

Supporting Information

Minimizing base-stoichiometry in Pd(0)/g-C₃N₄O catalyzed Suzuki-Miyaura cross-coupling reaction

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Calculation of green chemistry matrices:

Our work: 1-Bromo-4-nitrobenzene (0.101g, 0.5 mmol) and Phenylboronic acid (0.0609g, 0.5 mmol) reacted in presence of Pd(0)/g-C₃N₄O (0.005g, 5 wt%), and K₂CO₃ (0.0345g, 0.25 mmol) in Ethanol medium (2 mL, 0.004g) (excluding water) to form 4-Nitrobiphenyl (0.0978g, 0.49 mmol).

Mass intensity = Total mass used in the process/ Mass of product

$$= (0.101+0.0609+0.005+0.0345+0.004)\text{g}/0.0978\text{g}$$

$$= 2.1$$

Mass productivity = (1/Mass intensity) x 100

$$= (1/2.1) \times 100$$

$$= 47.6\%$$

E-factor = Mass intensity-1

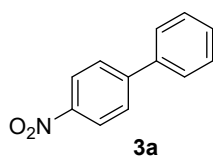
$$= 2.1-1$$

$$= 1.1$$

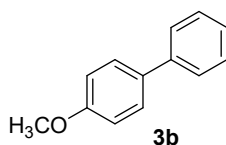
General procedure for synthesis of biaryls:

A mixture of aryl bromide (0.5 mmol), arylboronic acid (0.5 mmol), Pd(0)/g-C₃N₄O (5wt%), K₂CO₃ (0.25 mmol), and EtOH:H₂O (1:1) (4 mL) were taken in a 50 mL round bottomed flask and stirred at room temperature for 5 hours. After completion of the reaction (monitoring by TLC), the reaction mixture was diluted with water (10 mL) and extracted with ethyl acetate (3 x 20 mL). The combined organic layers were washed with brine solution (20 mL) and the organic layer was dried over anhydrous sodium sulfate. The crude was obtained by evaporating the solvent under reduced pressure in a rotary evaporator. To obtain the desired product, purification of the crude was done by column chromatography using silica gel (60-120 mesh) and hexane:ethyl acetate as solvent system.

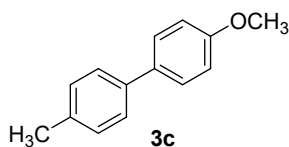
^1H and ^{13}C NMR analytical data:



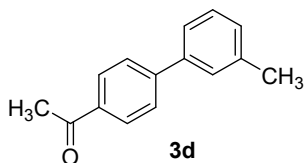
4-nitro-1,1'-biphenyl (3a)^{1,2}: Obtained by general procedure, white solid (98% yield), ^1H NMR (400 MHz, CDCl_3) δ 8.30–8.24 (m, 2H), 7.75–7.68 (m, 2H), 7.63–7.59 (m, 2H), 7.52–7.41 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 147.7, 147.2, 138.8, 129.3, 127.9, 127.6, 127.5, 124.6.



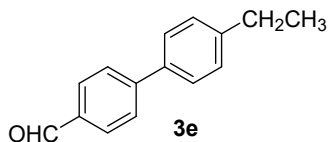
4-methoxy-1,1'-biphenyl (3b)^{1,2}: Obtained by general procedure, white solid (96% yield), ^1H NMR (400 MHz, CDCl_3) δ 7.58–7.49 (m, 4H), 7.46–7.37 (m, 2H), 7.36–7.26 (m, 1H), 7.01–6.94 (m, 2H), 3.85 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 159.2, 141.1, 134.1, 128.9, 128.3, 126.9, 126.7, 114.7, 55.4.



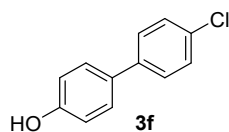
4-methoxy-4'-methyl-1,1'-biphenyl (3c)²: Obtained by general procedure, white solid (90% yield), ^1H NMR (400 MHz, CDCl_3) δ 7.54–7.41 (m, 4H), 7.22 (dd, $J = 8.4, 0.6$ Hz, 2H), 6.99–6.93 (m, 2H), 3.84 (s, 3H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 159.2, 138.0, 136.7, 134.1, 130.2, 128.4, 127.0, 114.7, 55.8, 21.5.



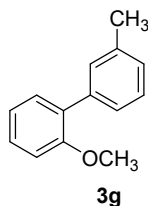
1-(3'-methyl-[1,1'-biphenyl]-4-yl)ethan-1-one (3d)³: Obtained by general procedure, white solid (85% yield), ^1H NMR (400 MHz, CDCl_3) δ 8.04–7.99 (m, 2H), 7.70–7.64 (m, 2H), 7.41 (dd, $J = 4.6, 4.1$ Hz, 2H), 7.35 (t, $J = 7.5$ Hz, 1H), 7.21 (dd, $J = 4.3, 3.7$ Hz, 1H), 2.62 (s, 3H), 2.43 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 198.3, 146.1, 140.0, 138.7, 135.8, 129.0, 128.2, 128.1, 127.4, 124.5, 124.4, 26.7, 21.9.



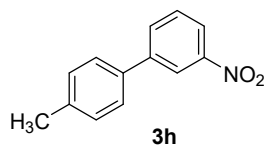
4'-ethyl-[1,1'-biphenyl]-4-carbaldehyde (3e)⁴: Obtained by general procedure, white solid (92% yield), ¹H NMR (400 MHz, CDCl₃) δ 10.03 (s, 1H), 7.95–7.90 (m, 2H), 7.76–7.71 (m, 2H), 7.60–7.53 (m, 2H), 7.34–7.28 (m, 2H), 2.80–2.61 (m, 2H), 1.31–1.21 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 192.1, 147.3, 145.0, 137.1, 135.1, 130.4, 128.6, 127.5, 127.4, 28.6, 15.8.



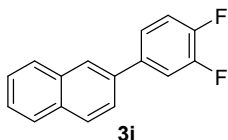
4'-chloro-[1,1'-biphenyl]-4-ol (3f)⁵: Obtained by general procedure, white solid (80% yield), ¹H NMR (400 MHz, CDCl₃) δ 7.49–7.32 (m, 6H), 6.93–6.86 (m, 2H), 4.94 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 155.2, 139.4, 132.9, 132.8, 129.0, 128.4, 128.1, 116.0.



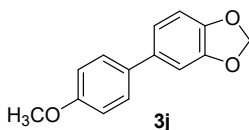
2-methoxy-3'-methyl-1,1'-biphenyl (3g)^{3a}: Obtained by general procedure, white solid (65% yield), ¹H NMR (400 MHz, CDCl₃) δ 7.44–7.33 (m, 5H), 7.24–7.18 (m, 1H), 7.11–7.01 (m, 2H), 3.85 (d, *J* = 1.3 Hz, 3H), 2.46 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 156.7, 138.7, 137.7, 131.0, 130.4, 130.3, 128.8, 128.0, 120.9, 111.2, 56.3, 21.9.



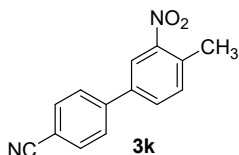
4'-methyl-3-nitro-1,1'-biphenyl (3h)⁶: Obtained by general procedure, white solid (63% yield), ¹H NMR (400 MHz, CDCl₃) δ 8.44–8.41 (m, 1H), 8.18–8.14 (m, 1H), 7.91–7.86 (m, 1H), 7.61–7.49 (m, 3H), 7.31–7.27 (m, 2H), 2.41 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 148.8, 142.9, 138.7, 135.8, 133.0, 130.0, 129.9, 129.7, 127.1, 121.8, 21.3.



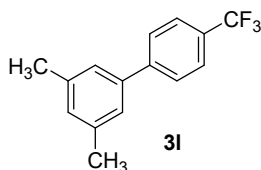
2-(3,4-difluorophenyl)naphthalene (3i)⁷: Obtained by general procedure, white solid (74% yield), ¹H NMR (400 MHz, CDCl₃) δ 7.96 (s, 1H), 7.93–7.80 (m, 3H), 7.64 (dd, *J* = 8.5, 1.7 Hz, 1H), 7.56–7.38 (m, 4H), 7.31–7.18 (m, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 151.5, 149.5, 138.4, 136.6, 132.8, 128.8, 128.4, 127.9, 126.68 (d, *J*_{C-F} = 25.0 Hz), 126.43, 125.9, 125.1, 123.4; ¹⁹F NMR (376 MHz, CDCl₃) δ -137.34(s), -140.01(s).



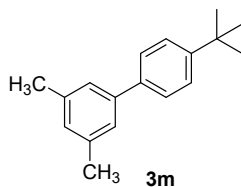
5-(4-methoxyphenyl)benzo[1,3]dioxole (3j)⁸: Obtained by general procedure, white solid (91% yield), ¹H NMR (400 MHz, CDCl₃) δ 7.48–7.39 (m, 2H), 7.04–6.91 (m, 4H), 6.88–6.82 (m, 1H), 6.01–5.94 (m, 2H), 3.83 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 159.0, 148.2, 146.7, 135.4, 133.8, 127.9, 120.3, 114.3, 108.7, 107.5, 101.1, 55.8.



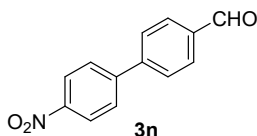
4'-methyl-3'-nitro-[1,1'-biphenyl]-4-carbonitrile (3k)⁹: Obtained by general procedure, white solid (68% yield), ¹H NMR (600 MHz, CDCl₃) δ 7.92–7.83 (m, 1H), 7.48–7.32 (m, 5H), 7.13 (dd, *J* = 9.6, 5.3 Hz, 1H), 6.98–6.86 (m, 1H), 2.43–2.28 (m, 3H); ¹³C NMR (150 MHz, CDCl₃) δ 149.4, 142.6, 137.9, 133.6, 133.4, 132.6, 131.0, 127.3, 122.9, 118.2, 111.7, 19.9.



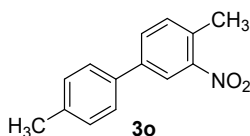
3,5-dimethyl-4'-(trifluoromethyl)-1,1'-biphenyl (3l)¹⁰: Obtained by general procedure, colourless liquid (70% yield), ¹H NMR (400 MHz, CDCl₃) δ 7.66 (s, 4H), 7.20 (s, 2H), 7.05 (d, *J* = 0.5 Hz, 1H), 2.42–2.36 (m, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 143.2, 138.6, 130.5, 130.1, 129.8, 127.6, 127.4, 125.2, 122.77 (*J*_{C-F} = 270 Hz), 120.07, 21.4; ¹⁹F NMR (377 MHz, CDCl₃) δ -62.6.



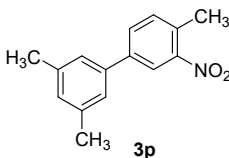
4'-(tert-butyl)-3,5-dimethyl-1,1'-biphenyl (3m)¹¹: Obtained by general procedure, colourless liquid (68% yield), ¹H NMR (500 MHz, CDCl₃) δ 7.63–7.59 (m, 2H), 7.55–7.52 (m, 2H), 7.29 (d, *J* = 6.8 Hz, 2H), 7.06 (s, 1H), 2.46 (s, 6H), 1.45 (s, 9H); ¹³C NMR (125 MHz, CDCl₃) δ 150.0, 141.1, 138.1, 128.6, 126.8, 126.6, 125.5, 124.9, 34.4, 31.3, 21.4.



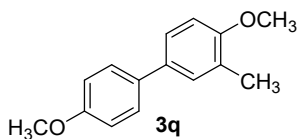
4'-nitro-[1,1'-biphenyl]-4-carbaldehyde (3n)¹²: Obtained by general procedure, white solid (86% yield), ¹H NMR (500 MHz, CDCl₃) δ 10.11 (s, 1H), 8.35 (d, *J* = 8.9 Hz, 2H), 8.03 (d, *J* = 8.4 Hz, 2H), 7.83–7.77 (m, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 191.5, 147.7, 145.9, 144.4, 136.2, 130.3, 128.1, 128.0, 124.2.



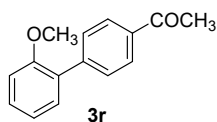
4,4'-dimethyl-3-nitro-1,1'-biphenyl (3o)¹³: Obtained by general procedure, white solid (64% yield), ¹H NMR (500 MHz, CDCl₃) δ 8.21 (d, *J* = 1.9 Hz, 1H), 7.73 (dd, *J* = 7.9, 1.9 Hz, 1H), 7.54–7.49 (m, 2H), 7.39 (dd, *J* = 11.7, 4.6 Hz, 1H), 7.30 (dd, *J* = 10.8, 3.0 Hz, 2H), 2.65 (s, 3H), 2.44 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 149.5, 140.1, 138.1, 135.5, 133.1, 131.8, 131.0, 129.7, 126.6, 122.6, 21.0, 20.0.



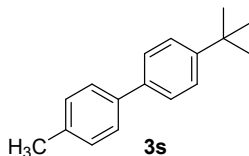
3',4,5'-trimethyl-3-nitro-1,1'-biphenyl (3p): Obtained by general procedure, colourless liquid (61% yield), ¹H NMR (500 MHz, CDCl₃) δ 8.22 (d, *J* = 1.9 Hz, 1H), 7.73 (dd, *J* = 7.9, 2.0 Hz, 1H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.24 (s, 2H), 7.08–7.05 (m, 1H), 2.66 (s, 3H), 2.42 (s, 6H); ¹³C NMR (125 MHz, CDCl₃) δ 149.4, 140.5, 138.6, 138.4, 133.0, 131.9, 131.2, 129.8, 124.7, 122.8, 21.3, 20.0. Anal. for C₁₅H₁₅NO₂, calcd C, 74.67; H, 6.27; N, 5.81; found C, 74.16; H, 6.37; N, 5.39.



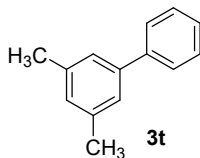
4,4'-dimethoxy-3-methyl-1,1'-biphenyl (3q)¹⁴: Obtained by general procedure, white solid (68% yield), ¹H NMR (500 MHz, CDCl₃) δ 7.55–7.48 (m, 2H), 7.38 (dd, *J* = 6.0, 2.2 Hz, 2H), 7.02–6.97 (m, 2H), 6.94–6.88 (m, 1H), 3.90 (s, 3H), 3.88 (s, 3H), 2.32 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 158.5, 156.8, 133.6, 133.0, 129.1, 127.6, 126.7, 124.8, 114.0, 110.1, 55.3, 55.2, 16.3.



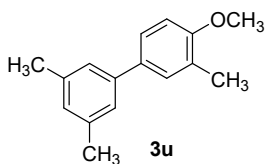
1-(2'-methoxy-[1,1'-biphenyl]-4-yl)ethan-1-one (3r)¹⁵: Obtained by general procedure, white solid (64% yield), ¹H NMR (500 MHz, CDCl₃) δ 8.08–7.99 (m, 2H), 7.73–7.62 (m, 2H), 7.46–7.34 (m, 2H), 7.15–6.98 (m, 2H), 3.85 (s, 3H), 2.66 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 197.8, 156.4, 143.5, 135.4, 130.6, 129.6, 129.4, 129.3, 128.0, 120.9, 111.3, 55.5, 26.5.



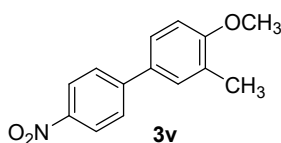
4-(tert-butyl)-4'-methyl-1,1'-biphenyl (3s)¹⁵: Obtained by general procedure, white solid (98% yield), ¹H NMR (500 MHz, CDCl₃) δ 7.60–7.49 (m, 6H), 7.31–7.26 (m, 2H), 2.44 (s, 3H), 1.42 (s, 9H); ¹³C NMR (125 MHz, CDCl₃) δ 149.8, 138.2, 136.6, 129.3, 126.8, 126.5, 125.6, 125.5, 34.4, 31.3, 21.0.



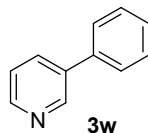
3,5-dimethyl-1,1'-biphenyl (3t)¹⁶: Obtained by general procedure, colourless liquid (84% yield), ¹H NMR (500 MHz, CDCl₃) δ 7.73 (d, *J* = 7.1 Hz, 2H), 7.60–7.46 (m, 4H), 7.37 (s, 2H), 7.14 (s, 1H), 2.53 (s, 6H); ¹³C NMR (125 MHz, CDCl₃) δ 141.5, 141.3, 138.2, 128.9, 128.7, 127.2, 127.1, 125.1, 21.4.



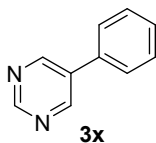
4-methoxy-3,3',5'-trimethyl-1,1'-biphenyl (3u): Obtained by general procedure, colourless liquid (72% yield), ^1H NMR (500 MHz, CDCl_3) δ 7.49–7.46 (m, 3H), 7.28 (s, 1H), 7.05–6.95 (m, 2H), 3.94 (s, 3H), 2.47 (s, 6H), 2.39(s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 157.2, 138.1, 133.5, 129.5, 129.0, 128.2, 126.7, 125.3, 124.7, 110.0, 55.3, 21.4, 16.3. Anal. for $\text{C}_{16}\text{H}_{18}\text{O}$, calcd C, 84.91; H, 8.02; found C, 84.58; H, 8.18.



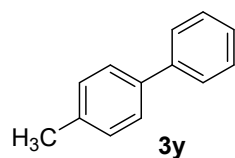
4-methoxy-3-methyl-4'-nitro-1,1'-biphenyl (3v)¹⁷: Obtained by general procedure, white solid (72% yield), ^1H NMR (500 MHz, CDCl_3) δ 8.31–8.24 (m, 2H), 7.74–7.68 (m, 2H), 7.46 (d, $J = 5.9$ Hz, 2H), 6.95 (d, $J = 8.4$ Hz, 1H), 3.92 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 158.6, 147.4, 146.3, 130.5, 129.5, 127.4, 126.9, 125.8, 124.0, 110.2, 55.4, 16.3.



3-phenylpyridine (3w)²: Obtained by general procedure, colourless liquid (82% yield), ^1H NMR (400 MHz, CDCl_3) δ 8.87–8.80 (m, 1H), 8.60–8.53 (m, 1H), 7.89–7.82 (m, 1H), 7.59–7.53 (m, 2H), 7.49–7.30 (m, 4H); ^{13}C NMR (125 MHz, CDCl_3) δ 148.5, 148.4, 137.9, 136.8, 134.5, 129.3, 128.2, 127.2, 123.8.



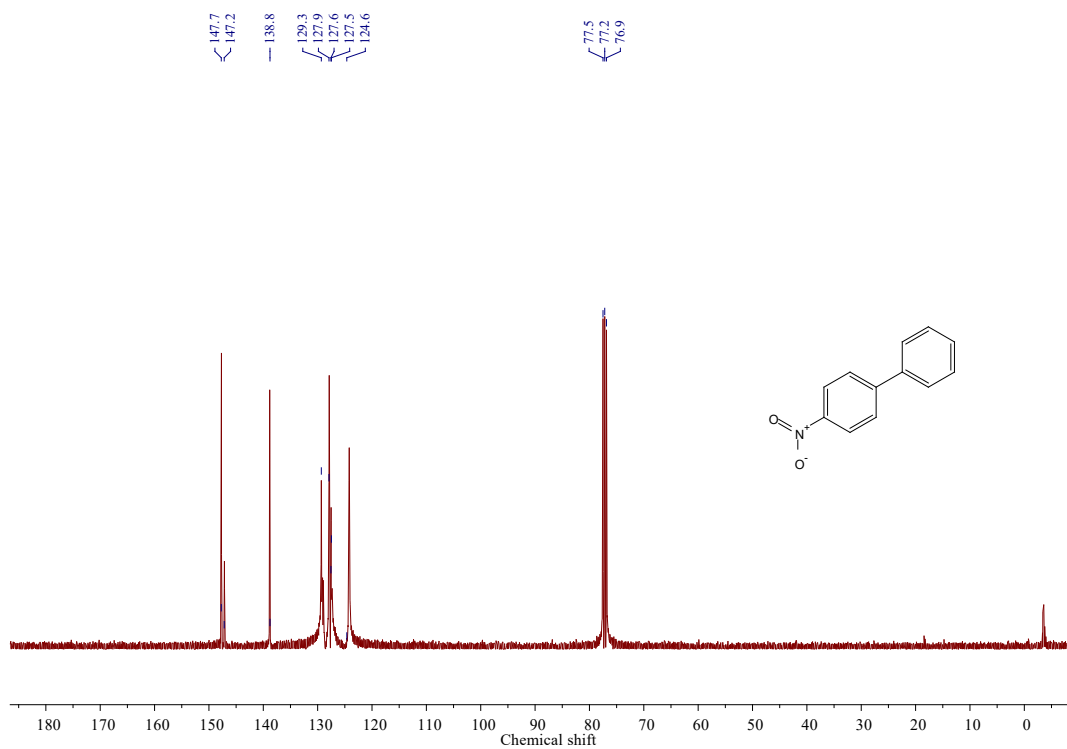
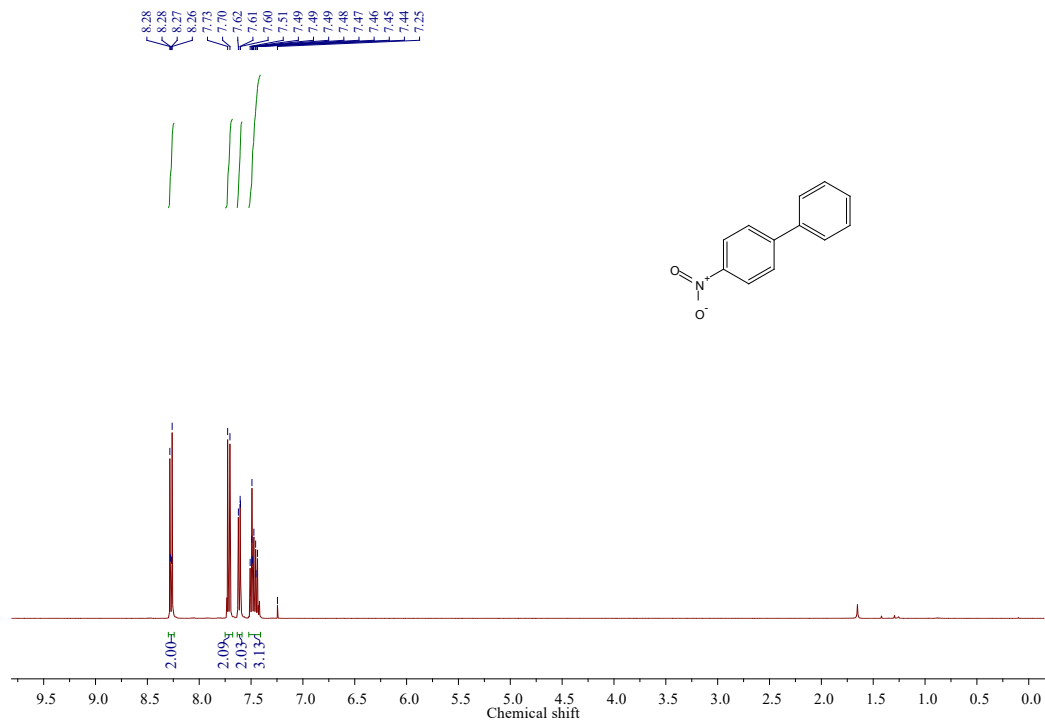
5-phenylpyrimidine (3x)¹: Obtained by general procedure, colourless liquid (74% yield), ^1H NMR (400 MHz, CDCl_3) δ 9.20–9.16 (m, 1H), 8.93 (d, $J = 3.3$ Hz, 2H), 7.58–7.54 (m, 2H), 7.52–7.42 (m, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 157.6, 157.5, 155.0, 134.4, 134.3, 129.6, 129.1, 127.1.



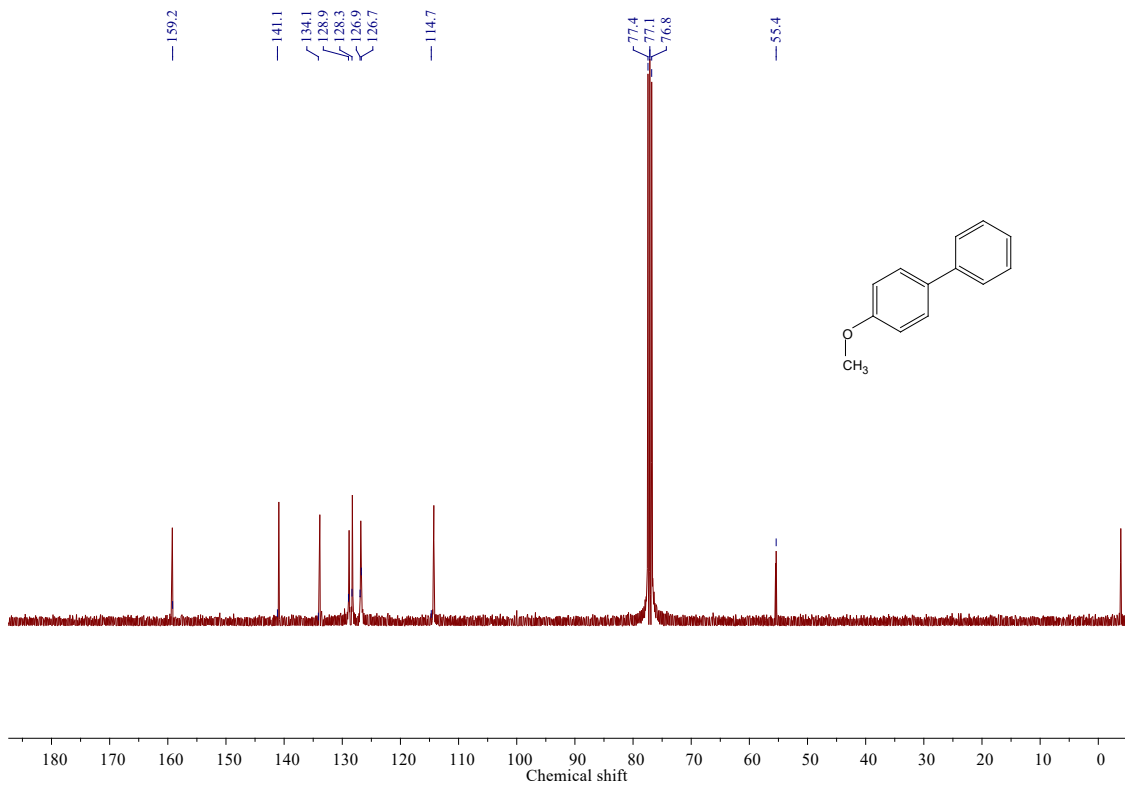
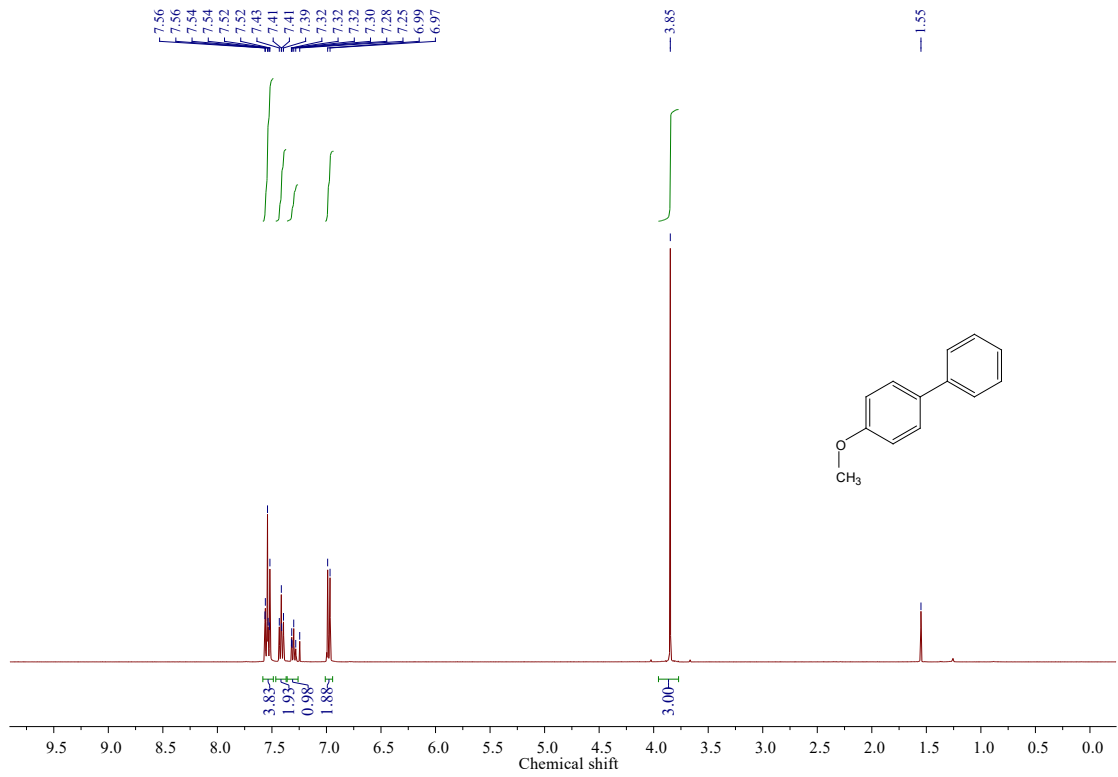
4-methyl-1,1'-biphenyl (3y)^{1,2}: Obtained by general procedure, white solid (81% yield), ¹H NMR (500 MHz, CDCl₃) δ 7.66–7.61 (m, 2H), 7.55 (d, *J* = 8.1 Hz, 2H), 7.48 (dd, *J* = 10.6, 4.8 Hz, 2H), 7.40–7.35 (m, 1H), 7.32–7.28 (m, 2H), 2.45 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 141.1, 138.3, 136.9, 129.4, 128.6, 126.9, 126.8, 21.0.

^1H and ^{13}C NMR spectra

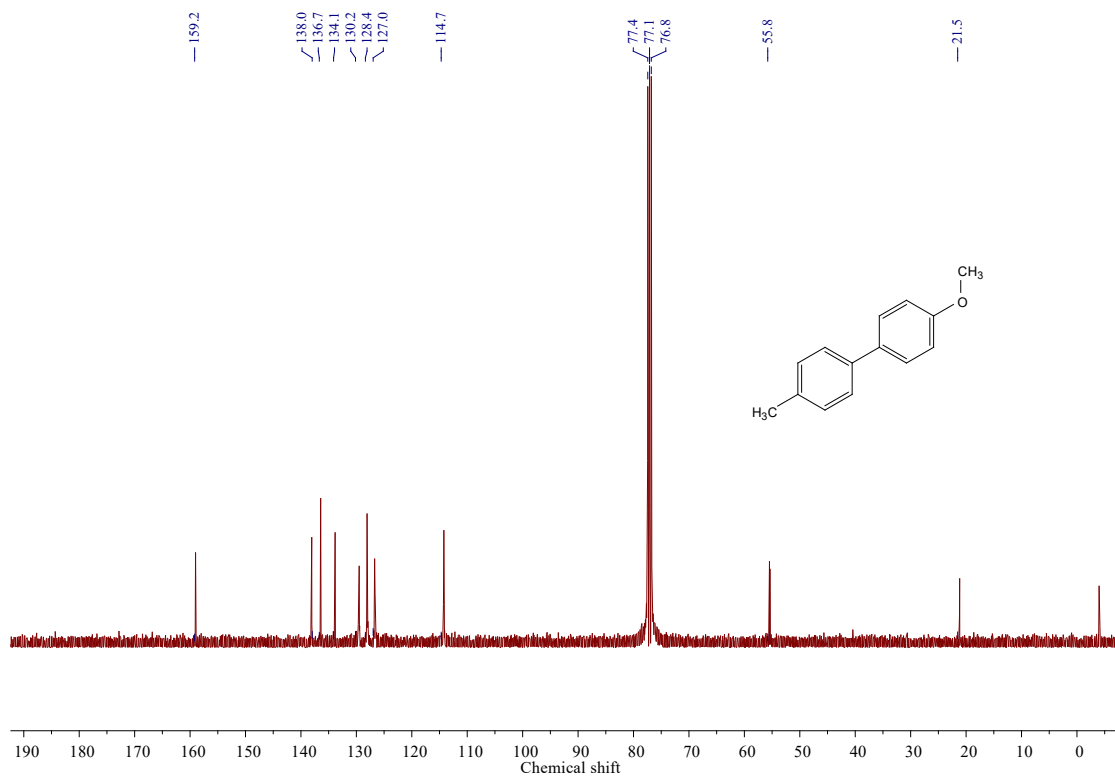
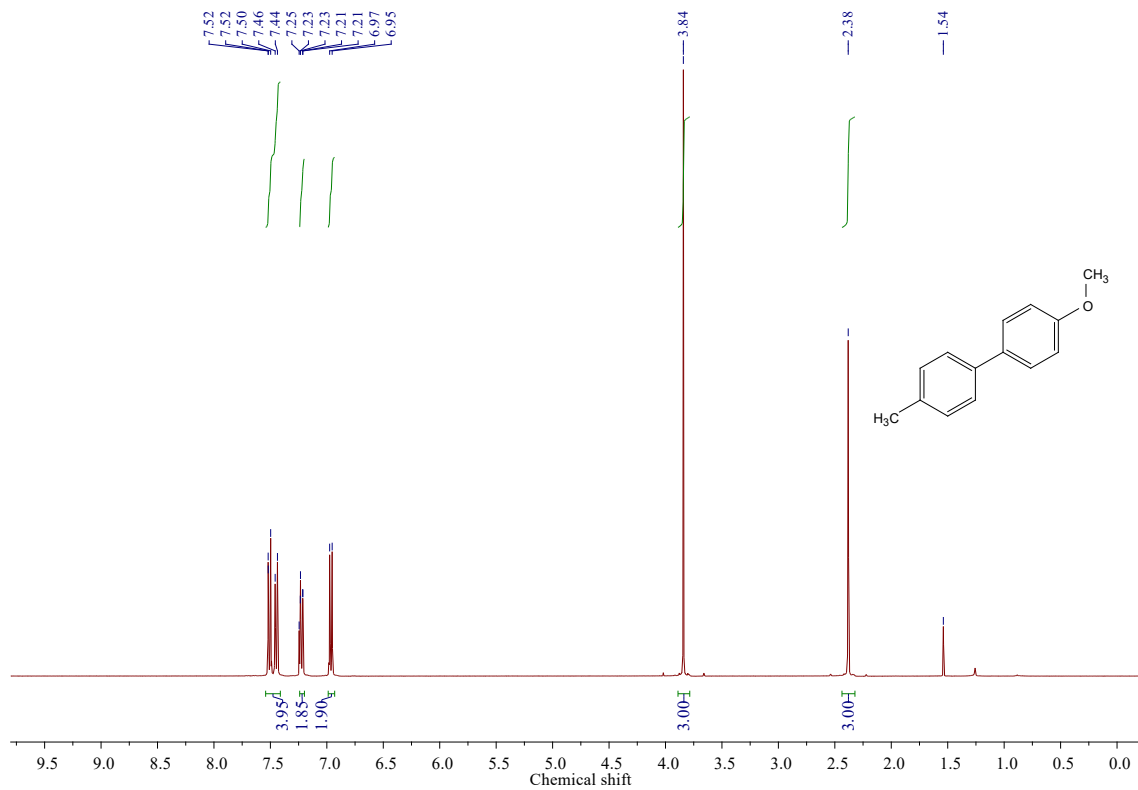
3a.



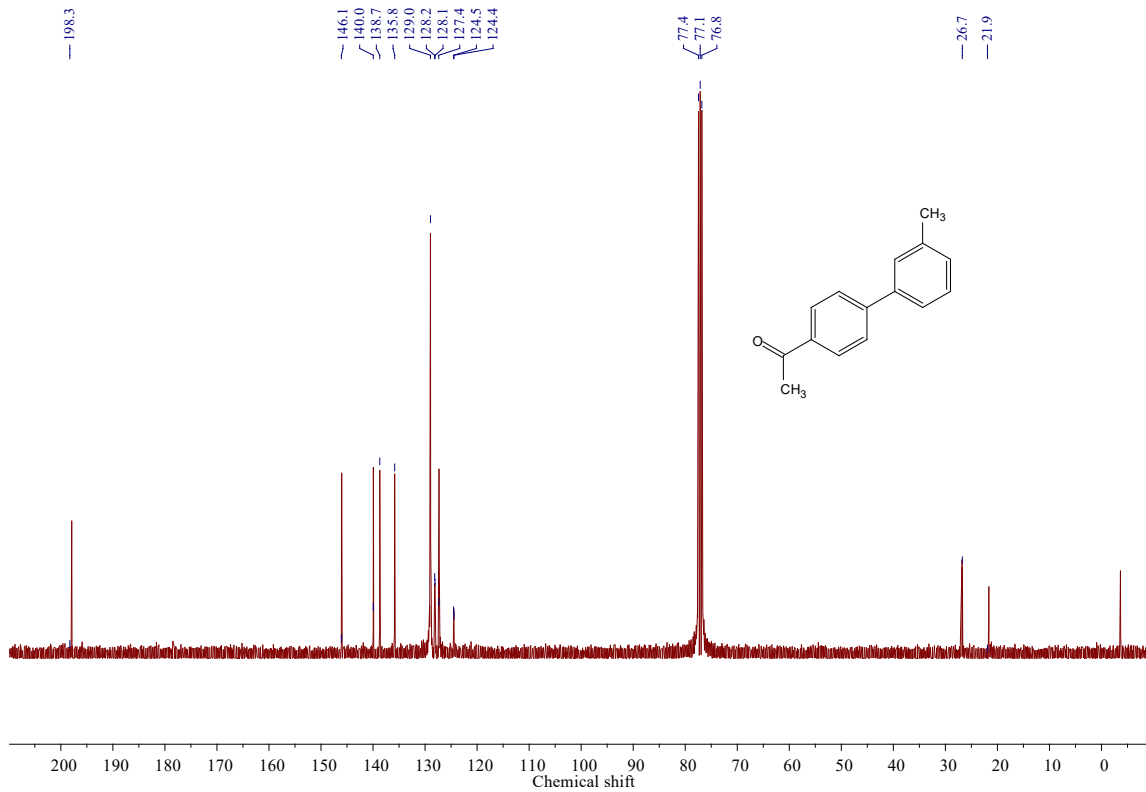
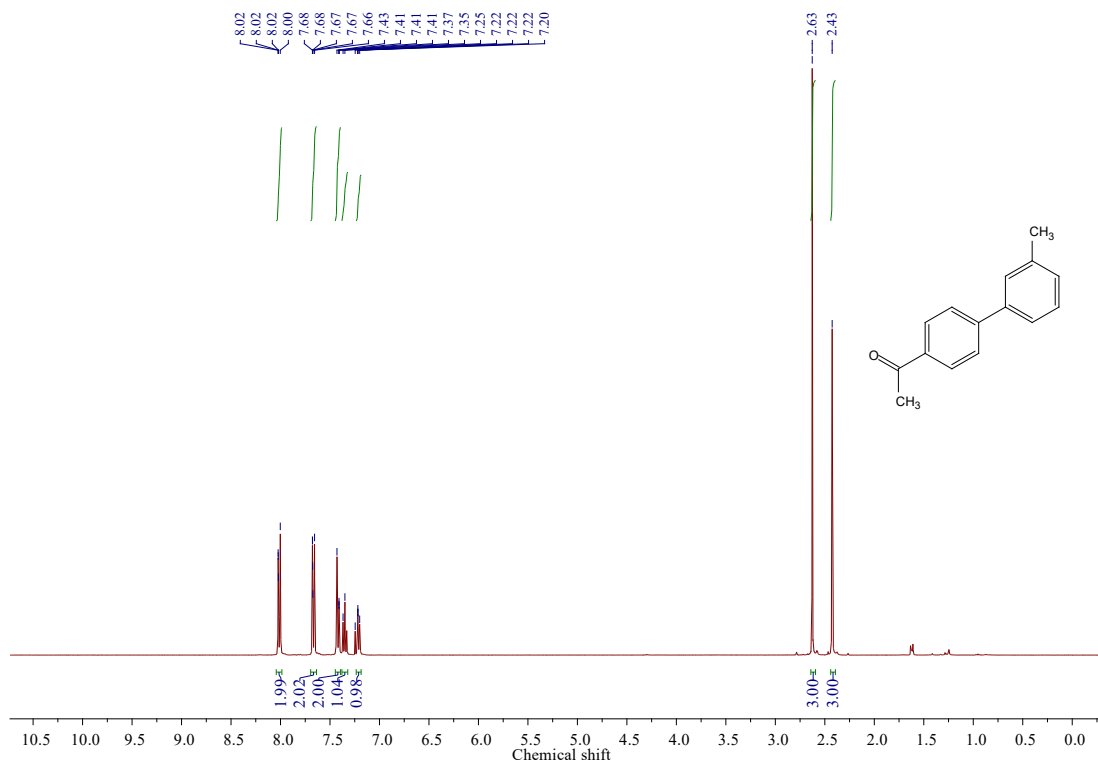
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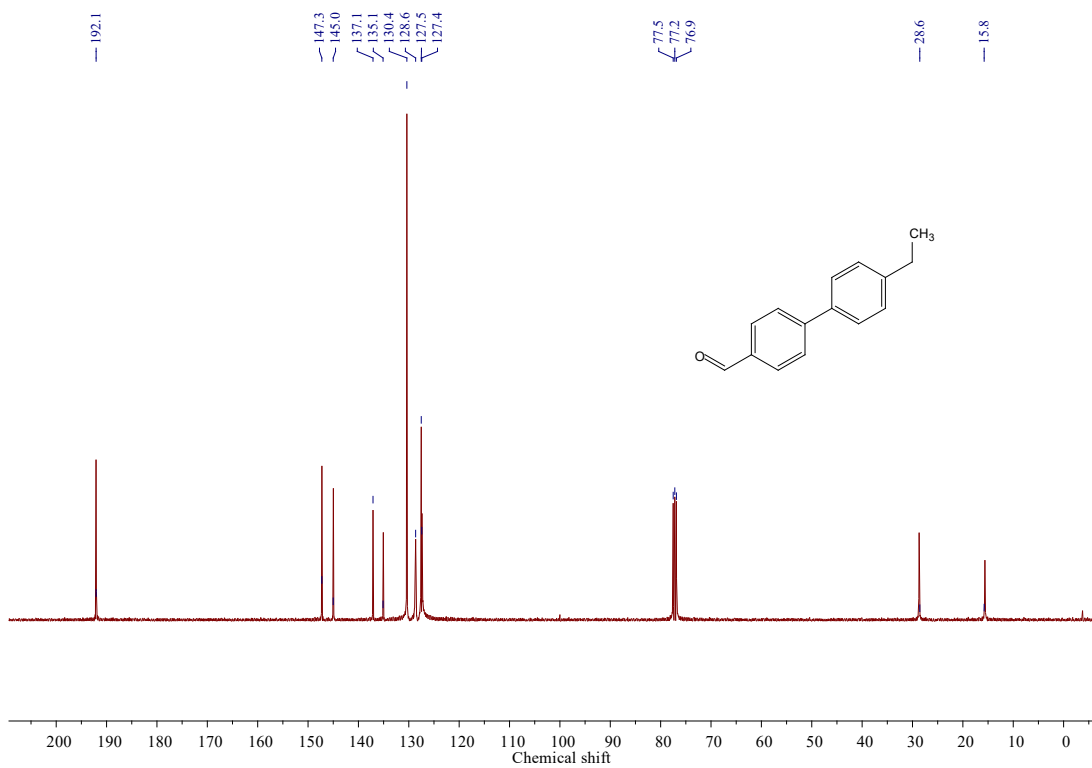
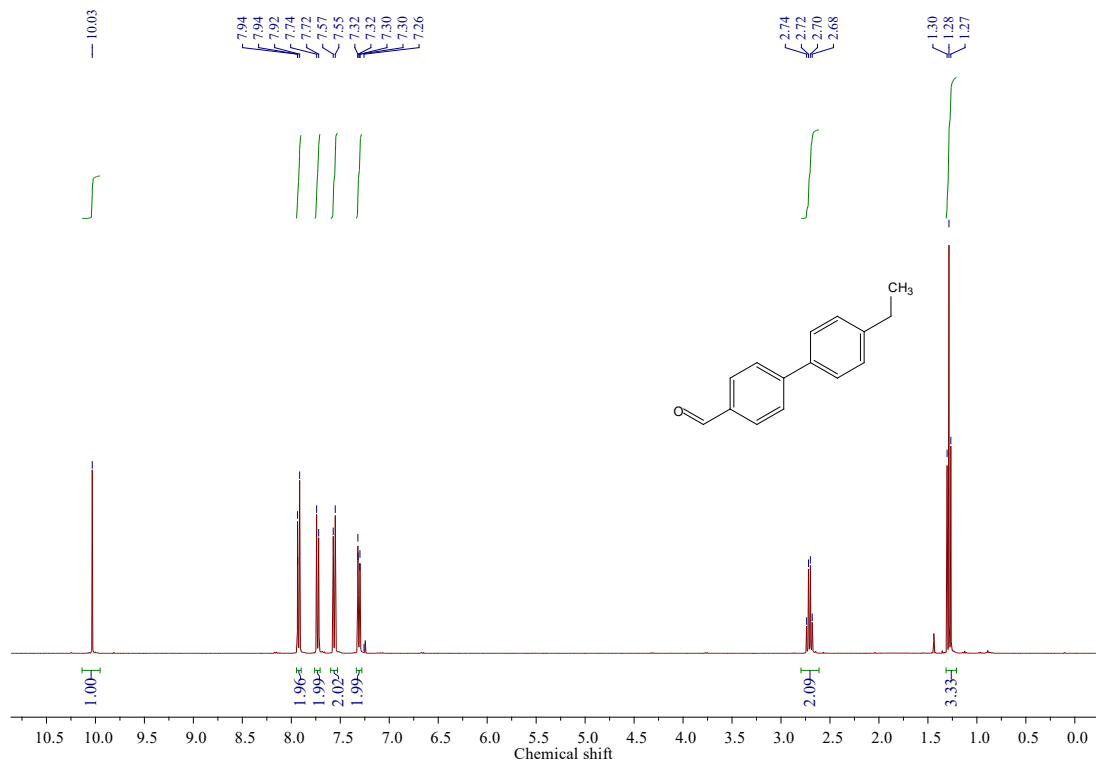
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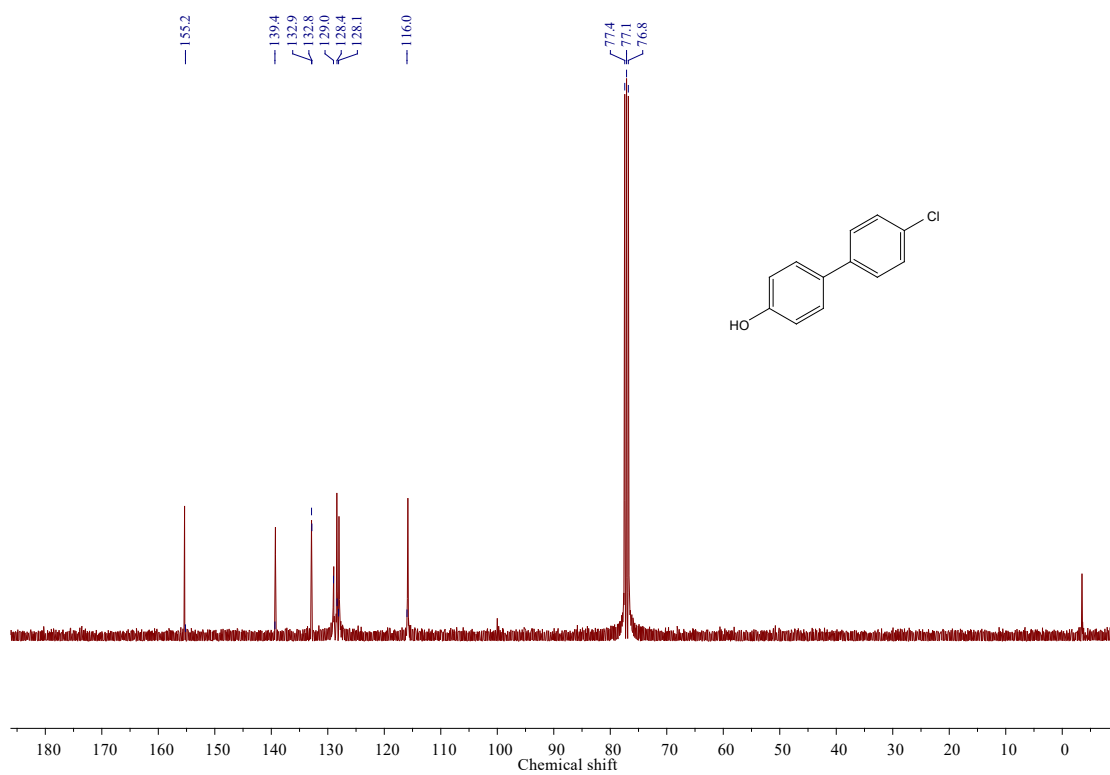
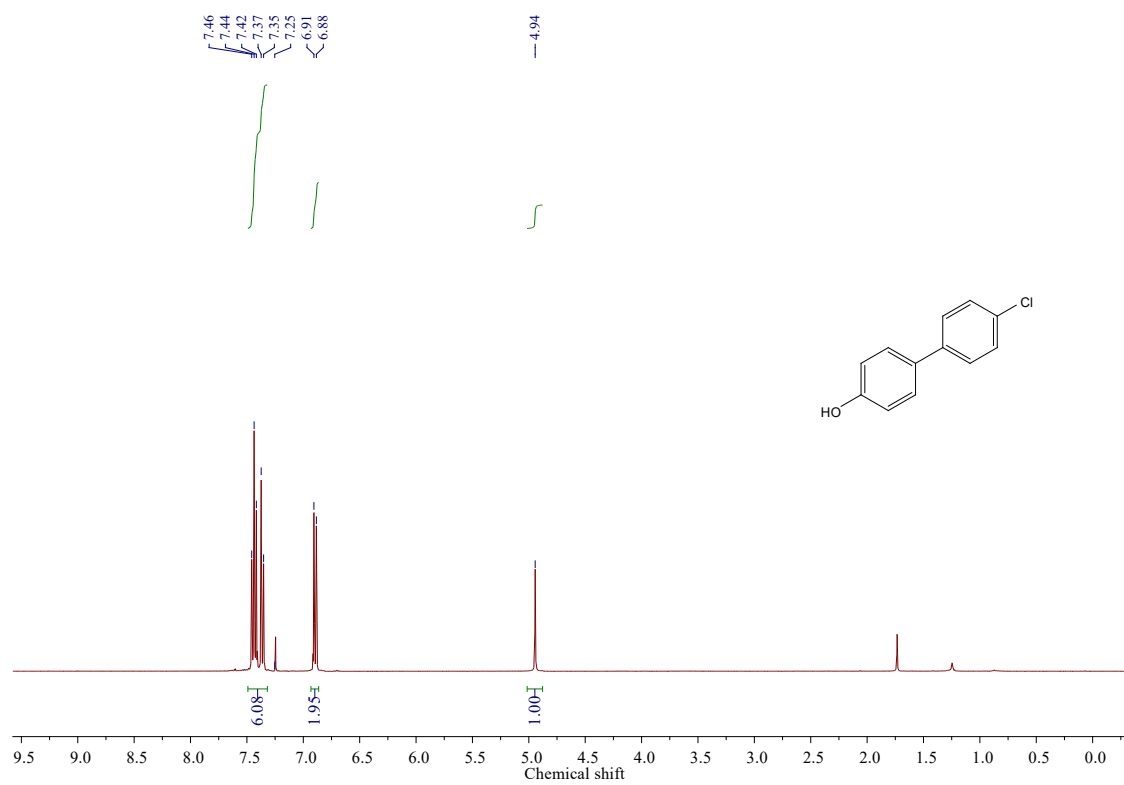
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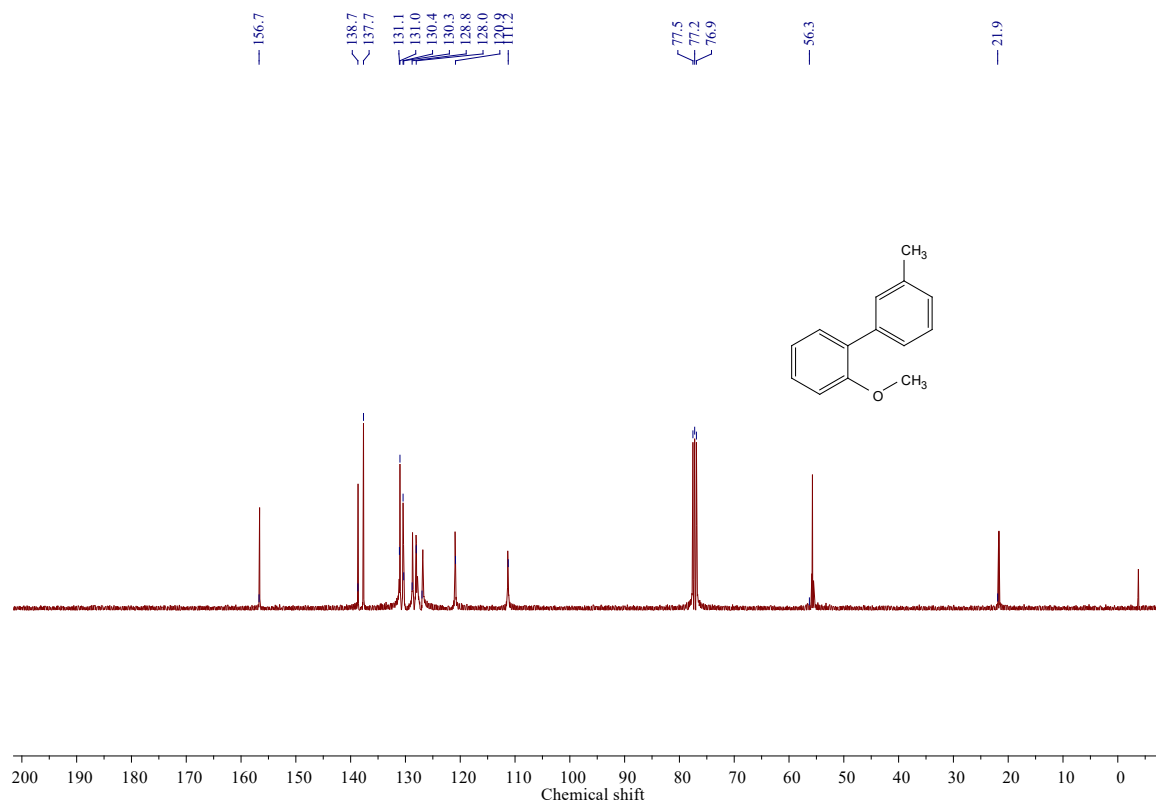
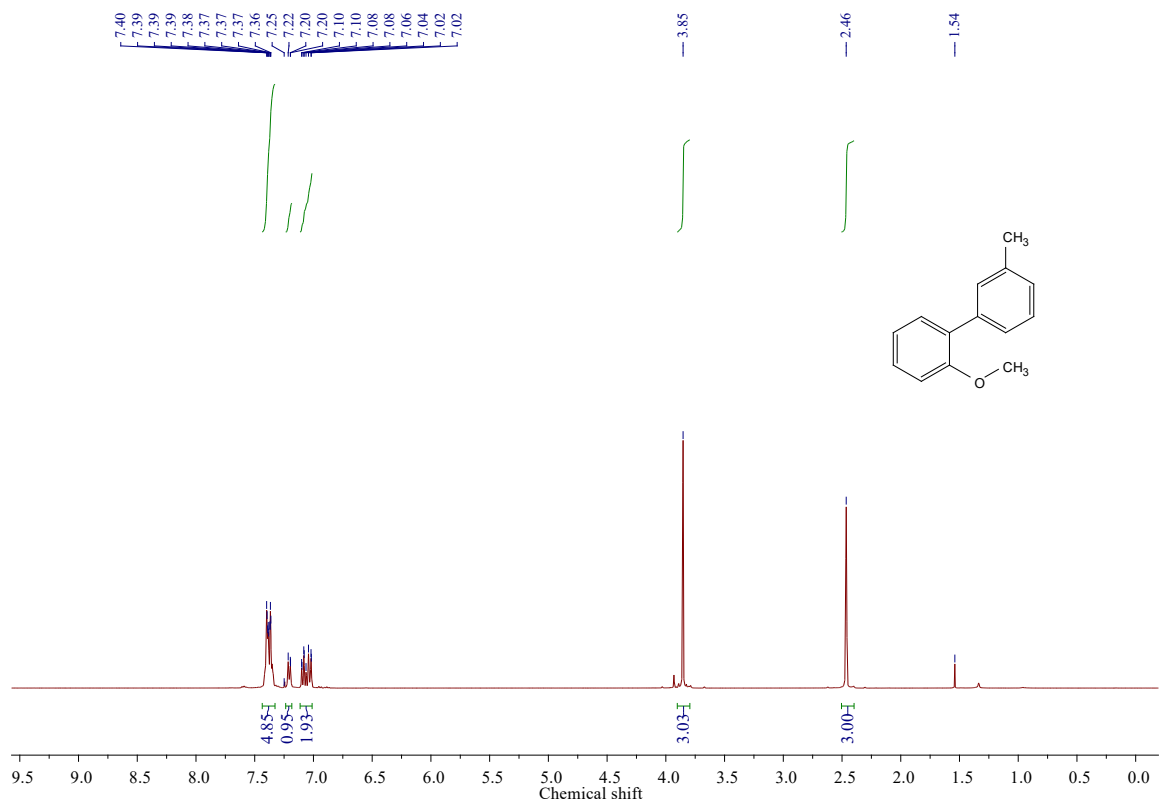
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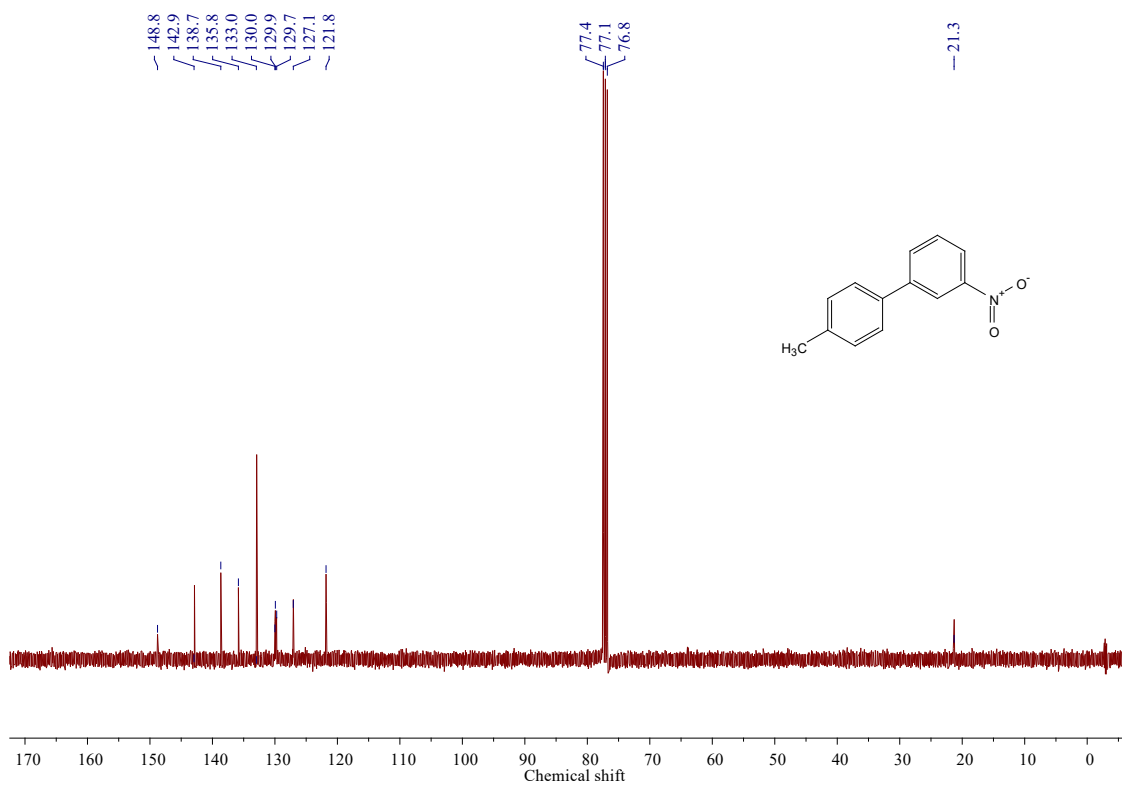
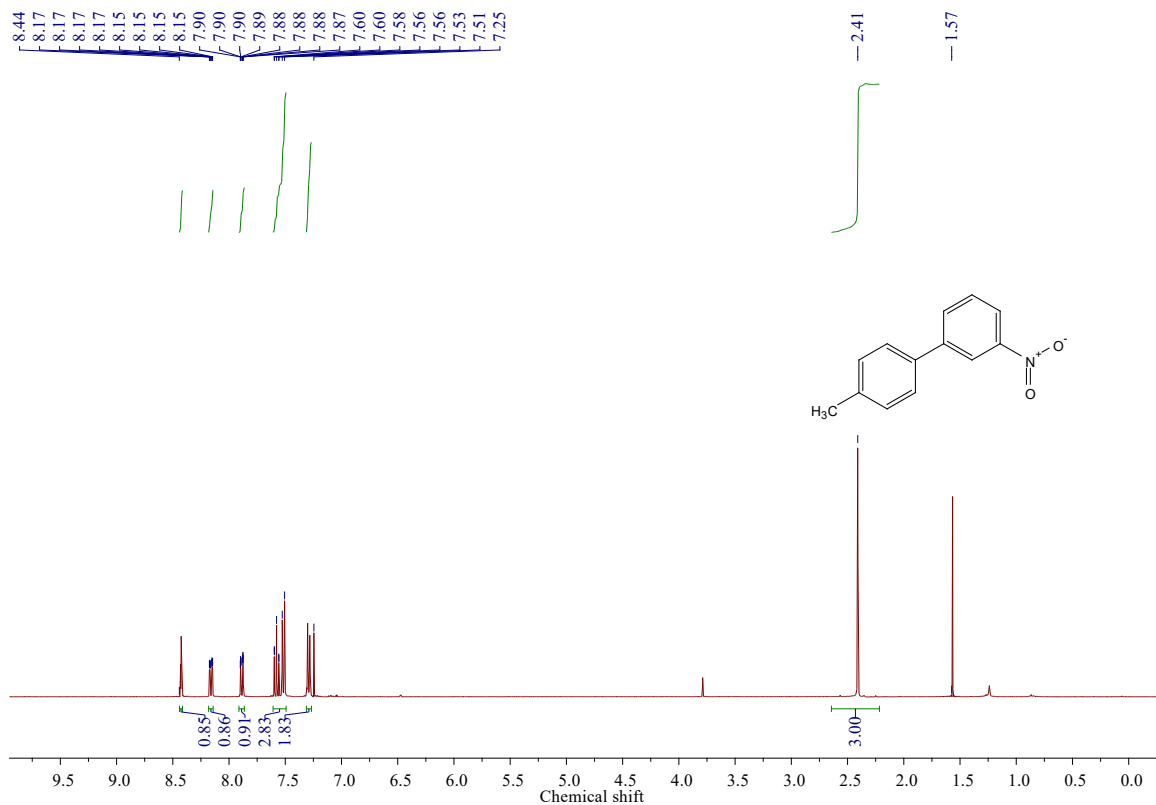
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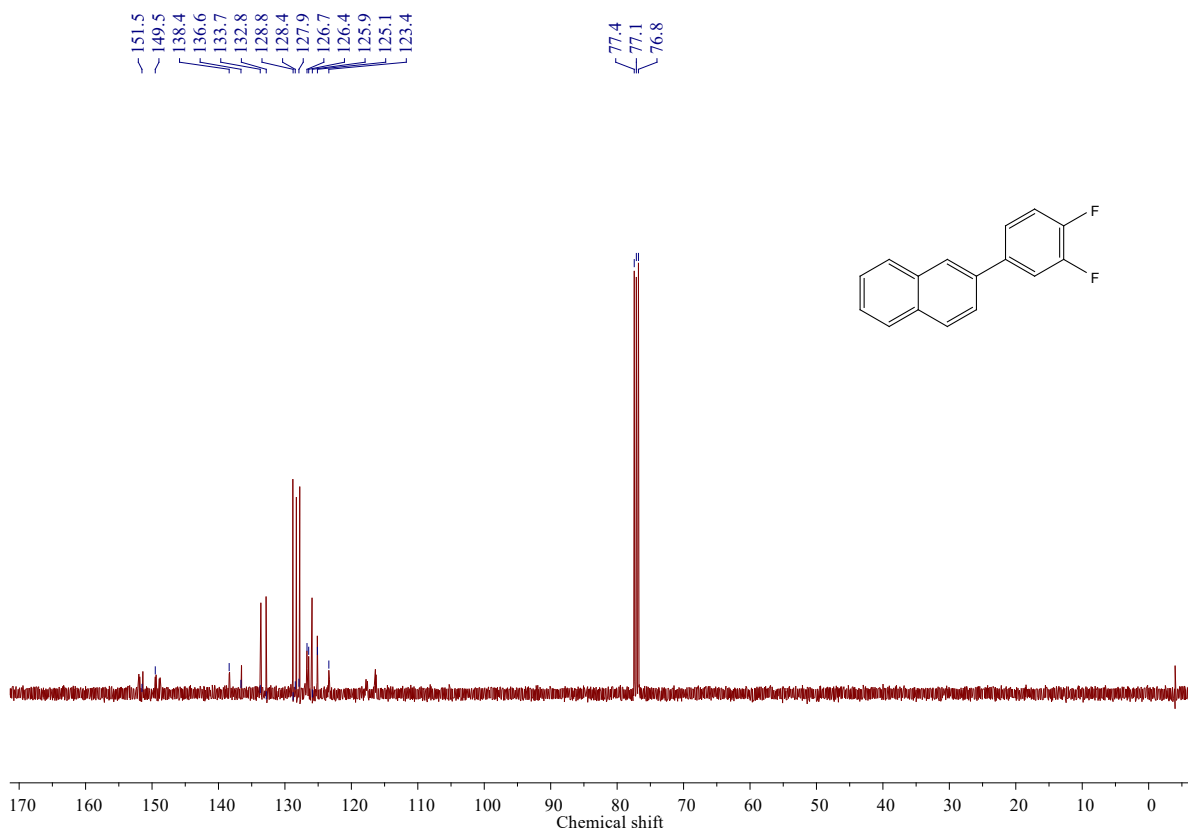
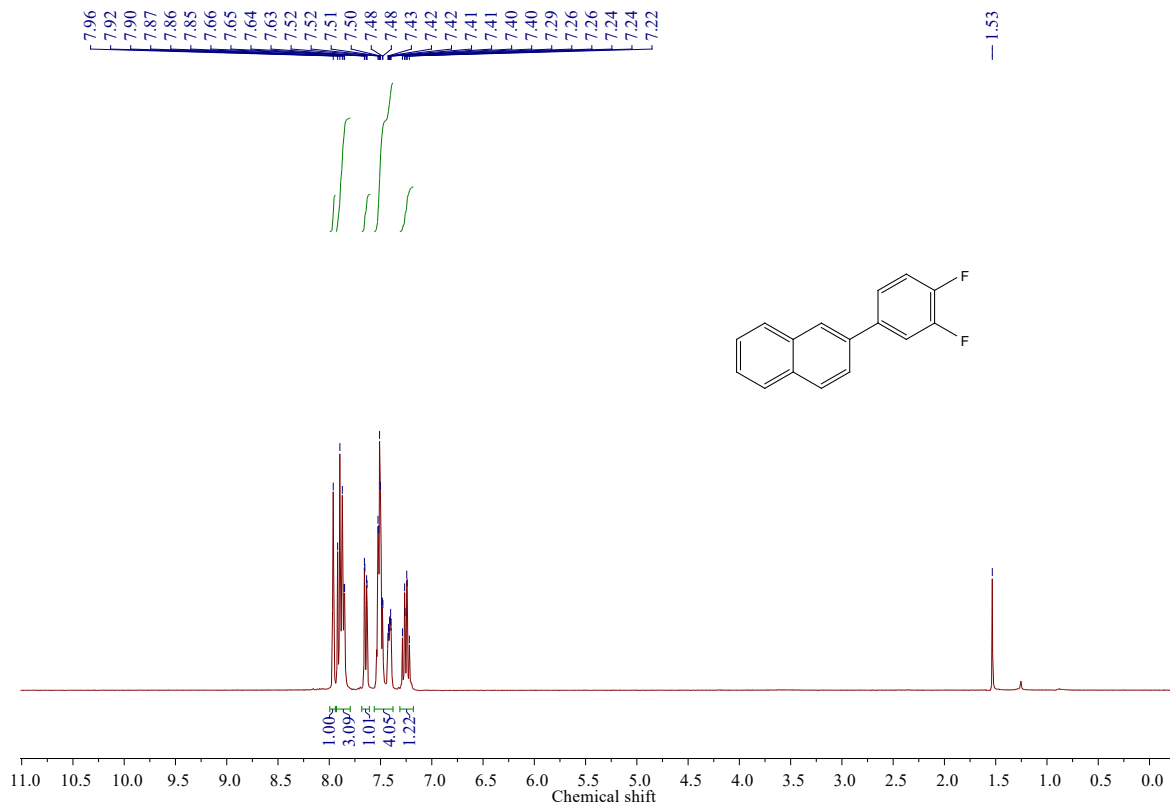
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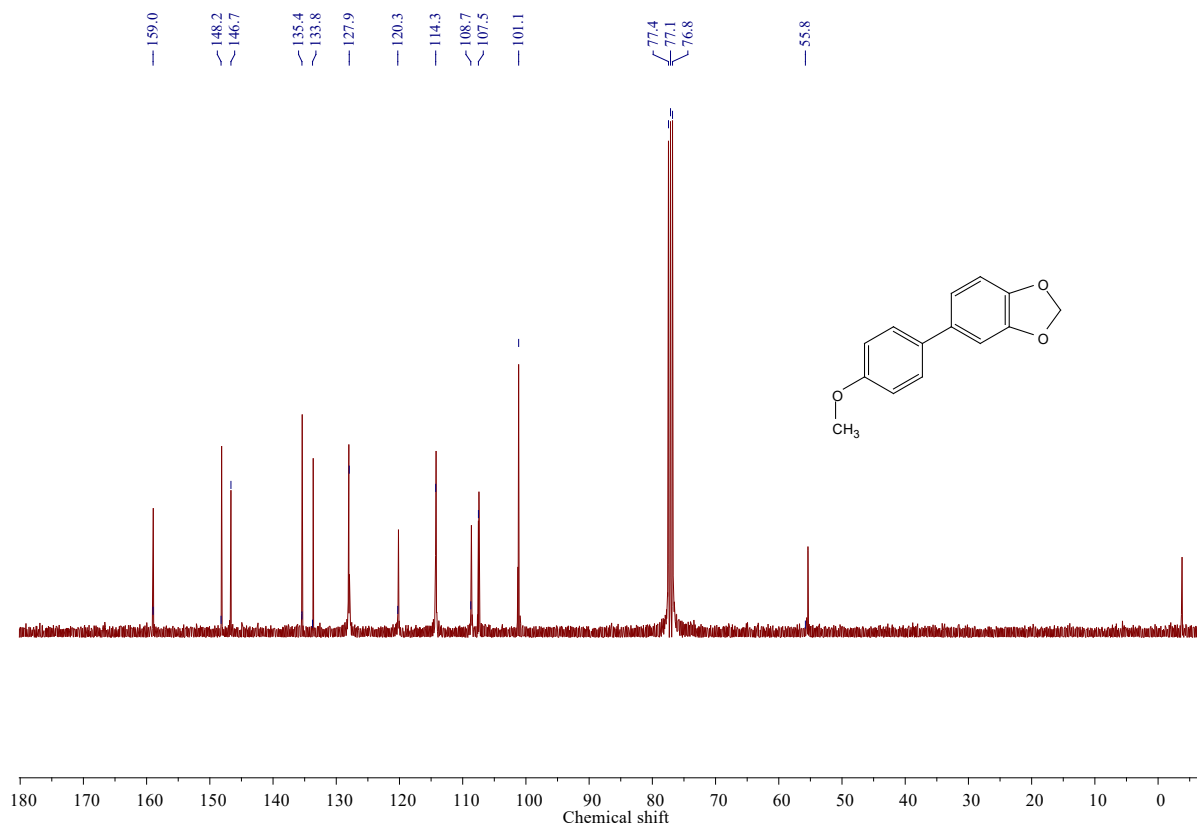
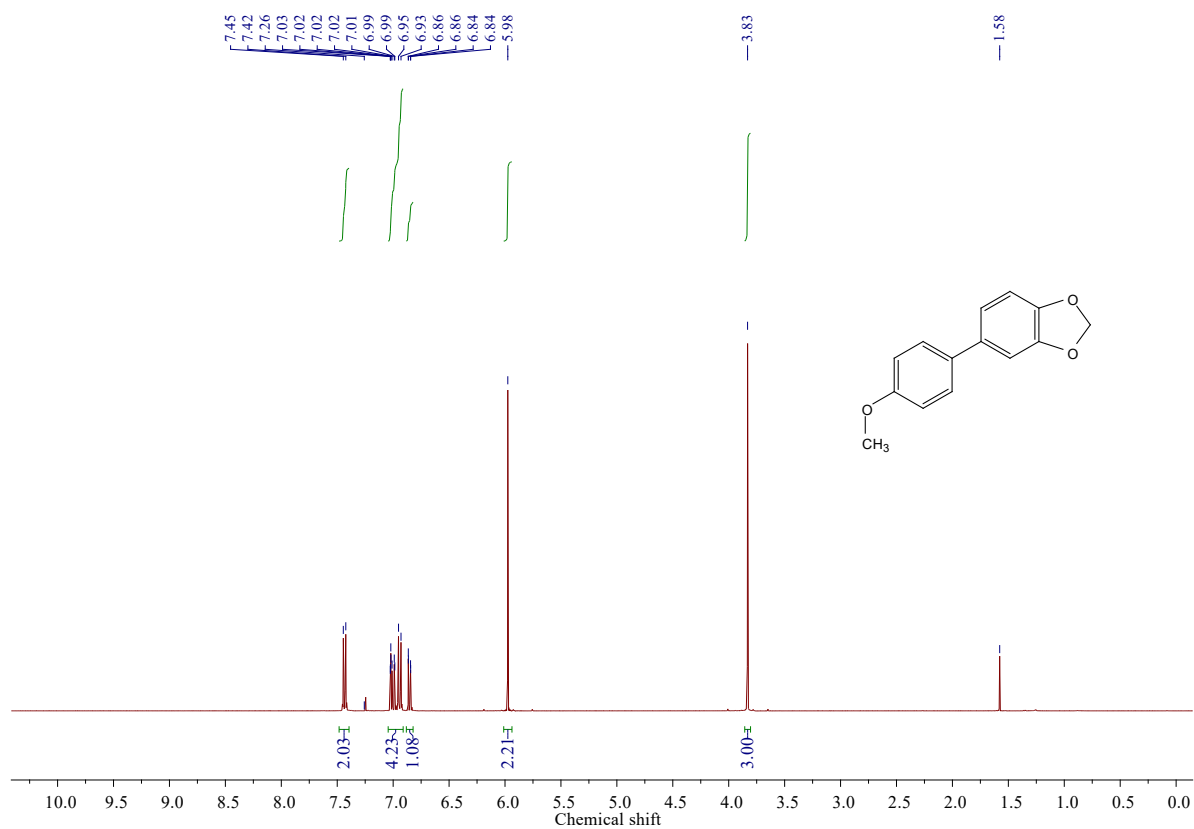
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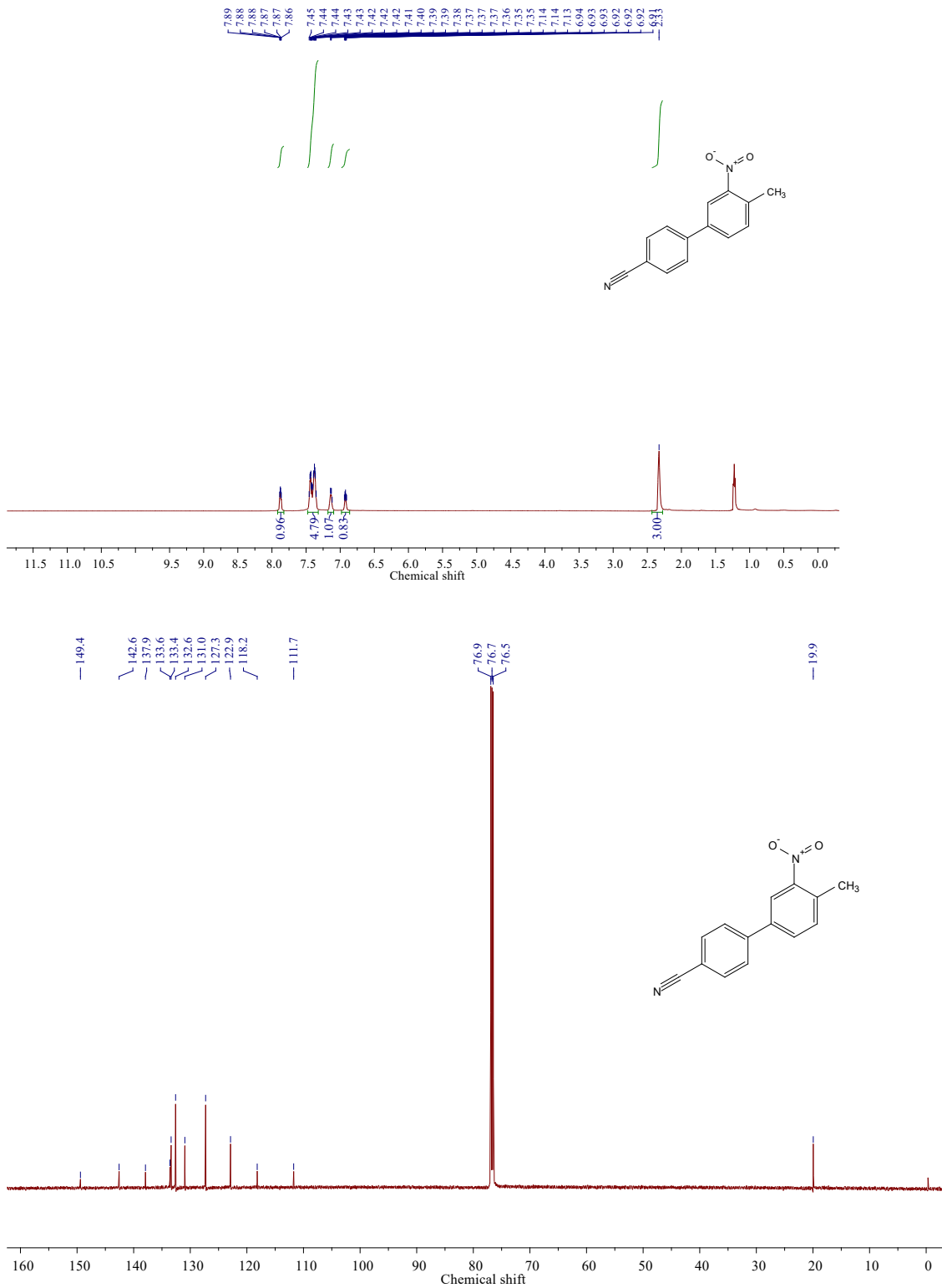
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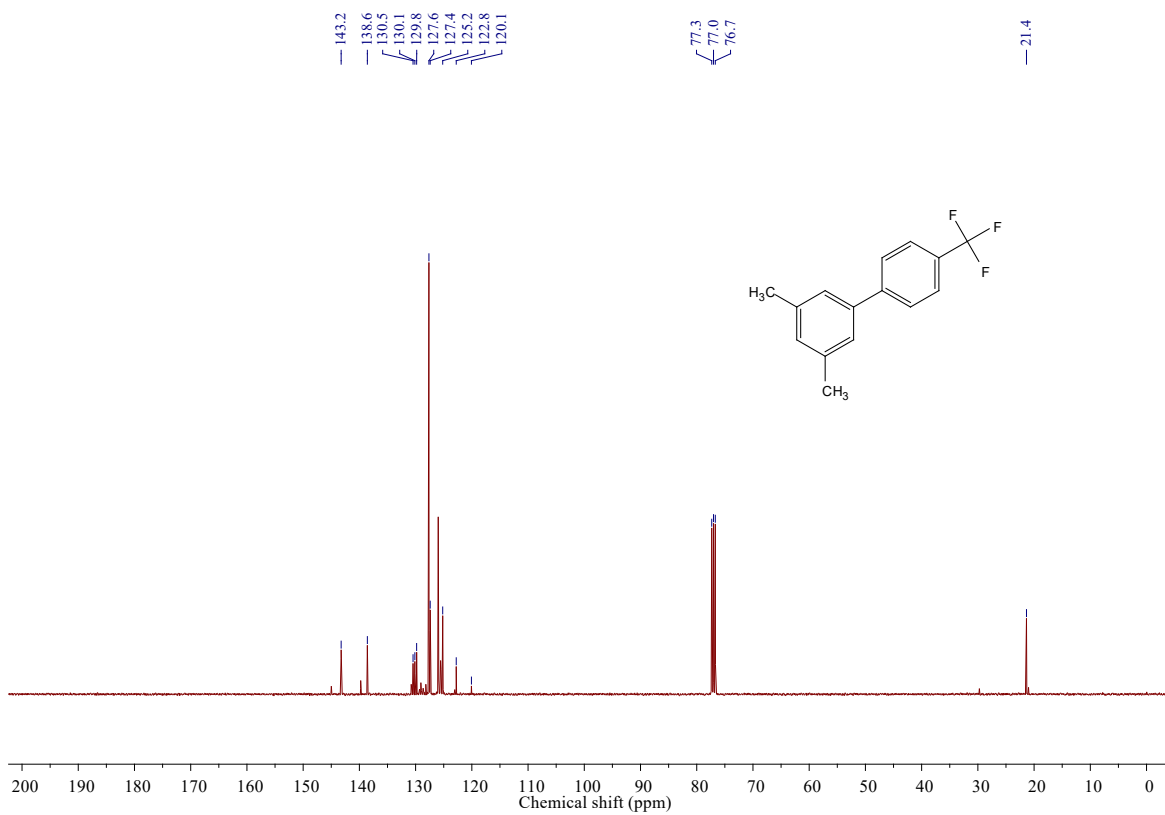
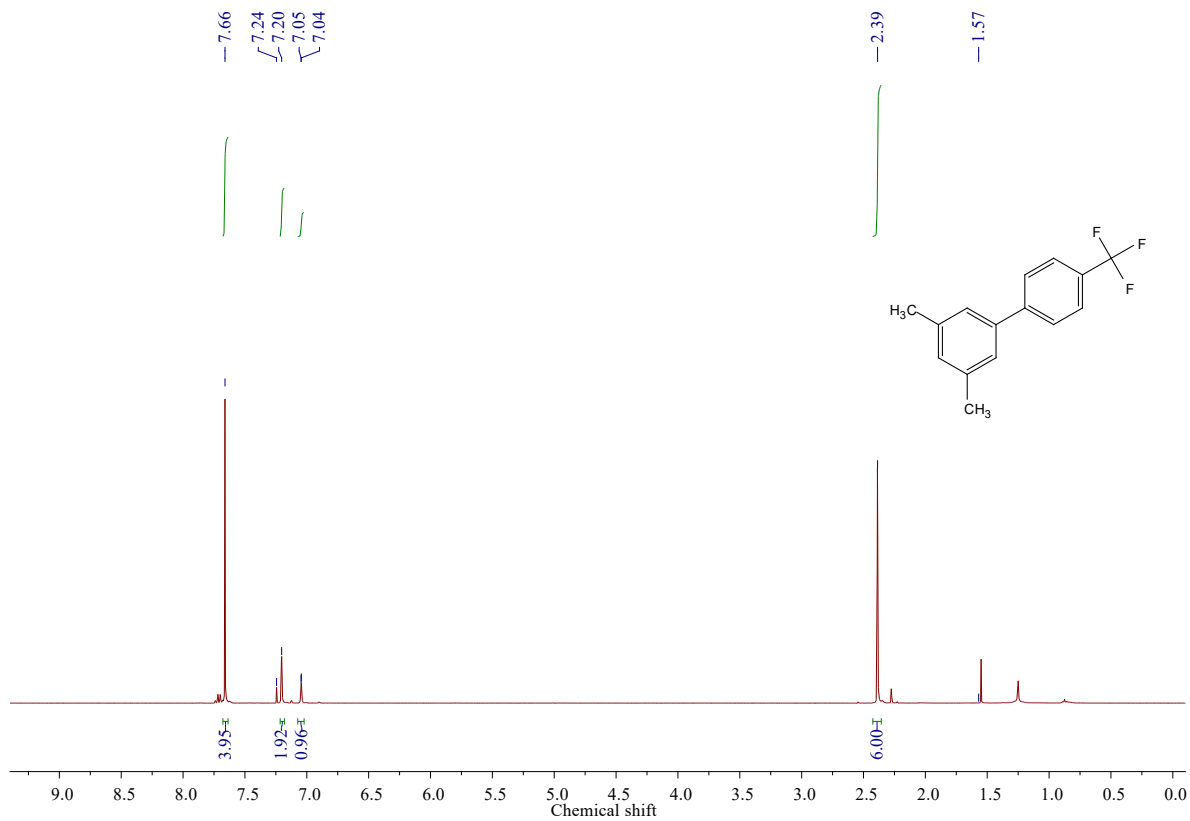
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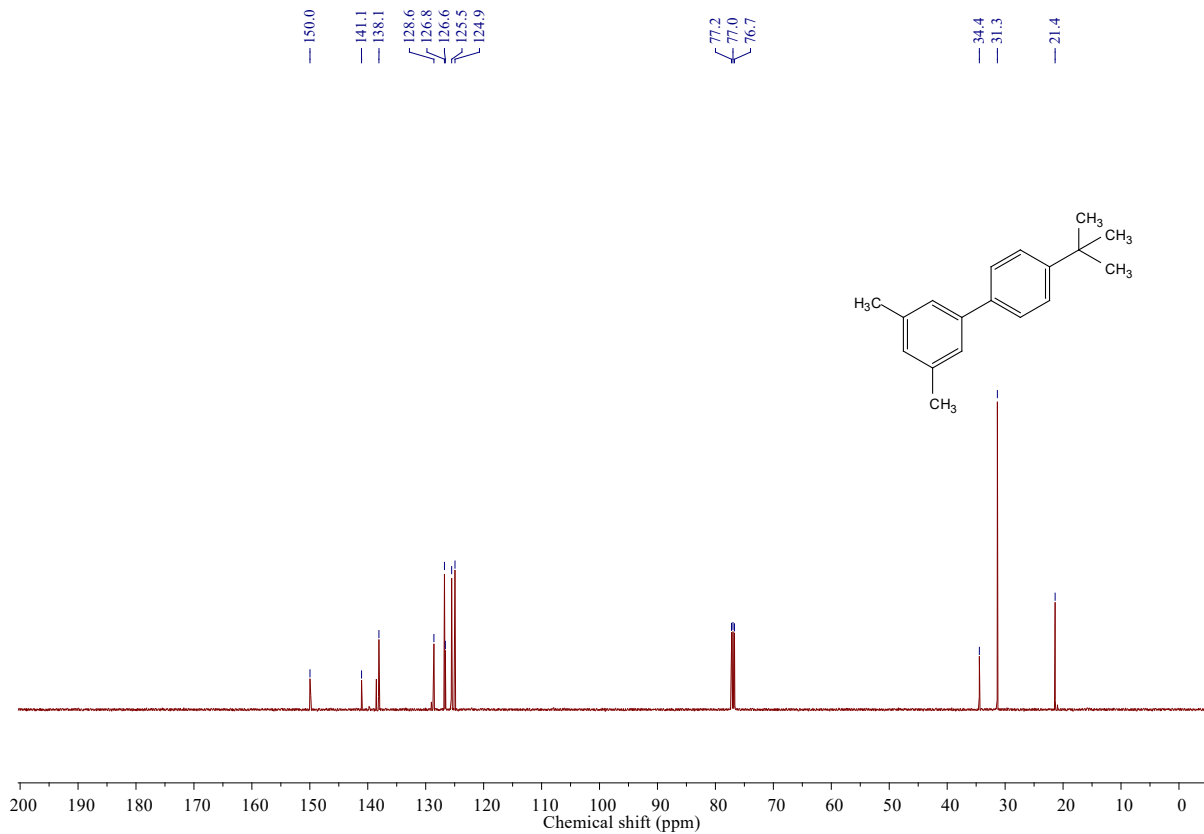
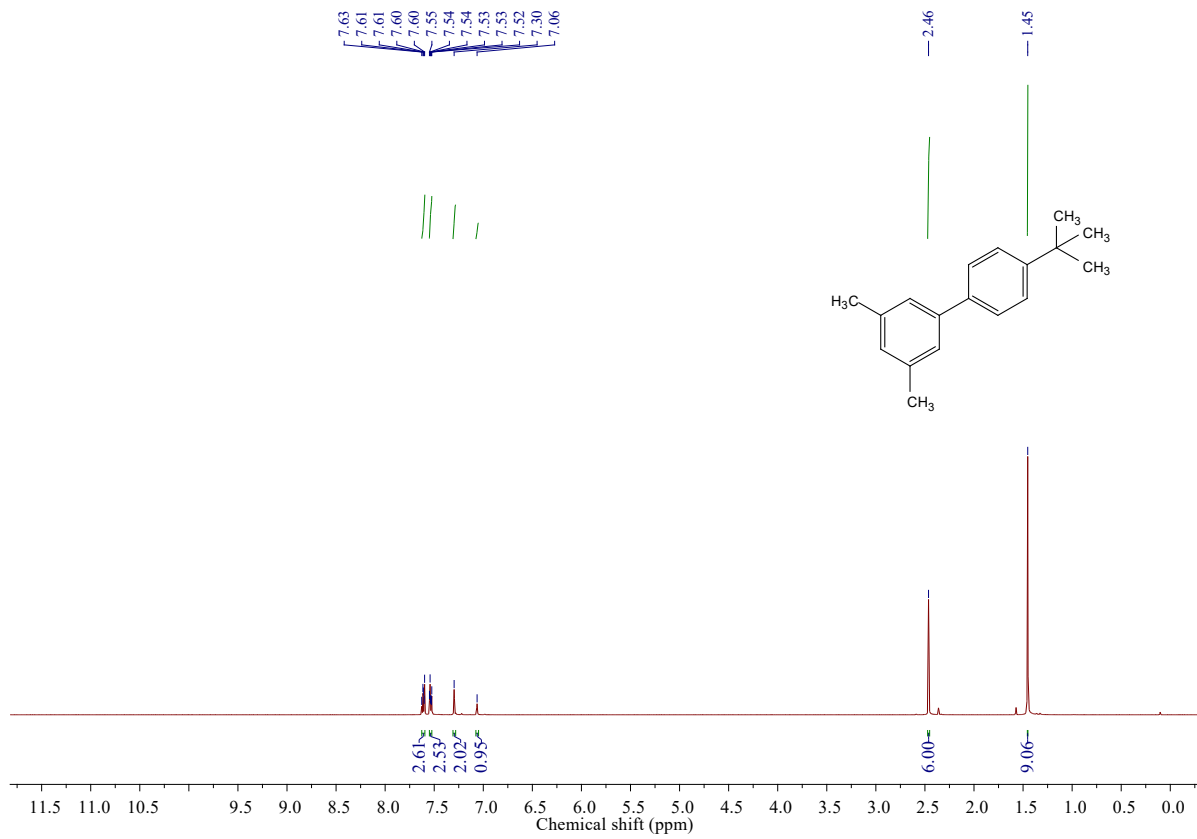
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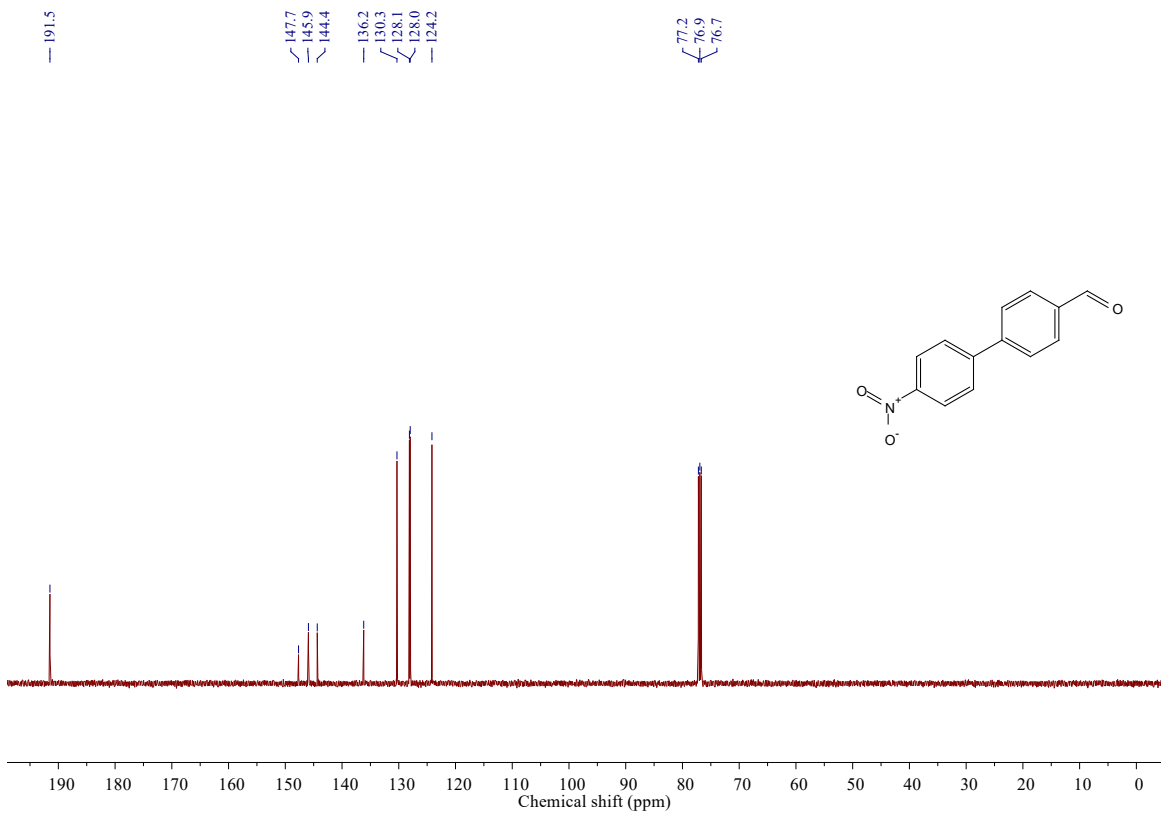
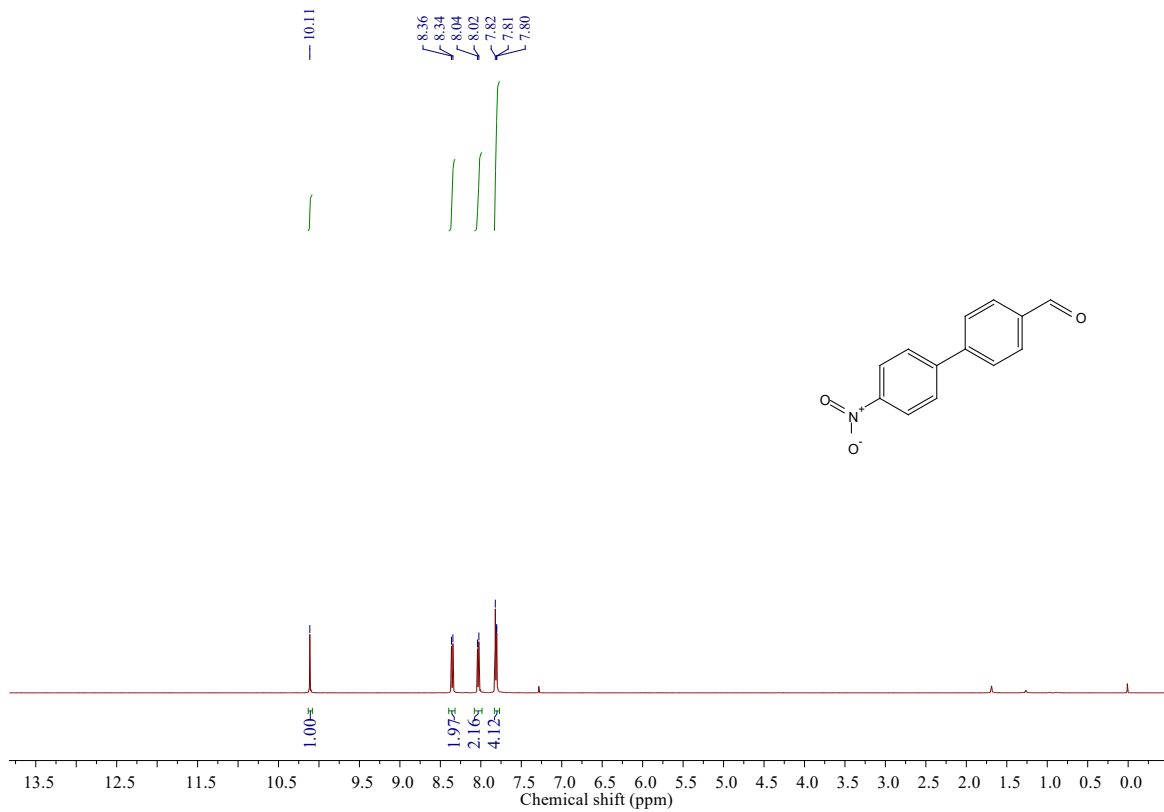
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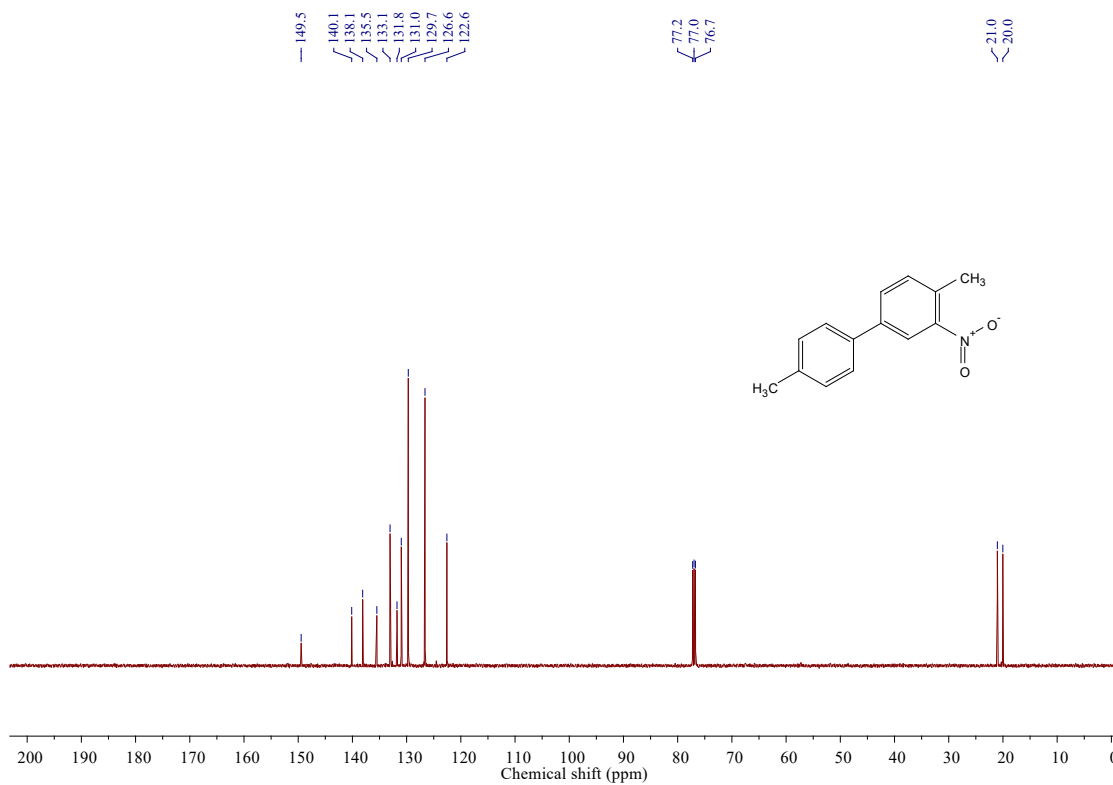
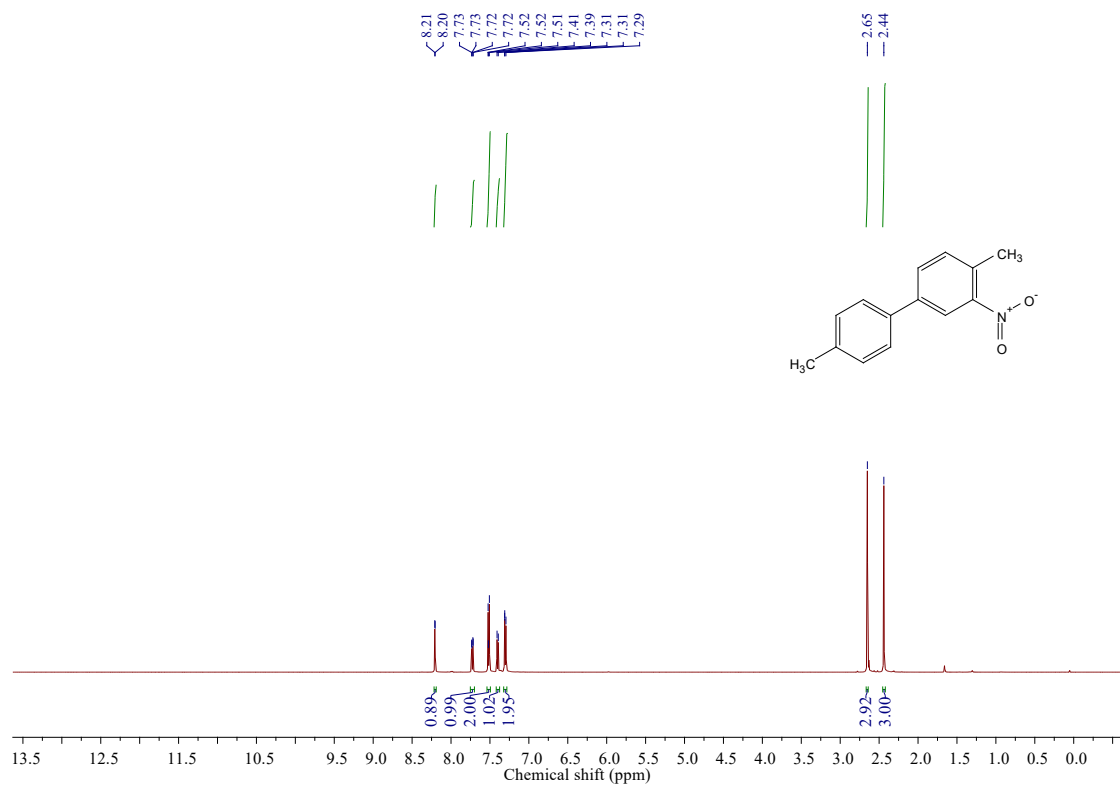
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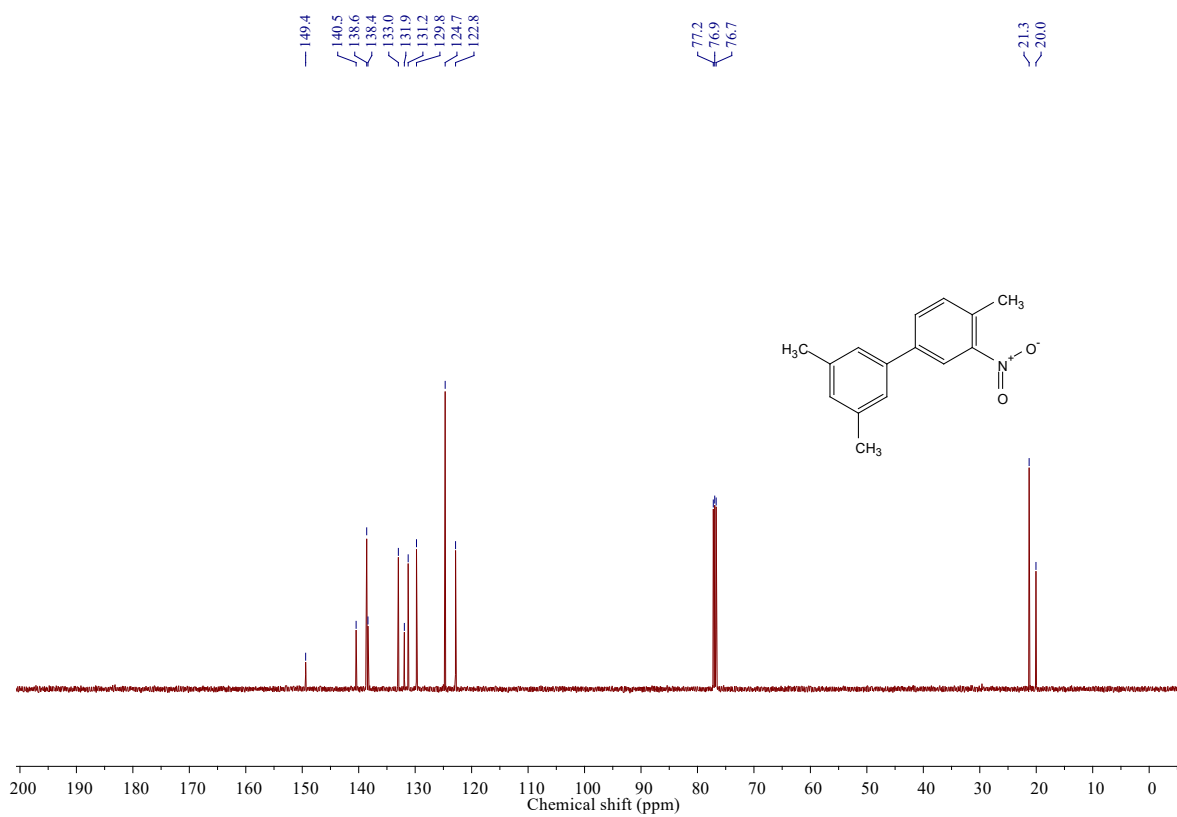
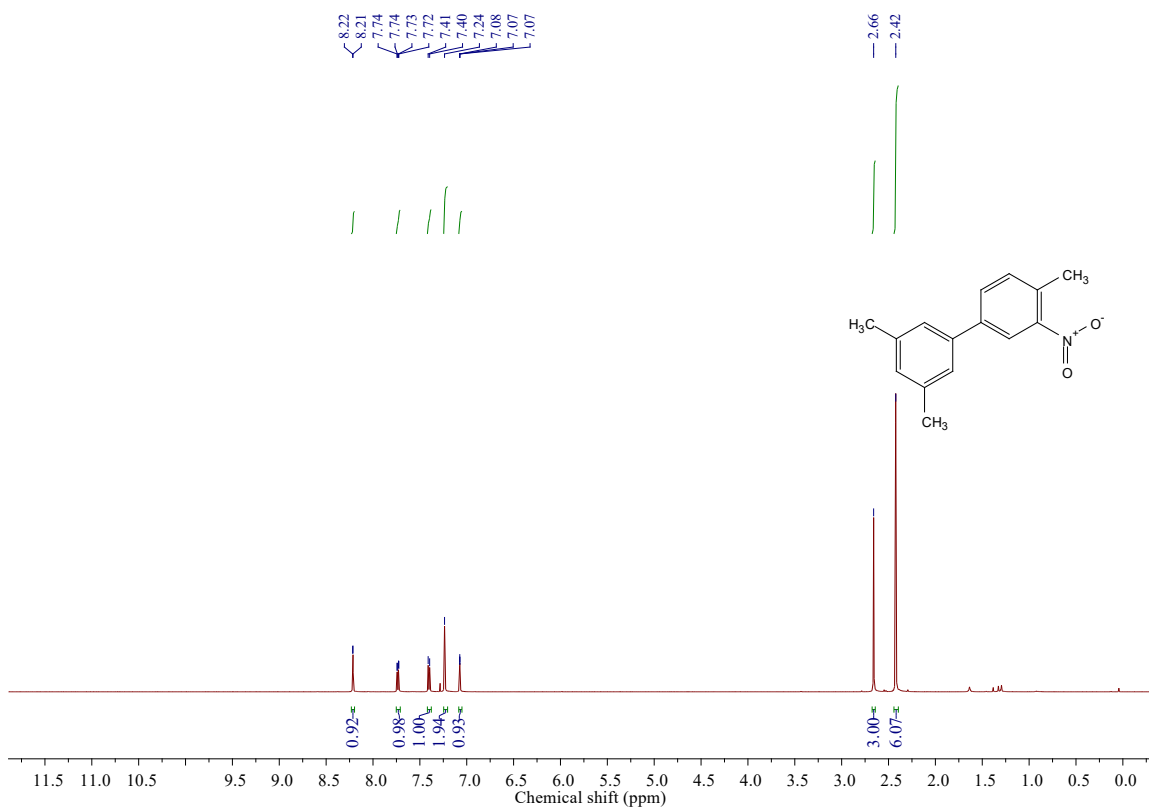
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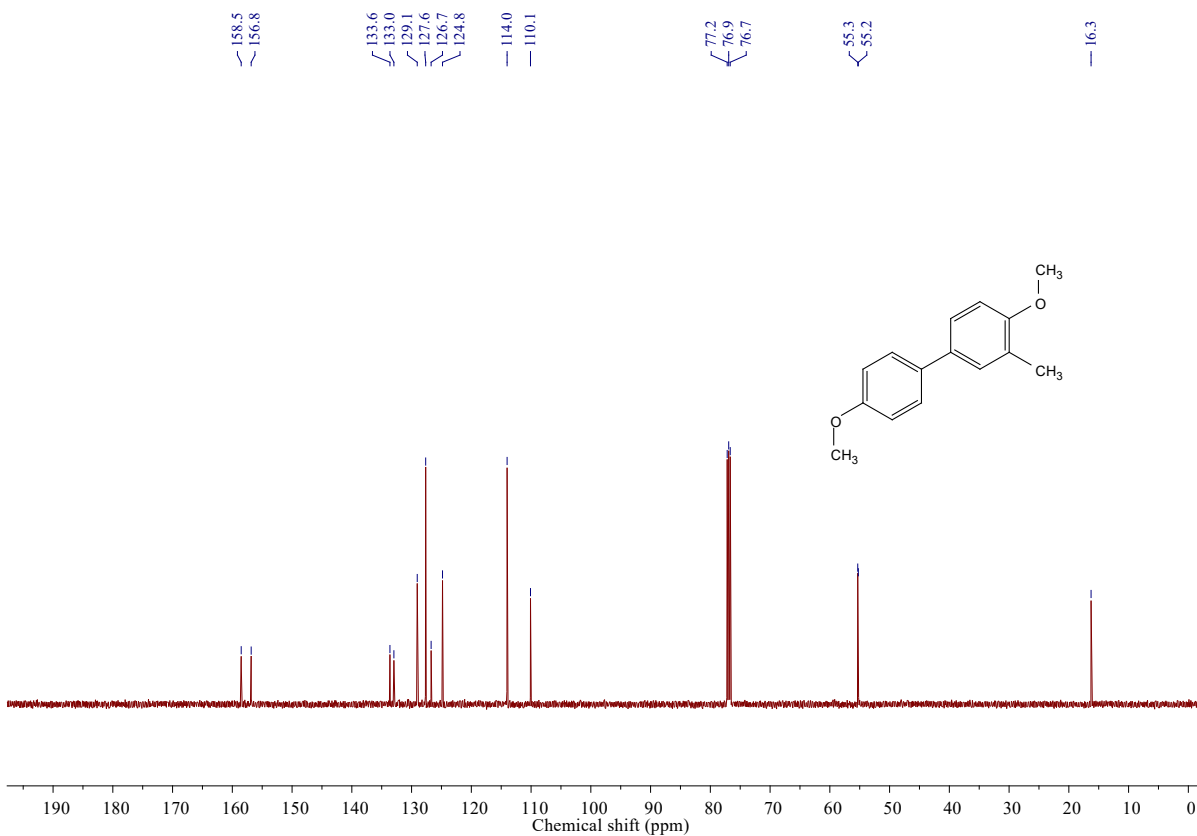
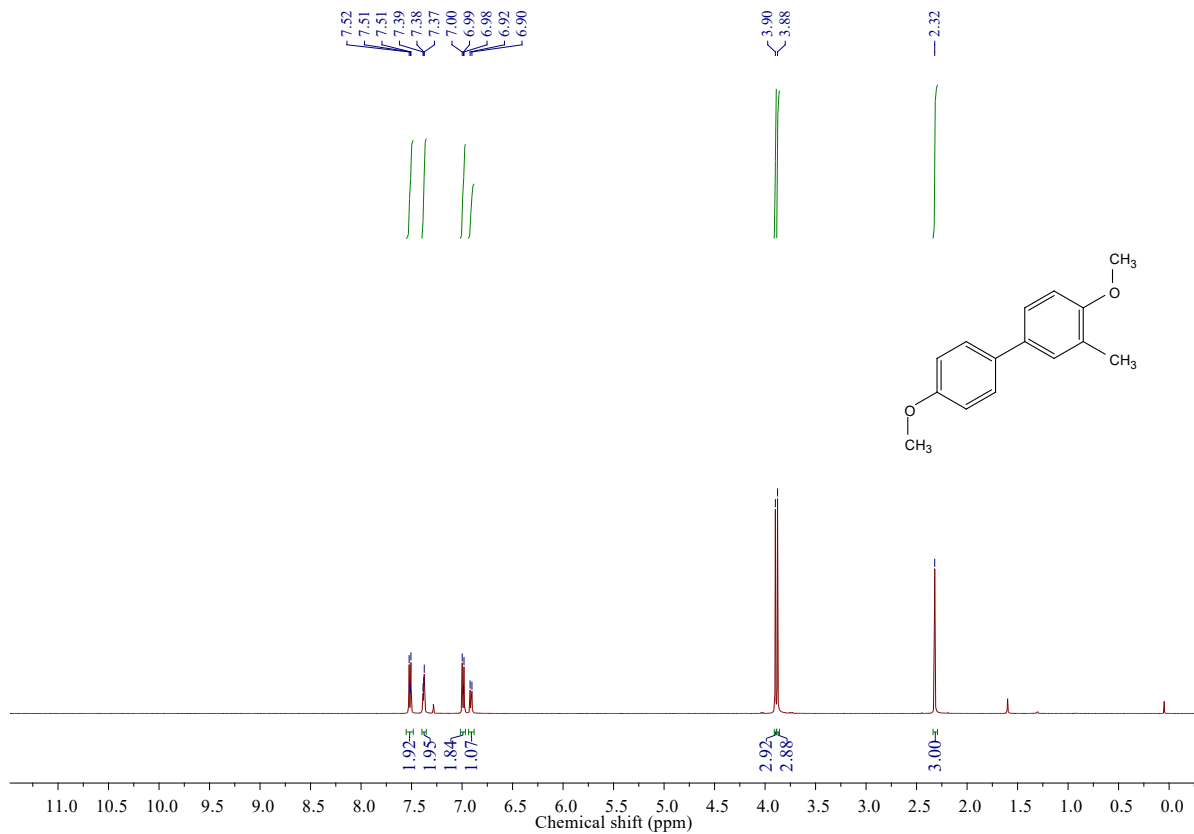
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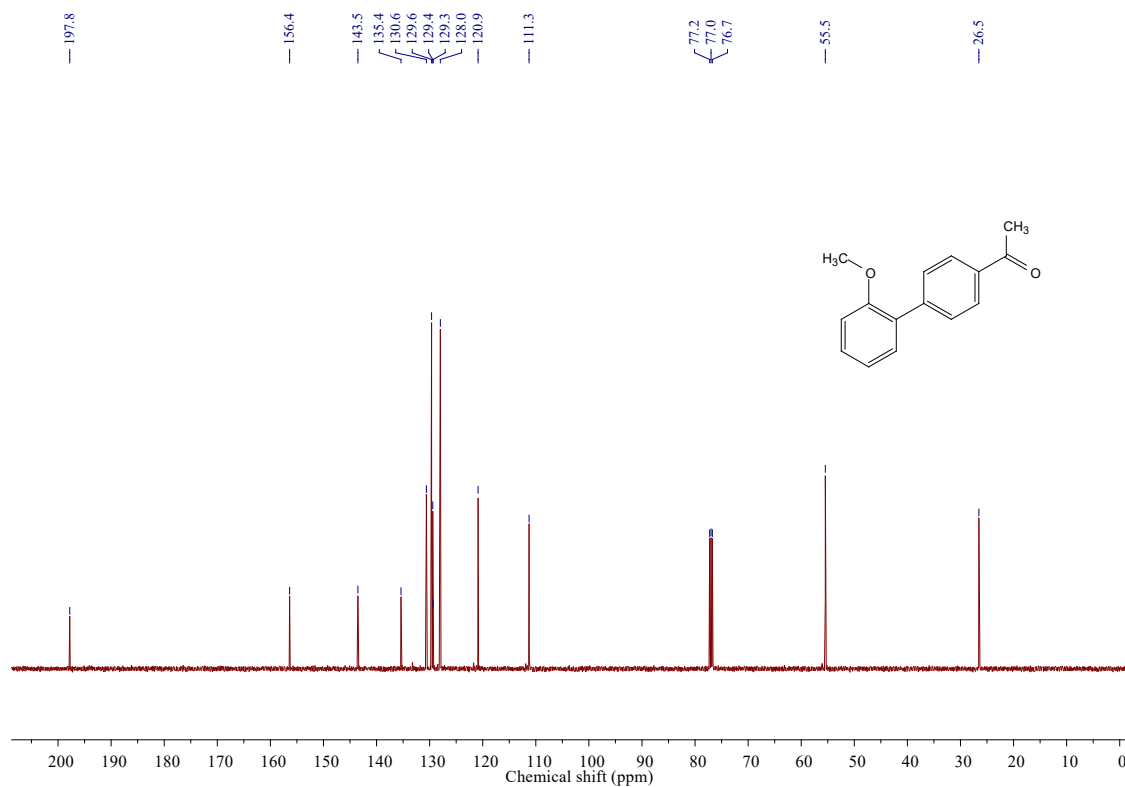
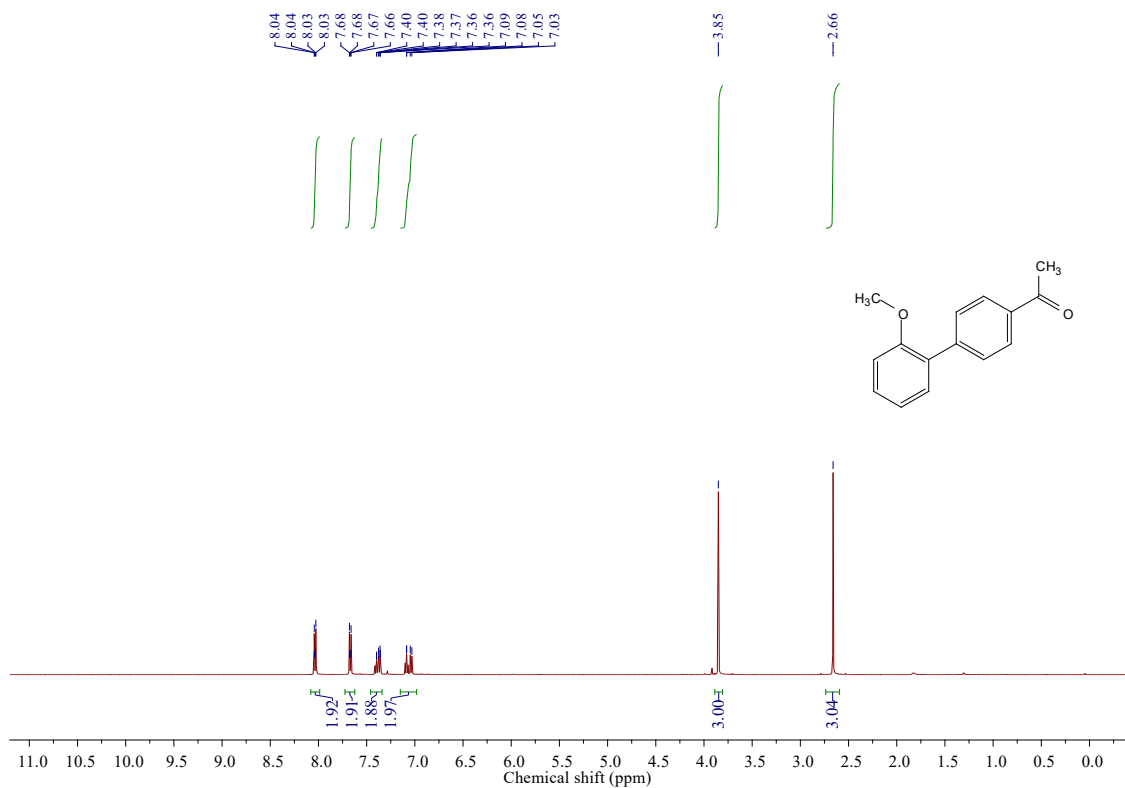
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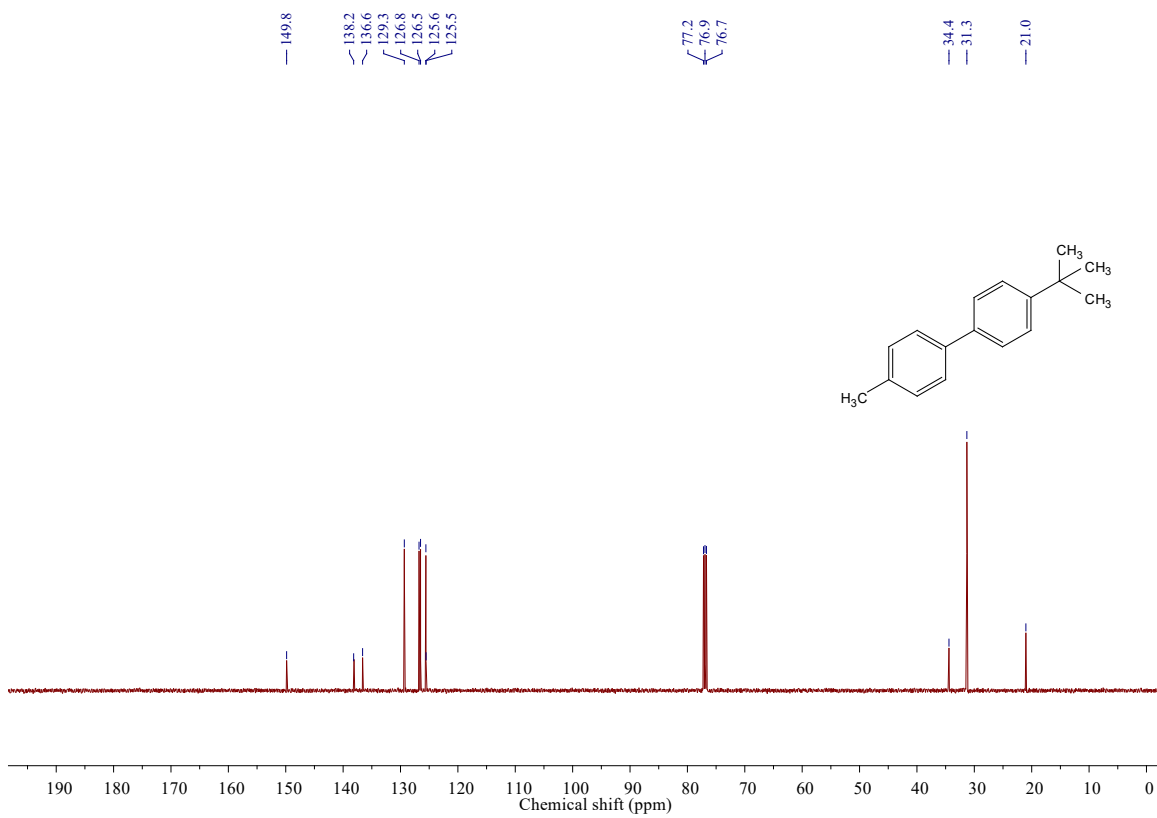
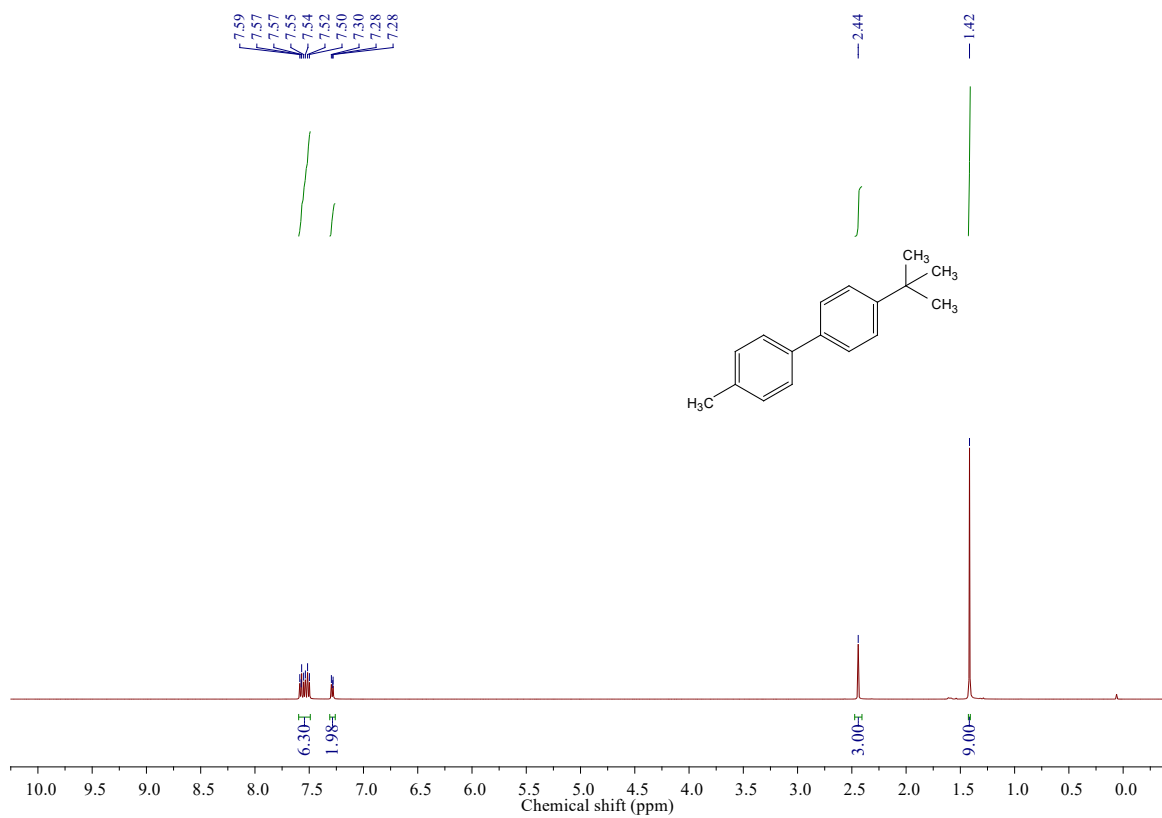
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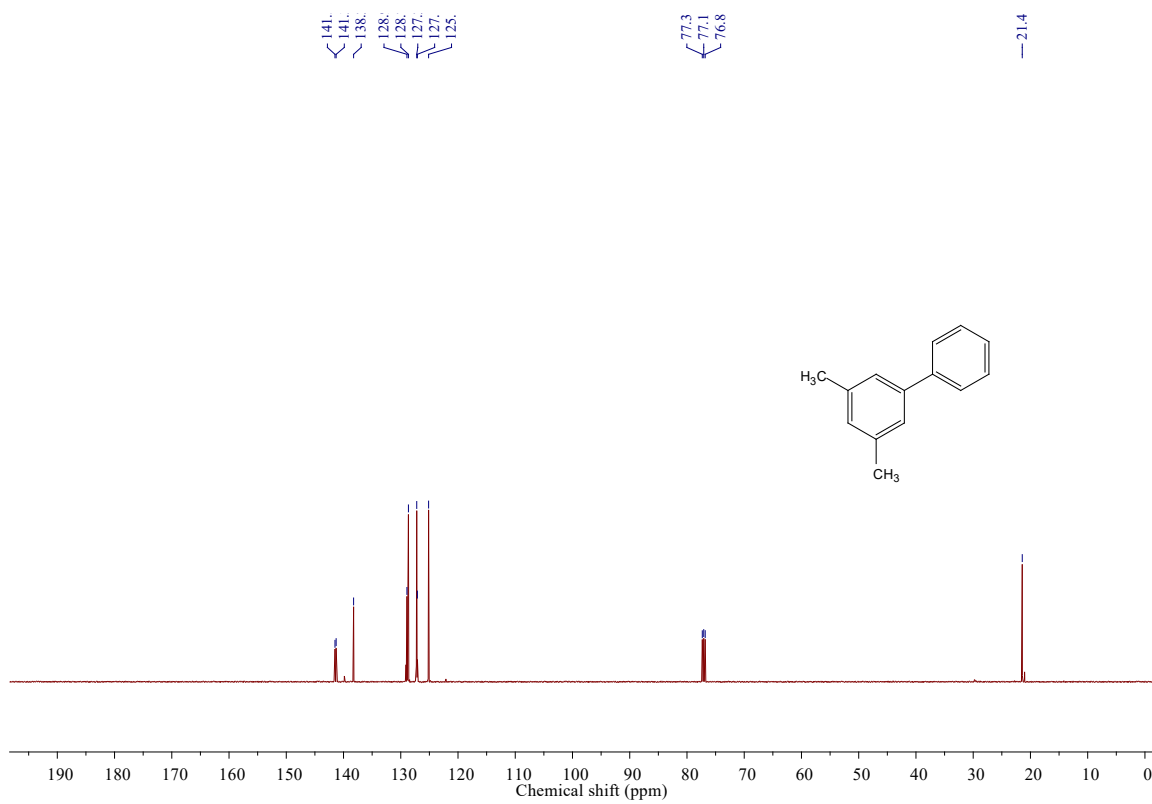
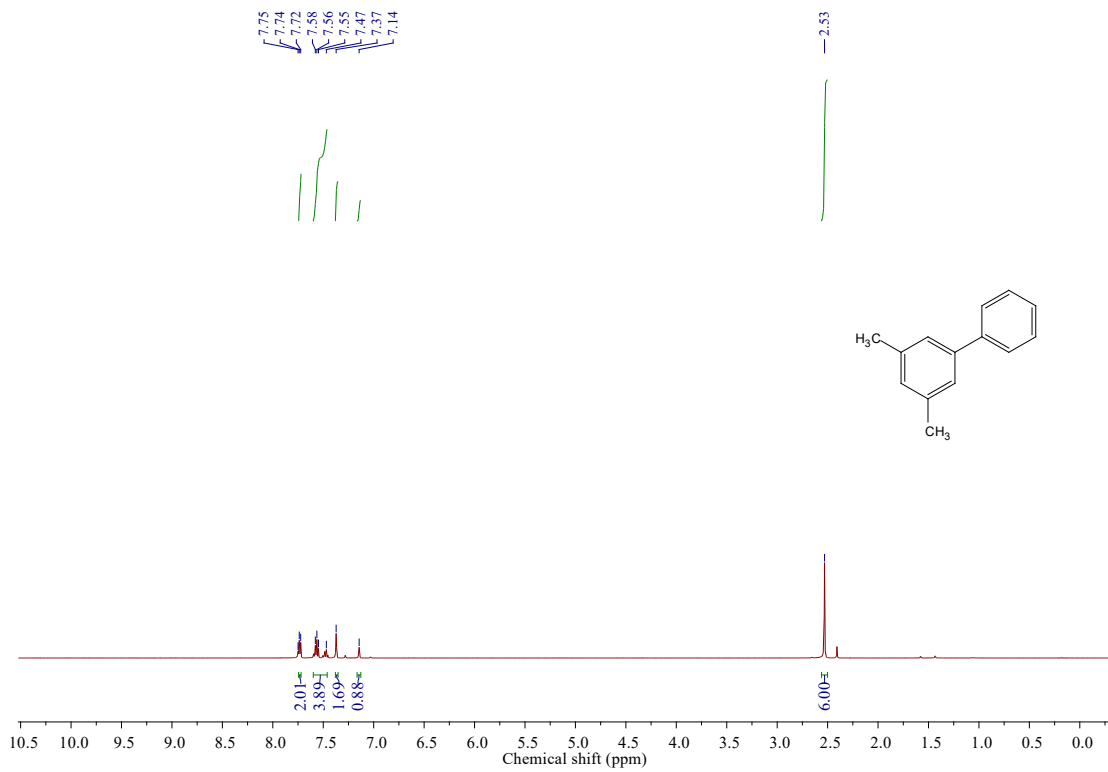
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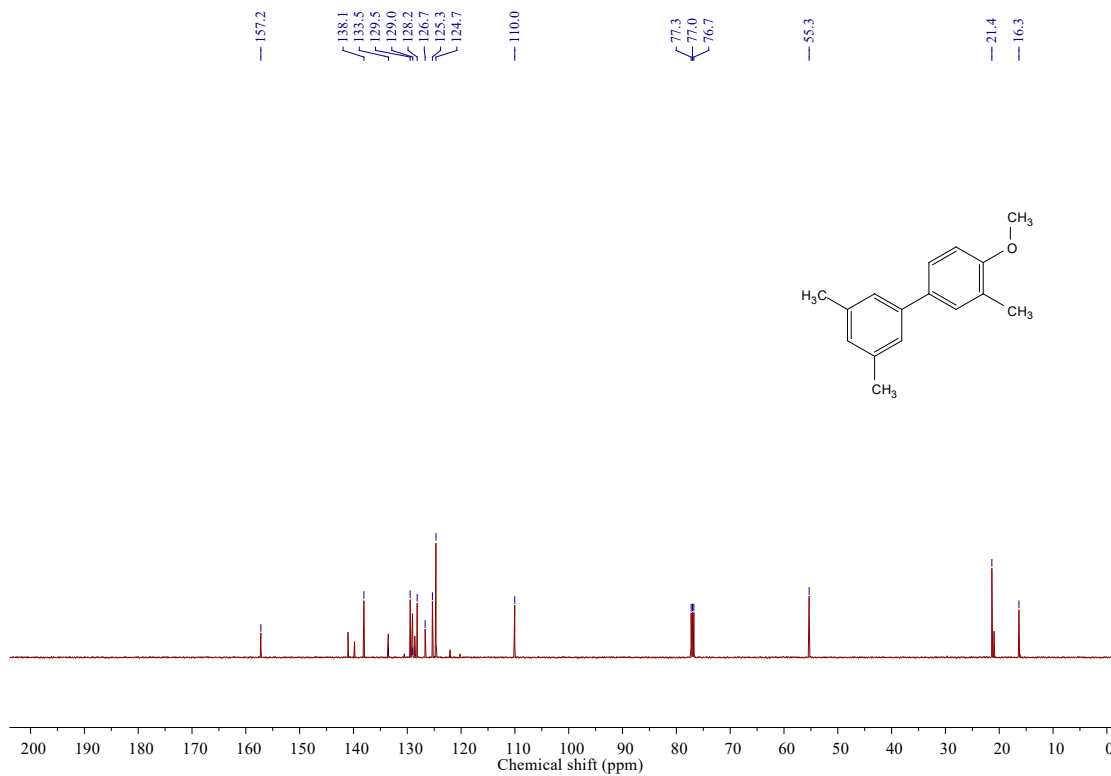
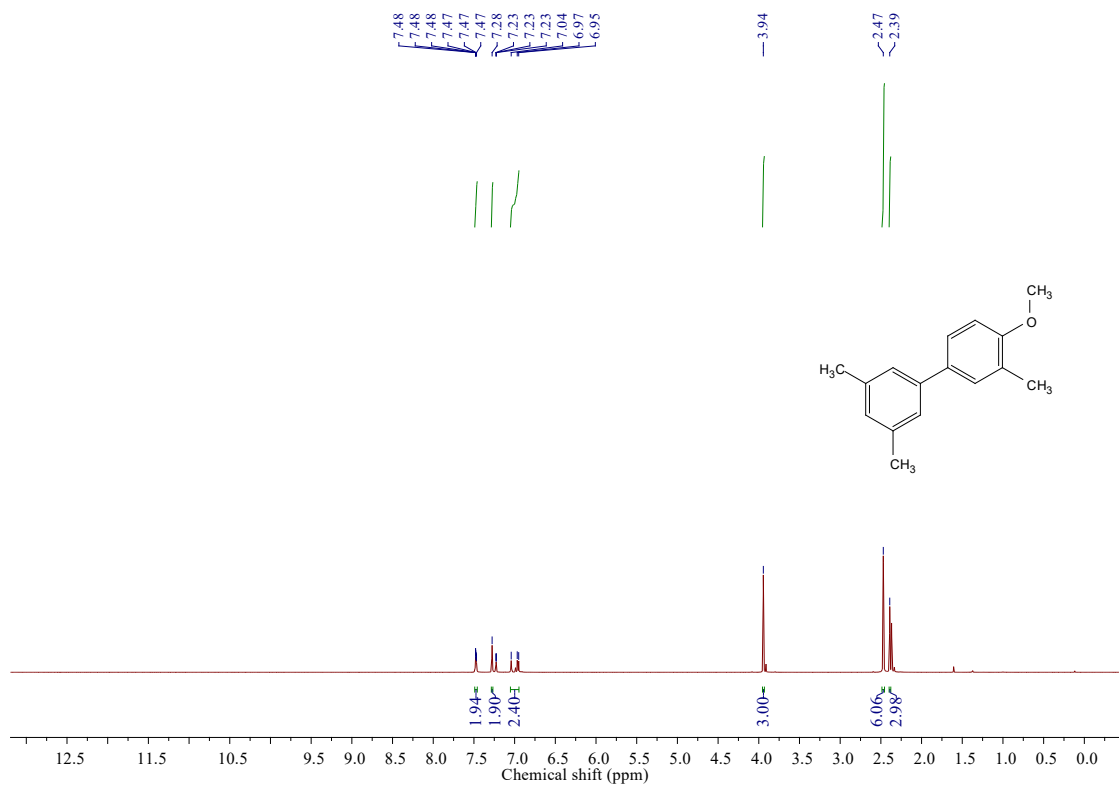
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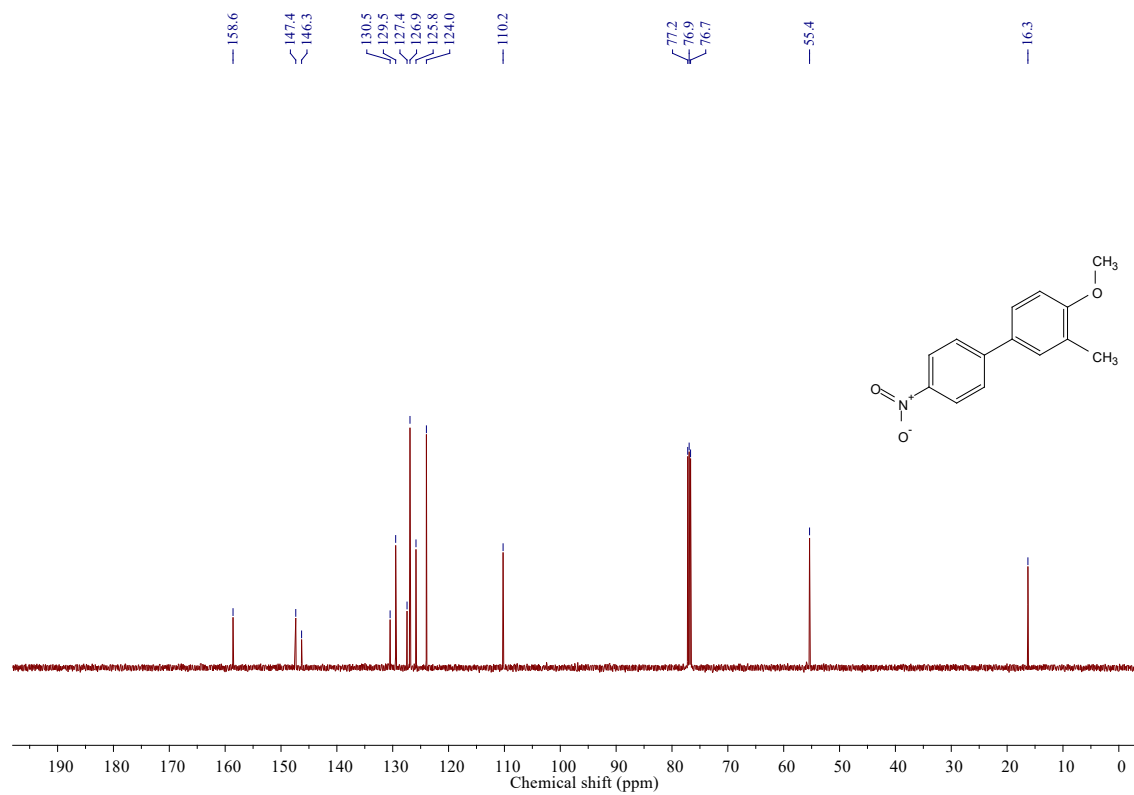
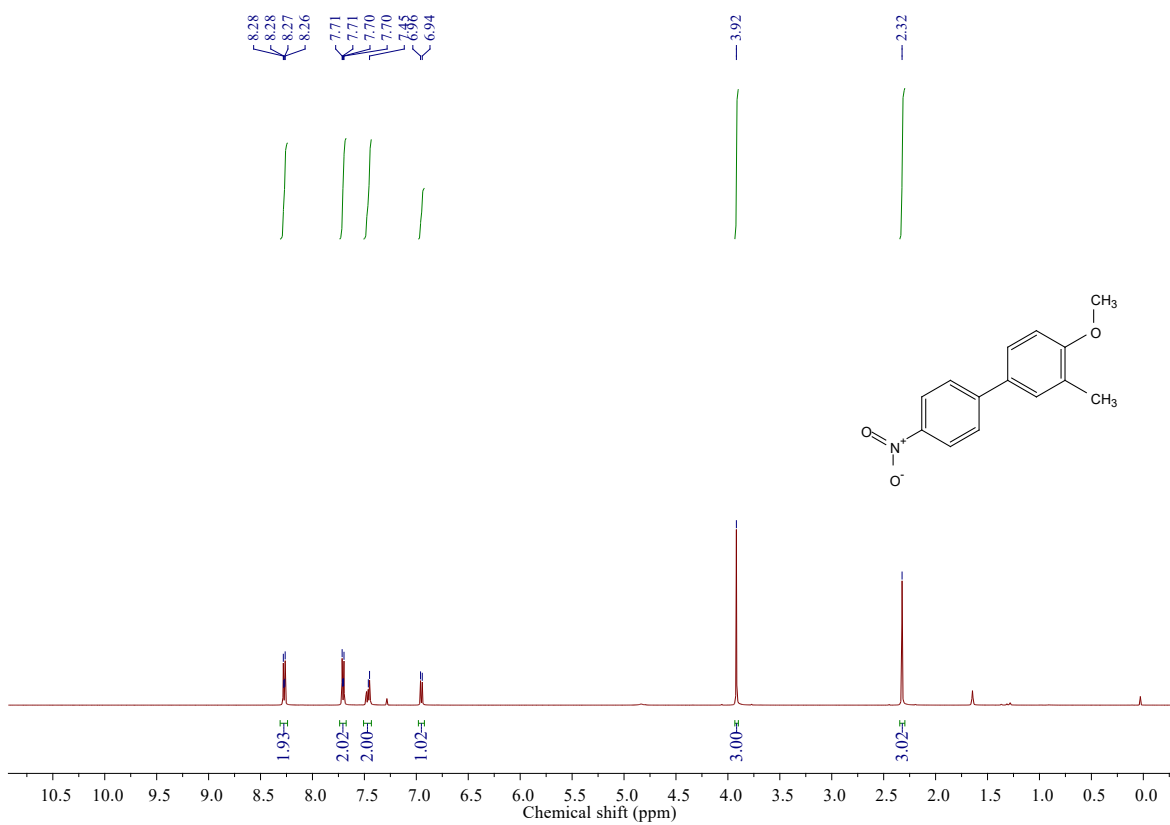
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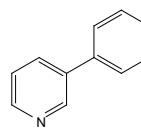
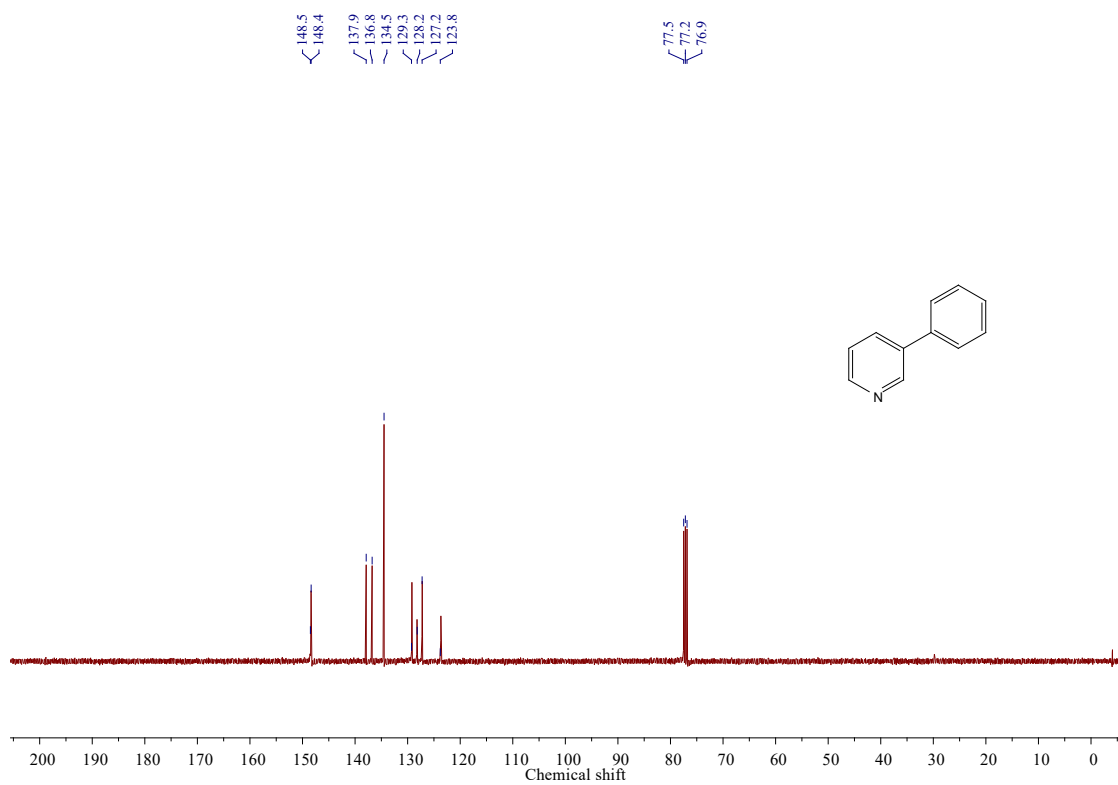
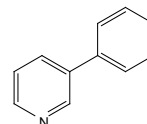
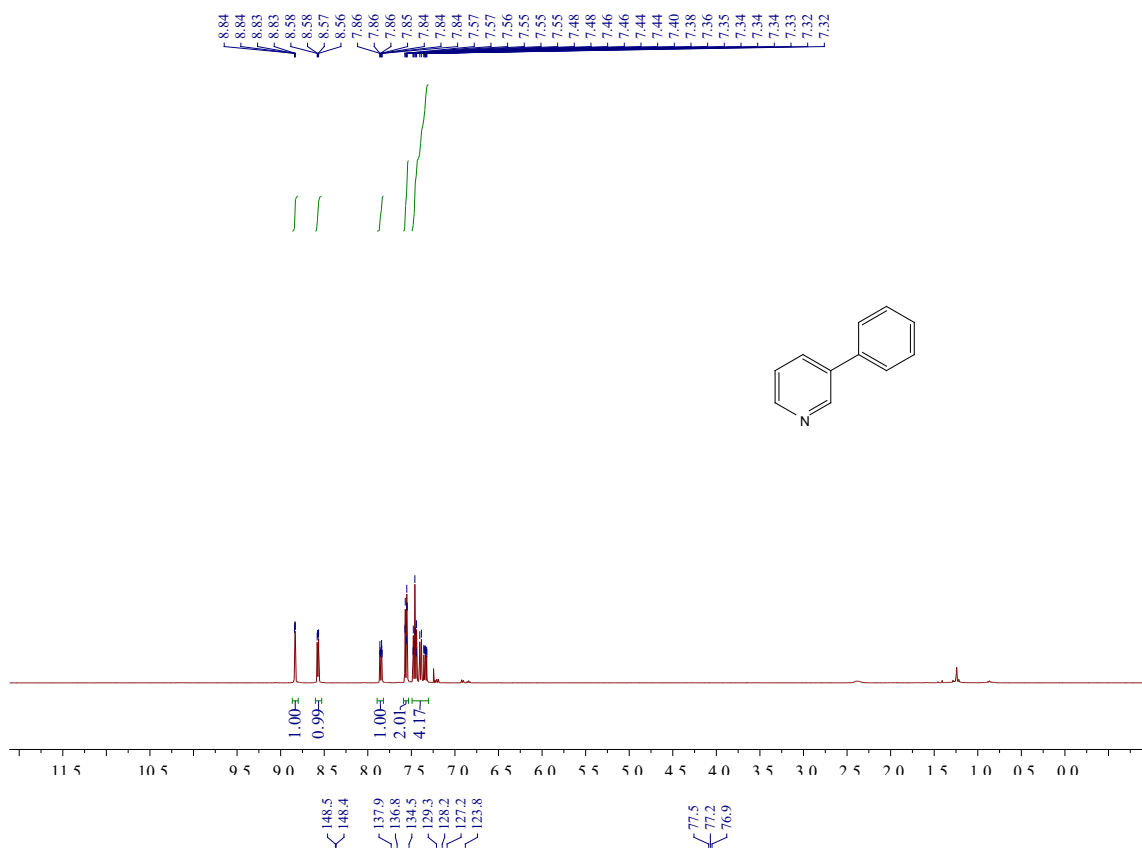
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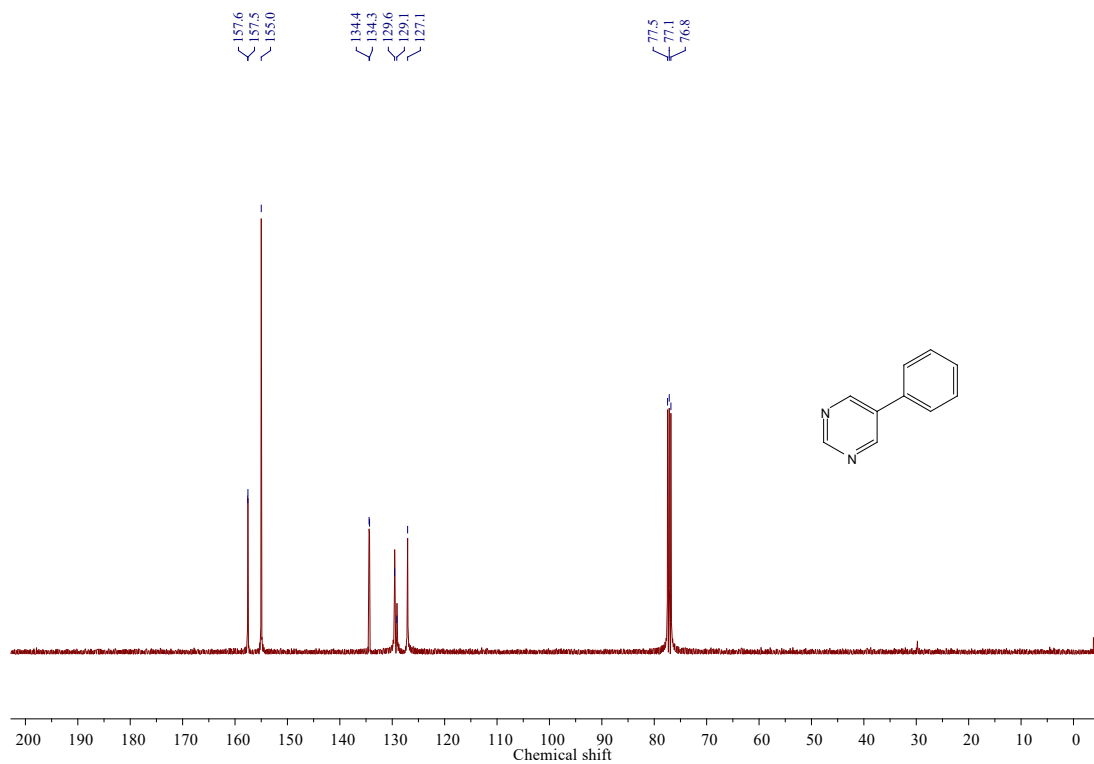
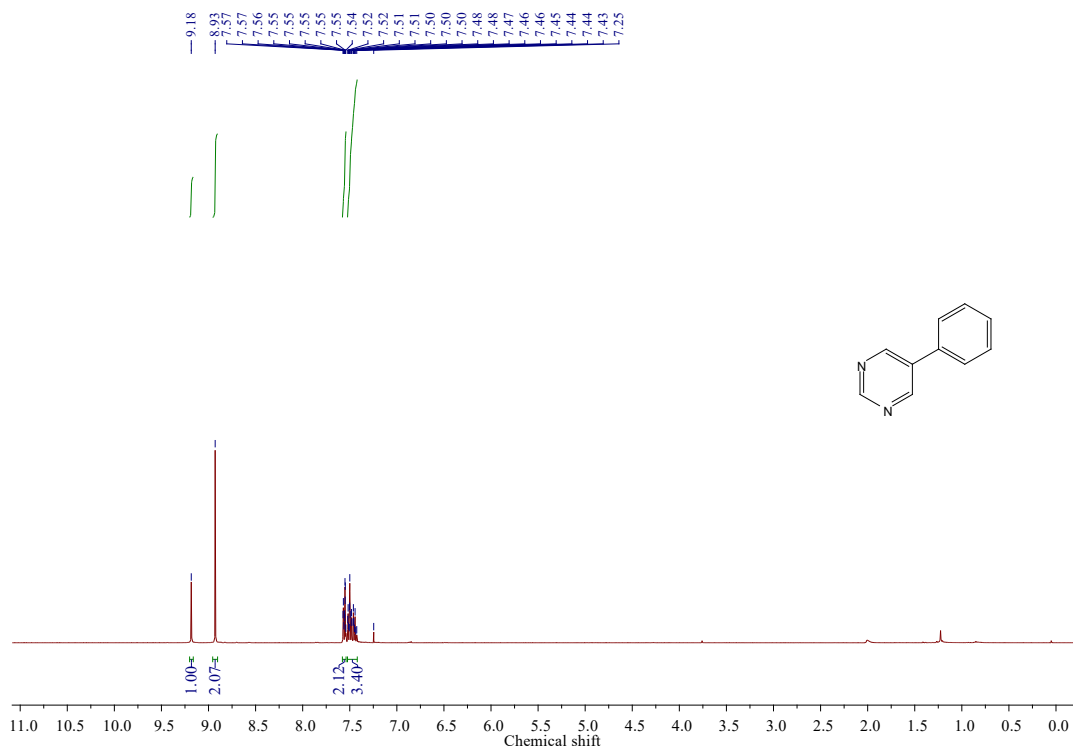
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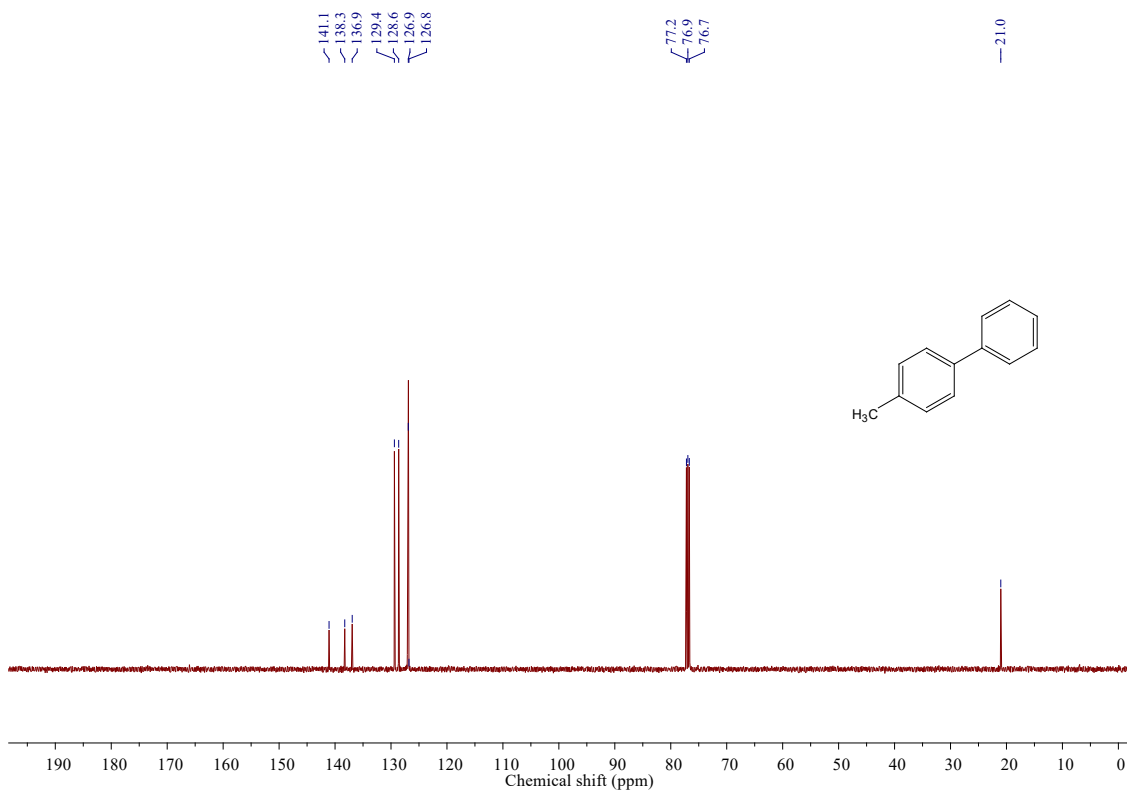
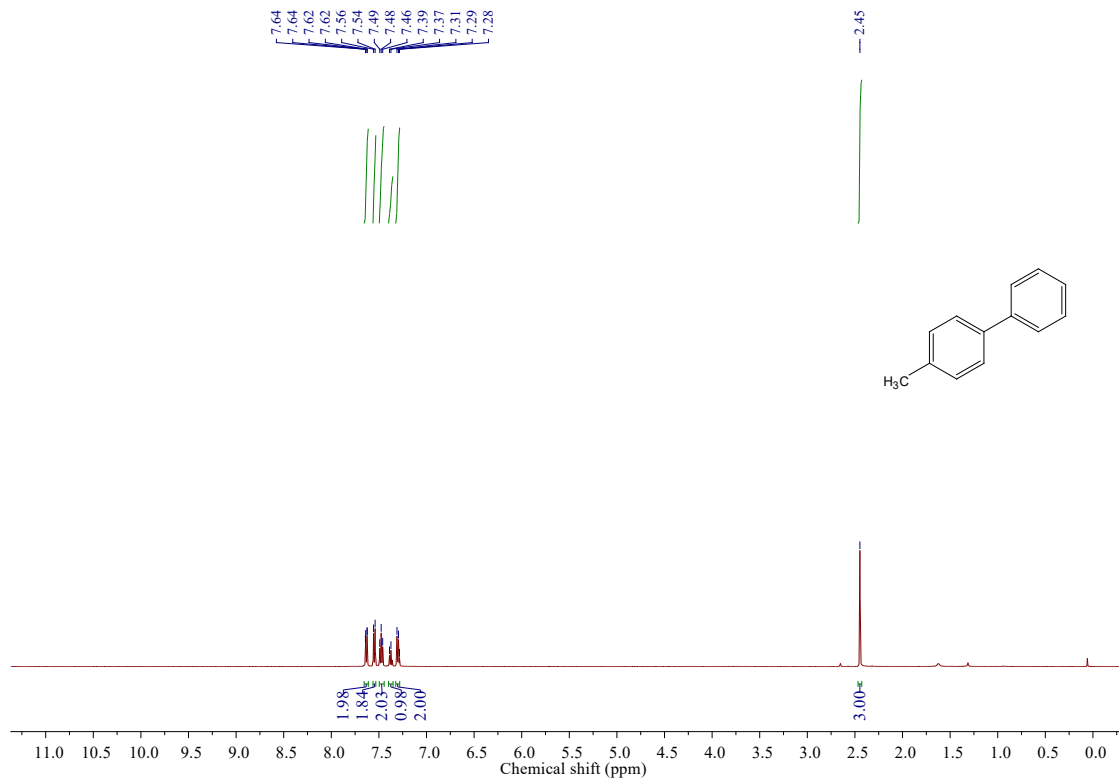
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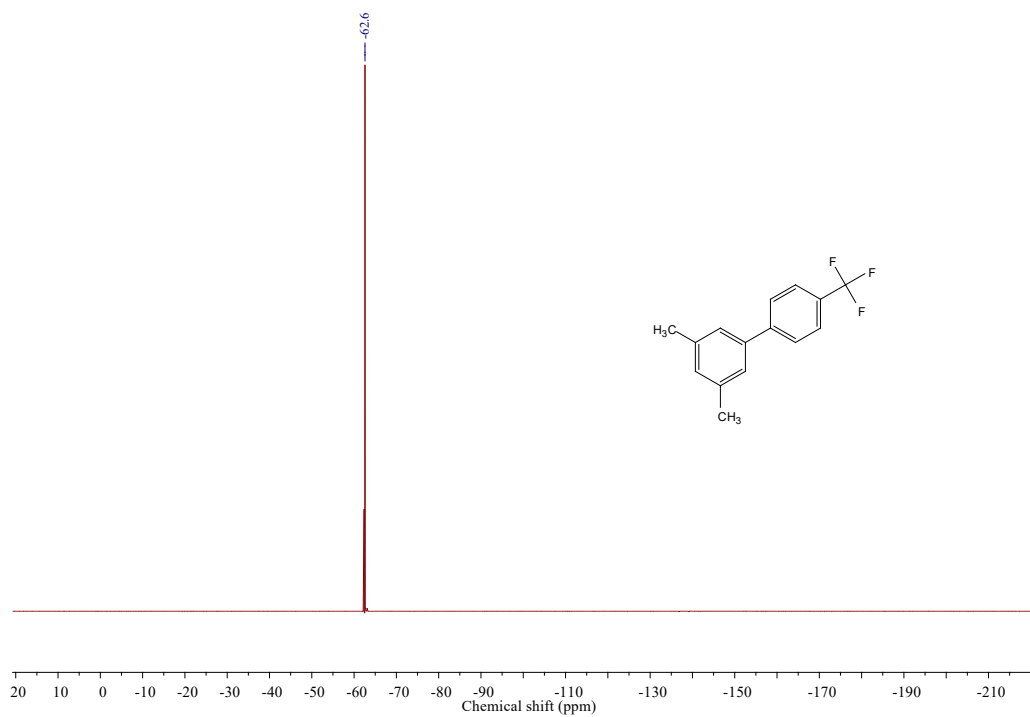


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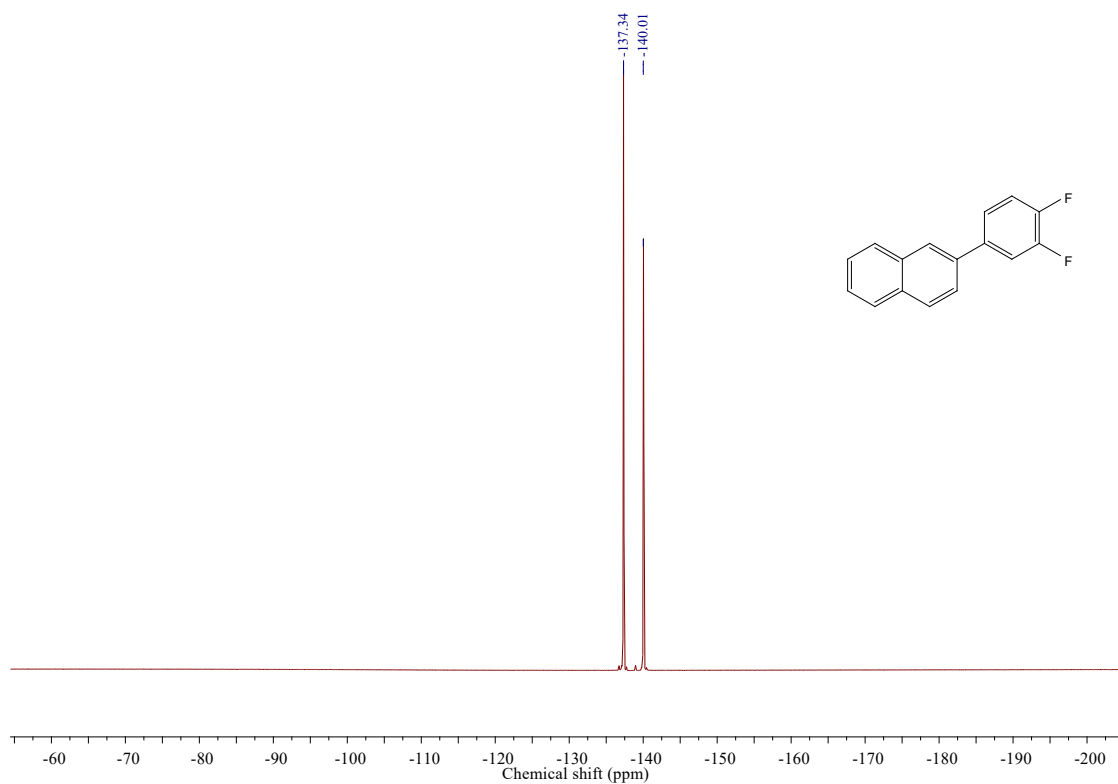


¹⁹F NMR spectra

3l.



3i.



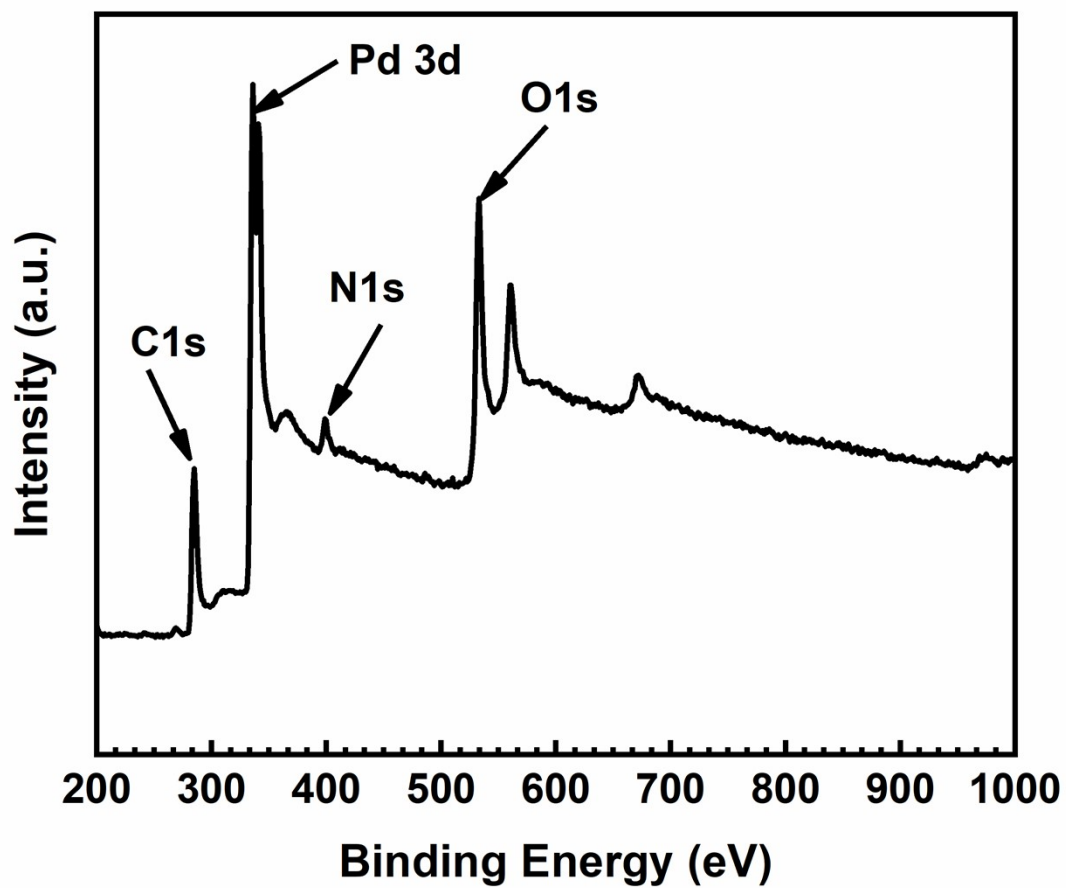


Figure S1: XPS survey spectrum of Pd(0)/g-C₃N₄O

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