

Supporting Information

Metal-free Synthesis 3-methylthiofurans from Homopropargylic Alcohols and DMSO via tandem sulfenylation/cyclization reaction in one-pot manner

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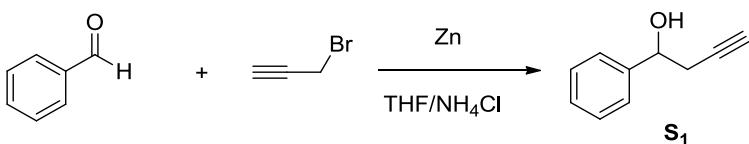
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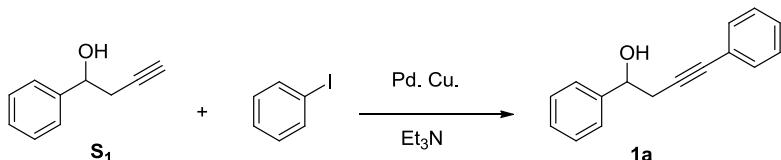
General remark

¹H NMR and ¹³C NMR spectra were recorded on 400MHz and 100MHz in CDCl₃ (BRUKER 400M or JNM-ECS 400M). All chemical shifts are given as δ value (ppm) with reference to tetramethylsilane (TMS) as an internal standard. All compounds were further characterized by HRMS; copies of their ¹H NMR and ¹³C NMR spectra are provided. Products were purified by flash chromatography on 200–300 mesh silica gels. All melting points were determined without correction. Unless otherwise noted, commercially available reagents and solvents were used without further purification.

General procedure for the synthesis of homopropargylic alcohols^[1-2]:



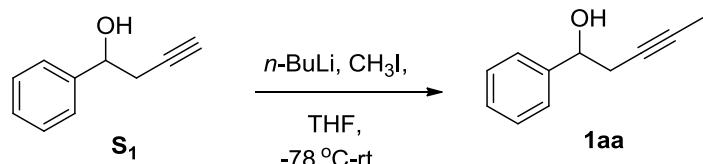
- Aldehyde (1.0 equiv.) was dissolved in anhydrous THF. A sample was taken out for analysis and propargyl bromide (2.0 equiv.) was added. Another sample was taken out for analysis and saturated aqueous NH₄Cl was added. Portions of activated zinc dust (2.0 equiv.) were added slowly on at 0°C and the resulting suspension was stirred overnight at this temperature. The THF layer was separated from the aqueous layer, which was extracted with diethyl ether for 3 times. The combined organic layers were washed with brine, dried over Na₂SO₄, filtered and concentrated in vacuo. The crude product was directly used in the next step without further purification; the residue was purified by column chromatography (silica gel, appropriate mixture of *n*-hexane/ethyl acetate) to obtain **S1**.



- To a dried schlenk flask was added Pd(PPh₃)₂Cl₂ (0.2 mmol), CuI (0.2 mmol), iodoarene (11.0 mmol), **S1** (10.0 mmol) and freshly distilled Et₃N under argon. The resulting mixture was stirred for 16 h at rt. 50 mL of EtOAc were added and the mixture filtered. After removal of solvent using rotary evaporator, the crude

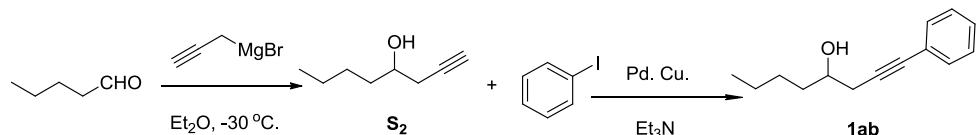
compound was purified by SiO₂ chromatography to give **1a-1z**, **1ac** and **1ad**.

1aa were prepared in the method^[3]



n-BuLi (2.5 M in hexanes, 6 mL, 15 mmol) was slowly added to a stirred solution of the propargyl alcohol (876 mg, 6 mmol) prepared in dry THF (20 mL) at -78 °C under Ar. After being stirred at -78 °C for 1 h, the reaction mixture was treated with CH₃I (1.12 mL, 18 mmol) and then allowed to warm to rt over night. The reaction mixture was then cooled to -78 °C again, quenched with sat NH₄Cl (aq) (10 mL) and extracted with Et₂O (3 * 20 mL). The combined organic phases were dried over Na₂SO₄, filtered and concentrated in vacuo. The residue was purified by column chromatography on silica gel to give **1aa** as yellow oil.

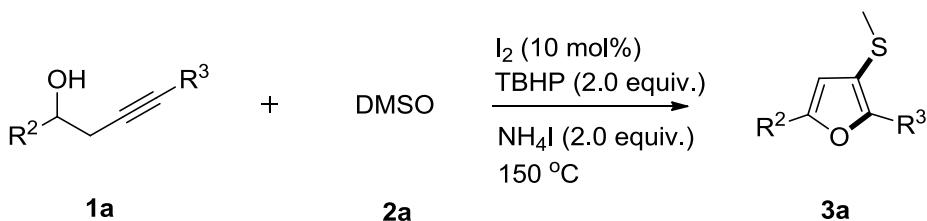
1ab were prepared in the method^[4]



Under an argon atmosphere, magnesium turnings (0.67 g, 27.5 mmol) and mercury chloride (0.34 g, 1.3 mmol) were mixed in dry diethyl ether (40 mL) in a 250 mL round-bottom flask. To the solution, propargyl bromide (2.0 mL, 25 mmol) was then added dropwise at 60 °C over about 1 h. The reaction was kept at the same temperature until the yellow solution turned cloudy. This solution was cooled to -30 °C and a solution of valeraldehyde (6 mmol) in Et₂O (12 ml) was added dropwise. After addition the reaction was moved to room temperature for further 30 min then quenched with sat. NH₄Cl (aq). The aqueous layer was extracted with ether and the extracts were combined with the above organic layer. The combined solution was dried over Na₂SO₄. After evaporation of the solvent the residue was purified by column chromatography (silica gel, appropriate mixture of *n*-hexane/ethyl acetate) to afford **S₂**.

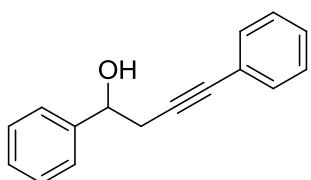
To a dried schlenk flask was added **S₂** (10.0 mmol), Pd(PPh₃)₂Cl₂ (0.2 mmol), CuI (0.4 mmol), iodoarene (11.0 mmol) and freshly distilled Et₃N (50 ml) under argon. The resulting mixture was stirred for 16 h at rt. The reaction mixture was quenched with sat. NH₄Cl (aq) and 50 mL of ethyl acetate were added and the mixture filtered. After removal of solvent using rotary evaporator, the crude compound was purified by column chromatography on silica gel to give **1ab**.

General procedure for synthesis of *S*-substituted furans from homopropargylic alcohols and DMSO:



The homopropargylic alcohols (**1a**, 0.3 mmol), iodine (0.03 mmol), TBHP (0.6 mmol), NH₄I (0.6 mmol) were mixed in DMSO (2 mL) and this mixture was carried out under N₂ at 150 °C for 4 h. The reaction mixture was cooled to room temperature and then washed with saturated sodium thiosulfate (10 mL), extracted with ethyl acetate (15 ml × 3). The combined organic phase was dried over anhydrous Na₂SO₄. The solvent was evaporated in vacuo and the crude product was purified by column chromatography, eluting with petroleum ether/EtOAc (10:1) to afford the desired 3-(methylthio)-2,5-diphenylfuran **3a**.

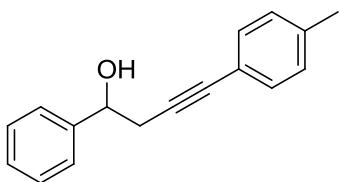
The data of products:



1,4-diphenylbut-3-yn-1-ol (1a)

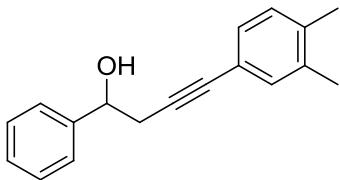
Yellow solid. ¹H NMR (400 MHz, CDCl₃, ppm): δ = 7.42-7.25 (m, 10 H), 4.92-4.89

(m, 1 H), 2.83-2.82 (d, $J = 4.0$ Hz, 2 H), 2.61 (s, 1 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 142.78, 131.72, 128.49, 128.31, 128.04, 127.96, 125.88, 123.33, 86.12, 83.24, 72.66, 30.61$.



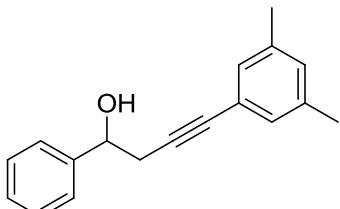
1-phenyl-4-(p-tolyl)but-3-yn-1-ol (1b)

Yellow solid. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.43\text{-}7.41$ (m, 2 H), 7.38-7.34 (m, 2 H), 7.31-7.28 (m, 3 H), 7.09-7.07 (d, $J = 8.0$ Hz, 2 H), 4.94-4.90 (m, 1 H), 2.84-2.83 (d, $J = 4.0$ Hz, 2 H), 2.60-2.59 (d, $J = 4.0$ Hz, 1 H), 2.33 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 142.85, 138.16, 131.70, 131.63, 129.16, 129.08, 128.56, 127.98, 125.93, 120.27, 85.32, 83.43, 72.81, 30.77, 21.57$.



4-(3,4-dimethylphenyl)-1-phenylbut-3-yn-1-ol (1c)

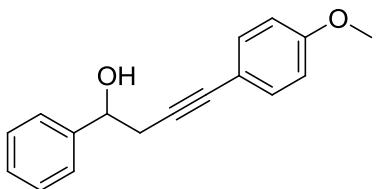
Yellow solid. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.44\text{-}7.41$ (m, 2 H), 7.38-7.36 (m, 2 H), 7.35-7.30 (m, 1 H), 7.17-7.10 (m, 1 H), 7.14-7.12 (m, 1 H), 7.05-7.03 (d, $J = 8.0$ Hz, 1 H), 4.93-4.91 (m, 1 H), 2.84-2.82 (m, 2 H), 2.57 (s, 1 H), 2.24 (s, 3 H), 2.21 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 142.85, 136.97, 136.66, 132.82, 129.67, 129.18, 128.53, 127.97, 125.92, 120.54, 84.99, 83.57, 72.77, 30.81, 19.82, 19.67$.



4-(3,5-dimethylphenyl)-1-phenylbut-3-yn-1-ol (1d)

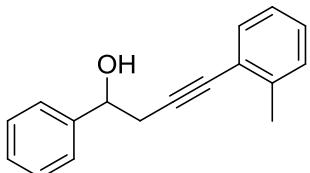
Yellow solid. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.42\text{-}7.41$ (d, $J = 4.0$ Hz, 2 H), 7.37-7.34 (m, 2 H), 7.31-7.29 (m, 1 H), 7.02 (s, 2 H), 6.91 (s, 1 H), 4.92-4.84 (m, 1 H), 2.83-2.82 (d, $J = 4.0$ Hz, 2 H), 2.58-2.55 (m, 1 H), 2.26 (s, 6 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 142.84, 137.91, 130.04, 129.47, 128.54, 127.98, 125.92, 122.94$,

85.30, 83.63, 72.76, 30.77, 21.20.



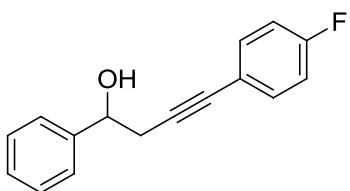
4-(4-methoxyphenyl)-1-phenylbut-3-yn-1-ol (1e)

Yellow solid. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.43\text{-}7.41$ (m, 2 H), 7.37-7.32 (m, 2 H), 7.30-7.23 (m, 3 H), 6.81-6.79 (d, $J = 8.8$ Hz, 2 H), 4.93-4.89 (m, 1 H), 3.77 (s, 3 H) 2.83-2.81 (m, 2 H), 2.55-2.54 (d, $J = 3.2$ Hz, 1 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 159.39$, 142.79, 133.06, 128.44, 127.89, 125.83, 115.39, 113.90, 84.40, 83.08, 72.66, 55.28, 30.70.



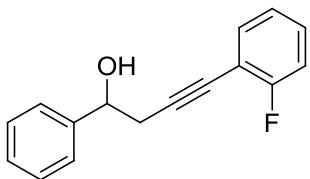
1-phenyl-4-(o-tolyl)but-3-yn-1-ol (1f)

Yellow solid. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.44\text{-}7.42$ (d, $J = 8.0$ Hz, 2 H), 7.37-7.28 (m, 3 H), 7.22-7.19 (m, 1 H), 7.17-7.10 (m, 2 H), 7.09-7.07 (m, 1 H), 4.95-4.91 (m, 1 H), 2.92-2.90 (d, $J = 6.0$ Hz, 2 H), 2.50-2.49 (d, $J = 3.2$ Hz, 1 H), 2.32 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 142.73$, 140.16, 132.02, 129.38, 128.48, 128.00, 127.94, 125.92, 125.50, 123.06, 89.75, 82.16, 72.70, 30.64, 20.71.



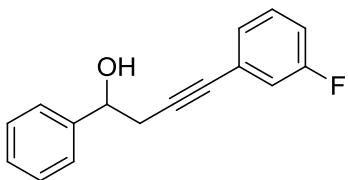
4-(4-fluorophenyl)-1-phenylbut-3-yn-1-ol (1g)

Yellow solid. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.45\text{-}7.31$ (m, 7 H), 7.01-6.96 (m, 2 H), 4.97-4.93 (m, 1 H), 2.86-2.85 (d, $J = 6.4$ Hz, 2 H), 2.48-2.47 (d, $J = 2.4$ Hz, 1 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 161.08\text{-}163.56$ (d, $J = 248$ Hz, 1 C), 142.69, 133.46-133.54 (d, $J = 8$ Hz, 1 C), 128.49, 127.99, 125.80, 119.33-119.36 (d, $J = 3$ Hz, 1 C), 115.61-115.39 (d, $J = 23$ Hz, 2 C), 85.69-85.71 (d, $J = 2$ Hz, 1 C), 82.11, 72.63, 30.49.



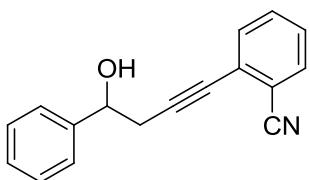
4-(2-fluorophenyl)-1-phenylbut-3-yn-1-ol (1h)

Yellow solid. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.45\text{-}7.31$ (m, 7 H), 7.01- 6.96 (m, 2 H), 4.97-4.93 (m, 1 H), 2.96-2.85 (d, $J = 6.4$ Hz, 2 H), 2.48-2.47 (d, $J = 2.4$ Hz, 1 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 163.56\text{-}161.08$ (d, $J = 248$ Hz, 1 C), 142.69, 133.46-133.54 (d, $J = 8$ Hz, 2 C), 128.49, 127.99, 125.80, 119.36, 119.33, 115.61- 115.39 (d, $J = 23$ Hz, 2 C), 85.71-85.69 (d, $J = 2$ Hz, 1 C), 82.11, 72.63, 30.49.



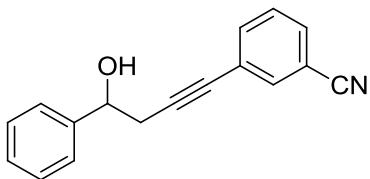
4-(3-fluorophenyl)-1-phenylbut-3-yn-1-ol (1i)

Yellow solid. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.43\text{-}7.37$ (m, 4 H), 7.35-7.31 (m, 1 H), 7.29-7.20 (m, 1 H), 7.16-7.14 (d, $J = 8.0$ Hz, 1 H), 7.08-7.05 (d, $J = 2.4$ Hz, 1 H) 7.01-6.97 (m, 1 H), 4.95-4.91 (m, 1 H), 2.86-2.84 (d, $J = 8.0$ Hz, 2 H), 2.60 (s, 1 H), 2.02 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 163.64\text{-}161.19$ (d, $J = 245$ Hz, 1 C), 142.74, 129.90, 128.60, 128.12, 127.61, 125.92, 125.30-125.20 (d, $J = 10$ Hz, 1 C), 118.74, 118.63, 118.51-118.41 (d, $J = 10$ Hz, 1 C), 115.55-115.30 (d, $J = 25$ Hz, 1 C), 87.36, 82.04, 72.76-72.61 (d, $J=5$ Hz, 1 C), 30.51.



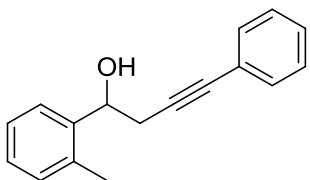
2-(4-hydroxy-4-phenylbut-1-yn-1-yl)benzonitrile (1j)

Yellow solid. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.61\text{-}7.59$ (m, 1 H), 7.52-7.45 (m, 4 H), 7.38-7.33 (m, 3 H), 7.31-7.27 (m, 1 H), 5.04-5.00 (m, 1 H), 2.94-2.92 (d, $J = 6.4$ Hz, 2 H), 2.79-2.78 (d, $J = 4$ Hz, 1 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 142.55$, 132.39, 132.37, 132.21, 128.51, 128.04, 127.95, 127.50, 125.82, 118.05, 115.43, 93.92, 79.48, 72.41, 30.72.



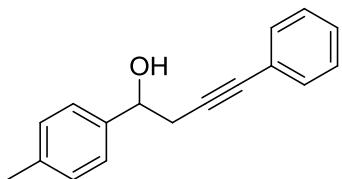
3-(4-hydroxy-4-phenylbut-1-yn-1-yl)benzonitrile (1k)

Yellow solid. ¹H NMR (400 MHz, CDCl₃, ppm): δ = 7.58-7.62 (d, *J*=12.0 Hz, 1 H), 7.44-7.55 (m, 2 H), 7.37-7.42 (m, 5 H), 7.31-7.34 (m, 1 H), 4.95-4.98 (m, 1 H), 2.87-2.89 (m, 2 H), 2.46 (s, 1 H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ = 142.64, 135.82, 135.14, 135.04, 131.19, 129.17, 128.55, 125.95, 125.04, 118.22, 112.76, 89.20, 80.88, 72.74, 30.40.



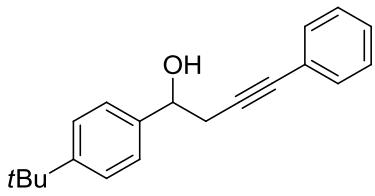
4-phenyl-1-(o-tolyl)but-3-yn-1-ol (1l)

Yellow solid. ¹H NMR (400 MHz, CDCl₃, ppm): δ = 7.47-7.45 (d, *J* = 8.0 Hz, 1 H), 7.31-7.29 (m, 2 H), 7.19-7.04 (m, 6 H), 5.09-5.06 (m, 1 H), 2.73-2.72 (d, *J* = 4.0 Hz, 2 H), 2.51 (s, 1 H), 2.28 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ = 140.92, 134.75, 131.87, 131.74, 130.60, 130.42, 128.32, 126.38, 125.37, 125.42, 86.40, 83.06, 69.25, 29.51, 19.32.



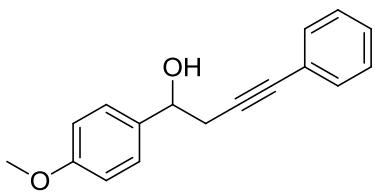
4-phenyl-1-(p-tolyl)but-3-yn-1-ol (1m)

Yellow solid. ¹H NMR (400 MHz, CDCl₃, ppm): δ = 7.40-7.37 (m, 2 H), 7.33-7.31 (d, *J* = 8.0 Hz, 2 H), 7.29-7.27 (m, 3 H), 7.24-7.17 (m, 2 H), 4.94-4.92 (m, 1 H), 2.85-2.83 (d, *J* = 8.0 Hz, 2 H), 2.43-2.42 (d, *J* = 4.0 Hz, 1 H), 2.35 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ = 139.85, 137.72, 131.93, 131.59, 129.45, 128.99, 128.55, 128.13, 127.85, 126.02, 125.64, 123.39, 86.21, 83.21, 72.82, 30.69.



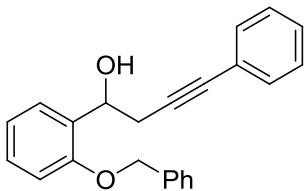
1-(4-(tert-butyl)phenyl)-4-phenylbut-3-yn-1-ol (1n)

Yellow solid. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.41\text{-}7.36$ (m, 6 H), 7.29-7.27 (m, 3 H), 4.95-4.91 (m, 1 H), 2.87-2.85 (d, $J = 6.4$ Hz, 2 H), 2.41-2.40 (d, $J = 3.6$ Hz, 1 H), 1.32 (s, 9 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 150.92, 139.3, 131.68, 128.24, 127.97, 125.55, 125.39, 123.34, 86.26, 83.13, 72.45, 34.57, 31.36, 30.48$.



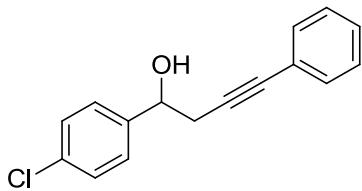
1-(4-methoxyphenyl)-4-phenylbut-3-yn-1-ol (1o)

Yellow solid. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.40\text{-}7.35$ (m, 4 H), 7.29-7.27 (m, 3 H), 6.92-6.89 (m, 2 H), 4.93-4.89 (m, 1 H), 3.81(s, 3 H), 2.85-2.83 (m, 2 H), 2.41 (s, 1 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 159.38, 135.00, 131.79, 128.36, 127.20, 127.10, 113.95, 113.87, 86.21, 83.21, 72.48, 55.46, 30.67$.



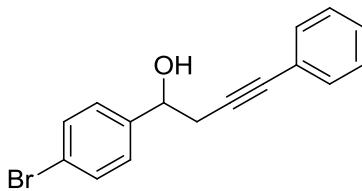
1-(2-(benzyloxy)phenyl)-4-phenylbut-3-yn-1-ol (1p)

Yellow solid. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.51\text{-}7.50$ (d, $J = 1.6$ Hz, 1 H), 7.49-7.347 (m, 7 H), 7.27-7.23 (m, 4 H), 7.03-6.99 (m, 1 H), 6.96-6.93(d, $J = 8.4$ Hz, 1 H), 5.25-5.24(m, 1 H), 5.12(s, 2 H), 3.04-2.99 (m, 1 H), 2.91-2.84 (m, 2 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 155.46, 136.76, 131.68, 130.96, 128.74, 128.69, 128.20, 128.10, 127.82, 127.28, 127.14, 123.55, 121.03, 111.75, 86.61, 82.92, 70.13, 69.01, 28.70$.



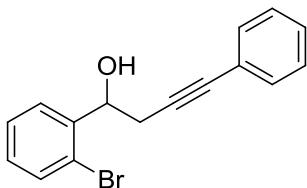
1-(4-chlorophenyl)-4-phenylbut-3-yn-1-ol (1q)

Yellow solid. ¹H NMR (400 MHz, CDCl₃, ppm): δ = 7.36-7.26 (m, 9 H), 4.90-4.87 (m, 1 H), 2.81-2.79 (d, *J* = 6.4 Hz, 2 H), 2.68-2.67 (d, *J* = 2.0 Hz, 1 H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ = 141.20, 133.59, 131.67, 128.58, 128.33, 128.15, 127.28, 123.11, 85.54, 83.51, 71.94, 30.58.



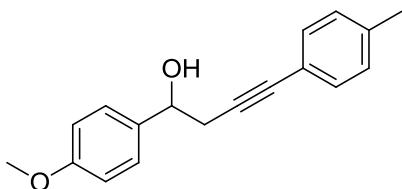
1-(4-bromophenyl)-4-phenylbut-3-yn-1-ol (1r)

Yellow solid. ¹H NMR (400 MHz, CDCl₃, ppm): δ = 7.50-7.48 (d, *J* = 8.4 Hz, 2 H), 7.38-7.36 (m, 2 H), 7.31-7.27 (m, 5 H), 4.93-4.87 (m, 1 H), 2.83-2.81 (m, 2 H), 2.54-2.53 (d, *J* = 3.6 Hz, 1 H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ = 141.68, 131.67, 131.53, 128.32, 128.15, 127.59, 123.07, 121.75, 85.41, 83.57, 71.97, 30.56.



1-(2-bromophenyl)-4-phenylbut-3-yn-1-ol (1s)

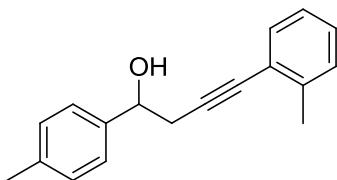
Yellow solid. ¹H NMR (400 MHz, CDCl₃, ppm): δ = 7.67-7.65 (m, 1 H), 7.54-7.52 (d, *J* = 8.0 Hz, 1 H), 7.41-7.34 (m, 3 H), 7.29-7.28 (m, 3 H), 7.18-7.14 (m, 1 H), 5.30 (s, 1 H), 3.05-3.00 (m, 1 H), 2.79-2.74 (m, 1 H), 2.66 (s, 1 H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ = 141.47, 132.67, 131.73, 129.24, 128.27, 128.06, 127.67, 127.46, 123.19, 121.84, 85.46, 83.46, 28.87.



1-(4-methoxyphenyl)-4-(p-tolyl)but-3-yn-1-ol (1t)

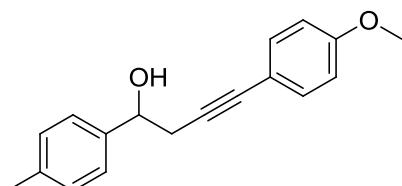
Yellow solid. ¹H NMR (400 MHz, CDCl₃, ppm): δ = 7.34-7.32 (m, 2 H), 7.27-7.26 (d,

J = 8.0 Hz, 2 H), 7.08-7.06 (d, *J* = 7.6 Hz, 2 H), 6.89-6.87 (m, 2 H), 4.88-4.84 (m, 1 H), 3.78 (s, 3 H), 2.81-2.80 (m, 2 H), 2.57-2.56 (d, *J* = 3.2 Hz, 1 H), 2.31 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ = 159.28, 138.01, 135.06, 131.57, 129.03, 127.10, 120.28, 113.82, 85.43, 83.22, 72.30, 55.30, 30.61, 21.44.



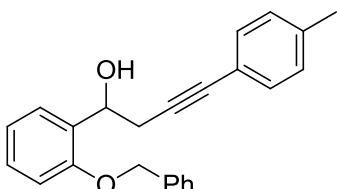
4-(o-tolyl)-1-(p-tolyl)but-3-yn-1-ol (1u)

Yellow solid. ¹H NMR (400 MHz, CDCl₃, ppm): δ = 7.34-7.29 (m, 3 H), 7.19-7.14 (m, 4 H), 7.09-7.05 (m, 1 H), 4.90-4.86 (m, 1 H), 2.88-2.87 (d, *J* = 6.4 Hz, 2 H), 2.52 (s, 1 H), 2.32 (s, 6 H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ = 140.17, 139.88, 137.59, 132.04, 129.38, 129.15, 127.96, 125.89, 125.50, 123.18, 90.03, 82.06, 72.61, 30.59, 21.18, 20.72.



4-(4-methoxyphenyl)-1-(p-tolyl)but-3-yn-1-ol (1v)

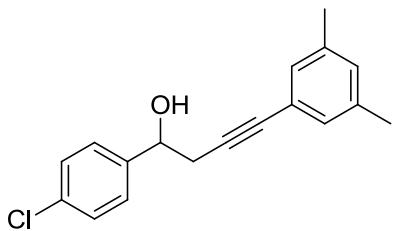
Yellow solid. ¹H NMR (400 MHz, CDCl₃, ppm): δ = 7.33-7.31 (m, 4 H), 7.18-7.17 (d, *J* = 7.6 Hz, 2 H), 6.82-6.80 (d, *J* = 8.8 Hz, 2 H), 4.91-4.89 (m, 1 H), 3.79 (s, 3 H), 2.83-2.81 (d, *J* = 7.2 Hz, 2 H), 2.44 (s, 1 H), 2.35 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ = 159.37, 139.85, 137.56, 133.05, 129.11, 125.74, 115.44, 113.88, 84.50, 82.97, 72.52, 55.27, 30.67, 21.15.



1-(2-(benzyloxy)phenyl)-4-(p-tolyl)but-3-yn-1-ol (1w)

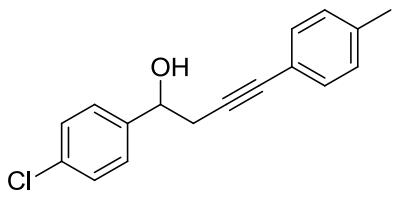
Yellow solid. ¹H NMR (400 MHz, CDCl₃, ppm): δ = 7.50-7.48 (m, 1 H), 7.42-7.30 (m, 5 H), 7.26-7.22 (m, 3 H), 7.07-7.05 (d, *J* = 8.0 Hz, 2 H), 7.02-6.98 (m, 1 H), 6.94-6.92 (m, 1 H), 5.25-5.21 (m, 1 H), 5.11 (s, 2 H), 3.03-3.01 (m, 1 H), 2.91-2.81 (m, 2 H), 2.31 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ = 155.46, 137.84, 136.79, 131.56,

131.03, 128.97, 128.74, 128.65, 128.09, 127.28, 127.12, 121.02, 120.46, 111.74, 85.77, 83.02, 70.12, 68.69, 28.76, 21.44.



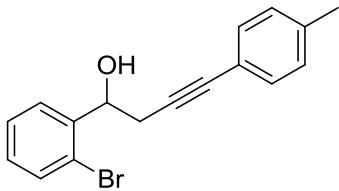
1-(4-chlorophenyl)-4-(3,5-dimethylphenyl)but-3-yn-1-ol (1x)

Yellow solid. ¹H NMR (400 MHz, CDCl₃, ppm): δ = 7.50-7.48 (m, 1 H), 7.43-7.33 (m, 7 H), 7.27-7.23 (m, 4 H), 7.03-6.99 (m, 1 H), 6.95-6.93 (m, 1 H), 5.25-5.24 (m, 1 H), 5.12 (s, 2 H), 3.04-2.99 (m, 1 H), 2.91-2.84 (m, 2 H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ = 155.46, 136.76, 131.68, 130.96, 128.74, 128.69, 128.20, 128.10, 127.82, 127.28, 127.14, 123.55, 121.03, 86.61, 82.92, 70.13, 69.01, 28.70.



1-(4-chlorophenyl)-4-(p-tolyl)but-3-yn-1-ol (1y)

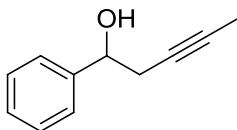
Yellow solid. ¹H NMR (400 MHz, CDCl₃, ppm): δ = 7.35-7.32 (m, 4 H), 7.28-7.26 (d, J = 8.0 Hz, 2 H), 7.11-7.09 (d, J = 8.0 Hz, 2 H), 4.93-4.91 (m, 1 H), 2.82-2.80 (m, 2 H), 2.58 (s, 1 H), 2.34 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ = 141.27, 138.31, 133.63, 131.74, 131.49, 129.29, 129.02, 128.77, 128.51, 127.45, 127.20, 120.04, 84.72, 83.73, 72.16, 30.76, 21.59.



1-(2-bromophenyl)-4-(p-tolyl)but-3-yn-1-ol (1z)

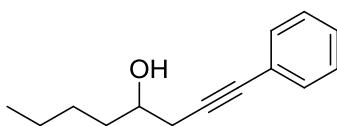
Yellow solid. ¹H NMR (400 MHz, CDCl₃, ppm): δ = 7.68-7.65 (m, 1 H), 7.55-7.53 (m, 1 H), 7.39-7.35 (m, 1 H), 7.32-7.30 (d, J = 8.0 Hz, 2 H), 7.19-7.17 (m, 1 H), 7.16-7.09 (m, 2 H), 5.32-5.28 (m, 1 H), 3.06-3.00 (m, 1 H), 2.79 (s, 3 H), 2.64-2.63 (m, 1 H), 2.34 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ = 141.55, 138.23, 132.82, 132.63, 131.81, 131.54, 129.27, 128.97, 127.56, 121.90, 120.13, 84.66, 83.67, 71.14, 29.02,

21.59.



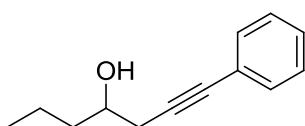
1-phenylpent-3-yn-1-ol (1aa)

Colorless oil. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.38\text{-}7.32$ (m, 4 H), 7.30-7.27 (m, 1 H), 4.81-4.78 (m, 1 H), 2.58-2.54 (m, 2 H), 2.51 (s, 1 H), 1.81-1.79 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 142.81, 128.35, 127.71, 125.68, 78.63, 75.23, 72.58, 29.96, 3.49$.



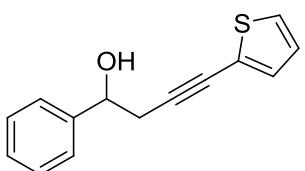
1-phenyloct-1-yn-4-ol (1ab)

Colorless oil. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.42\text{-}7.39$ (m, 2 H), 7.29-7.25 (m, 3 H), 3.85-3.80 (m, 1 H), 2.67-2.51 (m, 2 H), 2.06 (s, 1 H), 1.63-1.57 (m, 2 H), 1.48-1.33 (m, 4 H), 0.94-0.90 (m, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 131.66, 128.25, 127.90, 123.44, 86.28, 83.00, 69.96, 38.55, 28.43, 18.87, 14.03$.



1-phenylhept-1-yn-4-ol (1ac)

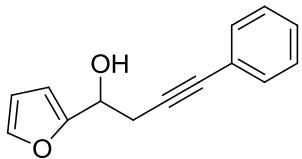
Colorless oil. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.42\text{-}7.40$ (m, 2 H), 7.29-7.27 (m, 3 H), 3.84 (s, 1 H), 2.67-2.51 (m, 2 H), 2.08 (s, 1 H), 1.61-1.54 (m, 2 H), 1.51-1.38 (m, 2 H), 0.97-0.94 (m, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 131.66, 128.26, 127.91, 123.44, 86.27, 83.02, 70.23, 36.10, 28.42, 27.82, 22.66, 14.04$.



1-phenyl-4-(thiophen-2-yl)but-3-yn-1-ol (1ad)

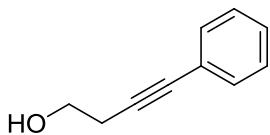
Yellow solid. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.44\text{-}7.31$ (m, 5 H), 7.20-7.14 (m, 2 H), 6.95-6.93 (m, 1 H), 4.94-4.92 (m, 1 H), 2.88-2.86 (d, $J = 6.0$ Hz, 1 H), 2.54 (s, 1 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 142.64, 131.64, 128.51, 128.00$,

126.85, 126.53, 125.80, 95.23, 90.23, 72.55, 30.86.



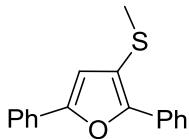
1-(furan-2-yl)-4-phenylbut-3-yn-1-ol (1ae)

Colorless oil. ^1H NMR (400 MHz, CDCl_3 , ppm): δ = 7.44-7.41 (m, 3 H), 7.32-7.28 (m, 3 H), 6.42-6.38 (m, 2 H), 5.00-4.96 (m, 1 H), 3.03-3.02 (d, J = 6.4 Hz, 2 H), 2.58-2.57 (d, J = 5.2 Hz, 1 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ = 154.94, 142.24, 131.70, 128.25, 128.04, 123.21, 110.28, 106.61, 85.18, 83.33, 66.40, 27.19.



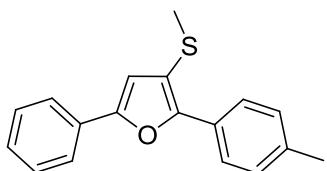
4-phenylbut-3-yn-1-ol (1af)

Colorless oil. ^1H NMR (400 MHz, CDCl_3 , ppm): δ = 7.43-7.40 (m, 2 H), 7.29-7.28 (m, 3 H), 3.82-4.79 (m, 2 H), 2.70-2.67 (m, 2 H), 2.11 (s, 1 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ = 131.67, 128.27, 127.95, 123.35, 86.40, 82.46, 61.17, 23.82.



3-(methylthio)-2,5-diphenylfuran (3a)

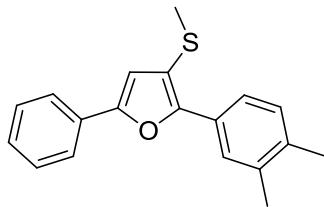
Colorless oil. ^1H NMR (400 MHz, CDCl_3 , ppm): δ = 8.02-8.00 (m, 2 H), 7.71-7.68 (m, 2 H), 7.43-7.35 (m, 4 H), 7.29-7.24 (m, 2 H), 6.74 (s, 1 H), 2.43 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ = 152.46, 149.48, 130.78, 130.21, 128.82, 128.56, 127.83, 127.41, 125.50, 123.89, 117.07, 109.78, 18.28; HRMS calcd for $\text{C}_{17}\text{H}_{15}\text{OS} [\text{M}+\text{H}]^+$ 267.0838; found: 267.0839.



3-(methylthio)-5-phenyl-2-(p-tolyl)furan (3b)

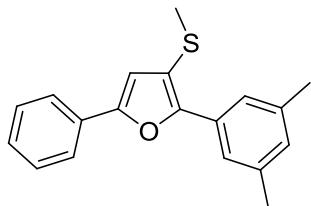
White solid, melting point: 55-56 °C. ^1H NMR (400 MHz, CDCl_3 , ppm): δ = 7.92-7.90 (d, J = 8.0 Hz, 2 H), 7.72-7.70 (m, 2 H), 7.41-7.37 (m, 2 H), 7.28-7.22 (m, 3

H), 6.75 (s, 1 H), 2.43 (s, 3 H), 2.38 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ = 152.08, 150.00, 137.37, 130.27, 129.22, 128.77, 128.00, 127.66, 125.50, 123.78, 116.04, 109.89, 21.38, 18.37; HRMS calcd for $\text{C}_{18}\text{H}_{17}\text{OS}$ $[\text{M}+\text{H}]^+$ 281.0995; found: 281.0991.



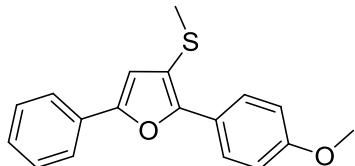
2-(3,4-dimethylphenyl)-3-(methylthio)-5-phenylfuran (3c)

White solid, melting point: 58-60 °C. ^1H NMR (400 MHz, CDCl_3 , ppm): δ = 7.77-7.72 (m, 2 H), 7.72-7.70 (m, 2 H), 7.41-7.37 (m, 2 H), 7.28-7.26 (m, 1 H), 7.22-7.18 (m, 1 H), 6.75 (s, 1 H), 2.43 (s, 3 H), 2.33 (s, 3 H), 2.29 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ = 152.01, 150.07, 136.68, 136.16, 130.32, 129.78, 128.76, 128.39, 127.62, 126.69, 123.78, 123.20, 115.93, 109.82, 20.01, 19.69, 18.35; HRMS calcd for $\text{C}_{19}\text{H}_{19}\text{OS}$ $[\text{M}+\text{H}]^+$ 295.1151; found: 295.1148.



2-(3,5-dimethylphenyl)-3-(methylthio)-5-phenylfuran (3d)

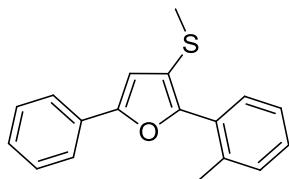
Colorless oil. ^1H NMR (400 MHz, CDCl_3 , ppm): δ = 7.73-7.71 (m, 2 H), 7.62 (s, 2 H), 7.41-7.38 (m, 2 H), 7.29-7.25 (m, 1 H), 6.94 (s, 1 H), 6.75 (s, 1 H), 2.45 (s, 3 H), 2.38 (s, 6 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ = 152.22, 149.73, 138.00, 130.58, 130.28, 129.25, 128.76, 127.69, 123.83, 123.35, 116.68, 109.58, 21.53, 18.22; HRMS calcd for $\text{C}_{19}\text{H}_{19}\text{OS}$ $[\text{M}+\text{H}]^+$ 295.1151; found: 295.1146.



2-(4-methoxyphenyl)-3-(methylthio)-5-phenylfuran (3e)

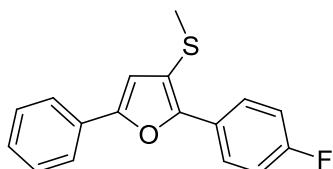
Colorless oil. ^1H NMR (400 MHz, CDCl_3 , ppm): δ = 7.98-7.96 (m, 2 H), 7.70-7.69 (d,

$J = 4$ Hz, 2 H), 7.41-7.37 (m, 2 H), 7.28-7.24 (m, 1 H), 6.99-6.97 (m, 2 H), 6.75 (s, 1 H), 3.85 (s, 3 H), 2.43 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 159.05$, 151.78, 150.16, 130.30, 128.75, 127.55, 127.09, 123.68, 114.85, 113.98, 113.88, 110.09, 55.34, 18.50; HRMS calcd for $\text{C}_{18}\text{H}_{17}\text{O}_2\text{S}$ $[\text{M}+\text{H}]^+$ 297.0944; found: 297.0940.



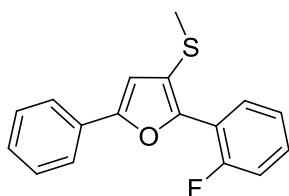
3-(methylthio)-5-phenyl-2-(o-tolyl)furan (3f)

Colorless oil. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.69$ -7.67 (d, $J = 7.2$ Hz, 2 H), 7.54-7.53 (d, $J = 6.8$ Hz, 1 H), 7.40-7.36 (m, 2 H), 7.30-7.23 (m, 4 H), 6.80 (s, 1 H), 2.47 (s, 3 H), 2.35 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 152.98$, 151.77, 137.35, 130.81, 130.39, 130.19, 129.64, 128.79, 128.76, 127.68, 125.49, 123.66, 117.56, 108.83, 20.90, 18.48; HRMS calcd for $\text{C}_{18}\text{H}_{17}\text{OS}$ $[\text{M}+\text{H}]^+$ 281.0995; found: 281.0997.



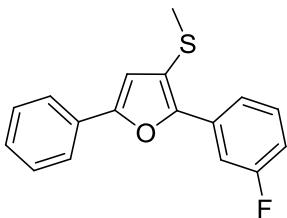
2-(4-fluorophenyl)-3-(methylthio)-5-phenylfuran (3g)

White solid, melting point: 54-55 °C. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 8.02$ -7.98 (m, 2 H), 7.71-7.69 (d, $J = 7.6$ Hz, 2 H), 7.42-7.39 (m, 2 H), 7.31-7.25 (m, 1 H), 7.15-7.11 (m, 2 H), 6.76 (s, 1 H), 2.45 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 163.23$ -160.76 (d, $J = 246$ Hz, 1 C), 152.41, 148.88, 130.06, 128.79, 127.84, 127.36-127.28 (d, $J = 7.9$ Hz, 1 C), 127.04-127.01 (d, $J = 3$ Hz, 1 C), 123.81, 116.41, 115.64-115.43 (d, $J = 21.5$ Hz, 1 C), 109.85, 18.31; HRMS calcd for $\text{C}_{17}\text{H}_{14}\text{FOS}$ $[\text{M}+\text{H}]^+$ 285.0744; found: 285.0747.



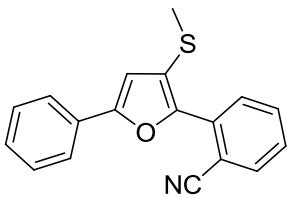
2-(2-fluorophenyl)-3-(methylthio)-5-phenylfuran (3h)

Colorless oil. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.76\text{-}7.70$ (m, 3 H), $7.42\text{-}7.38$ (m, 2 H), $7.35\text{-}7.32$ (m, 1 H), $7.30\text{-}7.27$ (m, 1 H), $7.24\text{-}7.15$ (m, 2 H), 6.81 (s, 1 H), 2.42 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 160.54\text{-}158.04$ (d, $J = 250$ Hz, 1 C), 153.79 , 146.04 , 130.11 , $130.07\text{-}130.05$ (d, $J = 13$ Hz, 1 C), 129.92 , 129.84 , 128.76 , 127.90 , 123.91 , 119.29 , $118.58\text{-}118.45$ (d, $J = 5$ Hz, 1 C), $116.41\text{-}116.19$ (d, $J = 22$ Hz, 1 C), 108.95 , $18.30\text{-}18.29$ (d, $J = 1$ Hz, 1 C); HRMS calcd for $\text{C}_{17}\text{H}_{14}\text{FOS}$ [$\text{M+H}]^+$ 285.0744; found: 285.0741.



2-(3-fluorophenyl)-3-(methylthio)-5-phenylfuran (3i)

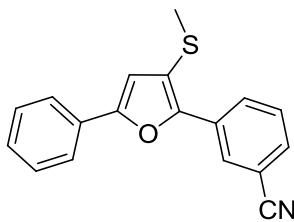
Colorless oil. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.82\text{-}7.80$ (d, $J = 8.0$ Hz, 1 H), $7.77\text{-}7.72$ (m, 3 H), $7.44\text{-}7.39$ (m, 3 H), $7.33\text{-}7.29$ (m, 1 H), $7.00\text{-}6.96$ (m, 1 H), 6.78 (s, 1 H), 2.49 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 164.16\text{-}161.73$ (d, $J = 243$ Hz, 1 C), 152.84 , $147.91\text{-}147.88$ (d, $J = 3$ Hz, 1 C), $132.68\text{-}132.60$ (d, $J = 8$ Hz, 1 C), $130.10\text{-}130.01$ (d, $J = 8$ Hz, 1 C), 129.89 , 128.82 , 128.05 , 123.94 , $120.93\text{-}120.90$ (d, $J = 3$ Hz, 1 C), 118.37 , $114.15\text{-}113.93$ (d, $J = 21$ Hz, 1 C), $112.14\text{-}111.90$ (d, $J = 23$ Hz, 1 C), 109.61 , 18.09 ; HRMS calcd for $\text{C}_{17}\text{H}_{14}\text{FOS}$ [$\text{M+H}]^+$ 285.0744; found: 285.0740.



2-(3-(methylthio)-5-phenylfuran-2-yl)benzonitrile (3j)

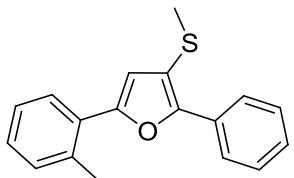
White solid, melting point: 95-96 °C. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 8.02\text{-}8.00$ (d, $J = 8.0$ Hz, 1 H), $7.86\text{-}7.84$ (d, $J = 7.6$ Hz, 2 H), $7.80\text{-}7.78$ (d, $J = 7.6$ Hz, 1 H), $7.65\text{-}7.61$ (m, 1 H), $7.46\text{-}7.39$ (m, 2 H), $7.37\text{-}7.30$ (m, 2 H), 6.83 (s, 1 H), 2.50 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 154.73$, 145.78 , 134.76 , 132.89 , 132.33 , 129.59 , 128.90 , 128.43 , 128.14 , 127.33 , 124.42 , 121.17 , 119.15 , 108.98 , 108.86 ,

17.99; HRMS calcd for C₁₈H₁₄NOS [M+H]⁺ 292.0791; found: 292.0796.



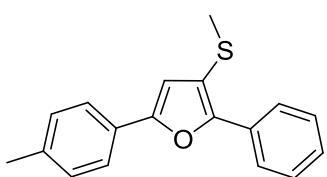
3-(3-(methylthio)-5-phenylfuran-2-yl)benzonitrile (3k)

White solid, melting point: 103-104 °C. ¹H NMR (400 MHz, CDCl₃, ppm): δ = 8.30 (s, 1 H), 8.24-8.21 (m, 1 H), 7.74-7.71 (m, 2 H), 7.53-7.52 (m, 2 H), 7.45-7.42 (m, 2 H), 7.35-7.31 (m, 1 H), 6.79 (s, 1 H), 2.50 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ = 153.55, 146.47, 131.84, 130.10, 129.59, 129.34, 128.95, 128.89, 128.46, 128.37, 124.06, 119.69, 118.85, 112.83, 109.51, 18.04; HRMS calcd for C₁₈H₁₄NOS [M+H]⁺ 292.0791; found: 292.0795.



3-(methylthio)-2-phenyl-5-(o-tolyl)furan (3l)

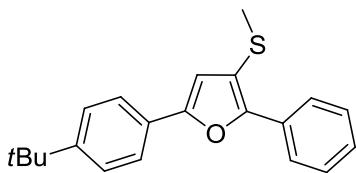
Colorless oil. ¹H NMR (400 MHz, CDCl₃, ppm): δ = 8.04-8.01 (m, 2 H), 7.75-7.74 (d, J = 7.2 Hz, 1 H), 7.45-7.41 (m, 2 H), 7.30-7.22 (m, 4 H), 6.66 (s, 1 H), 2.56 (s, 3 H), 2.46 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ = 152.29, 149.40, 134.70, 131.36, 130.75, 129.51, 128.54, 127.88, 127.39, 126.98, 126.12, 125.47, 116.39, 113.32, 22.04, 18.37; HRMS calcd for C₁₈H₁₇OS [M+H]⁺ 281.0995; found: 281.0991.



3-(methylthio)-2-phenyl-5-(p-tolyl)furan (3m)

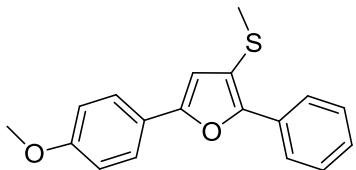
Colorless oil. ¹H NMR (400 MHz, CDCl₃, ppm): δ = 8.02-8.00 (m, 2 H), 7.62-7.60 (d, J = 8.4 Hz, 2 H), 7.45-7.41 (m, 2 H), 7.30-7.28 (d, J = 7.2 Hz, 1 H), 7.23-7.20 (m, 2 H), 6.71 (s, 1 H), 2.45 (s, 3 H), 2.37 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ = 152.69, 149.07, 137.74, 130.81, 129.47, 128.49, 127.50, 127.24, 125.39, 123.83, 116.89, 109.02, 21.35, 18.25; HRMS calcd for C₁₈H₁₇OS [M+H]⁺ 281.0995; found:

281.0993.



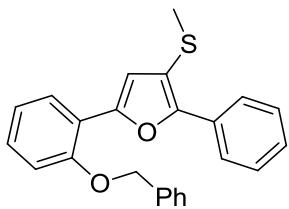
5-(4-(tert-butyl)phenyl)-3-(methylthio)-2-phenylfuran (3n)

Colorless oil. ^1H NMR (400 MHz, CDCl_3 , ppm): δ = 8.02-8.00 (d, J = 6.8 Hz, 2 H), 7.66-7.64 (d, J = 8.4 Hz, 2 H), 7.43-7.41 (m, 4 H), 7.29-7.23 (m, 1 H), 6.72 (s, 1 H), 2.45 (s, 3 H), 1.34 (s, 9 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ = 152.67, 150.99, 149.14, 130.83, 128.49, 127.49, 127.23, 125.70, 125.39, 123.70, 116.89, 109.13, 34.71, 31.28, 18.25; HRMS calcd for $\text{C}_{21}\text{H}_{23}\text{OS} [\text{M}+\text{H}]^+$ 323.1464; found: 323.1470.



5-(4-methoxyphenyl)-3-(methylthio)-2-phenylfuran (3o)

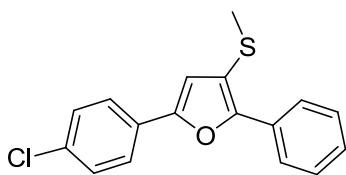
Colorless oil. ^1H NMR (400 MHz, CDCl_3 , ppm): δ = 8.01-8.00 (d, J = 7.2 Hz, 2 H), 7.67-7.64 (m, 2 H), 7.45-7.41 (m, 2 H), 7.30-7.25 (m, 1 H), 6.96-6.93 (m, 2 H), 6.64 (s, 1 H), 3.85 (s, 3 H), 2.46 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ = 159.42, 152.54, 148.83, 130.83, 128.46, 127.12, 125.34, 125.30, 123.24, 116.85, 114.25, 108.19, 55.36, 18.24; HRMS calcd for $\text{C}_{18}\text{H}_{17}\text{O}_2\text{S} [\text{M}+\text{H}]^+$ 297.0944; found: 297.0939.



5-(2-(benzyloxy)phenyl)-3-(methylthio)-2-phenylfuran (3p)

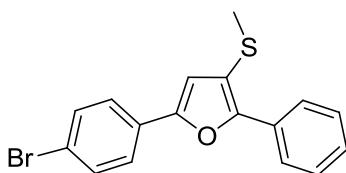
White solid, melting point: 67-69 °C. ^1H NMR (400 MHz, CDCl_3 , ppm): δ = 7.97-7.96 (d, J = 7.6 Hz, 3 H), 7.51-7.50 (d, J = 7.2 Hz, 2 H), 7.44-7.36 (m, 5 H), 7.28-7.22 (m, 2 H), 7.08-7.02 (m, 3 H), 5.18 (s, 2 H), 2.30 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ = 154.78, 148.80, 147.55, 136.71, 130.89, 128.67, 128.48, 128.39, 128.24, 127.88, 127.05, 125.94, 125.34, 121.13, 119.51, 117.22, 114.24, 112.23, 70.61, 17.61; HRMS calcd for $\text{C}_{24}\text{H}_{21}\text{O}_2\text{S} [\text{M}+\text{H}]^+$ 373.1257; found:

373.1259.



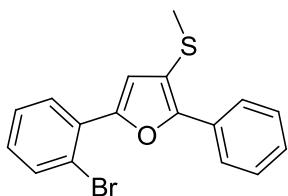
5-(4-chlorophenyl)-3-(methylthio)-2-phenylfuran (3q)

White solid, melting point: 60-62 °C. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 8.03\text{-}8.02$ (d, $J = 1.0$ Hz, 2 H), 7.67-7.66 (m, 2 H), 7.50-7.46 (m, 2 H), 7.42-7.41 (d, $J = 2.4$ Hz, 2 H), 7.40-7.32 (m, 1 H), 6.79 (s, 1 H), 2.49 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 151.33, 149.73, 133.43, 130.51, 129.01, 128.64, 128.54, 127.55, 125.49, 125.02, 117.15, 110.16, 18.23$; HRMS calcd for $\text{C}_{17}\text{H}_{14}\text{ClOS}$ $[\text{M}+\text{H}]^+$ 301.0449; found: 301.0445.



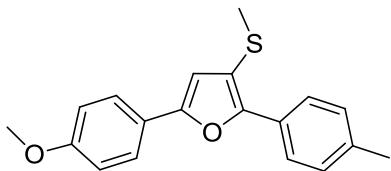
5-(4-bromophenyl)-3-(methylthio)-2-phenylfuran (3r)

Colorless oil. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 8.01\text{-}7.99$ (d, $J = 7.2$ Hz, 2 H), 7.59-7.51 (m, 4 H), 7.46-7.42 (m, 2 H), 7.32-7.29 (m, 1 H), 6.77 (s, 1 H), 2.46 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 151.34, 149.81, 131.93, 130.49, 129.06, 128.54, 127.57, 125.50, 125.27, 121.55, 117.15, 110.28, 18.24$; HRMS calcd for $\text{C}_{17}\text{H}_{14}\text{BrOS}$ $[\text{M}+\text{H}]^+$ 344.9943; found: 344.9949.



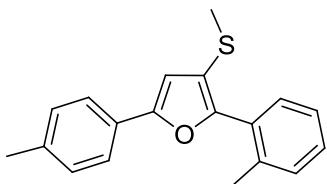
5-(2-bromophenyl)-3-(methylthio)-2-phenylfuran (3s)

Colorless oil. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 8.04\text{-}8.02$ (m, 2 H), 7.89-7.86 (m, 1 H), 7.68-7.65 (m, 1 H), 7.46-7.42 (m, 2 H), 7.40-7.36 (m, 1 H), 7.33-7.29 (m, 2 H), 7.16-7.12 (m, 1 H), 2.48 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 149.77, 149.74, 134.28, 130.55, 130.47, 128.70, 128.63, 128.54, 127.64, 127.50, 125.65, 119.58, 116.57, 115.31, 18.34$; HRMS calcd for $\text{C}_{17}\text{H}_{14}\text{BrOS}$ $[\text{M}+\text{H}]^+$ 344.9943; found: 344.9939.



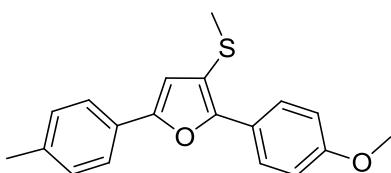
5-(4-methoxyphenyl)-3-(methylthio)-2-(p-tolyl)furan (3t)

Colorless oil. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.90\text{-}7.88$ (d, $J = 8.0$ Hz, 2 H), 7.66-7.63 (d, $J = 9.2$ Hz, 2 H), 7.26-7.23 (m, 2 H), 6.95-6.93 (d, $J = 8.8$ Hz, 2 H), 6.63 (s, 1 H), 3.84 (s, 3 H), 2.44 (s, 3 H), 2.38 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 159.32, 152.18, 149.35, 137.08, 129.15, 128.10, 125.34, 125.26, 123.35, 115.87, 114.23, 108.32, 55.35, 21.33, 18.34$; HRMS calcd for $\text{C}_{19}\text{H}_{19}\text{O}_2\text{S}$ $[\text{M}+\text{H}]^+$ 311.1101; found: 311.1107.



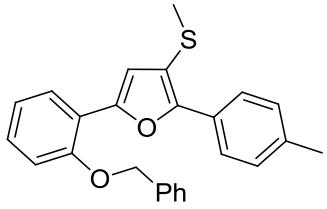
3-(methylthio)-2-(o-tolyl)-5-(p-tolyl)furan (3u)

Colorless oil. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.58\text{-}7.55$ (d, $J = 8.0$ Hz, 2 H), 7.54-7.52 (d, $J = 6.4$ Hz, 1 H), 7.28-7.24 (m, 3 H), 7.19-7.17 (d, $J = 8.0$ Hz, 2 H), 6.73 (s, 1 H), 2.46 (s, 3 H), 2.35 (s, 3 H), 2.34 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 153.24, 151.37, 137.61, 137.33, 130.80, 130.18, 129.74, 129.48, 128.67, 127.74, 125.47, 123.66, 117.44, 108.11, 21.33, 20.92, 18.47$; HRMS calcd for $\text{C}_{19}\text{H}_{19}\text{OS}$ $[\text{M}+\text{H}]^+$ 295.1151; found: 295.1155.



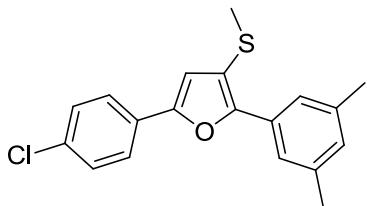
2-(4-methoxyphenyl)-3-(methylthio)-5-(p-tolyl)furan (3v)

White solid, melting point: 59-60 °C. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.97\text{-}7.95$ (d, $J = 8.8$ Hz, 2 H), 7.60-7.58 (d, $J = 8.4$ Hz, 2 H), 7.21-7.18 (d, $J = 8.4$ Hz, 2 H), 6.98-6.96 (d, $J = 8.8$ Hz, 2 H), 6.69 (s, 1 H), 3.84 (s, 3 H), 2.42 (s, 3 H), 2.36 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 158.96, 152.05, 149.76, 137.46, 129.43, 127.64, 127.02, 123.78, 123.67, 114.76, 113.95, 109.35, 55.33, 21.32, 18.48$; HRMS calcd for $\text{C}_{19}\text{H}_{19}\text{O}_2\text{S}$ $[\text{M}+\text{H}]^+$ 311.1101; found: 311.1106.



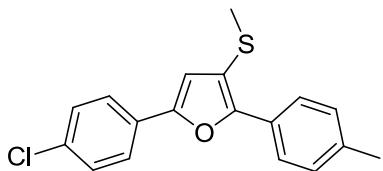
5-(2-(benzyloxy)phenyl)-3-(methylthio)-2-(p-tolyl)furan (3w)

White solid, melting point: 70-72 °C. ¹H NMR (400 MHz, CDCl₃, ppm): δ = 7.97-7.95 (m, 1 H), 7.88-7.86 (d, *J* = 8.4 Hz, 2 H), 7.52-7.51 (d, *J* = 7.2 Hz, 2 H), 7.43-7.36 (m, 3 H), 7.25-7.22 (m, 3 H), 7.08-7.01 (m, 3 H), 5.20 (s, 2 H), 2.38 (s, 3 H), 2.30 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ = 154.68, 148.42, 148.08, 137.00, 136.72, 129.15, 128.65, 128.21, 128.14, 127.84, 125.87, 125.37, 121.09, 119.60, 116.22, 114.38, 112.21, 70.57, 21.34, 17.72; HRMS calcd for C₂₅H₂₃O₂S [M+H]⁺ 387.1414; found: 387.1409.



5-(4-chlorophenyl)-2-(3,5-dimethylphenyl)-3-(methylthio)furan (3x)

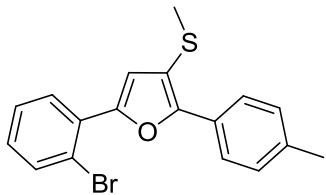
White solid, melting point: 53-55 °C. ¹H NMR (400 MHz, CDCl₃, ppm): δ = 7.65-7.63 (d, *J* = 8.8 Hz, 2 H), 7.59 (s, 2 H), 7.38-7.36 (d, *J* = 8.4 Hz, 2 H), 6.95 (s, 1 H), 6.74 (s, 1 H), 2.45 (s, 3 H), 2.39 (s, 6 H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ = 151.12, 150.03, 138.04, 133.30, 130.35, 129.41, 128.97, 128.73, 125.00, 123.35, 116.81, 110.00, 21.49, 18.19; HRMS calcd for C₁₉H₁₈ClOS [M+H]⁺ 329.0762; found: 329.0767.



5-(4-chlorophenyl)-3-(methylthio)-2-(p-tolyl)furan (3y)

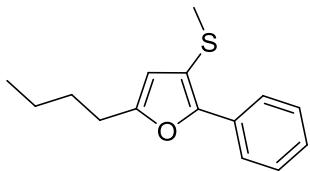
White solid, melting point: 50-52 °C. ¹H NMR (400 MHz, CDCl₃, ppm): δ = 7.90-7.88 (d, *J* = 8.0 Hz, 2 H), 7.64-7.62 (d, *J* = 8.4 Hz, 2 H), 7.37-7.35 (d, *J* = 8.8 Hz, 2 H), 7.25-7.23 (d, *J* = 6.8 Hz, 2 H), 6.74 (s, 1 H), 2.44 (s, 3 H), 2.39 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ = 150.98, 150.28, 137.57, 133.27, 129.23, 128.98,

128.73, 127.76, 125.51, 124.95, 116.17, 110.30, 21.37, 18.33; HRMS calcd for C₁₈H₁₆ClOS [M+H]⁺ 315.0605; found: 315.0608.



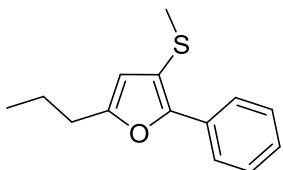
5-(2-bromophenyl)-3-(methylthio)-2-(p-tolyl)furan (3z)

Colorless oil. ¹H NMR (400 MHz, CDCl₃, ppm): δ = 7.93-7.91 (d, *J* = 8.4 Hz, 2 H), 7.87-7.85 (m, 1 H), 7.66-7.64 (m, 1 H), 7.39-7.35 (m, 1 H), 7.29 (s, 1 H), 7.25-7.23 (m, 2 H), 7.14-7.10 (m, 1 H), 2.45 (s, 3 H), 2.38 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ = 150.28, 149.37, 137.67, 134.27, 130.62, 130.29, 129.24, 128.55, 127.74, 127.49, 125.67, 119.49, 115.67, 115.49, 21.39, 18.43; HRMS calcd for C₁₈H₁₆BrOS [M+H]⁺ 359.0100; found: 359.0106.



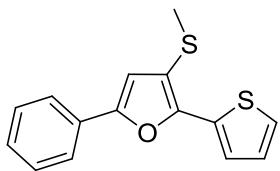
5-butyl-3-(methylthio)-2-phenylfuran (3ab)

Colorless oil. ¹H NMR (400 MHz, CDCl₃, ppm): δ = 7.90-7.89 (m, 2 H), 7.41-7.36 (m, 2 H), 7.24-7.20 (m, 1 H), 6.11 (s, 1 H), 2.66-2.62 (m, 2 H), 2.38 (s, 3 H), 1.70-1.62 (m, 2 H), 1.45-1.36 (m, 2 H), 0.96-0.93 (m, 3 H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ = 155.71, 148.34, 131.11, 128.38, 126.80, 125.13, 115.03, 109.66, 30.05, 27.82, 22.30, 18.17, 13.84; HRMS calcd for C₁₅H₁₉OS [M+H]⁺ 247.1151; found: 247.1154.



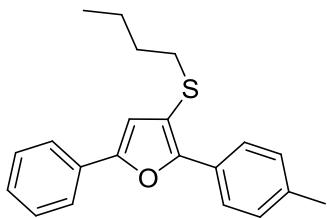
3-(methylthio)-2-phenyl-5-propylfuran (3ac)

Colorless oil. ¹H NMR (400 MHz, CDCl₃, ppm): δ = 7.90-7.88 (d, *J* = 8.0 Hz, 2 H), 7.40-7.37 (m, 2 H), 7.24-7.21 (m, 2 H), 6.12 (s, 1 H), 2.64-2.60 (m, 2 H), 2.39 (s, 3 H), 1.73-1.68 (m, 2 H), 1.01-0.98 (m, 3 H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ = 155.52, 148.37, 131.08, 128.36, 126.80, 125.12, 114.98, 109.78, 30.11, 21.26, 18.17, 13.77; HRMS calcd for C₁₄H₁₇OS [M+H]⁺ 233.0995; found: 233.0991.



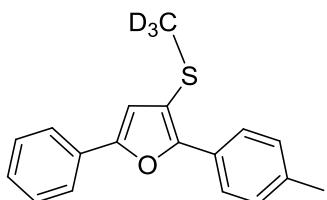
3-(methylthio)-5-phenyl-2-(thiophen-2-yl)furan (3ad)

Colorless oil. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.70\text{-}7.68$ (d, $J = 7.6$ Hz, 2 H), 7.61-7.60 (m, 1 H), 7.42-7.38 (m, 2 H), 7.31-7.25 (m, 2 H), 7.11-7.09 (m, 1 H), 6.75 (s, 1 H), 2.45 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 152.23, 147.52, 132.42, 129.96, 128.78, 127.81, 127.34, 125.10, 124.16, 123.80, 115.61, 110.05, 18.42$; HRMS calcd for $\text{C}_{15}\text{H}_{13}\text{OS}_2$ $[\text{M}+\text{H}]^+$ 273.0403; found: 273.0405.



3-(butylthio)-5-phenyl-2-(p-tolyl)furan (3ah)

Colorless oil. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 8.00\text{-}7.98$ (d, $J = 8.4$ Hz, 2 H), 7.73-7.71 (m, 2 H), 7.42-7.38 (m, 2 H), 7.29-7.22 (m, 4 H), 6.75 (s, 1 H), 2.84-2.81 (m, 2 H), 2.39 (s, 3 H), 1.65-1.57 (m, 2 H), 1.46-1.37 (m, 2 H), 0.90-0.86 (m, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 151.90, 151.26, 137.40, 130.31, 129.41, 129.13, 128.75, 128.05, 127.62, 125.64, 123.77, 114.32, 111.22, 35.18, 31.61, 21.84, 21.37, 13.64$; HRMS calcd for $\text{C}_{21}\text{H}_{23}\text{OS}$ $[\text{M}+\text{H}]^+$ 323.1464; found: 323.1460.

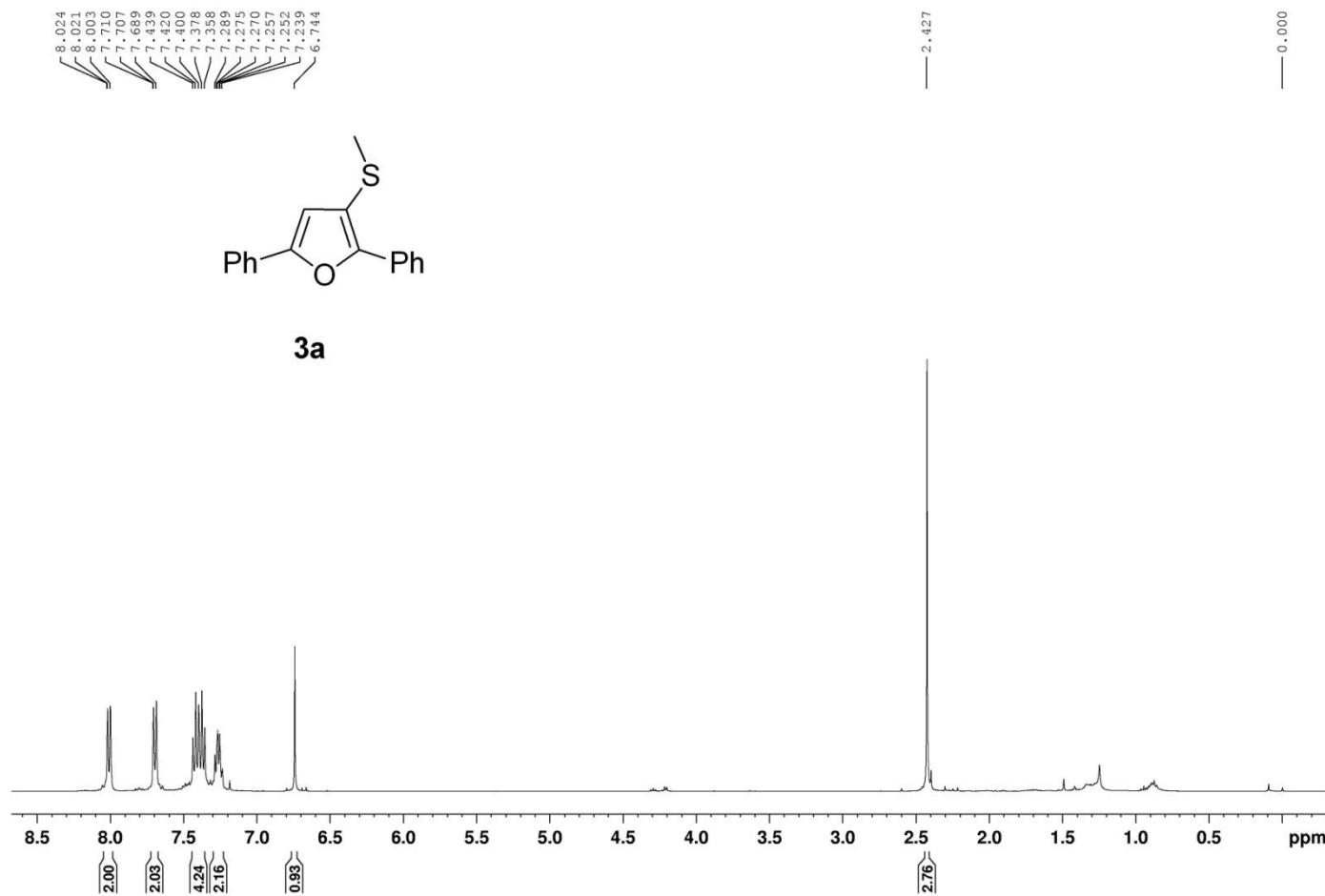


3-(methylthio)-5-phenyl-2-(p-tolyl)furan (3aj)

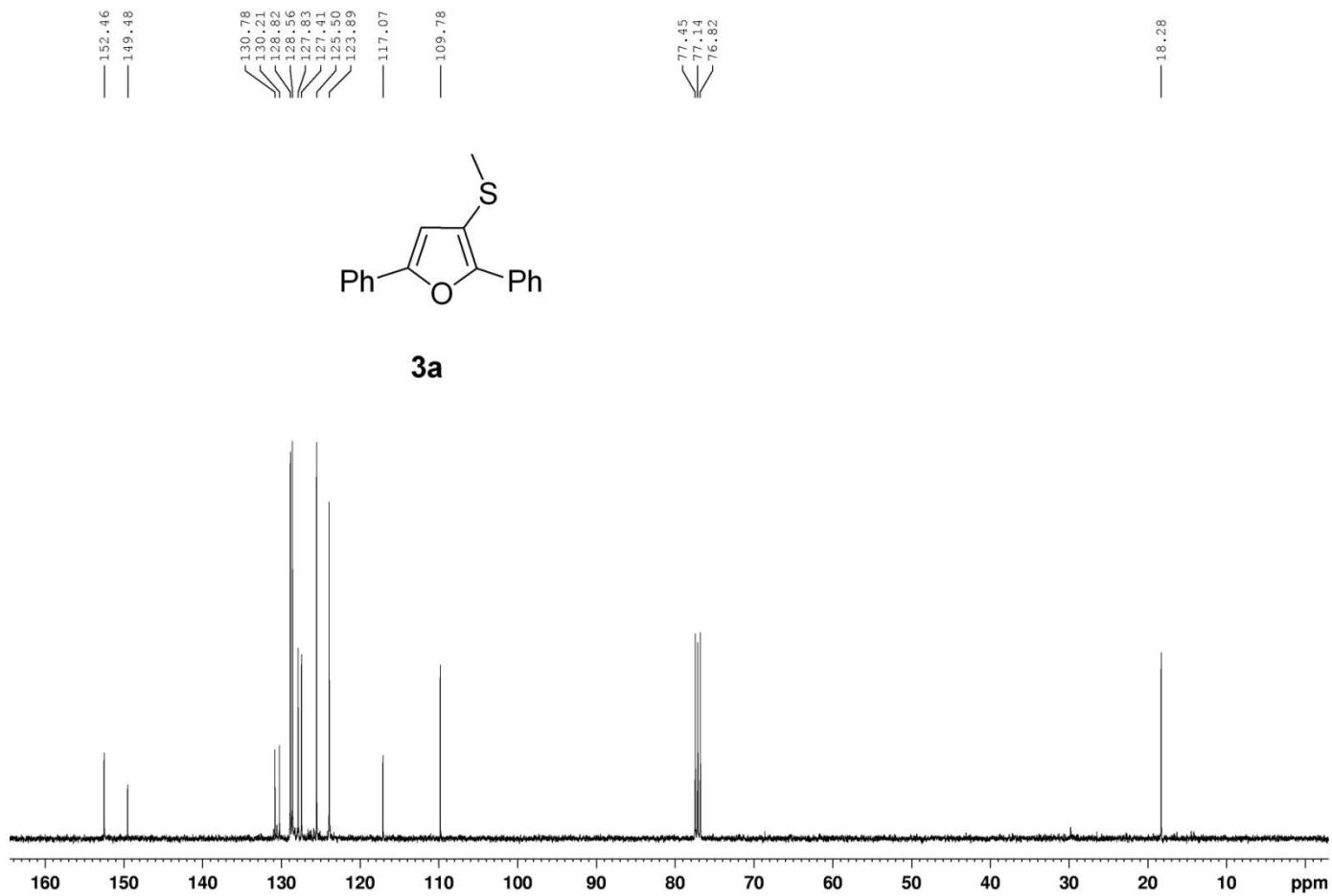
Colorless oil. ^1H NMR (400 MHz, CDCl_3 , ppm): $\delta = 7.96\text{-}7.94$ (d, $J = 8.0$ Hz, 2 H), 7.76-7.74 (d, $J = 7.6$ Hz, 2 H), 7.46-7.42 (m, 2 H), 7.33-7.27 (m, 4 H), 6.80 (s, 1 H), 2.43 (s, 3 H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): $\delta = 152.07, 150.00, 137.34, 130.27, 129.19, 128.75, 127.99, 127.64, 125.48, 123.76, 115.94, 109.87, 21.35$; HRMS calcd for $\text{C}_{18}\text{H}_{15}\text{D}_3\text{OS}$ $[\text{M}+\text{H}]^+$ 283.1183; found: 283.1180.

Refrence:

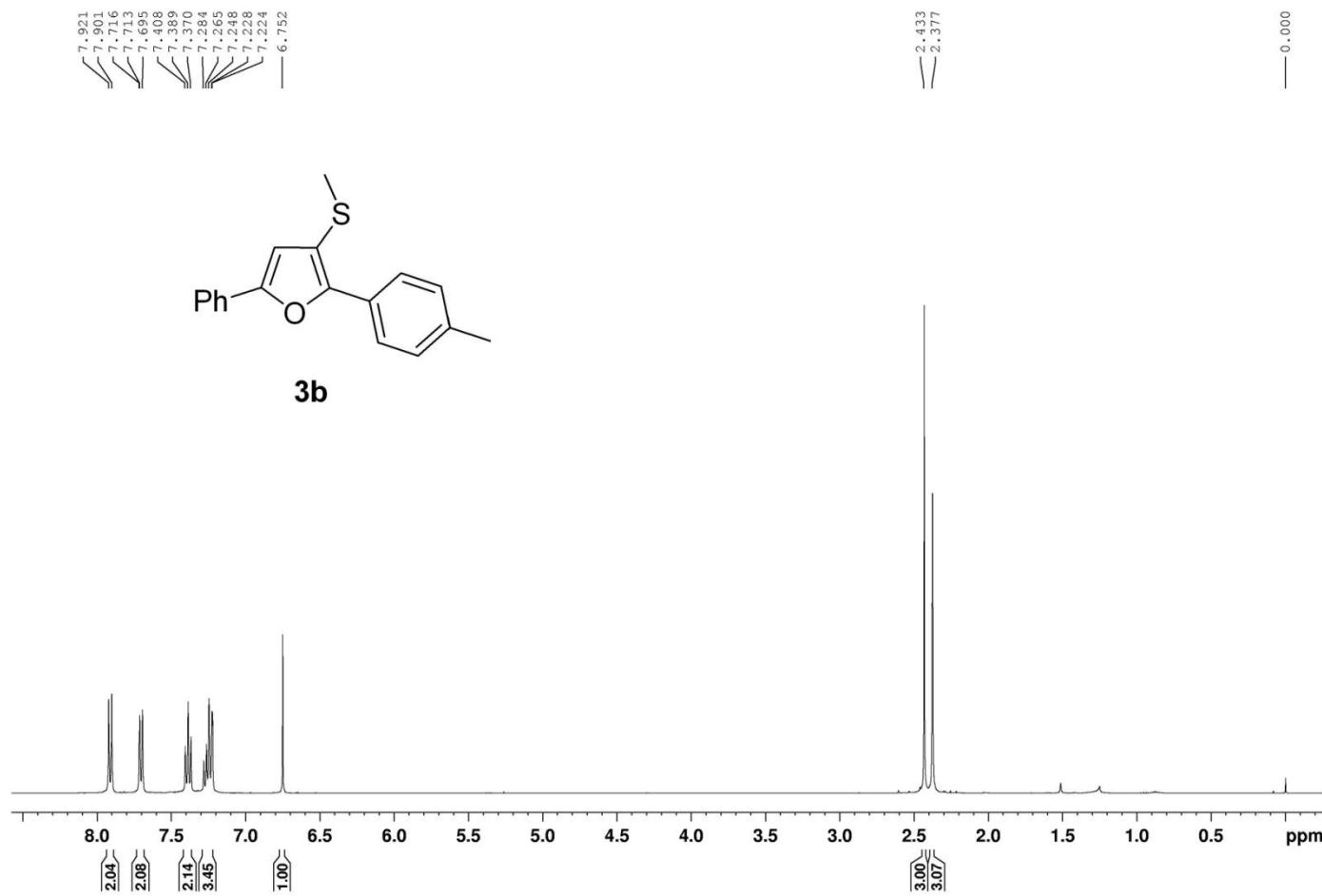
- [1] P. Gao, Y. -W. Shen, R. Fang, X.-H. Hao, Z.-H. Qiu, F. Yang, X.-B. Yan, Q. Wang, X.-J. Gong, X.-Y. Liu and Y. -M. Liang, *Angew. Chem., Int. Ed.*, 2014, **53**, 7629.
- [2] D.-Q. Chen, P. Gao, P.-X. Zhou, X.-R. Song, Y.-F. Qiu, X.-Y. Liu and Y.-M. Liang, *Chem. Commun.*, 2015, **51**, 6637.
- [3]. Y .-Z. Chen, L.-Z. Wu, M.-L. Peng, D. Zhang, L.-P. Zhang and C.-H. Tung, *Tetrahedron* 2006, **62**, 10688.
- [4]. A. Arrault, F. Touzeau, G. Guillaumet, J.-Y. M éour, *Synthesis* 1999, **7**, 1241.

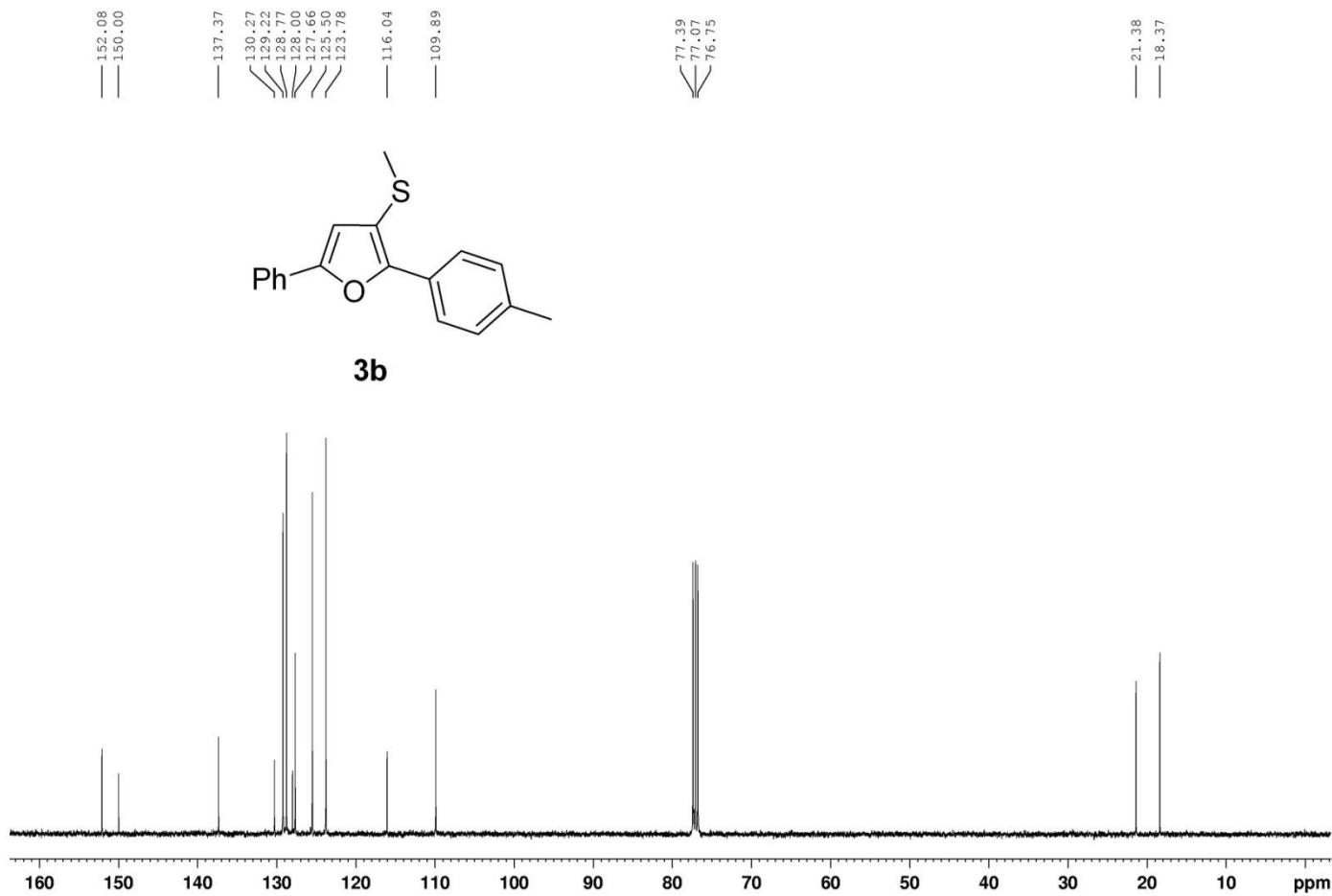


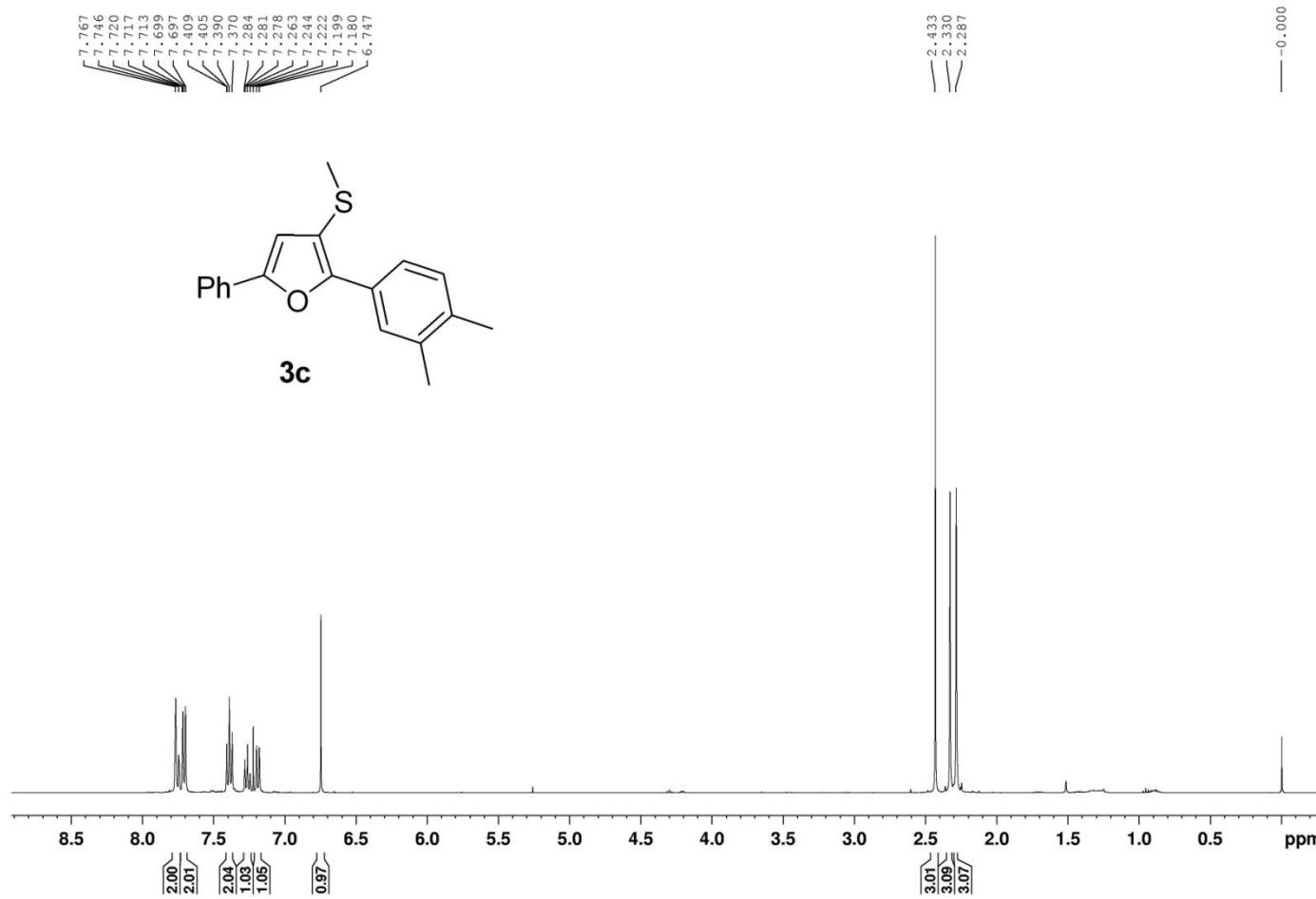
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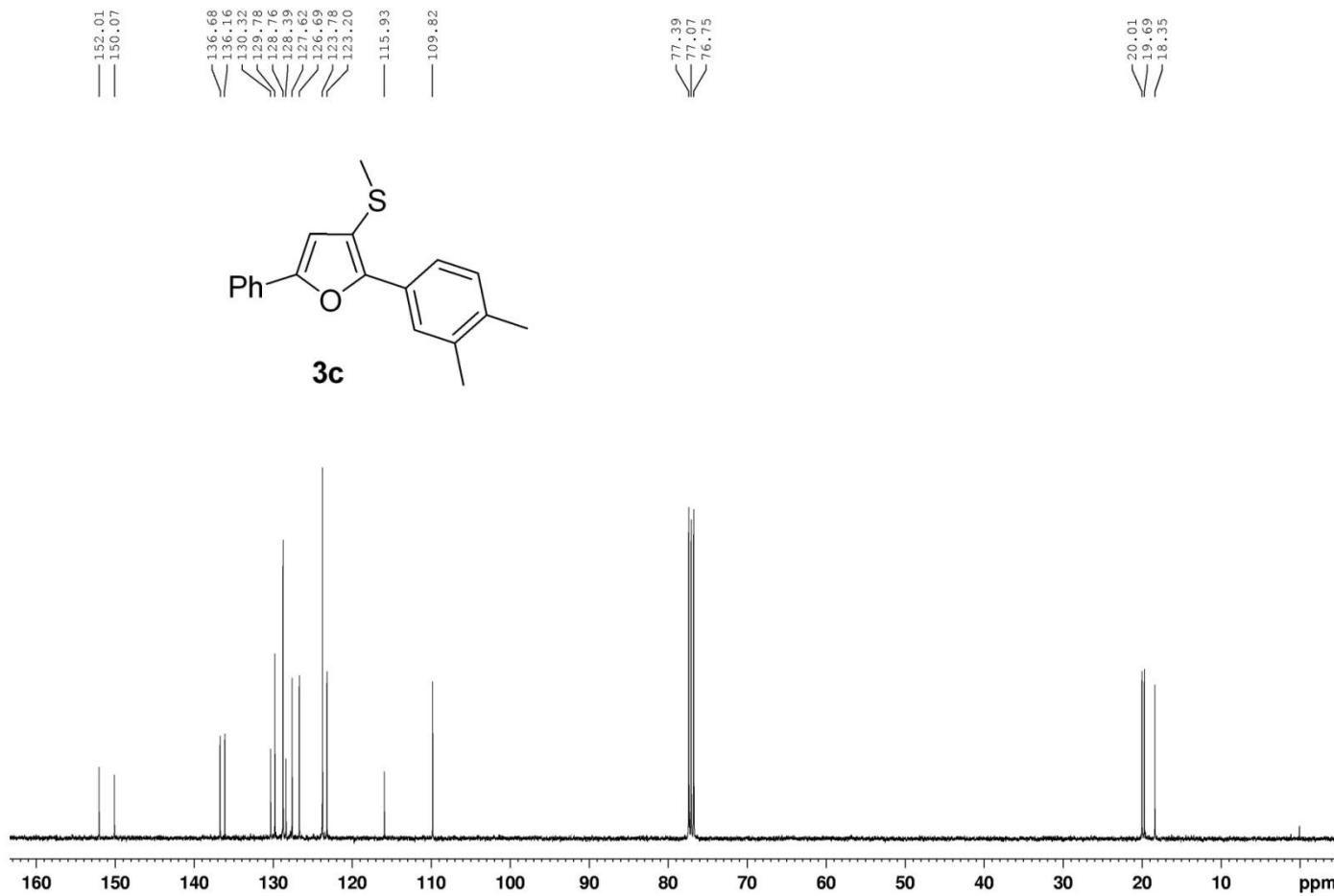


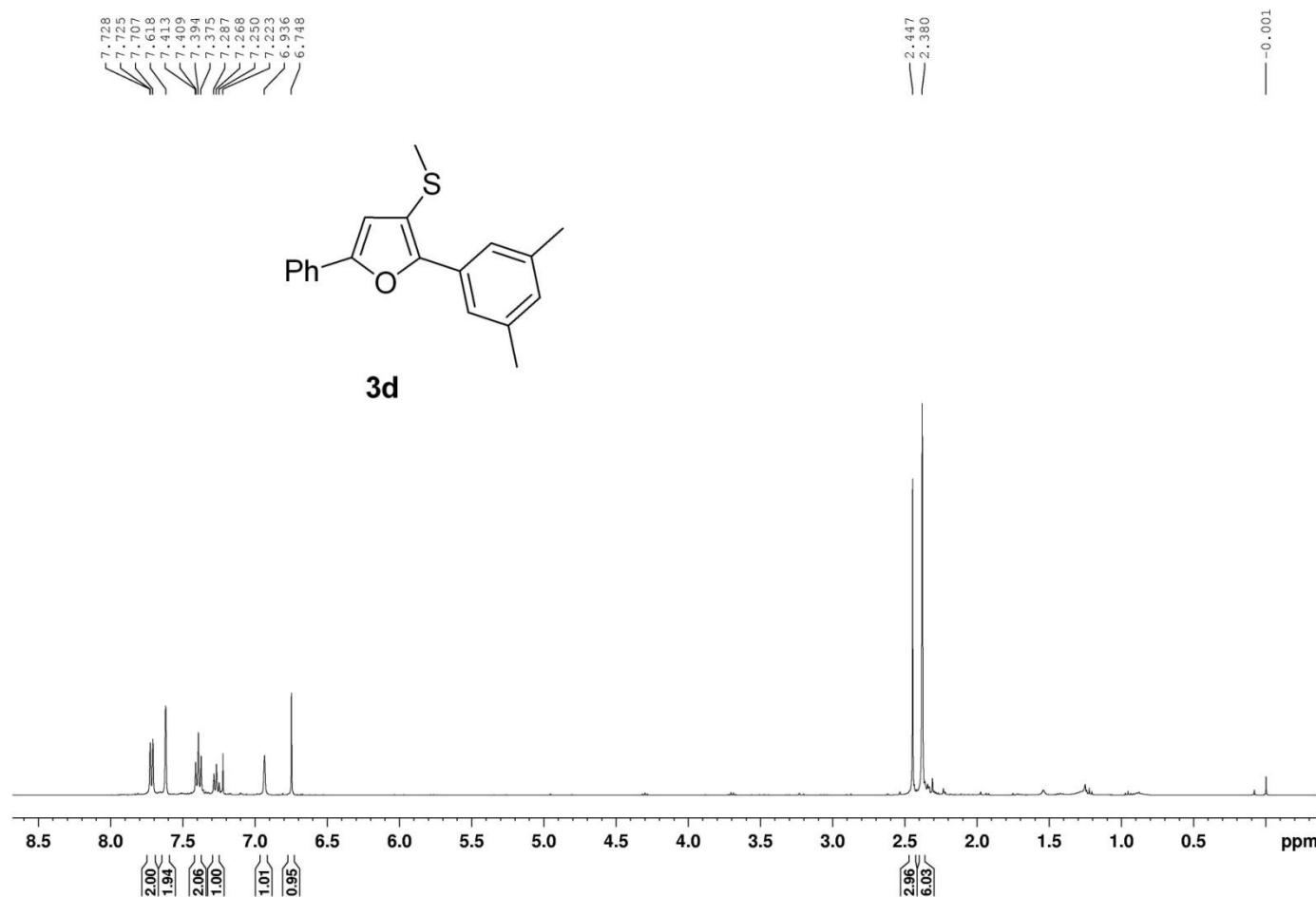
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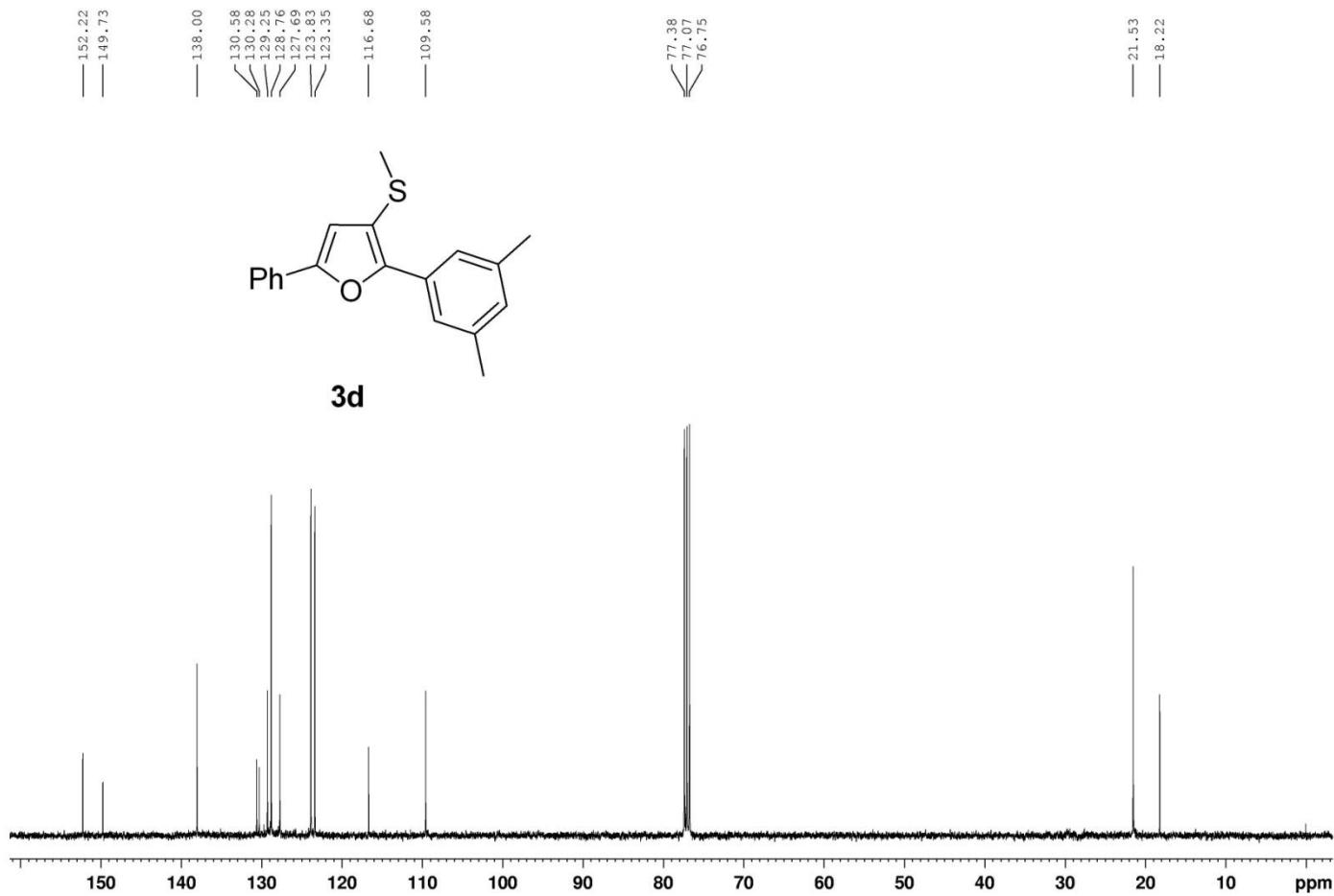


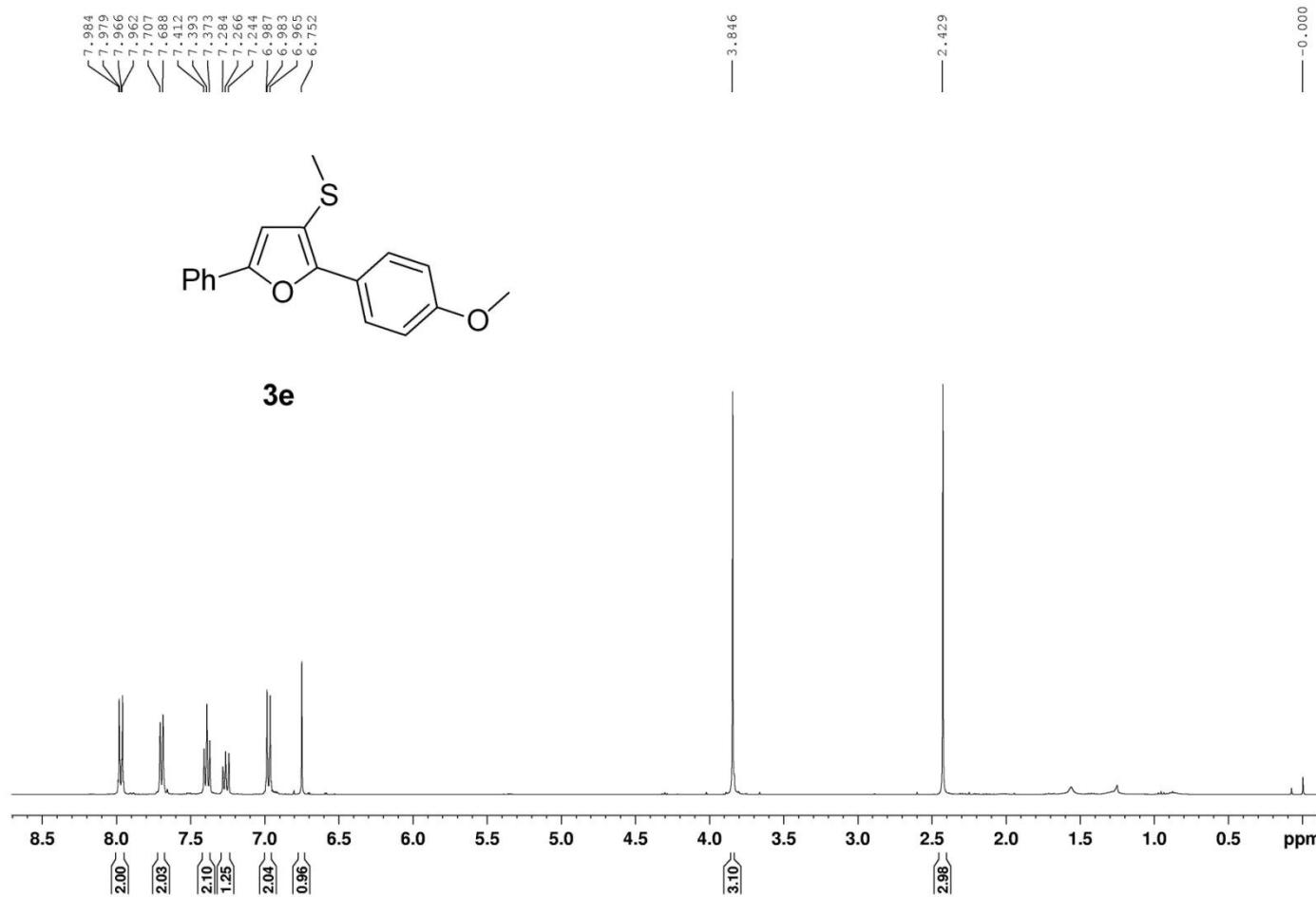


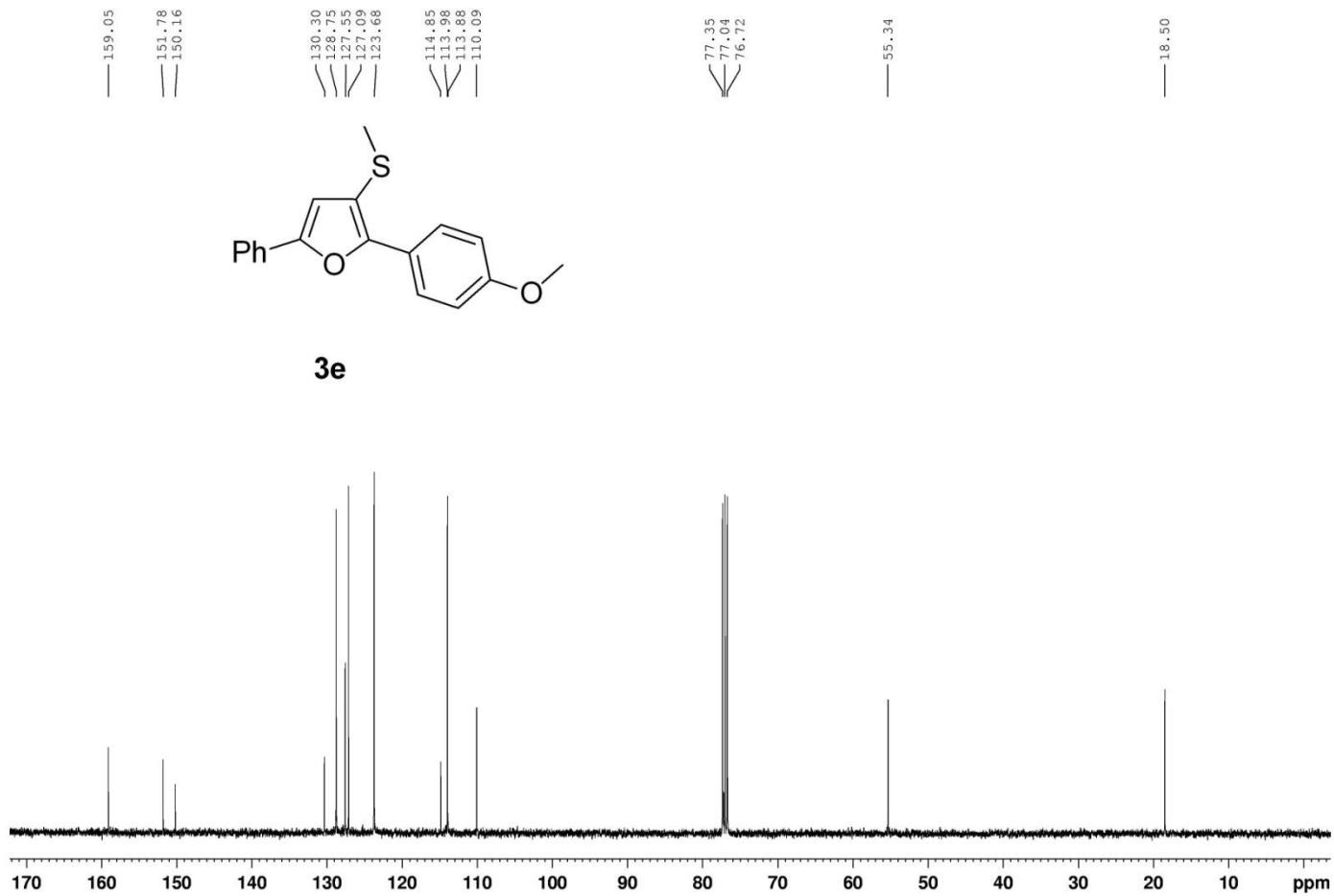


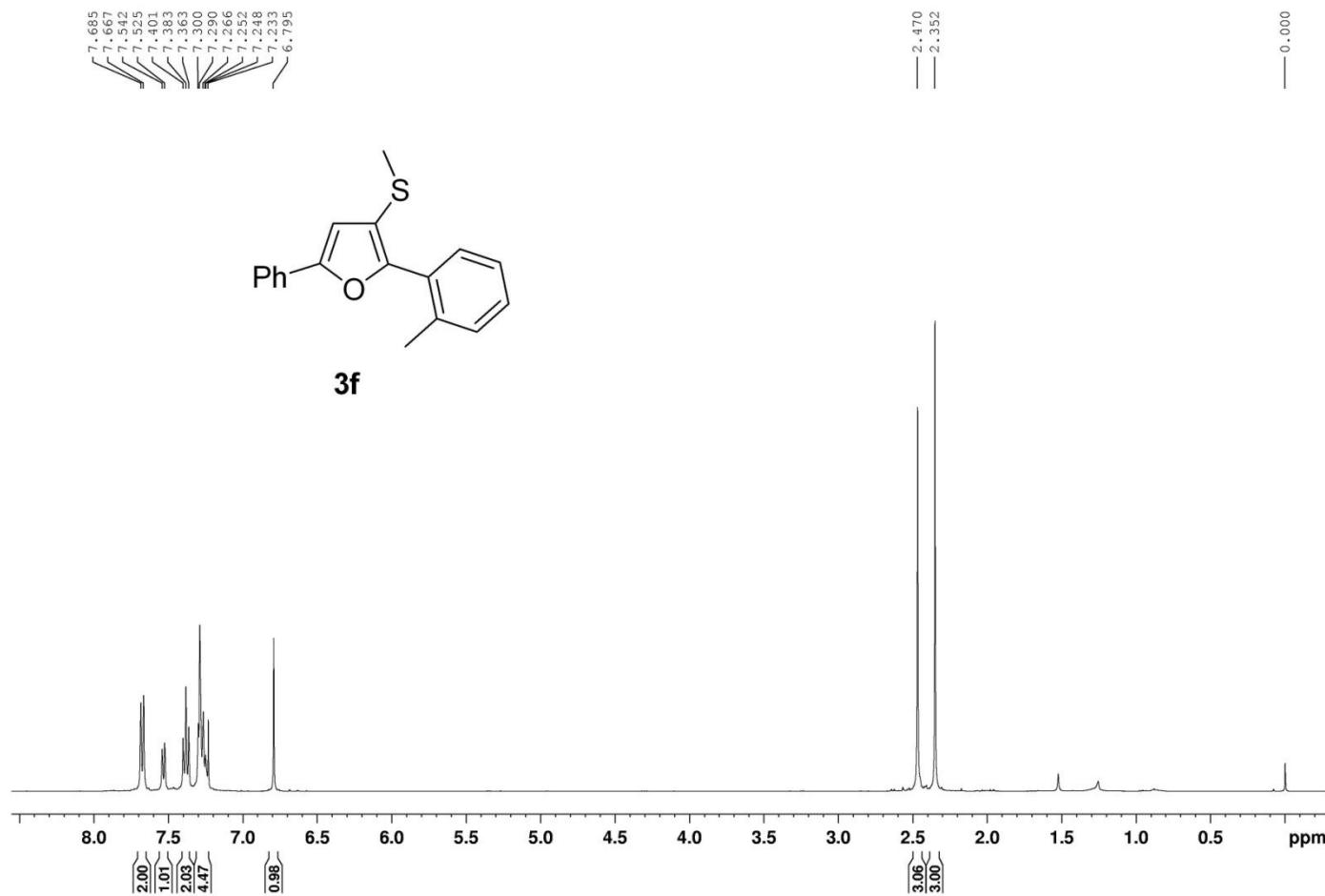


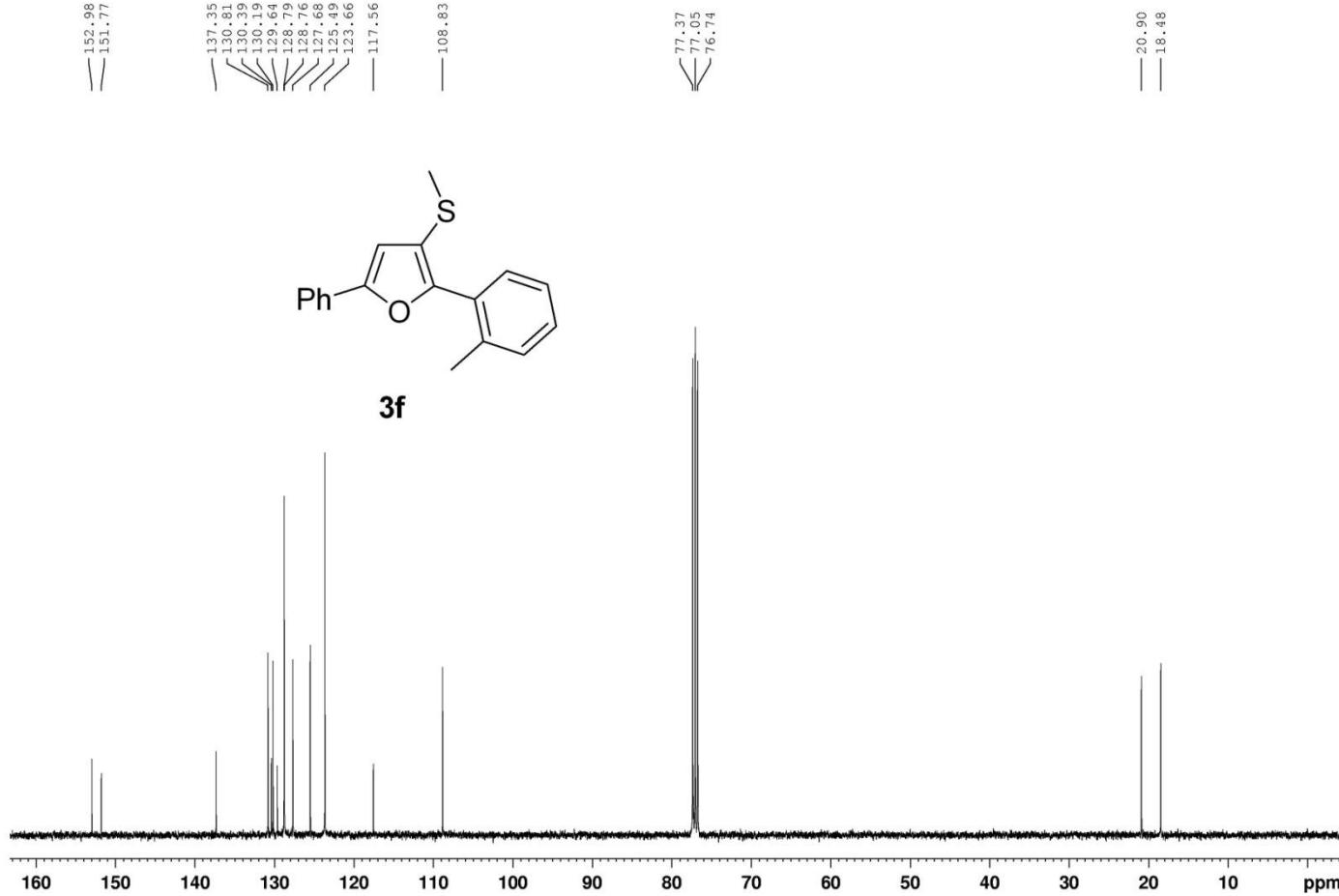


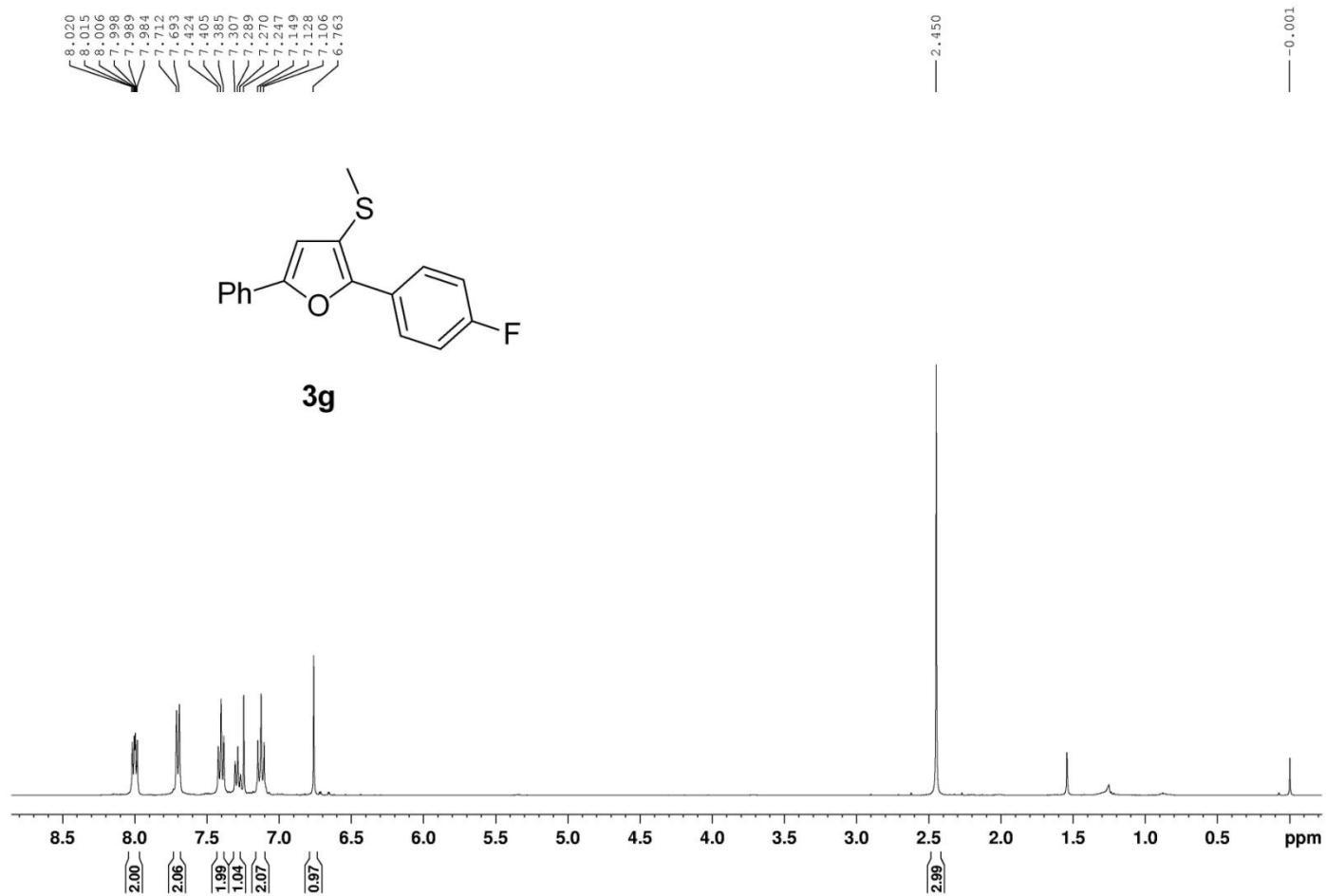


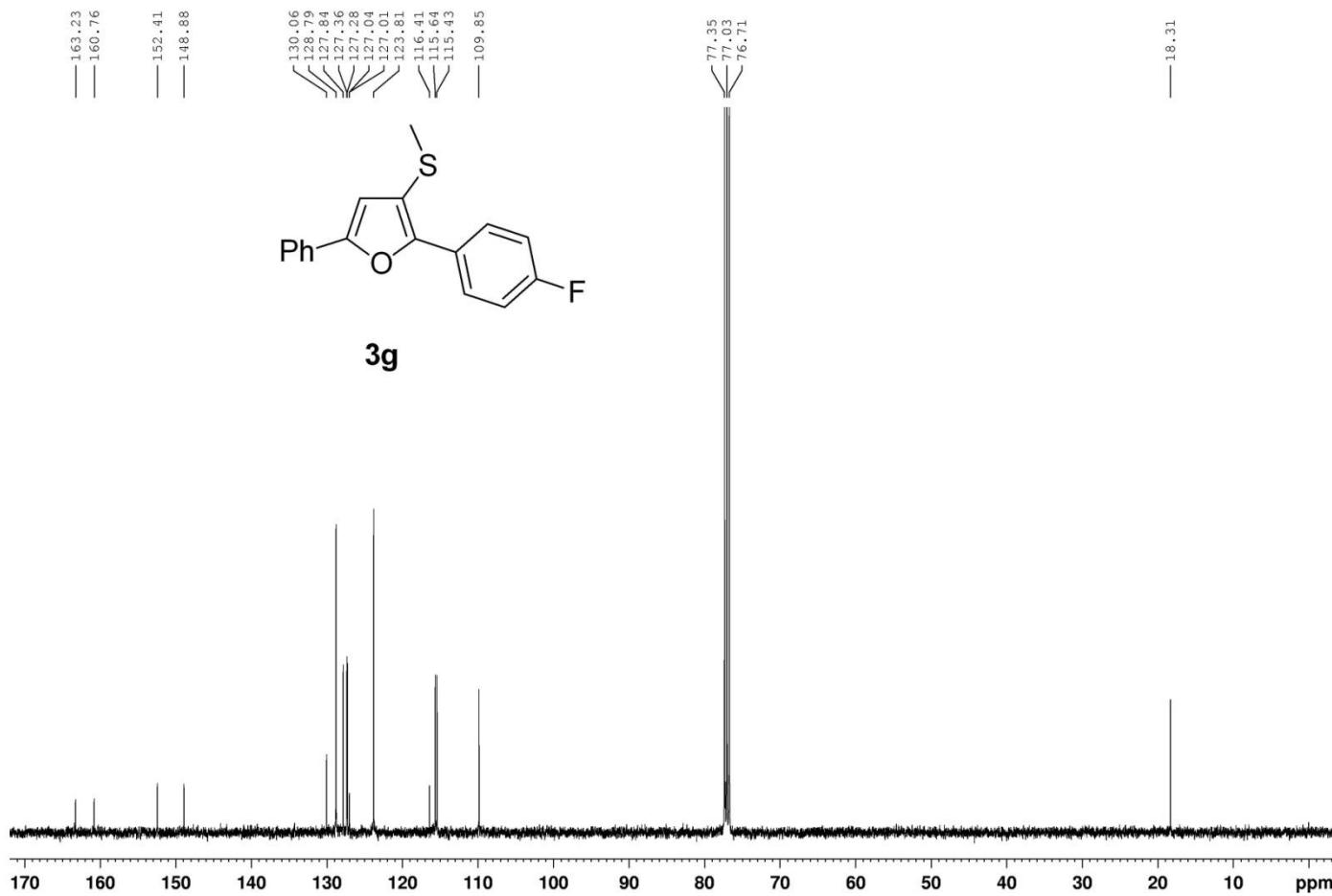


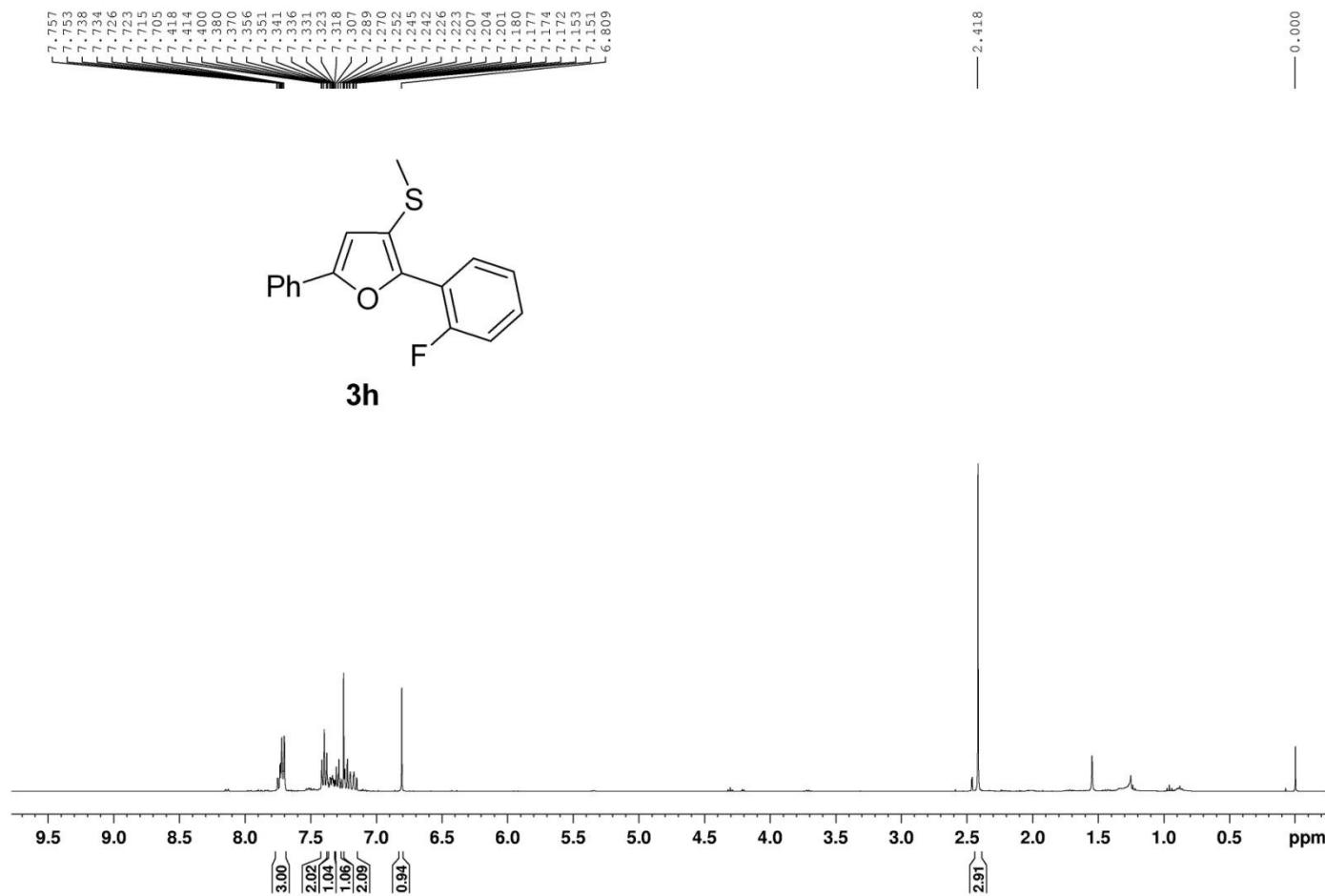


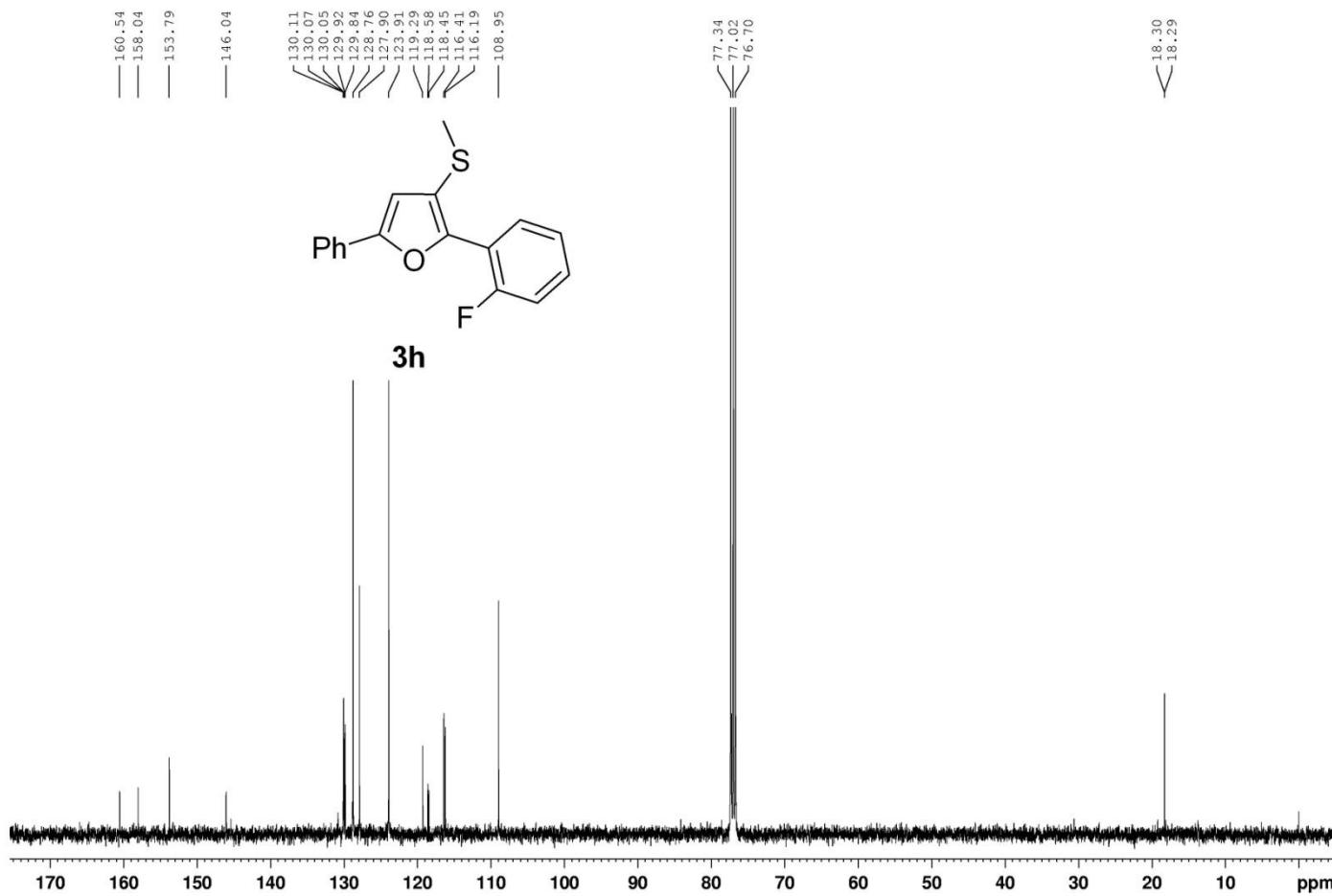


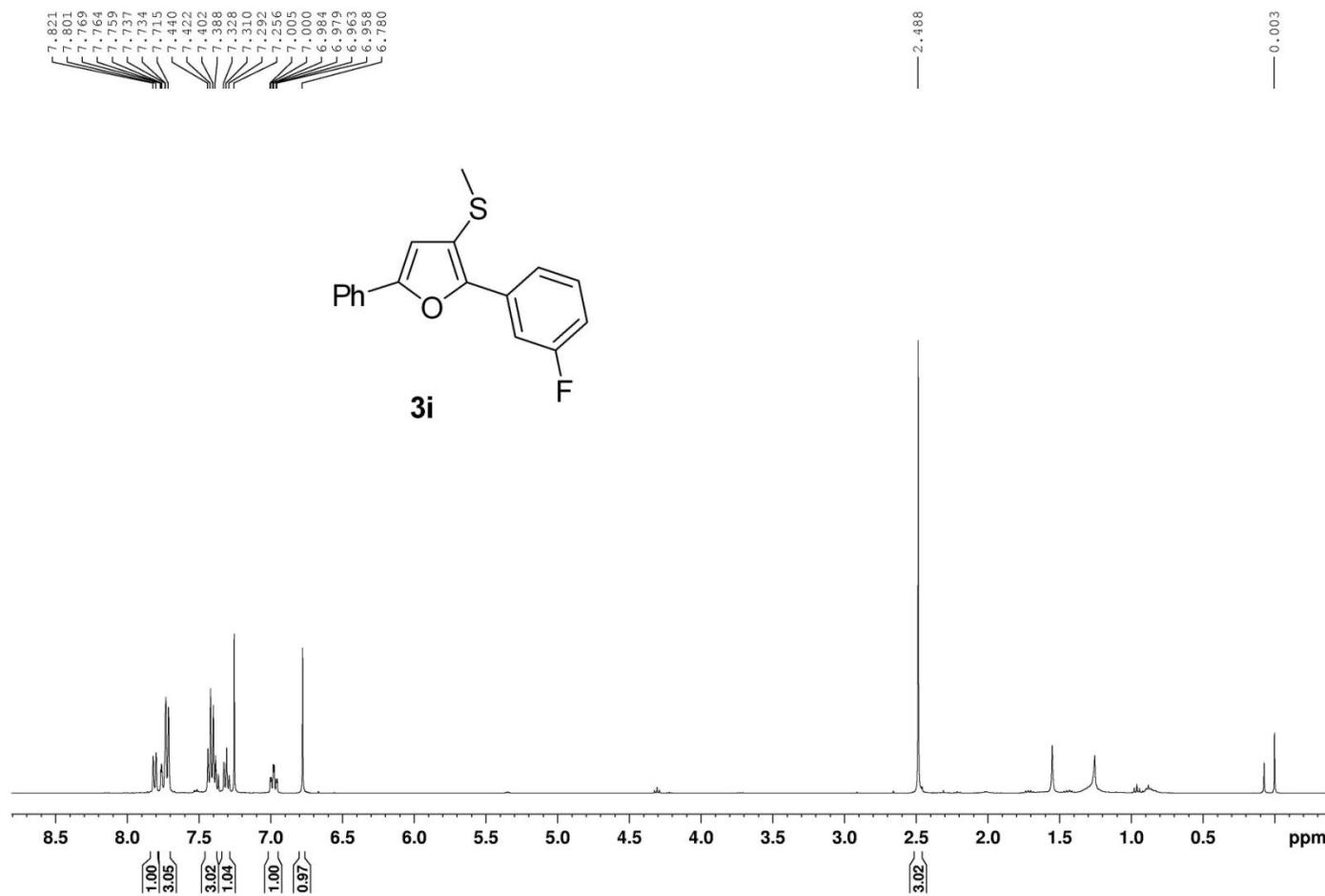


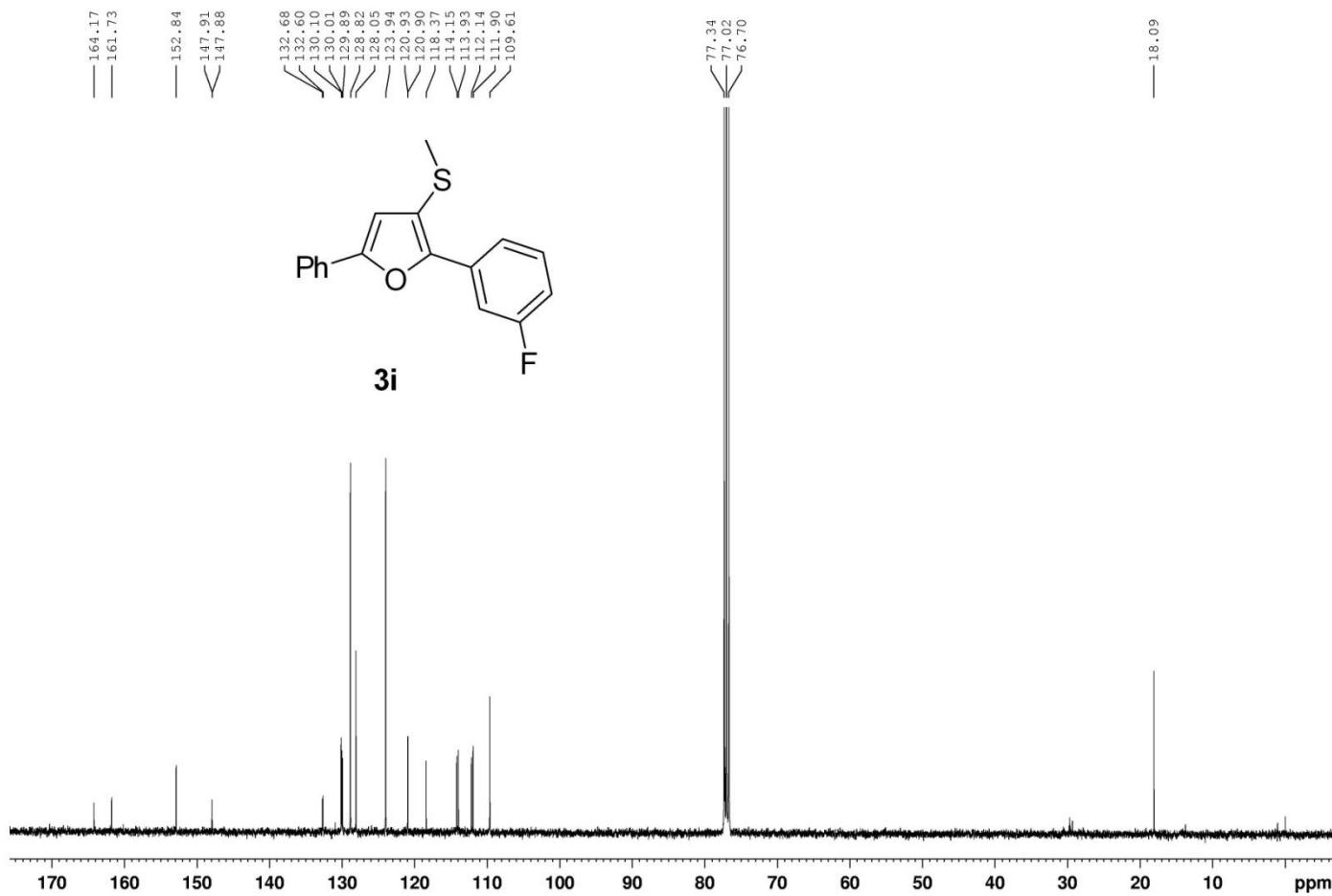


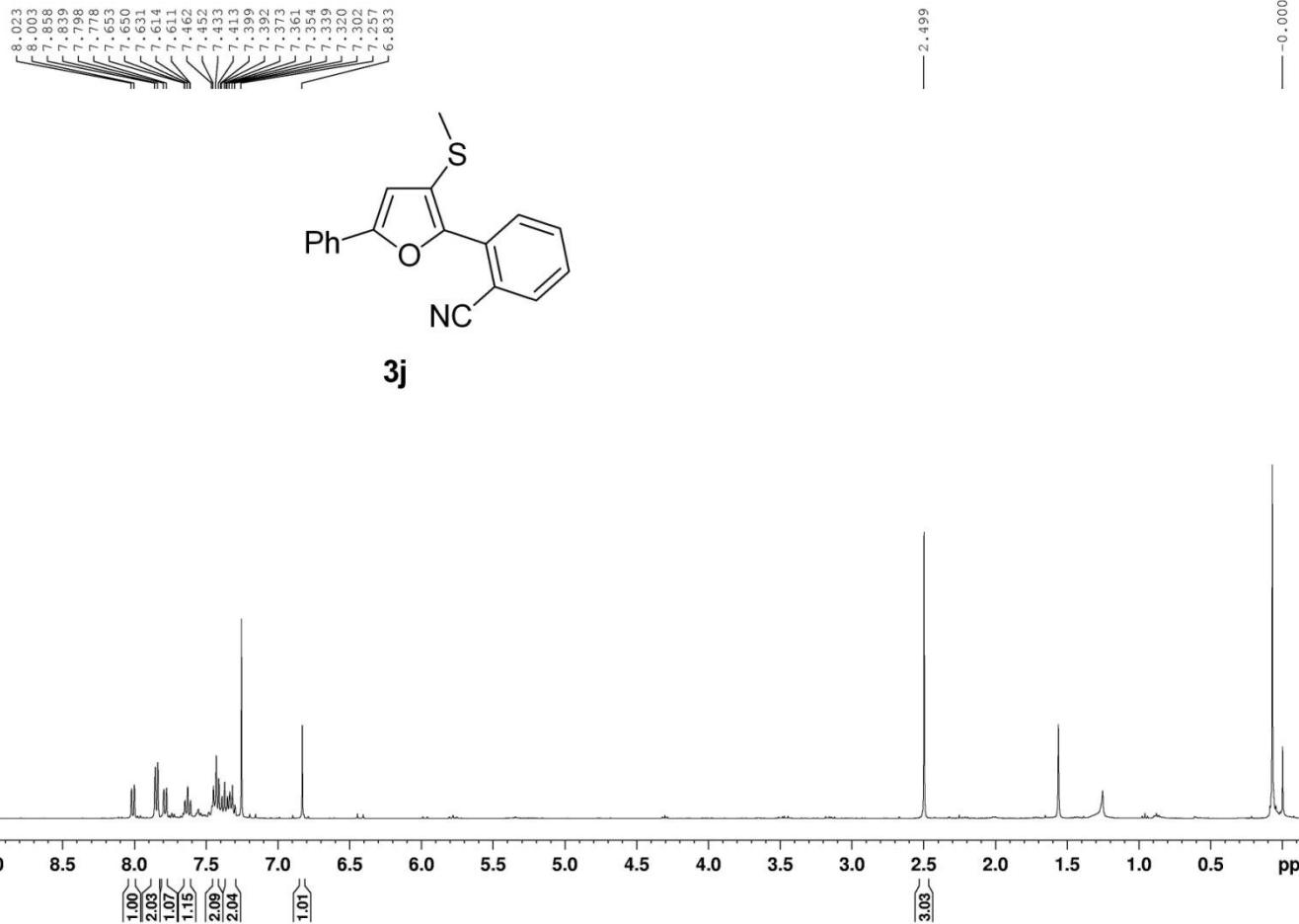


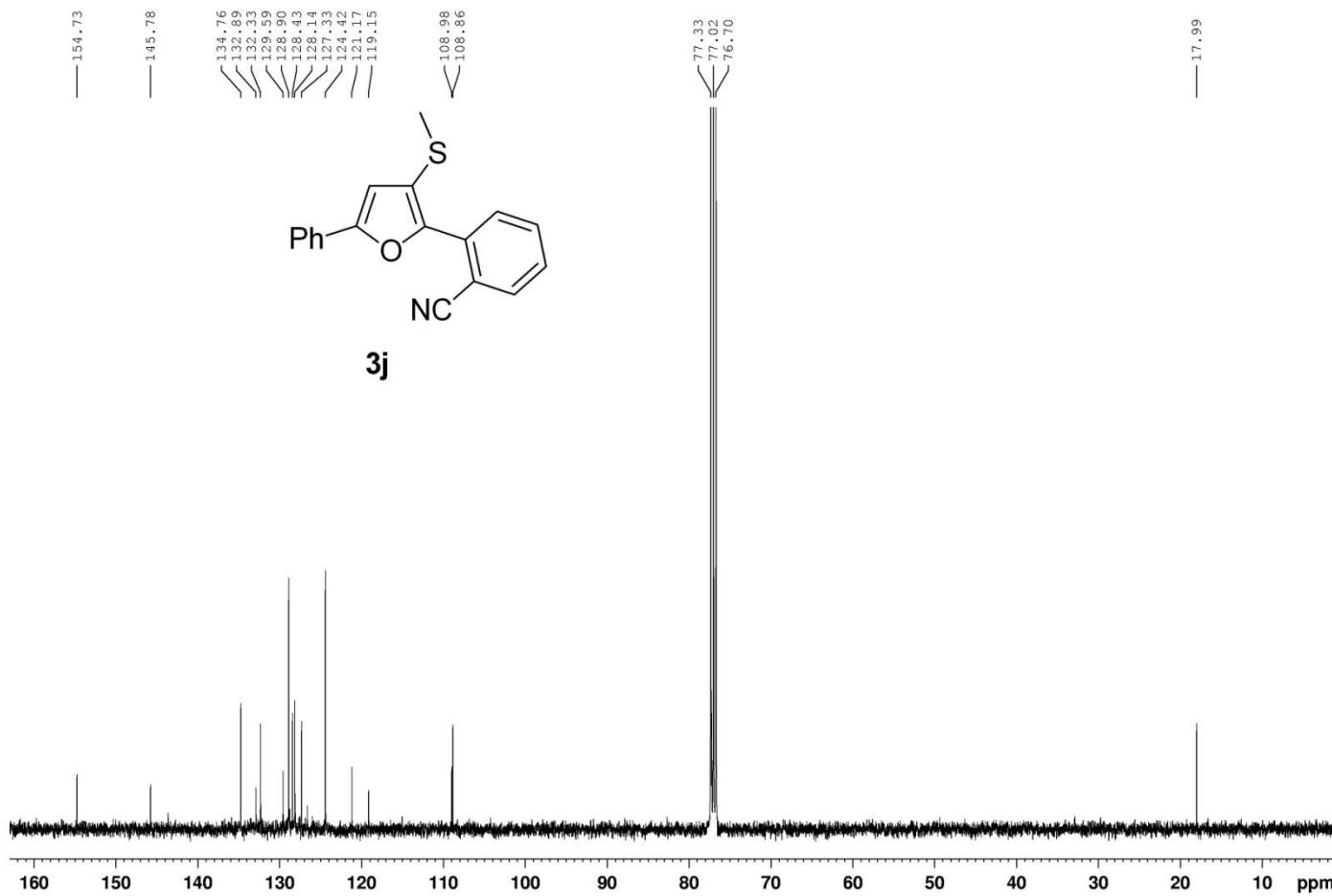


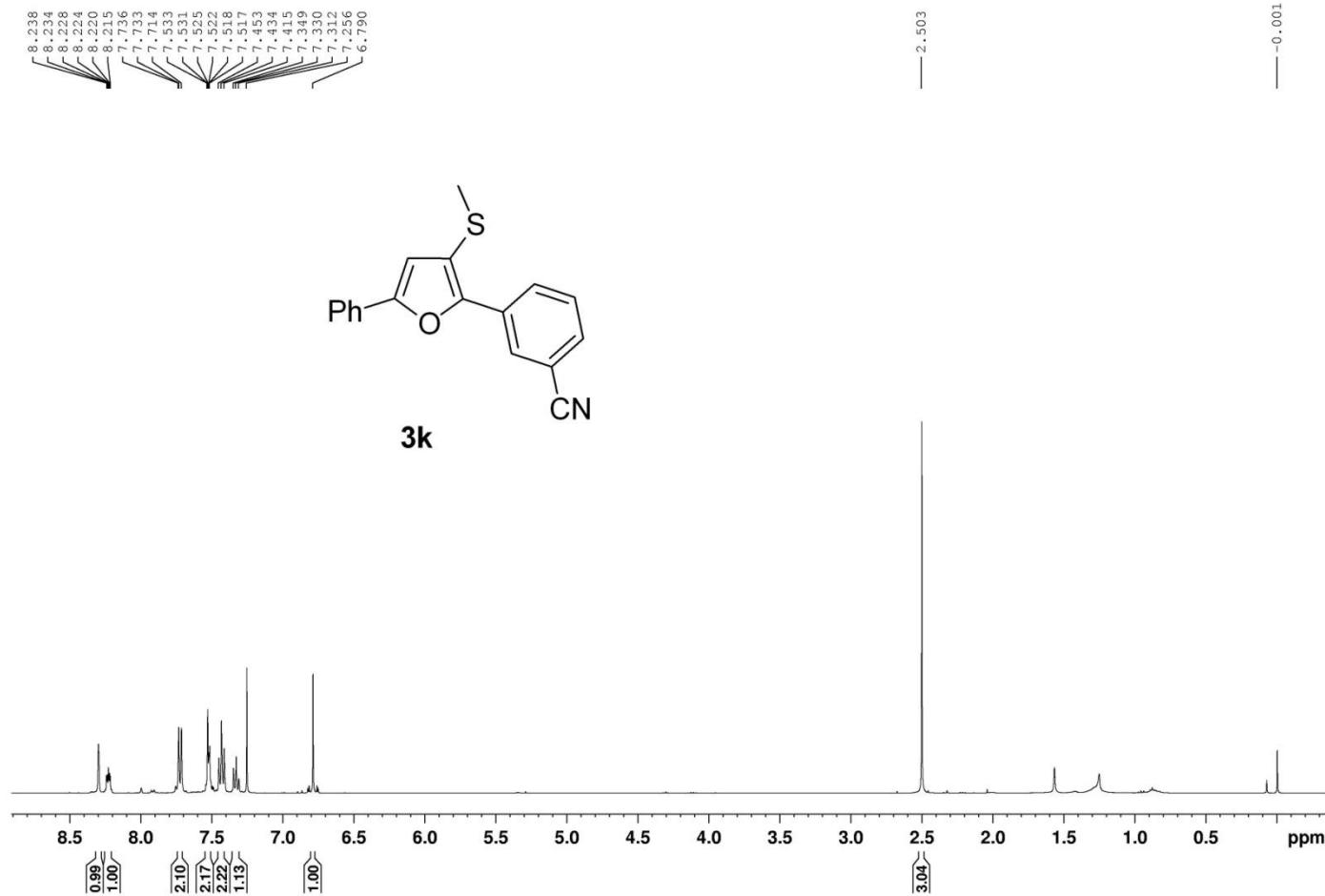


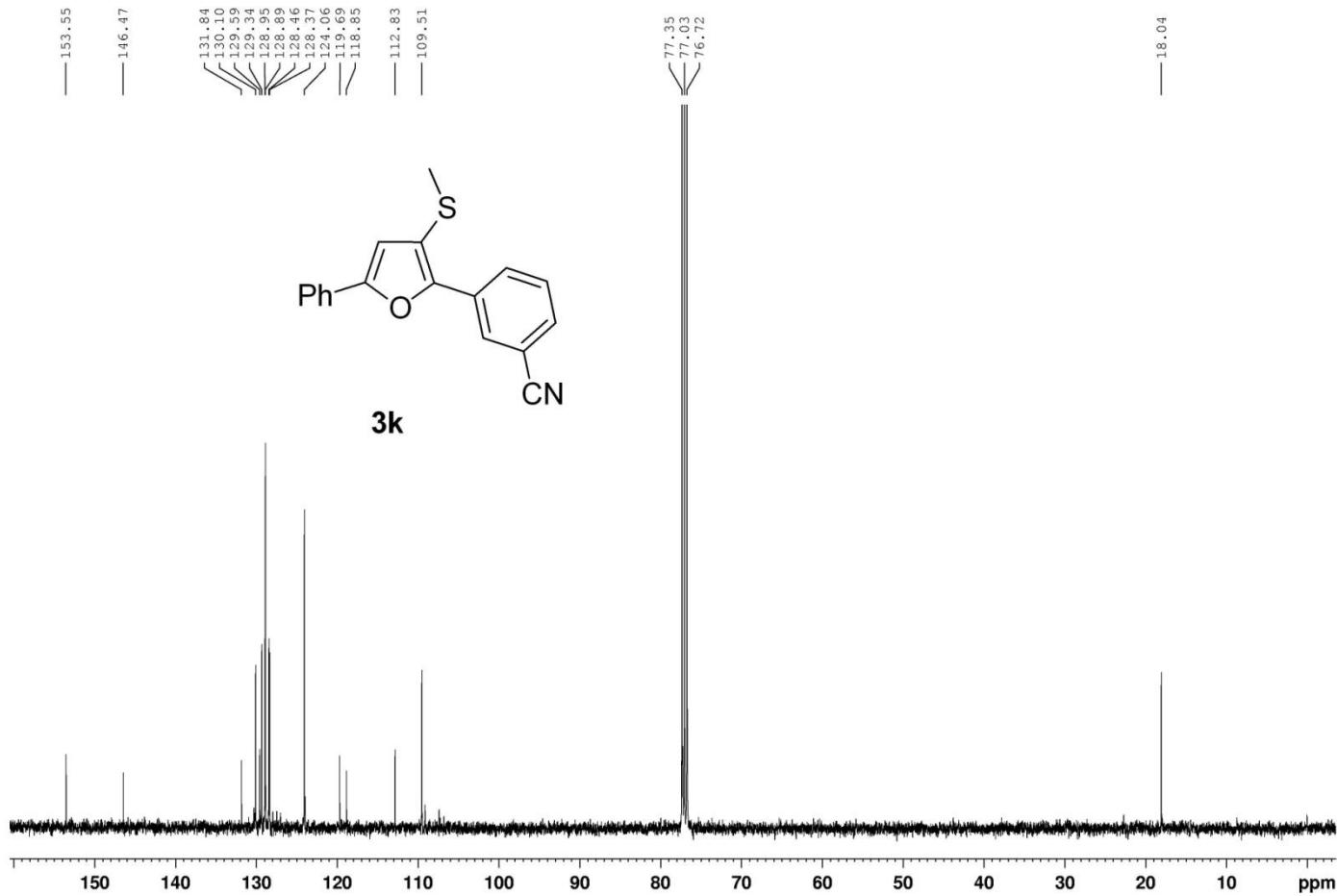


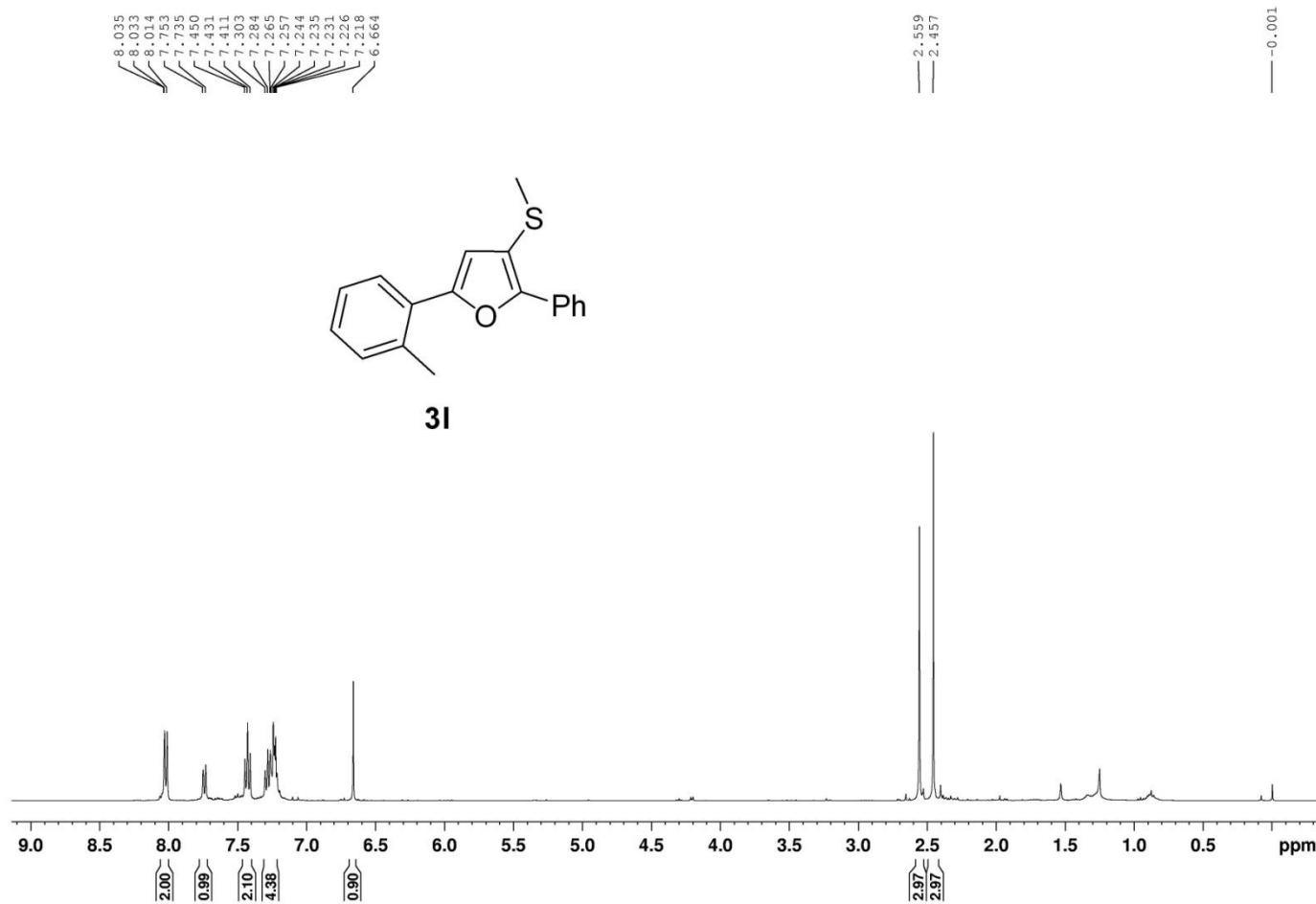


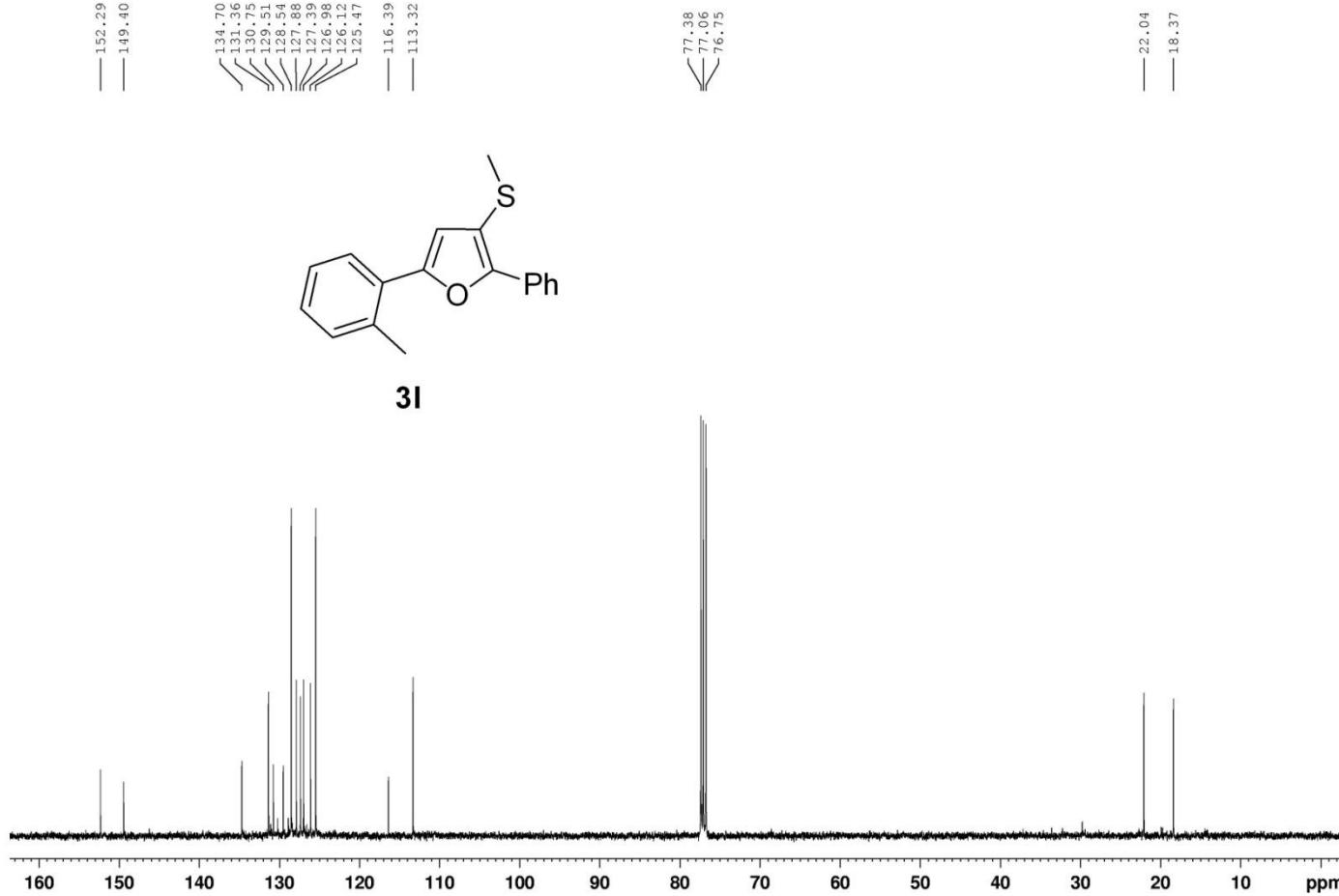


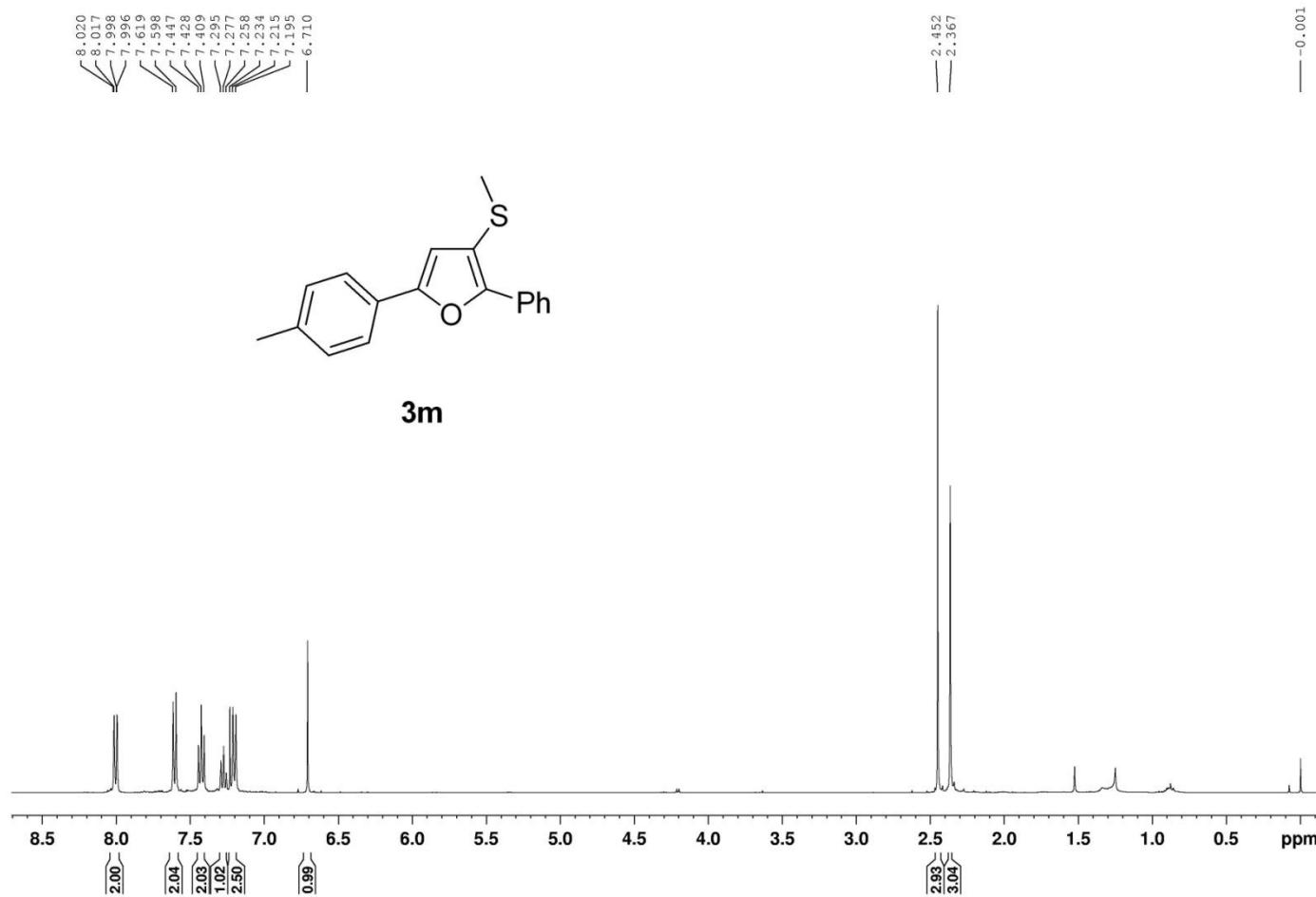


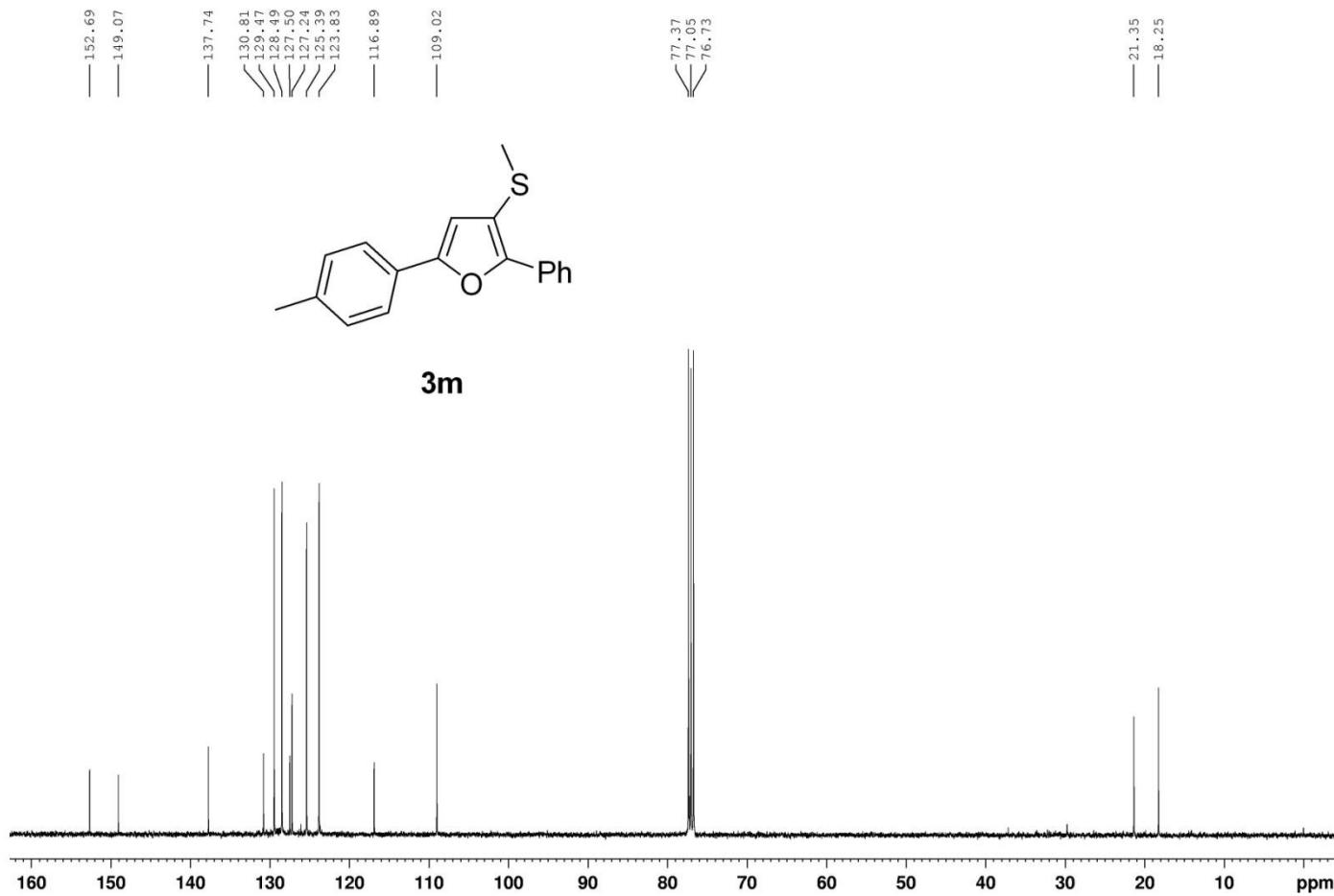


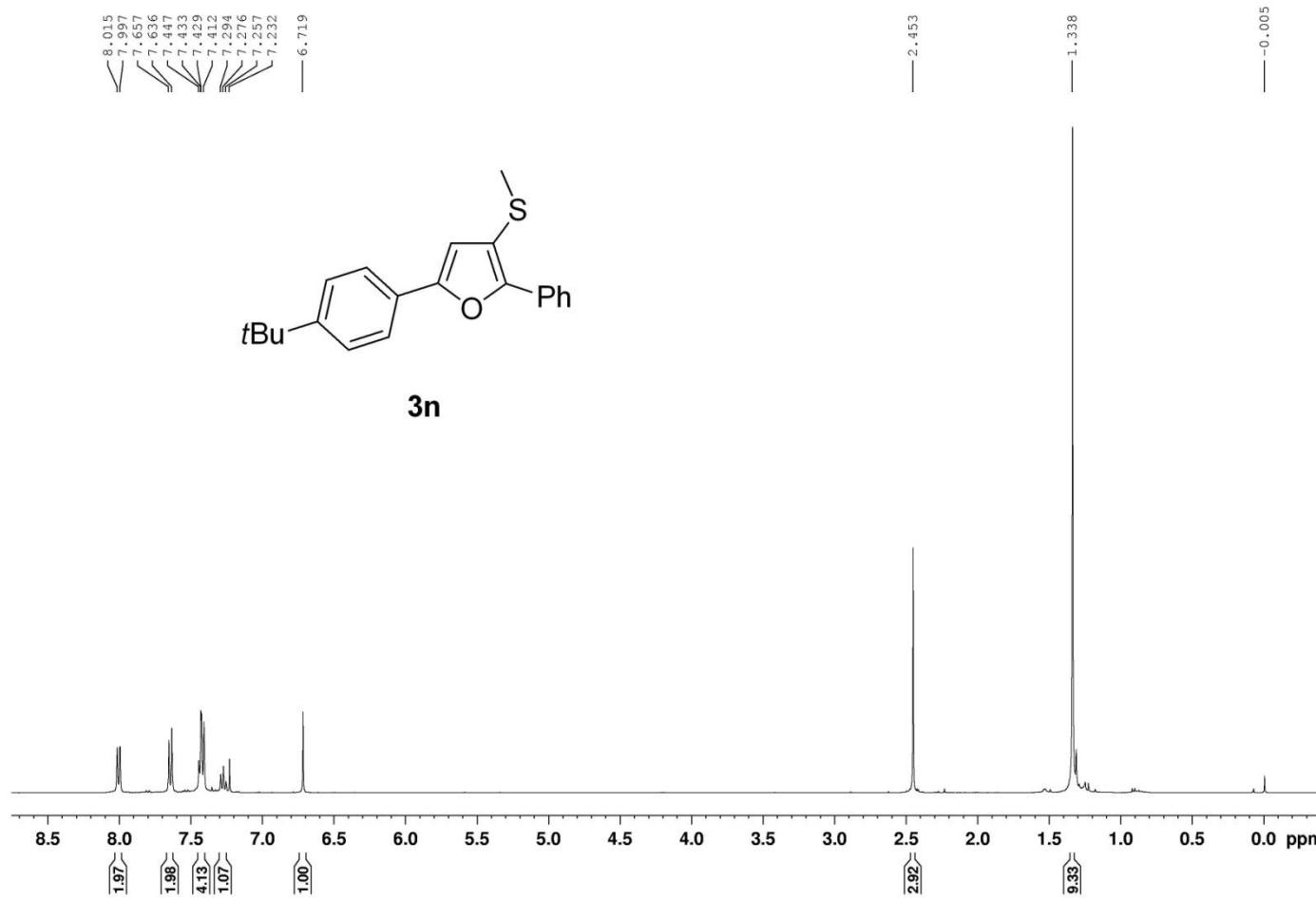


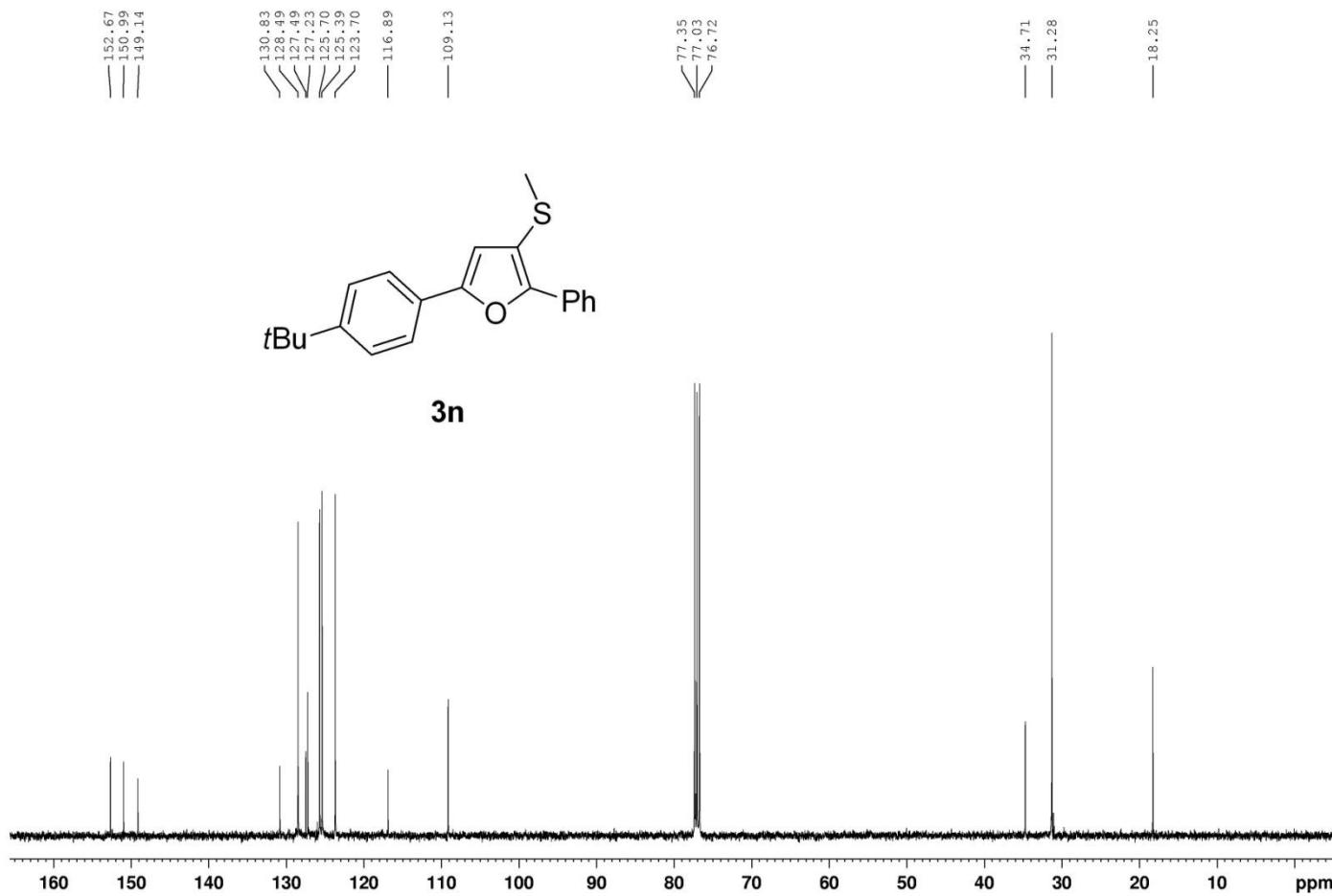










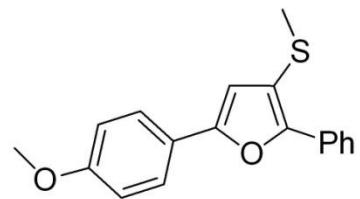


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7.644
7.450
7.431
7.411
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7.254
6.956
6.939
6.934
6.644

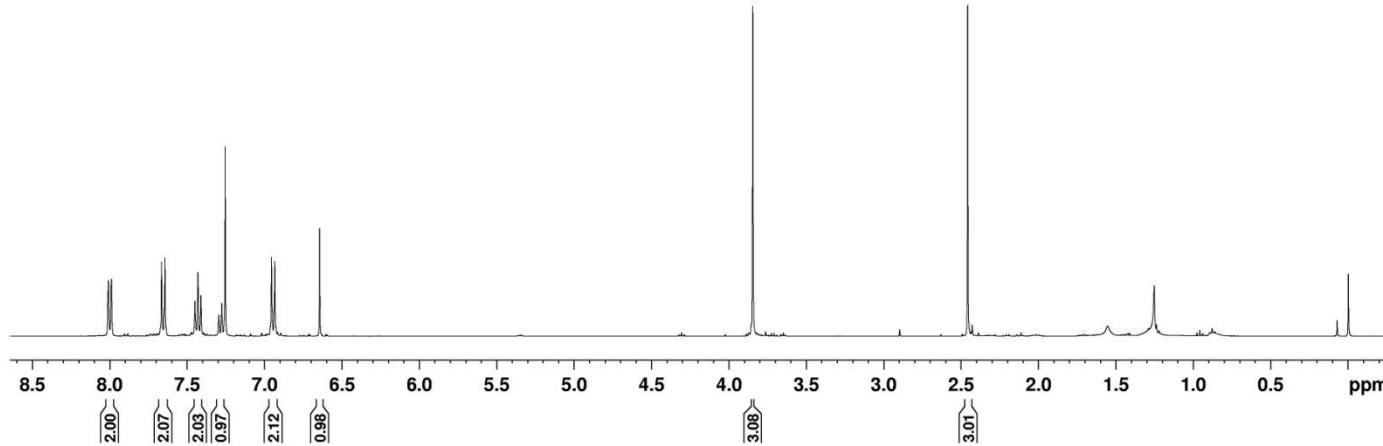
— 3.846

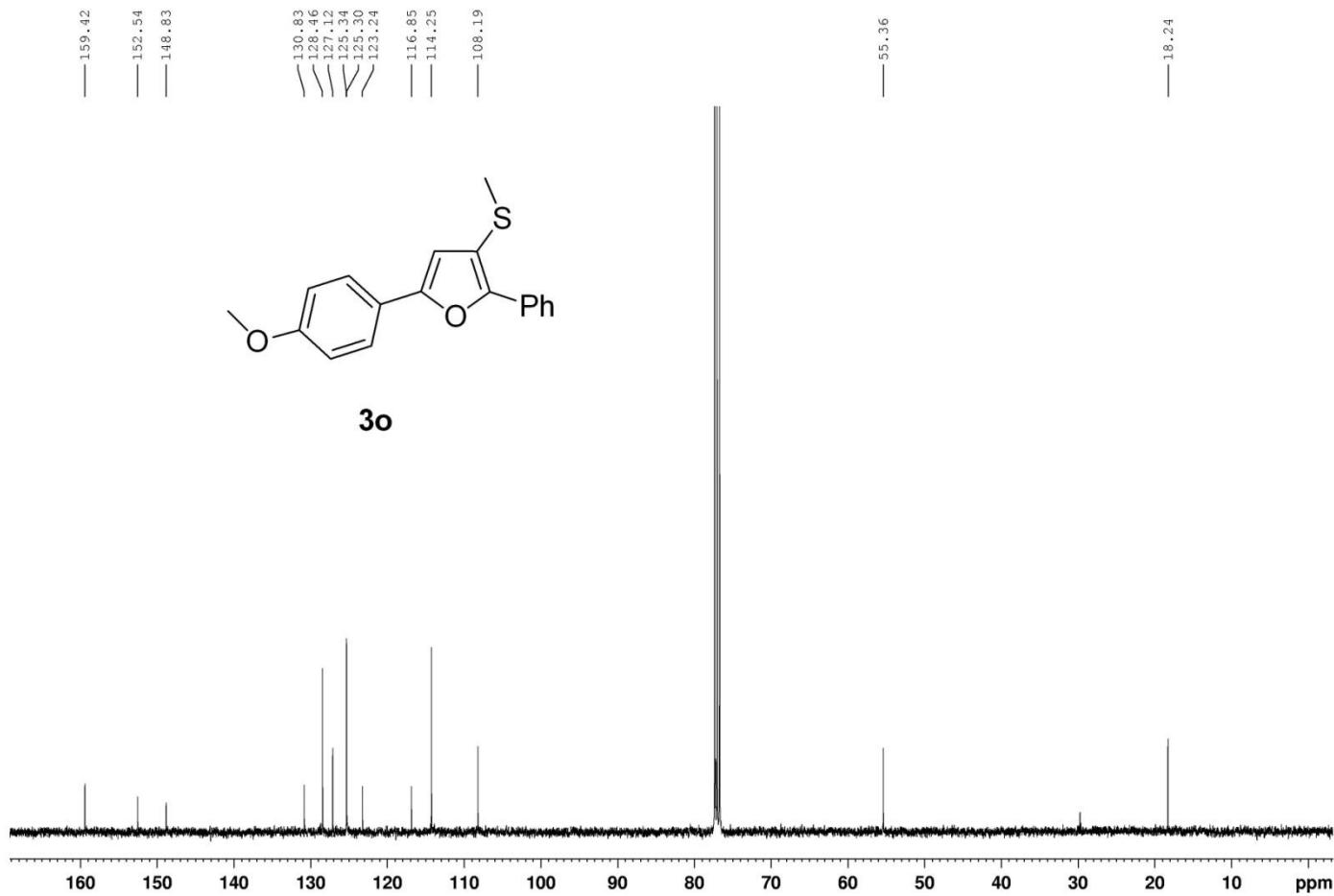
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— 0.001



3o



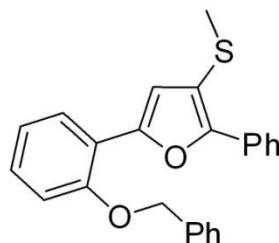


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7.241
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7.224
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7.062
7.035
7.015

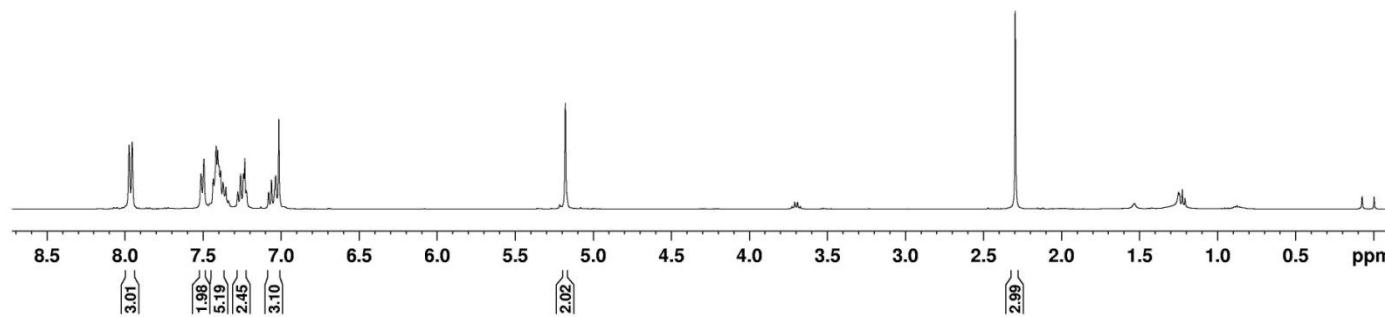
— 5.181

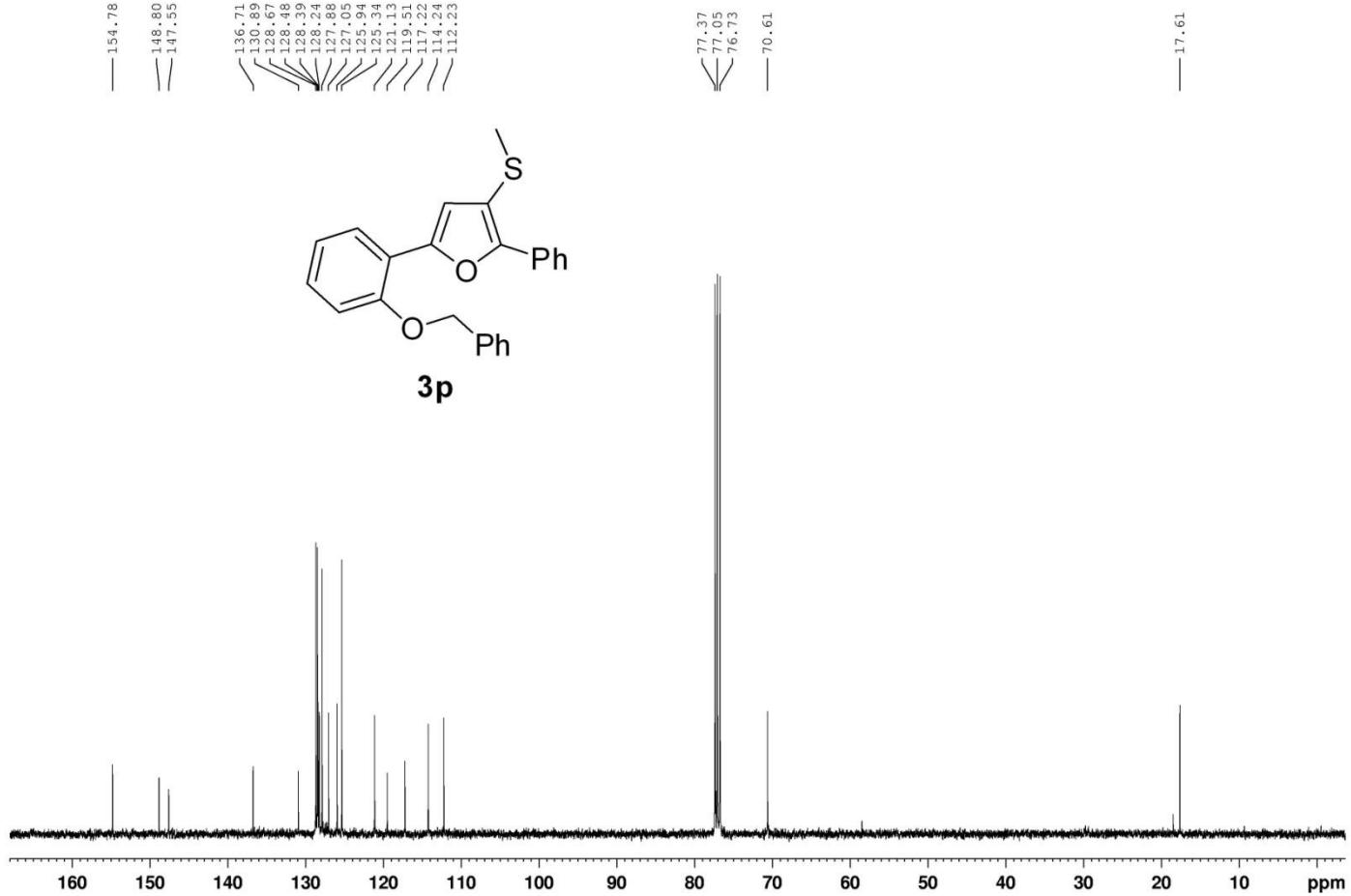
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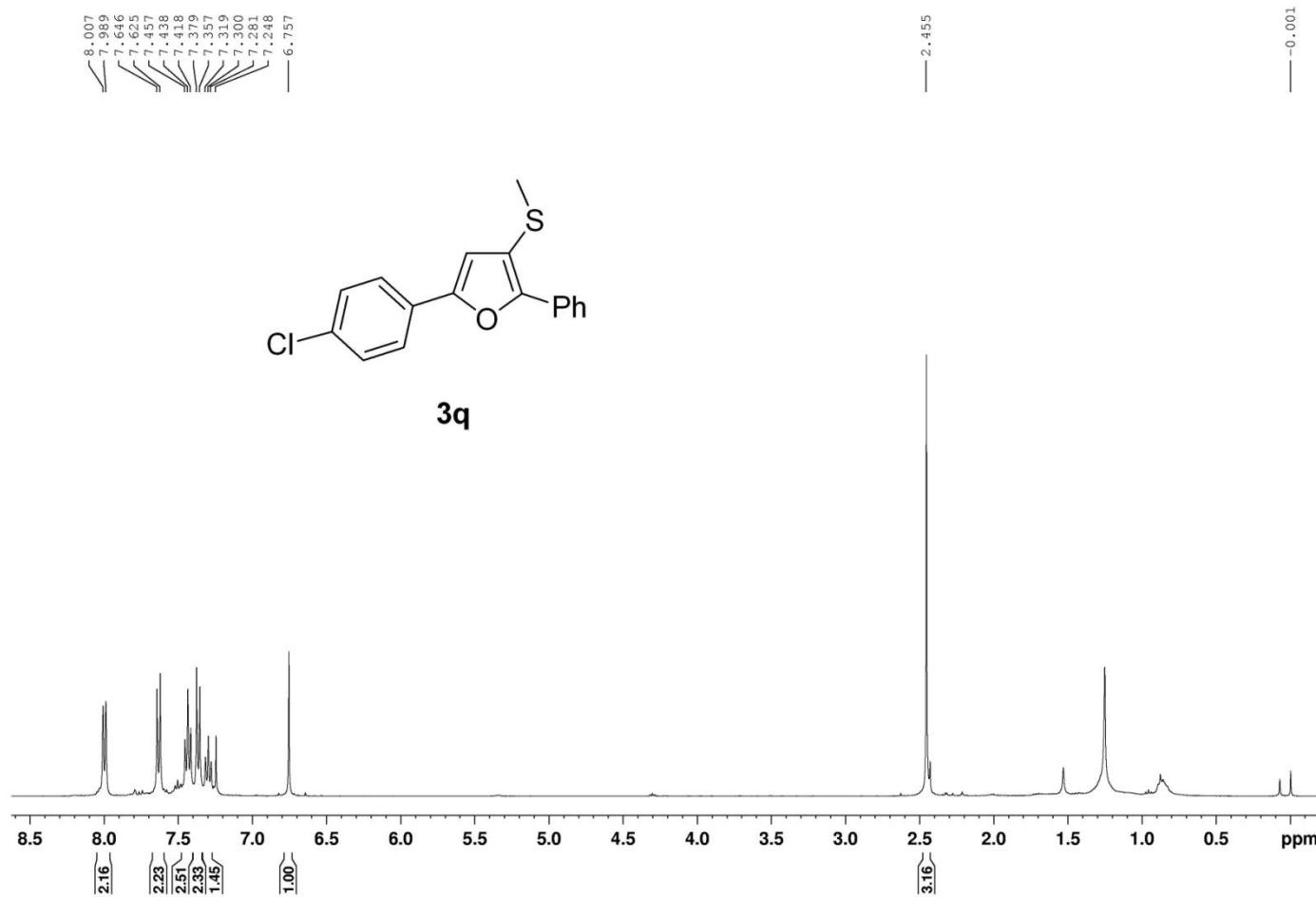
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3p







— 151.33
— 149.73

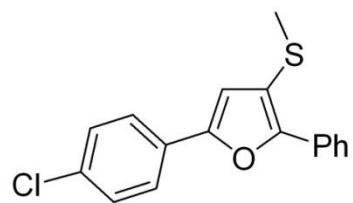
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128.54
127.55
127.49
125.49
125.02

— 117.15

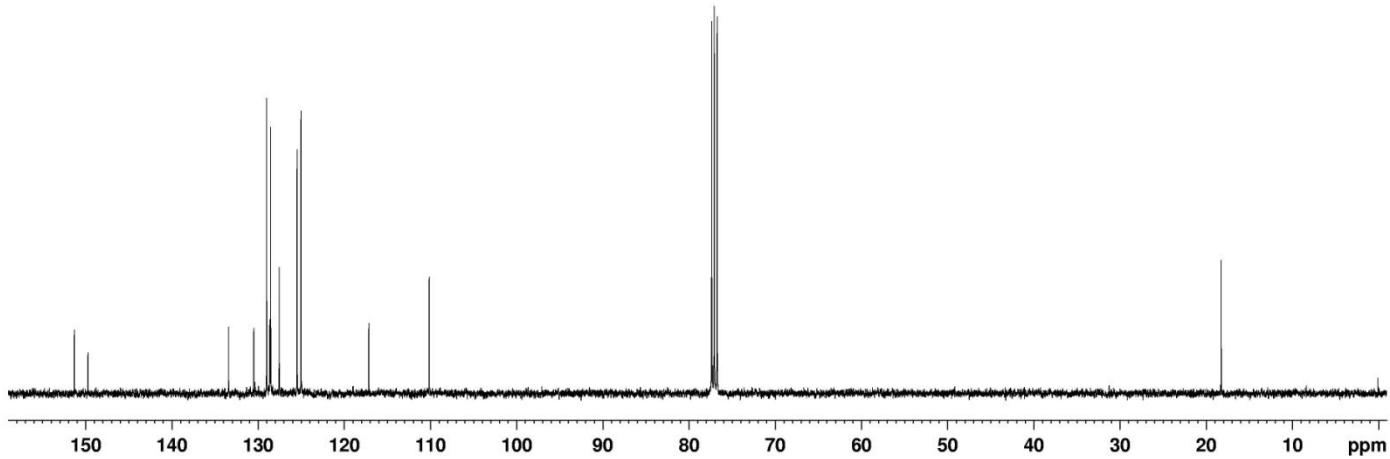
— 110.16

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77.05
76.73

— 18.23



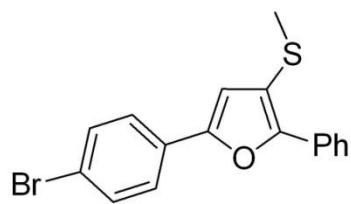
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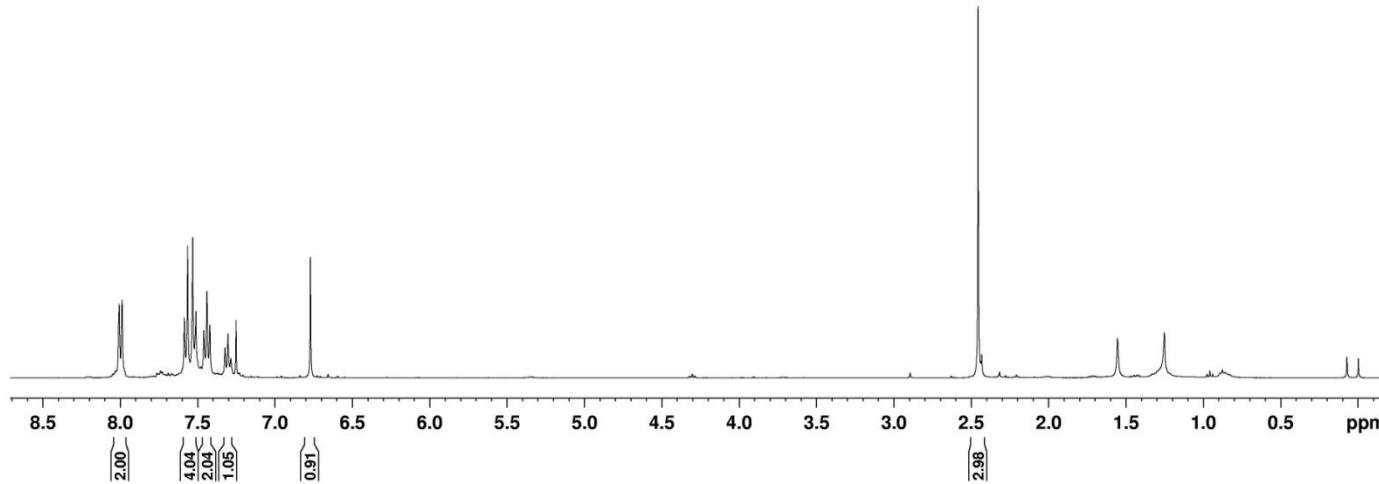
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6.773

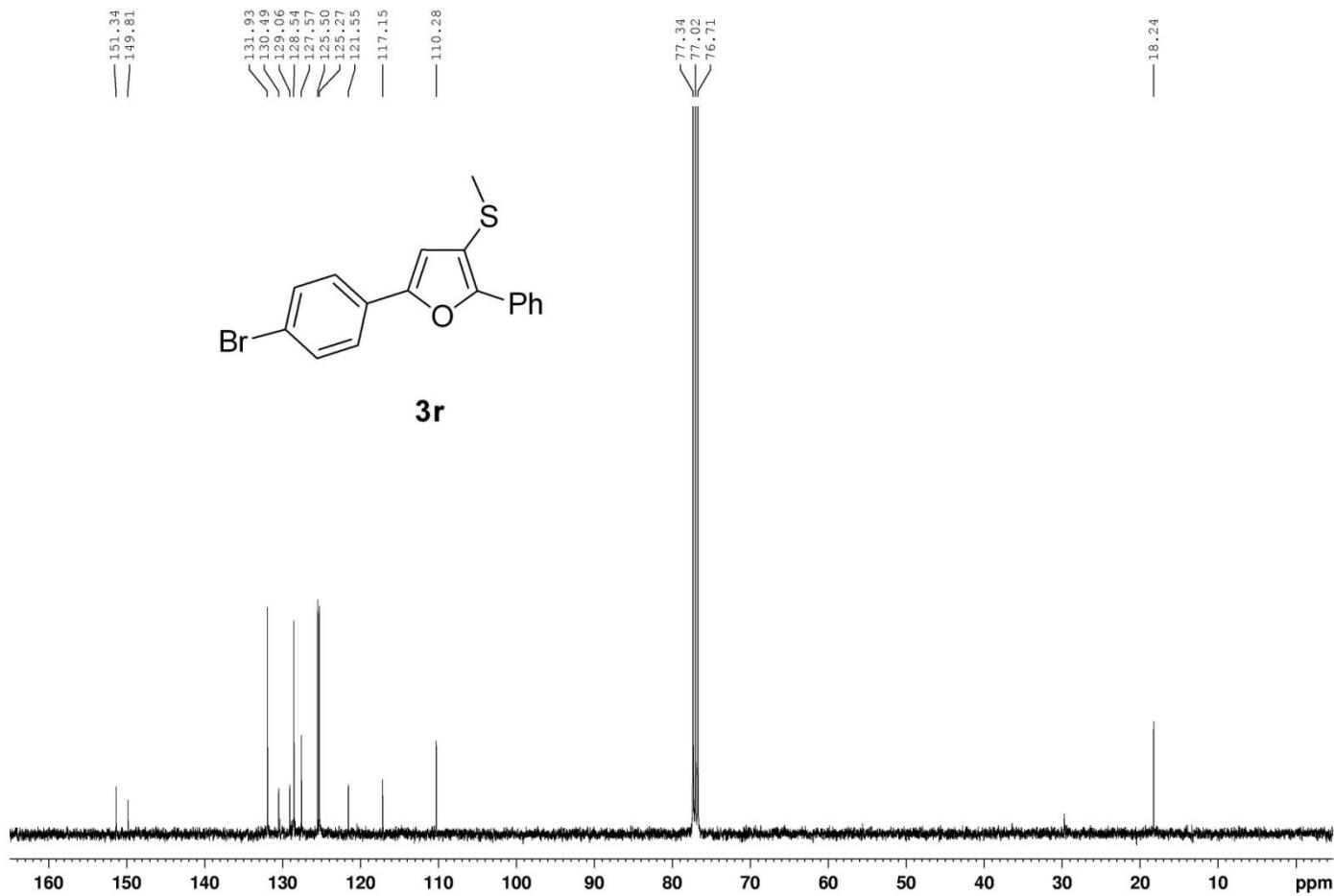
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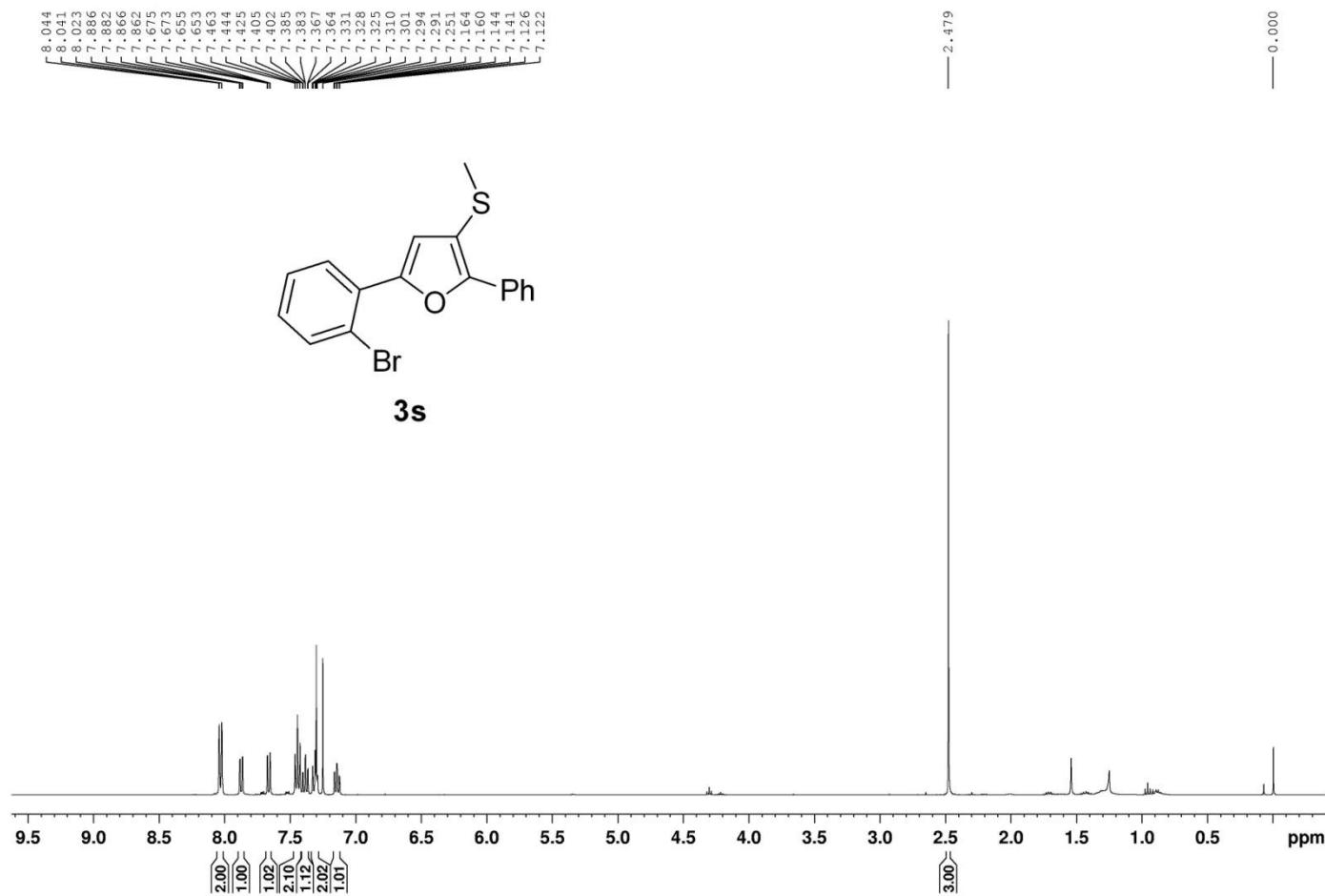
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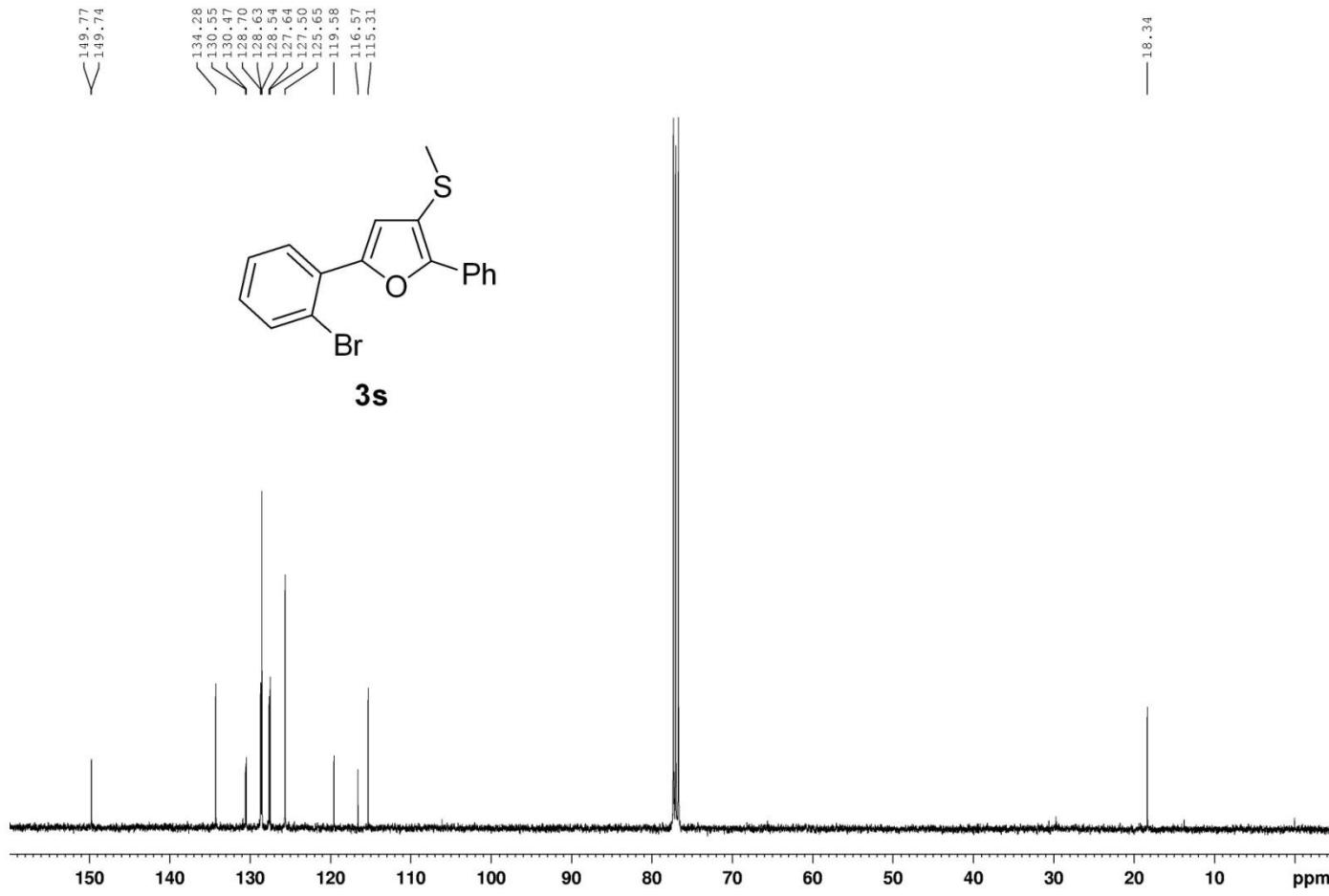


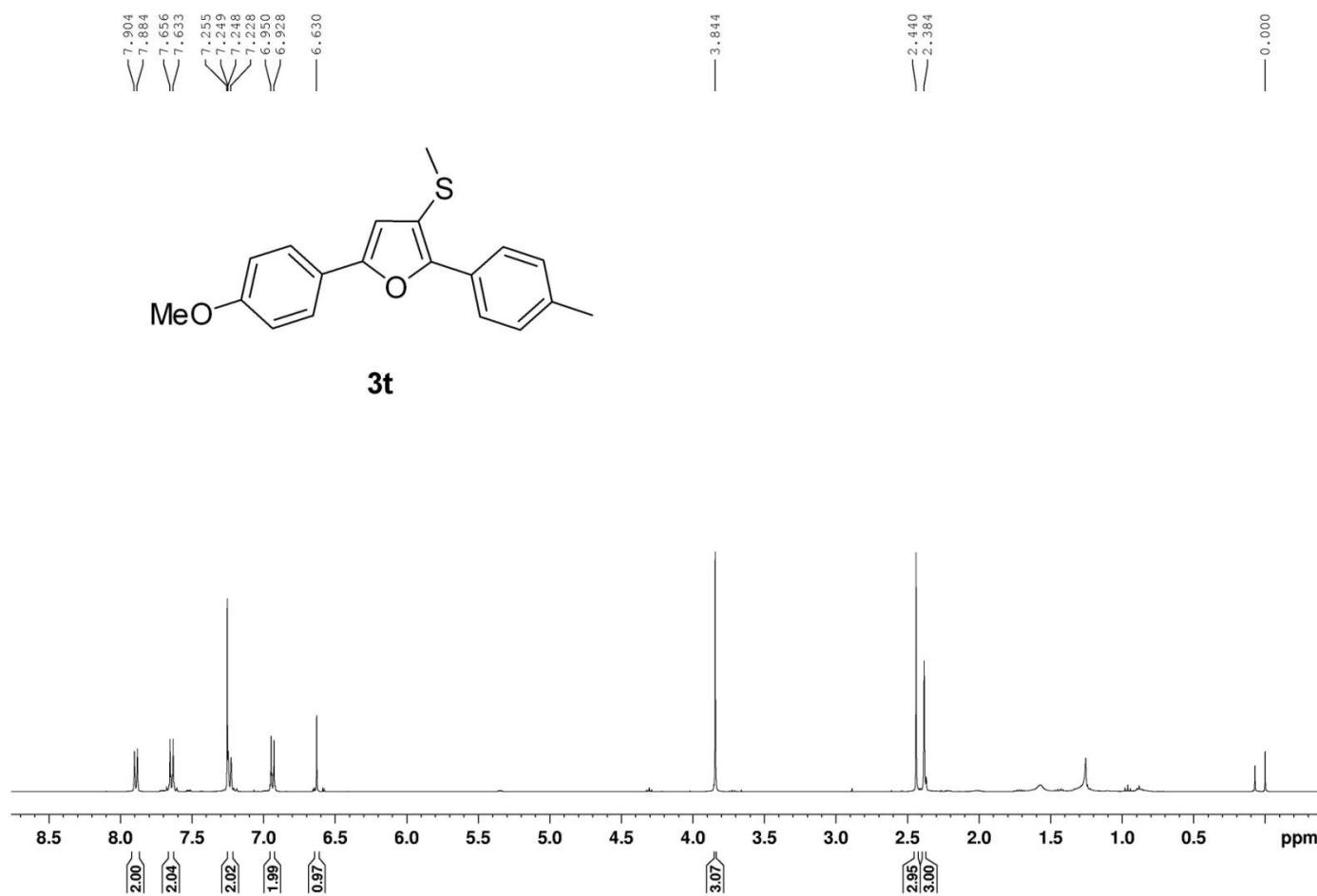
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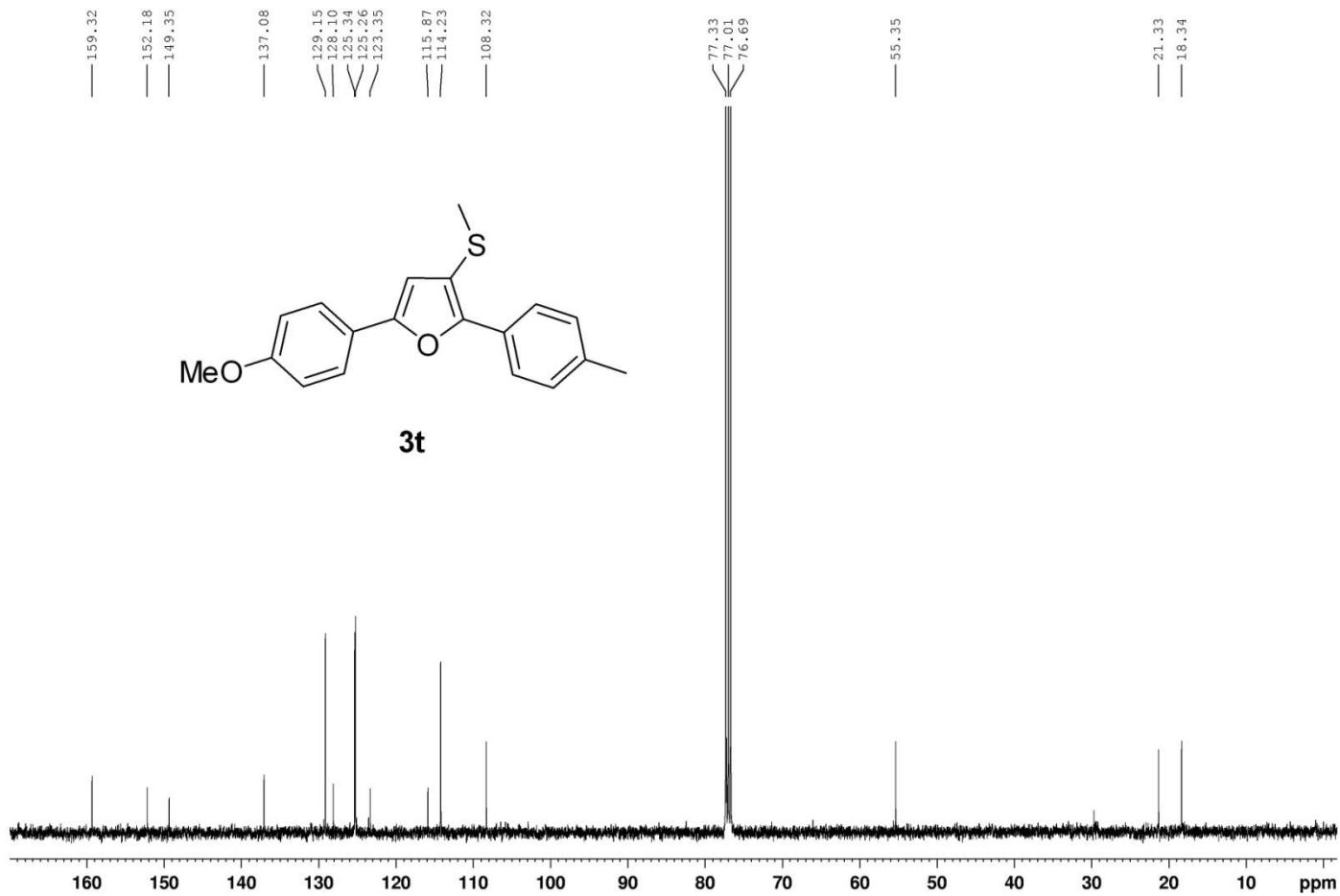


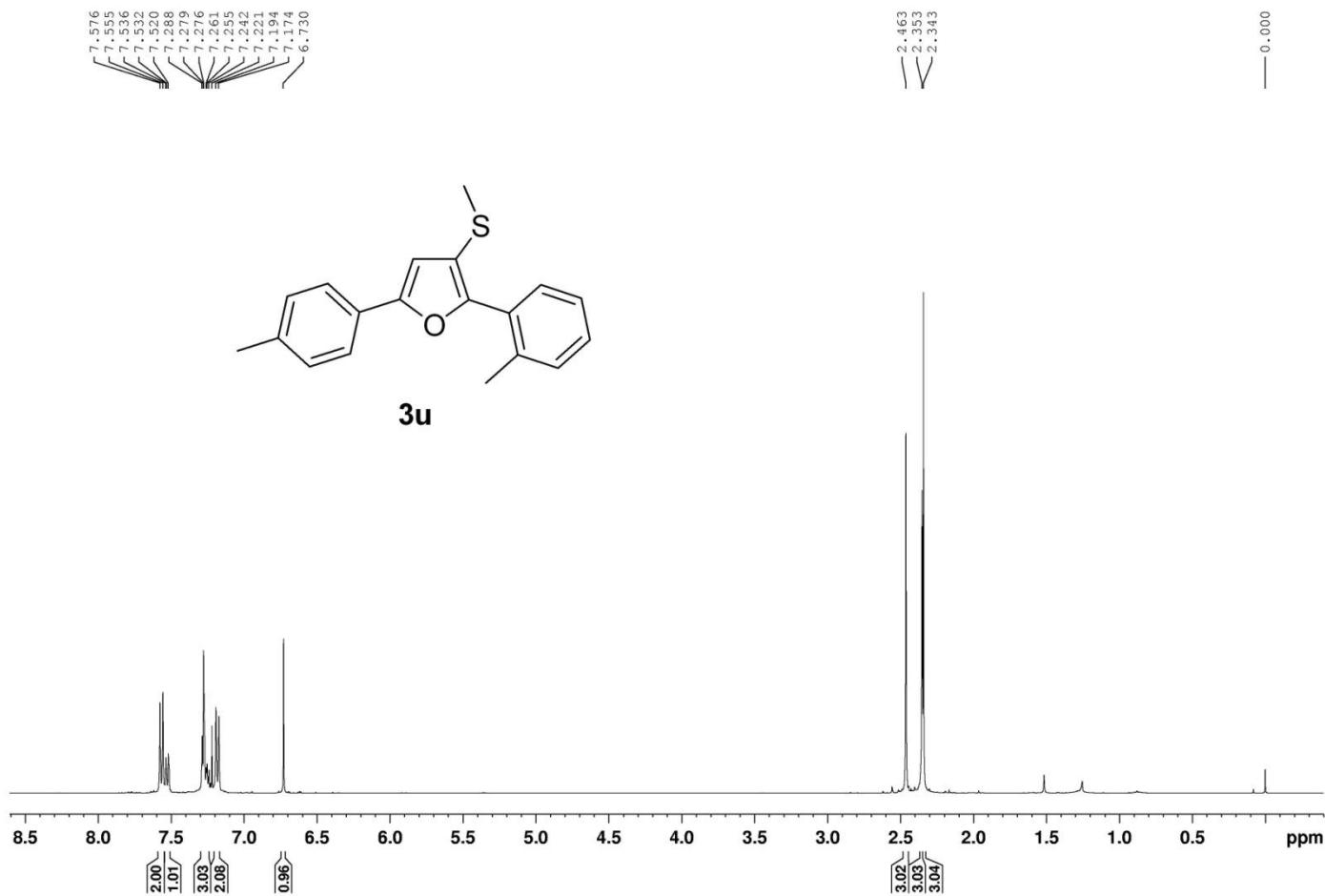


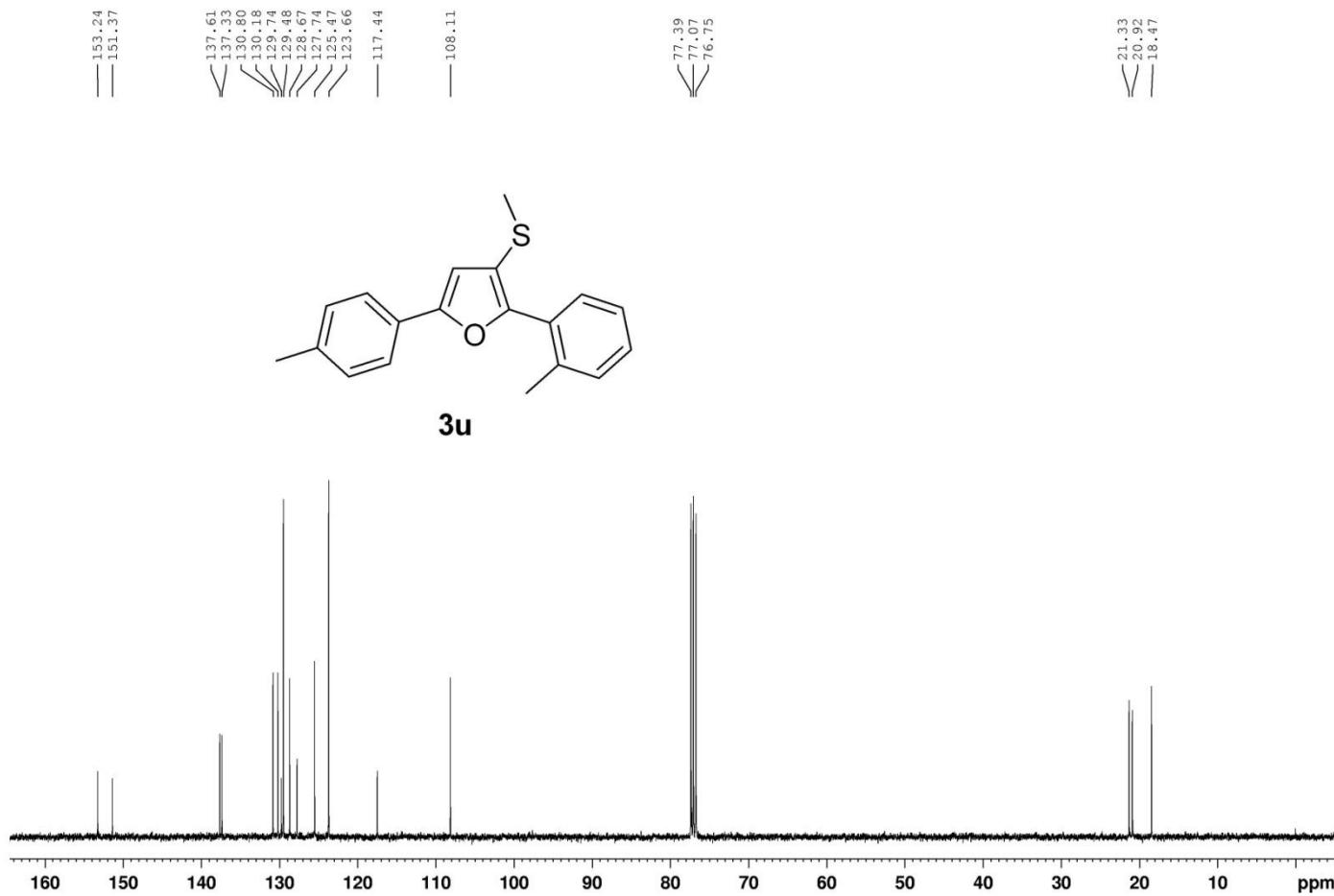


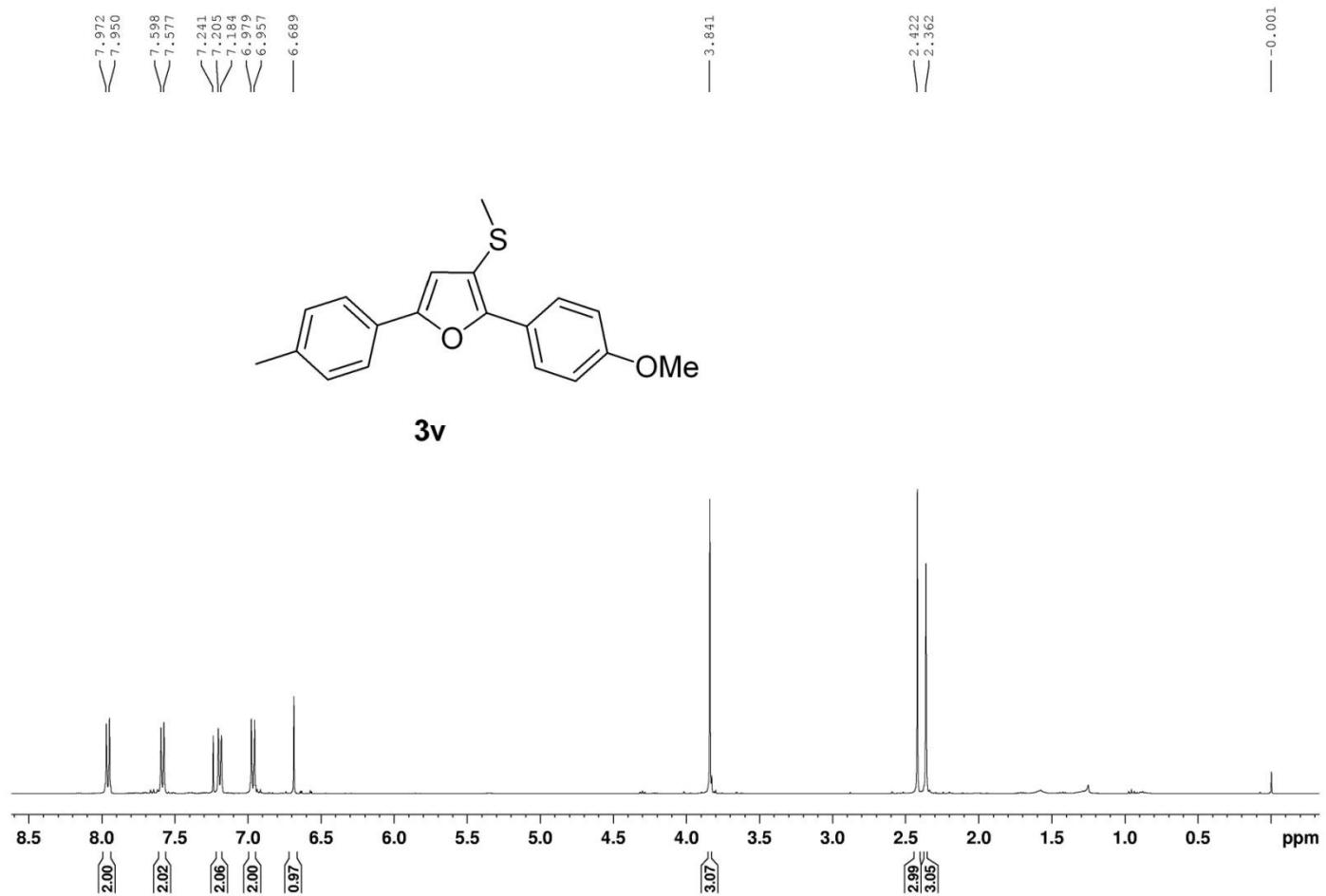


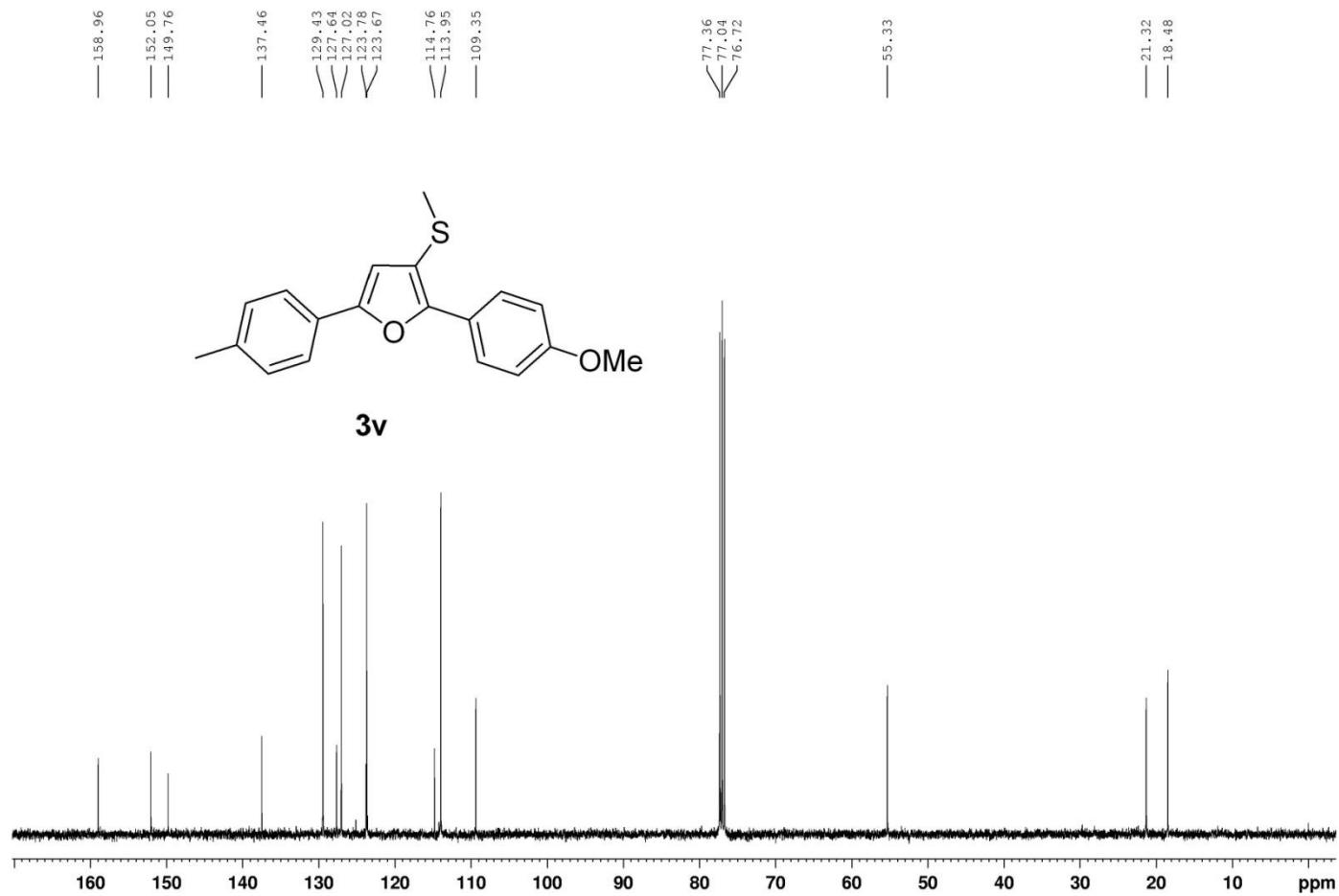


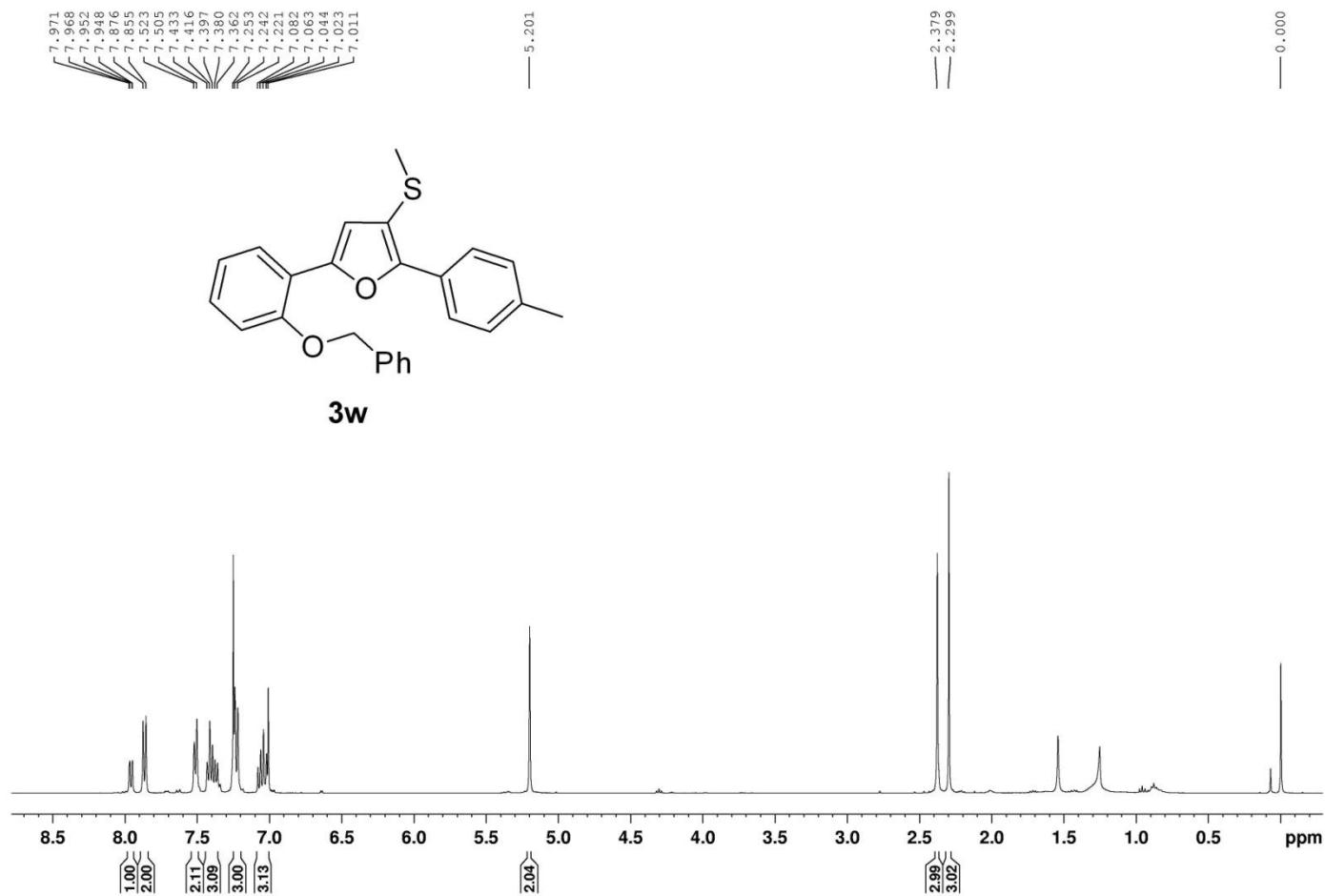


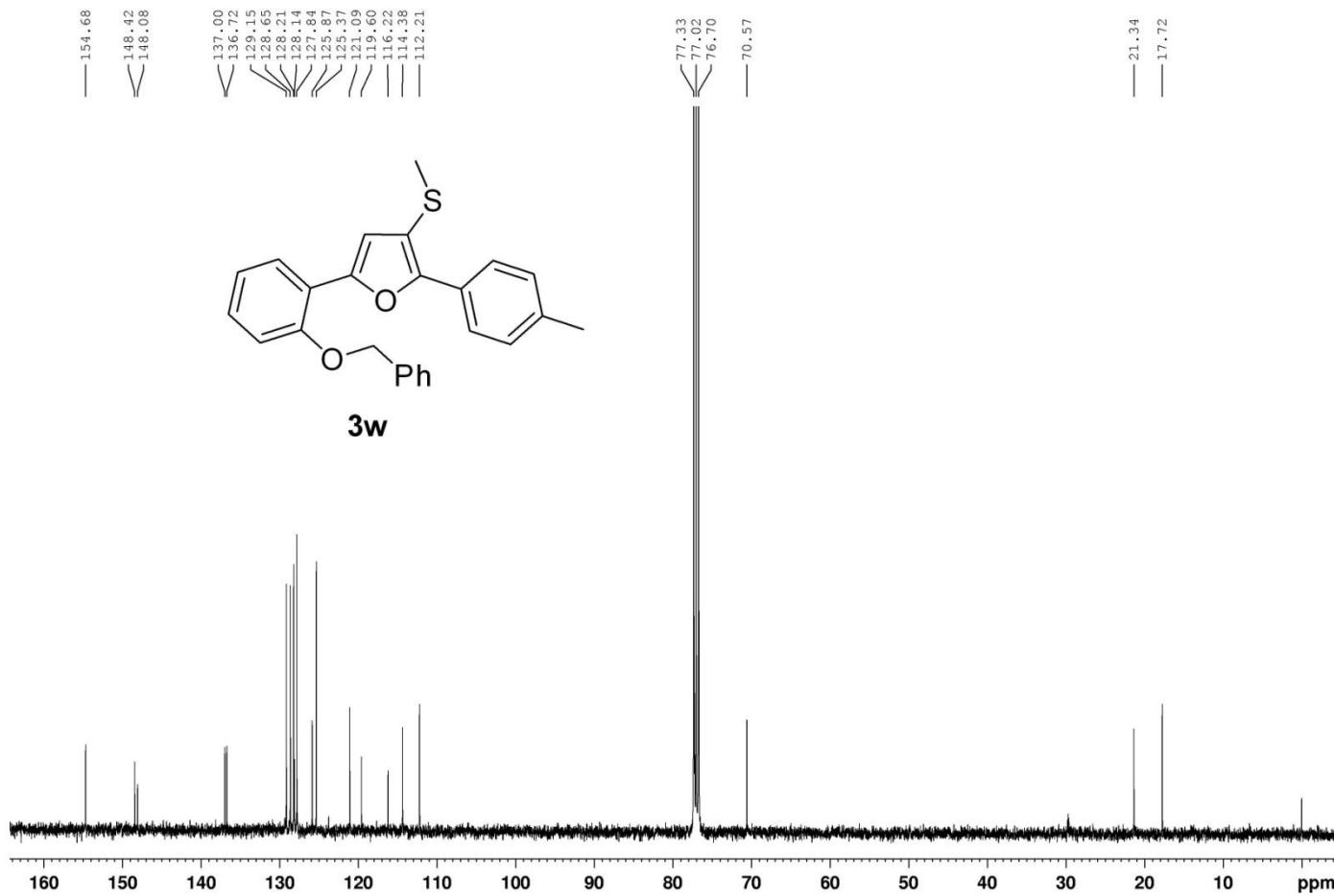










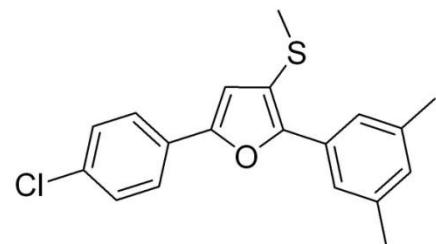


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7.379
7.358
7.252

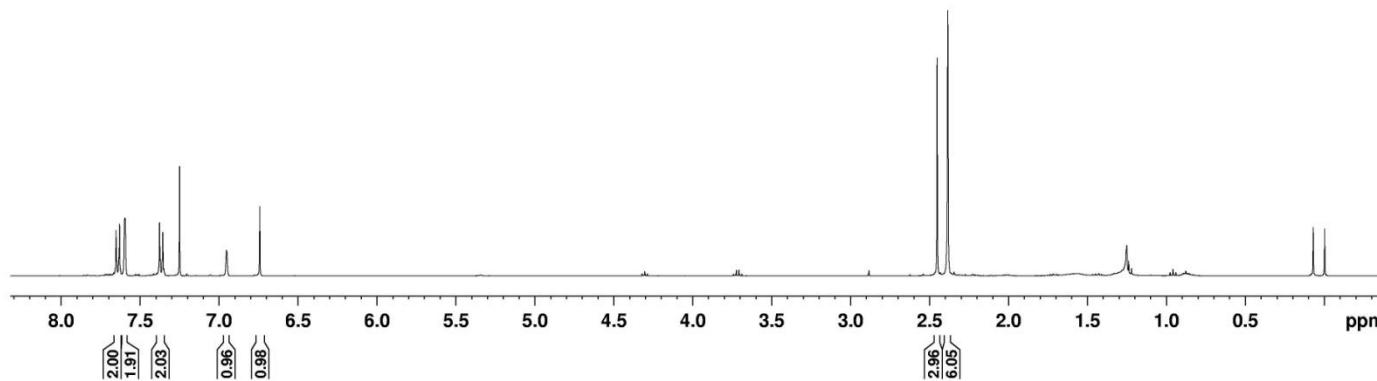
— 6.954
— 6.744

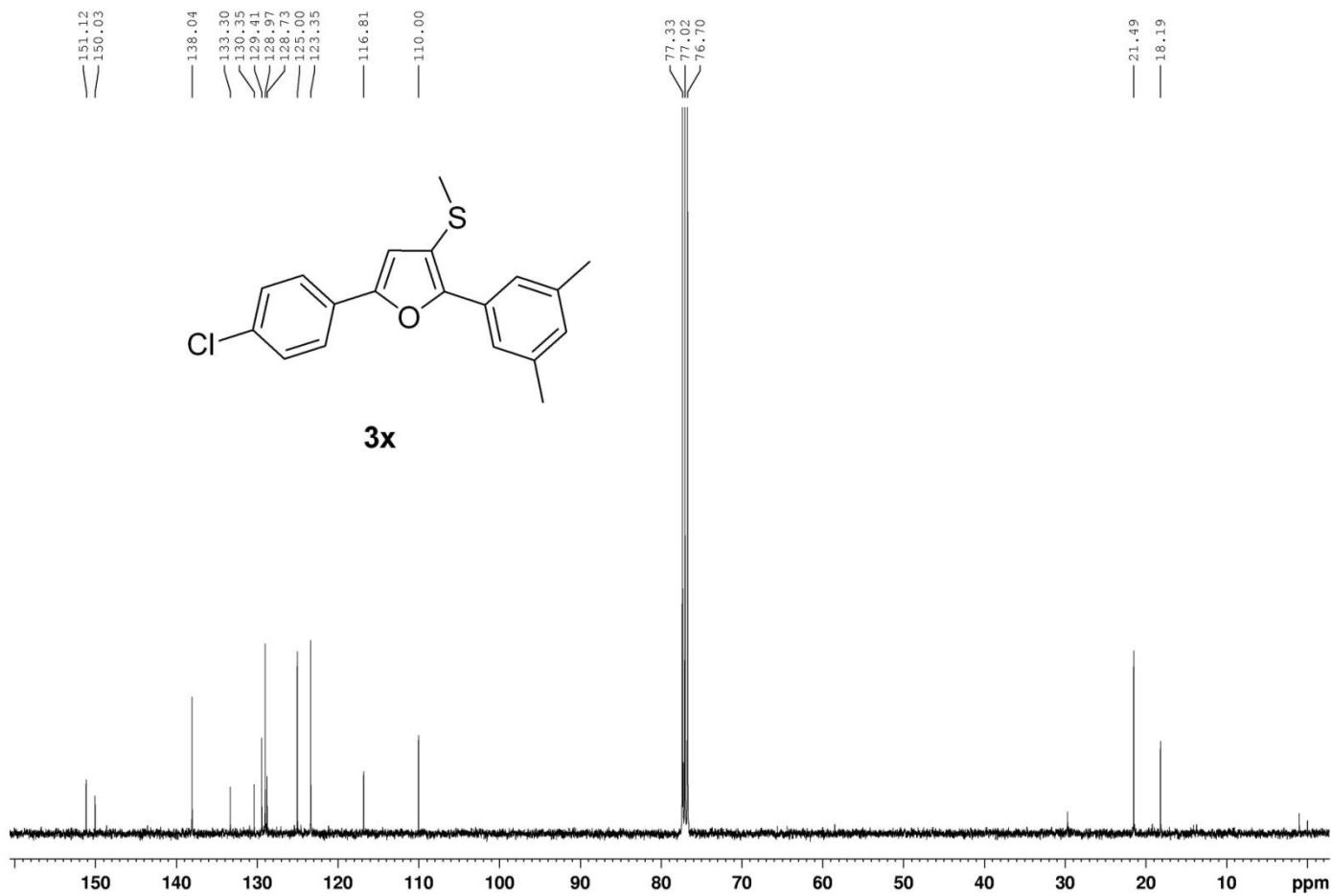
— 2.453
— 2.386

— -0.001



3x



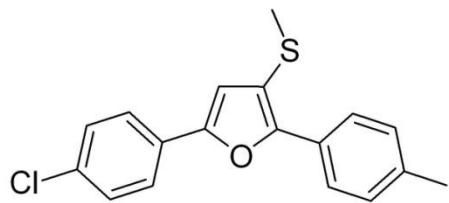


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7.618
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7.253
7.236

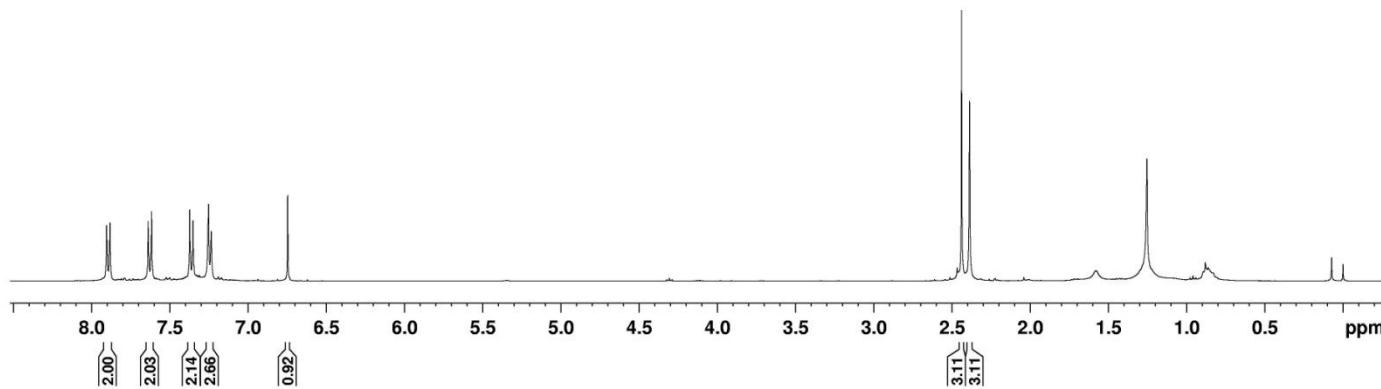
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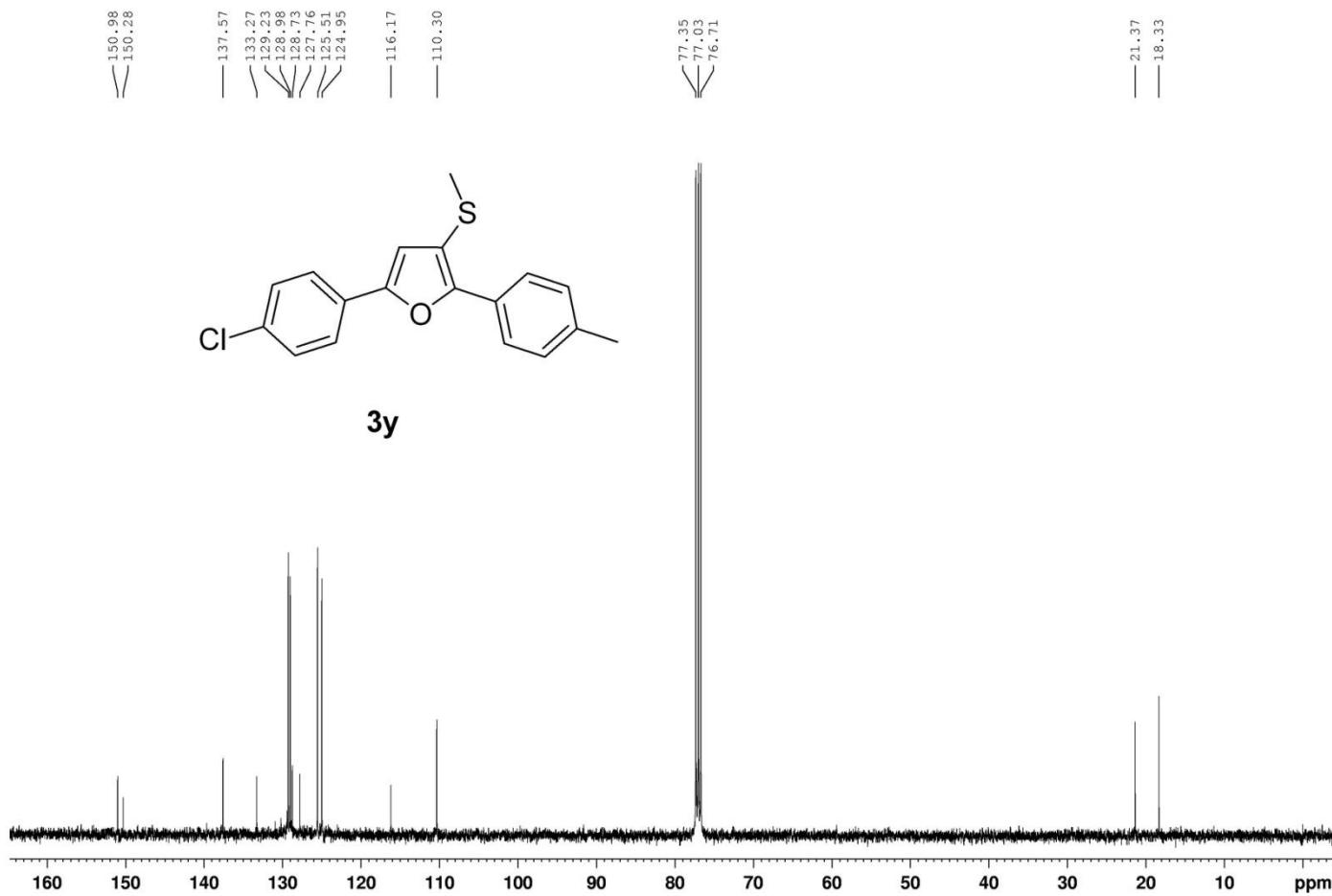
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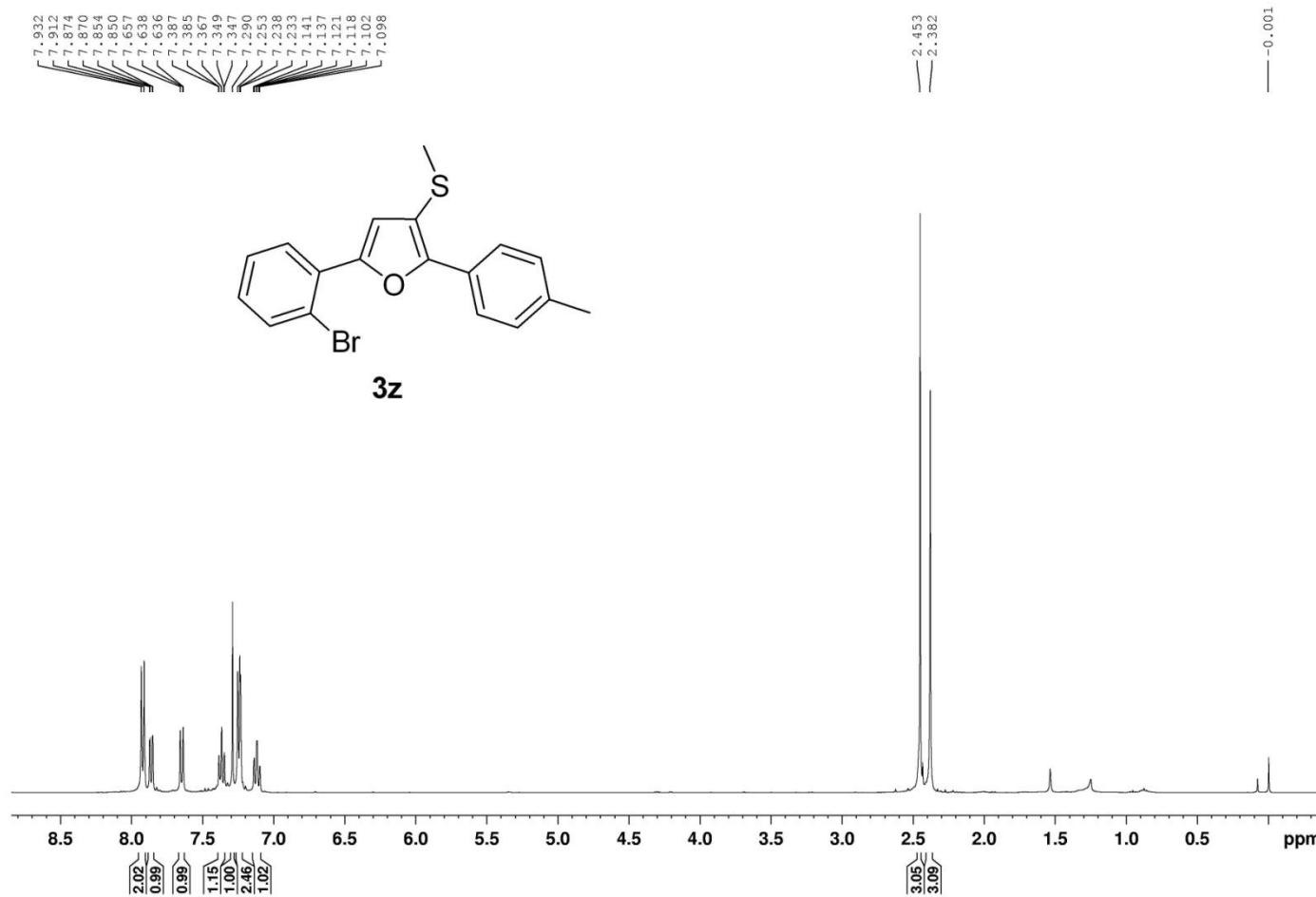
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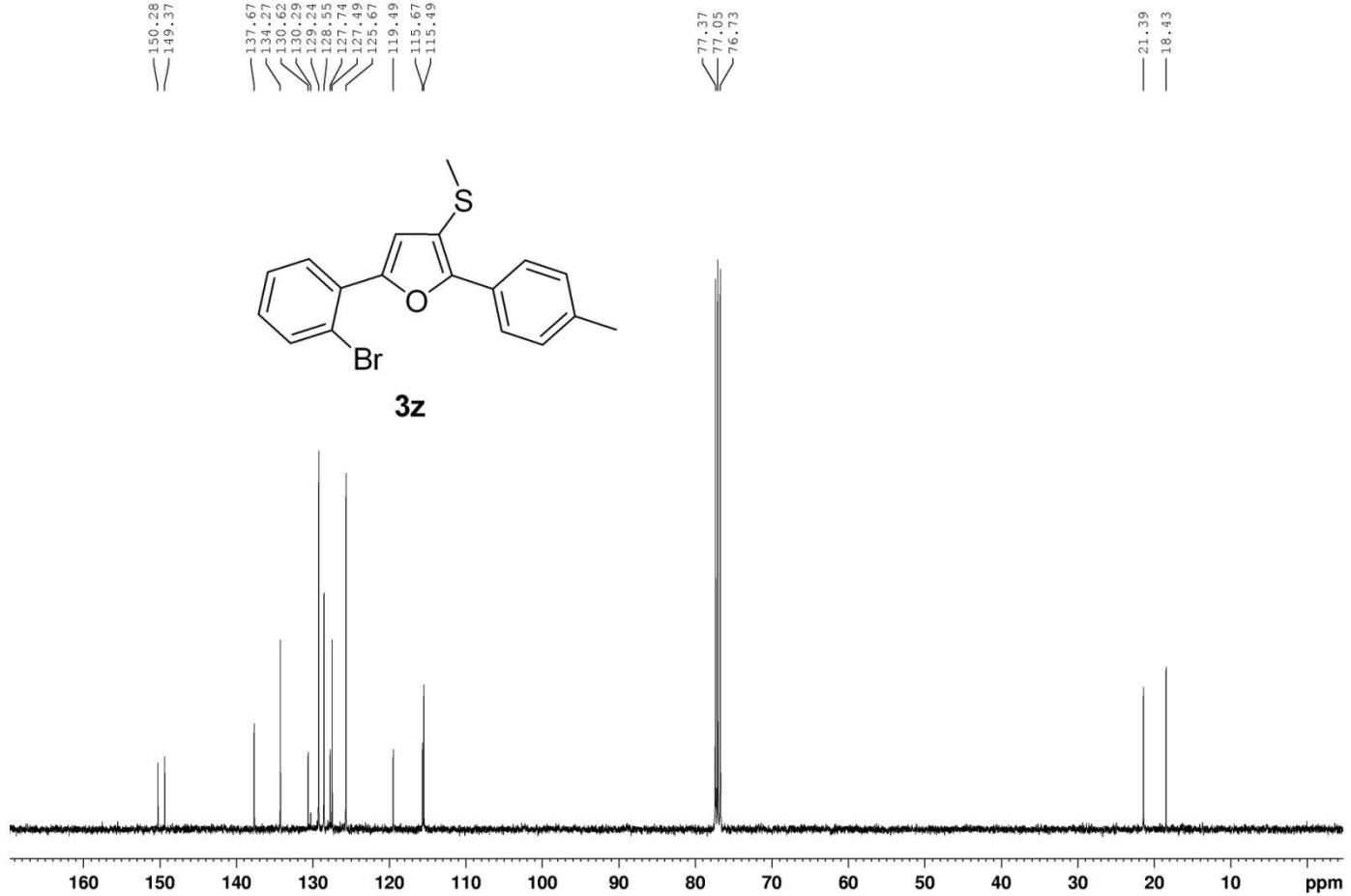


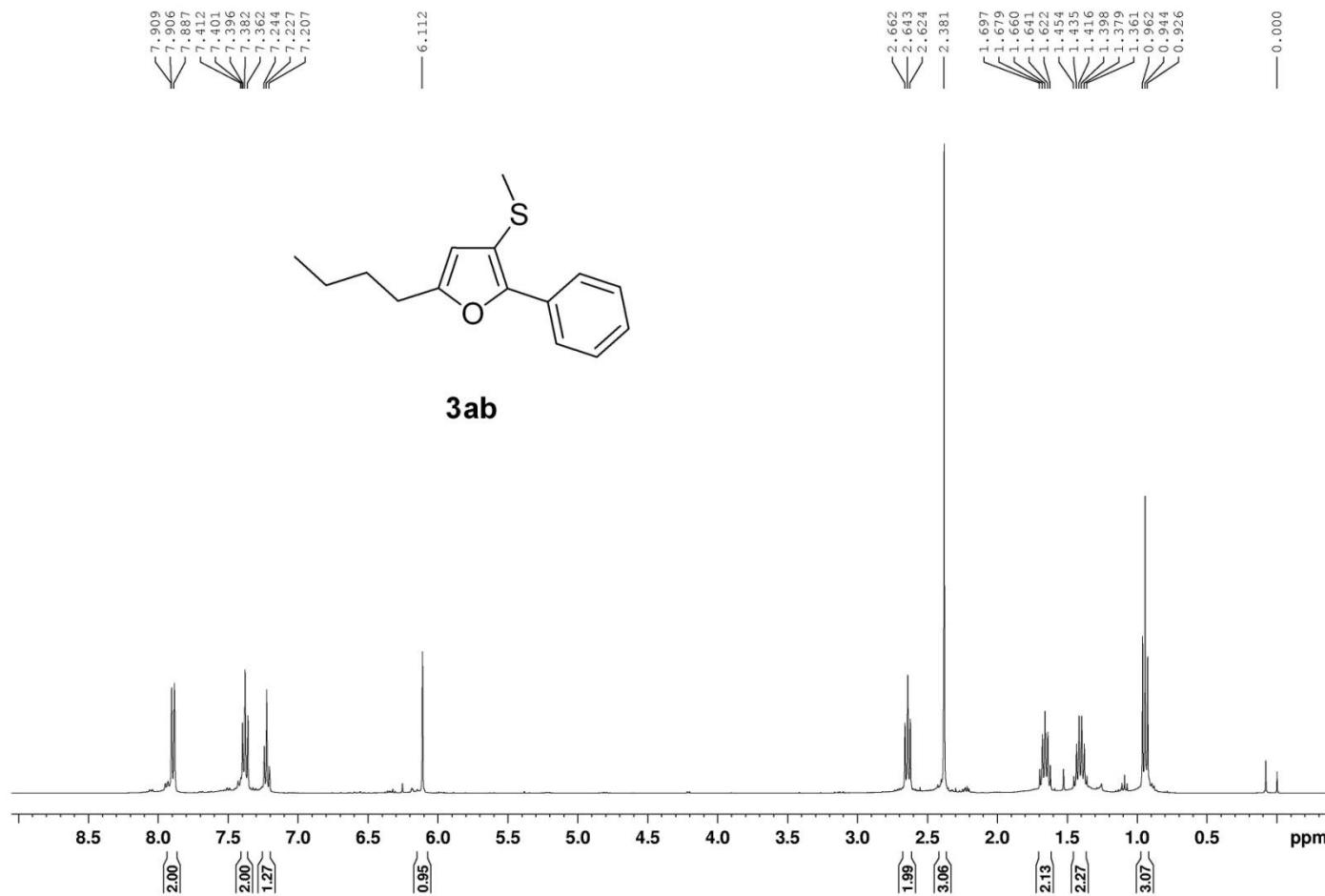
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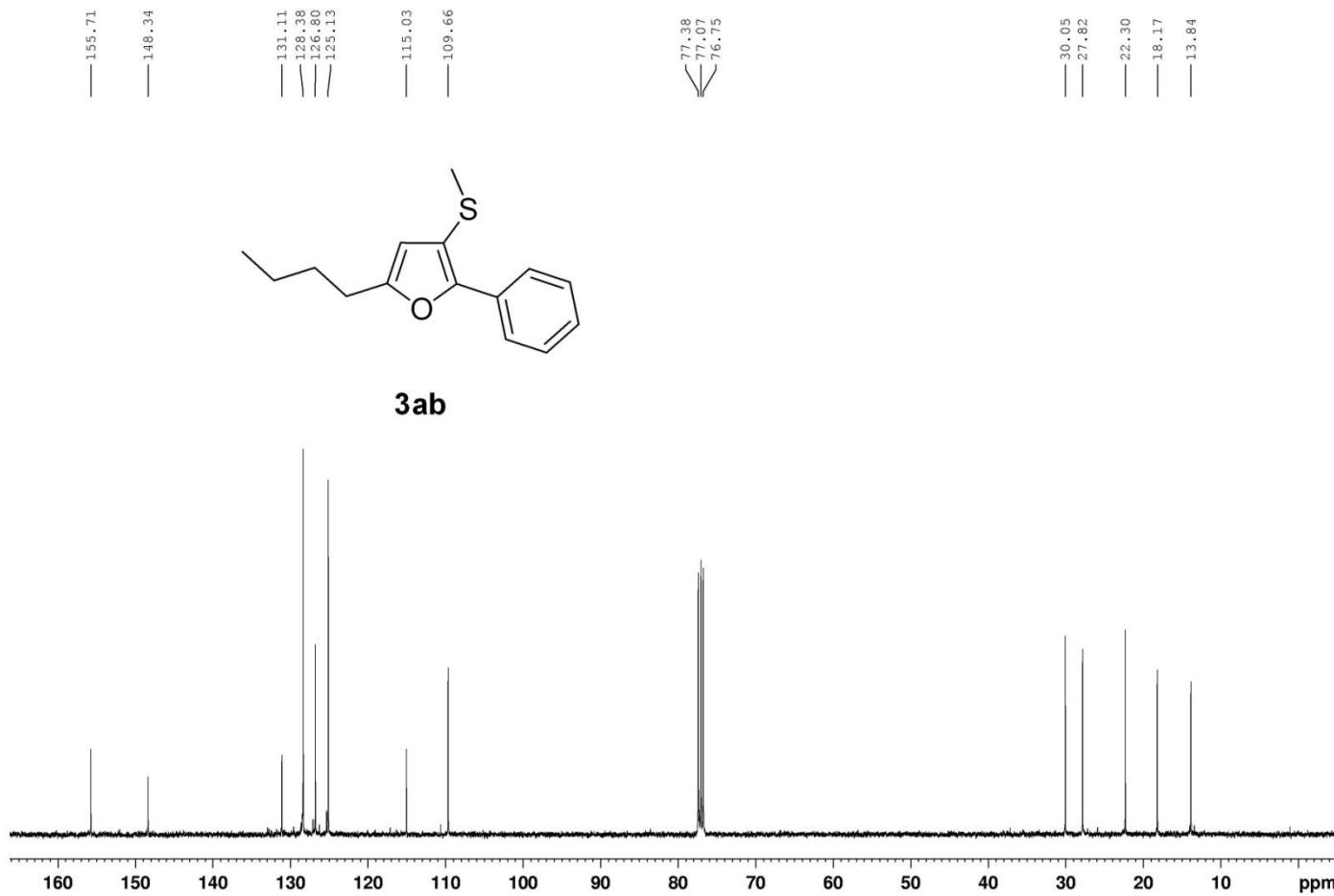


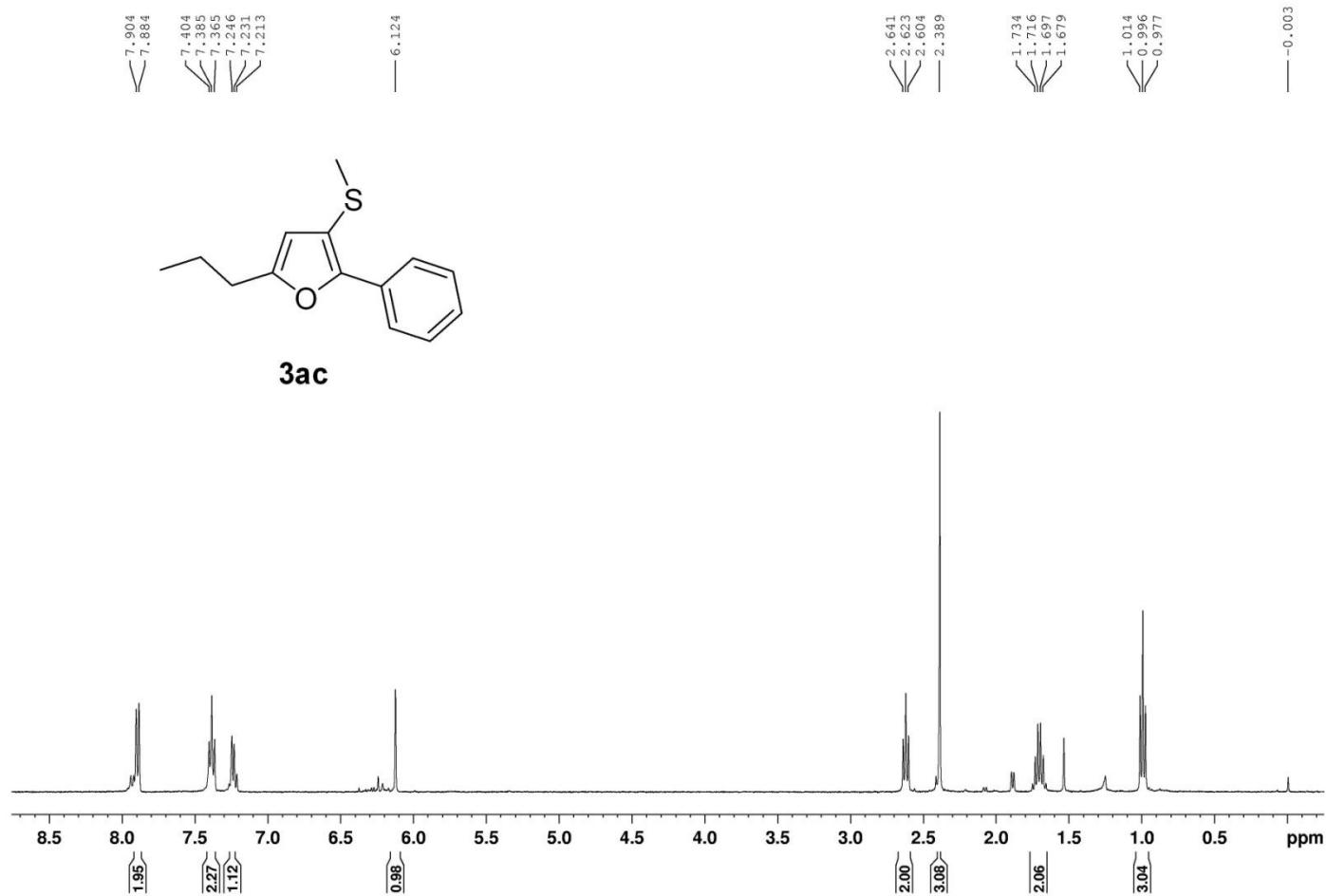


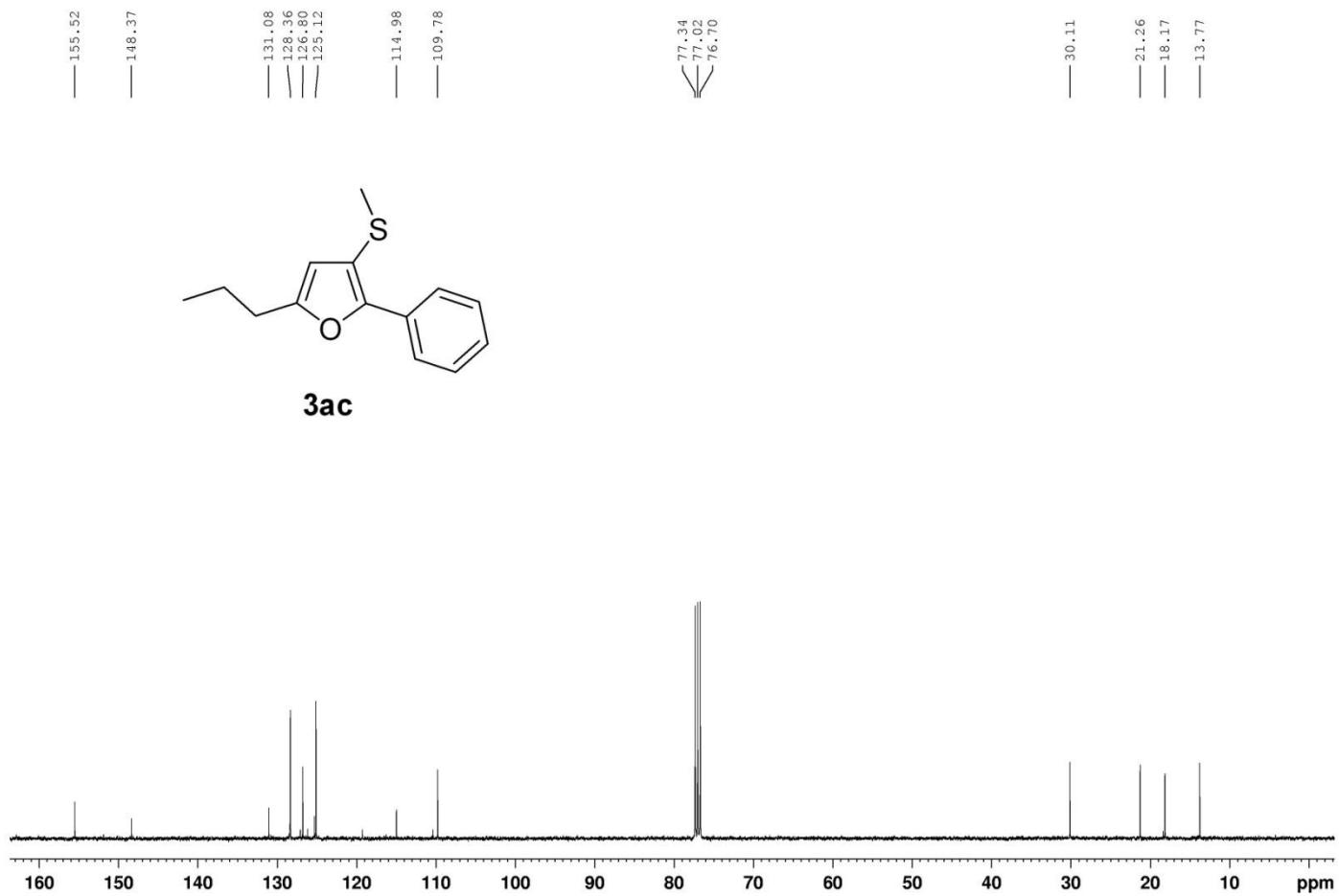


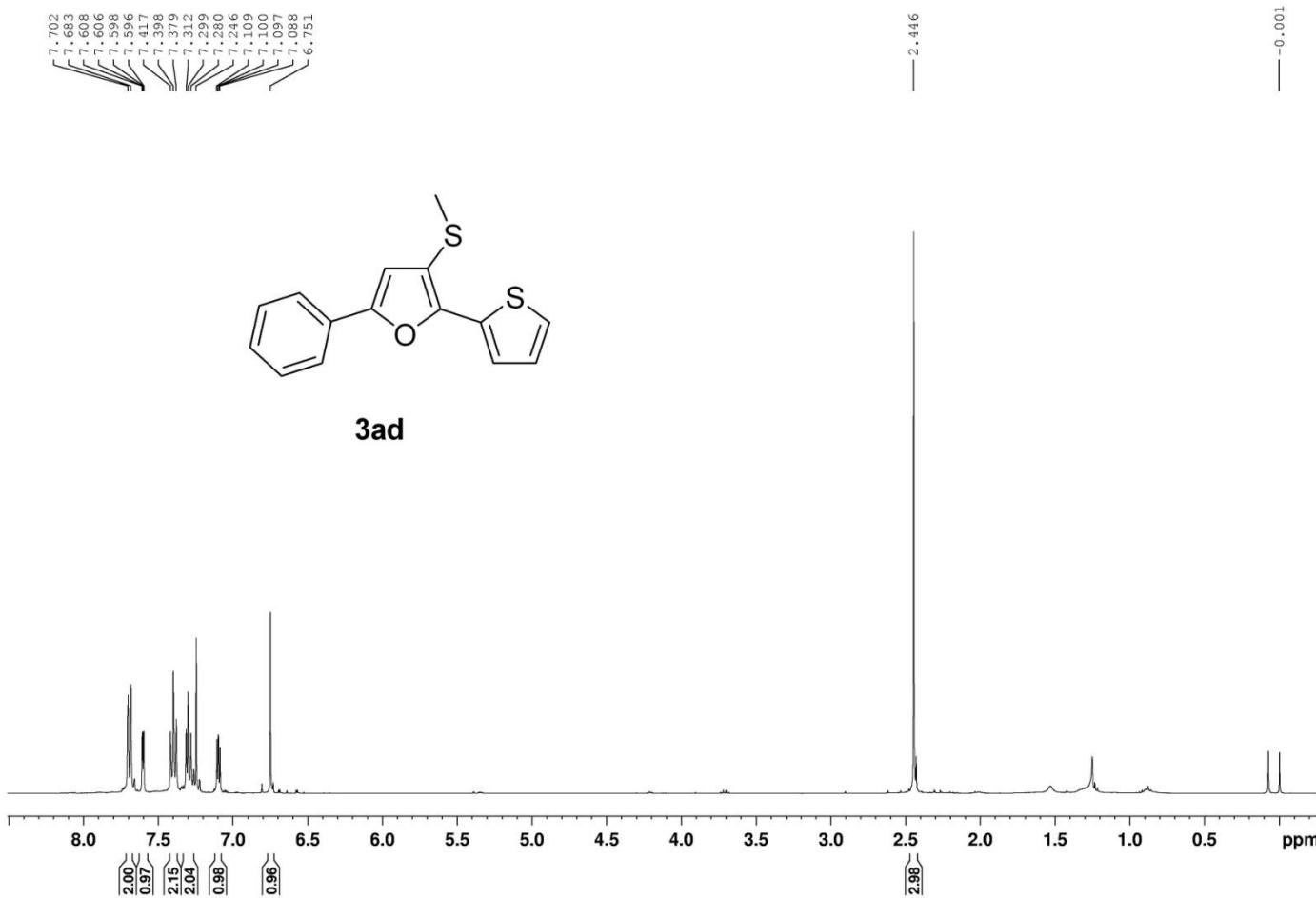


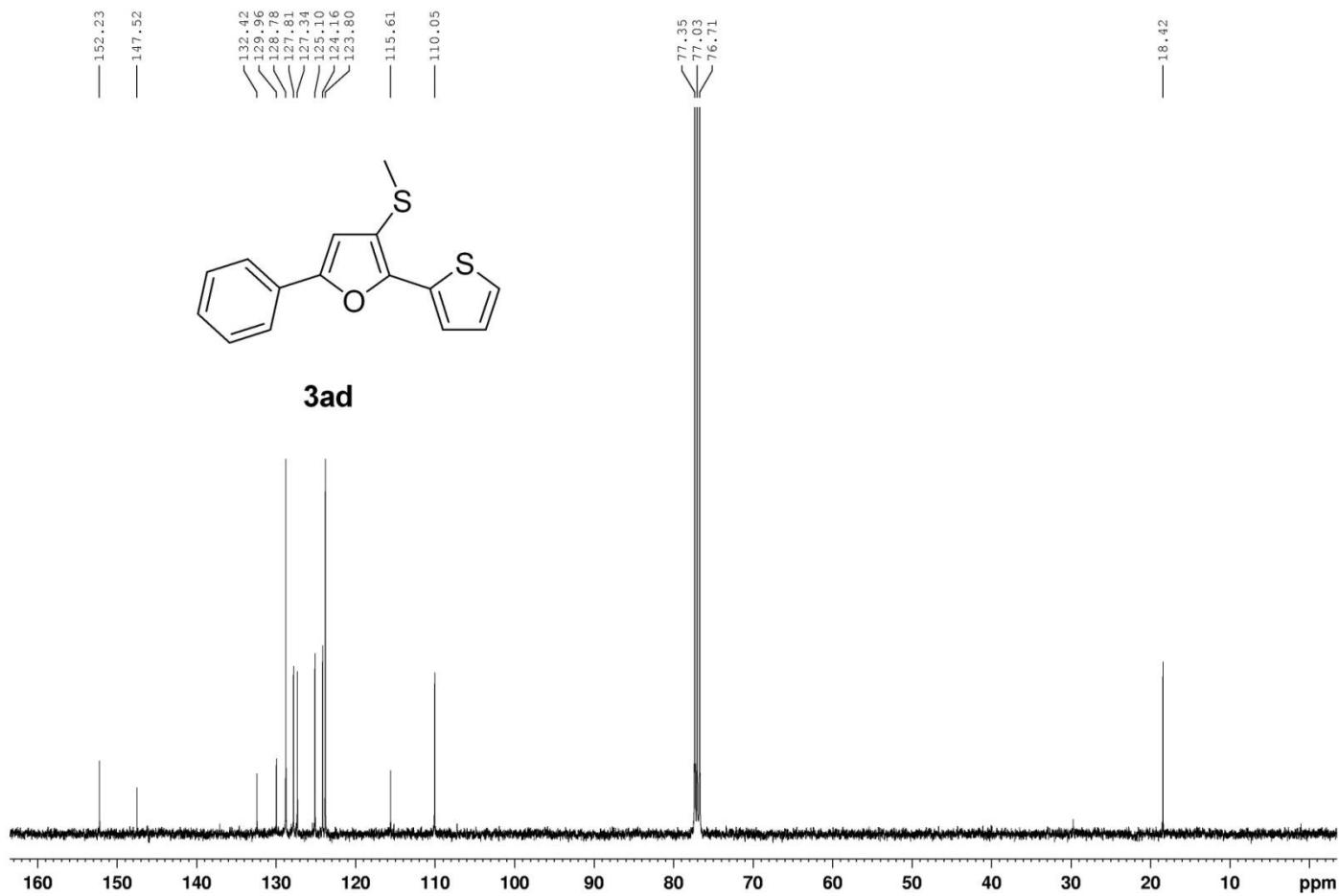








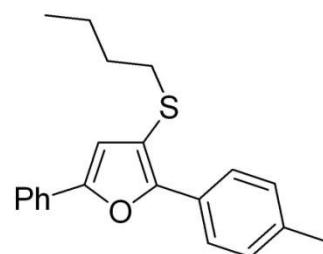




8.000
7.979
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7.705
7.418
7.399
7.379
7.292
7.273
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7.244
7.229
7.217
6.747

2.844
2.826
2.807
2.385

0.000



3ah

