

Electronic Supplementary Information

Rhodium-Catalyzed Oxidative Decarbonylative Heck-type Coupling
of Aromatic Aldehydes with Terminal Alkenes

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1. General information

Thin-layer chromatography (TLC) was performed using E. Merck Silica Gel 60 F254 precoated plates (0.25 mm). The developed chromatography was analyzed by UV lamp (254 nm). Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker Avance 400 spectrometer at ambient temperature. Chemical shifts for ¹H NMR spectra are reported in parts per million (ppm) from tetramethylsilane with the solvent resonance as the internal standard (chloroform: δ 7.26 ppm). Chemical shifts for ¹³C NMR spectra are reported in parts per million (ppm) from tetramethylsilane with the solvent as the internal standard (CDCl₃: δ 77.16 ppm). Data are reported as following: chemical shift, multiplicity (s = singlet, d = doublet, dd = doublet of doublets, t = triplet, q = quartet, m = multiplet, br = broad signal), coupling constant (Hz), and integration. The (*E*) to (*Z*) ratios were determined from the relative integration of the crude ¹H NMR spectra of the resolved olefinic protons.

2. General experimental procedures

A general experimental procedure is described as following:

An oven-dried reaction vessel was charged with [Rh(COD)Cl]₂ (0.01 mmol, 2.4 mg, 5 mol%), styrene (0.8 mmol, 83.3 mg), *p*-cyanobenzaldehyde (**1a**, 0.2 mmol, 26.2 mg), benzoyl chloride (0.04 mmol, 5.6 mg, 20 mol%), chlorobenzene (0.5 mL), and TBP (2.5 equiv, 0.5 mmol, 94 μ L) under air. The vessel was sealed and heated at 140 °C (oil bath temperature) for 12 h. The resulting mixture was cooled to room temperature, transferred to silica gel column directly and eluted with petroleum ether and ethyl acetate (30:1) to give products **3a** (34.0 mg, 83 % yield).

3. Condition optimization

Table S1. Optimization of the oxidants

$\text{Ar-CHO} + \text{Ph-CH=CH}_2 \xrightarrow[140\text{ }^\circ\text{C, 12 h}]{\text{Additive, [O]}} \text{Ar-CH=CH-Ph}$

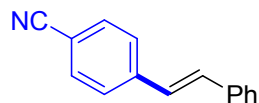
1a, Ar = *p*NC-C₆H₄ **2a** **3a**

Entry	Oxidant (equiv)	Yield (%) ^[b]
1	TBP	83
2	TBHP	< 5
3	K ₂ S ₂ O ₈	< 5
4	BPO (benzoyl peroxide)	trace
5	DCP (dicumyl peroxide)	< 5
6	H ₂ O ₂	trace

To a solution of **1a** (0.2 mmol, 1 equiv.) and **2a** (0.8 mmol, 4 equiv.) in chlorobenzene (0.5 mL), oxidant (0.5 mmol, 2.5 equiv.) was added with vigorous stirring. The reaction mixture was stirring at 140°C for 12 h under argon. ^[b] Isolated yields.

4. Spectra data of products 3a-3t

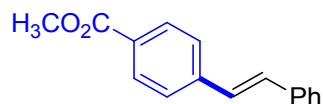
(3a)¹



Yellow solid, mp 116-118 °C. ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.64 (d, *J* = 8.4 Hz, 2H), 7.59 (d, *J* = 8.4 Hz, 2H), 7.54 (d, *J* = 7.6 Hz, 2H), 7.41 (t, *J* = 7.2 Hz, 2H), 7.33 (t, *J* = 7.2 Hz, 1H), 7.22 (d, *J* = 16.4 Hz, 1H), 7.095 (d, *J* = 16 Hz, 1H);

¹³C NMR (100 MHz, CDCl₃) δ 141.78, 136.25, 132.46, 132.35, 128.87, 128.67, 126.94, 126.86, 126.67, 119.10, 110.47; MS (EI) *m/z* (%): 205(100)[M]⁺, 190(26), 177(12), 165(8), 152(6).

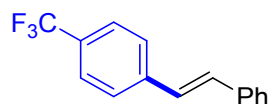
(3b)¹



White solid, mp 157-159 °C. ¹H NMR (400 MHz, CDCl₃, TMS) δ 8.03 (d, *J* = 7.2 Hz, 2H), 7.53 - 7.58 (m, 4H), 7.38 (t, *J* = 6.4 Hz, 2H), 7.31 (d, *J* = 6.4 Hz, 1H), 7.23 (d, *J* = 16.8 Hz, 1H), 7.13 (d, *J* = 16.4 Hz, 1H), 3.93 (s, 3H);

¹³C NMR (100 MHz, CDCl₃) δ 166.97, 141.89, 136.81, 131.30, 130.12, 128.96, 128.87, 128.33, 127.62, 126.89, 126.41, 52.18; MS (EI) *m/z* (%): 238(90)[M]⁺, 207(55), 178(100), 165(4), 152(18).

(3c)²



White solid, mp 133-135 °C. ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.60 (s, 4H), 7.54 (d, *J* = 7.2 Hz, 2H), 7.39 (t, *J* = 6.8 Hz, 2H), 7.31 (t, *J* = 6.8 Hz, 1H), 7.20 (d, *J* = 16.4 Hz, 1H), 7.12 (d, *J* = 16 Hz, 1H);

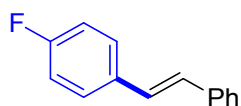
¹³C NMR (100 MHz, CDCl₃) δ 140.88, 136.72, 131.28, 129.32 (d, *J* = 32 Hz), 128.93,

¹ Sugihara T., Satoh T., Miura M., et al. *Advanced Synthesis & Catalysis*, **2004**, 346(13 - 15): 1765-1772.

² Chen Z., Luo M., Wen Y., et al. *Organic letters*, **2014**, 16(11): 3020-3023.

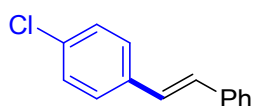
128.42, 127.20, 126.90, 126.69, 125.74 (q, $J = 4$ Hz), 123.03. MS (EI) m/z (%): 248(100)[M]⁺, 233(18), 227(16), 179(76), 152(9).

(3d)²



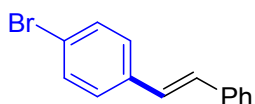
White solid, mp 124-126 °C. ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.51 - 7.49 (m, 4H), 7.36 (t, $J = 7.2$ Hz, 2H), 7.27 (t, $J = 7.2$ Hz, 1H), 7.00 - 7.12 (m, 4H); ¹³C NMR (100 MHz, CDCl₃) δ 162.42 (d, $J = 246$ Hz), 137.24, 133.59 (d, $J = 3$ Hz), 128.82, 128.57, 128.09 (d, $J = 8$ Hz), 127.78, 127.55, 126.56, 115.72 (d, $J = 22$ Hz). MS (EI) m/z (%): 198(100)[M]⁺, 183(42), 177(21), 165(7), 152(5).

(3e)²



White solid, mp 126-128 °C. ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.51 (d, $J = 6.8$ Hz, 2H), 7.45 (d, $J = 8$ Hz, 2H), 7.28 - 7.39 (m, 5H), 7.07 (dd, $J = 16.4$ Hz, $J = 18.4$ Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 137.05, 135.91, 133.25, 129.37, 128.94, 128.85, 127.98, 127.77, 127.43, 126.66; MS (EI) m/z (%): 214(79)[M]⁺, 199(6), 178(100), 163(4), 152(13).

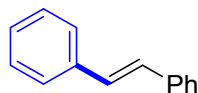
(3f)¹



White solid, mp 137-139 °C. ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.47-7.52 (m, 4H), 7.37 - 7.39 (m, 4H), 7.26 - 7.30 (m, 1H), 7.11 (d, $J = 16.4$ Hz, 1H), 7.04 (d, $J = 16.4$ Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 137.03, 136.36, 131.89, 129.50, 128.87, 128.09, 128.03, 127.49, 126.68, 121.42; MS (EI) m/z (%): 258(75)[$M-1$]⁺, 243(4), 178(100),

165(4), 152(10).

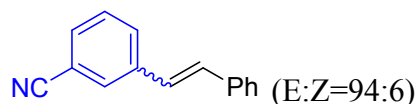
(3g)¹



White solid, mp 123-125 °C. ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.52 (d, *J* = 7.6 Hz, 4H), 7.36 (t, *J* = 8.0 Hz, 4H), 7.24 - 7.28 (m, 2H), 7.12 (s, 2H);

¹³C NMR (100 MHz, CDCl₃) δ 137.43, 128.81, 128.80, 127.75, 126.64. MS (EI) *m/z* (%): 180(100)[M]⁺, 165(60), 152(16).

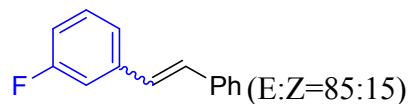
(3h)³



Yellow solid, mp 62-64 °C. ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.78 (s, 1H), 7.72 (m, 1H), 7.24 - 7.54 (m, 7H), 7.17 (d, *J* = 16.4 Hz, 1H), 7.06 (d, *J* = 16.4 Hz, 1H);

¹³C NMR (100 MHz, CDCl₃) δ 138.75, 136.50, 130.84, 130.67, 130.00, 129.61, 128.98, 128.59, 126.92, 126.33, 118.93, 113.09. MS (EI) *m/z* (%): 204(100)[M-1]⁺, 190(35), 176(15), 165(10), 152(9).

(3i)⁴



White solid, mp 69-71 °C. ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.52 (d, *J* = 6.8 Hz, 2H), 7.21 - 7.39 (m, 6H), 7.05 - 7.14 (m, 2H), 6.96 - 6.98 (m, 1H);

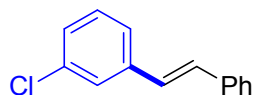
¹³C NMR (100 MHz, CDCl₃) δ 163.33 (d, *J* = 244.0 Hz), 139.50 (d, *J* = 7.0 Hz), 136.96, 130.26, 130.18, 130.15, 128.88, 128.82, 128.14, 127.76, 127.62, 127.59, 126.79, 126.65, 122.61, 122.58, 114.51 (d, *J* = 22.0 Hz), 112.91 (d, *J* = 21.0 Hz). MS

³ Cui X., Zhou Y., Wang N., et al. *Tetrahedron Letters*, **2007**, 48(1): 163-167.

⁴ Zhu X., Liu J., Chen T., et al. *ChemInform*, **2012**, 43(31): no.

(EI) m/z (%): 198(100)[M]⁺, 183(50), 178(30), 170(8), 152(5).

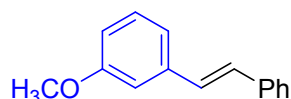
(3j)⁵



White solid, mp 73-75 °C. ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.51 (m, 3H), 7.37 (m, 3H), 7.24 - 7.31 (m, 3H), 7.12 (d, *J* = 16 Hz, 1H), 7.04 (d, *J* = 16 Hz, 1H);

¹³C NMR (100 MHz, CDCl₃) δ 124.86, 126.39, 126.77, 127.27, 127.59, 128.15, 128.86, 129.98, 130.17, 134.72, 136.88, 139.30. MS (EI) m/z (%): 214(61)[M]⁺, 199(8), 178(100), 163(5), 152(10).

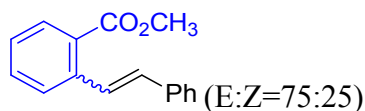
(3k)⁶



Yellow oil. ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.52 (d, *J* = 7.2 Hz, 2H), 7.35 - 7.38 (m, 2H), 7.29 (d, *J* = 7.2 Hz, 2H), 7.05 - 7.13 (m, 4H), 6.83 (d, *J* = 8 Hz, 1H), 3.85 (s, 3H);

¹³C NMR (100 MHz, CDCl₃) δ 159.98, 138.88, 137.31, 129.76, 129.10, 128.81, 128.67, 127.81, 126.66, 119.35, 113.40, 111.83, 55.36. MS (EI) m/z (%): 210(100)[M]⁺, 194(25), 179(40), 165(50), 152(24).

(3l)⁷



Yellow oil. ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.52 (d, *J* = 7.2 Hz, 1H), 7.00 - 7.39 (m, 9H), 6.65 (d, *J* = 12.4, 1H), 3.89 (s, 3H);

¹³C NMR (100 MHz, CDCl₃) δ 167.61, 139.77, 136.77, 132.11, 131.21, 130.54,

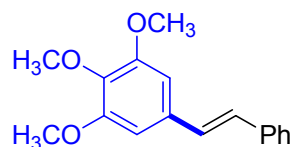
⁵ Zhang Y.-G., Liu X.-L., He Z.-Y., et al. *Chemistry-A European Journal*, **2014**, 20(10): 2765-2769.

⁶ Pavia C., Giacalone F., Bivona L. A., et al. *Journal of Molecular Catalysis A: Chemical*, **2014**, 387: 57-62.

⁷ Shahzad S. A., Wirth T., *Angewandte Chemie International Edition*, **2009**, 48(14): 2588-2591.

129.66, 129.34, 128.79, 128.13, 127.53, 127.22, 127.05, 52.15. MS (EI) m/z (%): 238(92)[M]⁺, 206(33), 195(13), 178(100), 165(13), 152(20).

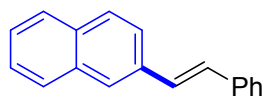
(3m)⁸



Light yellow solid, mp 105-107 °C. ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.51 (d, *J* = 7.2 Hz, 2H), 7.37 (t, *J* = 7.2 Hz, 2H), 7.27 (t, *J* = 7.6 Hz, 1H), 6.99 - 7.08 (dd, *J* = 17.2 Hz, *J* = 17.6 Hz, 2H), 6.75 (s, 2H), 3.93 (s, 6H), 3.87 (s, 3H);

¹³C NMR (100 MHz, CDCl₃) δ 56.24, 61.12, 103.60, 126.56, 127.75, 128.32, 128.74, 128.84, 133.21, 137.31, 137.97, 153.51. MS (EI) m/z (%): 270(100)[M]⁺, 255(88), 195(18), 180(11), 165(19), 152(20).

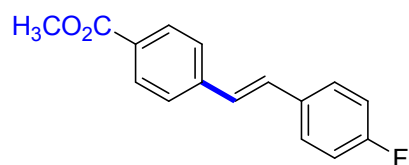
(3n)¹



White solid, mp 147-149 °C. ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.82 - 7.86 (m, 4H), 7.75 (d, *J* = 8.4 Hz, 1H), 7.57 (d, *J* = 7.2 Hz, 2H), 7.46 (t, *J* = 6.4 Hz, 2H), 7.39 (t, *J* = 6.8 Hz, 2H), 7.22 - 7.30 (m, 3H);

¹³C NMR (100 MHz, CDCl₃) δ 137.46, 134.92, 133.82, 133.15, 129.12, 128.88, 128.86, 128.44, 128.13, 127.83, 127.82, 126.78, 126.67, 126.47, 126.03, 123.61. MS (EI) m/z (%): 230(100)[M]⁺, 215(24), 202(12), 189(5), 152(10).

(3o)⁹



White solid, mp 144-146 °C. ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.03 (d, *J* = 8.4 Hz,

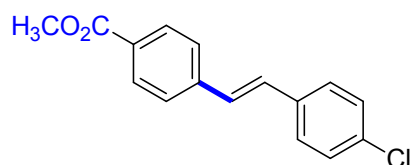
⁸ McNulty J., Das P. *European Journal of Organic Chemistry*, **2009**, 2009(24): 4031-4035.

⁹ Palmer B. D., Sutherland H. S., Blaser A., et al. *Journal of Medicinal Chemistry*, **2015**, 58(7): 3036-3059.

2H), 7.55 (d, $J = 8.0$ Hz, 2H), 7.49 - 7.53 (dd, $J = 5.6$ Hz, $J = 5.6$ Hz, 2H), 7.18 (d, $J = 16.4$ Hz, 1H), 7.02 - 7.09 (dd, $J = 16.4$ Hz, $J = 8.4$ Hz, 3H), 3.93 (s, 3H);

^{13}C NMR (100 MHz, CDCl_3) δ 166.94, 162.74 (d, $J = 247$ Hz), 141.71, 133.02 (d, $J = 3$ Hz), 130.13, 129.00, 128.43 (d, $J = 8$ Hz), 127.40, 127.38, 126.34, 115.86 (d, $J = 21$ Hz), 52.19. MS (EI) m/z (%): 256(100)[M] $^+$, 225(75), 207(4), 196(70), 177(20), 151(4).

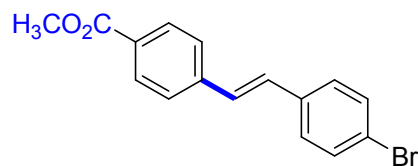
(3p)⁶



White solid, mp 161-163 °C. ^1H NMR (400 MHz, CDCl_3 , TMS) δ 8.03 (d, $J = 7.2$ Hz, 2H), 7.56 (d, $J = 7.2$ Hz, 2H), 7.46 (d, $J = 6.8$ Hz, 2H), 7.35 (d, $J = 7.6$ Hz, 2H), 7.17 (d, $J = 16.4$ Hz, 1H), 7.09 (d, $J = 16.4$ Hz, 1H), 3.93 (s, 3H);

^{13}C NMR (100 MHz, CDCl_3) δ 166.92, 141.51, 133.94, 130.16, 129.93, 129.21, 129.06, 128.22, 128.03, 126.46, 52.23. MS (EI) m/z (%): 272(85)[M] $^+$, 241(47), 213(9), 178(100), 152(12).

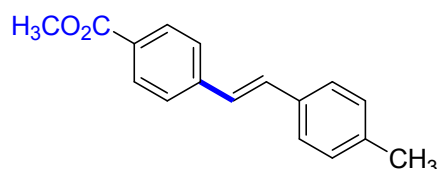
(3q)⁶



Yellow solid, mp 179-181°C. ^1H NMR (400 MHz, CDCl_3 , TMS) δ 8.03 (d, $J = 7.6$ Hz, 2H), 7.56 (d, $J = 8.0$ Hz, 2H), 7.51 (d, $J = 8.0$ Hz, 2H), 7.40 (d, $J = 8.0$ Hz, 2H), 7.09 - 7.18 (dd, $J = 18.4$ Hz, $J = 16.4$ Hz, 2H), 3.93 (s, 3H);

^{13}C NMR (100 MHz, CDCl_3) δ 166.97, 141.55, 135.84, 132.07, 130.22, 130.06, 129.32, 128.42, 128.37, 126.53, 122.20, 52.26. MS (EI) m/z (%): 316(58)[$\text{M}-1$] $^+$, 285(29), 258(4), 205(4), 192(4), 178(100), 152(15).

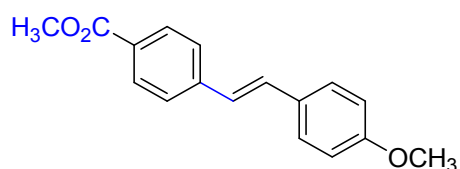
(3r)⁶



White solid, mp 163-165 °C. ^1H NMR (400 MHz, CDCl_3 , TMS) δ 8.02 (d, $J = 8.0$ Hz, 2H), 7.55 (d, $J = 7.6$ Hz, 2H), 7.43 (d, $J = 7.6$ Hz, 2H), 7.18 - 7.22 (m, 3H), 7.08 (d, $J = 16.4$ Hz, 1H), 3.92 (s, 3H), 2.37 (s, 3H);

^{13}C NMR (100 MHz, CDCl_3) δ 167.01, 142.13, 138.36, 134.05, 131.26, 130.10, 129.60, 128.74, 126.82, 126.62, 126.28, 52.16, 21.46. MS (EI) m/z (%): 252(100)[M] $^+$, 221(36), 193(37), 178(94), 152(10).

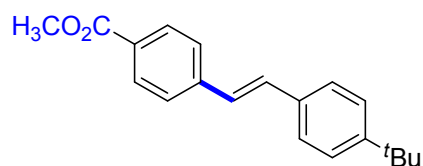
(3s)¹⁰



White solid, mp 170-172 °C. ^1H NMR (400 MHz, CDCl_3 , TMS) δ 8.01 (d, $J = 8.0$ Hz, 2H), 7.54 (d, $J = 8$ Hz, 2H), 7.48 (d, $J = 8.4$ Hz, 2H), 7.18 (d, $J = 16.4$ Hz, 1H), 6.91 - 7.02 (dd, $J = 8$ Hz, $J = 16.4$ Hz, 3H), 3.92 (s, 3H), 3.84 (s, 3H);

^{13}C NMR (100 MHz, CDCl_3) δ 167.08, 159.92, 142.35, 130.92, 130.15, 129.68, 128.60, 128.23, 126.15, 125.55, 114.37, 55.48, 52.18. MS (EI) m/z (%): 268(100)[M] $^+$, 273(19), 209(10), 194(20), 178(18), 165(50), 152(5).

(3t)¹¹



White solid, mp 102-104 °C. ^1H NMR (400 MHz, CDCl_3 , TMS) δ 8.02 (d, $J = 8.0$ Hz, 2H), 7.56 (d, $J = 8.4$ Hz, 2H), 7.48 (d, $J = 8.0$ Hz, 1H), 7.41 (d, $J = 8.0$ Hz, 2H), 7.20

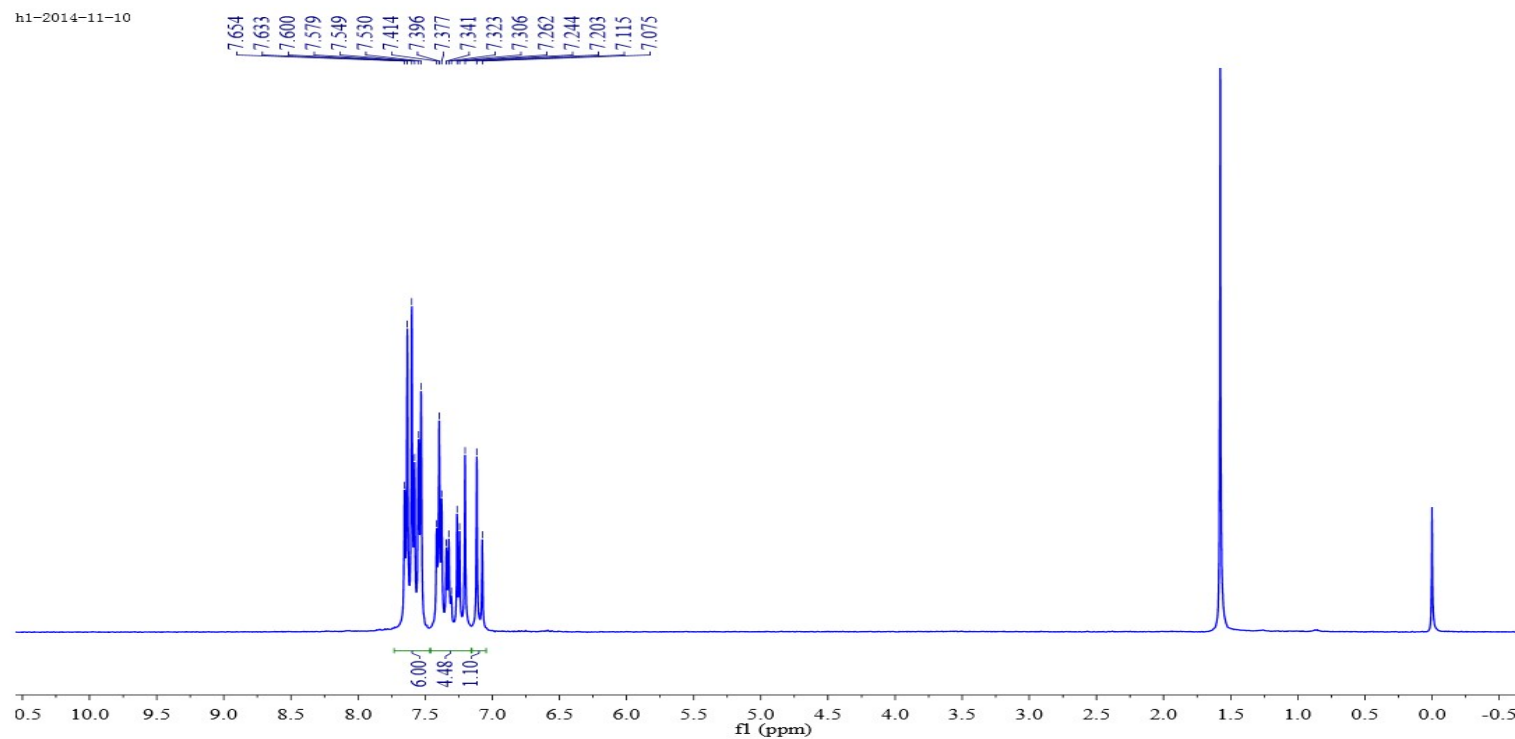
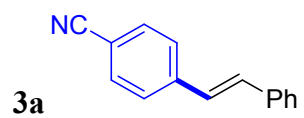
¹⁰ Hong F.-J., Low Y. Y., Chong K. W., et al. *The Journal of Organic Chemistry*, **2014**, 79(10): 4528-4543.

¹¹ Jana D., Ghorai B. K. *Tetrahedron*, **2012**, 68(36): 7309-7316.

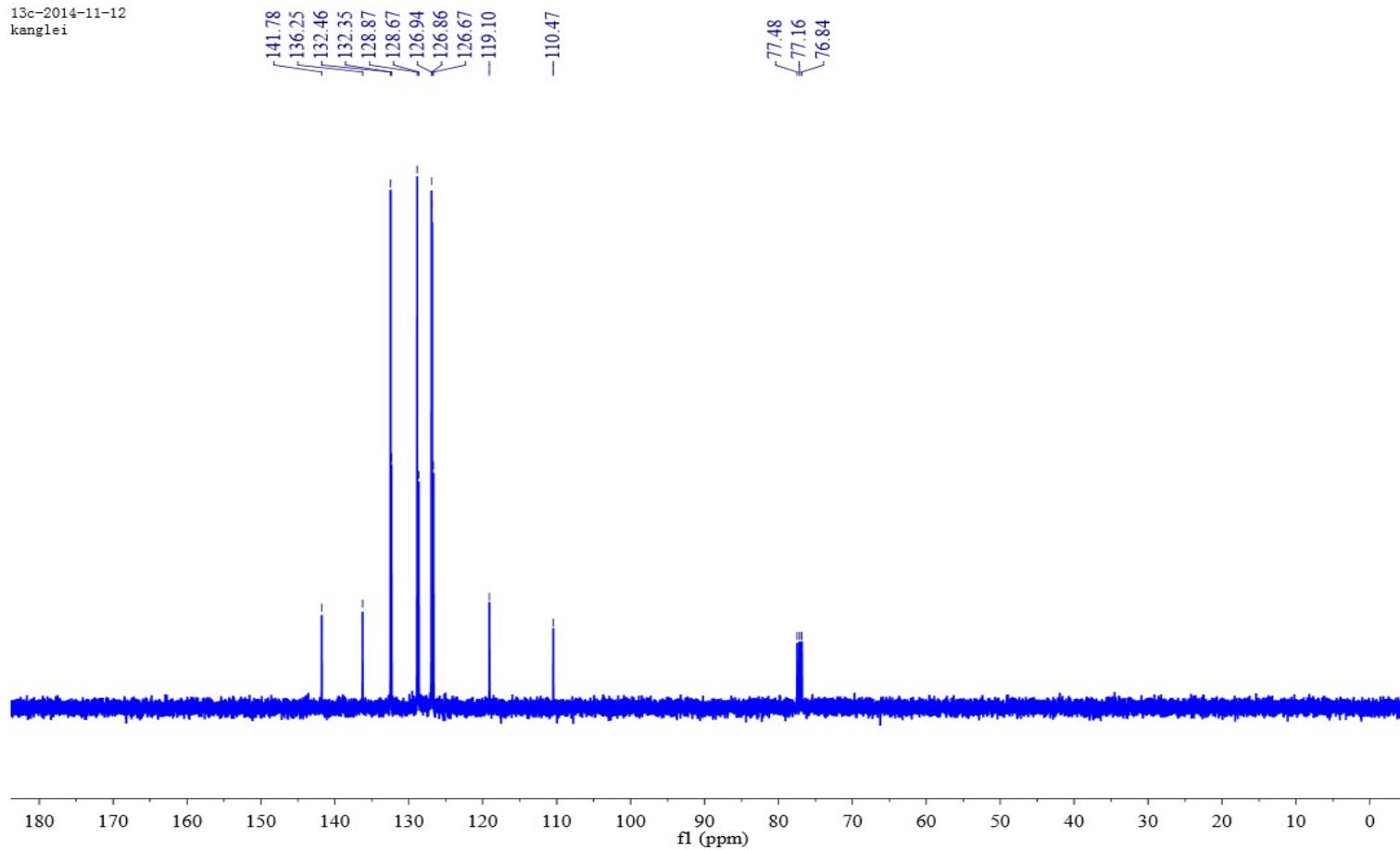
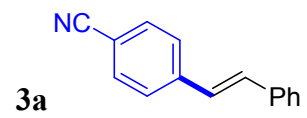
(d, $J = 16.4$ Hz, 1H), 7.09 (d, $J = 16.4$ Hz, 1H), 3.92 (s, 3H), 1.34 (s, 9H);

^{13}C NMR (100 MHz, CDCl_3) δ 167.06, 151.65, 142.20, 134.12, 130.14, 128.81, 126.91, 126.69, 126.34, 125.86, 52.20, 34.83, 31.40. MS (EI) m/z (%): 294(155)[M]⁺, 279(100), 263(5), 247(15), 220(5), 191(5), 178(20).

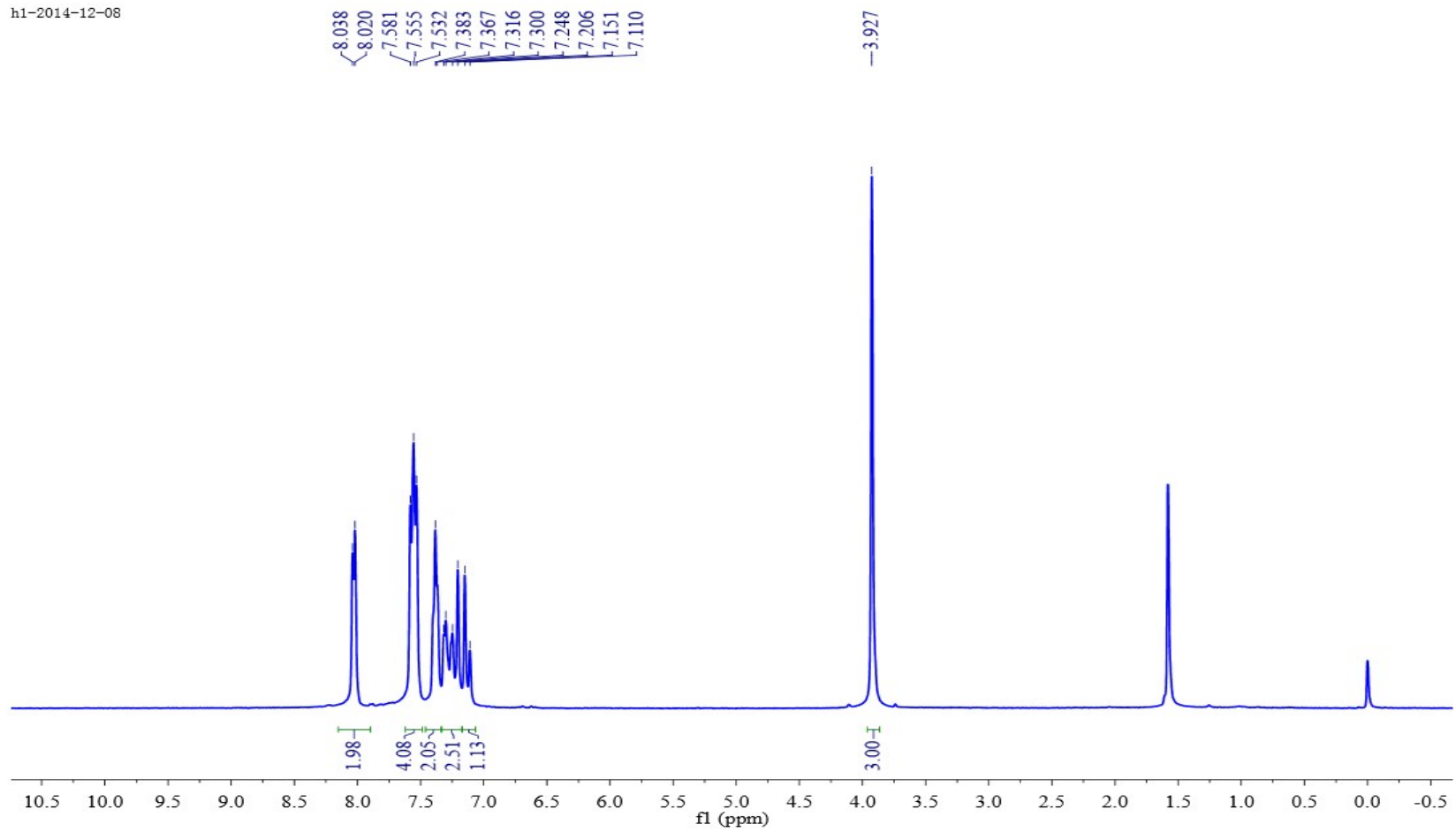
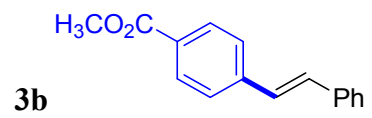
5. Copies of ¹H and ¹³C NMR spectra of products 3a-3t



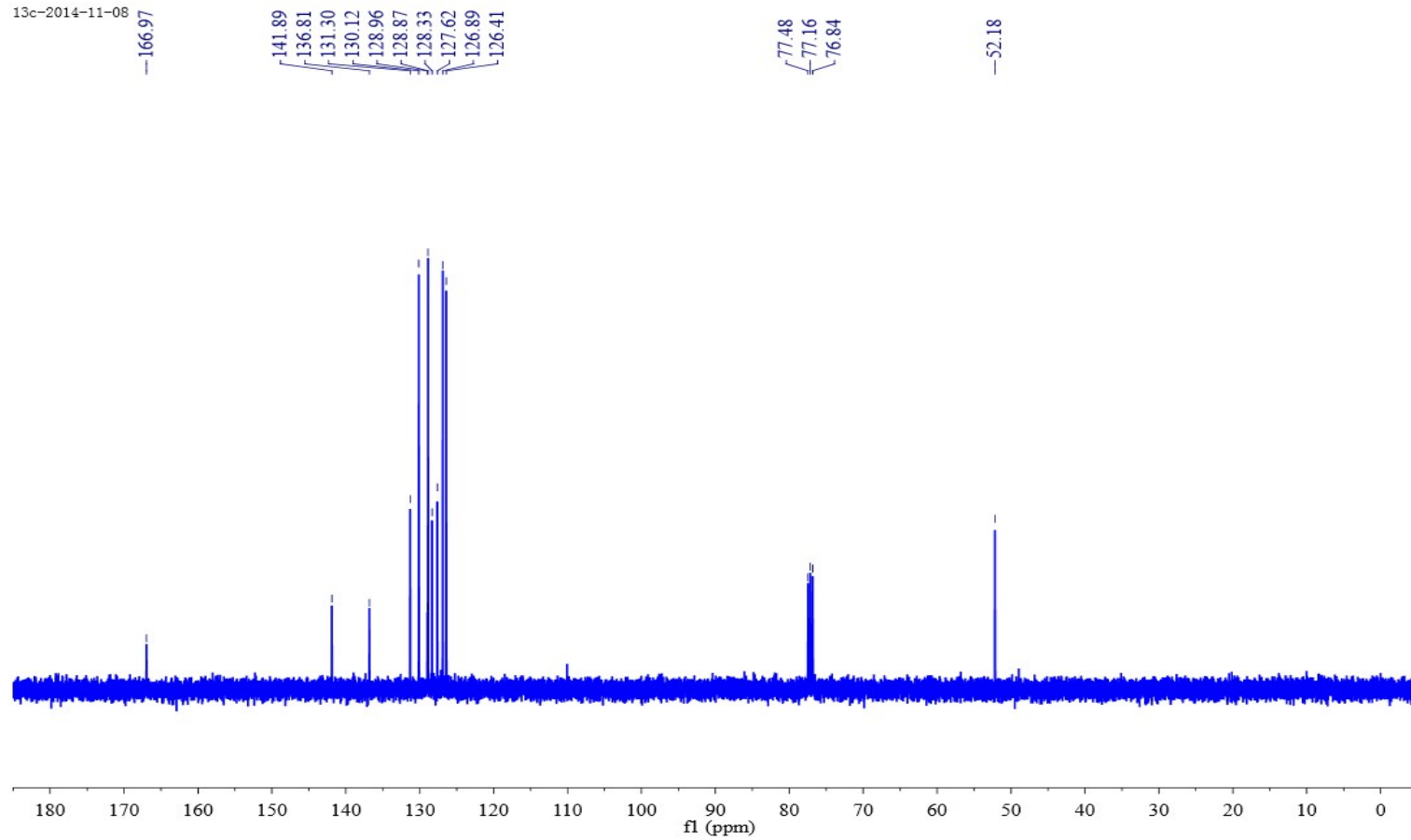
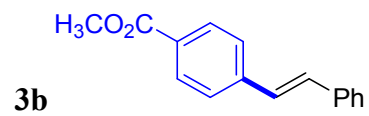
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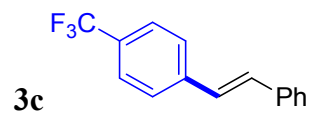


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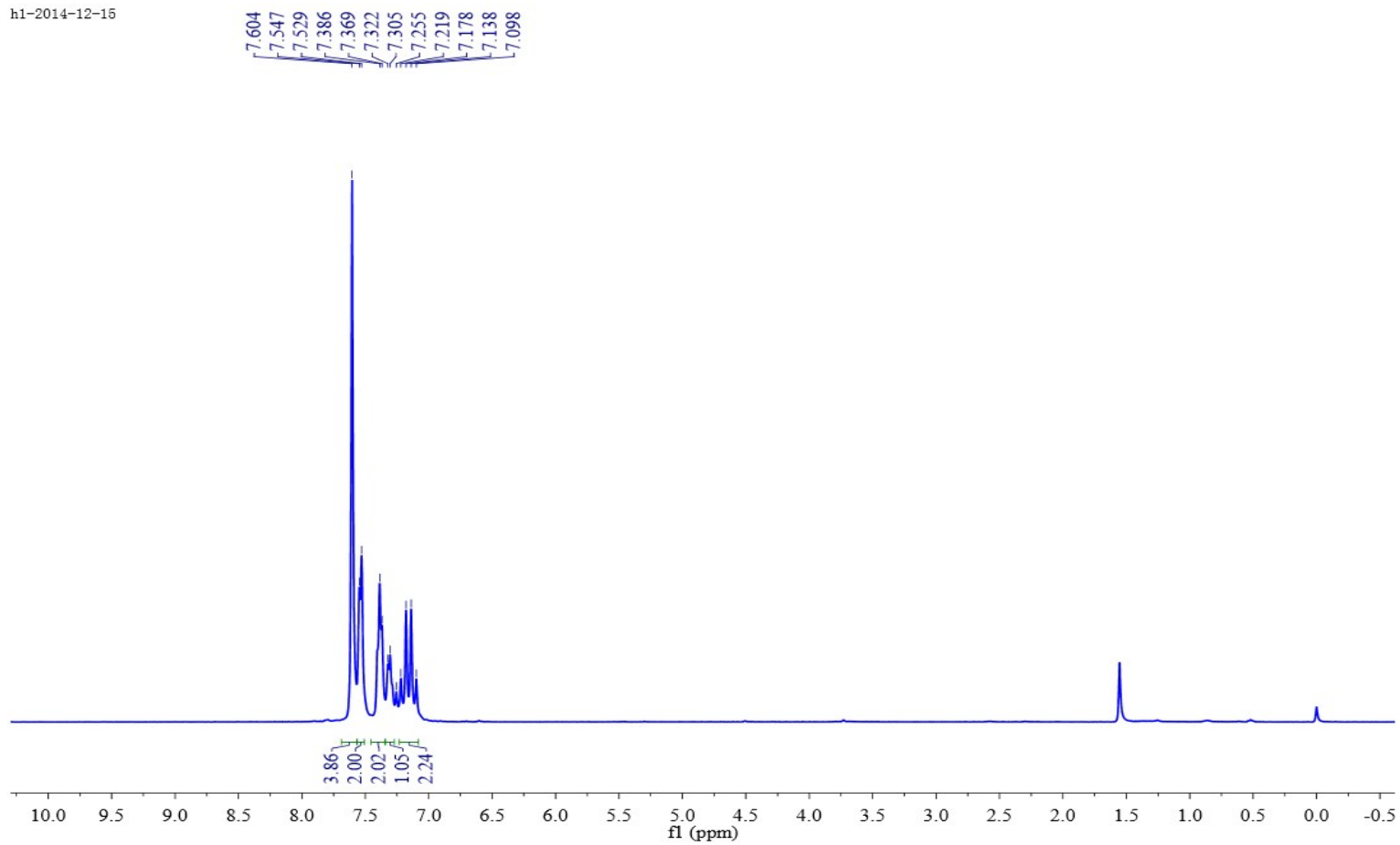


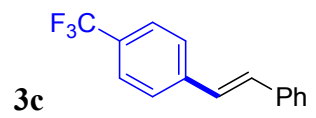
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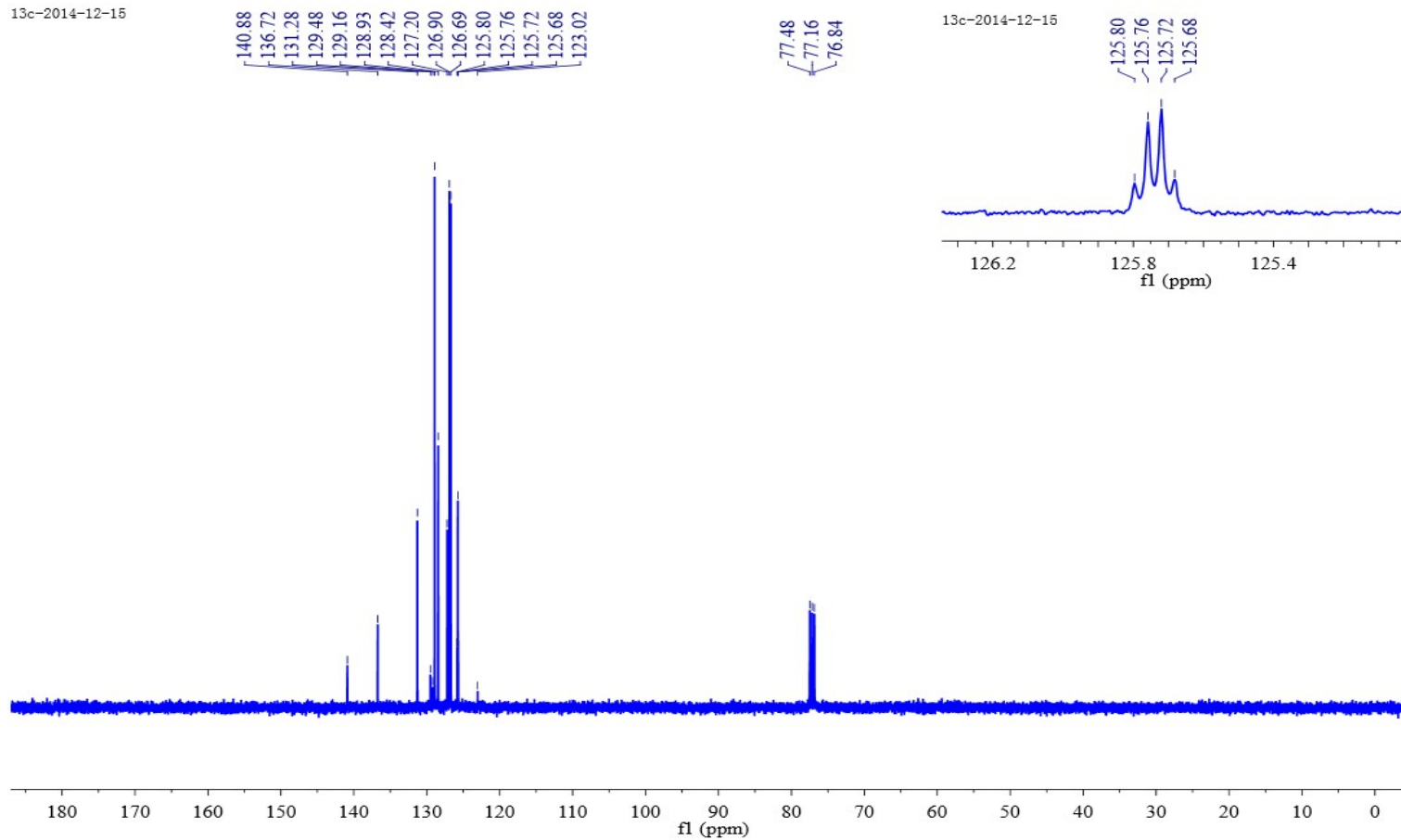


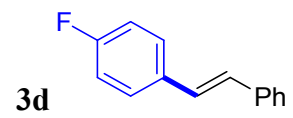
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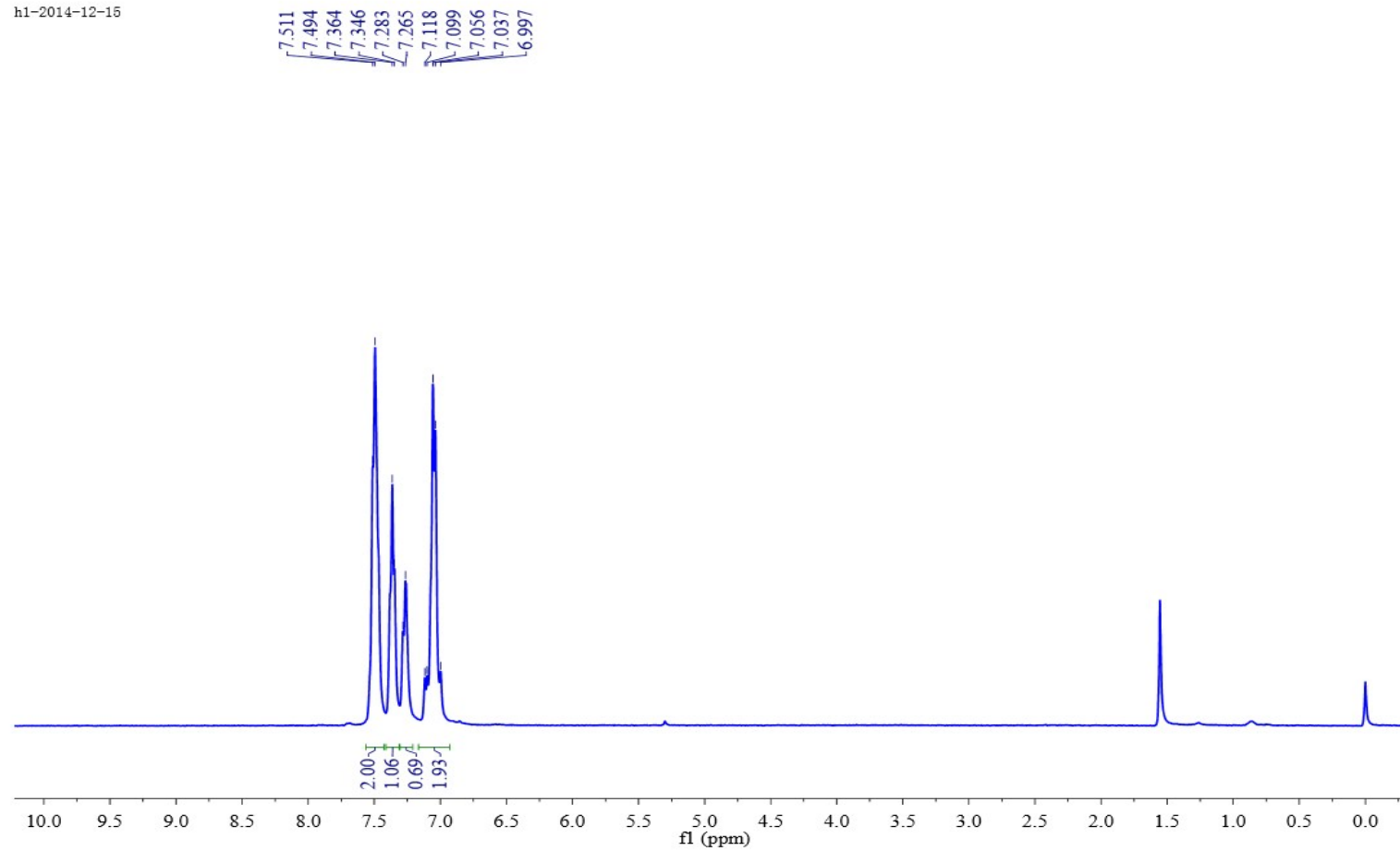


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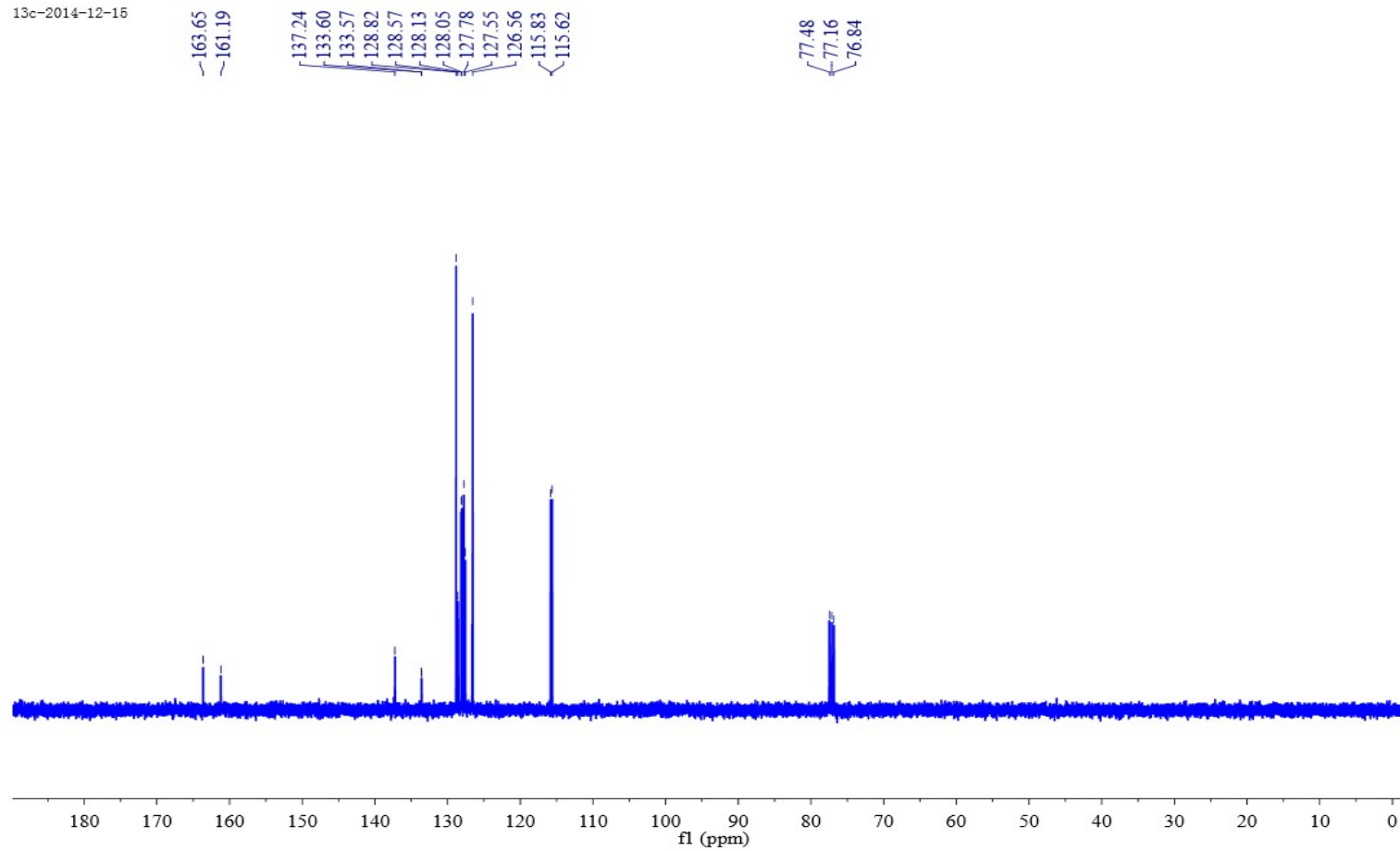
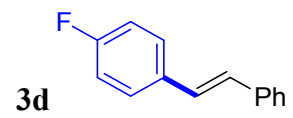




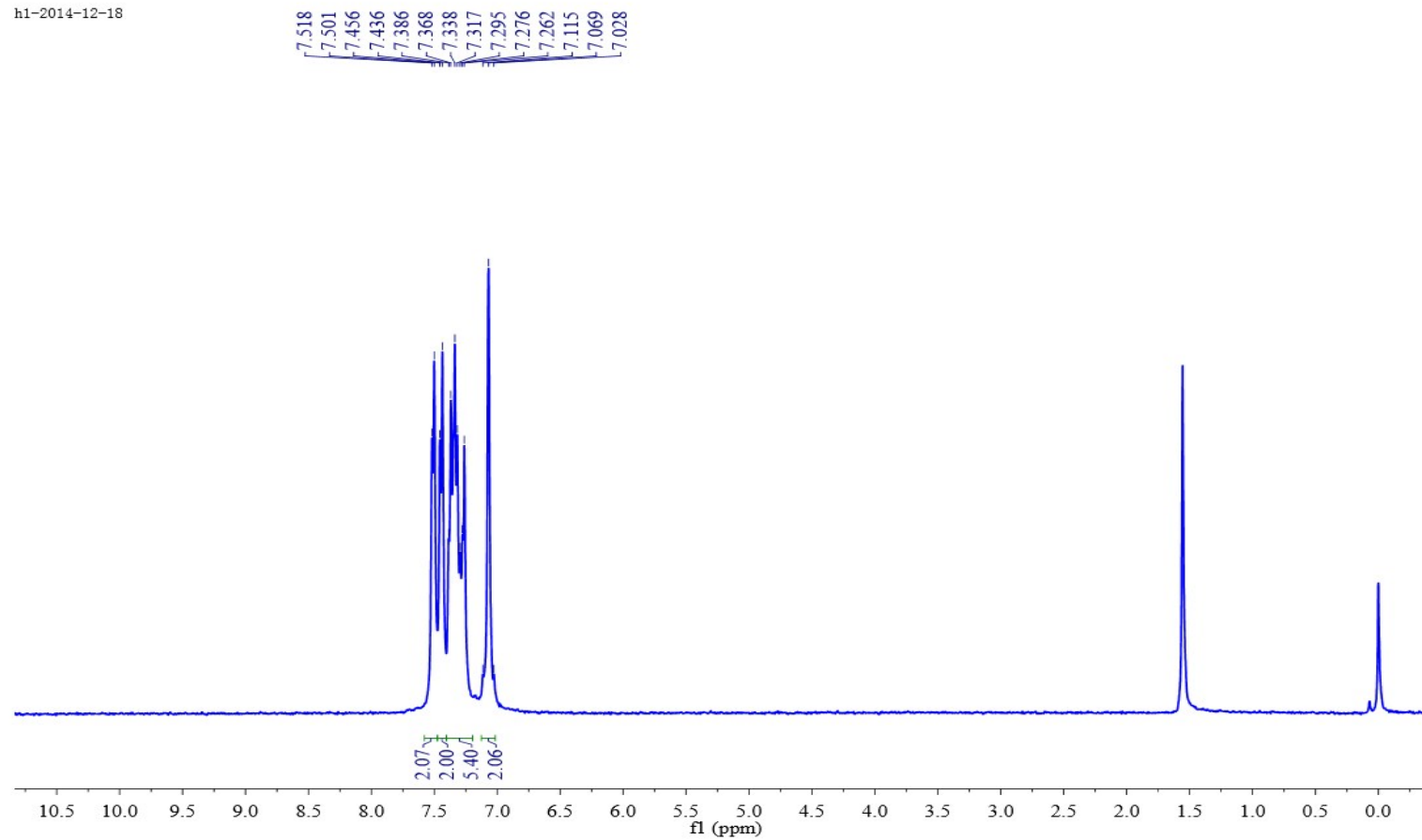
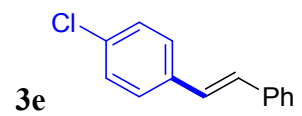
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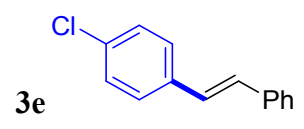


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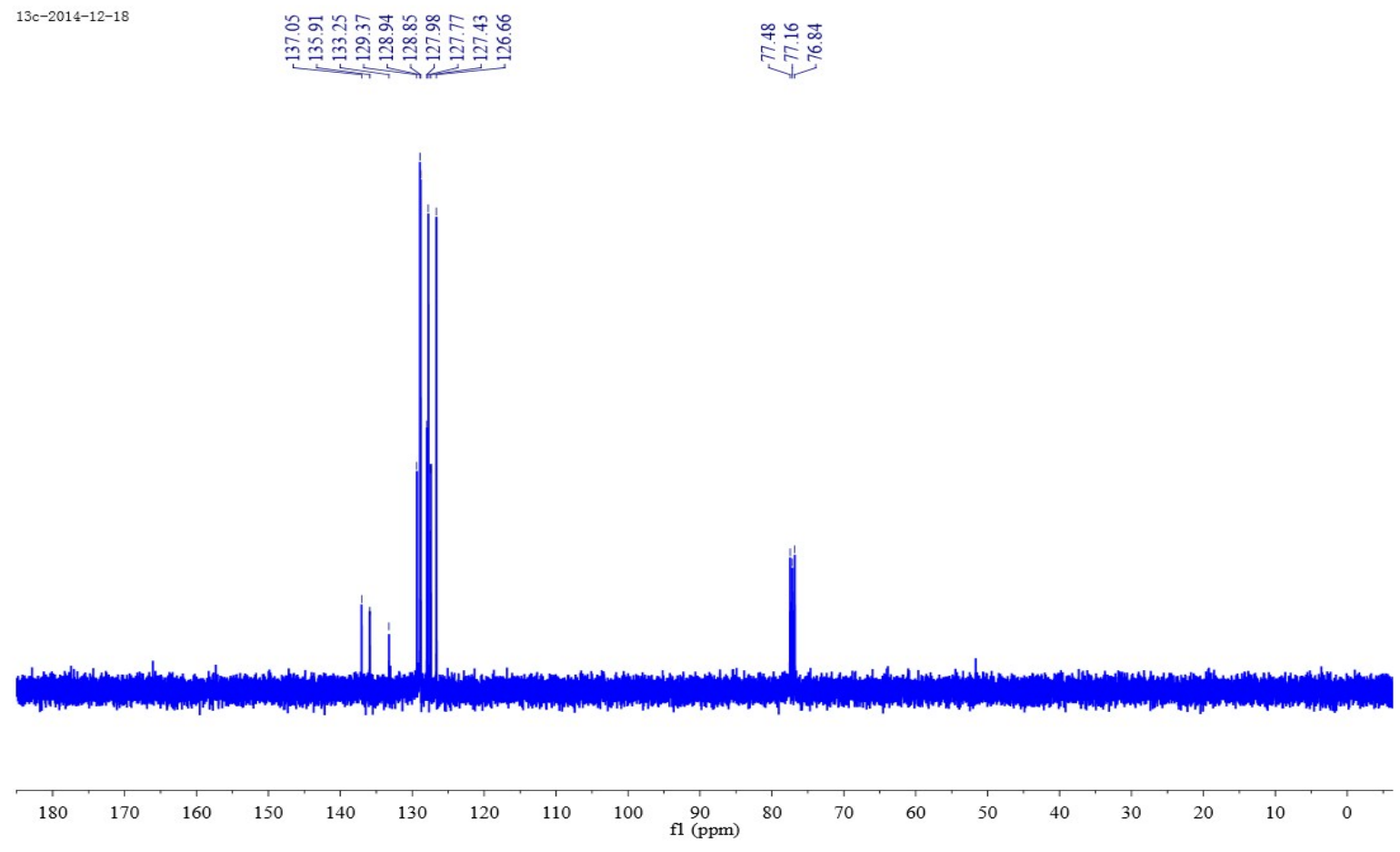


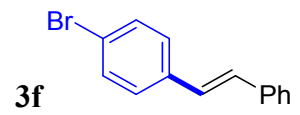
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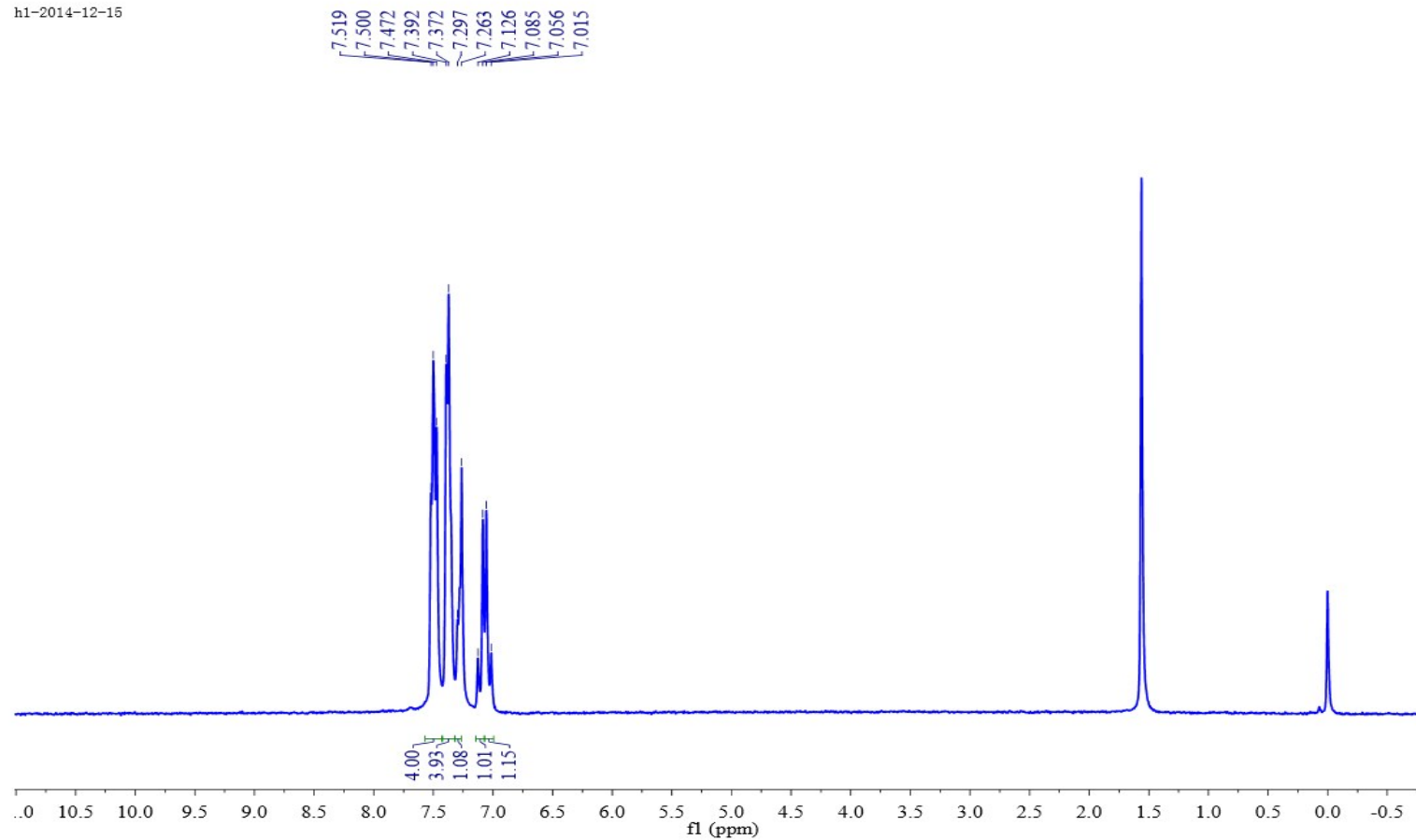


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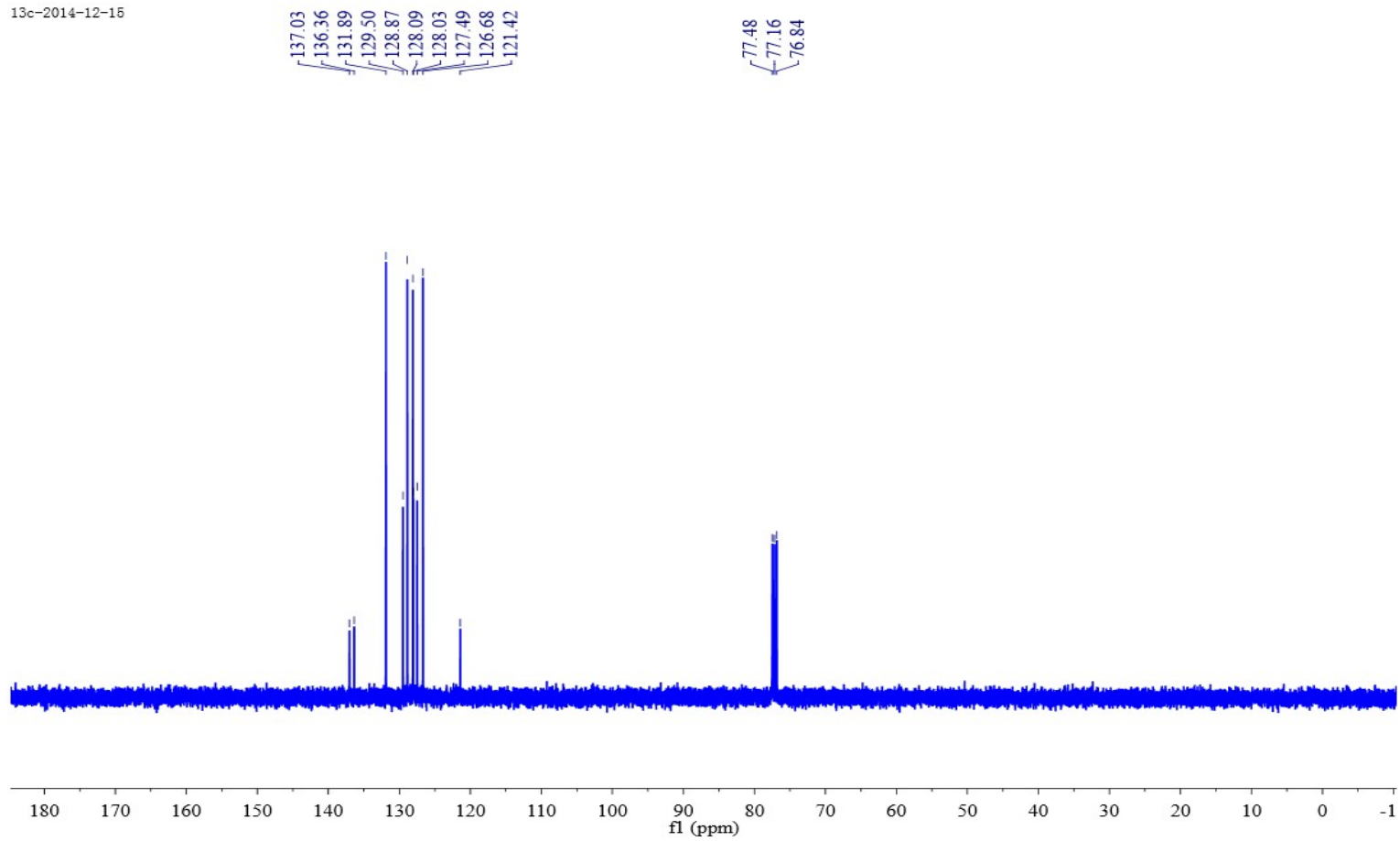
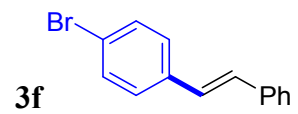


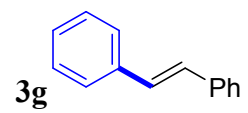


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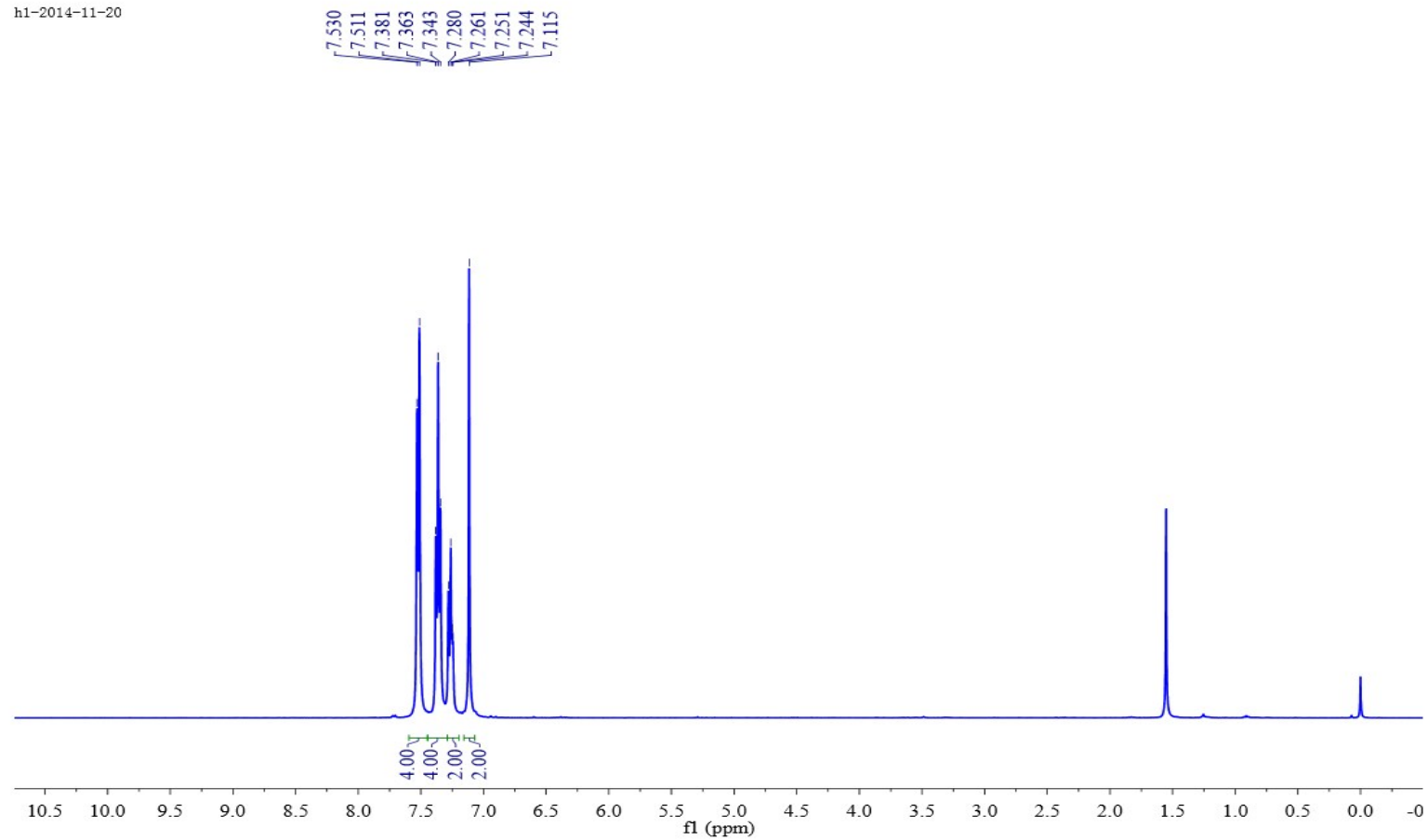


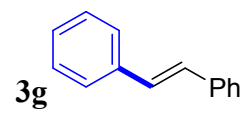
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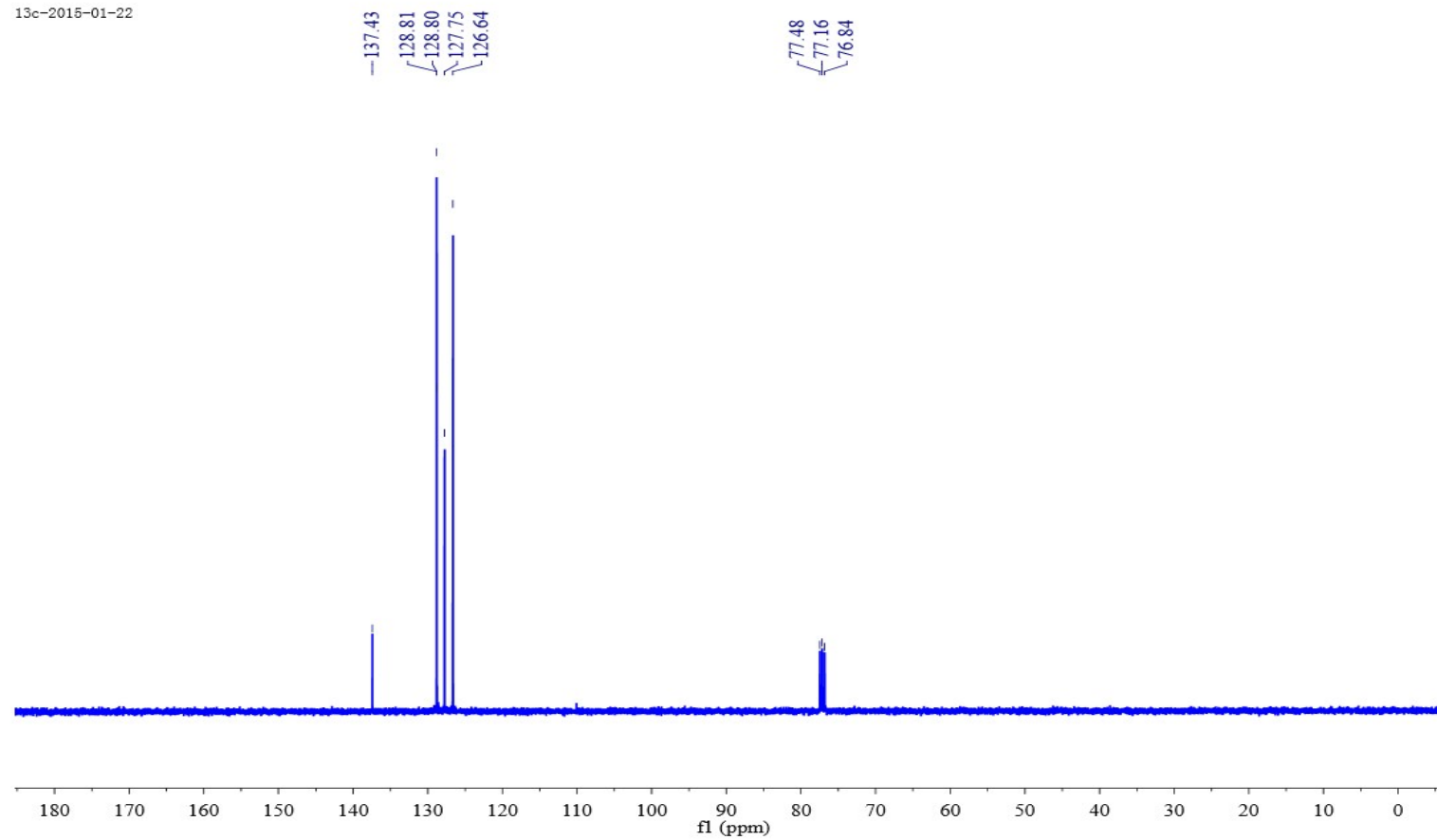


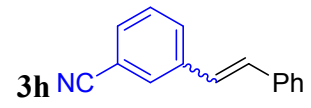
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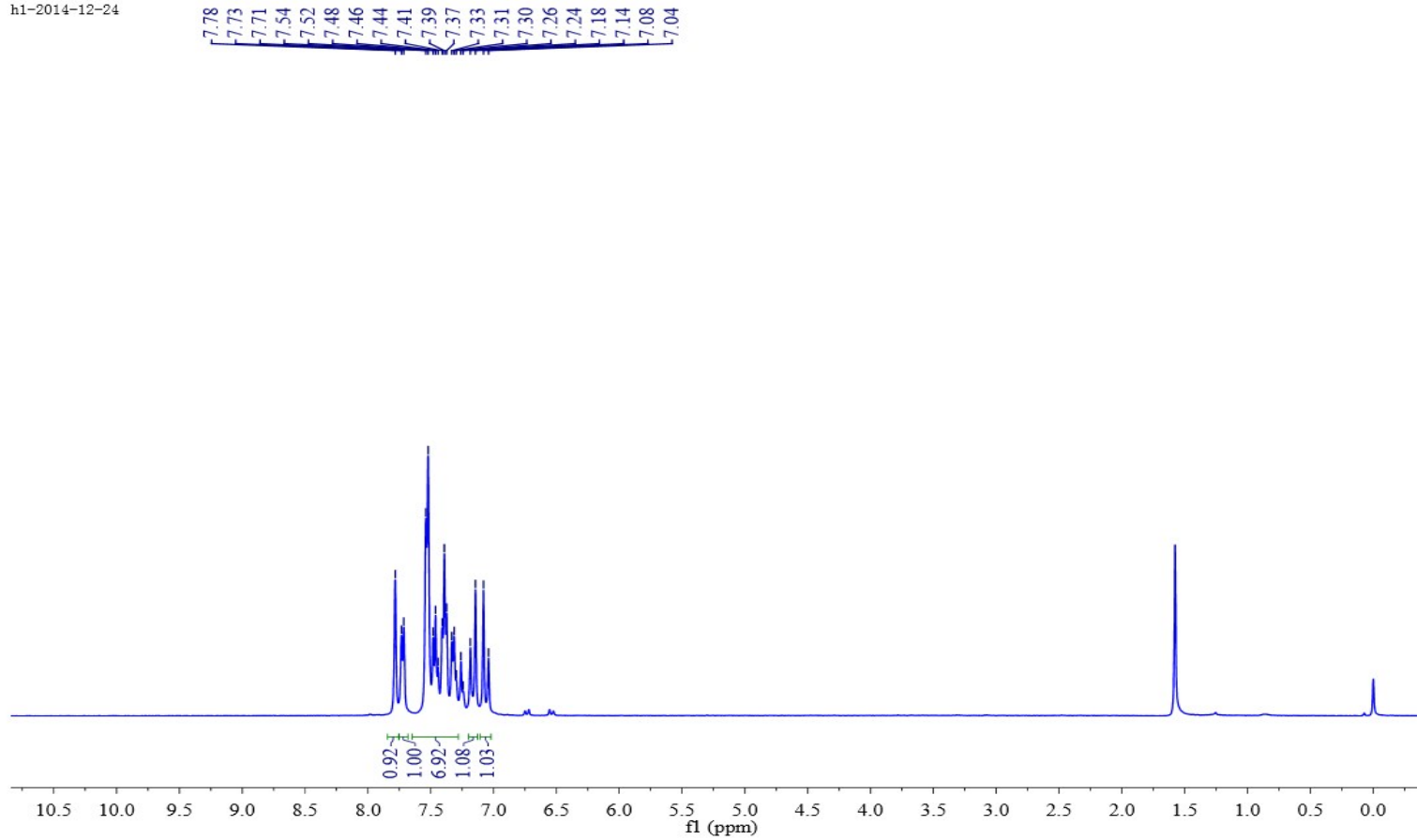


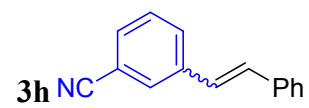
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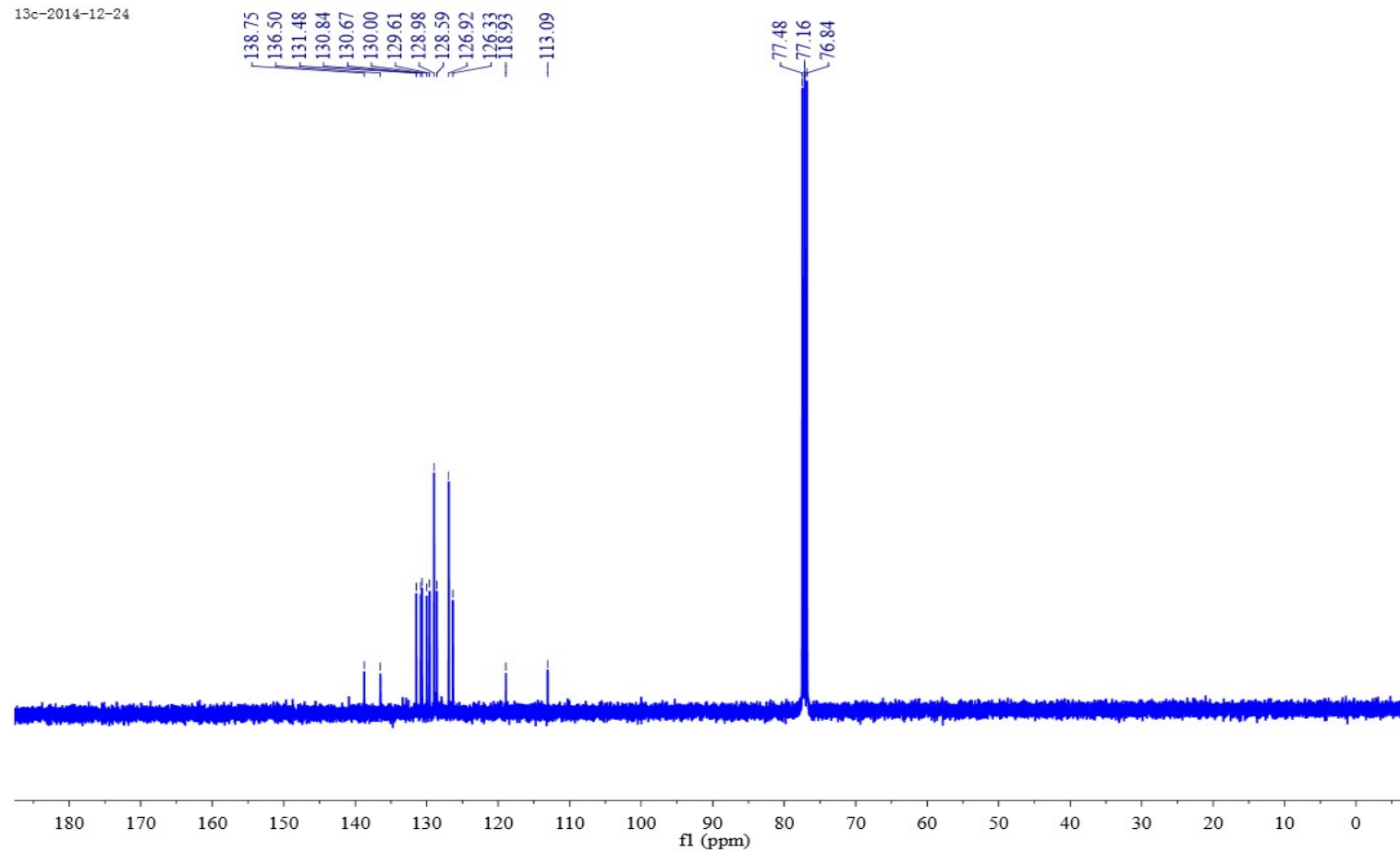


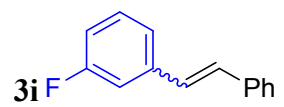
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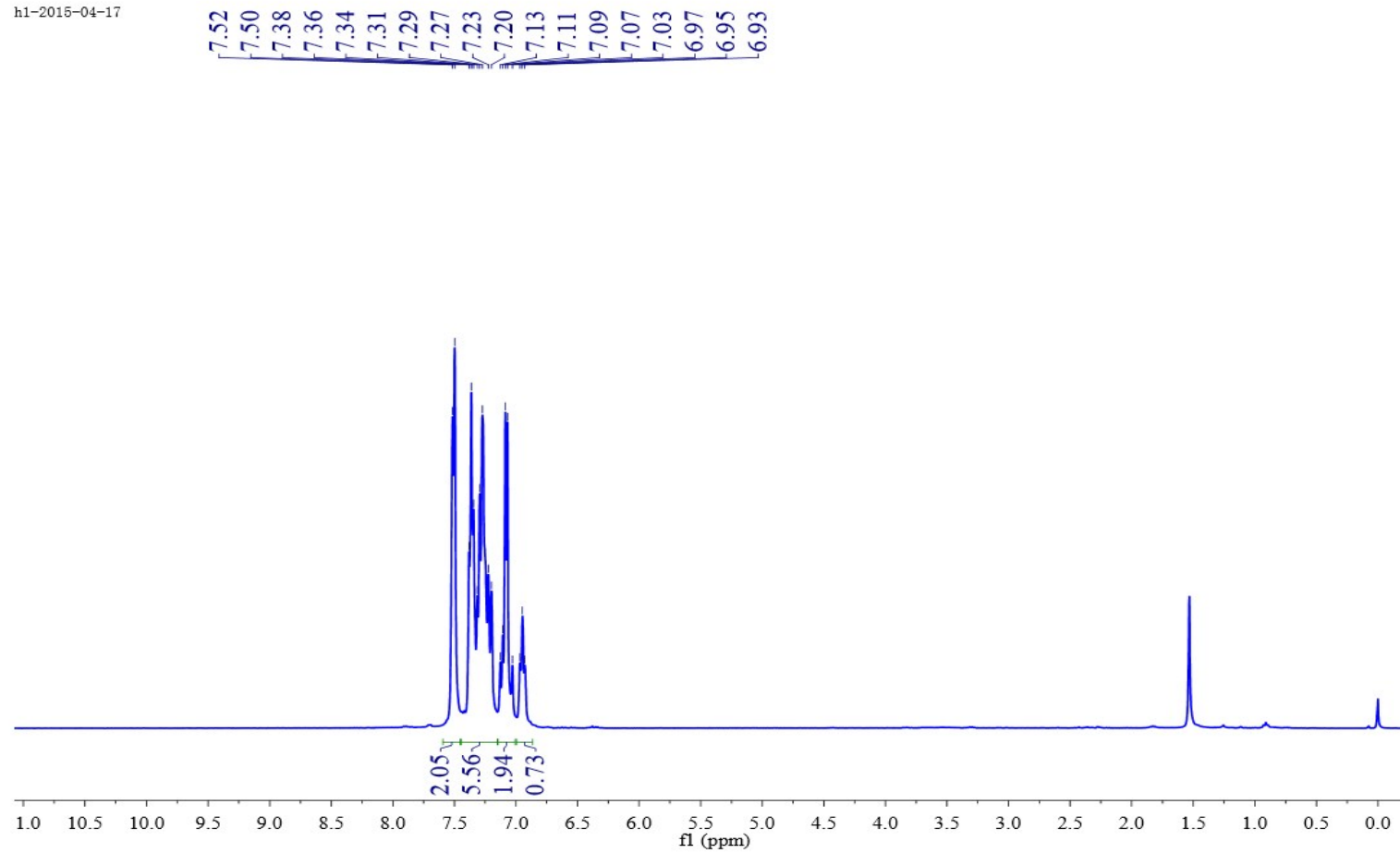


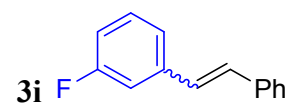
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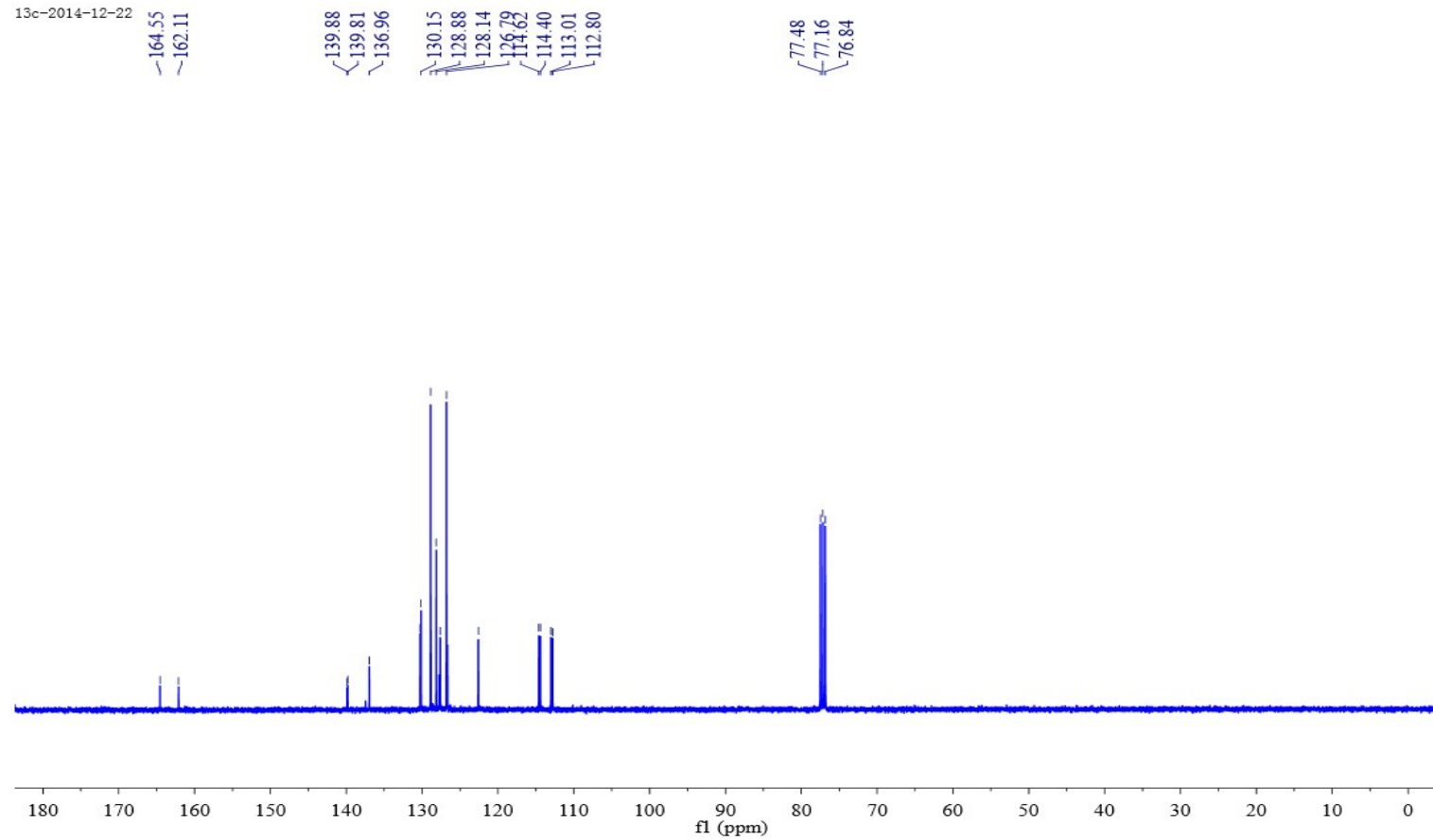


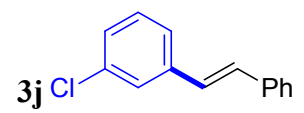
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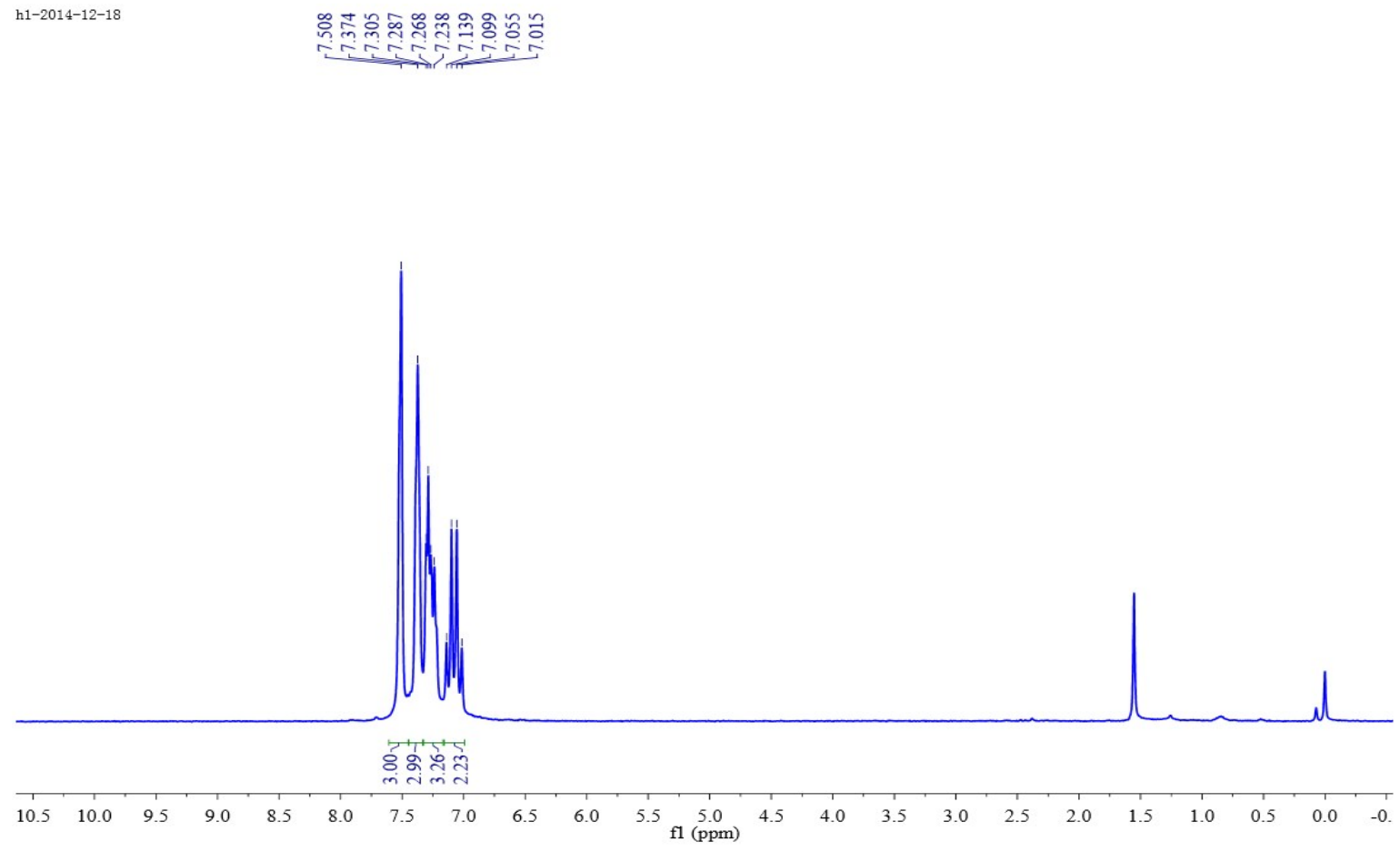


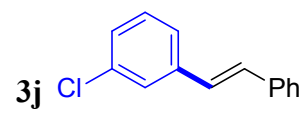
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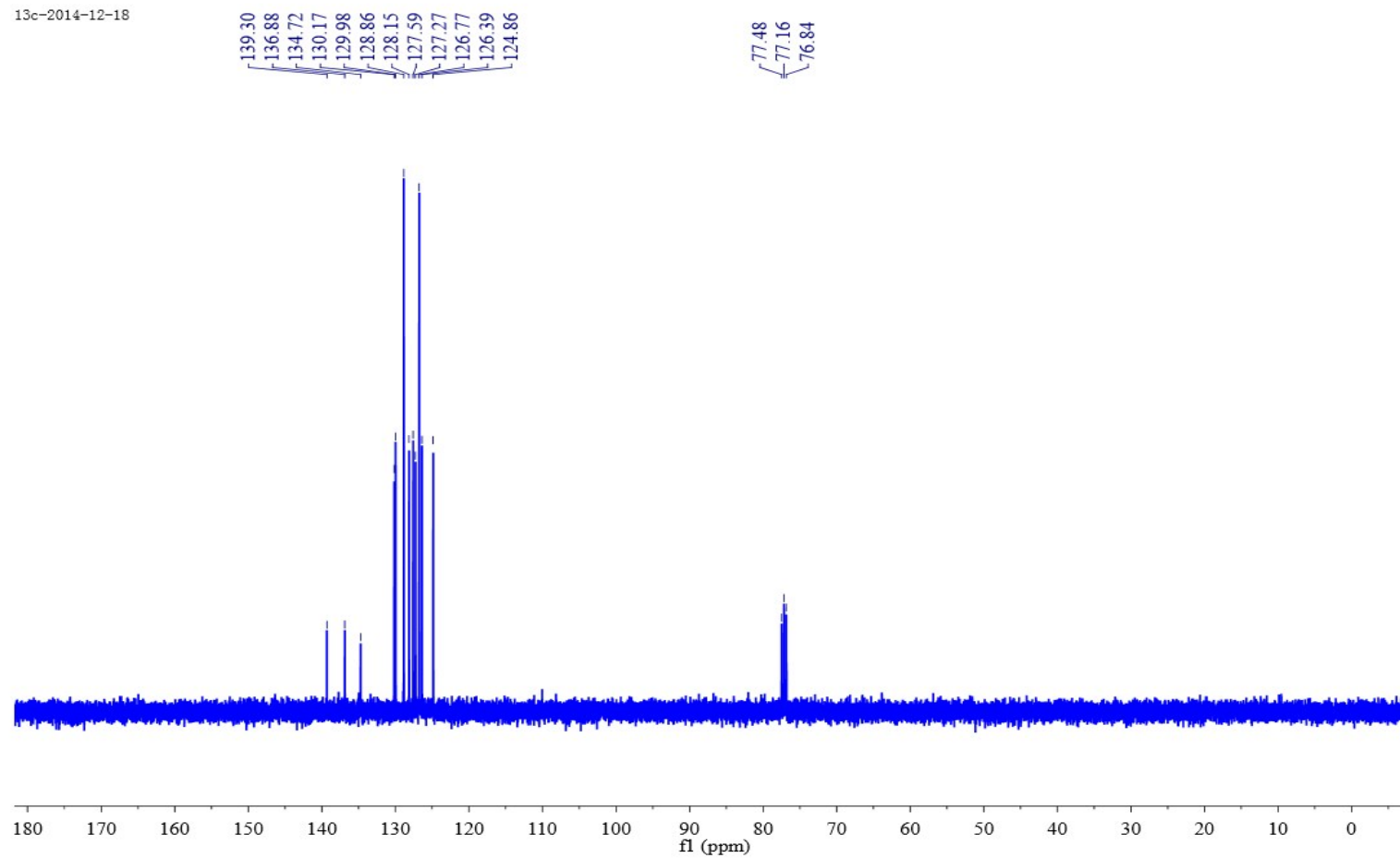


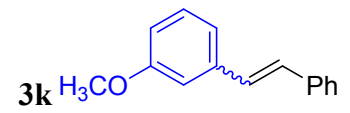
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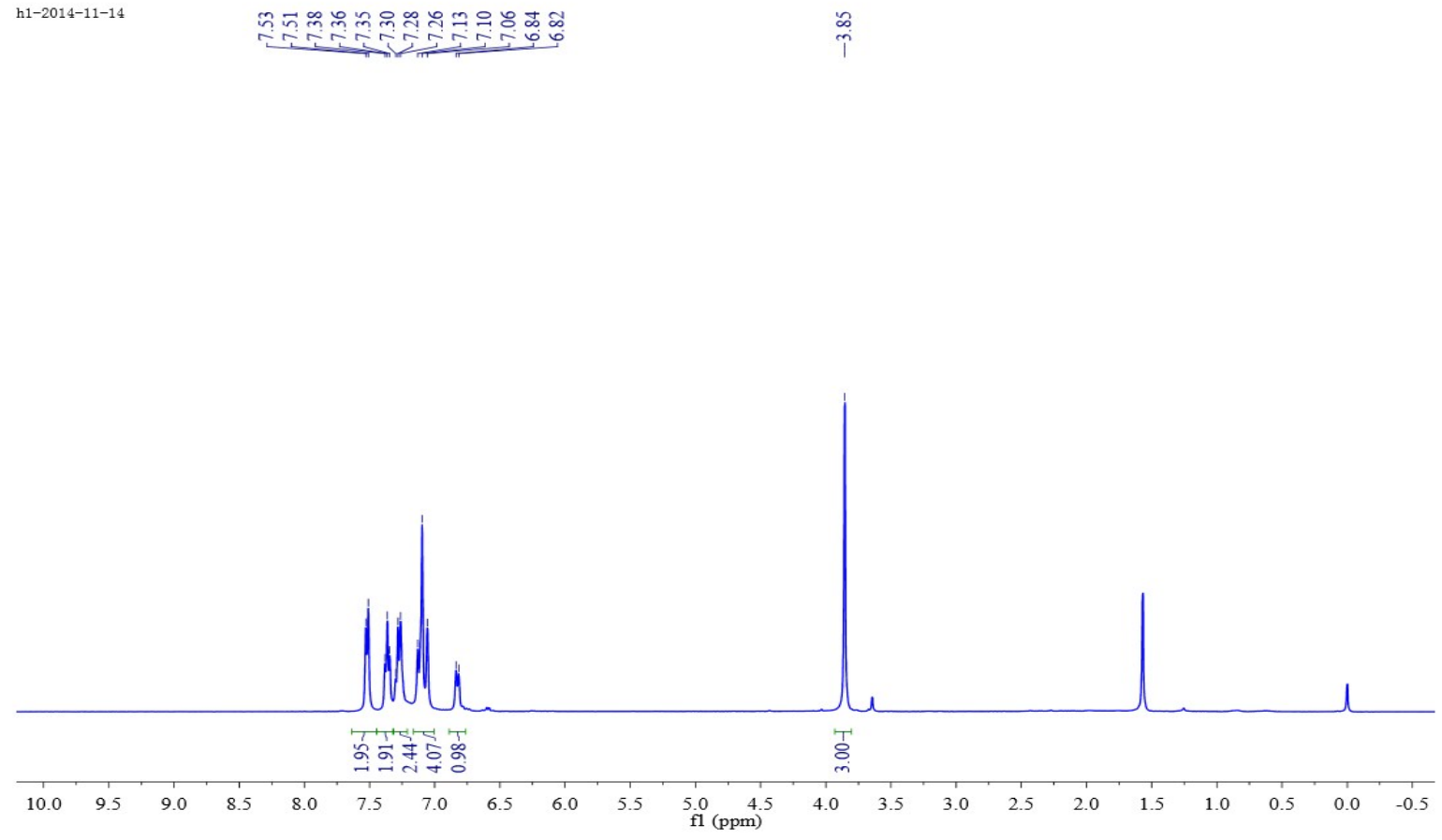


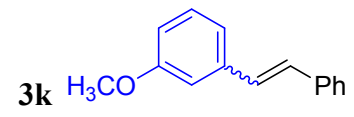
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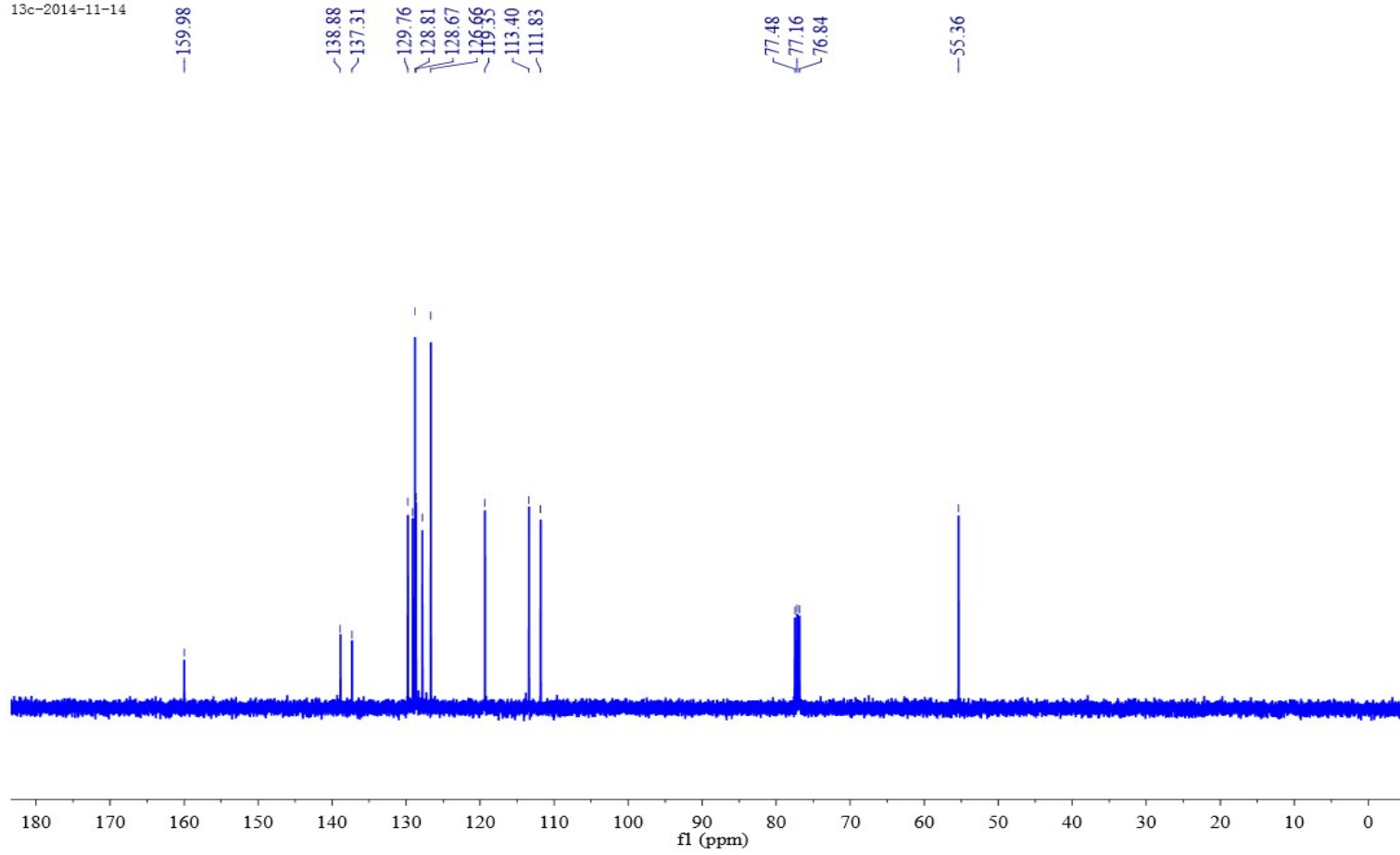


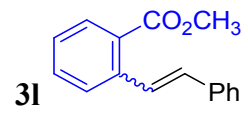
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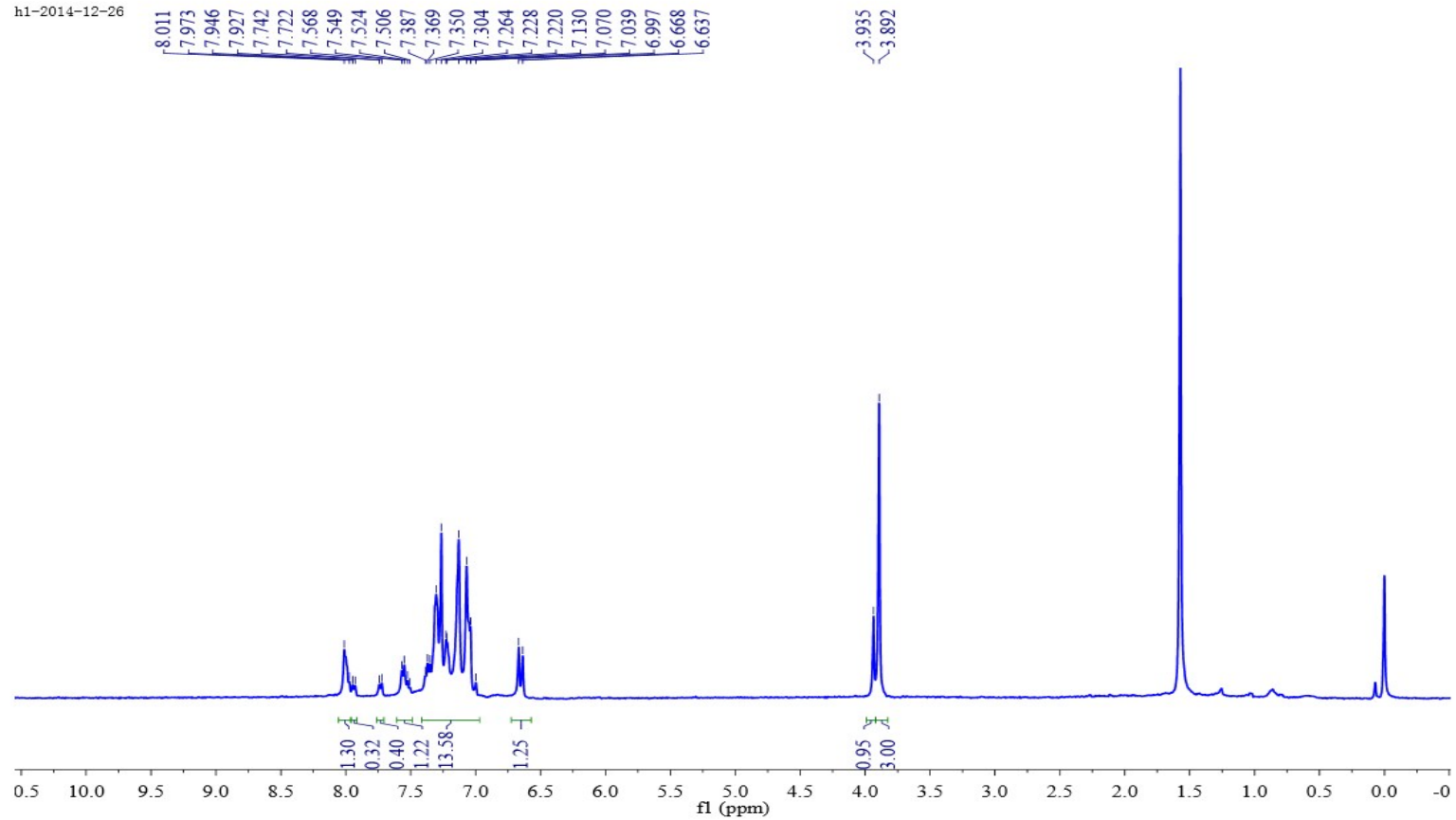


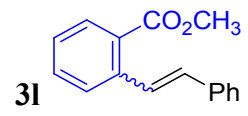
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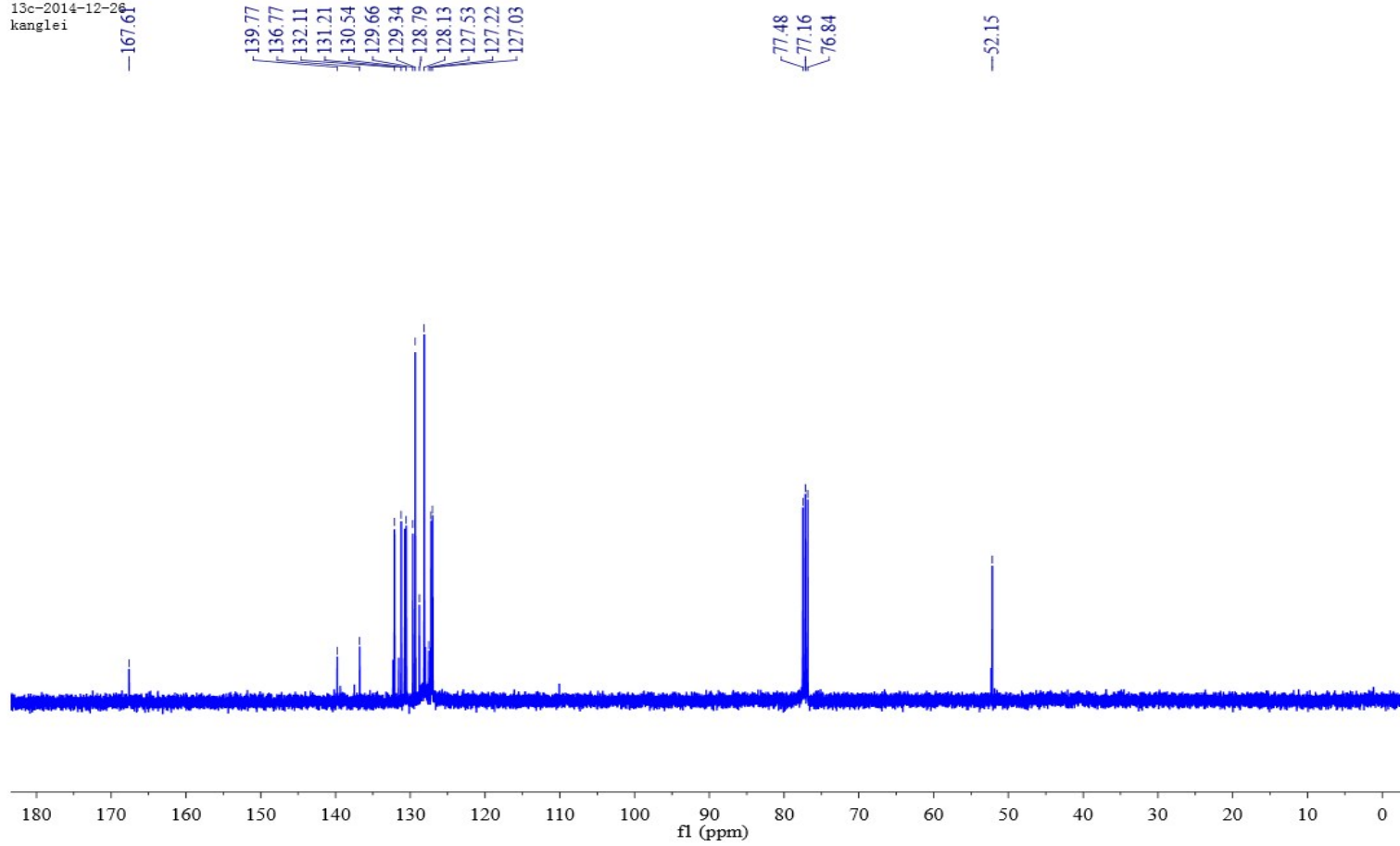


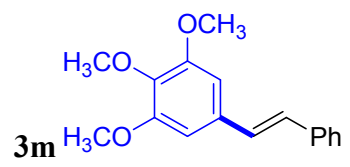
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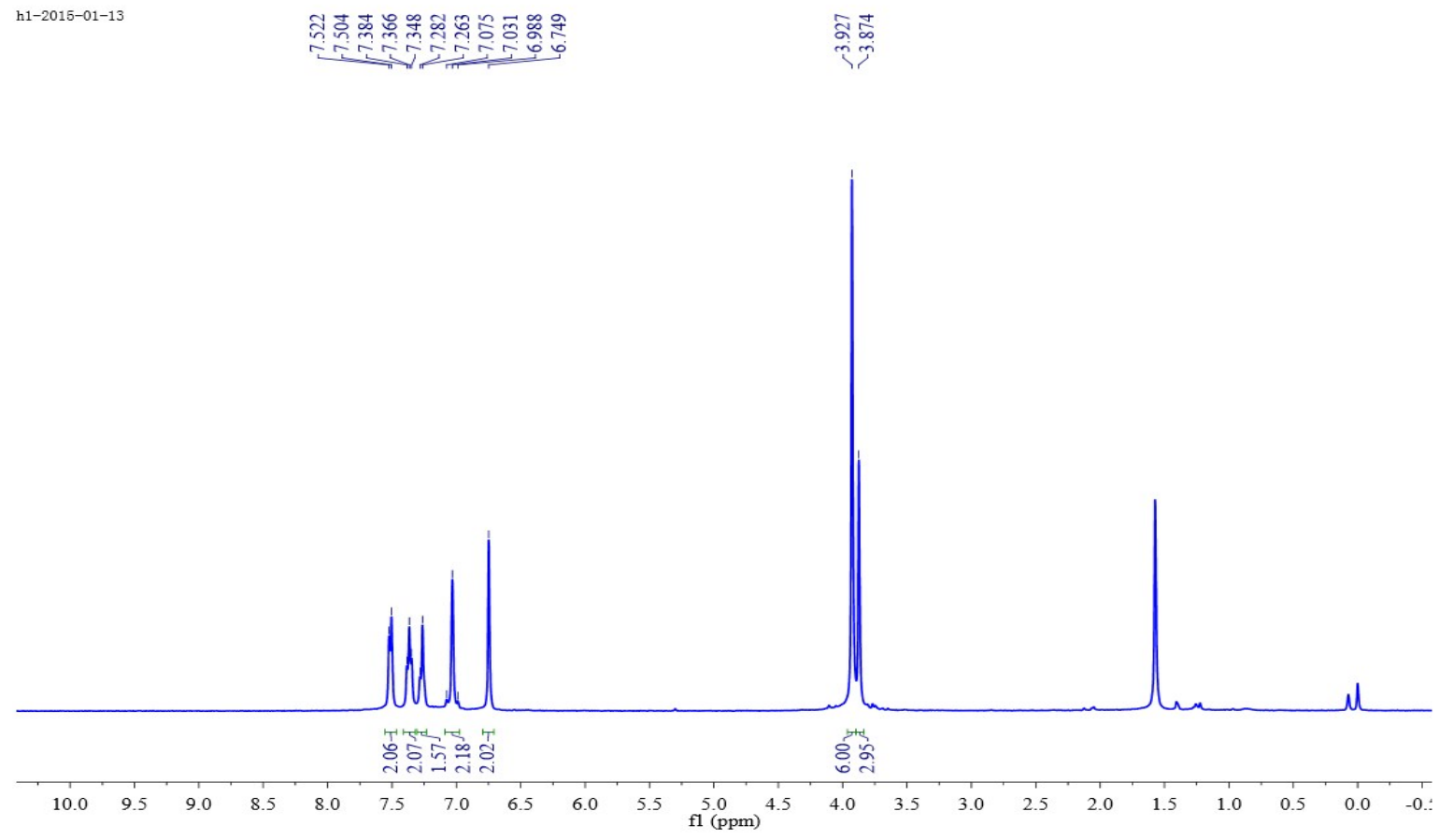


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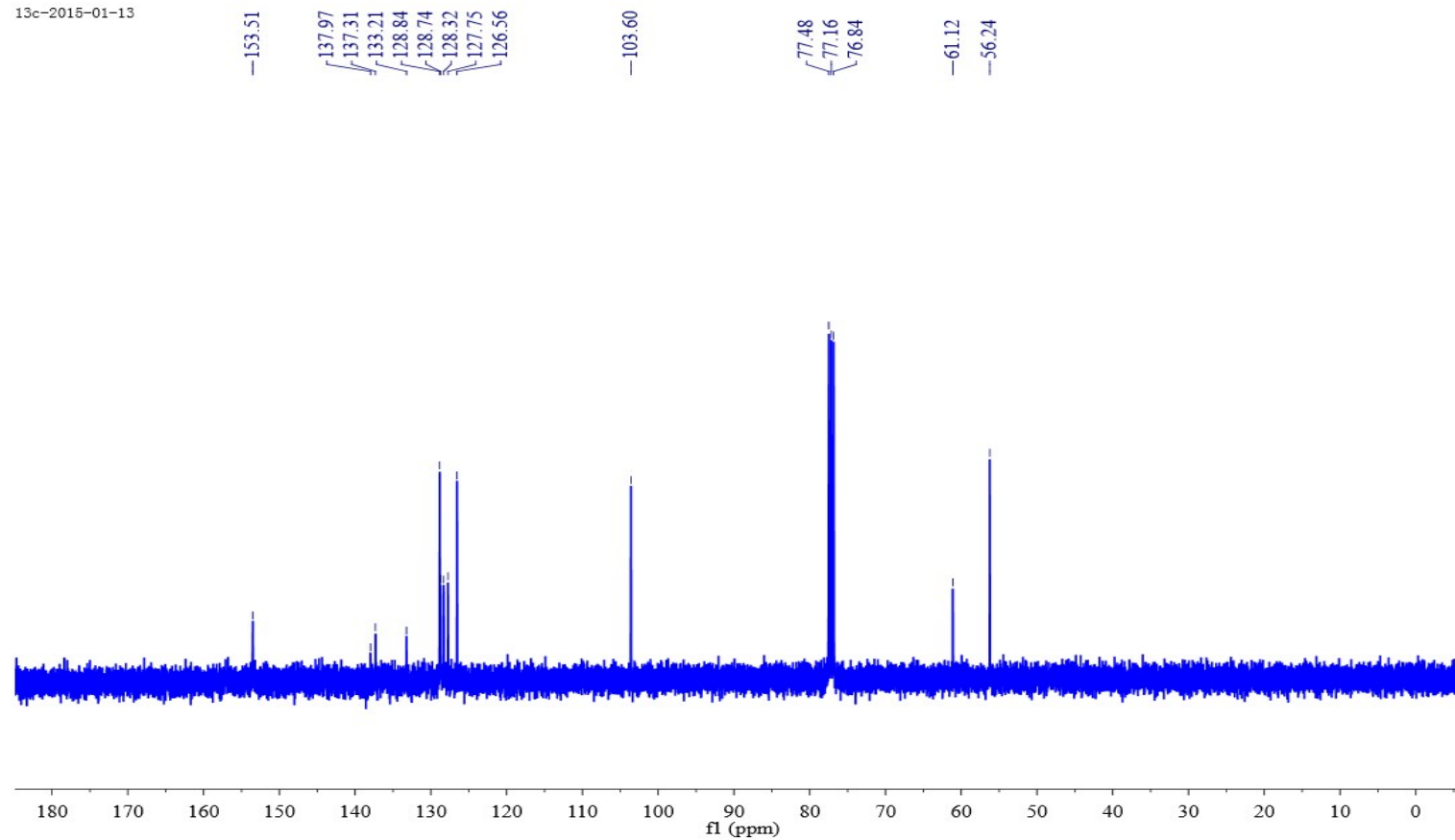
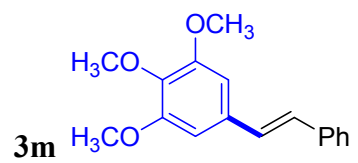


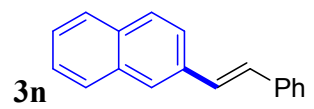


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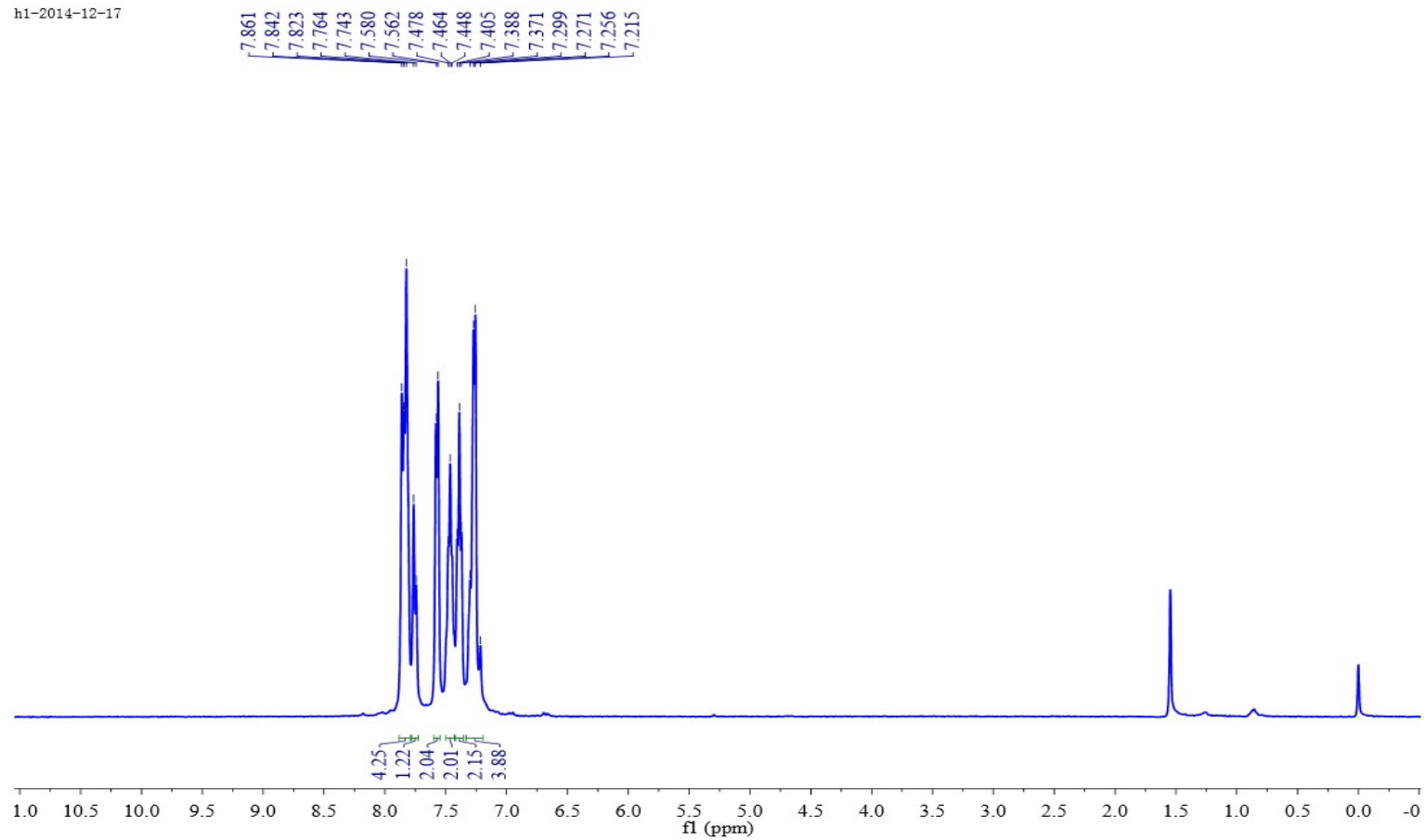


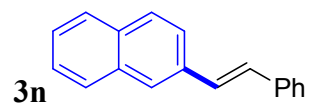
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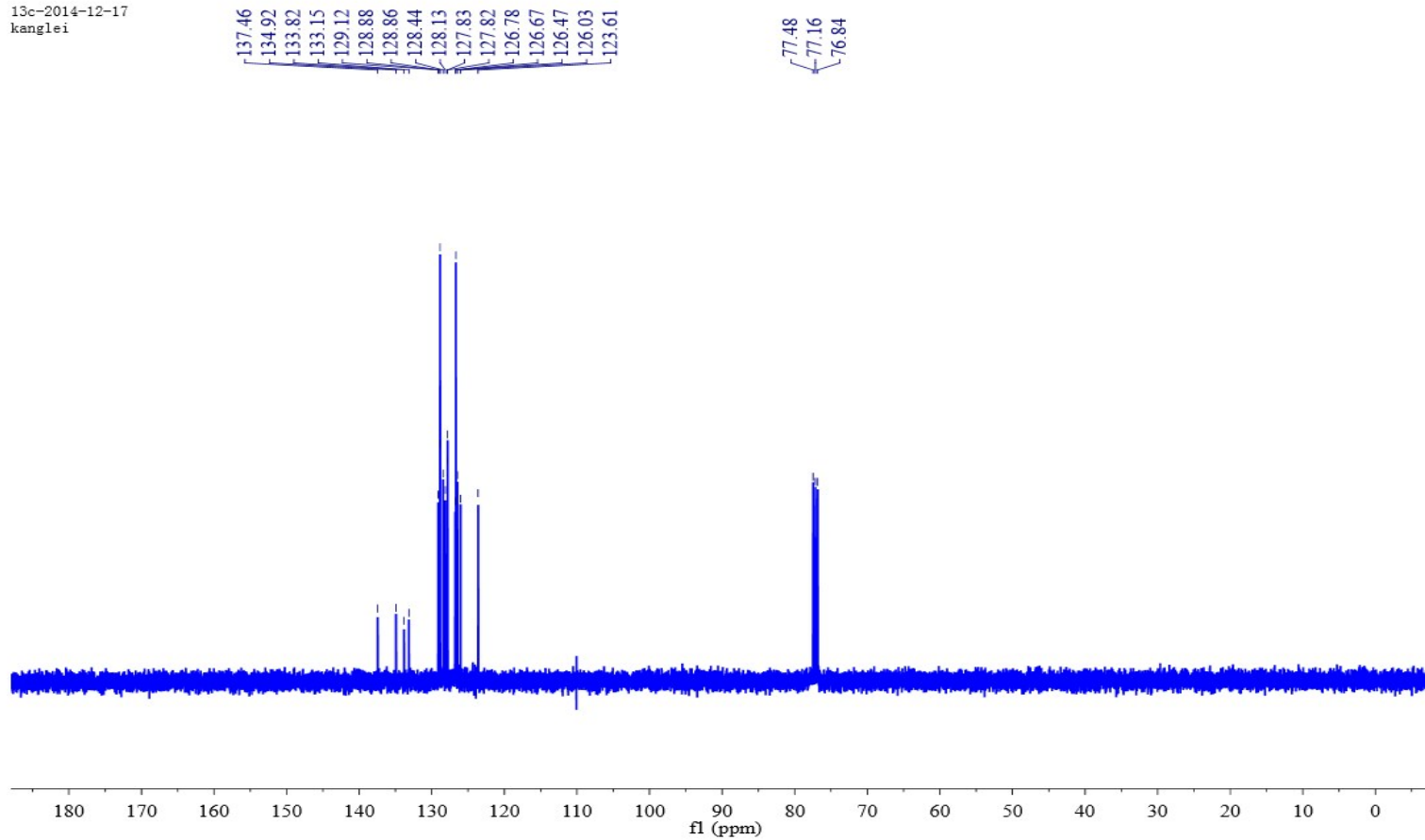


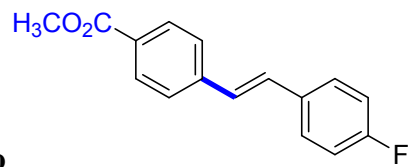
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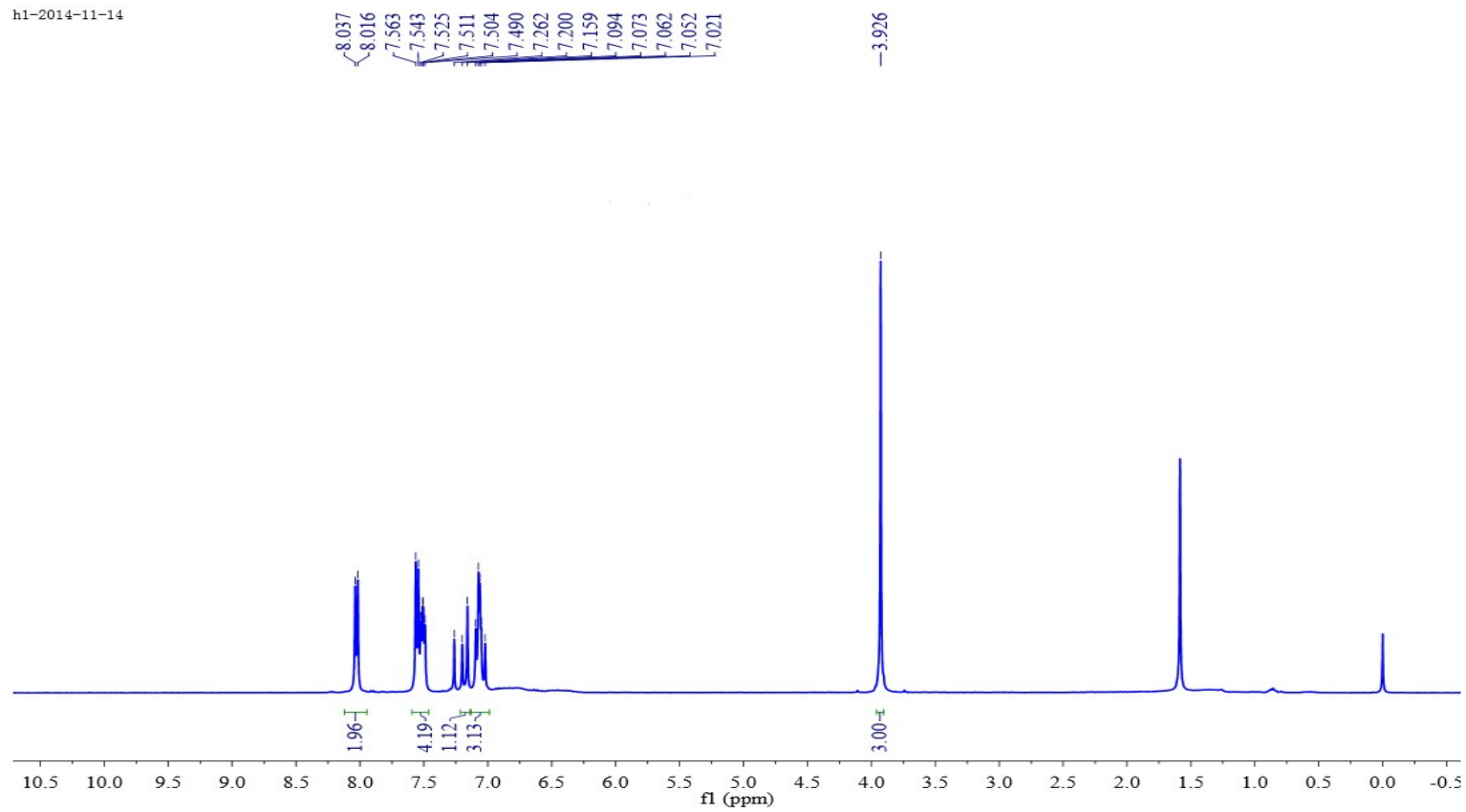
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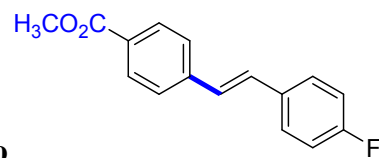




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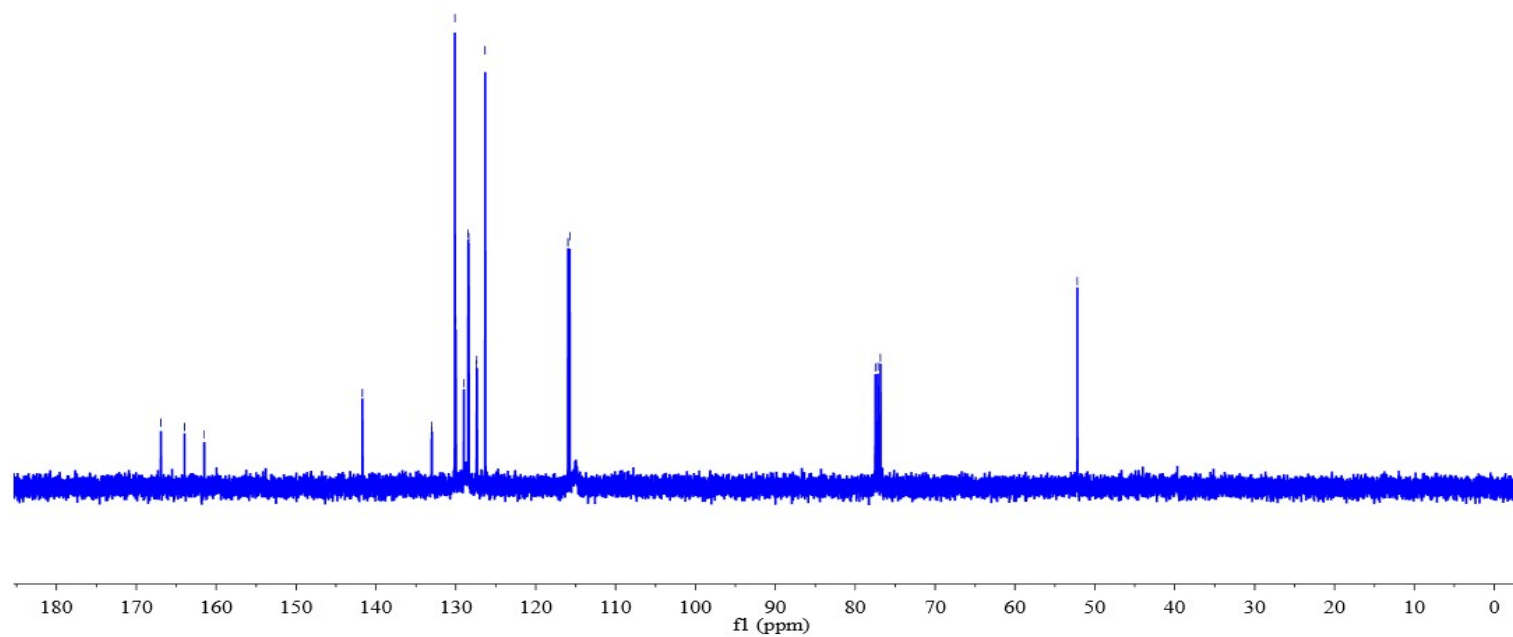
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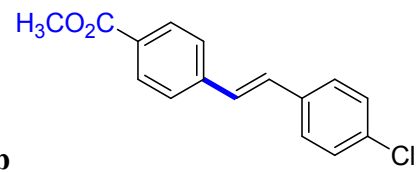
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~166.94
~163.97
~161.50

~141.71
~130.13
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~128.47
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~127.38
~116.34
~115.96
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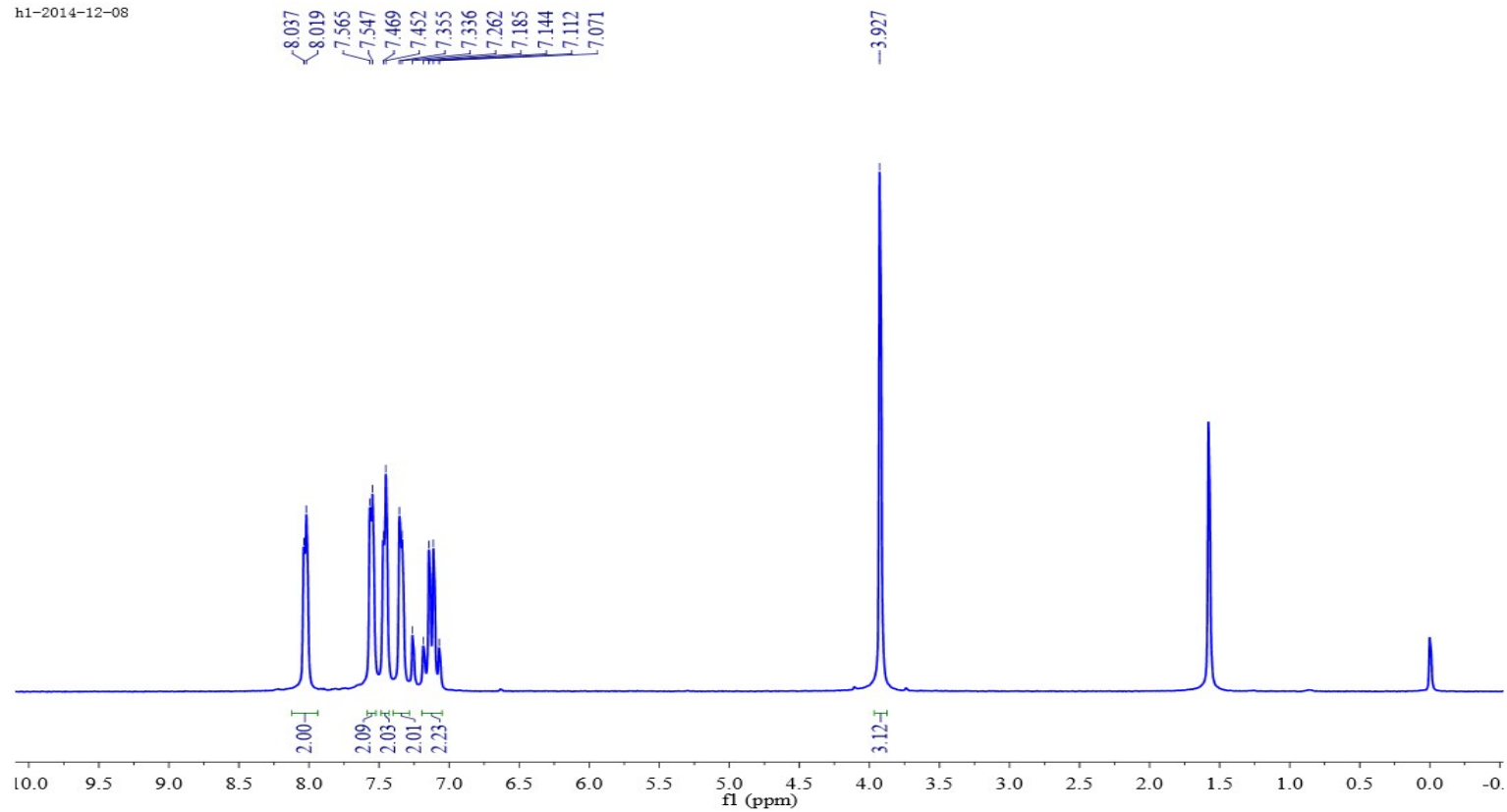
~77.48
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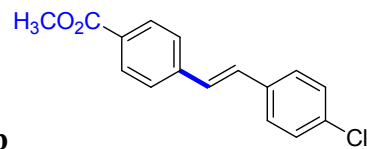
~52.19





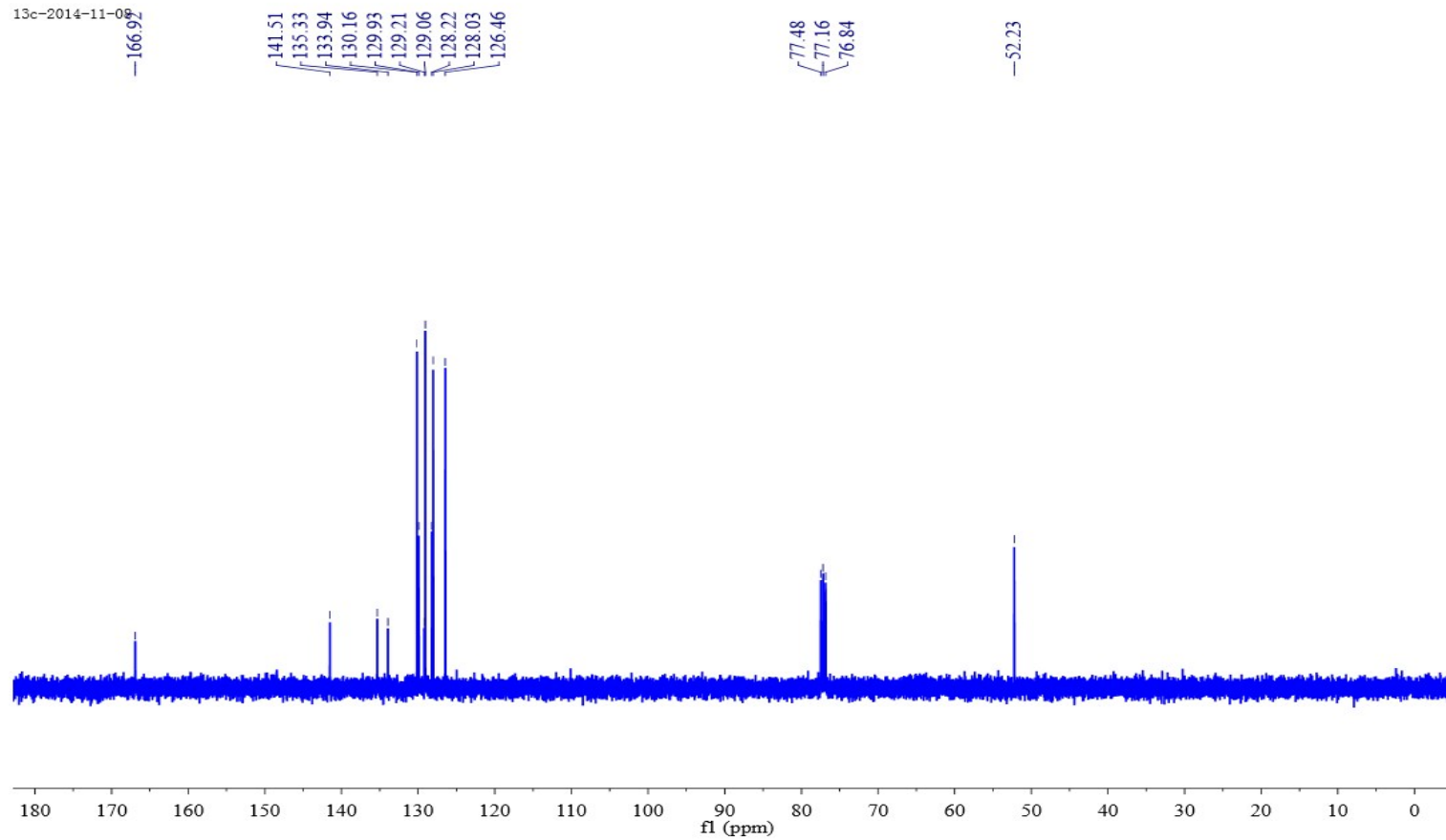
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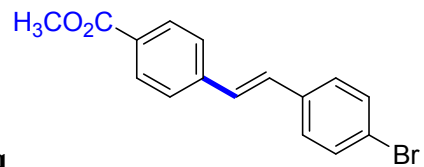




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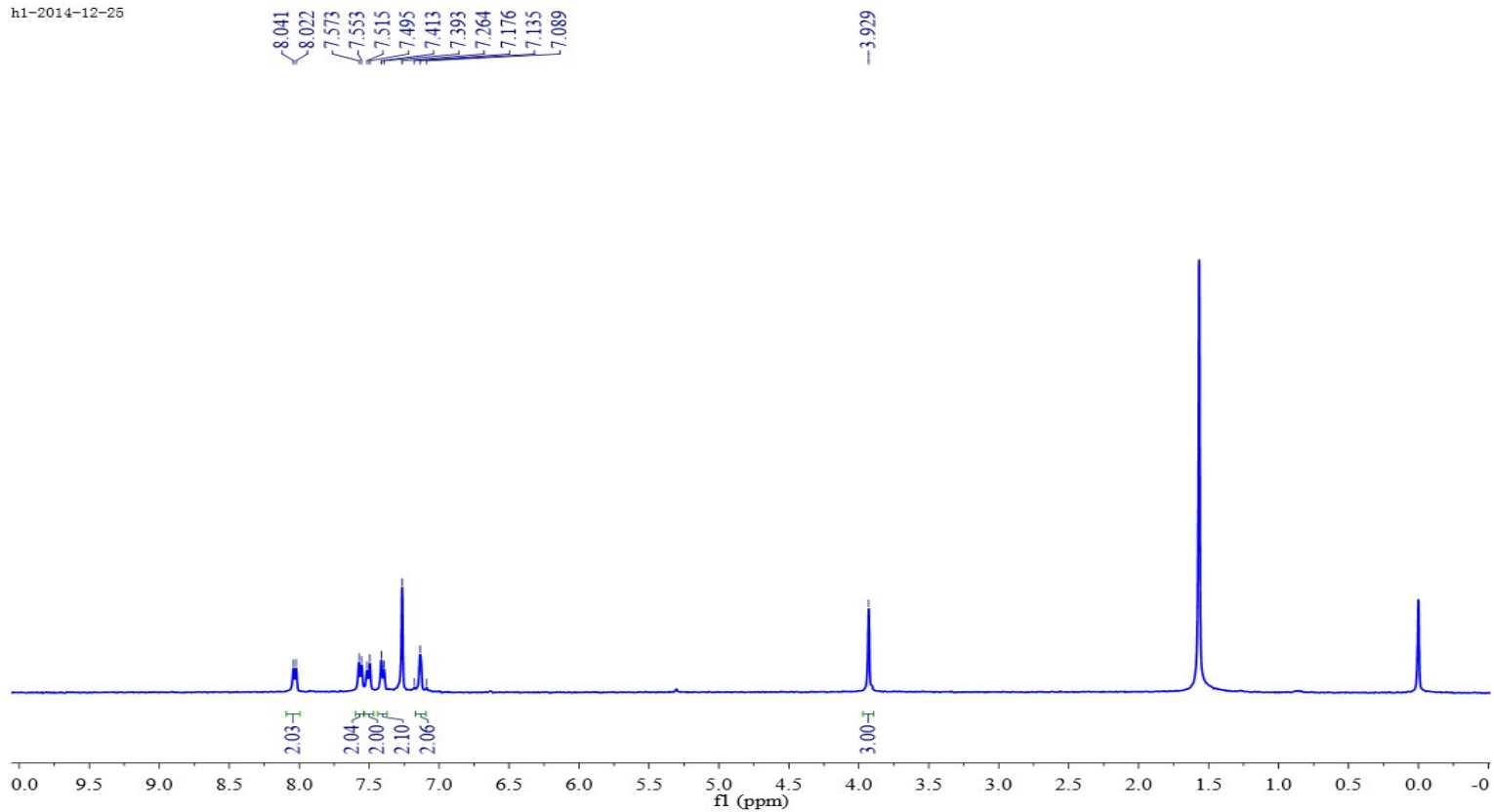
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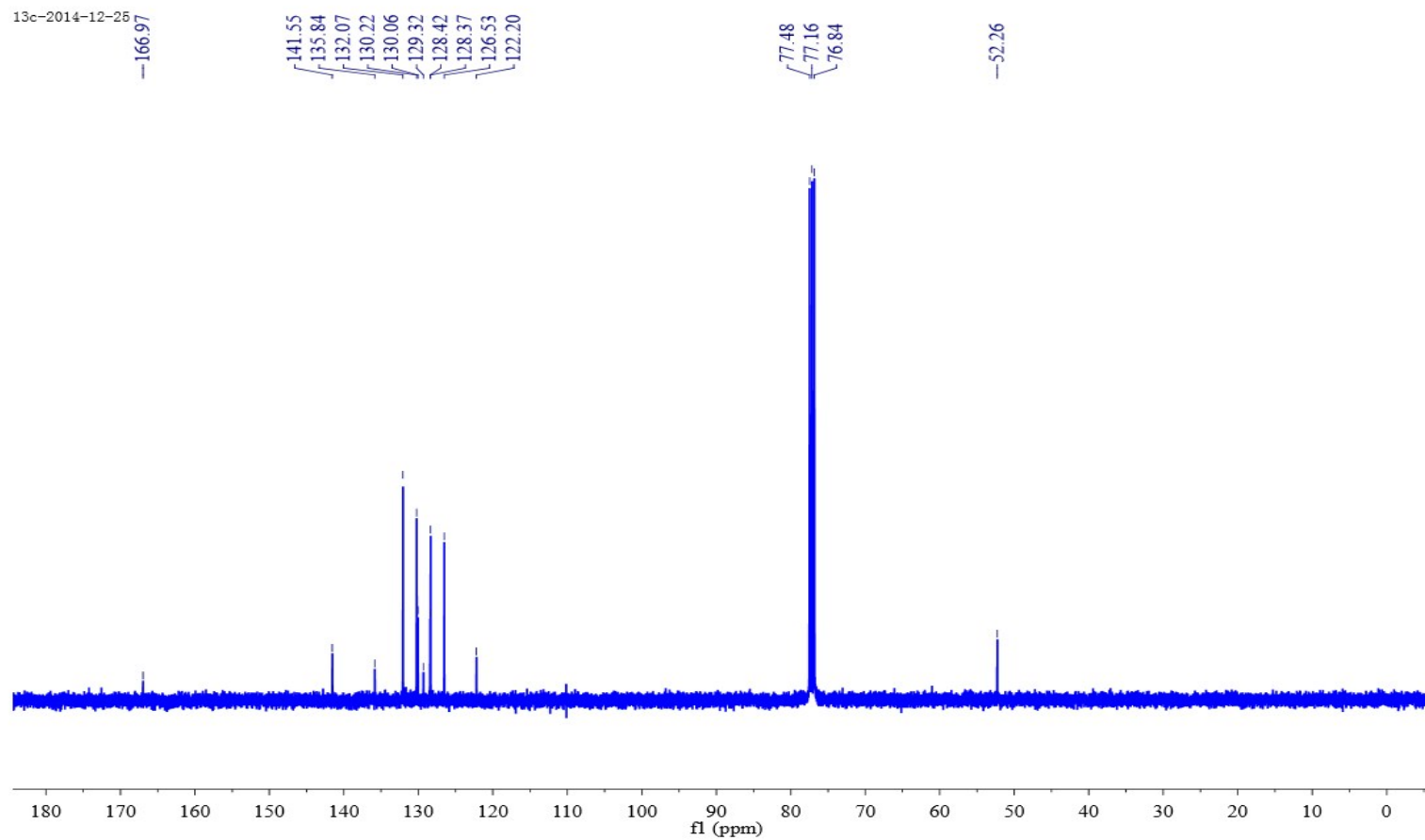
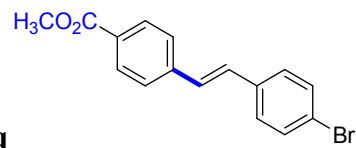


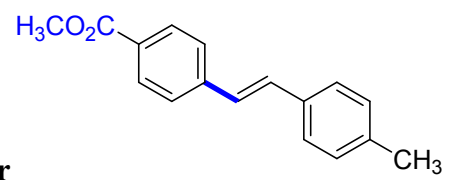


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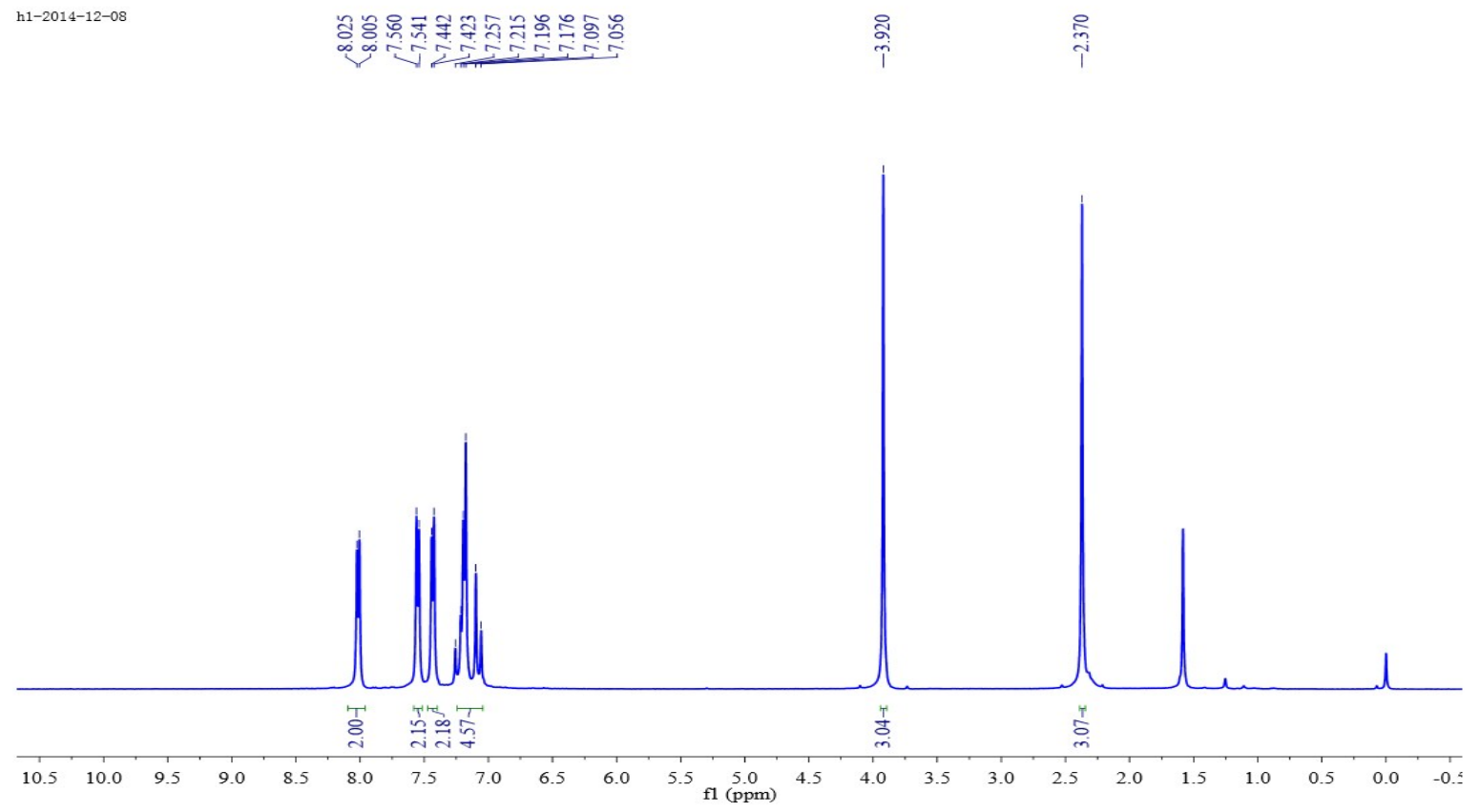
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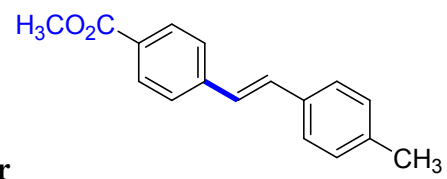




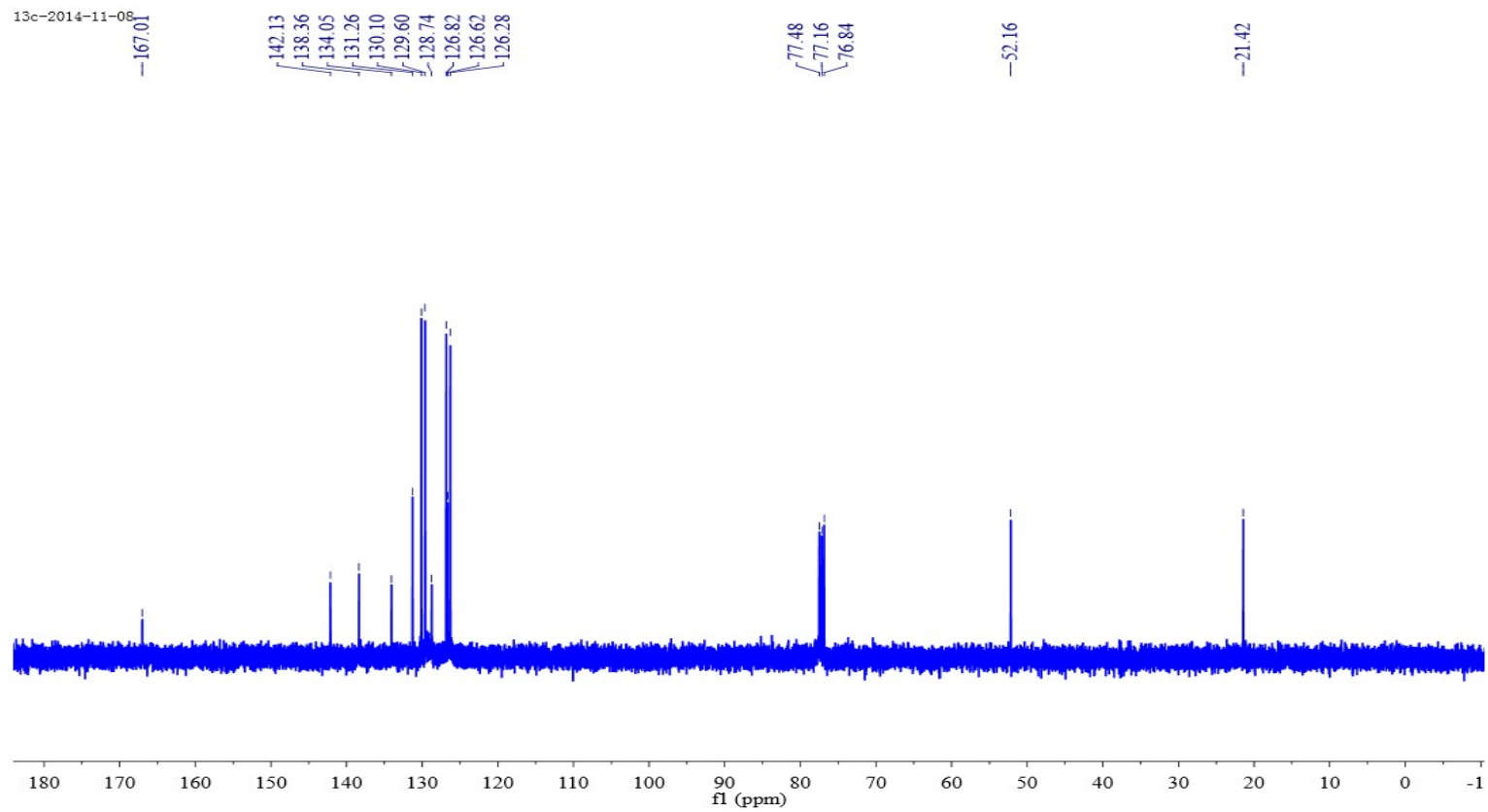


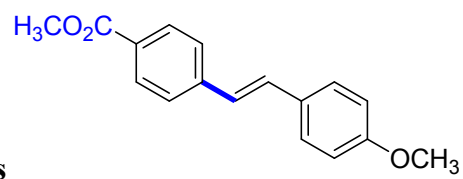
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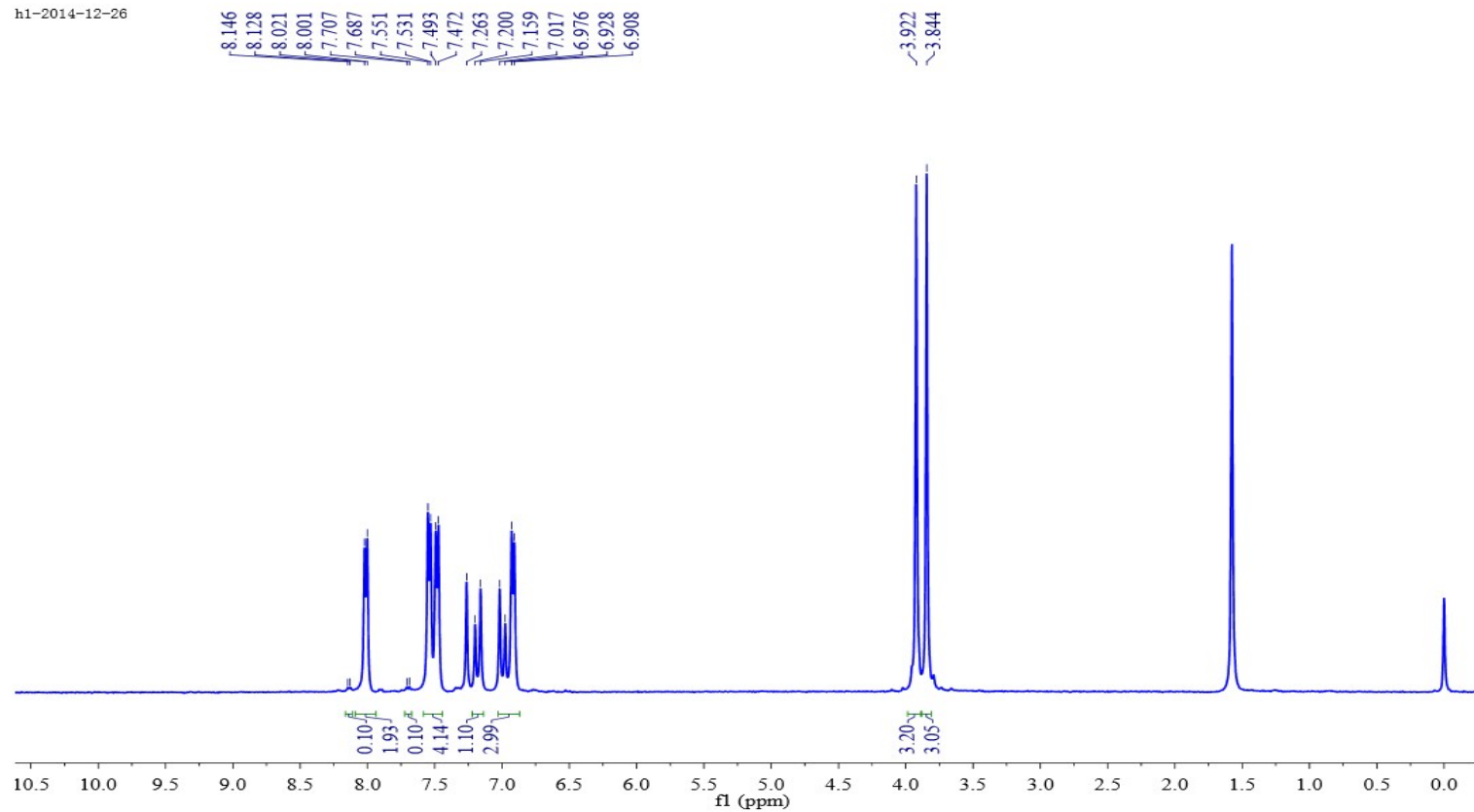


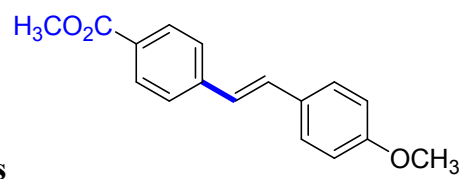
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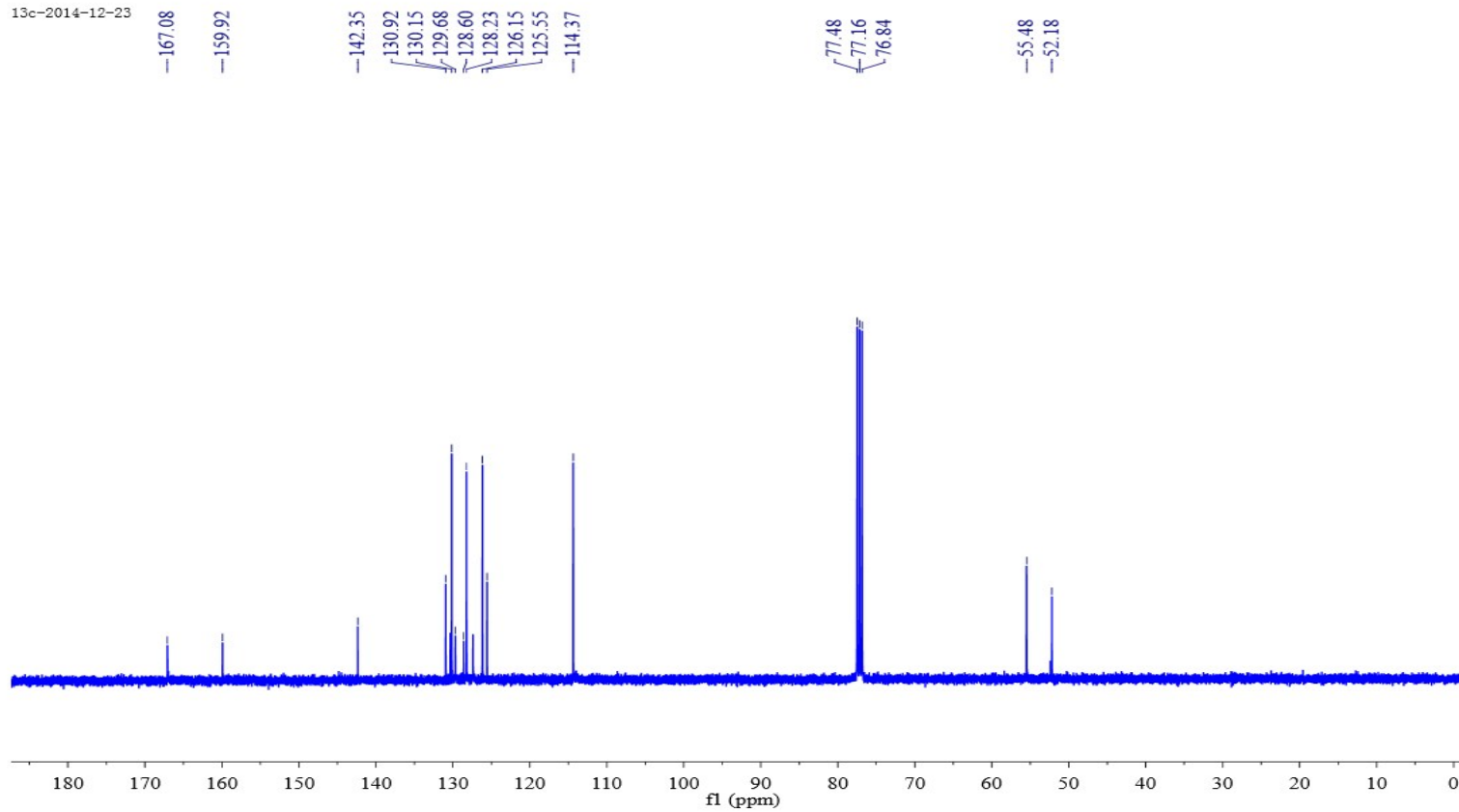
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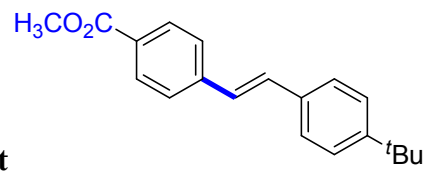




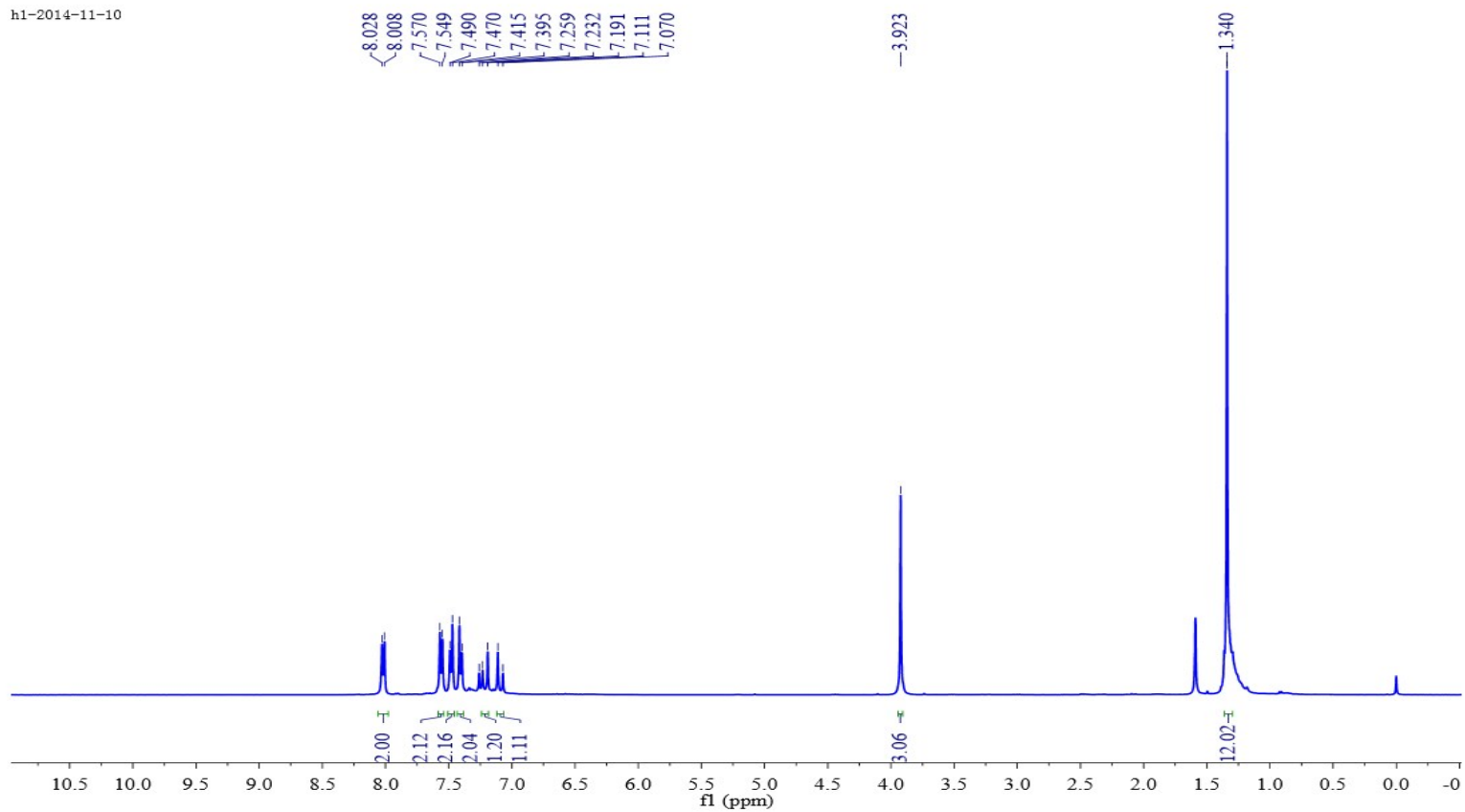
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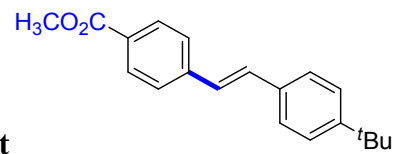
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h1-2014-11-10





3t

