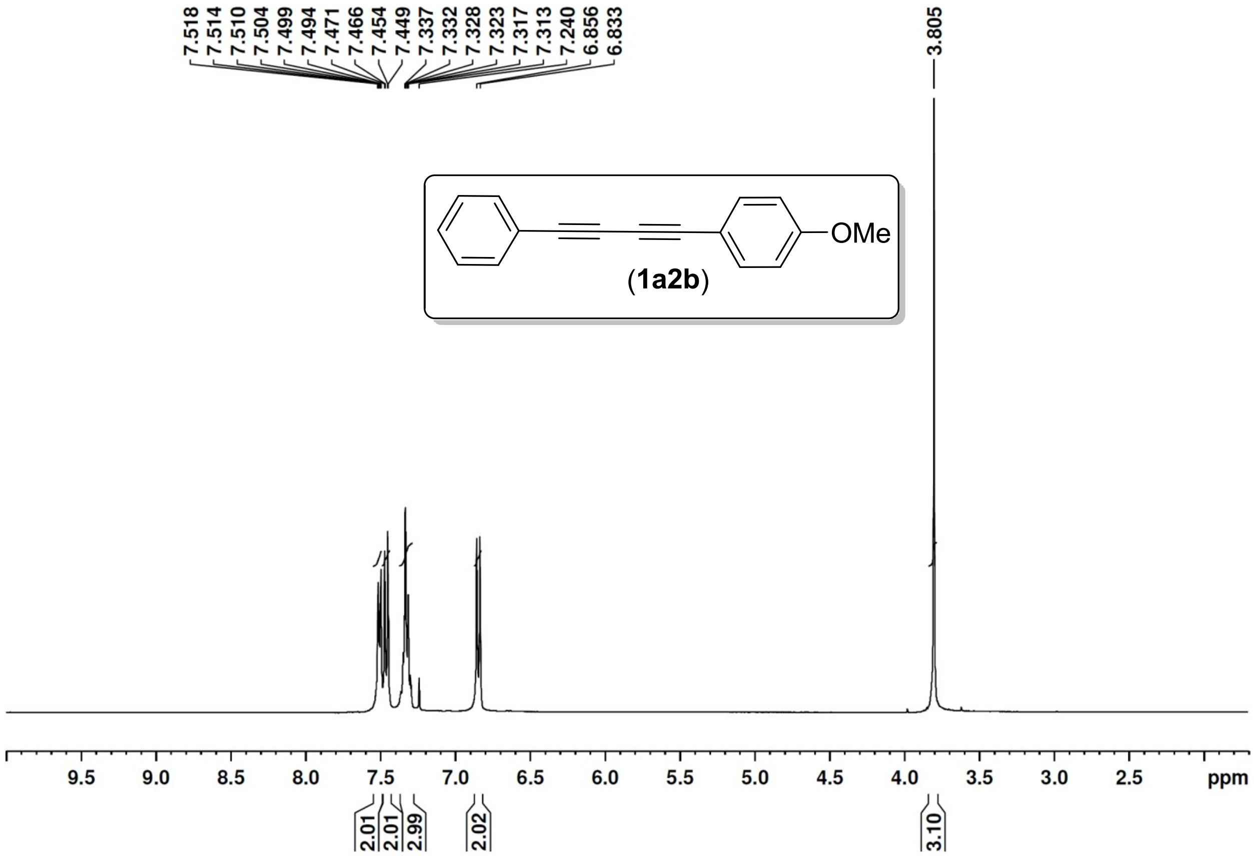
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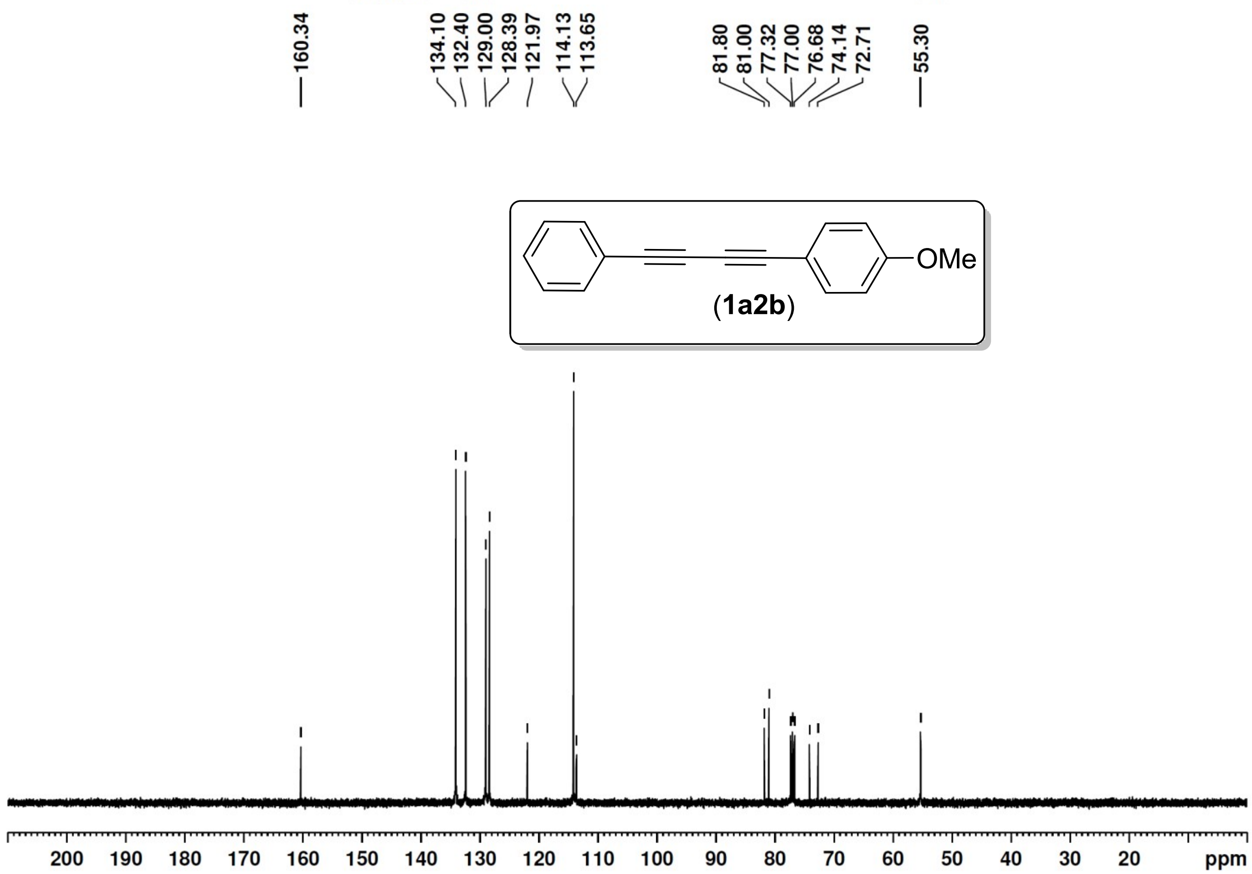
3.10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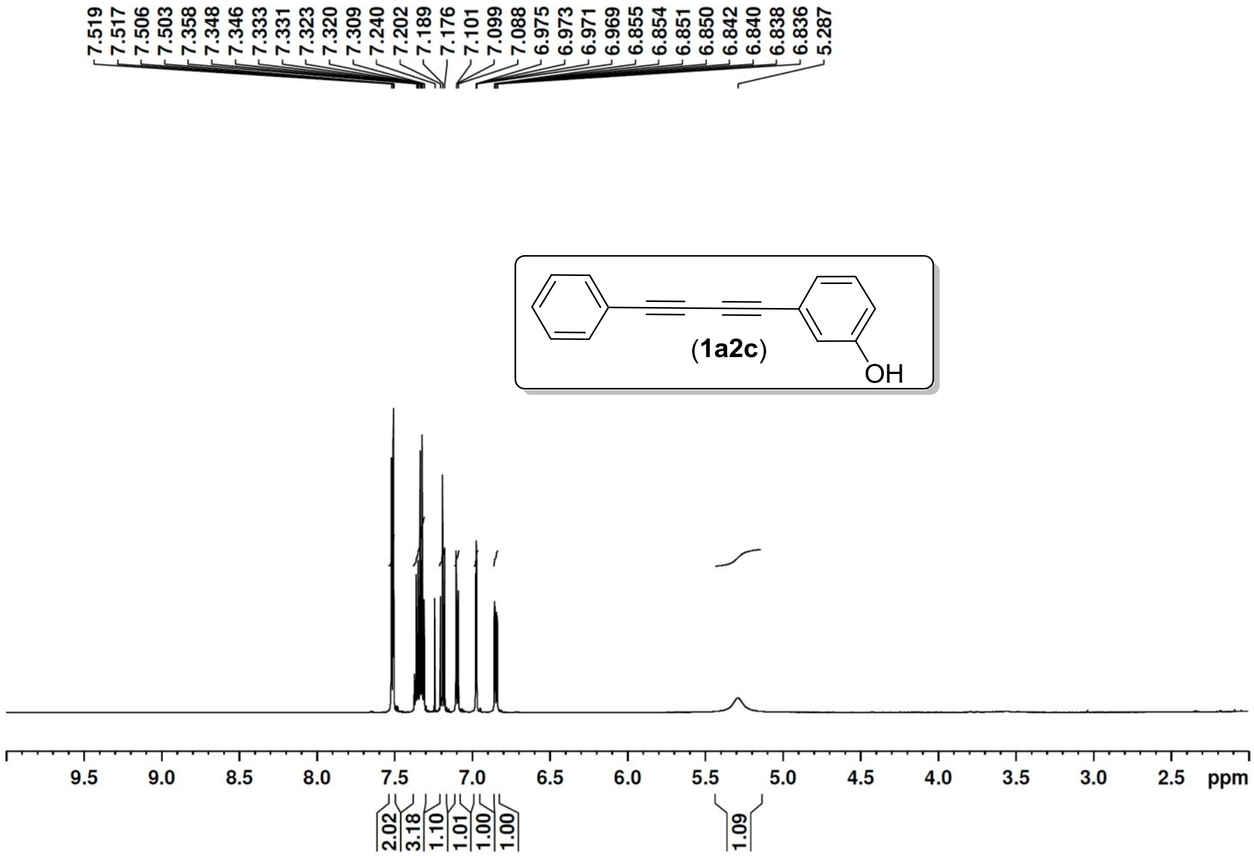
ppm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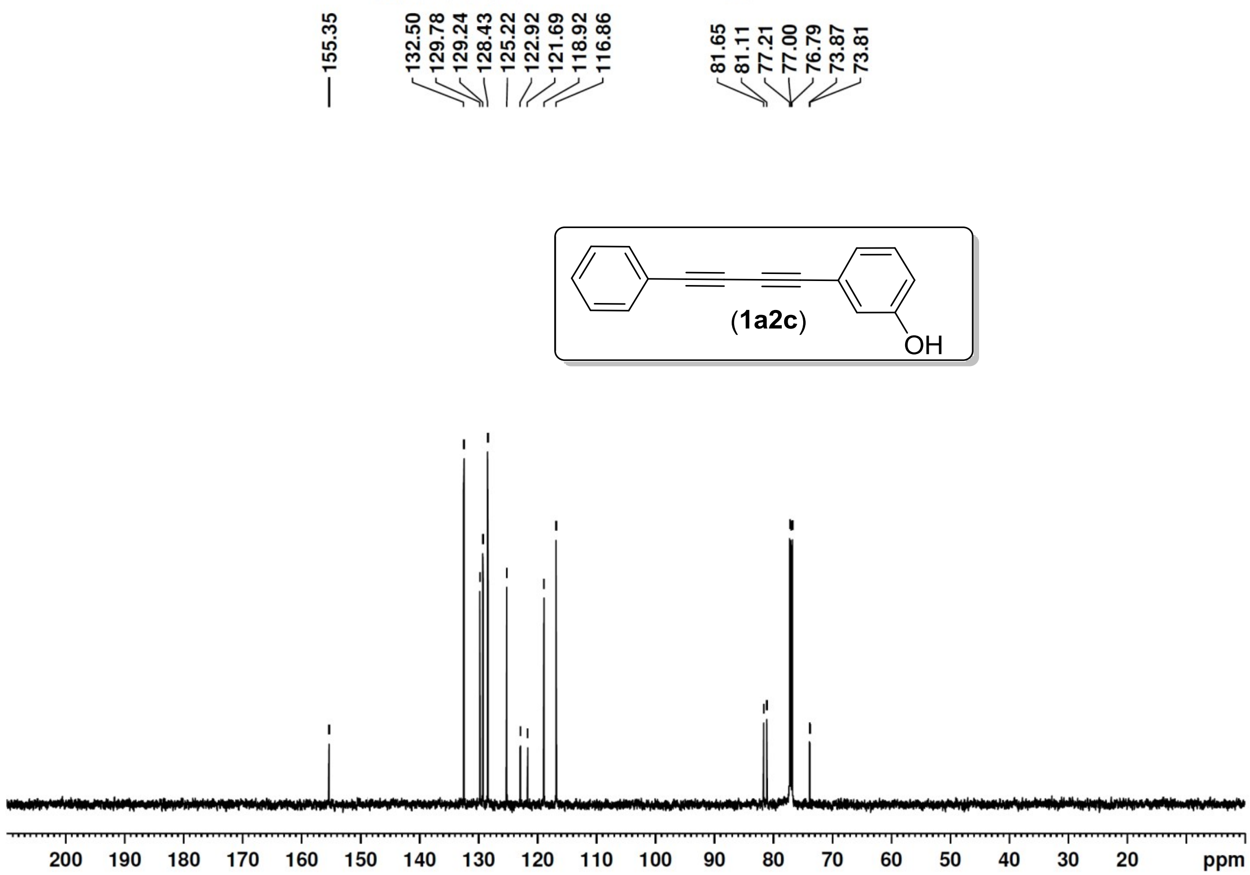
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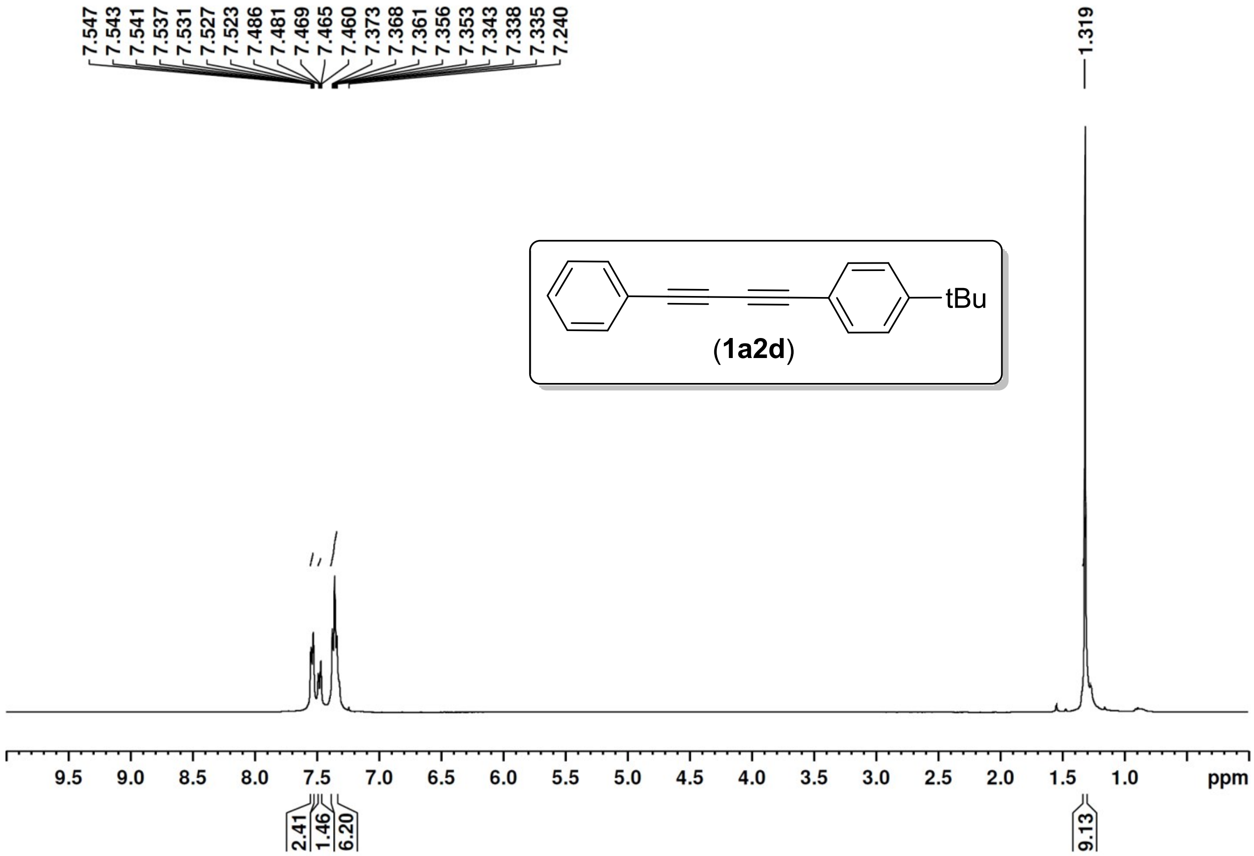
ppm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ppm
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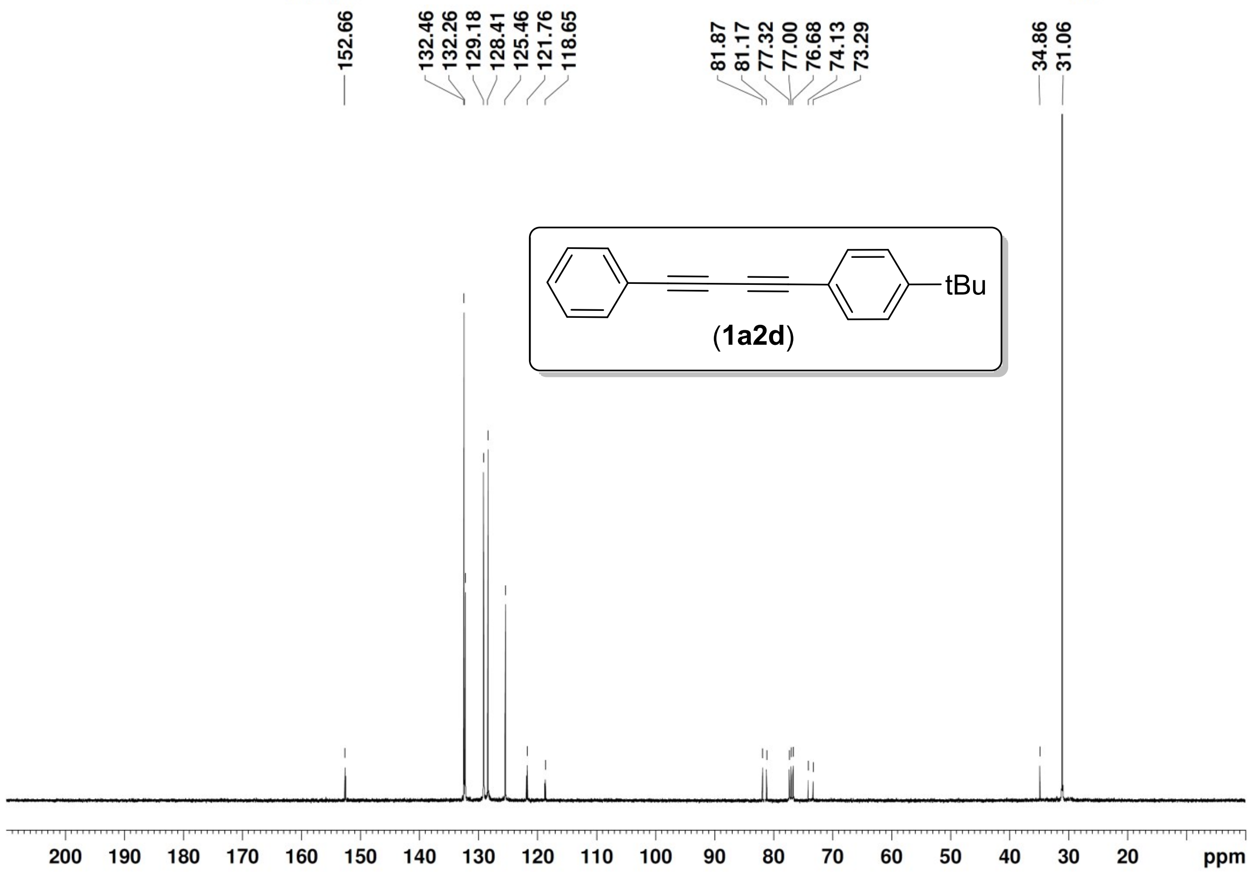
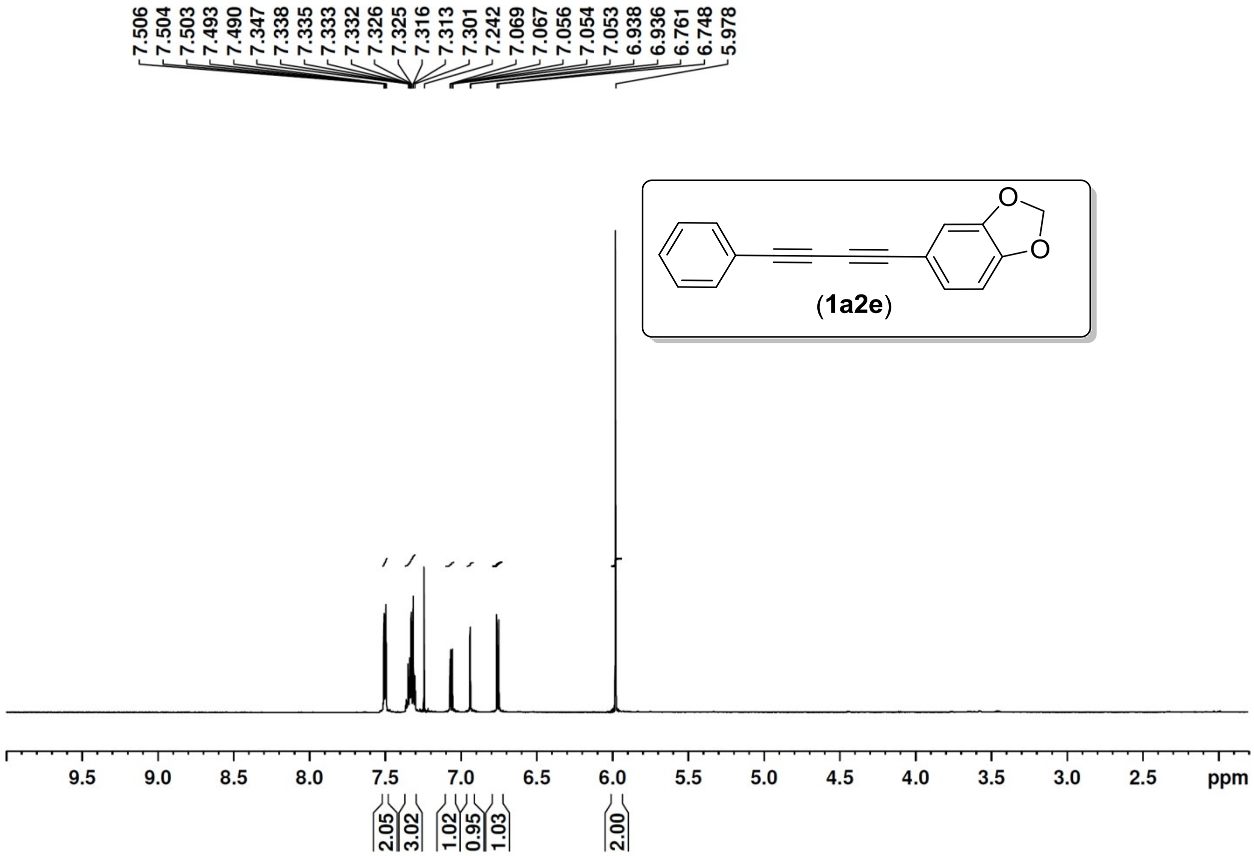
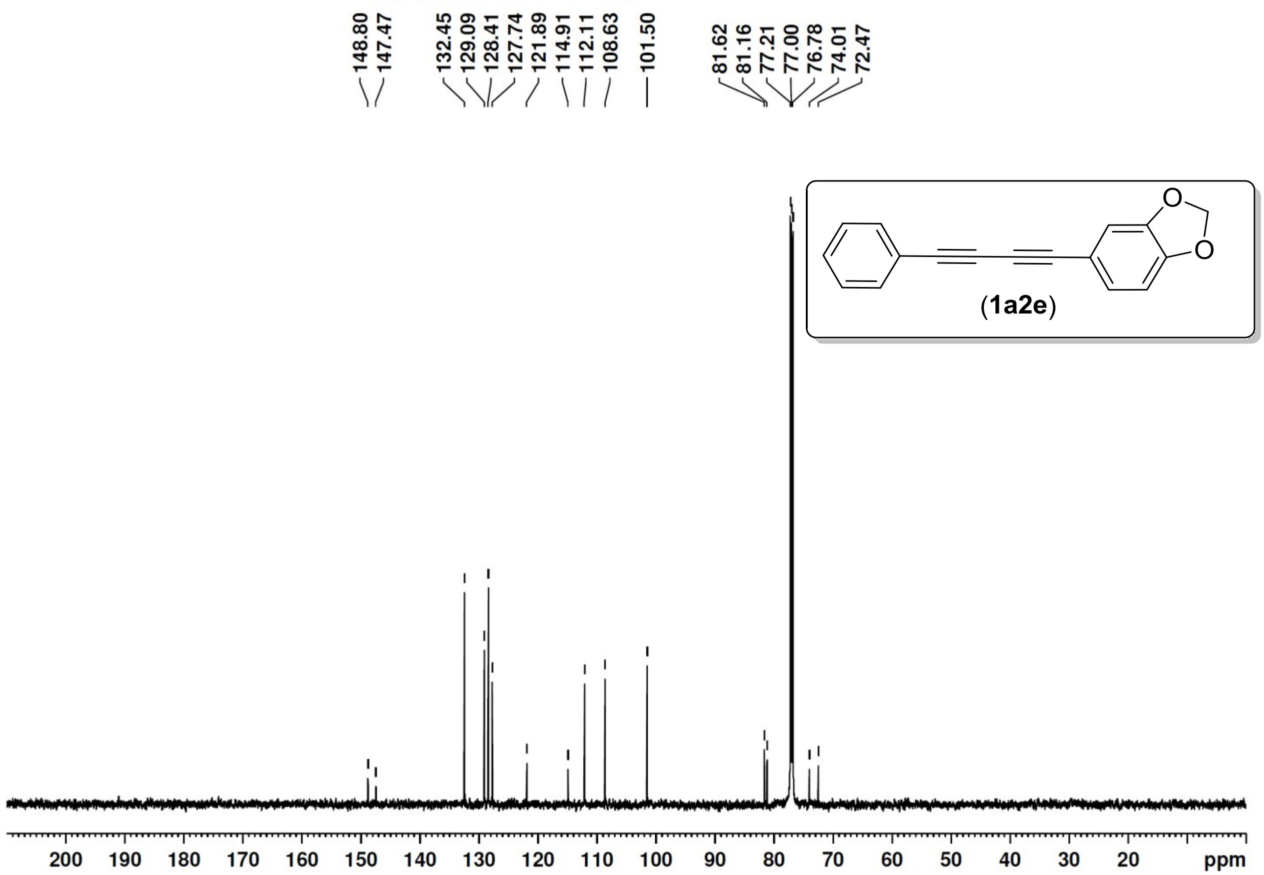
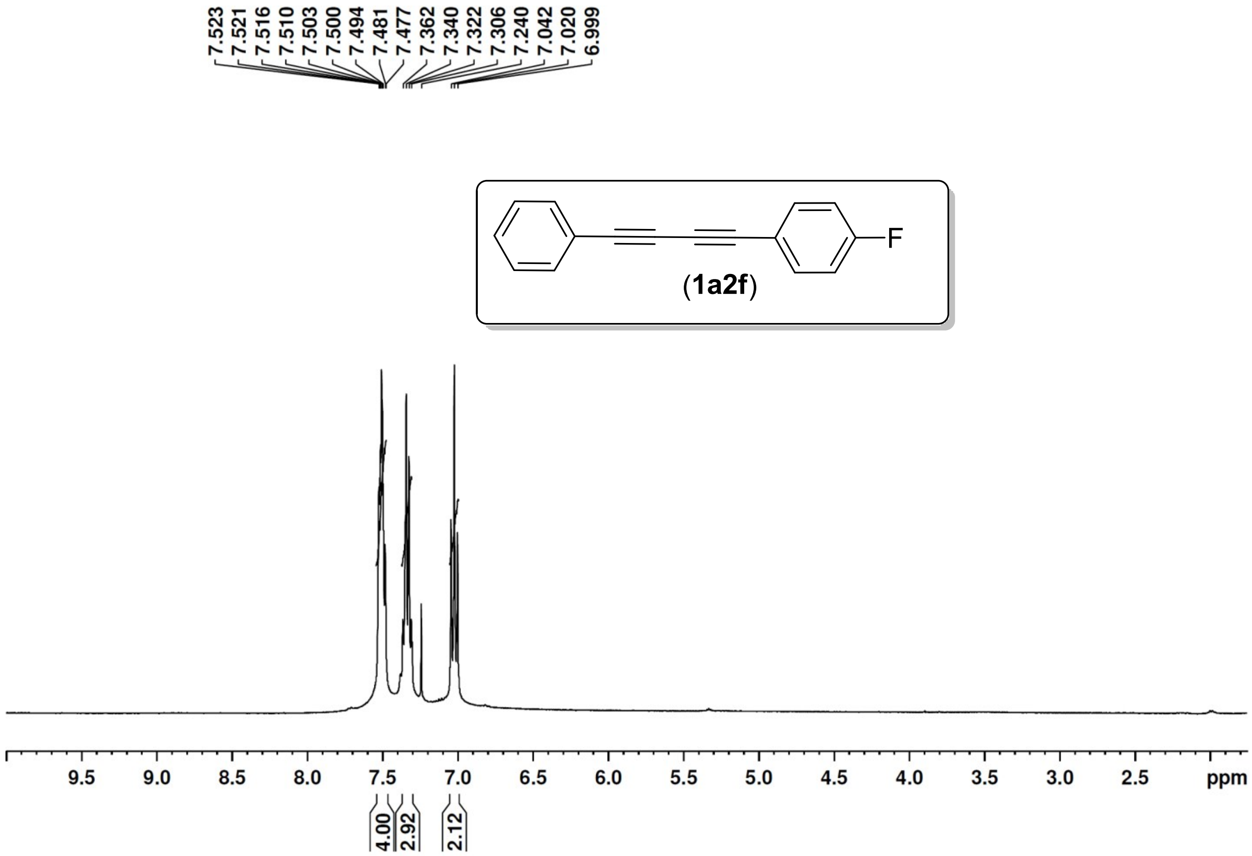
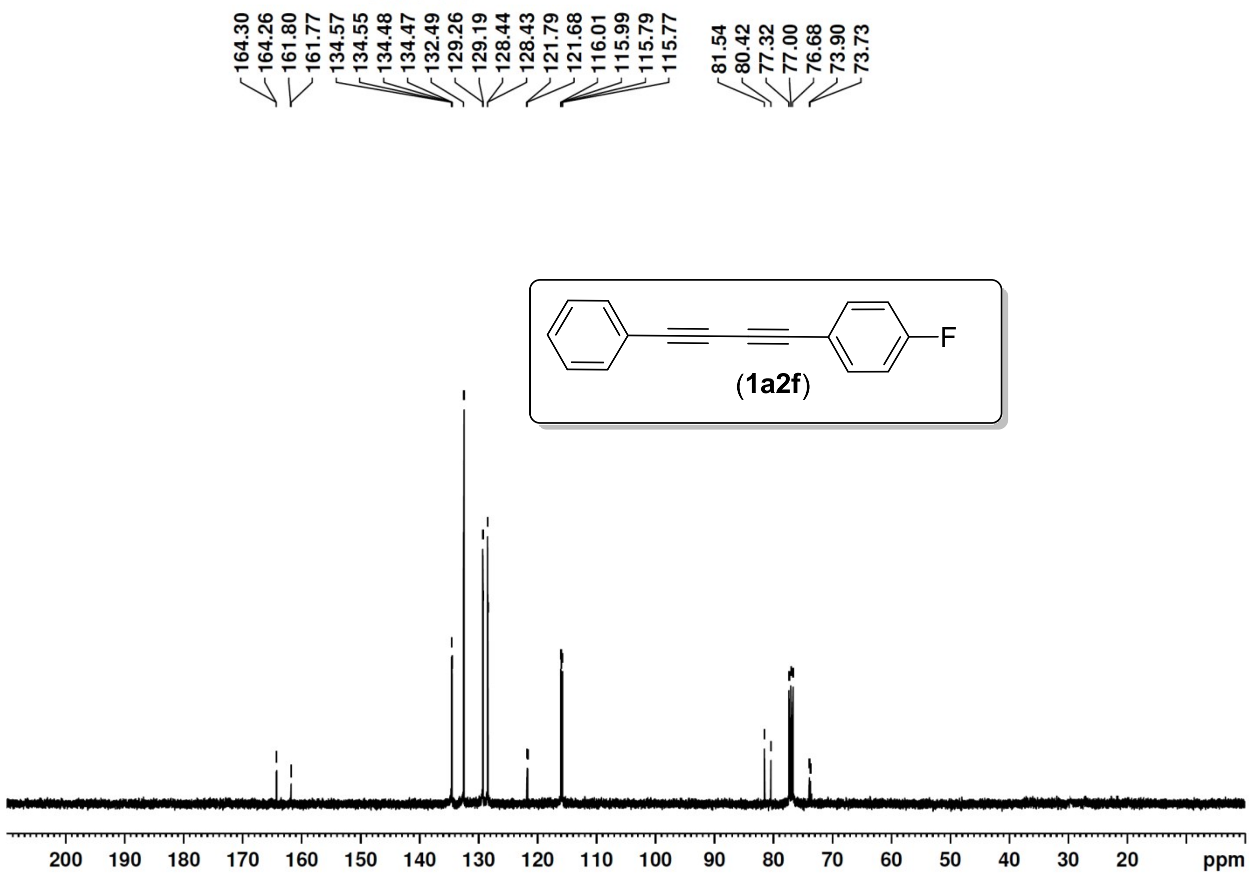
6.20
9.13

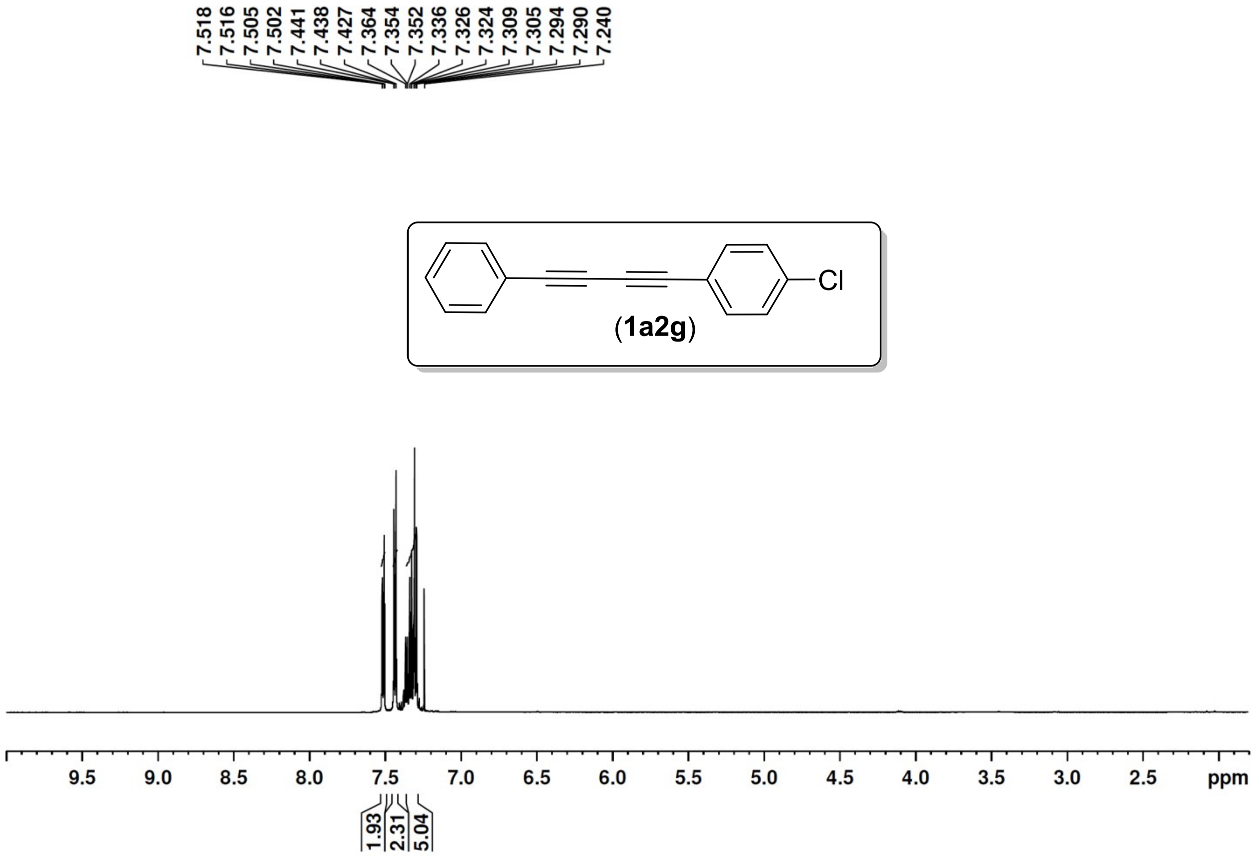

152.66
132.46
132.26
129.18
128.41
125.46
121.76
118.65
81.87
81.17
77.32
77.00
76.68
74.13
73.29
34.86
31.06
tBu
(1a2d)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

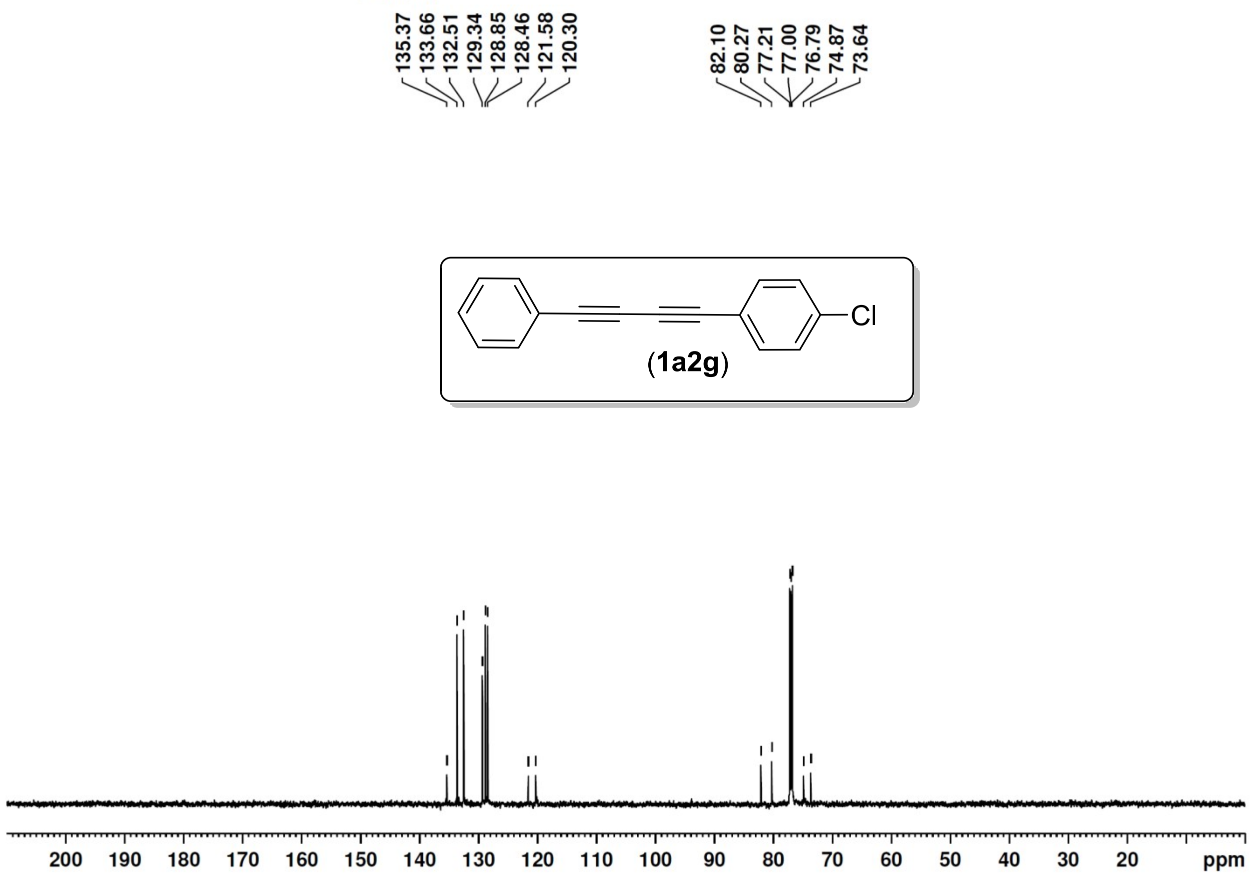

7.506
7.504
7.503
7.493
7.490
7.347
7.338
7.335
7.333
7.332
7.326
7.325
7.316
7.313
7.301
7.242
7.069
7.067
7.056
7.054
7.053
6.938
6.936
6.761
6.748
5.978
(1a2e)
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 ppm
2.05
3.02
1.02
0.95
1.03
2.00

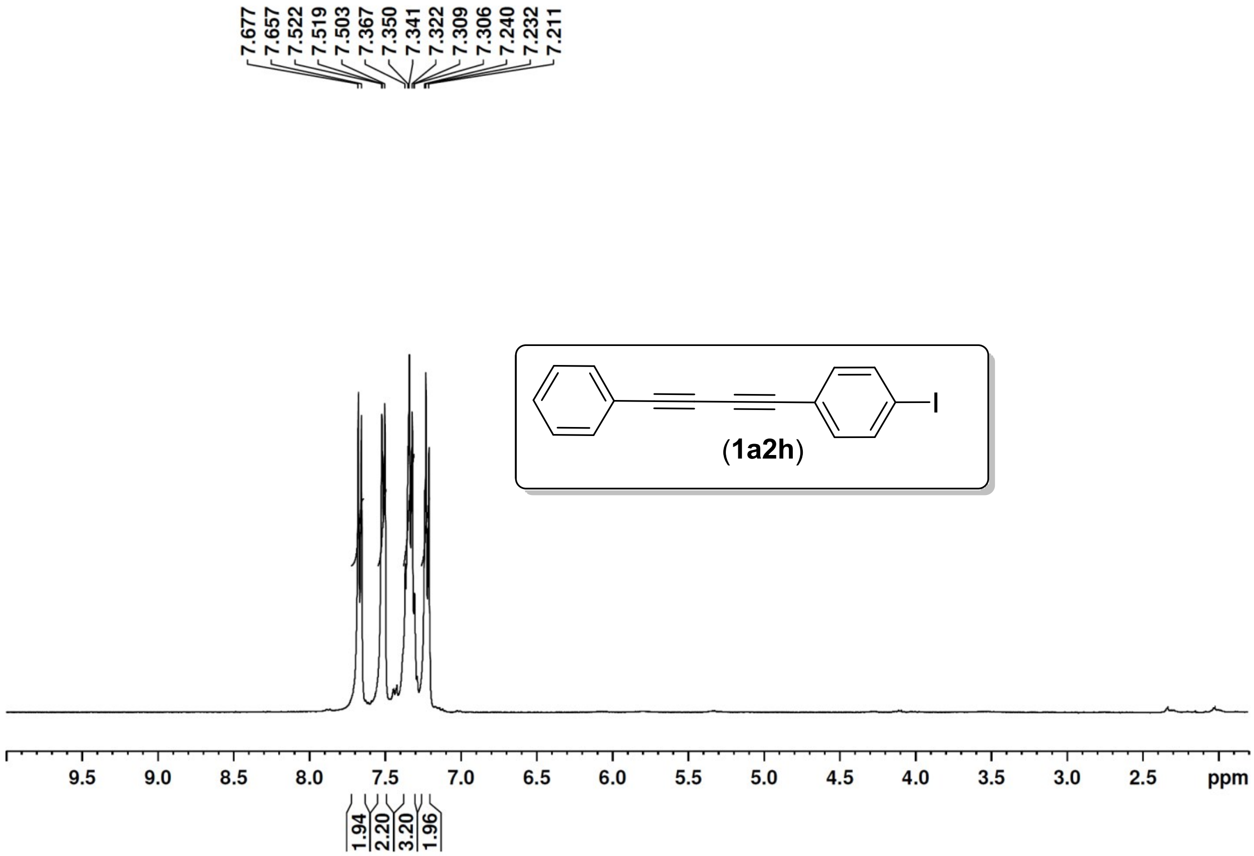

148.80
147.47
132.45
129.09
128.41
127.74
121.89
114.91
112.11
108.63
101.50
81.62
81.16
77.21
77.00
76.78
74.01
72.47
(1a2e)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

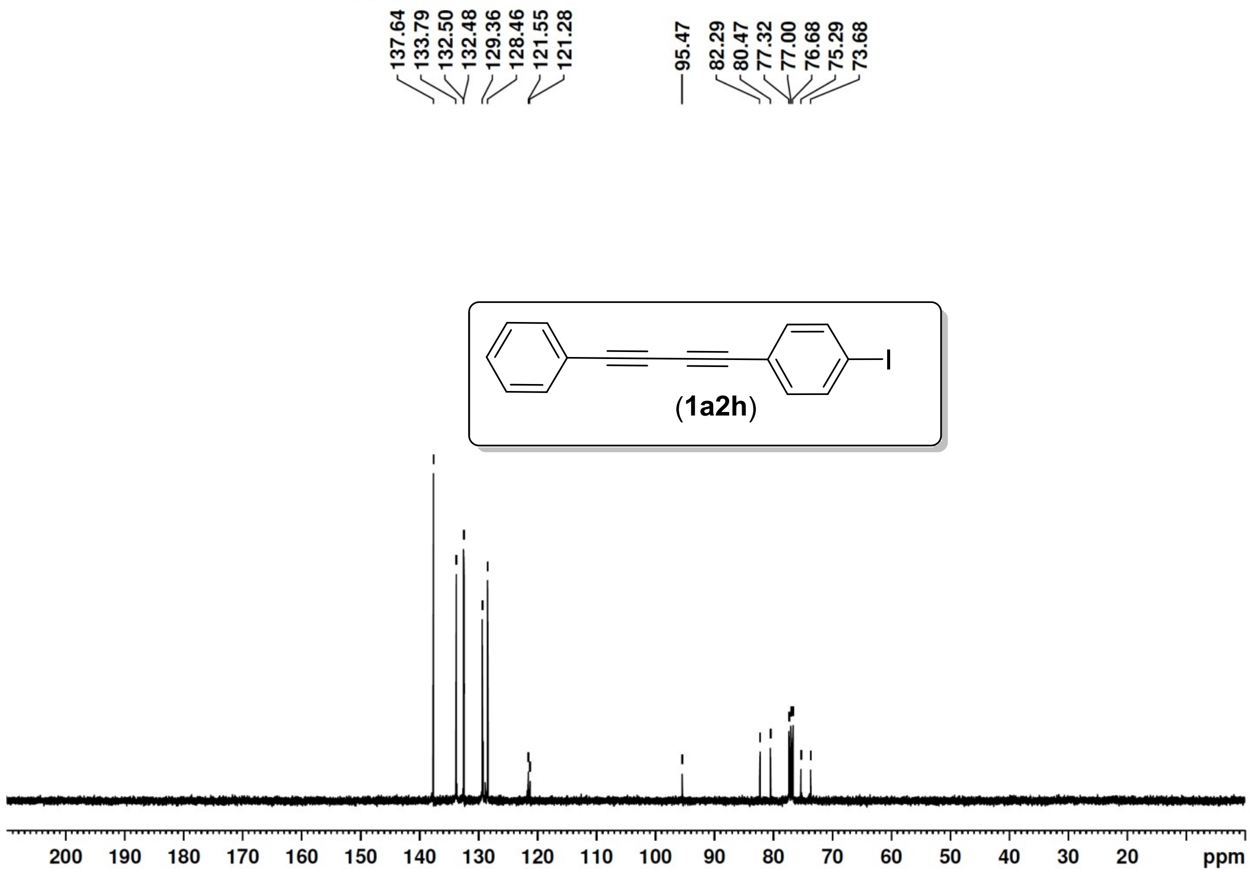

7.523
7.521
7.516
7.510
7.503
7.500
7.494
7.481
7.477
7.362
7.340
7.322
7.306
7.240
7.042
7.020
6.999
F
(1a2f)
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 ppm
4.00
2.92
2.12

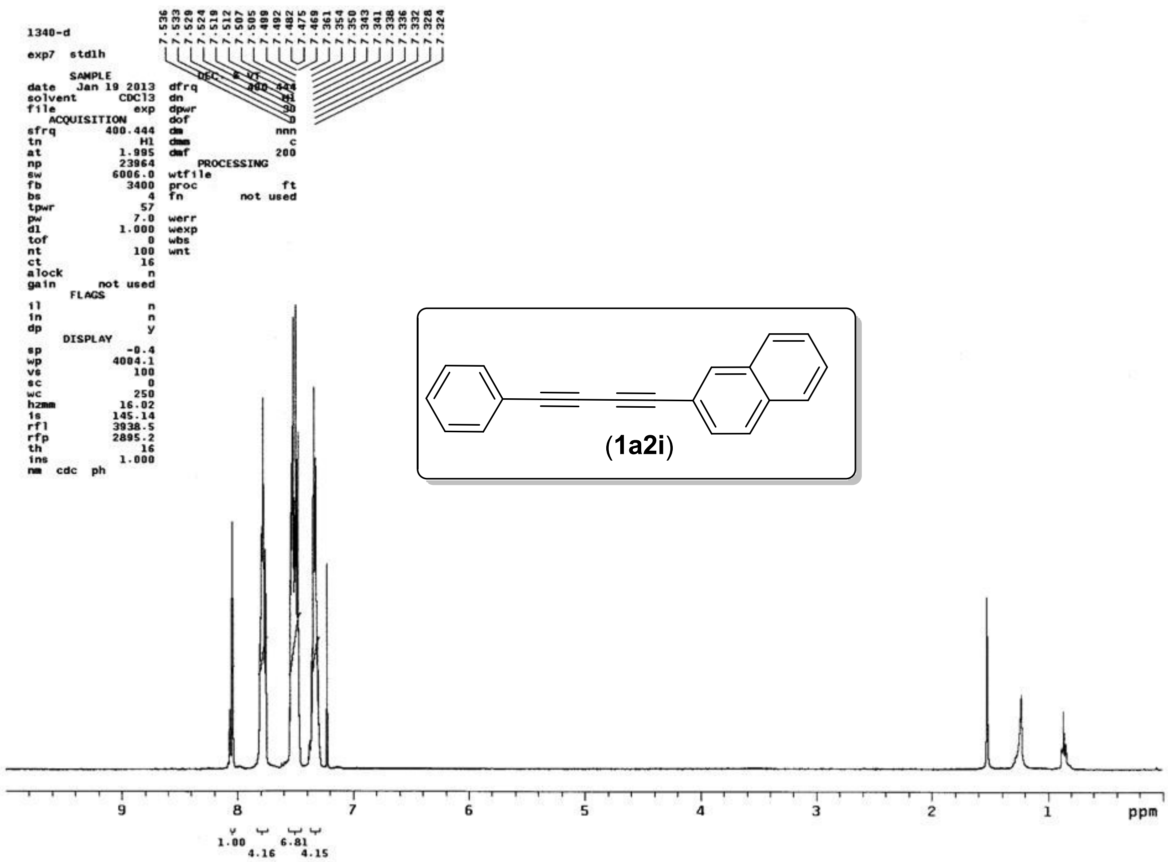

164.30
164.26
161.80
161.77
134.57
134.55
134.48
134.47
132.49
129.26
129.19
128.44
128.43
121.79
121.68
116.01
115.99
115.79
115.77
81.54
80.42
77.32
77.00
76.68
73.90
73.73
F
(1a2f)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


7.518
7.516
7.505
7.502
7.441
7.438
7.427
7.364
7.354
7.352
7.336
7.326
7.324
7.309
7.305
7.294
7.290
7.240
Cl
(1a2g)
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 ppm
1.93
2.31
5.04

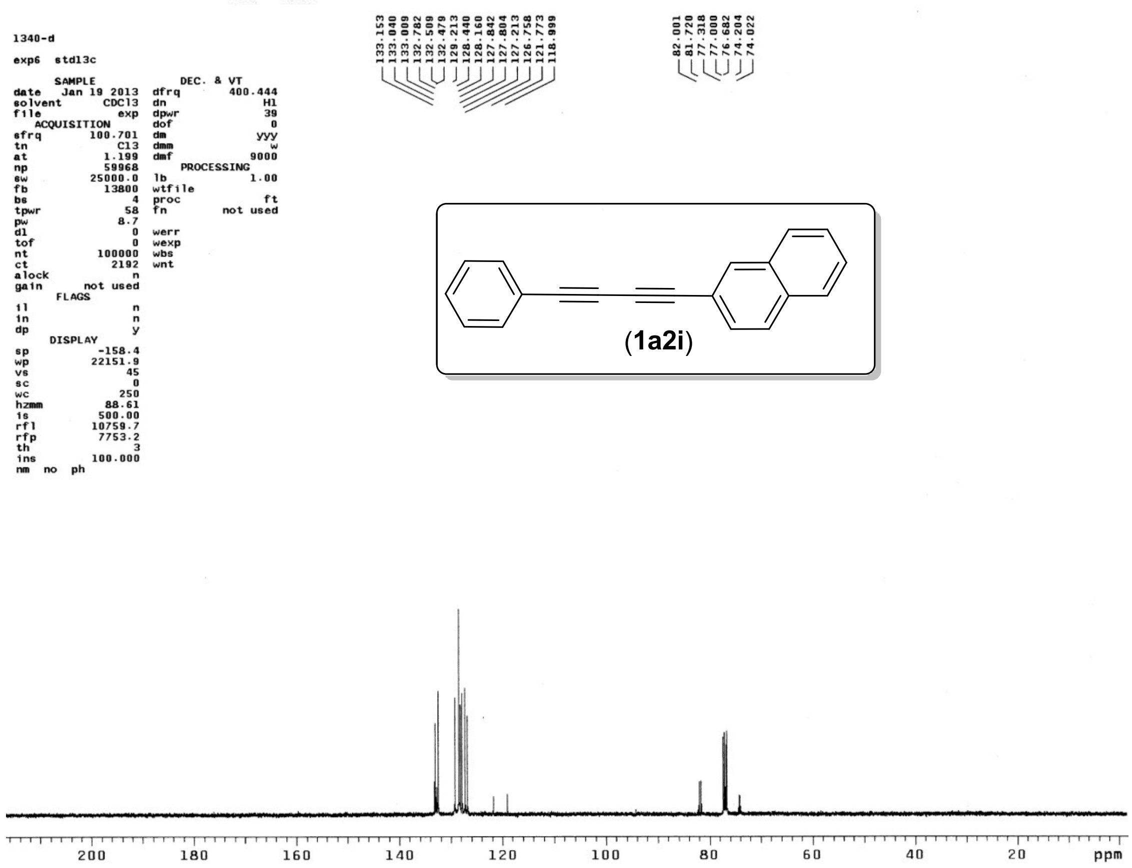

135.37
133.66
132.51
129.34
128.85
128.46
121.58
120.30
82.10
80.27
77.21
77.00
76.79
74.87
73.64
Cl
(1a2g)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


7.677
7.657
7.522
7.519
7.503
7.367
7.350
7.341
7.322
7.309
7.306
7.240
7.232
7.211
(1a2h)
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 ppm
1.94
2.20
3.20
1.96

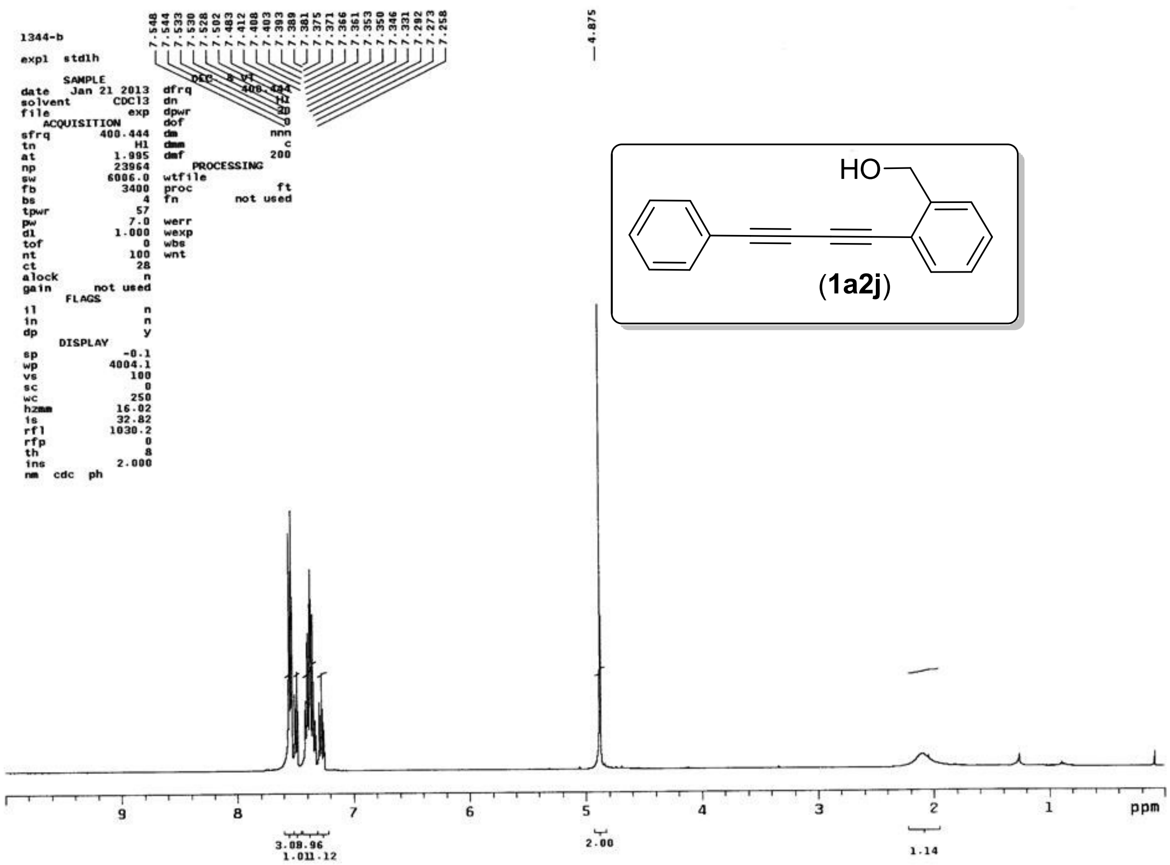

137.64
133.79
132.50
132.48
129.36
128.46
121.55
121.28
95.47
82.29
80.47
77.32
77.00
76.68
75.29
73.68
(1a2h)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

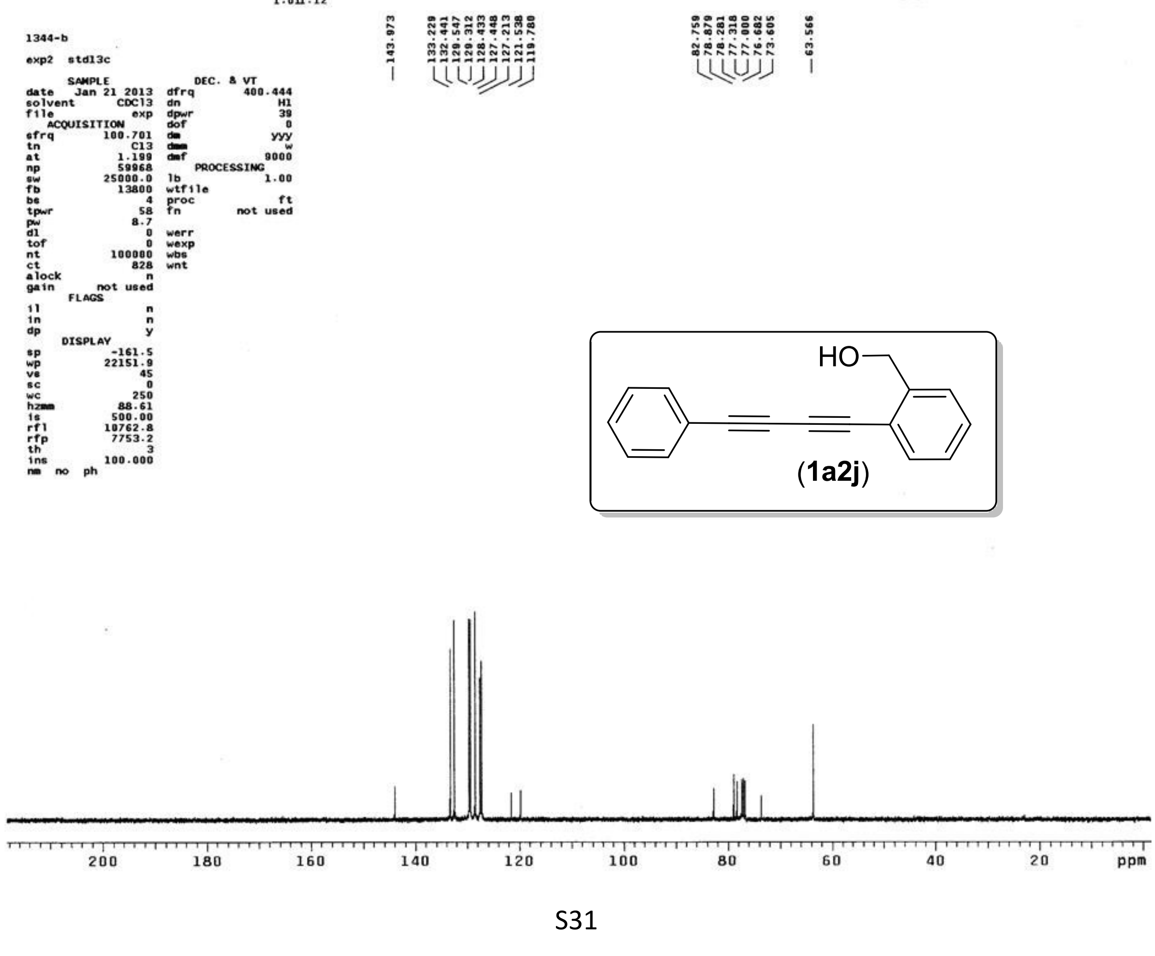


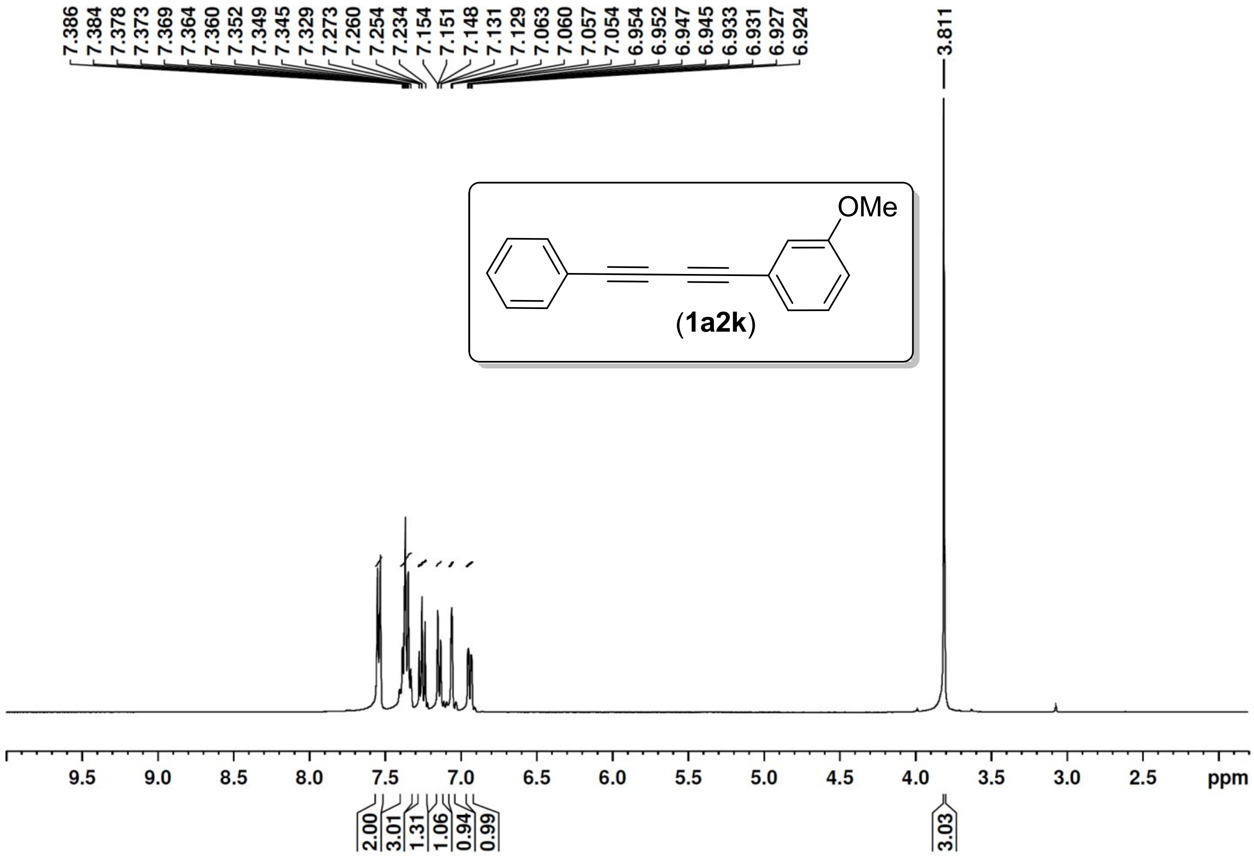
7.536 7.533 7.529 7.524 7.519 7.512 7.507 7.505 7.499 7.492 7.482 7.475 7.469 7.361 7.354 7.350 7.343 7.341 7.338 7.336 7.332 7.328 7.324
1340-d
exp7 std1h
SAMPLE
date Jan 19 2013
solvent CDCl3
file exp
ACQUISITION
sfrq 400.444
tn H1
at 1.995
np 23964
sw 6006.0
fb 3400
bs 4
tpwr 57
pw 7.0
d1 1.000
tof 0
nt 100
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -0.4
wp 4004.1
vs 100
sc 0
wc 250
hzmm 16.02
is 145.14
rfl 3938.5
rfp 2895.2
th 16
ins 1.000
nm cdc ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
(1a2i)
9 8 7 6 5 4 3 2 1 ppm
1.00 4.16 6.81 4.15

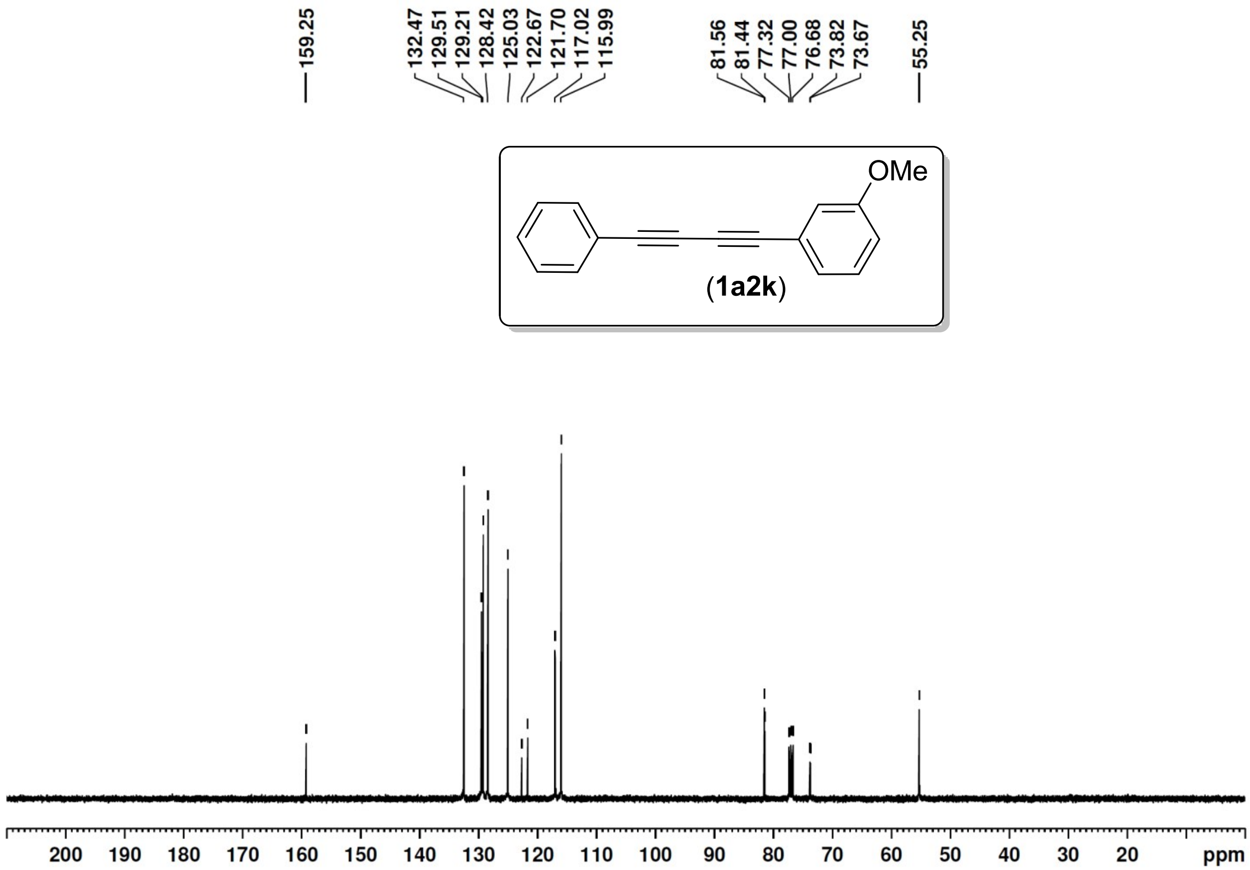
133.153 133.040 133.009 132.782 132.509 132.479 129.213 128.440 128.160 127.842 127.804 127.213 126.758 121.773 118.999
82.001 81.720 77.318 77.000 76.682 74.204 74.022
1340-d
exp6 std13c
SAMPLE
date Jan 19 2013
solvent CDCl3
file exp
ACQUISITION
sfrq 100.701
tn C13
at 1.199
np 59968
sw 25000.0
fb 13800
bs 4
tpwr 58
pw 8.7
d1 0
tof 0
nt 100000
ct 2192
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -158.4
wp 22151.9
vs 45
sc 0
wc 250
hzmm 88.61
is 500.00
rfl 10759.7
rfp 7753.2
th 3
ins 100.000
nm no ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 39
dof 0
dm yyy
dmm w
dmf 9000
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
(1a2i)
200 180 160 140 120 100 80 60 40 20 ppm


1344-b
exp1 std1h
SAMPLE
date Jan 21 2013
solvent CDCl3
file exp
ACQUISITION
sfrq 400.444
tn H1
at 1.995
np 23964
sw 6006.0
fb 3400
bs 4
tpwr 57
pw 7.0
d1 1.000
tof 0
nt 100
ct 28
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -0.1
wp 4004.1
vs 100
sc 0
wc 250
hzmm 16.02
is 32.82
rfl 1030.2
rfp 0
th 8
ins 2.000
nm cdc ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
7.548 7.544 7.533 7.530 7.528 7.502 7.483 7.412 7.408 7.403 7.393 7.389 7.381 7.375 7.371 7.366 7.361 7.353 7.350 7.346 7.331 7.292 7.273 7.258
4.875
HO
(1a2j)
9 8 7 6 5 4 3 2 1 ppm
3.08.96
1.011.12
2.00
1.14

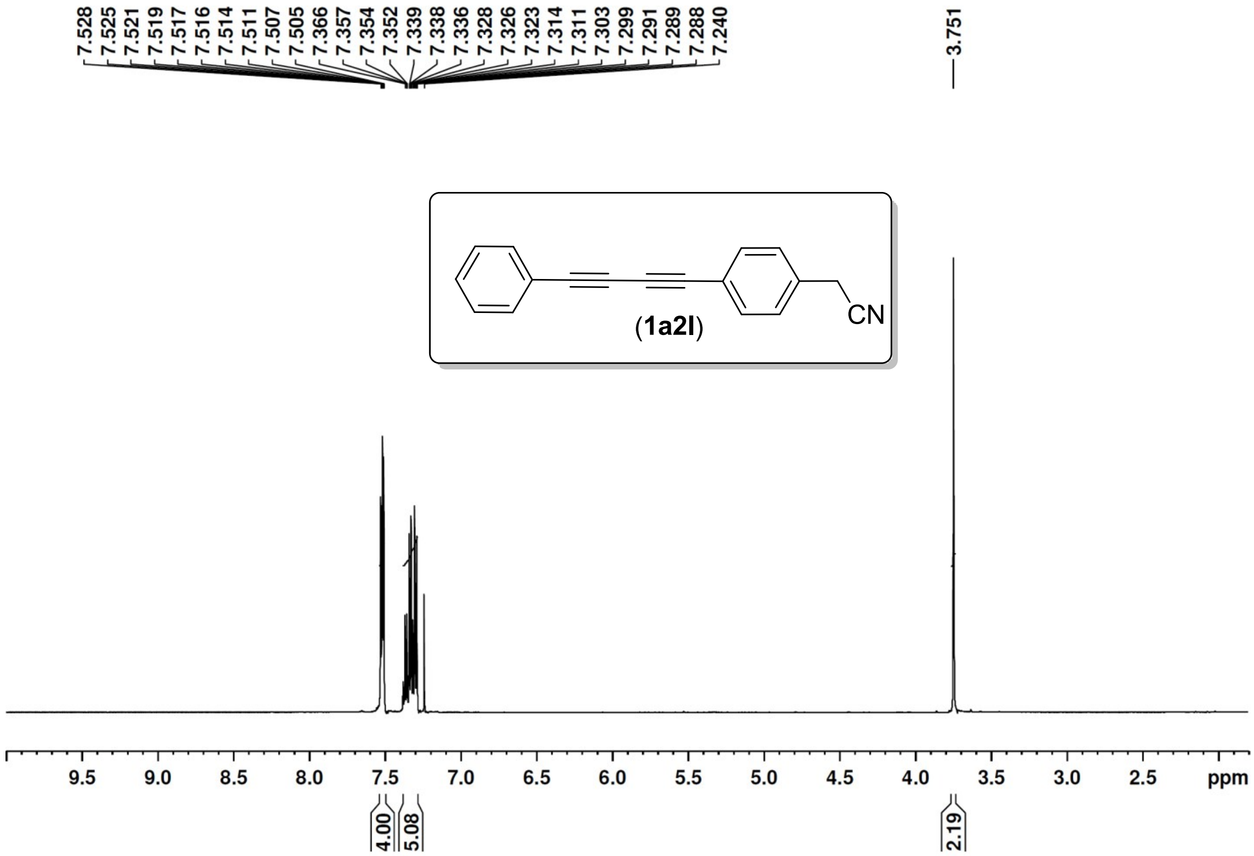

1344-b
exp2 std13c
SAMPLE
date Jan 21 2013
solvent CDCl3
file exp
ACQUISITION
sfrq 100.701
tn C13
at 1.199
np 59968
sw 25000.0
fb 13800
bs 4
tpwr 58
pw 8.7
d1 0
tof 0
nt 100000
ct 828
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -161.5
wp 22151.9
vs 45
sc 0
wc 250
hzmm 88.61
is 500.00
rfl 10762.8
rfp 7753.2
th 3
ins 100.000
nm no ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 39
dof 0
dm yyy
dmm w
dmf 9000
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
143.973
133.229 132.441 129.547 129.312 128.433 127.448 127.213 121.538 119.780
82.759 78.879 78.281 77.318 77.000 76.682 73.605
63.566
HO
(1a2j)
200 180 160 140 120 100 80 60 40 20 ppm

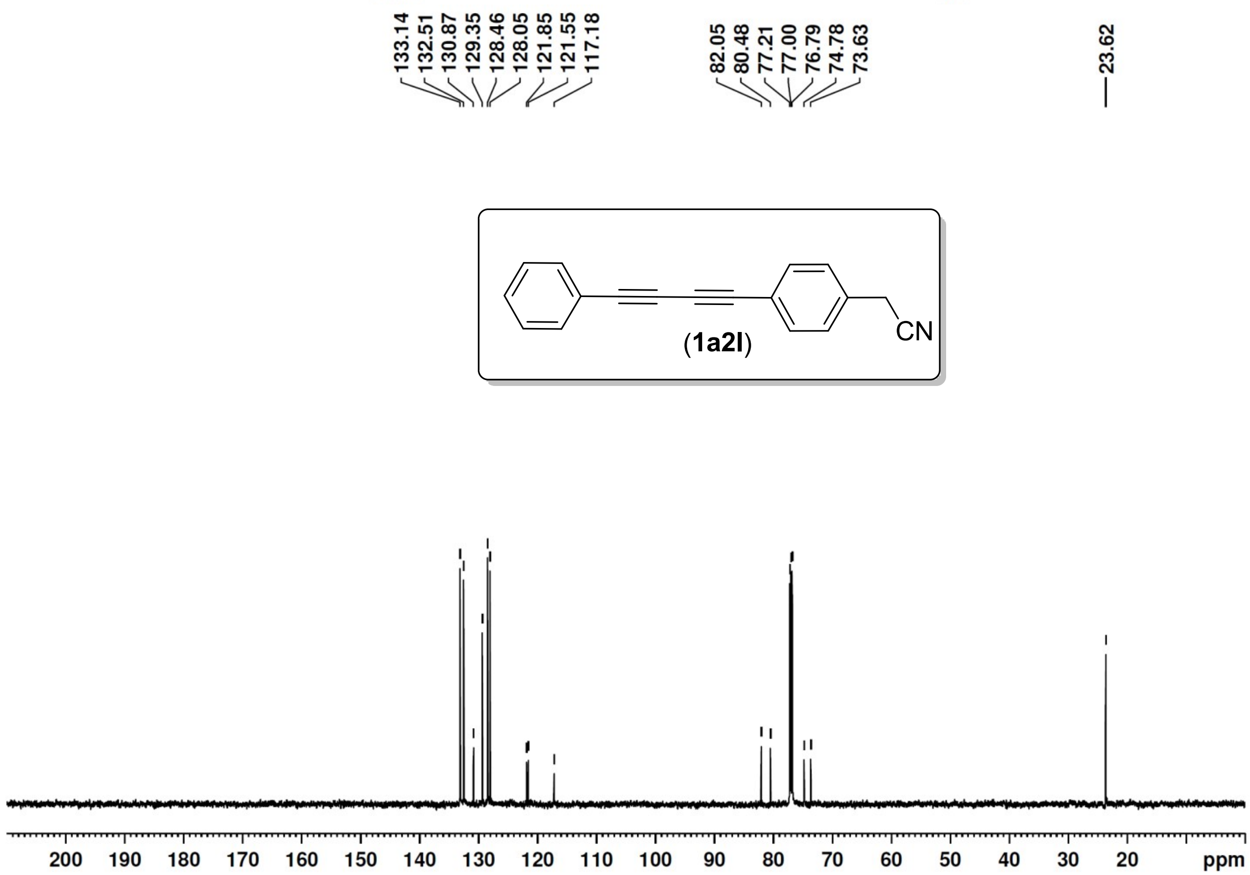


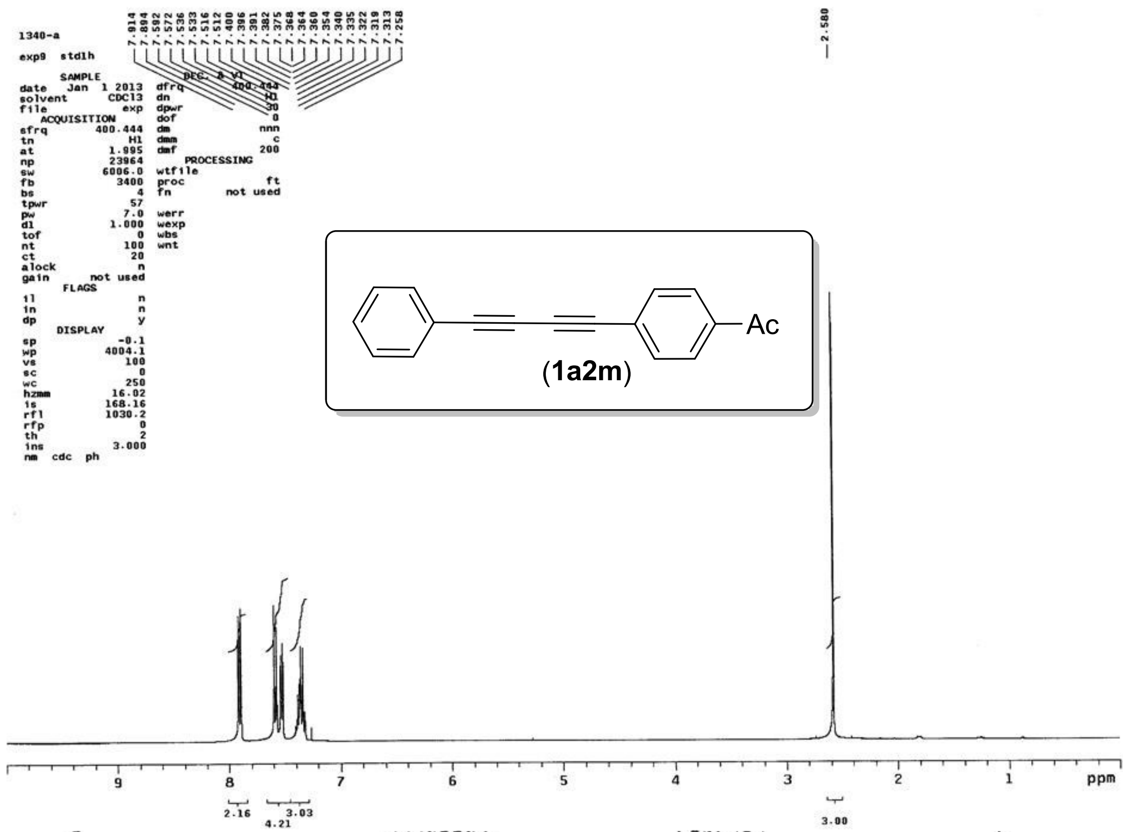
7.386 7.384 7.378 7.373 7.369 7.364 7.360 7.352 7.349 7.345 7.329 7.273 7.260 7.254 7.234 7.154 7.151 7.148 7.131 7.129 7.063 7.060 7.057 7.054 6.954 6.952 6.947 6.945 6.933 6.931 6.927 6.924
3.811
OMe
(1a2k)
2.00 3.01 1.31 1.06 0.94 0.99
3.03
ppm

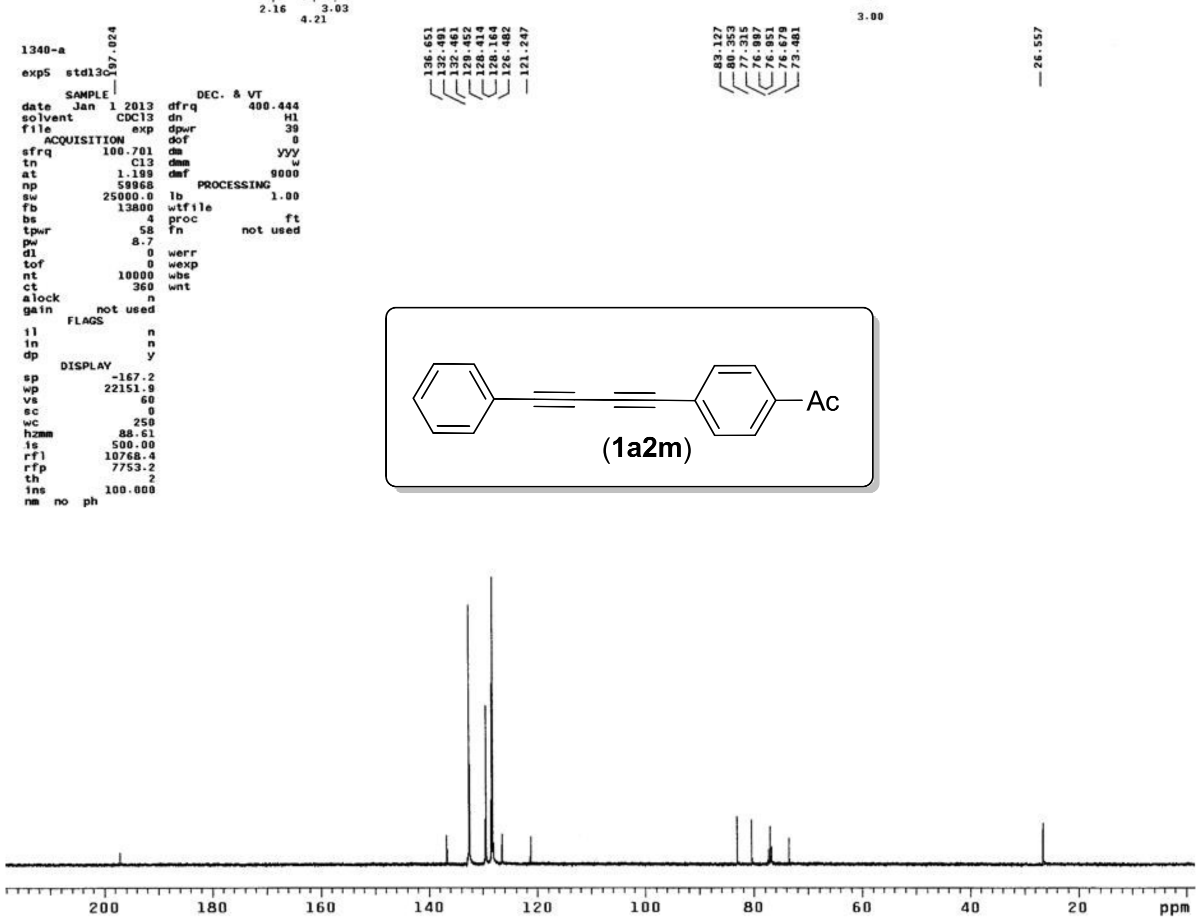



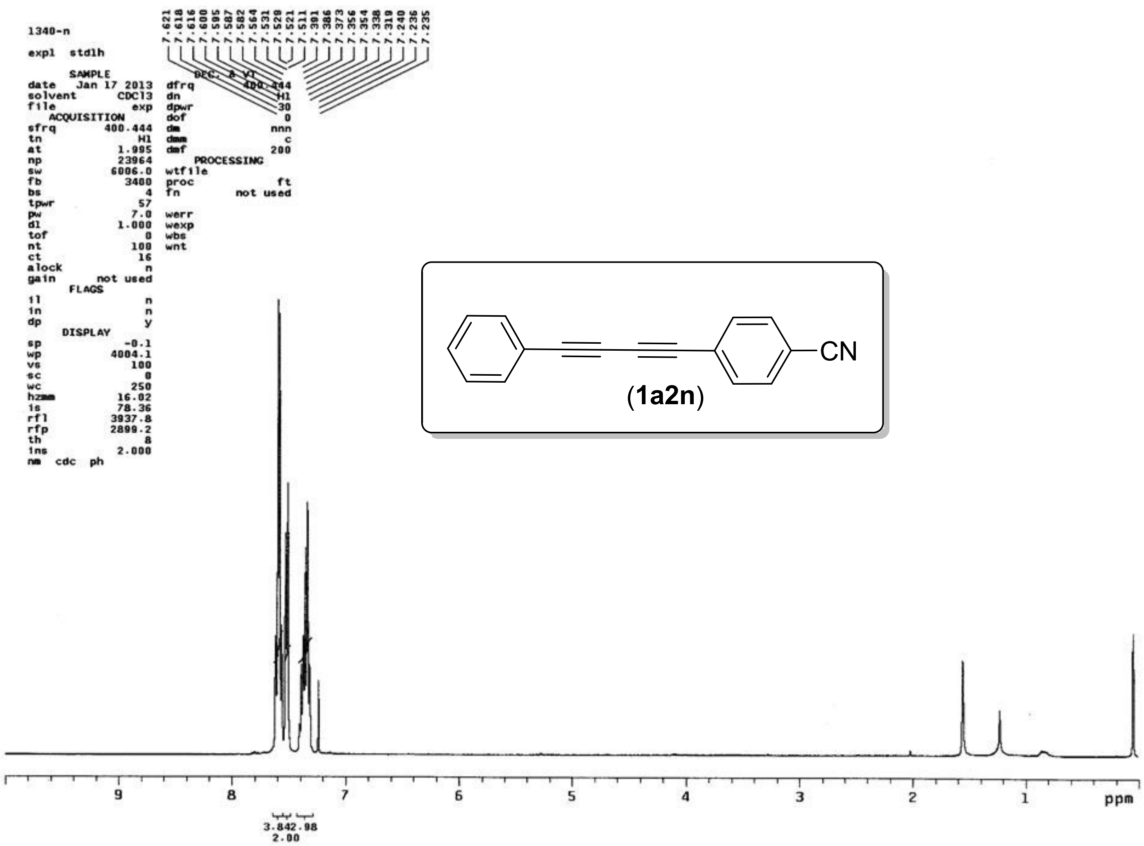
159.25
132.47 129.51 129.21 128.42 125.03 122.67 121.70 117.02 115.99
81.56 81.44 77.32 77.00 76.68 73.82 73.67
55.25
OMe
(1a2k)
ppm

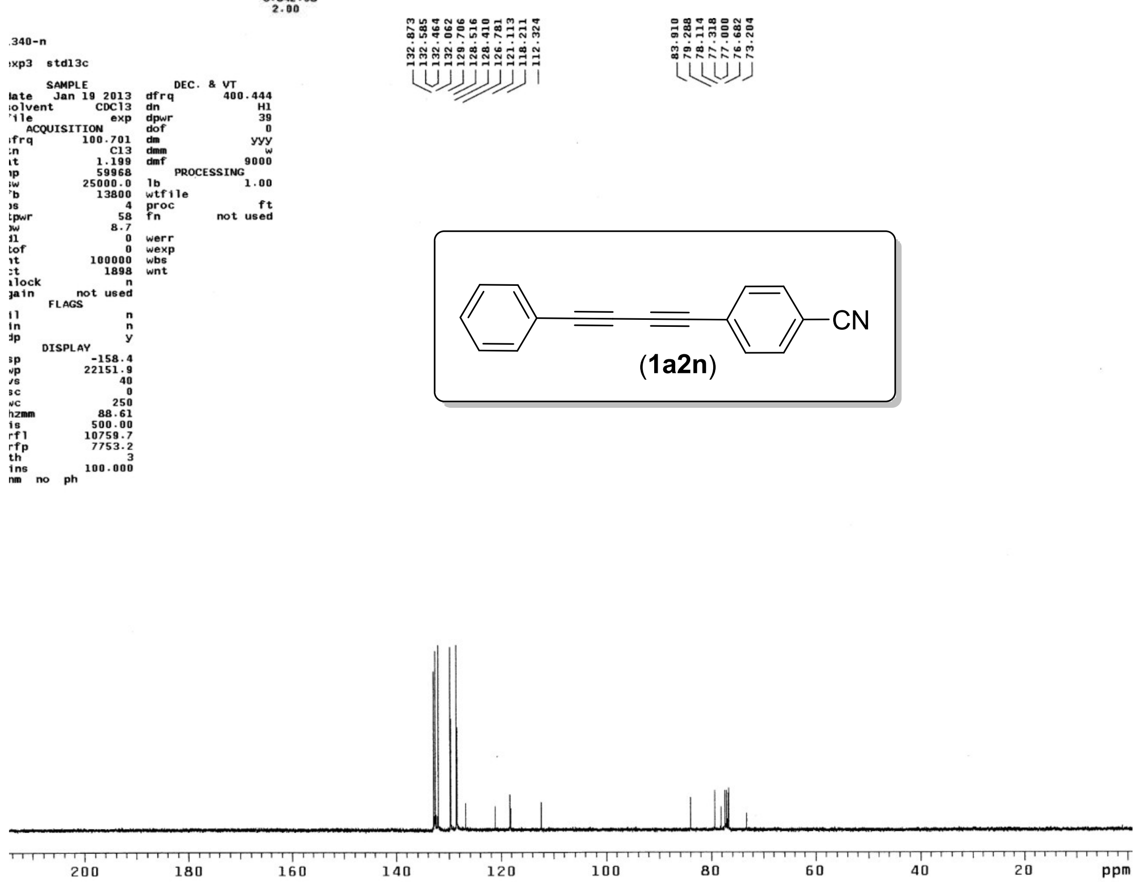

7.528
7.525
7.521
7.519
7.517
7.516
7.514
7.511
7.507
7.505
7.366
7.357
7.354
7.352
7.339
7.338
7.336
7.328
7.326
7.323
7.314
7.311
7.303
7.299
7.291
7.289
7.288
7.240
3.751
CN
(1a2l)
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm
4.00
5.08
2.19

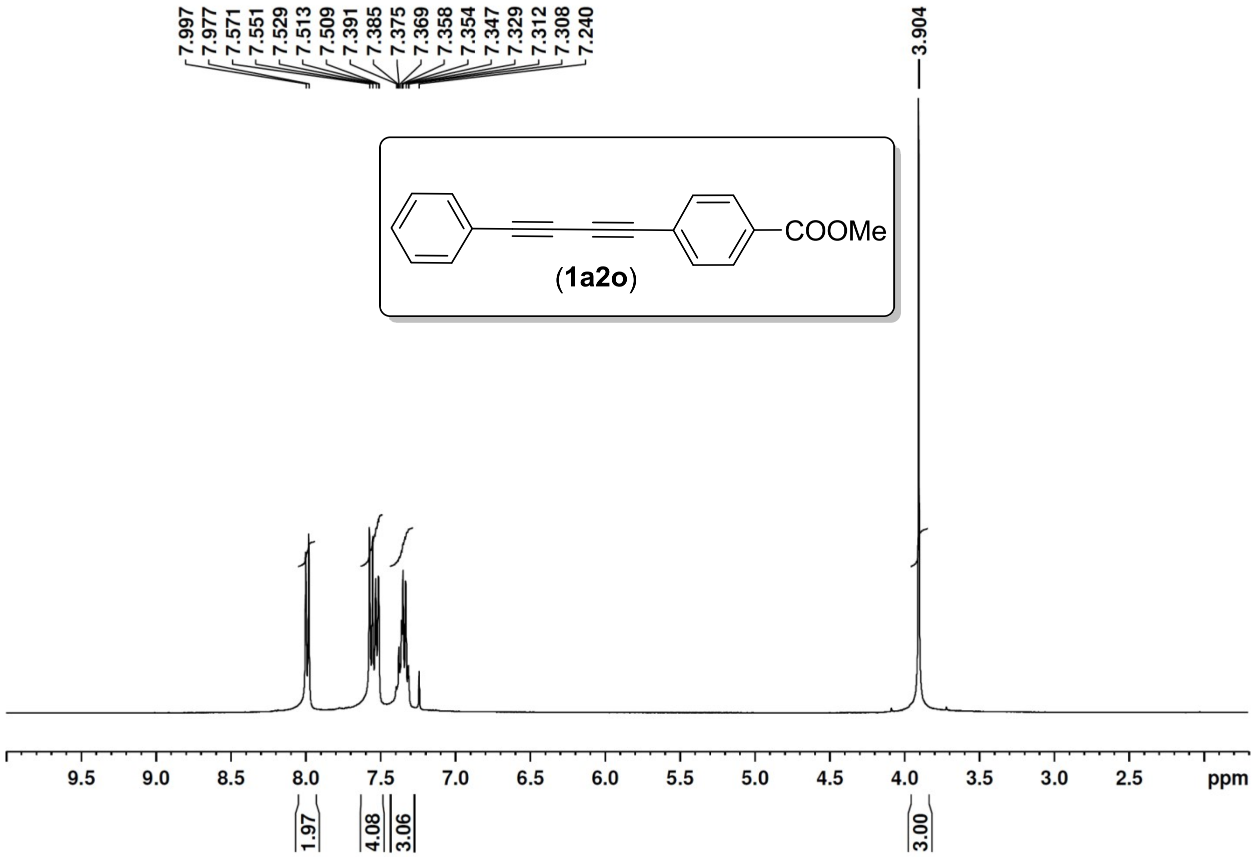

133.14
132.51
130.87
129.35
128.46
128.05
121.85
121.55
117.18
82.05
80.48
77.21
77.00
76.79
74.78
73.63
23.62
CN
(1a2l)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

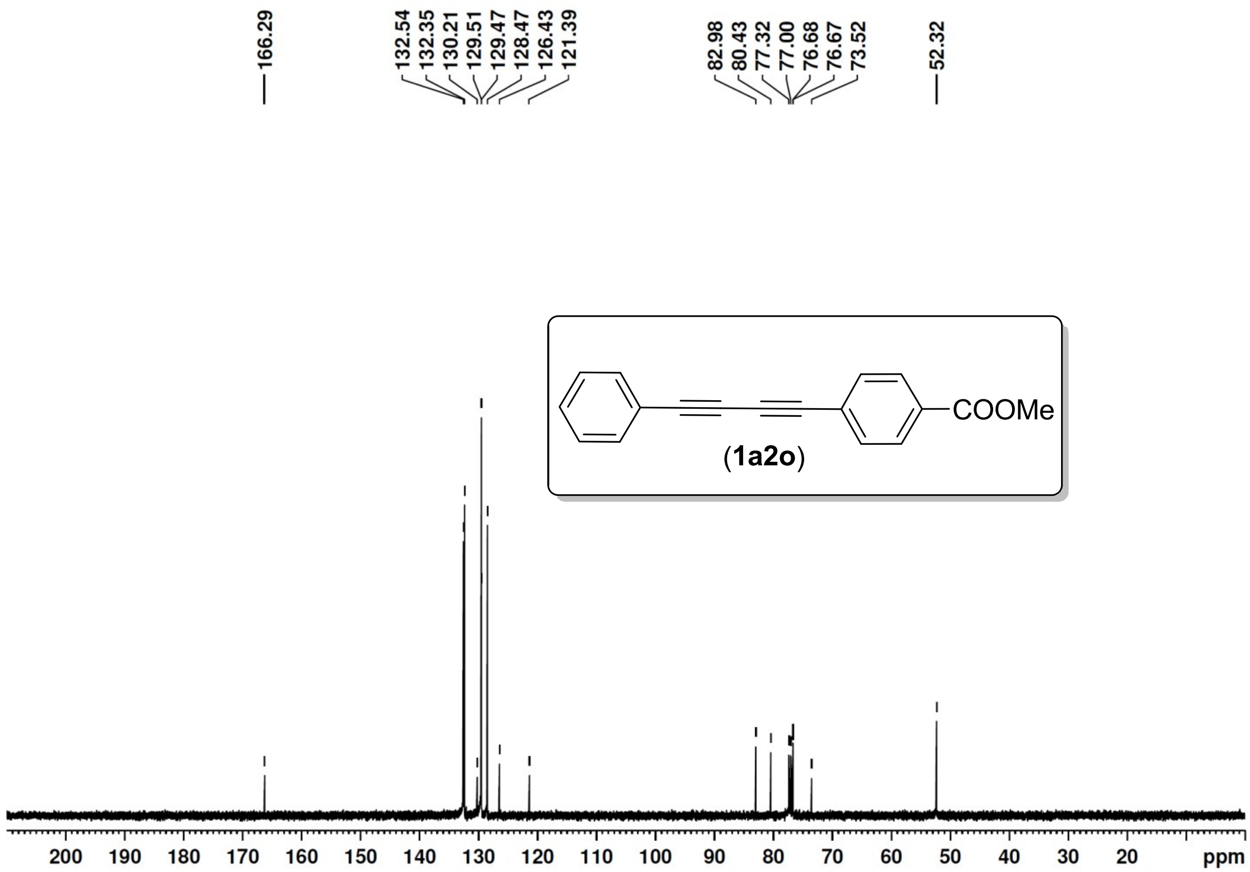

1340-a
exp9 std1h
7.914 7.894 7.592 7.572 7.536 7.533 7.516 7.512 7.400 7.396 7.391 7.382 7.375 7.368 7.364 7.360 7.354 7.340 7.335 7.322 7.319 7.313 7.258
2.580
Ac
(1a2m)
2.16
4.21
3.03
3.00
ppm

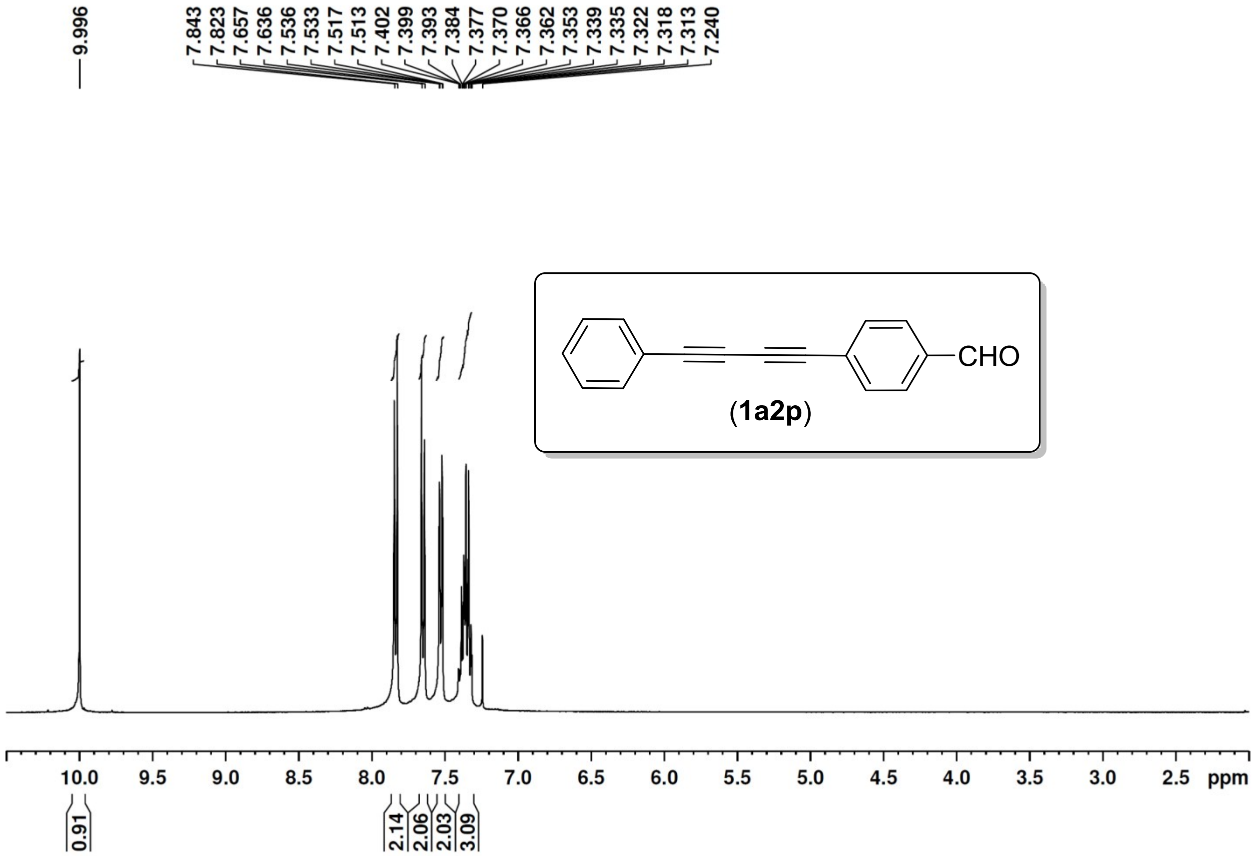

1340-a
exp5 std13c
197.024
136.651 132.491 132.461 129.452 128.414 128.164 126.482 121.247
83.127 80.353 77.315 76.997 76.951 76.679 73.481
26.557
Ac
(1a2m)
ppm

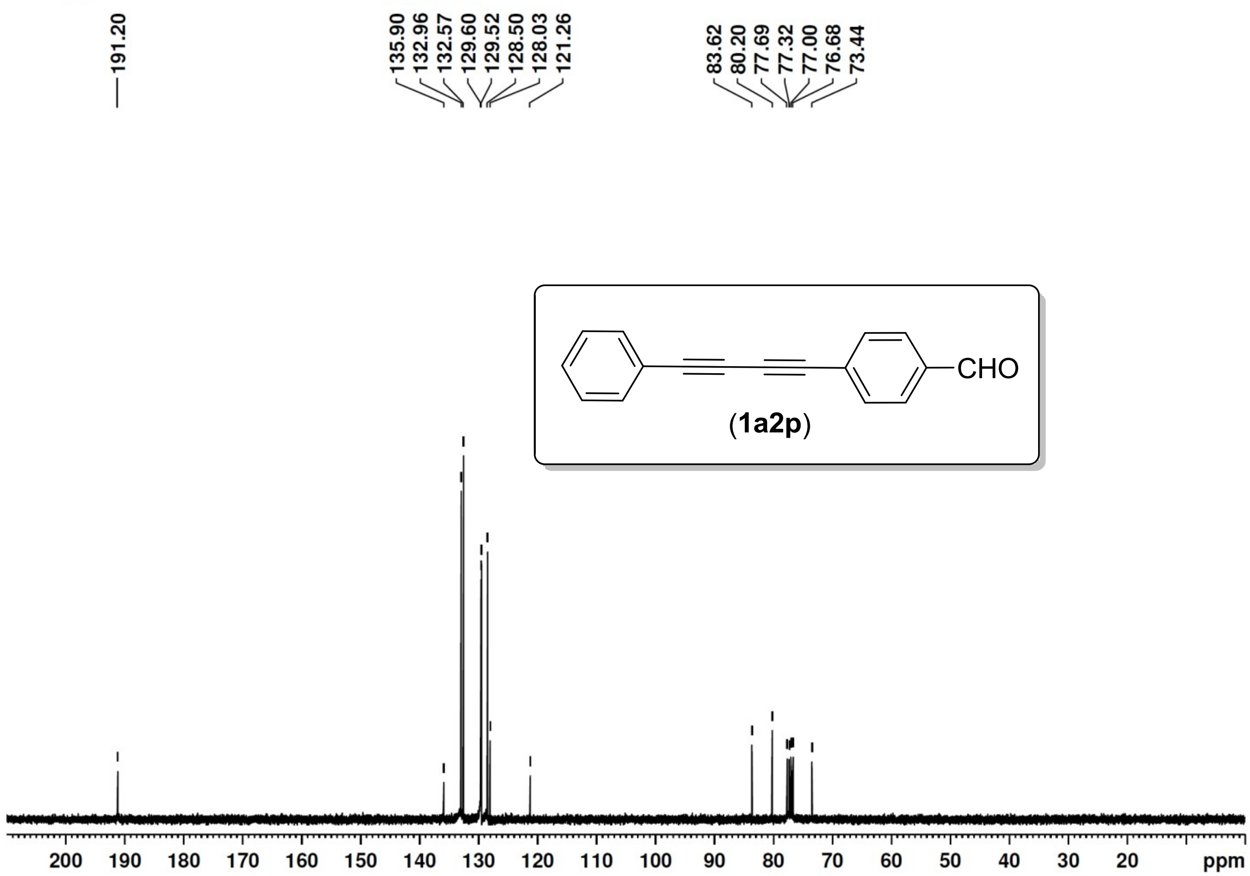

1340-n
expl stdlh
7.621 7.618 7.616 7.600 7.595 7.587 7.582 7.564 7.531 7.529 7.521 7.511 7.386 7.373 7.356 7.354 7.338 7.319 7.240 7.236 7.235
CN
(1a2n)
9 8 7 6 5 4 3 2 1 ppm
3.84 2.98
2.00

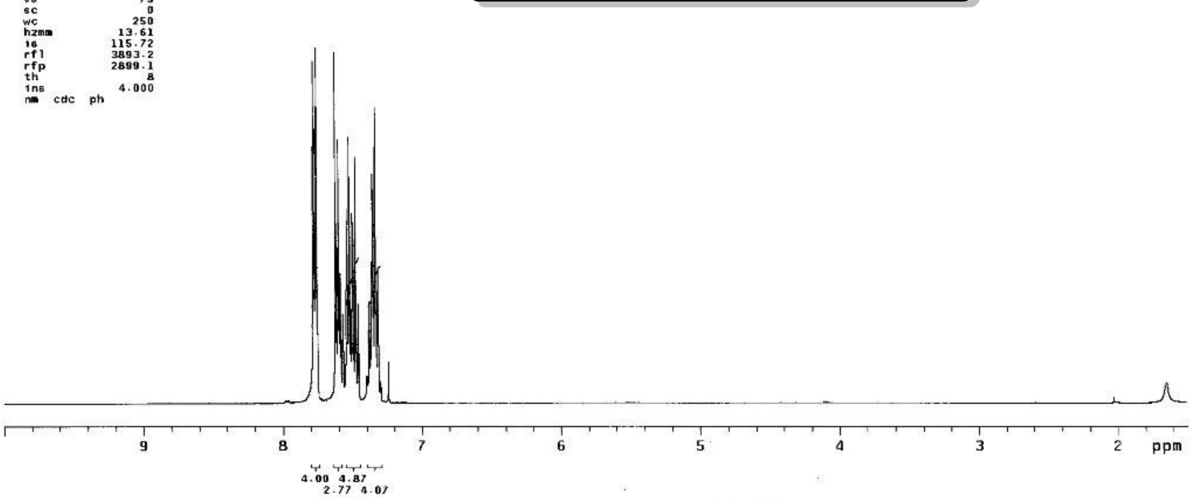

1340-n
exp3 stdl3c
132.873 132.585 132.464 132.062 129.706 128.516 128.410 126.781 121.113 118.211 112.324
83.910 79.288 78.114 77.318 77.000 76.682 73.204
CN
(1a2n)
200 180 160 140 120 100 80 60 40 20 ppm

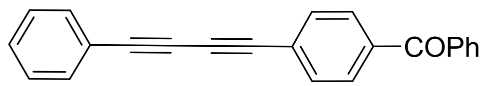

7.997
7.977
7.571
7.551
7.529
7.513
7.509
7.391
7.385
7.375
7.369
7.358
7.354
7.347
7.329
7.312
7.308
7.240
3.904
COOMe
(1a2o)
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 ppm
1.97
4.08
3.06
3.00


166.29
132.54
132.35
130.21
129.51
129.47
128.47
126.43
121.39
82.98
80.43
77.32
77.00
76.68
76.67
73.52
52.32
COOMe
(1a2o)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

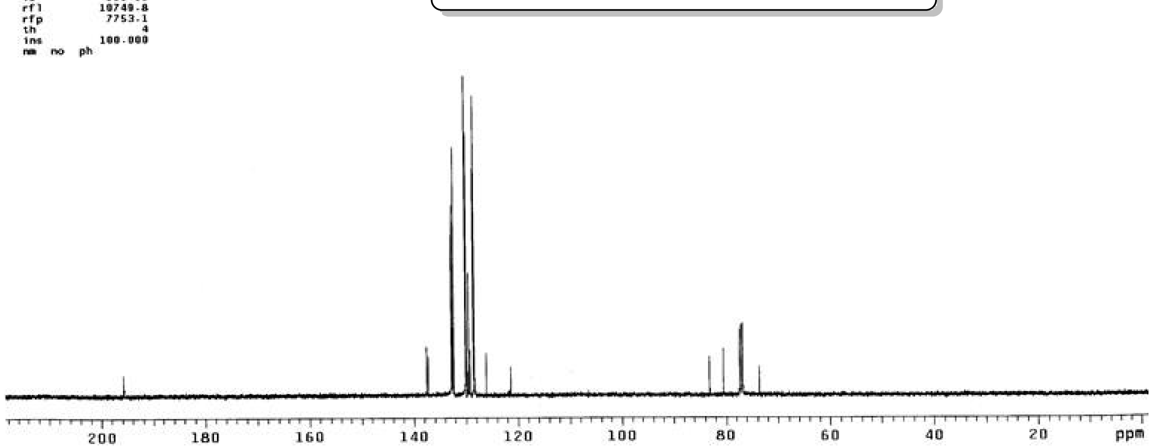

9.996
7.843
7.823
7.657
7.636
7.536
7.533
7.517
7.513
7.402
7.399
7.393
7.384
7.377
7.370
7.366
7.362
7.353
7.339
7.335
7.322
7.318
7.313
7.240
CHO
(1a2p)
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 ppm
0.91
2.14
2.06
2.03
3.09

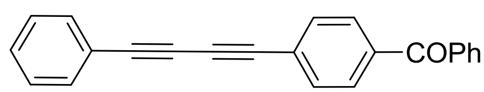

191.20
135.90
132.96
132.57
129.60
129.52
128.50
128.03
121.26
83.62
80.20
77.69
77.32
77.00
76.68
73.44
CHO
(1a2p)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


1401-1
exp4 std1h
7.761 7.758 7.754 7.618 7.601 7.598 7.592 7.587 7.571 7.568 7.540 7.537 7.521 7.517 7.509 7.503 7.496 7.476 7.457 7.378 7.370 7.365 7.360 7.356 7.351 7.337 7.333
4.00 2.77 4.87 4.07
9 8 7 6 5 4 3 2 ppm

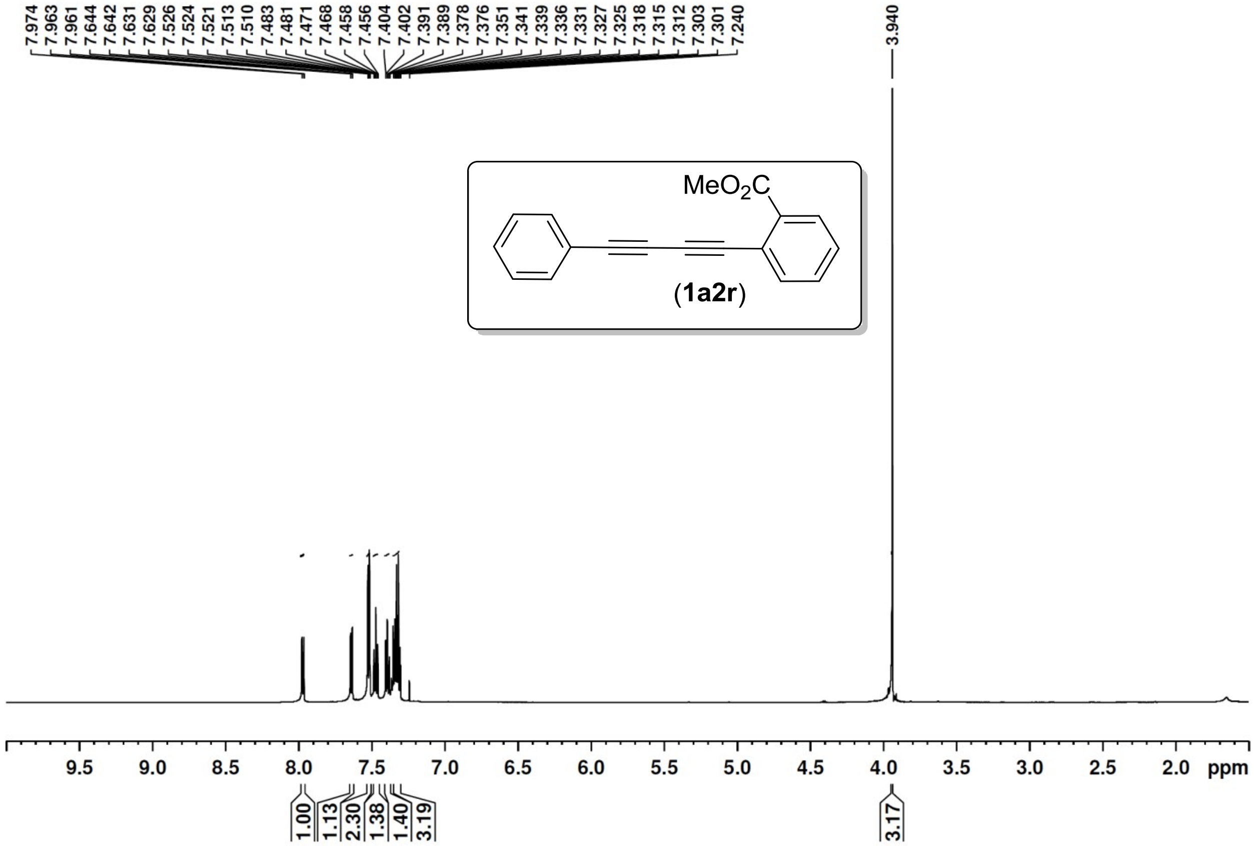

COPh

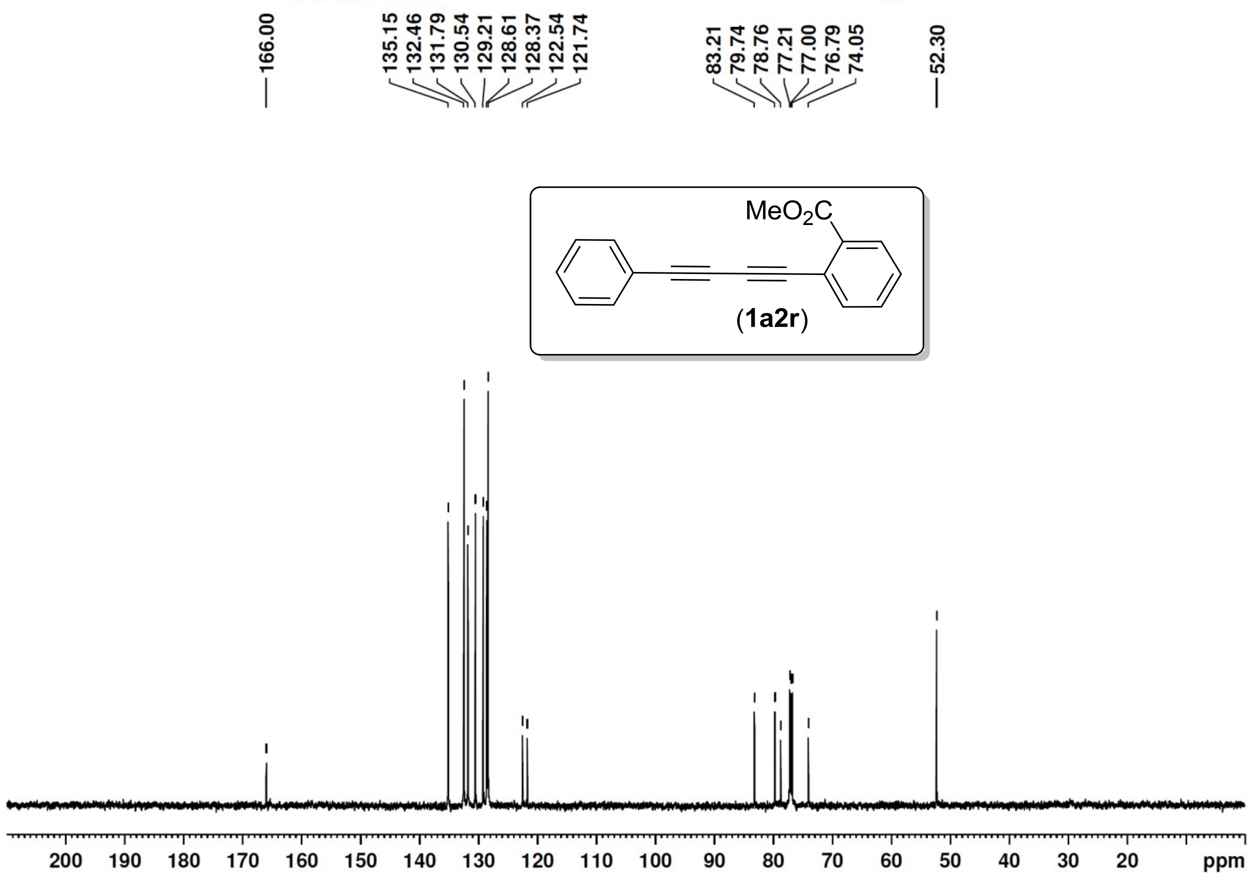
(**1a2q**)

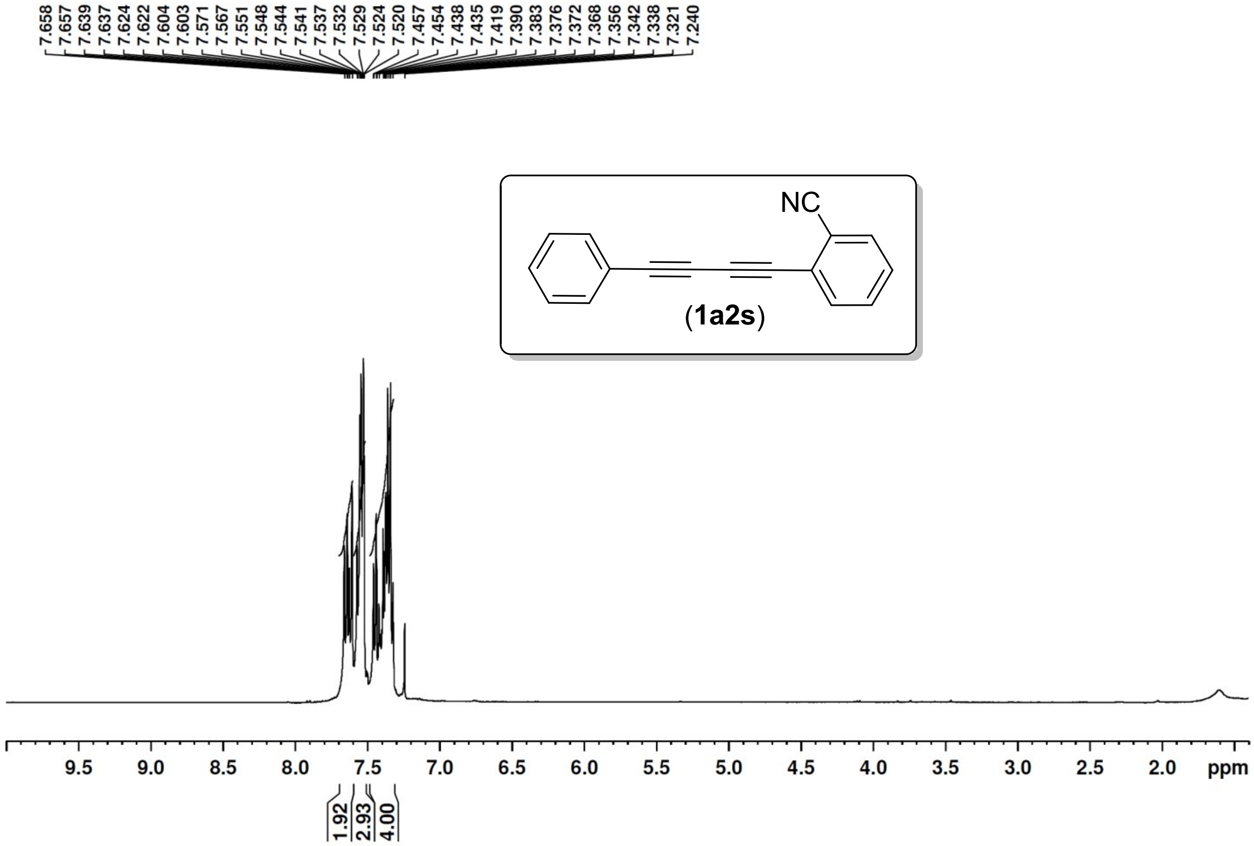

1401-1
exp5 std13c
195.680
137.504 137.148 132.647 132.541 132.457 132.275 129.995 129.934 129.487 129.176 128.472 128.404 128.373 125.971 121.402
83.069 80.478 77.318 77.000 76.765 76.682 73.583
200 180 160 140 120 100 80 60 40 20 ppm

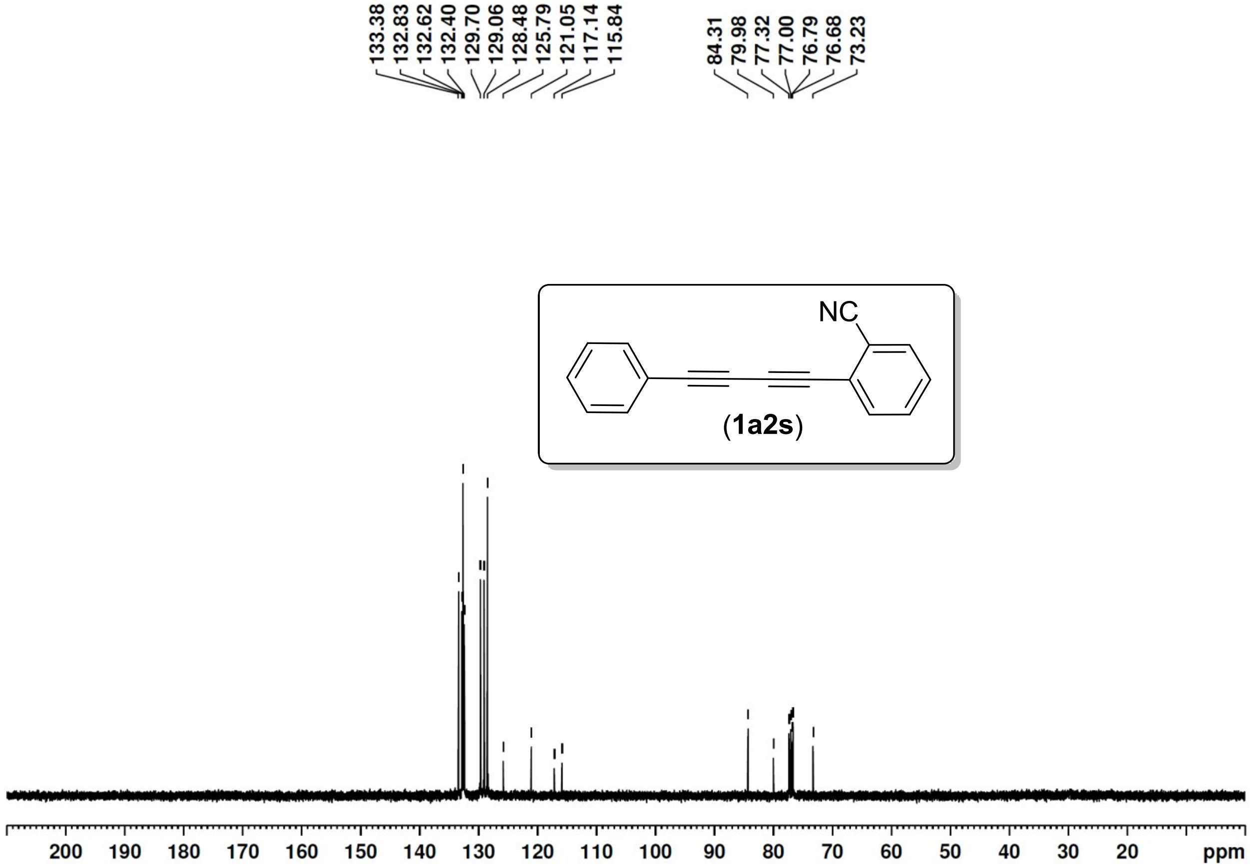

COPh

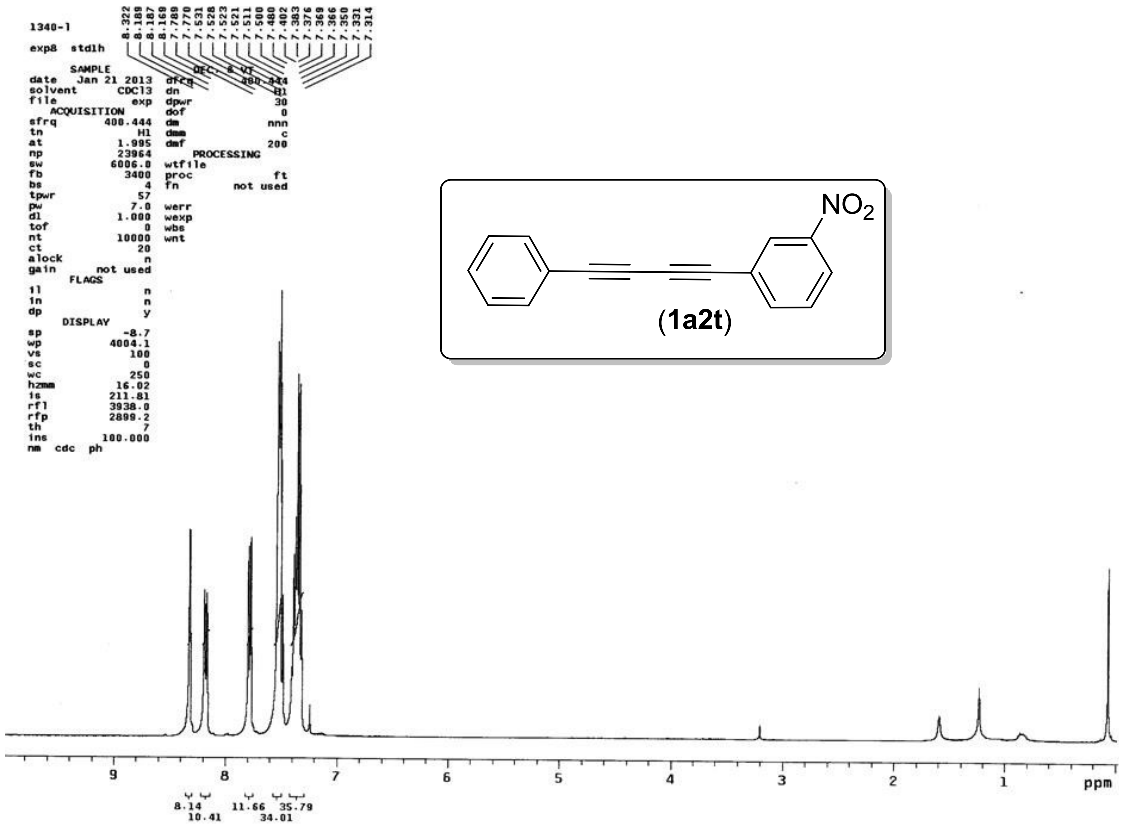
(**1a2q**)


7.974
7.963
7.961
7.644
7.642
7.631
7.629
7.526
7.524
7.521
7.513
7.510
7.483
7.481
7.471
7.468
7.458
7.456
7.404
7.402
7.391
7.389
7.378
7.376
7.351
7.341
7.339
7.336
7.331
7.327
7.325
7.318
7.315
7.312
7.303
7.301
7.240
3.940
MeO2C
(1a2r)
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm
1.00
1.13
2.30
1.38
1.40
3.19
3.17

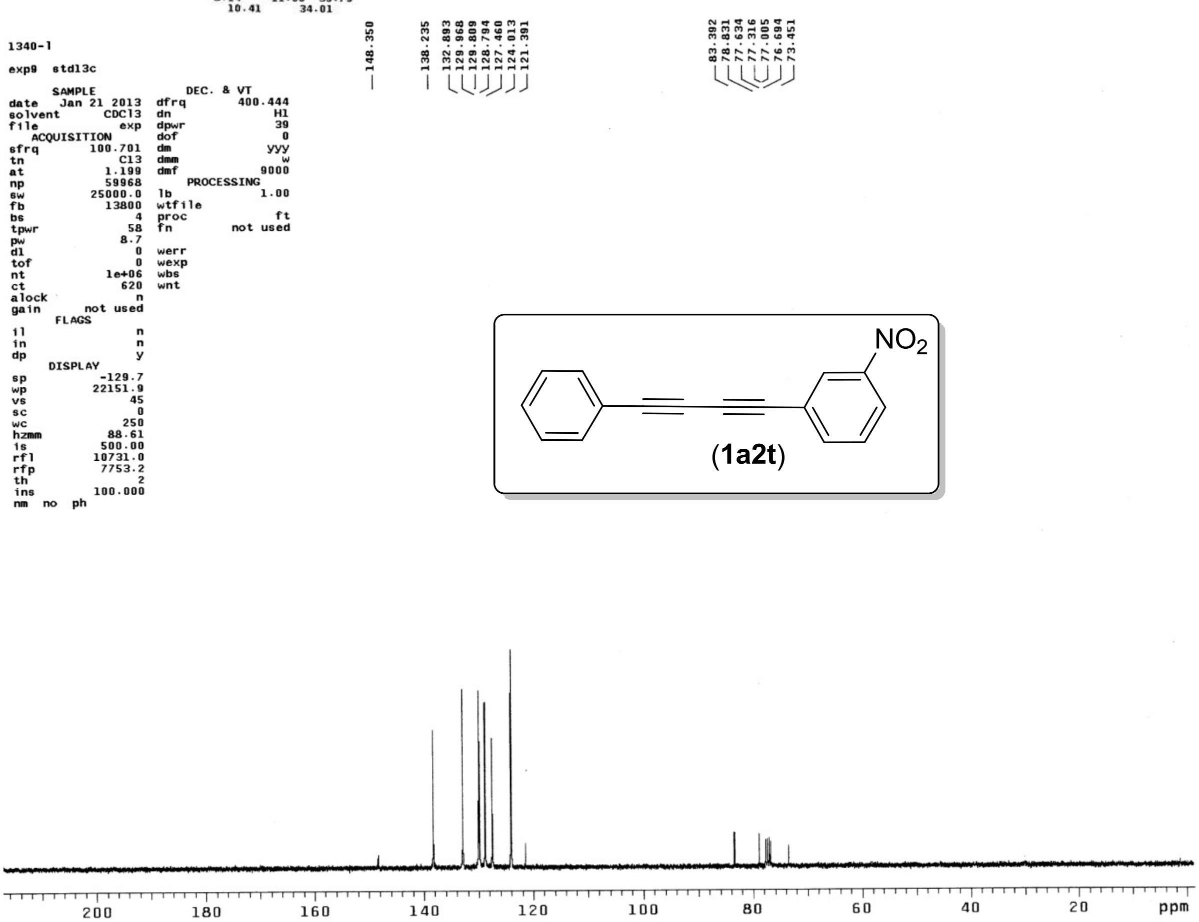

166.00
135.15
132.46
131.79
130.54
129.21
128.61
128.37
122.54
121.74
83.21
79.74
78.76
77.21
77.00
76.79
74.05
52.30
MeO2C
(1a2r)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


7.658 7.657 7.639 7.637 7.624 7.622 7.604 7.603 7.571 7.567 7.551 7.548 7.544 7.541 7.537 7.532 7.529 7.524 7.520 7.457 7.454 7.438 7.435 7.419 7.390 7.383 7.376 7.372 7.368 7.356 7.342 7.338 7.321 7.240
NC
(1a2s)
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 ppm
1.92 2.93 4.00

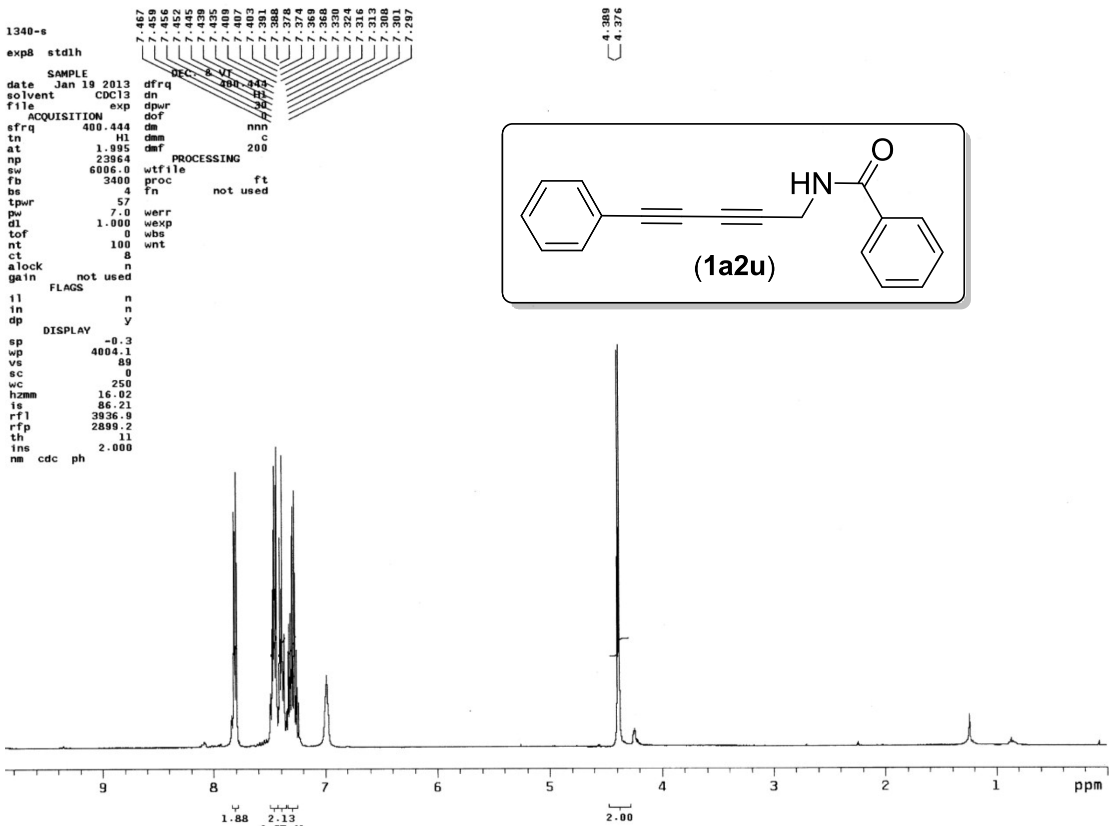

133.38 132.83 132.62 132.40 129.70 129.06 128.48 125.79 121.05 117.14 115.84
84.31 79.98 77.32 77.00 76.79 76.68 73.23
NC
(1a2s)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

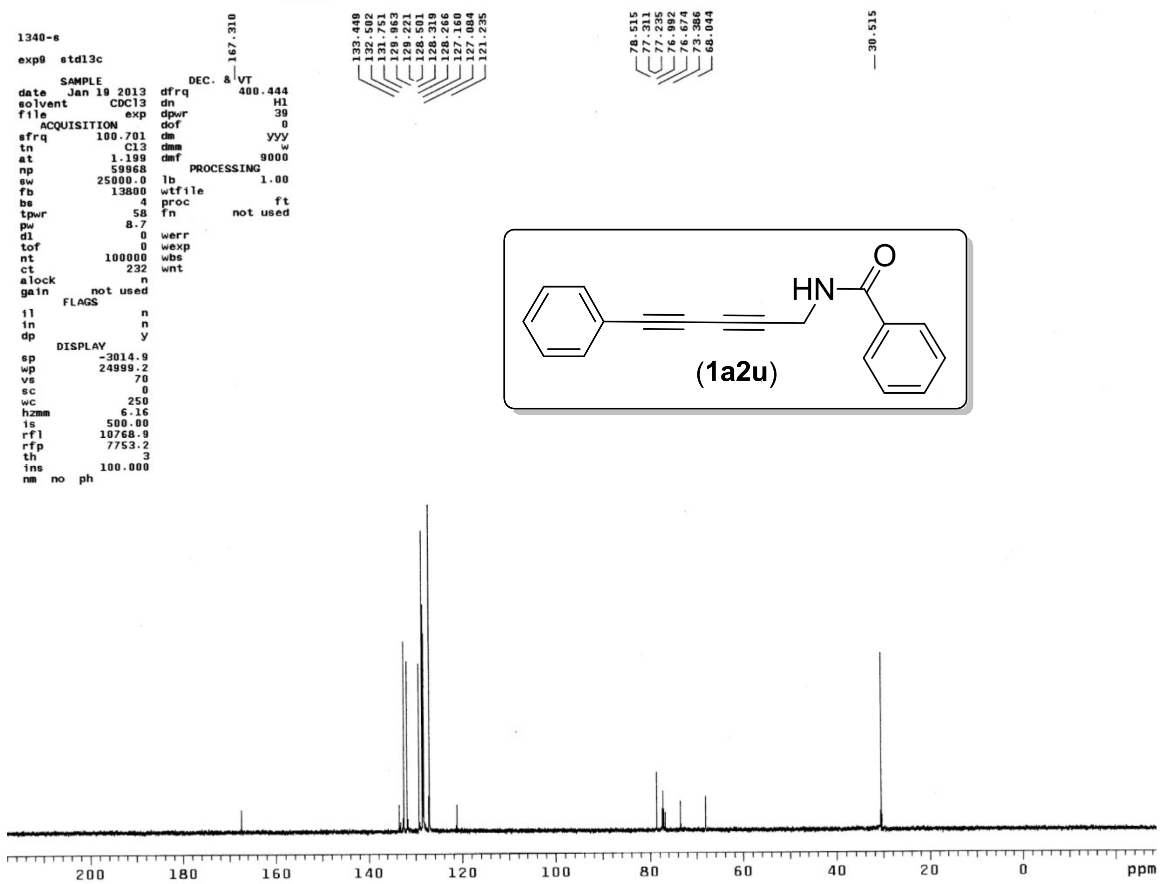

1340-1
exp8 stdlh
8.322 8.188 8.187 8.169 7.789 7.770 7.531 7.528 7.523 7.521 7.511 7.508 7.480 7.402 7.383 7.376 7.369 7.366 7.350 7.331 7.314
SAMPLE
date Jan 21 2013
solvent CDCl3
file exp
ACQUISITION
sfrq 400.444
tn H1
at 1.995
np 23964
sw 6006.0
fb 3400
bs 4
tpwr 57
pw 7.0
d1 1.000
tof 0
nt 10000
ct 20
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -8.7
wp 4004.1
vs 100
sc 0
wc 250
hzmm 16.02
is 211.81
rfl 3938.0
rfp 2899.2
th 7
ins 100.000
nm cdc ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
9 8 7 6 5 4 3 2 1 ppm
8.14 10.41 11.66 34.01 35.79

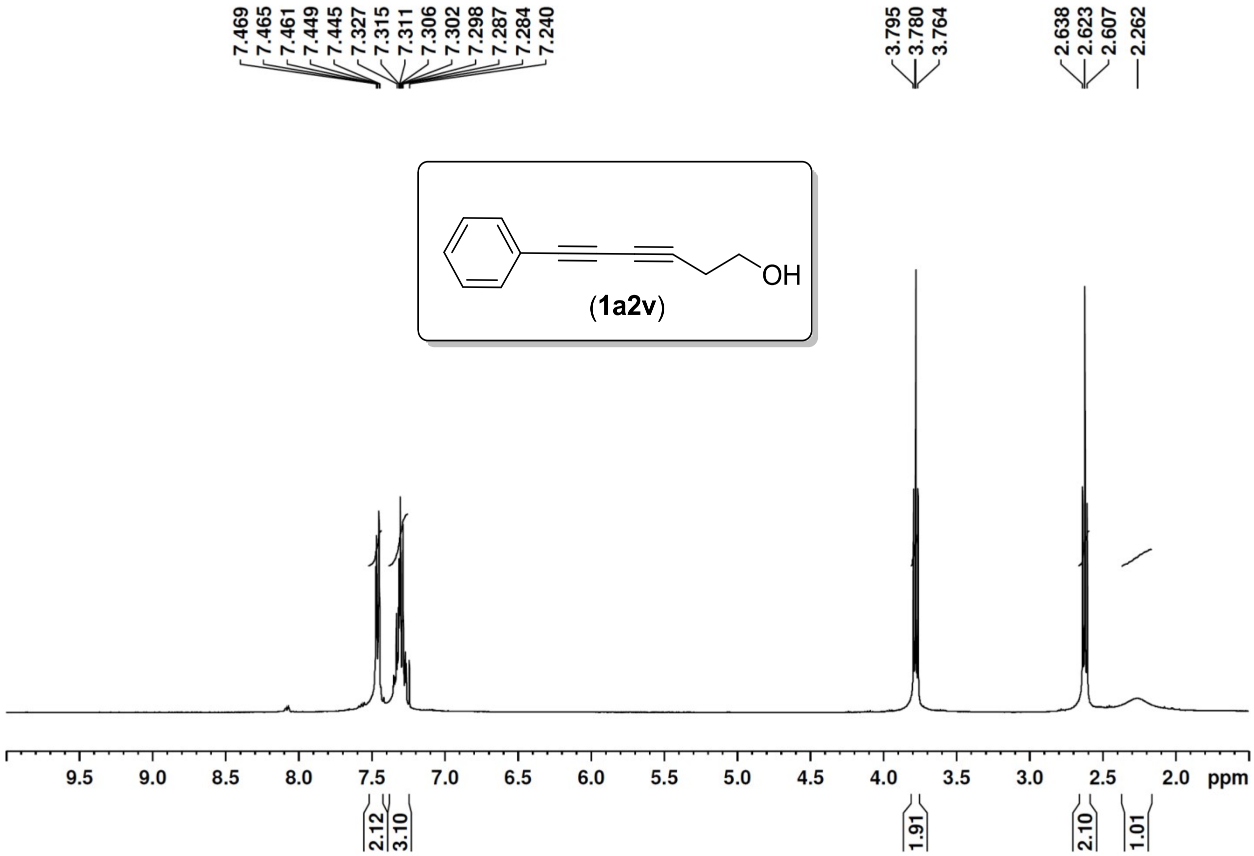
(**1a2t**)


148.350
138.235 132.893 129.968 129.809 128.794 127.460 124.013 121.391
83.392 78.831 77.634 77.316 77.005 76.694 73.451
1340-1
exp9 stdl3c
SAMPLE
date Jan 21 2013
solvent CDCl3
file exp
ACQUISITION
sfrq 100.701
tn C13
at 1.199
np 59968
sw 25000.0
fb 13800
bs 4
tpwr 58
pw 8.7
d1 0
tof 0
nt 1e+06
ct 620
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -129.7
wp 22151.9
vs 45
sc 0
wc 250
hzmm 88.61
is 500.00
rfl 10731.0
rfp 7753.2
th 2
ins 100.000
nm no ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 39
dof 0
dm yyy
dmm w
dmf 9000
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
200 180 160 140 120 100 80 60 40 20 ppm

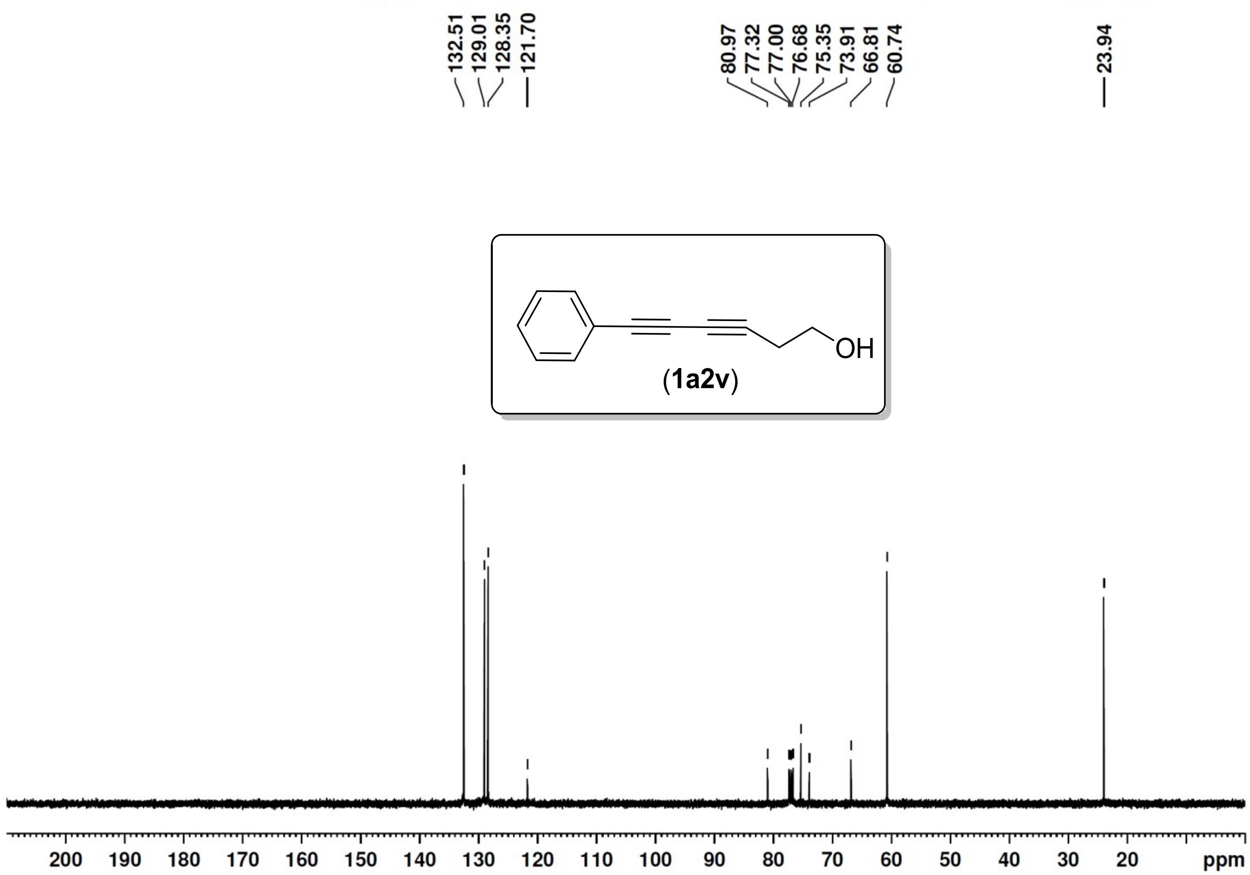
(**1a2t**)


1340-s
exp8 std1h
(1a2u)
ppm

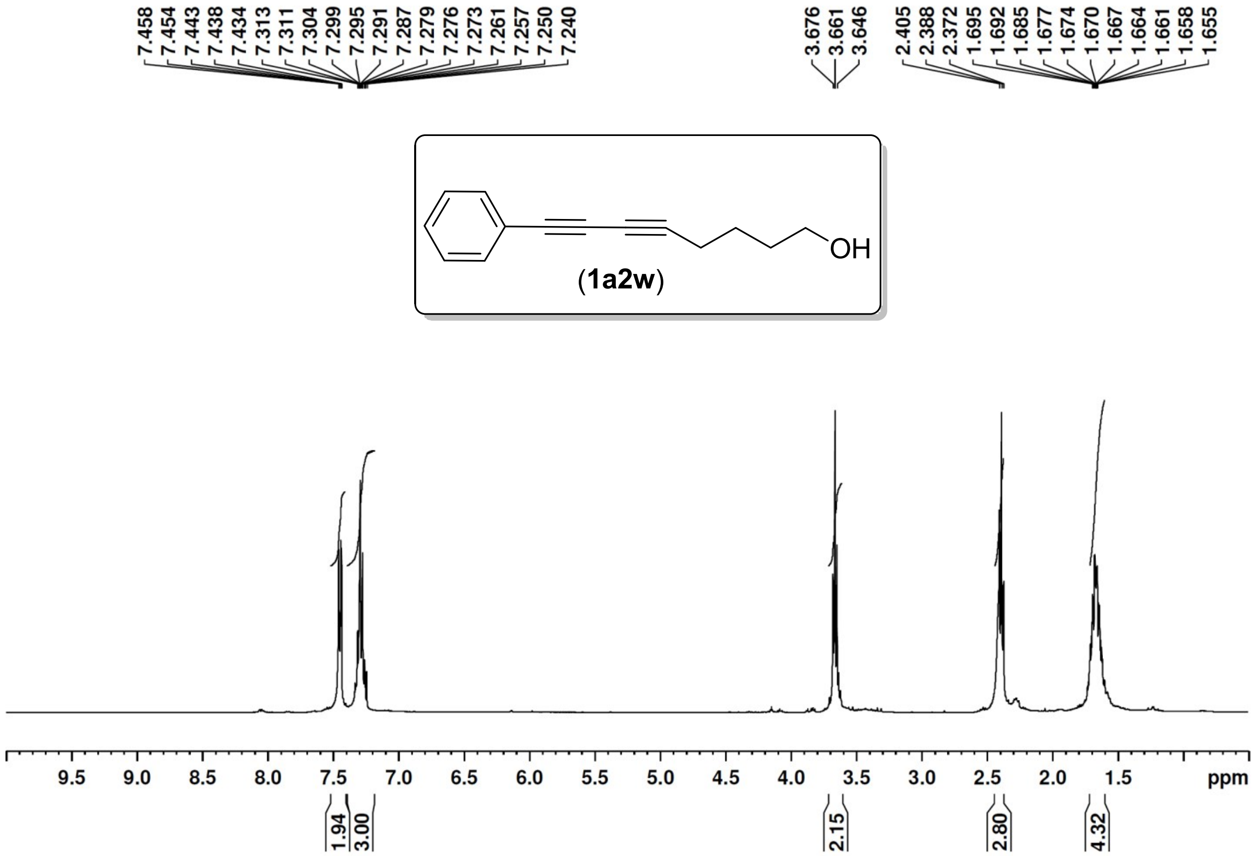

1340-s
exp9 std13c
(1a2u)
ppm



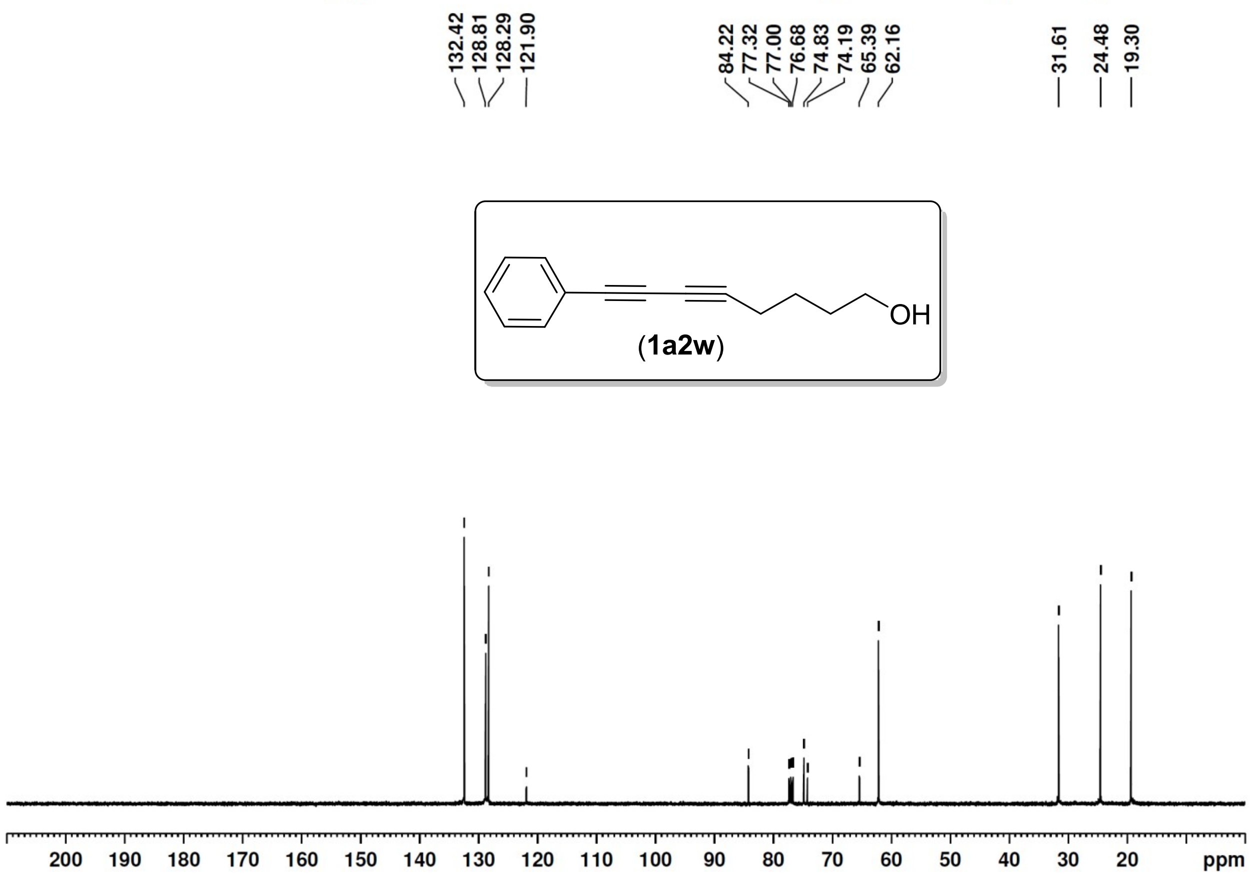
7.469
7.465
7.461
7.449
7.445
7.327
7.315
7.311
7.306
7.302
7.298
7.287
7.284
7.240
3.795
3.780
3.764
2.638
2.623
2.607
2.262
OH
(1a2v)
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 ppm
2.12
3.10
1.91
2.10
1.01

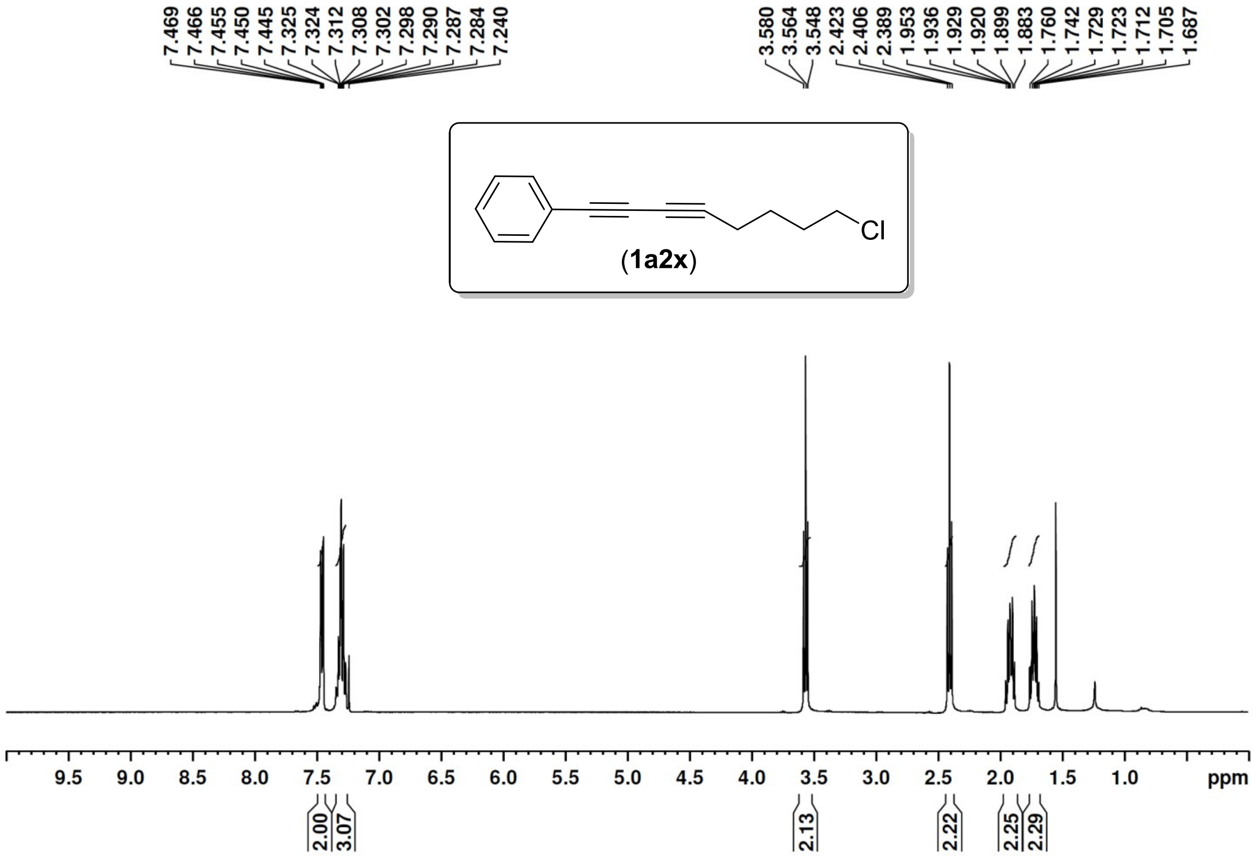
132.51
129.01
128.35
121.70
80.97
77.32
77.00
76.68
75.35
73.91
66.81
60.74
23.94
OH
(1a2v)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

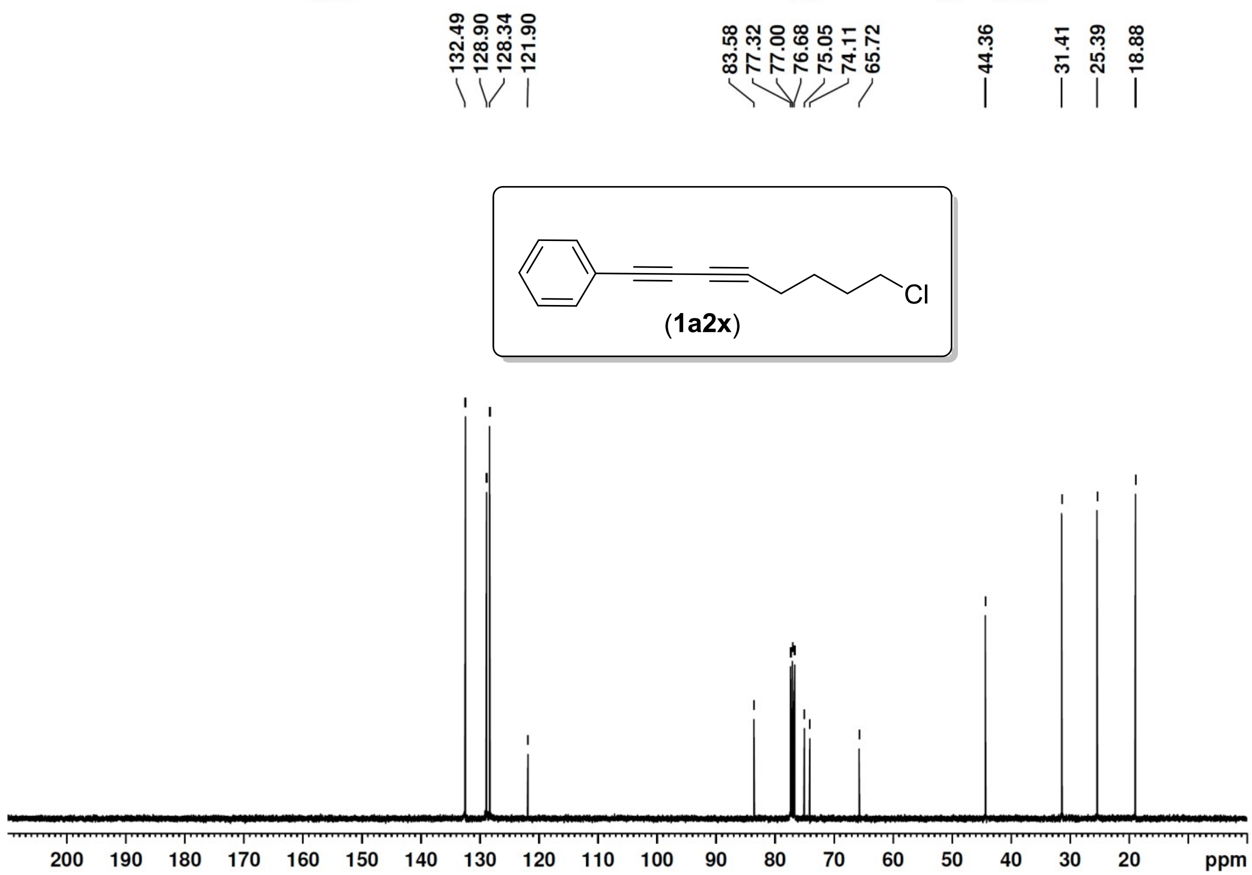


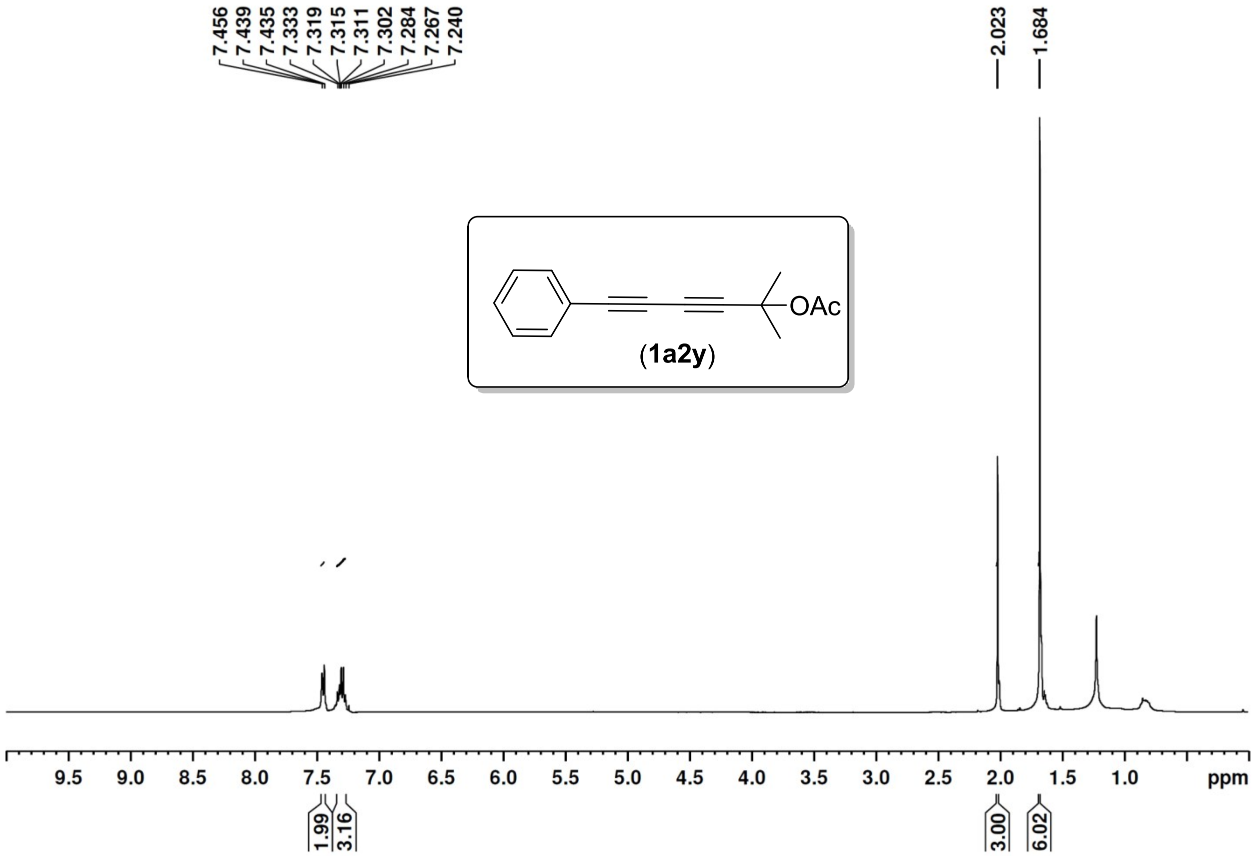
7.458 7.454 7.443 7.438 7.434 7.313 7.311 7.304 7.299 7.295 7.291 7.287 7.279 7.276 7.273 7.261 7.257 7.250 7.240
3.676 3.661 3.646
2.405 2.388 2.372 1.695 1.692 1.685 1.677 1.674 1.670 1.667 1.664 1.661 1.658 1.655
OH
(1a2w)
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 ppm
1.94 3.00 2.15 2.80 4.32

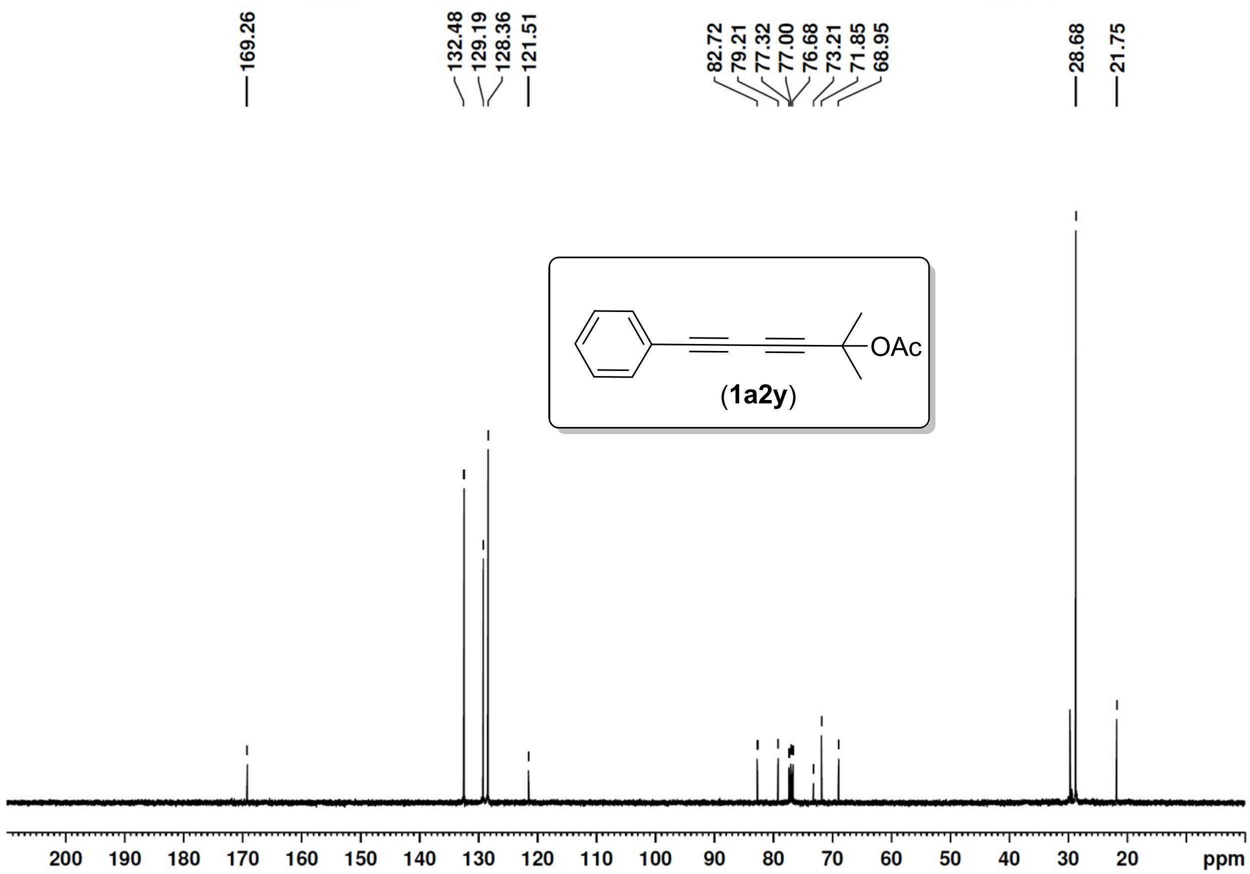



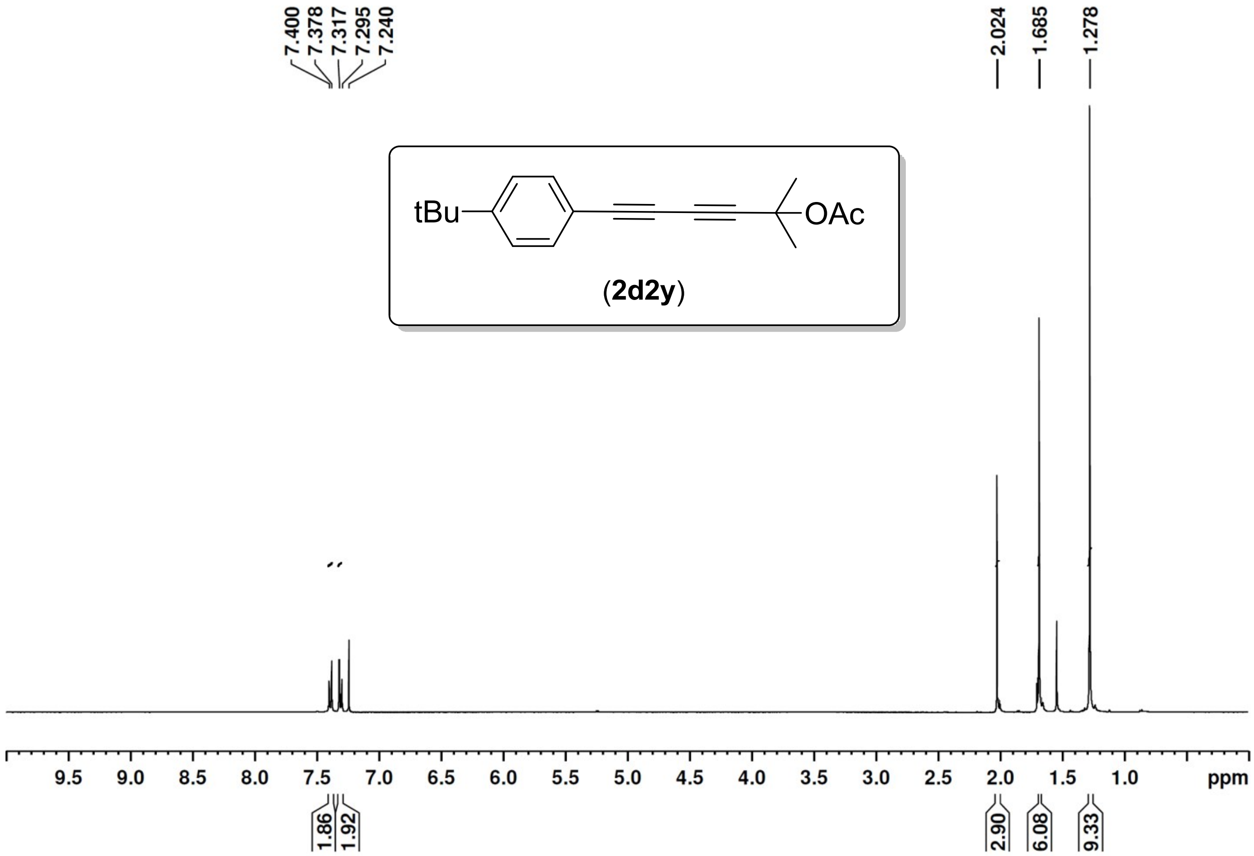
132.42 128.81 128.29 121.90
84.22 77.32 77.00 76.68 74.83 74.19 65.39 62.16
31.61 24.48 19.30
OH
(1a2w)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

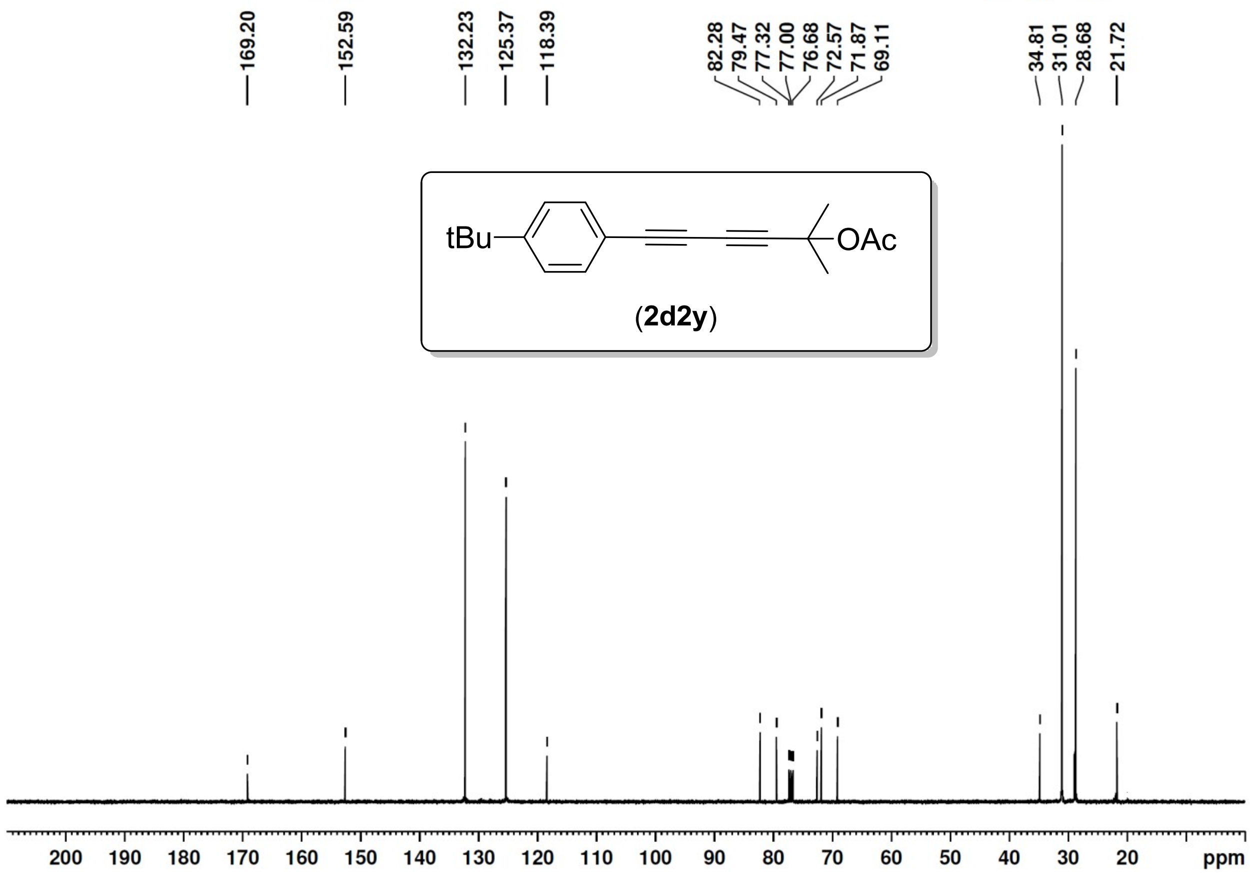

7.469 7.466 7.455 7.450 7.445 7.325 7.324 7.312 7.308 7.302 7.298 7.290 7.287 7.284 7.240
3.580 3.564 3.548 2.423 2.406 2.389 1.953 1.936 1.929 1.920 1.899 1.883 1.760 1.742 1.729 1.723 1.712 1.705 1.687
Cl
(1a2x)
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.00 3.07 2.13 2.22 2.25 2.29

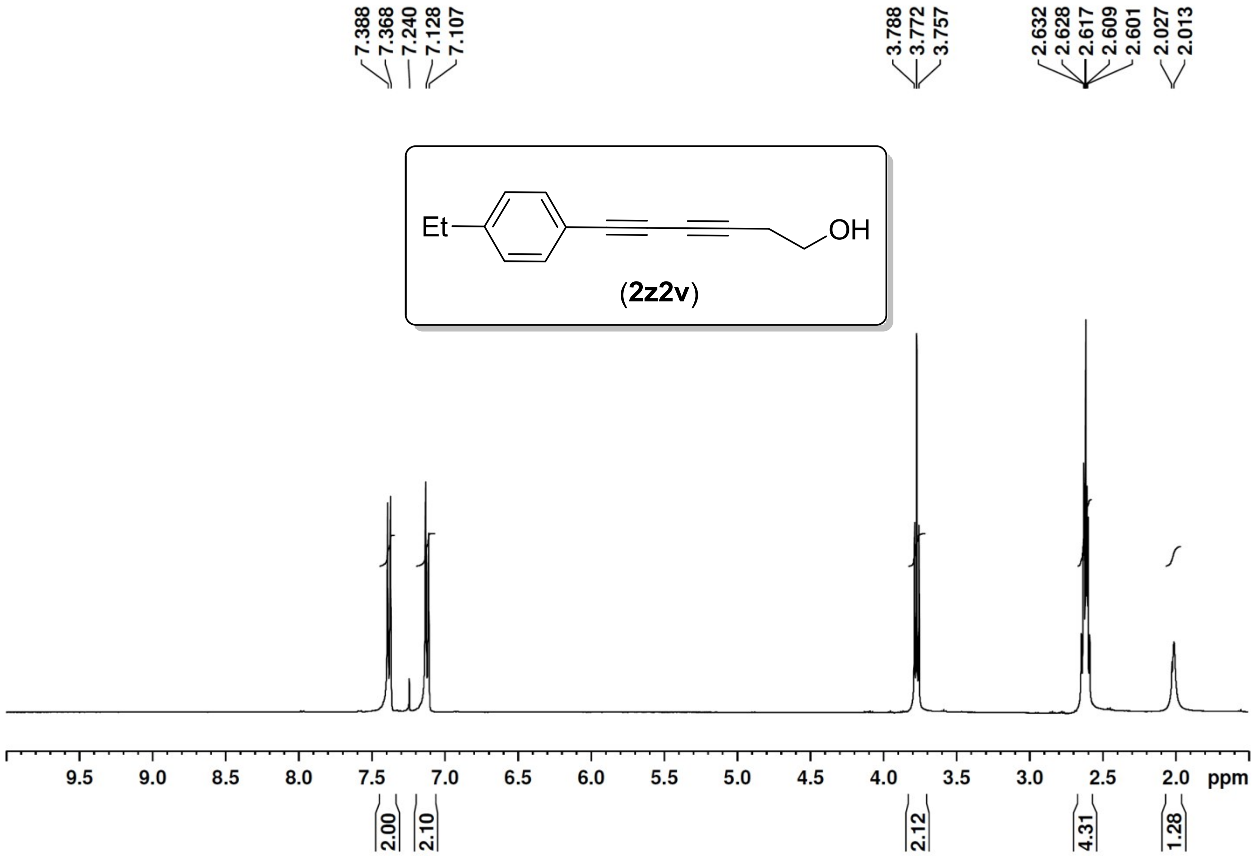

132.49 128.90 128.34 121.90
83.58 77.32 77.00 76.68 75.05 74.11 65.72
44.36
31.41 25.39 18.88
Cl
(1a2x)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

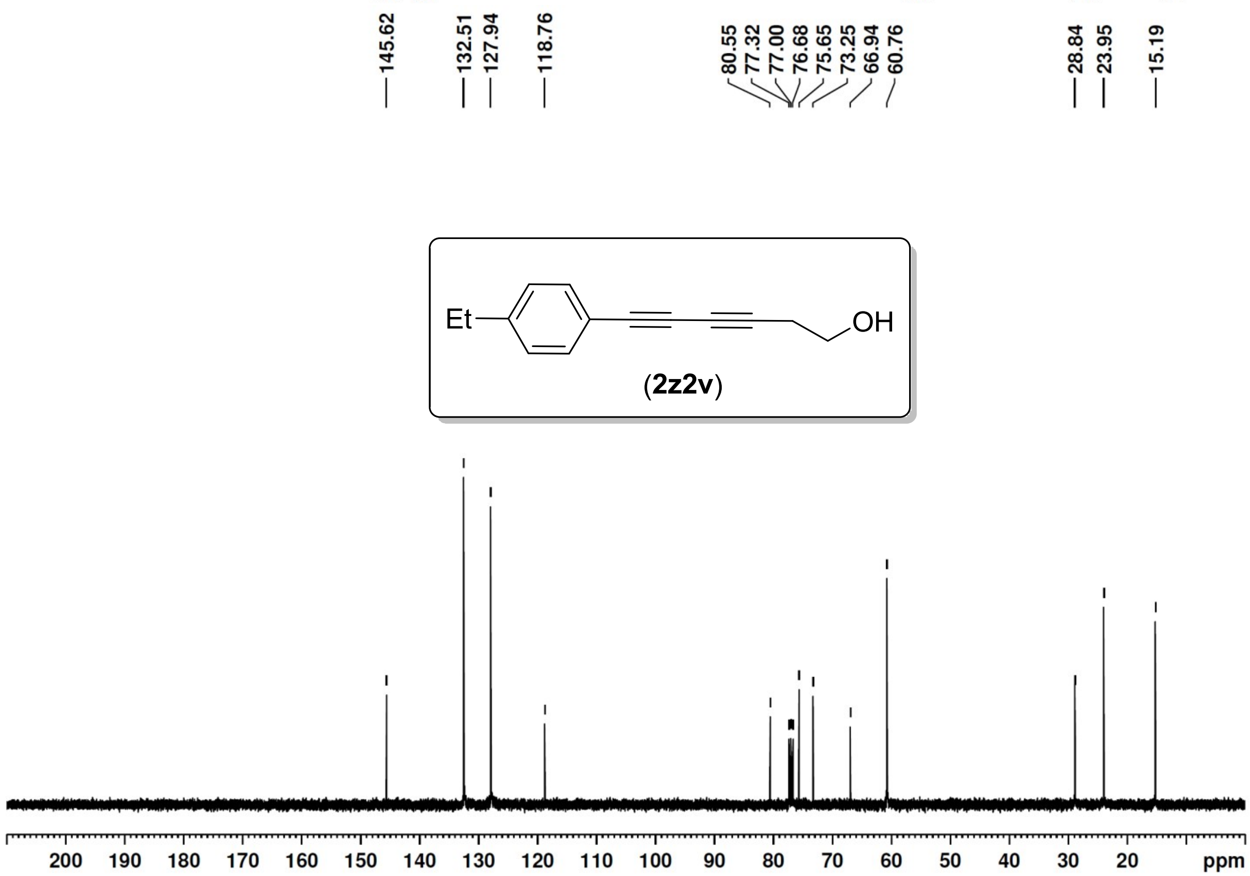

7.456
7.439
7.435
7.333
7.319
7.315
7.311
7.302
7.284
7.267
7.240
2.023
1.684
OAc
(1a2y)
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.99
3.16
3.00
6.02

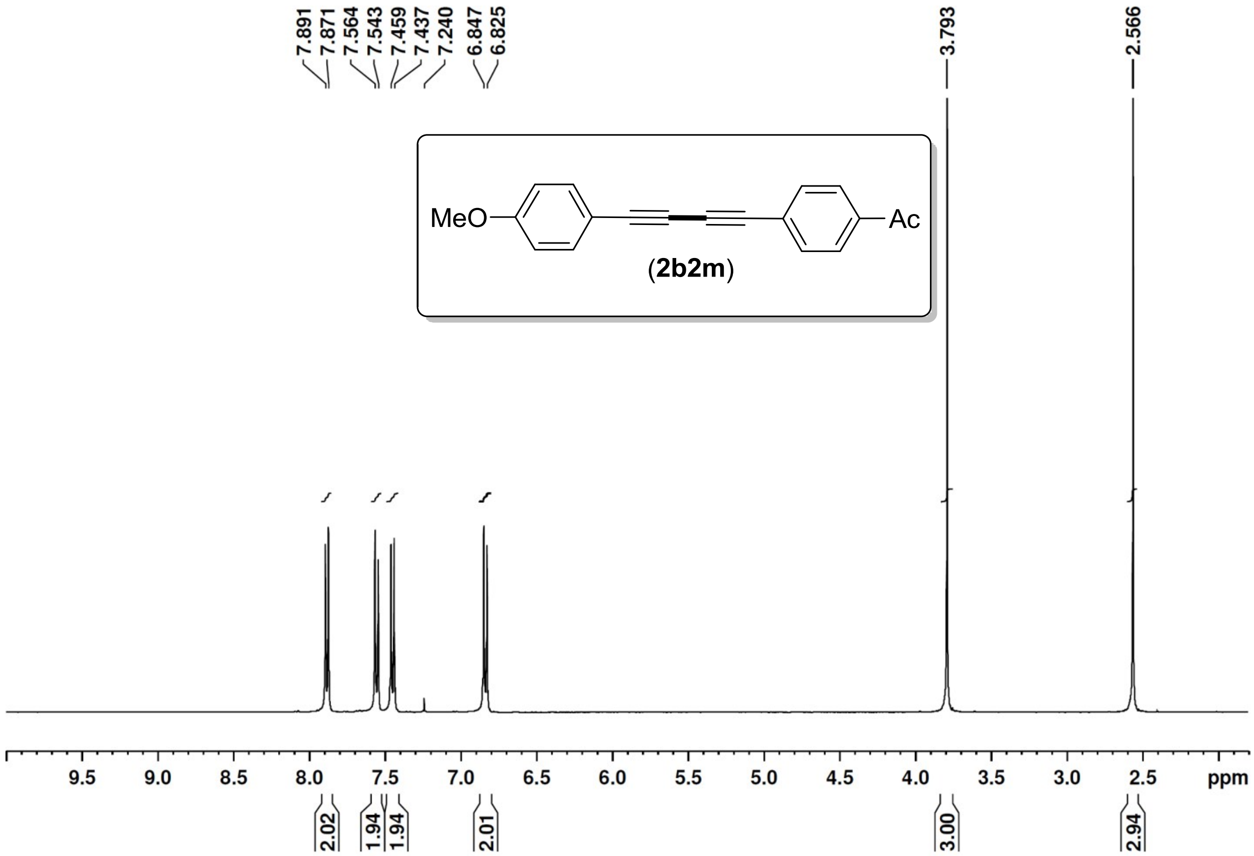

169.26
132.48
129.19
128.36
121.51
82.72
79.21
77.32
77.00
76.68
73.21
71.85
68.95
28.68
21.75
OAc
(1a2y)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

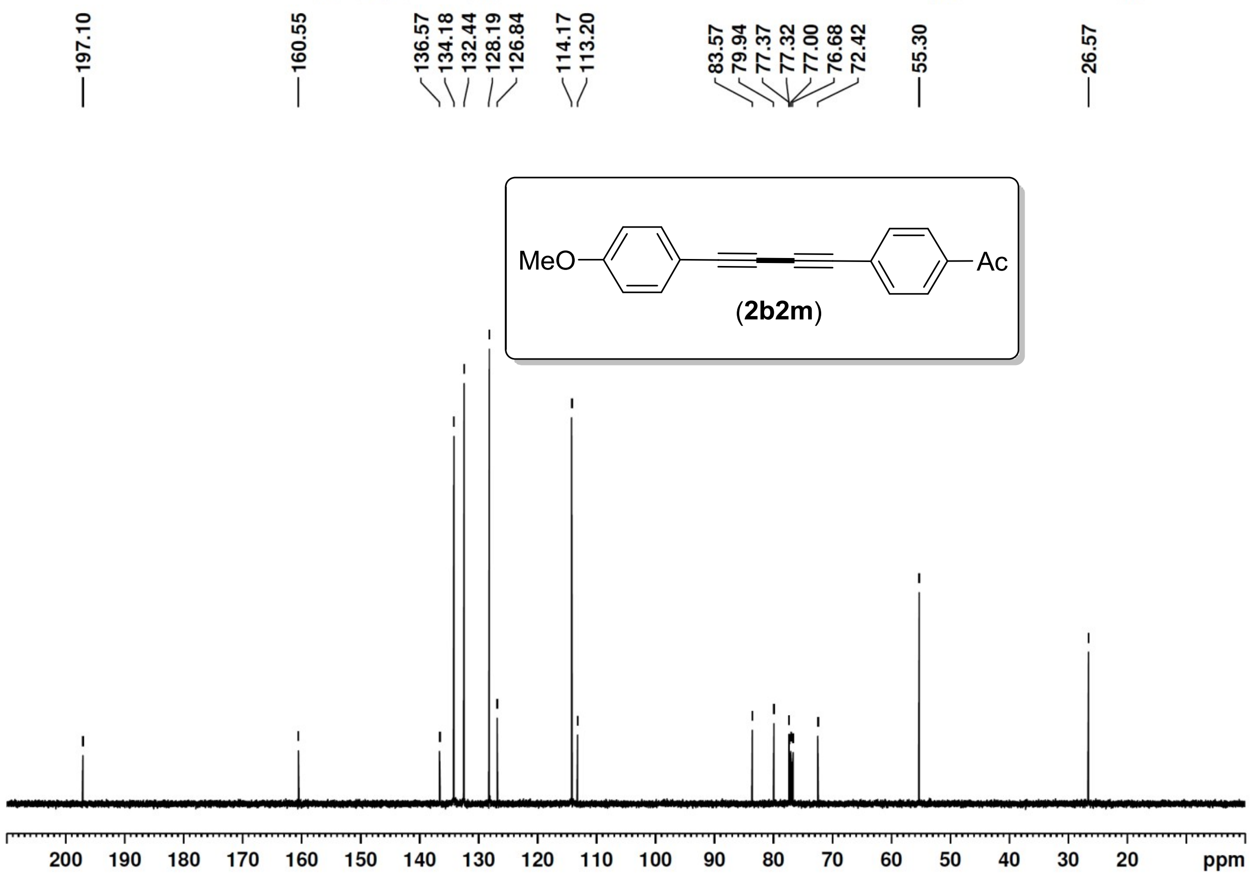

7.400
7.378
7.317
7.295
7.240
2.024
1.685
1.278
tBu
OAc
(2d2y)
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.86
1.92
2.90
6.08
9.33

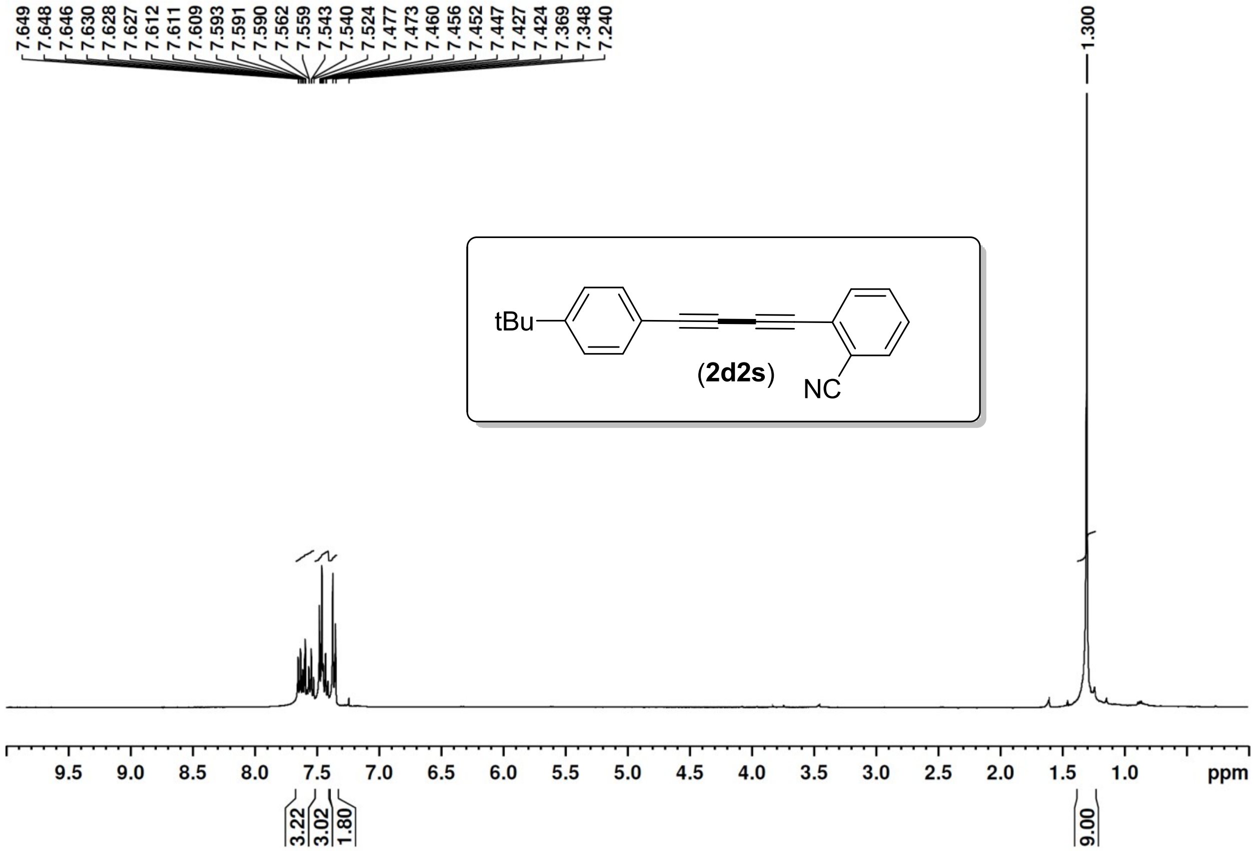

169.20
152.59
132.23
125.37
118.39
82.28
79.47
77.32
77.00
76.68
72.57
71.87
69.11
34.81
31.01
28.68
21.72
tBu
OAc
(2d2y)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

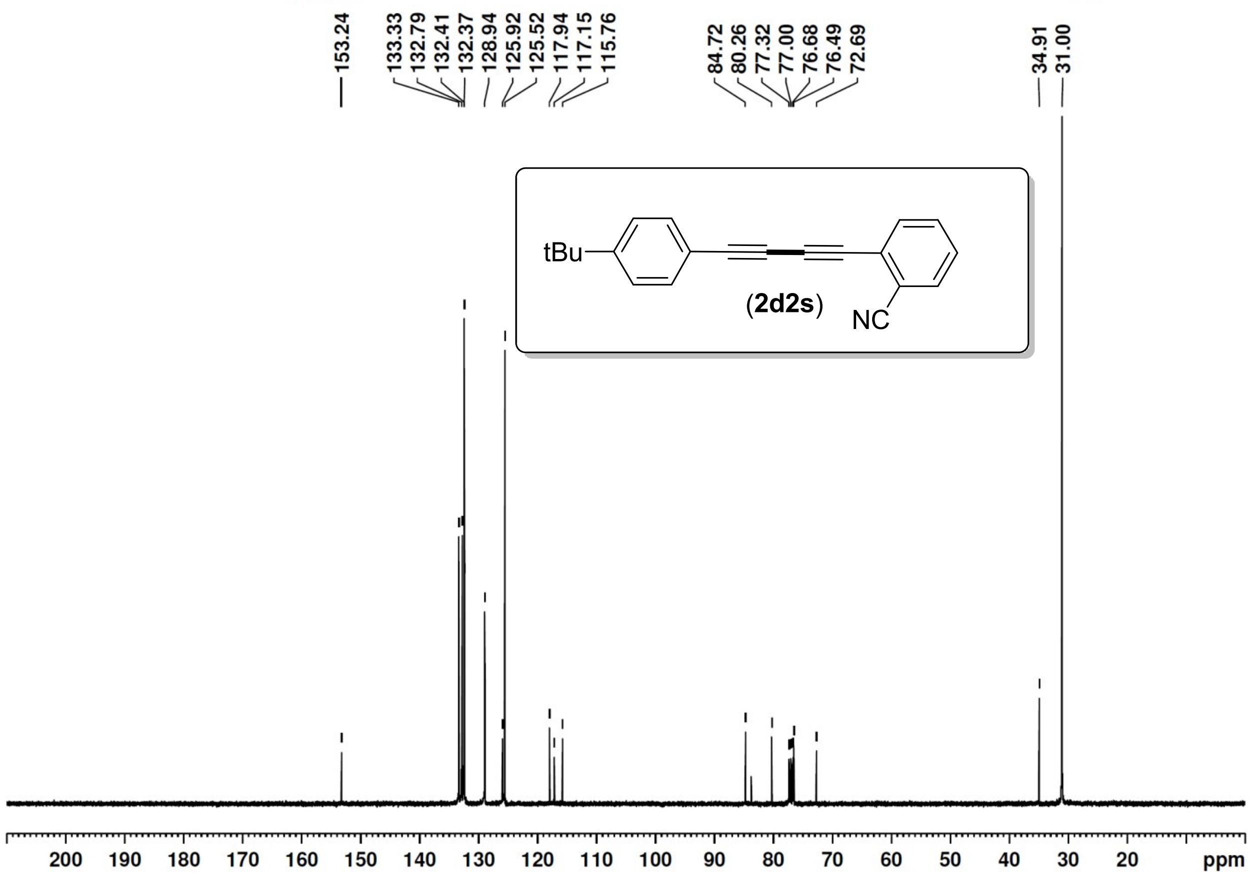

7.388
7.368
7.240
7.128
7.107
3.788
3.772
3.757
2.632
2.628
2.617
2.609
2.601
2.027
2.013
Et
OH
(2z2v)
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm
2.00
2.10
2.12
4.31
1.28

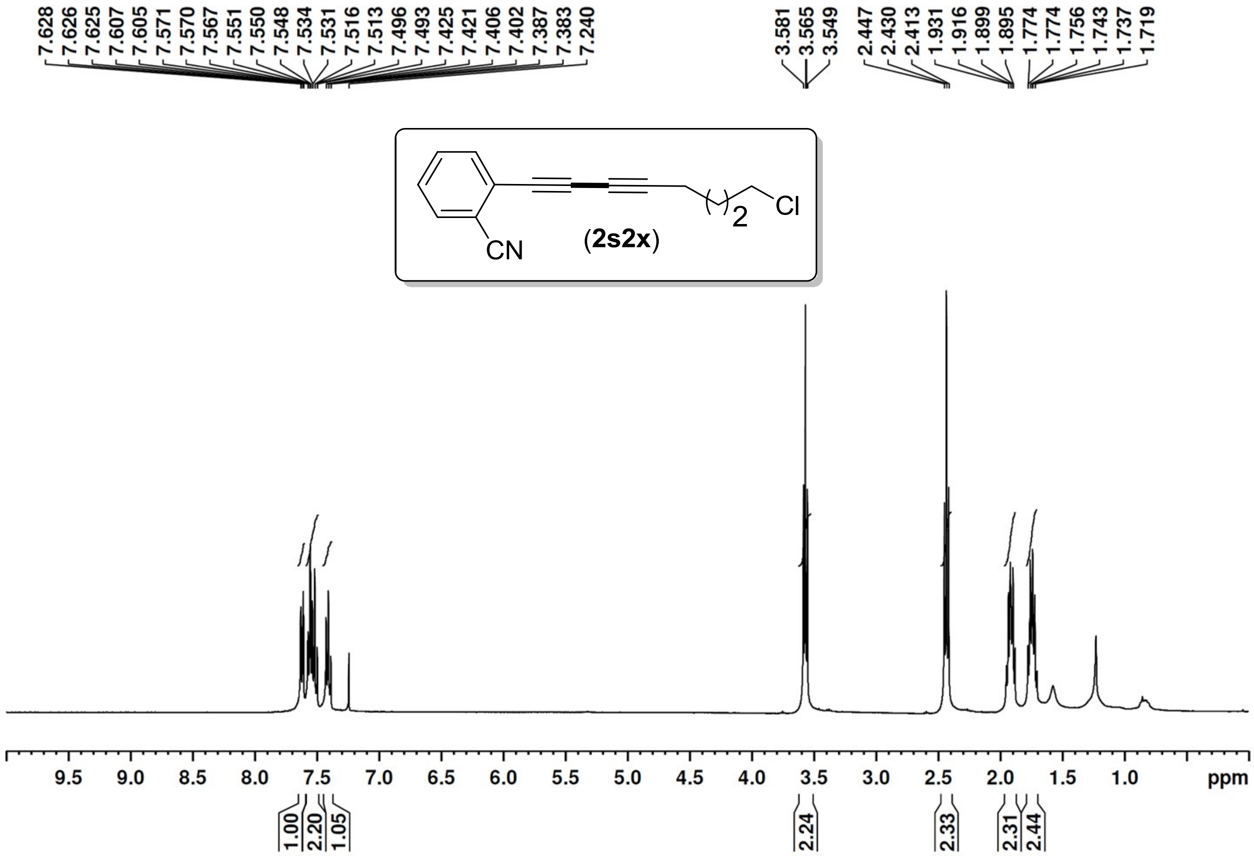

145.62
132.51
127.94
118.76
80.55
77.32
77.00
76.68
75.65
73.25
66.94
60.76
28.84
23.95
15.19
Et
OH
(2z2v)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

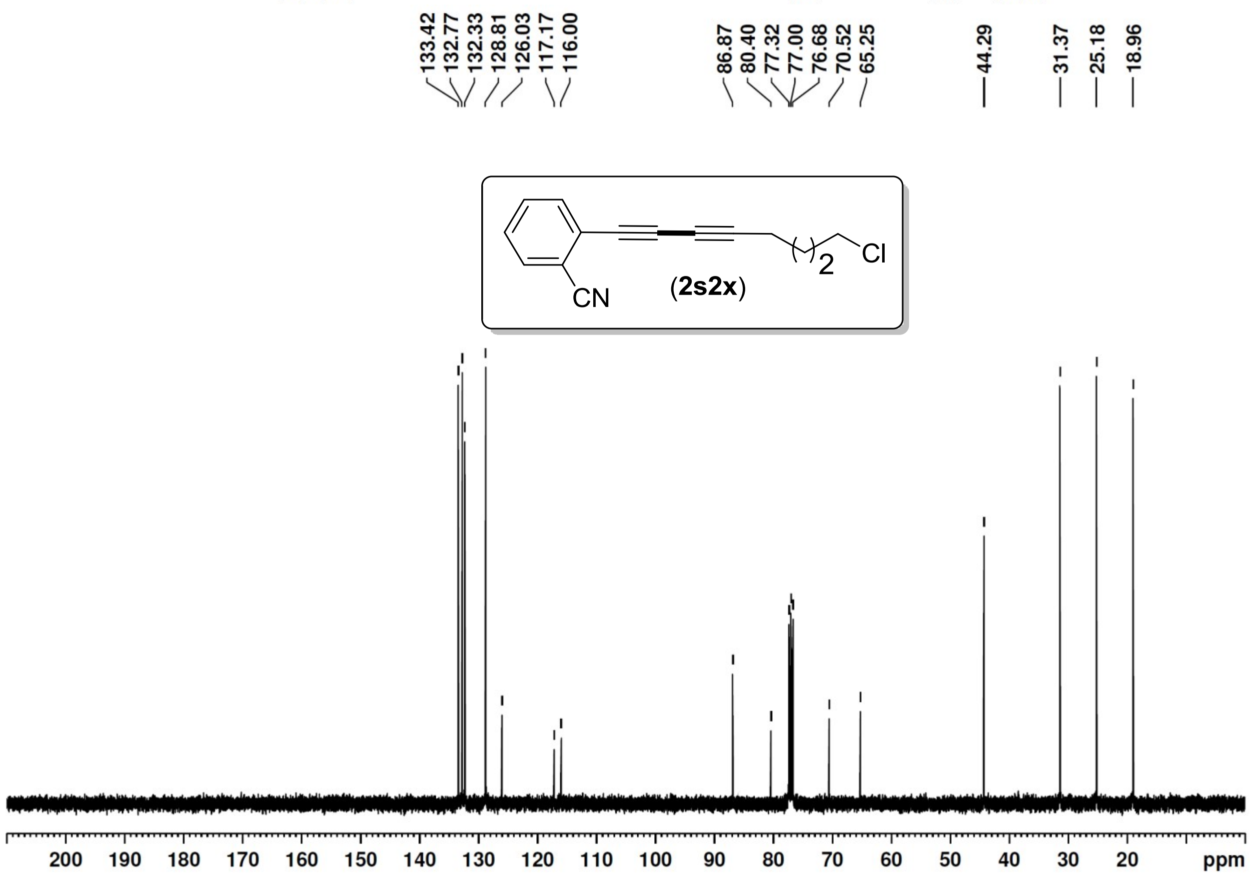

7.891
7.871
7.564
7.543
7.459
7.437
7.240
6.847
6.825
3.793
2.566
MeO
Ac
(2b2m)
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 ppm
2.02
1.94
1.94
2.01
3.00
2.94


197.10
160.55
136.57
134.18
132.44
128.19
126.84
114.17
113.20
83.57
79.94
77.37
77.32
77.00
76.68
72.42
55.30
26.57
MeO
Ac
(2b2m)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


7.649
7.648
7.646
7.630
7.628
7.627
7.612
7.611
7.609
7.593
7.591
7.590
7.562
7.559
7.543
7.540
7.524
7.477
7.473
7.460
7.456
7.452
7.447
7.427
7.424
7.369
7.348
7.240
1.300
tBu
(2d2s)
NC
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
3.22
3.02
1.80
9.00


153.24
133.33
132.79
132.41
132.37
128.94
125.92
125.52
117.94
117.15
115.76
84.72
80.26
77.32
77.00
76.68
76.49
72.69
34.91
31.00
tBu
(2d2s)
NC
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


7.628
7.626
7.625
7.607
7.605
7.571
7.570
7.567
7.551
7.550
7.548
7.534
7.531
7.516
7.513
7.496
7.493
7.425
7.421
7.406
7.402
7.387
7.383
7.240
3.581
3.565
3.549
2.447
2.430
2.413
1.931
1.916
1.899
1.895
1.774
1.774
1.756
1.743
1.737
1.719
CN
(2s2x)
2
Cl
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.00
2.20
1.05
2.24
2.33
2.31
2.44


133.42
132.77
132.33
128.81
126.03
117.17
116.00
86.87
80.40
77.32
77.00
76.68
70.52
65.25
44.29
31.37
25.18
18.96
CN
(2s2x)
2
Cl
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm