

Supporting Information

Transition-metal-free isofunctional reaction of α,β -unsaturated ketones/nitriles

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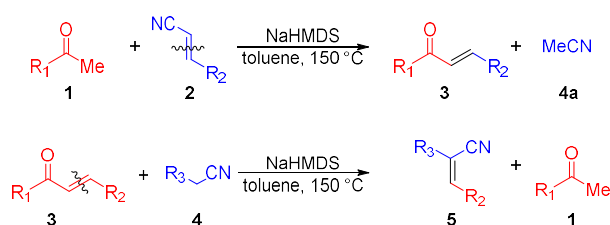
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I. General experimental information

NMR spectra were obtained on an Agilent 400-MR DD2 or a Bruker AV II-400 spectrometer. The ^1H NMR (400 MHz) chemical shifts were measured relative to CDCl_3 as the internal reference (CDCl_3 : $\delta = 7.26$ ppm). The ^{13}C NMR (100 MHz) chemical shifts were given using CDCl_3 as the internal standard (CDCl_3 : $\delta = 77.16$ ppm). High resolution mass spectra (HR-MS) were obtained with a Shimadzu LCMS-IT-TOF (ESI). Unless otherwise noted, all reagents were obtained from commercial suppliers and used without further purification.

II. General procedure

Alkenyl nitrile or alkenyl ketone C=C cleavage.



A 25 mL Schlenk tube with a magnetic stir bar was charged with **1** (0.5 mmol), **2** (0.5 mmol), NaHMDS (0.1 mL, 0.2 mmol, 2.0 M in toluene), toluene (2.0 mL). The Schlenk tube was then sealed with a Teflon lined cap and the mixture was heated at 150 °C for 20 hours. The reaction solution was then cooled to ambient temperature, neutralized with glacial acetic acid. The mixture diluted with 3 mL ethyl acetate, follow by washed with water (5 mL \times 3) and brine. The organic phases were dried over Na_2SO_4 , filtered, and concentrated in vacuo. The residue was purified by column chromatography on silica gel to provide the desired product **3**. This procedure can also be applied to the reaction of **3** and **4**.

III. Experimental procedures

3.1 Screening of Michael adducts.

Table S1. Screening bases for retro Michael of X1^a.

Entry	Base	1b ^b	2a ^b	3b ^b
1	KOH	ND	ND	trace
2	NaH	ND	ND	trace
3	CsOH	ND	ND	8%
4	Cs ₂ CO ₃	ND	ND	ND
5	LDA	ND	ND	ND
6	K ₃ PO ₄	ND	ND	ND
7	<i>t</i> -BuOK	ND	ND	14%
8	CH ₃ ONa	ND	ND	ND
9	BuLi	ND	ND	ND
10	TEA	ND	ND	ND
11	Pyridine	ND	ND	ND

^areaction conditions: **X1** (0.5 mmol) and base (0.5 mmol) were stirred in dioxane (2 mL) at 140 °C for 20 h. ^b Isolated yields.

Table S2. Screening of Michael adducts^{a, b}.

Michael adduct	σ -bond cleavage product	Michael adduct	σ -bond cleavage product	Michael adduct	σ -bond cleavage product
X1	14%	X4	25%	X7	ND
X2	11%	X5	32%	X8	ND
MA-1	41%	MA-2	34%	X9	ND
X3	27%	X6	ND		

^areaction conditions: Michael adduct (0.5 mmol) and *t*-BuOK (0.5 mmol) were stirred in dioxane (2 mL) at 140 °C for 20 h. ^bIsolated yields.

3.2 Optimization of Reaction Conditions.

Table S3. Screening of bases^a.

1a + 2a $\xrightarrow[\text{dioxane, 140 } ^\circ\text{C, 20 h}]{\text{base}}$ 3a

Entry	Base	Quantity	Yield ^b
1	NaOH	1.0	21%
2	NaH	1.0	31%
3	CsOH	1.0	26%
4	Piperidine	1.0	ND
5	<i>t</i> -BuOK	1.0	37%
6	<i>t</i> -BuONa	1.0	39%
7	CH ₃ COONa	1.0	ND
8	NaHMDS	1.0	56%
9	NaHMDS	0.2	50%
10	NaHMDS	0.4	64%
11	NaHMDS	0.6	61%
12	NaHMDS	0.8	57%
13	NaHMDS	1.2	49%
14	NaHMDS	1.4	41%

^areaction conditions: **1a** (0.5 mmol), **2a** (0.5 mmol) and base were stirred in dioxane (2 mL) at 140 °C for 20 h. ^b Isolated yields.

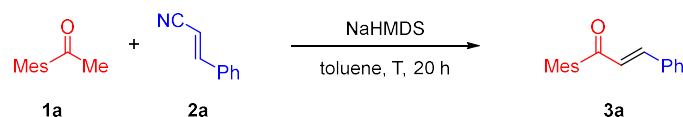
Table S4. Screening of solvents^a.

1a + 2a $\xrightarrow[\text{solvent, 140 } ^\circ\text{C, 20 h}]{\text{NaHMDS}}$ 3a

Entry	Solvent	Yield ^b
1	dioxane	64%
2	DMSO	14%
3	DEM	56%
4	DMF	20%
5	EA	ND
6	EtOH	ND
7	THF	trace
8	toluene	79%
9	cyclohexane	41%
10	mesitylene	75%
11	PhCl	48%

^areaction conditions: **1a** (0.5 mmol), **2a** (0.5 mmol) and NaHMDS (0.4 eq.) were stirred in solvent (2 mL) at 140 °C for 20 h. ^b Isolated yields.

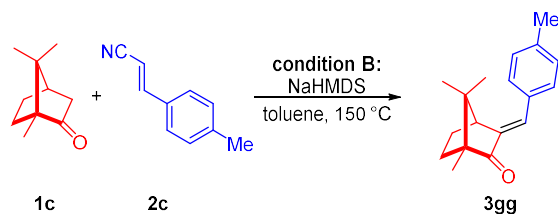
Table S5. Screening of temperature^a.



Entry	T	Yield ^b
1	110 °C	17%
2	130 °C	55%
3	140 °C	79%
4 (condition B)	150 °C	84%
5	160 °C	82%

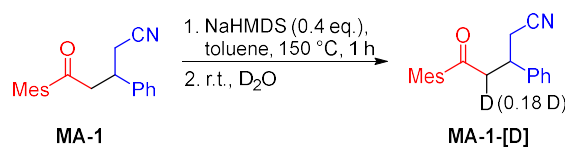
^areaction conditions: **1a** (0.5 mmol), **2a** (0.5 mmol) and NaHMDS (0.4 eq.) were stirred in toluene (2 mL) for 20 h. ^b Isolated yields.

3.3 A scale-up experiment of 3ff.

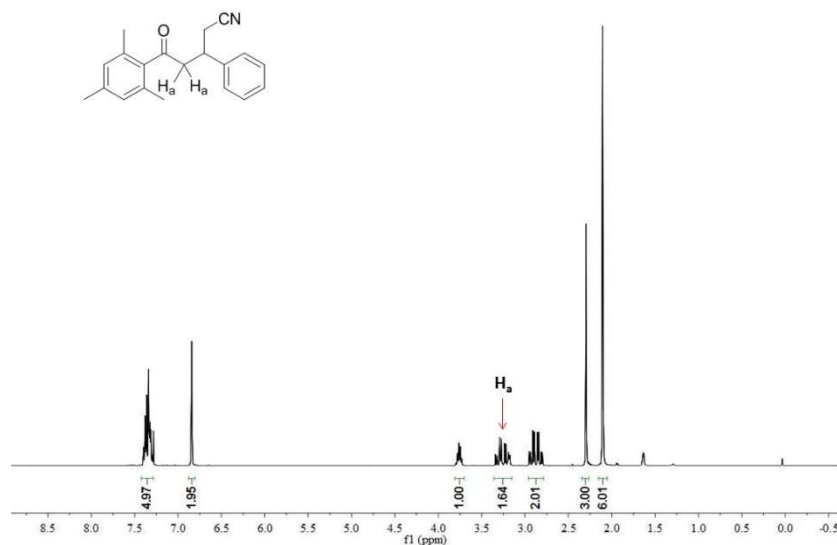


A 250 mL Schlenk tube with a magnetic stir bar was charged with **1c** (1.68 g, 10.0 mmol), **2c** (1.43 g, 10 mmol), NaHMDS (2 mL, 4 mmol, 2M in toluene), toluene (40.0 mL). The Schlenk tube was then sealed with a Teflon lined cap and the mixture was heated at 150 °C for 20 hours. The reaction solution was then cooled to ambient temperature, neutralize with glacial acetic acid. The mixture diluted with 30 mL ethyl acetate, follow by washed with water (50 mL \times 3) and brine. The organic phases were dried over Na₂SO₄, filtered, and concentrated in vacuo. The residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 50/1) to provide the product **3gg** (1.81 g) in 72% yield.

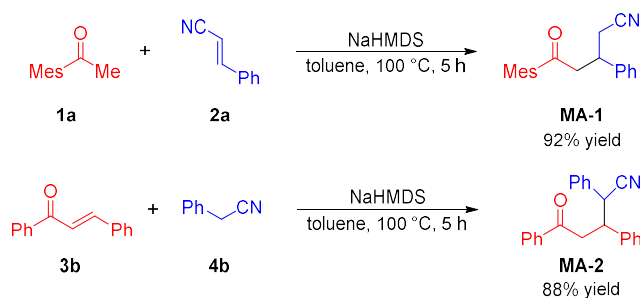
3.4 Deuterium-labeled experiment.



A 25 mL Schlenk tube with a magnetic stir bar was charged with **MA-1** (146 mg, 0.5 mmol), NaHMDS (0.1 mL, 0.2 mmol, 2.0 M in toluene), toluene (2.0 mL). The Schlenk tube was then sealed with a Teflon lined cap and the mixture was heated at 150 °C for 1 hours. The reaction solution was then cooled to ambient temperature, quenched with deuterium oxide (D₂O), diluted with 3 mL ethyl acetate, filtered through a celite pad and anhydrous Na₂SO₄, followby the filtrate was collected and concentrated. The residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 30/1).

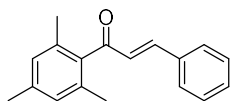


3.5 Capture of the intermediate MA-1 or MA-2.



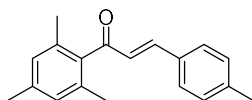
A 25 mL Schlenk tube with a magnetic stir bar was charged with **1a** (81 mg, 0.5 mmol) or **3b** (104 mg, 0.5 mmol), **2a** (65 mg, 0.5 mmol) or **4b** (59mg, 0.5 mmol), NaHMDS (0.1 mL, 0.2 mmol, 2.0 M in toluene), toluene (2.0 mL). The Schlenk tube was then sealed with a Teflon lined cap and the mixture was heated at 100 °C for 5 hours. The reaction solution was then cooled to ambient temperature, neutralized with glacial acetic acid. The mixture diluted with 3 mL ethyl acetate, follow by washed with water (5 mL \times 3) and brine. The organic phases were dried over Na₂SO₄, filtered, and concentrated in vacuo. The residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 30/1) to provide the **MA-1** (134 mg) in 92% yield or **MA-2** (143 mg) in 88% yield.

IV. Experimental data for the described substances



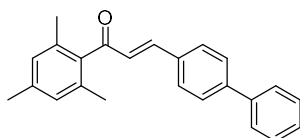
(E)-1-mesityl-3-phenylprop-2-en-1-one (3a) [55800-30-1]^[1]

The title compound was prepared according to general procedure. Yield 84% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.54 – 7.48 (m, 2H), 7.39 (dd, *J* = 5.1, 1.9 Hz, 3H), 7.18 (d, *J* = 16.3 Hz, 1H), 6.93 (d, *J* = 16.3 Hz, 1H), 6.89 (s, 2H), 2.33 (s, 3H), 2.19 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 201.42, 146.69, 138.39, 137.11, 134.48, 134.11, 130.79, 128.97, 128.48, 128.45, 128.38, 21.15, 19.33. HR-MS (ESI⁺, *m/z*): calcd for C₁₈H₁₈ONa [M+Na]⁺: 273.1255, found: 273.1259.



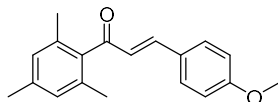
(E)-1-mesityl-3-(p-tolyl)prop-2-en-1-one (3c) [1322214-75-4]^[2]

The title compound was prepared according to general procedure. Yield 77% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.43 (d, *J* = 8.1 Hz, 2H), 7.24 – 7.17 (m, 3H), 6.94 (d, *J* = 16.4 Hz, 1H), 6.92 (s, 2H), 2.40 (s, 3H), 2.36 (s, 3H), 2.23 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 201.46, 146.86, 141.42, 138.29, 137.27, 134.10, 131.75, 129.74, 128.53, 128.38, 127.54, 21.54, 21.17, 19.35. HR-MS (ESI⁺, *m/z*): calcd for C₁₉H₂₀ONa [M+Na]⁺: 287.1412, found: 287.1410.



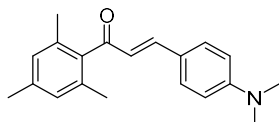
(E)-3-([1,1'-biphenyl]-4-yl)-1-mesitylprop-2-en-1-one (3d)

The title compound was prepared according to general procedure. Yield 71% as a yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 7.72 – 7.64 (m, 4H), 7.61 (d, *J* = 8.4 Hz, 2H), 7.51 (t, *J* = 7.5 Hz, 2H), 7.43 (t, *J* = 7.3 Hz, 1H), 7.36 (d, *J* = 16.2 Hz, 1H), 7.09 (d, *J* = 16.2 Hz, 1H), 7.00 (s, 2H), 2.42 (s, 3H), 2.34 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 201.17, 146.21, 143.56, 139.98, 138.47, 137.40, 134.21, 133.50, 129.13, 129.06, 128.59, 128.37, 128.12, 127.64, 127.10, 21.30, 19.52. HR-MS (ESI⁺, *m/z*): calcd for C₂₄H₂₂ONa [M+Na]⁺: 349.1568, found: 349.1565.



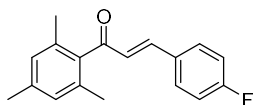
(E)-1-mesityl-3-(4-methoxyphenyl)prop-2-en-1-one (3e)

The title compound was prepared according to general procedure. Yield 91% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.48 (d, *J* = 8.8 Hz, 2H), 7.16 (d, *J* = 16.2 Hz, 1H), 6.95 – 6.91 (m, 2H), 6.91 (s, 2H), 6.84 (d, *J* = 16.2 Hz, 1H), 3.86 (s, 3H), 2.35 (s, 3H), 2.22 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 201.36, 161.87, 146.61, 138.20, 137.35, 134.08, 130.26, 128.33, 127.14, 126.32, 114.45, 55.44, 21.15, 19.33. HR-MS (ESI⁺, *m/z*): calcd for C₁₉H₂₀O₂Na [M+Na]⁺: 303.1361, found: 303.1359.



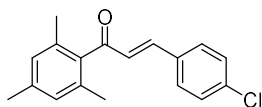
(E)-3-(4-(dimethylamino)phenyl)-1-mesitylprop-2-en-1-one (3f) [1432505-79-7]^[3]

The title compound was prepared according to general procedure. Yield 92% as a yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 7.42 (d, *J* = 8.9 Hz, 2H), 7.13 (d, *J* = 16.0 Hz, 1H), 6.90 (s, 2H), 6.78 (d, *J* = 16.0 Hz, 1H), 6.67 (d, *J* = 8.9 Hz, 2H), 3.05 (s, 6H), 2.34 (s, 3H), 2.22 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 201.28, 152.16, 147.81, 137.85, 134.09, 130.38, 128.21, 123.60, 122.09, 111.80, 40.11, 21.13, 19.32. HR-MS (ESI⁺, *m/z*): calcd for C₂₀H₂₃NONa [M+Na]⁺: 316.1677, found: 316.1676.



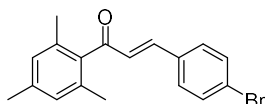
(E)-3-(4-fluorophenyl)-1-mesitylprop-2-en-1-one (3g)

The title compound was prepared according to general procedure. Yield 61% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.55 – 7.48 (m, 2H), 7.18 (d, *J* = 16.2 Hz, 1H), 7.13 – 7.05 (m, 2H), 6.91 (s, 2H), 6.88 (d, *J* = 16.3 Hz, 1H), 2.35 (s, 3H), 2.22 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 201.12, 165.44, 162.93, 145.25, 138.45, 137.05, 134.09, 130.72 (d, *J* = 3.4 Hz), 130.41 (d, *J* = 8.6 Hz), 128.42, 128.20 (d, *J* = 2.3 Hz), 116.27, 116.05, 21.14, 19.33. ¹⁹F NMR (376 MHz, CDCl₃) δ -108.59 (s). HR-MS (ESI⁺, *m/z*): calcd for C₁₈H₁₇FONa [M+Na]⁺: 291.1161, found: 291.1157.



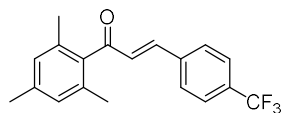
(E)-3-(4-chlorophenyl)-1-mesitylprop-2-en-1-one (3h)

The title compound was prepared according to general procedure. Yield 67% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.46 (d, *J* = 8.5 Hz, 2H), 7.37 (d, *J* = 8.5 Hz, 2H), 7.16 (d, *J* = 16.3 Hz, 1H), 6.92 (d, *J* = 16.3 Hz, 1H), 6.91 (s, 2H), 2.35 (s, 3H), 2.21 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 201.07, 145.01, 138.52, 136.97, 136.73, 134.10, 132.98, 129.61, 129.25, 128.82, 128.44, 21.16, 19.35. HR-MS (ESI⁺, *m/z*): calcd for C₁₈H₁₇ClONa [M+Na]⁺: 307.0866, found: 307.0863.



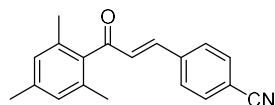
(E)-3-(4-bromophenyl)-1-mesitylprop-2-en-1-one (3i)

The title compound was prepared according to general procedure. Yield 66% as a yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 7.45 (d, *J* = 8.5 Hz, 2H), 7.32 (d, *J* = 8.5 Hz, 2H), 7.15 (d, *J* = 16.3 Hz, 1H), 6.94 (d, *J* = 16.2 Hz, 1H), 6.88 (s, 2H), 2.31 (s, 3H), 2.21 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 200.76, 144.96, 138.47, 137.08, 134.07, 133.42, 132.19, 129.83, 128.93, 128.51, 125.08, 21.21, 19.43. HR-MS (ESI⁺, *m/z*): calcd for C₁₈H₁₇BrONa [M+Na]⁺: 351.0360, found: 351.0364.



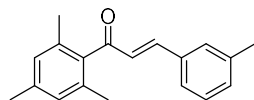
(*E*)-1-mesityl-3-(4-(trifluoromethyl)phenyl)prop-2-en-1-one (3j)

The title compound was prepared according to general procedure. Yield 55% as a yellow solid. ^1H NMR (400 MHz, CDCl_3) δ 7.69 – 7.61 (m, 4H), 7.24 (d, $J = 16.3$ Hz, 1H), 7.01 (d, $J = 16.3$ Hz, 1H), 6.93 (s, 2H), 2.36 (s, 3H), 2.23 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 200.89, 144.28, 138.72, 137.90, 136.78, 134.13, 132.12 (q, $J = 32.7$ Hz), 130.46, 128.55, 128.51, 125.89 (q, $J = 3.8$ Hz), 123.76 (q, $J = 272.3$ Hz), 21.14, 19.35. ^{19}F NMR (376 MHz, CDCl_3) δ -62.93 (s). HR-MS (ESI^+ , m/z): calcd for $\text{C}_{19}\text{H}_{17}\text{F}_3\text{ONa}$ $[\text{M}+\text{Na}]^+$: 341.1129, found: 341.1135.



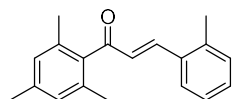
(*E*)-4-(3-mesityl-3-oxoprop-1-en-1-yl)benzonitrile (3k)

The title compound was prepared according to general procedure. Yield 32% as a yellow solid. ^1H NMR (400 MHz, CDCl_3) δ 7.68 (d, $J = 8.4$ Hz, 2H), 7.61 (d, $J = 8.4$ Hz, 2H), 7.19 (d, $J = 16.3$ Hz, 1H), 6.99 (d, $J = 16.3$ Hz, 1H), 6.92 (s, 2H), 2.34 (s, 3H), 2.20 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 200.65, 143.55, 138.85, 138.81, 136.60, 134.12, 132.64, 131.17, 128.74, 128.55, 118.28, 113.72, 21.16, 19.38. HR-MS (ESI^+ , m/z): calcd for $\text{C}_{19}\text{H}_{17}\text{N}\text{ONa}$ $[\text{M}+\text{Na}]^+$: 298.1208, found: 298.1211.



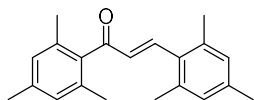
(*E*)-1-mesityl-3-(*m*-tolyl)prop-2-en-1-one (3l)

The title compound was prepared according to general procedure. Yield 78% as a yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.36 (d, $J = 6.9$ Hz, 2H), 7.30 (m, $J = 11.4, 4.9$ Hz, 1H), 7.25 (s, 1H), 7.20 (d, $J = 16.3$ Hz, 1H), 6.97 (d, $J = 16.2$ Hz, 1H), 6.93 (s, 2H), 2.39 (s, 3H), 2.36 (s, 3H), 2.24 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 201.41, 146.95, 138.68, 138.34, 137.23, 134.45, 134.10, 131.65, 129.25, 128.89, 128.40, 128.31, 125.61, 21.28, 21.17, 19.34. HR-MS (ESI^+ , m/z): calcd for $\text{C}_{19}\text{H}_{20}\text{ONa}$ $[\text{M}+\text{Na}]^+$: 287.1412, found: 287.1418.



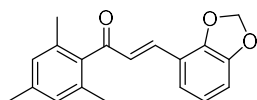
(*E*)-1-mesityl-3-(*o*-tolyl)prop-2-en-1-one (3m)

The title compound was prepared according to general procedure. Yield 75% as a white solid. ^1H NMR (400 MHz, CDCl_3) δ 7.64 (d, $J = 6.3$ Hz, 1H), 7.61 (d, $J = 15.8$ Hz, 1H), 7.35 – 7.17 (m, 3H), 6.94 (s, 2H), 6.90 (d, $J = 16.1$ Hz, 1H), 2.36 (s, 3H), 2.32 (s, 3H), 2.27 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 201.21, 144.35, 138.38, 137.90, 137.37, 134.00, 133.44, 130.88, 130.49, 129.45, 128.43, 126.69, 126.51, 21.18, 19.64, 19.43. HR-MS (ESI^+ , m/z): calcd for $\text{C}_{19}\text{H}_{20}\text{ONa}$ $[\text{M}+\text{Na}]^+$: 287.1412, found: 287.1413.



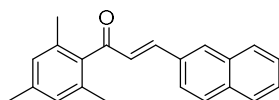
(E)-1,3-dimesitylprop-2-en-1-one (3n)

The title compound was prepared according to general procedure. Yield 71% as a white solid. ^1H NMR (400 MHz, CDCl_3) δ 7.43 (d, $J = 16.6$ Hz, 1H), 6.93 (s, 1H), 6.91 (s, 1H), 6.59 (d, $J = 16.6$ Hz, 1H), 2.35 (s, 3H), 2.31 (s, 3H), 2.30 (s, 6H), 2.28 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 201.80, 146.22, 138.81, 138.31, 137.02, 136.71, 133.93, 133.76, 130.85, 129.27, 128.35, 21.16, 21.09, 21.02, 19.30. HR-MS (ESI^+ , m/z): calcd for $\text{C}_{21}\text{H}_{24}\text{ONa}$ $[\text{M}+\text{Na}]^+$: 315.1725, found: 315.1727.



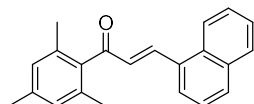
(E)-3-(benzo[d][1,3]dioxol-4-yl)-1-mesitylprop-2-en-1-one (3o)

The title compound was prepared according to general procedure. Yield 83% as a yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.16 (d, $J = 1.4$ Hz, 2H), 6.92 – 6.82 (m, 5H), 6.06 (s, 2H), 2.34 (s, 3H), 2.22 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 201.58, 148.06, 146.77, 140.87, 138.36, 137.10, 134.11, 130.84, 128.39, 122.74, 121.84, 117.50, 110.18, 101.53, 21.16, 19.39. HR-MS (ESI^+ , m/z): calcd for $\text{C}_{19}\text{H}_{18}\text{O}_3\text{Na}$ $[\text{M}+\text{Na}]^+$: 317.1154, found: 317.1152.



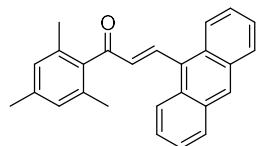
(E)-1-mesityl-3-(naphthalen-2-yl)prop-2-en-1-one (3p)

The title compound was prepared according to general procedure. Yield 82% as a yellow solid. ^1H NMR (400 MHz, CDCl_3) δ 7.91 – 7.79 (m, 4H), 7.72 (dd, $J = 8.6, 1.7$ Hz, 1H), 7.58 – 7.48 (m, 2H), 7.40 (d, $J = 16.2$ Hz, 1H), 7.10 (d, $J = 16.2$ Hz, 1H), 6.96 (s, 2H), 2.39 (s, 3H), 2.28 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 201.37, 146.81, 138.43, 137.25, 134.47, 134.18, 133.27, 132.03, 130.78, 128.84, 128.62, 128.62, 128.47, 127.85, 127.57, 126.86, 123.53, 21.21, 19.43. HR-MS (ESI^+ , m/z): calcd for $\text{C}_{22}\text{H}_{20}\text{ONa}$ $[\text{M}+\text{Na}]^+$: 323.1412, found: 323.1410.



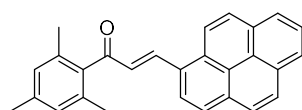
(E)-1-mesityl-3-(naphthalen-1-yl)prop-2-en-1-one (3q)

The title compound was prepared according to general procedure. Yield 87% as a yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 8.14 (d, $J = 16.0$ Hz, 1H), 7.97 – 7.83 (m, 4H), 7.55 (m, 3H), 7.07 (d, $J = 16.0$ Hz, 1H), 6.98 (s, 2H), 2.39 (s, 3H), 2.32 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 201.21, 143.55, 138.54, 137.29, 134.14, 133.72, 131.72, 131.42, 131.05, 130.90, 128.88, 128.54, 127.04, 126.29, 125.53, 125.42, 122.94, 21.22, 19.50. HR-MS (ESI^+ , m/z): calcd for $\text{C}_{22}\text{H}_{20}\text{ONa}$ $[\text{M}+\text{Na}]^+$: 323.1412, found: 323.1409.



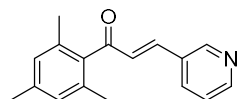
(*E*)-3-(anthracen-9-yl)-1-mesitylprop-2-en-1-one (3r)

The title compound was prepared according to general procedure. Yield 77% as a yellow solid. ^1H NMR (400 MHz, CDCl_3) δ 8.39 (s, 1H), 8.21 (d, $J = 16.5$ Hz, 1H), 8.14 – 8.08 (m, 2H), 8.02 – 7.94 (m, 2H), 7.55 – 7.45 (m, 4H), 7.04 (s, 2H), 6.96 (d, $J = 16.5$ Hz, 1H), 2.48 (s, 6H), 2.41 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 201.16, 144.37, 138.71, 137.33, 137.01, 134.18, 131.20, 129.24, 129.00, 128.90, 128.82, 128.66, 126.60, 125.40, 124.73, 21.26, 19.63. HR-MS (ESI^+ , m/z): calcd for $\text{C}_{26}\text{H}_{22}\text{ONa}$ $[\text{M}+\text{Na}]^+$: 373.1568, found: 373.1562.



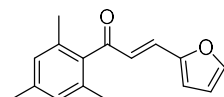
(*E*)-1-mesityl-3-(pyren-1-yl)prop-2-en-1-one (3s)

The title compound was prepared according to general procedure. Yield 79% as an orange solid. ^1H NMR (400 MHz, CDCl_3) δ 8.41 (d, $J = 15.9$ Hz, 1H), 8.32 – 8.25 (m, 1H), 8.21 – 8.13 (m, 2H), 8.12 – 7.97 (m, 6H), 7.23 (d, $J = 15.9$ Hz, 1H), 7.03 (s, 2H), 2.44 (s, 3H), 2.38 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 201.07, 143.22, 138.57, 137.55, 134.22, 132.97, 131.22, 130.51, 130.21, 129.88, 128.78, 128.73, 128.62, 128.10, 127.29, 126.29, 126.16, 125.92, 125.07, 124.77, 124.44, 124.31, 121.90, 21.29, 19.62. HR-MS (ESI^+ , m/z): calcd for $\text{C}_{28}\text{H}_{22}\text{ONa}$ $[\text{M}+\text{Na}]^+$: 397.1568, found: 397.1574.



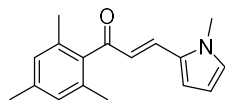
(*E*)-1-mesityl-3-(pyridin-3-yl)prop-2-en-1-one (3t)

The title compound was prepared according to general procedure. Yield 67% as a yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 8.69 (d, $J = 2.0$ Hz, 1H), 8.62 (dd, $J = 4.8, 1.5$ Hz, 1H), 7.87 (dt, $J = 8.0, 1.9$ Hz, 1H), 7.35 (dd, $J = 8.0, 4.8$ Hz, 1H), 7.20 (d, $J = 16.4$ Hz, 1H), 6.99 (d, $J = 16.3$ Hz, 1H), 6.91 (s, 2H), 2.34 (s, 3H), 2.21 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 200.74, 151.37, 150.10, 142.49, 138.73, 136.64, 134.38, 134.11, 130.27, 130.14, 128.51, 123.80, 21.15, 19.36. HR-MS (ESI^+ , m/z): calcd for $\text{C}_{17}\text{H}_{17}\text{NONa}$ $[\text{M}+\text{Na}]^+$: 274.1208, found: 274.1211.



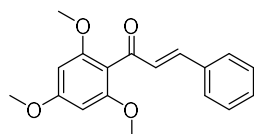
(*E*)-3-(furan-2-yl)-1-mesitylprop-2-en-1-one (3u)

The title compound was prepared according to general procedure. Yield 61% as a yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.55 (d, $J = 1.4$ Hz, 1H), 6.96 (d, $J = 16.0$ Hz, 1H), 6.90 (s, 2H), 6.82 (d, $J = 16.0$ Hz, 1H), 6.64 (d, $J = 3.4$ Hz, 1H), 6.50 (dd, $J = 3.4, 1.8$ Hz, 1H), 2.34 (s, 3H), 2.21 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 200.88, 150.89, 145.45, 138.34, 137.00, 134.14, 132.53, 128.35, 125.93, 116.23, 112.69, 21.13, 19.29. HR-MS (ESI^+ , m/z): calcd for $\text{C}_{16}\text{H}_{16}\text{O}_2\text{Na}$ $[\text{M}+\text{Na}]^+$: 263.1048, found: 263.1051.



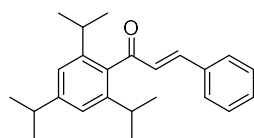
(E)-1-mesityl-3-(1-methyl-1H-pyrrol-2-yl)prop-2-en-1-one (3v)

The title compound was prepared according to general procedure. Yield 73% as a yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 7.23 (d, *J* = 15.8 Hz, 1H), 6.91 (s, 2H), 6.84 – 6.79 (m, 1H), 6.74 (dd, *J* = 4.0, 1.5 Hz, 1H), 6.71 (d, *J* = 15.8 Hz, 1H), 6.22 (dd, *J* = 3.9, 2.6 Hz, 1H), 3.63 (s, 3H), 2.34 (s, 3H), 2.25 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 200.40, 138.08, 137.78, 133.98, 133.91, 129.39, 128.53, 128.36, 123.05, 113.87, 109.99, 34.53, 21.17, 19.39. HR-MS (ESI⁺, *m/z*): calcd for C₁₇H₁₉NONa [M+Na]⁺: 276.1364, found: 276.1360.



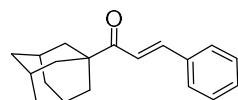
(E)-3-phenyl-1-(2,4,6-trimethoxyphenyl)prop-2-en-1-one (3x) [145629-35-2]^[4]

The title compound was prepared according to general procedure. Yield 47% as a yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 7.52 (dd, *J* = 6.7, 2.9 Hz, 2H), 7.41 – 7.33 (m, 4H), 6.98 (d, *J* = 16.1 Hz, 1H), 6.18 (s, 2H), 3.86 (s, 3H), 3.77 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 194.25, 162.49, 158.86, 144.07, 135.00, 130.21, 129.05, 128.83, 128.36, 111.73, 90.75, 55.91, 55.48. HR-MS (ESI⁺, *m/z*): calcd for C₁₈H₁₈O₄Na [M+Na]⁺: 321.1103, found: 321.1109.



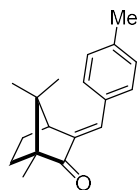
(E)-3-phenyl-1-(2,4,6-triisopropylphenyl)prop-2-en-1-one (3y) [862510-37-0]^[5]

The title compound was prepared according to general procedure. Yield 56% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.54 (dd, *J* = 6.7, 2.9 Hz, 2H), 7.44 – 7.37 (m, 3H), 7.27 (d, *J* = 16.2 Hz, 1H), 7.10 (s, 2H), 7.04 (dd, *J* = 16.2, 0.9 Hz, 1H), 2.98 (hept, *J* = 6.9 Hz, 1H), 2.85 (hept, *J* = 6.7 Hz, 2H), 1.33 (d, *J* = 6.9 Hz, 6H), 1.23 (d, *J* = 6.3 Hz, 12H). ¹³C NMR (101 MHz, CDCl₃) δ 201.97, 149.67, 146.66, 144.93, 135.40, 134.49, 130.73, 129.72, 129.01, 128.46, 121.05, 34.40, 31.05, 24.83, 24.05, 23.84. HR-MS (ESI⁺, *m/z*): calcd for C₂₄H₃₀ONa [M+Na]⁺: 357.2194, found: 357.2197.



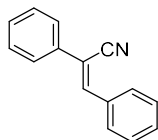
(E)-1-((3r,5r,7r)-adamantan-1-yl)-3-phenylprop-2-en-1-one (3aa) [116440-60-9]^[6]

The title compound was prepared according to general procedure. Yield 39% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.69 (d, *J* = 15.6 Hz, 1H), 7.59 (dd, *J* = 6.5, 3.1 Hz, 2H), 7.43 – 7.35 (m, 3H), 7.18 (d, *J* = 15.6 Hz, 1H), 2.10 (s, 3H), 1.91 (d, *J* = 2.7 Hz, 6H), 1.78 (q, *J* = 12.3 Hz, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 203.74, 142.83, 135.08, 130.14, 128.83, 128.27, 120.34, 45.53, 38.06, 36.62, 28.00. HR-MS (ESI⁺, *m/z*): calcd for C₁₉H₂₂ONa [M+Na]⁺: 289.1568, found: 289.1573.



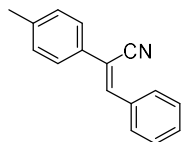
1,7,7-trimethyl-3-((E)-4-methylbenzylidene)bicyclo[2.2.1]heptan-2-one (3gg) [36861-47-9]^[7]

The title compound was prepared according to general procedure. Yield 78% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.29 (d, *J* = 8.1 Hz, 2H), 7.13 (s, 1H), 7.11 (d, *J* = 8.0 Hz, 2H), 3.01 (d, *J* = 4.2 Hz, 1H), 2.28 (s, 3H), 2.08 (tt, *J* = 10.8, 4.2 Hz, 1H), 1.69 (td, *J* = 12.3, 3.1 Hz, 1H), 1.46 (m, 2H), 0.94 (s, 3H), 0.91 (s, 3H), 0.71 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 208.27, 141.27, 138.94, 132.84, 129.79, 129.41, 127.58, 57.08, 49.26, 46.74, 30.78, 25.96, 21.42, 20.57, 18.39, 9.34. HR-MS (ESI⁺, *m/z*): calcd for C₁₈H₂₂ONa [M+Na]⁺: 277.1568, found: 277.1565.



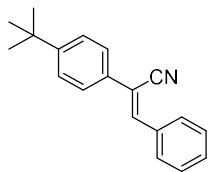
(Z)-2,3-diphenylacrylonitrile (5a) [6114-57-4]^[8]

The title compound was prepared according to general procedure. Yield 63% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.92 (dd, *J* = 7.8, 1.2 Hz, 2H), 7.75 – 7.68 (m, 2H), 7.57 (s, 1H), 7.54 – 7.39 (m, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 142.27, 134.50, 133.74, 130.55, 129.28, 129.22, 129.08, 128.98, 126.02, 118.01, 111.74. HR-MS (ESI⁺, *m/z*): calcd for C₁₅H₁₁NNa [M+Na]⁺: 228.0789, found: 228.0793.



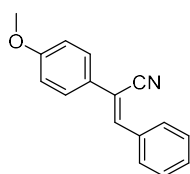
(Z)-3-phenyl-2-(p-tolyl)acrylonitrile (5b) [25125-30-8]^[9]

The title compound was prepared according to general procedure. Yield 64% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.93 – 7.88 (m, 2H), 7.60 (d, *J* = 8.3 Hz, 2H), 7.53 (s, 1H), 7.52 – 7.44 (m, 3H), 7.28 (d, *J* = 7.9 Hz, 2H), 2.43 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 141.23, 139.43, 133.88, 131.67, 130.34, 129.77, 129.20, 128.94, 125.88, 118.12, 111.67, 21.25. HR-MS (ESI⁺, *m/z*): calcd for C₁₆H₁₃NNa [M+Na]⁺: 242.0946, found: 242.0949.



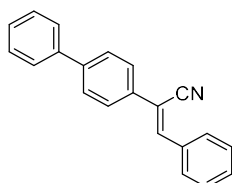
(Z)-2-(4-(*tert*-butyl)phenyl)-3-phenylacrylonitrile (5c) [2744214-14-8]^[9]

The title compound was prepared according to general procedure. Yield 63% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.92 (d, *J* = 6.9 Hz, 2H), 7.69 – 7.62 (m, 2H), 7.55 (s, 1H), 7.53 – 7.43 (m, 5H), 1.39 (s, 9H). ¹³C NMR (101 MHz, CDCl₃) δ 152.65, 141.39, 133.90, 131.66, 130.36, 129.21, 128.94, 126.04, 125.76, 118.09, 111.59, 34.78, 31.23. HR-MS (ESI⁺, *m/z*): calcd for C₁₉H₁₉NNa [M+Na]⁺: 284.1415, found: 284.1414.



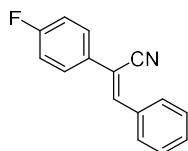
(Z)-2-(4-methoxyphenyl)-3-phenylacrylonitrile (5d) [54648-50-9]^[8]

The title compound was prepared according to general procedure. Yield 59% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.89 (dd, *J* = 5.1, 3.7 Hz, 2H), 7.68 – 7.61 (m, 2H), 7.52 – 7.41 (m, 4H), 7.02 – 6.96 (m, 2H), 3.88 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 160.46, 140.16, 133.98, 130.15, 129.06, 128.91, 127.35, 127.00, 118.15, 114.45, 111.32, 55.46. HR-MS (ESI⁺, *m/z*): calcd for C₁₆H₁₃NONa [M+Na]⁺: 258.0895, found: 258.0990.



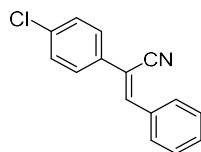
(Z)-2-([1,1'-biphenyl]-4-yl)-3-phenylacrylonitrile (5e) [54648-46-3]^[9]

The title compound was prepared according to general procedure. Yield 71% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.98 – 7.91 (m, 2H), 7.83 – 7.77 (m, 2H), 7.74 – 7.69 (m, 2H), 7.68 – 7.64 (m, 2H), 7.62 (s, 1H), 7.55 – 7.47 (m, 5H), 7.44 – 7.39 (m, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 142.06, 141.90, 139.98, 133.77, 133.37, 130.57, 129.31, 129.00, 128.95, 127.86, 127.69, 127.06, 126.42, 117.98, 111.34. HR-MS (ESI⁺, *m/z*): calcd for C₂₁H₁₅NNa [M+Na]⁺: 304.1102, found: 304.1104.



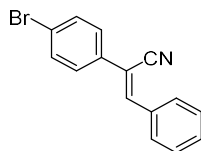
(Z)-2-(4-fluorophenyl)-3-phenylacrylonitrile (5f) [2558-28-3]^[8]

The title compound was prepared according to general procedure. Yield 71% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.90 (dd, *J* = 7.5, 1.7 Hz, 2H), 7.73 – 7.64 (m, 2H), 7.55 – 7.44 (m, 4H), 7.21 – 7.13 (m, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 164.46, 161.97, 142.17 (d, *J* = 1.8 Hz), 133.57, 130.64, 129.23, 129.01, 127.89 (d, *J* = 8.4 Hz), 117.88, 116.16 (d, *J* = 22.1 Hz), 110.64. ¹⁹F NMR (376 MHz, CDCl₃) δ -111.63 (s). HR-MS (ESI⁺, *m/z*): calcd for C₁₅H₁₀FNNa [M+Na]⁺: 246.0695, found: 246.0692.



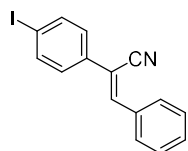
(Z)-2-(4-chlorophenyl)-3-phenylacrylonitrile (5g) [6269-09-6]^[8]

The title compound was prepared according to general procedure. Yield 70% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.91 (dd, *J* = 7.4, 1.9 Hz, 2H), 7.63 (d, *J* = 8.6 Hz, 2H), 7.54 (s, 1H), 7.53 – 7.46 (m, 3H), 7.44 (d, *J* = 8.6 Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 142.58, 135.24, 133.44, 132.96, 130.83, 129.34, 129.29, 129.05, 127.25, 117.72, 110.49. HR-MS (ESI⁺, *m/z*): calcd for C₁₅H₁₀ClNNa [M+Na]⁺: 262.0399, found: 262.0402.



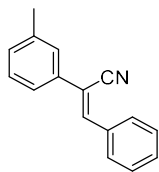
(Z)-2-(4-bromophenyl)-3-phenylacrylonitrile (5h) [112158-67-5]^[8]

The title compound was prepared according to general procedure. Yield 52% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.94 – 7.87 (m, 2H), 7.63 – 7.53 (m, 5H), 7.53 – 7.45 (m, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 142.61, 133.44, 132.25, 130.85, 129.34, 129.05, 127.50, 123.45, 117.62, 110.60. HR-MS (ESI⁺, *m/z*): calcd for C₁₅H₁₀BrNNa [M+Na]⁺: 305.9894, found: 305.9899.



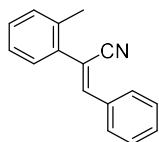
(Z)-2-(4-iodophenyl)-3-phenylacrylonitrile (5i) [2744214-15-9]^[9]

The title compound was prepared according to general procedure. Yield 53% as a yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 7.91 (dd, *J* = 7.4, 2.0 Hz, 2H), 7.80 (d, *J* = 8.6 Hz, 2H), 7.56 (s, 1H), 7.53 – 7.46 (m, 3H), 7.43 (d, *J* = 8.6 Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 142.60, 138.19, 134.05, 133.43, 130.88, 129.37, 129.06, 127.60, 117.59, 110.70, 95.13. HR-MS (ESI⁺, *m/z*): calcd for C₁₅H₁₀INNa [M+Na]⁺: 353.9756, found: 353.9754.



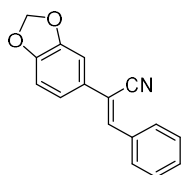
(Z)-3-phenyl-2-(*m*-tolyl)acrylonitrile (5j) [54648-48-5]^[9]

The title compound was prepared according to general procedure. Yield 61% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.91 (dd, *J* = 7.8, 1.8 Hz, 2H), 7.54 – 7.47 (m, 3H), 7.40 – 7.28 (m, 4H), 7.18 (s, 1H), 2.52 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 146.90, 136.30, 135.45, 133.62, 130.90, 130.64, 129.40, 129.18, 129.11, 128.98, 126.52, 117.92, 111.23, 20.13. HR-MS (ESI⁺, *m/z*): calcd for C₁₆H₁₃NNa [M+Na]⁺: 242.0946, found: 242.0943.



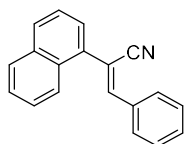
(Z)-3-phenyl-2-(*o*-tolyl)acrylonitrile (5k) [87968-36-3]^[9]

The title compound was prepared according to general procedure. Yield 51% as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.95 – 7.88 (m, 2H), 7.56 (s, 1H), 7.54 – 7.43 (m, 5H), 7.36 (t, *J* = 7.7 Hz, 1H), 7.24 (d, *J* = 7.5 Hz, 1H), 2.45 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 142.06, 138.89, 134.42, 133.81, 130.46, 130.02, 129.25, 128.95, 126.73, 123.09, 118.11, 111.83, 21.47. HR-MS (ESI⁺, *m/z*): calcd for C₁₆H₁₃NNa [M+Na]⁺: 242.0946, found: 242.0945.



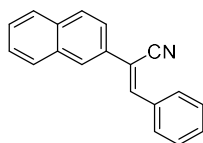
(Z)-2-(benzo[*d*][1,3]dioxol-5-yl)-3-phenylacrylonitrile (5l) [6443-78-3]^[10]

The title compound was prepared according to general procedure. Yield 64% as a yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 7.91 – 7.85 (m, 2H), 7.51 – 7.43 (m, 3H), 7.41 (s, 1H), 7.22 (dd, *J* = 8.2, 1.9 Hz, 1H), 7.15 (d, *J* = 1.9 Hz, 1H), 6.89 (d, *J* = 8.2 Hz, 1H), 6.04 (s, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 148.65, 148.50, 140.67, 133.79, 130.31, 129.14, 128.94, 128.76, 120.72, 118.08, 111.32, 108.65, 105.89, 101.70. HR-MS (ESI⁺, *m/z*): calcd for C₁₆H₁₁NO₂Na [M+Na]⁺: 272.0687, found: 272.0691.



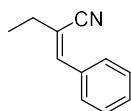
(Z)-2-(naphthalen-1-yl)-3-phenylacrylonitrile (5m) [27869-65-4]^[9]

The title compound was prepared according to general procedure. Yield 81% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.25 (d, *J* = 8.0 Hz, 1H), 8.01 (dd, *J* = 7.6, 1.3 Hz, 2H), 7.96 (d, *J* = 8.1 Hz, 2H), 7.67 – 7.49 (m, 7H), 7.39 (s, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 147.88, 133.88, 133.65, 133.57, 130.87, 130.82, 129.89, 129.29, 129.10, 128.79, 127.36, 127.11, 126.55, 125.44, 124.59, 118.67, 109.89. HR-MS (ESI⁺, *m/z*): calcd for C₁₉H₁₃NNa [M+Na]⁺: 278.0946, found: 278.0950.



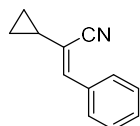
(Z)-2-(naphthalen-2-yl)-3-phenylacrylonitrile (5n) [27869-60-9]^[9]

The title compound was prepared according to general procedure. Yield 78% as a yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 8.20 (s, 1H), 8.01 – 7.86 (m, 5H), 7.79 (dd, *J* = 8.7, 1.0 Hz, 1H), 7.70 (s, 1H), 7.61 – 7.44 (m, 5H). ¹³C NMR (101 MHz, CDCl₃) δ 142.18, 133.82, 133.41, 133.29, 131.65, 130.58, 129.36, 129.01, 128.93, 128.52, 127.72, 127.08, 127.00, 126.31, 122.50, 118.10, 111.75. HR-MS (ESI⁺, *m/z*): calcd for C₁₉H₁₃NNa [M+Na]⁺: 278.0946, found: 278.0945.



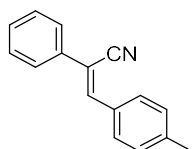
(Z)-2-benzylidenebutanenitrile (5o) [85520-67-8]^[9]

The title compound was prepared according to general procedure. Yield 36% as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.75 (dd, *J* = 7.8, 1.3 Hz, 2H), 7.47 – 7.37 (m, 3H), 6.96 (s, 1H), 2.46 (qd, *J* = 7.5, 1.2 Hz, 2H), 1.28 (t, *J* = 7.5 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 142.51, 133.86, 129.83, 128.79, 128.54, 118.76, 112.94, 29.63, 13.02. HR-MS (ESI⁺, *m/z*): calcd for C₁₁H₁₁NNa [M+Na]⁺: 180.0789, found: 180.0793.



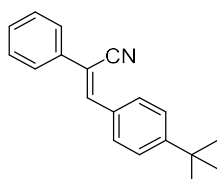
(Z)-2-cyclopropyl-3-phenylacrylonitrile (5p) [2744214-17-1]^[9]

The title compound was prepared according to general procedure. Yield 49% as a yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 7.71 (dd, *J* = 8.0, 1.3 Hz, 2H), 7.45 – 7.35 (m, 3H), 7.06 (s, 1H), 1.78 (ddd, *J* = 15.8, 8.0, 4.9 Hz, 1H), 0.99 – 0.85 (m, 4H). ¹³C NMR (101 MHz, CDCl₃) δ 141.43, 133.85, 129.61, 128.78, 128.38, 116.84, 114.40, 16.11, 6.46. HR-MS (ESI⁺, *m/z*): calcd for C₁₂H₁₁NNa [M+Na]⁺: 192.0789, found: 192.0785.



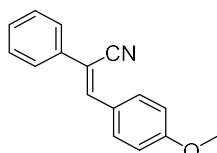
(Z)-2-phenyl-3-(p-tolyl)acrylonitrile (5q) [25125-29-5]^[8]

The title compound was prepared according to general procedure. Yield 71% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.84 (d, *J* = 8.2 Hz, 2H), 7.73 – 7.68 (m, 2H), 7.53 (s, 1H), 7.44 (m, 3H), 7.31 (d, *J* = 8.1 Hz, 2H), 2.45 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 142.31, 141.16, 134.68, 131.05, 129.71, 129.36, 129.06, 129.01, 125.93, 118.28, 110.41, 21.61. HR-MS (ESI⁺, *m/z*): calcd for C₁₆H₁₃NNa [M+Na]⁺: 242.0946, found: 242.0941.



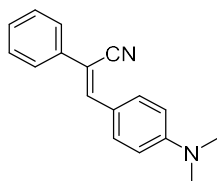
(Z)-3-(4-(tert-butyl)phenyl)-2-phenylacrylonitrile (5r) [347909-78-8]^[8]

The title compound was prepared according to general procedure. Yield 69% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.92 (d, *J* = 8.3 Hz, 2H), 7.73 (d, *J* = 7.5 Hz, 2H), 7.60 – 7.52 (m, 3H), 7.52 – 7.39 (m, 3H), 1.42 (s, 9H). ¹³C NMR (101 MHz, CDCl₃) δ 154.22, 142.18, 134.69, 131.05, 129.28, 129.09, 129.06, 125.99, 125.96, 118.33, 110.58, 35.05, 31.20. HR-MS (ESI⁺, *m/z*): calcd for C₁₉H₁₉NNa [M+Na]⁺: 284.1415, found: 284.1416.



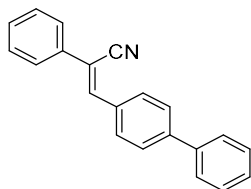
(Z)-3-(4-methoxyphenyl)-2-phenylacrylonitrile (5s) [5432-07-5]^[8]

The title compound was prepared according to general procedure. Yield 83% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.91 (d, *J* = 8.7 Hz, 2H), 7.67 (d, *J* = 7.4 Hz, 2H), 7.51 – 7.36 (m, 4H), 6.99 (d, *J* = 8.8 Hz, 2H), 3.87 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 161.43, 141.88, 134.82, 131.24, 129.04, 128.77, 126.51, 125.77, 118.61, 114.41, 108.54, 55.45. HR-MS (ESI⁺, *m/z*): calcd for C₁₆H₁₃NONa [M+Na]⁺: 258.0895, found: 258.0899.



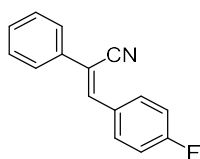
(Z)-3-(4-(dimethylamino)phenyl)-2-phenylacrylonitrile (5t) [1222-61-3]^[11]

The title compound was prepared according to general procedure. Yield 88% as a yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 7.89 (d, *J* = 8.9 Hz, 2H), 7.69 – 7.63 (m, 2H), 7.47 – 7.41 (m, 3H), 7.35 (m, 1H), 6.75 (d, *J* = 9.0 Hz, 2H), 3.08 (s, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 151.68, 142.58, 135.59, 131.30, 128.90, 127.97, 125.46, 121.60, 119.49, 111.63, 104.51, 40.06. HR-MS (ESI⁺, *m/z*): calcd for C₁₇H₁₆N₂Na [M+Na]⁺: 271.1211, found: 271.1207.



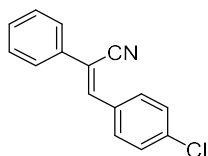
(Z)-3-([1,1'-biphenyl]-4-yl)-2-phenylacrylonitrile (5u) [27869-56-3]^[12]

The title compound was prepared according to general procedure. Yield 73% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.02 (d, *J* = 8.3 Hz, 2H), 7.78 – 7.71 (m, 4H), 7.70 – 7.65 (m, 2H), 7.60 (s, 1H), 7.56 – 7.47 (m, 4H), 7.47 – 7.39 (m, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 143.22, 141.72, 139.96, 134.55, 132.67, 129.85, 129.21, 129.11, 128.98, 128.04, 127.54, 127.12, 126.01, 118.18, 111.33. HR-MS (ESI⁺, *m/z*): calcd for C₂₁H₁₅NNa [M+Na]⁺: 304.1102, found: 304.1107.



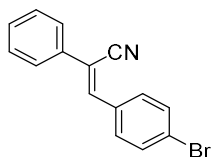
(Z)-3-(4-fluorophenyl)-2-phenylacrylonitrile (5v) [37629-63-3]^[8]

The title compound was prepared according to general procedure. Yield 63% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.92 (dd, *J* = 8.7, 5.3 Hz, 2H), 7.75 – 7.65 (m, 2H), 7.52 (s, 1H), 7.45 (m, 3H), 7.18 (t, *J* = 8.6 Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 163.71 (d, *J* = 252.8 Hz), 140.86, 134.28, 131.39 (d, *J* = 8.6 Hz), 130.01 (d, *J* = 3.4 Hz), 129.30, 129.12, 125.96, 117.95, 116.17 (d, *J* = 21.9 Hz), 111.44. ¹⁹F NMR (376 MHz, CDCl₃) δ -108.27 (s). HR-MS (ESI⁺, *m/z*): calcd for C₁₅H₁₀FNNa [M+Na]⁺: 246.0695, found: 246.0691.



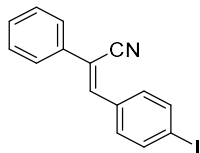
(Z)-3-(4-chlorophenyl)-2-phenylacrylonitrile (5w) [37629-64-4]^[8]

The title compound was prepared according to general procedure. Yield 65% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.85 (d, *J* = 8.5 Hz, 2H), 7.73 – 7.67 (m, 2H), 7.52 – 7.41 (m, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 140.66, 136.46, 134.17, 132.17, 130.48, 129.45, 129.26, 129.14, 126.02, 117.75, 112.32. HR-MS (ESI⁺, *m/z*): calcd for C₁₅H₁₀ClNNa [M+Na]⁺: 262.0399, found: 262.0404.



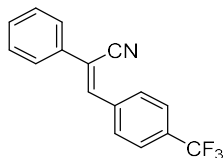
(Z)-3-(4-bromophenyl)-2-phenylacrylonitrile (5x) [73151-37-8]^[8]

The title compound was prepared according to general procedure. Yield 48% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.78 (d, *J* = 8.5 Hz, 2H), 7.70 (dd, *J* = 8.2, 1.3 Hz, 2H), 7.62 (d, *J* = 8.6 Hz, 2H), 7.51 – 7.40 (m, 4H). ¹³C NMR (101 MHz, CDCl₃) δ 140.71, 134.15, 132.59, 132.23, 130.64, 129.47, 129.15, 126.02, 124.86, 117.74, 112.45. HR-MS (ESI⁺, *m/z*): calcd for C₁₅H₁₀BrNNa [M+Na]⁺: 305.9894, found: 305.9898.



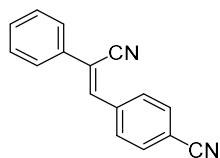
(Z)-3-(4-iodophenyl)-2-phenylacrylonitrile (5y) [862676-16-2]^[8]

The title compound was prepared according to general procedure. Yield 51% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.83 (d, *J* = 8.5 Hz, 2H), 7.69 (d, *J* = 6.9 Hz, 2H), 7.63 (d, *J* = 8.6 Hz, 2H), 7.51 – 7.41 (m, 4H). ¹³C NMR (101 MHz, CDCl₃) δ 140.85, 138.20, 134.14, 133.13, 130.64, 129.49, 129.15, 126.03, 117.72, 112.53, 96.99. HR-MS (ESI⁺, *m/z*): calcd for C₁₅H₁₀INNa [M+Na]⁺: 353.9756, found: 353.9759.



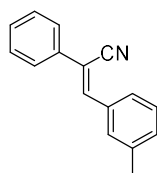
(Z)-2-phenyl-3-(4-(trifluoromethyl)phenyl)acrylonitrile (5z) [147728-28-7]^[8]

The title compound was prepared according to general procedure. Yield 60% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.00 (d, *J* = 8.6 Hz, 2H), 7.79 – 7.69 (m, 4H), 7.59 (s, 1H), 7.55 – 7.43 (m, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 140.12, 137.02, 133.80, 131.84 (q, *J* = 32.8 Hz), 129.85, 129.40, 129.22, 126.17, 125.92 (q, *J* = 3.8 Hz), 123.72 (q, *J* = 272.5 Hz), 117.39, 114.50. ¹⁹F NMR (376 MHz, CDCl₃) δ -62.94 (s). HR-MS (ESI⁺, *m/z*): calcd for C₁₆H₁₀F₃NNa [M+Na]⁺: 296.0663, found: 296.0661.



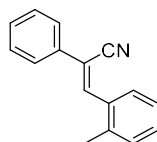
(Z)-4-(2-cyano-2-phenylvinyl)benzonitrile (5aa) [61469-71-4]^[8]

The title compound was prepared according to general procedure. Yield 40% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.99 (d, *J* = 8.3 Hz, 2H), 7.77 (d, *J* = 8.5 Hz, 2H), 7.72 (dd, *J* = 7.9, 1.7 Hz, 2H), 7.56 (s, 1H), 7.54 – 7.44 (m, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 139.41, 137.85, 133.55, 132.64, 130.14, 129.60, 129.29, 126.23, 118.26, 117.18, 115.36, 113.50. HR-MS (ESI⁺, *m/z*): calcd for C₁₆H₁₀N₂Na [M+Na]⁺: 253.0742, found: 253.0743.



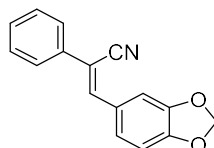
(Z)-2-phenyl-3-(*m*-tolyl)acrylonitrile (5bb) [67366-25-0]^[8]

The title compound was prepared according to general procedure. Yield 69% as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.76 (d, *J* = 7.8 Hz, 1H), 7.75 – 7.68 (m, 3H), 7.54 (s, 1H), 7.44 (m, 4H), 7.29 (d, *J* = 6.8 Hz, 1H), 2.46 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 142.47, 138.68, 134.60, 133.71, 131.44, 130.01, 129.16, 129.08, 128.89, 126.41, 126.00, 118.10, 111.37, 21.44. HR-MS (ESI⁺, *m/z*): calcd for C₁₆H₁₃NNa [M+Na]⁺: 242.0946, found: 242.0945.



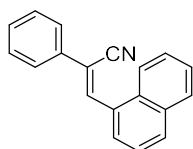
(Z)-2-phenyl-3-(*o*-tolyl)acrylonitrile (5cc) [2248161-99-9]^[8]

The title compound was prepared according to general procedure. Yield 63% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.00 – 7.94 (m, 1H), 7.79 (s, 1H), 7.73 (dd, *J* = 8.0, 1.0 Hz, 2H), 7.53 – 7.43 (m, 3H), 7.40 – 7.33 (m, 2H), 7.30 (d, *J* = 2.3 Hz, 1H), 2.43 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 141.16, 137.50, 134.35, 133.19, 130.48, 130.26, 129.31, 129.11, 128.12, 126.44, 126.14, 117.76, 113.91, 19.98. HR-MS (ESI⁺, *m/z*): calcd for C₁₆H₁₃NNa [M+Na]⁺: 242.0946, found: 242.0942.



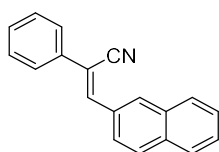
(Z)-3-(benzo[*d*][1,3]dioxol-5-yl)-2-phenylacrylonitrile (5dd) [37629-66-6]^[8]

The title compound was prepared according to general procedure. Yield 70% as a yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 7.81 (d, *J* = 8.1 Hz, 1H), 7.75 – 7.62 (m, 3H), 7.52 – 7.38 (m, 3H), 7.00 – 6.84 (m, 2H), 6.06 (s, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 147.60, 147.06, 134.29, 133.96, 129.31, 129.08, 126.00, 122.08, 119.52, 117.90, 116.50, 112.21, 110.33, 101.42. HR-MS (ESI⁺, *m/z*): calcd for C₁₆H₁₁NO₂Na [M+Na]⁺: 272.0687, found: 272.0689.



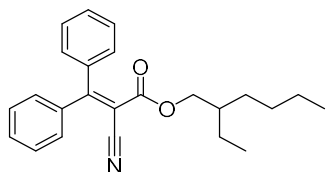
(Z)-3-(naphthalen-1-yl)-2-phenylacrylonitrile (5ee) [27869-54-1]^[8]

The title compound was prepared according to general procedure. Yield 42% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.32 (s, 1H), 8.13 (d, *J* = 7.2 Hz, 1H), 8.06 – 7.92 (m, 3H), 7.82 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.66 – 7.57 (m, 3H), 7.51 (m, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 140.32, 134.19, 133.54, 131.58, 131.17, 130.79, 129.50, 129.19, 129.00, 127.08, 127.00, 126.47, 126.24, 125.58, 123.40, 117.75, 115.35. HR-MS (ESI⁺, *m/z*): calcd for C₁₉H₁₃NNa [M+Na]⁺: 278.0946, found: 278.0951.



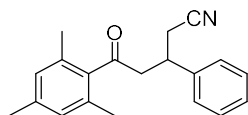
(Z)-3-(naphthalen-2-yl)-2-phenylacrylonitrile (5ff) [27869-55-2]^[8]

The title compound was prepared according to general procedure. Yield 49% as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.31 (s, 1H), 8.12 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.98 – 7.91 (m, 2H), 7.89 (d, *J* = 6.9 Hz, 1H), 7.79 – 7.73 (m, 2H), 7.70 (s, 1H), 7.62 – 7.54 (m, 2H), 7.48 (m, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 142.23, 134.63, 134.13, 133.12, 131.27, 130.48, 129.23, 129.12, 128.81, 128.74, 127.79, 127.68, 126.84, 126.03, 125.28, 118.23, 111.60. HR-MS (ESI⁺, *m/z*): calcd for C₁₉H₁₃NNa [M+Na]⁺: 278.0946, found: 278.0949.



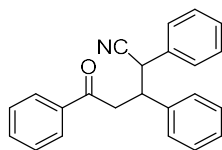
2-ethylhexyl-2-cyano-3,3-diphenylacrylate (5hh) [6197-30-4]^[13]

The title compound was prepared according to general procedure. Yield 67% as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.55 – 7.36 (m, 8H), 7.19 (d, *J* = 7.1 Hz, 2H), 4.12 – 3.97 (m, 2H), 1.50 (m, 1H), 1.26 (m, 8H), 0.93 (t, *J* = 7.0 Hz, 3H), 0.86 (t, *J* = 7.5 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 168.85, 162.96, 138.71, 138.39, 131.42, 130.38, 130.27, 129.32, 128.53, 128.30, 116.82, 104.16, 68.56, 38.56, 30.13, 28.88, 23.50, 22.92, 14.09, 10.97. HR-MS (ESI⁺, *m/z*): calcd for C₂₄H₂₇NO₂Na [M+Na]⁺: 384.1939, found: 384.1945.



5-mesityl-5-oxo-3-phenylpentanenitrile (MA-1)

The titled compound was obtained through the reaction described in Section 3.5 of Chapter 3. Yield 92% as a colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 7.42 – 7.29 (m, 5H), 6.86 (s, 2H), 3.82 – 3.72 (m, 1H), 3.27 (qd, J = 18.8, 6.9 Hz, 2H), 2.87 (qd, J = 16.8, 6.3 Hz, 2H), 2.31 (s, 3H), 2.12 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 207.76, 141.08, 138.79, 138.71, 132.57, 129.01, 128.67, 127.79, 127.40, 118.19, 48.81, 36.43, 24.38, 21.08, 18.99. HR-MS (ESI^+ , m/z): calcd for $\text{C}_{20}\text{H}_{21}\text{NONa}$ $[\text{M}+\text{Na}]^+$: 314.1521, found: 314.1516.



5-mesityl-5-oxo-3-phenylpentanenitrile (MA-2)

The titled compound was obtained through the reaction described in Section 3.5 of Chapter 3. Yield 88% as a yellow solid. ^1H NMR (400 MHz, CDCl_3) δ 8.01 (d, J = 7.1 Hz, 2H), 7.62 (t, J = 7.4 Hz, 1H), 7.51 (t, J = 7.6 Hz, 2H), 7.32 – 7.29 (m, 3H), 7.27 – 7.23 (m, 3H), 7.20 – 7.14 (m, 2H), 7.14 – 7.08 (m, 2H), 4.53 (d, J = 5.3 Hz, 1H), 3.88 (dt, J = 8.6, 5.1 Hz, 1H), 3.78 (dd, J = 17.8, 8.5 Hz, 1H), 3.56 (dd, J = 17.8, 5.0 Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 197.64, 138.25, 136.59, 134.17, 133.61, 128.78, 128.66, 128.62, 128.33, 128.09, 128.05, 127.72, 119.37, 45.33, 43.07, 41.68. HR-MS (ESI^+ , m/z): calcd for $\text{C}_{23}\text{H}_{19}\text{NONa}$ $[\text{M}+\text{Na}]^+$: 348.1364, found: 348.1360.

V. Crystal data and structure refinement for MA-1

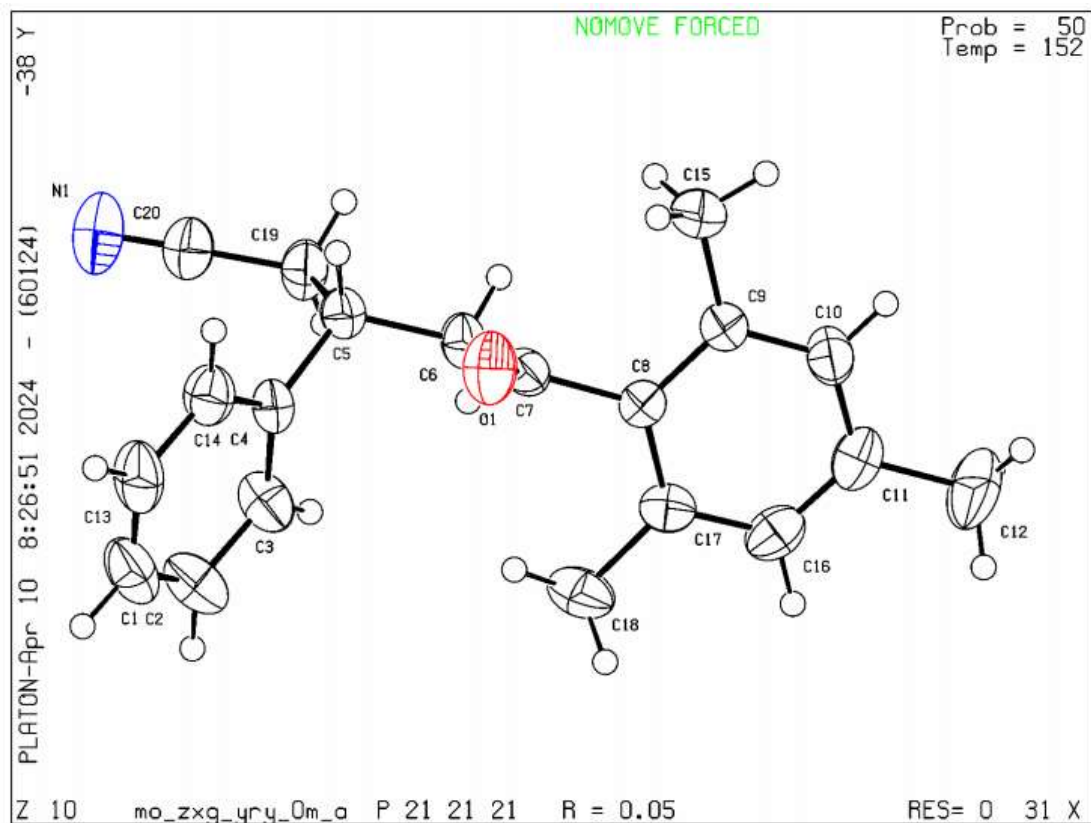


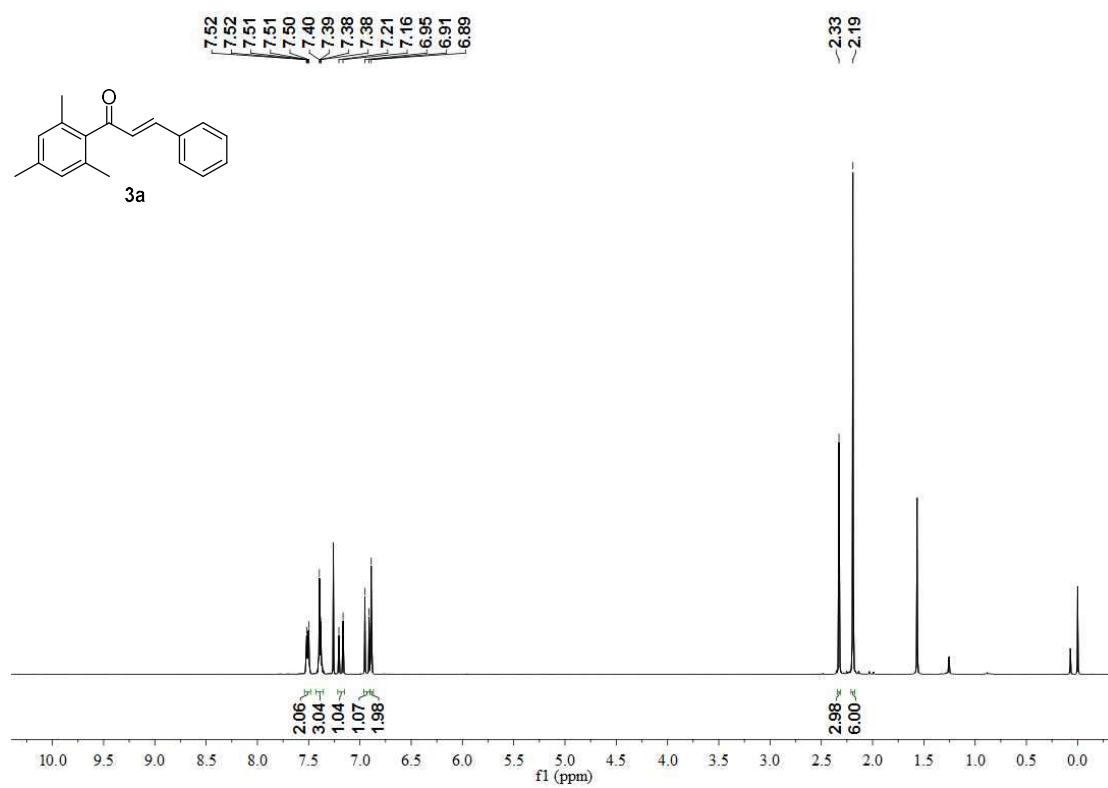
Table S6 Crystal data and structure refinement for MA-1.

Identification code	MA-1 (CCDC 2348624)
Empirical formula	C ₂₀ H ₂₁ NO
Formula weight	291.38
Temperature/K	152.0
Crystal system	orthorhombic
Space group	P2 ₁ 2 ₁ 2 ₁
<i>a</i> /Å	9.3333(11)
<i>b</i> /Å	9.4374(9)
<i>c</i> /Å	19.037(2)
<i>α</i> /°	90
<i>β</i> /°	90
<i>γ</i> /°	90
Volume/Å ³	1676.8(3)
<i>Z</i>	4
<i>ρ</i> _{calc} /cm ³	1.154
<i>μ</i> /mm ⁻¹	0.070

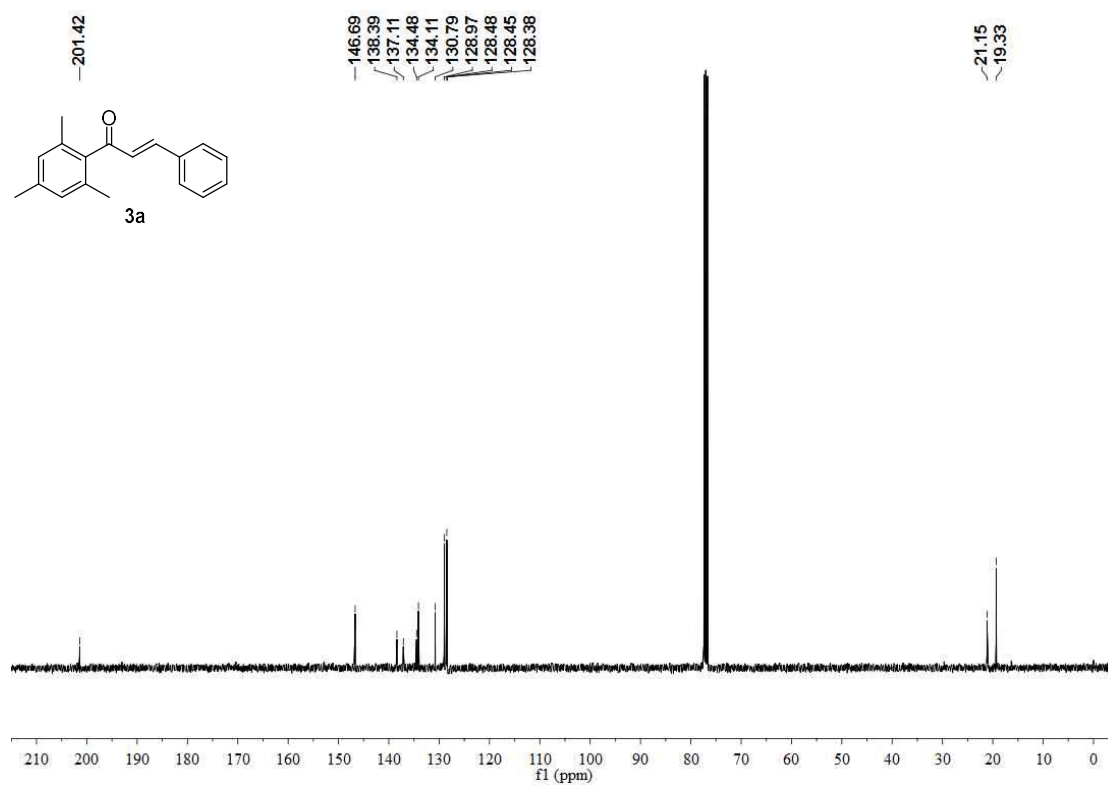
$F(000)$	624.0
Crystal size/mm ³	0.48 × 0.43 × 0.37
Radiation	MoK α (λ = 0.71073)
2 θ range for data collection/°	4.28 to 55.038
Index ranges	-12 ≤ h ≤ 12, -11 ≤ k ≤ 12, -24 ≤ l ≤ 24
Reflections collected	25250
Independent reflections	3819 [R _{int} = 0.0884, R _{sigma} = 0.0552]
Data/restraints/parameters	3819/0/202
Goodness-of-fit on F ²	1.046
Final R indexes [I ≥ 2 σ (I)]	R ₁ = 0.0475, wR ₂ = 0.1067
Final R indexes [all data]	R ₁ = 0.0688, wR ₂ = 0.1188
Largest diff. peak/hole / e Å ⁻³	0.18/-0.23
Flack parameter	-0.2(10)

VI. Copies of NMR spectra

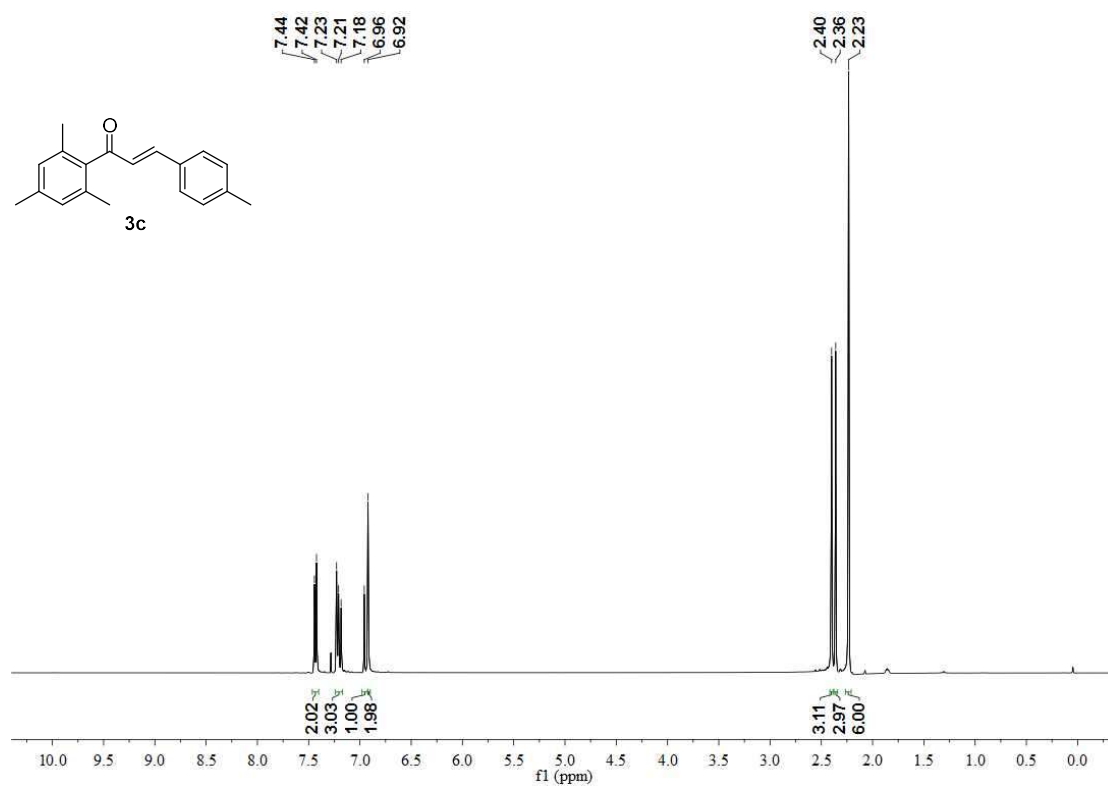
^1H NMR



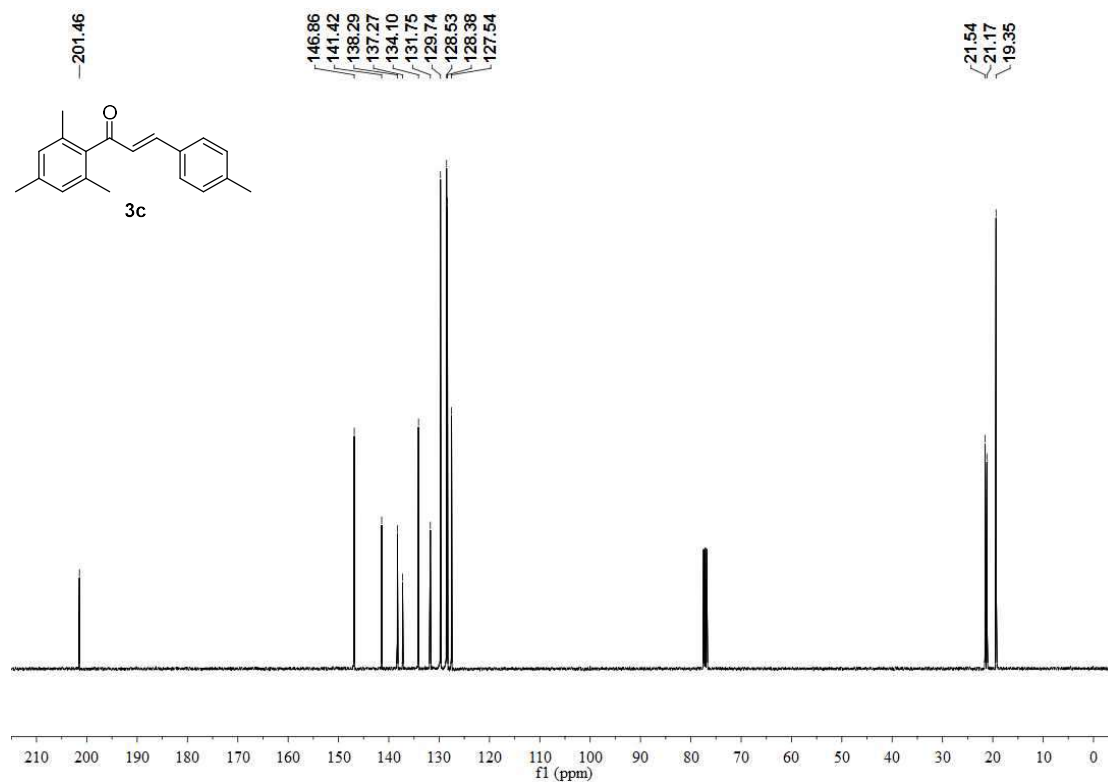
^{13}C NMR



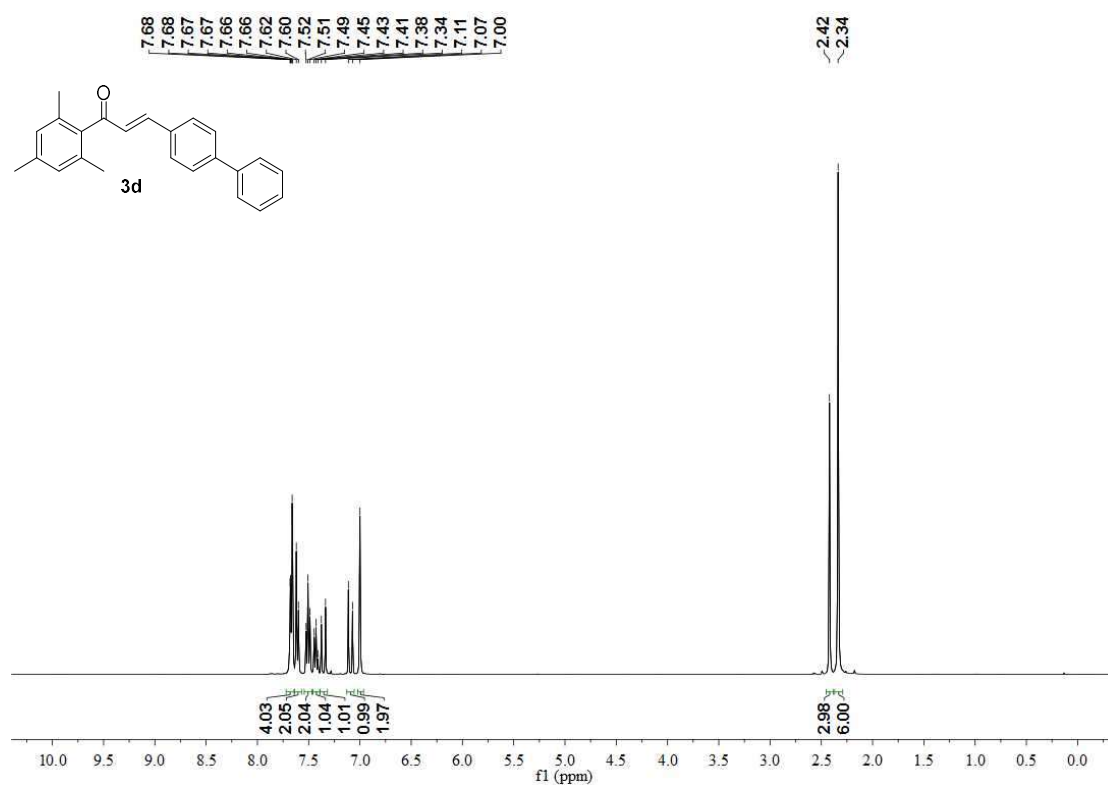
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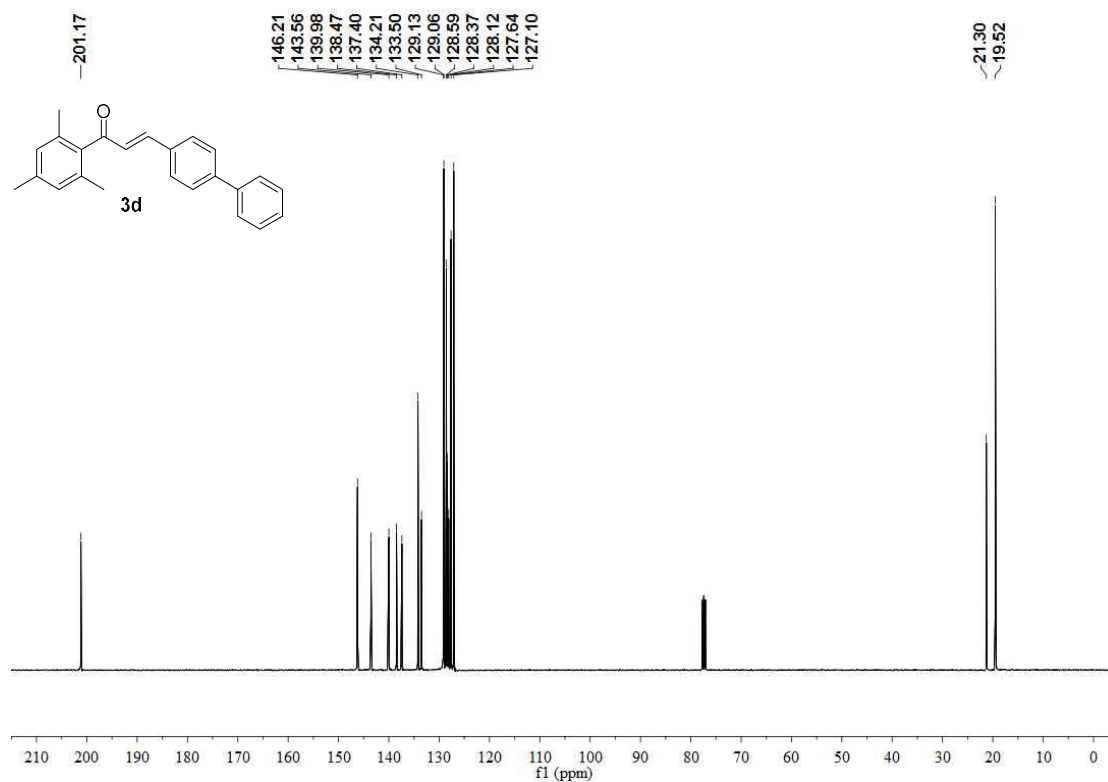
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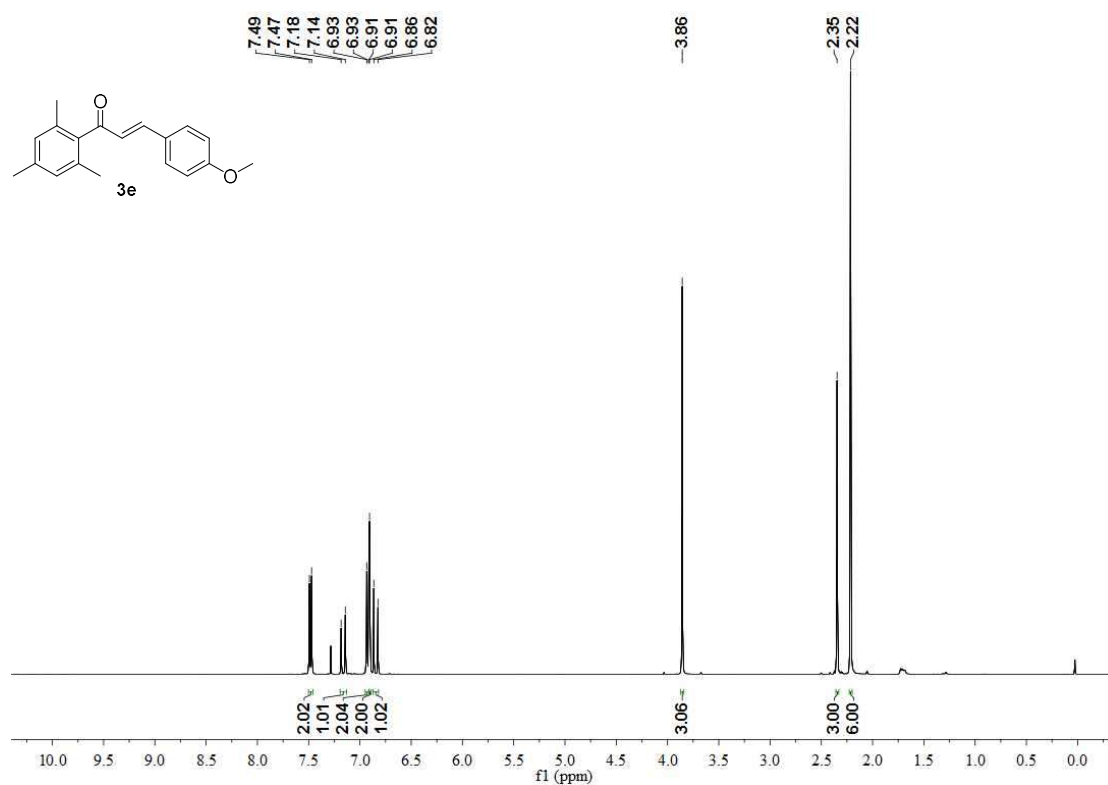
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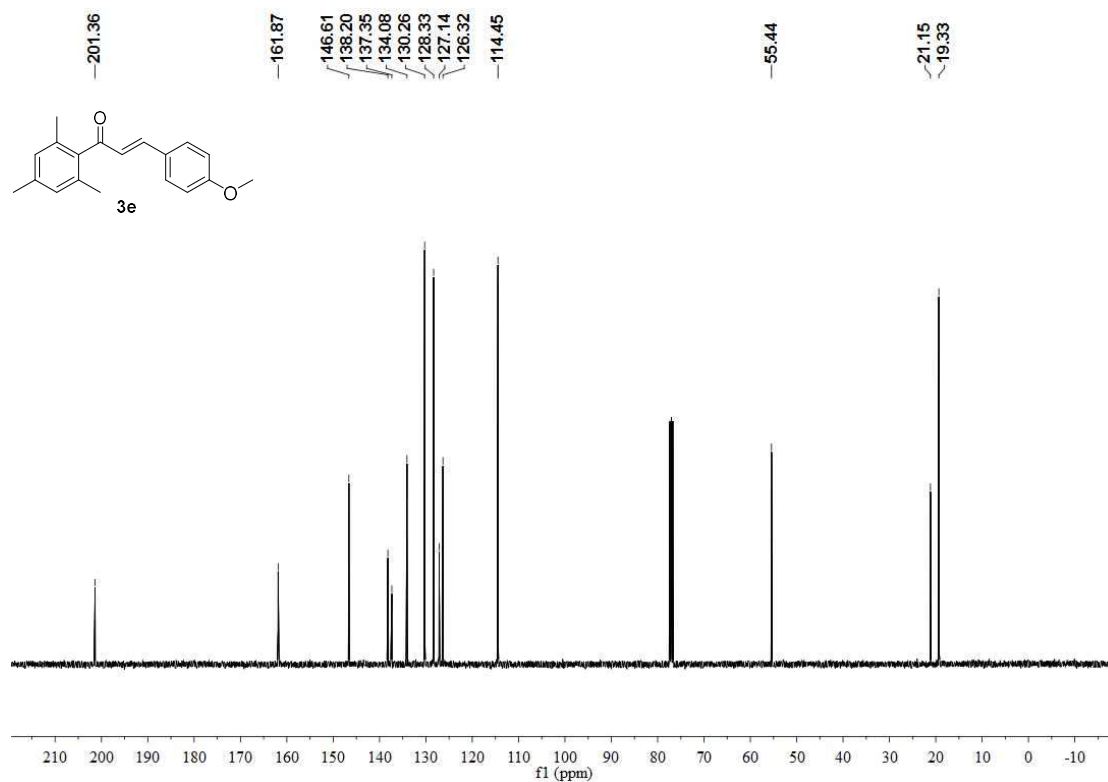
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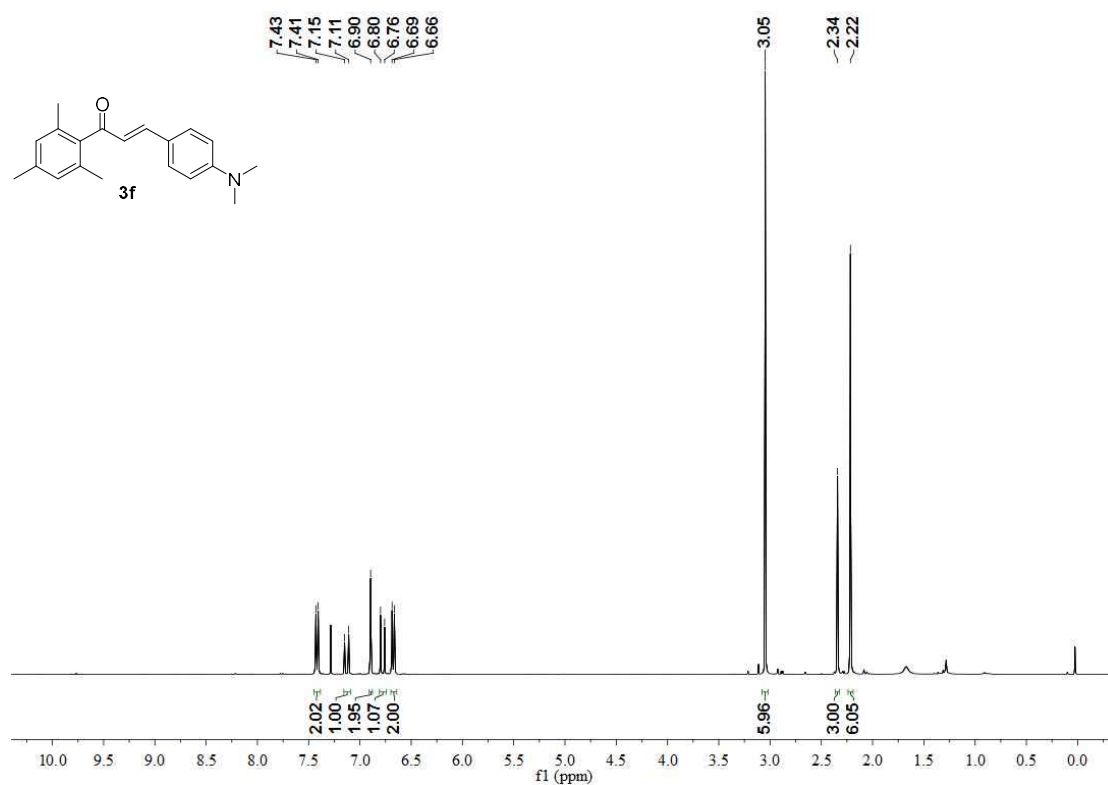
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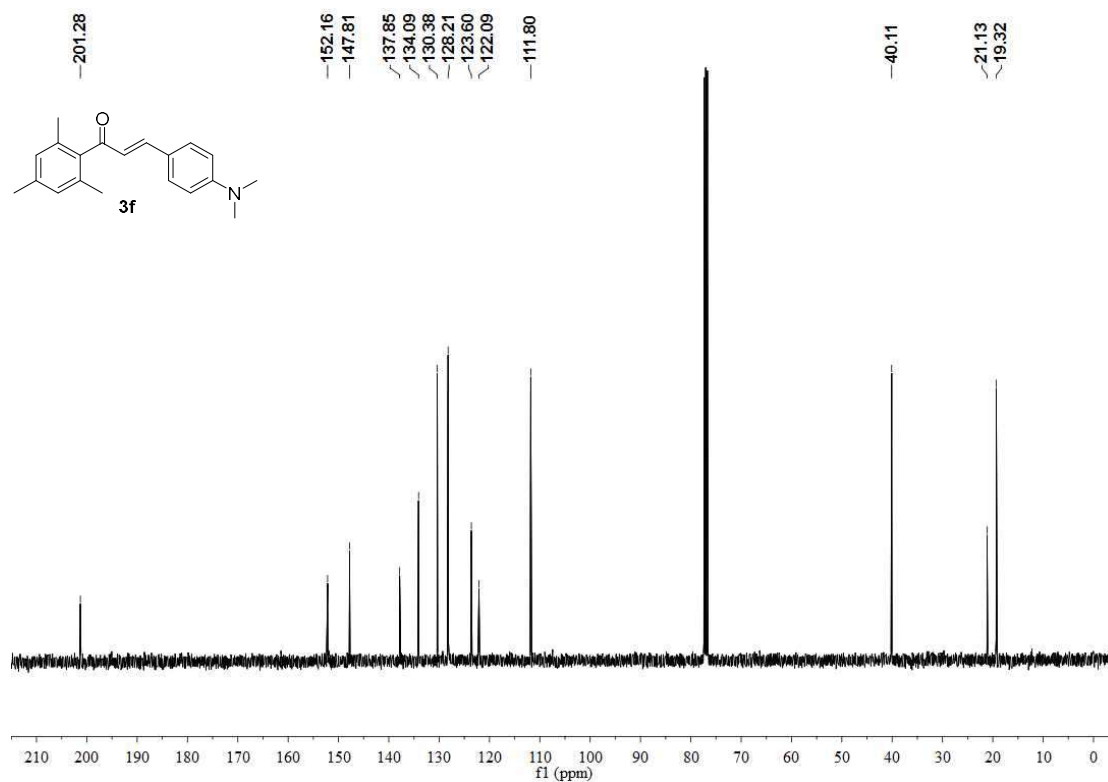
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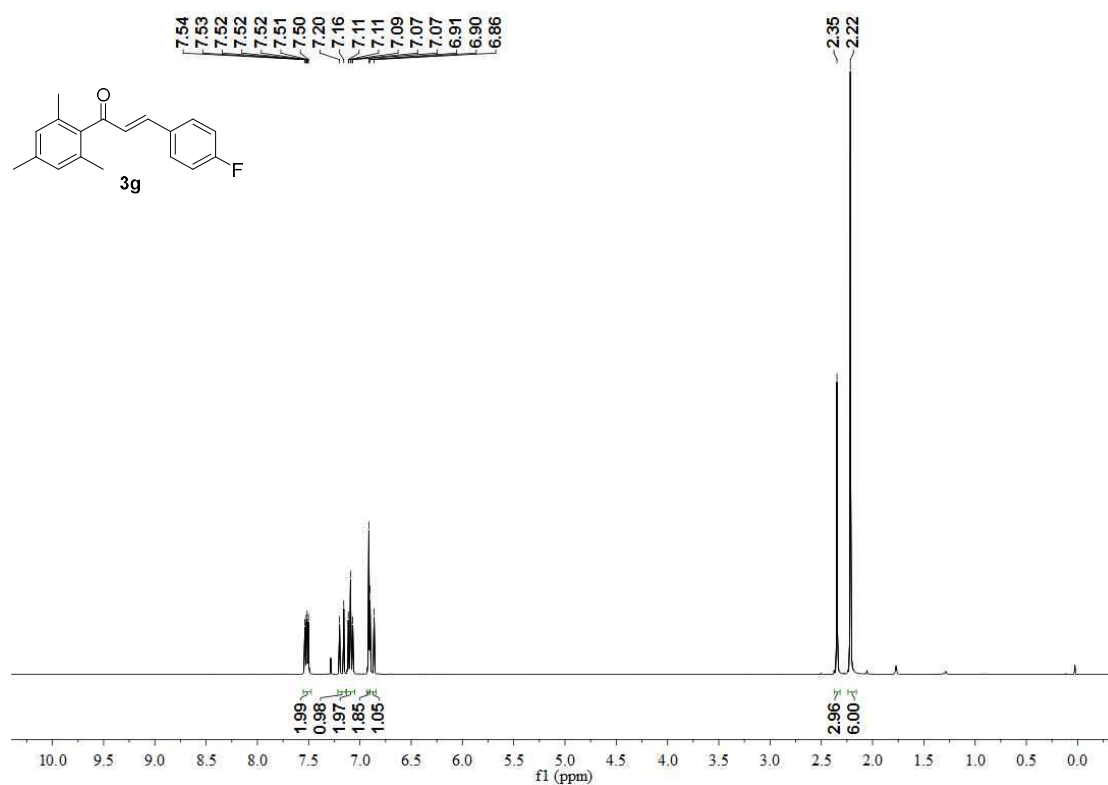
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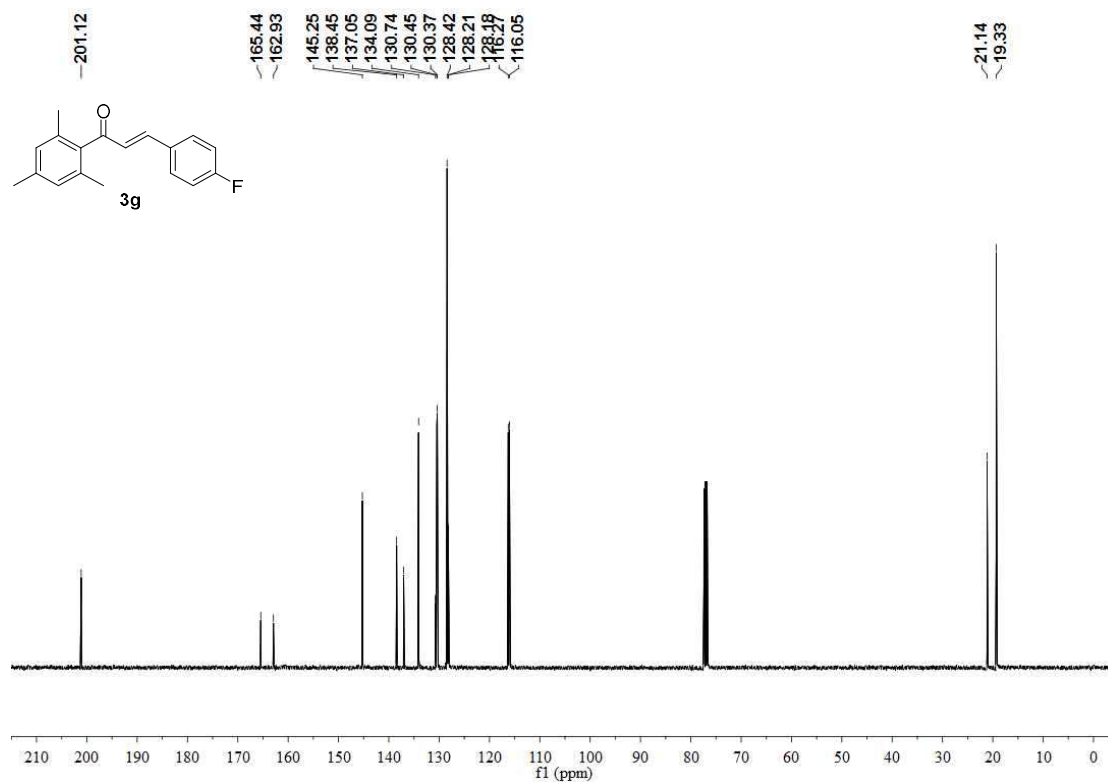
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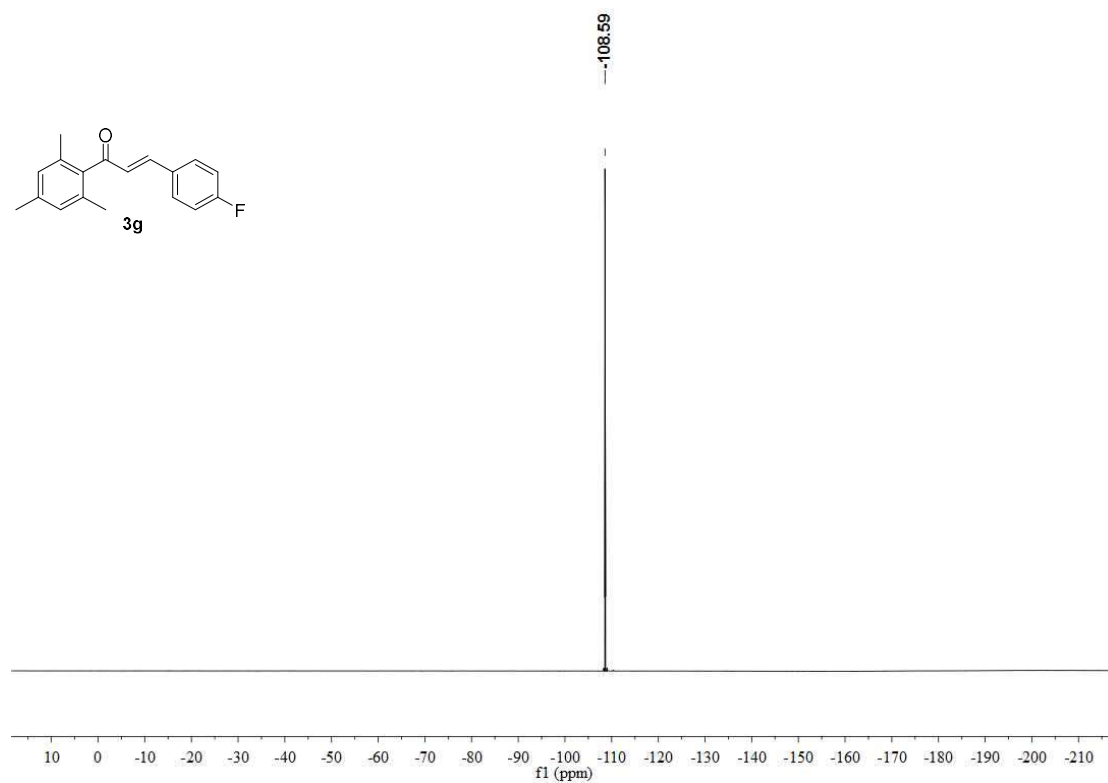
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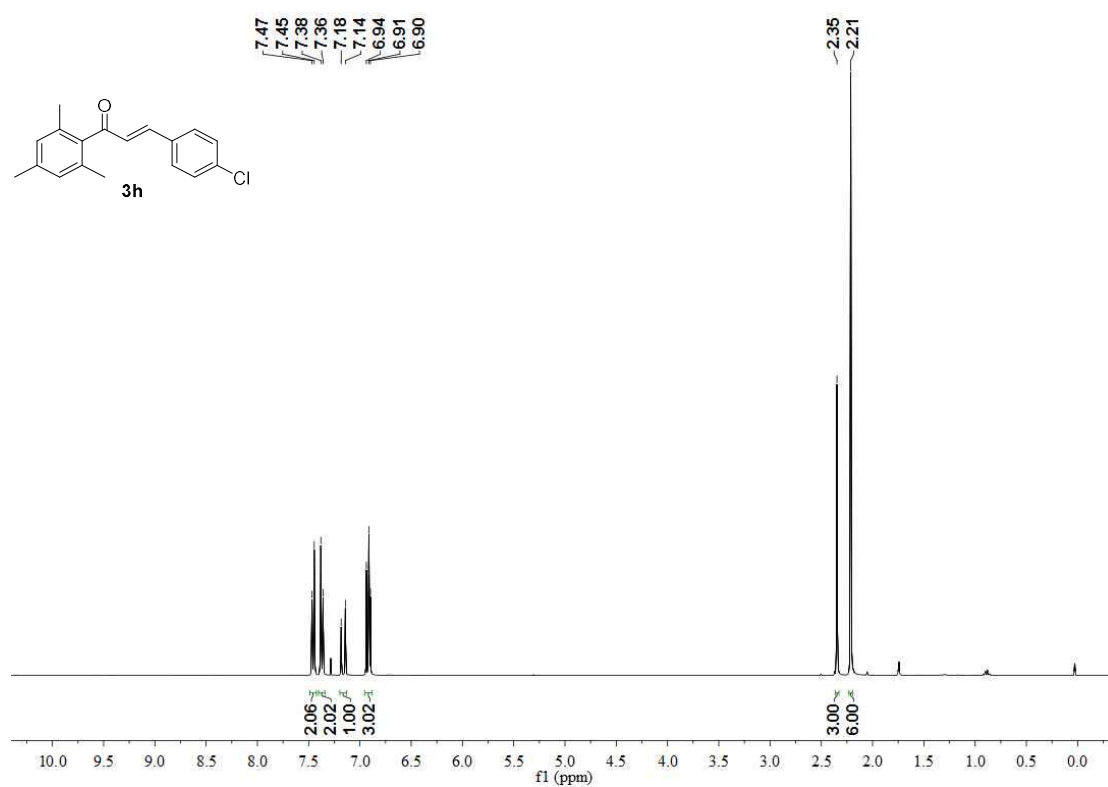
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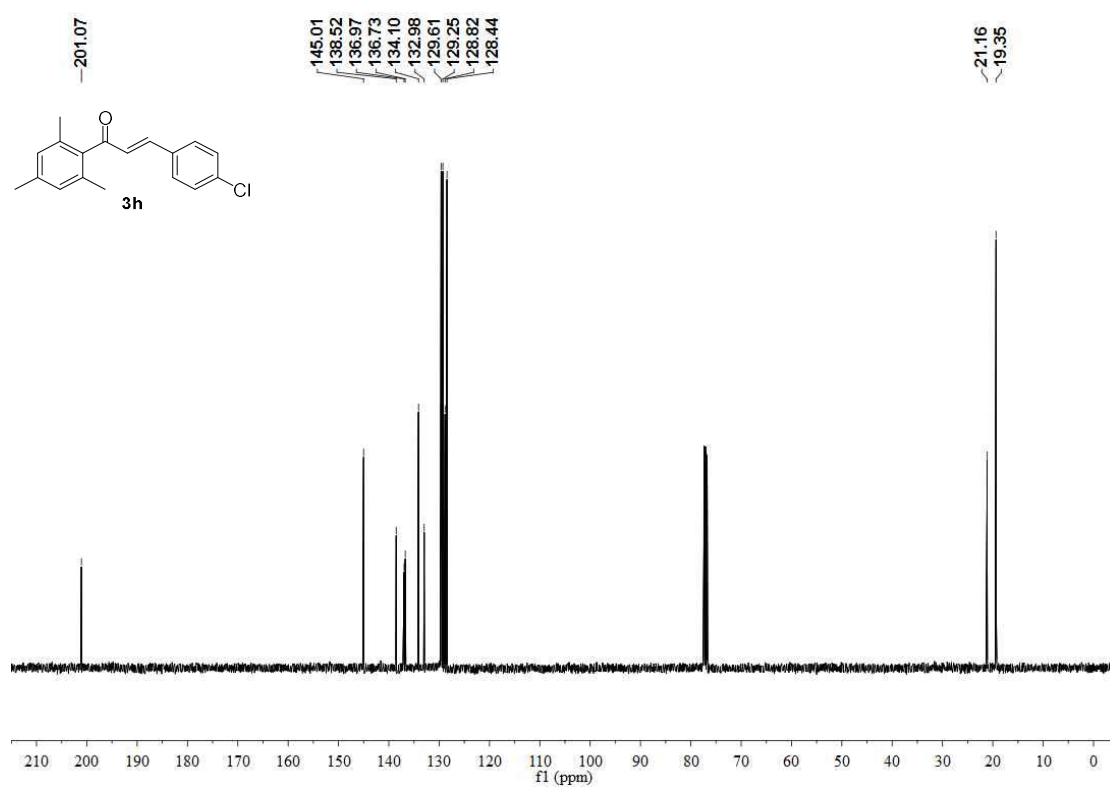
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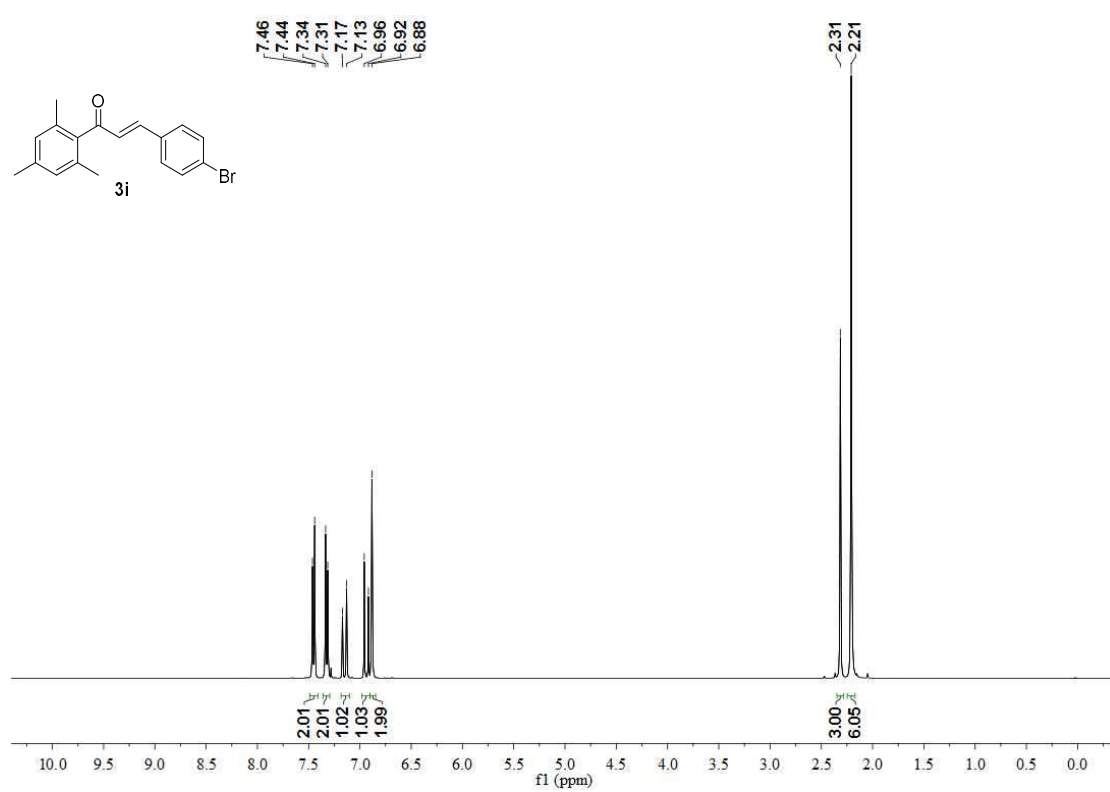
^1H NMR



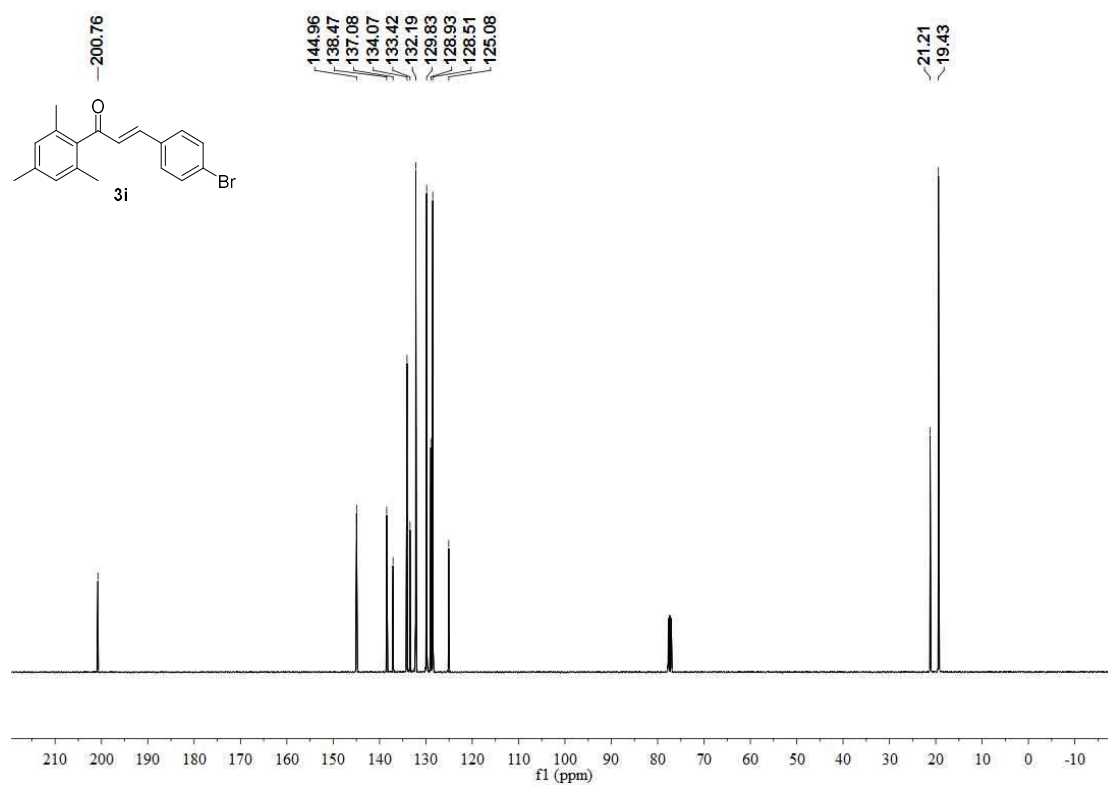
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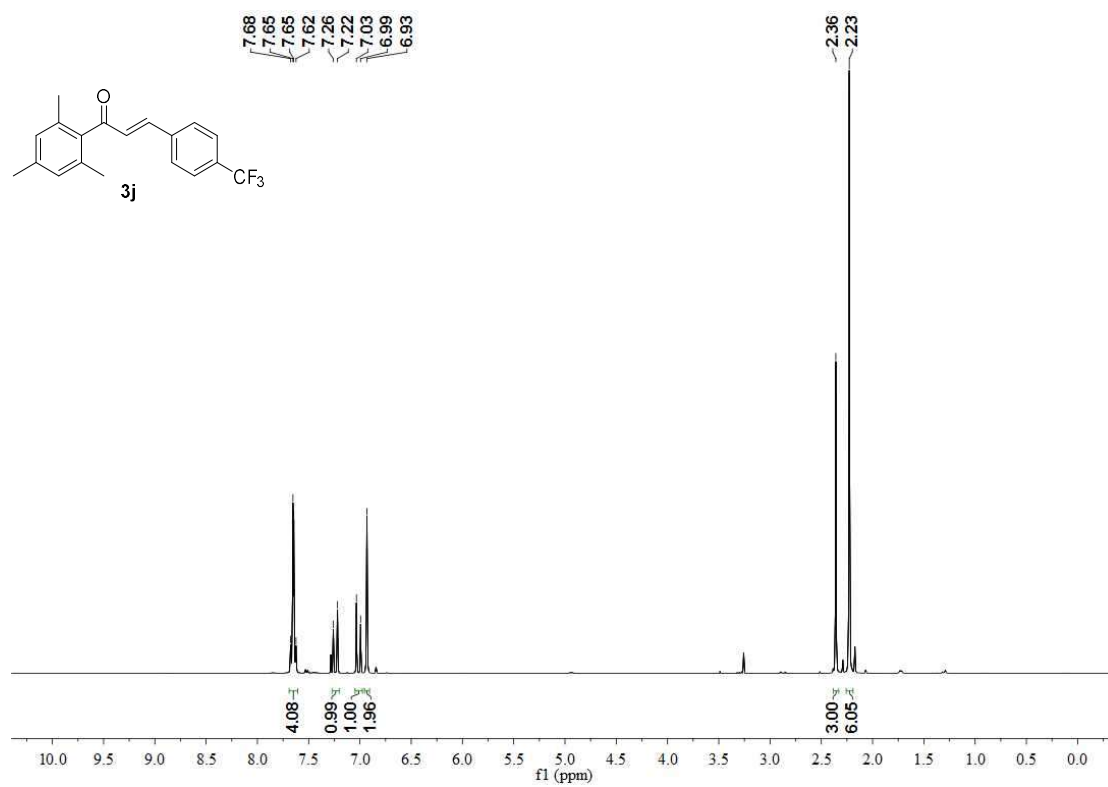
^1H NMR



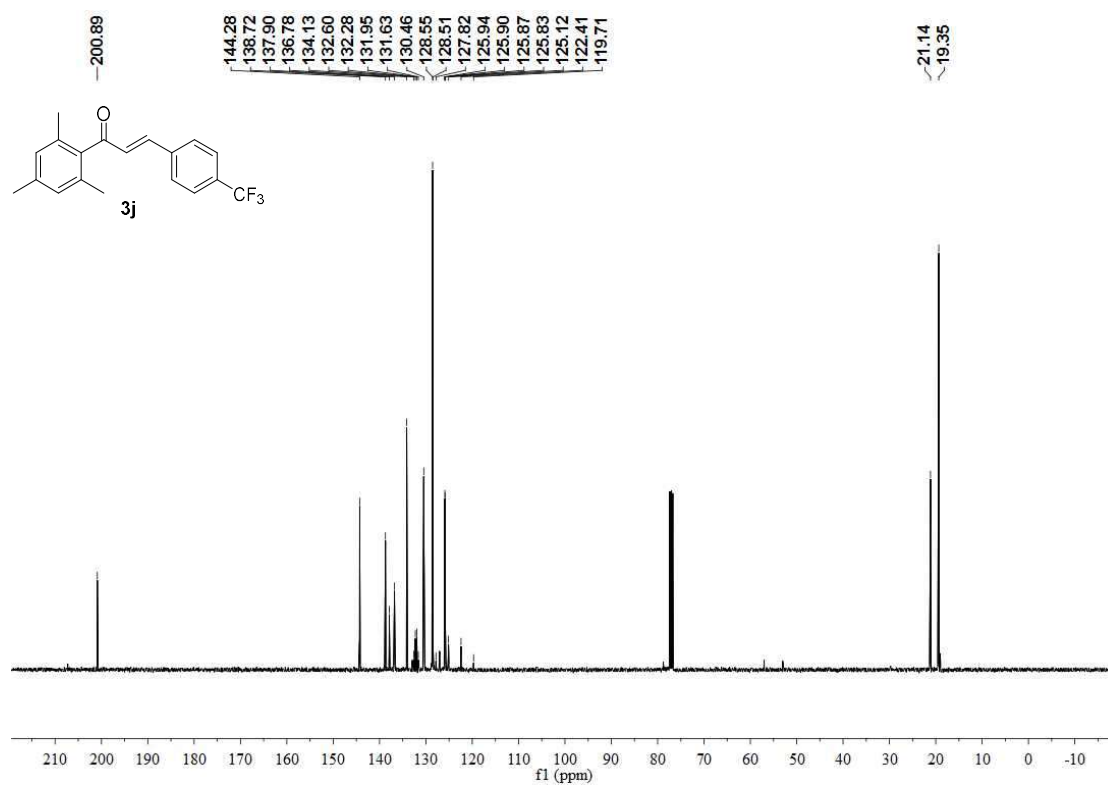
¹³C NMR



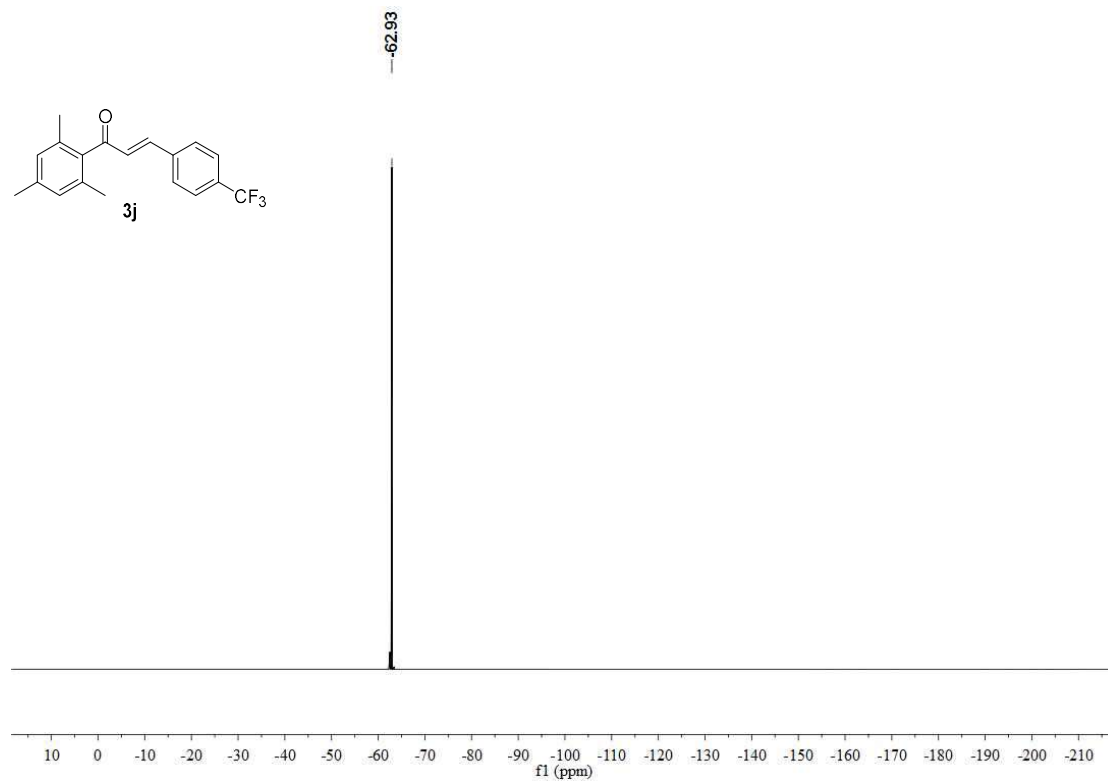
¹H NMR



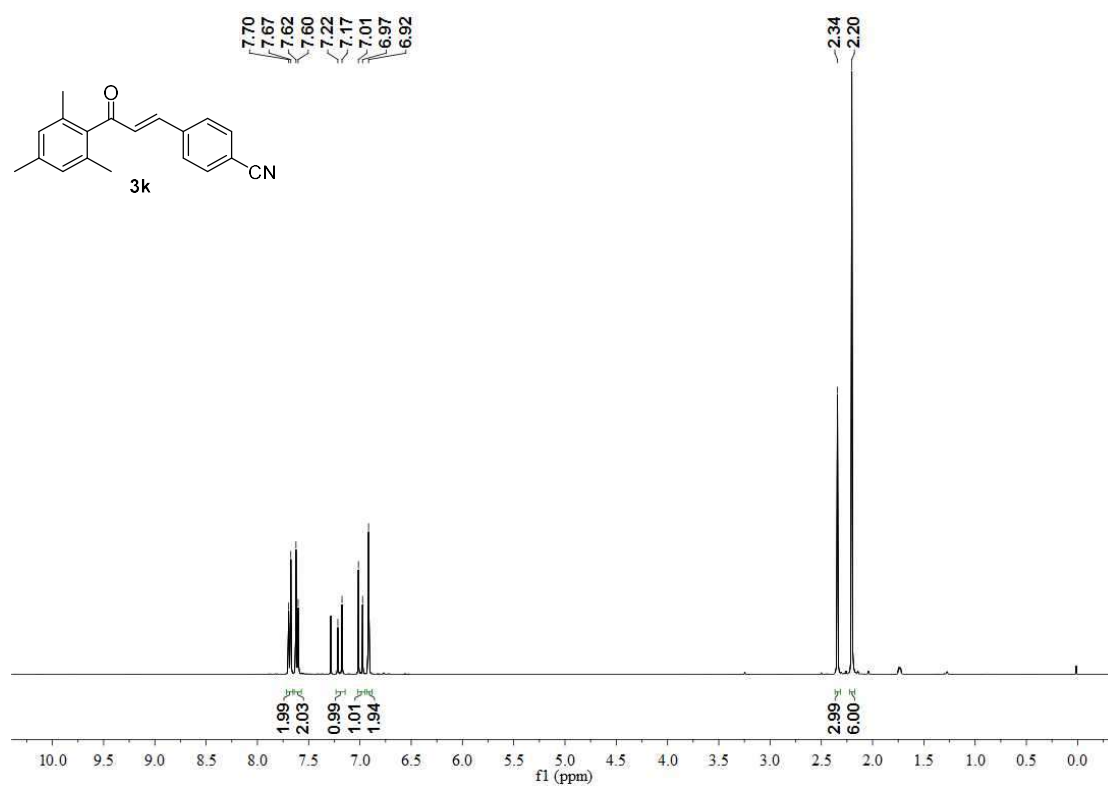
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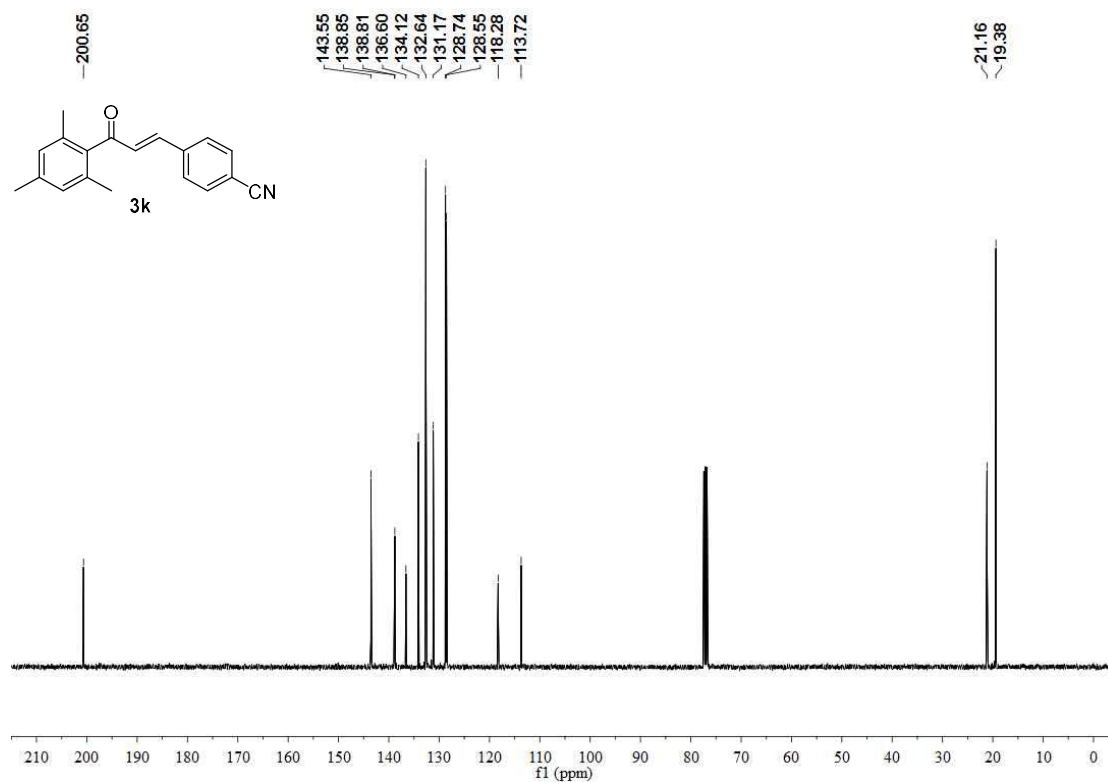
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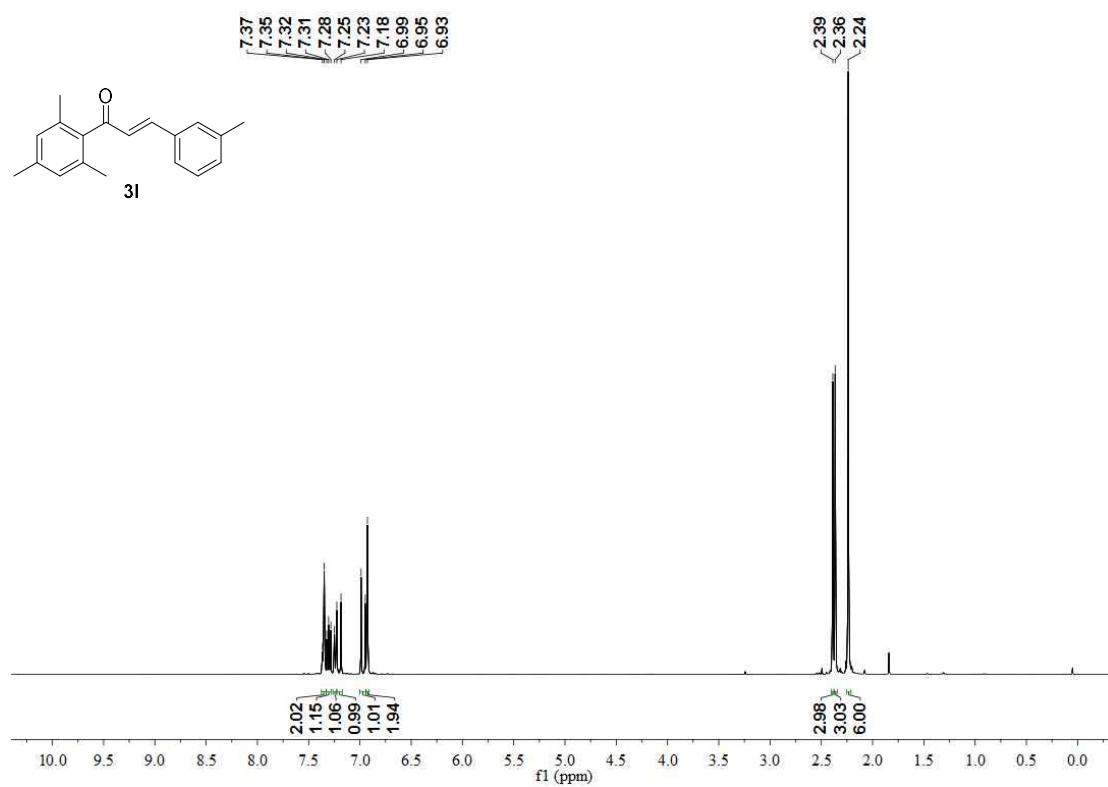
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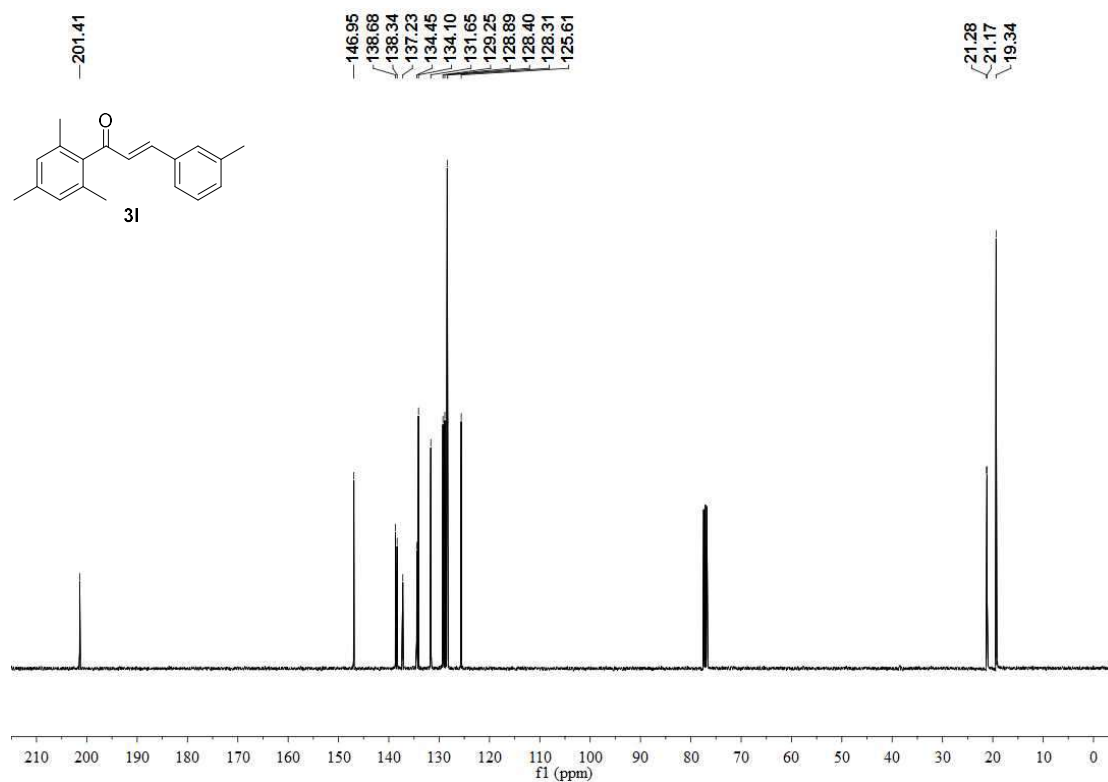
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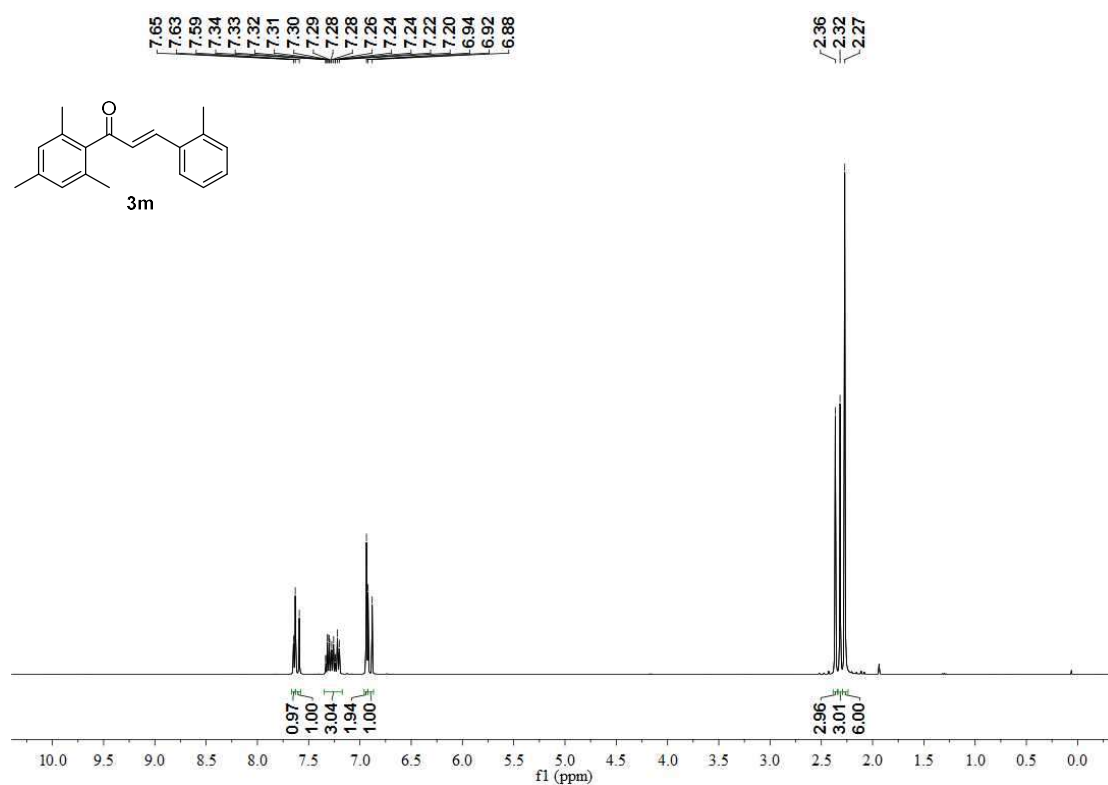
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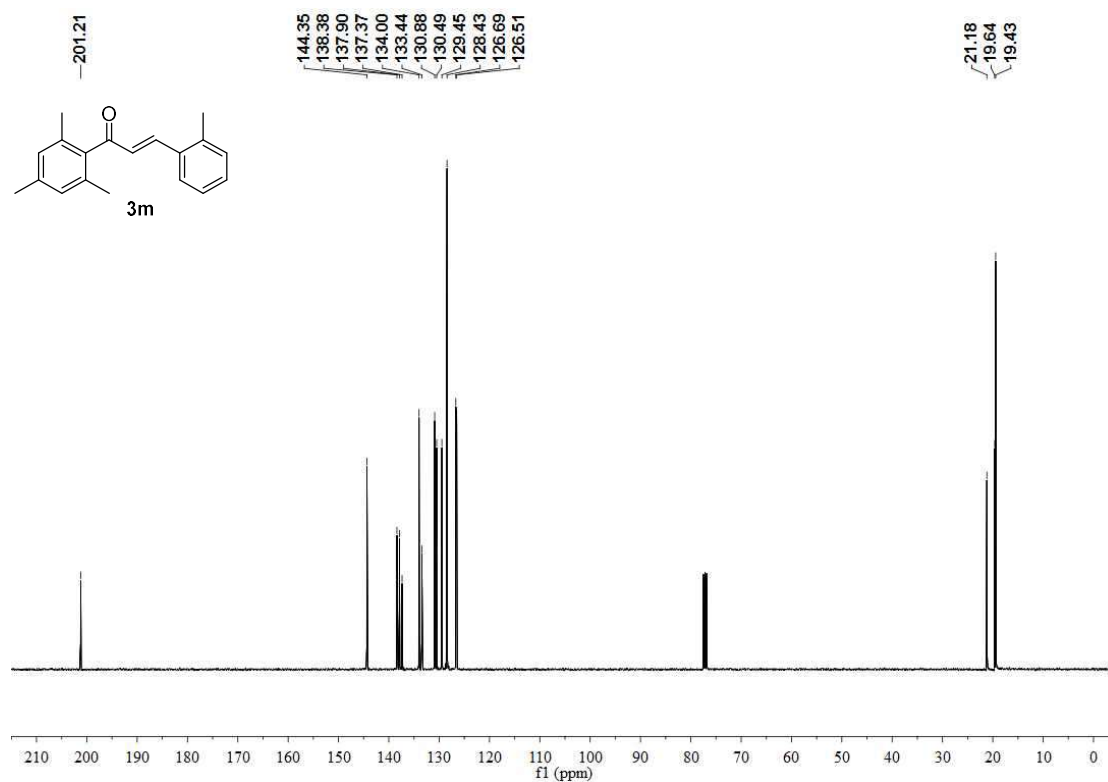
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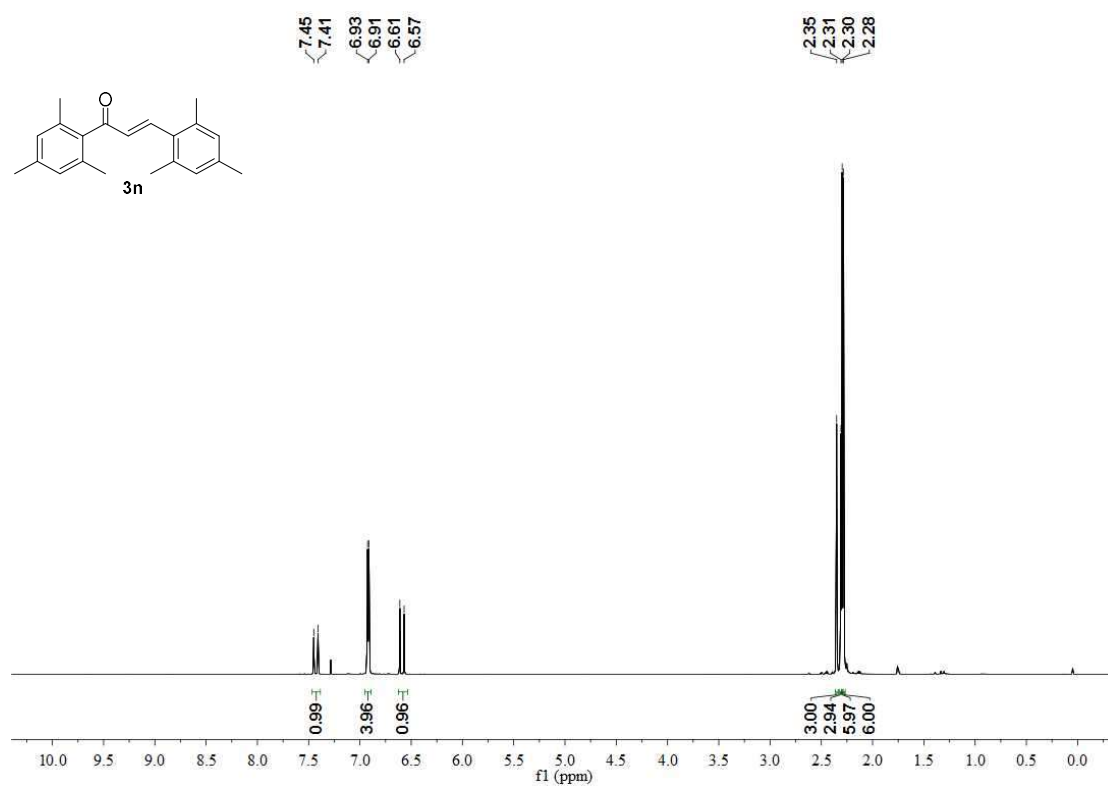
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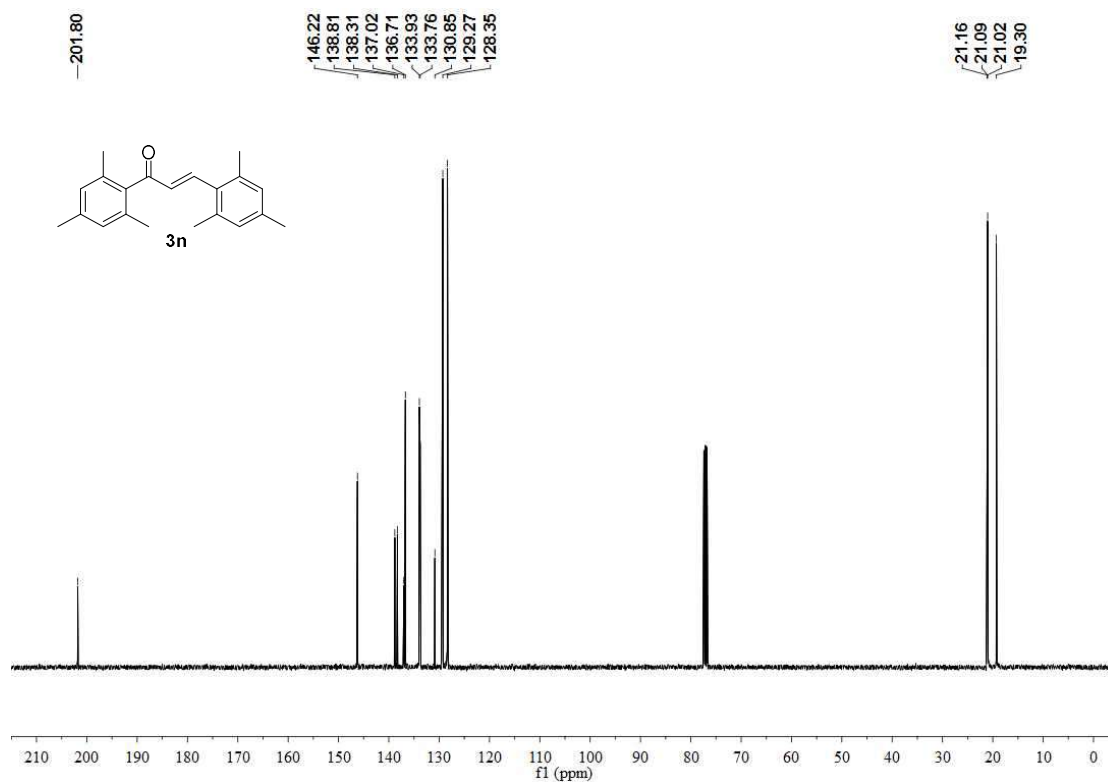
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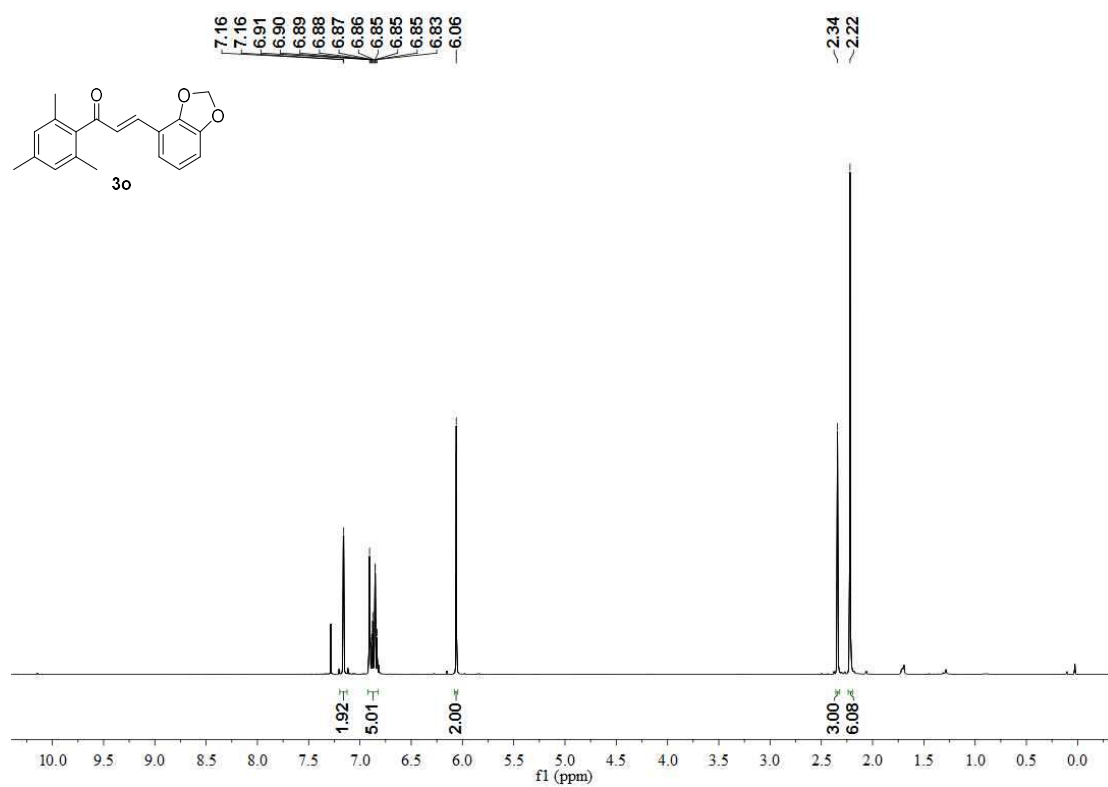
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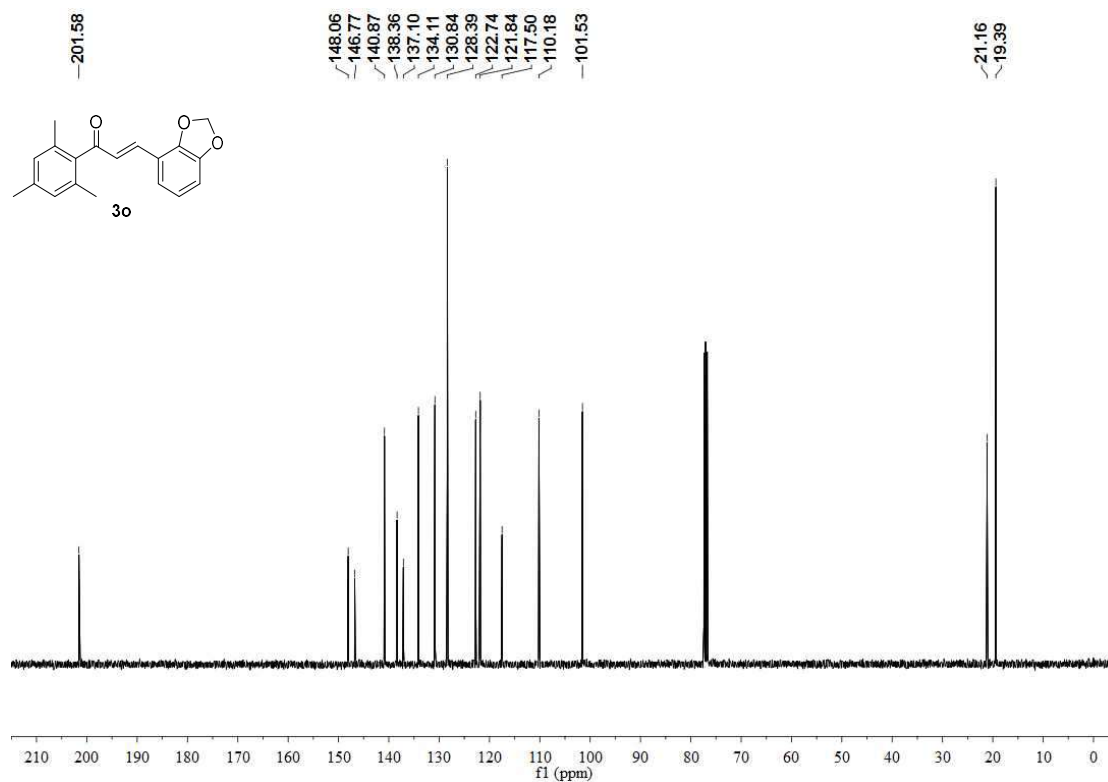
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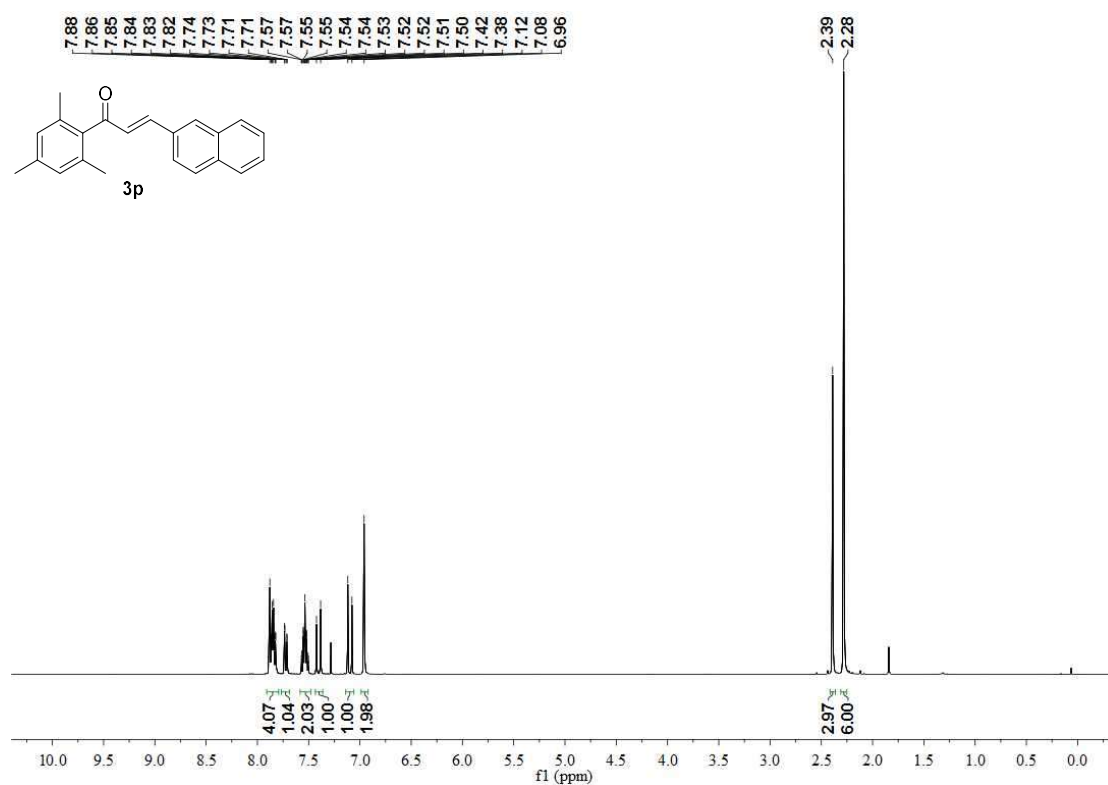
¹H NMR



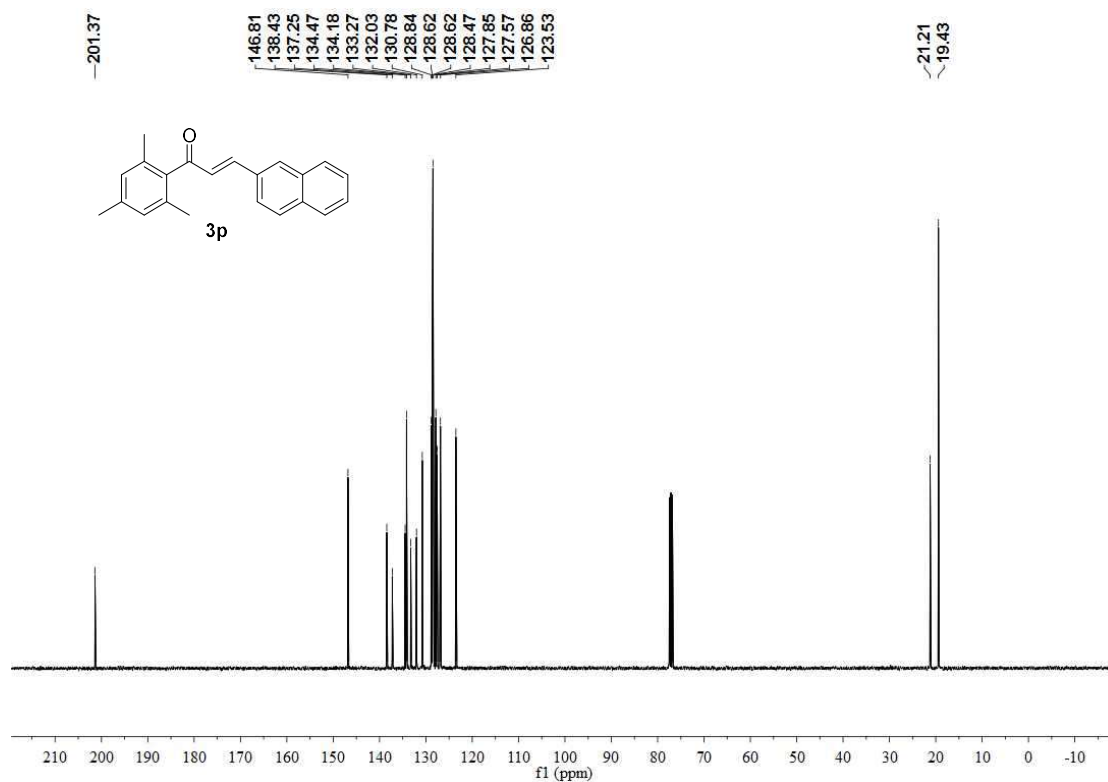
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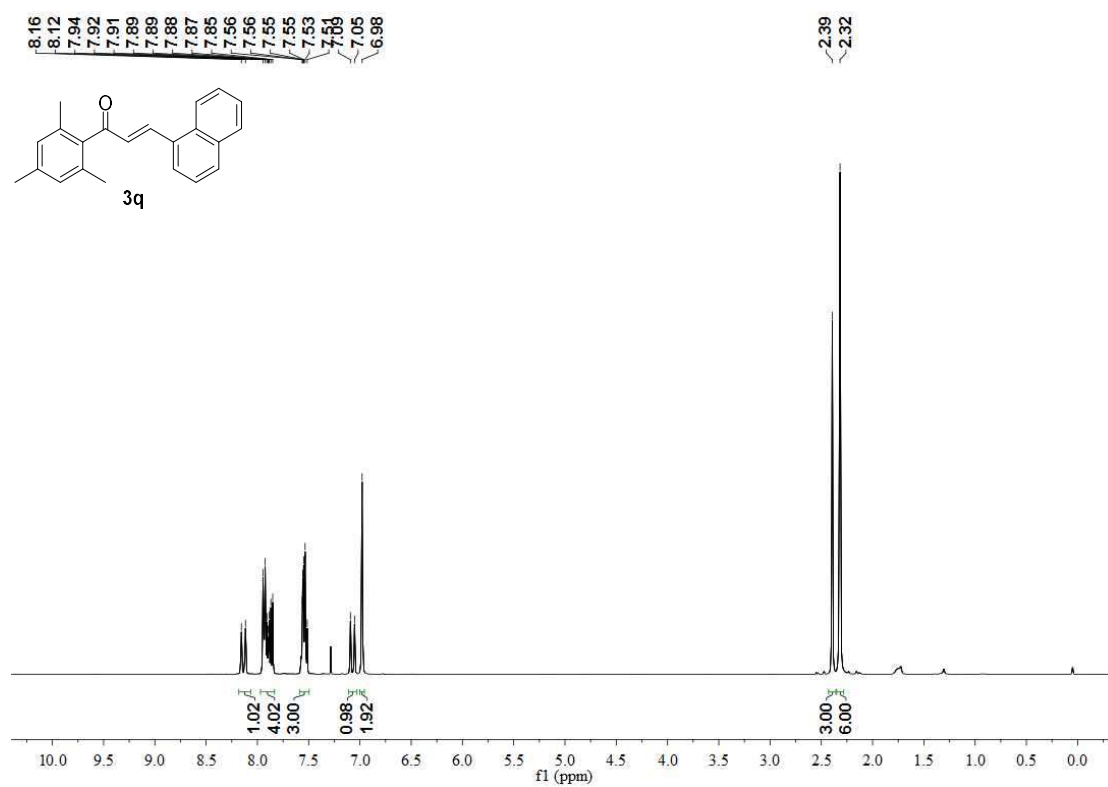
¹H NMR



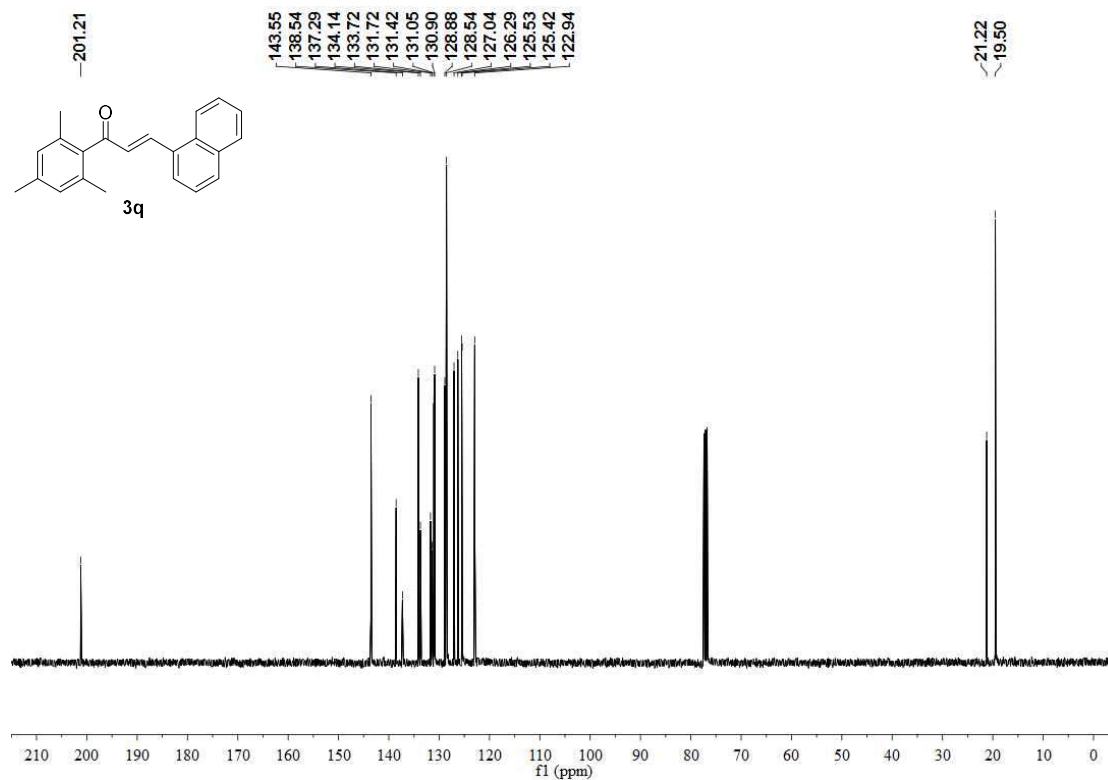
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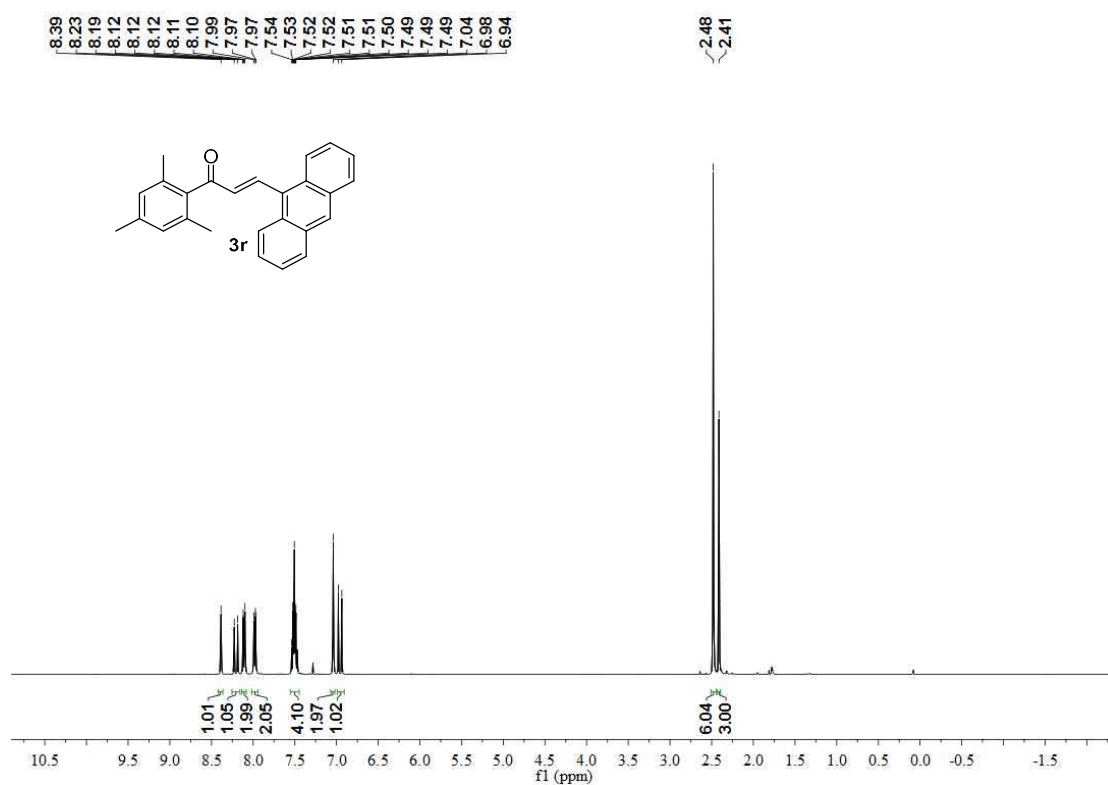
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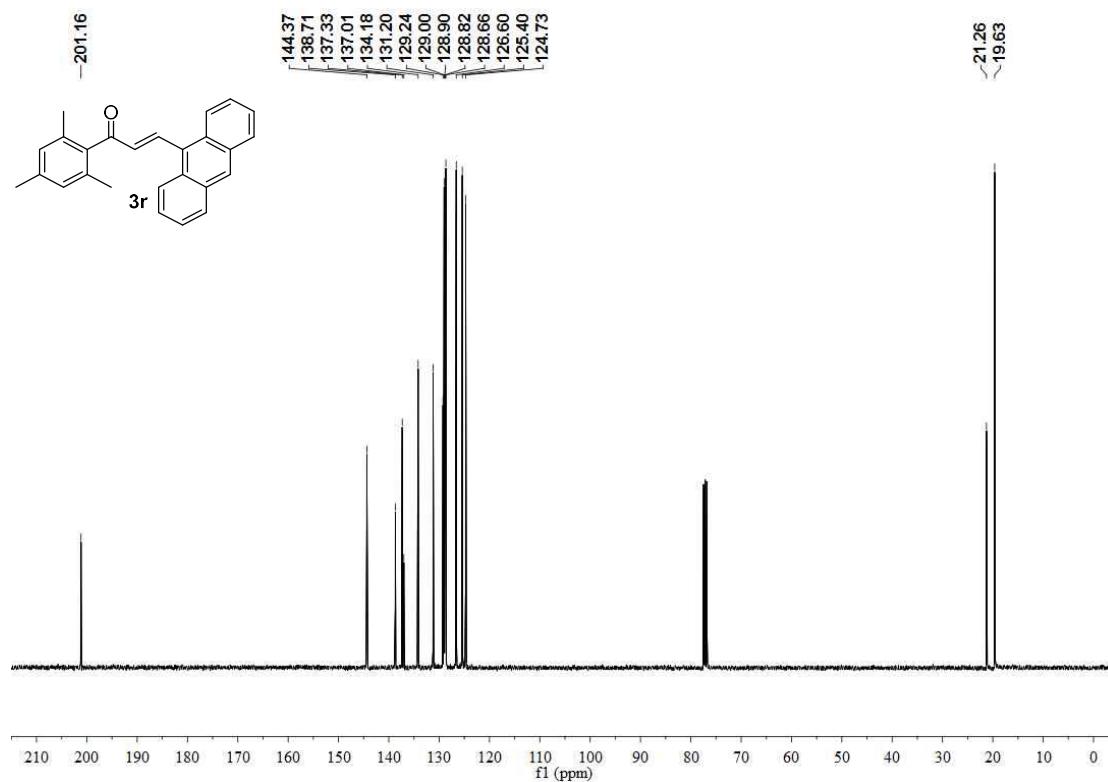
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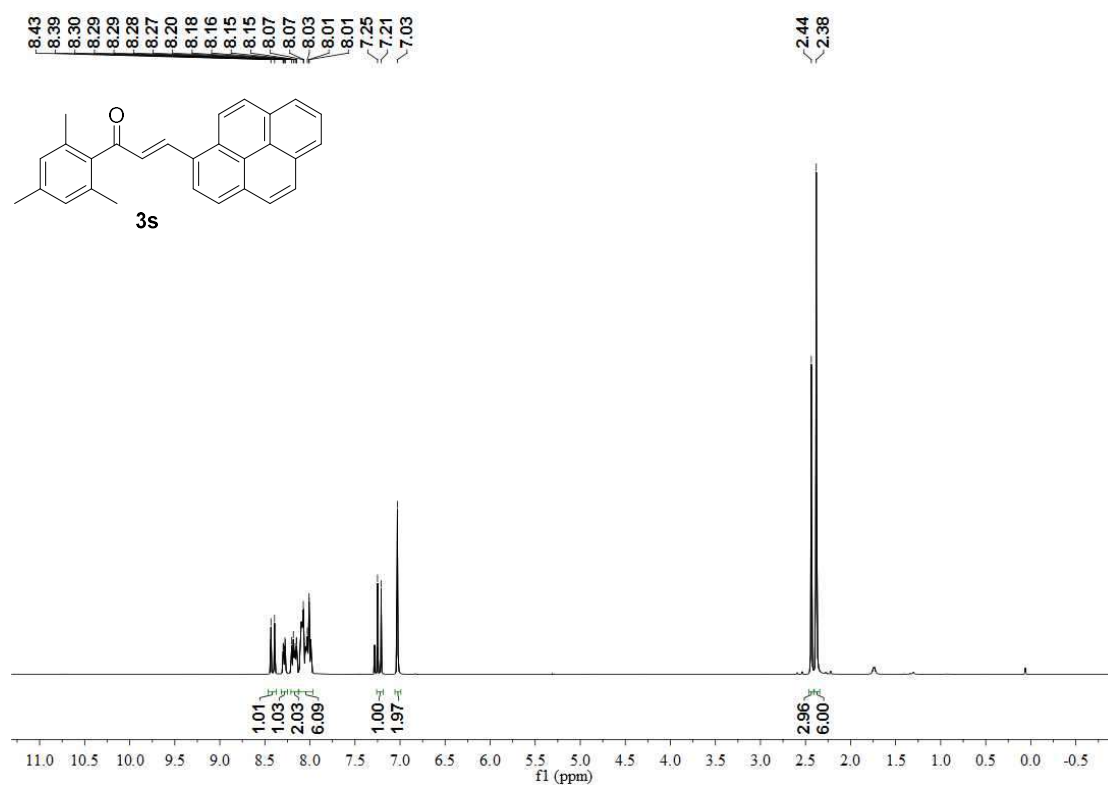
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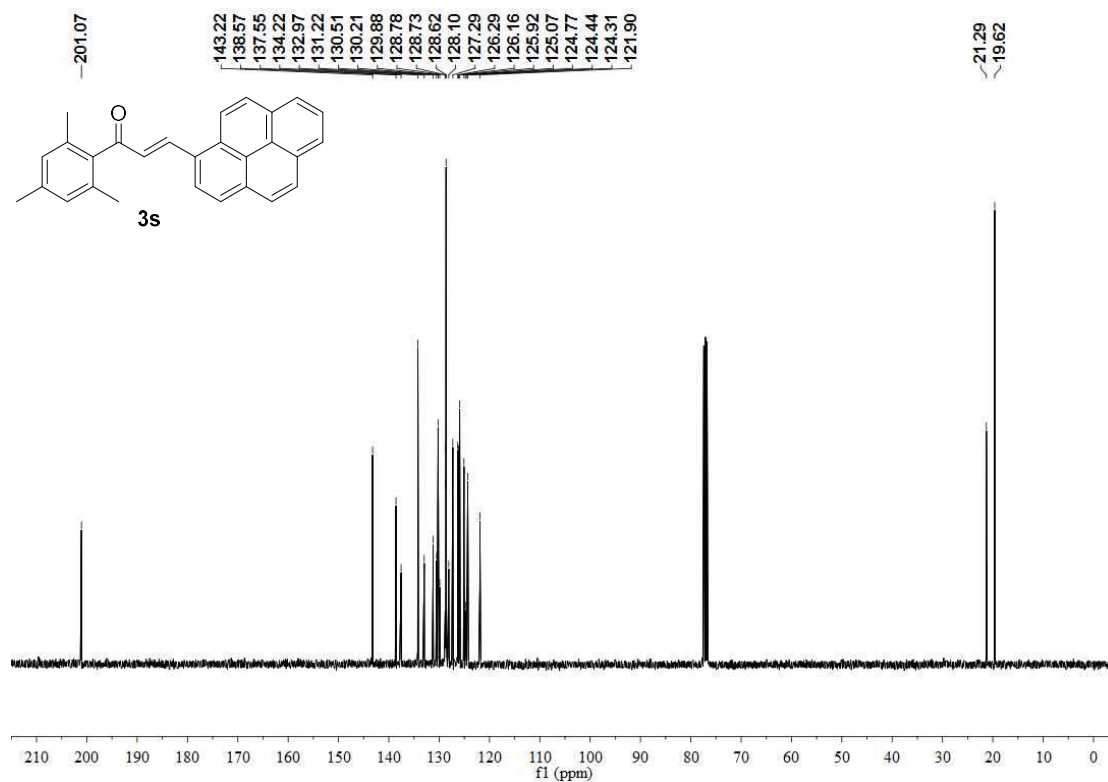
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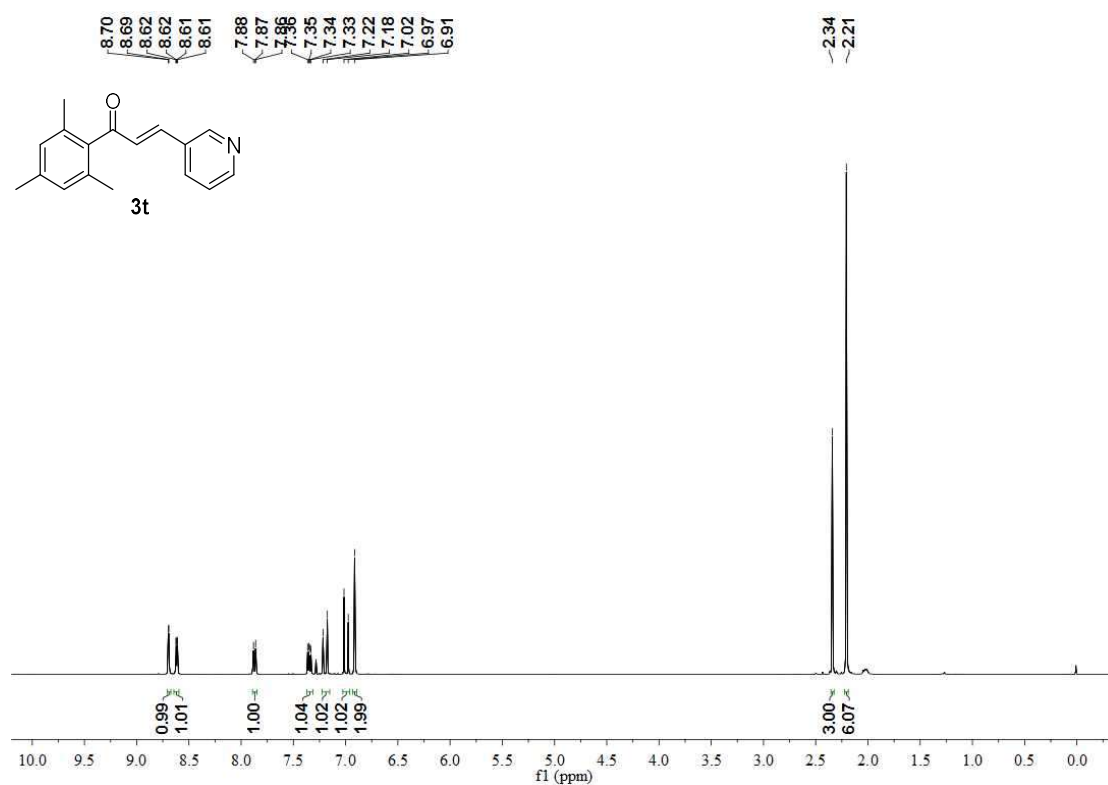
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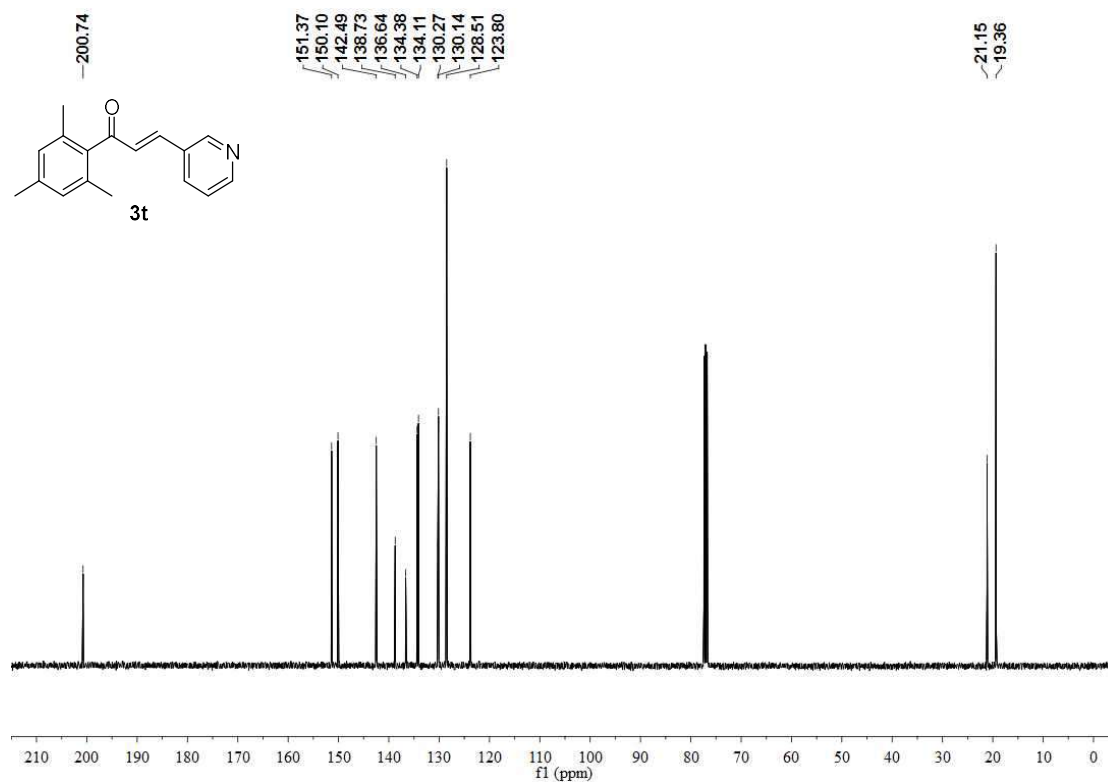
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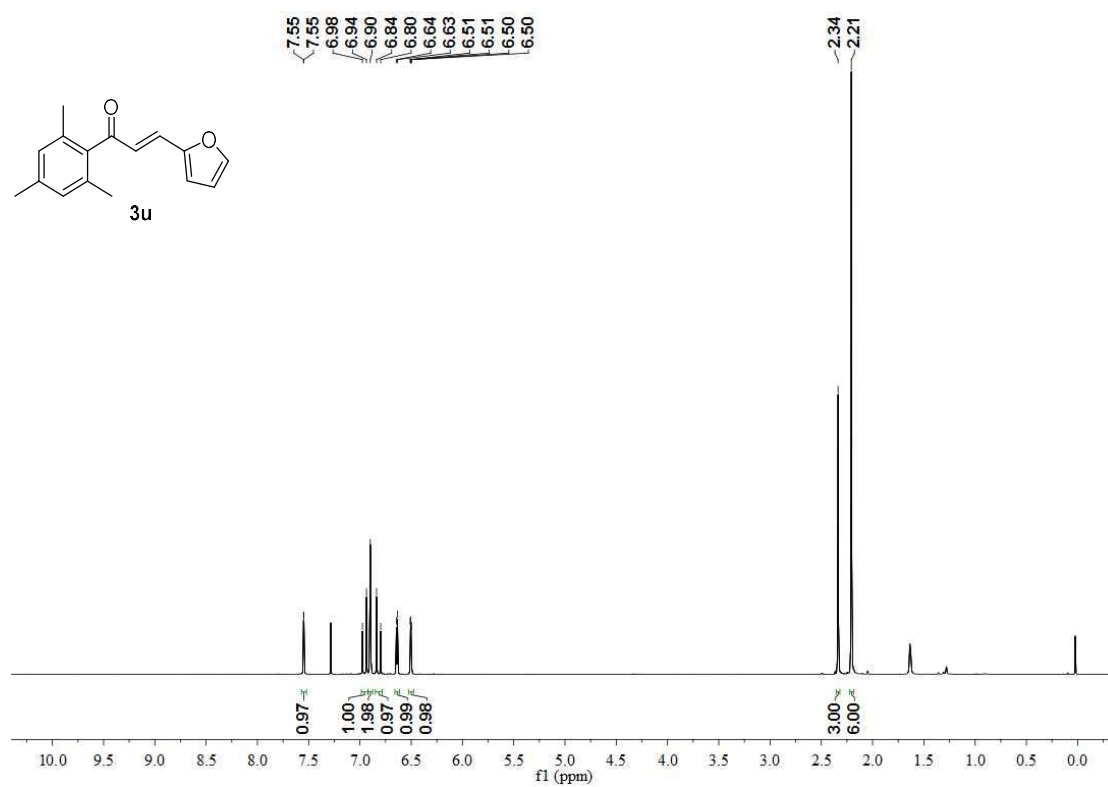
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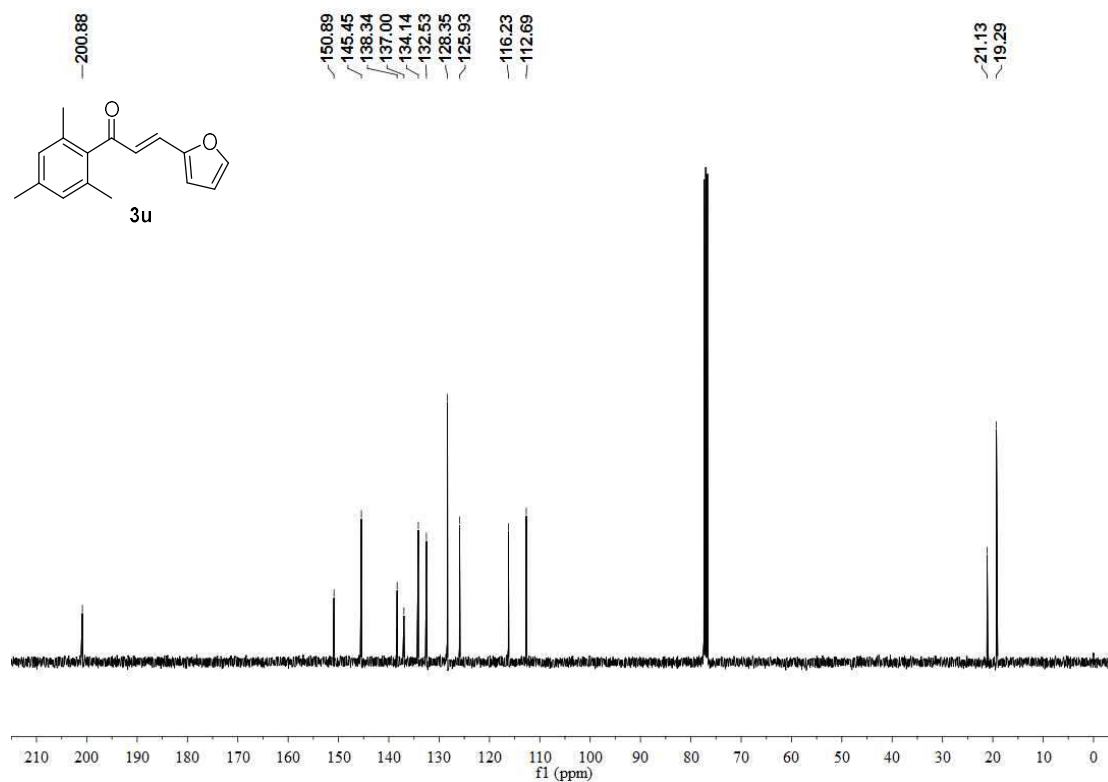
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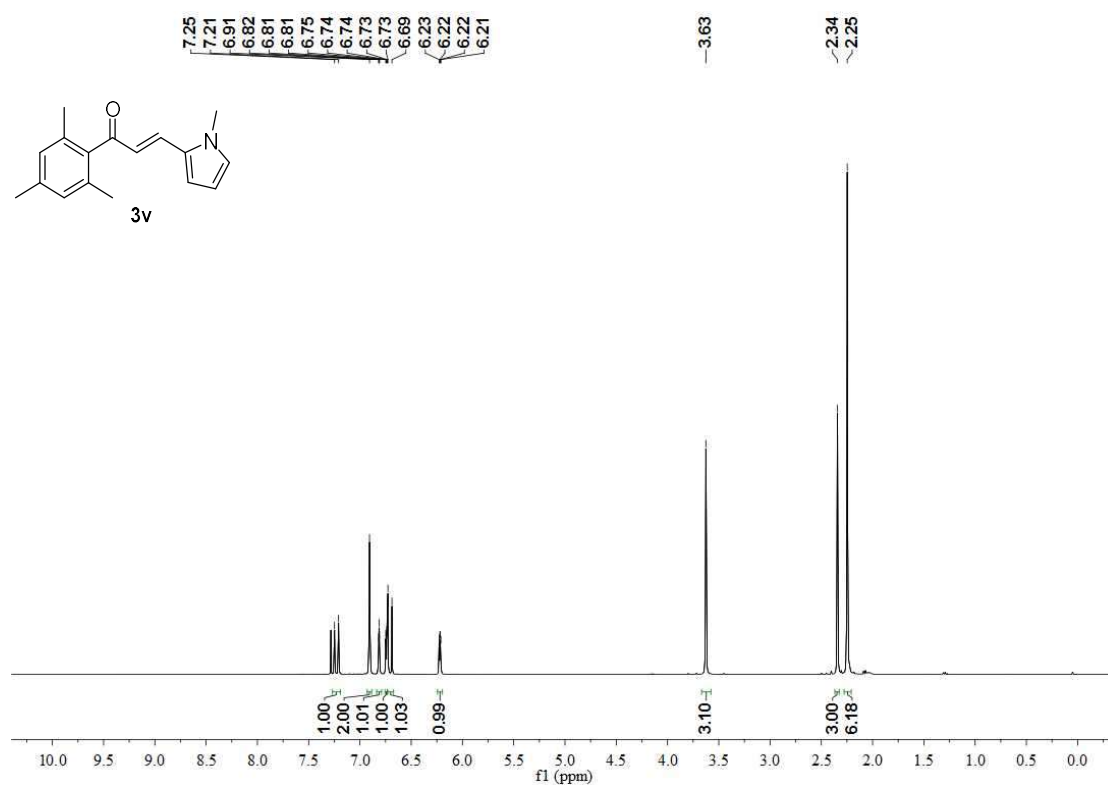
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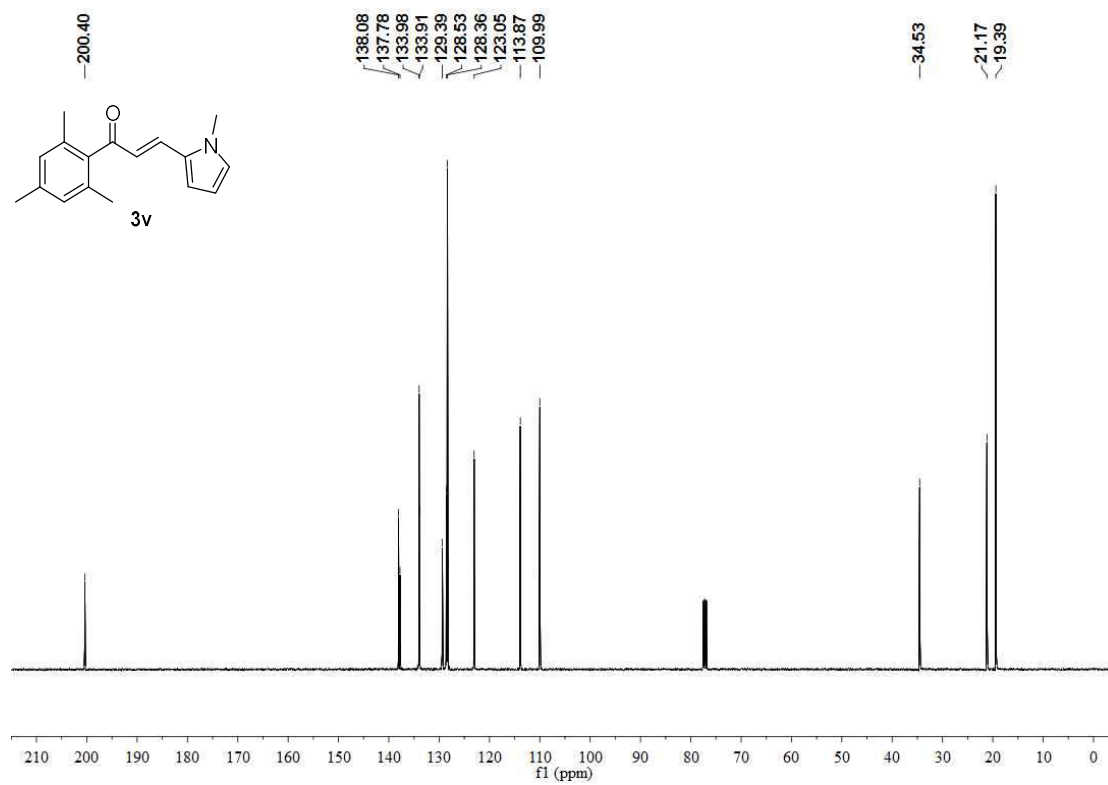
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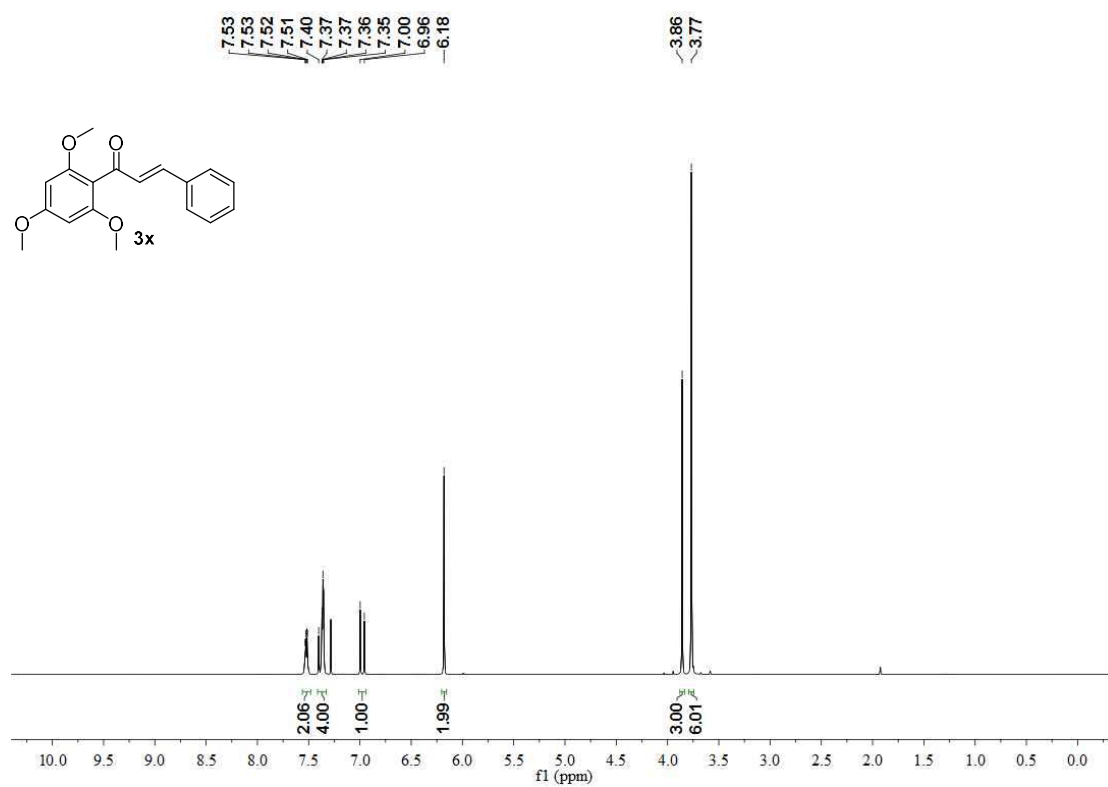
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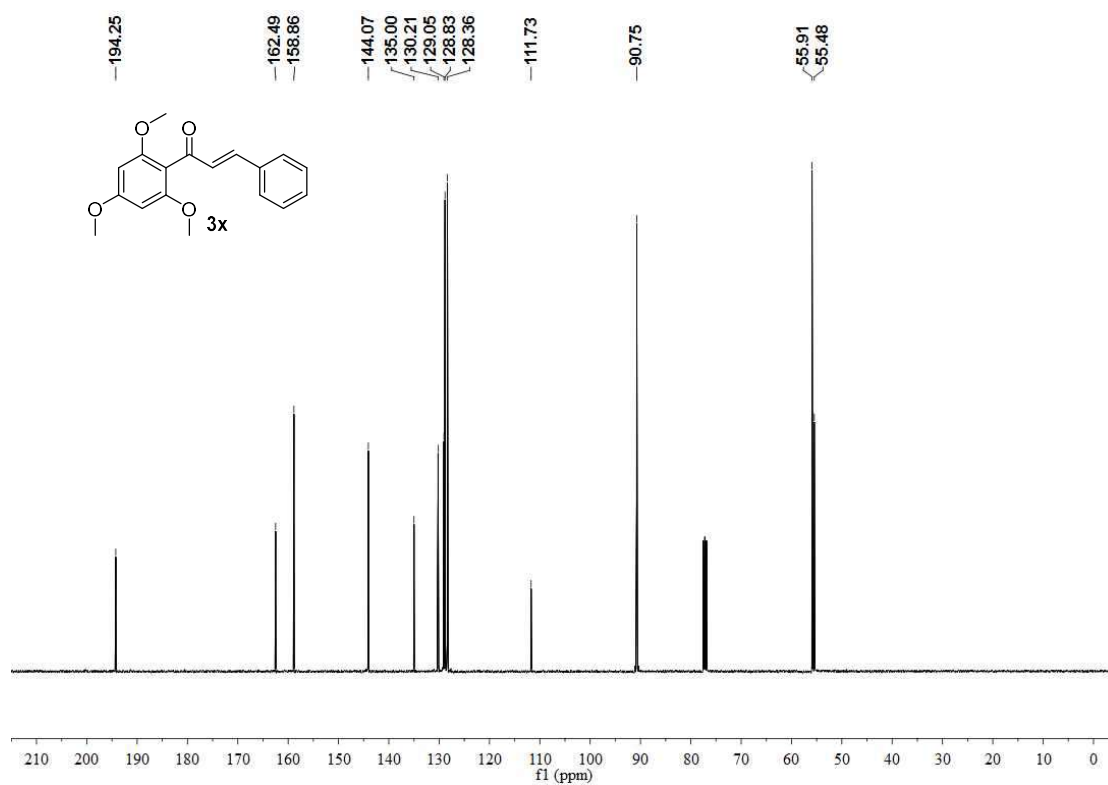
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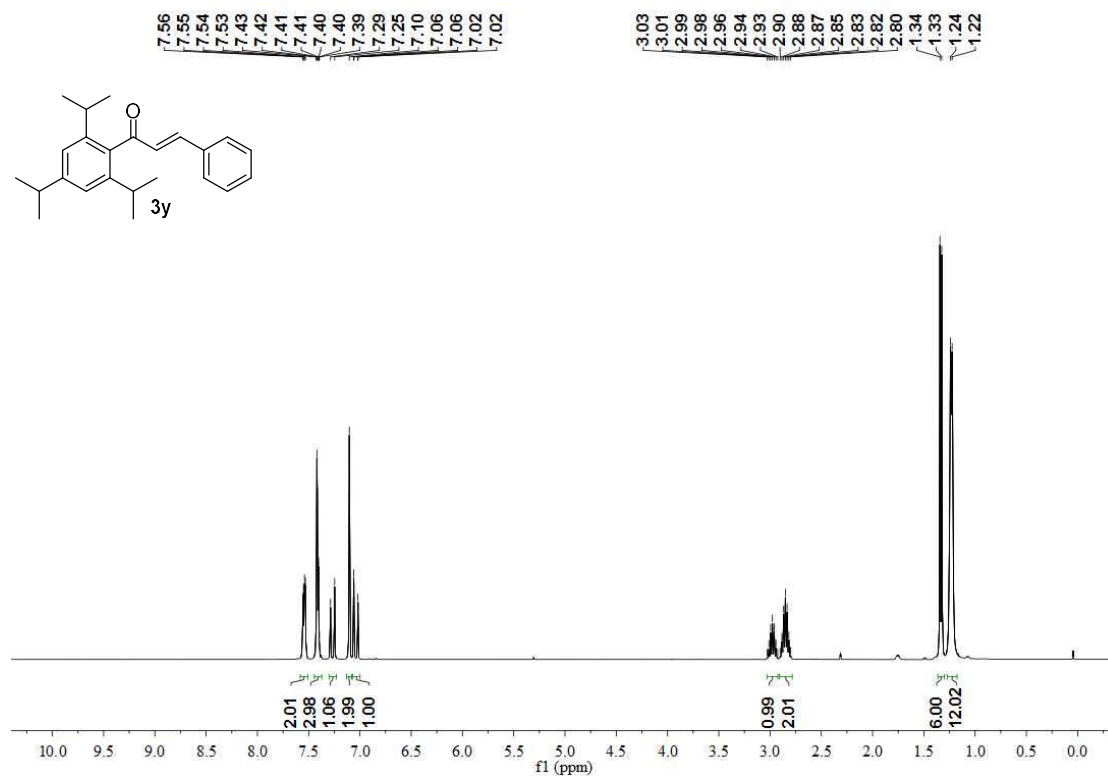
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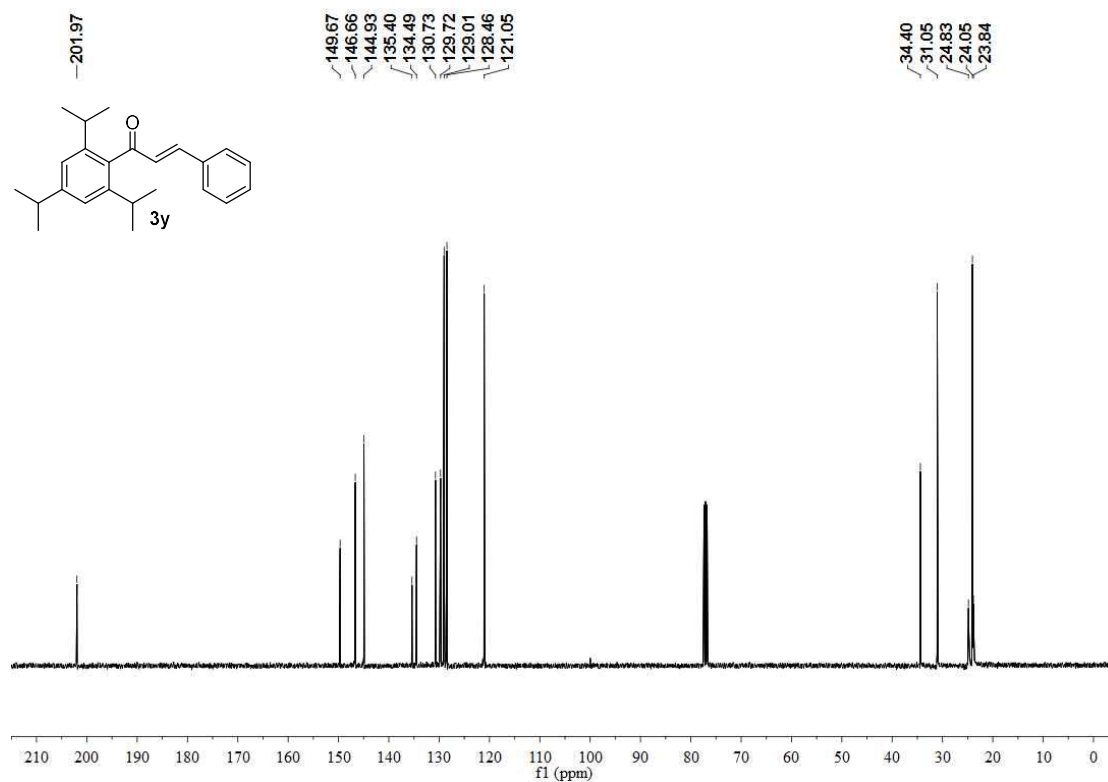
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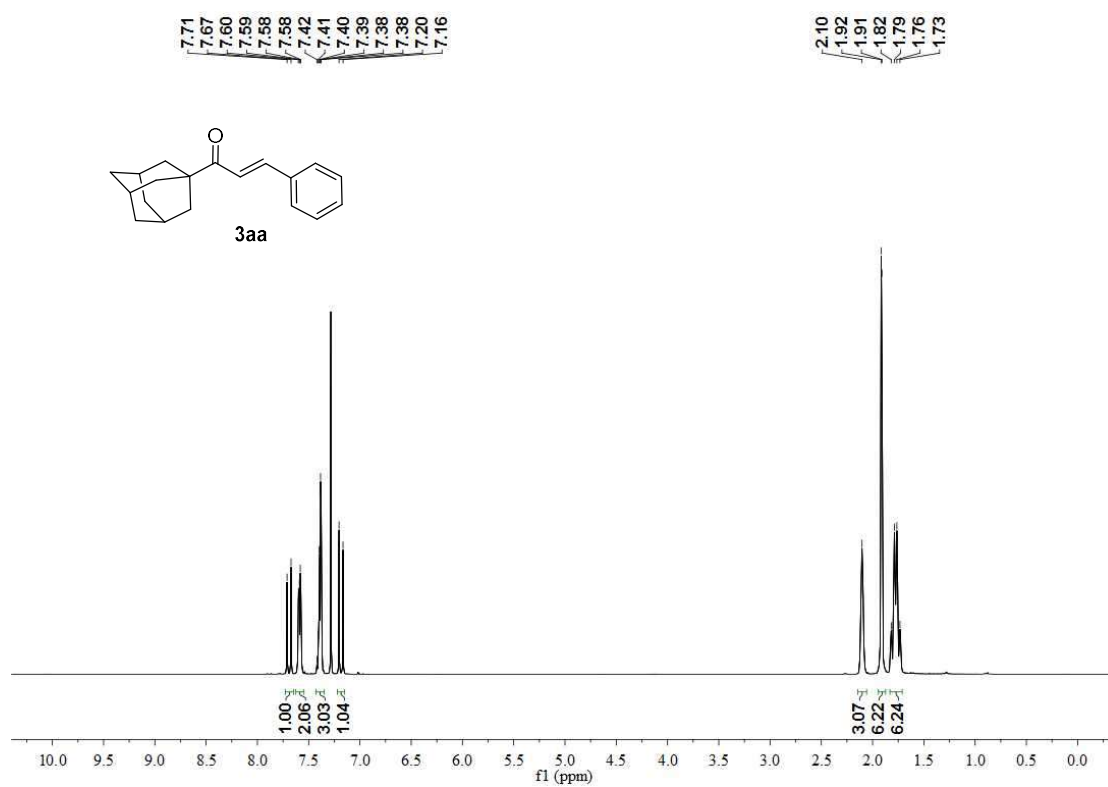
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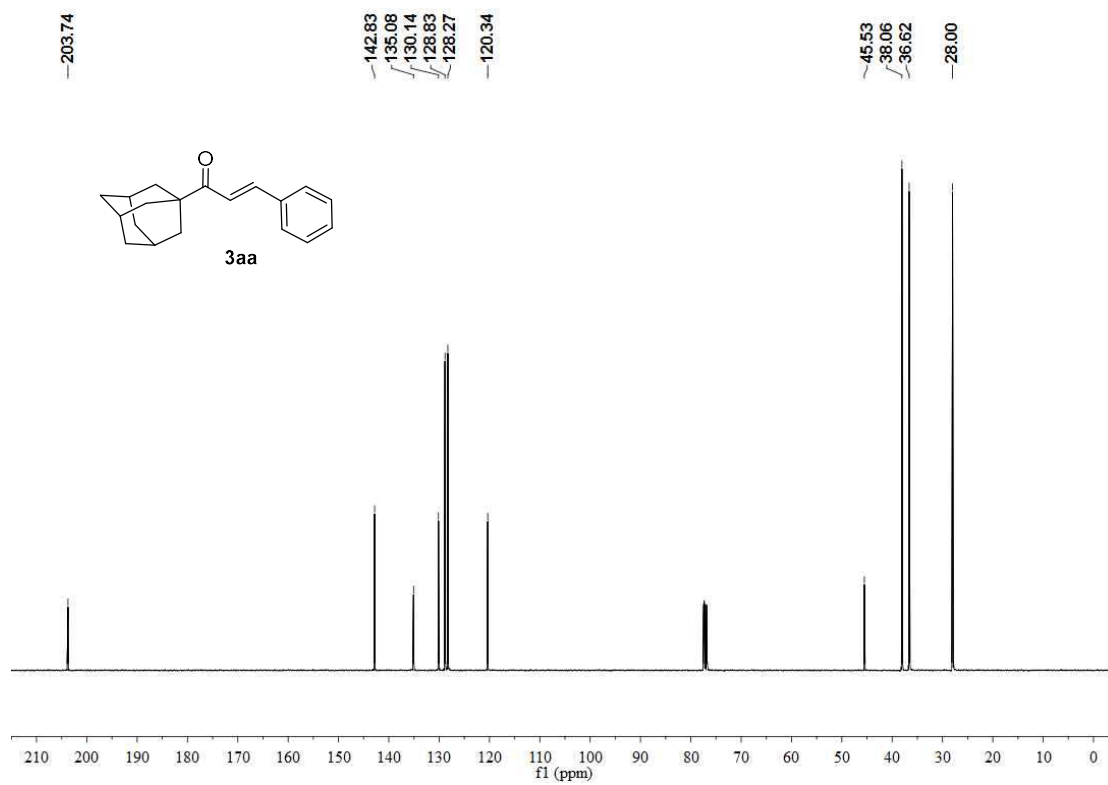
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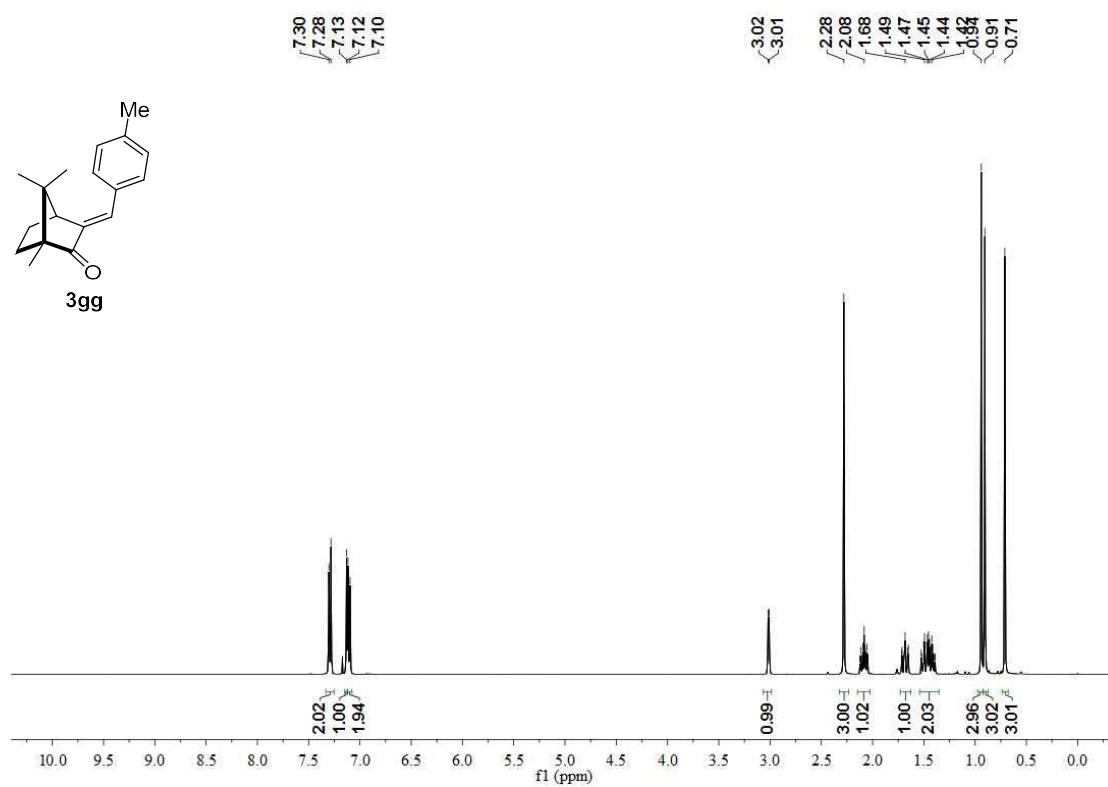
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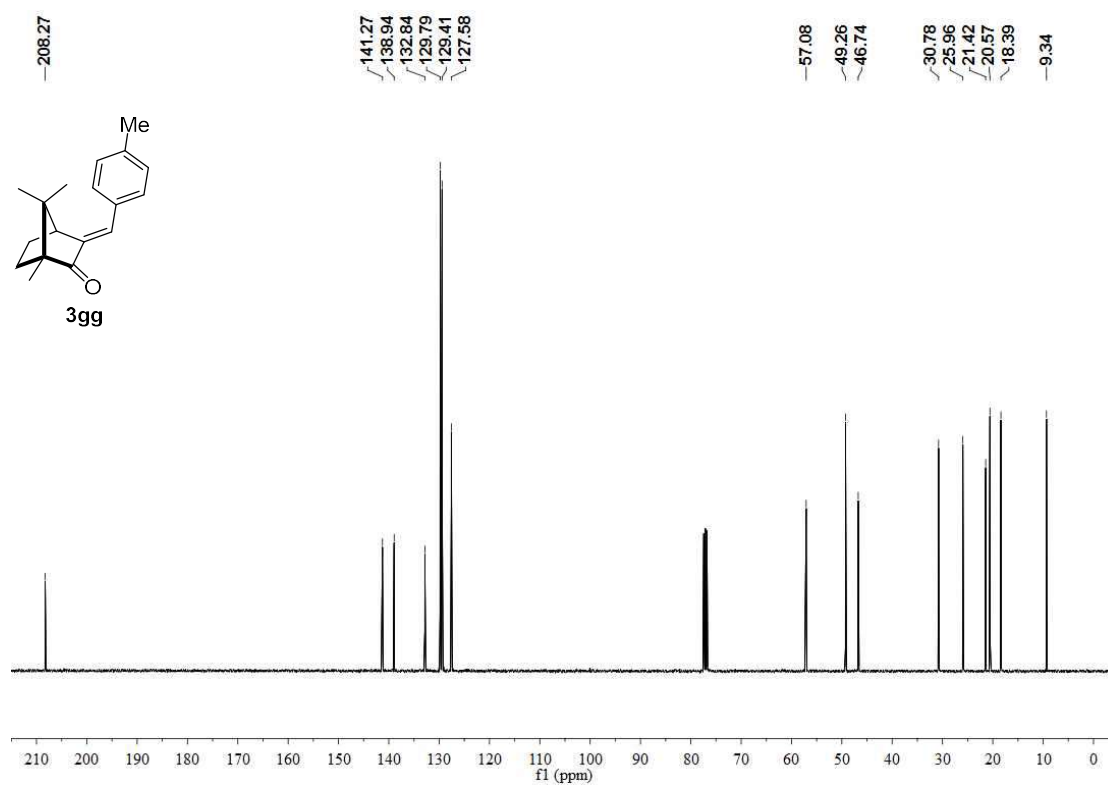
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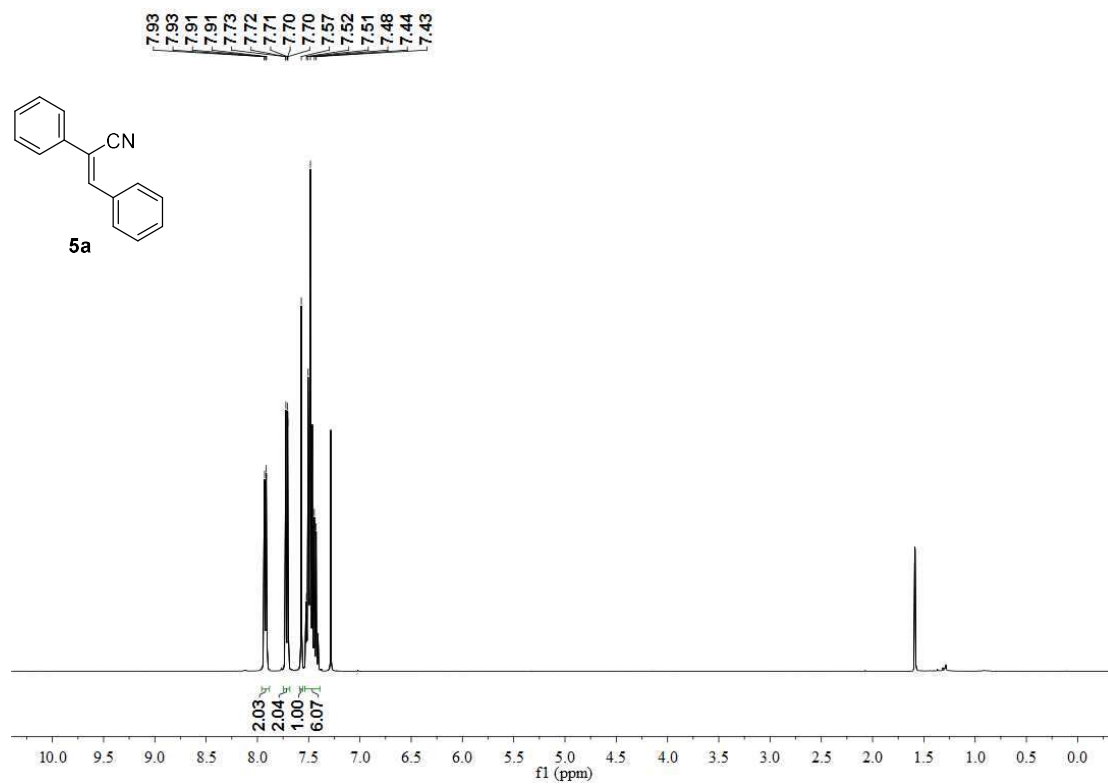
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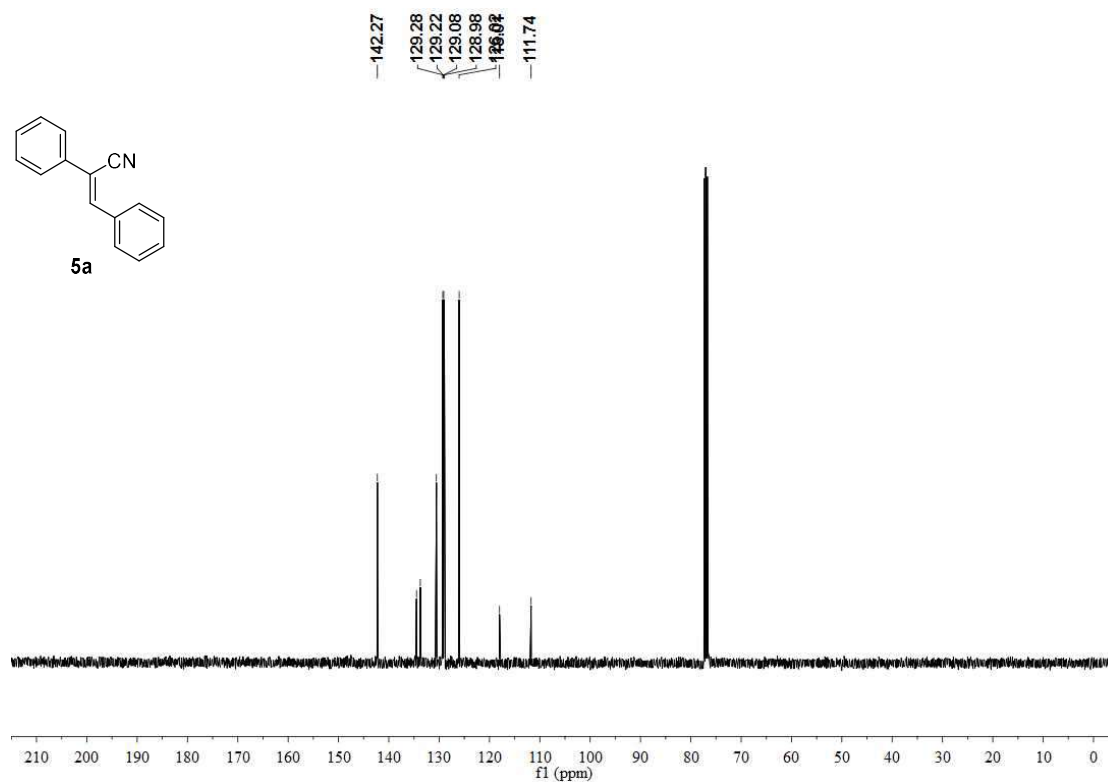
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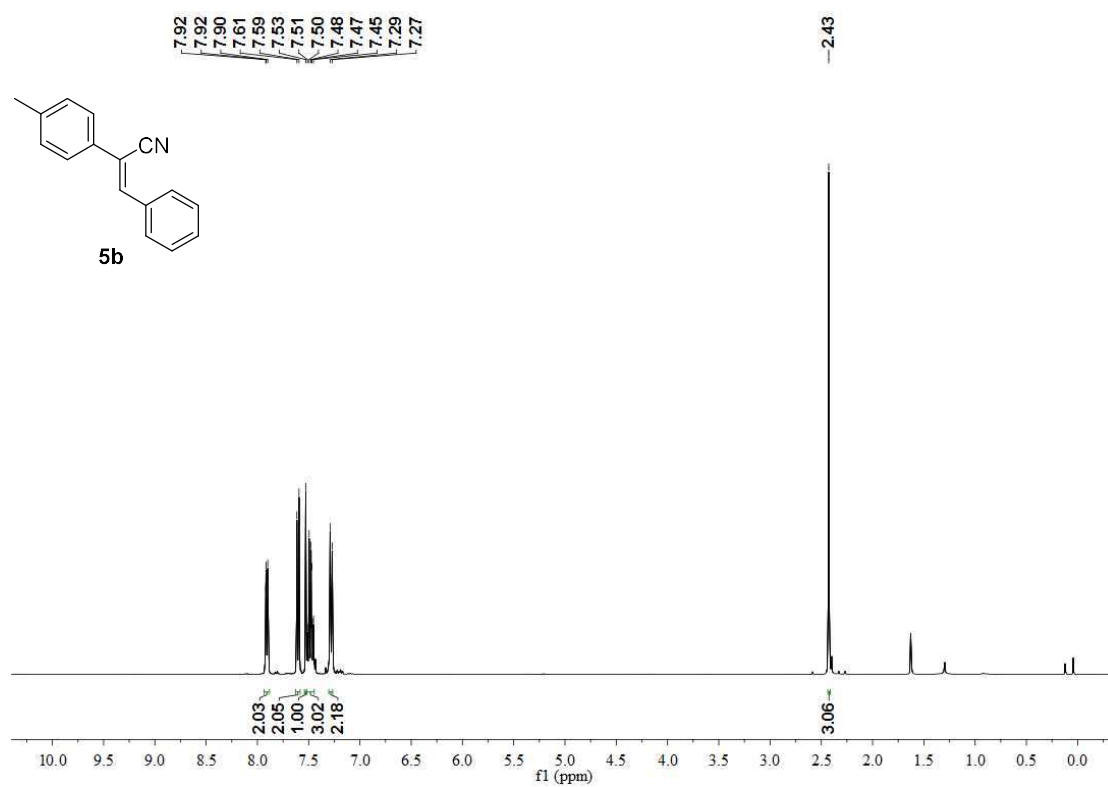
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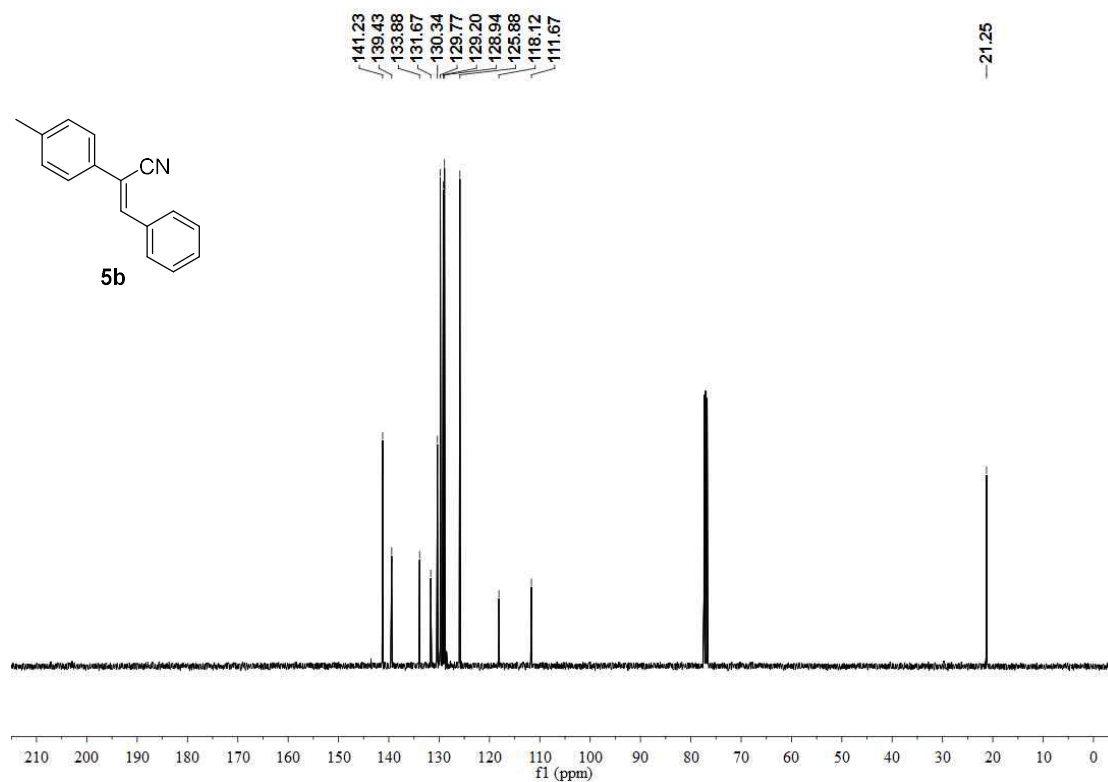
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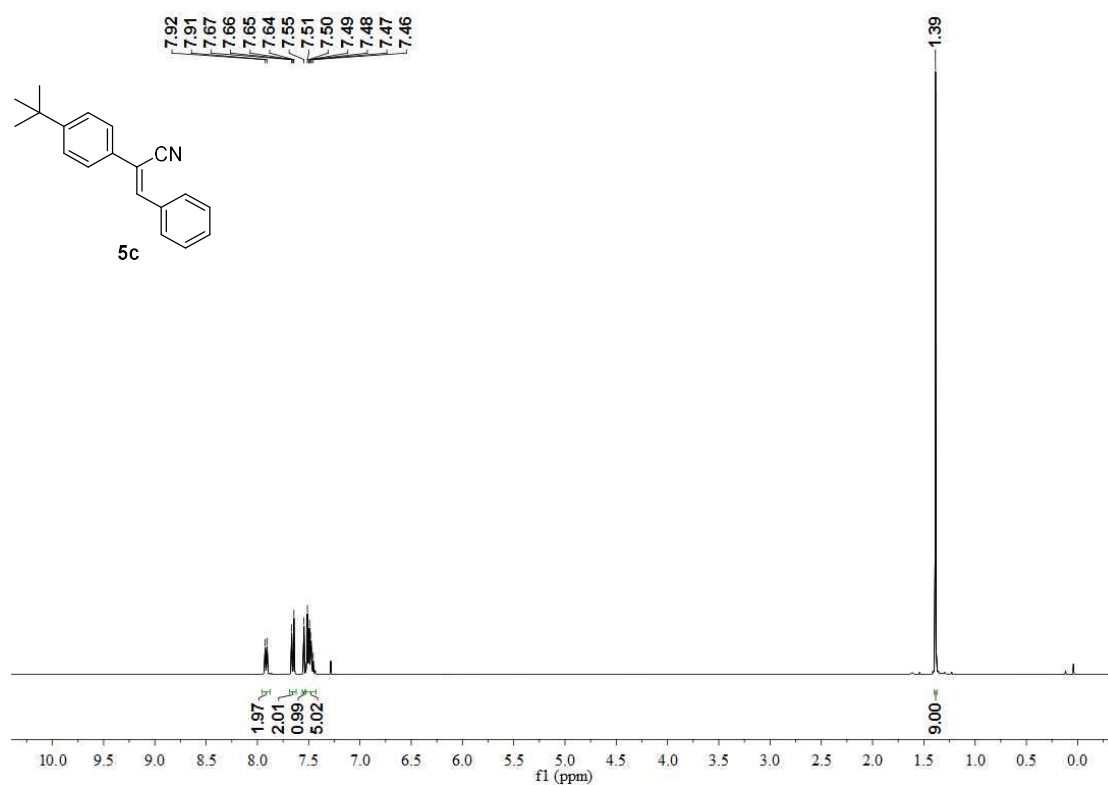
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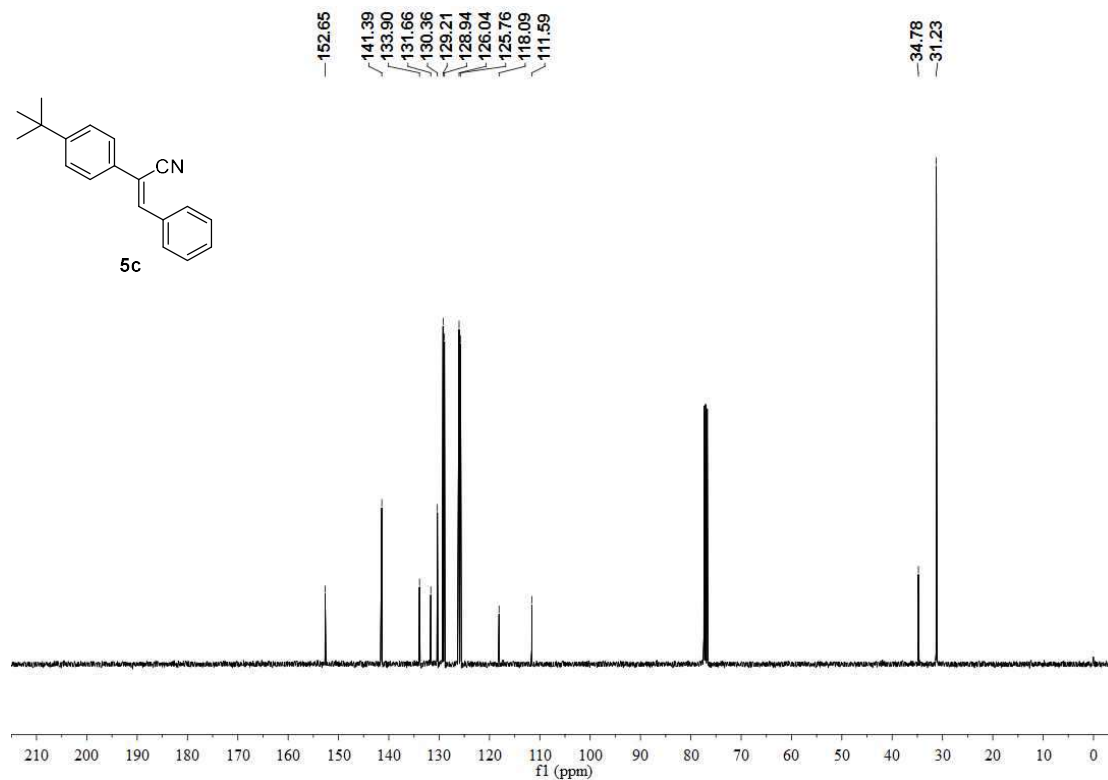
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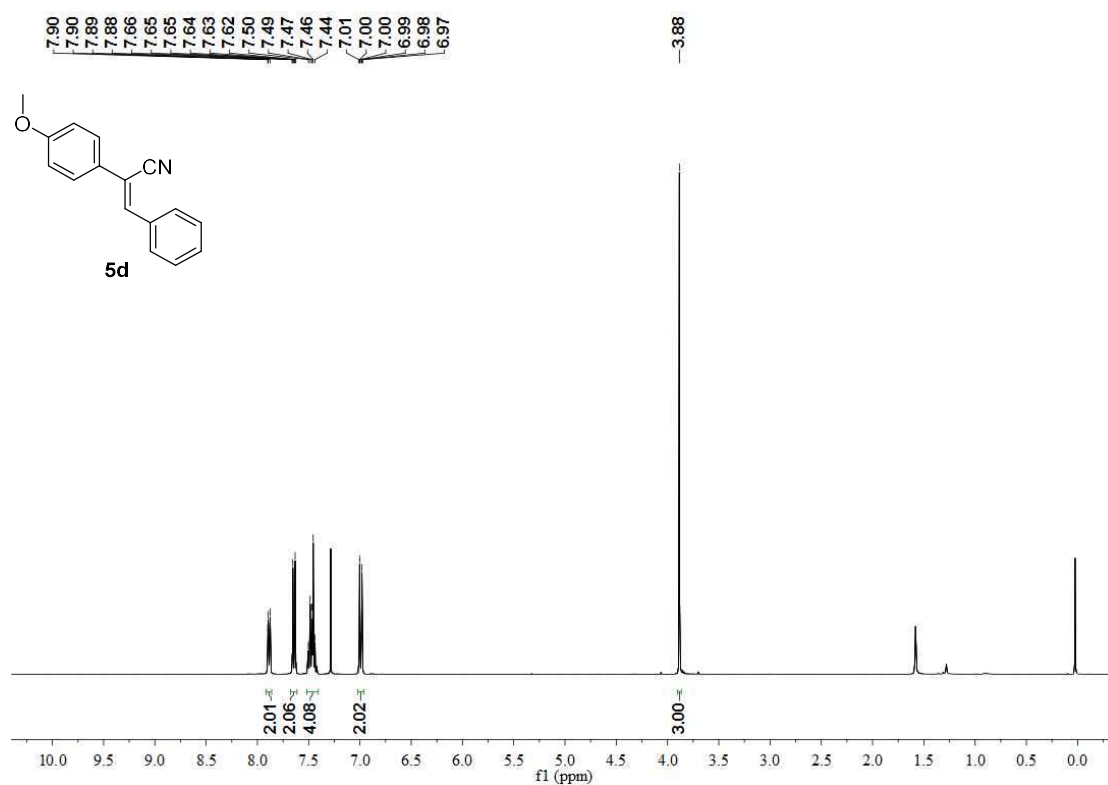
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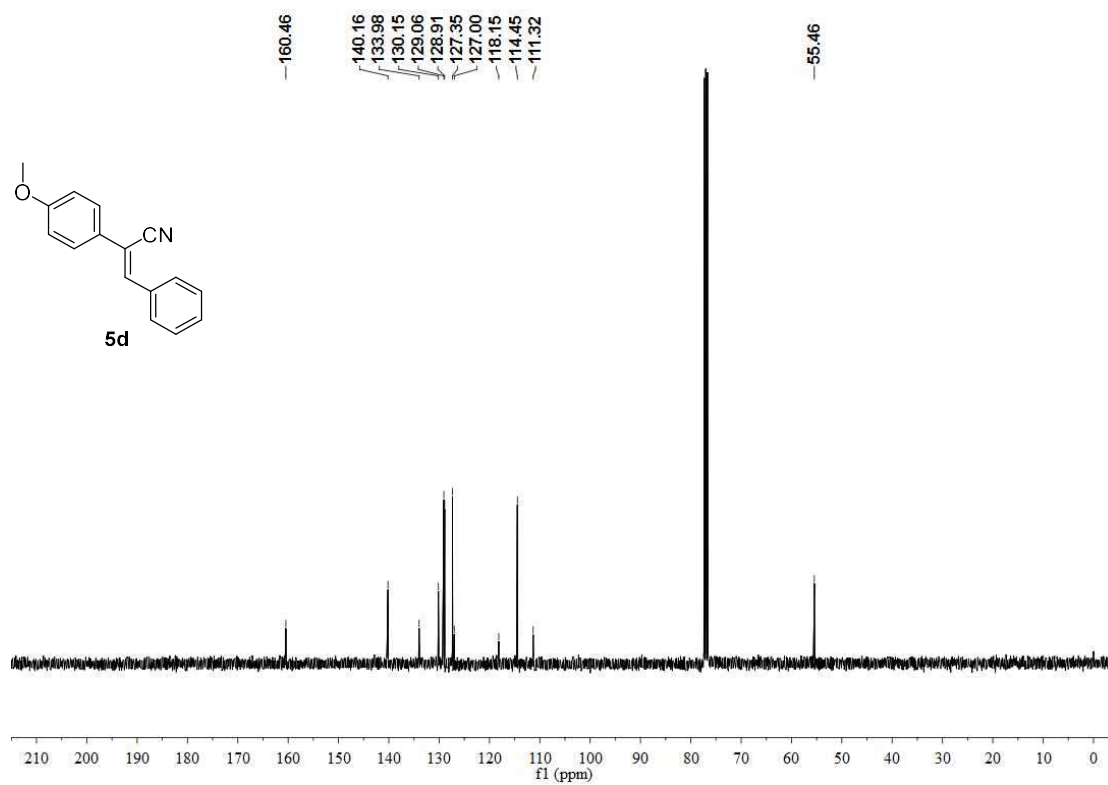
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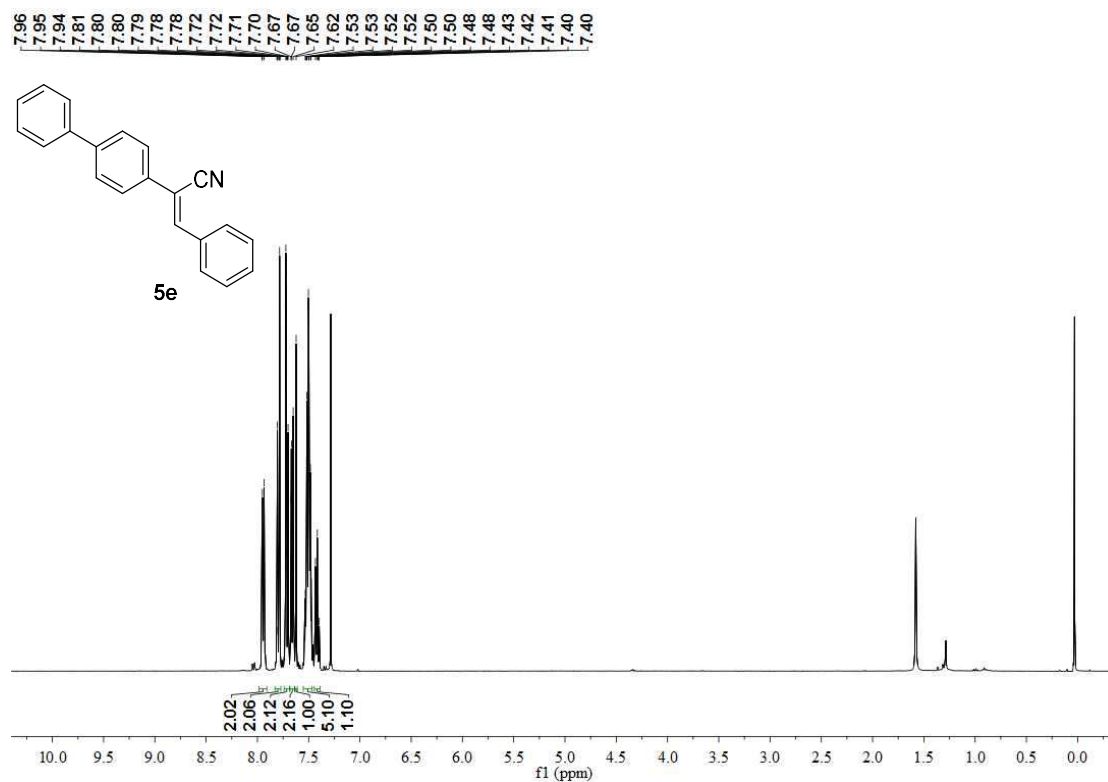
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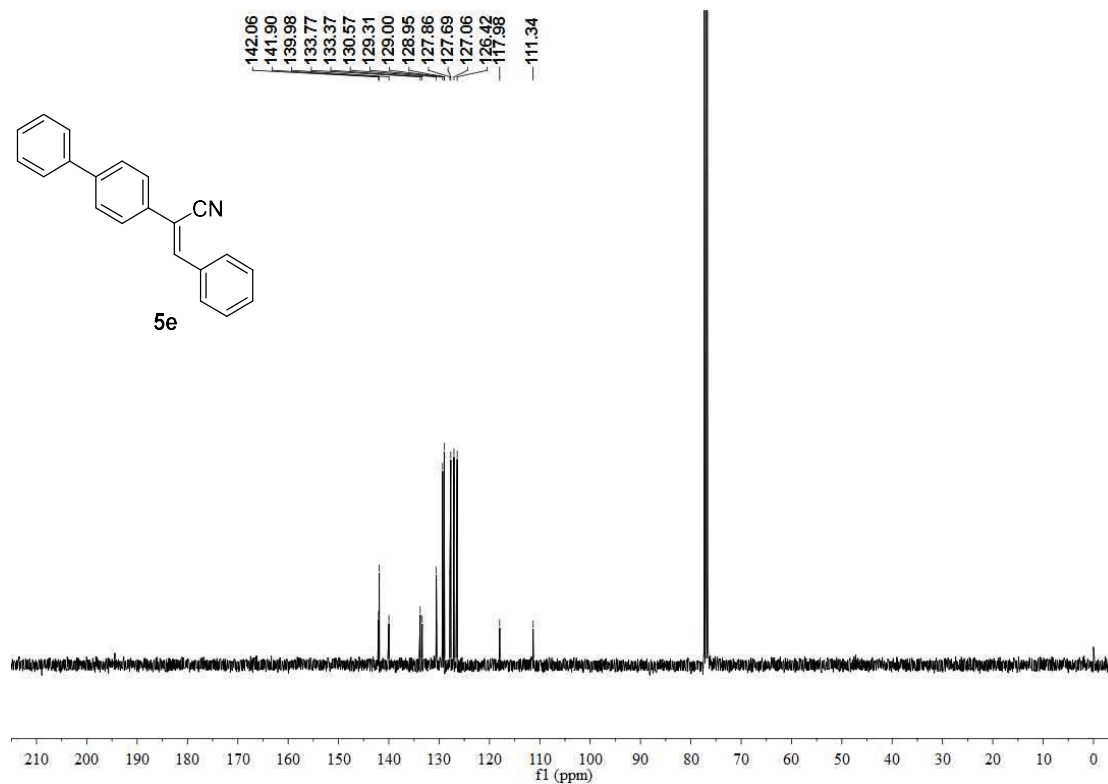
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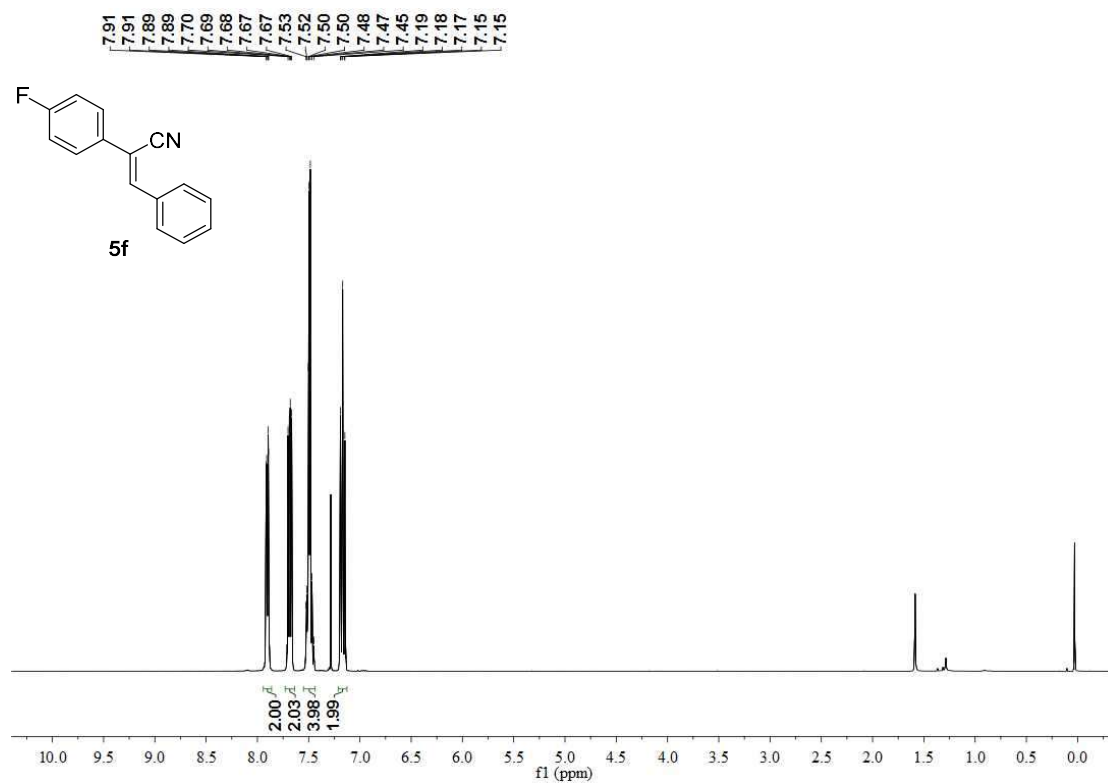
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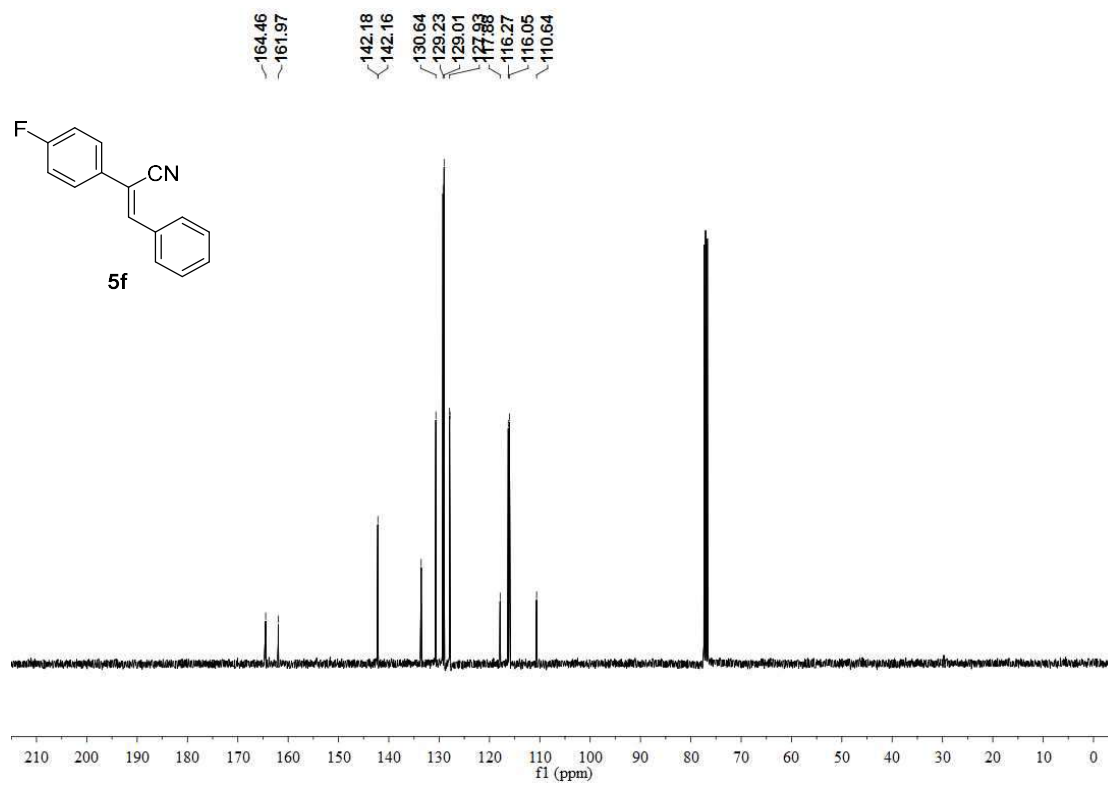
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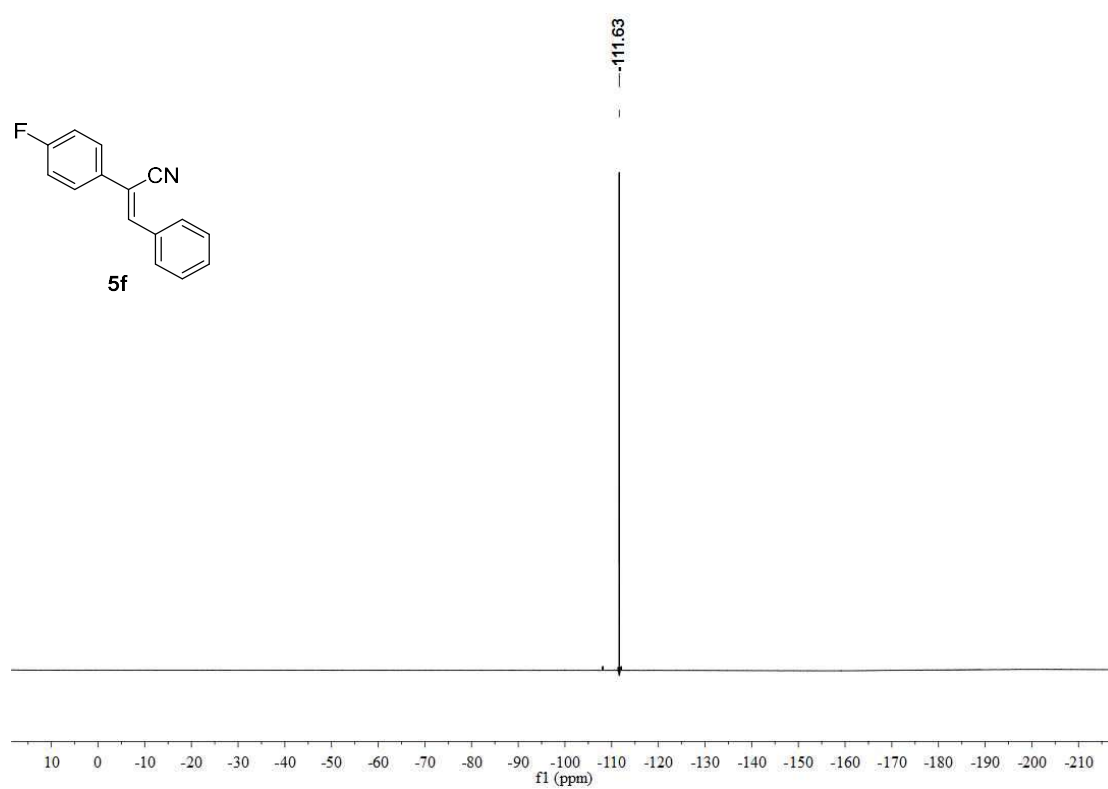
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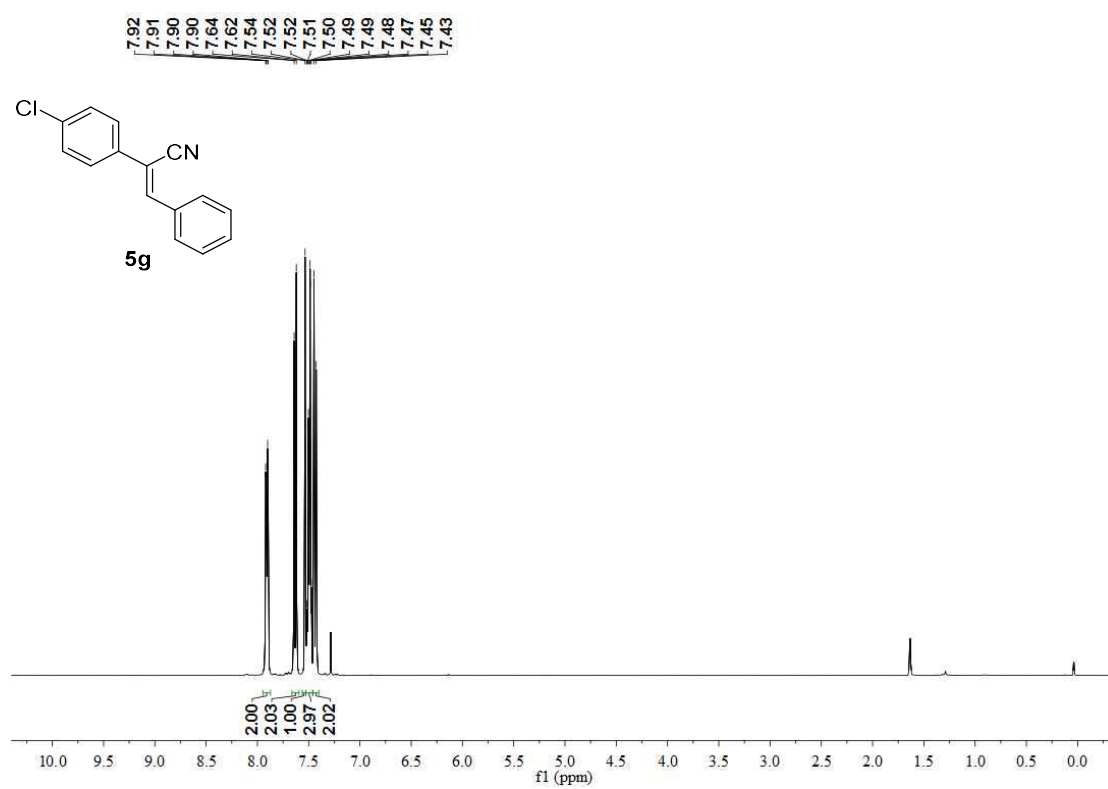
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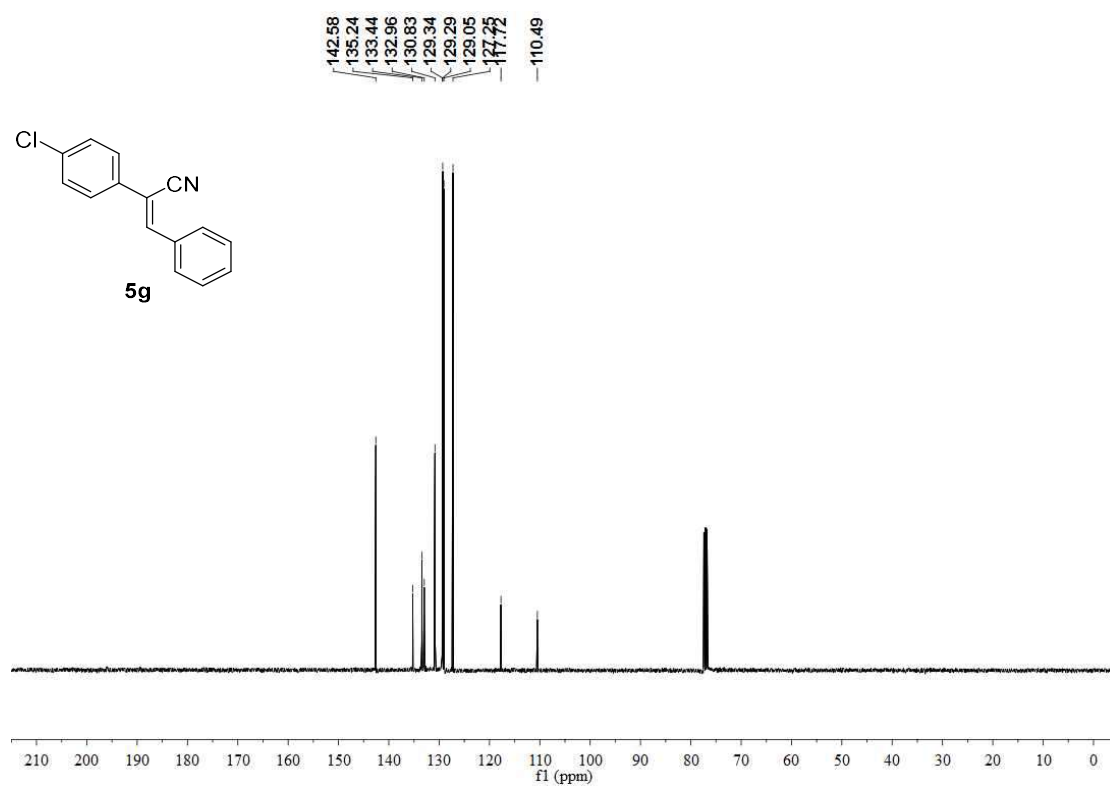
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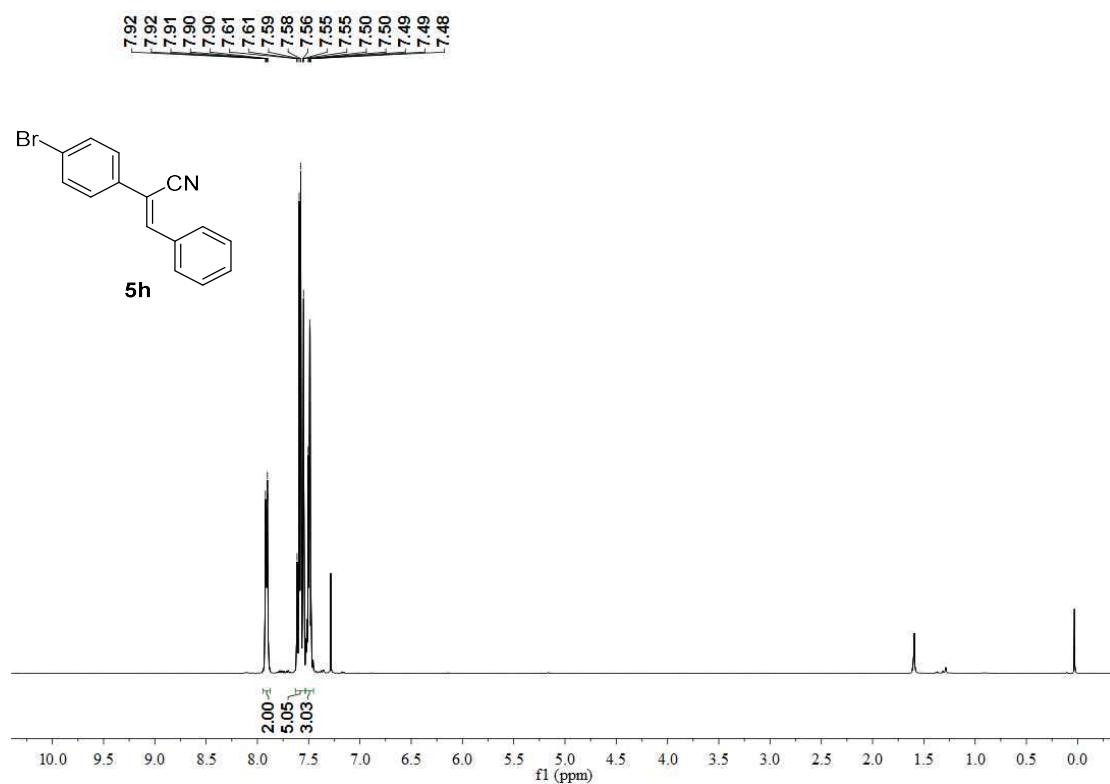
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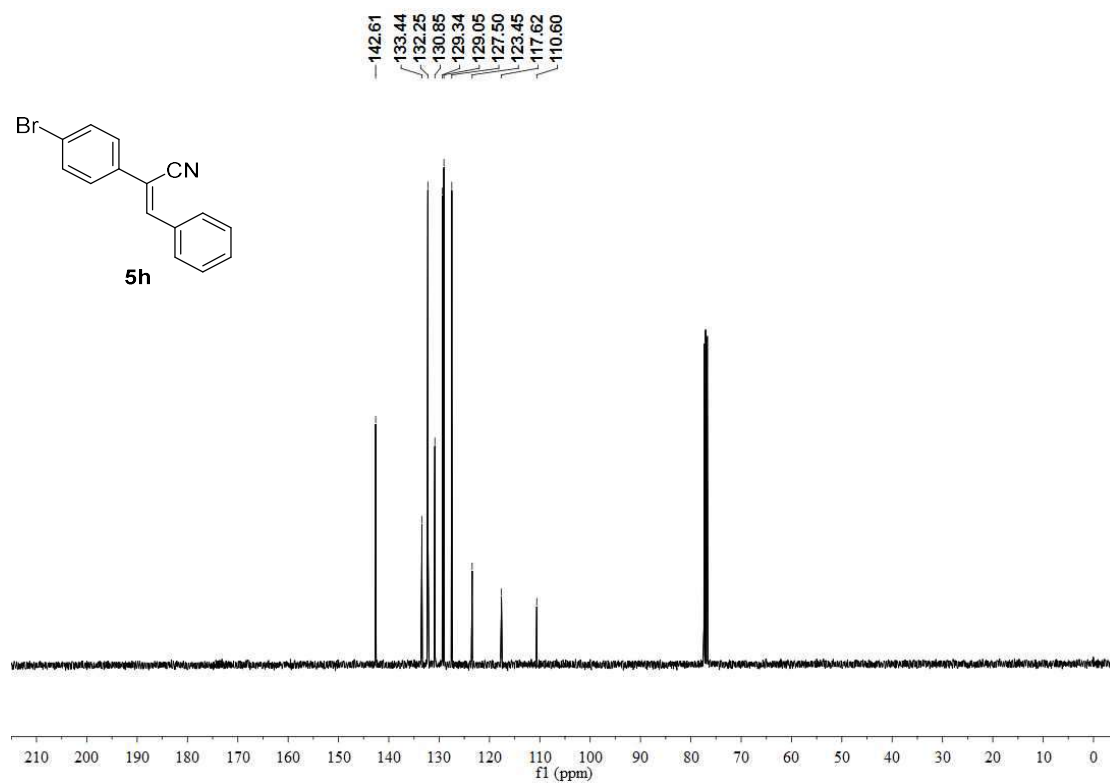
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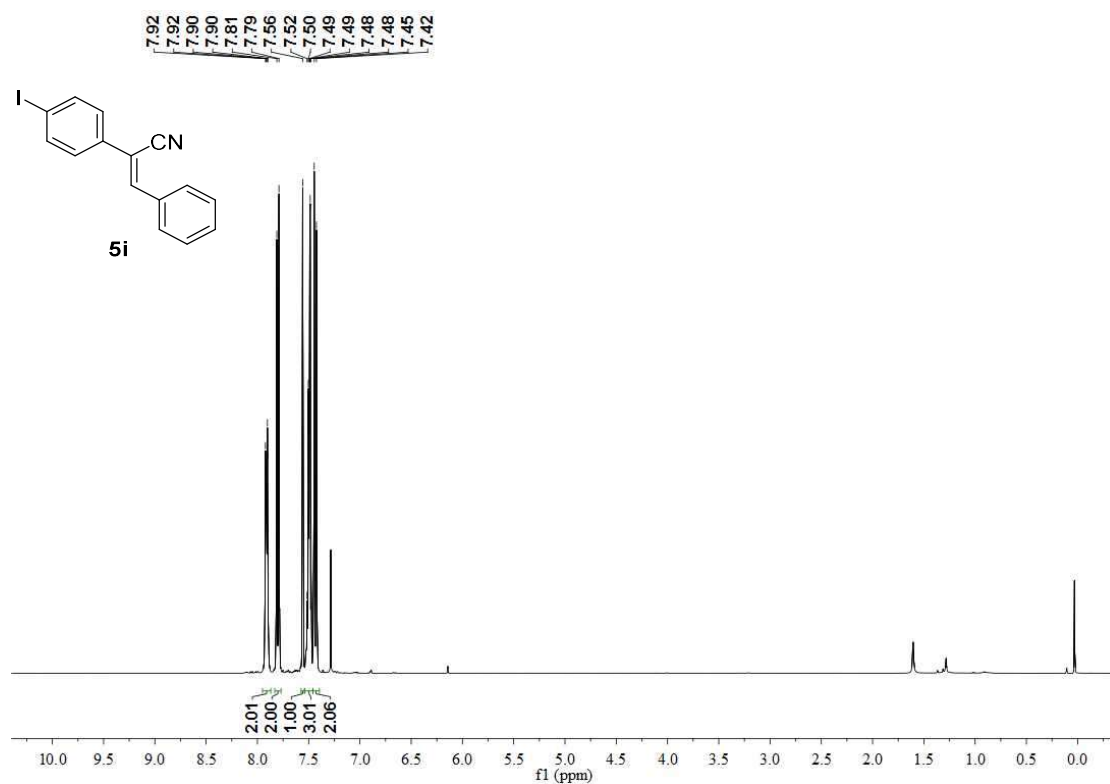
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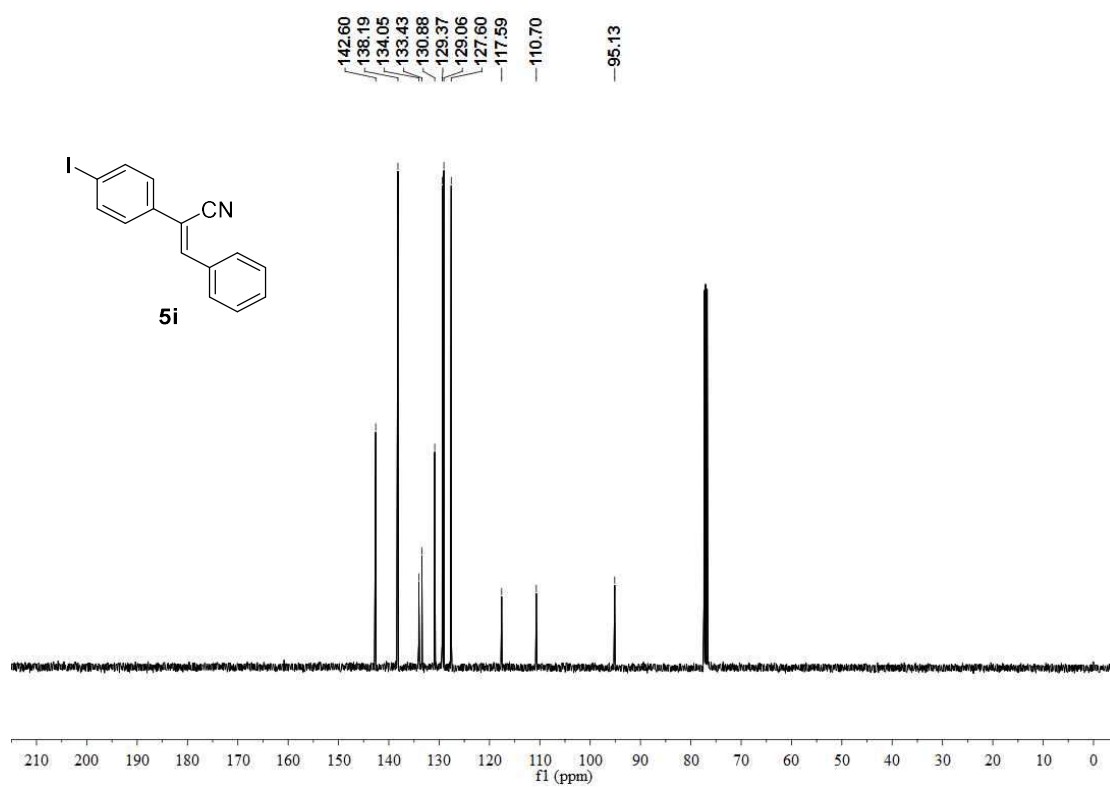
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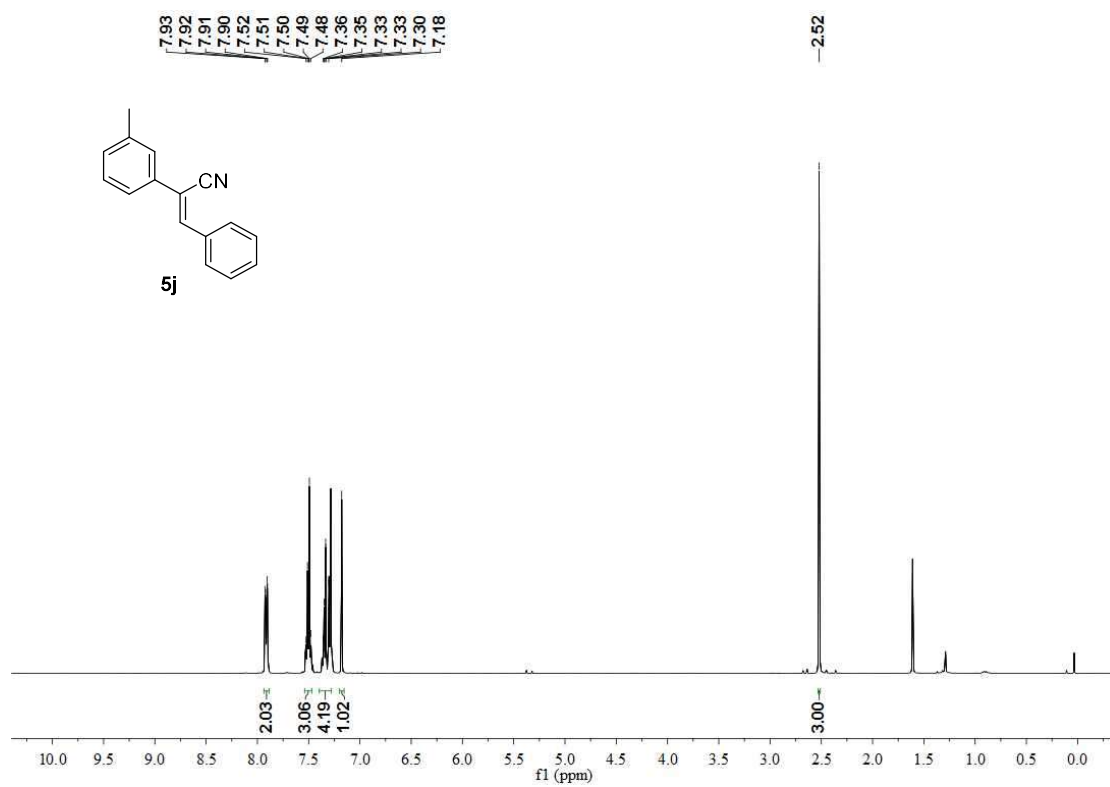
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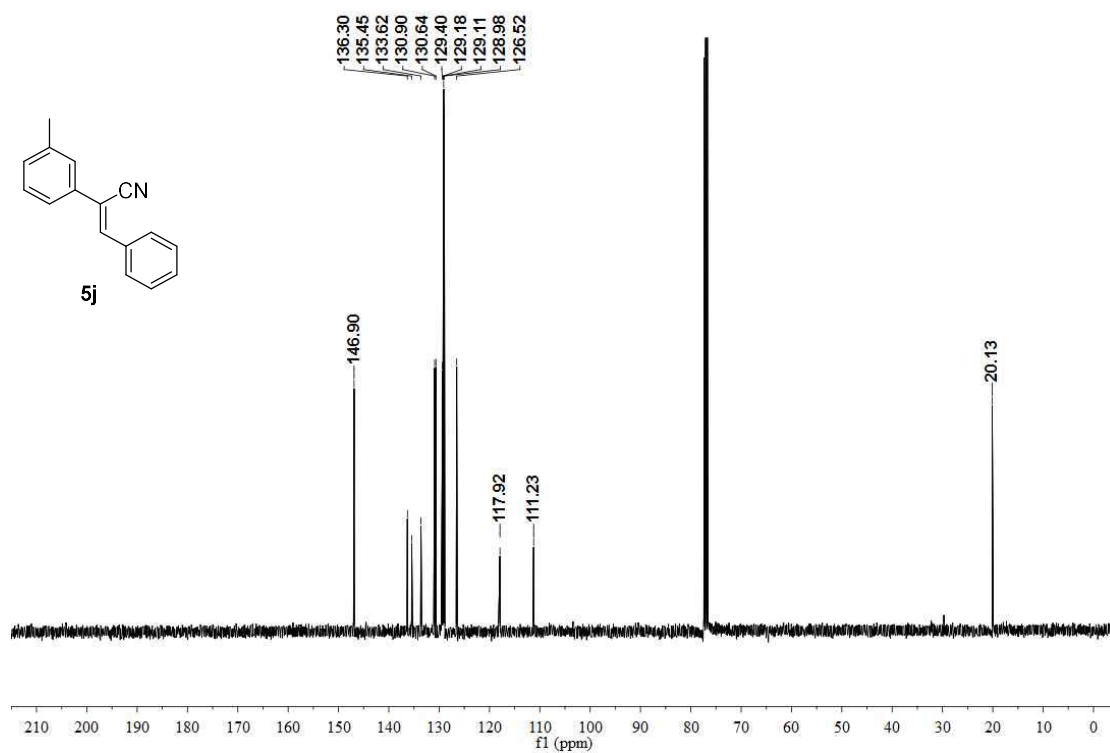
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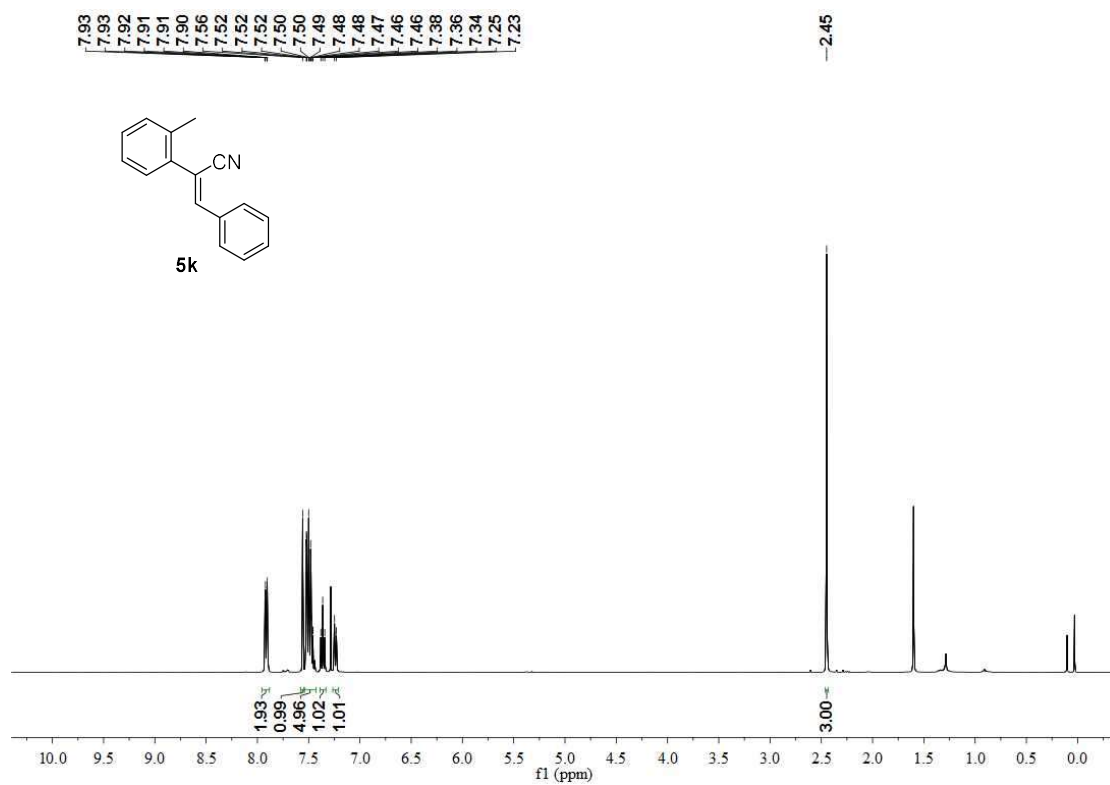
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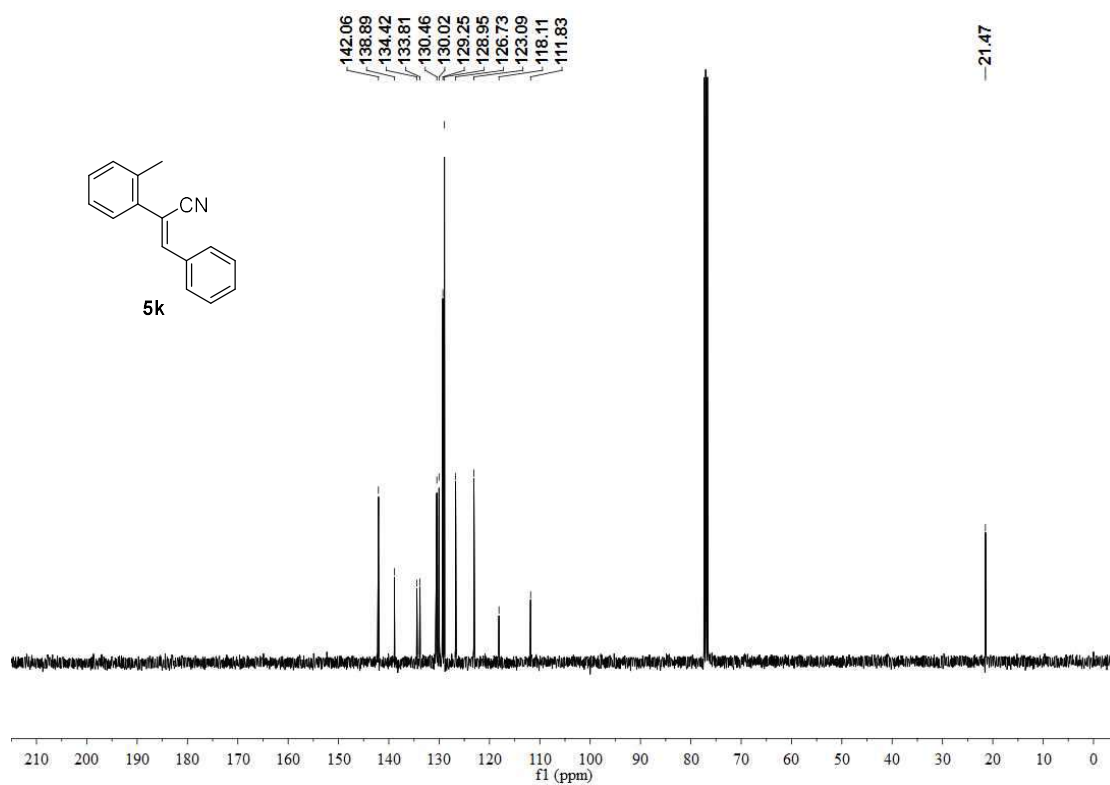
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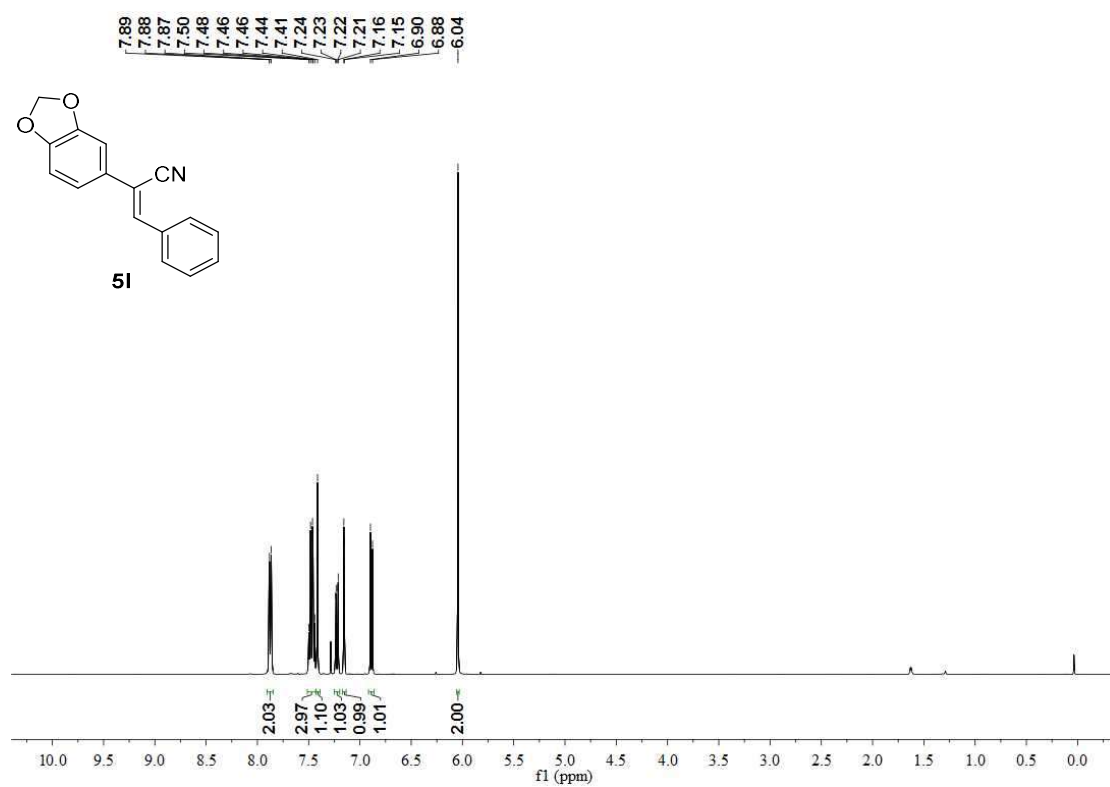
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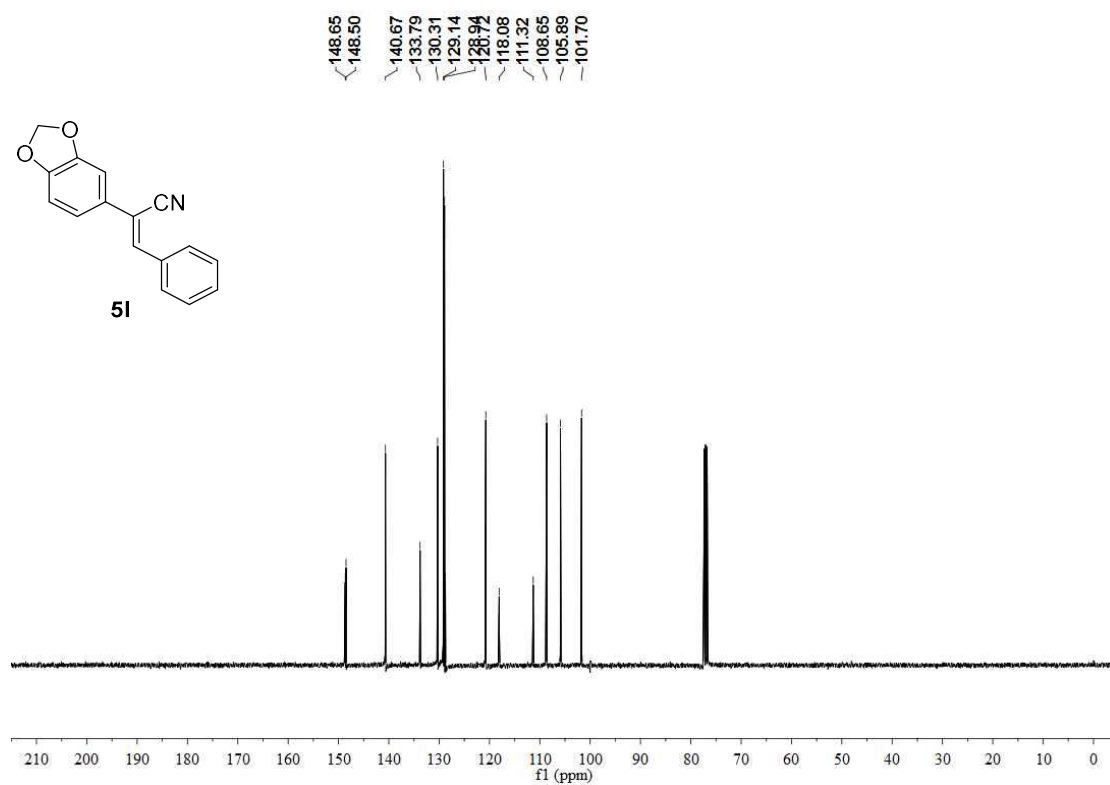
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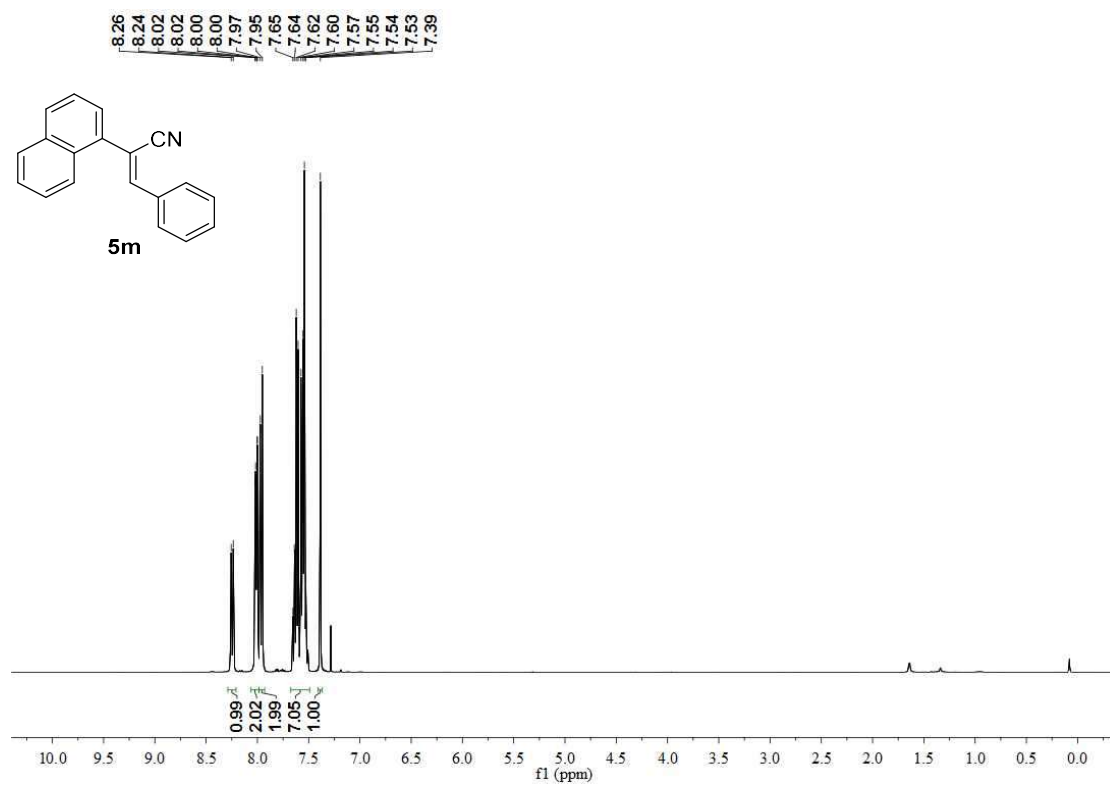
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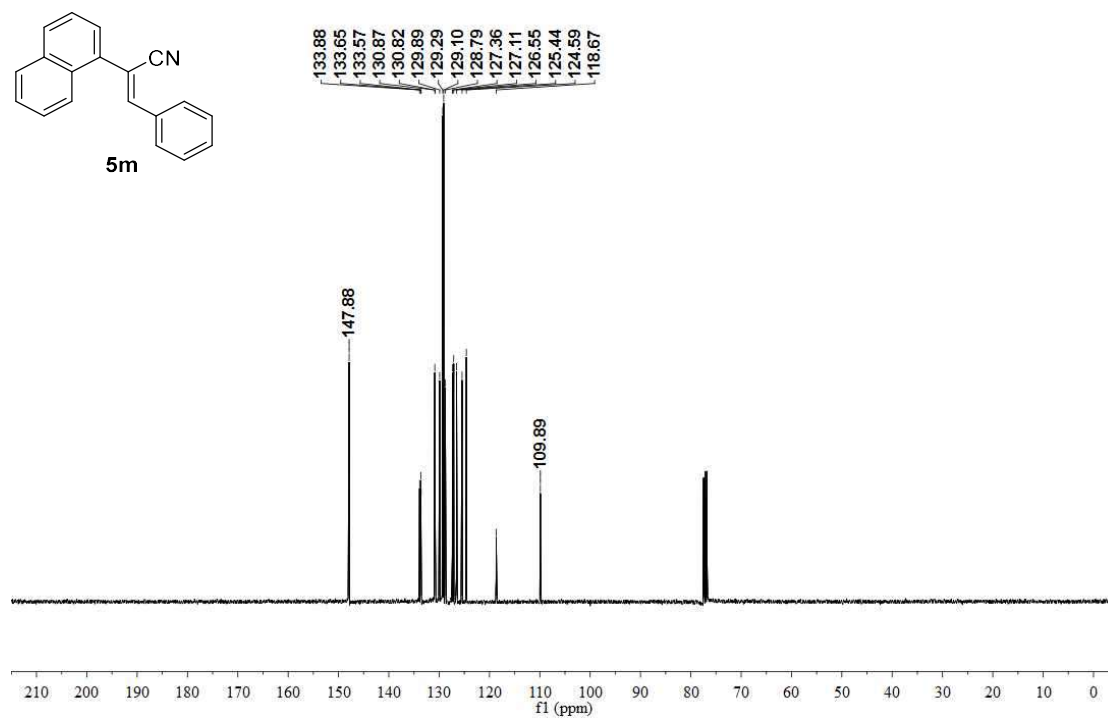
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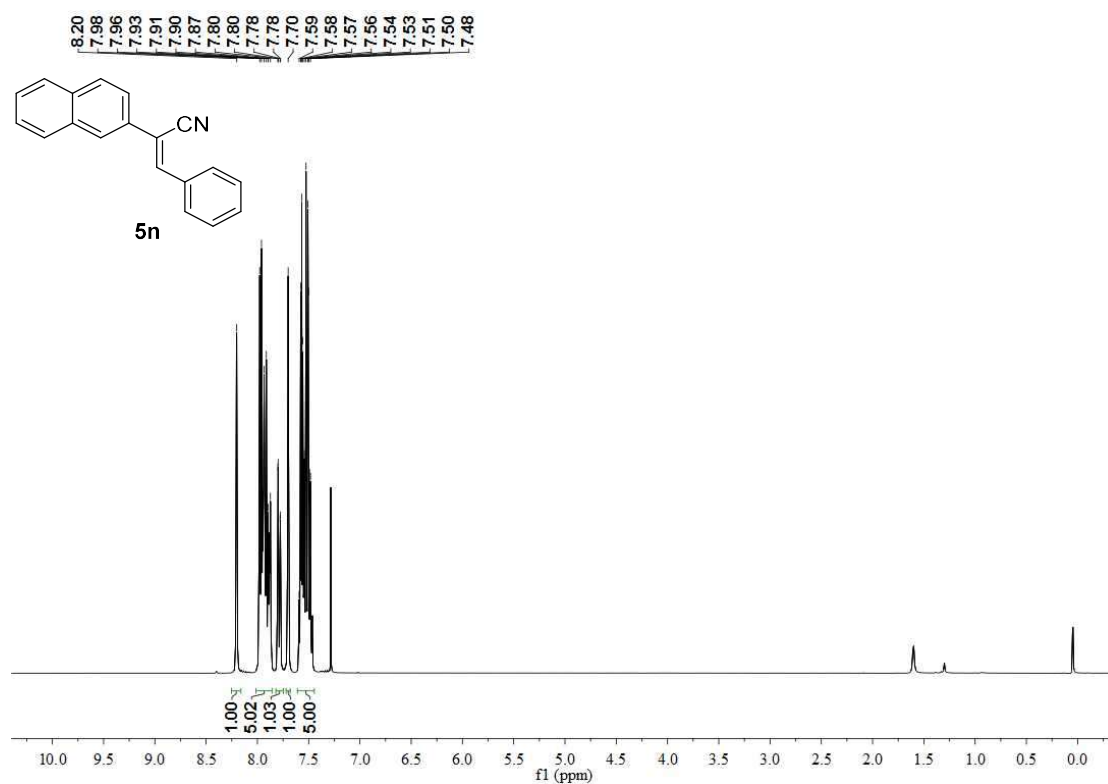
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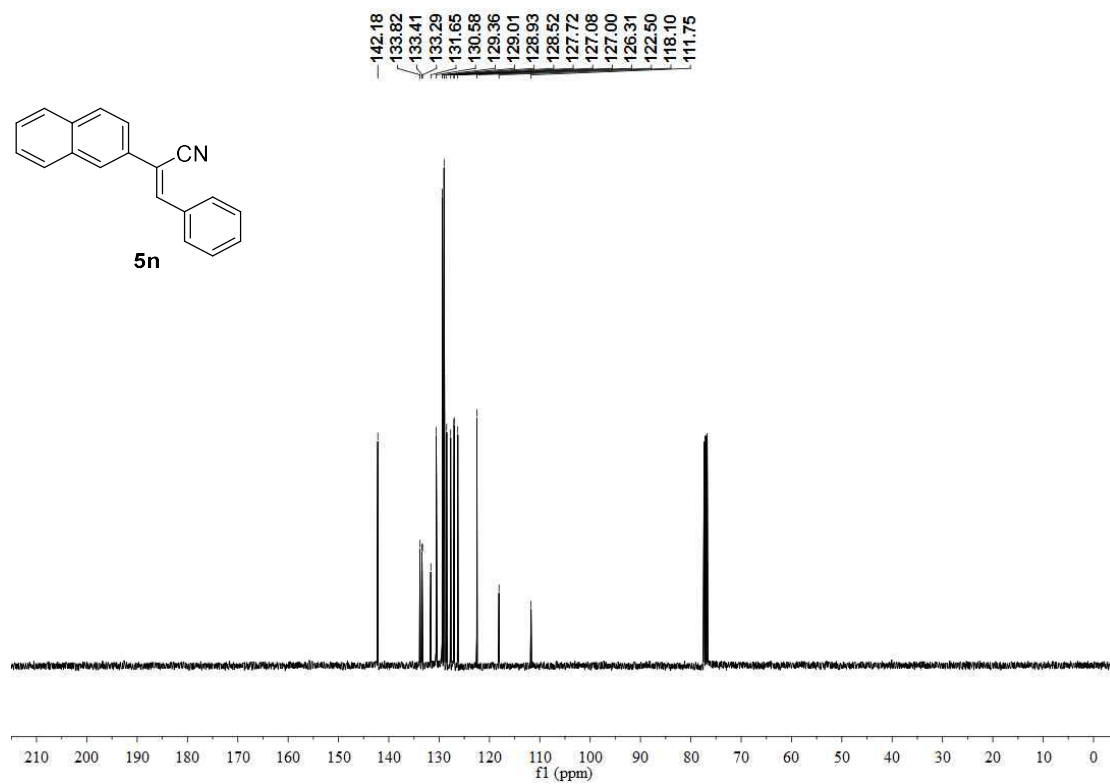
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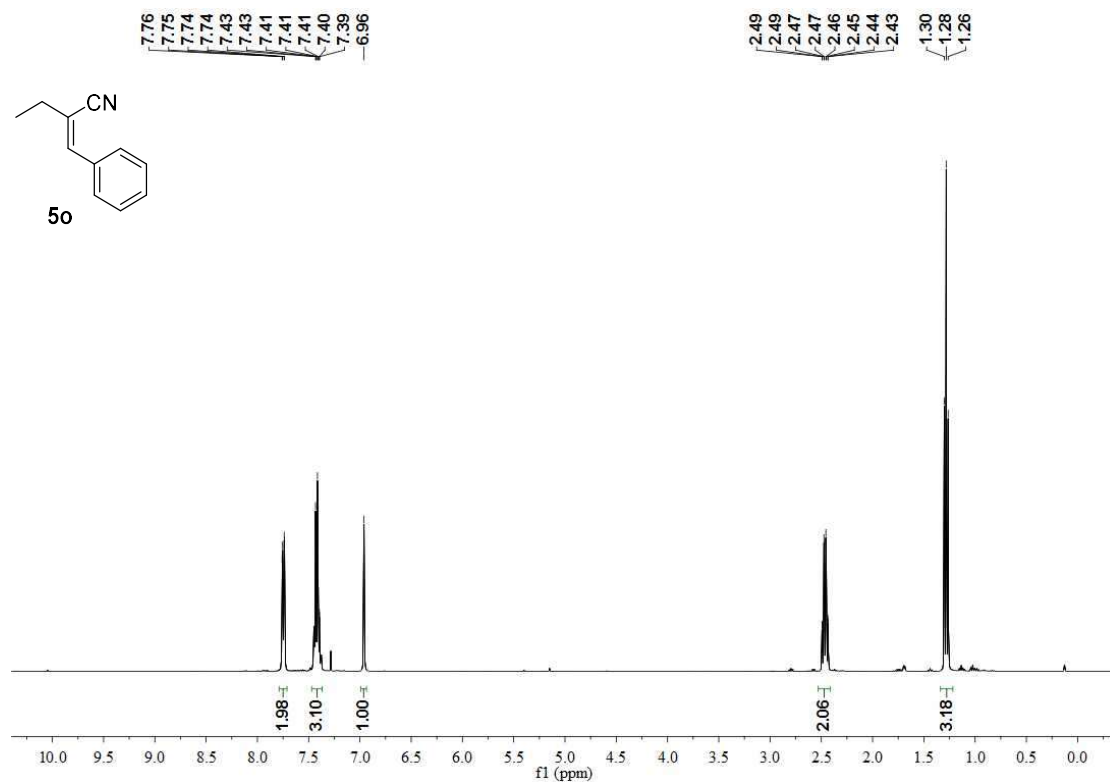
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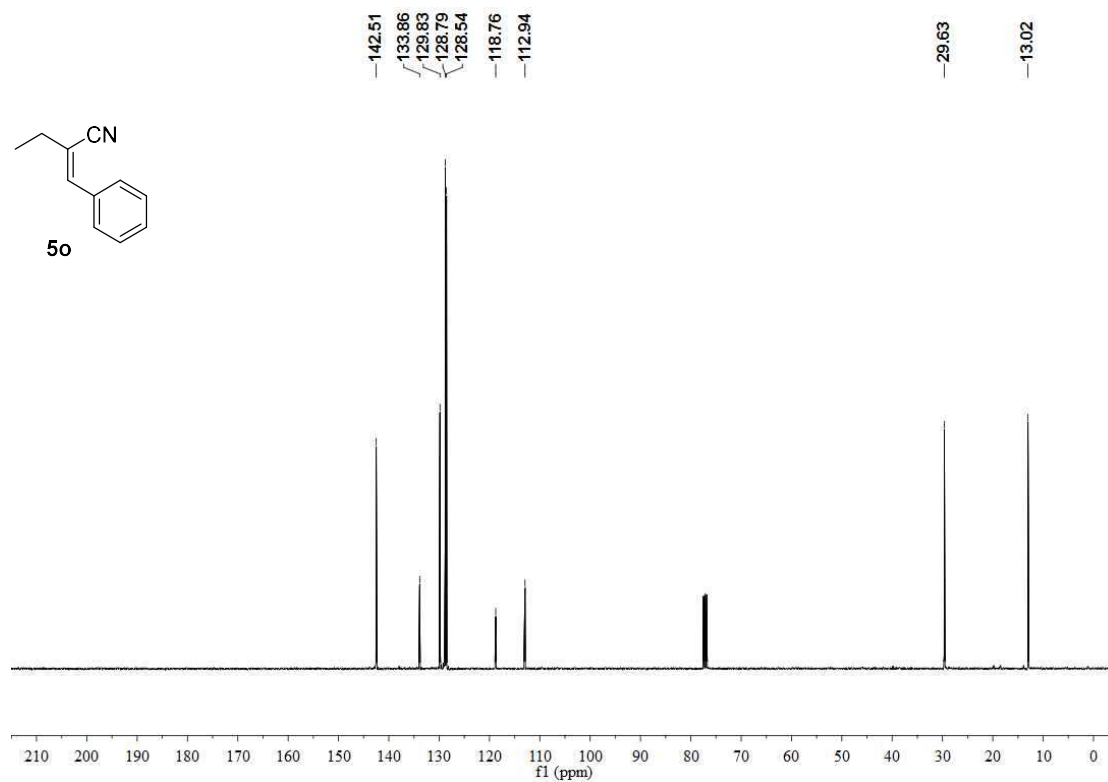
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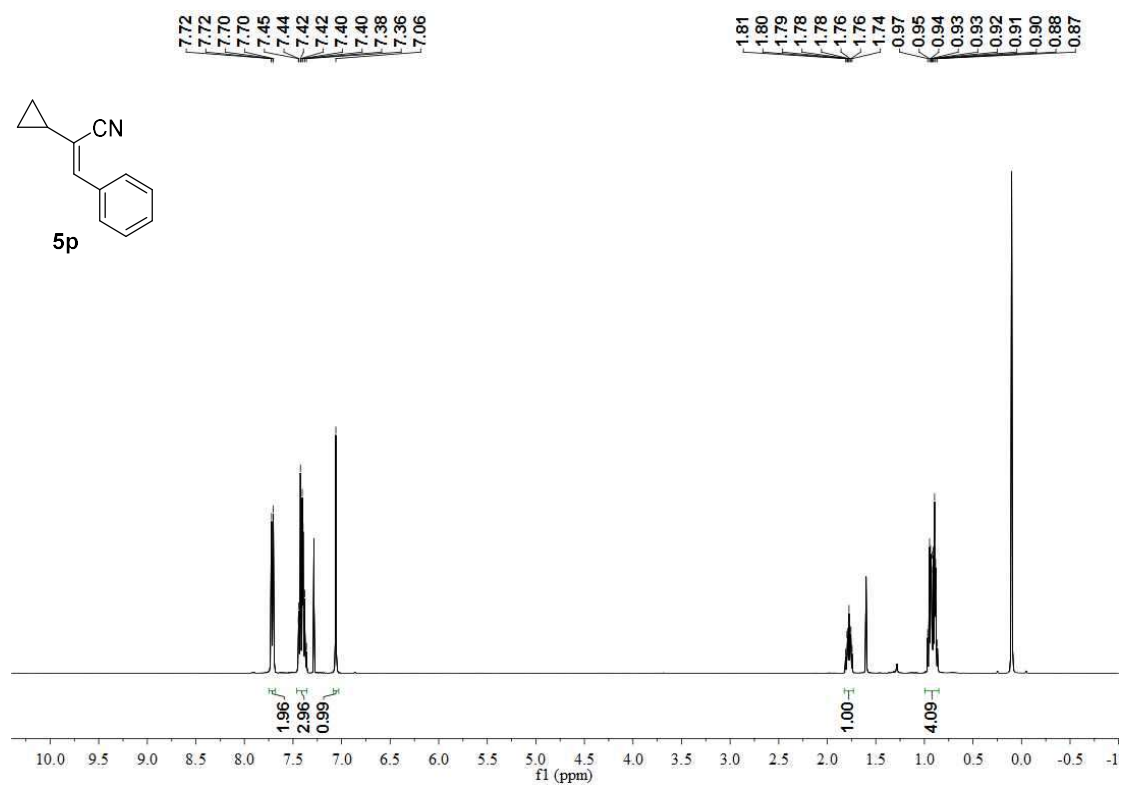
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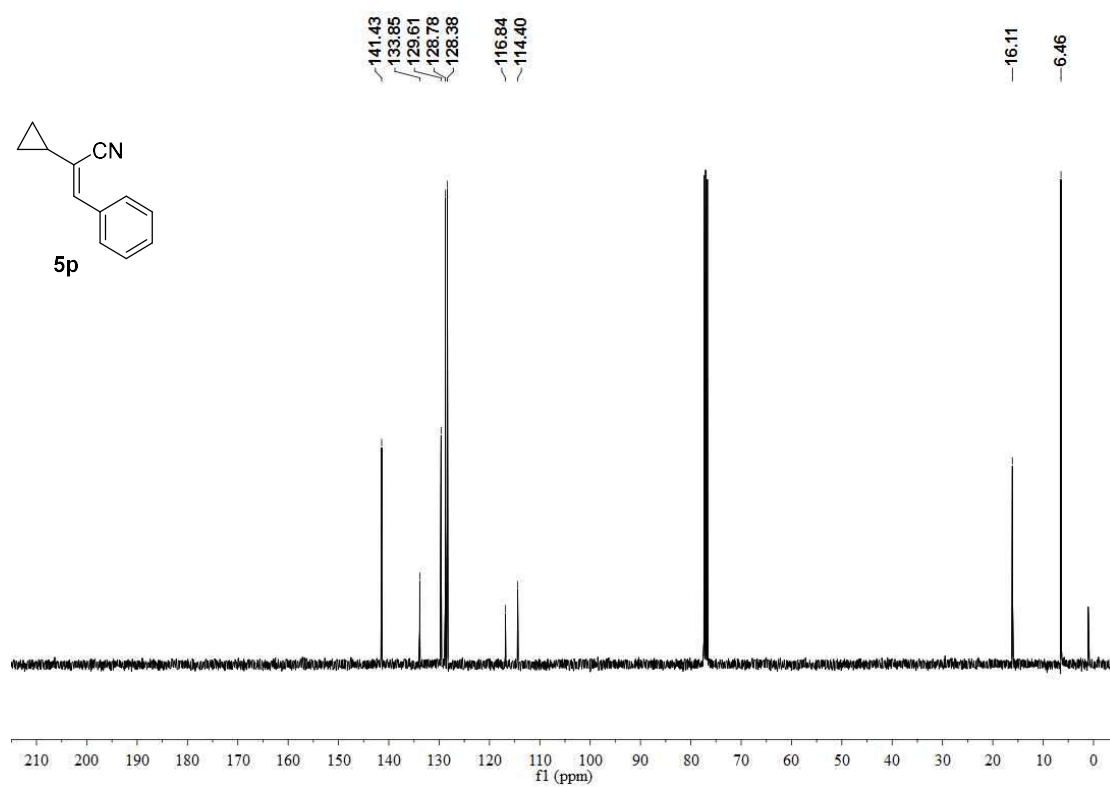
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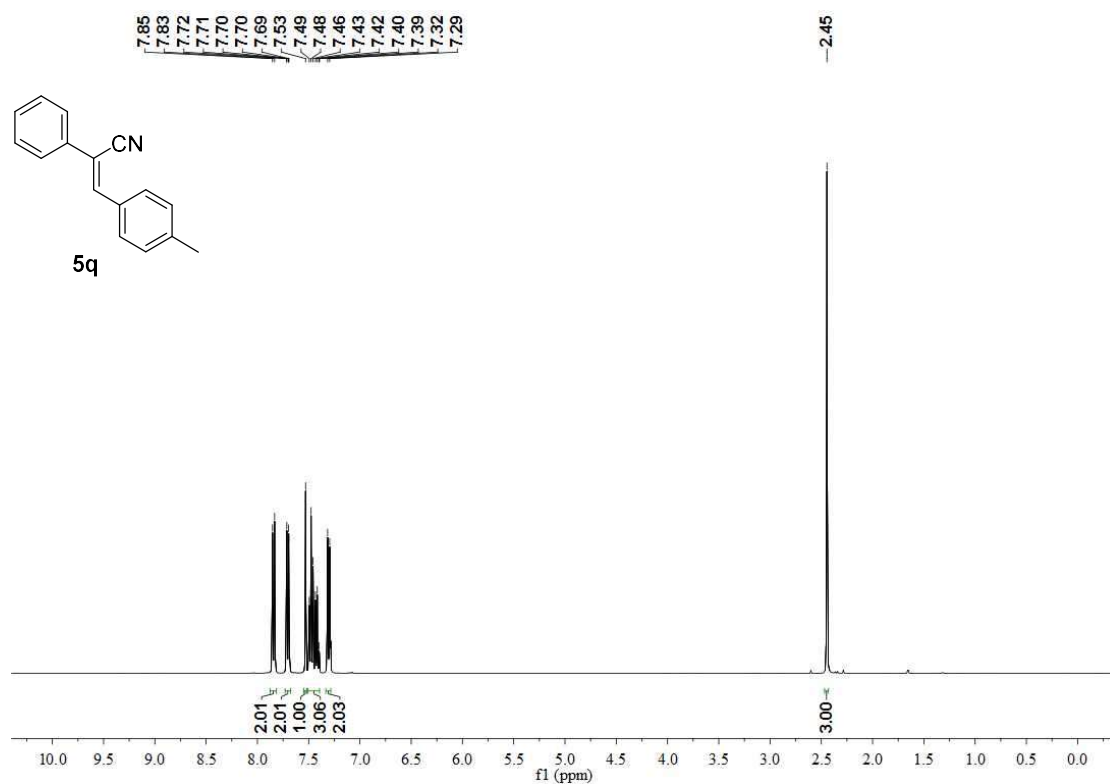
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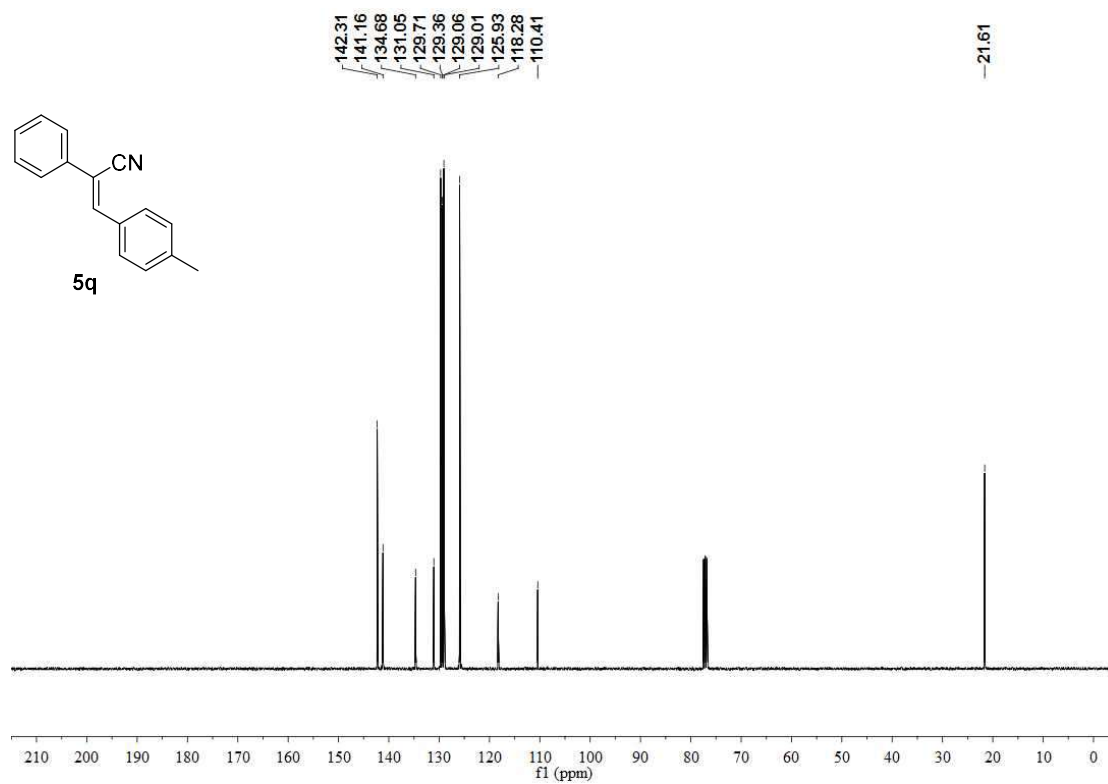
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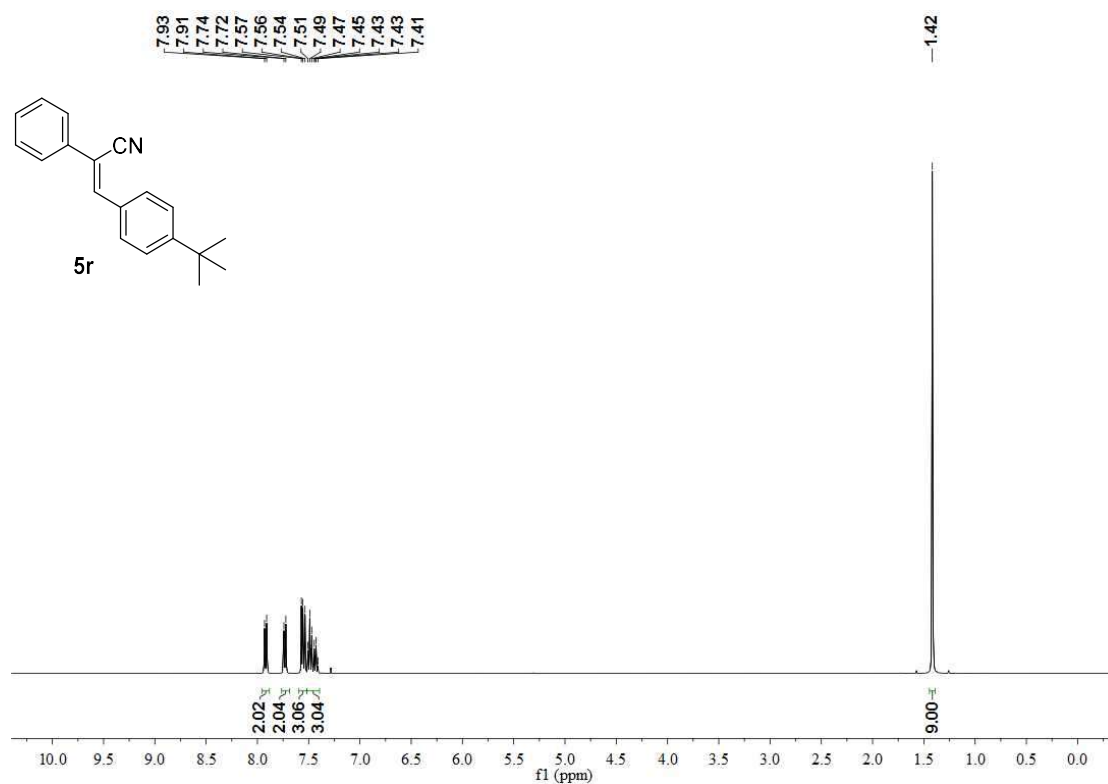
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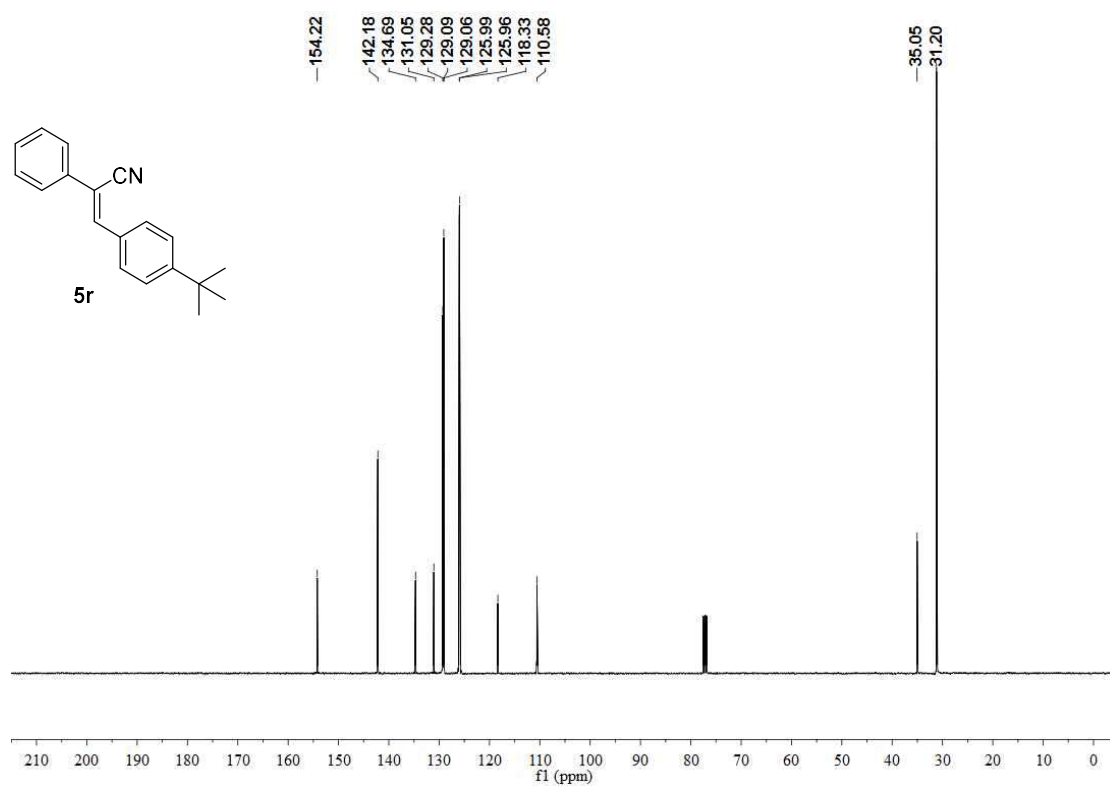
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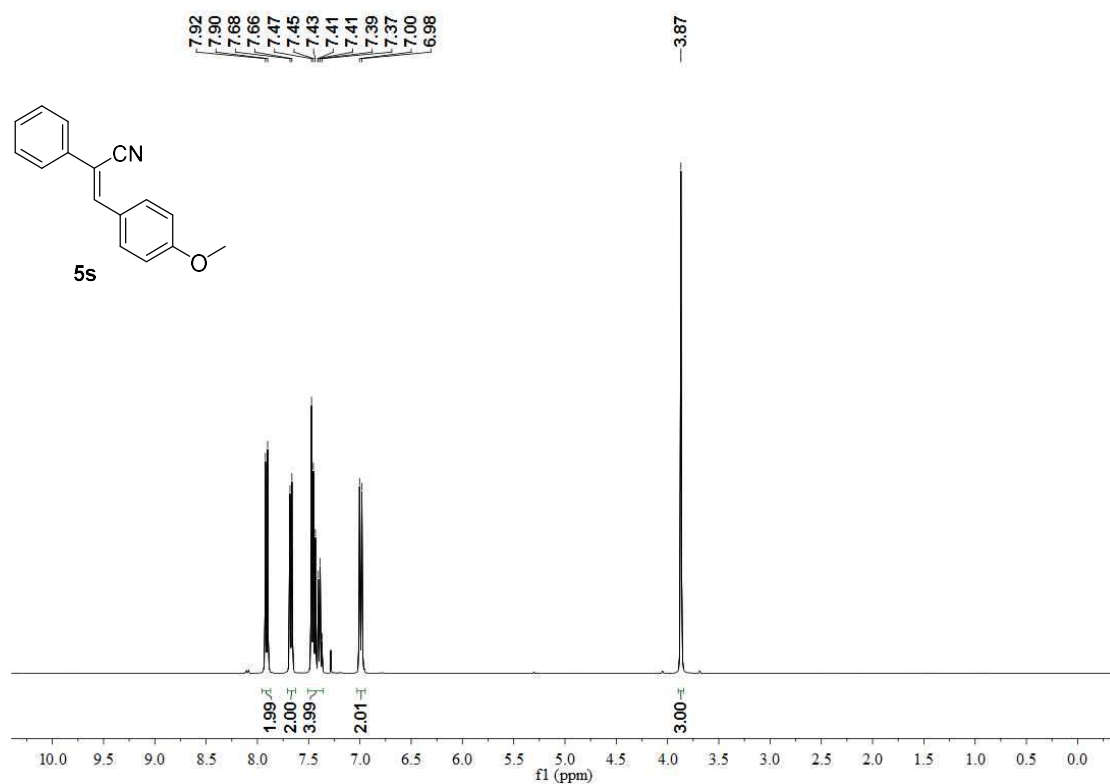
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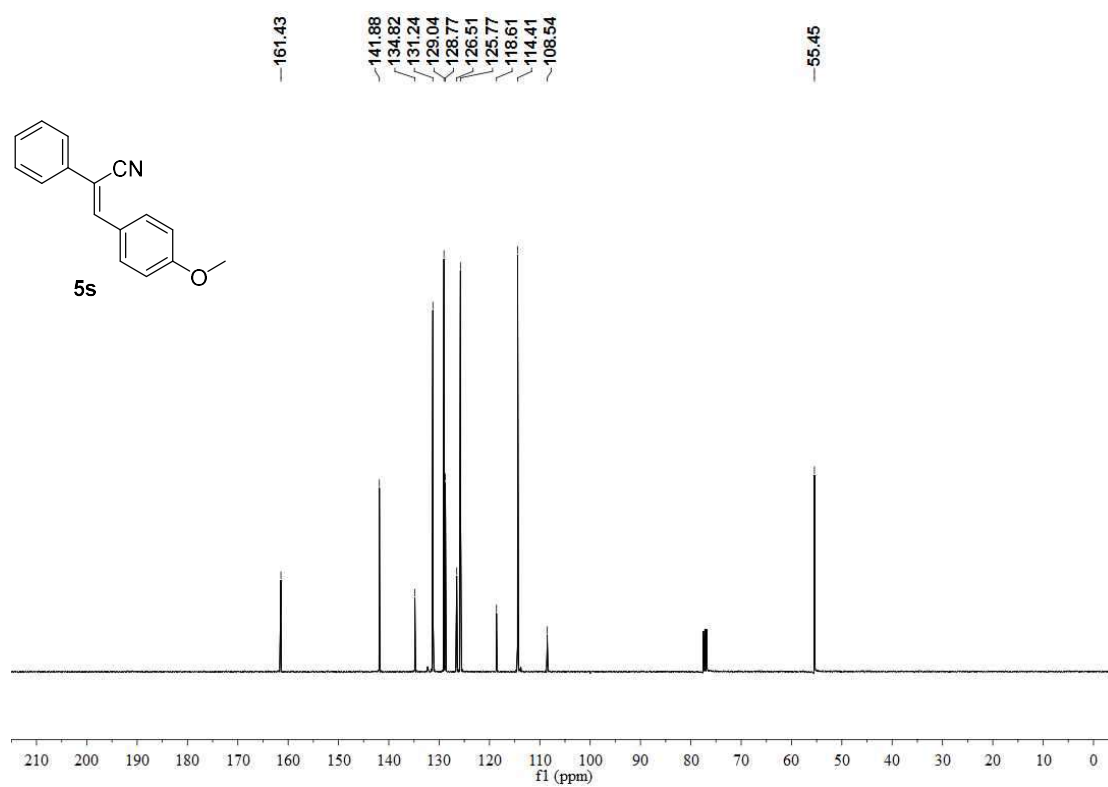
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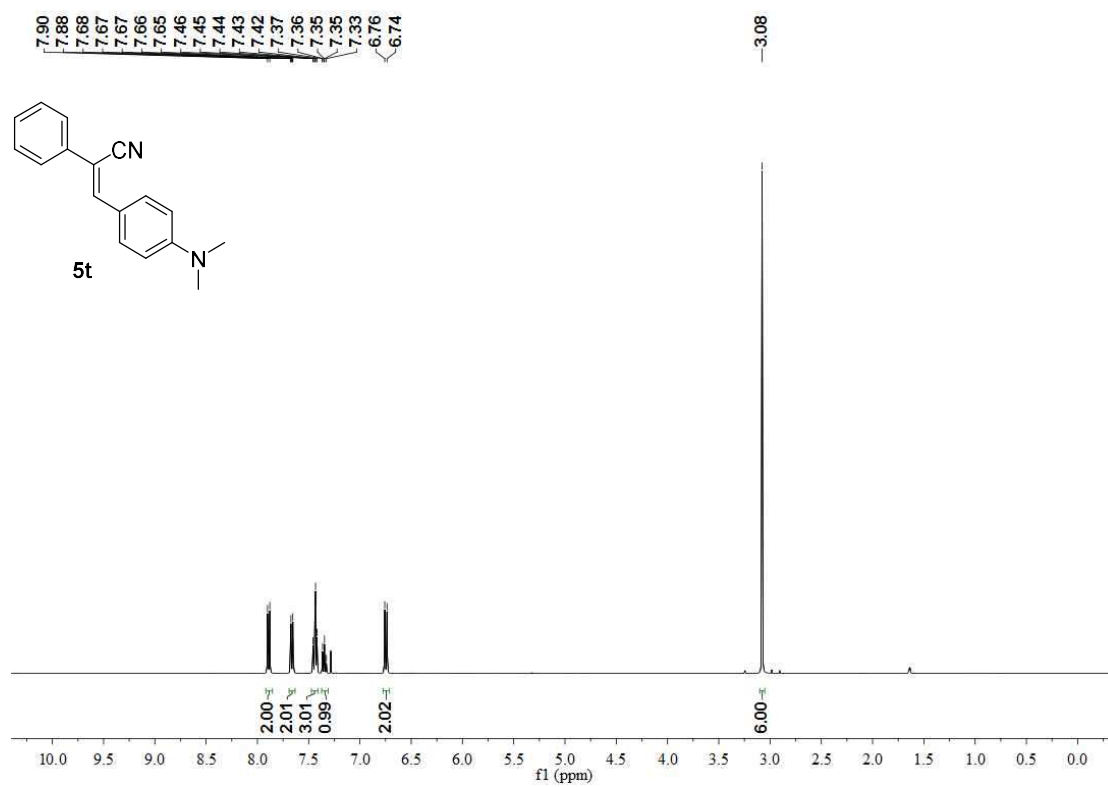
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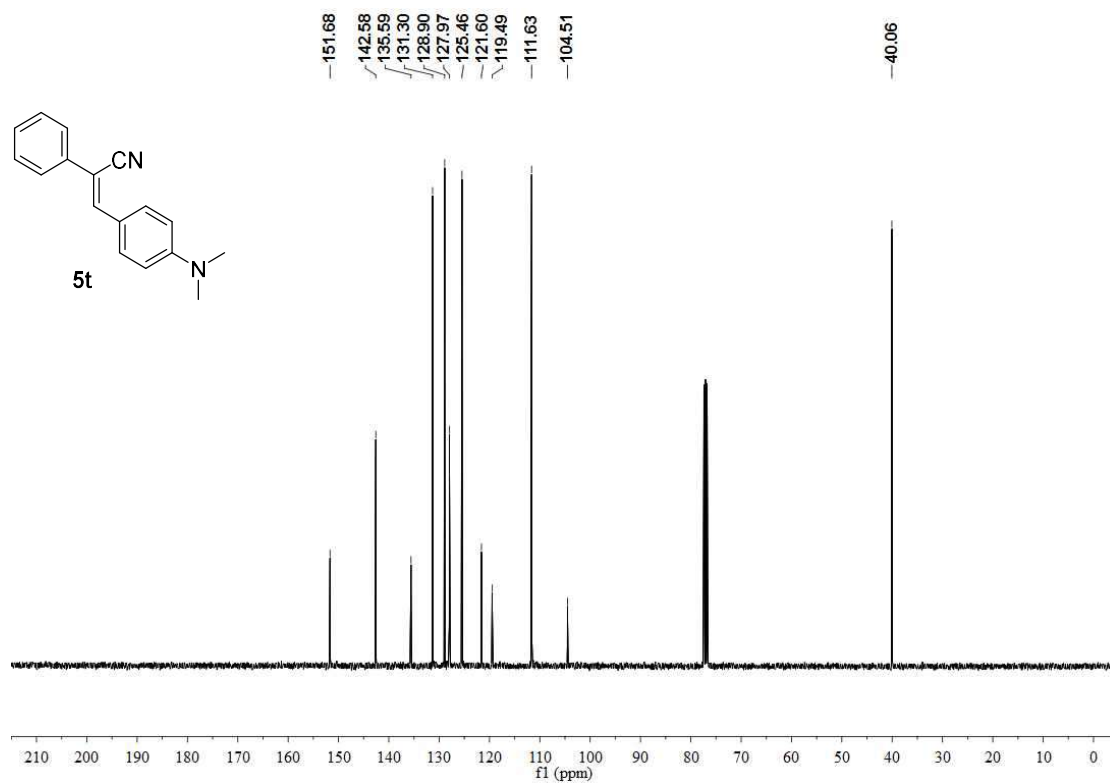
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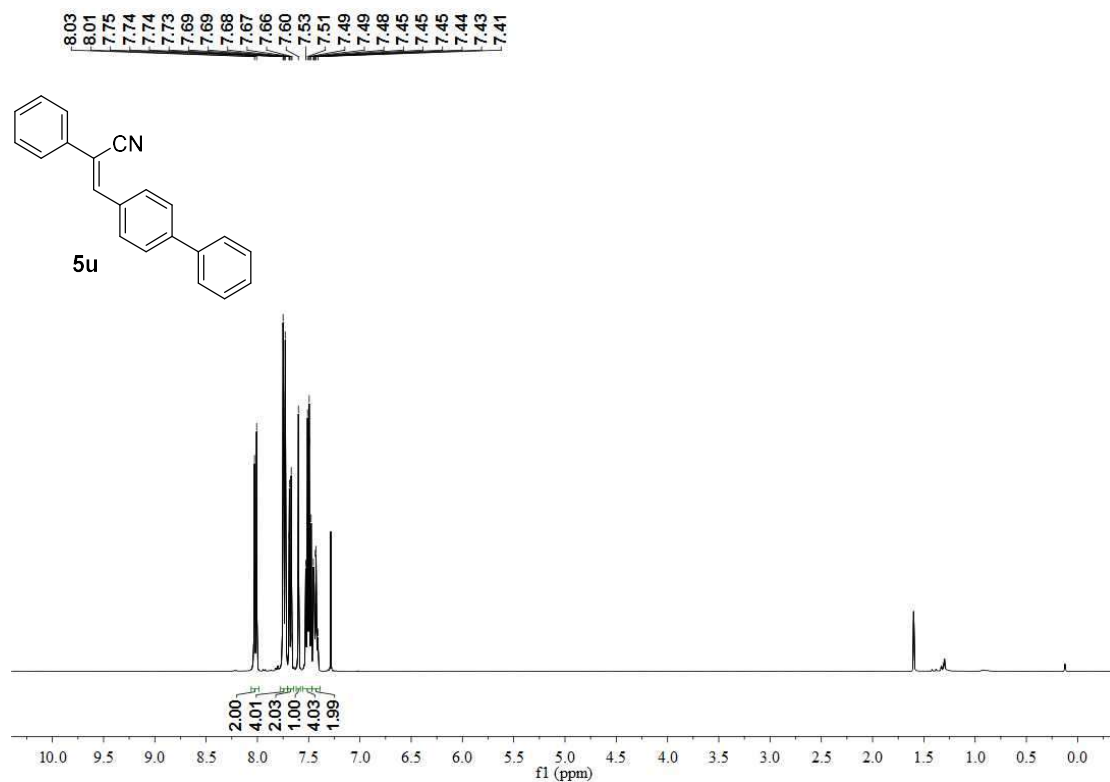
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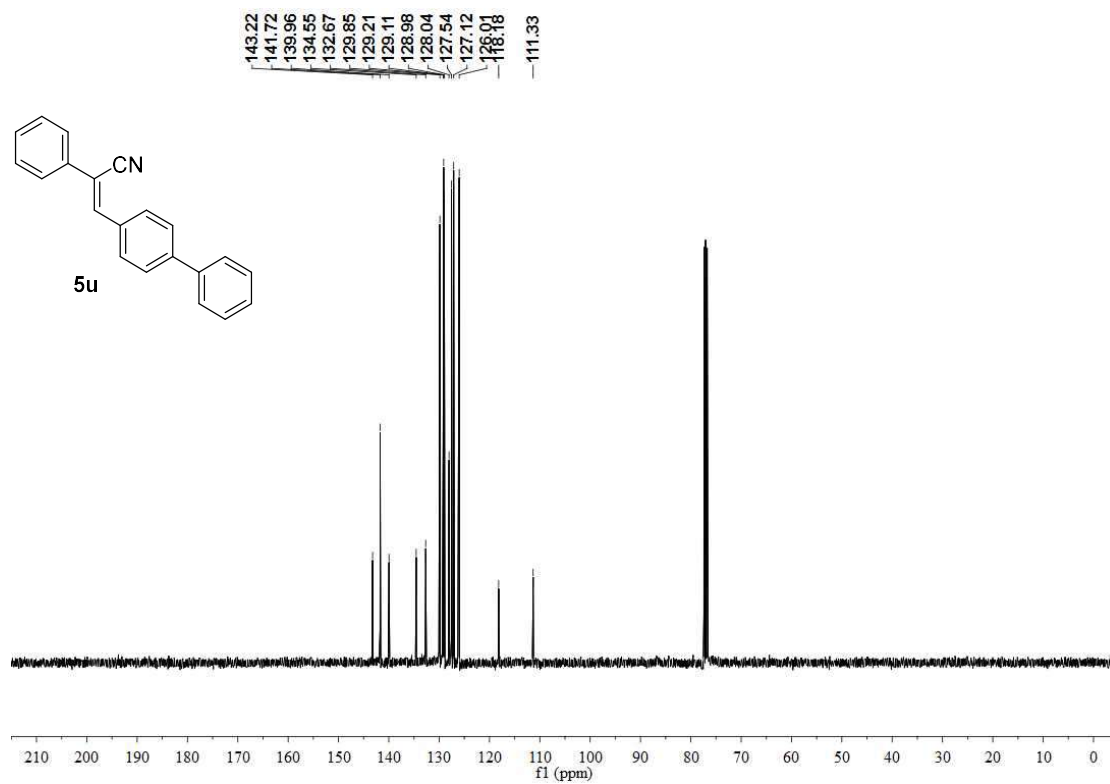
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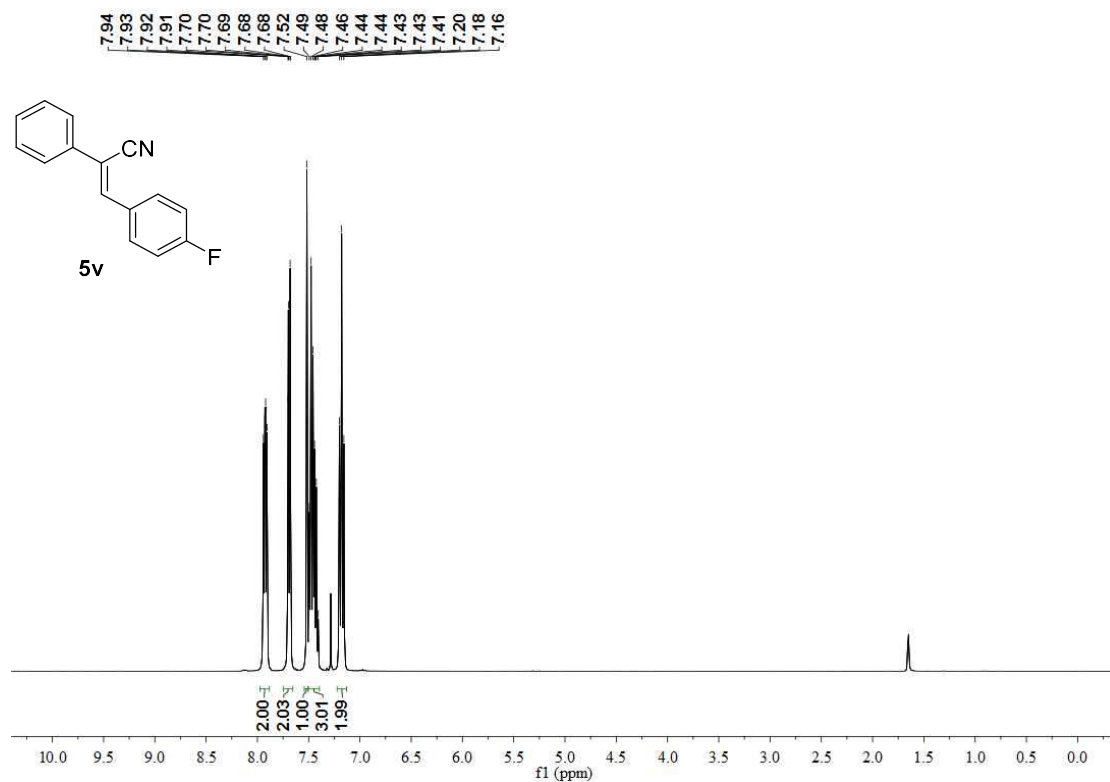
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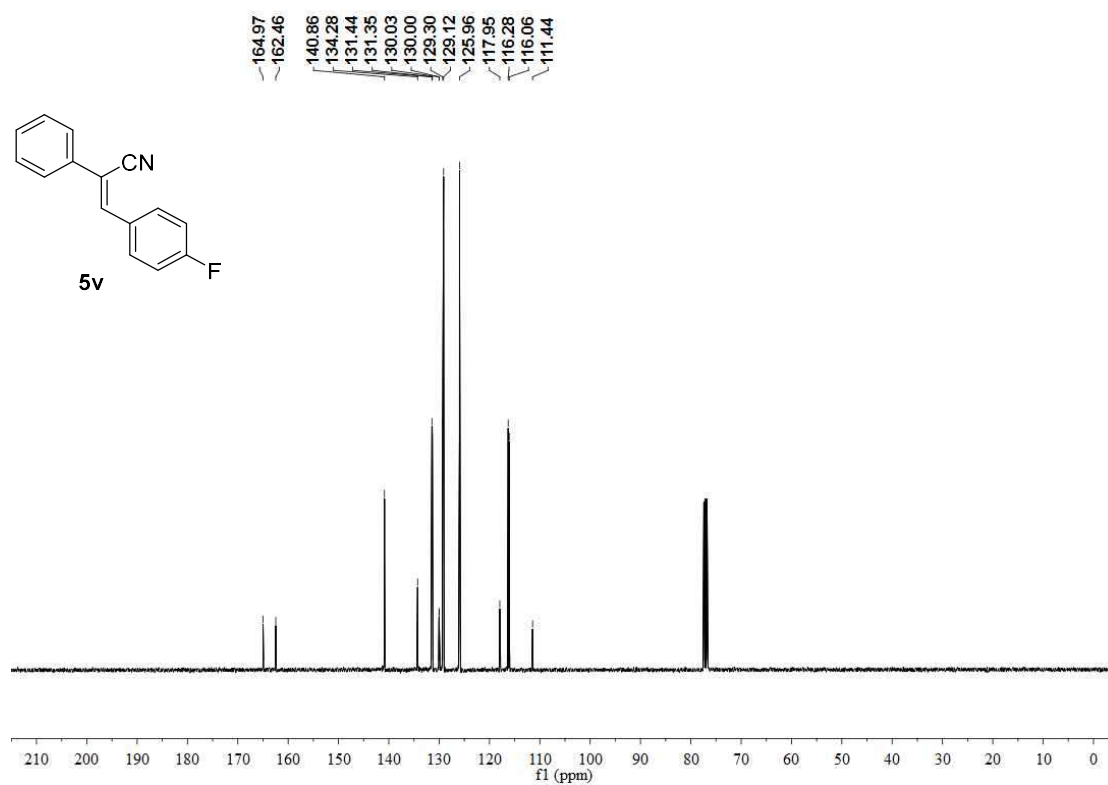
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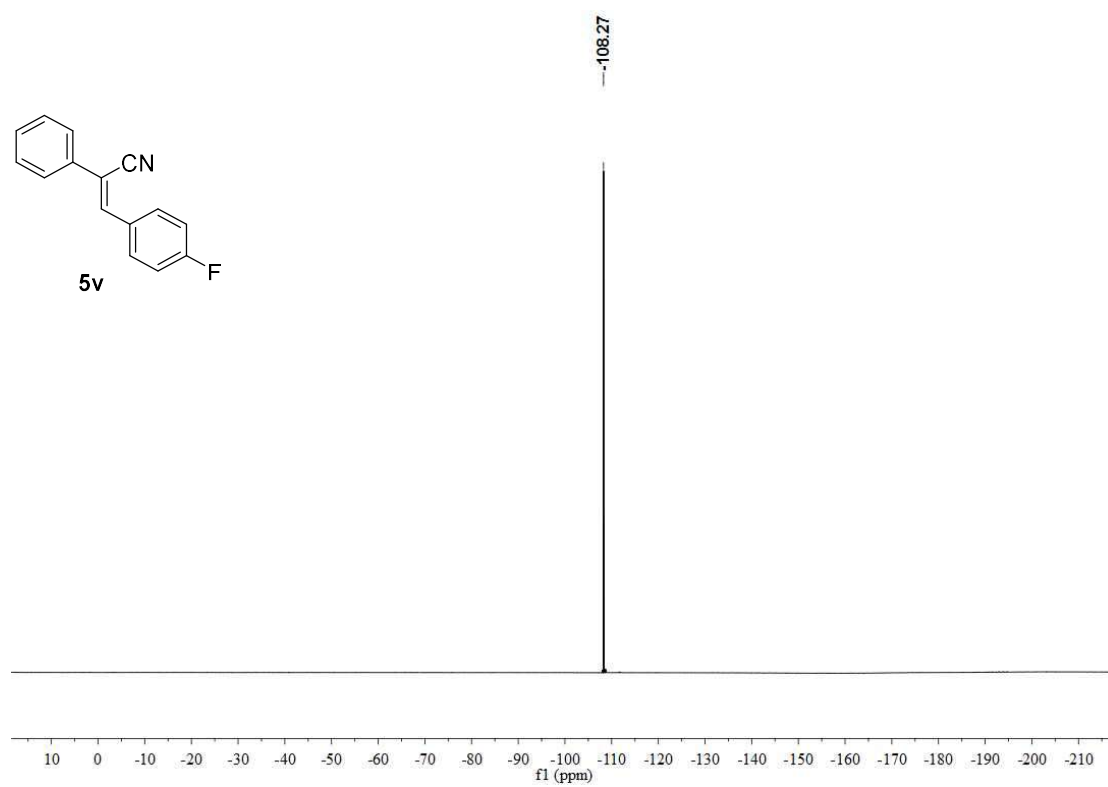
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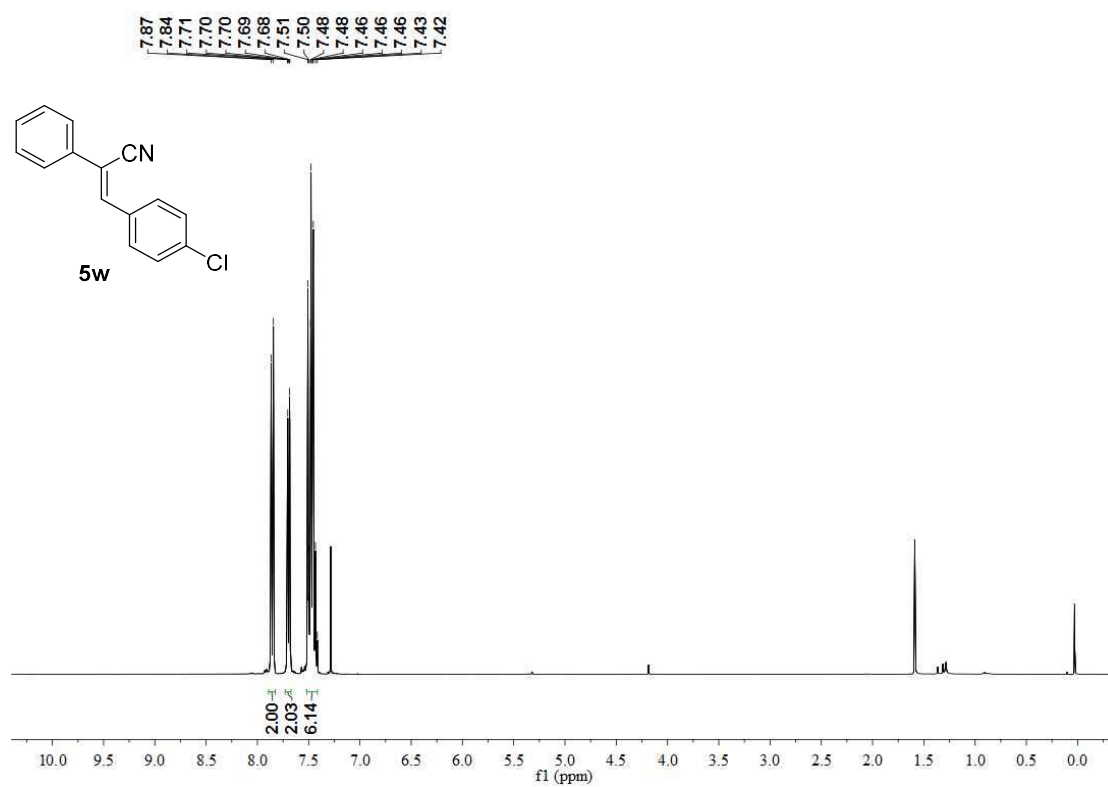
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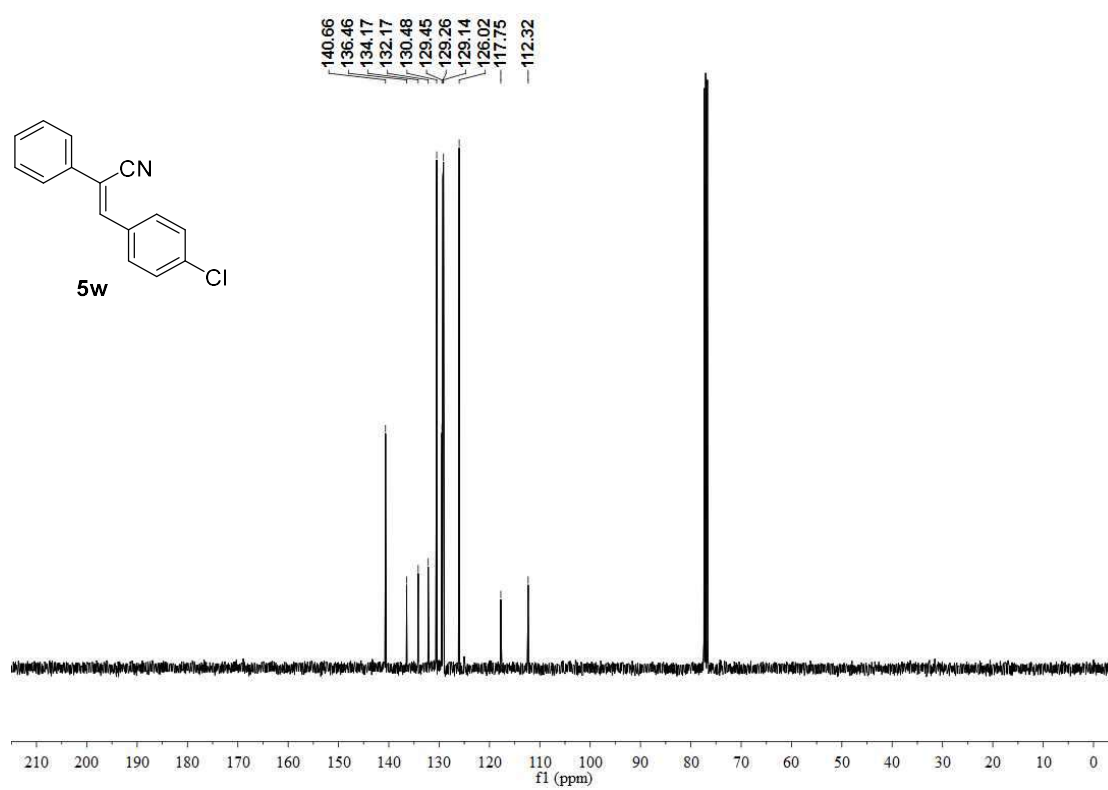
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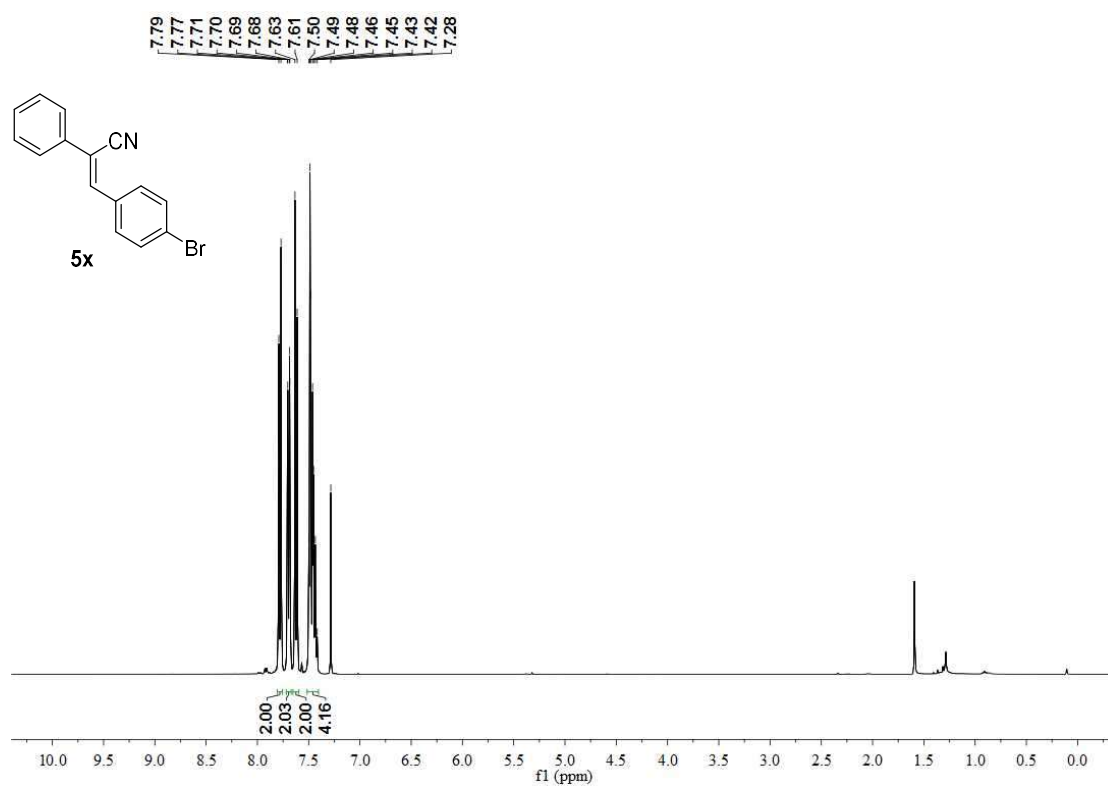
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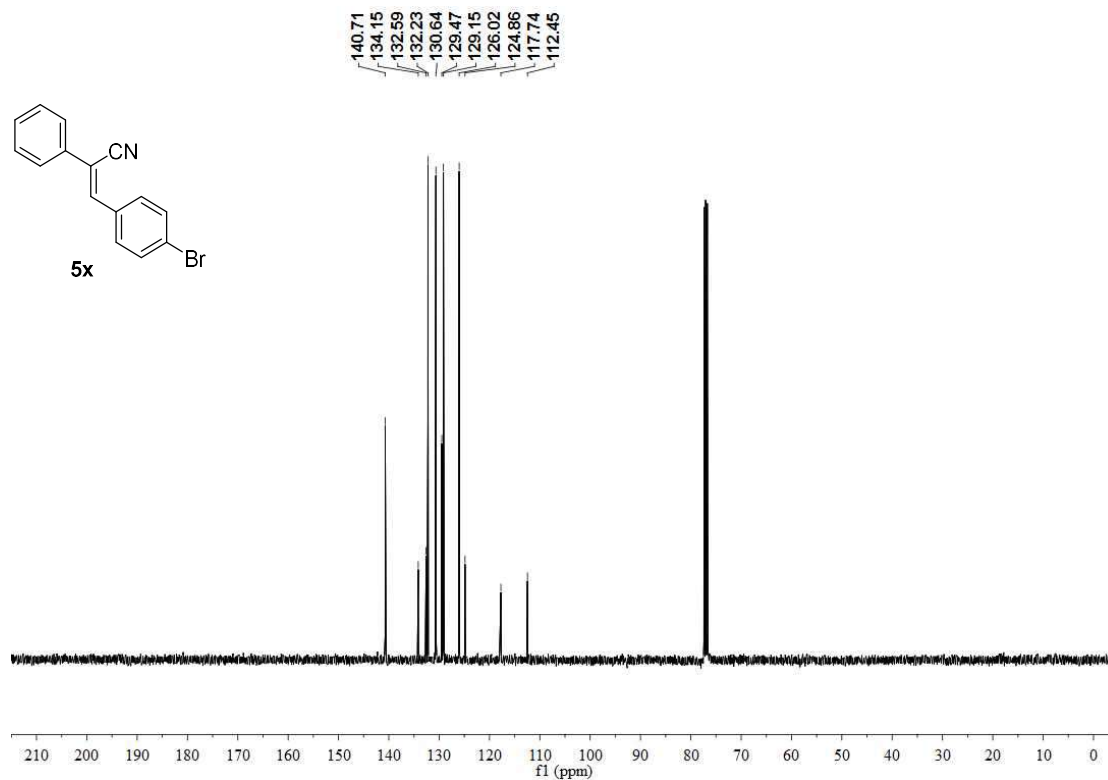
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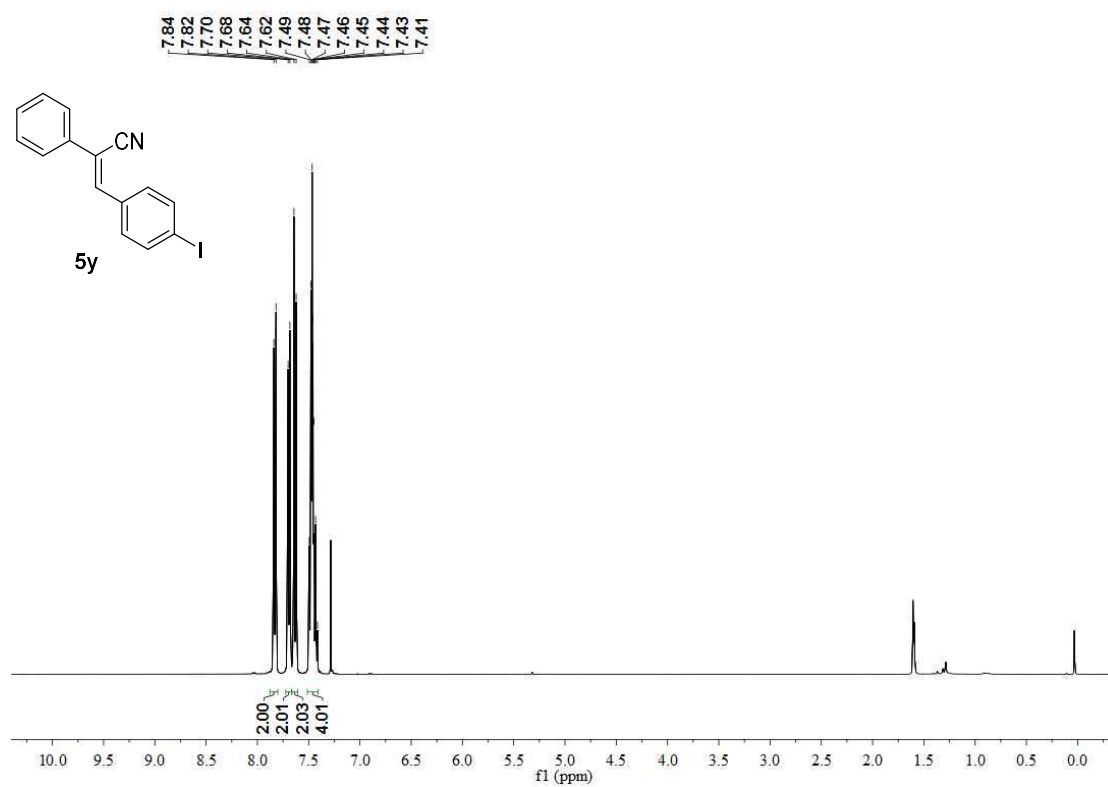
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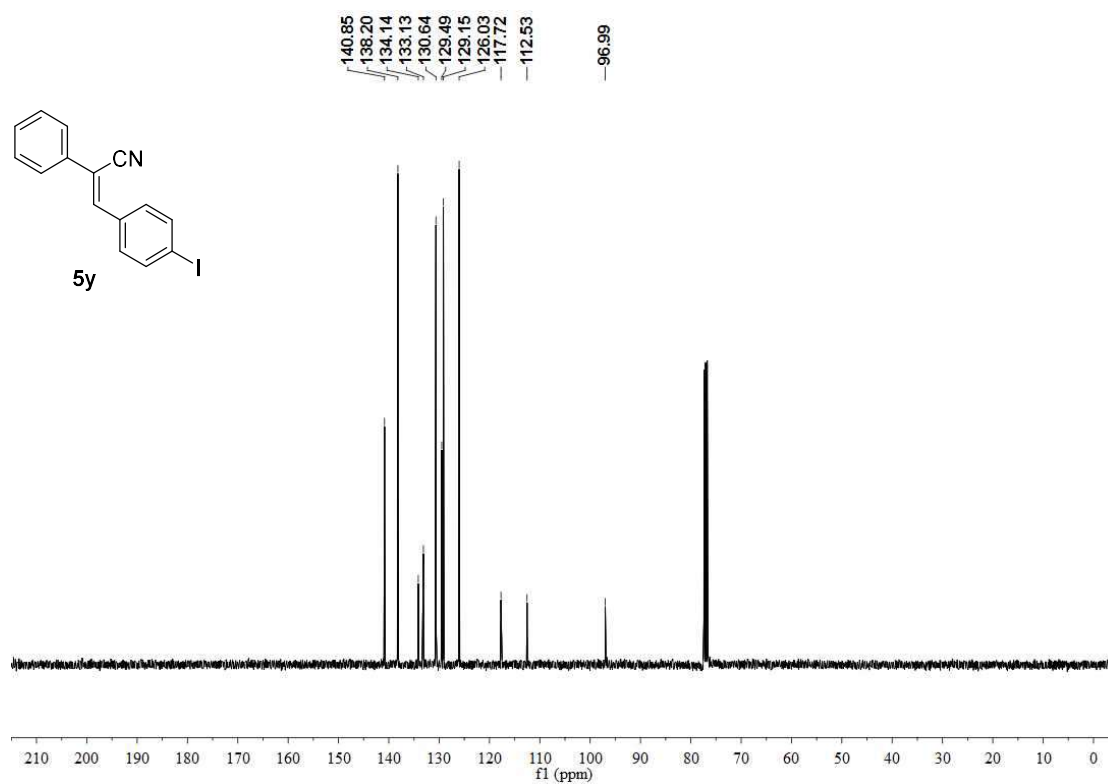
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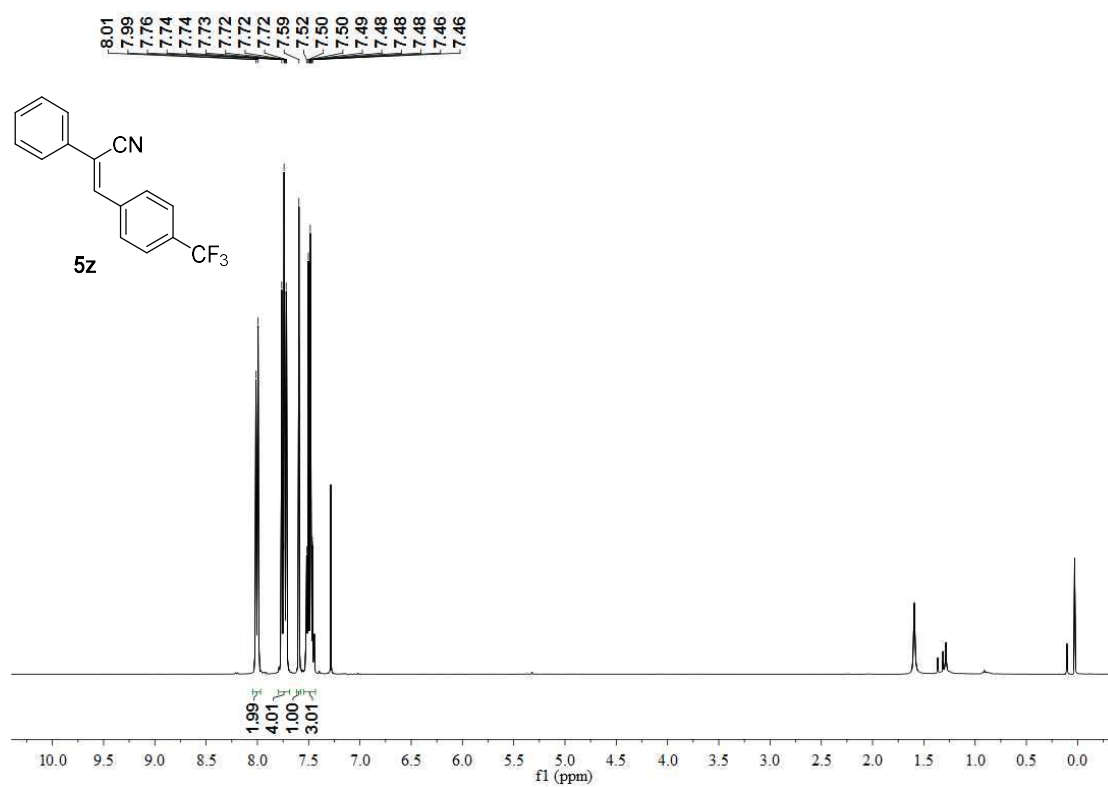
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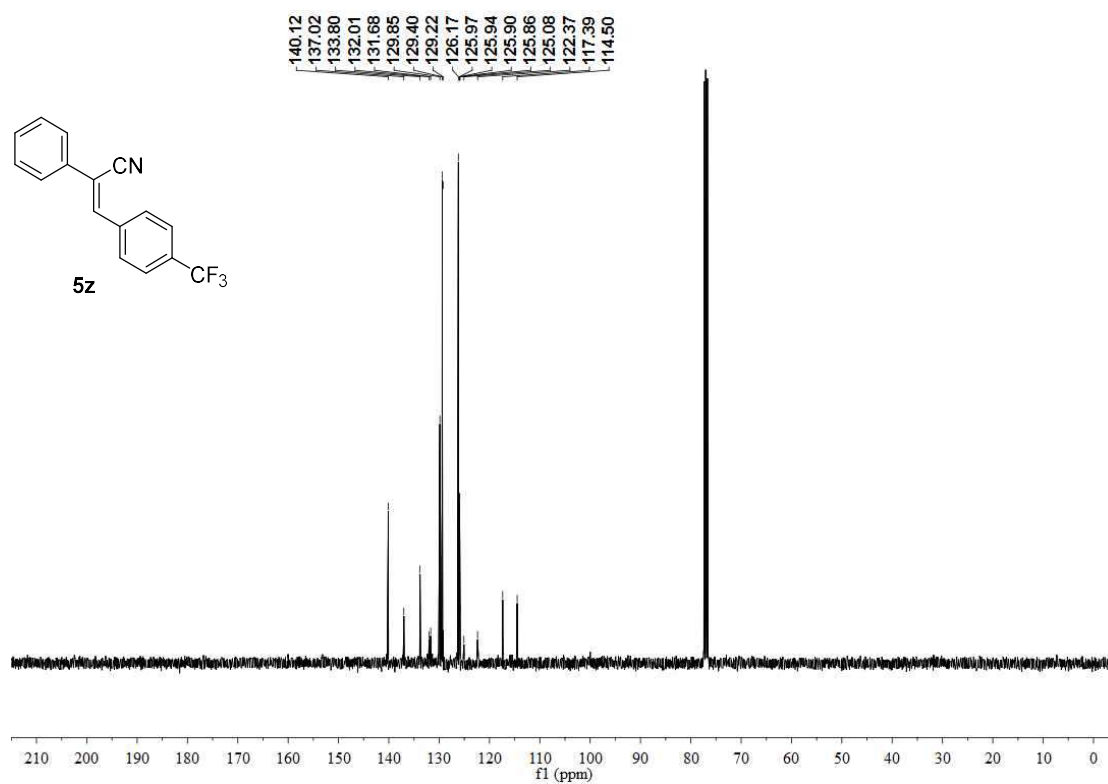
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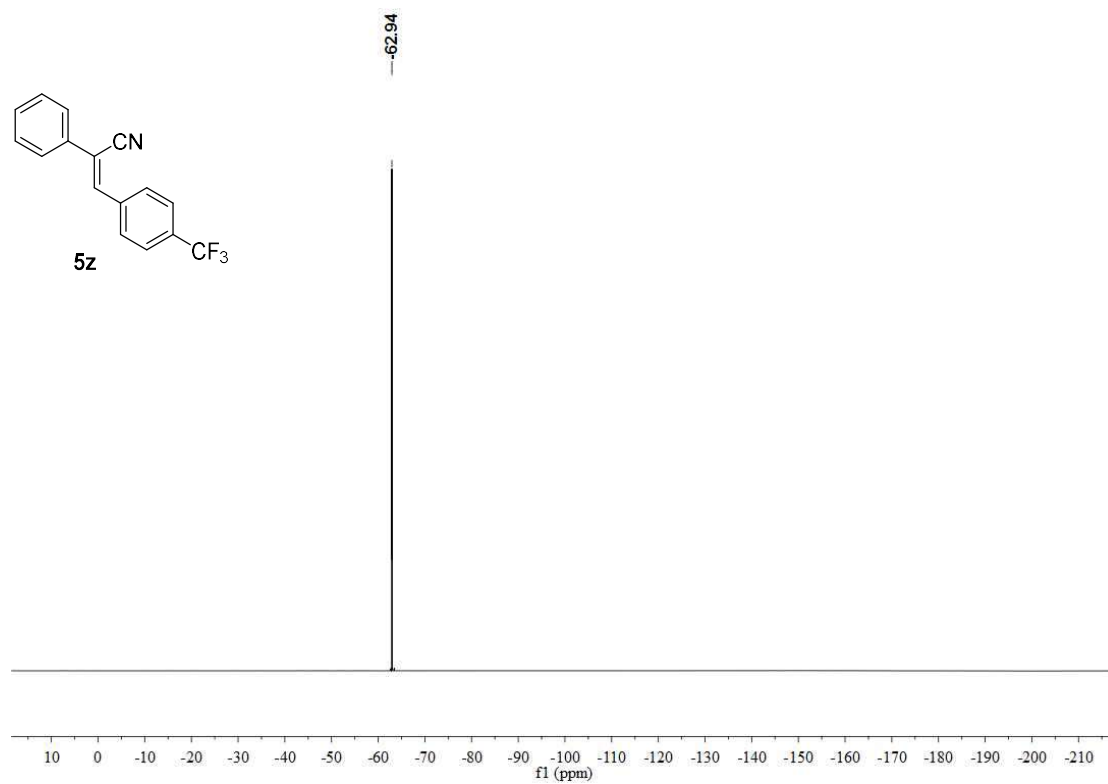
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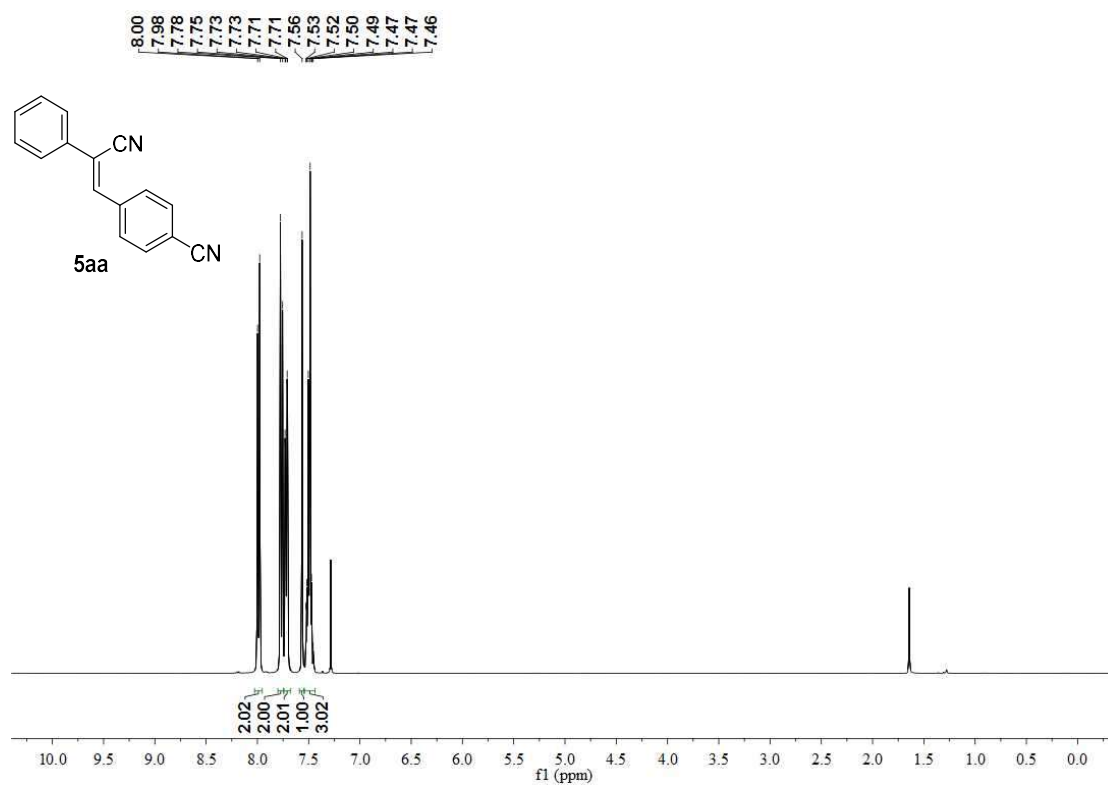
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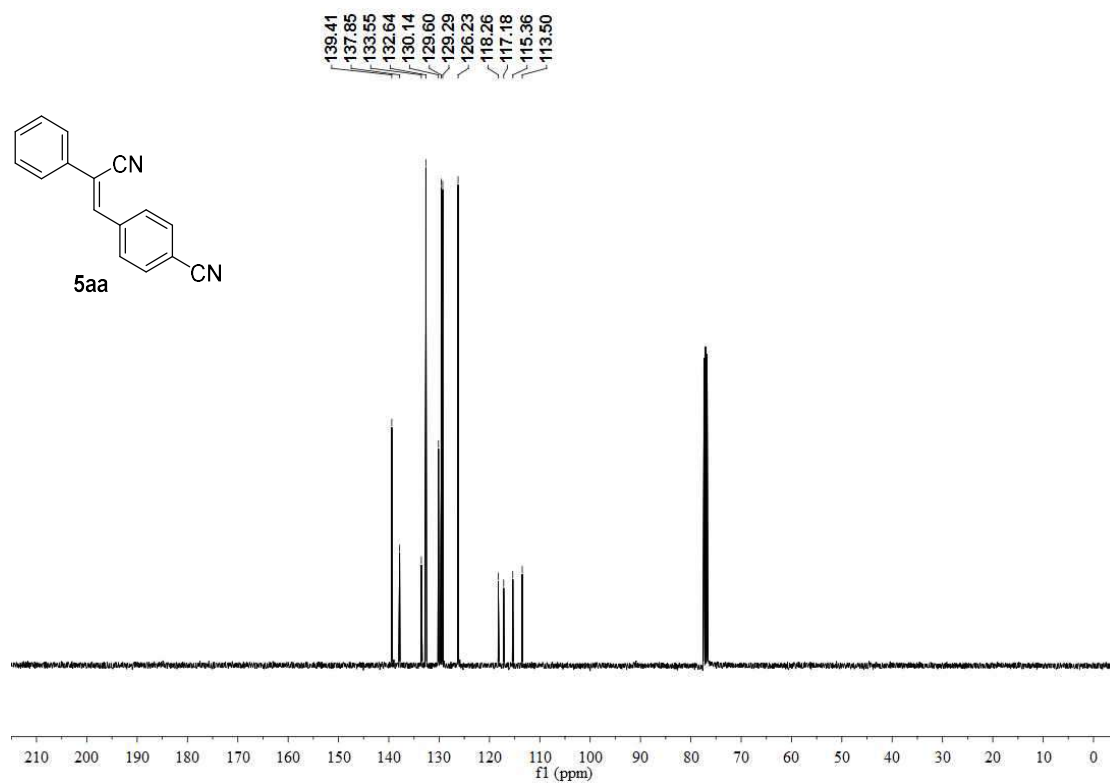
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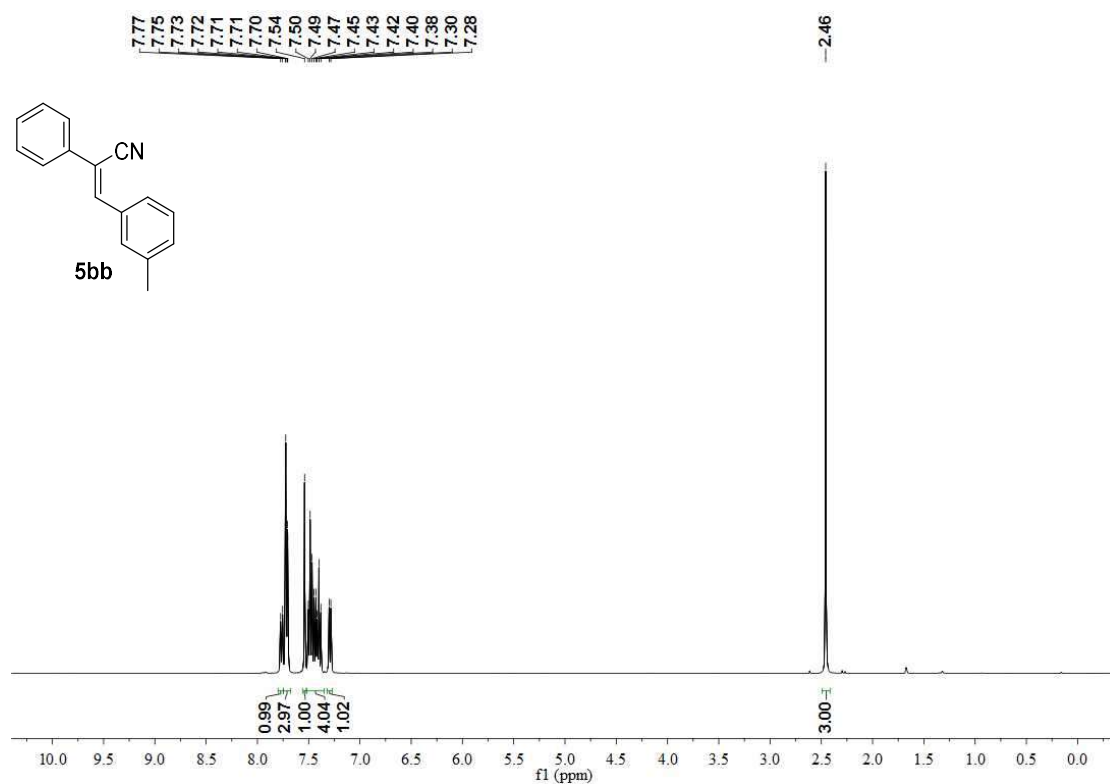
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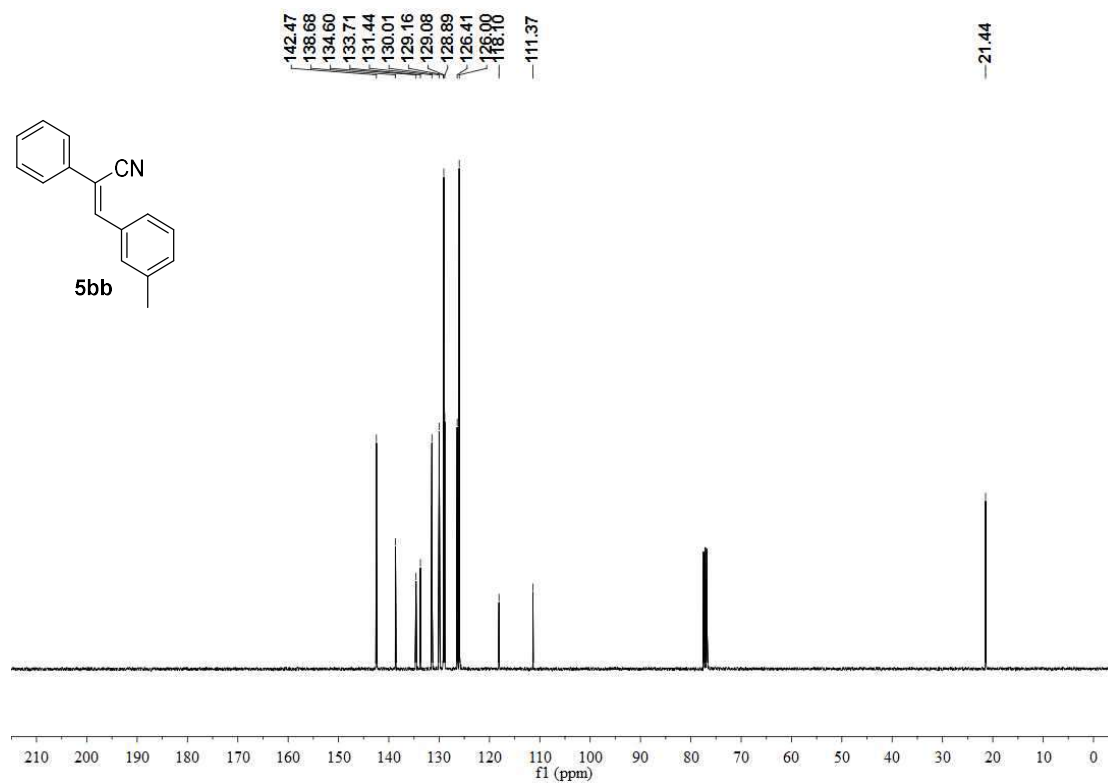
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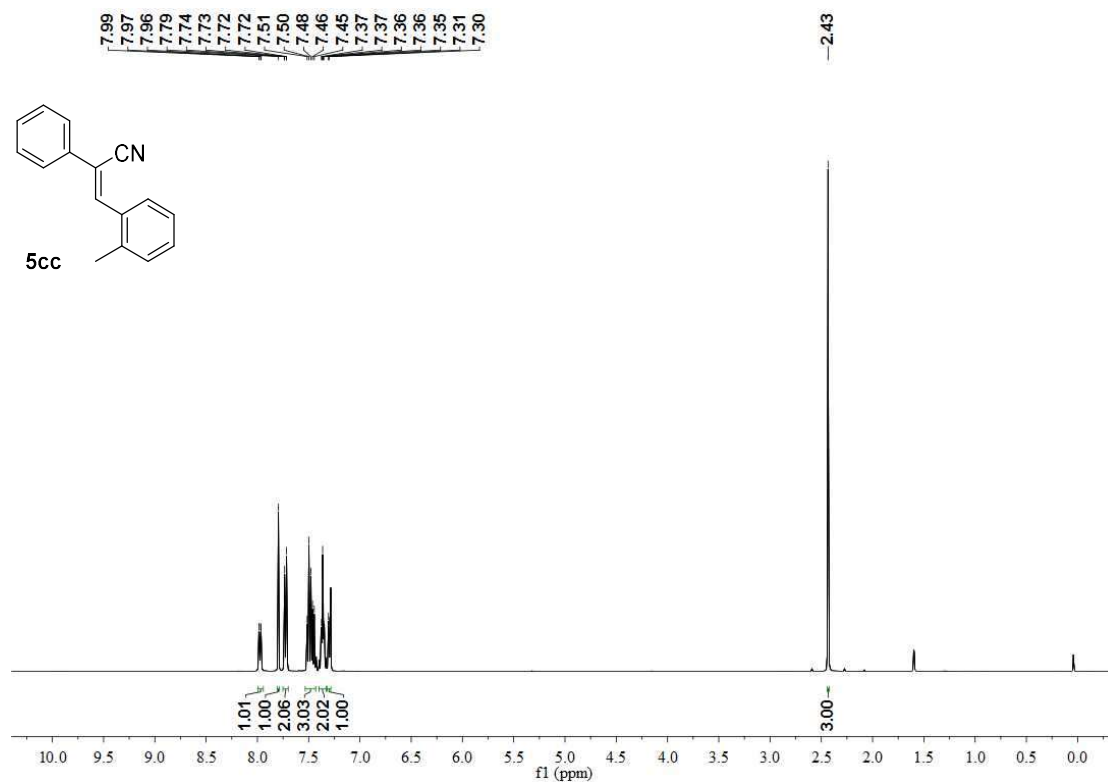
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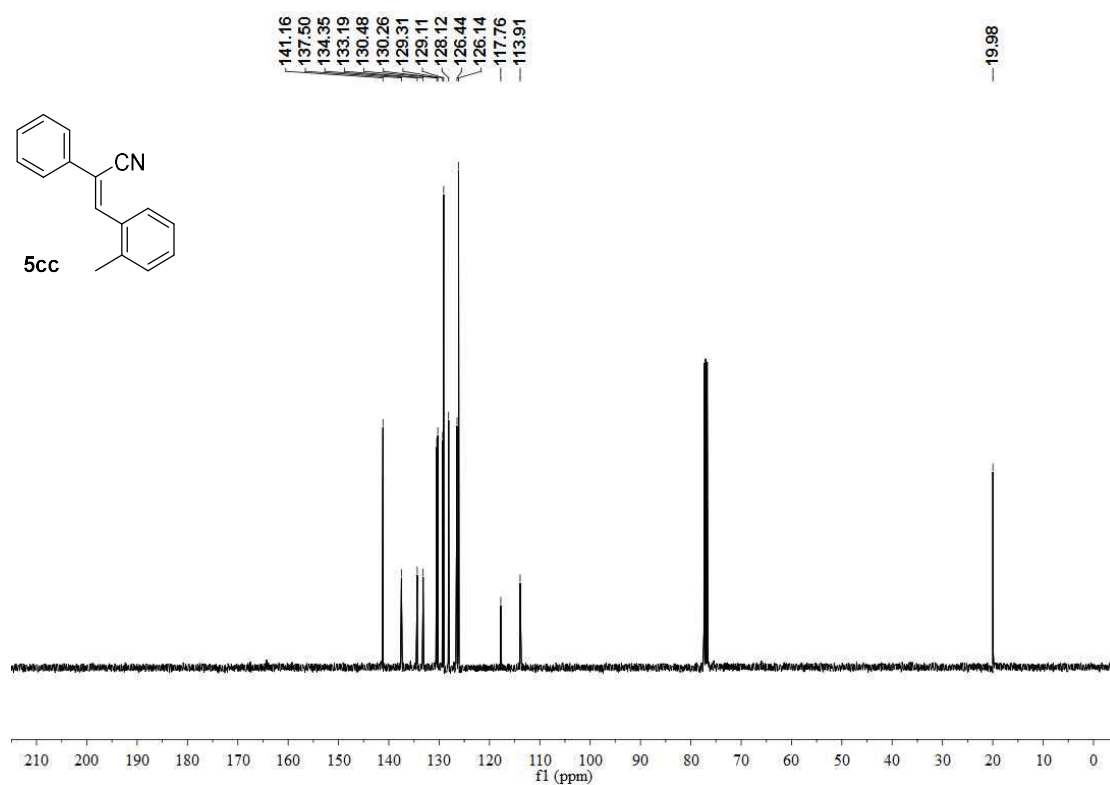
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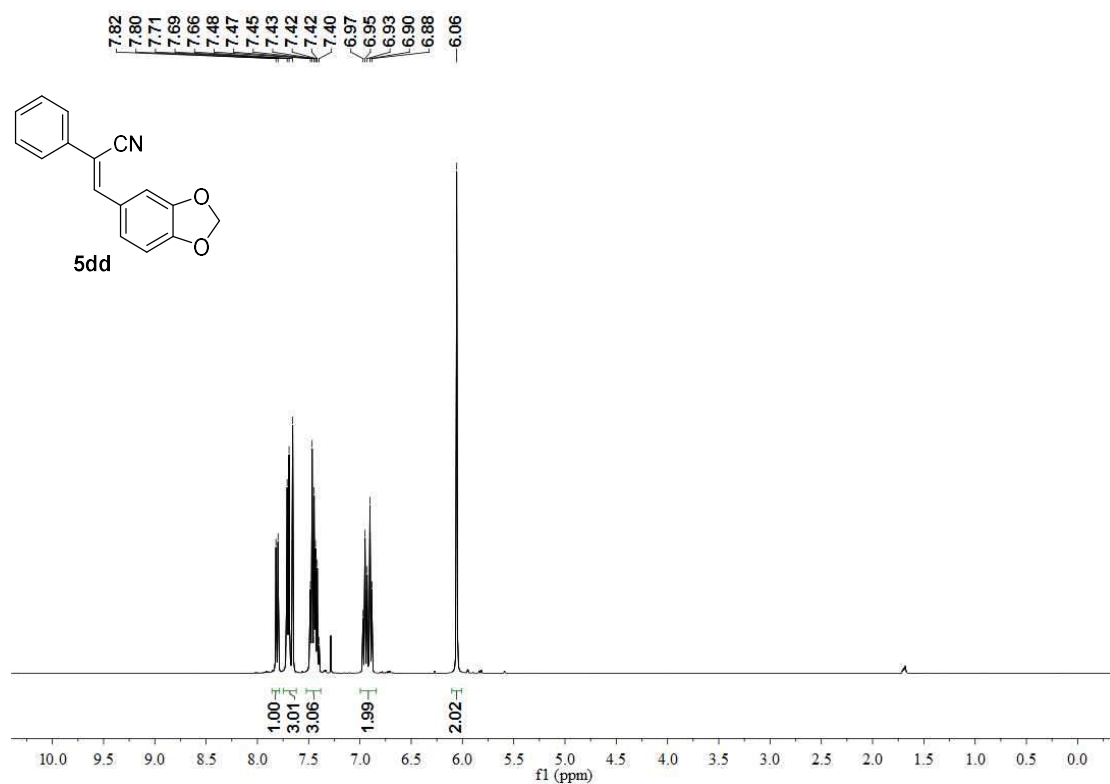
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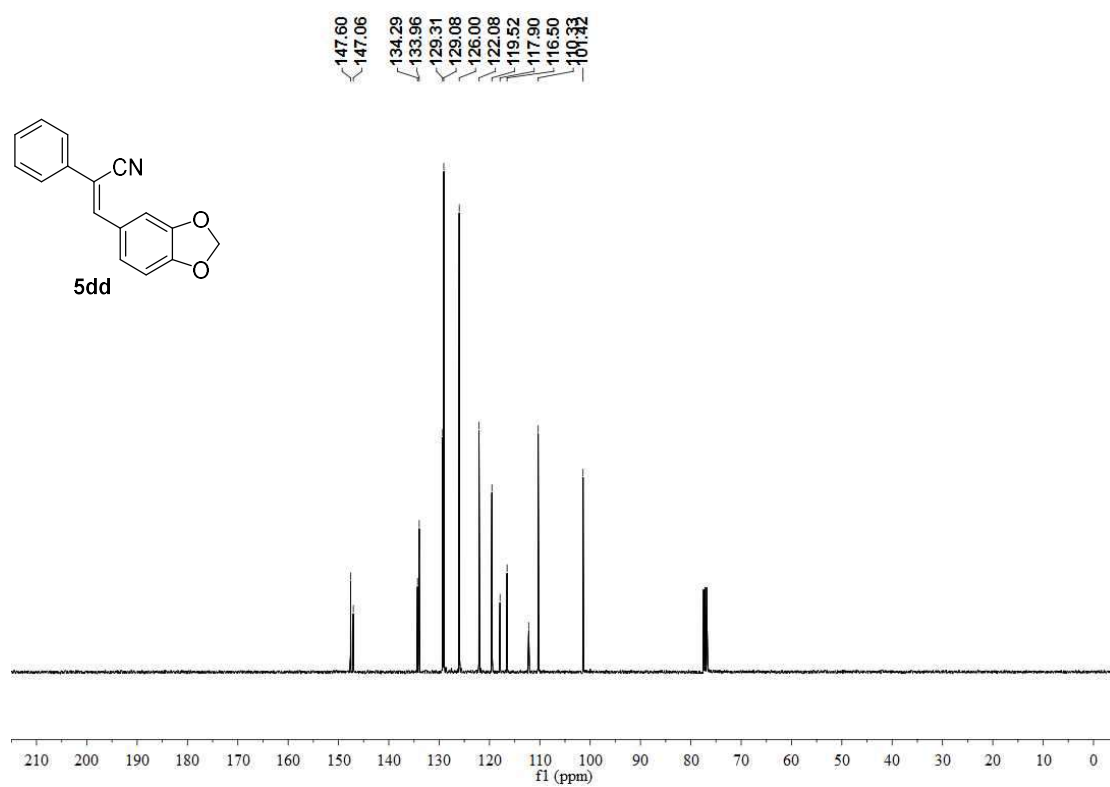
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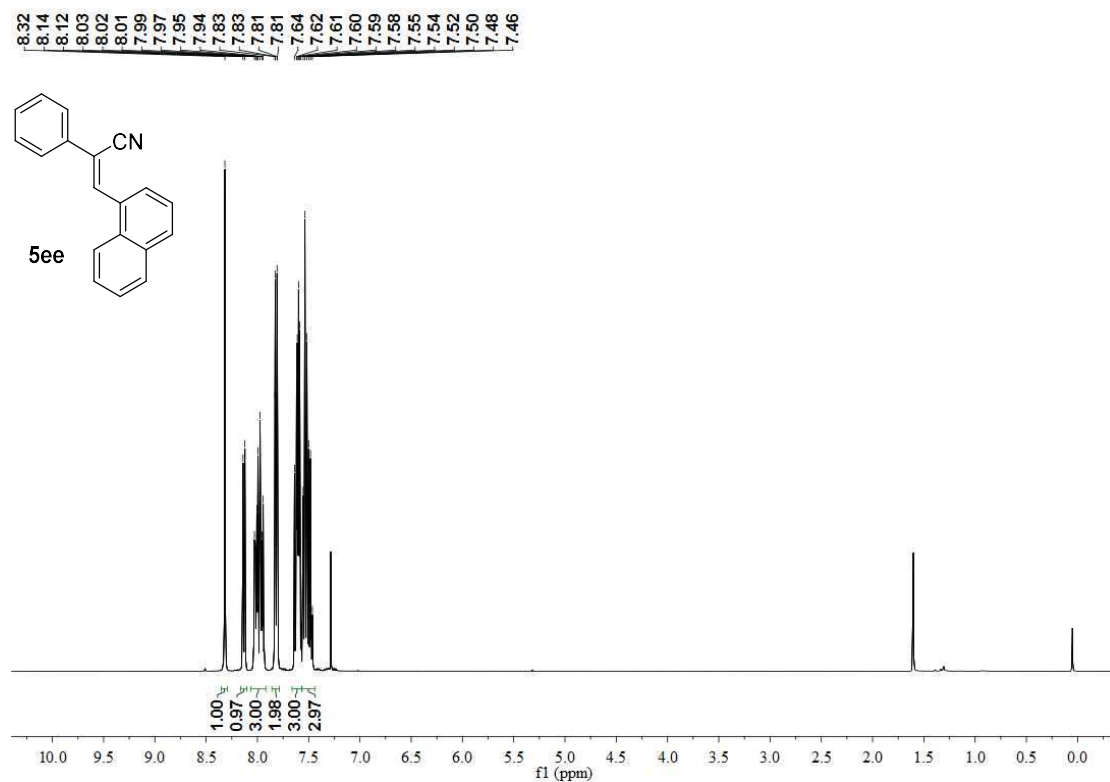
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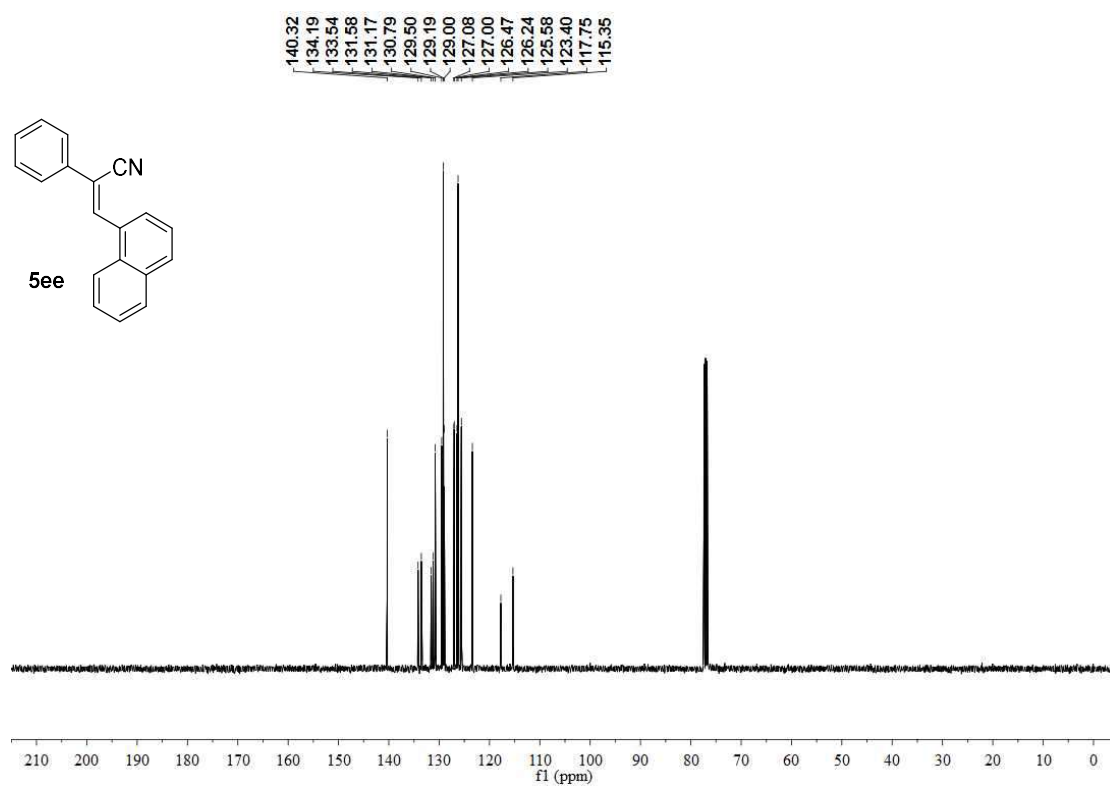
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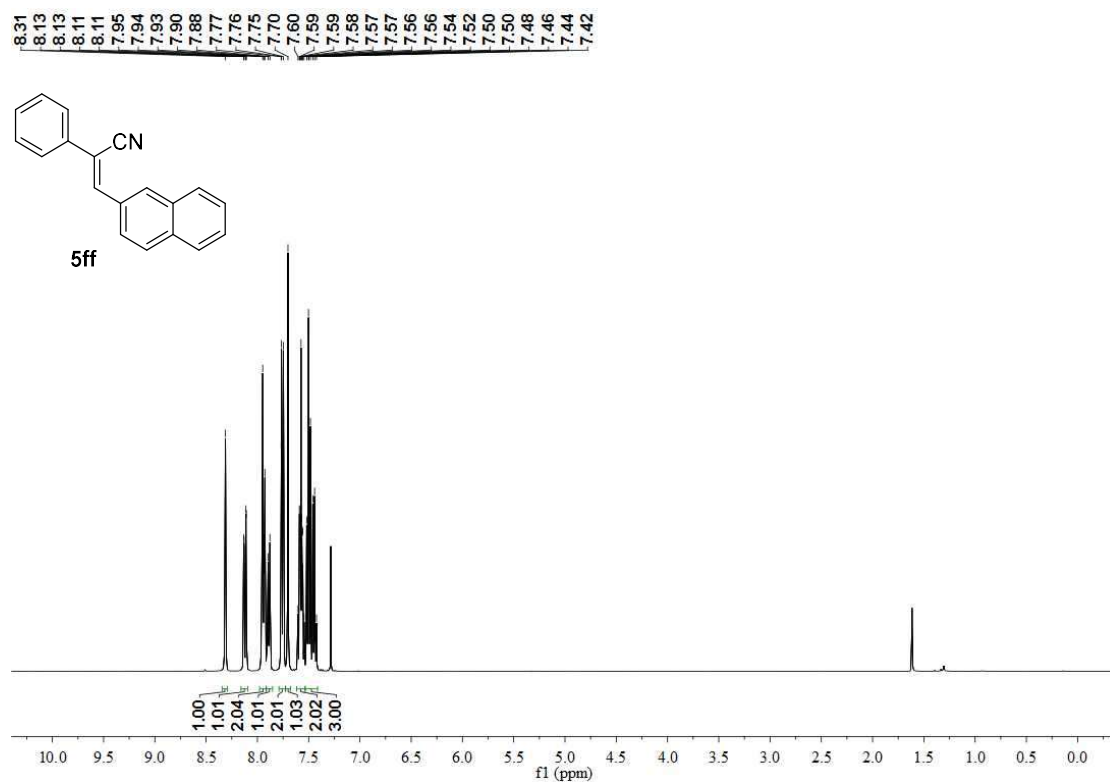
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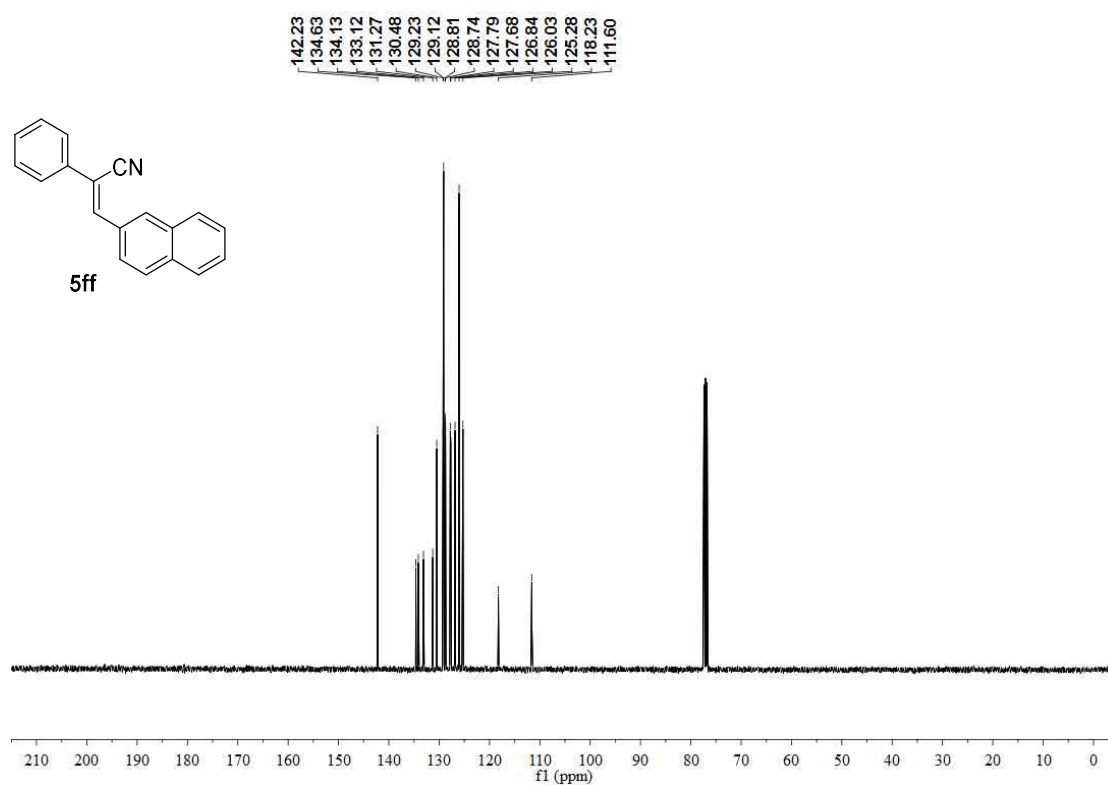
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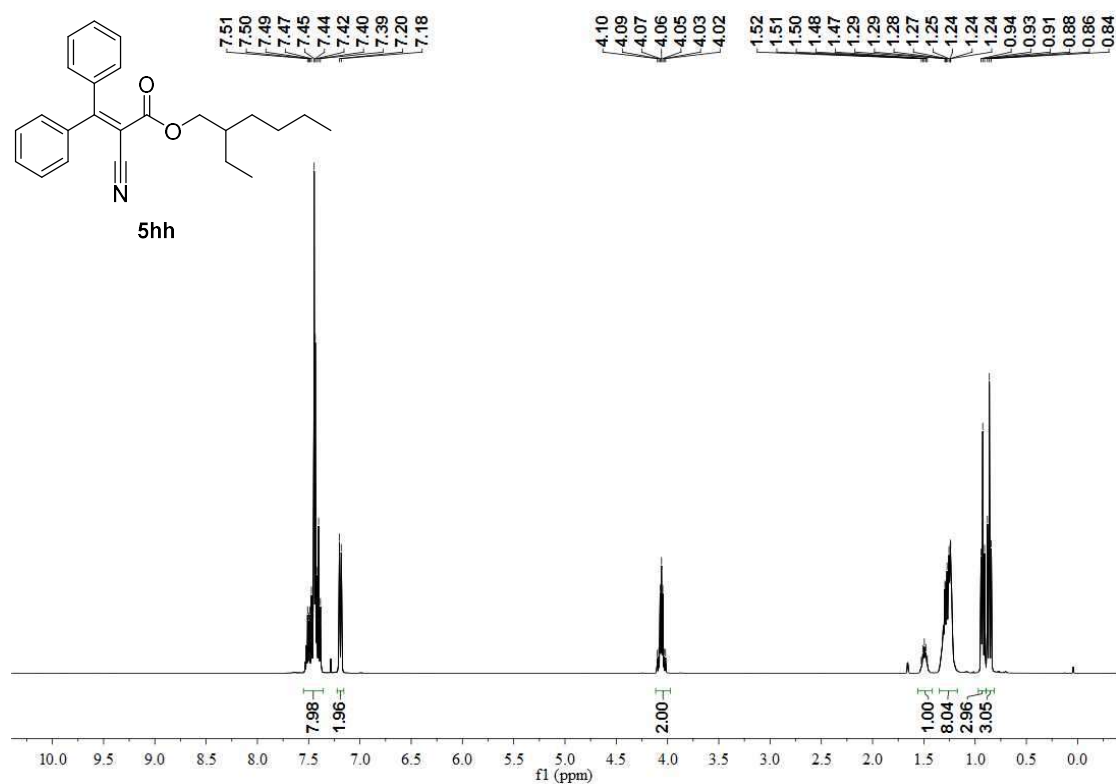
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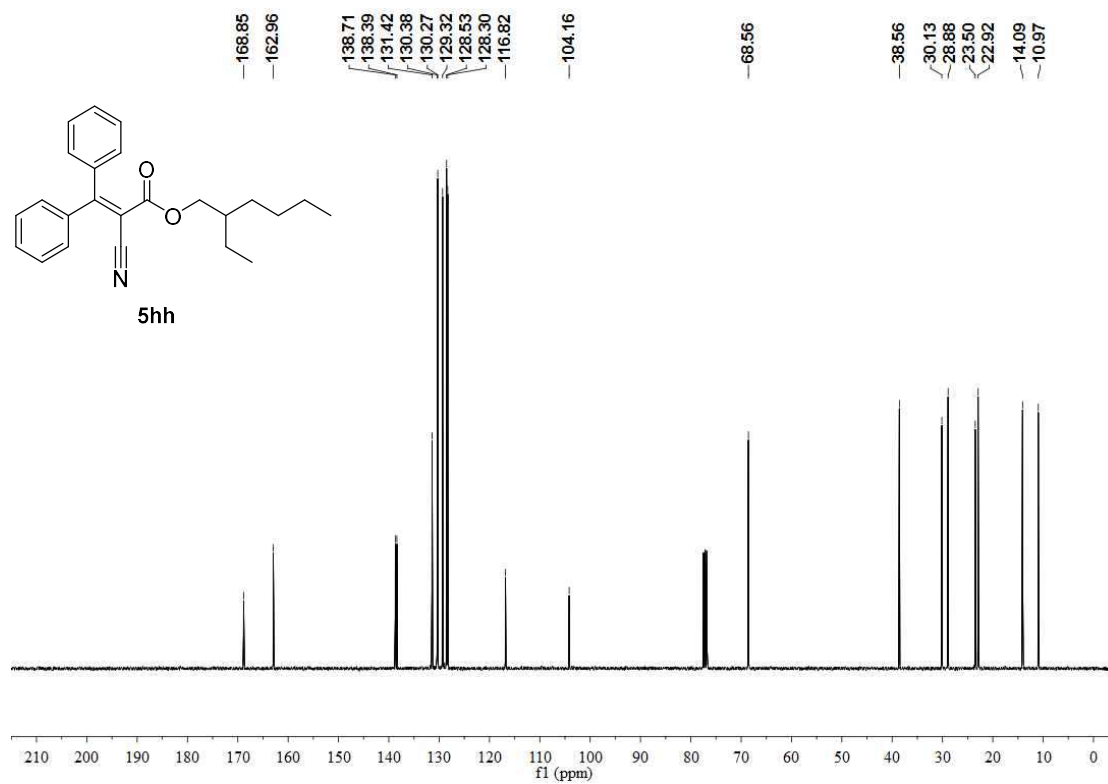
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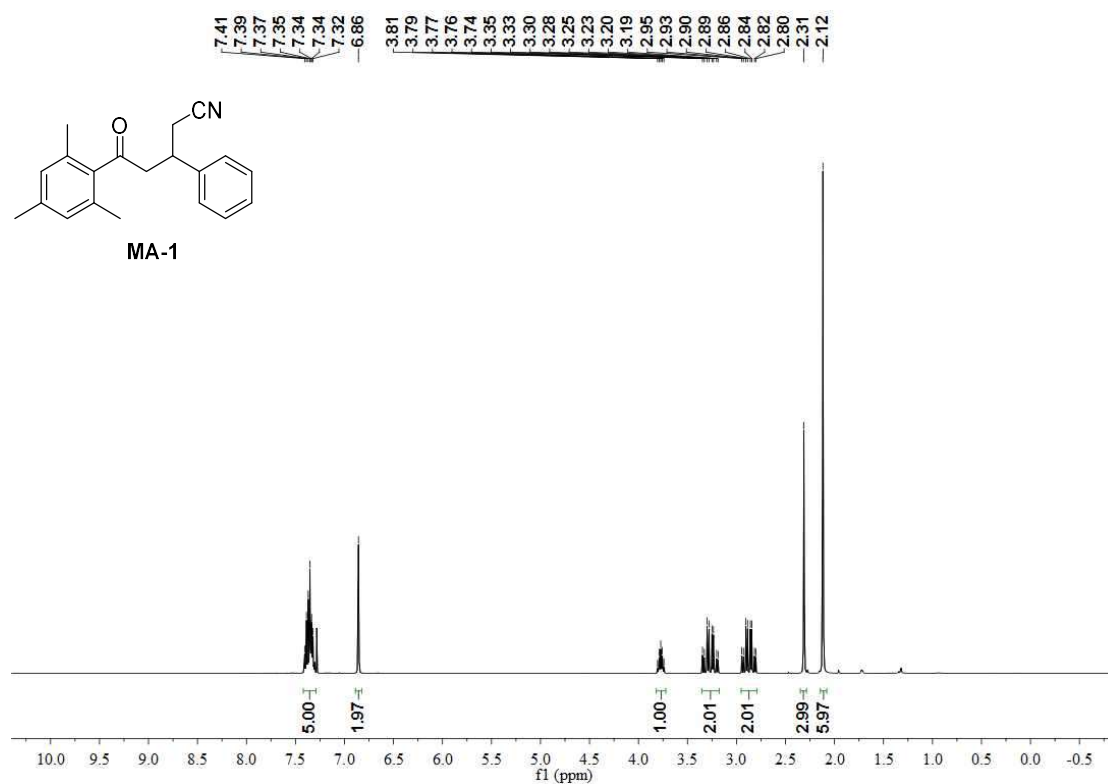
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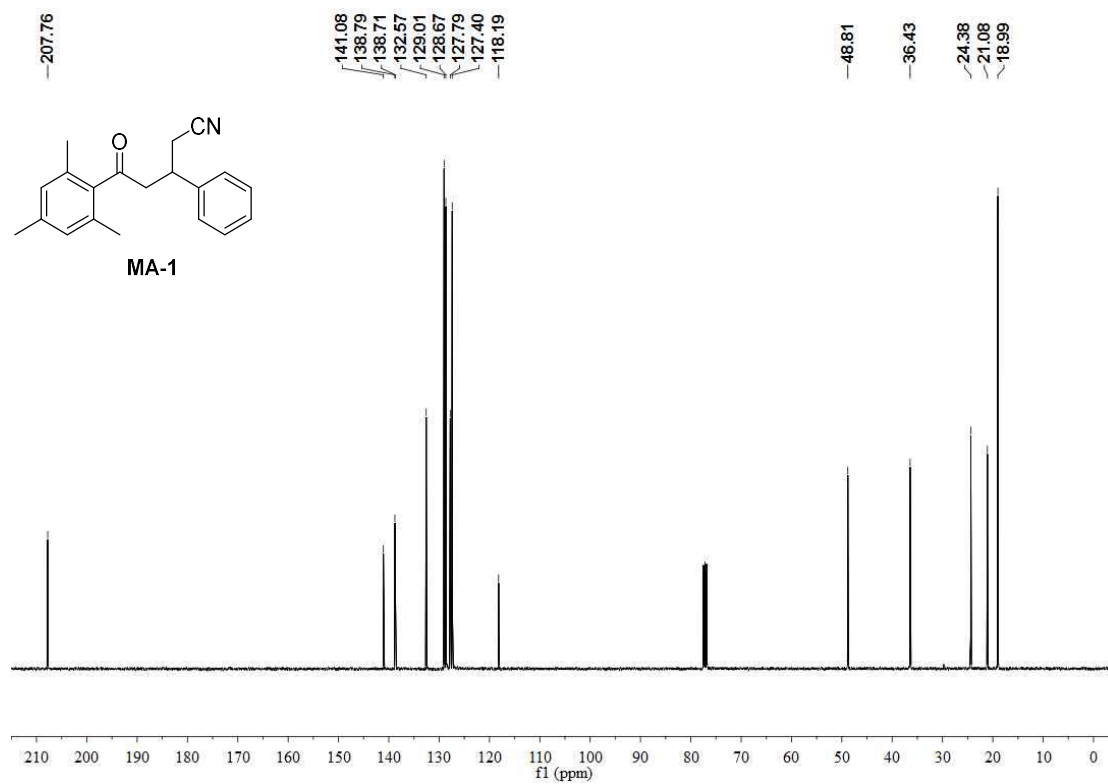
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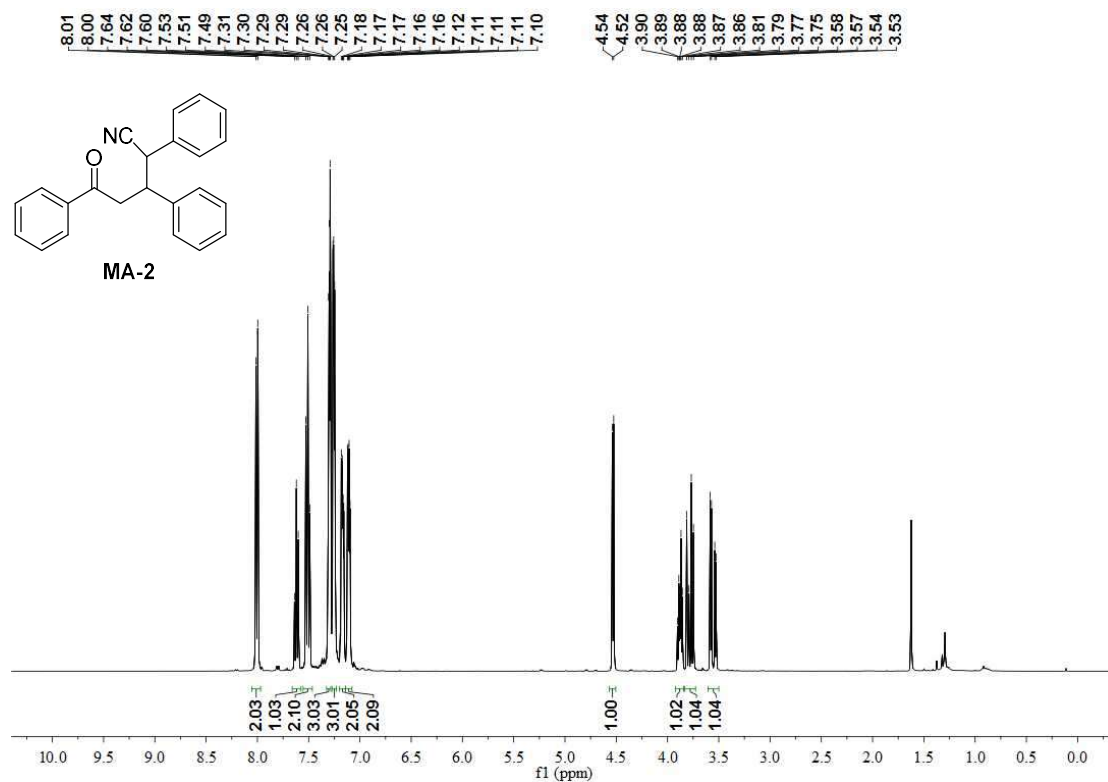
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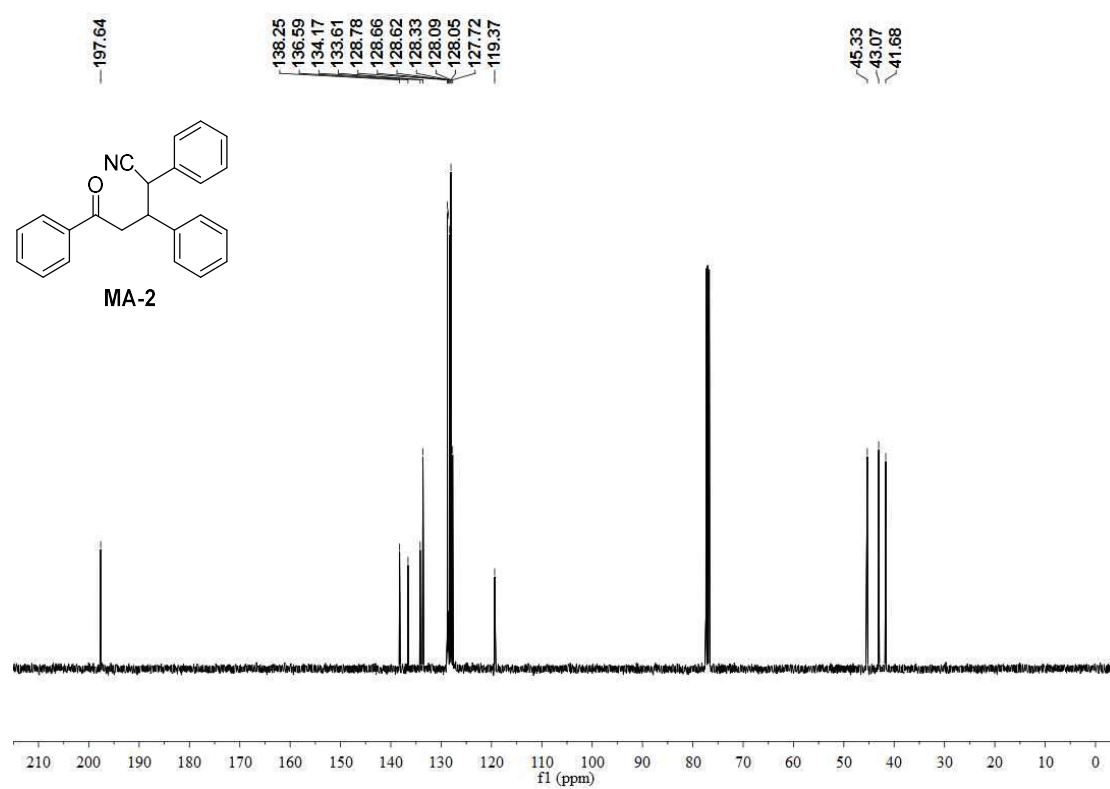
^{13}C NMR



^1H NMR



¹³C NMR



VII. References

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