

Visible Light-Promoted Pd-catalyzed Regioselective 1,4-Dicarbofunctionalization of 1,3-Butadiene with Unactivated Bromides and Malononitriles

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Table of Contents

1.	General Information.....	3
2.	Optimization of Reaction Conditions.....	4
3.	Synthesis of the Substrates and Unsuccessful Examples.....	5
4.	Typical Reaction Setup.....	5
5.	General Experimental Procedure A (GPA)	6
6.	General Experimental Procedure B (GPB)	6
7.	Analytical Data for the Products.....	7
8.	Copies of NMR Spectra of the Products.....	28

1. General Information

NMR spectra were recorded on a Bruker-400/500 MHz spectrometer. Chemical shifts (δ) are given in ppm relative to TMS. The residual solvent signals were used as references and the chemical shifts converted to the TMS scale (CDCl_3 : $\delta\text{H} = 7.26$ ppm, $\delta\text{C} = 77.16$ ppm). The high resolution mass spectra were recorded on a Thermo LTQ Orbitrap XL (ESI-TOF) or a P-SIMS-Gly of Bruker Daltonics Inc (EI+). The reaction mixture is irradiated in a photoreactor (Commercial supplier: Bibby Scientific Ltd., website: <http://www.rogertech.cn/cpzszong.asp>). Light was emitted by OHSP-350UV- 455 nm lamps, 0.5 cm away. To maintain the temperature, the reactor has a self-cooling system.

Materials: Starting-materials were purchased from commercial suppliers (Aldrich, Alfa, TCI, Adamas-beta, Energy chemical) and used as supplied unless otherwise stated. All solvents were purified and dried according to standard methods prior to use, unless stated otherwise. The oxindoles were synthesized according to relevant literature.

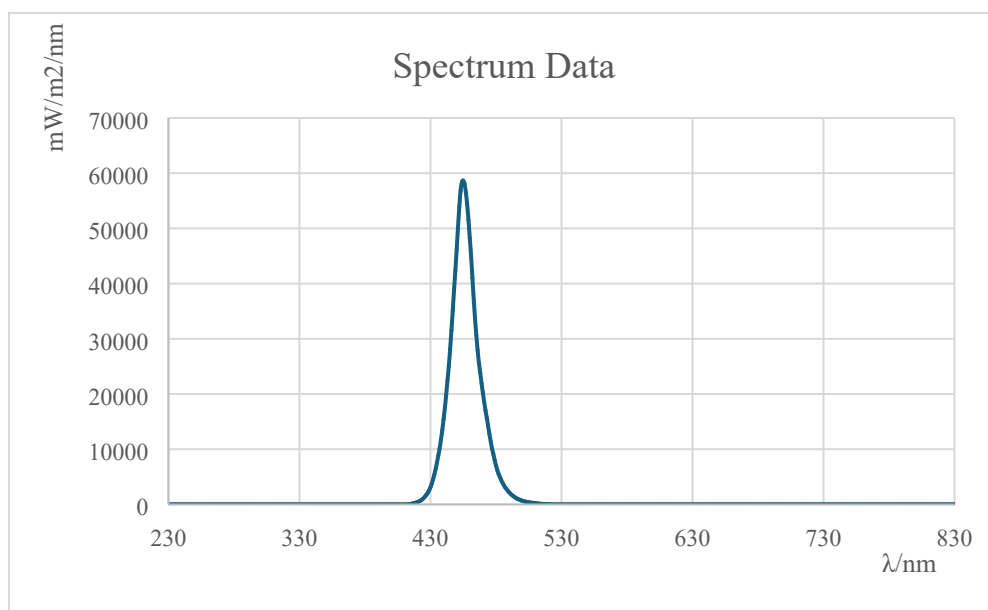
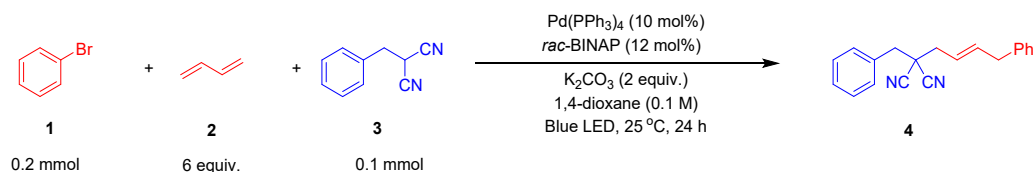


Figure S1. Spectrum Data of Blue LED

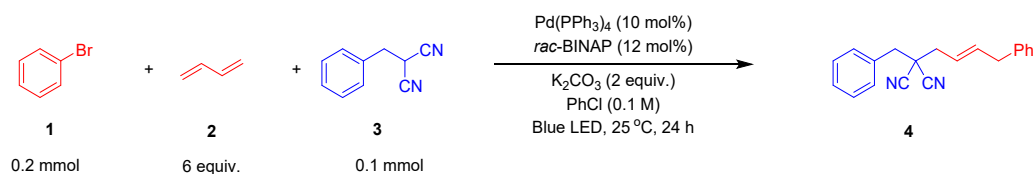
2. Optimization of Reaction Conditions

Table S1. Optimization of reaction conditions



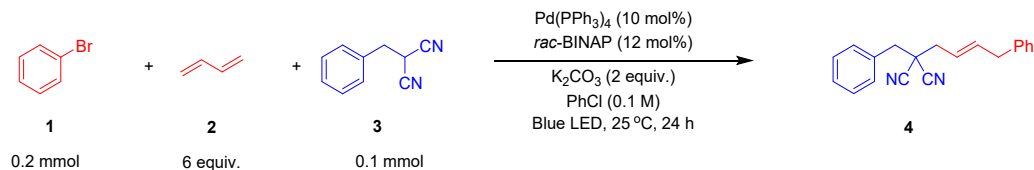
Entry	Variation	Yield ^b	r.r
1	None	35	>20:1
2	PhH instead of 1,4-dioxane	51	>20:1
3	PhCl instead of 1,4-dioxane	89(85)	>20:1
4	PhCF ₃ instead of 1,4-dioxane	70	>20:1
5	PhCN instead of 1,4-dioxane	53	>20:1

Table S2. Optimization of reaction conditions



Entry	Variation	Yield	r.r
1	DPEPhos instead of <i>rac</i> -BINAP	60	>20:1
2	Xantphos instead of <i>rac</i> -BINAP	66	>20:1
3	SEGphos instead of <i>rac</i> -BINAP	55	>20:1
4	Cs ₂ CO ₃ instead of K ₂ CO ₃	65	>20:1
5	KHCO ₃ instead of K ₂ CO ₃	60	>20:1
6	DIPEA instead of K ₂ CO ₃	80	>20:1
7	Pd(PPh ₃) ₄ (5 mol %)/ <i>rac</i> -BINAP (6 mol %)	70	>20:1

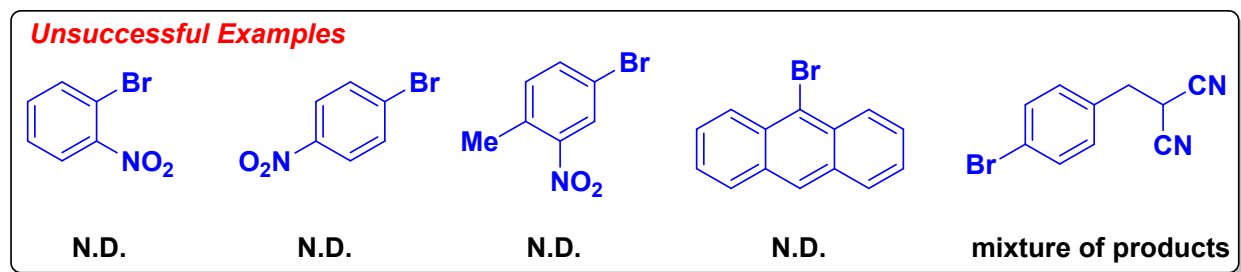
Table S3. Control Experiments



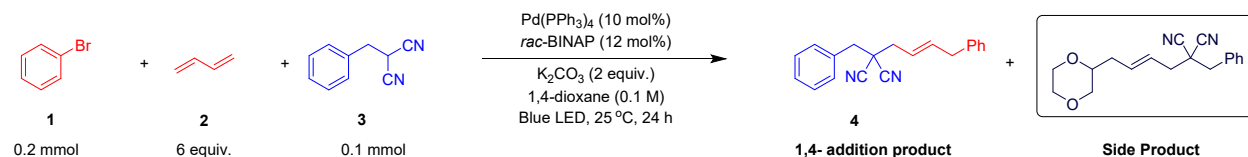
Entry	Conditions	Result
1	without $\text{Pd(PPh}_3)_4$	N.D.
2	without blue LEDs	N.D.
3	Ir(ppy)_3 instead of $\text{Pd(PPh}_3)_4$	N.D.
4	100 °C instead of blue LEDs	traces

3. Synthesis of the Substrates and Unsuccessful examples

All the malononitrile substrates were synthesized according to the relevant literature.¹ All the alkyl and aryl bromides are commercially available and few of them were synthesized following suitable literature.



The three-component side product of bromobenzene, 1,3-butadiene and 1,4-dioxane



4. Typical Reaction Setup

The reaction was performed in a commercially available photoreactor system (Bibby Scientific Ltd.) equipped with a self-cooling mechanism. The system comprises three main components: a constant-temperature bath, a circulating pump, and the photoreactor unit. The temperature of the cooling liquid is maintained by the thermostatic bath, which continuously circulates the liquid through the metal chamber of the photoreactor. This circulation efficiently removes excess heat and ensures a stable reaction temperature throughout the process. As the design is fixed, the

distance between the reaction vessel and the light source is not adjustable. All equipment used is standard and readily accessible. A schematic representation of the setup is provided in **Figure S2**.



Figure S2. Reactor temperature, pump, and photoreactor

The whole equipment as shown in **Figure S3a**. The typical reaction setup was shown in **Figure S3b**.

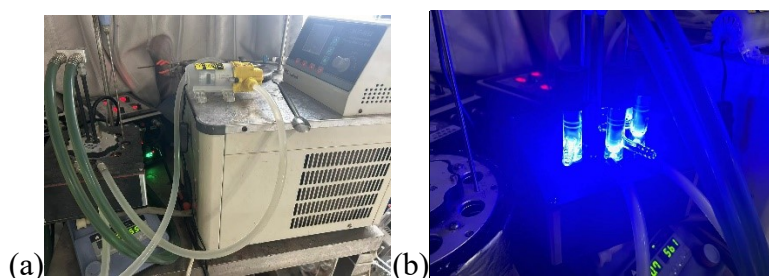


Figure S3. (a) Whole equipment. (b) Typical reaction setup

5. General Experimental Procedure A (GPA)

To a flame-dried and N₂-purged Schlenk tube (10 mL) were added Pd(PPh₃)₄ (0.01 mmol, 11.6 mg), *rac*-BINAP (0.012 mmol, 7.5 mg), malononitrile substrate (0.1 mmol) and K₂CO₃ (0.2 mmol, 27.6 mg). The Schlenk tube was then evacuated and filled with N₂. This cycle was repeated three times followed by addition of the chlorobenzene solvent (1.0 mL), aryl bromide (0.2 mmol) and 1,3-butadiene (0.6 mmol) sequentially. The mixture was stirred at 25 °C, then irradiated under blue LED (455 nm). After 24 hours the mixture was concentrated in vacuo. The residue was purified through flash chromatography to provide the product.

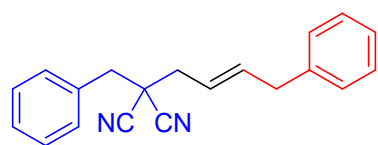
6. General Experimental Procedure B (GPB)

To a flame-dried and N₂-purged Schlenk tube (10 mL) were added Pd(PPh₃)₄ (0.01 mmol, 11.6 mg), *rac*-BINAP (0.012 mmol, 7.5 mg), malononitrile substrate (0.1 mmol) and K₂CO₃ (0.2 mmol, 27.6 mg). The Schlenk tube was then evacuated and filled with N₂. This cycle was repeated three

times followed by the addition of the 1,4-dioxane solvent (1.0 mL), alkyl bromide (0.2 mmol) and 1,3-butadiene (0.6 mmol), sequentially. The mixture was stirred at 25 °C, then irradiated under blue LED (455 nm). After 14 hours the mixture was concentrated in vacuo. The residue was purified through flash chromatography to provide the desired product.

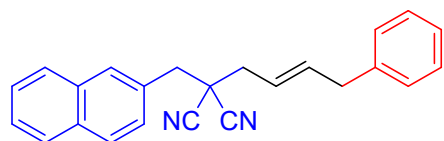
7. Analytical Data for the Products

(*E*)-2-benzyl-2-(4-phenylbut-2-en-1-yl)malononitrile (4)



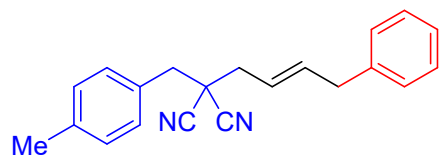
¹H NMR (400 MHz, Chloroform-*d*) δ 7.42 – 7.29 (m, 7H), 7.23 – 7.18 (m, 3H), 5.97 (dt, *J* = 14.9, 6.9, 1.2 Hz, 1H), 5.65 (dt, *J* = 14.9, 7.3, 1.5 Hz, 1H), 3.46 (dd, *J* = 6.9, 1.5 Hz, 2H), 3.17 (s, 2H), 2.67 (dd, *J* = 7.3, 1.1 Hz, 2H). **¹³C NMR** (126 MHz, CDCl₃) δ 139.19, 138.49, 132.01, 130.26, 129.34, 129.20, 129.04, 128.84, 128.65, 128.58, 128.39, 126.47, 121.49, 115.11, 42.67, 40.43, 39.65, 38.98. **HRMS** (ESI) *m/z* (M+Na)⁺ calculated for C₂₀H₁₈N₂Na: 309.1368, found: 309.1370.

(*E*)-2-(naphthalen-2-ylmethyl)-2-(4-phenylbut-2-en-1-yl)malononitrile (5)



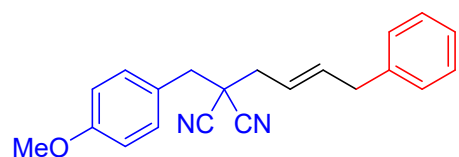
¹H NMR (400 MHz, Chloroform-*d*) δ 7.92 – 7.78 (m, 4H), 7.54 – 7.43 (m, 3H), 7.35 – 7.26 (m, 2H), 7.21 (dd, *J* = 9.3, 7.5 Hz, 3H), 5.97 (dt, *J* = 14.9, 6.8, 1.2 Hz, 1H), 5.67 (dt, *J* = 15.0, 7.3, 1.5 Hz, 1H), 3.46 (d, *J* = 6.9 Hz, 2H), 3.34 (s, 2H), 2.70 (dd, *J* = 7.4, 1.2 Hz, 2H). **¹³C NMR** (101 MHz, CDCl₃) δ 139.19, 138.55, 133.25, 133.19, 129.74, 129.41, 128.86, 128.65, 128.58, 128.38, 128.05, 127.78, 127.47, 126.72, 126.66, 126.46, 121.46, 115.17, 42.78, 40.45, 39.56, 38.99. **HRMS** (ESI) *m/z* (M+Na)⁺ calculated for C₂₄H₂₀N₂Na: 359.1524, found: 359.1525.

(*E*)-2-(4-methylbenzyl)-2-(4-phenylbut-2-en-1-yl)malononitrile (6)



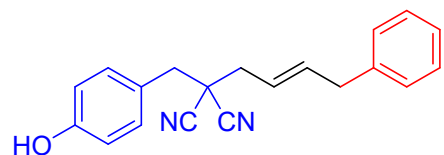
¹H NMR (400 MHz, Chloroform-*d*) δ 7.33 – 7.24 (m, 3H), 7.23 – 7.17 (m, 6H), 5.96 (dt, *J* = 15.0, 6.9, 1.2 Hz, 1H), 5.70 – 5.59 (m, 1H), 3.45 (dd, *J* = 6.8, 1.4 Hz, 2H), 3.14 (s, 2H), 2.65 (dd, *J* = 7.3, 1.1 Hz, 2H), 2.62 (d, *J* = 1.1 Hz, 2H), 2.35 (s, 3H). **¹³C NMR** (126 MHz, CDCl₃) δ 139.22, 138.70, 138.37, 130.11, 129.70, 128.94, 128.64, 128.57, 128.39, 126.44, 121.57, 115.21, 42.35, 40.35, 39.75, 38.97, 21.22. **HRMS** (ESI) *m/z* (M+Na)⁺ calculated for C₂₁H₂₀N₂Na: 323.1524, found: 323.1531.

(*E*)-2-(4-methoxybenzyl)-2-(4-phenylbut-2-en-1-yl)malononitrile (7)



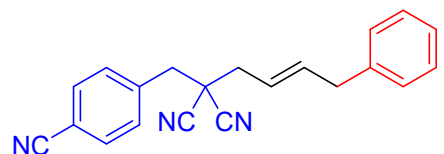
¹H NMR (400 MHz, Chloroform-*d*) δ 7.37 – 7.16 (m, 7H), 6.95 – 6.86 (m, 2H), 6.03 – 5.91 (m, 1H), 5.64 (dtt, J = 14.9, 7.3, 1.5 Hz, 1H), 3.80 (s, 3H), 3.46 (d, J = 6.8 Hz, 2H), 3.13 (s, 2H), 2.65 (dd, J = 7.3, 1.2 Hz, 2H). **¹³C NMR** (126 MHz, CDCl₃) δ 159.96, 139.22, 138.34, 131.41, 128.64, 128.57, 126.44, 123.96, 121.58, 115.24, 114.39, 55.33, 42.02, 40.26, 39.92, 38.97. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₁H₂₁N₂O: 317.1654, found: 317.1656.

(E)-2-(4-hydroxybenzyl)-2-(4-phenylbut-2-en-1-yl)malononitrile (8)



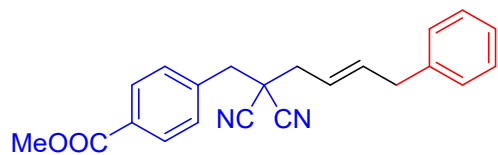
¹H NMR (400 MHz, Chloroform-*d*) δ 7.31 (m, 2H), 7.25 – 7.16 (m, 5H), 6.88 – 6.78 (m, 2H), 5.98 (dtt, J = 15.0, 6.9, 1.2 Hz, 1H), 5.65 (dtt, J = 15.0, 7.3, 1.5 Hz, 1H), 5.14 (s, 1H), 3.46 (d, J = 6.9 Hz, 2H), 3.12 (s, 2H), 2.67 (dq, J = 7.3, 1.0 Hz, 2H). **¹³C NMR** (101 MHz, CDCl₃) δ 156.12, 139.16, 138.42, 131.58, 128.62, 128.54, 126.43, 124.05, 121.46, 115.88, 115.17, 42.02, 40.28, 39.95, 38.94. **HRMS** (ESI) m/z (M+Na)⁺ calculated for C₂₀H₁₈N₂ONa: 325.1317, found: 325.1319.

(E)-2-(4-cyanobenzyl)-2-(4-phenylbut-2-en-1-yl)malononitrile (9)



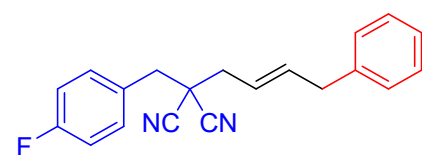
¹H NMR (400 MHz, Chloroform-*d*) δ 7.76 – 7.65 (m, 2H), 7.52 – 7.47 (m, 2H), 7.35 – 7.28 (m, 2H), 7.25 – 7.14 (m, 3H), 6.07 – 5.96 (m, 1H), 5.64 (dtt, J = 14.9, 7.3, 1.6 Hz, 1H), 3.48 (d, J = 6.8 Hz, 2H), 3.21 (s, 2H), 2.81 – 2.67 (m, 2H). **¹³C NMR** (126 MHz, CDCl₃) δ 139.20, 138.95, 137.21, 132.90, 132.78, 131.02, 128.69, 128.53, 126.54, 120.84, 118.17, 114.49, 113.09, 42.41, 40.75, 39.29, 38.96. **HRMS** (ESI) m/z (M+Na)⁺ calculated for C₂₁H₁₇N₃Na: 334.1320, found: 334.1322.

Methyl (E)-4-(2,2-dicyano-6-phenylhex-4-en-1-yl)benzoate (10)



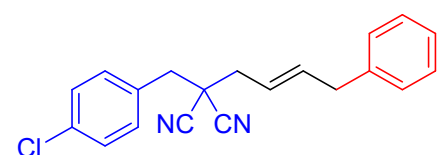
¹H NMR (400 MHz, Chloroform-*d*) δ 8.14 – 8.01 (m, 2H), 7.49 – 7.37 (m, 2H), 7.36 – 7.15 (m, 5H), 5.99 (dtt, J = 14.9, 6.9, 1.1 Hz, 1H), 5.65 (dtt, J = 14.8, 7.3, 1.5 Hz, 1H), 3.93 (s, 3H), 3.47 (d, J = 6.8 Hz, 2H), 3.22 (s, 2H), 2.70 (dd, J = 7.4, 1.1 Hz, 2H). **¹³C NMR** (126 MHz, CDCl₃) δ 166.49, 139.05, 138.85, 136.90, 130.70, 130.31, 130.24, 129.97, 129.91, 128.65, 128.54, 126.48, 121.14, 114.77, 52.33, 42.41, 40.60, 39.32, 38.96. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₂H₂₁N₂O₂: 345.1603, found: 345.1605.

(E)-2-(4-fluorobenzyl)-2-(4-phenylbut-2-en-1-yl)malononitrile (11)



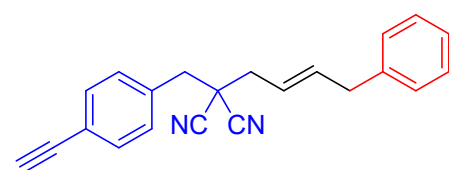
^1H NMR (400 MHz, Chloroform-*d*) δ 7.33 (m, 4H), 7.27 – 7.18 (m, 3H), 7.15 – 7.05 (m, 2H), 6.00 (m, 1H), 5.66 (dt, J = 14.9, 7.3, 1.5 Hz, 1H), 3.48 (d, J = 6.8 Hz, 2H), 3.16 (s, 2H), 2.70 (d, J = 7.3 Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 163.04 (d, J = 248.2 Hz), 139.12, 138.66, 132.00, 131.93, 128.66, 128.56, 127.87, 127.85, 126.48, 121.29, 116.17, 116.00, 114.96, 41.88, 40.44, 39.77 (d, J = 1.9 Hz), 38.96. ^{19}F NMR (376 MHz, CDCl_3) δ -112.67. HRMS (ESI) m/z ($\text{M}+\text{Na}$) $^+$ calculated for $\text{C}_{20}\text{H}_{17}\text{FN}_2\text{Na}$: 327.1273, found: 327.1274.

(E)-2-(4-chlorobenzyl)-2-(4-phenylbut-2-en-1-yl)malononitrile (12)



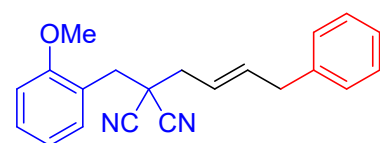
^1H NMR (400 MHz, Chloroform-*d*) δ 7.41 – 7.34 (m, 2H), 7.34 – 7.26 (m, 4H), 7.26 – 7.16 (m, 3H), 5.99 (dt, J = 14.9, 6.9, 1.2 Hz, 1H), 5.71 – 5.58 (m, 1H), 3.47 (d, J = 6.8 Hz, 2H), 3.14 (s, 2H), 2.69 (dd, J = 7.2, 1.1 Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 139.08, 138.75, 135.07, 131.53, 130.46, 129.28, 128.66, 128.55, 126.49, 121.20, 114.86, 41.98, 40.48, 39.56, 38.96. HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calculated for $\text{C}_{20}\text{H}_{18}\text{ClN}_2$: 321.1159, found: 321.1162.

(E)-2-(4-ethynylbenzyl)-2-(4-phenylbut-2-en-1-yl)malononitrile (13)



^1H NMR (400 MHz, Chloroform-*d*) δ 7.57 – 7.49 (m, 2H), 7.37 – 7.27 (m, 4H), 7.27 – 7.17 (m, 3H), 6.00 (dt, J = 14.9, 6.9, 1.2 Hz, 1H), 5.65 (dt, J = 15.0, 7.3, 1.5 Hz, 1H), 3.48 (dd, J = 6.8, 1.4 Hz, 2H), 3.18 (s, 2H), 3.14 (s, 1H), 2.69 (dd, J = 7.2, 1.1 Hz, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 139.10, 138.71, 132.71, 132.62, 130.22, 128.65, 128.55, 126.48, 122.86, 121.25, 114.88, 82.84, 42.42, 40.47, 39.42, 38.95. HRMS (ESI) m/z ($\text{M}+\text{Na}$) $^+$ calculated for $\text{C}_{22}\text{H}_{18}\text{N}_2\text{Na}$: 333.1368, found: 333.1369.

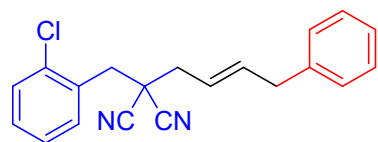
(E)-2-(2-methoxybenzyl)-2-(4-phenylbut-2-en-1-yl)malononitrile (14)



^1H NMR (400 MHz, Chloroform-*d*) δ 7.40 – 7.28 (m, 4H), 7.23 (td, J = 5.3, 2.8 Hz, 3H), 6.99 (td, J = 7.5, 1.1 Hz, 1H), 6.94 (d, J = 8.2 Hz, 1H), 5.98 (dt, J = 15.0, 6.8, 1.2 Hz, 1H), 5.68 (dt, J = 14.9, 7.3, 1.5 Hz, 1H), 3.85 (s, 3H), 3.48 (d, J = 6.8 Hz, 2H), 3.31 (s, 2H), 2.69 (dd, J = 7.3, 1.1 Hz, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.83, 139.34, 137.98, 131.80, 130.21, 128.59, 126.38, 122.07,

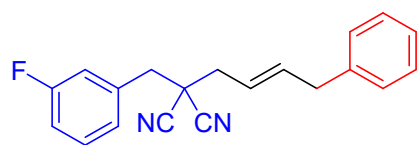
120.81, 120.75, 115.48, 110.92, 55.31, 40.41, 39.18, 38.98, 36.13. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₁H₂₁N₂O: 317.1654, found: 317.1659.

(E)-2-(2-chlorobenzyl)-2-(4-phenylbut-2-en-1-yl)malononitrile (15)



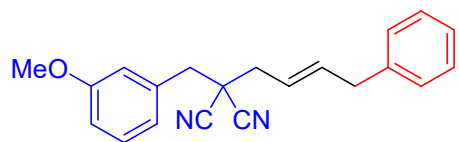
¹H NMR (400 MHz, Chloroform-*d*) δ 7.57 – 7.49 (m, 1H), 7.49 – 7.41 (m, 1H), 7.37 – 7.24 (m, 4H), 7.21 (td, J = 7.1, 6.6, 1.4 Hz, 3H), 6.00 (dtt, J = 14.9, 6.8, 1.2 Hz, 1H), 5.66 (dtt, J = 15.0, 7.3, 1.5 Hz, 1H), 3.46 (d, J = 7.3 Hz, 2H), 3.44 (s, 2H), 2.74 (dq, J = 7.4, 1.0 Hz, 2H). **¹³C NMR** (101 MHz, CDCl₃) δ 139.13, 138.60, 135.13, 131.90, 130.28, 130.25, 130.22, 129.31, 128.62, 128.59, 128.37, 127.48, 126.43, 121.47, 114.92, 40.52, 38.99, 38.95, 38.76. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₀H₁₈ClN₂: 321.1159, found: 321.1163.

(E)-2-(3-fluorobenzyl)-2-(4-phenylbut-2-en-1-yl)malononitrile (16)



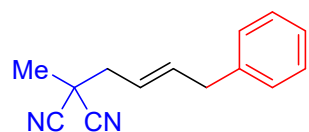
¹H NMR (400 MHz, Chloroform-*d*) δ 7.42 – 7.32 (m, 1H), 7.36 – 7.25 (m, 2H), 7.25 – 7.18 (m, 3H), 7.15 (dt, J = 7.7, 1.3 Hz, 1H), 7.09 (dtt, J = 8.8, 7.6, 1.5 Hz, 2H), 5.99 (dtt, J = 15.0, 6.9, 1.2 Hz, 1H), 5.65 (dtt, J = 14.9, 7.3, 1.5 Hz, 1H), 3.47 (d, J = 6.9 Hz, 2H), 3.16 (s, 2H), 2.69 (dd, J = 7.3, 1.1 Hz, 2H). **¹³C NMR** (126 MHz, CDCl₃) δ 162.80 (d, J = 247.7 Hz), 139.10, 138.78, 134.29, 134.23, 130.70, 130.63, 128.66, 128.56, 128.37, 126.49, 125.98, 125.95, 121.21, 117.35, 117.17, 116.06, 115.89, 114.85, 42.19 (d, J = 1.9 Hz), 40.53, 39.41, 38.96. **¹⁹F NMR** (376 MHz, Chloroform-*d*) δ -111.61 (td, J = 8.9, 6.0 Hz). **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₀H₁₈FN₂: 305.1454, found: 305.1465.

(E)-2-(3-methoxybenzyl)-2-(4-phenylbut-2-en-1-yl)malononitrile (17)



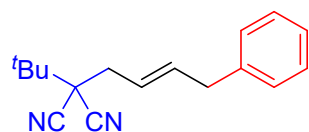
¹H NMR (400 MHz, Chloroform-*d*) δ 7.45 – 7.26 (m, 4H), 7.26 – 6.99 (m, 4H), 7.03 – 6.87 (m, 4H), 5.98 (dtt, J = 15.0, 6.8, 1.2 Hz, 1H), 5.65 (dtt, J = 14.9, 7.3, 1.6 Hz, 1H), 3.82 (q, J = 1.8, 1.3 Hz, 1H), 3.81 (s, 3H), 3.47 (d, J = 6.8 Hz, 2H), 3.16 (s, 2H), 2.67 (dd, J = 7.2, 1.1 Hz, 2H). **¹³C NMR** (101 MHz, CDCl₃) δ 159.88, 139.16, 138.47, 133.32, 130.03, 128.62, 128.55, 128.50, 126.43, 122.44, 121.47, 115.70, 115.14, 114.39, 55.30, 42.67, 40.40, 39.45, 38.96. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₁H₂₁N₂O: 317.1654, found: 317.1667.

(E)-2-methyl-2-(4-phenylbut-2-en-1-yl)malononitrile (18)



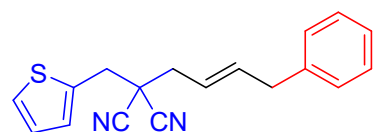
¹H NMR (400 MHz, Chloroform-*d*) δ 7.35 – 7.28 (m, 2H), 7.25 – 7.16 (m, 3H), 5.97 (dt, J = 14.9, 6.8, 1.2 Hz, 1H), 5.60 (dt, J = 14.9, 7.3, 1.5 Hz, 1H), 3.45 (d, J = 6.8 Hz, 2H), 2.65 (dd, J = 7.37, 1.06 Hz, 2H), 1.76 (s, 3H). **¹³C NMR** (101 MHz, CDCl₃) δ 139.11, 138.50, 131.21, 129.30, 128.63, 128.54, 128.36, 126.44, 125.91, 121.35, 115.99, 41.94, 38.91, 31.97, 24.04. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₁₄H₁₅N₂: 211.1235, found: 211.1245.

(E)-2-(tert-butyl)-2-(4-phenylbut-2-en-1-yl)malononitrile (19)



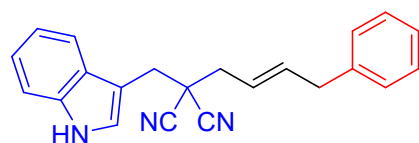
¹H NMR (400 MHz, Chloroform-*d*) δ 7.34 – 7.28 (m, 2H), 7.24 – 7.18 (m, 3H), 5.97 (dt, J = 14.8, 6.8, 1.2 Hz, 1H), 5.70 (dt, J = 15.0, 7.2, 1.5 Hz, 1H), 3.47 (dd, J = 6.7, 1.5 Hz, 2H), 2.56 (dq, J = 7.3, 0.9 Hz, 2H), 1.25 (s, 9H). **¹³C NMR** (126 MHz, CDCl₃) δ 139.36, 137.59, 128.60, 128.56, 126.34, 122.76, 115.06, 49.27, 38.93, 38.14, 35.68, 25.82. **HRMS** (ESI) m/z (M+Na)⁺ calculated for C₁₇H₂₀N₂Na: 275.1524, found: 275.1522.

(E)-2-(4-phenylbut-2-en-1-yl)-2-(thiophen-2-ylmethyl)malononitrile (20)



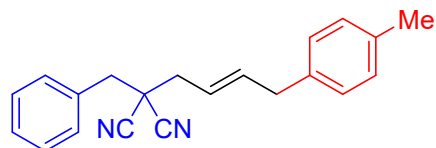
¹H NMR (500 MHz, Chloroform-*d*) δ 7.36 – 7.28 (m, 3H), 7.28 – 7.18 (m, 3H), 7.13 (dd, J = 3.7, 1.1 Hz, 1H), 7.04 (dd, J = 5.2, 3.5 Hz, 1H), 5.99 (dt, J = 15.0, 6.9, 1.3 Hz, 1H), 5.64 (dt, J = 14.9, 7.3, 1.6 Hz, 1H), 3.46 (d, J = 6.9 Hz, 2H), 3.43 (s, 2H), 2.69 (dd, J = 7.3, 1.1 Hz, 2H). **¹³C NMR** (126 MHz, CDCl₃) δ 139.11, 138.75, 133.84, 133.69, 132.71, 129.22, 128.65, 128.56, 127.65, 126.70, 126.47, 121.21, 115.00, 39.85, 39.63, 38.97, 36.83. **HRMS** (ESI) m/z (M+Na)⁺ calculated for C₁₈H₁₆N₂SNa: 315.0932, found: 315.0930.

(E)-2-((1H-indol-3-yl)methyl)-2-(4-phenylbut-2-en-1-yl)malononitrile (21)



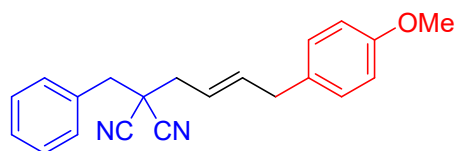
¹H NMR (500 MHz, Chloroform-*d*) δ 8.27 (s, 1H), 7.59 (d, J = 7.9 Hz, 1H), 7.37 (d, J = 8.1 Hz, 1H), 7.32 – 7.13 (m, 9H), 5.91 (ddd, J = 15.1, 7.6, 6.3 Hz, 1H), 5.63 (dt, J = 14.9, 7.3, 1.5 Hz, 1H), 3.43 (m, 4H), 2.66 (d, J = 7.3 Hz, 2H). **¹³C NMR** (126 MHz, CDCl₃) δ 139.31, 138.12, 135.85, 128.63, 128.60, 128.42, 127.31, 126.43, 124.75, 122.74, 121.88, 120.34, 118.53, 115.95, 111.58, 106.76, 40.18, 40.01, 38.96, 33.28. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₂H₂₀N₃: 326.1657, found: 326.1665.

(E)-2-benzyl-2-(4-(p-tolyl)but-2-en-1-yl)malononitrile (22)



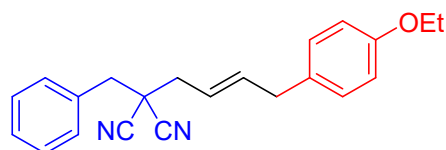
¹H NMR (400 MHz, Chloroform-*d*) δ 7.41 – 7.34 (m, 5H), 7.13 – 7.05 (m, 4H), 5.96 (dtt, J = 14.9, 6.9, 1.2 Hz, 1H), 5.69 – 5.58 (m, 1H), 3.42 (d, J = 6.8 Hz, 2H), 3.17 (s, 2H), 2.67 (dq, J = 7.3, 0.9 Hz, 2H), 2.32 (s, 3H). **¹³C NMR** (101 MHz, CDCl₃) δ 138.79, 136.07, 135.96, 132.01, 130.24, 129.31, 129.01, 128.81, 128.43, 121.17, 115.10, 42.65, 40.45, 39.62, 38.55, 21.05. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₁H₂₁N₂: 301.1705, found: 301.1692.

(*E*)-2-benzyl-2-(4-(4-methoxyphenyl)but-2-en-1-yl)malononitrile (23)



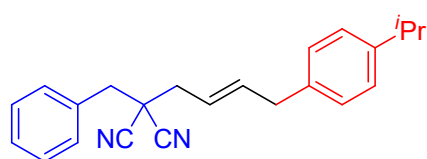
¹H NMR (400 MHz, Chloroform-*d*) δ 7.34 – 7.26 (m, 5H), 7.08 – 6.99 (m, 2H), 6.81 – 6.73 (m, 2H), 5.88 (dtt, J = 14.9, 6.7, 1.2 Hz, 1H), 5.55 (dtt, J = 14.9, 7.4, 1.5 Hz, 1H), 3.71 (s, 3H), 3.33 (d, J = 6.8 Hz, 2H), 3.10 (s, 2H), 2.65 – 2.55 (m, 2H). **¹³C NMR** (101 MHz, CDCl₃) δ 157.14, 137.86, 130.95, 130.11, 129.18, 128.45, 128.27, 128.11, 127.95, 127.79, 127.76, 120.03, 114.05, 112.96, 54.24, 41.60, 39.37, 38.60, 37.01. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₁H₂₁N₂O: 317.1654, found: 317.1656.

(*E*)-2-benzyl-2-(4-(4-ethoxyphenyl)but-2-en-1-yl)malononitrile (24)



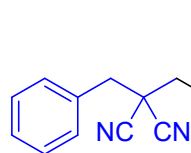
¹H NMR (500 MHz, Chloroform-*d*) δ 7.37 (qdt, J = 6.8, 4.6, 2.8 Hz, 5H), 7.12 – 7.04 (m, 2H), 6.87 – 6.79 (m, 2H), 6.00 – 5.90 (m, 1H), 5.62 (dtt, J = 14.9, 7.3, 1.6 Hz, 1H), 4.00 (q, J = 7.0 Hz, 2H), 3.39 (d, J = 6.8 Hz, 2H), 3.17 (s, 2H), 2.66 (d, J = 7.2 Hz, 2H), 1.39 (t, J = 7.0 Hz, 3H). **¹³C NMR** (126 MHz, CDCl₃) δ 157.59, 138.96, 132.04, 131.04, 130.24, 129.49, 129.05, 129.01, 128.87, 128.81, 121.06, 115.13, 115.05, 114.65, 114.44, 63.46, 42.66, 40.44, 39.64, 38.09, 14.90. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₂H₂₃N₂O: 331.1810, found: 331.1808.

(*E*)-2-benzyl-2-(4-(4-isopropylphenyl)but-2-en-1-yl)malononitrile (25)



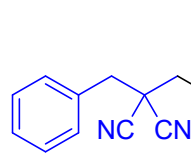
¹H NMR (400 MHz, Chloroform-*d*) δ 7.42 – 7.34 (m, 5H), 7.17 (d, J = 8.1 Hz, 2H), 7.15 – 7.09 (m, 2H), 5.97 (dtt, J = 15.1, 7.0, 1.1 Hz, 1H), 5.72 – 5.59 (m, 1H), 3.47 – 3.40 (m, 2H), 3.18 (s, 2H), 2.88 (p, J = 6.9 Hz, 1H), 2.72 – 2.65 (m, 2H), 1.23 (d, J = 6.9 Hz, 6H). **¹³C NMR** (101 MHz, CDCl₃) δ 147.04, 138.69, 136.49, 132.01, 130.24, 129.79, 129.14, 129.01, 128.82, 128.45, 126.67, 121.23, 115.12, 42.66, 40.46, 39.61, 38.56, 33.74, 24.07. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₃H₂₅N₂: 329.2018, found: 329.2014.

(E)-2-benzyl-2-(4-(4-chlorophenyl)but-2-en-1-yl)malononitrile (26)



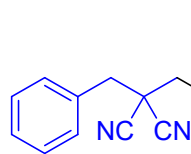
$^1\text{H NMR}$ (400 MHz, Chloroform-*d*) δ 7.41 – 7.35 (m, 5H), 7.29 – 7.24 (m, 2H), 7.17 – 7.09 (m, 2H), 6.00 – 5.88 (m, 1H), 5.65 (dtt, J = 14.9, 7.3, 1.5 Hz, 1H), 3.42 (d, J = 6.8 Hz, 2H), 3.19 (s, 2H), 2.67 (d, J = 7.3 Hz, 2H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 137.85, 137.59, 132.23, 131.90, 130.24, 129.92, 129.81, 129.14, 129.05, 128.88, 128.73, 122.01, 115.05, 42.76, 40.38, 39.68, 38.24. **HRMS** (ESI) m/z ($\text{M}+\text{Na}$) $^+$ calculated for $\text{C}_{20}\text{H}_{17}\text{ClN}_2\text{Na}$: 343.0978, found: 343.0980.

(E)-2-benzyl-2-(4-(4-cyanophenyl)but-2-en-1-yl)malononitrile (27)



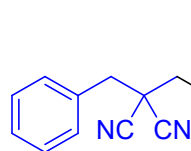
$^1\text{H NMR}$ (400 MHz, Chloroform-*d*) δ 7.64 – 7.56 (m, 2H), 7.41 – 7.34 (m, 5H), 7.34 – 7.28 (m, 2H), 5.94 (dtt, J = 14.9, 6.8, 1.2 Hz, 1H), 5.71 (dtt, J = 15.0, 7.3, 1.5 Hz, 1H), 3.52 (d, J = 6.8 Hz, 2H), 3.21 (s, 2H), 2.69 (dd, J = 7.3, 1.1 Hz, 2H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 144.71, 136.51, 132.44, 131.78, 130.22, 129.36, 129.07, 128.92, 123.16, 118.92, 114.98, 110.41, 42.86, 40.33, 39.71, 38.88. **HRMS** (ESI) m/z ($\text{M}+\text{H}$) $^+$ calculated for $\text{C}_{21}\text{H}_{19}\text{N}_3$: 312.1501, found: 312.1518.

(E)-2-benzyl-2-(4-(4-formylphenyl)but-2-en-1-yl)malononitrile (28)



$^1\text{H NMR}$ (400 MHz, Chloroform-*d*) δ 9.98 (s, 1H), 7.88 – 7.78 (m, 2H), 7.42 – 7.34 (m, 7H), 5.98 (dtt, J = 14.9, 6.8, 1.2 Hz, 1H), 5.71 (dtt, J = 15.0, 7.3, 1.5 Hz, 1H), 3.55 (d, J = 6.8 Hz, 2H), 3.21 (s, 2H), 2.69 (dd, J = 7.3, 1.1 Hz, 2H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 191.95, 146.39, 136.96, 134.95, 131.83, 130.22, 130.16, 129.24, 129.06, 128.89, 122.75, 115.01, 42.81, 40.36, 39.67, 39.01. **HRMS** (ESI) m/z ($\text{M}+\text{H}$) $^+$ calculated for $\text{C}_{21}\text{H}_{19}\text{N}_2\text{O}$: 315.1497, found: 315.1514.

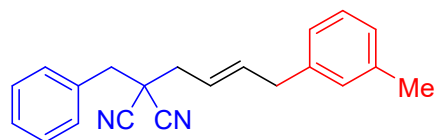
(E)-2-benzyl-2-(4-(4-(trifluoromethyl)phenyl)but-2-en-1-yl)malononitrile (29)



$^1\text{H NMR}$ (400 MHz, Chloroform-*d*) δ 7.56 (d, J = 8.0 Hz, 2H), 7.38 (dtt, J = 9.8, 6.3, 2.7 Hz, 6H), 7.31 (d, J = 8.0 Hz, 2H), 5.96 (ddd, J = 15.2, 7.6, 6.4 Hz, 1H), 5.75 – 5.65 (m, 1H), 3.52 (d, J = 6.9 Hz, 2H), 3.20 (s, 2H), 2.68 (d, J = 7.3 Hz, 2H). $^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 143.24, 137.20, 131.86, 130.22, 129.79, 129.13, 129.05, 128.89, 128.88, 125.55 (q, J = 3.8 Hz),

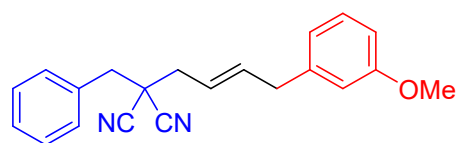
122.57, 115.03, 42.82, 40.37, 39.67, 38.65. **¹⁹F NMR** (376 MHz, CDCl₃) δ -62.34. **HRMS** (ESI) m/z (M+Na)⁺ calculated for C₂₁H₁₇F₃N₂Na: 377.1242, found: 377.1243.

(E)-2-benzyl-2-(4-(*m*-tolyl)but-2-en-1-yl)malononitrile (30)



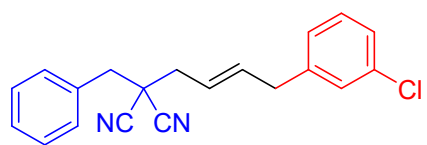
¹H NMR (400 MHz, Chloroform-*d*) δ 7.44 – 7.36 (m, 5H), 7.21 (t, *J* = 7.8 Hz, 1H), 7.09 – 6.98 (m, 3H), 6.05 – 5.93 (m, 1H), 5.67 (dt, *J* = 14.9, 7.3, 1.5 Hz, 1H), 3.44 (d, *J* = 6.9 Hz, 2H), 3.20 (s, 2H), 2.70 (d, *J* = 7.4 Hz, 2H), 2.35 (s, 3H). **¹³C NMR** (126 MHz, CDCl₃) δ 139.11, 138.59, 138.28, 132.02, 130.25, 129.36, 129.03, 128.83, 128.53, 127.18, 125.56, 121.37, 115.13, 42.68, 40.47, 39.67, 38.91, 21.41. **HRMS** (ESI) m/z (M+Na)⁺ calculated for C₂₁H₂₀N₂Na: 323.1524, found: 323.1522.

(E)-2-benzyl-2-(4-(3-methoxyphenyl)but-2-en-1-yl)malononitrile (31)



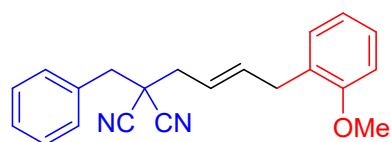
¹H NMR (400 MHz, Chloroform-*d*) δ 7.43 – 7.33 (m, 5H), 7.24 – 7.18 (m, 1H), 6.84 – 6.71 (m, 3H), 5.97 (dt, *J* = 15.0, 6.9, 1.1 Hz, 1H), 5.67 (dt, *J* = 15.0, 7.3, 1.5 Hz, 1H), 3.79 (s, 3H), 3.44 (d, *J* = 6.7 Hz, 2H), 3.18 (s, 2H), 2.68 (dd, *J* = 7.3, 1.1 Hz, 2H). **¹³C NMR** (101 MHz, CDCl₃) δ 159.85, 140.78, 138.25, 131.98, 130.24, 129.59, 129.02, 128.83, 121.62, 120.89, 115.10, 114.08, 111.96, 55.23, 42.70, 40.44, 39.66, 38.98. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₁H₂₁N₂O: 317.1654, found: 317.1654.

(E)-2-benzyl-2-(4-(3-chlorophenyl)but-2-en-1-yl)malononitrile (32)



¹H NMR (400 MHz, Chloroform-*d*) δ 7.43 – 7.35 (m, 6H), 7.25 – 7.17 (m, 3H), 7.09 (dd, *J* = 7.2, 1.7 Hz, 1H), 5.95 (ddd, *J* = 15.2, 7.7, 6.4 Hz, 1H), 5.73 – 5.63 (m, 1H), 3.45 (d, *J* = 6.9 Hz, 2H), 3.20 (s, 2H), 2.69 (d, *J* = 7.3 Hz, 2H). **¹³C NMR** (126 MHz, CDCl₃) δ 141.17, 137.49, 131.91, 130.23, 129.87, 129.79, 129.14, 129.04, 128.86, 128.67, 126.75, 126.66, 122.29, 115.03, 42.76, 40.38, 39.62, 38.51. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₀H₁₈ClN₂: 321.1159, found: 321.1164.

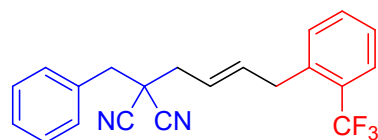
(E)-2-benzyl-2-(4-(2-methoxyphenyl)but-2-en-1-yl)malononitrile (33)



¹H NMR (400 MHz, Chloroform-*d*) δ 7.47 – 7.30 (m, 5H), 7.21 (ddd, *J* = 8.1, 7.4, 1.7 Hz, 1H), 7.14 (dd, *J* = 7.4, 1.8 Hz, 1H), 6.90 (td, *J* = 7.5, 1.1 Hz, 1H), 6.86 (dd, *J* = 8.2, 1.1 Hz, 1H), 5.97 (dt,

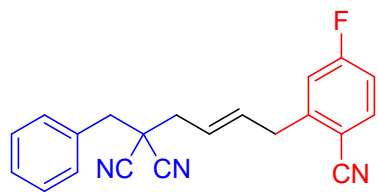
$J = 14.7, 6.6, 1.2$ Hz, 1H), 5.65 – 5.56 (m, 1H), 3.83 (s, 3H), 3.44 (d, $J = 6.7$ Hz, 2H), 3.16 (s, 2H), 2.66 (dq, $J = 7.3, 0.9$ Hz, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.24, 137.99, 132.09, 130.22, 129.80, 128.97, 128.75, 127.76, 127.63, 124.94, 120.81, 120.59, 120.45, 115.14, 110.32, 55.29, 42.54, 40.45, 39.55, 33.40. HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calculated for $\text{C}_{21}\text{H}_{21}\text{N}_2\text{O}$: 317.1654, found: 317.1648.

(*E*)-2-benzyl-2-(4-(2-(trifluoromethyl)phenyl)but-2-en-1-yl)malononitrile (34)



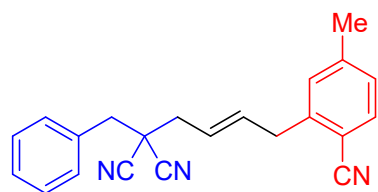
^1H NMR (400 MHz, Chloroform-*d*) δ 7.64 (dd, $J = 8.0, 1.4$ Hz, 1H), 7.51 – 7.47 (m, 1H), 7.40 – 7.31 (m, 7H), 6.02 – 5.91 (m, 1H), 5.62 (dt, $J = 14.9, 7.3, 1.6$ Hz, 1H), 3.64 (d, $J = 6.6$ Hz, 2H), 3.16 (s, 2H), 2.67 (dd, $J = 7.2, 1.1$ Hz, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 137.72, 137.70, 137.69, 137.43, 132.07, 131.95, 131.25, 130.22, 129.14, 129.03, 128.91, 128.84, 128.79, 126.64, 126.08 (q, $J = 5.7$ Hz), 122.24, 115.03, 42.61, 40.33, 39.54, 35.49 (q, $J = 2.0$ Hz). ^{19}F NMR (376 MHz, CDCl_3) δ -59.71. HRMS (ESI) m/z ($\text{M}+\text{Na}$) $^+$ calculated for $\text{C}_{21}\text{H}_{17}\text{F}_3\text{N}_2\text{Na}$: 377.1242, found: 377.1223.

(*E*)-2-benzyl-2-(4-(2-cyano-5-fluorophenyl)but-2-en-1-yl)malononitrile (35)



^1H NMR (400 MHz, Chloroform-*d*) δ 7.65 (dd, $J = 8.6, 5.4$ Hz, 1H), 7.43 – 7.37 (m, 5H), 7.13 – 7.01 (m, 2H), 5.94 (dt, $J = 16.4, 6.7, 1.1$ Hz, 1H), 5.81 – 5.70 (m, 1H), 3.68 (dd, $J = 6.7, 1.3$ Hz, 2H), 3.22 (s, 2H), 2.71 (dd, $J = 7.3, 1.1$ Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 165.16 (d, $J = 257.3$ Hz), 146.32, 146.25, 135.39, 135.31, 134.80, 130.24, 129.01, 128.95, 128.89, 128.84, 128.80, 124.16, 117.31, 117.17, 117.13, 115.11, 115.06, 114.92, 114.88, 108.63, 108.61, 42.58, 40.22, 39.54, 37.33, 0.04. ^{19}F NMR (471 MHz, CDCl_3) δ -102.18. HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calculated for $\text{C}_{21}\text{H}_{17}\text{FN}_3$: 330.1407, found: 330.1406.

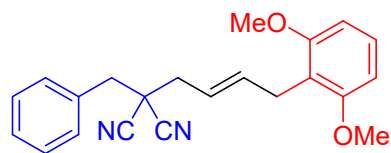
(*E*)-2-benzyl-2-(4-(2-cyano-5-methylphenyl)but-2-en-1-yl)malononitrile (36)



^1H NMR (400 MHz, Chloroform-*d*) δ 7.52 (d, $J = 7.8$ Hz, 1H), 7.42 – 7.37 (m, 5H), 7.19 (d, $J = 1.6$ Hz, 1H), 7.15 – 7.10 (m, 1H), 5.95 (dt, $J = 14.6, 6.7, 1.1$ Hz, 1H), 5.71 (dt, $J = 15.0, 7.3, 1.4$ Hz, 1H), 3.64 (dd, $J = 6.7, 1.4$ Hz, 2H), 3.21 (s, 2H), 2.70 (dd, $J = 7.3, 1.1$ Hz, 2H), 2.38 (s, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 144.19, 142.64, 135.94, 132.88, 132.03, 131.98, 130.54, 130.25, 129.03, 128.84, 128.01, 123.22, 118.25, 115.13, 115.03, 109.39, 42.54,

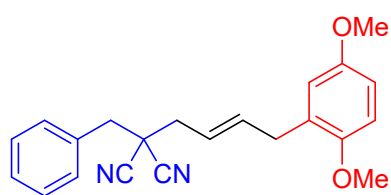
40.31, 39.62, 37.29, 21.79. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₂H₂₀N₃: 326.1657, found: 326.1656.

(E)-2-benzyl-2-(4-(2,6-dimethoxyphenyl)but-2-en-1-yl)malononitrile (37)



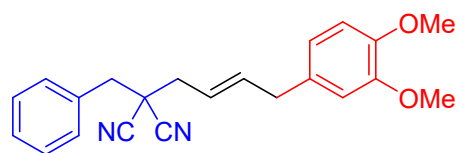
¹H NMR (400 MHz, Chloroform-*d*) δ 7.40 – 7.30 (m, 5H), 7.16 (t, J = 8.3 Hz, 1H), 6.55 (d, J = 8.3 Hz, 2H), 5.89 (dt, J = 15.1, 6.3, 1.2 Hz, 1H), 5.52 (dt, J = 14.9, 7.3, 1.6 Hz, 1H), 3.82 (s, 6H), 3.50 – 3.43 (m, 2H), 3.11 (s, 2H), 2.66 – 2.57 (m, 2H). **¹³C NMR** (101 MHz, CDCl₃) δ 158.14, 137.73, 132.26, 130.21, 128.92, 128.67, 127.55, 119.70, 115.45, 115.22, 103.70, 55.75, 42.38, 40.44, 39.51, 26.14. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₂H₂₃N₂O₂: 347.1760, found: 347.1764.

(E)-2-benzyl-2-(4-(2,5-dimethoxyphenyl)but-2-en-1-yl)malononitrile (38)



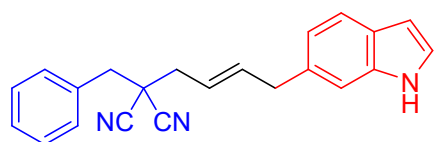
¹H NMR (400 MHz, Chloroform-*d*) δ 7.43 – 7.35 (m, 5H), 7.07 (d, J = 7.5 Hz, 1H), 6.99 (dt, J = 7.5, 3.7 Hz, 2H), 5.98 (dt, J = 15.3, 6.5, 1.2 Hz, 1H), 5.59 (dddd, J = 15.0, 7.3, 5.8, 1.6 Hz, 1H), 3.43 (dd, J = 6.5, 1.5 Hz, 2H), 3.19 (s, 2H), 2.69 (dd, J = 7.2, 1.1 Hz, 2H), 2.32 (s, 3H), 2.29 (s, 3H). **¹³C NMR** (101 MHz, CDCl₃) δ 137.93, 137.06, 135.69, 133.05, 132.03, 130.26, 130.24, 129.84, 129.81, 129.02, 128.82, 127.31, 121.22, 115.12, 42.70, 40.47, 39.70, 36.47, 20.95, 18.93, 18.91. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₂H₂₂N₂O₂: 347.1760, found: 347.1777.

(E)-2-benzyl-2-(4-(3,4-dimethoxyphenyl)but-2-en-1-yl)malononitrile (39)



¹H NMR (400 MHz, Chloroform-*d*) δ 7.43 – 7.33 (m, 5H), 6.81 (d, J = 7.9 Hz, 1H), 6.76 – 6.71 (m, 2H), 5.96 (dt, J = 14.9, 6.8, 1.2 Hz, 1H), 5.66 (dt, J = 15.0, 7.3, 1.5 Hz, 1H), 3.86 (s, 4H), 3.85 (s, 3H), 3.41 (dd, J = 6.9, 1.5 Hz, 2H), 3.19 (s, 2H), 2.68 (dd, J = 7.4, 1.1 Hz, 2H). **¹³C NMR** (101 MHz, CDCl₃) δ 149.05, 147.60, 138.64, 131.97, 131.69, 130.22, 129.02, 128.89, 128.84, 121.38, 120.39, 115.12, 111.70, 111.27, 55.94, 55.89, 42.78, 40.47, 39.78, 38.54. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₂H₂₃N₂O₂: 347.1760, found: 347.1757.

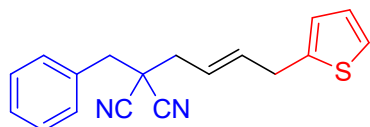
(E)-2-(4-(1H-indol-6-yl)but-2-en-1-yl)-2-benzylmalononitrile (40)



¹H NMR (400 MHz, Chloroform-*d*) δ 8.10 (s, 1H), 7.57 (d, J = 8.1 Hz, 1H), 7.41 – 7.34 (m, 5H), 7.23 (s, 1H), 7.15 (t, J =

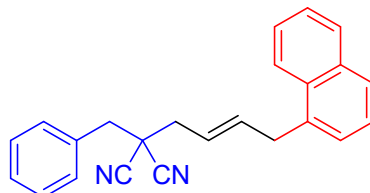
2.8 Hz, 1H), 6.96 (dd, $J = 8.0, 1.5$ Hz, 1H), 6.51 (ddd, $J = 3.1, 2.1, 0.9$ Hz, 1H), 6.02 (dtd, $J = 13.9, 6.9, 1.4$ Hz, 1H), 5.68 (dtt, $J = 14.9, 7.3, 1.5$ Hz, 1H), 3.56 (d, $J = 6.8$ Hz, 2H), 3.18 (s, 2H), 2.68 (d, $J = 7.3$ Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 139.27, 136.21, 133.08, 132.04, 130.24, 129.02, 128.82, 126.43, 124.09, 120.94, 120.91, 120.74, 115.19, 110.74, 102.46, 42.66, 40.54, 39.74, 39.20. HRMS (ESI) m/z ($\text{M}+\text{H}$) $^+$ calculated for $\text{C}_{22}\text{H}_{20}\text{N}_3$: 326.1657, found: 326.1661.

(E)-2-benzyl-2-(4-(thiophen-2-yl)but-2-en-1-yl)malononitrile (41)



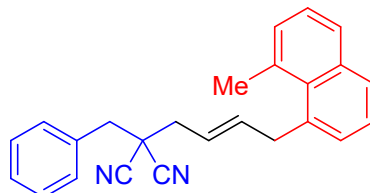
^1H NMR (500 MHz, Chloroform- d) δ 7.39 (tdd, $J = 7.0, 5.1, 1.8$ Hz, 6H), 7.17 (dd, $J = 5.2, 1.2$ Hz, 1H), 6.95 (dd, $J = 5.1, 3.4$ Hz, 1H), 6.85 (dd, $J = 3.3, 1.2$ Hz, 1H), 6.07 – 5.97 (m, 1H), 5.72 (dtt, $J = 14.9, 7.3, 1.5$ Hz, 1H), 3.66 (d, $J = 6.8$ Hz, 2H), 3.20 (s, 2H), 2.71 (d, $J = 7.4$ Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 141.83, 137.46, 131.96, 130.24, 129.79, 129.13, 129.03, 129.00, 128.94, 128.84, 128.78, 127.07, 126.85, 124.99, 124.03, 121.97, 115.02, 42.64, 40.25, 39.46, 32.99. HRMS (ESI) m/z ($\text{M}+\text{Na}$) $^+$ calculated for $\text{C}_{18}\text{H}_{16}\text{N}_2\text{SNa}$: 315.0932, found: 315.0931.

(E)-2-benzyl-2-(4-(naphthalen-1-yl)but-2-en-1-yl)malononitrile (42)



^1H NMR (400 MHz, Chloroform- d) δ 8.04 – 7.96 (m, 1H), 7.88 – 7.83 (m, 1H), 7.75 (d, $J = 8.1$ Hz, 1H), 7.56 – 7.32 (m, 8H), 7.30 – 7.25 (m, 2H), 6.10 (dtt, $J = 15.1, 6.3, 1.2$ Hz, 1H), 5.62 (dtt, $J = 15.0, 7.4, 1.6$ Hz, 1H), 3.90 (d, $J = 6.3$ Hz, 2H), 3.07 (s, 2H), 2.68 – 2.59 (m, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 138.13, 135.23, 133.91, 131.99, 131.77, 130.19, 129.15, 128.98, 128.86, 128.79, 127.41, 126.48, 126.14, 125.74, 125.70, 123.92, 121.78, 115.08, 42.50, 40.40, 39.59, 36.16. HRMS (ESI) m/z (M^+) calculated for $\text{C}_{24}\text{H}_{20}\text{N}_2$: 336.1626, found: 336.1613.

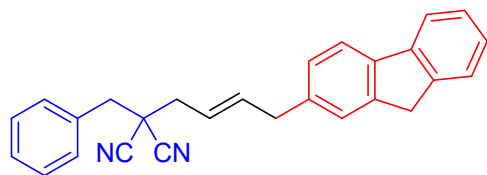
(E)-2-benzyl-2-(4-(8-methylnaphthalen-1-yl)but-2-en-1-yl)malononitrile (43)



^1H NMR (400 MHz, Chloroform- d) δ 7.72 (ddd, $J = 13.4, 8.0, 1.7$ Hz, 2H), 7.39 – 7.26 (m, 9H), 6.15 (dtt, $J = 15.3, 5.5, 1.2$ Hz, 1H), 5.30 (dtt, $J = 15.1, 7.4, 1.8$ Hz, 1H), 4.11 (dd, $J = 5.5, 1.7$ Hz, 2H), 3.05 (s, 2H), 2.91 (s, 3H), 2.62 (dd, $J = 7.4, 1.3$ Hz, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 139.90, 135.88, 135.70, 134.34, 132.29, 131.96, 130.24, 130.17, 129.80, 129.69, 129.14, 128.97, 128.90, 128.77, 128.23, 125.22, 125.11, 121.43, 115.04, 42.44, 40.29,

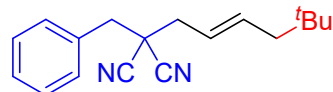
39.63, 39.58, 25.48. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₅H₂₃N₂: 351.1861, found: 351.1873.

(E)-2-(4-(9H-fluoren-2-yl)but-2-en-1-yl)-2-benzylmalononitrile (44)



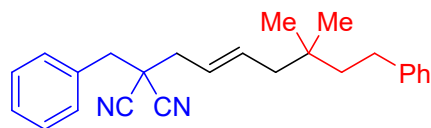
¹H NMR (400 MHz, Chloroform-*d*) δ 7.75 (dd, J = 14.8, 7.7 Hz, 2H), 7.54 (d, J = 7.4 Hz, 1H), 7.44 – 7.36 (m, 7H), 7.32 – 7.28 (m, 1H), 7.23 (dd, J = 7.8, 1.6 Hz, 1H), 6.10 – 5.96 (m, 1H), 5.79 – 5.64 (m, 1H), 3.89 (s, 2H), 3.55 (d, J = 6.9 Hz, 2H), 3.20 (s, 2H), 2.71 (d, J = 7.3 Hz, 2H). **¹³C NMR** (101 MHz, CDCl₃) δ 143.82, 143.23, 141.55, 140.14, 138.71, 137.82, 132.01, 130.25, 129.03, 128.84, 127.19, 126.76, 126.54, 125.30, 125.06, 121.43, 119.96, 119.78, 115.14, 42.70, 40.48, 39.71, 39.12, 36.84. **HRMS** (ESI) m/z (M+Na)⁺ calculated for C₂₇H₂₂N₂Na: 397.1681, found: 397.1685.

(E)-2-benzyl-2-(5,5-dimethylhex-2-en-1-yl)malononitrile (45)



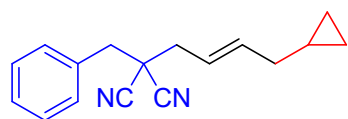
¹H NMR (400 MHz, Chloroform-*d*) δ 7.39 (qd, J = 5.4, 3.1 Hz, 5H), 5.86 (dtt, J = 15.2, 7.5, 1.2 Hz, 1H), 5.55 (dtt, J = 14.9, 7.3, 1.3 Hz, 1H), 3.19 (s, 2H), 2.74 – 2.60 (m, 2H), 2.00 (dd, J = 7.5, 1.2 Hz, 2H), 0.92 (s, 9H). **¹³C NMR** (101 MHz, CDCl₃) δ 137.39, 132.10, 130.24, 129.00, 128.78, 122.05, 115.19, 47.09, 42.67, 42.58, 40.65, 39.82, 30.98, 29.28. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₁₈H₂₃N₂: 267.1861, found: 267.1875.

(E)-2-benzyl-2-(5,5-dimethyl-7-phenylhept-2-en-1-yl)malononitrile (46)



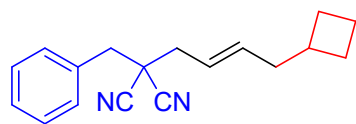
¹H NMR (400 MHz, Chloroform-*d*) δ 7.41 – 7.34 (m, 5H), 7.31 – 7.23 (m, 2H), 7.18 (d, J = 7.4 Hz, 3H), 5.93 – 5.81 (m, 1H), 5.65 – 5.53 (m, 1H), 3.17 (s, 2H), 2.71 – 2.64 (m, 2H), 2.63 – 2.51 (m, 2H), 2.10 (dd, J = 7.5, 1.2 Hz, 2H), 1.57 – 1.47 (m, 2H), 0.97 (s, 6H). **¹³C NMR** (101 MHz, CDCl₃) δ 143.15, 136.87, 132.06, 130.24, 129.01, 128.79, 128.39, 128.33, 125.66, 122.40, 115.17, 45.13, 44.22, 42.68, 40.70, 39.79, 33.63, 30.69, 26.90. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₅H₂₉N₂: 357.2331, found: 357.2341.

(E)-2-benzyl-2-(4-cyclopropylbut-2-en-1-yl)malononitrile (47)



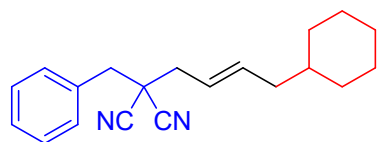
¹H NMR (500 MHz, Chloroform-*d*) δ 7.39 (qd, J = 5.5, 4.5, 2.4 Hz, 5H), 5.93 – 5.85 (m, 1H), 5.66 – 5.58 (m, 1H), 3.19 (s, 2H), 2.69 – 2.63 (m, 2H), 2.03 (td, J = 6.7, 1.4 Hz, 2H), 0.81 – 0.73 (m, 1H), 0.53 – 0.45 (m, 2H), 0.13 – 0.07 (m, 2H). **¹³C NMR** (101 MHz, CDCl₃) δ 139.22, 132.08, 130.23, 128.99, 128.78, 120.02, 115.13, 42.62, 40.56, 39.72, 37.15, 9.99, 4.26, 1.04. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₁₇H₁₉N₂: 251.1548, found: 251.1555.

(E)-2-benzyl-2-(4-cyclobutylbut-2-en-1-yl)malononitrile (48)



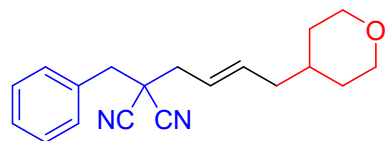
¹H NMR (400 MHz, Chloroform-*d*) δ 7.48 – 7.31 (m, 5H), 5.82 – 5.68 (m, 1H), 5.60 – 5.47 (m, 1H), 3.17 (s, 2H), 2.64 (d, J = 7.2 Hz, 2H), 2.38 (hept, J = 7.6 Hz, 1H), 2.24 – 2.16 (m, 2H), 2.06 (tdd, J = 10.1, 8.2, 5.2 Hz, 2H), 1.93 – 1.82 (m, 1H), 1.86 – 1.76 (m, 1H), 1.72 – 1.55 (m, 2H). **¹³C NMR** (101 MHz, CDCl₃) δ 138.17, 132.11, 130.23, 128.99, 128.77, 120.43, 115.13, 42.57, 40.58, 39.78, 39.65, 35.03, 27.79, 18.37. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₁₈H₂₁N₂: 265.1705, found: 265.1711.

(E)-2-benzyl-2-(4-cyclohexylbut-2-en-1-yl)malononitrile (49)



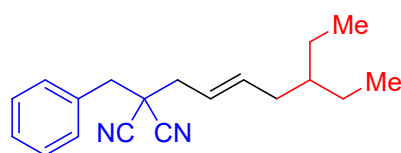
¹H NMR (400 MHz, Chloroform-*d*) δ 7.48 – 7.32 (m, 5H), 5.89 – 5.74 (m, 1H), 5.53 (dt, J = 14.9, 7.3, 1.4 Hz, 1H), 3.18 (s, 2H), 2.68 – 2.62 (m, 2H), 2.07 – 1.97 (m, 2H), 1.78 – 1.56 (m, 5H), 1.43 – 1.05 (m, 4H), 1.04 – 0.79 (m, 2H). **¹³C NMR** (101 MHz, CDCl₃) δ 138.85, 132.12, 128.99, 128.77, 120.84, 115.17, 42.63, 40.62, 40.54, 39.77, 37.66, 33.03, 26.47, 26.27. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₀H₂₅N₂: 293.2018, found: 293.2018.

(E)-2-benzyl-2-(4-(tetrahydro-2H-pyran-4-yl)but-2-en-1-yl)malononitrile (50)



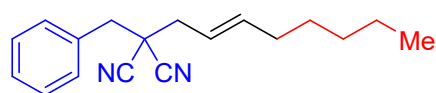
¹H NMR (400 MHz, Chloroform-*d*) δ 7.39 (dtd, J = 9.8, 4.8, 4.4, 2.2 Hz, 5H), 5.86 – 5.74 (m, 1H), 5.65 – 5.52 (m, 1H), 4.00 – 3.91 (m, 2H), 3.36 (td, J = 11.8, 1.9 Hz, 2H), 3.19 (s, 2H), 2.68 – 2.62 (m, 2H), 2.12 – 2.03 (m, 2H), 1.68 – 1.51 (m, 3H), 1.37 – 1.22 (m, 2H). **¹³C NMR** (126 MHz, CDCl₃) δ 137.58, 132.00, 130.22, 129.02, 128.82, 121.80, 115.11, 67.97, 42.76, 40.55, 39.97, 39.85, 34.99, 32.81. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₁₉H₂₃N₂O: 295.1810, found: 295.1824.

(E)-2-benzyl-2-(5-ethylhept-2-en-1-yl)malononitrile (51)



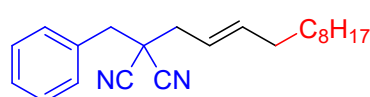
¹H NMR (500 MHz, Chloroform-*d*) δ 7.38 – 7.28 (m, 5H), 5.79 – 5.67 (m, 1H), 5.53 – 5.44 (m, 1H), 3.11 (s, 2H), 2.59 (d, *J* = 7.3 Hz, 2H), 2.02 (dd, *J* = 7.0, 4.6 Hz, 2H), 1.24 (dtd, *J* = 13.0, 7.2, 6.1, 2.8 Hz, 5H), 0.79 (t, *J* = 7.1 Hz, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 138.84, 132.12, 130.23, 128.99, 128.77, 128.73, 121.04, 115.16, 42.64, 40.65, 40.56, 40.46, 39.80, 35.86, 25.29, 10.96. HRMS (ESI) *m/z* (M+H)⁺ calculated for C₁₉H₂₄N₂O: 281.2018, found: 281.2020.

(E)-2-benzyl-2-(oct-2-en-1-yl)malononitrile (52)



¹H NMR (500 MHz, Chloroform-*d*) δ 7.45 – 7.34 (m, 5H), 5.87 – 5.77 (m, 1H), 5.55 (dtt, *J* = 14.8, 7.3, 1.5 Hz, 1H), 3.18 (s, 2H), 2.64 (d, *J* = 7.3 Hz, 2H), 2.19 – 2.04 (m, 2H), 1.41 (p, *J* = 7.3 Hz, 2H), 1.37 – 1.24 (m, 4H), 0.95 – 0.84 (m, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 140.32, 132.12, 130.23, 128.98, 128.76, 119.86, 115.15, 42.63, 40.59, 39.77, 32.52, 28.60, 22.46, 14.04. HRMS (ESI) *m/z* (M+H)⁺ calculated for C₁₈H₂₃N₂: 267.1861, found: 267.1837.

(E)-2-benzyl-2-(dodec-2-en-1-yl)malononitrile (53)



¹H NMR (500 MHz, Chloroform-*d*) δ 7.45 – 7.34 (m, 5H), 5.82 (dt, *J* = 15.1, 6.9 Hz, 1H), 5.54 (dtt, *J* = 14.8, 7.3, 1.5 Hz, 1H), 3.17 (s, 2H), 2.69 – 2.59 (m, 2H), 2.16 – 2.06 (m, 2H), 1.41 (p, *J* = 7.0 Hz, 2H), 1.36 – 1.22 (m, 12H), 0.93 – 0.83 (m, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 140.33, 132.13, 130.23, 128.98, 128.76, 128.72, 119.85, 115.15, 42.62, 40.59, 39.76, 32.57, 31.91, 29.63, 29.58, 29.45, 29.33, 29.11, 28.95, 22.70, 14.14. HRMS (ESI) *m/z* (M+Na)⁺ calculated for C₂₂H₃₀N₂Na: 345.2307, found: 345.2316.

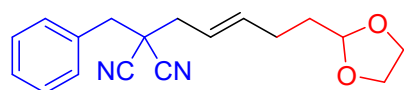
(E)-2-benzyl-2-(6-phenylhex-2-en-1-yl)malononitrile (54)



¹H NMR (400 MHz, Chloroform-*d*) δ 7.38 (ddt, *J* = 8.2, 5.3, 2.7 Hz, 5H), 7.32 – 7.24 (m, 2H), 7.18 (ddt, *J* = 7.5, 3.2, 1.9 Hz, 3H), 5.84 (ddd, *J* = 15.2, 7.5, 6.2 Hz, 1H), 5.57 (dtt, *J* = 15.0, 7.4, 1.5 Hz, 1H), 3.17 (s, 2H), 2.68 – 2.60 (m, 4H), 2.21 – 2.09 (m, 2H), 1.81 – 1.69 (m, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 142.08, 139.74, 132.07, 130.25, 129.01, 128.80, 128.50, 128.38, 125.85, 120.49, 115.14, 42.67, 40.58, 39.84,

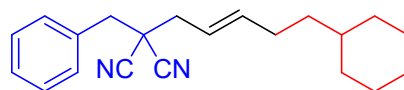
35.24, 32.06, 30.65. **HRMS** (ESI) m/z $(M+H)^+$ calculated for $C_{19}H_{23}N_2O$: 295.1810, found: 295.1824.

(E)-2-(5-(1,3-dioxolan-2-yl)pent-2-en-1-yl)-2-benzylmalononitrile (55)



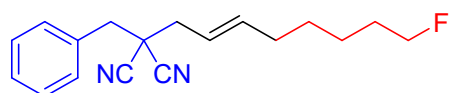
1H NMR (400 MHz, Chloroform- d) δ 7.42 – 7.37 (m, 5H), 5.86 (dtt, J = 14.8, 6.8, 1.2 Hz, 1H), 5.60 (dtt, J = 14.8, 7.3, 1.5 Hz, 1H), 4.89 (t, J = 4.6 Hz, 1H), 4.00 – 3.93 (m, 2H), 3.89 – 3.81 (m, 2H), 3.18 (s, 2H), 2.70 – 2.62 (m, 2H), 2.27 (q, J = 7.3, 6.7 Hz, 2H), 1.85 – 1.75 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.09, 132.05, 130.23, 129.00, 128.78, 120.49, 115.08, 103.79, 64.96, 42.57, 40.45, 39.65, 33.06, 27.01. **HRMS** (ESI) m/z $(M+Na)^+$ calculated for $C_{18}H_{20}N_2O_2Na$: 319.1422, found: 319.1434.

(E)-2-benzyl-2-(5-cyclohexylpent-2-en-1-yl)malononitrile (56)



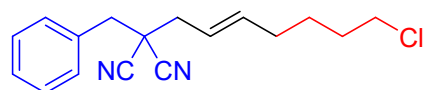
1H NMR (400 MHz, Chloroform- d) δ 7.38 (tq, J = 5.7, 3.7, 2.8 Hz, 5H), 5.87 – 5.76 (m, 1H), 5.54 (dtt, J = 14.8, 7.2, 1.5 Hz, 1H), 3.17 (s, 2H), 2.78 – 2.51 (m, 2H), 2.12 (q, J = 7.0 Hz, 2H), 1.80 – 1.49 (m, 5H), 1.34 – 1.09 (m, 6H), 0.96 – 0.80 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 140.58, 132.12, 130.23, 128.99, 128.77, 119.66, 115.15, 77.37, 77.06, 76.74, 42.62, 40.61, 39.80, 37.03, 36.57, 33.22, 29.93, 26.67, 26.57, 26.35. **HRMS** (ESI) m/z (M^+) calculated for $C_{21}H_{26}N_2$: 306.2096, found: 306.2105.

(E)-2-benzyl-2-(8-fluorooct-2-en-1-yl)malononitrile (57)



1H NMR (400 MHz, Chloroform- d) δ 7.44 – 7.34 (m, 5H), 5.82 (dtt, J = 14.9, 6.7, 1.2 Hz, 1H), 5.61 – 5.51 (m, 1H), 4.49 (t, J = 6.1 Hz, 1H), 4.38 (t, J = 6.1 Hz, 1H), 3.18 (s, 2H), 2.69 – 2.59 (m, 2H), 2.18 – 2.08 (m, 2H), 1.68 (ddt, J = 25.0, 7.9, 6.1 Hz, 2H), 1.48 – 1.32 (m, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 140.01, 132.07, 130.23, 129.00, 128.79, 120.13, 115.13, 84.17 (d, J = 163.9 Hz), 42.66, 40.57, 39.84, 32.42, 30.43, 30.24, 28.77, 28.62, 25.02, 24.97. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -218.14. **HRMS** (ESI) m/z $(M+H)^+$ calculated for $C_{18}H_{22}FN_2$: 285.1767, found: 285.1750.

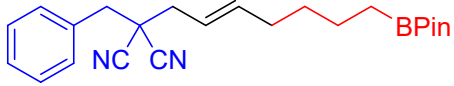
(E)-2-benzyl-2-(7-chlorohept-2-en-1-yl)malononitrile (58)



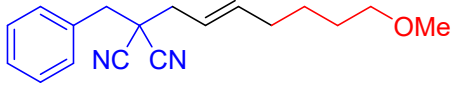
1H NMR (500 MHz, Chloroform- d) δ 7.44 – 7.35 (m, 5H), 5.85 – 5.78 (m, 1H), 5.58 (dtt, J = 14.9, 7.3, 1.5 Hz, 1H), 3.54 (t, J = 6.6 Hz, 2H), 3.18 (s, 2H), 2.64 (dd, J = 7.2, 1.1 Hz, 2H), 2.16 (qd, J = 7.3, 1.3 Hz, 2H), 1.84 –

1.77 (m, 2H), 1.58 (tt, $J = 9.9, 6.4$ Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 139.28, 132.02, 130.23, 129.01, 128.81, 120.74, 115.09, 44.80, 42.71, 40.52, 39.80, 31.87, 31.73, 26.11. HRMS (ESI) m/z (M+H) $^+$ calculated for $\text{C}_{17}\text{H}_{20}\text{ClN}_2$: 287.1315, found: 287.1324.

(E)-2-benzyl-2-(7-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hept-2-en-1-yl)malononitrile (59)

 ^1H NMR (400 MHz, Chloroform- d) δ 7.46 – 7.32 (m, 5H), 5.88 – 5.76 (m, 1H), 5.60 – 5.48 (m, 1H), 3.17 (s, 2H), 2.68 – 2.61 (m, 2H), 2.12 (d, $J = 6.8$ Hz, 2H), 1.49 – 1.36 (m, $J = 3.3$ Hz, 4H), 1.24 (s, 12H), 0.78 (d, $J = 7.6$ Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 140.28, 132.14, 130.23, 128.96, 128.74, 119.76, 115.14, 82.94, 77.29, 77.03, 76.78, 42.57, 40.60, 39.67, 32.37, 31.63, 24.82, 23.54. HRMS (ESI) m/z (M+Na) $^+$ calculated for $\text{C}_{23}\text{H}_{31}\text{BN}_2\text{O}_2\text{Na}$: 401.2376, found: 401.2388.

(E)-2-benzyl-2-(7-methoxyhept-2-en-1-yl)malononitrile (60)

 ^1H NMR (400 MHz, Chloroform- d) δ 7.39 (m, 5H), 5.82 (dt, $J = 14.7, 6.7, 1.2$ Hz, 1H), 5.56 (dt, $J = 15.1, 7.2, 1.5$ Hz, 1H), 3.38 (t, $J = 6.3$ Hz, 2H), 3.33 (s, 3H), 3.18 (s, 2H), 2.68 – 2.61 (m, 2H), 2.20 – 2.09 (m, 2H), 1.64 – 1.54 (m, 2H), 1.49 (tdd, $J = 9.7, 6.8, 5.0$ Hz, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 139.84, 132.08, 130.23, 128.99, 128.91, 128.84, 128.78, 128.69, 120.24, 115.12, 72.50, 58.60, 42.61, 40.55, 39.77, 32.34, 29.06, 25.51. HRMS (ESI) m/z (M+H) $^+$ calculated for $\text{C}_{18}\text{H}_{23}\text{N}_2\text{O}$: 283.1810, found: 283.1803.

(E)-2-(naphthalen-2-ylmethyl)-2-(6-phenylhex-2-en-1-yl)malononitrile (61)

 ^1H NMR (500 MHz, Chloroform- d) δ 7.90 – 7.82 (m, 4H), 7.51 (dt, $J = 6.8, 3.5$ Hz, 2H), 7.47 (dd, $J = 8.4, 1.9$ Hz, 1H), 7.28 (m, 2H), 7.17 (m, 3H), 5.84 (dt, $J = 14.3, 6.9$ Hz, 1H), 5.59 (dt, $J = 15.0, 7.3$ Hz, 1H), 3.34 (s, 2H), 2.82 – 2.39 (m, 4H), 2.16 (q, $J = 7.3$ Hz, 2H), 1.75 (p, $J = 7.5$ Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 142.10, 139.81, 133.26, 133.19, 129.73, 129.51, 128.86, 128.53, 128.40, 128.07, 127.80, 127.52, 126.72, 126.66, 125.86, 120.49, 115.21, 42.81, 40.63, 39.84, 35.26, 32.09, 30.68. HRMS (ESI) m/z (M+Na) $^+$ calculated for $\text{C}_{26}\text{H}_{24}\text{N}_2\text{Na}$: 387.1837, found: 387.1856.

(E)-2-(4-methylbenzyl)-2-(6-phenylhex-2-en-1-yl)malononitrile (62)

¹H NMR (400 MHz, Chloroform-*d*) δ 7.32 – 7.24 (m, 4H), 7.18 (m, 3H), 6.92 (d, *J* = 8.6 Hz, 2H), 5.83 (dtd, *J* = 13.6, 7.2, 6.4, 3.0 Hz, 1H), 5.62 – 5.50 (m, 1H), 3.81 (s, 3H), 3.13 (s, 2H), 2.68 – 2.59 (m, 4H), 2.15 (q, *J* = 7.1 Hz, 2H), 1.81 – 1.69 (m, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 142.08, 139.63, 138.66, 130.10, 129.68, 128.99, 128.49, 128.40, 128.36, 125.83, 120.55, 115.23, 42.34, 40.49, 39.96, 35.23, 32.05, 30.65, 21.21. HRMS (ESI) *m/z* (M+Na)⁺ calculated for C₂₃H₂₄N₂Na: 351.1837, found: 351.1842.

(*E*)-2-(4-methoxybenzyl)-2-(6-phenylhex-2-en-1-yl)malononitrile (63)

¹H NMR (400 MHz, Chloroform-*d*) δ 7.28 (ddd, *J* = 9.8, 5.4, 2.9 Hz, 5H), 7.18 (td, *J* = 5.6, 5.1, 2.3 Hz, 3H), 6.96 – 6.87 (m, 2H), 5.83 (dtd, *J* = 13.6, 7.2, 6.4, 3.0 Hz, 1H), 5.62 – 5.50 (m, 1H), 3.81 (s, 3H), 3.13 (s, 2H), 2.68 – 2.59 (m, 4H), 2.15 (q, *J* = 7.1 Hz, 2H), 1.81 – 1.69 (m, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 159.92, 142.08, 139.60, 131.40, 128.50, 128.37, 125.83, 124.02, 120.57, 115.27, 114.37, 55.32, 42.01, 40.41, 40.13, 35.23, 32.05, 30.66. HRMS (ESI) *m/z* (M+Na)⁺ calculated for C₂₃H₂₄N₂ONa: 345.1967, found: 345.1978.

(*E*)-2-(4-hydroxybenzyl)-2-(6-phenylhex-2-en-1-yl)malononitrile (64)

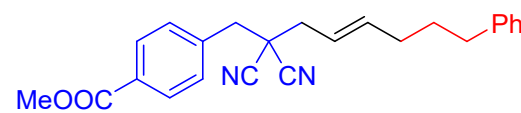
¹H NMR (400 MHz, Chloroform-*d*) δ 7.33 – 7.20 (m, 4H), 7.24 – 7.15 (m, 3H), 6.88 – 6.79 (m, 2H), 5.89 – 5.78 (m, 1H), 5.62 – 5.50 (m, 1H), 5.17 (s, 1H), 3.12 (s, 2H), 2.64 (m, 4H), 2.15 (q, *J* = 7.2 Hz, 2H), 1.81 – 1.69 (m, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 156.12, 142.07, 139.69, 131.59, 128.49, 128.37, 125.83, 124.12, 120.47, 115.87, 115.21, 42.01, 40.43, 40.20, 35.23, 32.05, 30.64. HRMS (ESI) *m/z* (M+H)⁺ calculated for C₂₂H₂₃N₂O: 331.1810, found: 331.1825.

(*E*)-2-(4-isocyanobenzyl)-2-(6-phenylhex-2-en-1-yl)malononitrile (65)

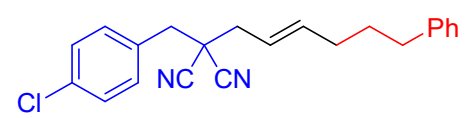
¹H NMR (400 MHz, Chloroform-*d*) δ 7.71 (d, *J* = 7.8 Hz, 2H), 7.51 (d, *J* = 7.8 Hz, 2H), 7.35 – 7.24 (m, 2H), 7.19 (t, *J* = 7.4 Hz, 3H), 5.87 (dt, *J* = 14.3, 6.7 Hz, 1H), 5.57 (dt, *J* = 14.6, 7.2 Hz, 1H), 3.22 (d, *J* = 6.9 Hz, 2H), 2.68 (dt, *J* = 26.5, 7.7 Hz, 4H), 2.17 (q, *J* = 7.2 Hz, 2H), 1.76 (p, *J* = 7.6 Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 141.97, 140.41, 137.33, 132.78, 131.04, 128.48, 128.40, 125.90, 119.92,

118.21, 114.55, 113.04, 42.41, 40.92, 39.53, 35.25, 32.07, 30.61. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₃H₂₂N₃: 340.1814, found: 340.1824.

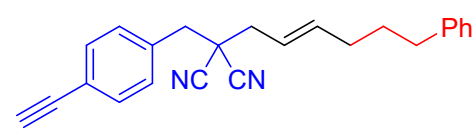
Methyl (E)-4-(2,2-dicyano-8-phenyloct-4-en-1-yl)benzoate (66)

 **¹H NMR** (400 MHz, Chloroform-*d*) δ 8.07 (d, J = 8.0 Hz, 2H), 7.46 (d, J = 8.1 Hz, 2H), 7.28 (m, 2H), 7.18 (m, 3H), 5.96 – 5.79 (m, 1H), 5.64 – 5.48 (m, 1H), 3.93 (s, 3H), 3.28 – 3.16 (m, 2H), 2.65 (h, J = 6.9, 6.0 Hz, 4H), 2.16 (q, J = 7.1 Hz, 2H), 1.75 (h, J = 7.4 Hz, 2H). **¹³C NMR** (101 MHz, CDCl₃) δ 166.51, 142.02, 140.08, 137.00, 130.67, 130.32, 130.24, 128.49, 128.39, 125.86, 120.19, 114.82, 52.33, 42.40, 40.75, 39.54, 35.24, 32.06, 30.63. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₄H₂₅N₂O₂: 373.1916, found: 373.1936.

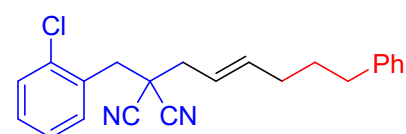
(E)-2-(4-chlorobenzyl)-2-(6-phenylhex-2-en-1-yl)malononitrile (67)

 **¹H NMR** (500 MHz, Chloroform-*d*) δ 7.44 – 7.35 (m, 2H), 7.37 – 7.24 (m, 4H), 7.25 – 7.15 (m, 3H), 5.90 – 5.81 (m, 1H), 5.57 (dddd, J = 15.0, 7.3, 5.8, 1.6 Hz, 1H), 3.15 (s, 2H), 2.71 – 2.57 (m, 4H), 2.17 (q, J = 7.2, 6.5 Hz, 2H), 1.81 – 1.72 (m, 2H). **¹³C NMR** (126 MHz, CDCl₃) δ 142.04, 140.00, 135.03, 131.55, 130.55, 129.28, 128.50, 128.40, 125.88, 120.24, 114.91, 41.98, 40.64, 39.81, 35.25, 32.08, 30.65. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₂H₂₂ClN₂: 349.1472, found: 349.1469.

(E)-2-(4-ethynylbenzyl)-2-(6-phenylhex-2-en-1-yl)malononitrile (68)

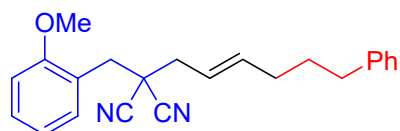
 **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.56 – 7.50 (m, 2H), 7.36 – 7.31 (m, 2H), 7.31 – 7.26 (m, 2H), 7.21 – 7.16 (m, 3H), 5.90 – 5.79 (m, 1H), 5.56 (dt, J = 14.9, 7.4, 1.5 Hz, 1H), 3.17 (s, 2H), 3.13 (s, 1H), 2.69 – 2.60 (m, 4H), 2.16 (q, J = 7.2 Hz, 2H), 1.75 (p, J = 6.9, 2H). **¹³C NMR** (101 MHz, CDCl₃) δ 142.02, 139.97, 132.70, 132.66, 130.21, 128.48, 128.37, 125.85, 122.82, 120.25, 114.90, 82.83, 78.41, 42.44, 40.63, 39.63, 35.23, 32.05, 30.63. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₄H₂₃N₂: 339.1861, found: 339.1866.

(E)-2-(2-chlorobenzyl)-2-(6-phenylhex-2-en-1-yl)malononitrile (69)

 **¹H NMR** (500 MHz, Chloroform-*d*) δ 7.57 – 7.53 (m, 1H), 7.49 – 7.45 (m, 1H), 7.36 – 7.32 (m, 2H), 7.30 (d, J = 7.7 Hz, 2H),

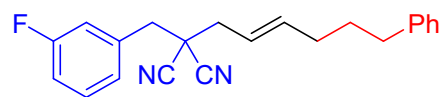
7.19 (dq, $J = 7.7, 2.8$ Hz, 3H), 5.87 (dt, $J = 15.1, 6.8$ Hz, 1H), 5.59 (dt, $J = 15.0, 7.3, 1.6$ Hz, 1H), 3.45 (s, 2H), 2.72 (d, $J = 7.3$ Hz, 2H), 2.68 – 2.63 (m, 2H), 2.17 (q, $J = 7.2$ Hz, 2H), 1.76 (ddd, $J = 15.1, 8.5, 6.9$ Hz, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 142.11, 139.89, 135.13, 131.93, 130.31, 130.29, 130.21, 128.51, 128.38, 127.49, 125.83, 120.50, 114.98, 40.67, 39.22, 38.77, 35.22, 32.05, 30.64. **HRMS** (ESI) m/z ($\text{M}+\text{H}$) $^+$ calculated for $\text{C}_{22}\text{H}_{22}\text{ClN}_2$: 349.1472, found: 349.1458.

(E)-2-(2-methoxybenzyl)-2-(6-phenylhex-2-en-1-yl)malononitrile (70)



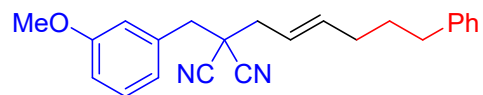
^1H NMR (500 MHz, Chloroform- d) δ 7.36 (m, 2H), 7.35 – 7.25 (m, 2H), 7.25 – 7.16 (m, 3H), 6.99 (td, $J = 7.5, 1.1$ Hz, 1H), 6.97 – 6.92 (m, 1H), 5.89 – 5.79 (m, 1H), 5.60 (dt, $J = 14.8, 7.3, 1.5$ Hz, 1H), 3.85 (s, 3H), 3.31 (s, 2H), 2.74 – 2.58 (m, 4H), 2.17 (q, $J = 6.9$ Hz, 2H), 1.76 (m, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 157.82, 142.18, 139.30, 131.80, 130.19, 128.53, 128.37, 128.29, 121.05, 120.81, 115.52, 110.92, 55.33, 40.58, 39.40, 36.12, 35.19, 32.04, 30.70. **HRMS** (ESI) m/z ($\text{M}+\text{H}$) $^+$ calculated for $\text{C}_{23}\text{H}_{25}\text{N}_2\text{O}$: 345.1967, found: 345.1968.

(E)-2-(3-fluorobenzyl)-2-(6-phenylhex-2-en-1-yl)malononitrile (71)



^1H NMR (500 MHz, Chloroform- d) δ 7.42 – 7.34 (m, 1H), 7.28 (m, 2H), 7.18 (m, 4H), 7.10 (m, 2H), 5.90 – 5.81 (m, 1H), 5.57 (dt, $J = 16.5, 7.3, 1.5$ Hz, 1H), 3.16 (s, 2H), 2.69 – 2.56 (m, 4H), 2.16 (q, $J = 7.3$ Hz, 2H), 1.75 (m, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 162.80 (d, $J = 247.7$ Hz), 142.04, 140.03, 134.34, 134.27, 130.69, 130.62, 128.50, 128.39, 125.98, 125.96, 125.87, 120.22, 117.35, 117.18, 116.04, 115.87, 114.88, 42.20 (d, $J = 2.0$ Hz), 40.69, 39.61, 35.25, 32.08, 30.65. ^{19}F NMR (471 MHz, CDCl_3) δ -111.65. **HRMS** (ESI) m/z ($\text{M}+\text{H}$) $^+$ calculated for $\text{C}_{22}\text{H}_{22}\text{FN}_2$: 333.1767, found: 333.1761.

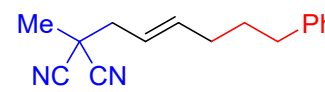
(E)-2-(3-methoxybenzyl)-2-(6-phenylhex-2-en-1-yl)malononitrile (72)



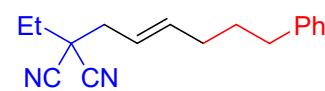
^1H NMR (500 MHz, Chloroform- d) δ 7.36 – 7.28 (m, 3H), 7.21 (m, 3H), 6.98 – 6.93 (m, 3H), 5.86 (ddd, $J = 14.6, 7.6, 5.7$ Hz, 1H), 5.59 (ddd, $J = 15.7, 8.4, 6.3$ Hz, 1H), 3.84 (d, $J = 2.1$ Hz, 3H), 3.17 (d, $J = 2.0$ Hz, 2H), 2.67 (t, $J = 8.0$ Hz, 4H), 2.17 (p, $J = 7.8$ Hz, 2H), 1.78 (m, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 159.89, 142.11, 139.75, 133.48, 130.05, 128.53, 128.51, 128.44, 128.41, 125.87, 122.49, 120.54,

115.78, 115.23, 114.33, 55.34, 42.65, 40.58, 39.73, 35.25, 32.08, 30.69. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₃H₂₅N₂O: 345.1967, found: 345.1967.

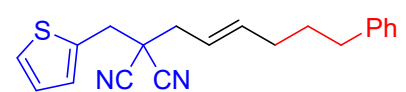
(E)-2-methyl-2-(6-phenylhex-2-en-1-yl)malononitrile (73)

 **¹H NMR** (500 MHz, Chloroform-*d*) δ 7.32 – 7.27 (m, 2H), 7.20 (m, 3H), 5.84 (dt, J = 14.9, 6.8, 1.2 Hz, 1H), 5.53 (dt, J = 15.0, 7.4, 1.5 Hz, 1H), 2.67 – 2.61 (m, 4H), 2.20 – 2.12 (m, 2H), 1.79 – 1.72 (m, 5H). **¹³C NMR** (126 MHz, CDCl₃) δ 142.08, 139.76, 128.51, 128.40, 125.87, 120.40, 116.08, 42.06, 35.26, 32.16, 32.04, 30.69, 24.00. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₁₆H₁₉N₂: 239.1548, found: 239.1550.

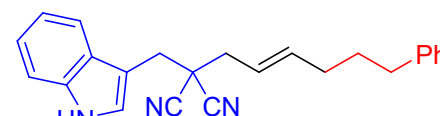
(E)-2-ethyl-2-(6-phenylhex-2-en-1-yl)malononitrile (74)

 **¹H NMR** (500 MHz, Chloroform-*d*) δ 7.33 – 7.26 (m, 2H), 7.24 – 7.16 (m, 3H), 5.83 (dt, J = 14.9, 6.7, 1.2 Hz, 1H), 5.54 (dt, J = 15.0, 7.3, 1.5 Hz, 1H), 2.71 – 2.59 (m, 4H), 2.22 – 2.11 (m, 2H), 1.98 (q, J = 7.4 Hz, 2H), 1.76 (p, J = 7.6 Hz, 2H), 1.29 (t, J = 7.4 Hz, 3H). **¹³C NMR** (126 MHz, CDCl₃) δ 142.11, 139.38, 128.51, 128.48, 128.41, 128.39, 125.85, 120.53, 115.42, 40.37, 39.16, 35.27, 32.04, 30.81, 30.70, 9.91. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₁₇H₂₁N₂: 253.1705, found: 253.1707.

(E)-2-(6-phenylhex-2-en-1-yl)-2-(thiophen-2-ylmethyl)malononitrile (75)

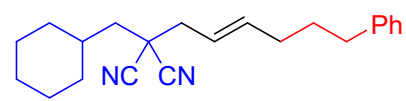
 **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.35 – 7.24 (m, 3H), 7.23 – 7.11 (m, 4H), 7.05 (dd, J = 5.2, 3.5 Hz, 1H), 5.85 (dt, J = 14.9, 6.8, 1.2 Hz, 1H), 5.55 (dt, J = 14.8, 7.2, 1.5 Hz, 1H), 3.43 (s, 2H), 2.69 – 2.55 (m, 4H), 2.16 (q, J = 7.2, 6.6 Hz, 2H), 1.75 (p, J = 7.5 Hz, 2H). **¹³C NMR** (101 MHz, CDCl₃) δ 142.06, 140.02, 132.80, 129.20, 128.51, 128.42, 128.39, 127.65, 126.66, 125.86, 120.22, 115.06, 39.99, 39.83, 36.78, 35.25, 32.09, 30.65. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₀H₂₁N₂S: 321.1425, found: 321.1426.

(E)-2-((1H-indol-3-yl)methyl)-2-(6-phenylhex-2-en-1-yl)malononitrile (76)

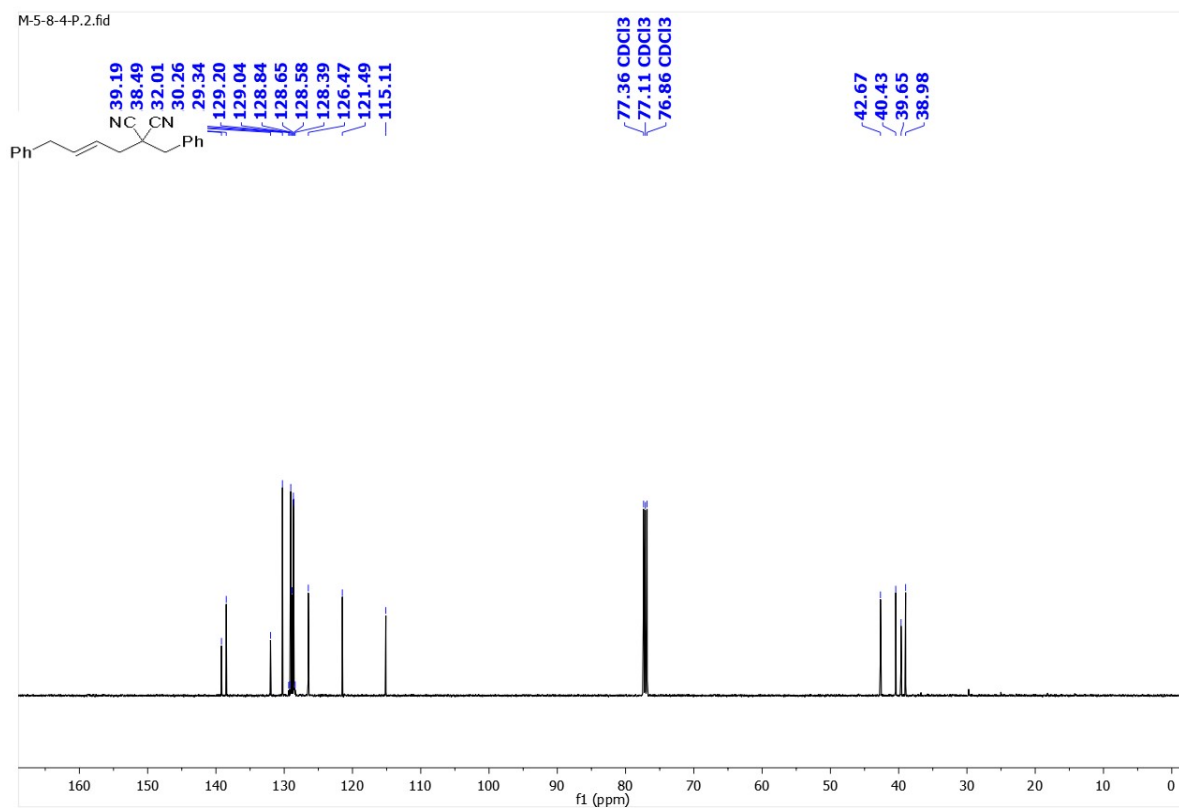
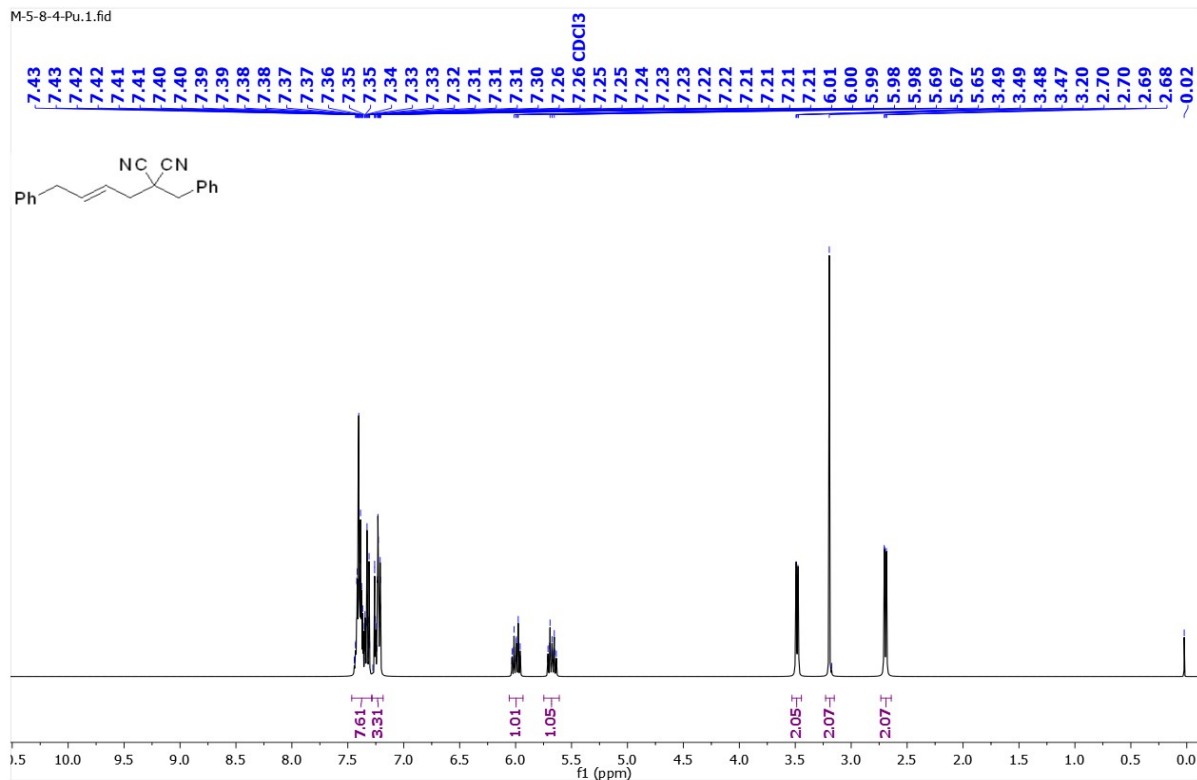
 **¹H NMR** (400 MHz, Chloroform-*d*) δ 8.29 (s, 1H), 7.64 (d, J = 7.8 Hz, 1H), 7.39 – 7.16 (m, 9H), 5.79 (dt, J = 14.0, 6.7 Hz, 1H), 5.56 (dt, J = 15.0, 7.2 Hz, 1H), 3.44 (d, J = 5.5 Hz, 2H), 2.63 (t, J = 8.8 Hz, 4H), 2.13 (q, J = 7.0 Hz, 2H), 1.73 (p, J = 7.2 Hz, 2H). **¹³C NMR** (101 MHz,

CDCl₃) δ 142.14, 139.37, 135.84, 128.51, 128.40, 128.37, 127.32, 125.82, 124.71, 122.74, 120.89, 120.33, 118.54, 115.96, 111.54, 106.88, 40.39, 40.20, 35.21, 33.31, 32.04, 30.66. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₄H₂₄N₃: 354.1970, found: 354.1975.

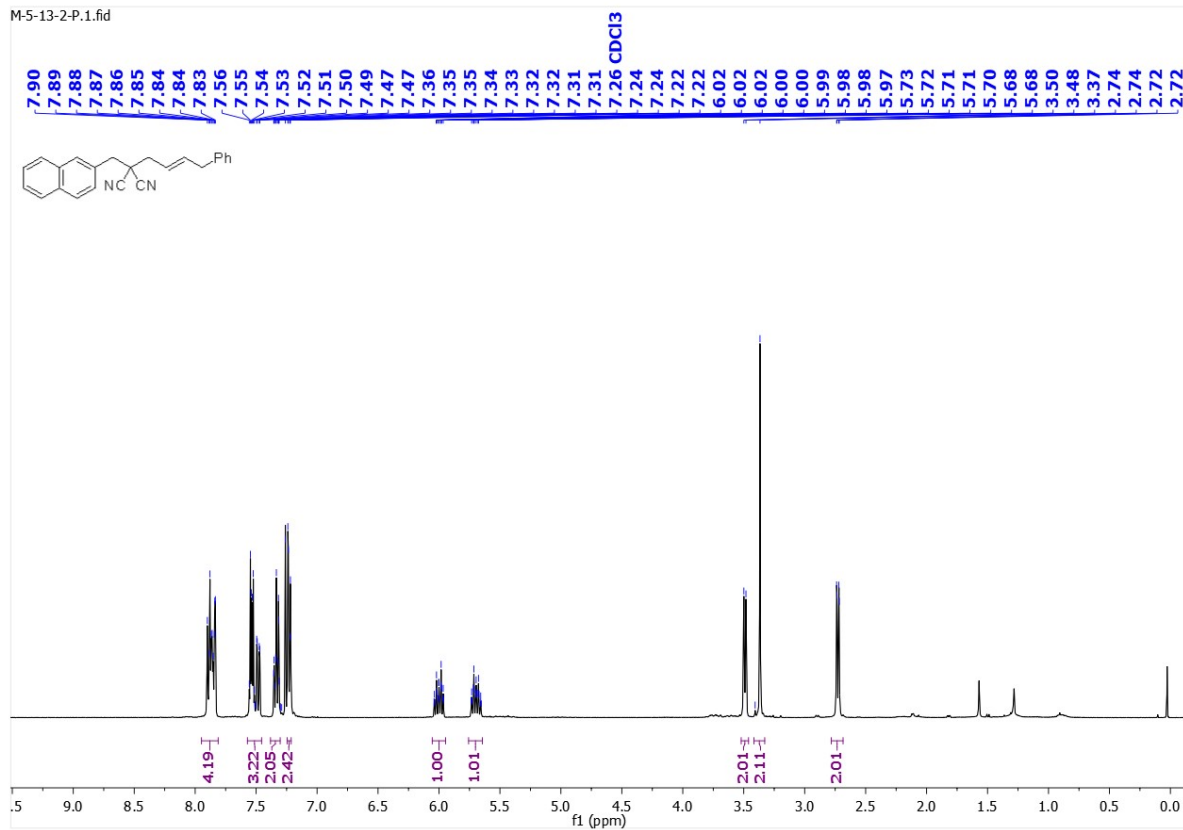
***(E)*-2-(cyclohexylmethyl)-2-(6-phenylhex-2-en-1-yl)malononitrile (77)**

 **¹H NMR** (500 MHz, Chloroform-*d*) δ 7.31 – 7.27 (m, 2H), 7.21 – 7.16 (m, 3H), 5.87 – 5.76 (m, 1H), 5.53 (dtt, *J* = 14.9, 7.3, 1.5 Hz, 1H), 2.68 – 2.60 (m, 4H), 2.17 – 2.11 (m, 2H), 1.95 – 1.91 (m, 2H), 1.83 – 1.66 (m, 8H), 1.39 – 1.02 (m, 5H). **¹³C NMR** (126 MHz, CDCl₃) δ 142.11, 139.51, 128.50, 128.37, 128.28, 128.07, 125.83, 120.55, 115.91, 43.94, 42.28, 36.32, 35.61, 35.23, 33.36, 32.03, 30.66, 25.90, 25.86, 1.06. **HRMS** (ESI) m/z (M+H)⁺ calculated for C₂₂H₂₉N₂: 321.2331, found: 321.2335.

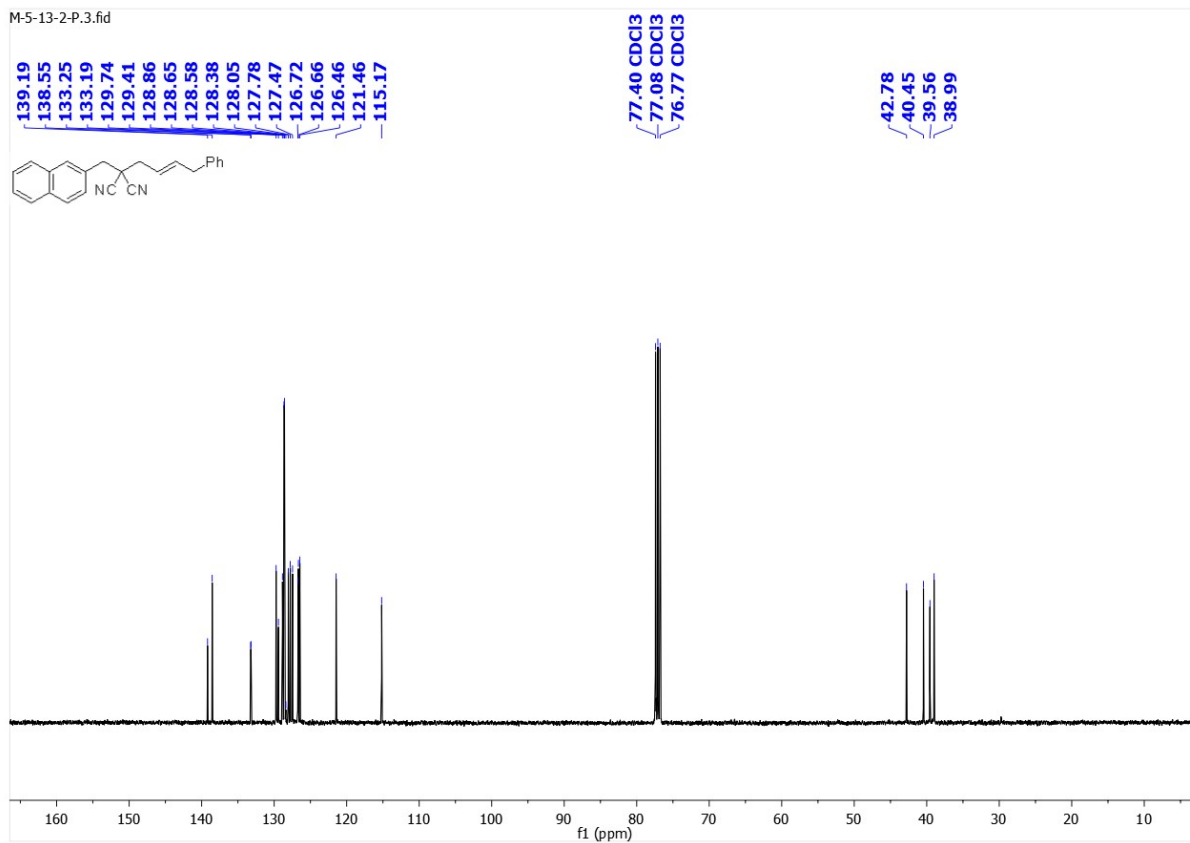
8. Copies of NMR Spectra of the Products



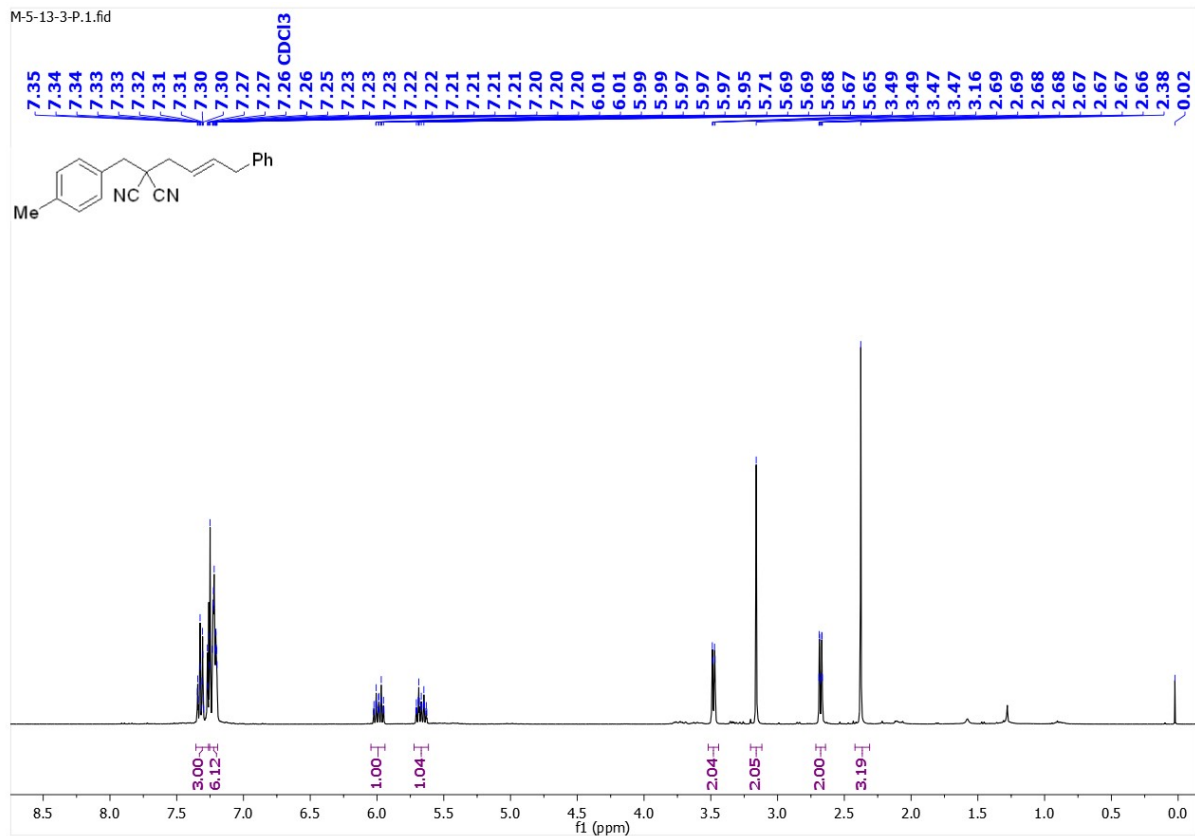
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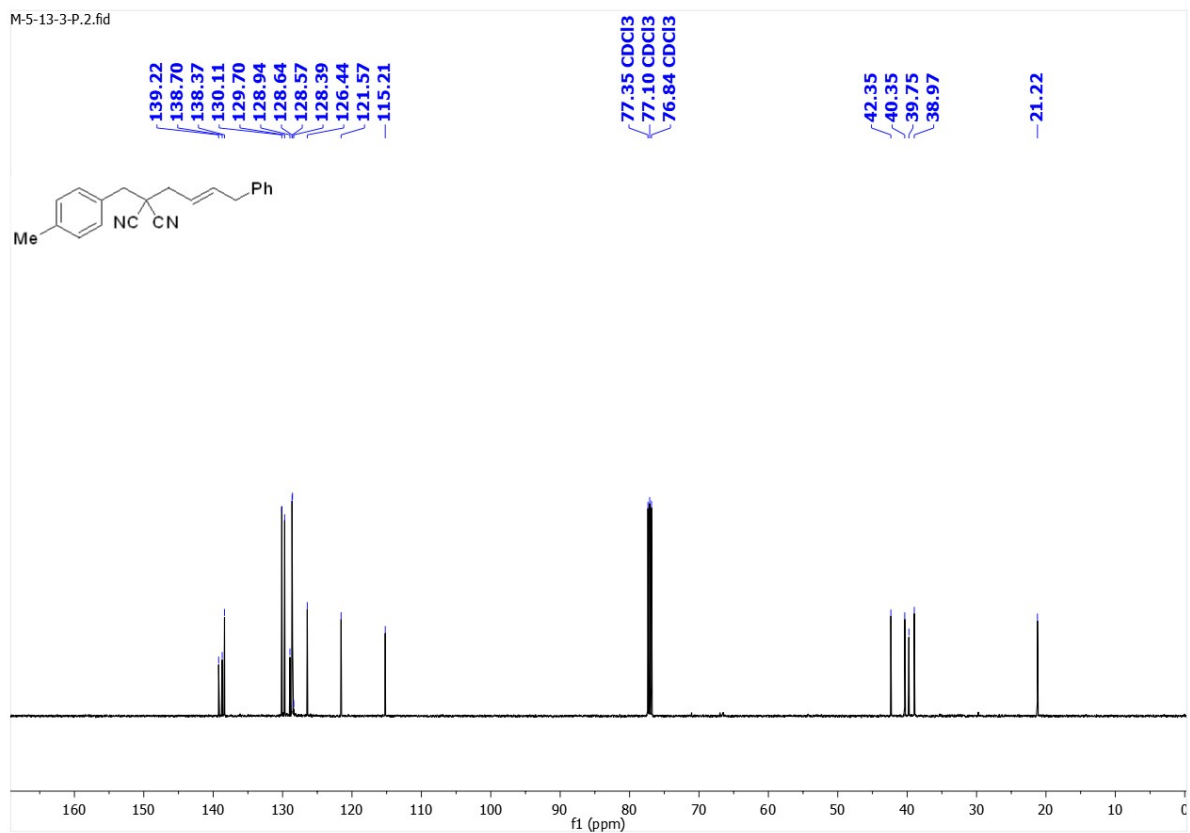
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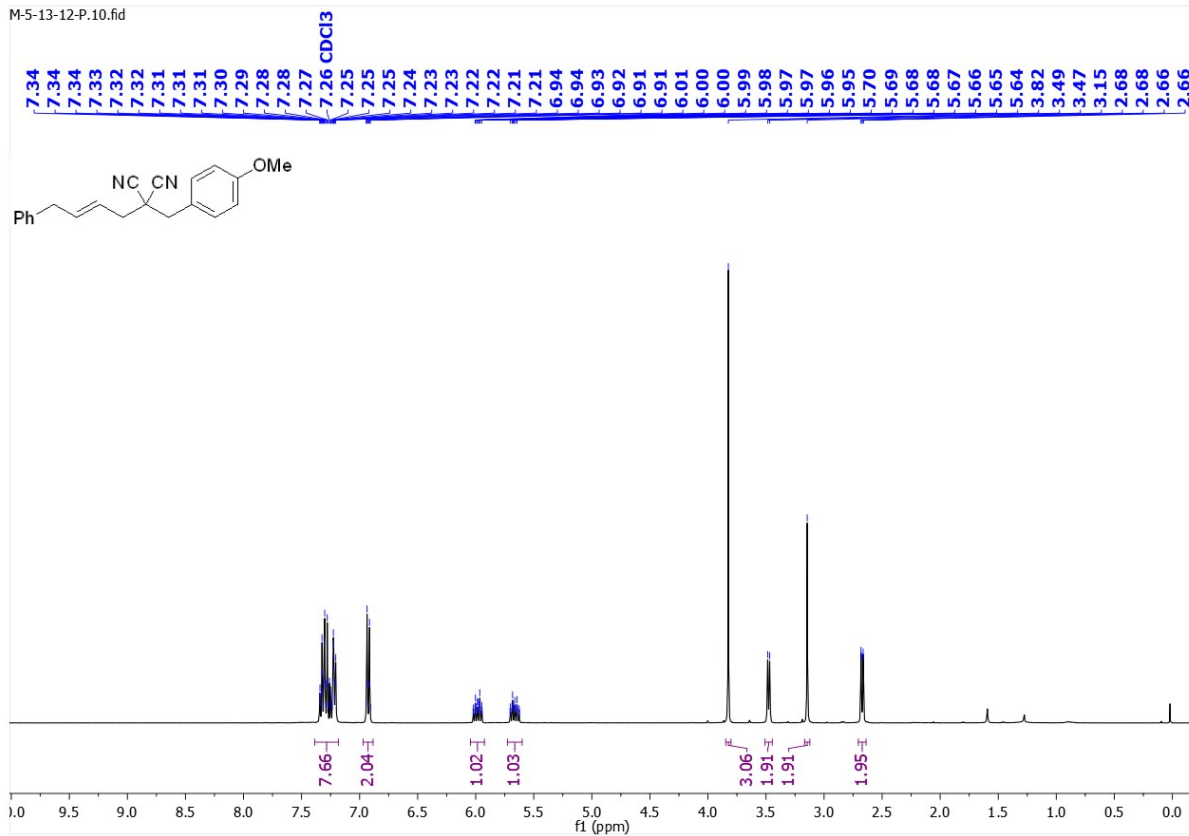
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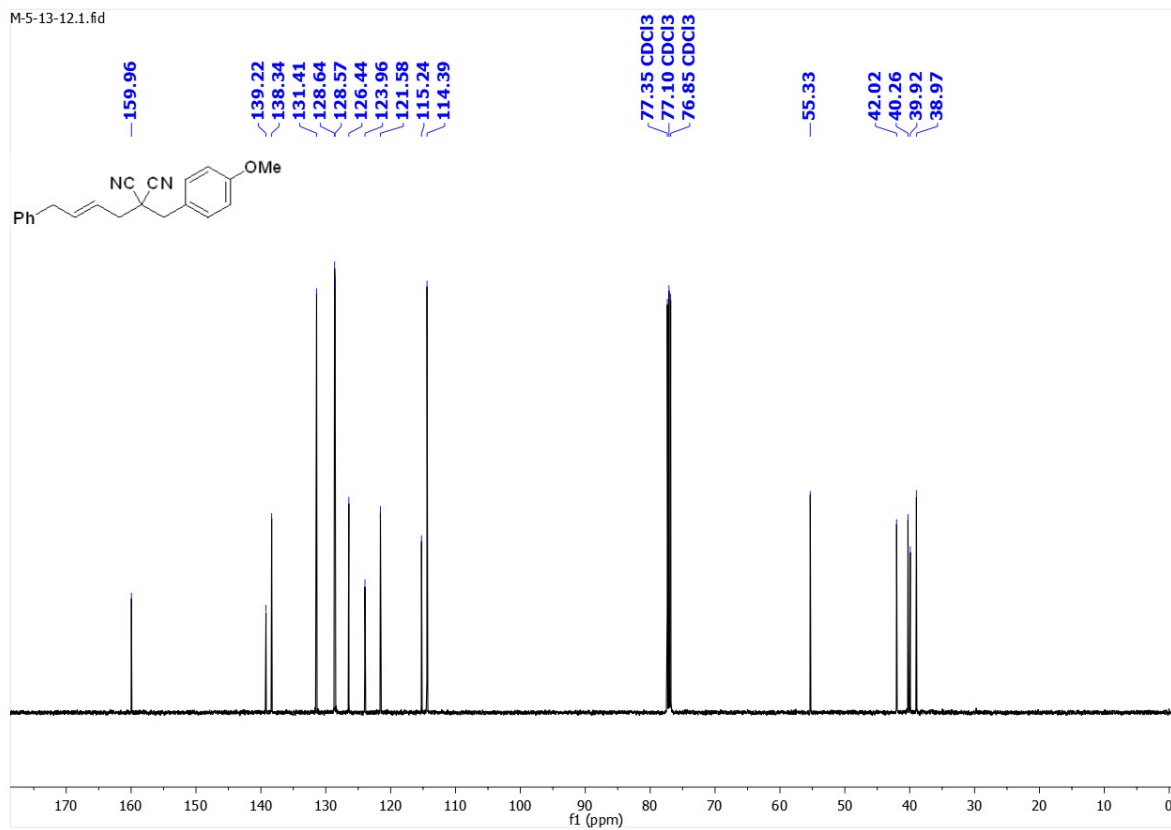
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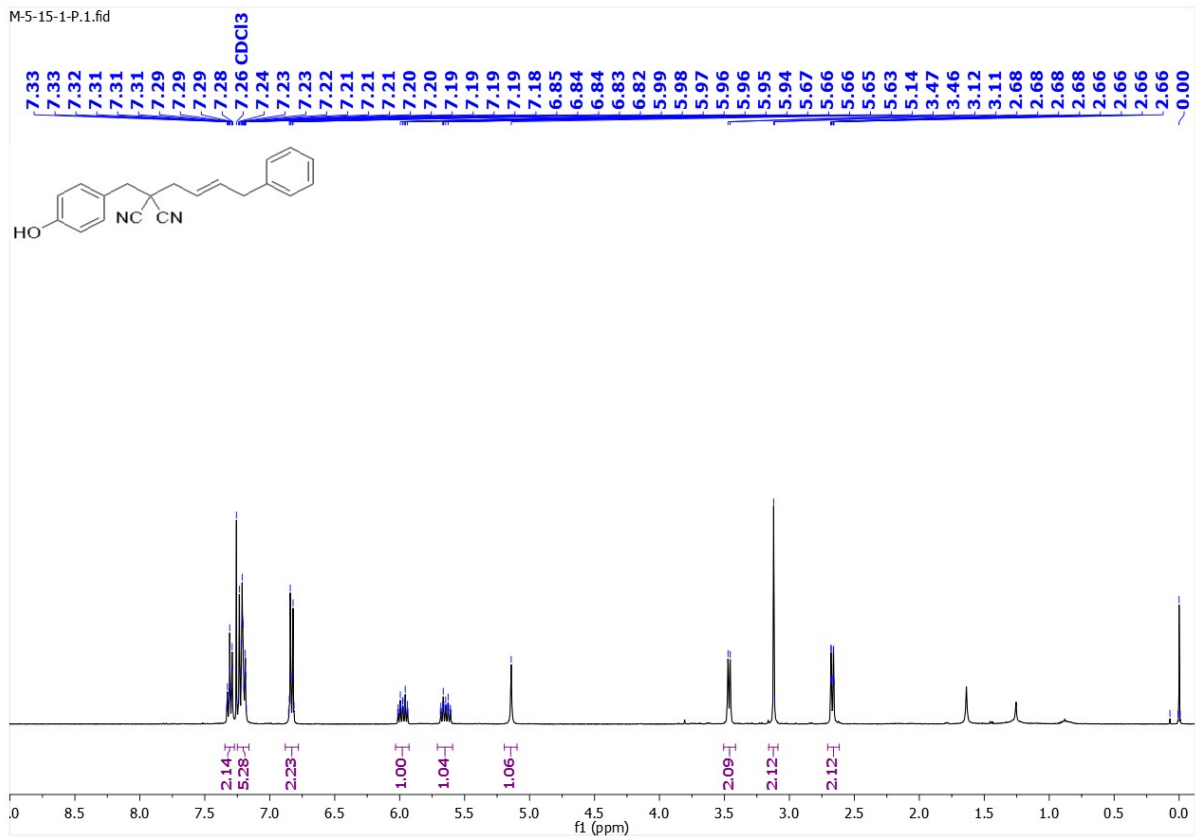
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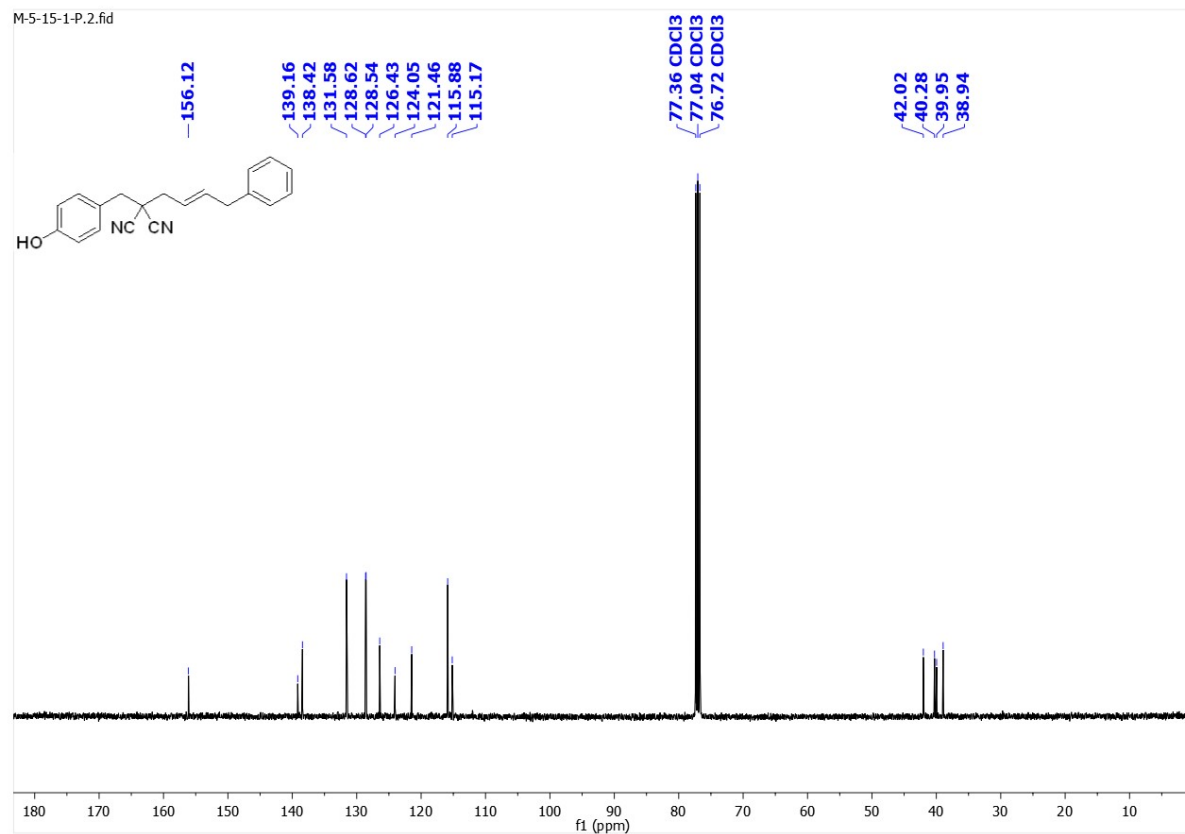
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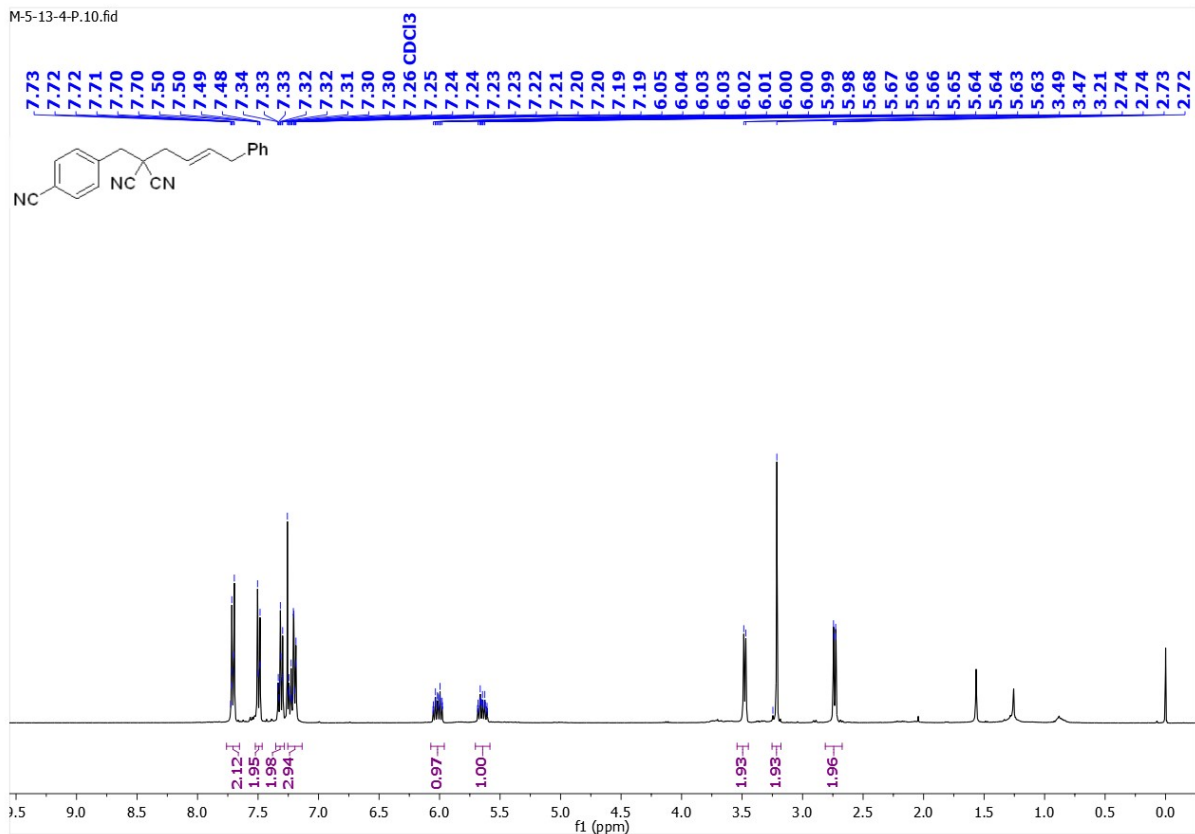
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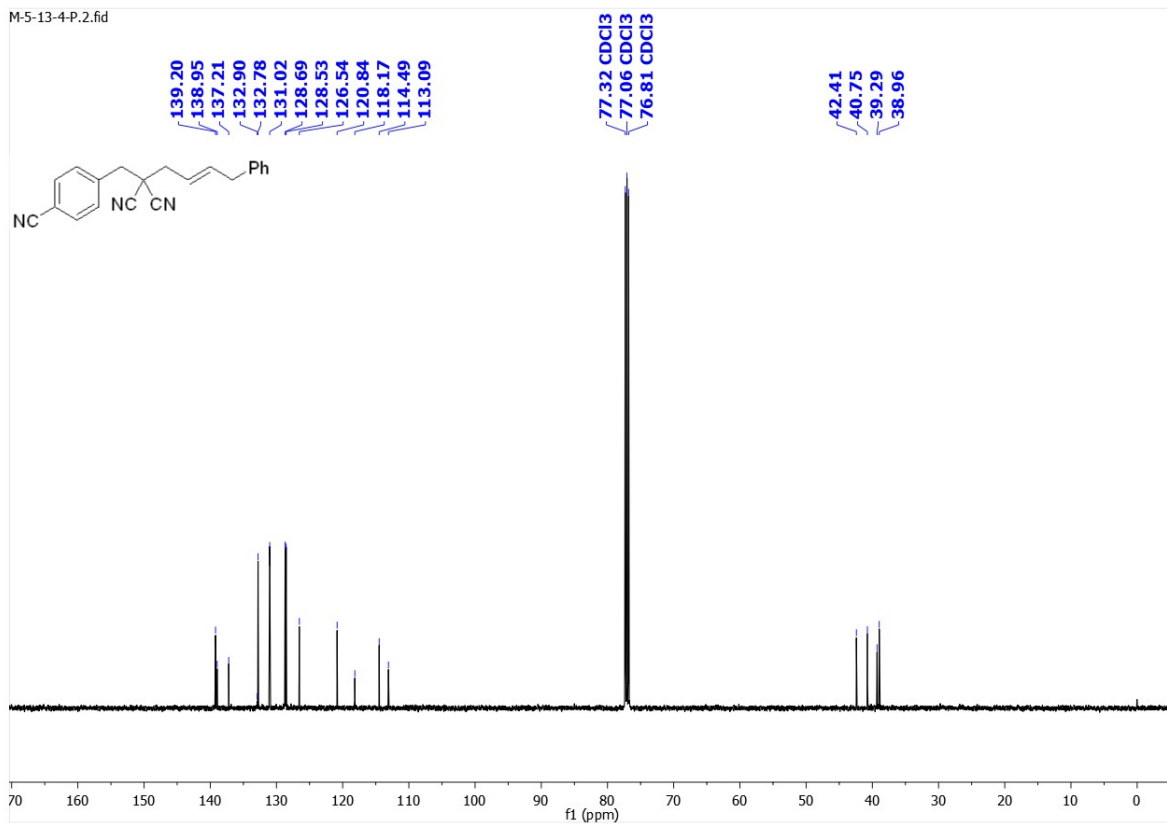
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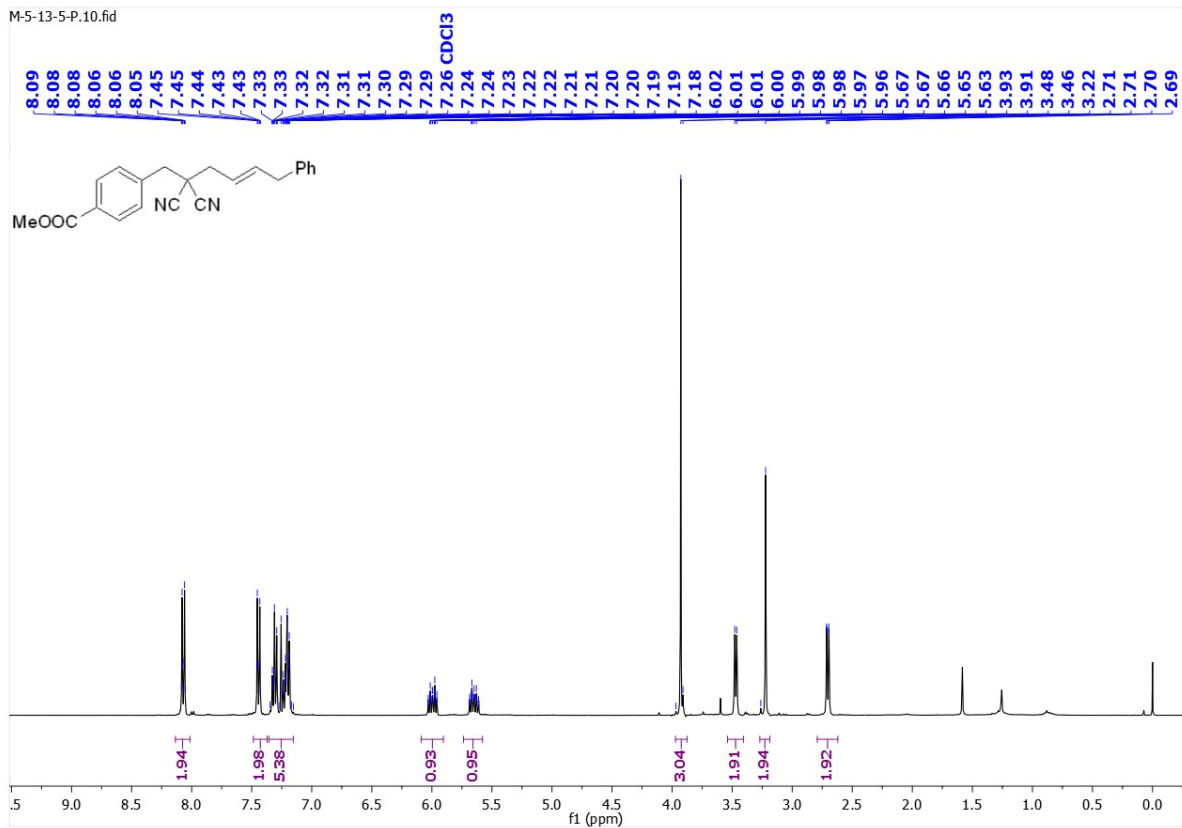
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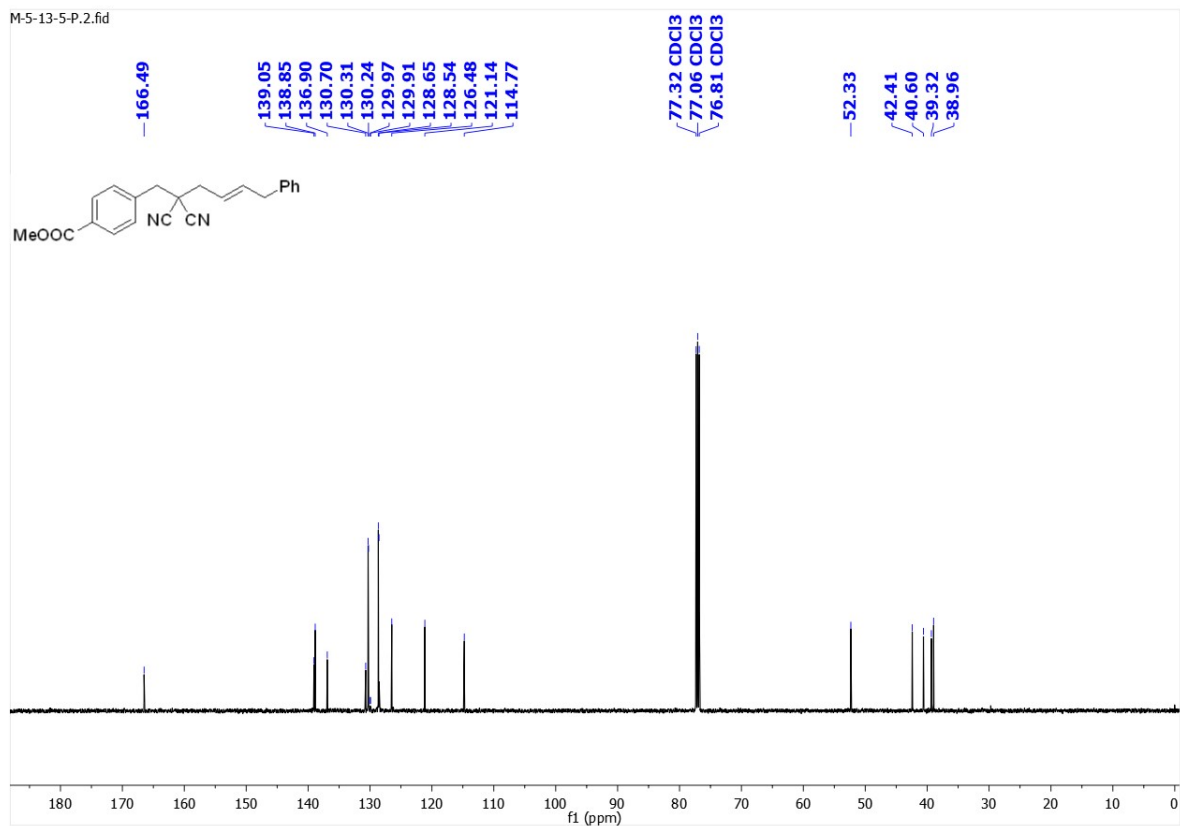
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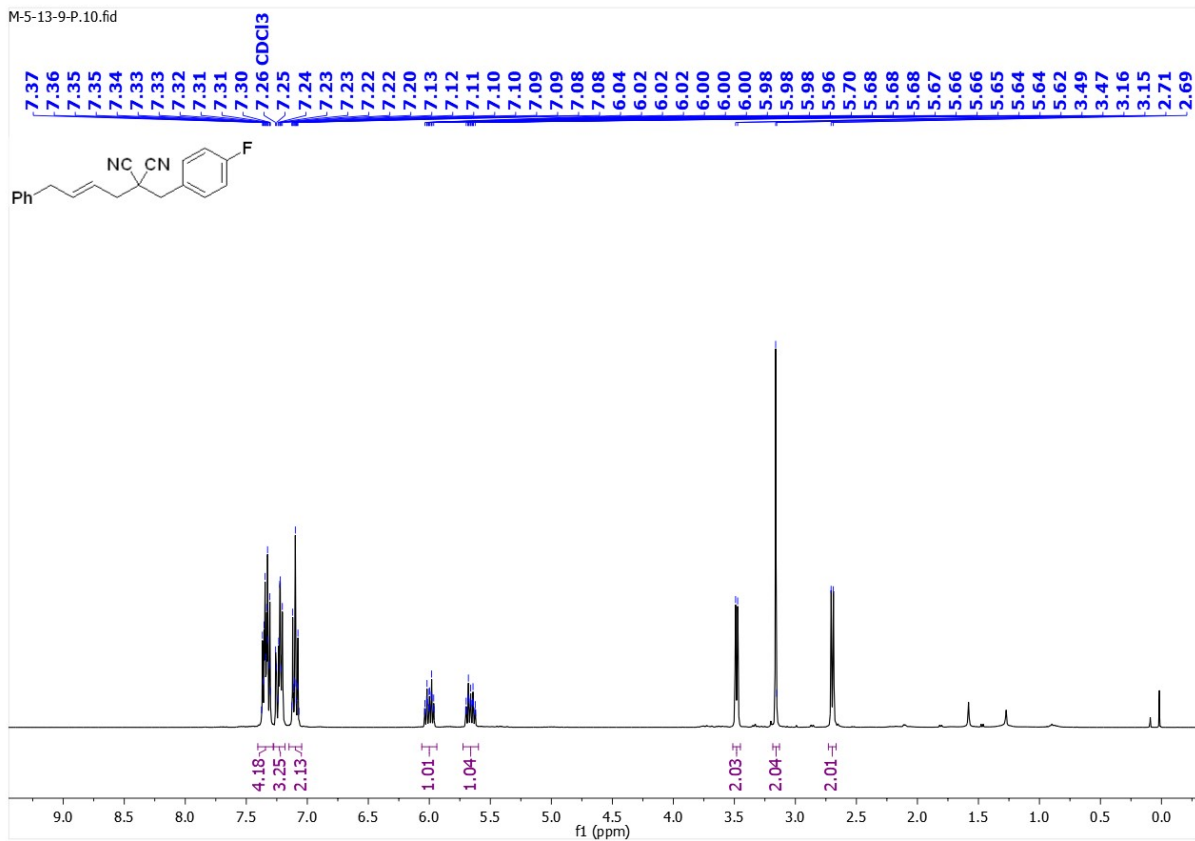
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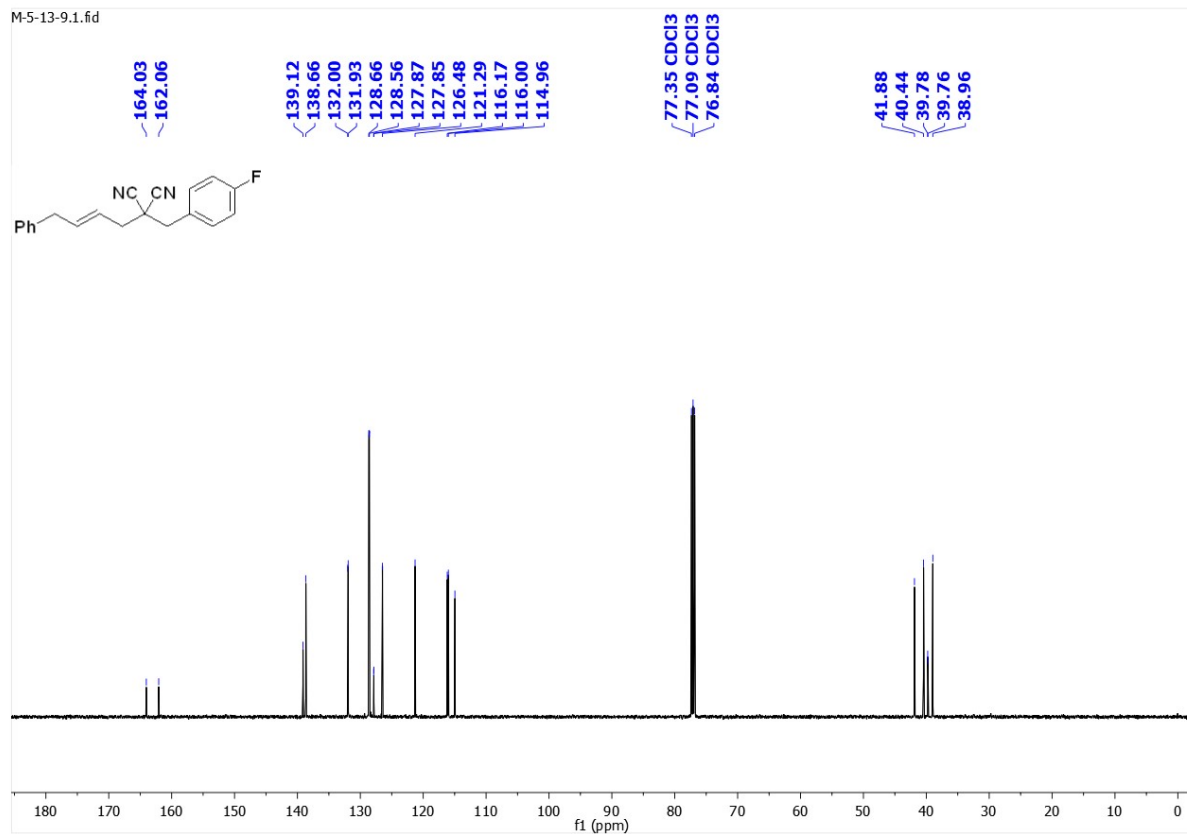
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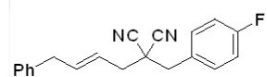
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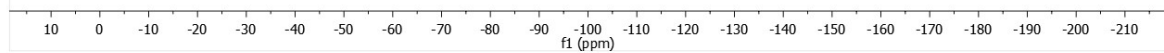
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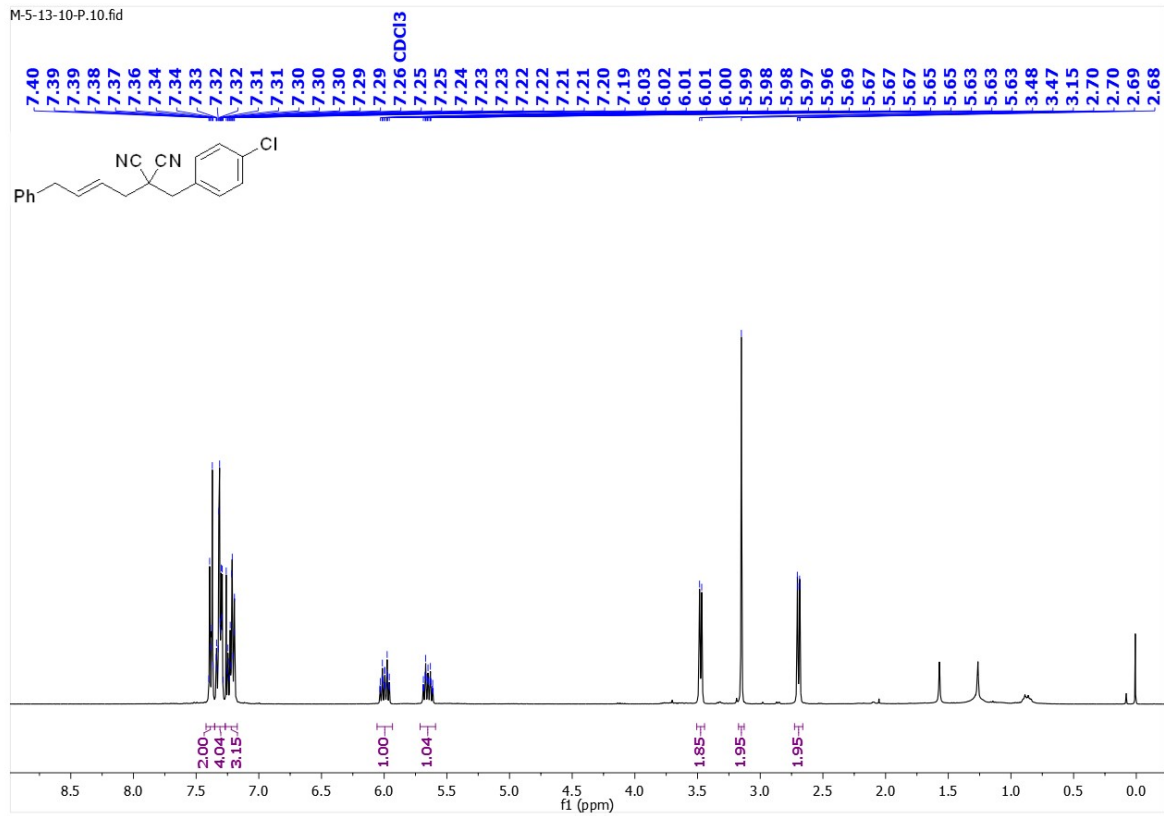
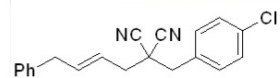
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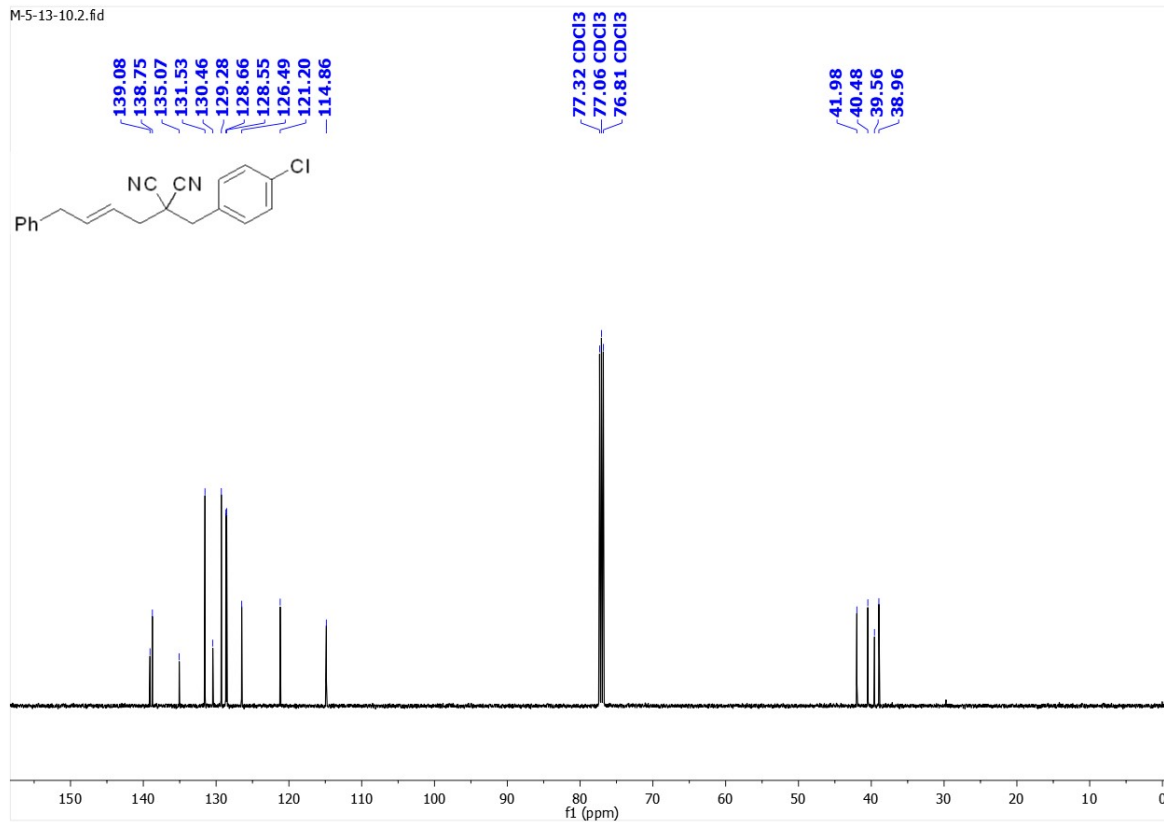
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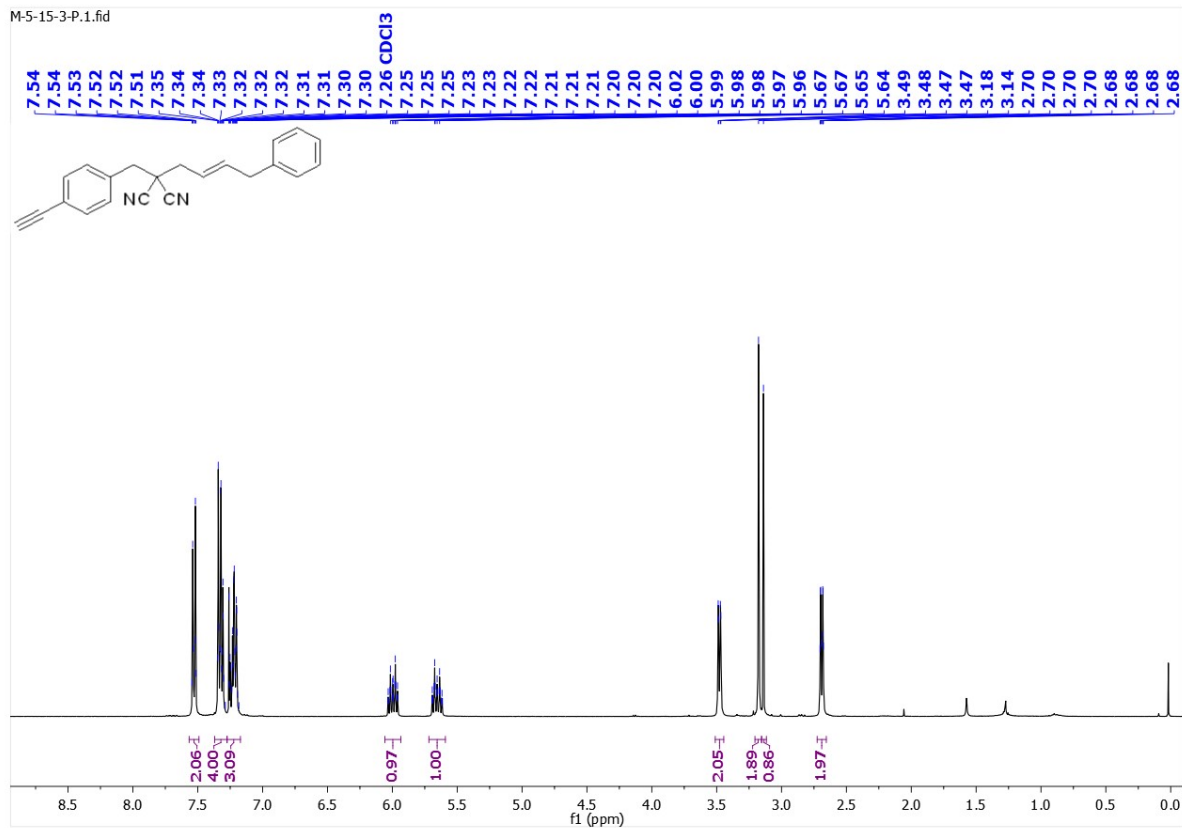
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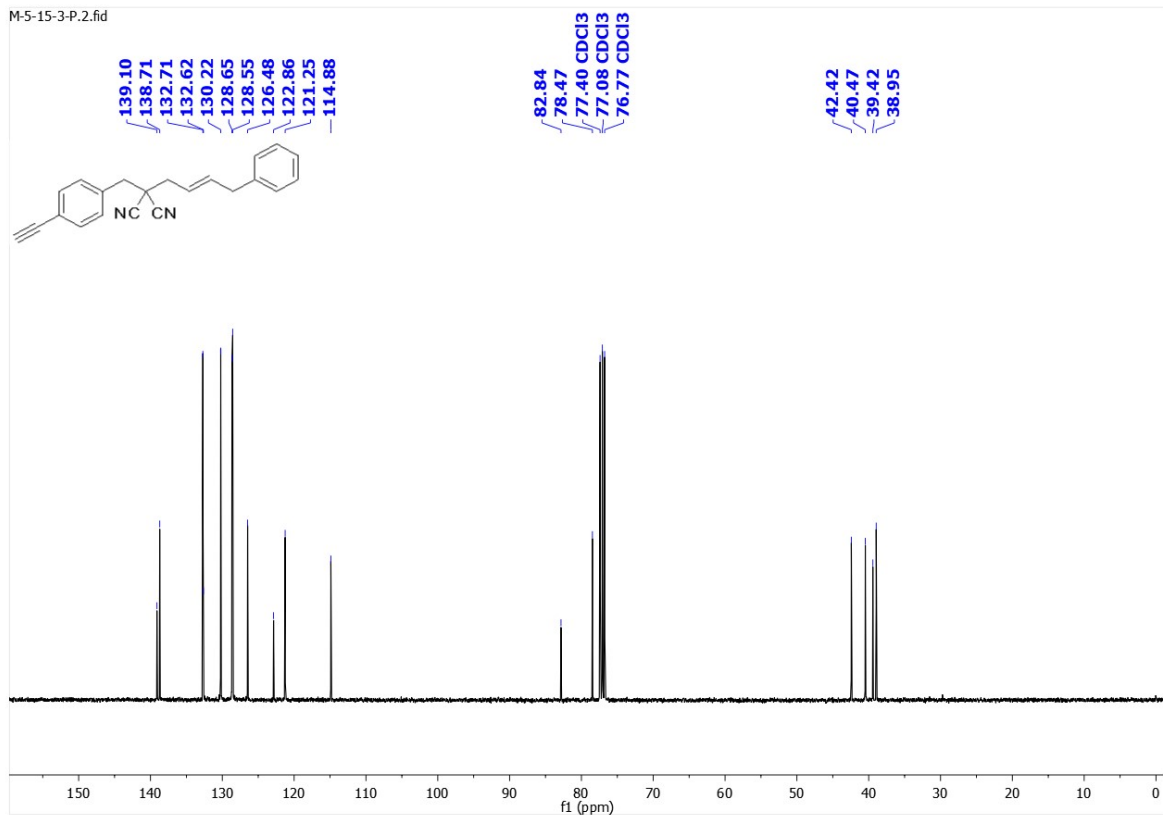
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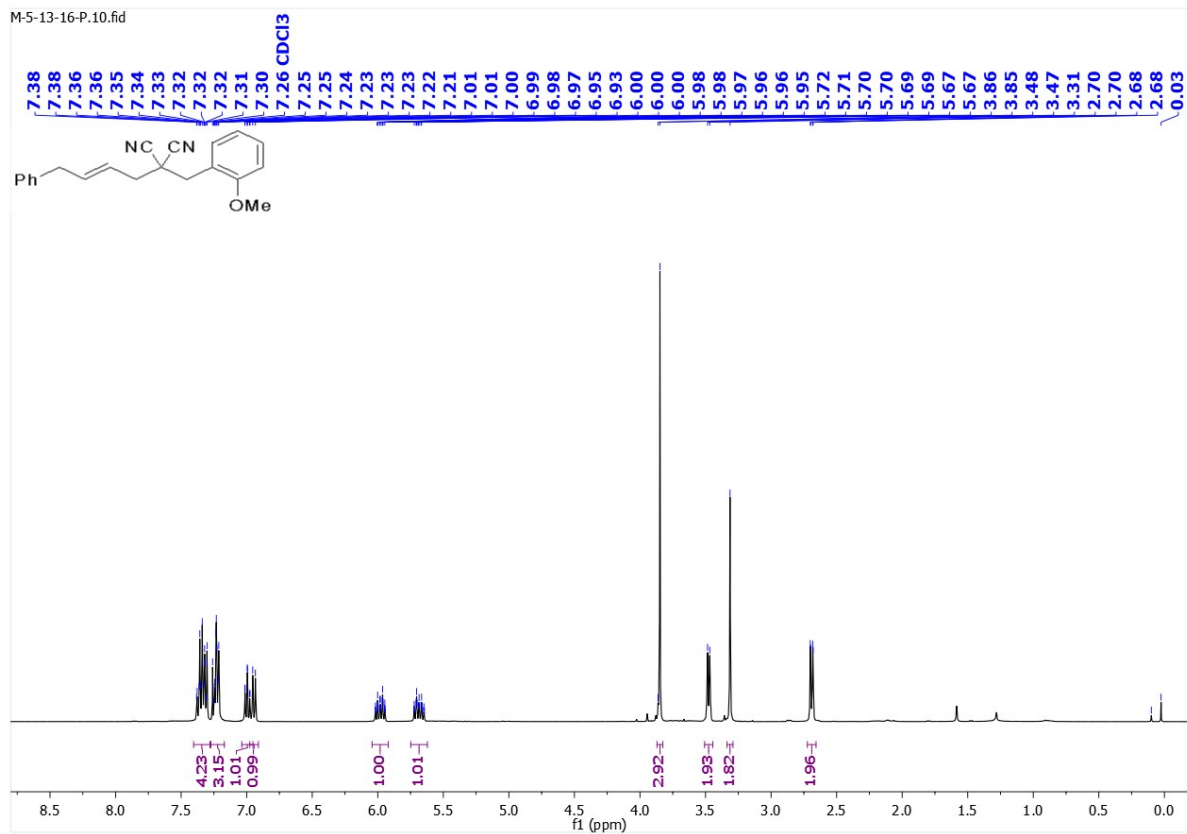
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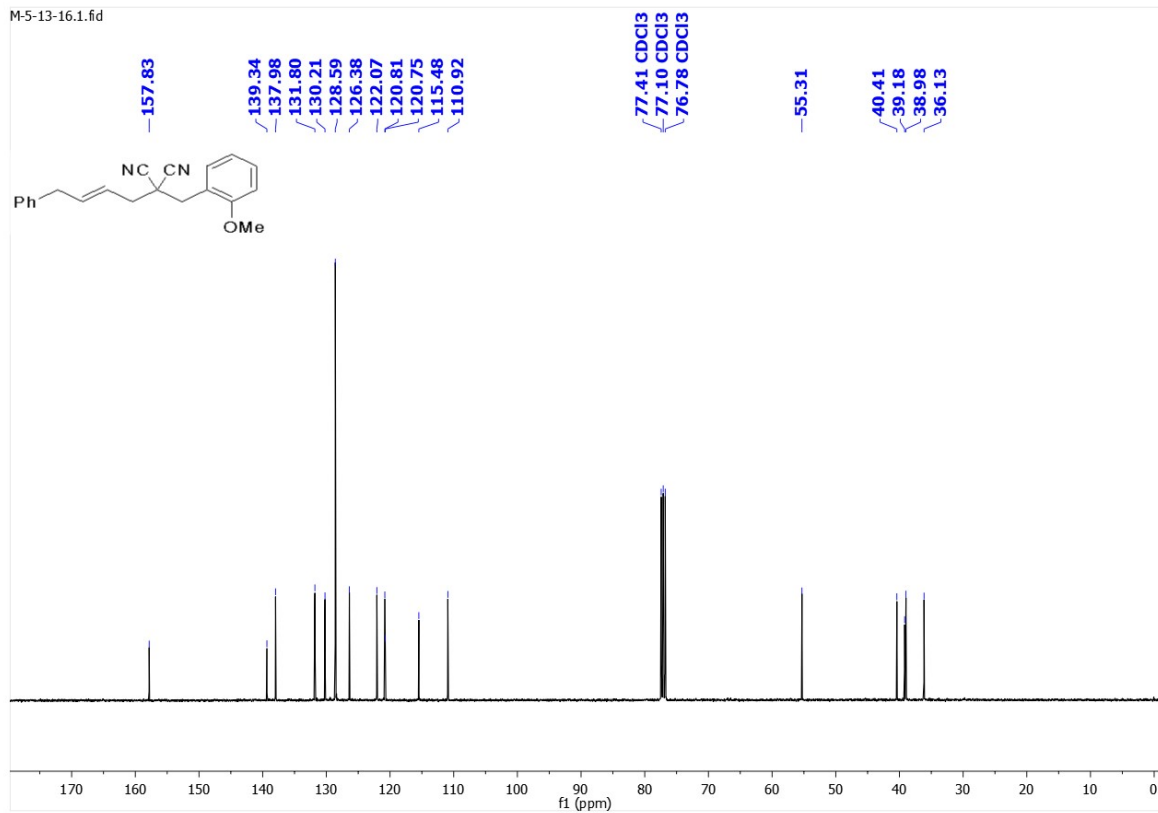
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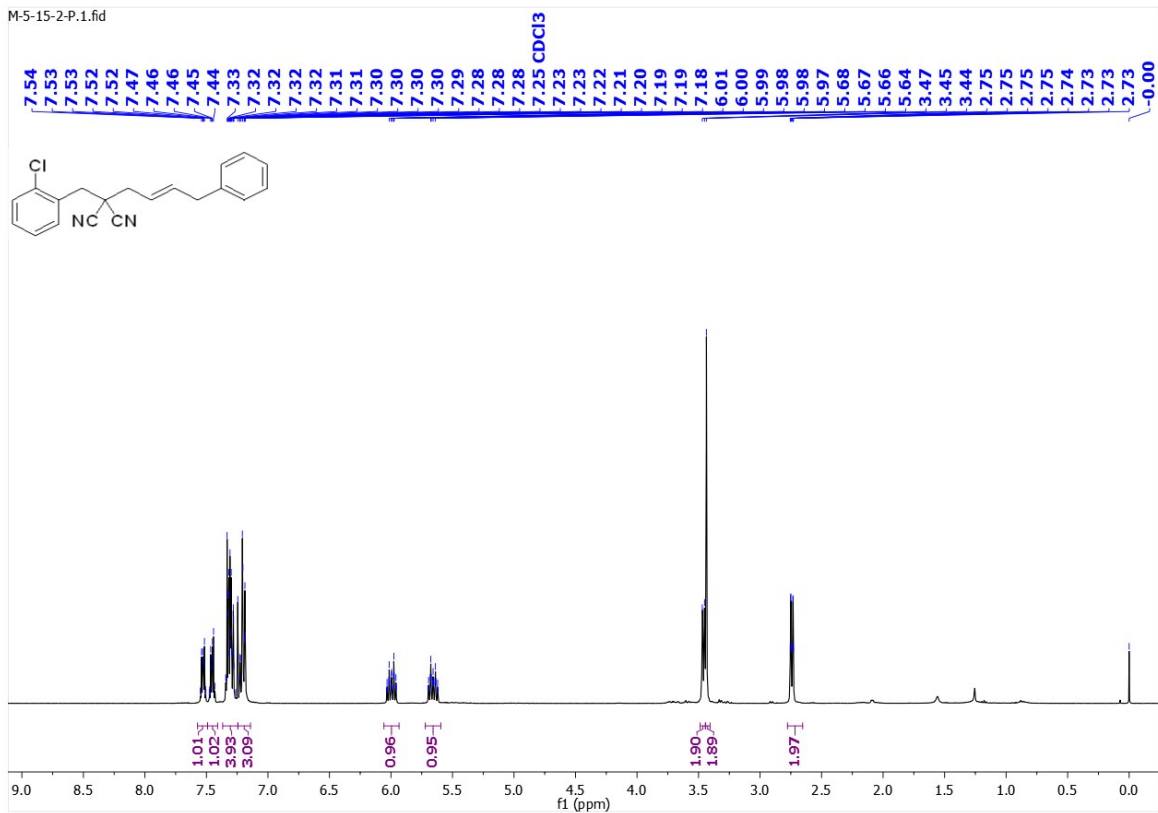
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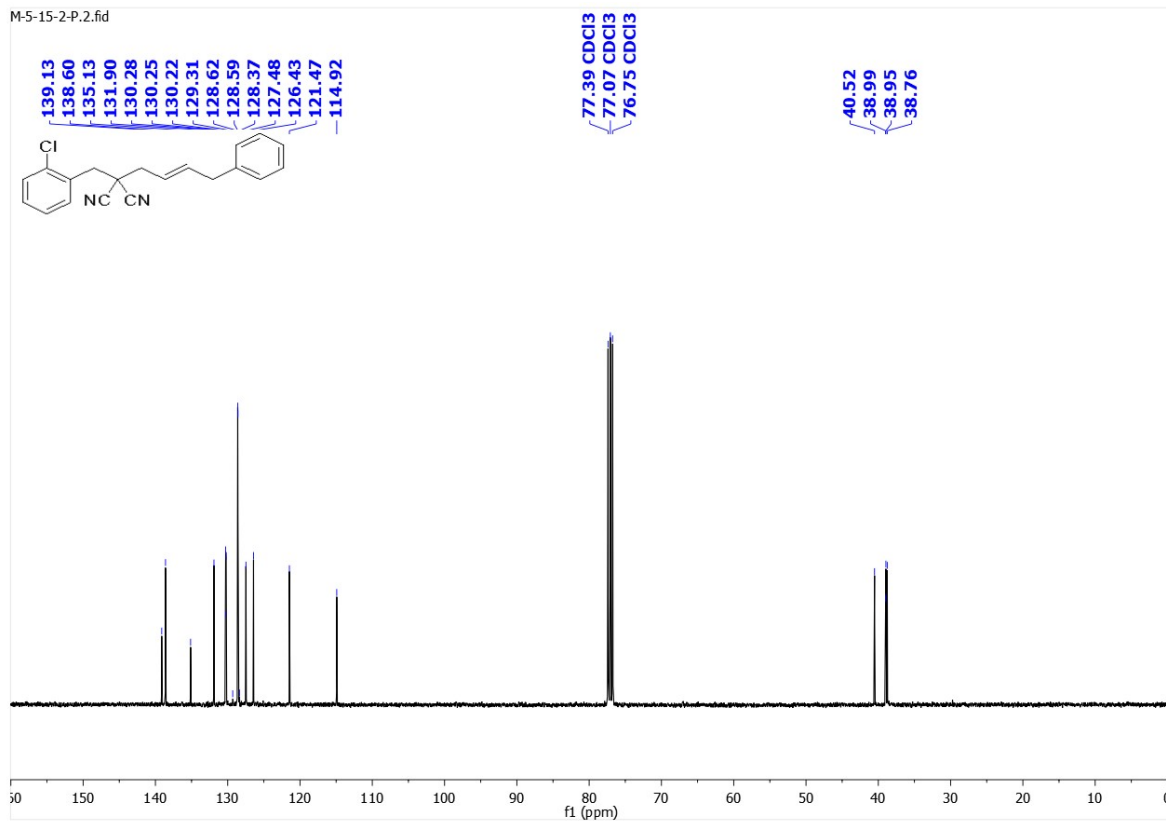
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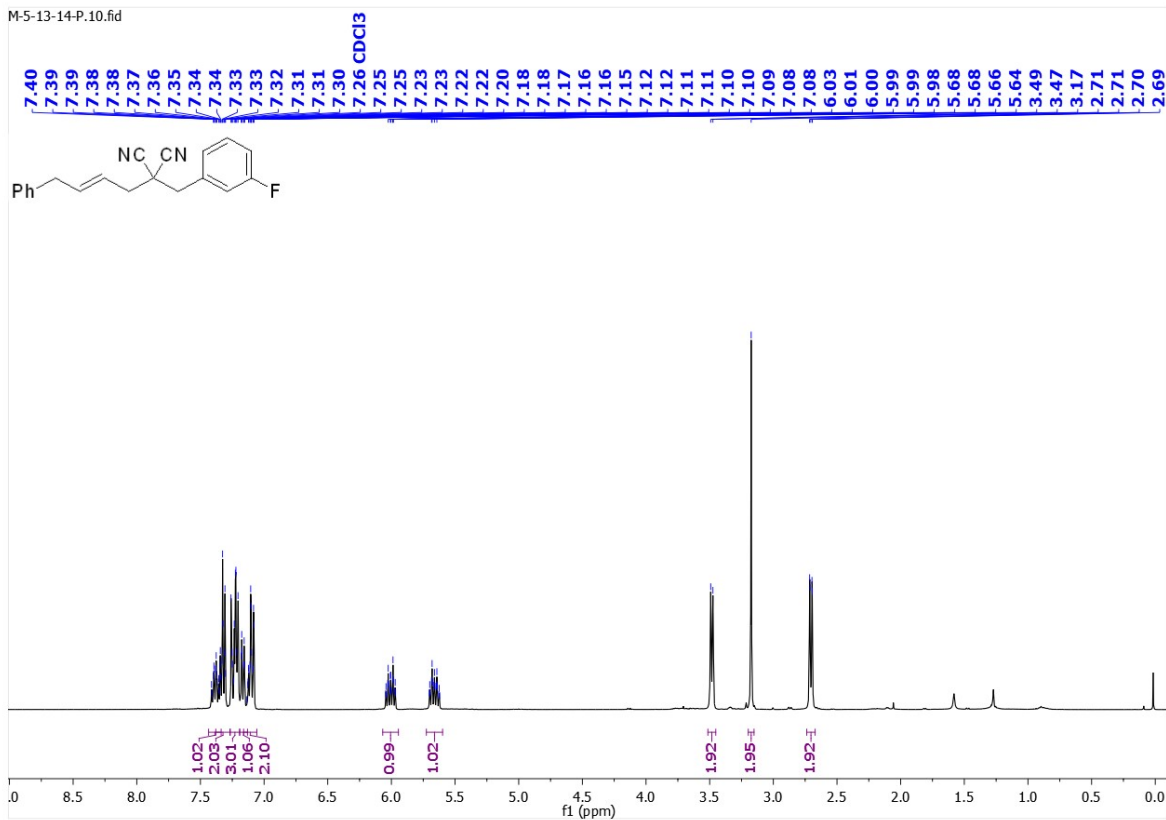
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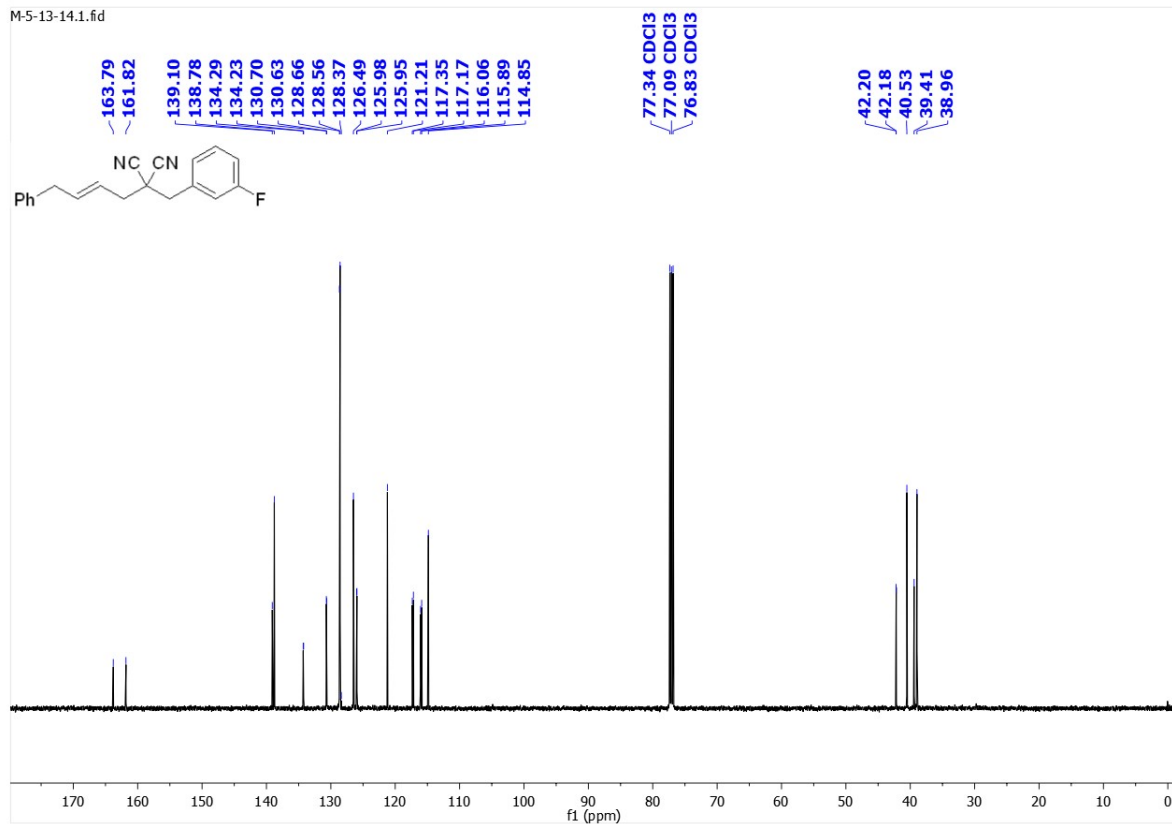
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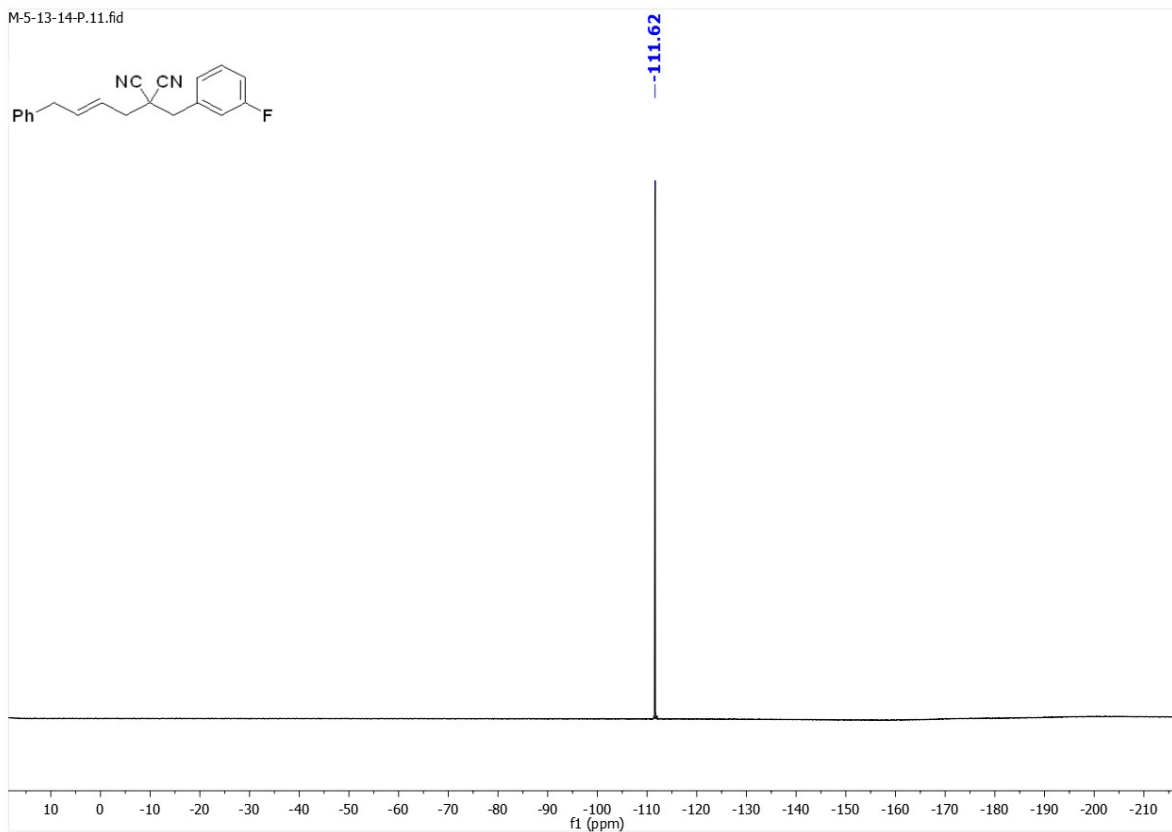
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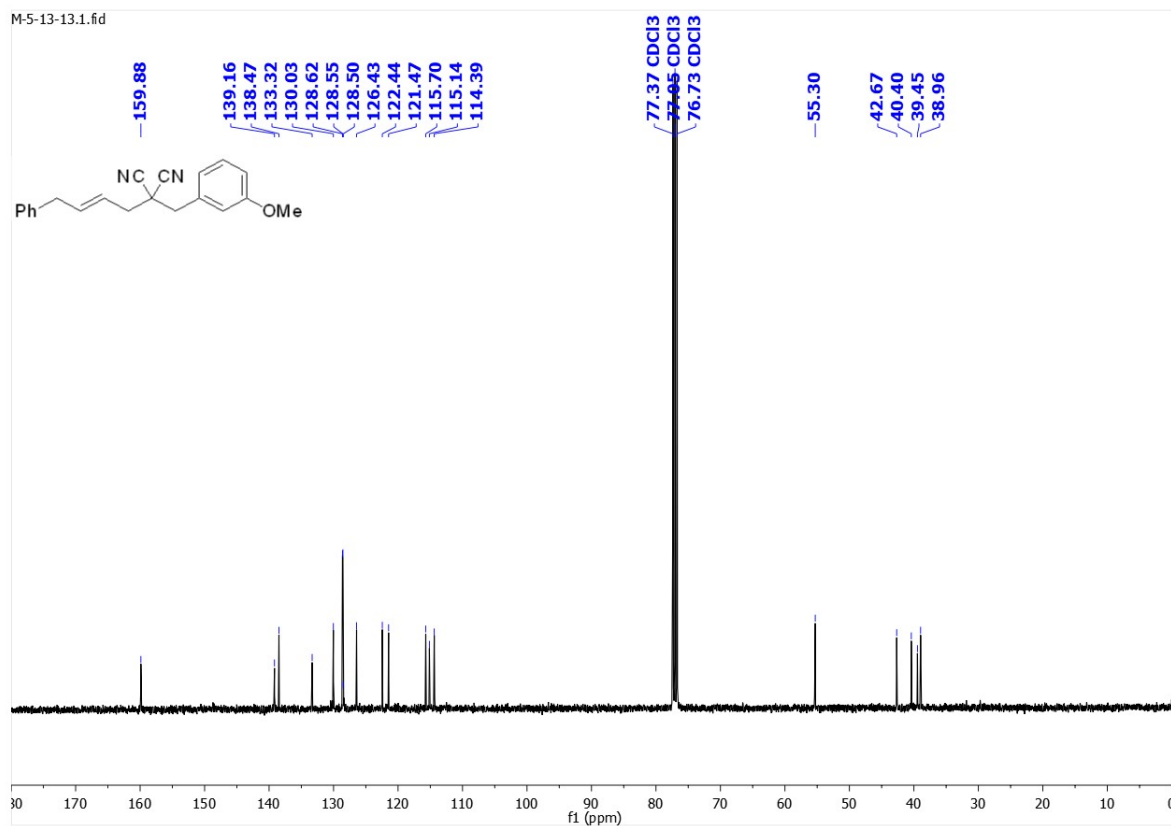
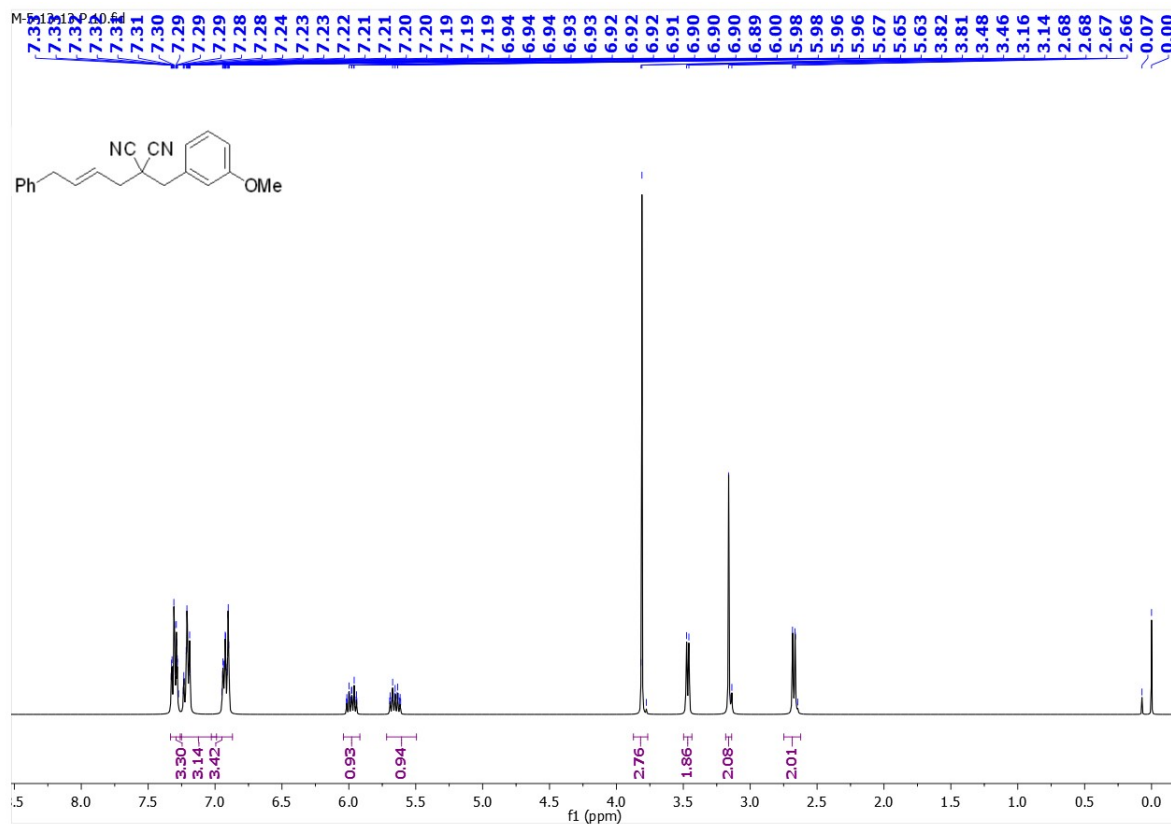


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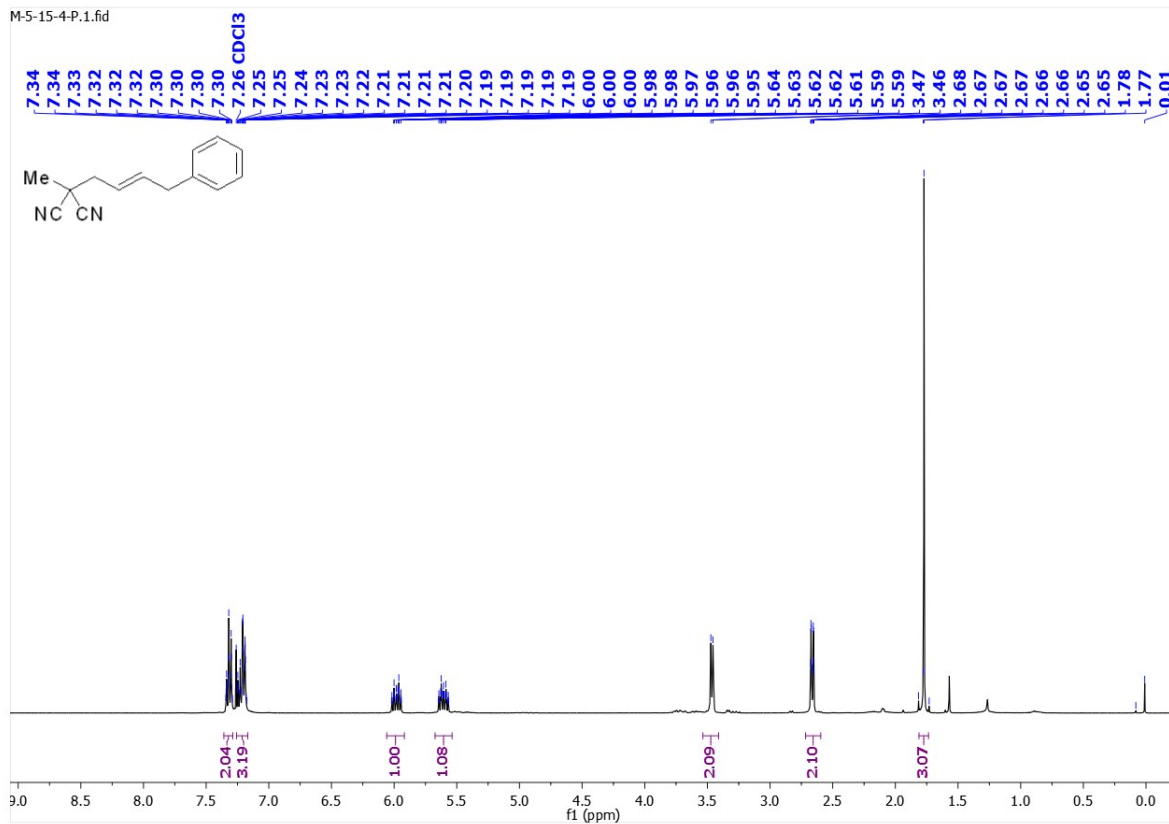


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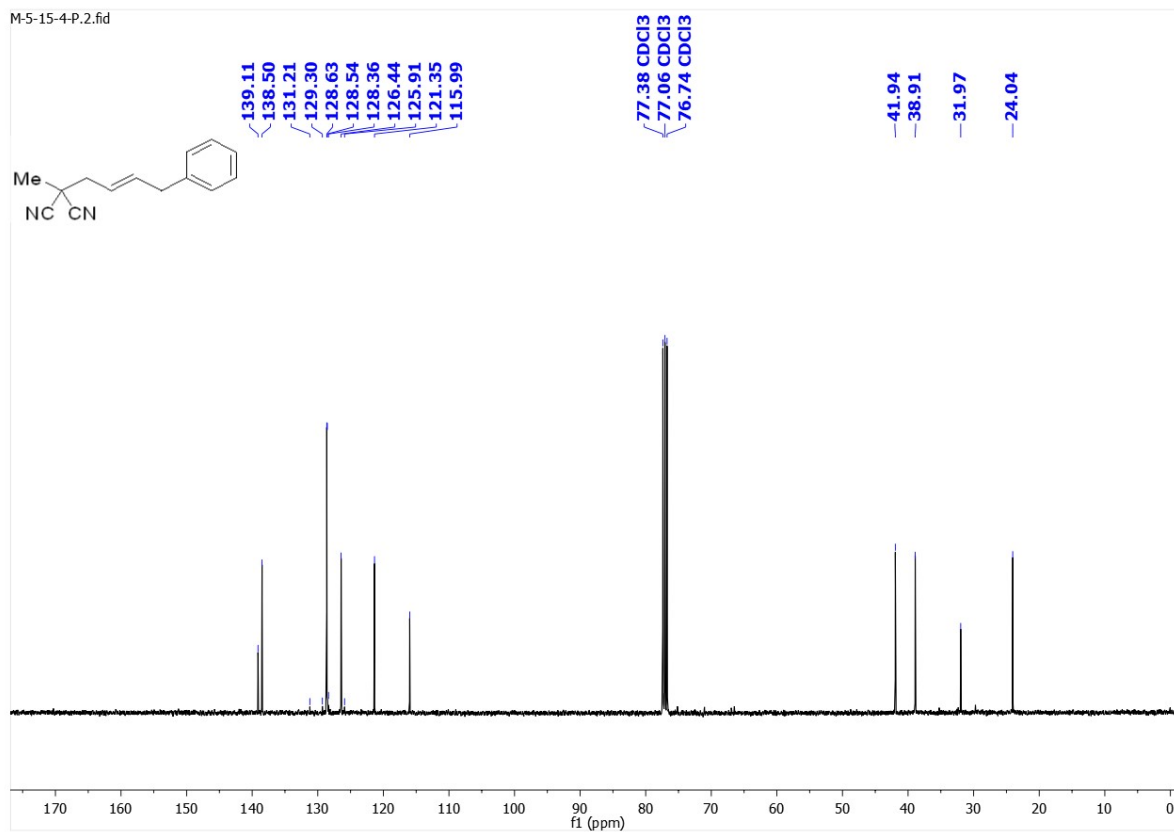




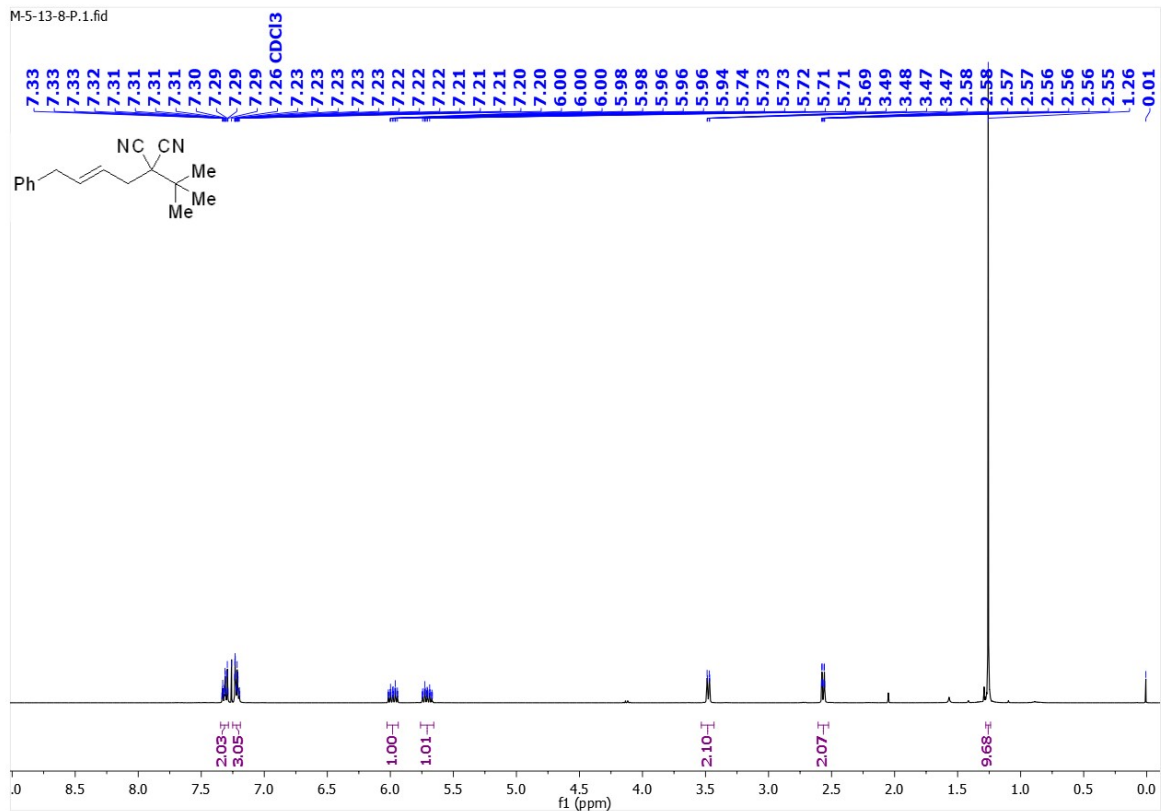
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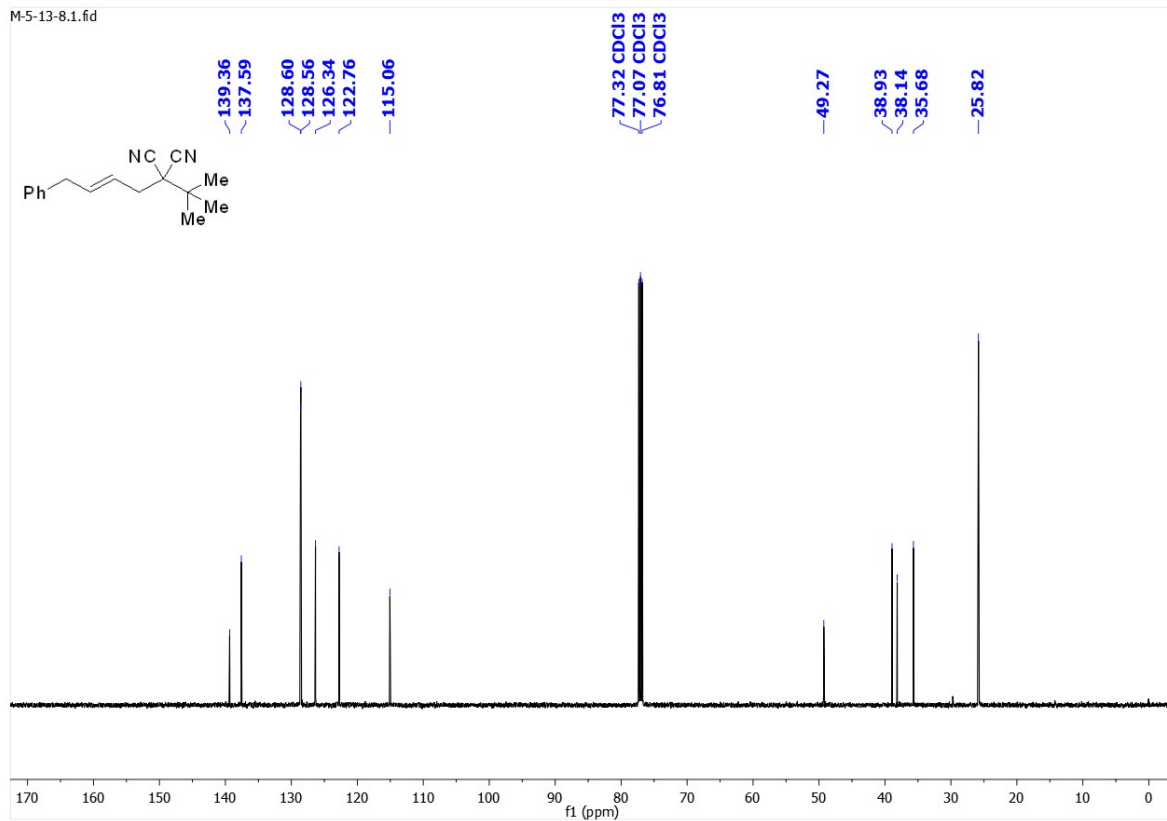
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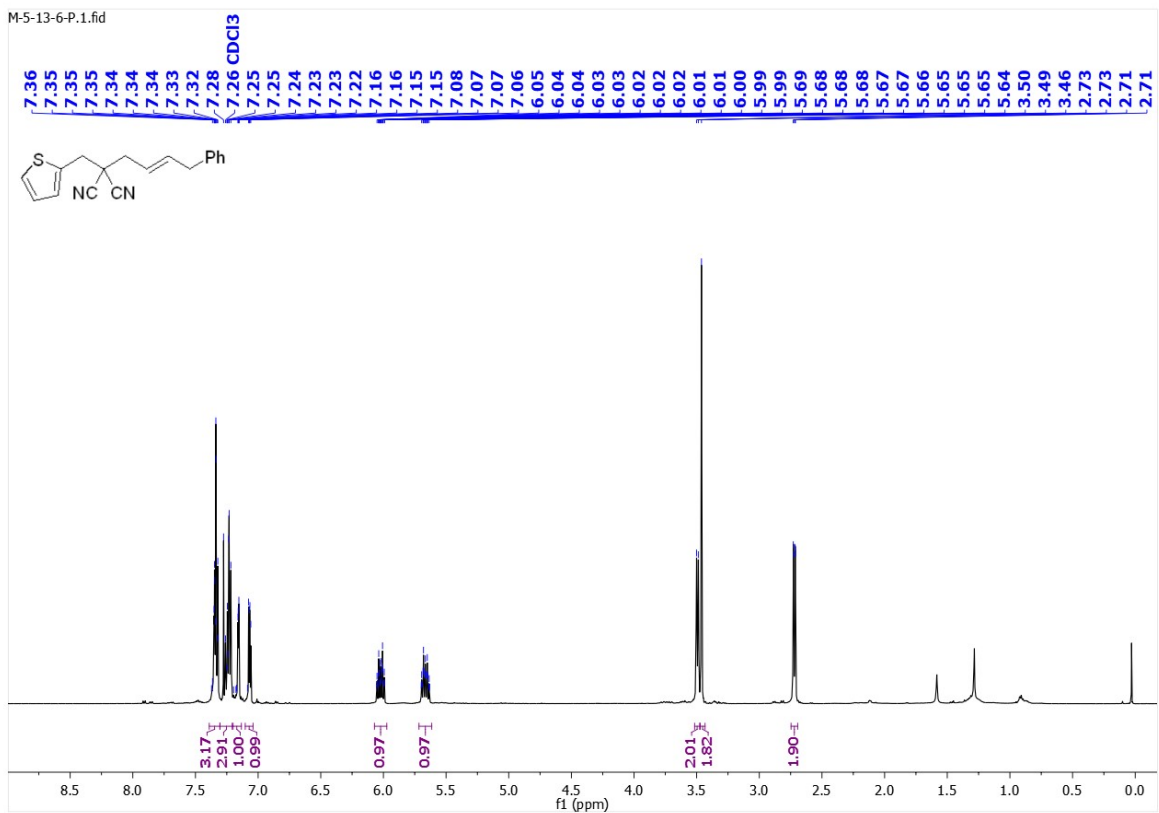
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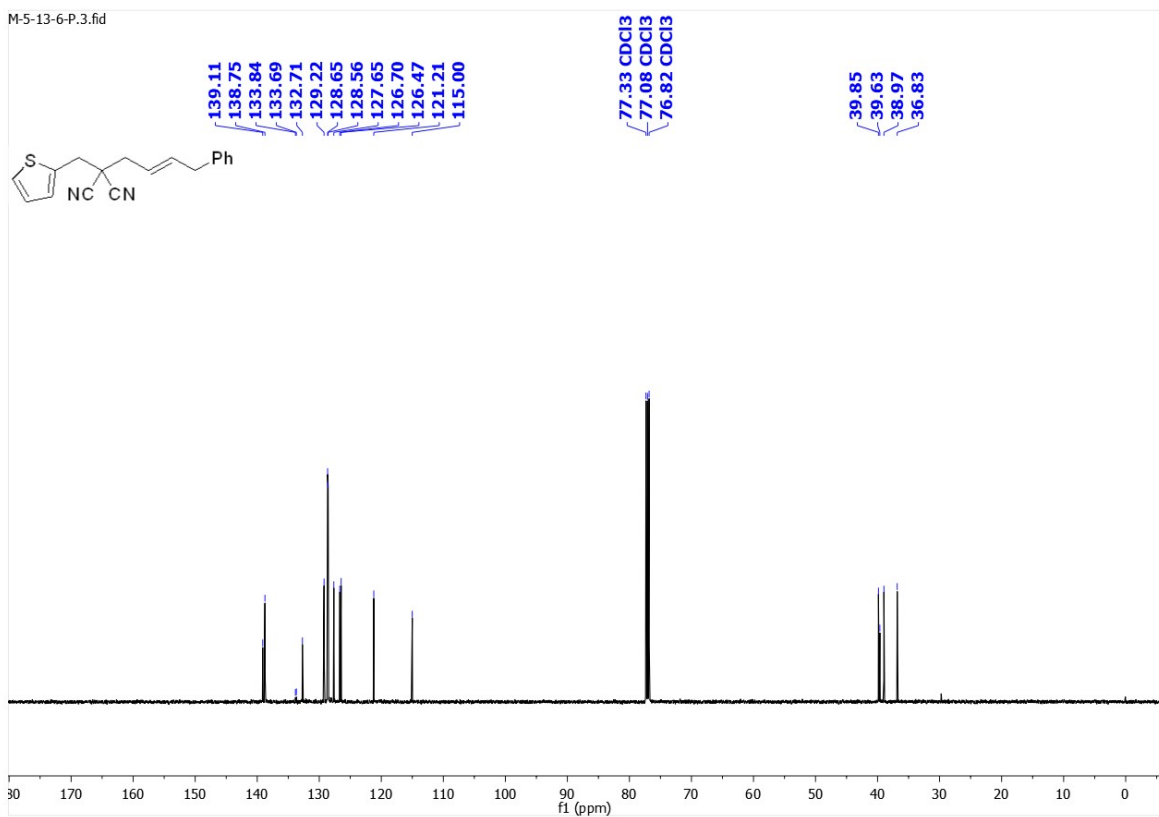
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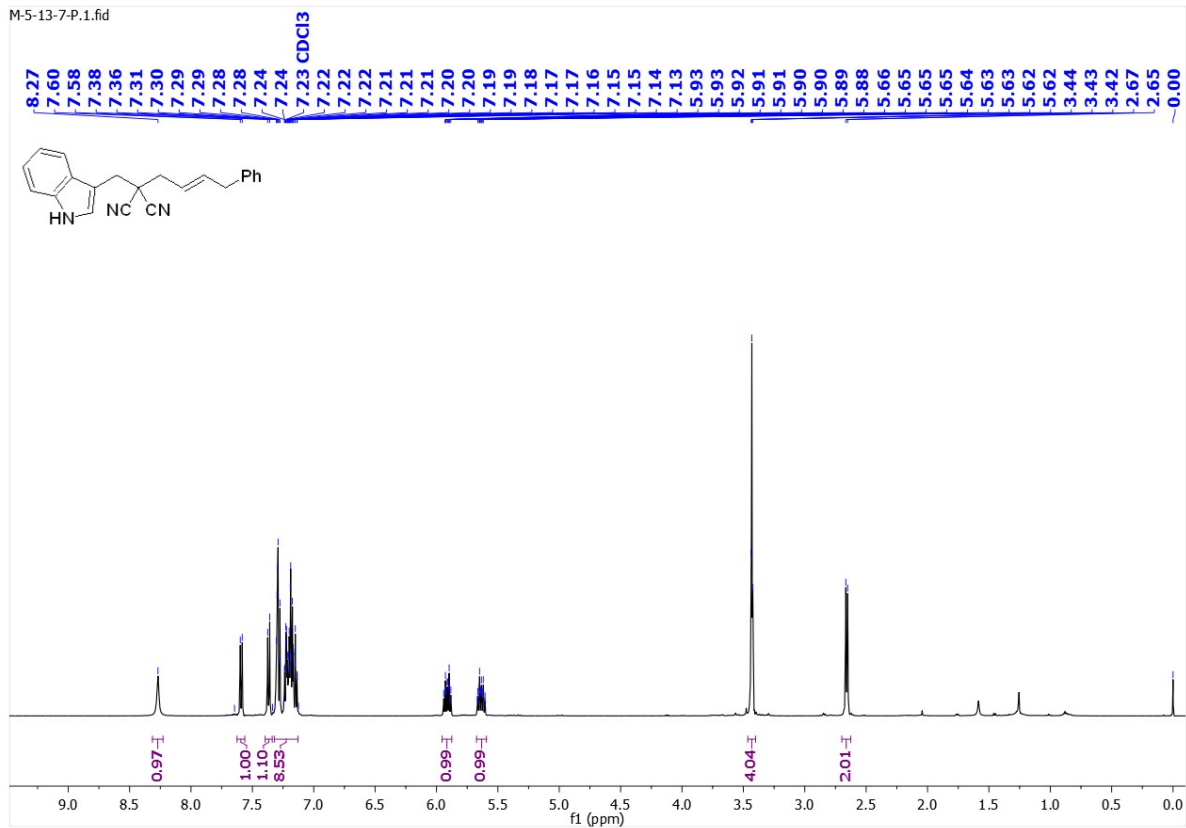
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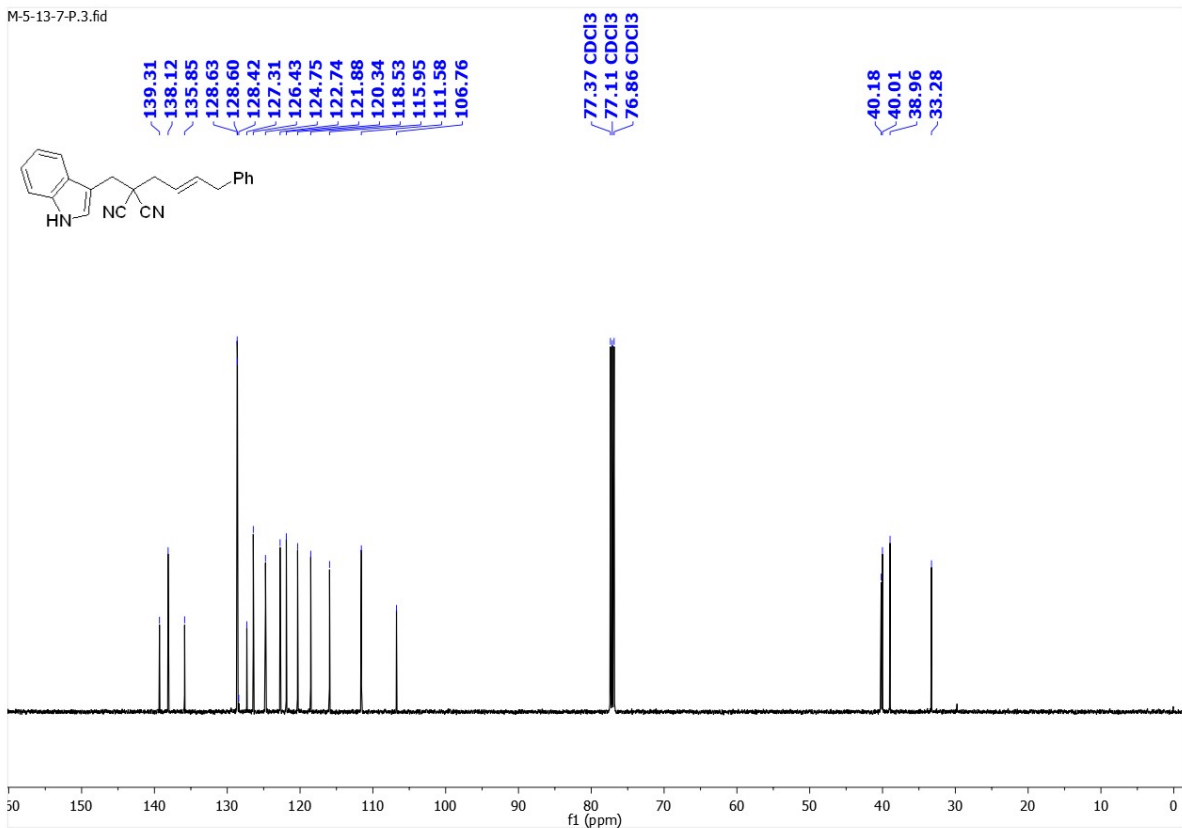
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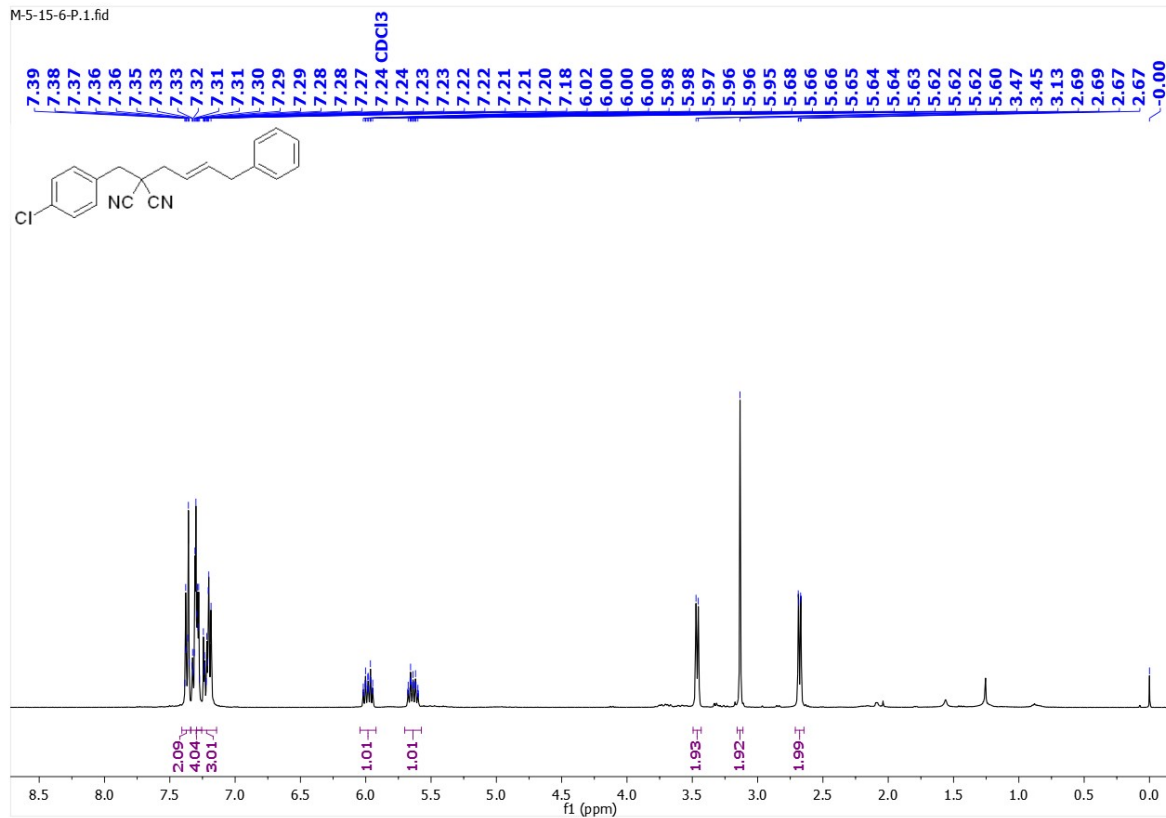
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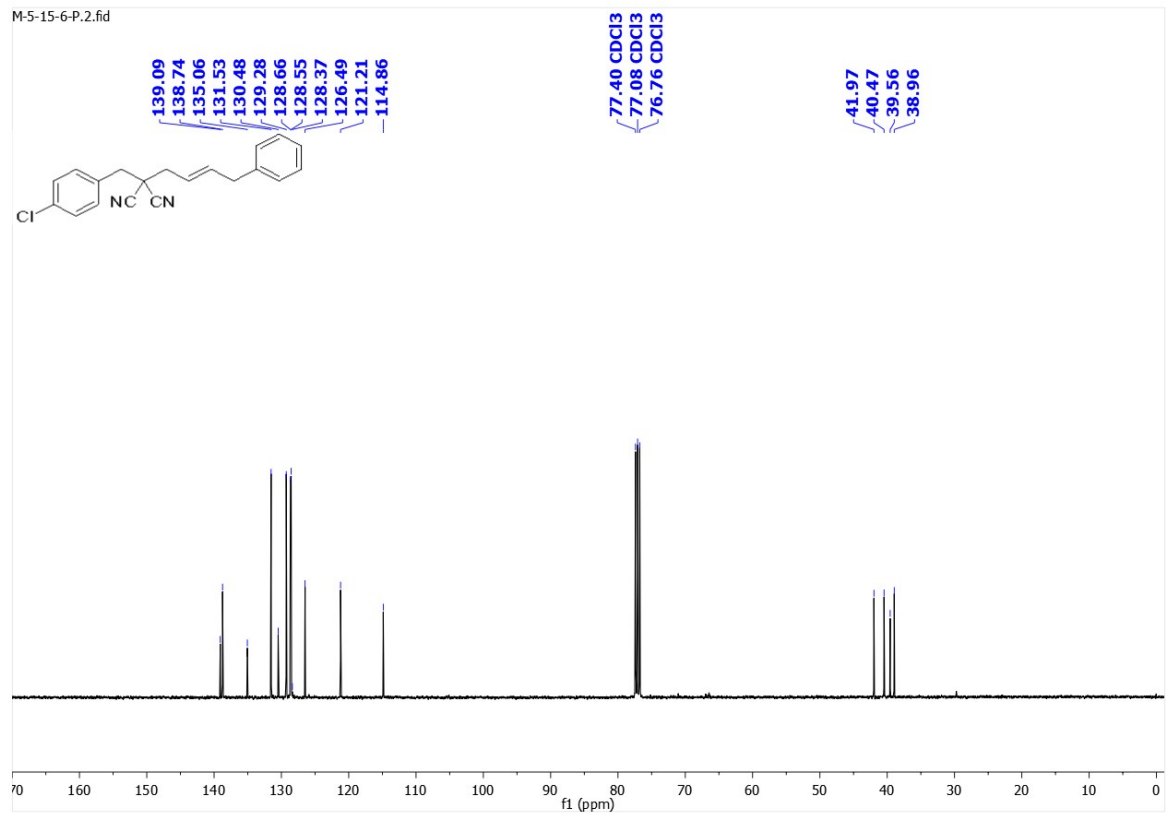
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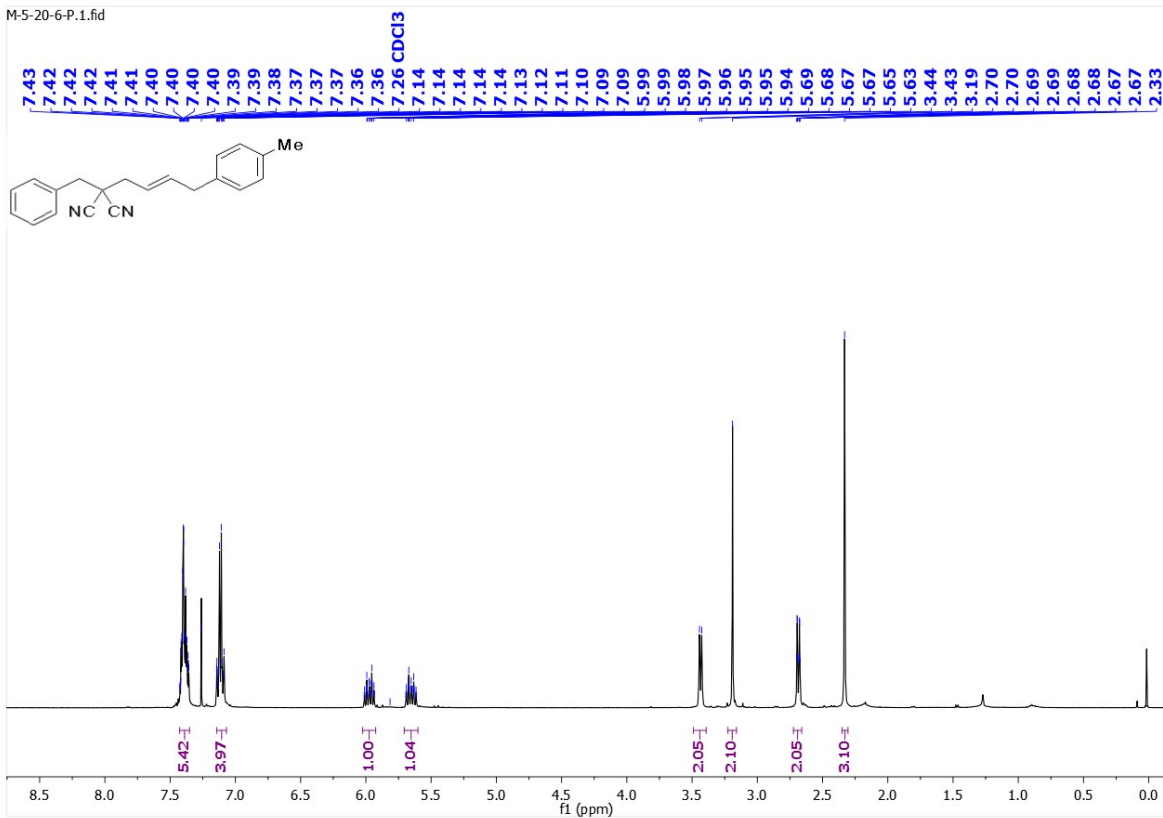
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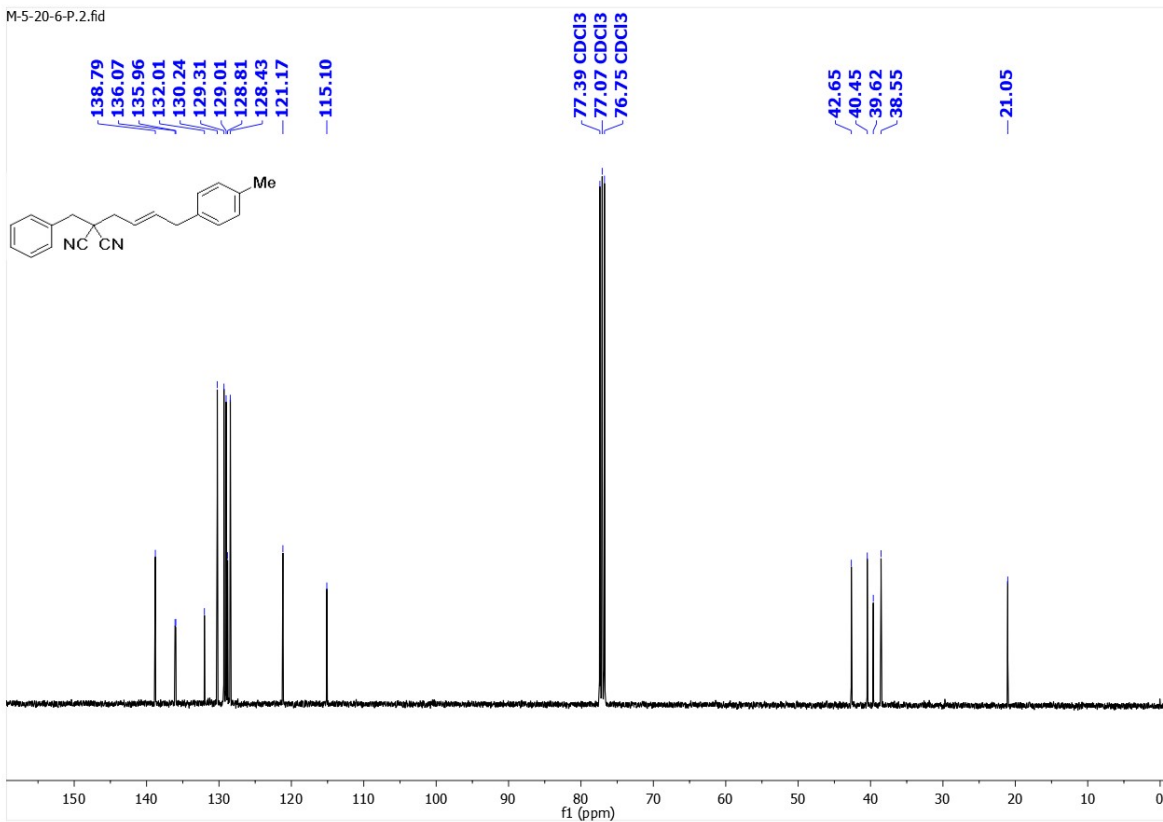
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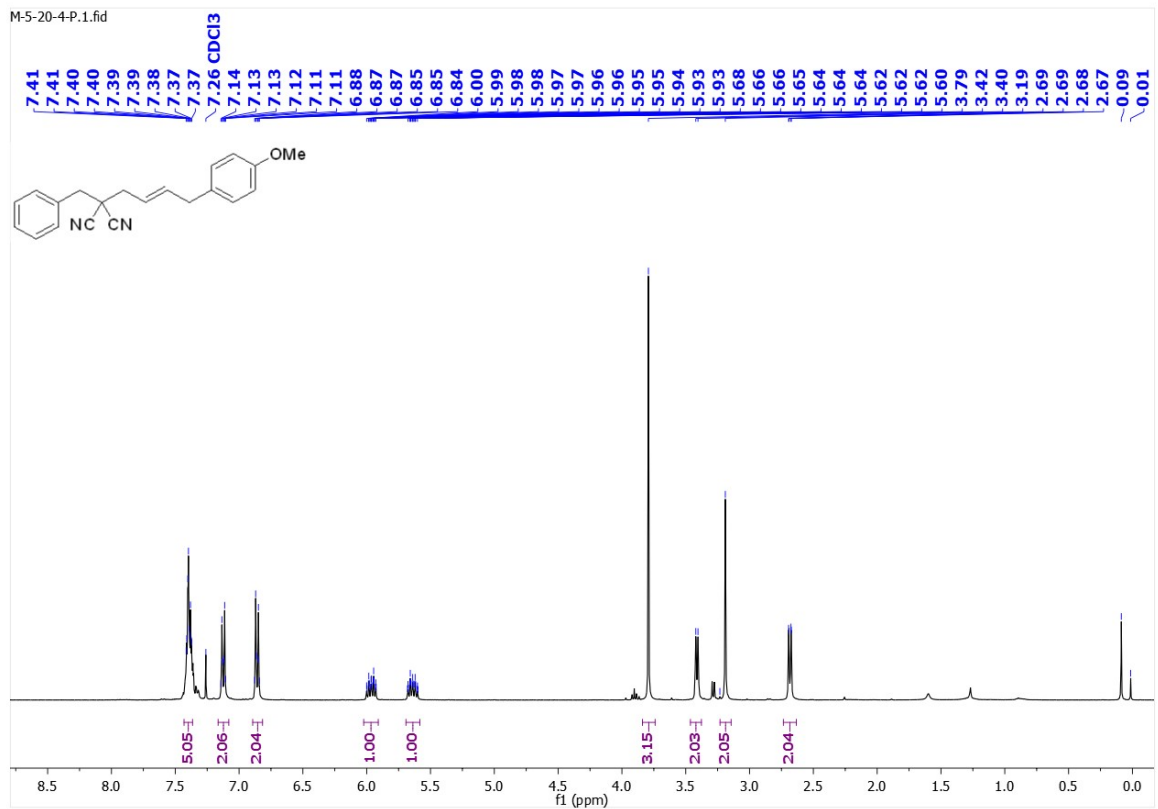
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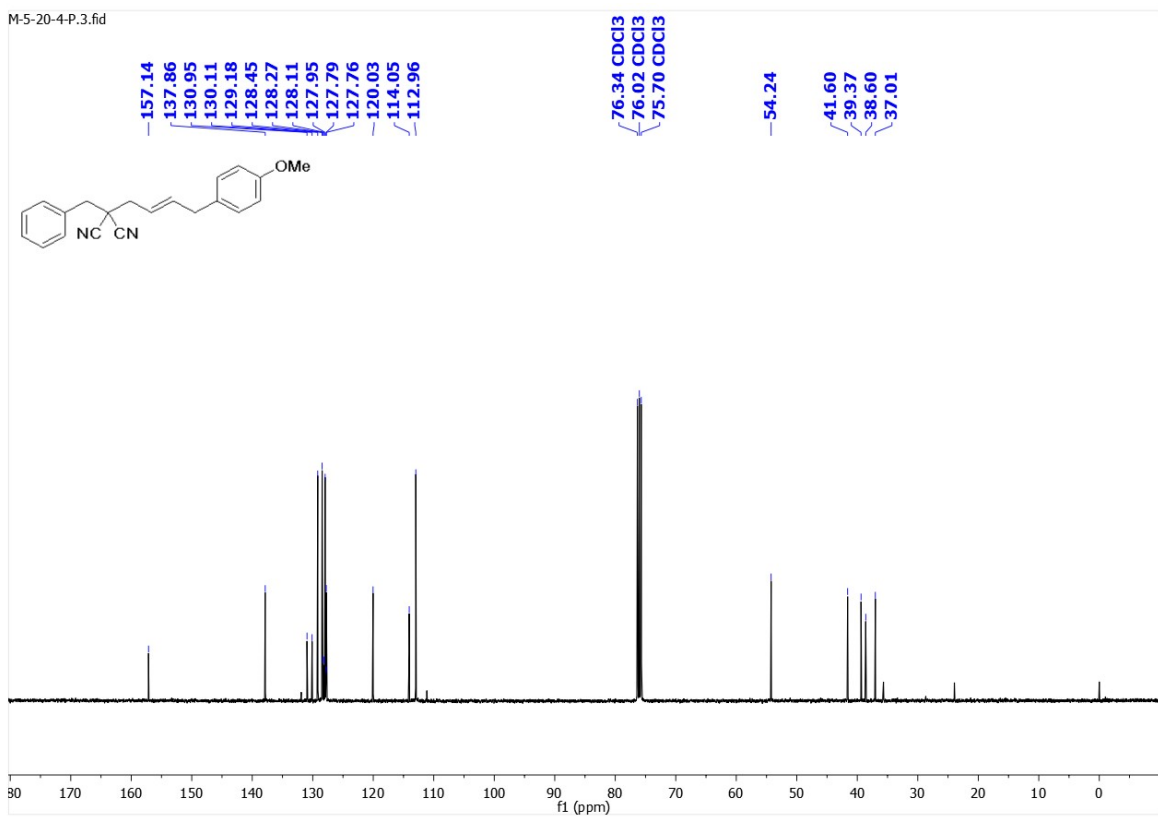
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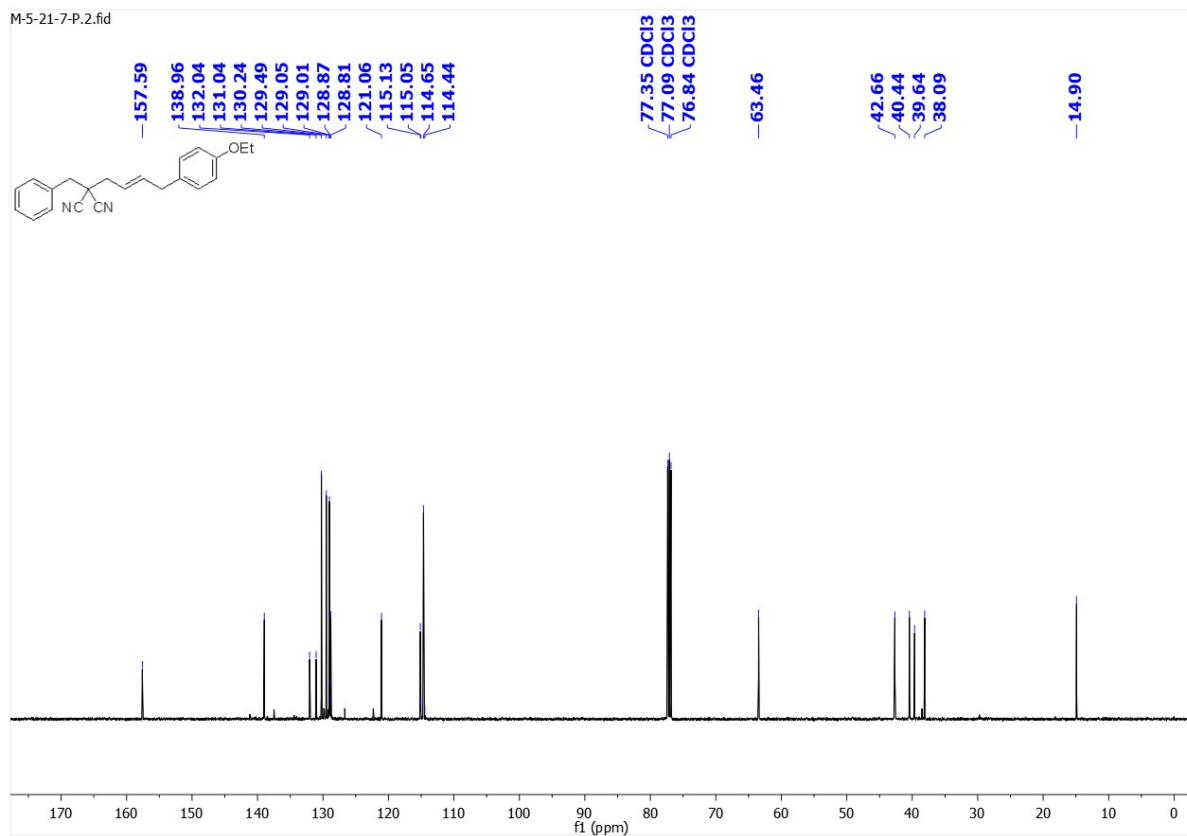
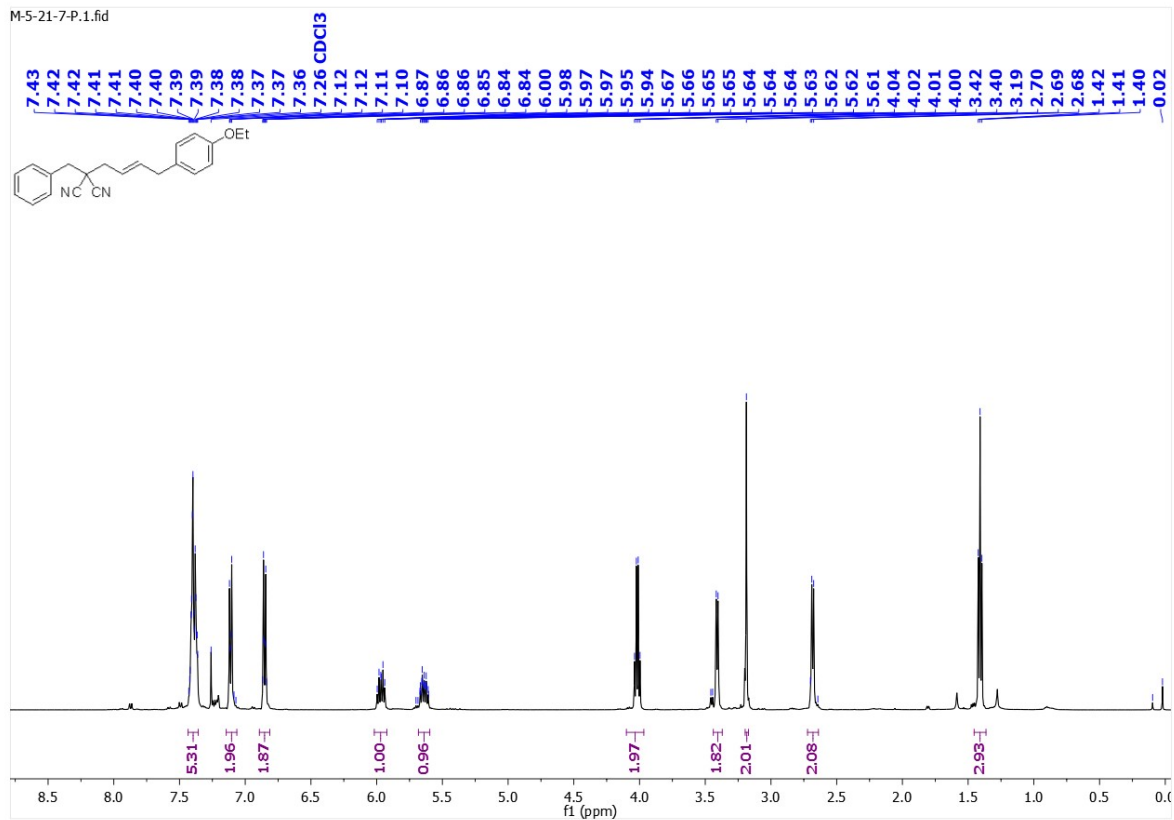


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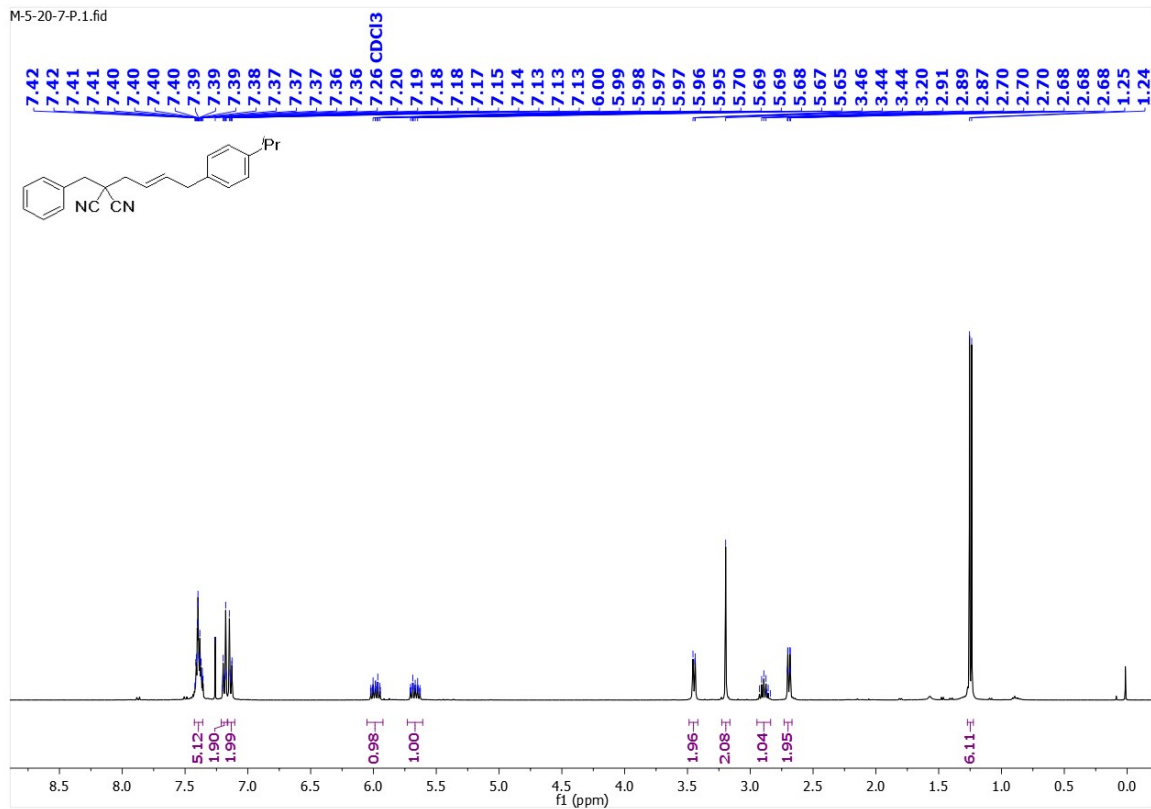


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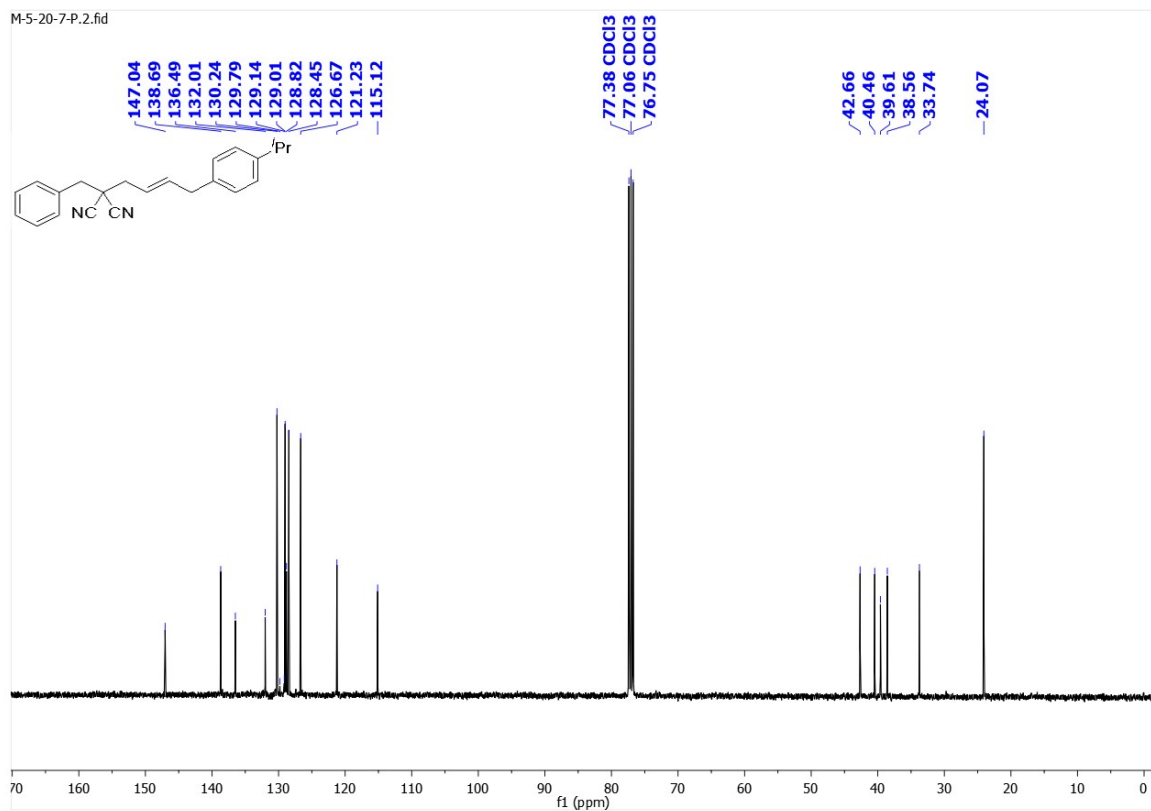




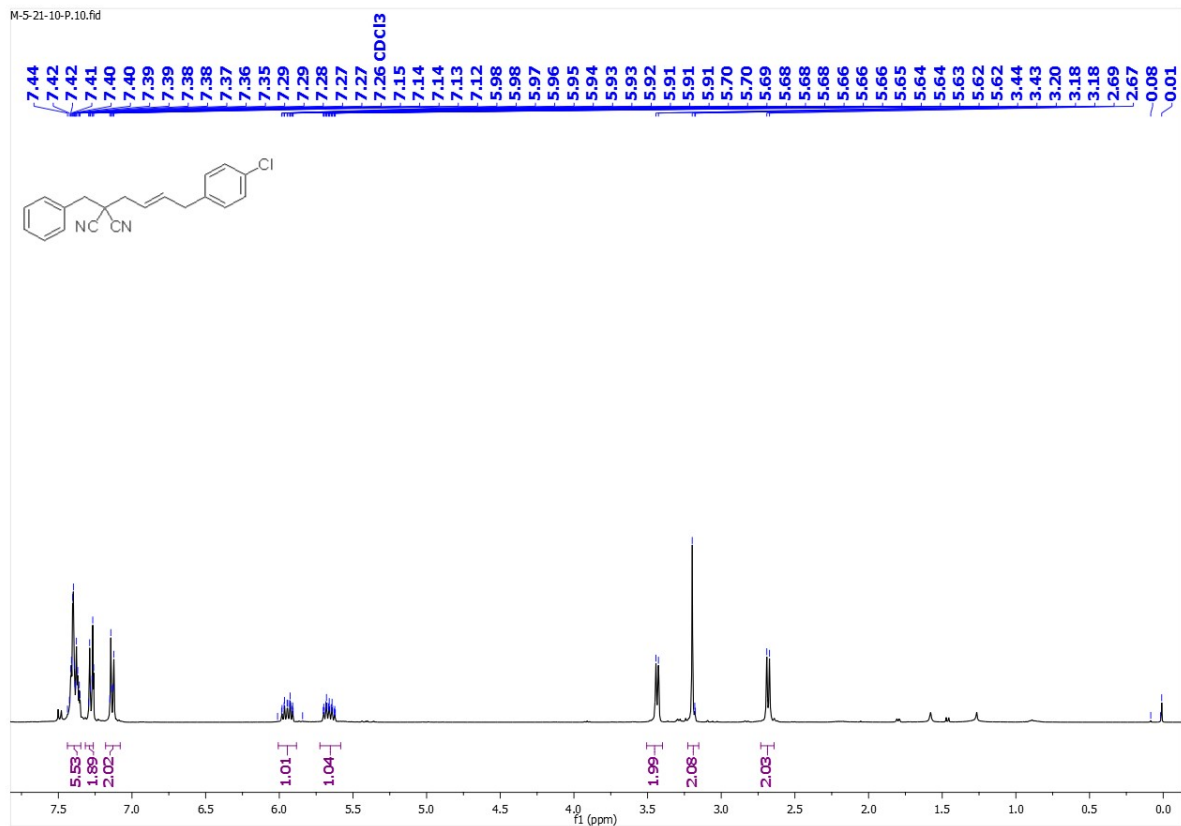
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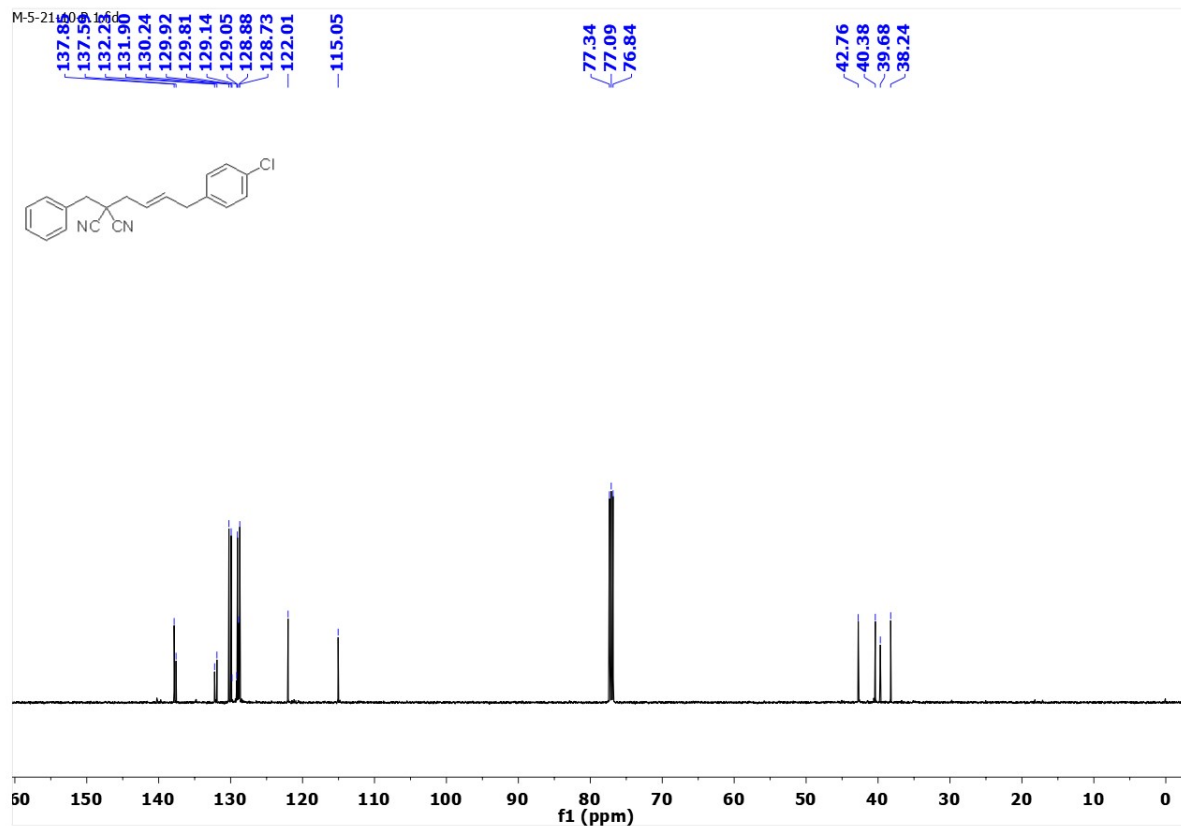
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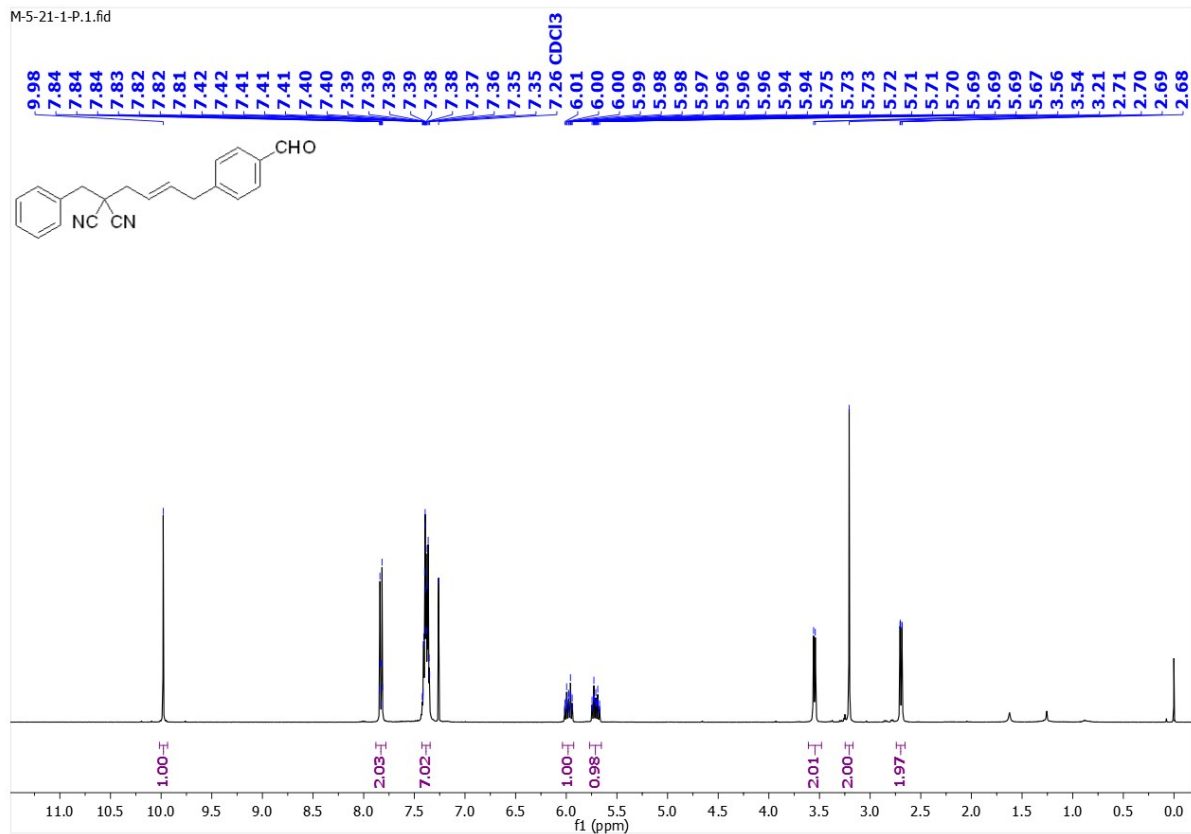
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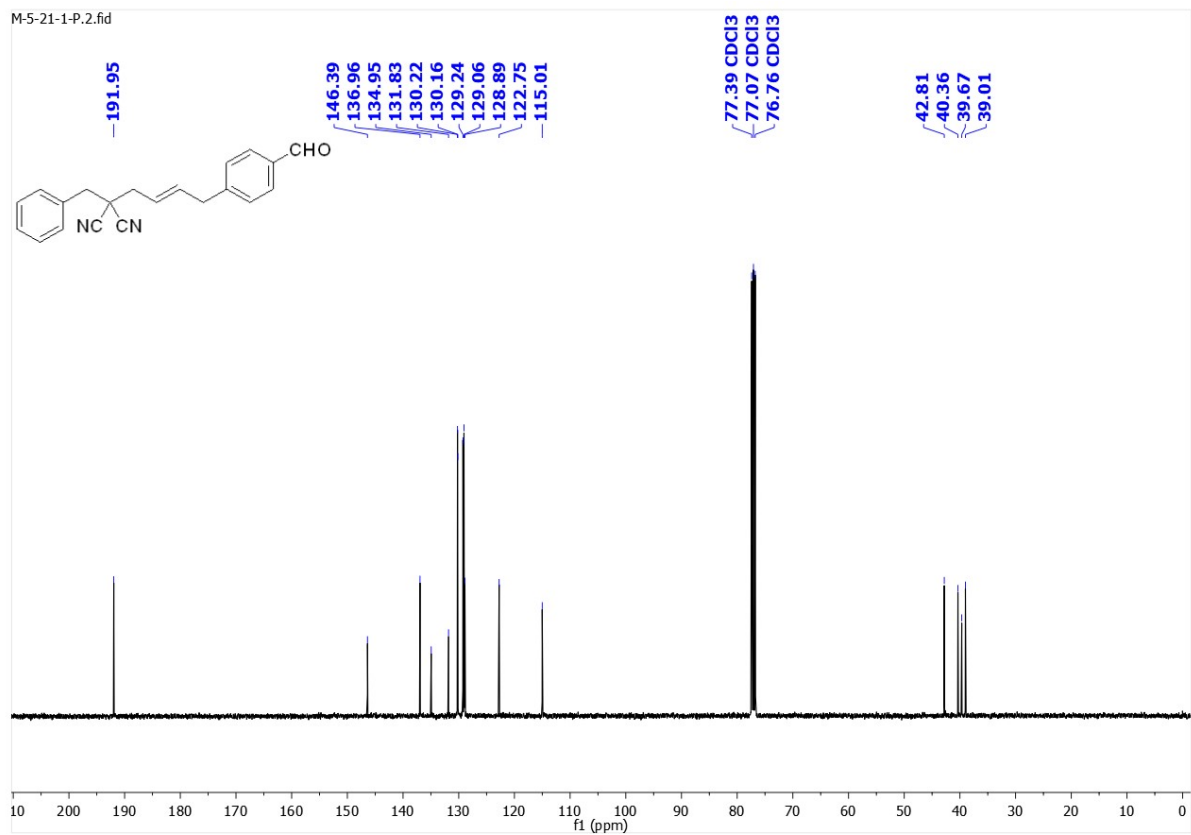
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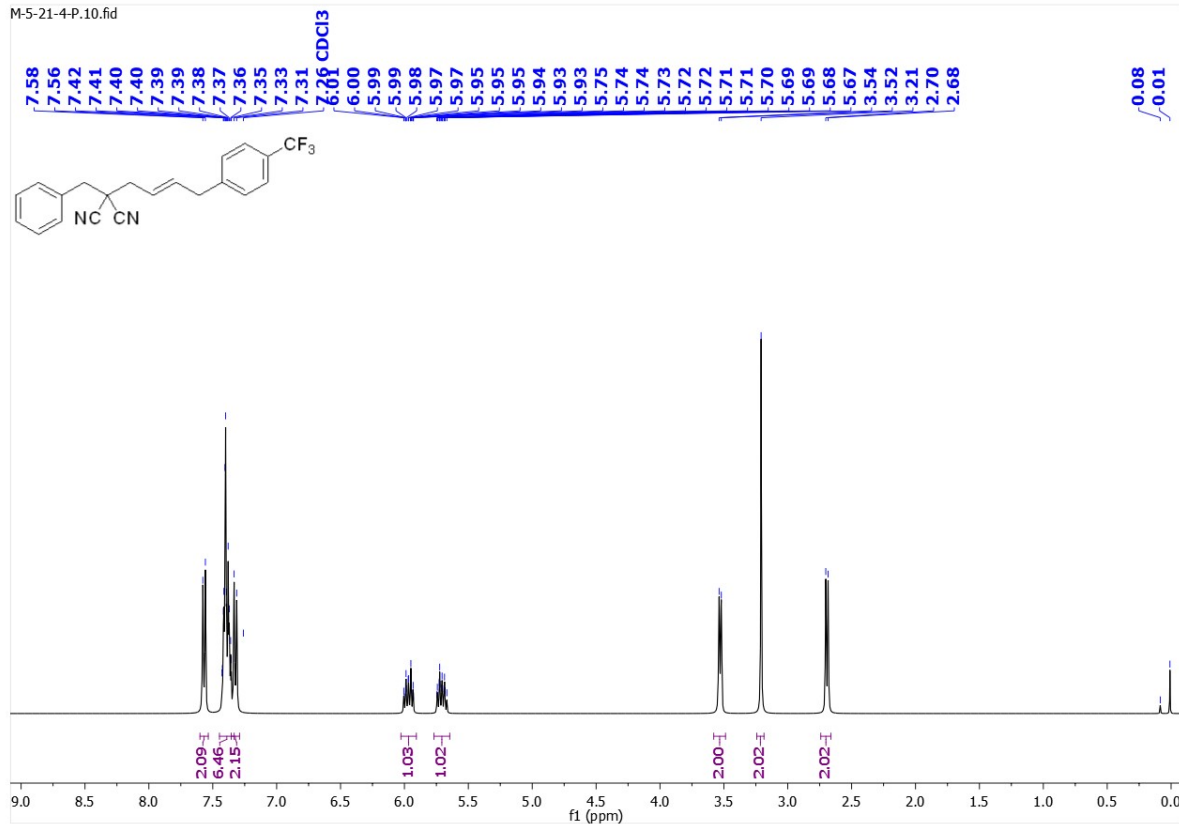
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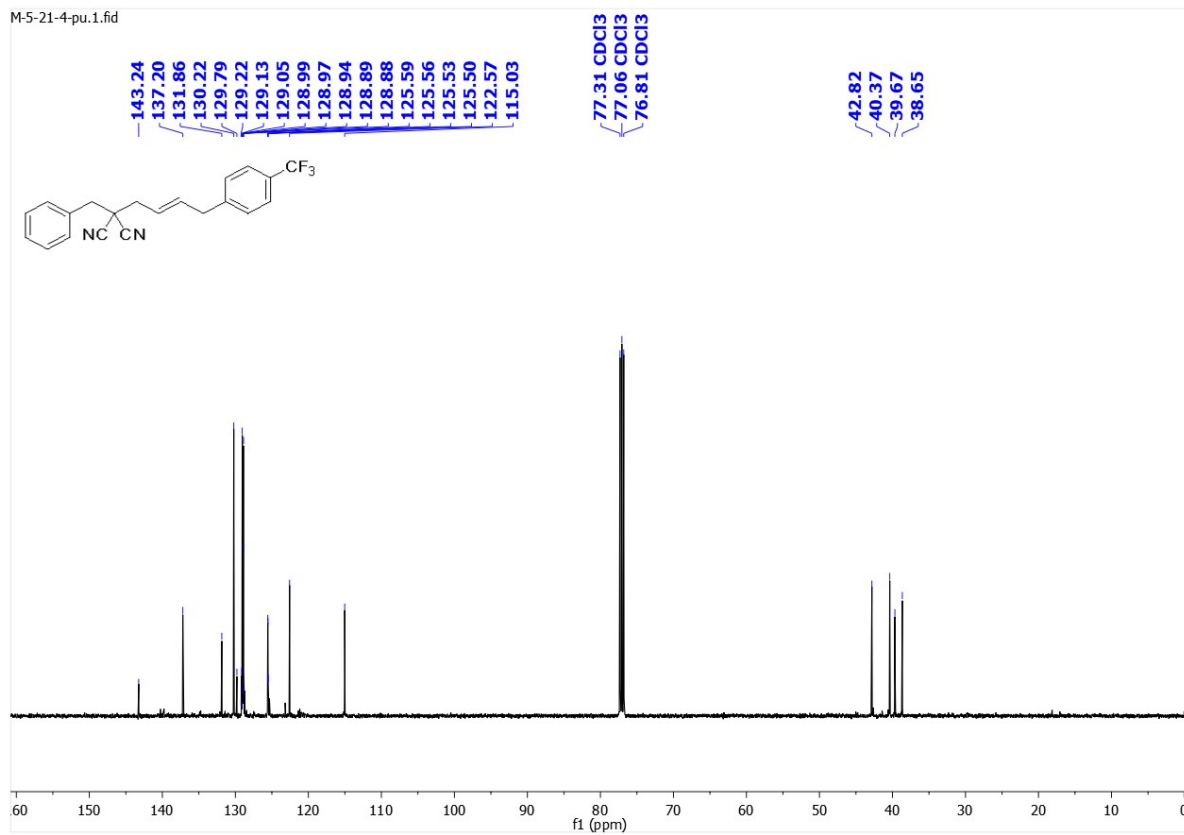
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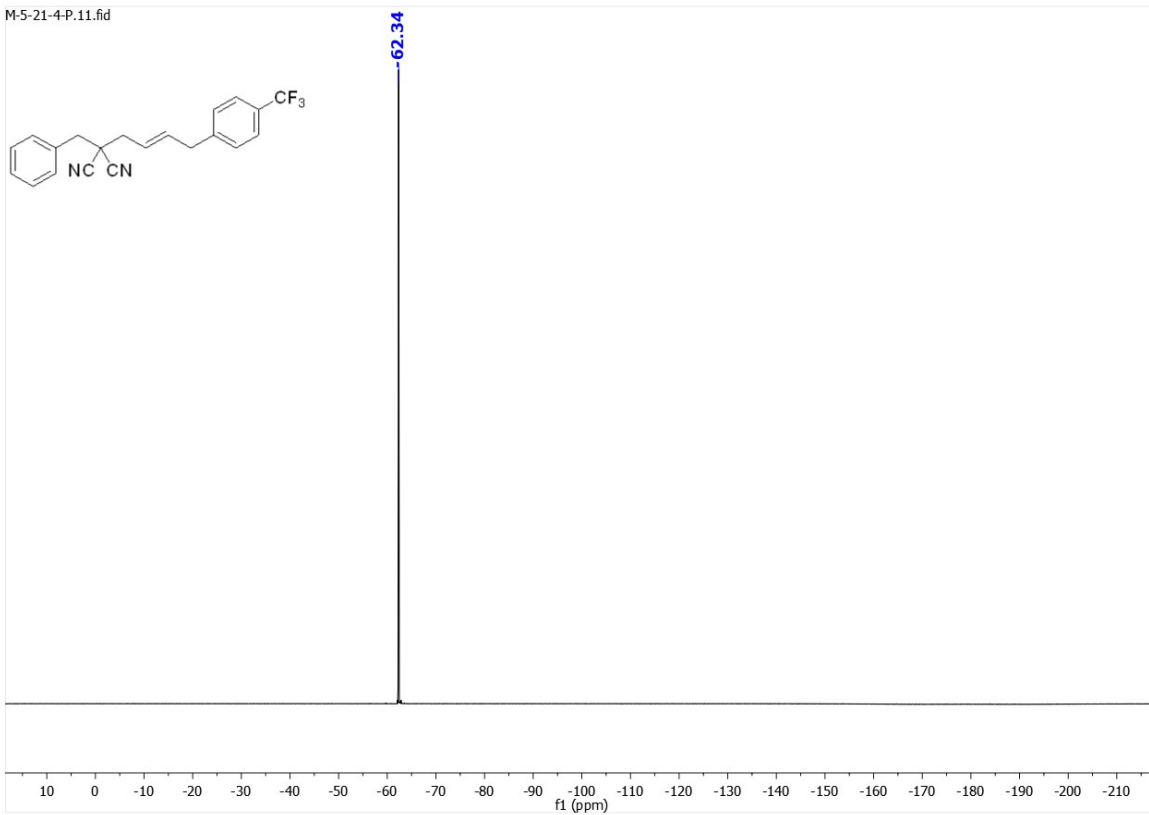
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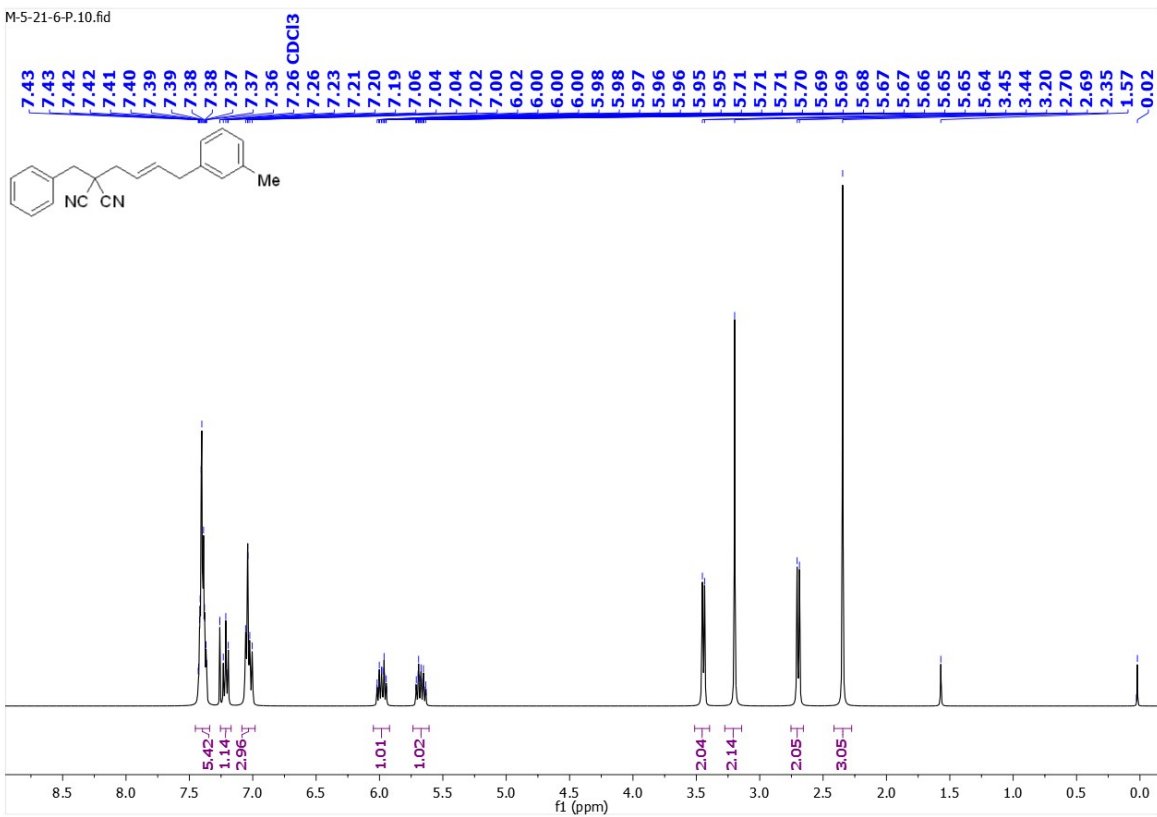
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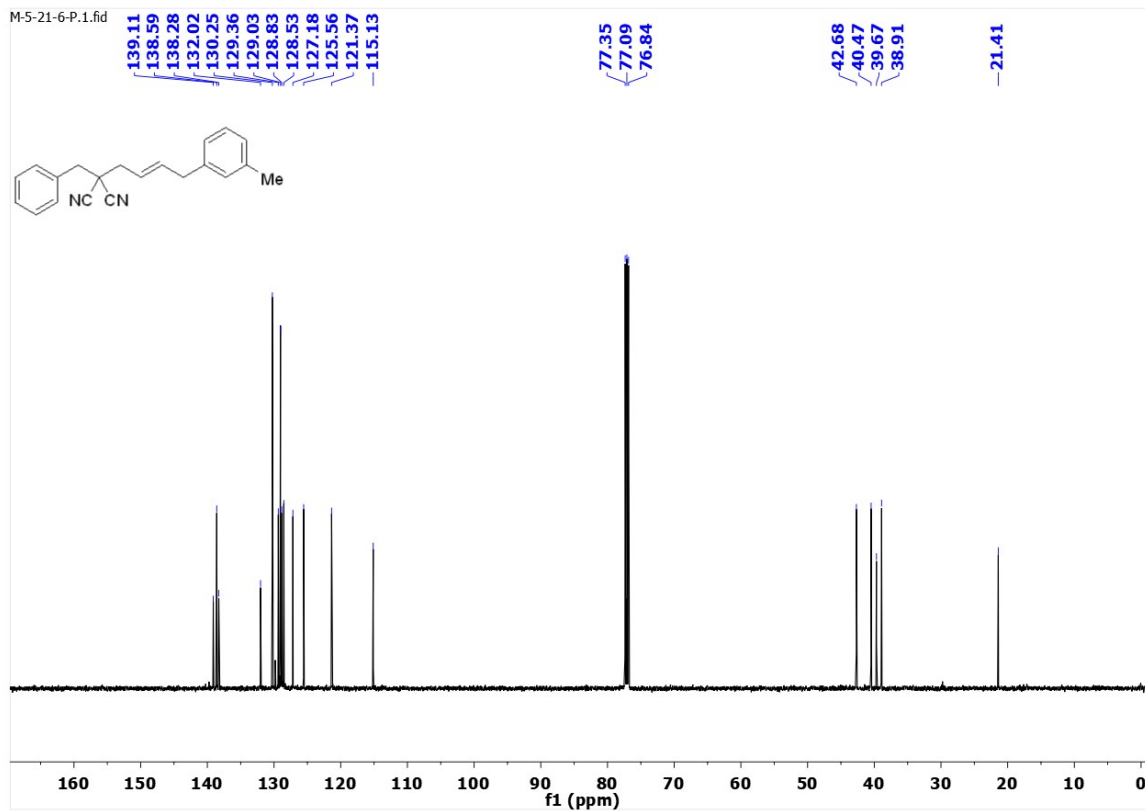
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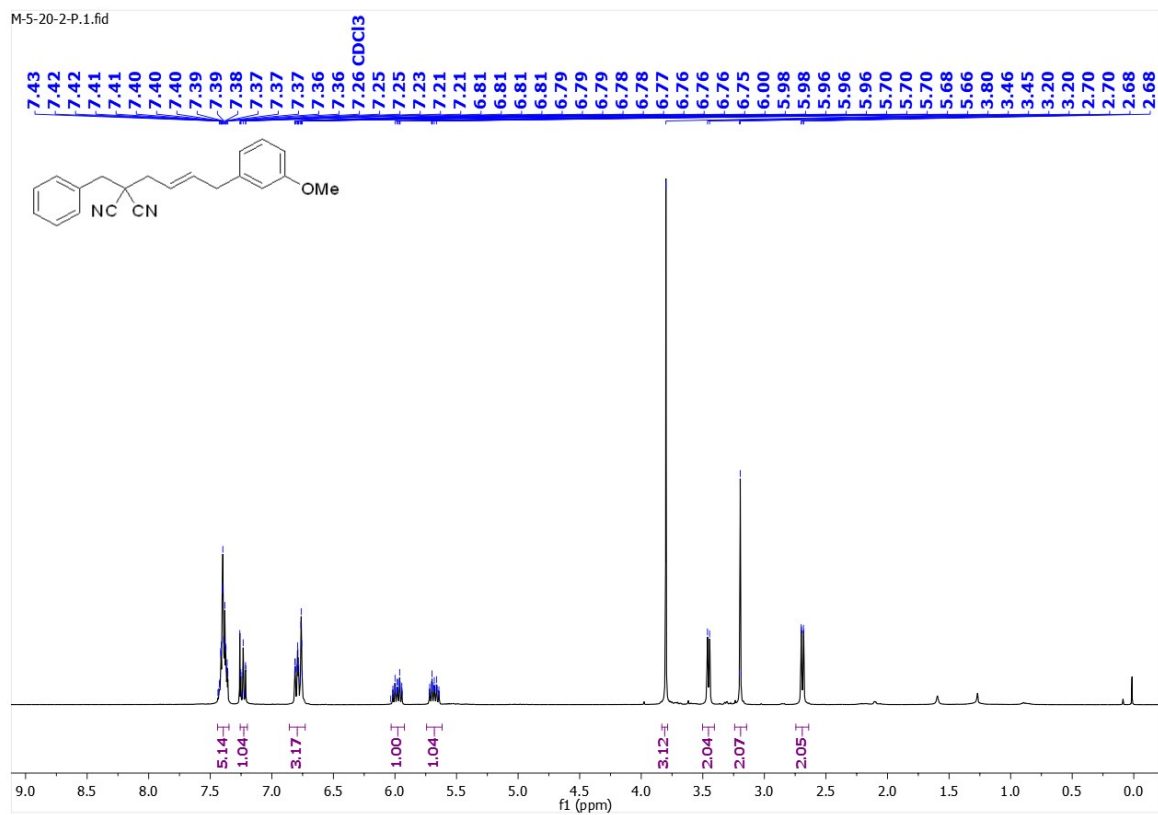
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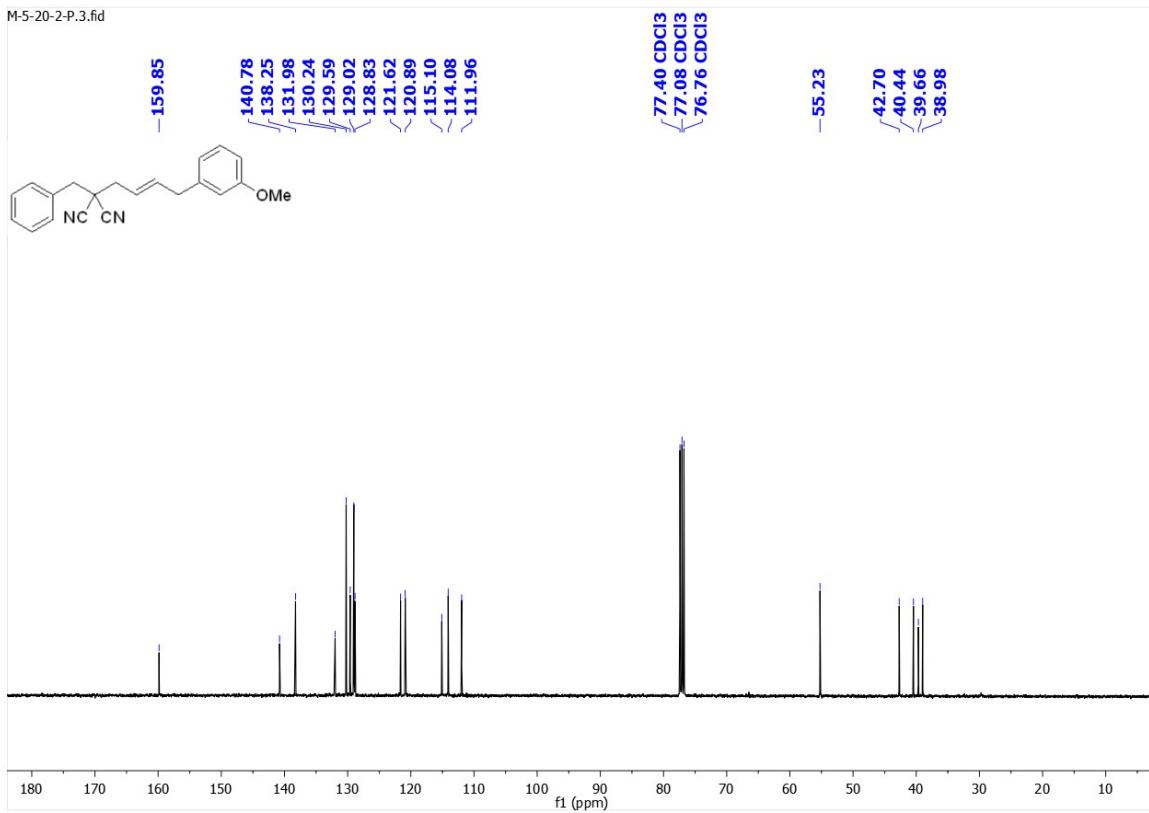
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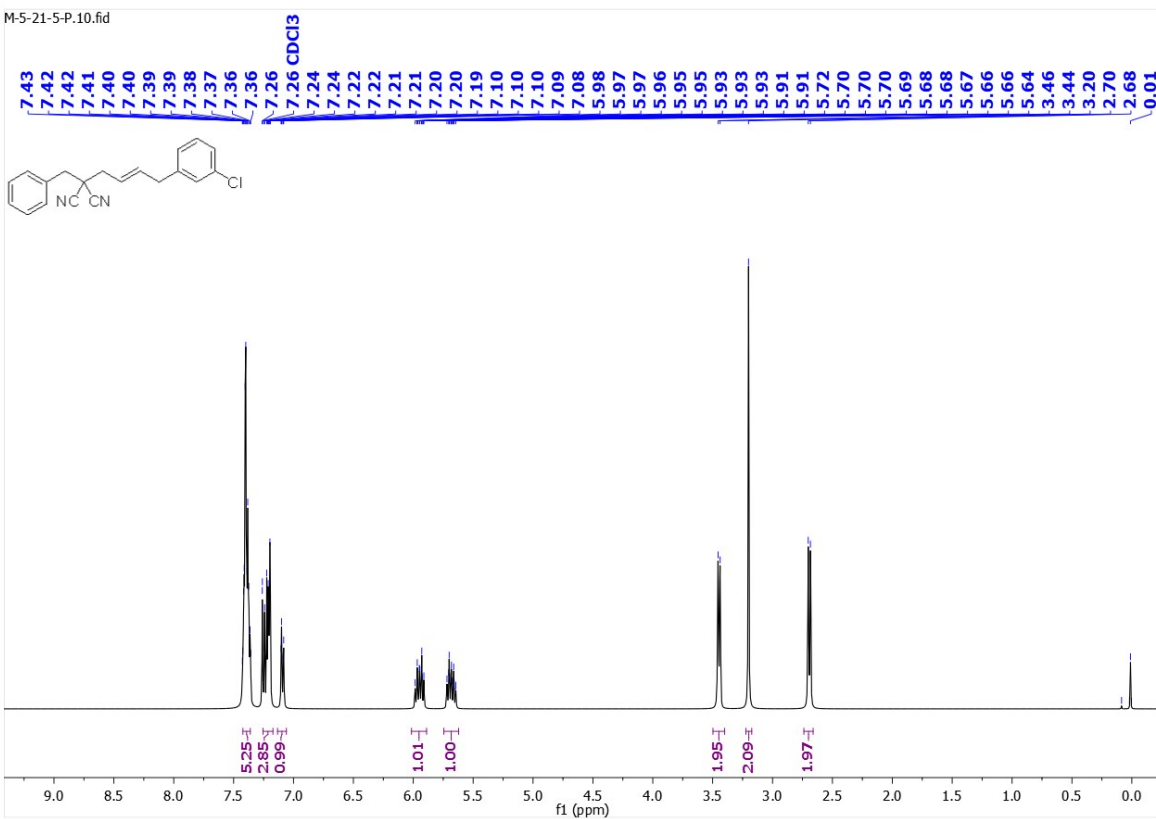
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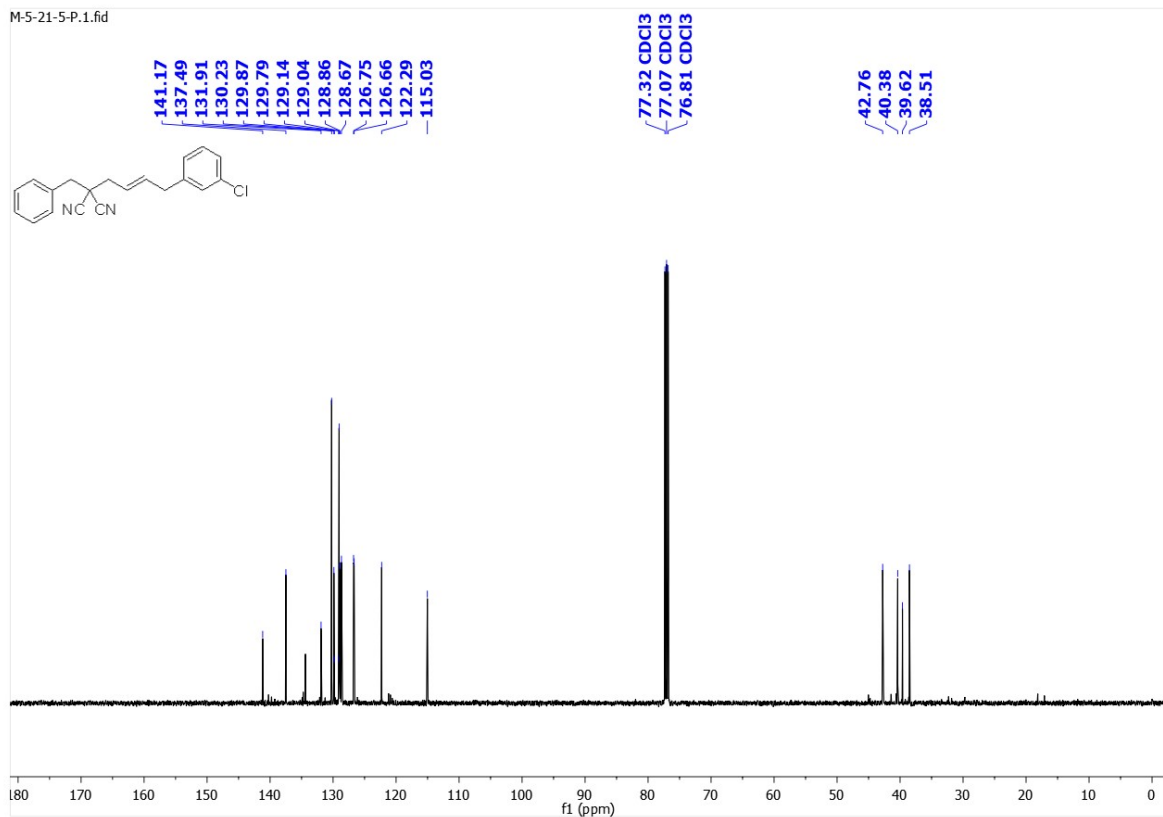
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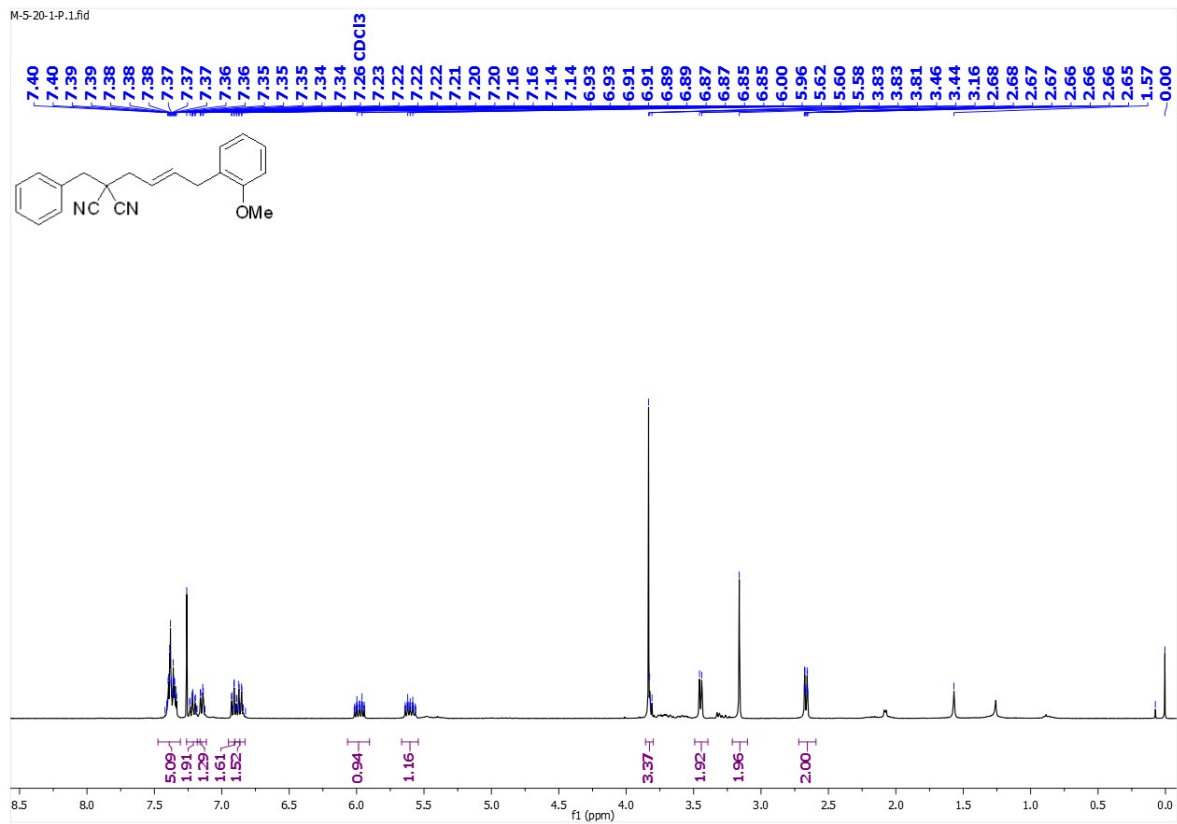
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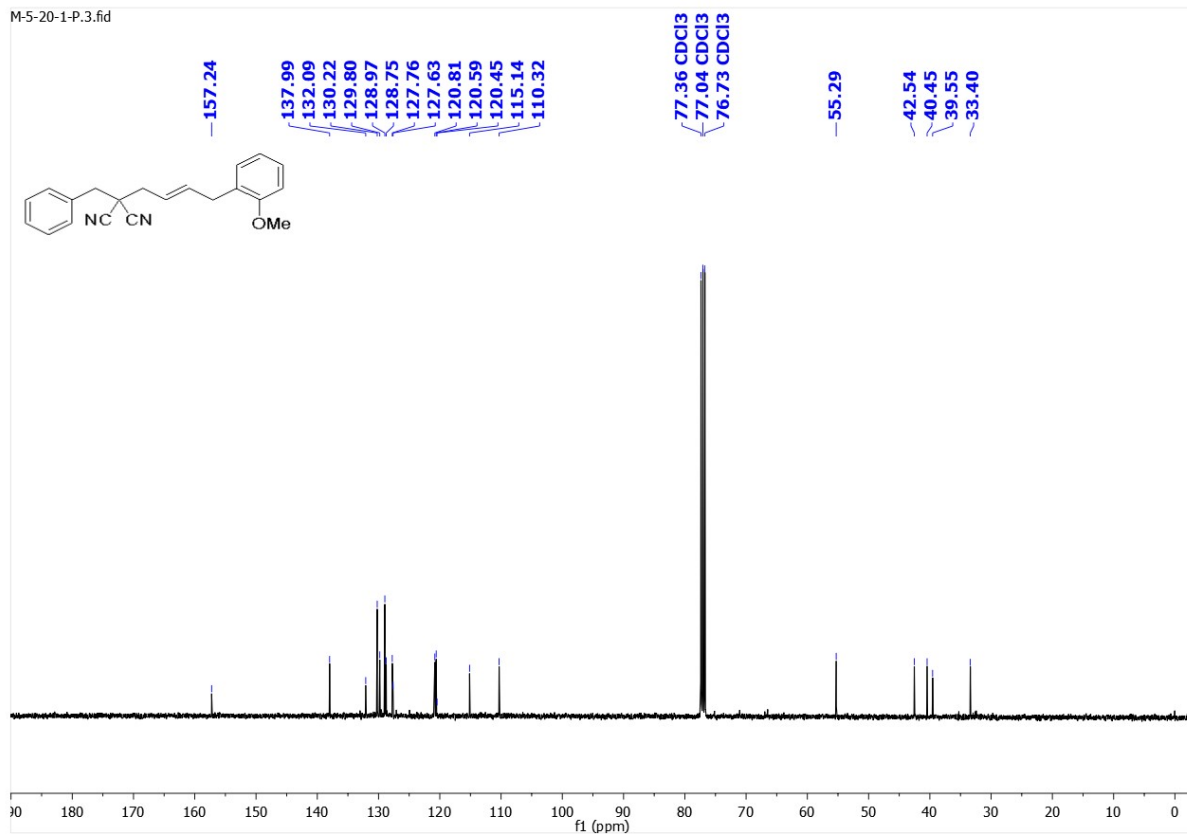
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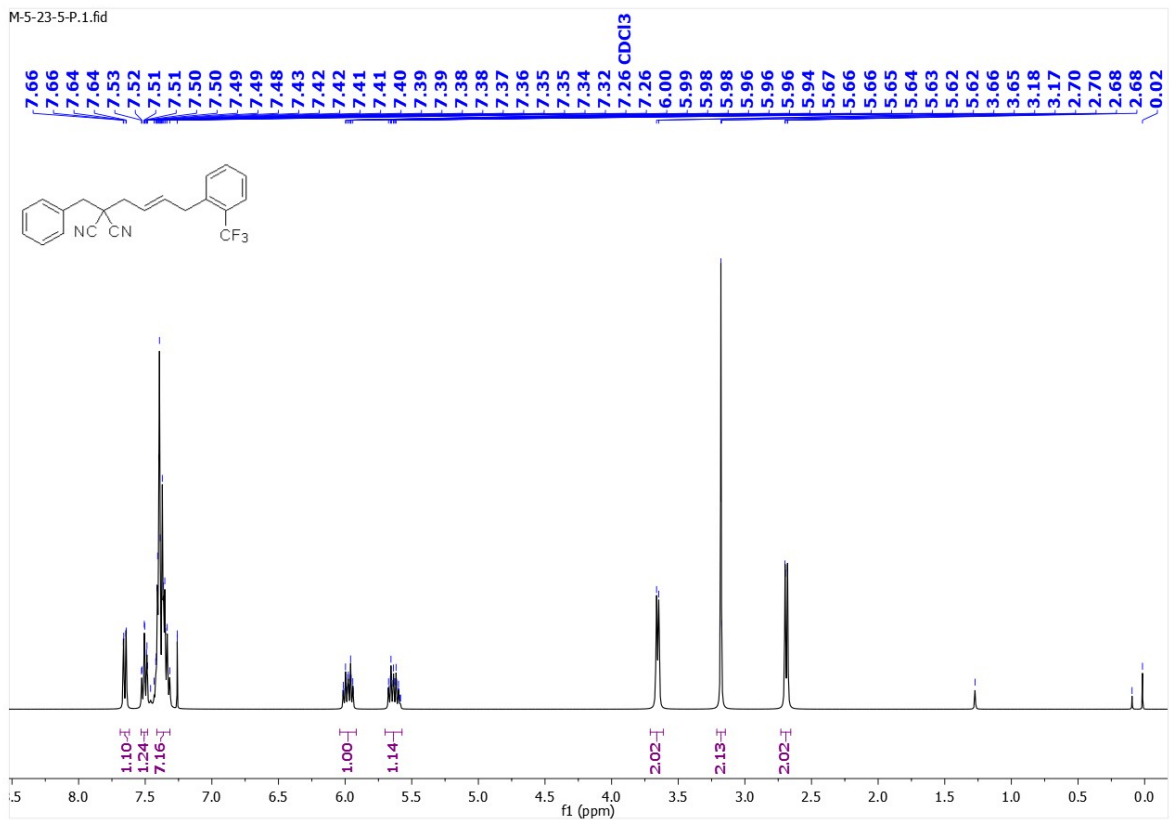
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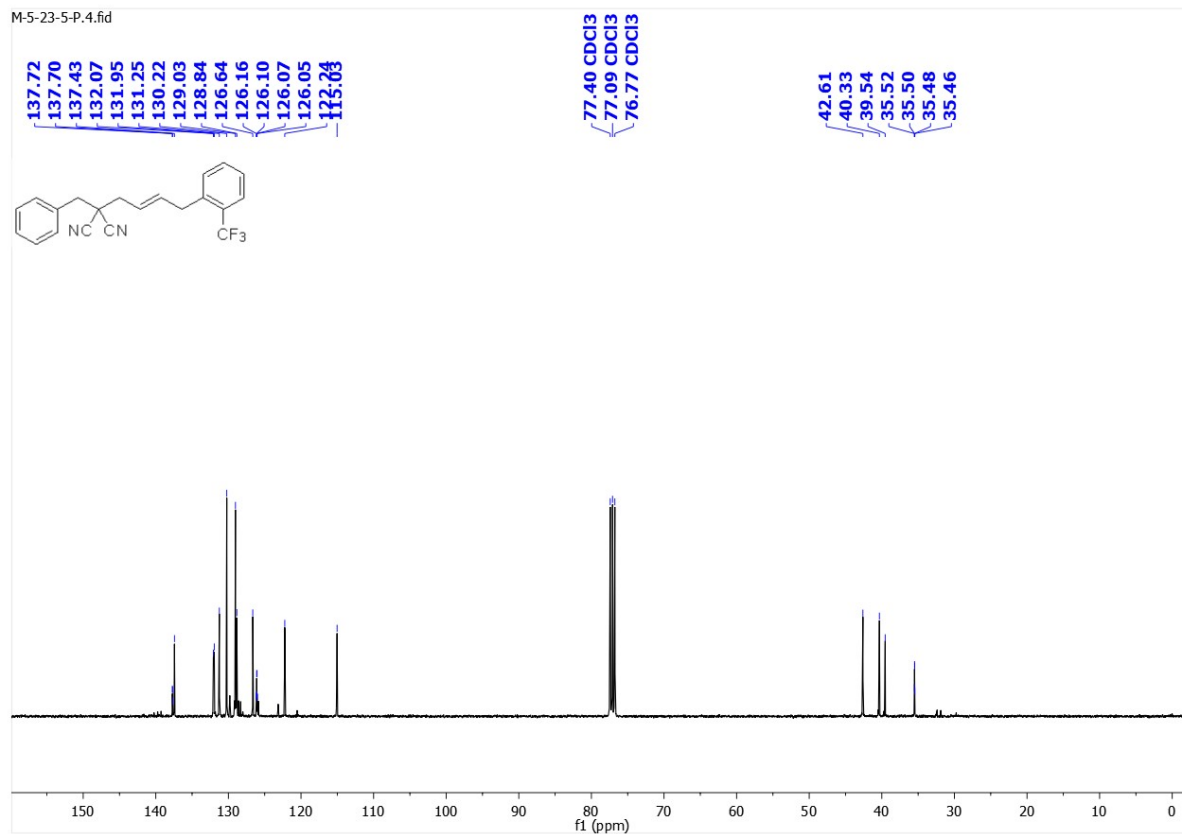
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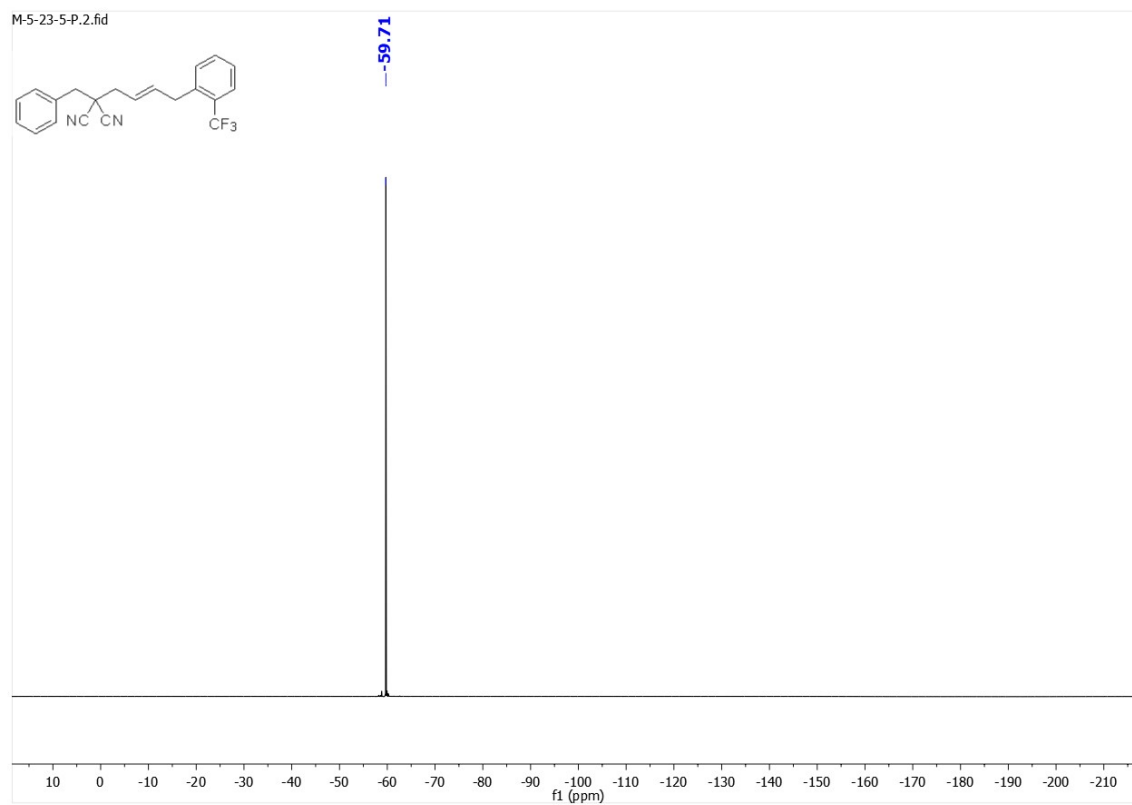
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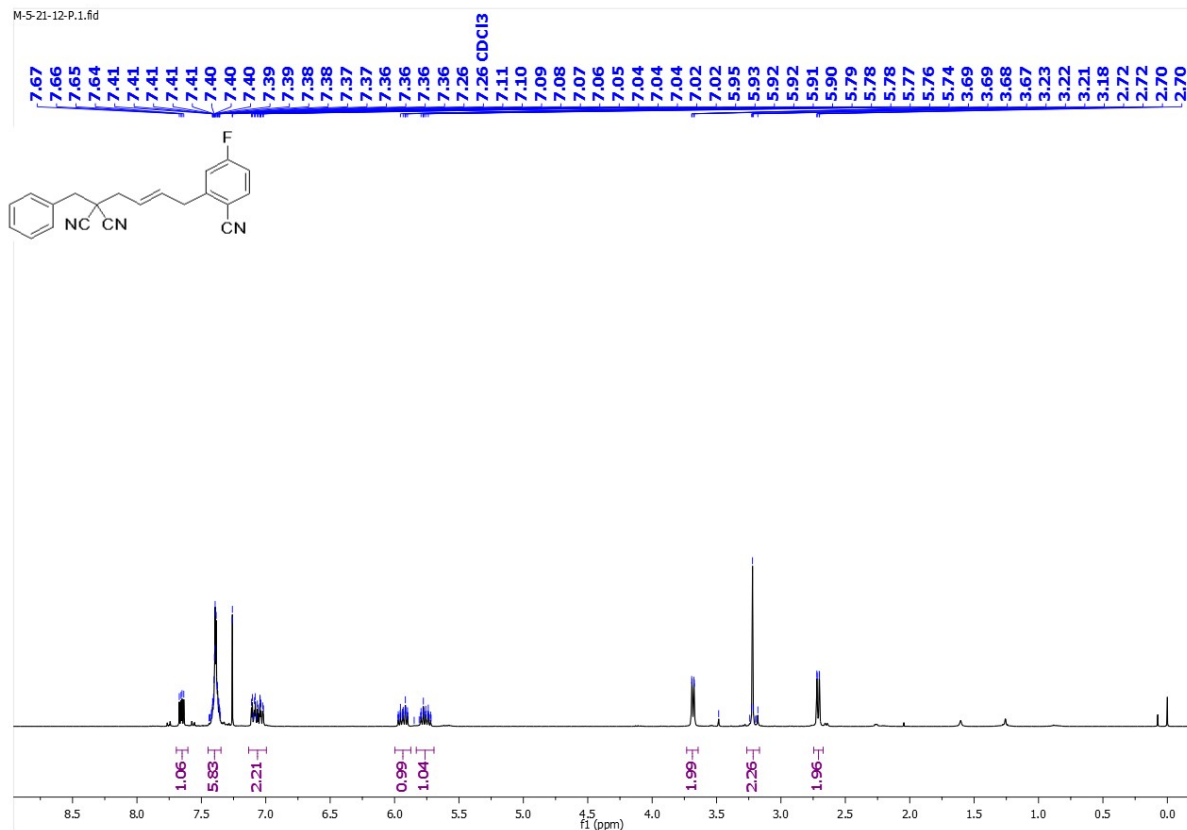
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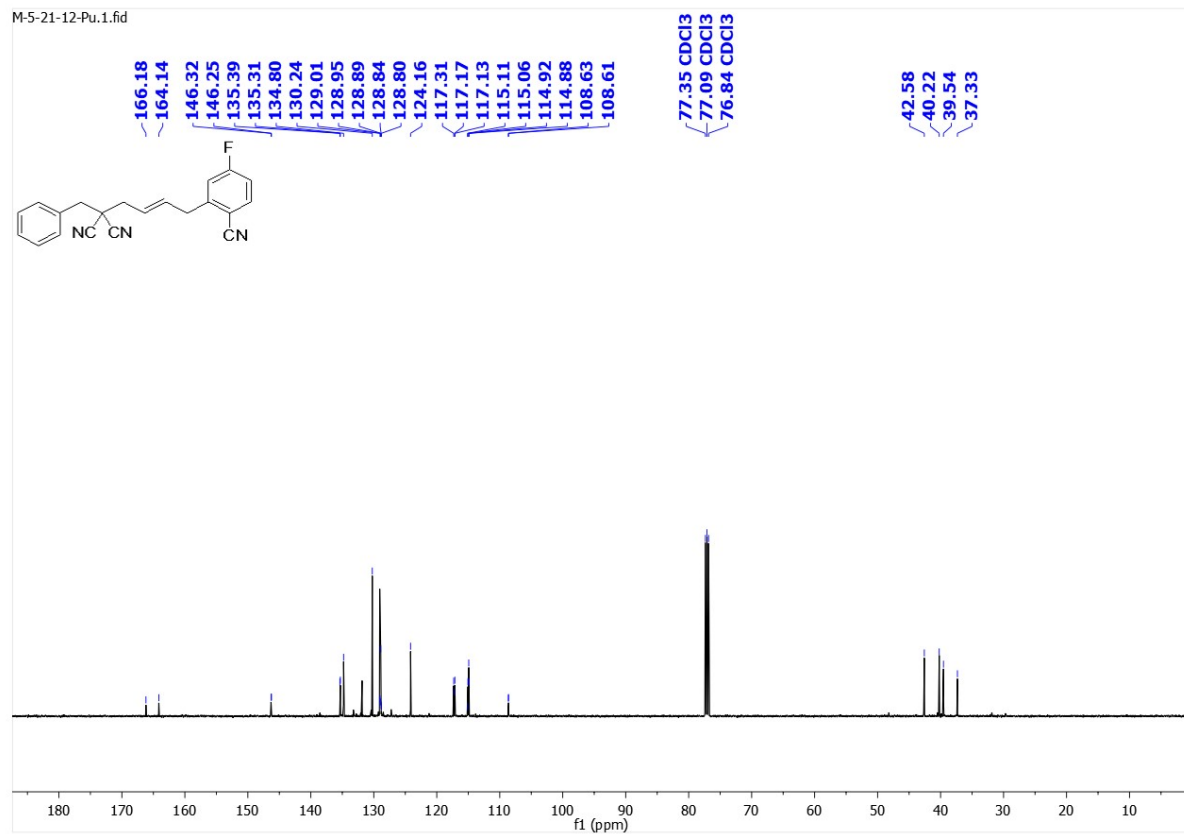
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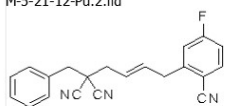
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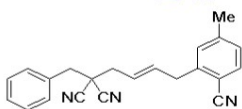
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-102.18

10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220
f1 (ppm)

M-5-21-13-P.1.fid



7.53 7.51 7.42 7.41 7.41 7.40 7.40 7.40 7.39 7.39 7.39 7.38 7.38 7.37 7.37 7.36 7.36 7.26 7.26 7.26 CDCl3 7.19 7.19 7.14 7.14 7.13 7.12 7.11 5.98 5.97 5.97 5.96 5.95 5.94 5.93 5.93 5.92 5.91 5.75 5.73 5.73 5.72 5.71 5.71 5.70 5.69 5.69 5.67 3.65 3.64 3.63 3.63 3.21 2.71 2.71 2.69 2.69 2.38 1.62 -0.00

8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

1.07
5.64
1.03
1.12

1.02
1.02

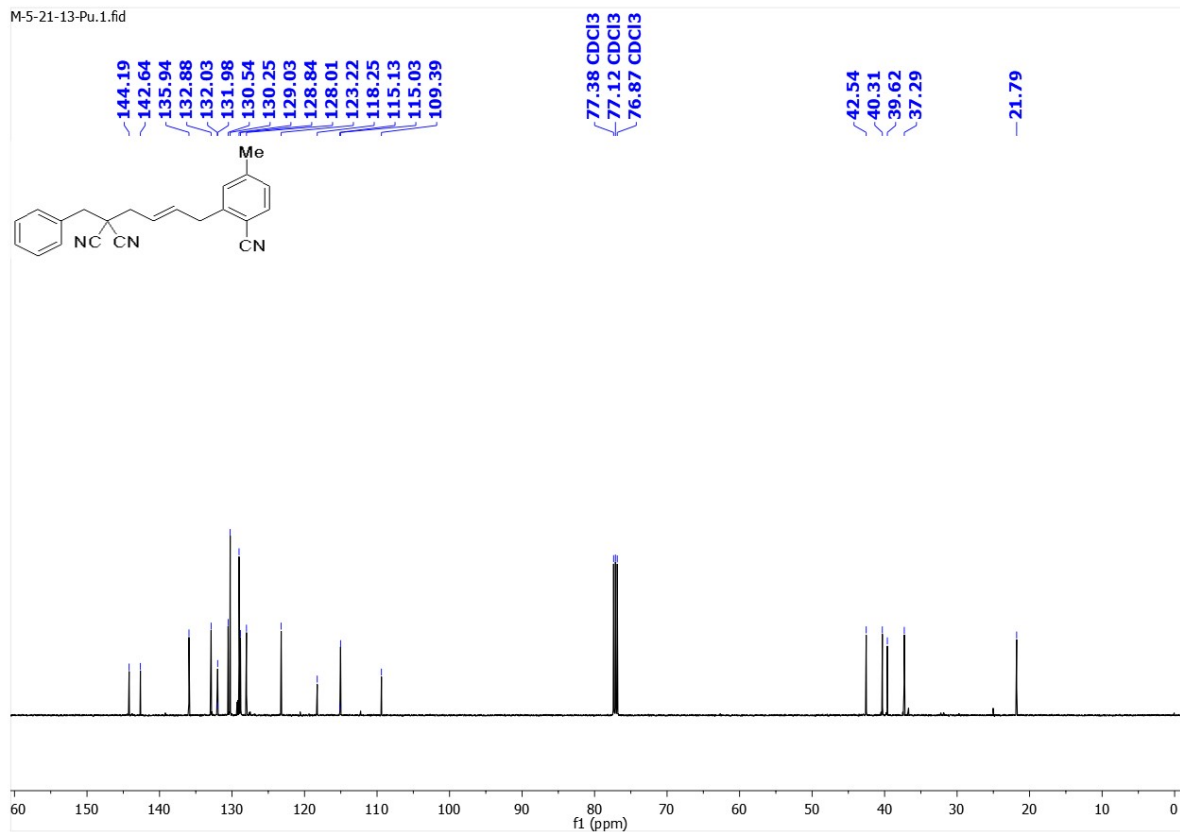
2.02

2.01

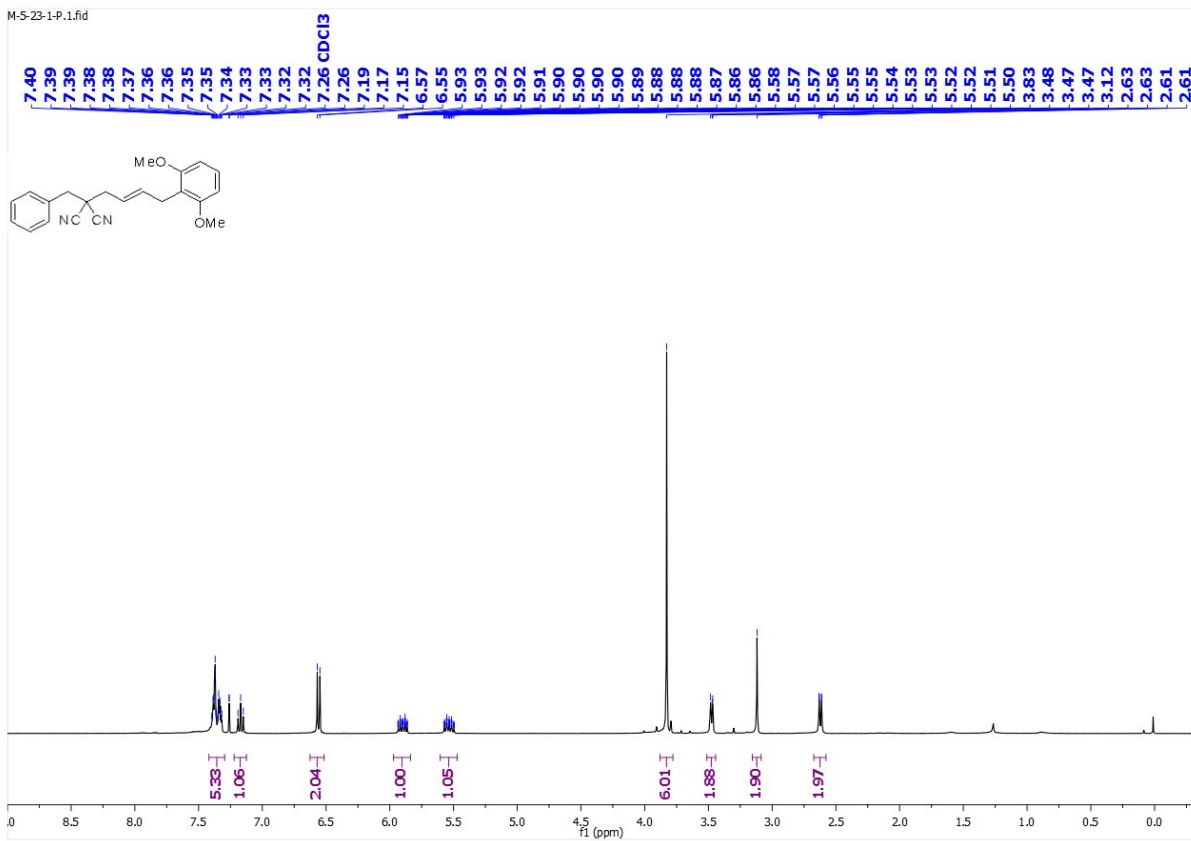
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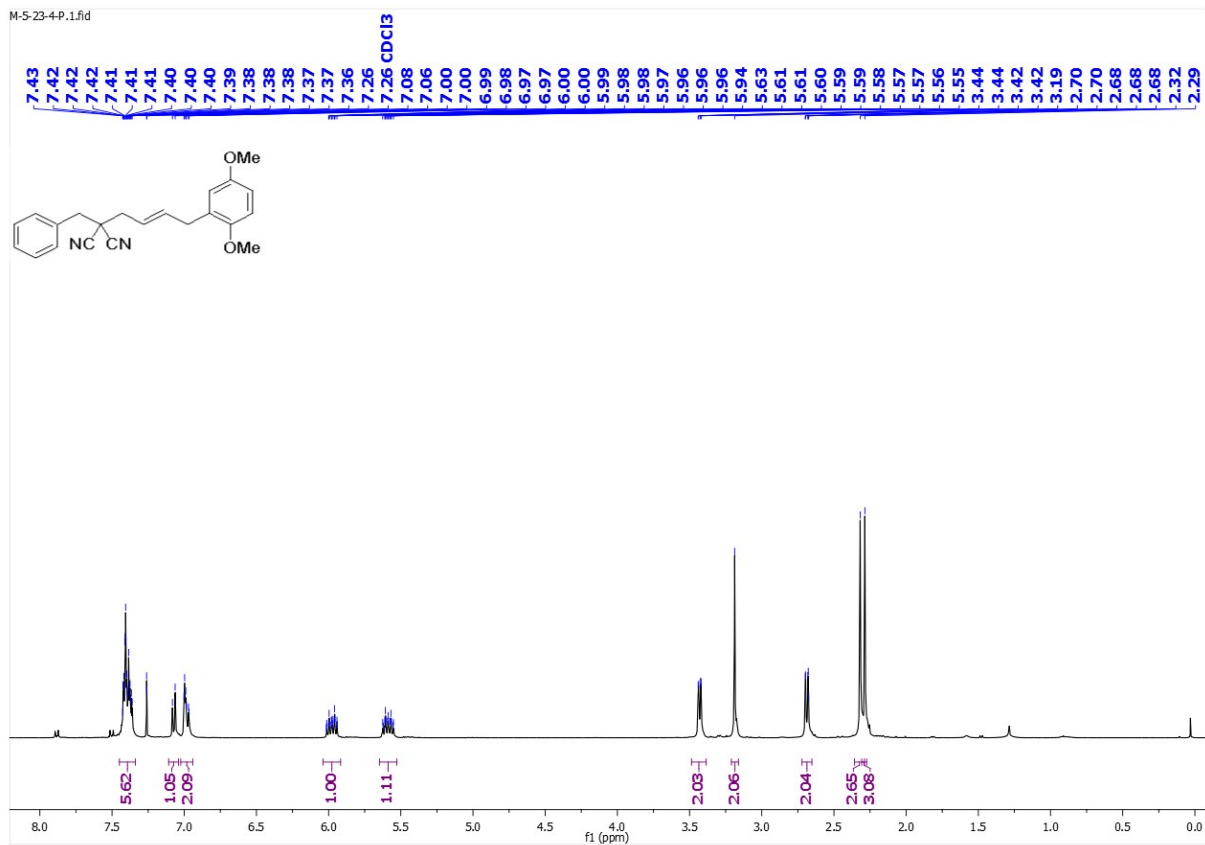
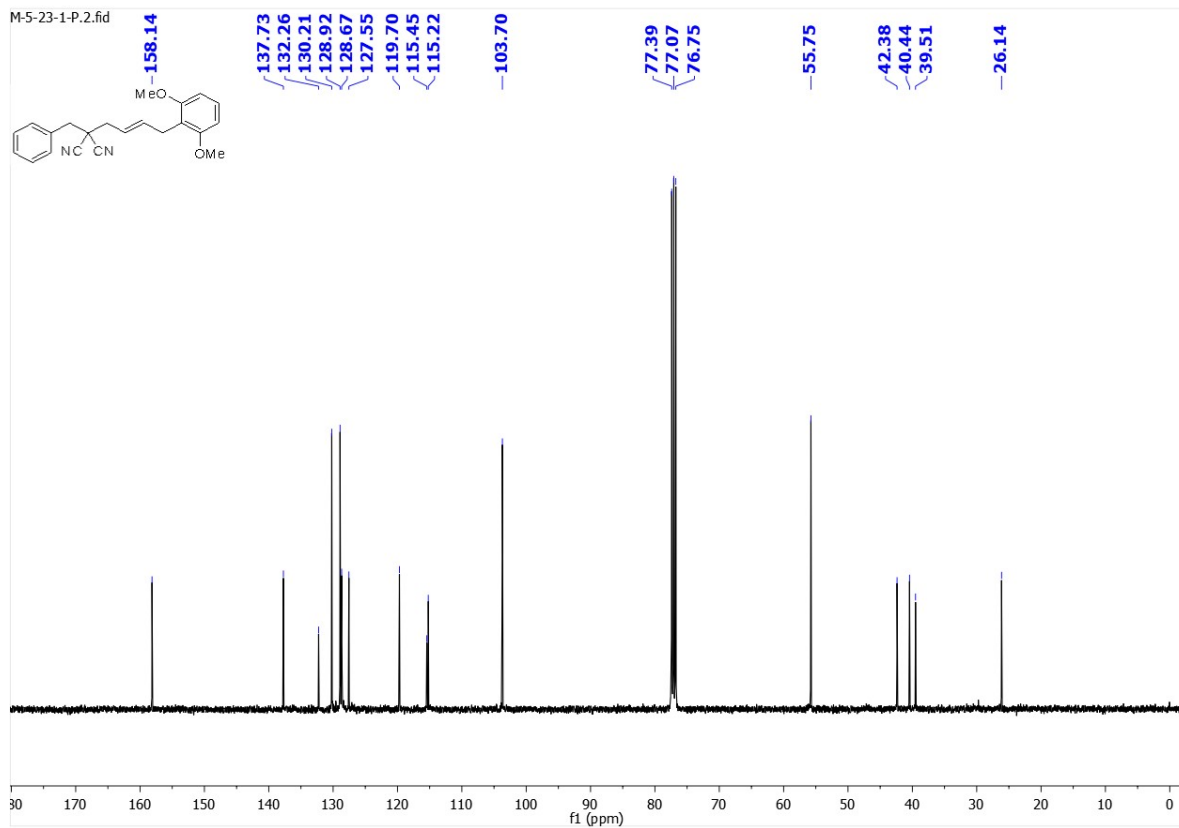
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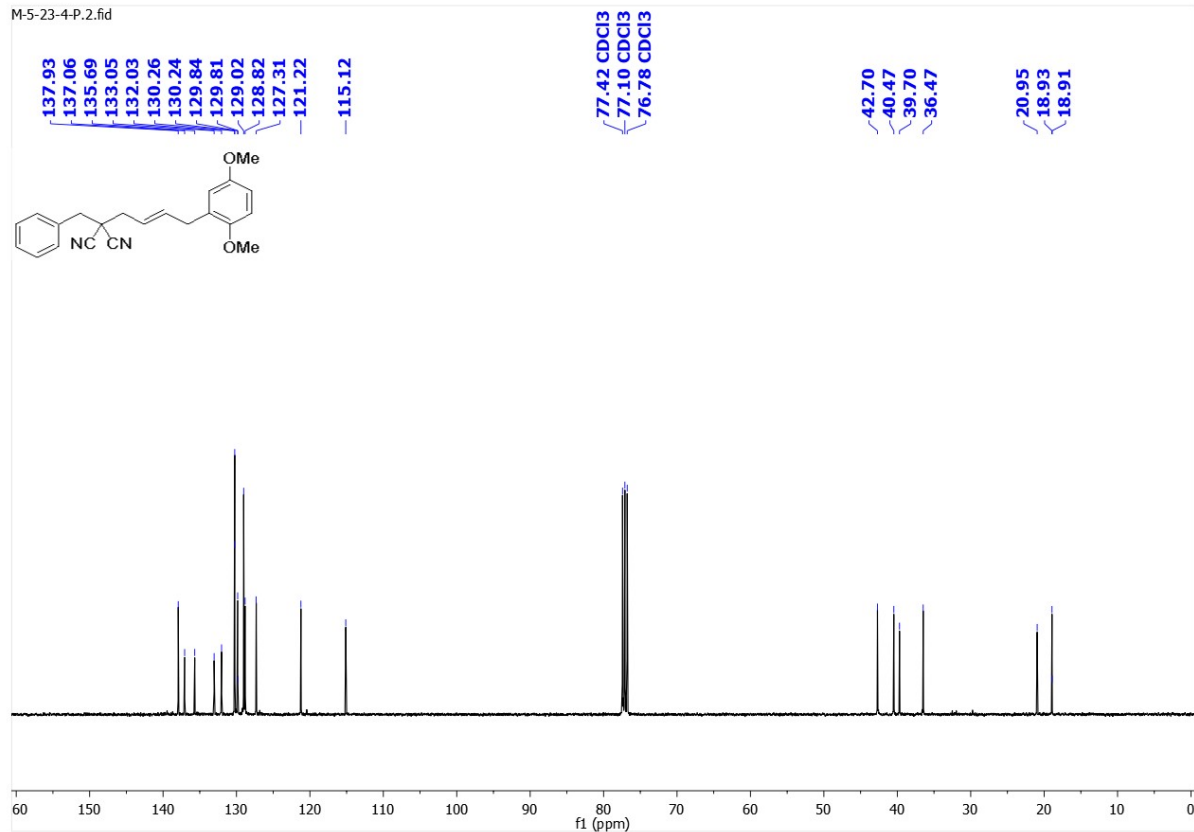


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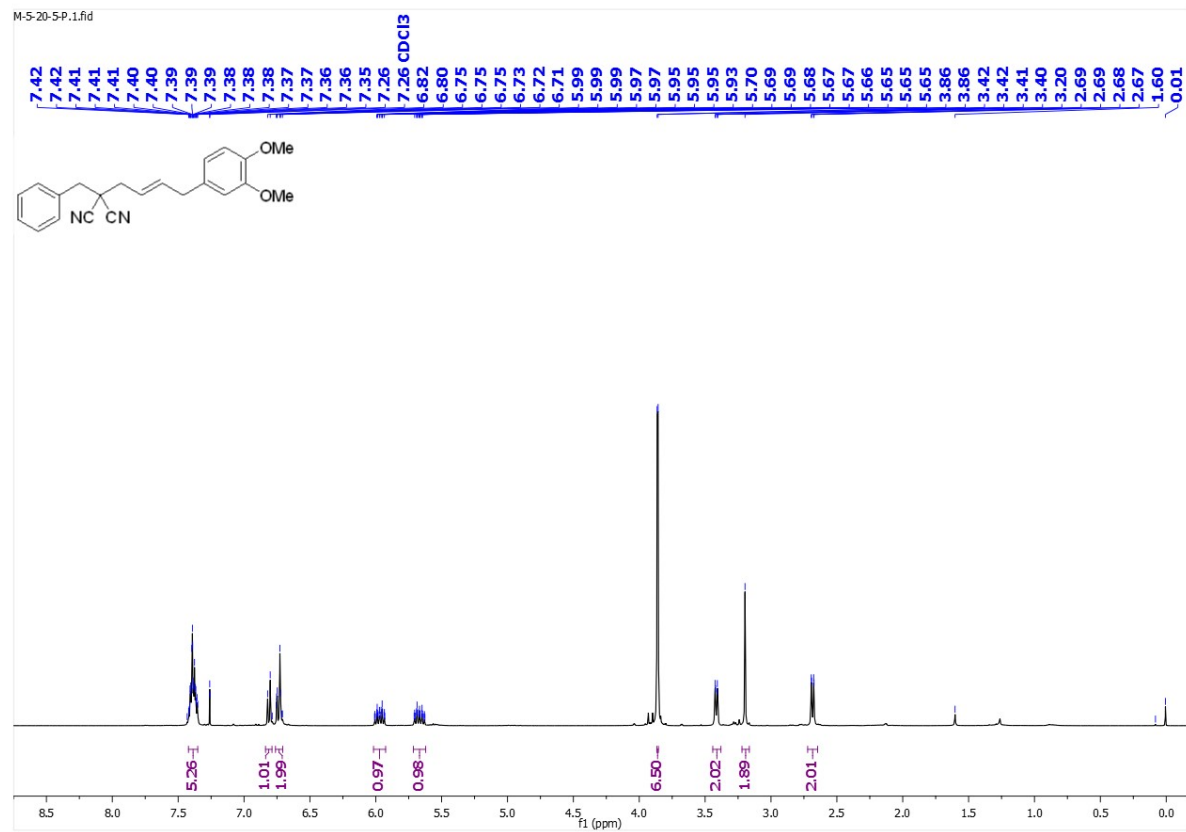




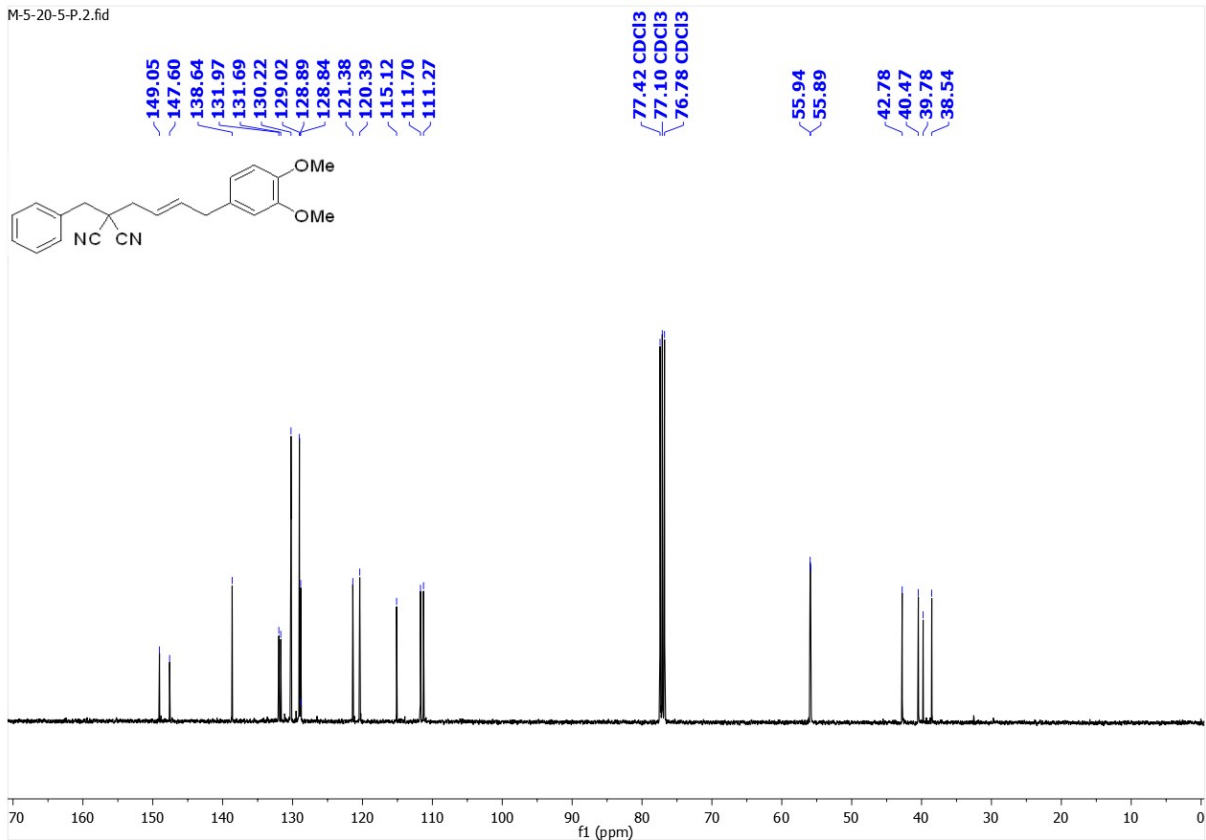
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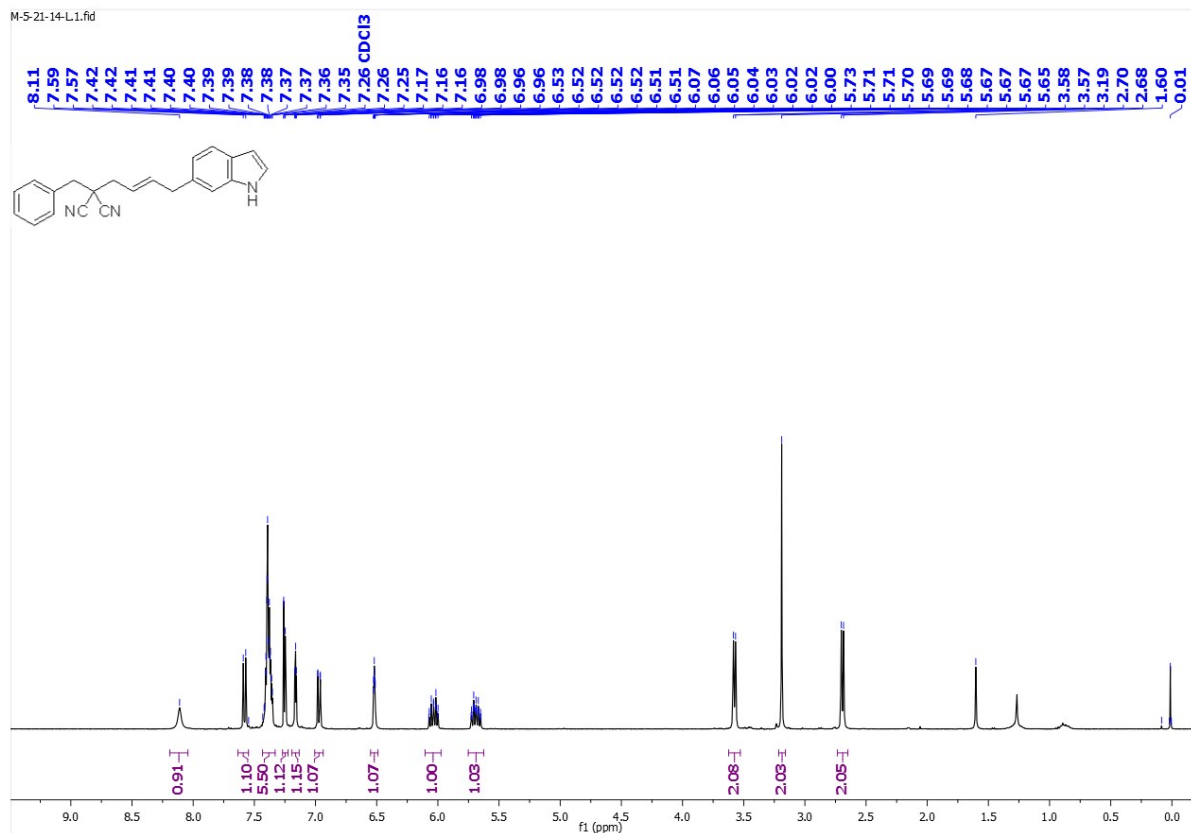
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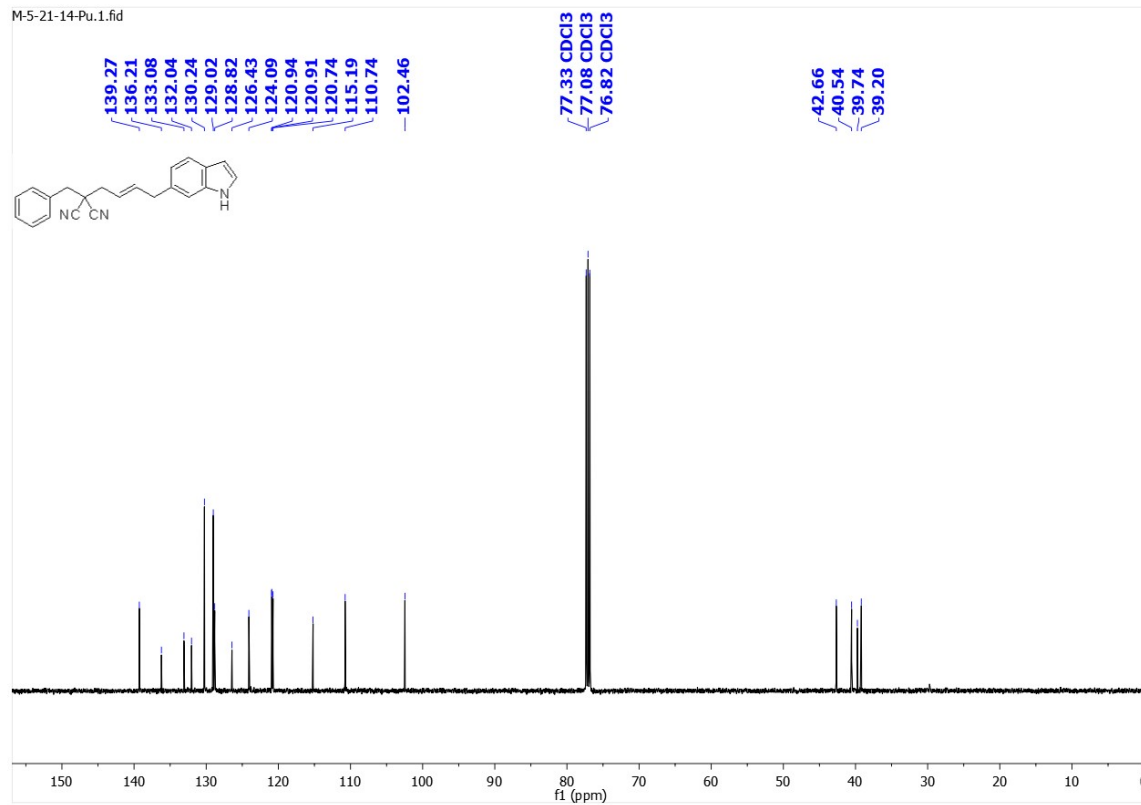
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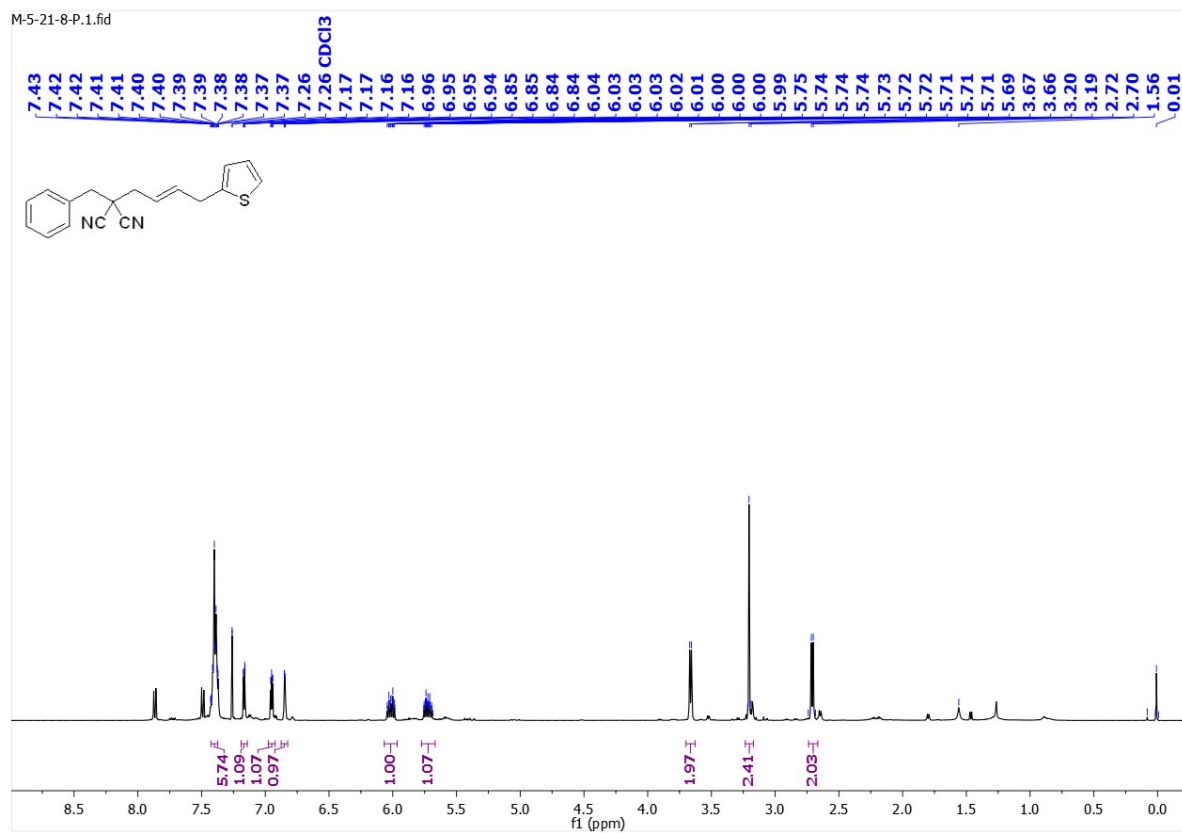
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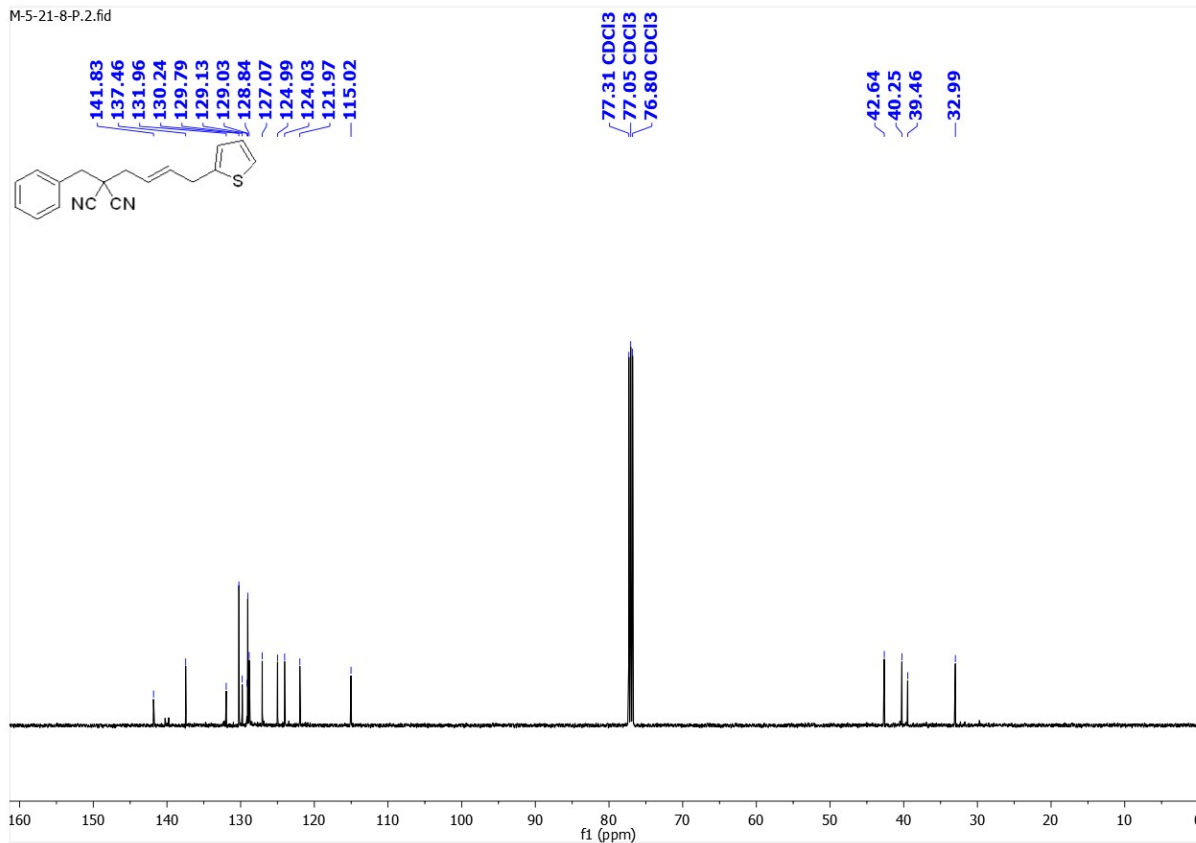
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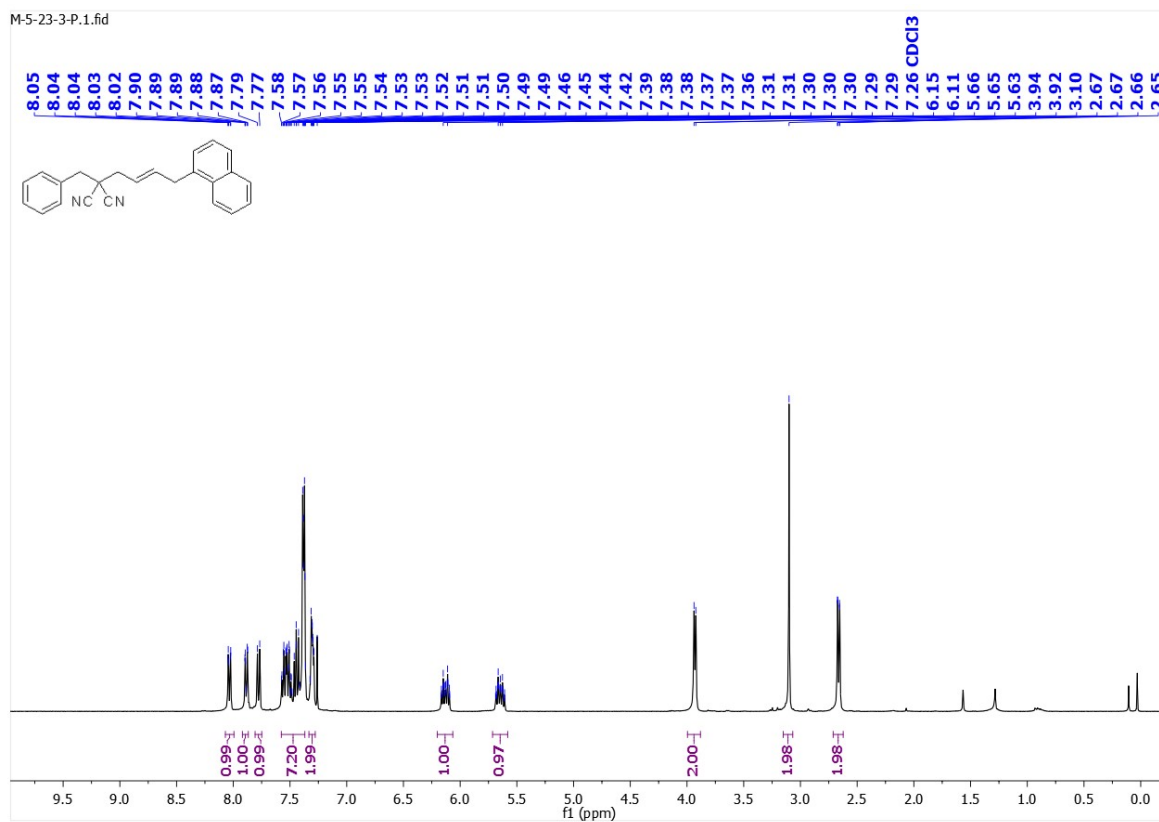
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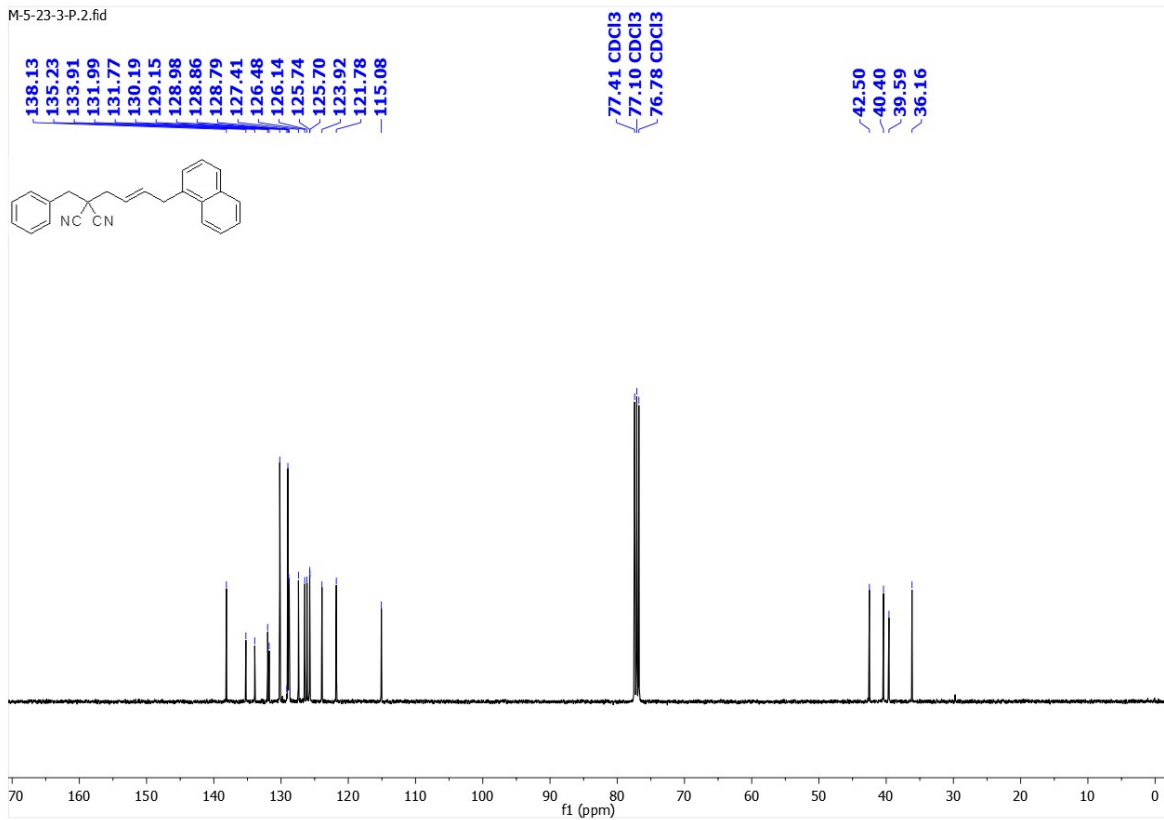
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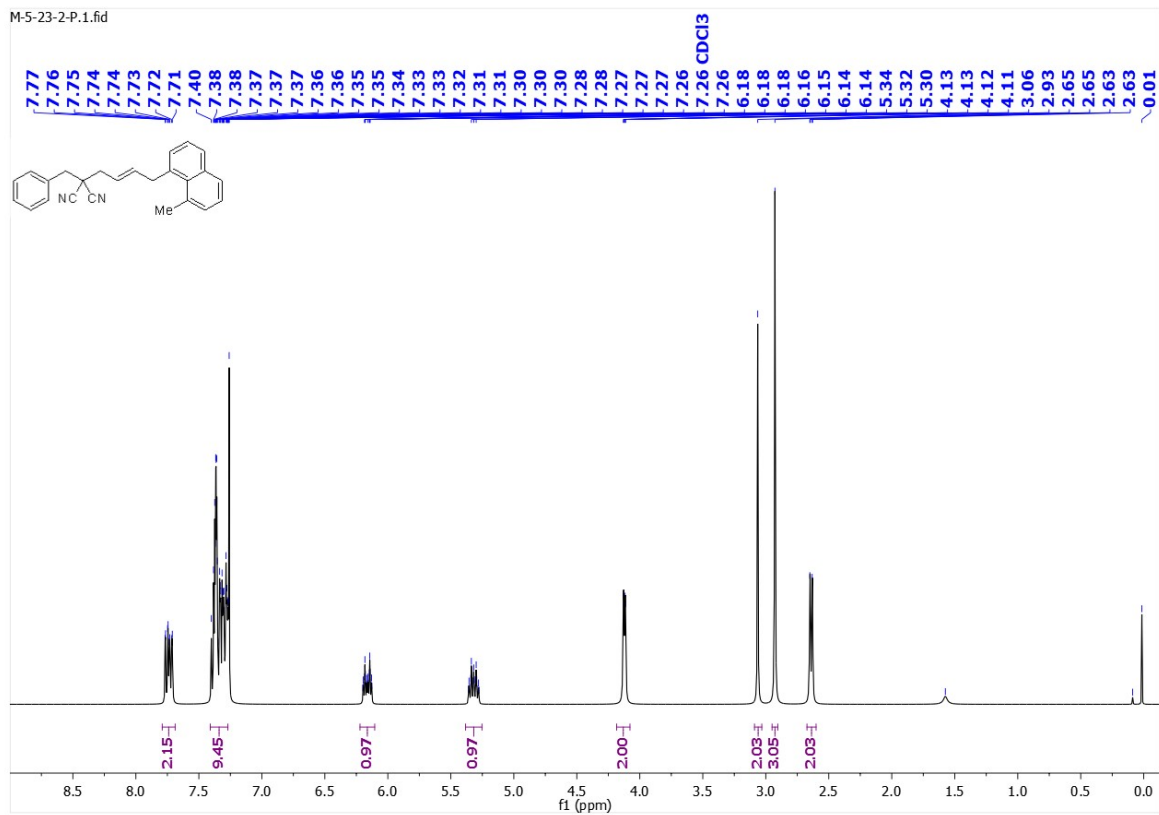
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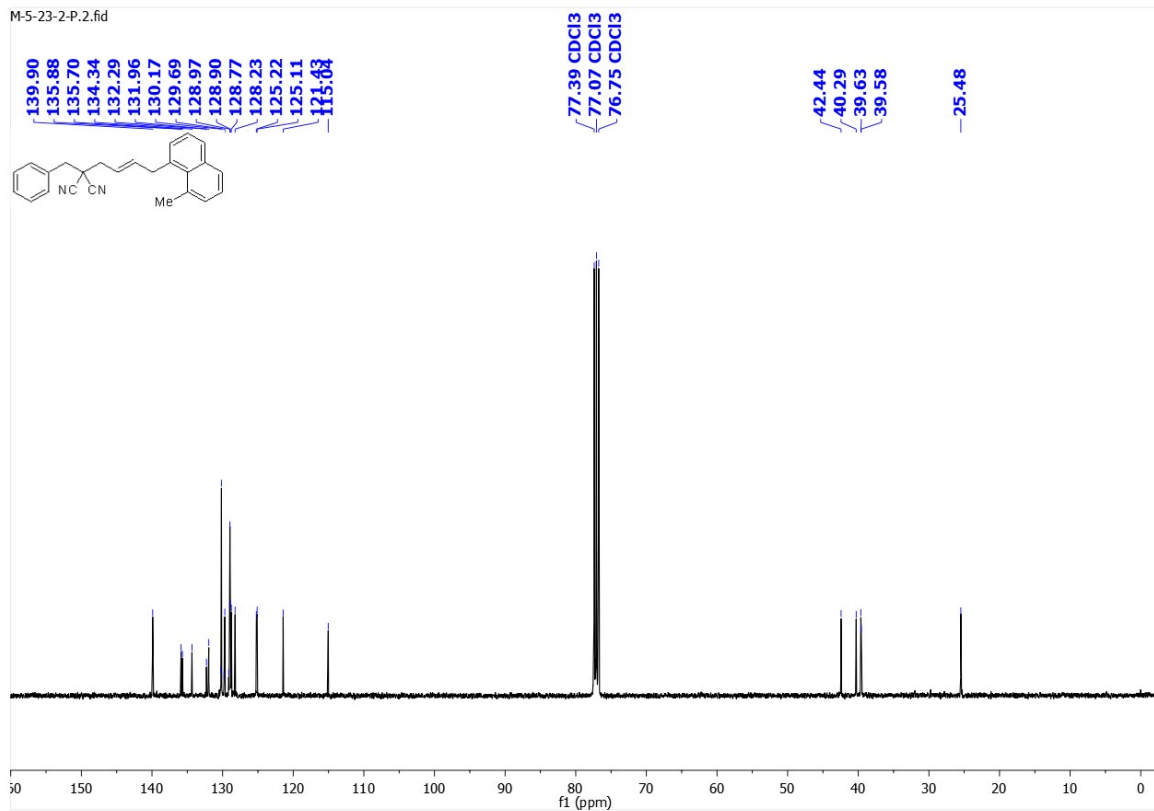
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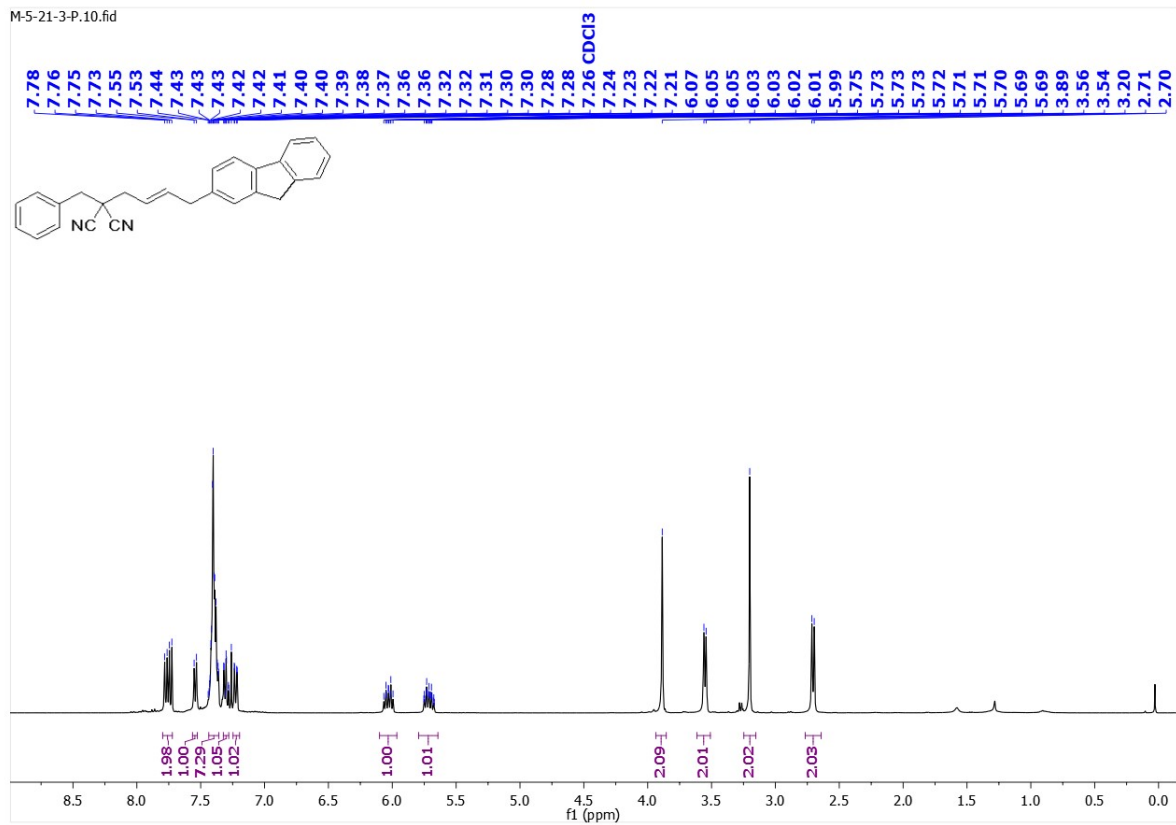
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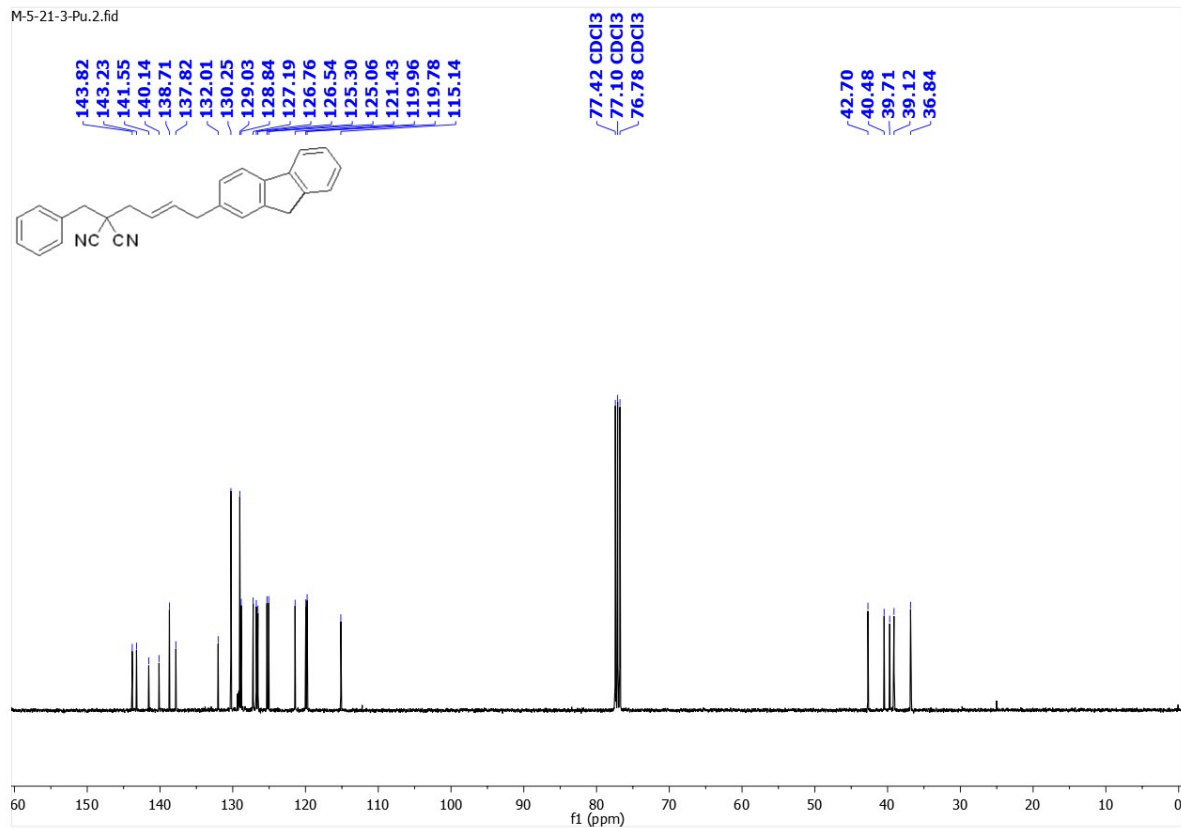
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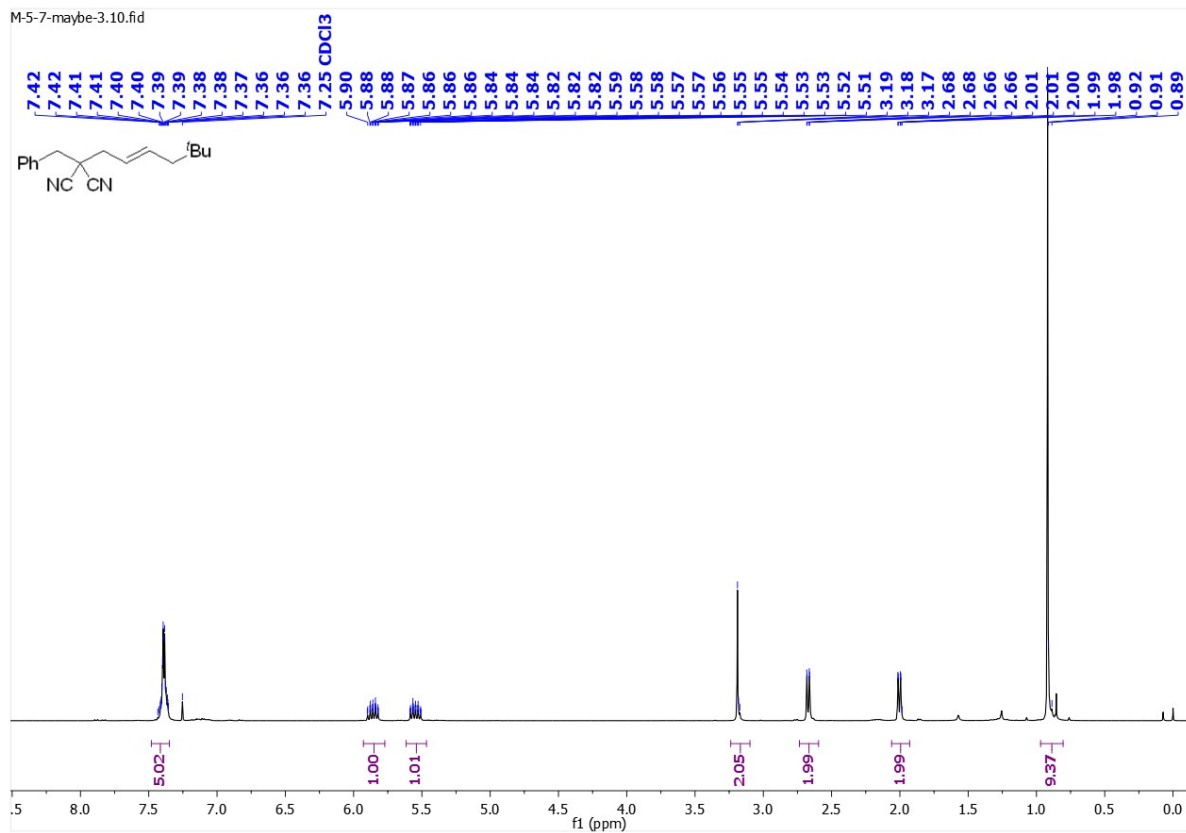
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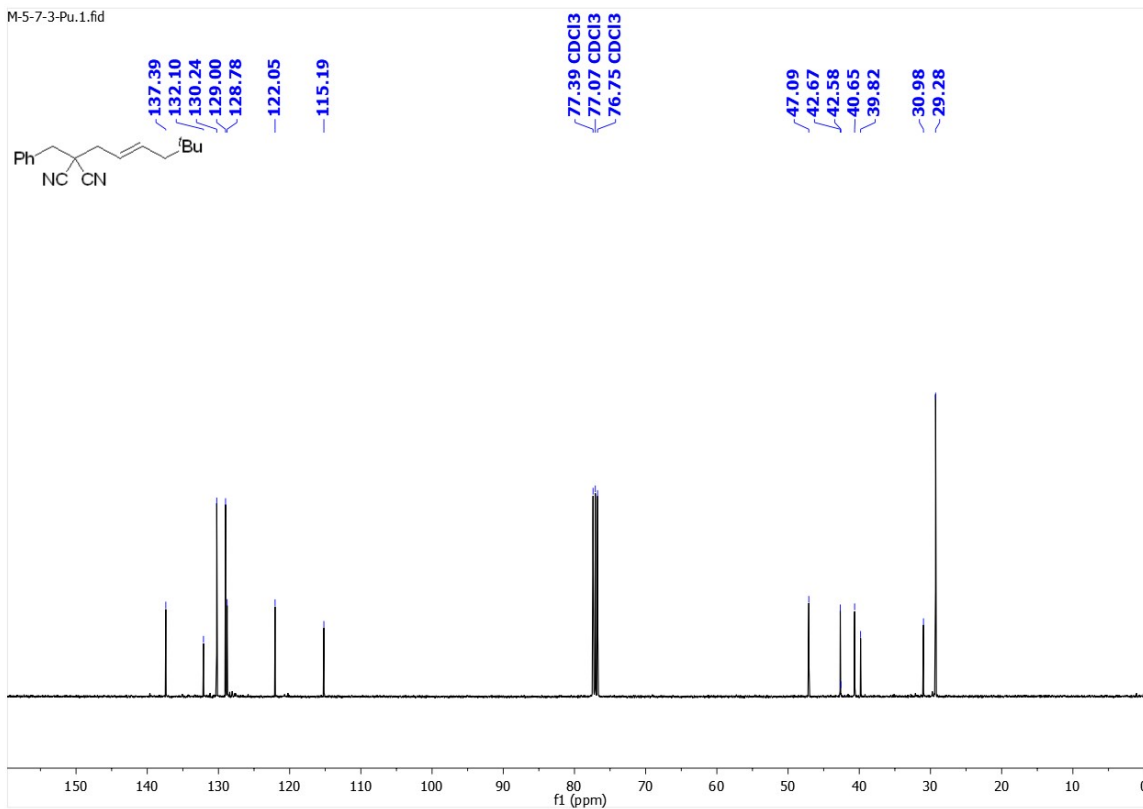
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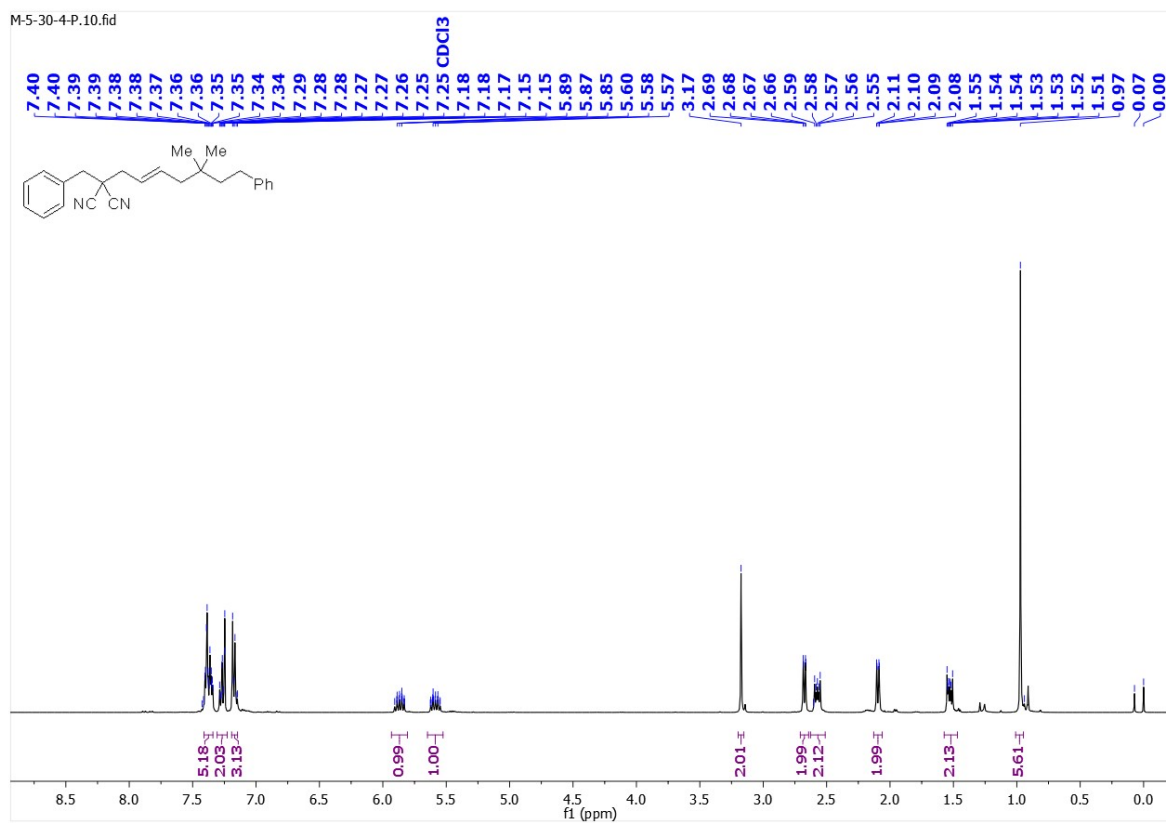
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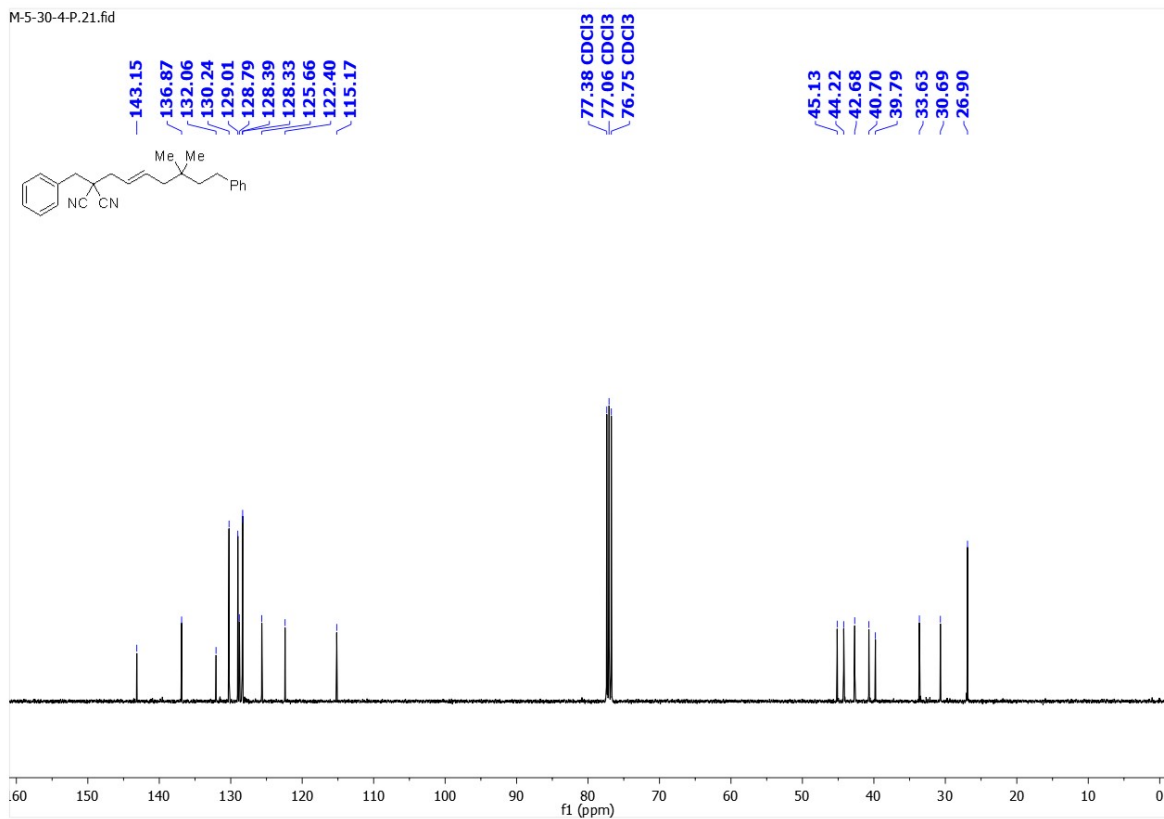
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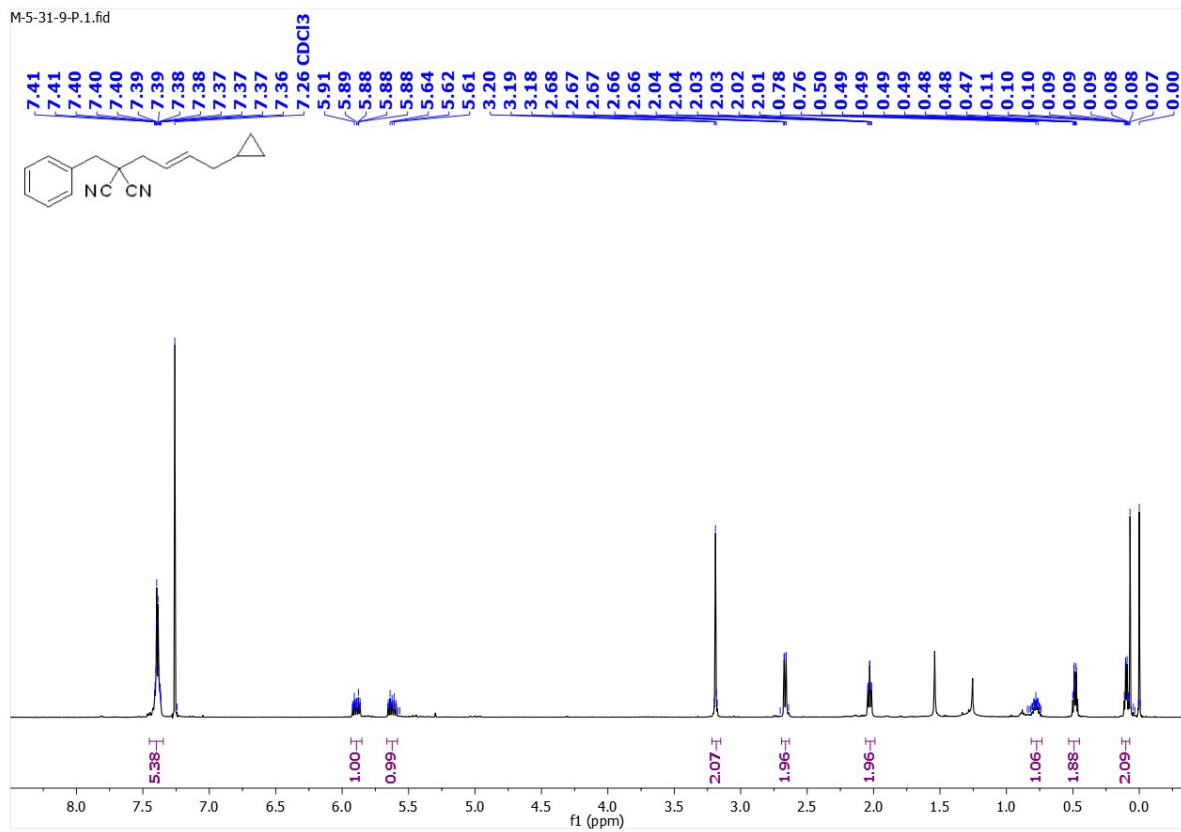
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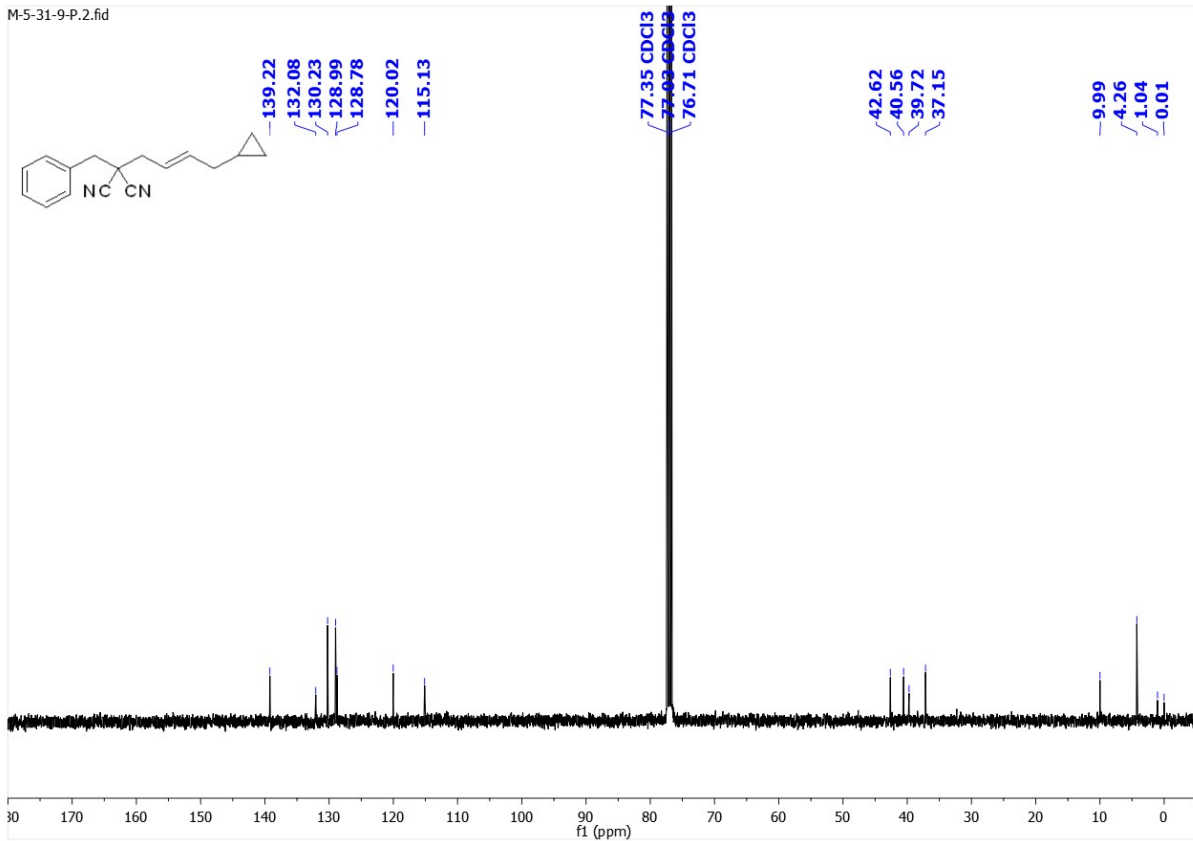
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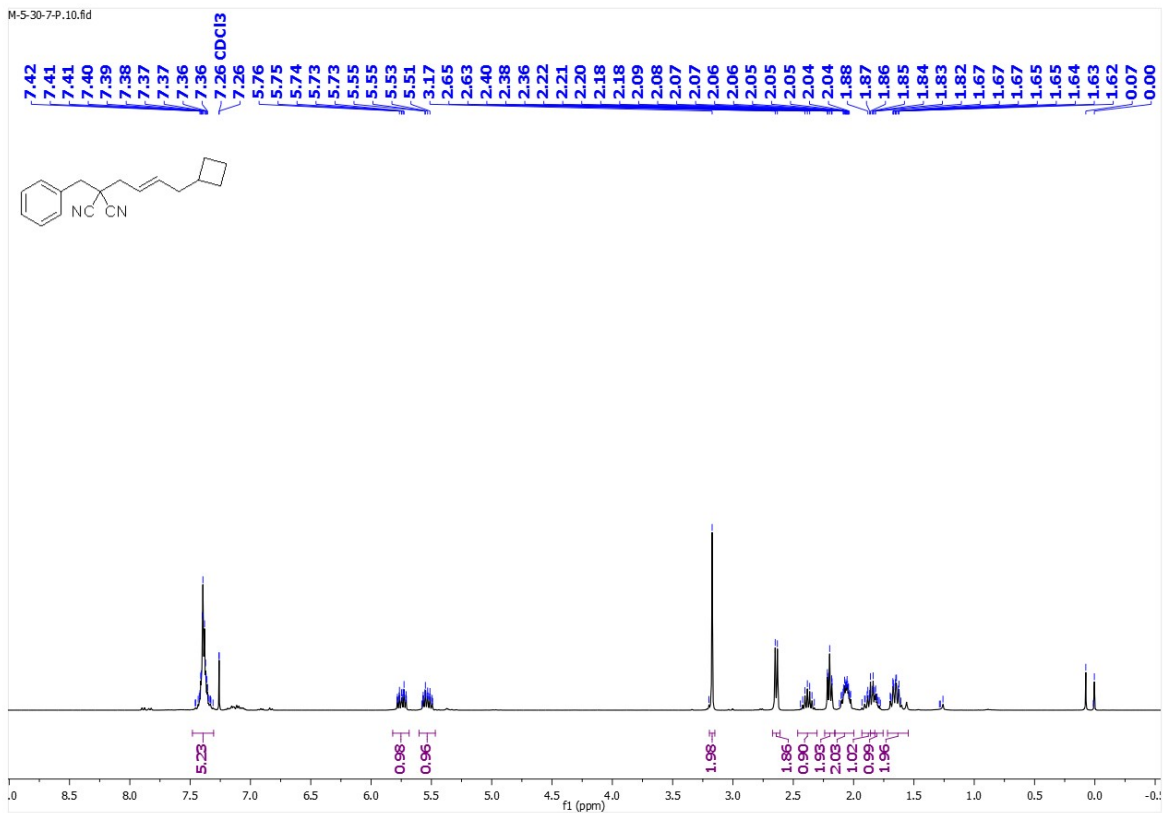
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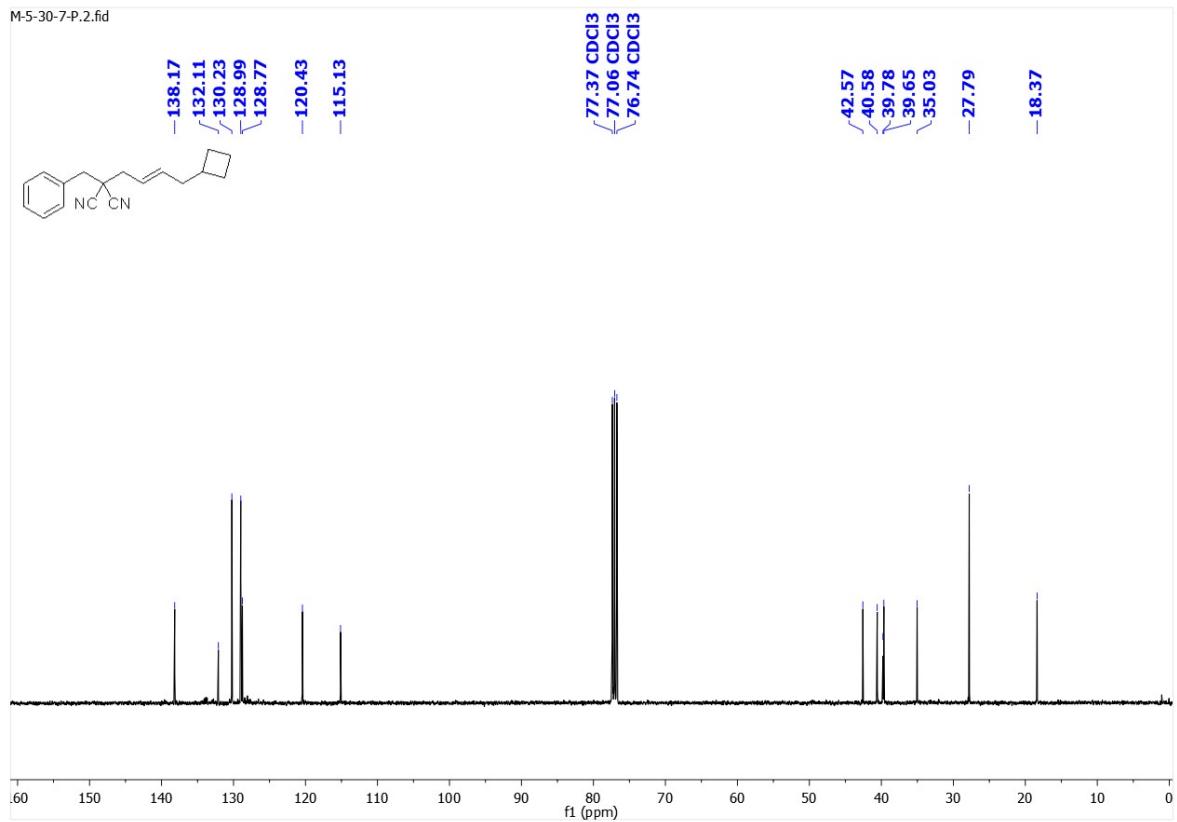
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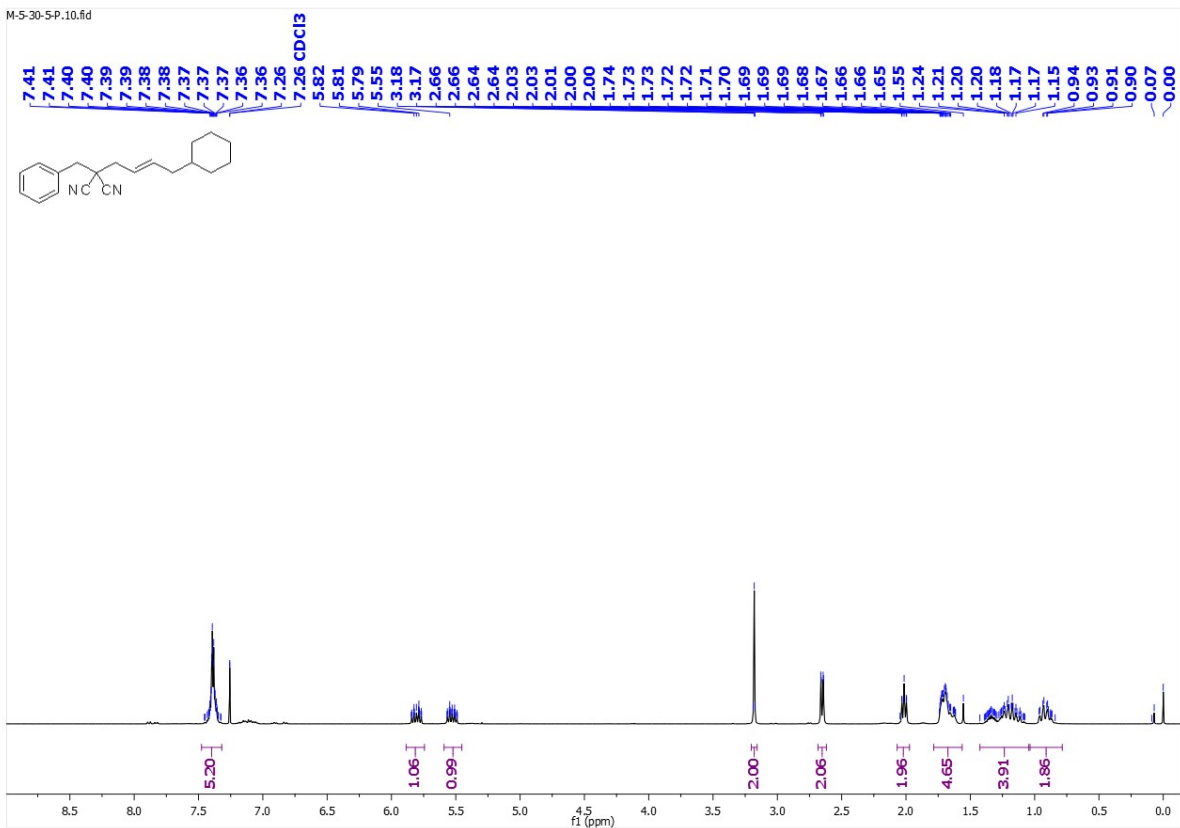
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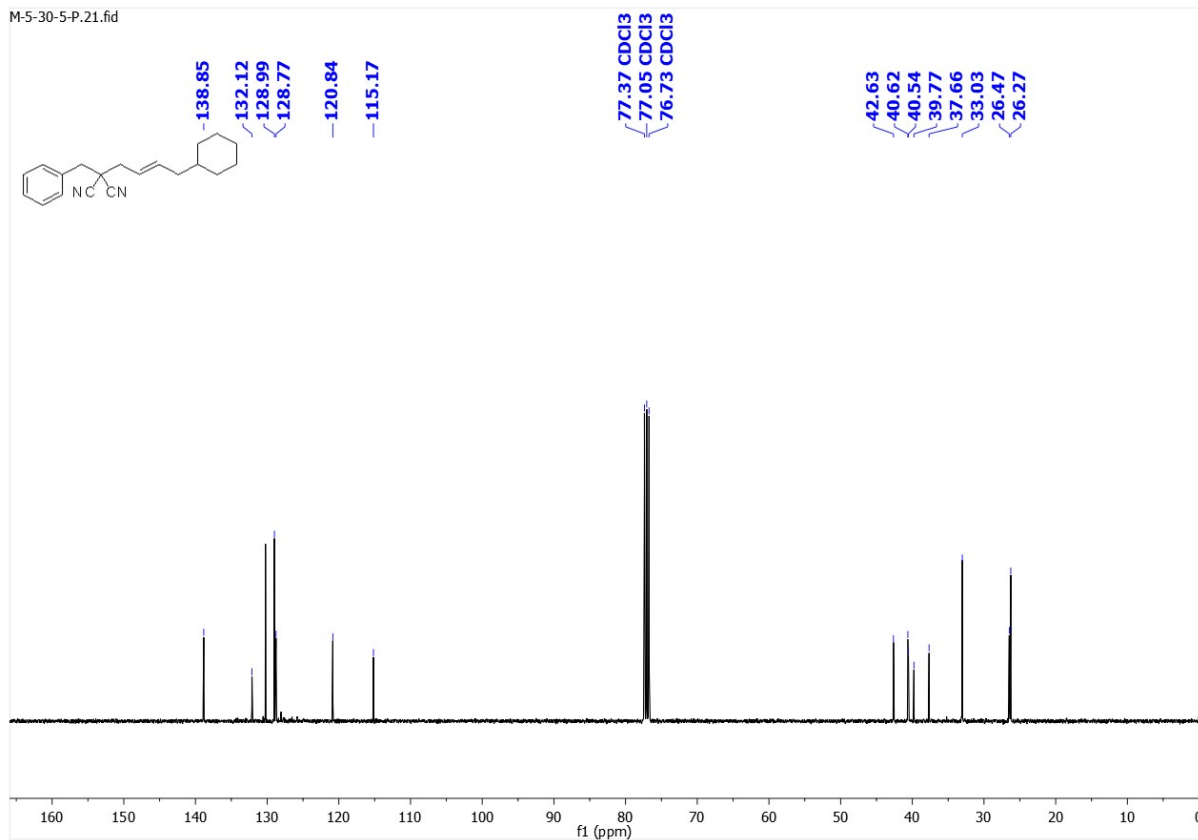
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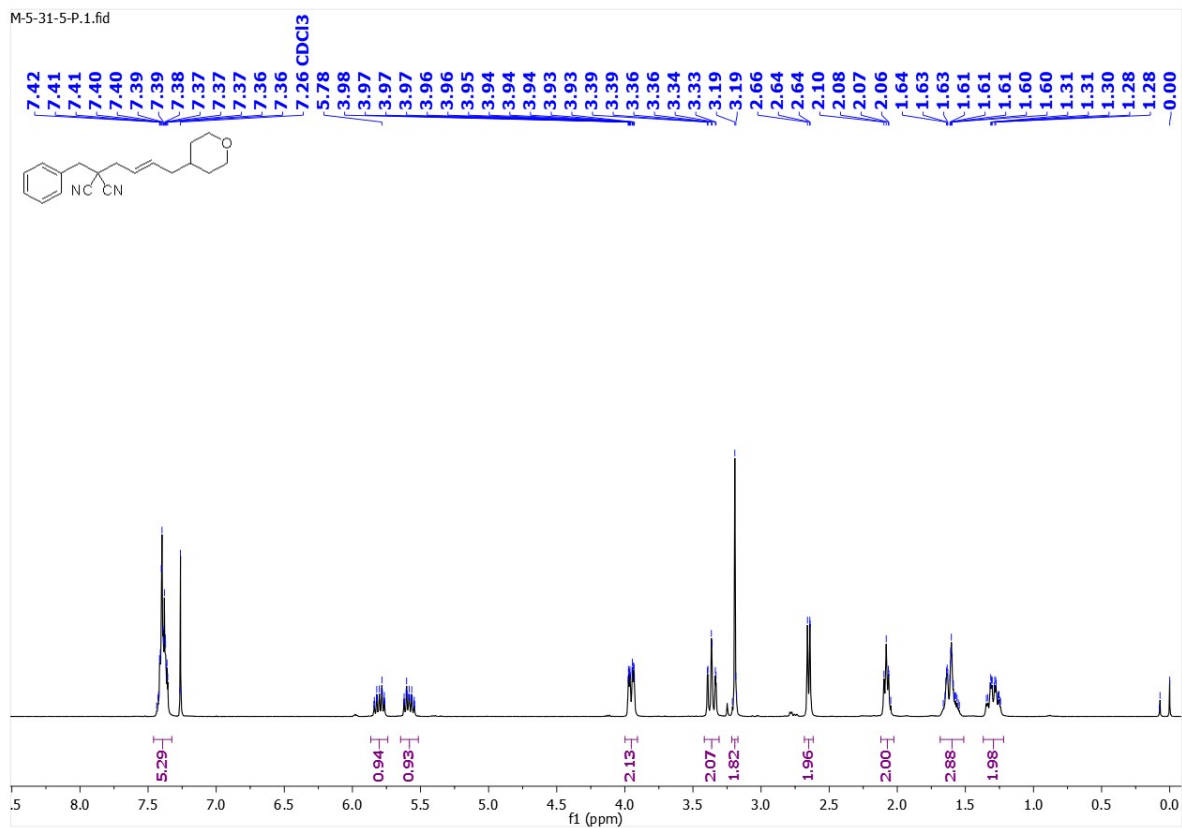
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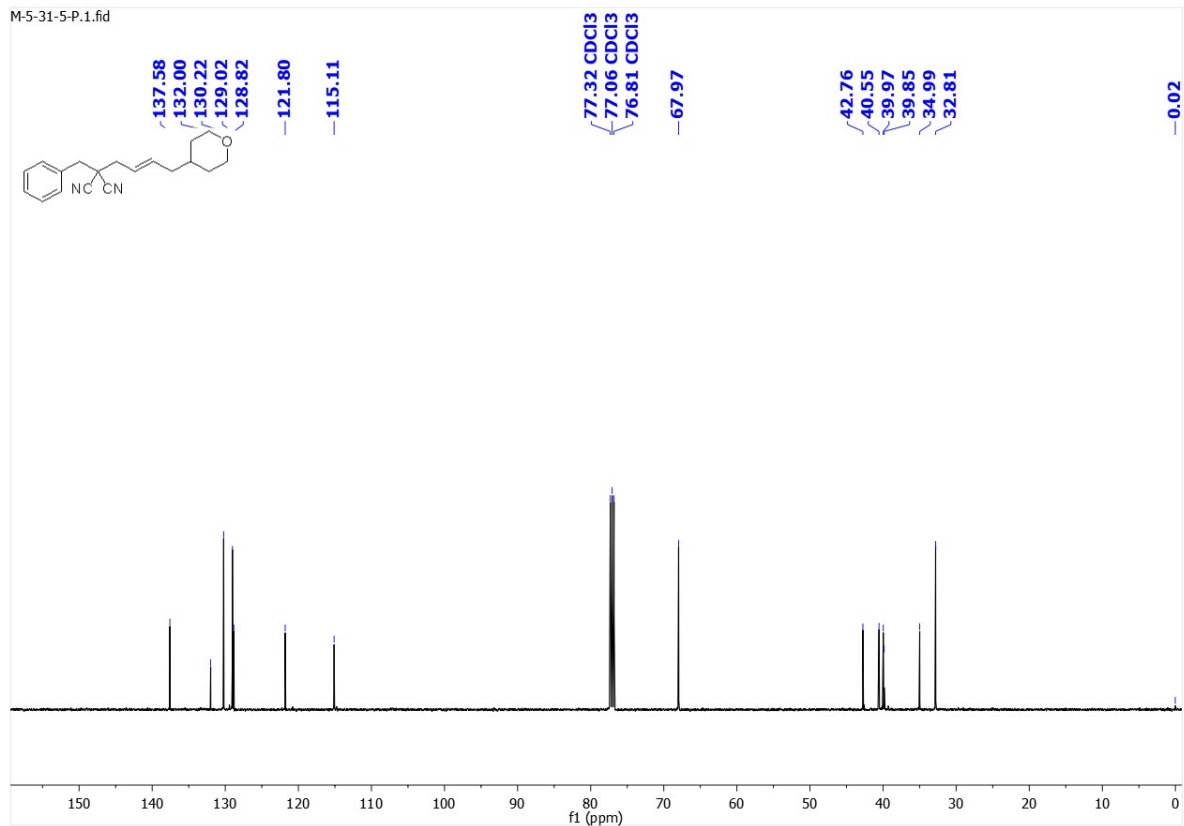
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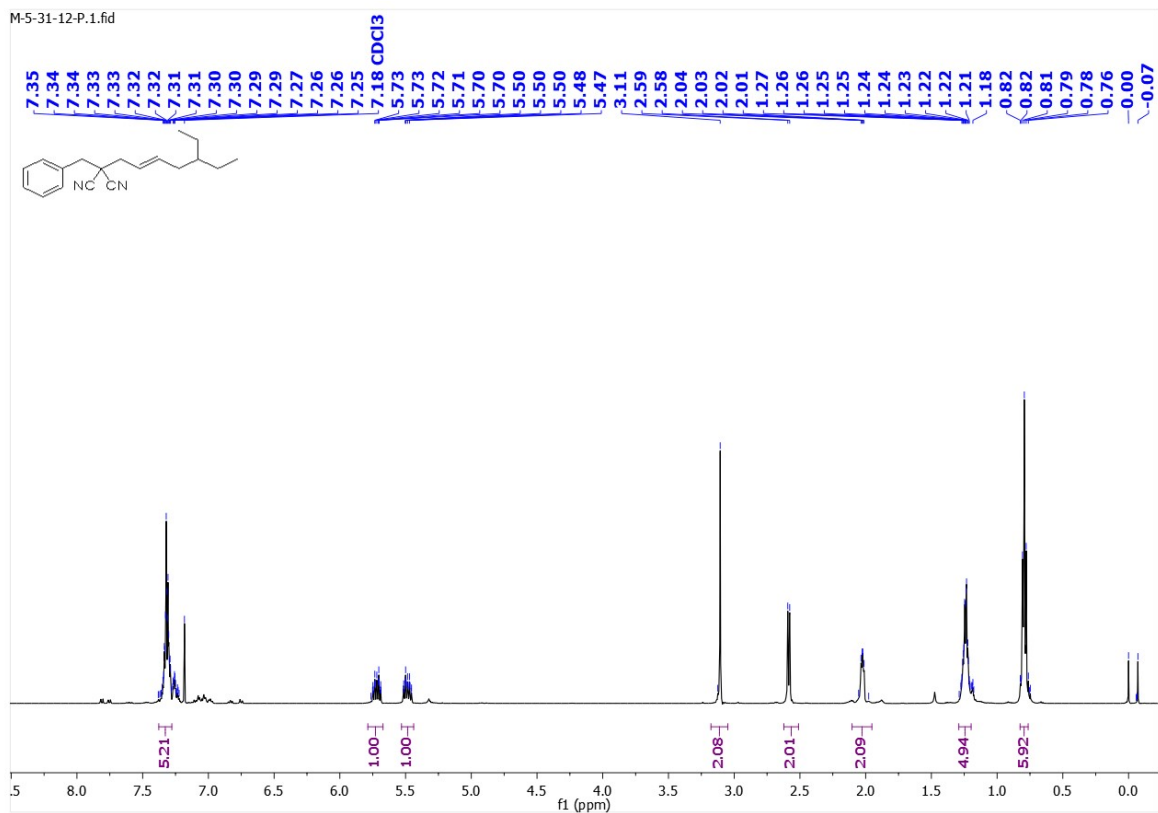
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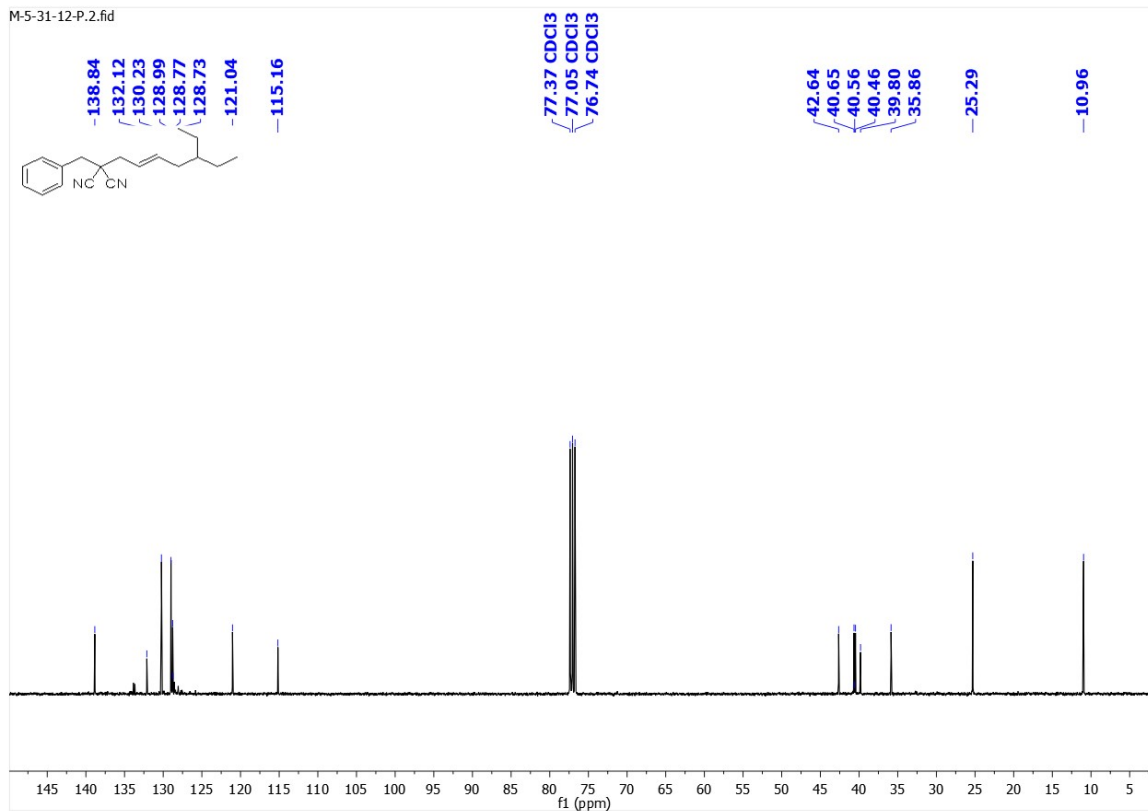
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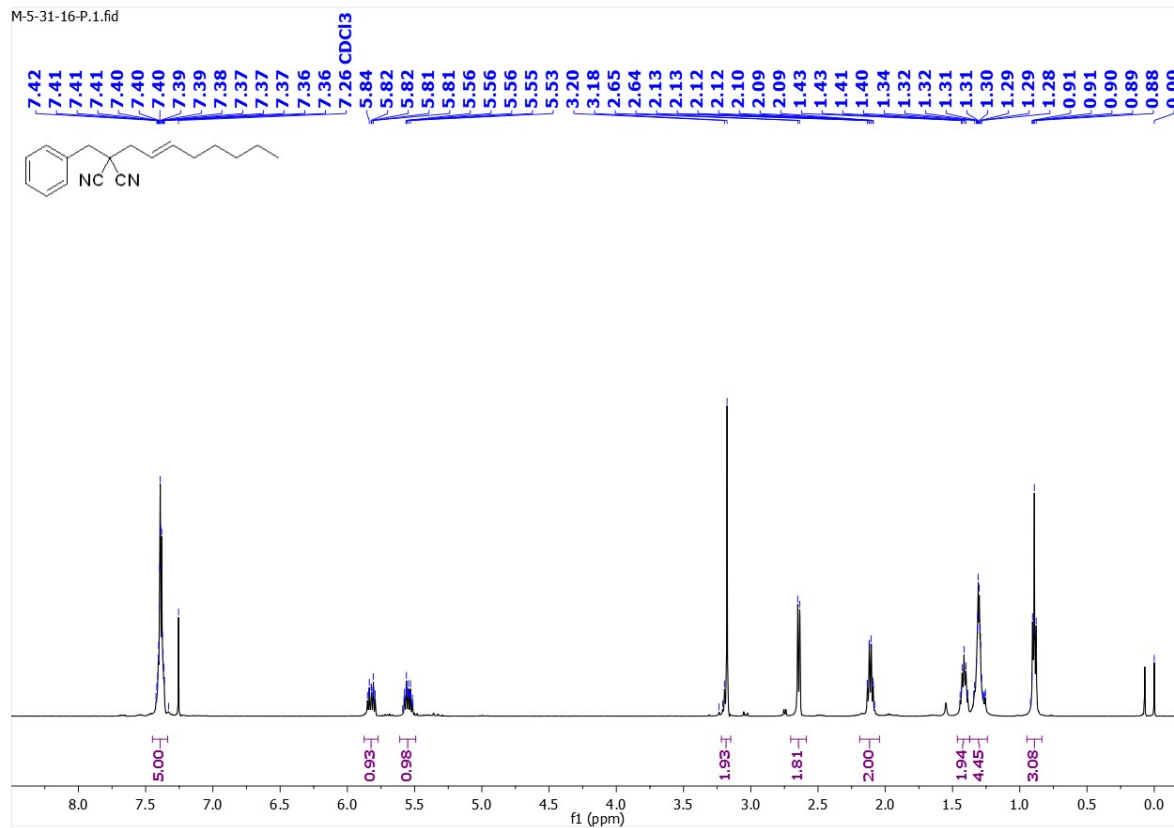
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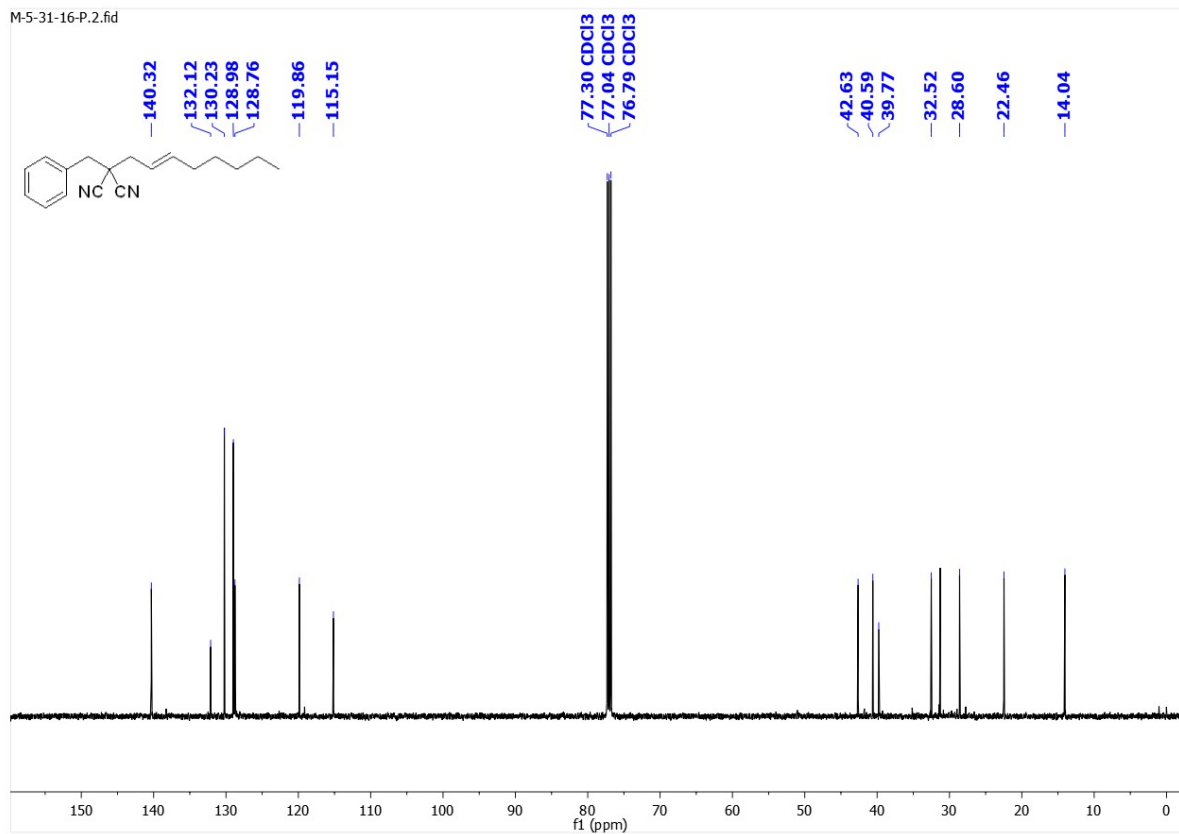
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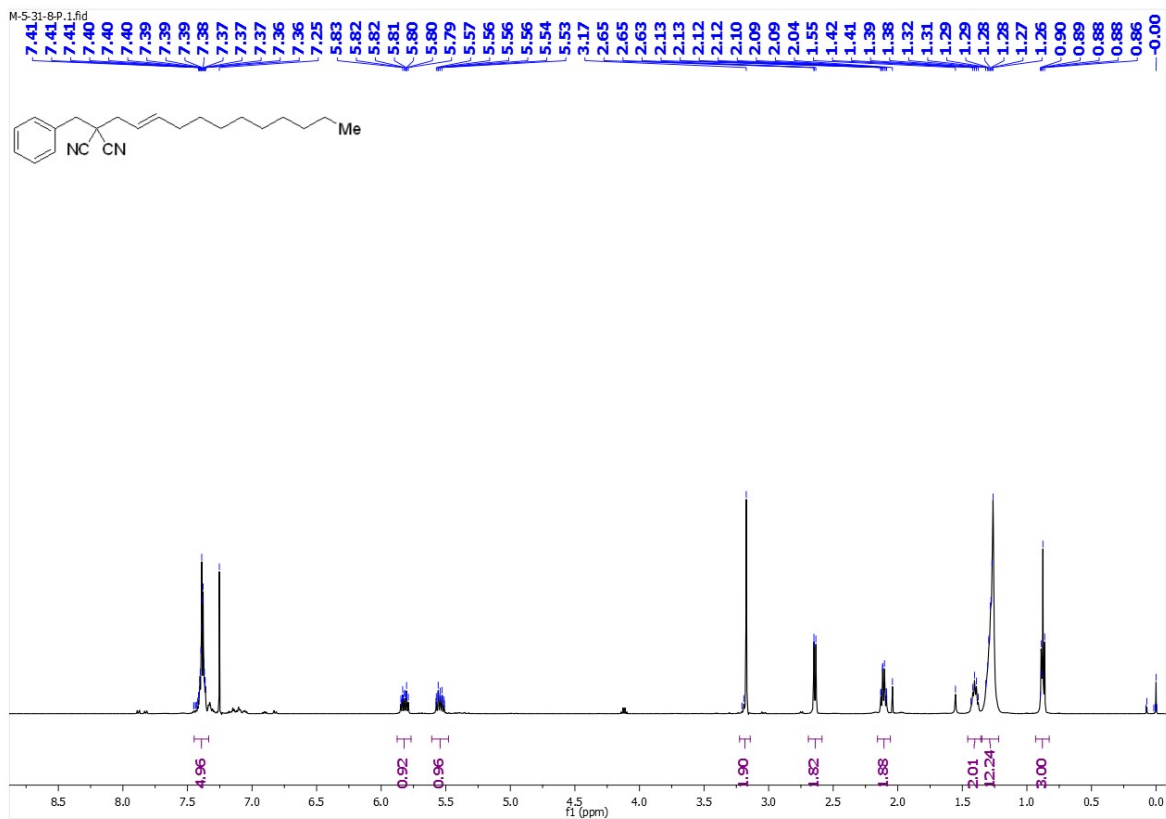
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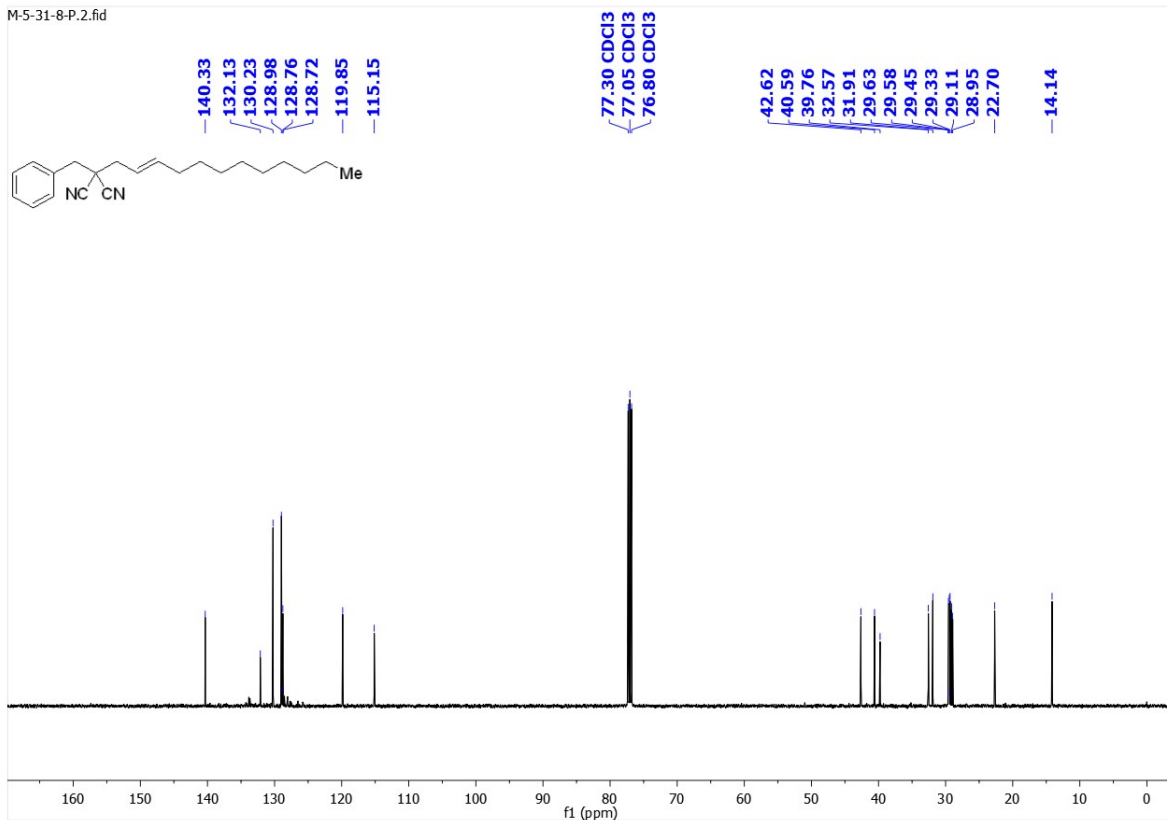
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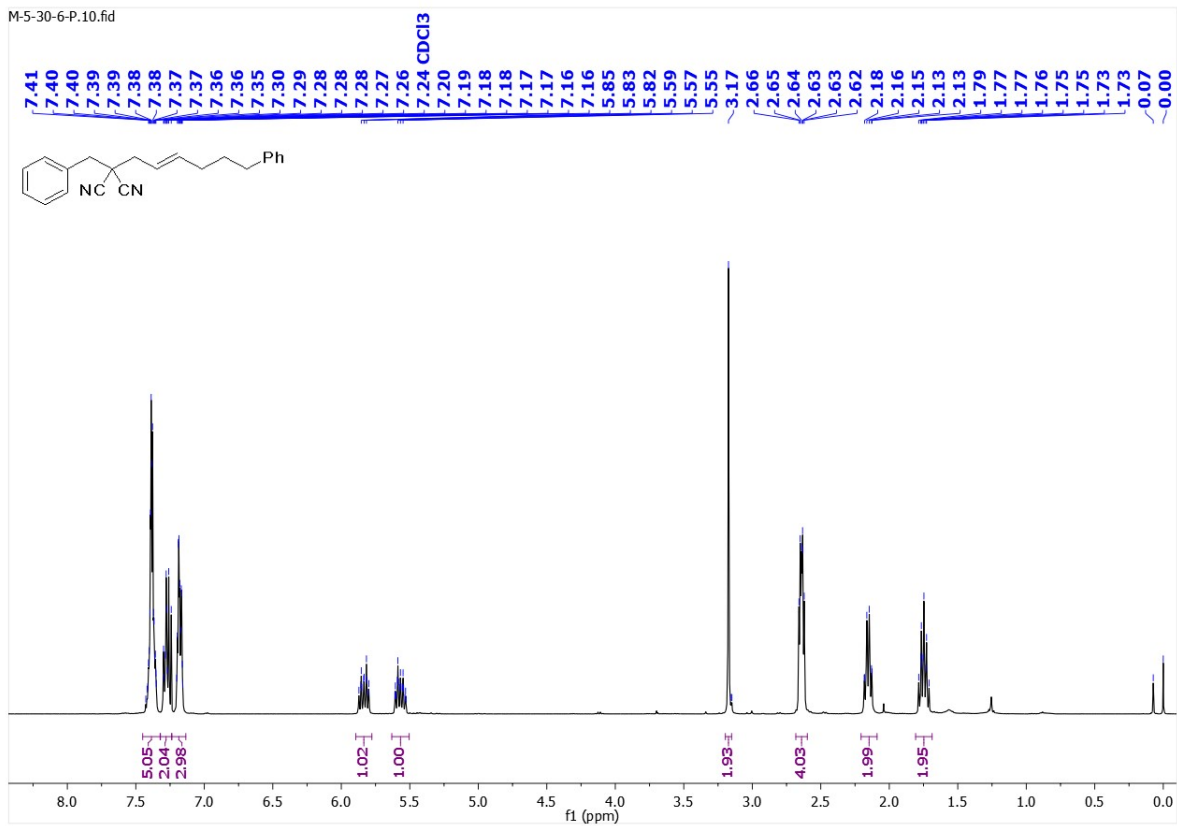
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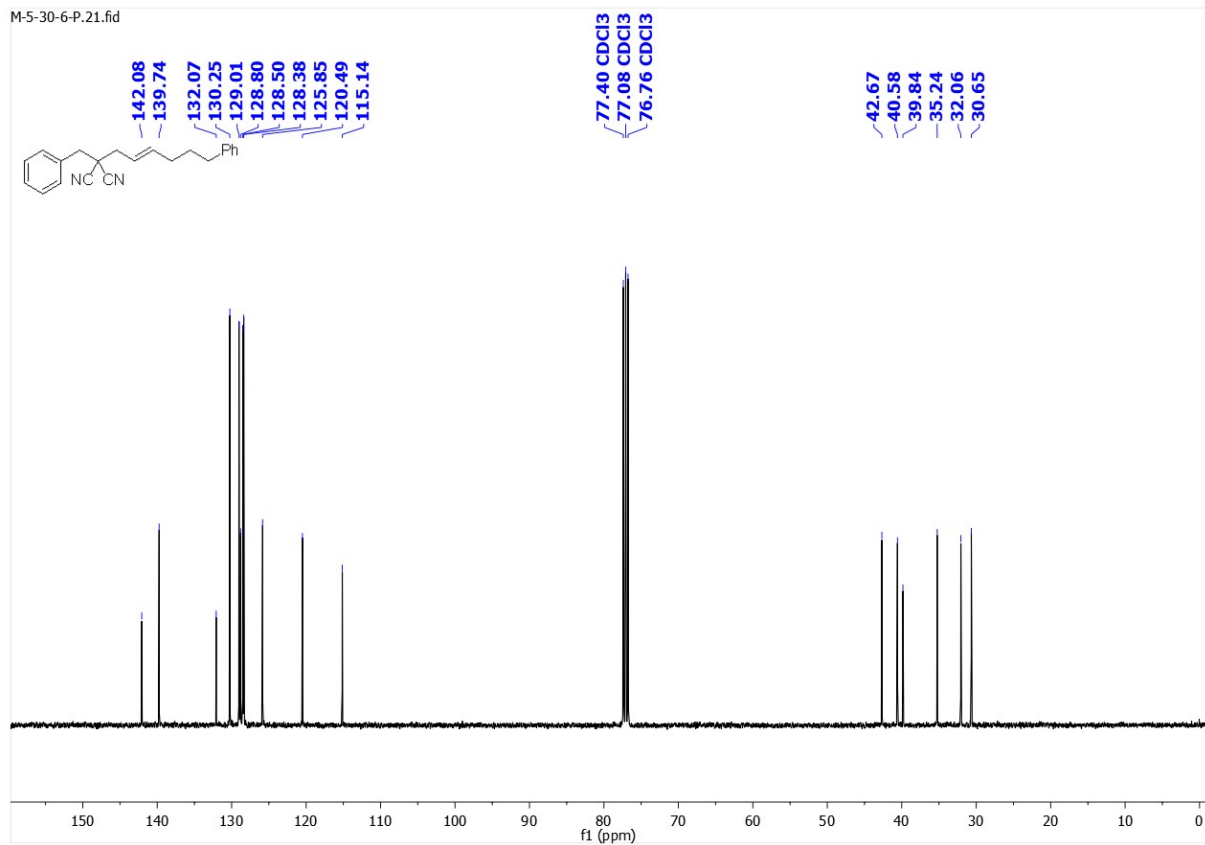
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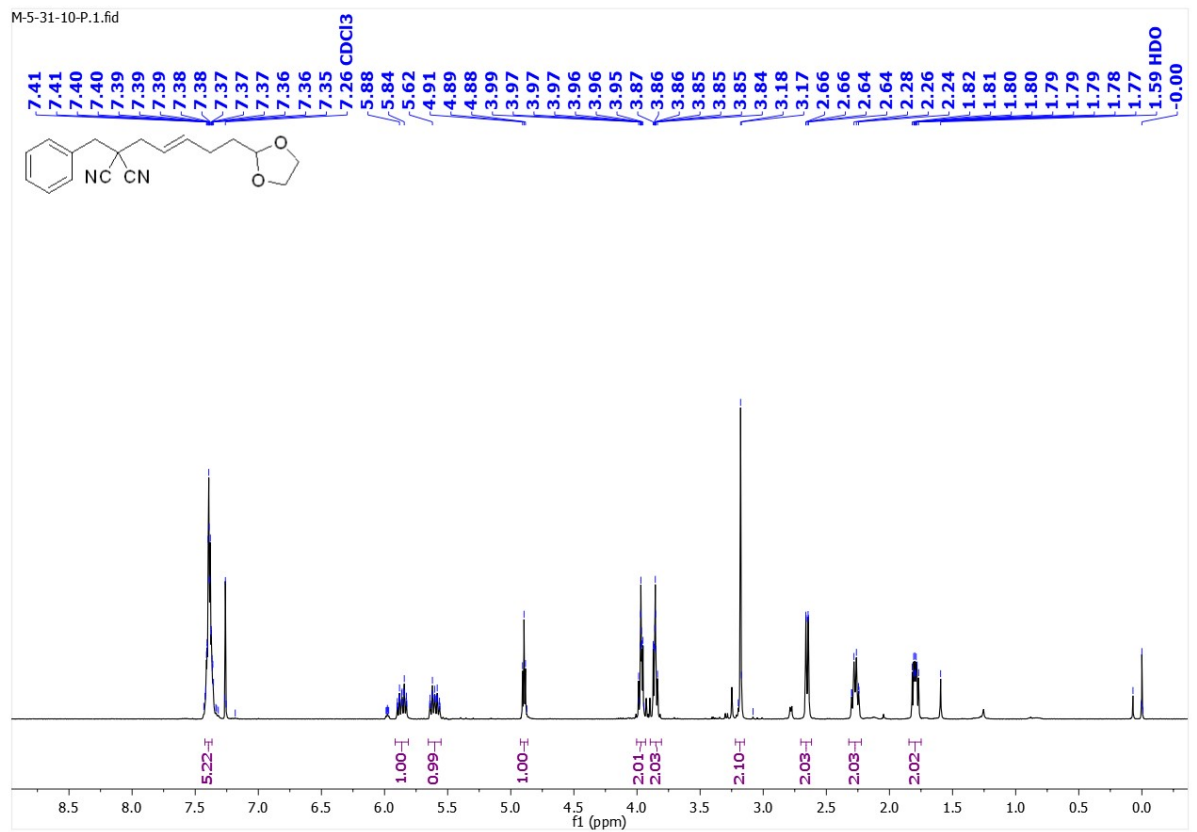
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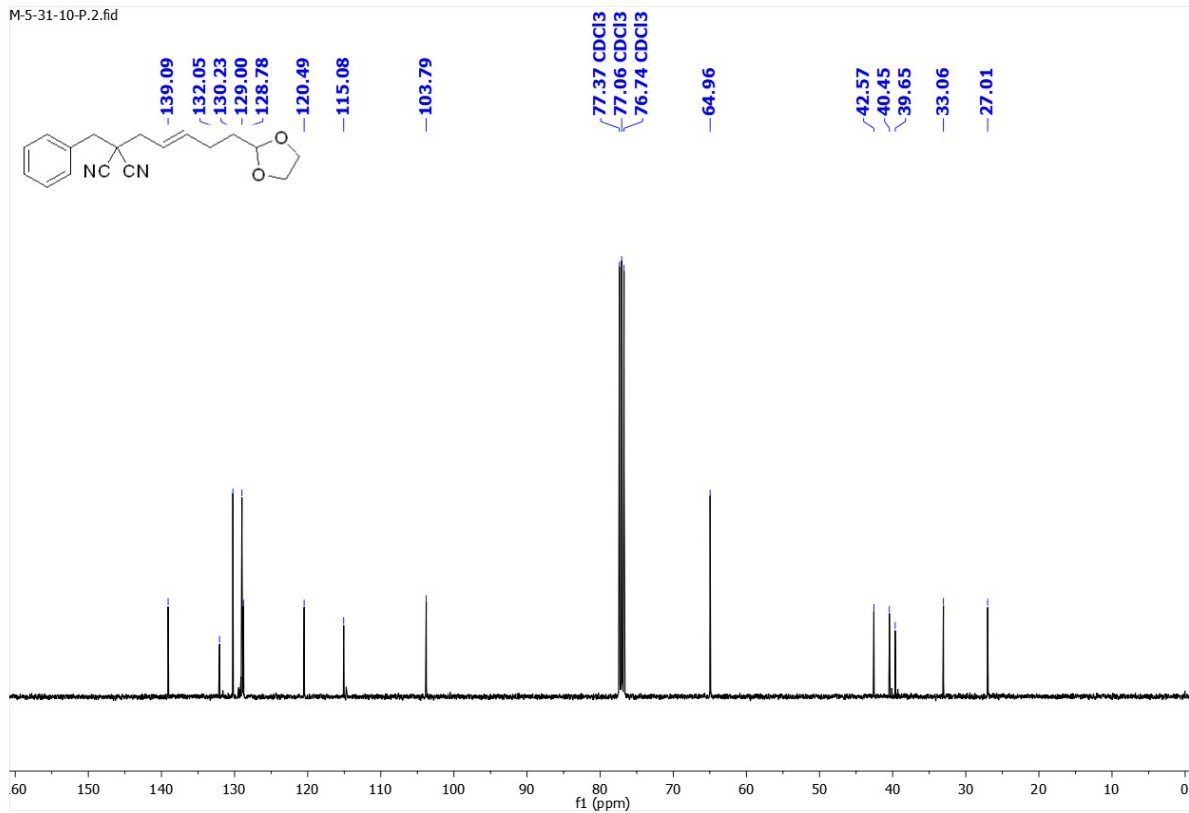
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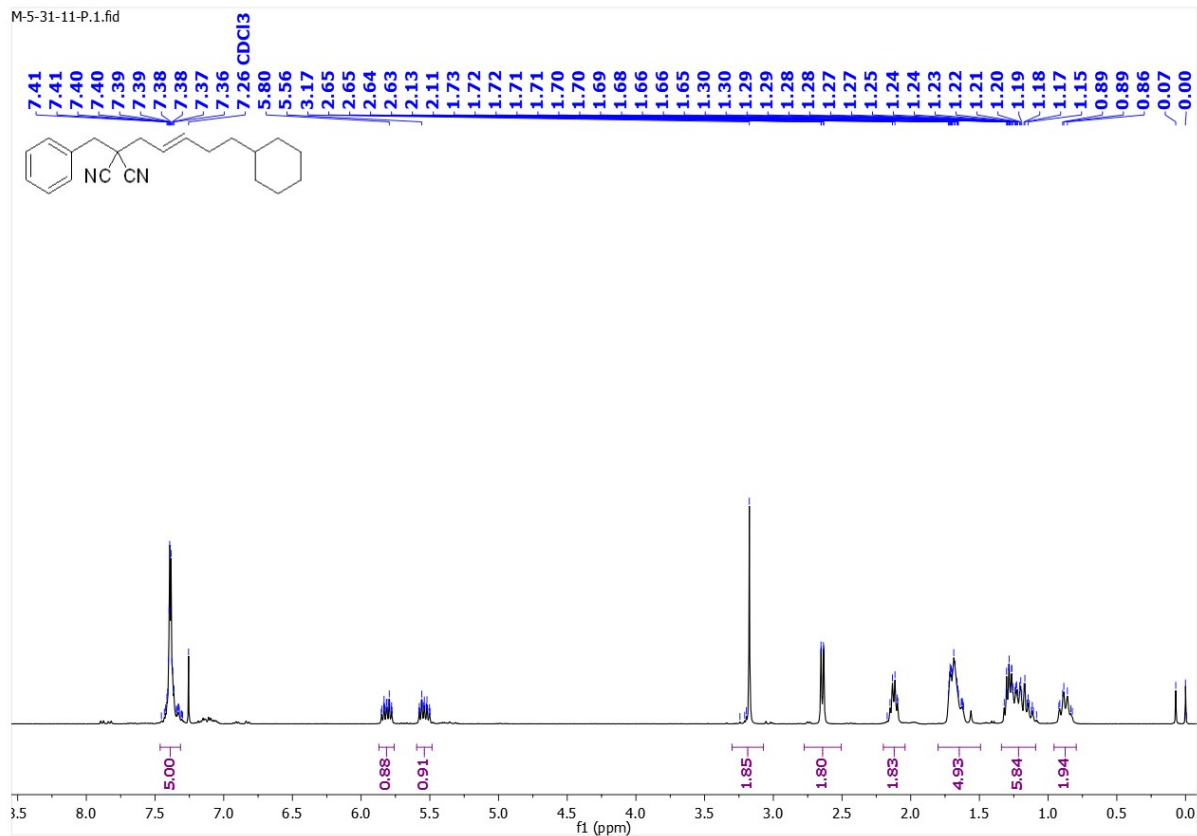
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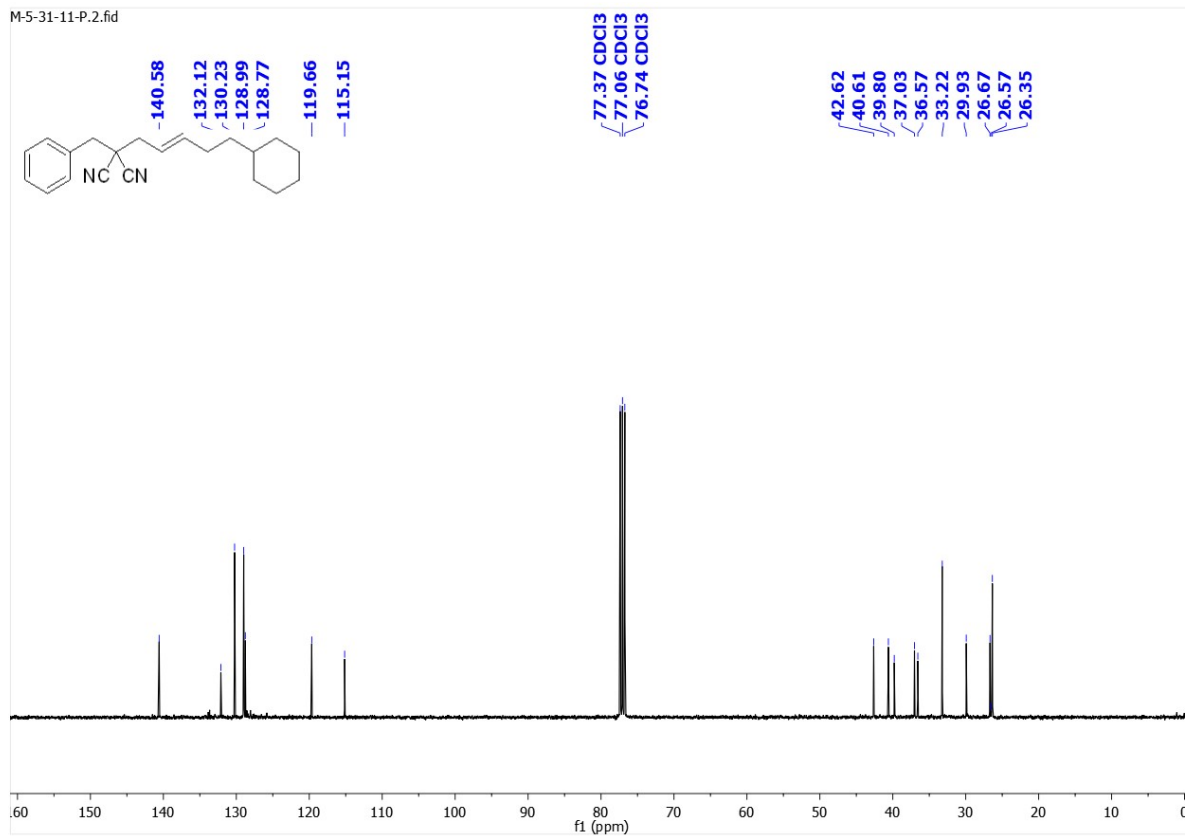
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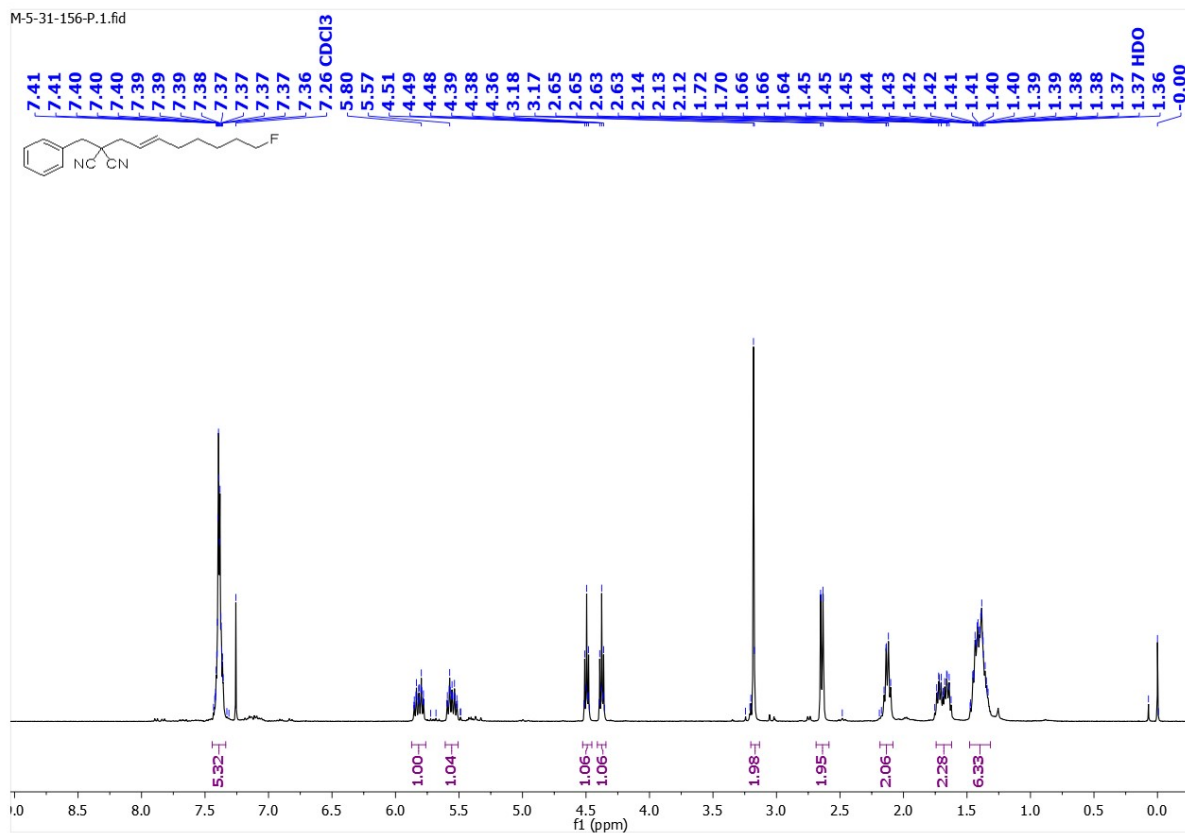
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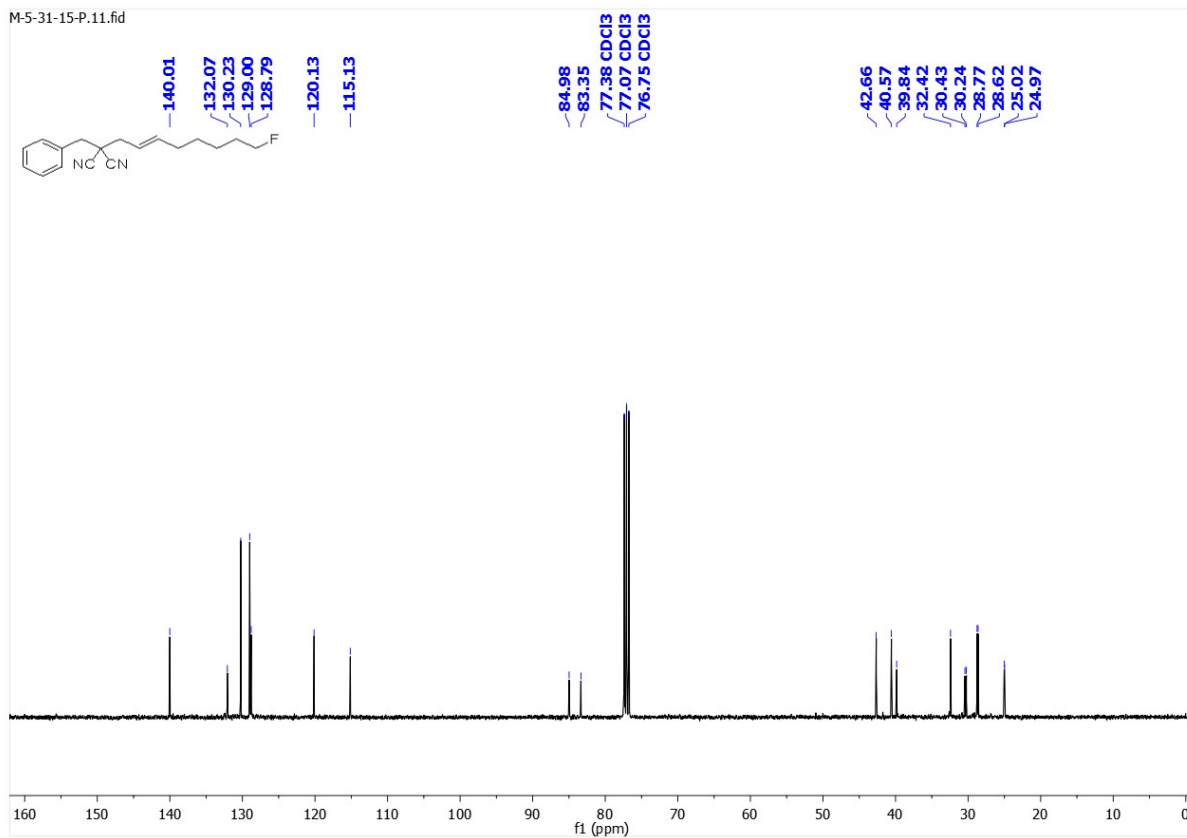
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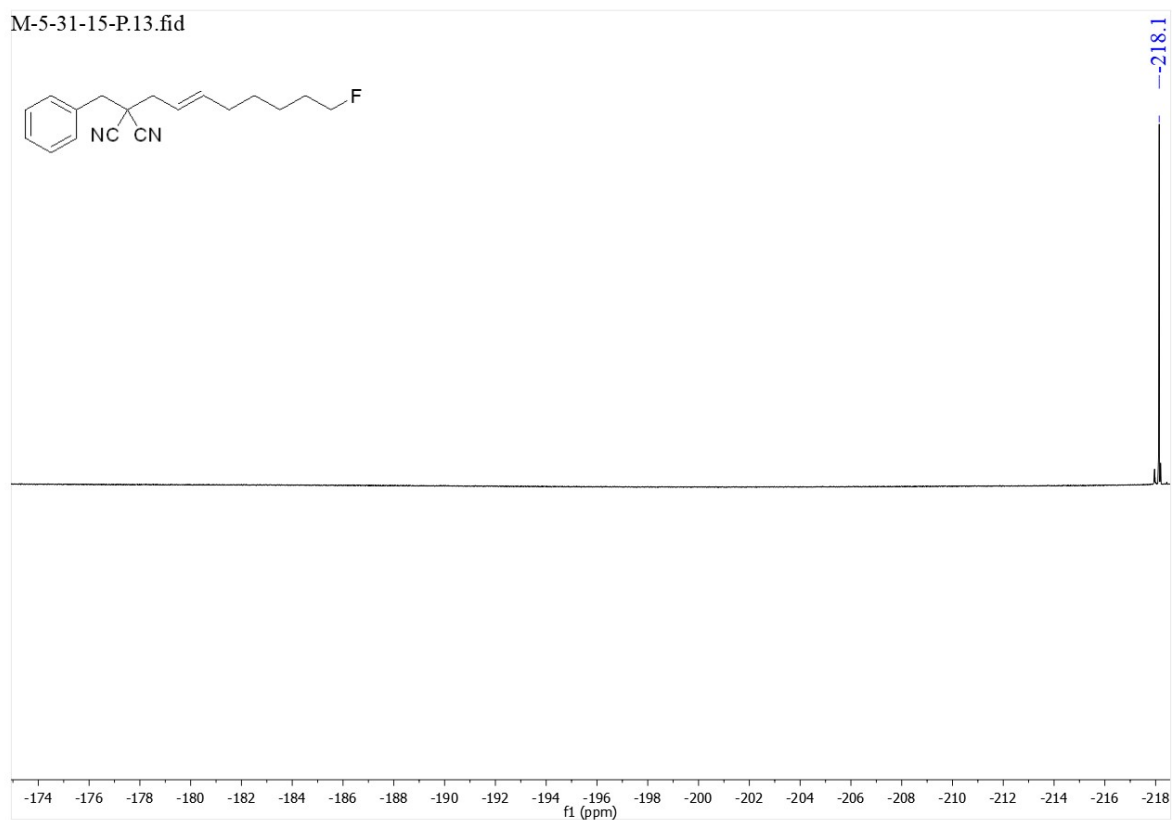
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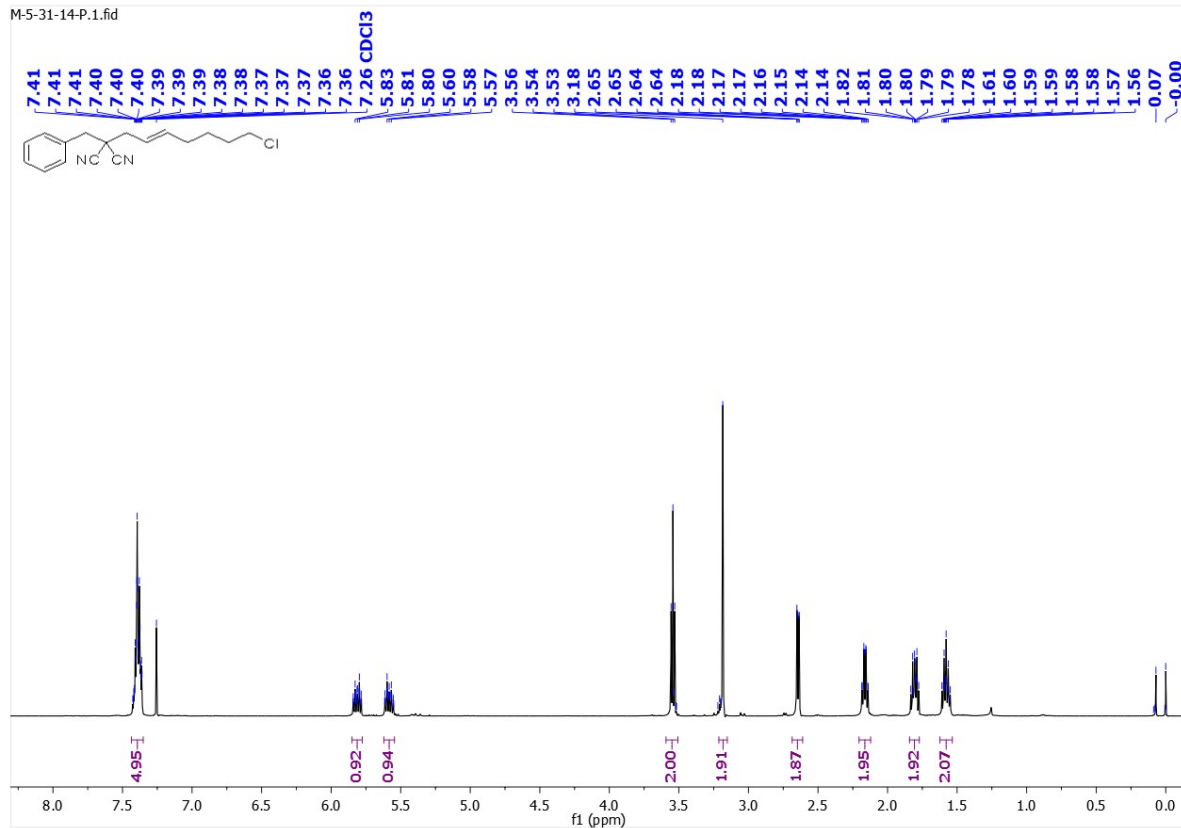
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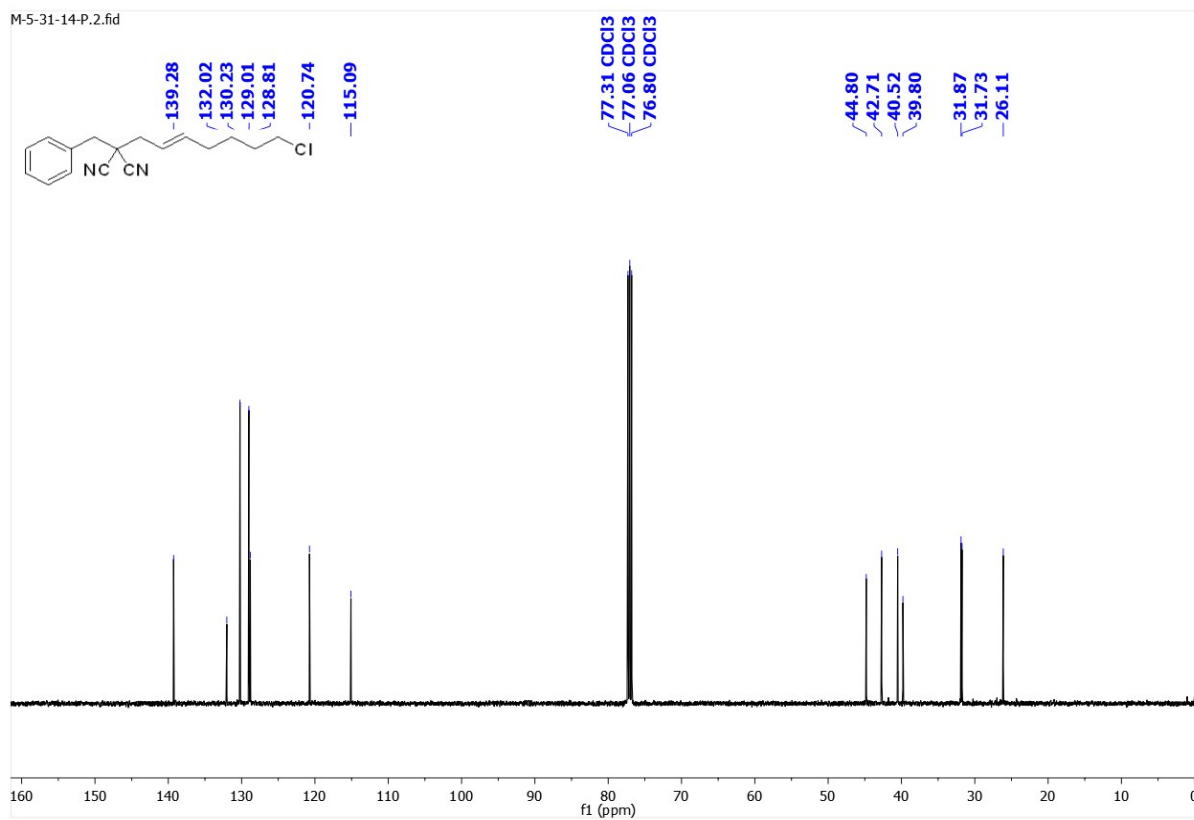
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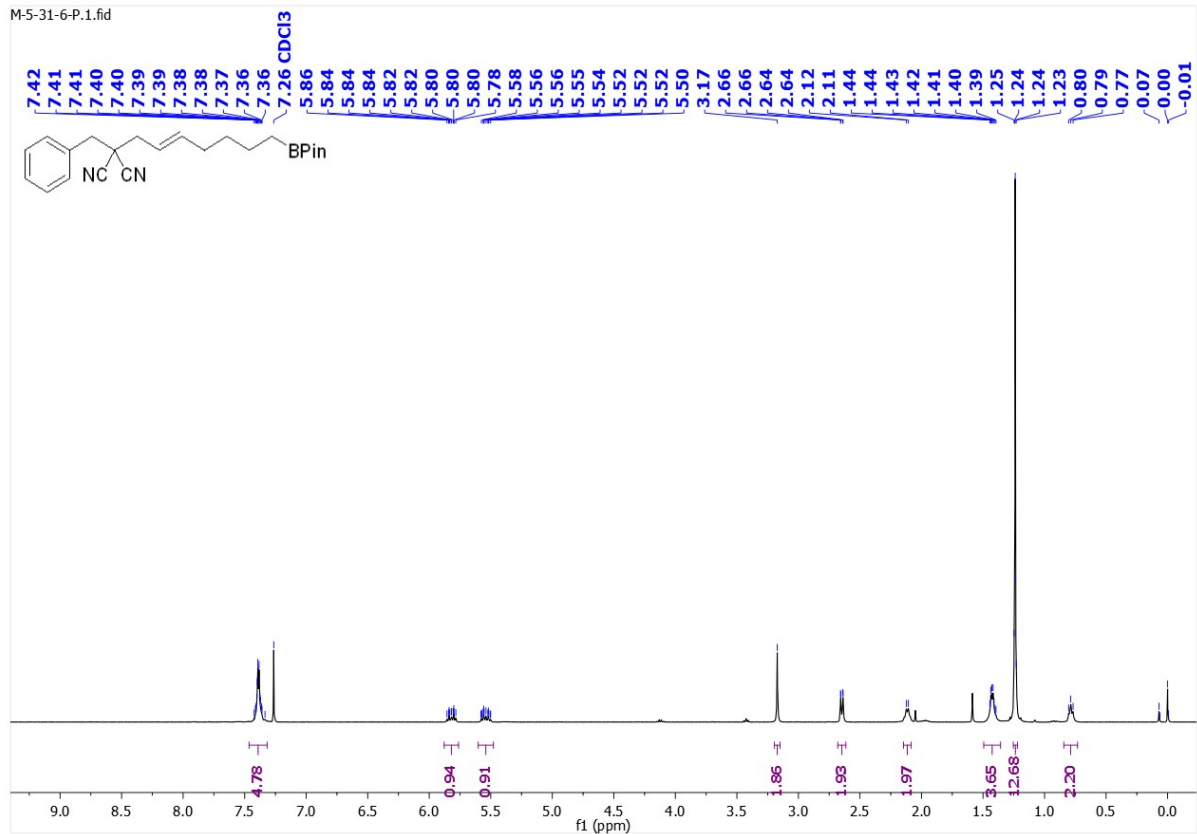
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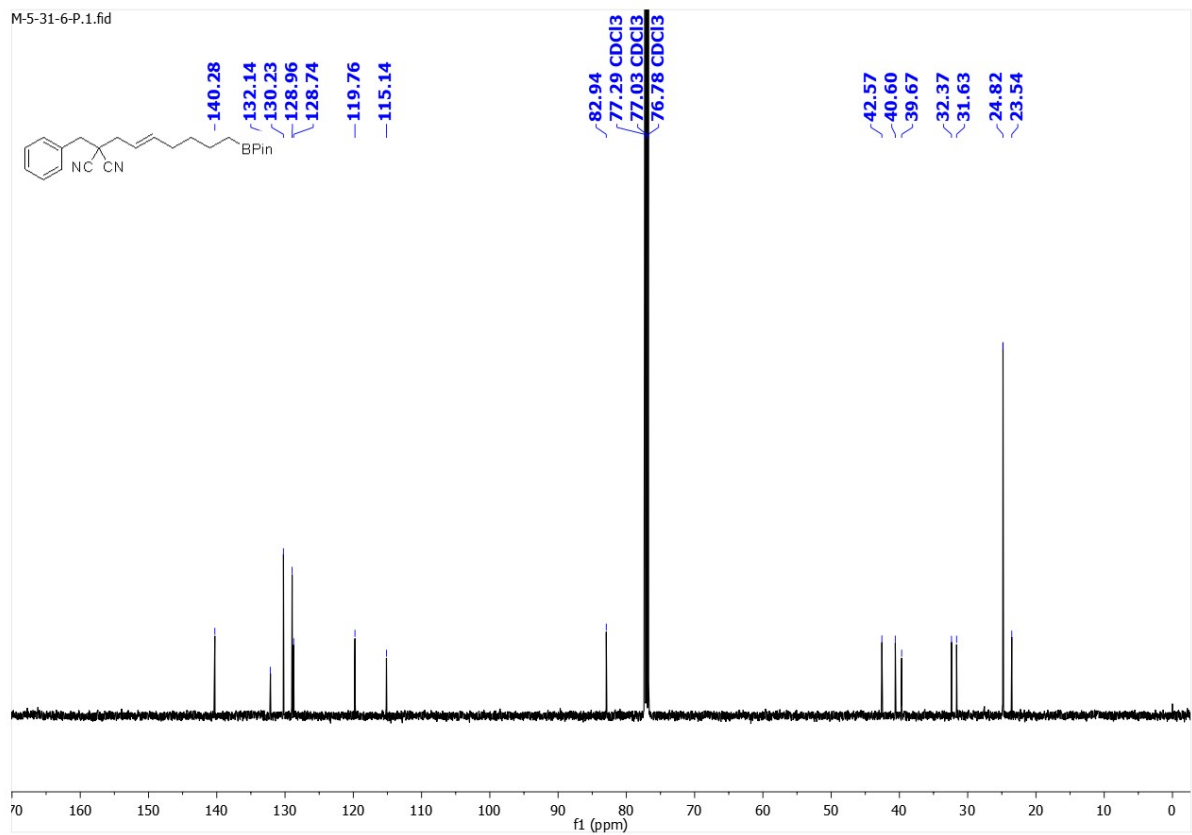
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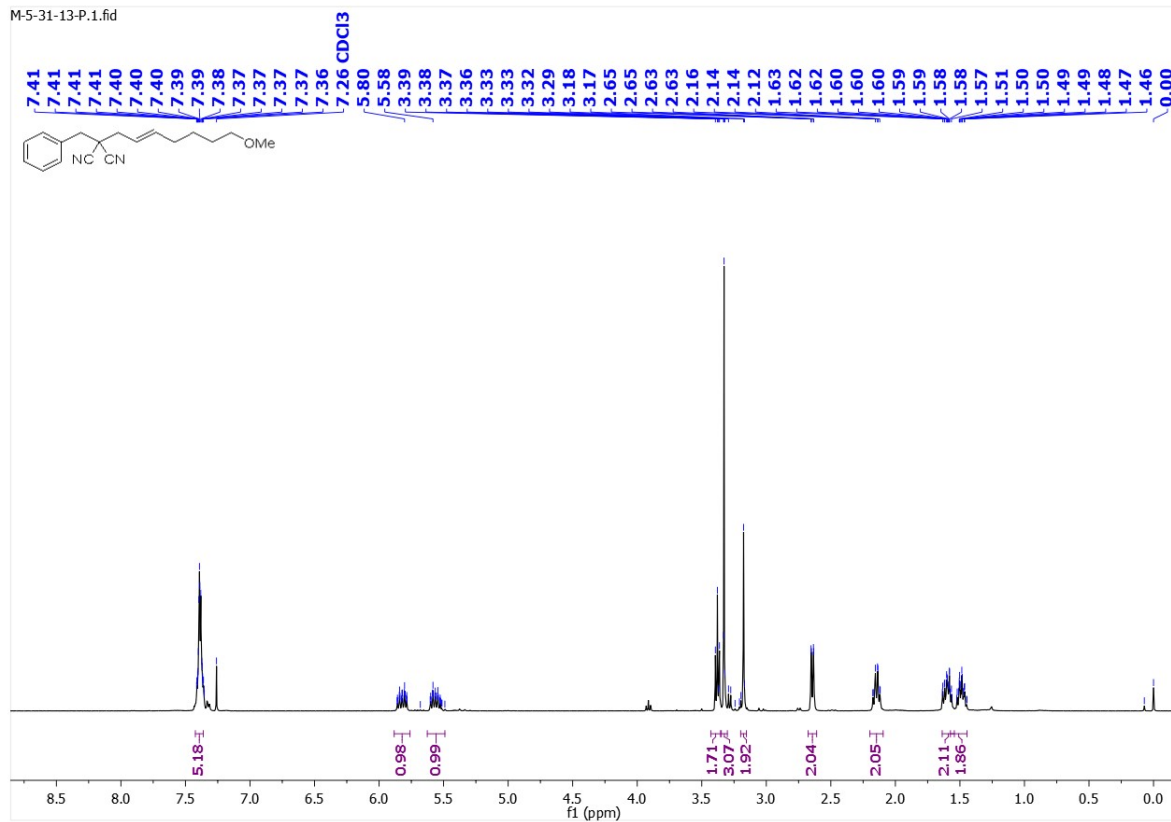
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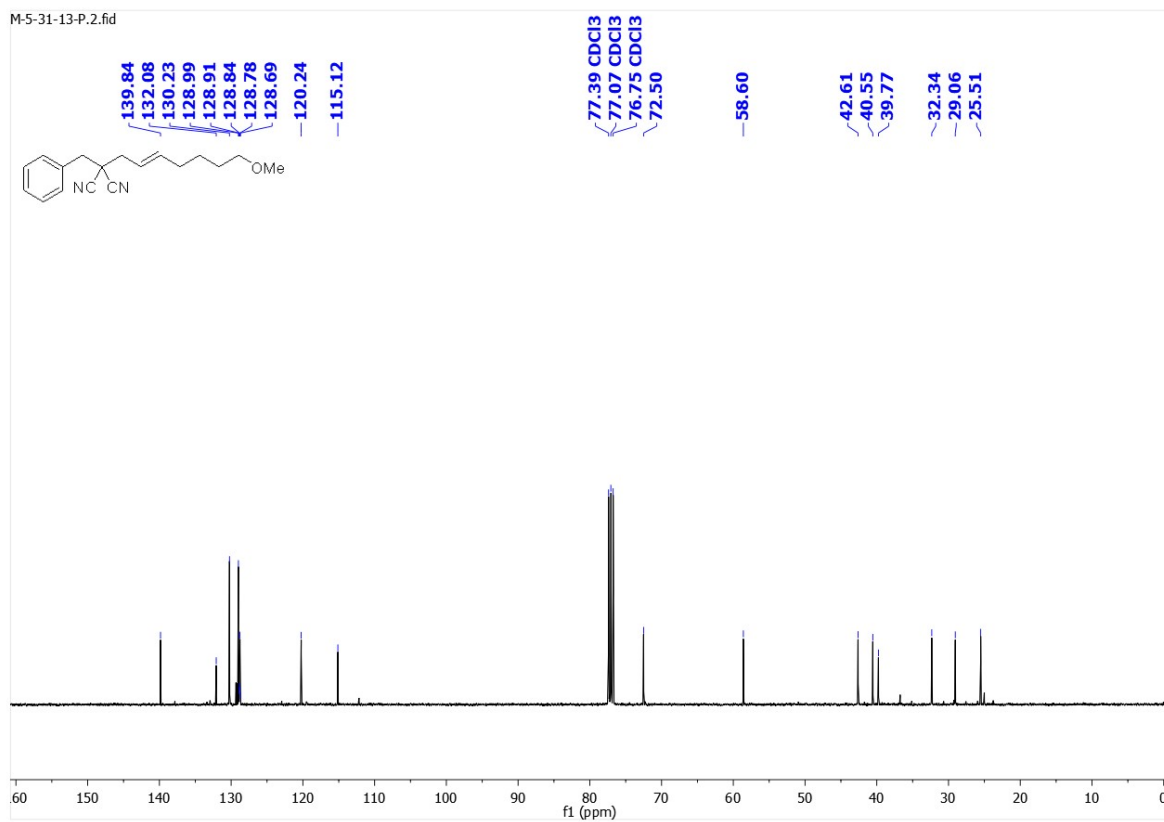
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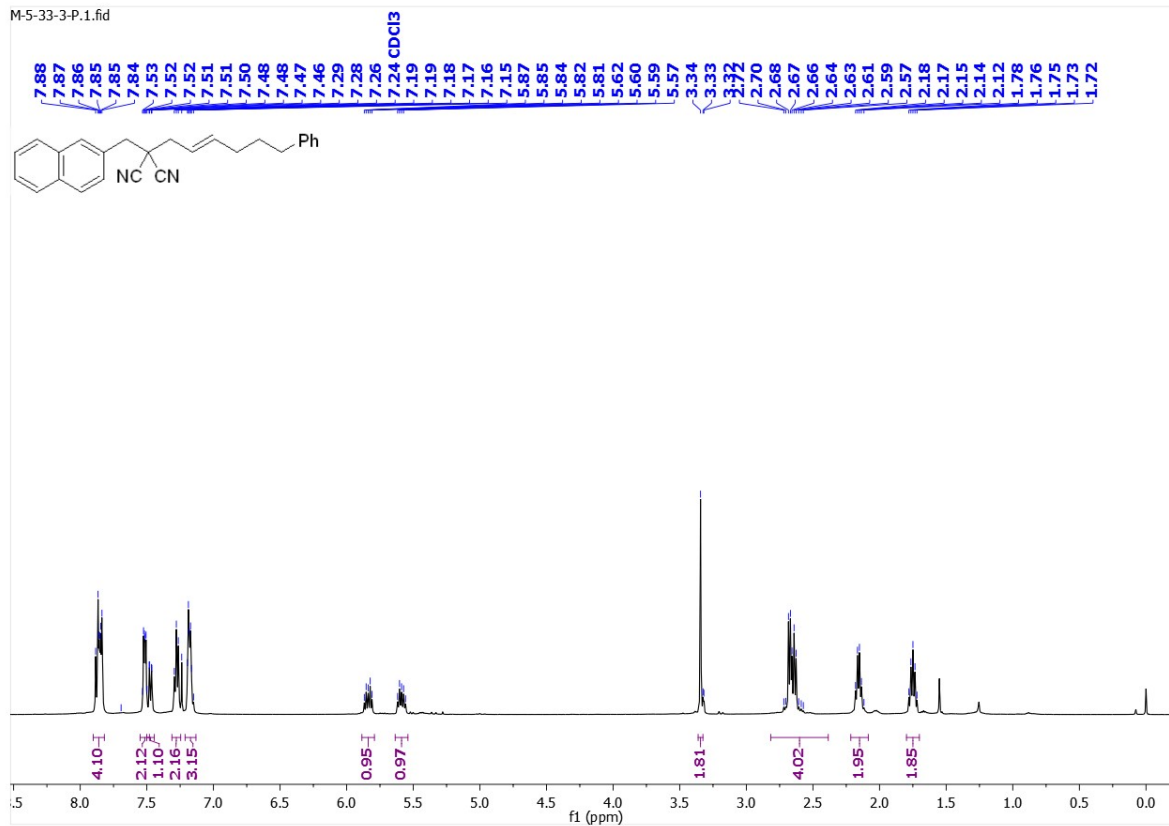
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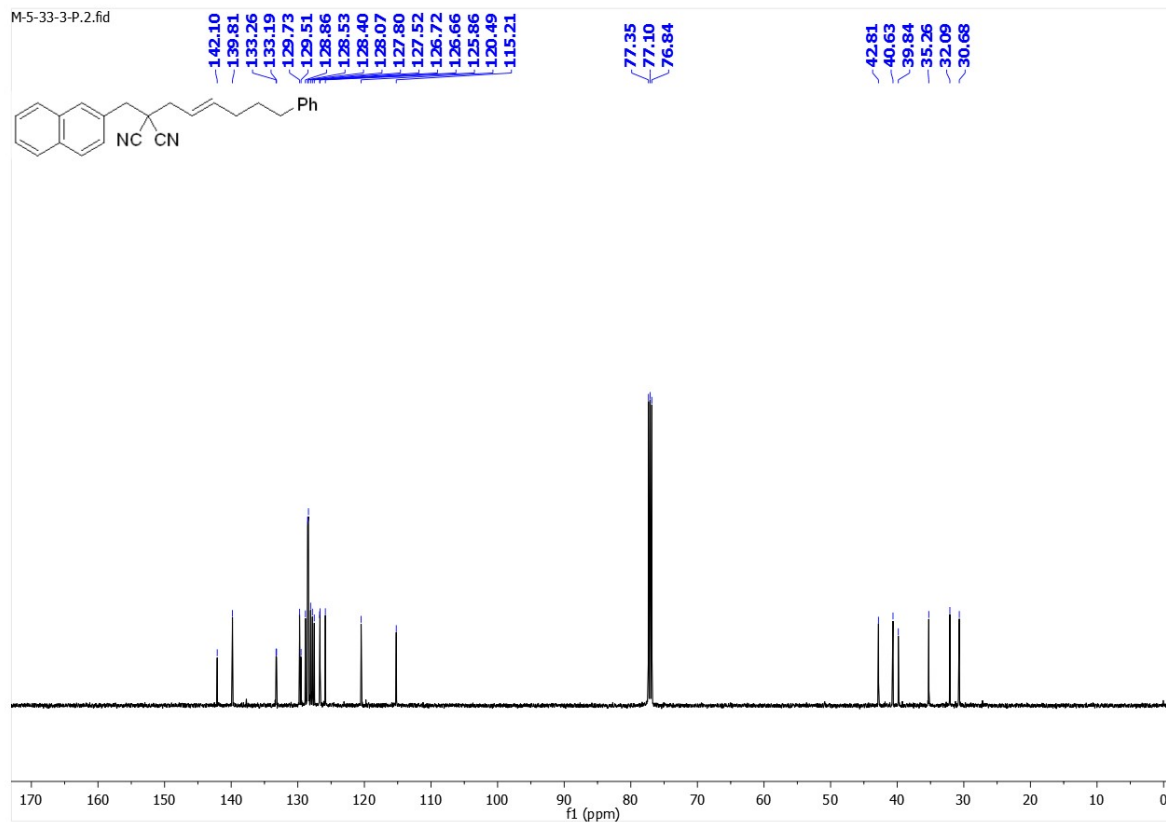
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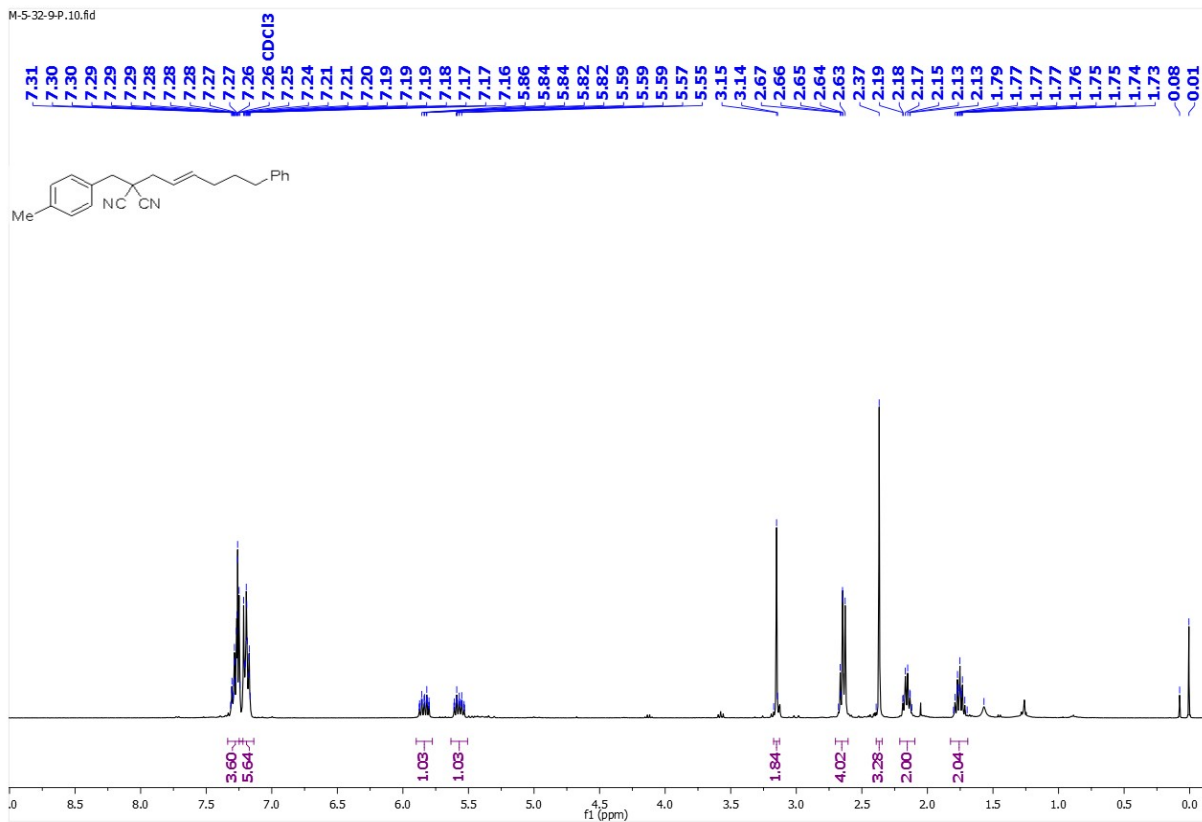
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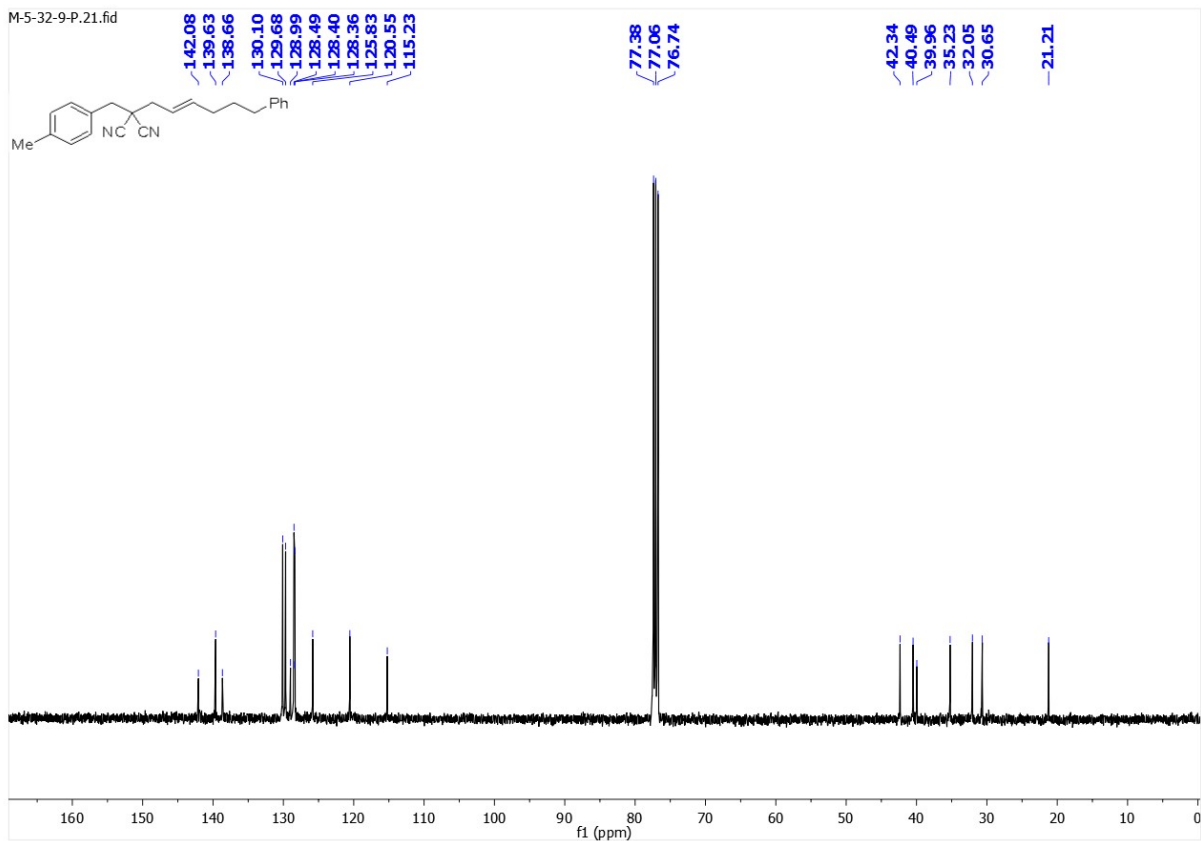
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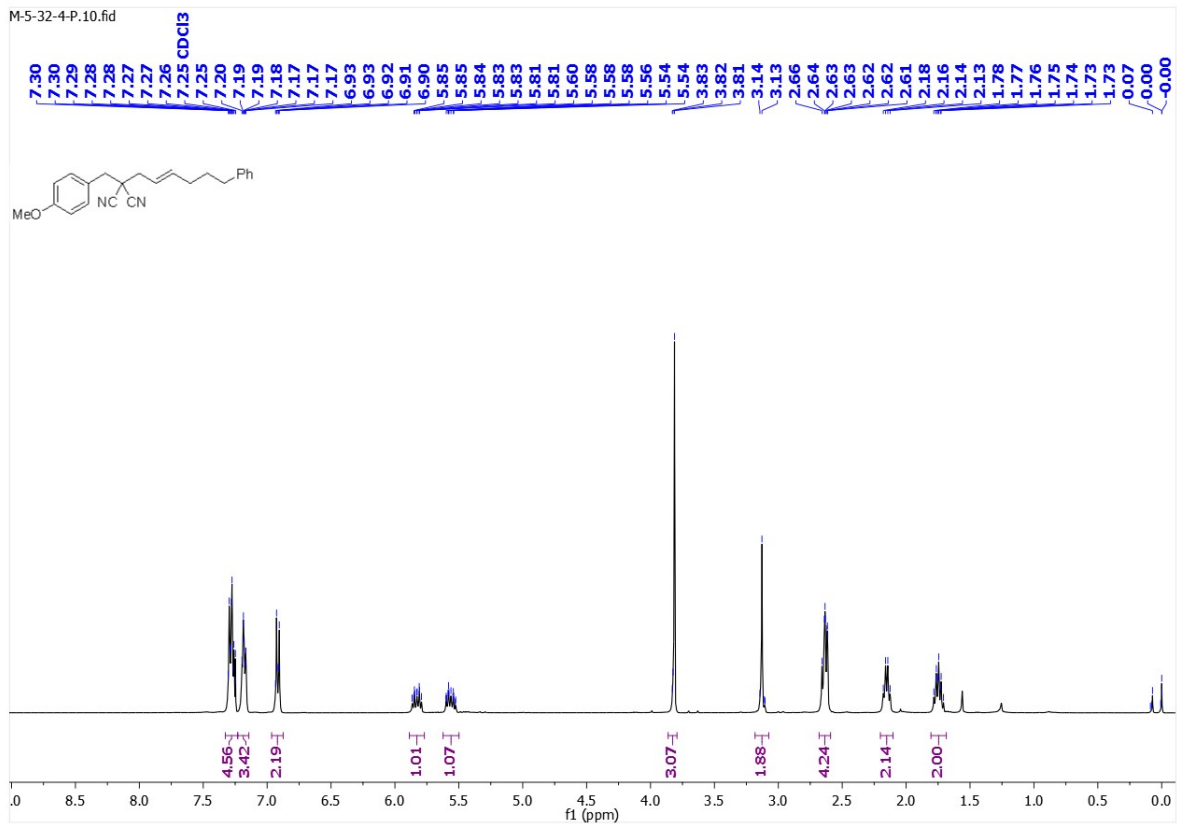
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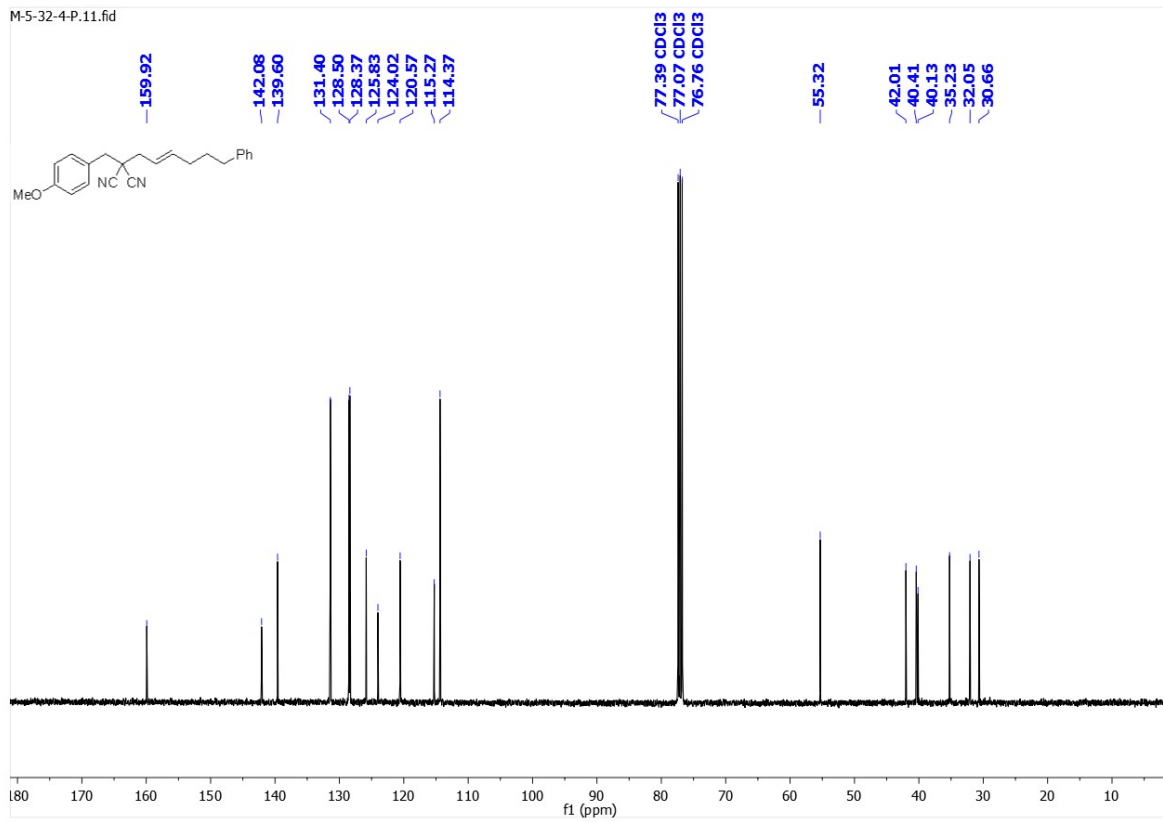
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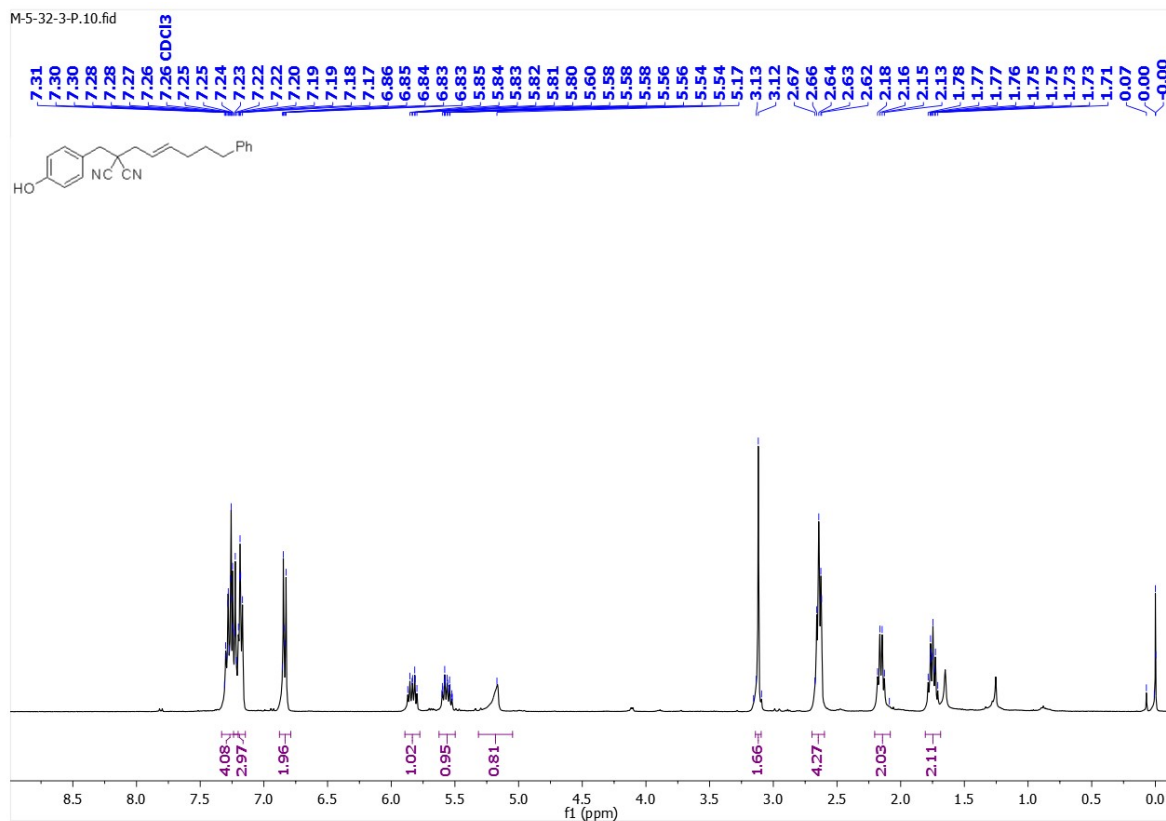
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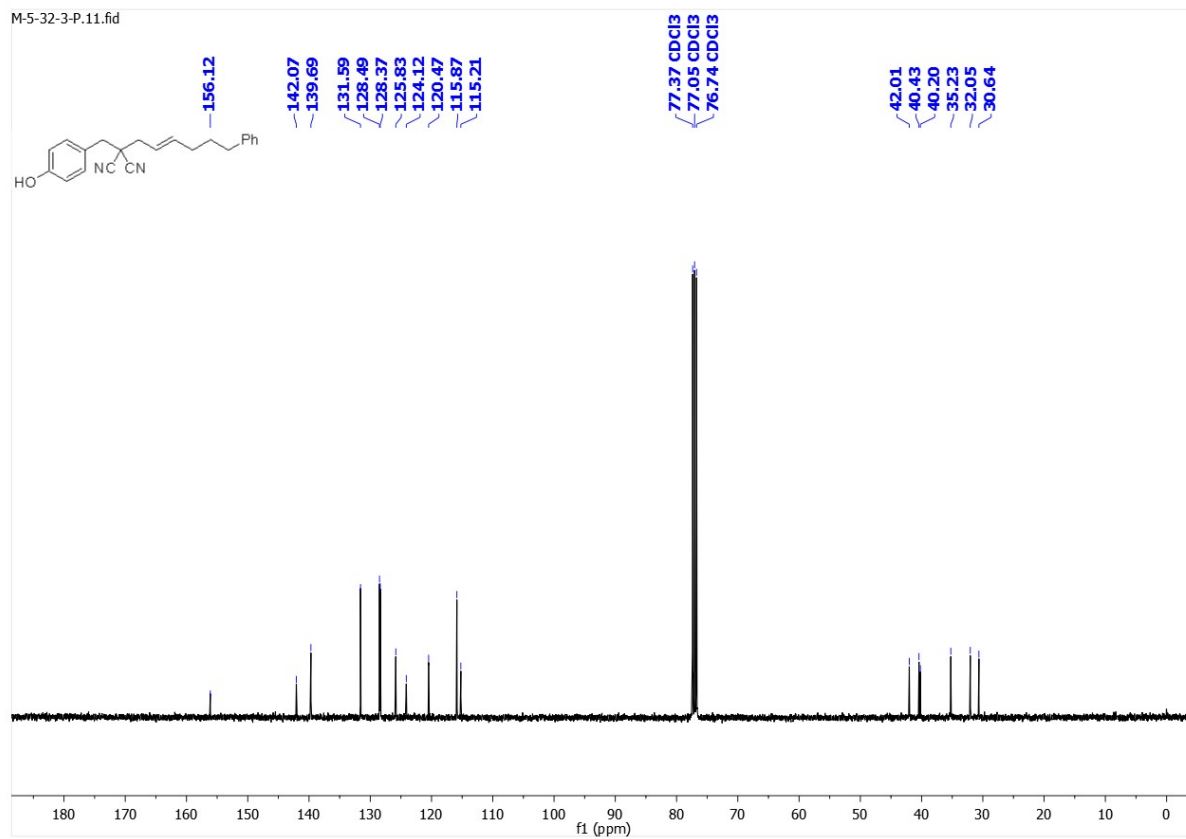
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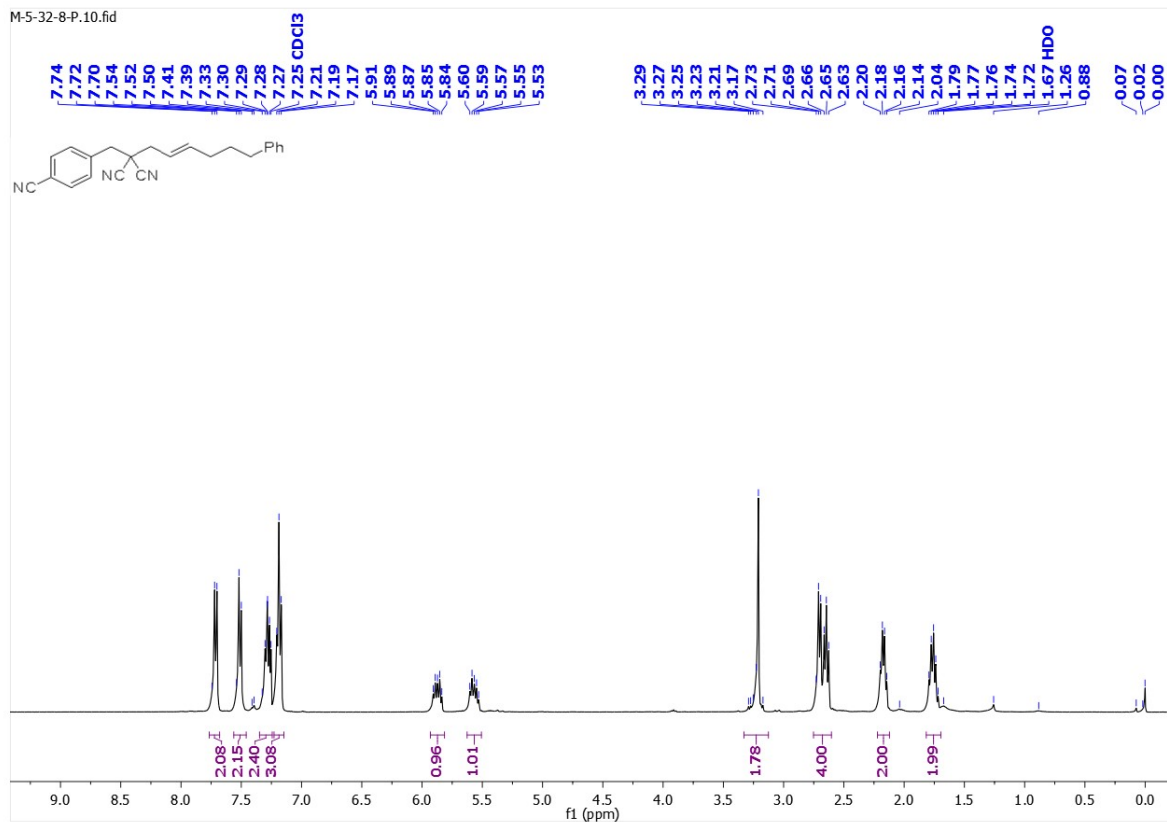
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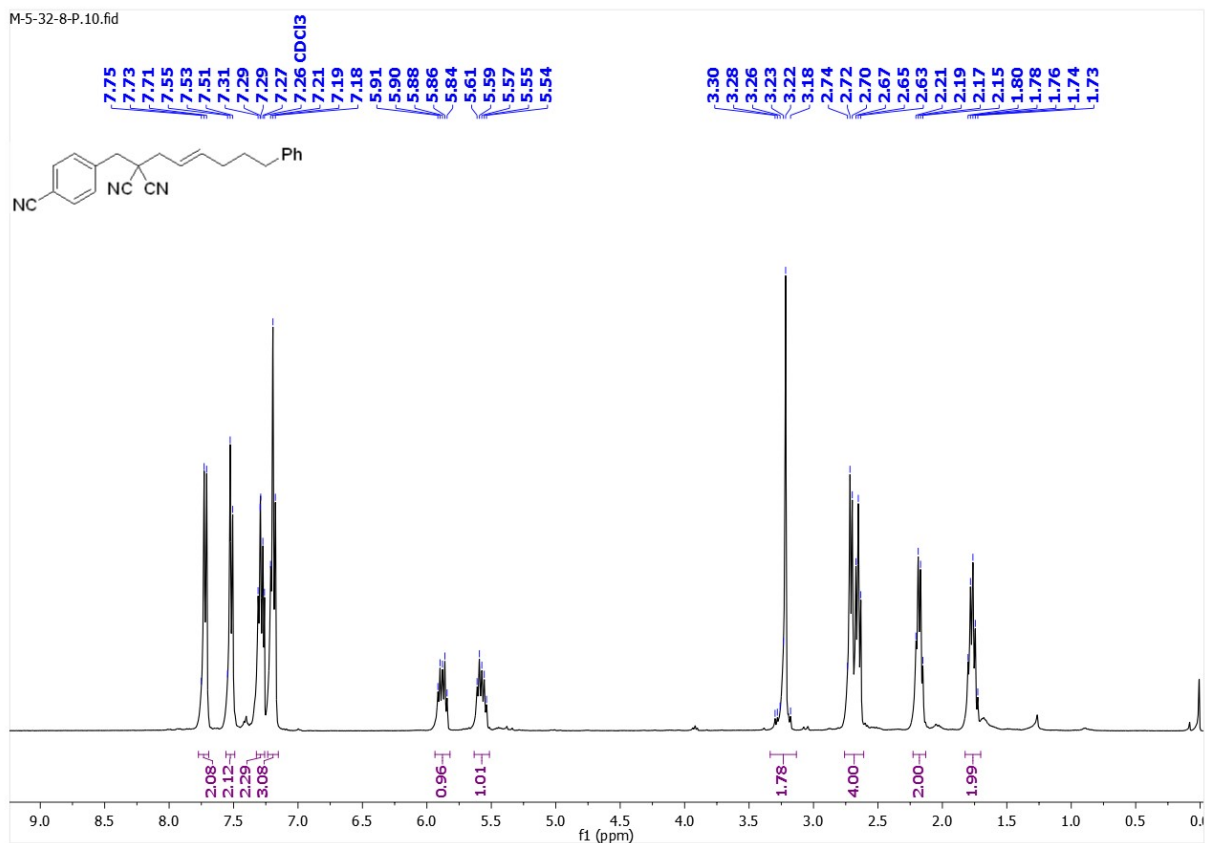
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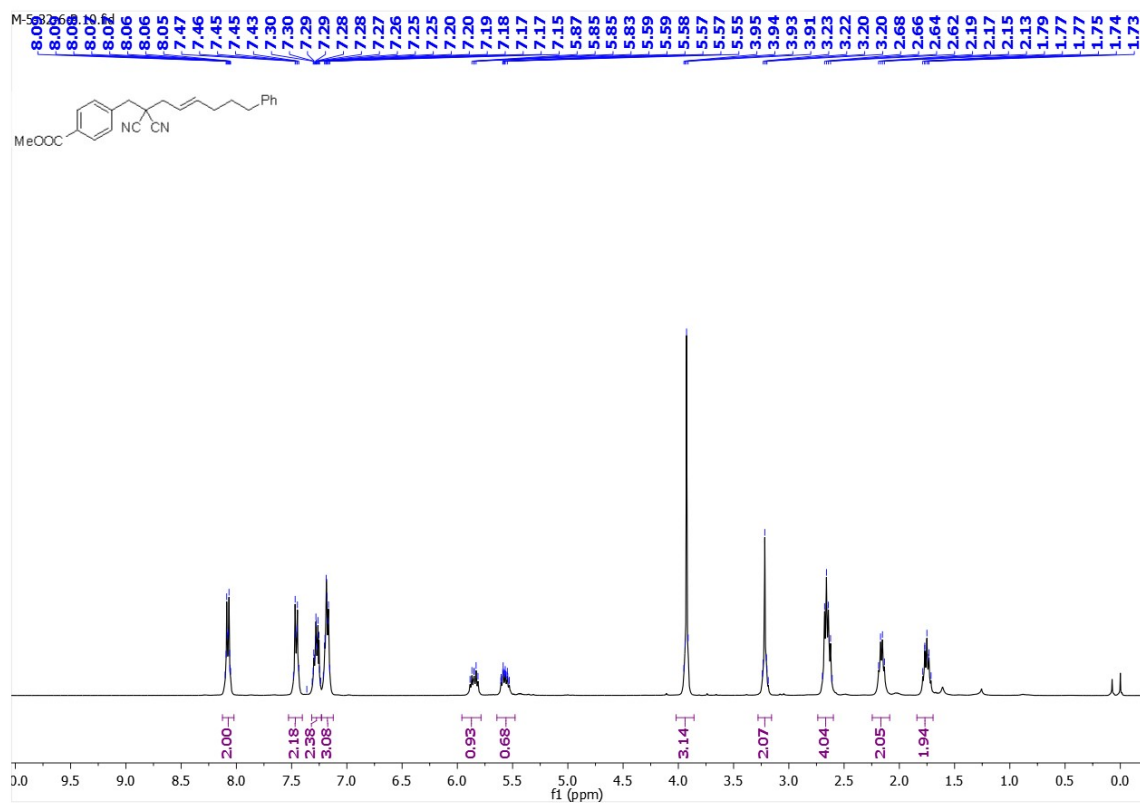
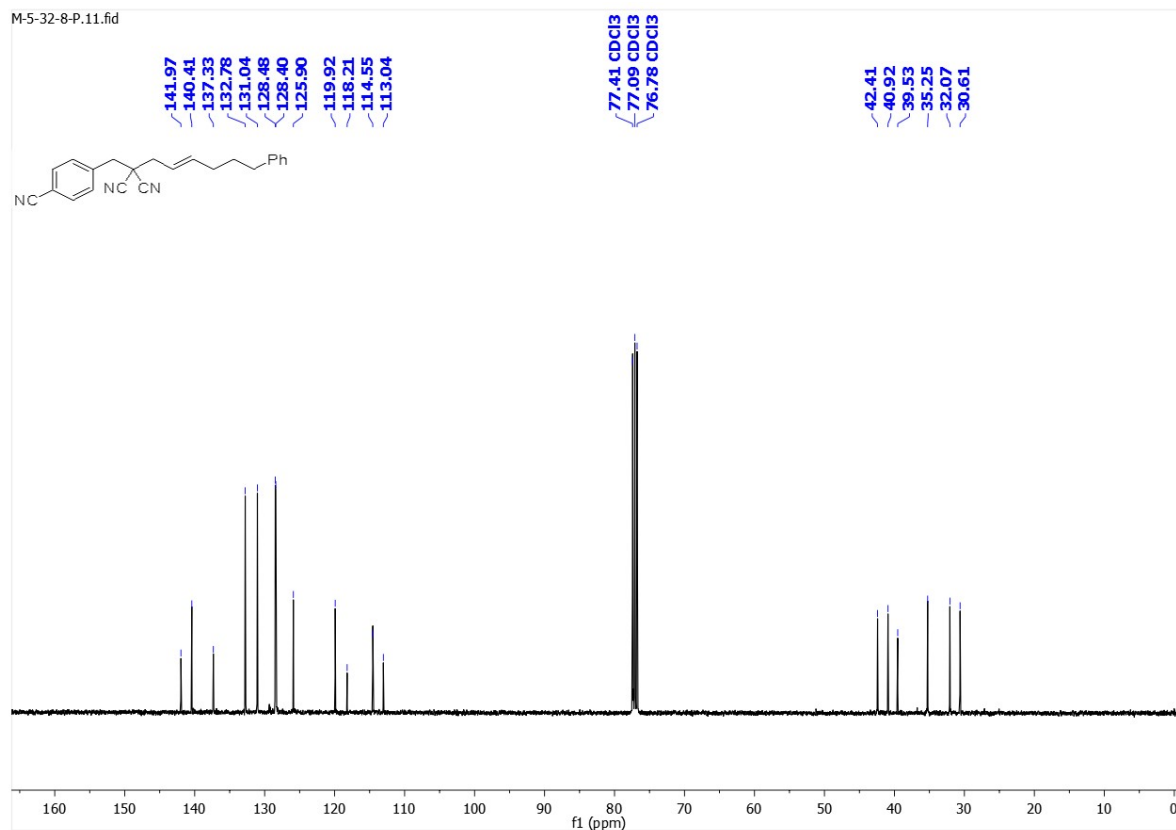
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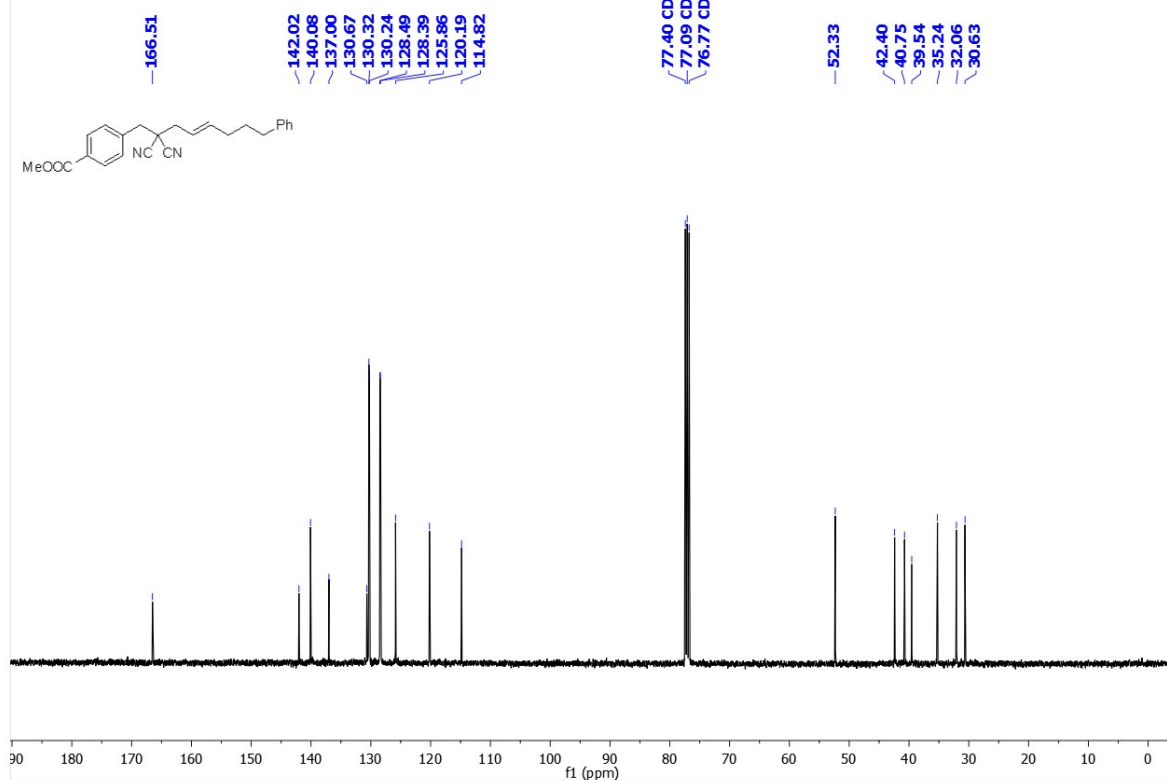
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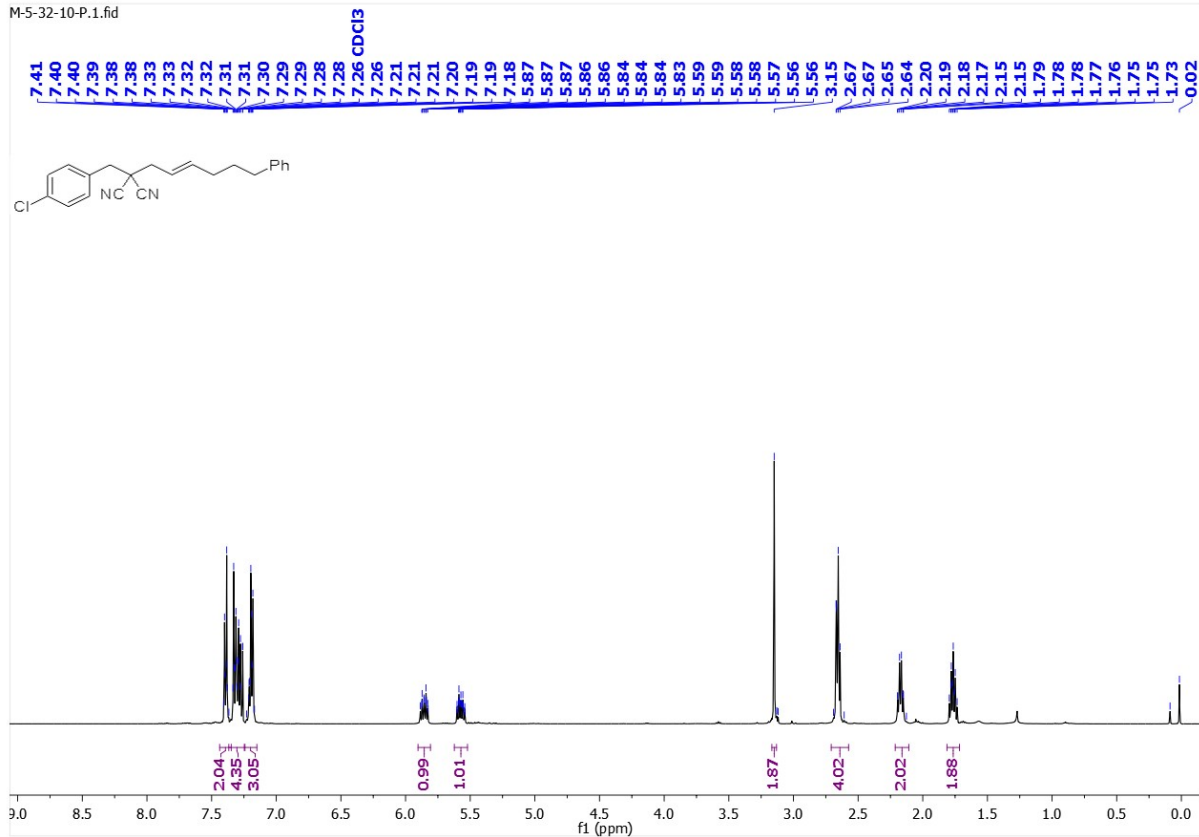
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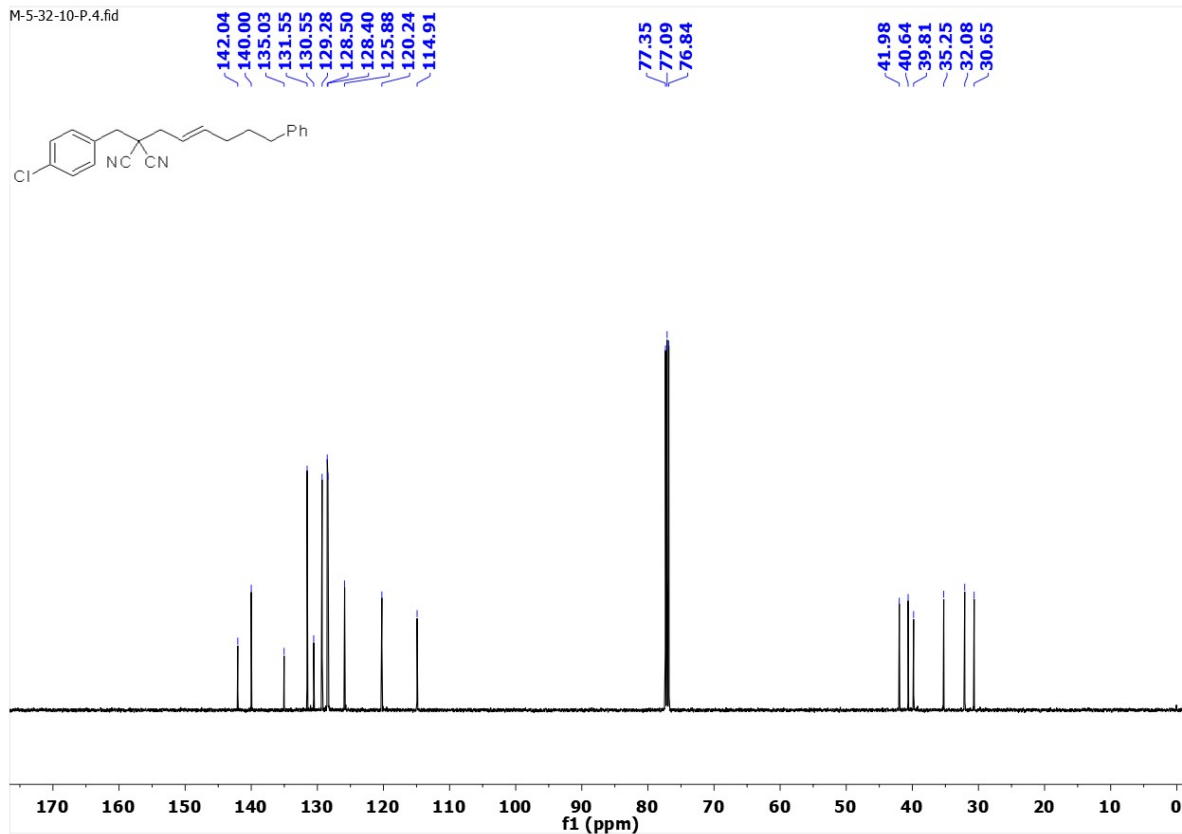
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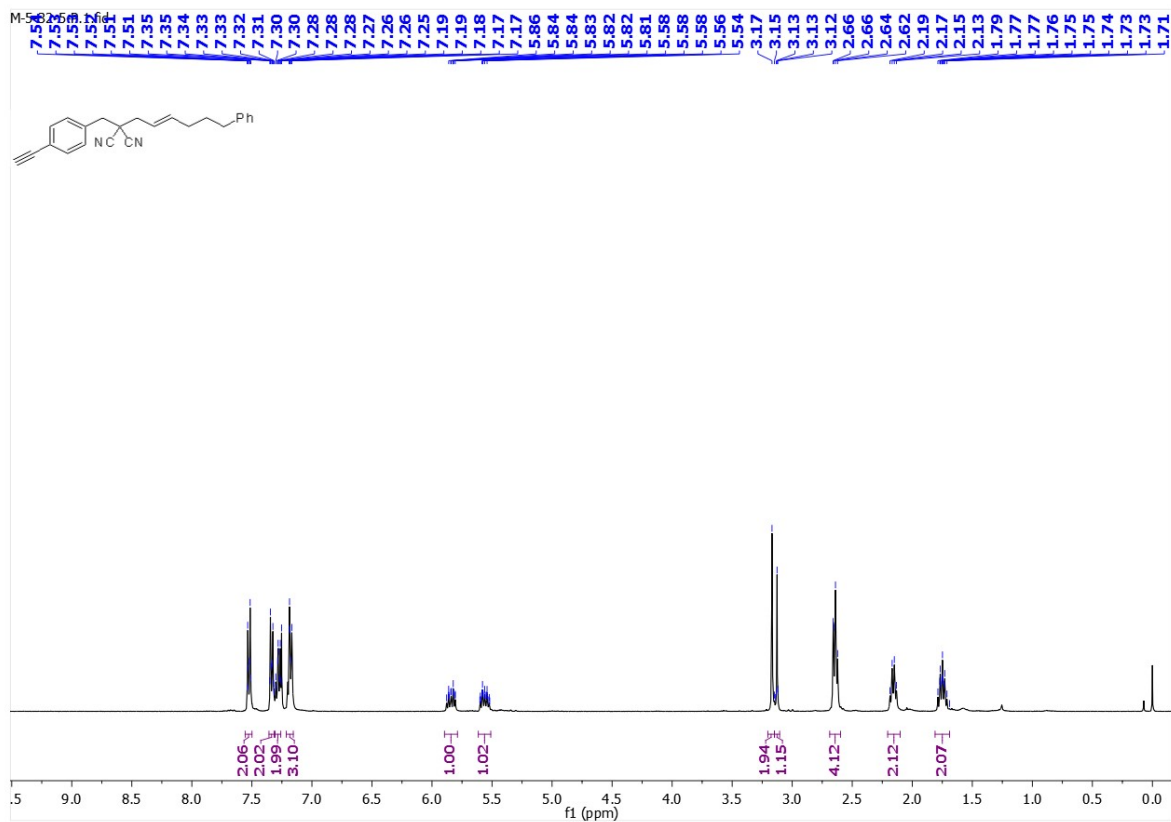
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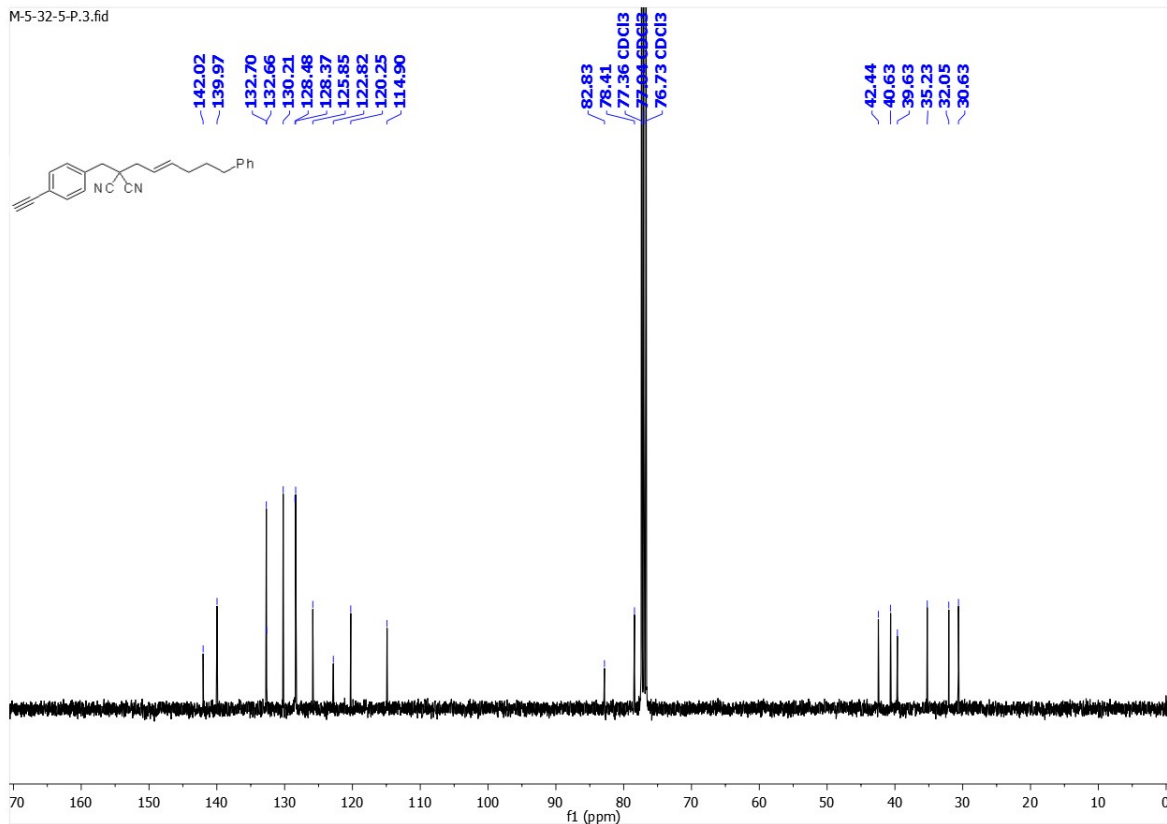
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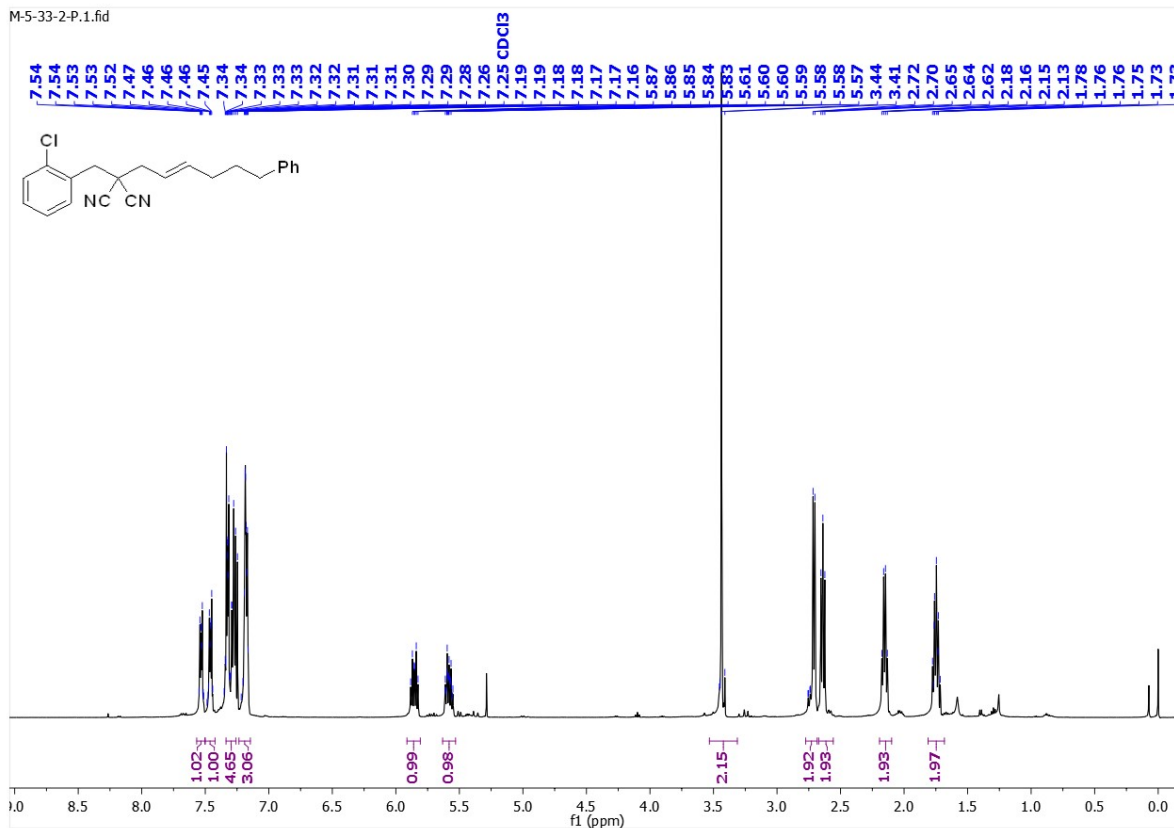
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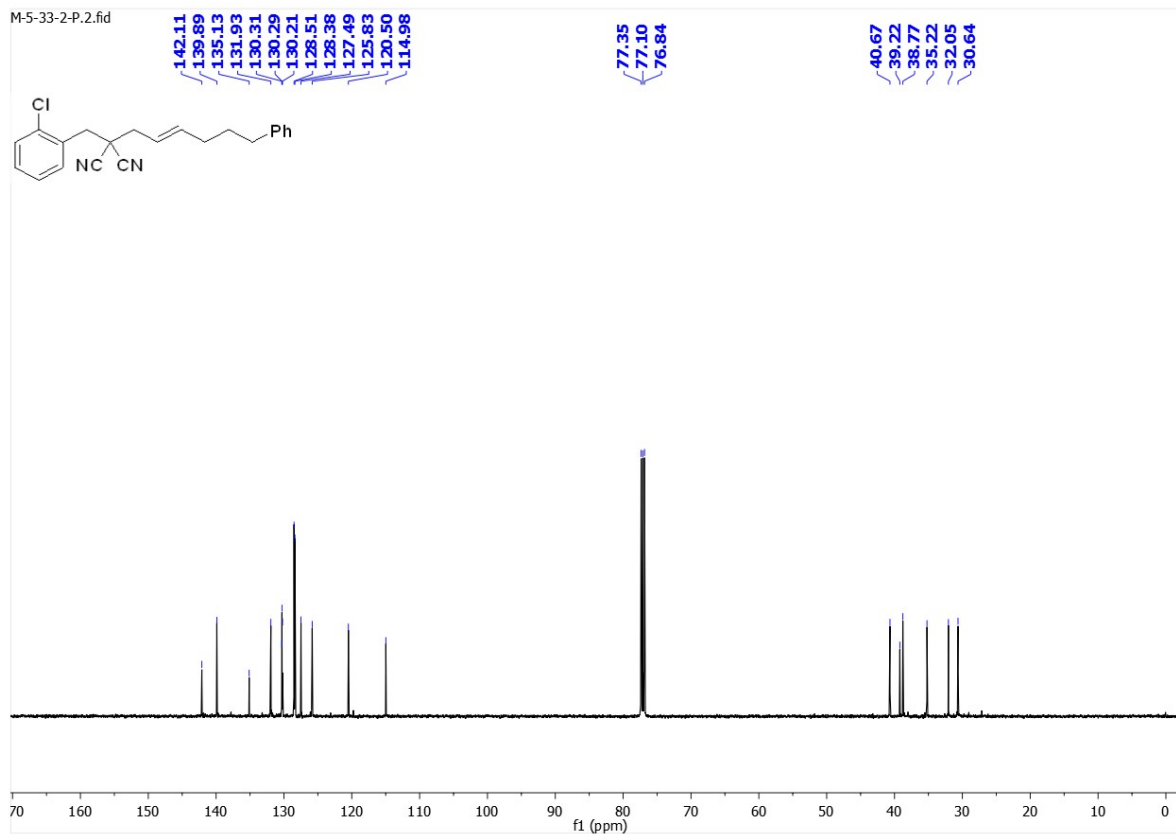
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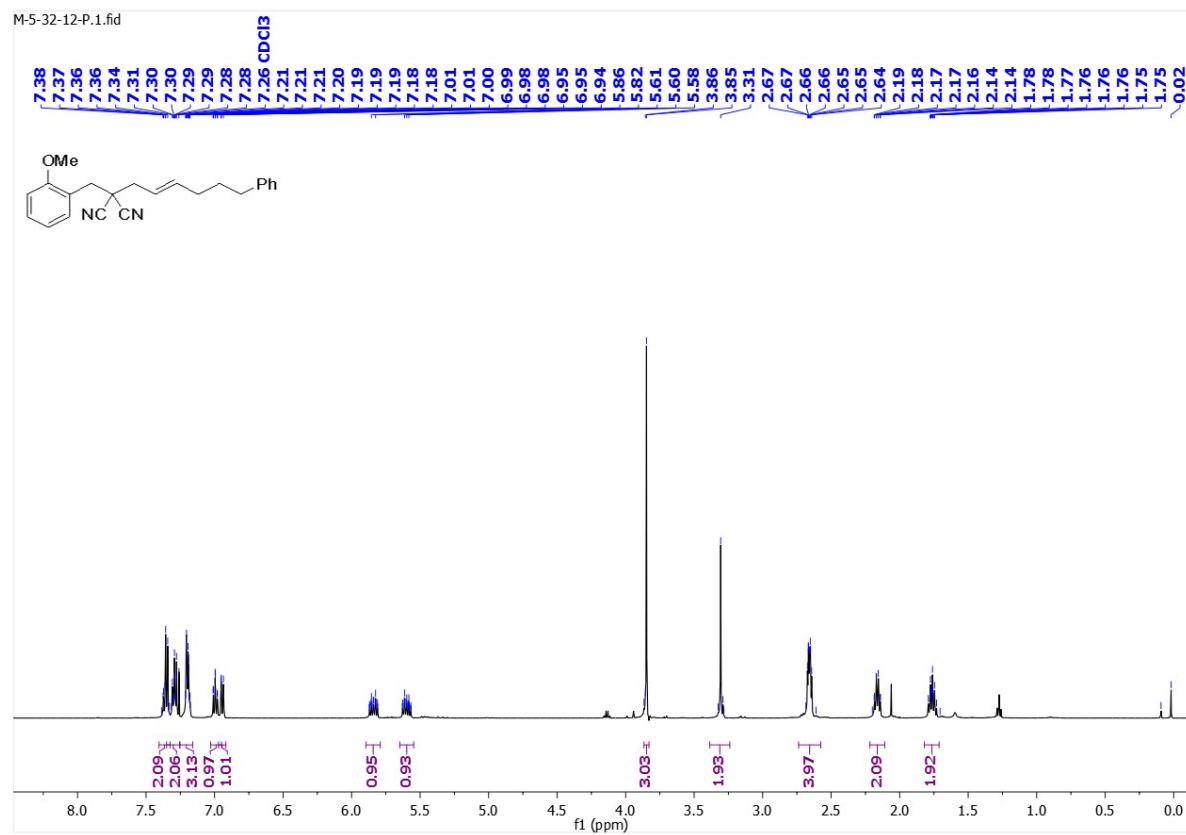
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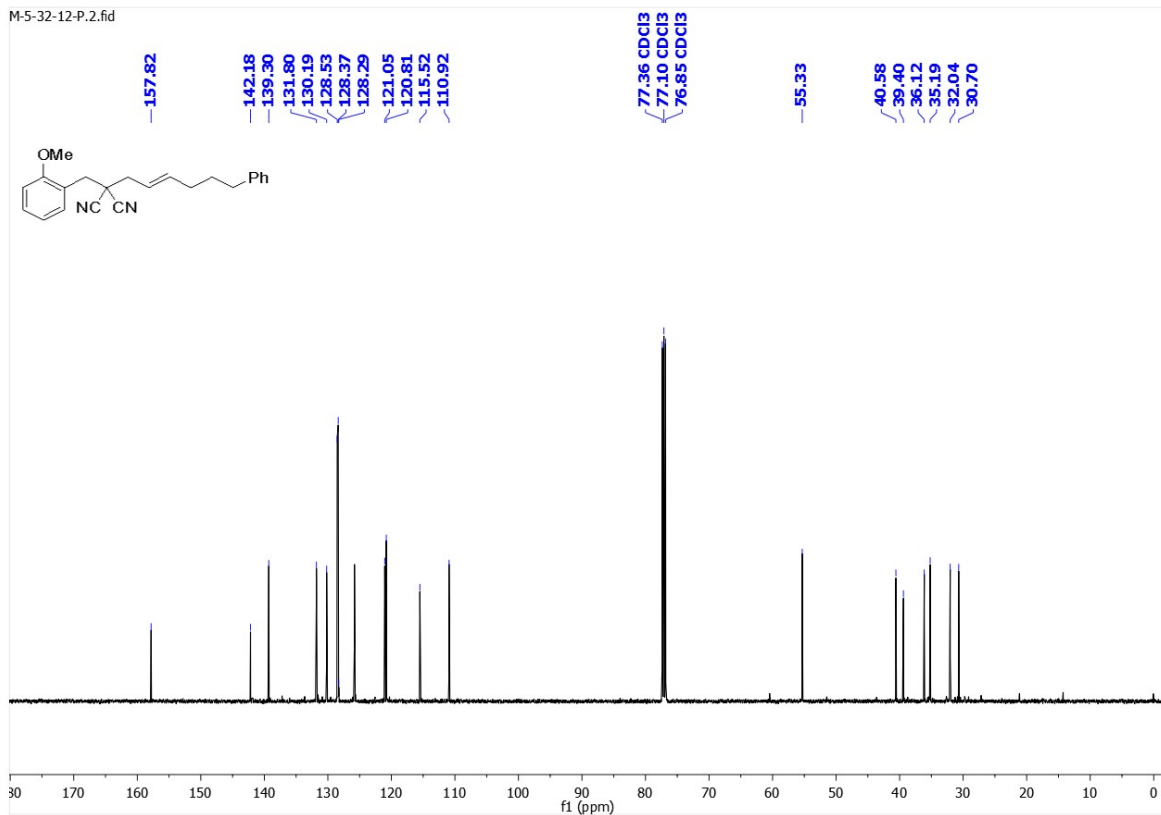
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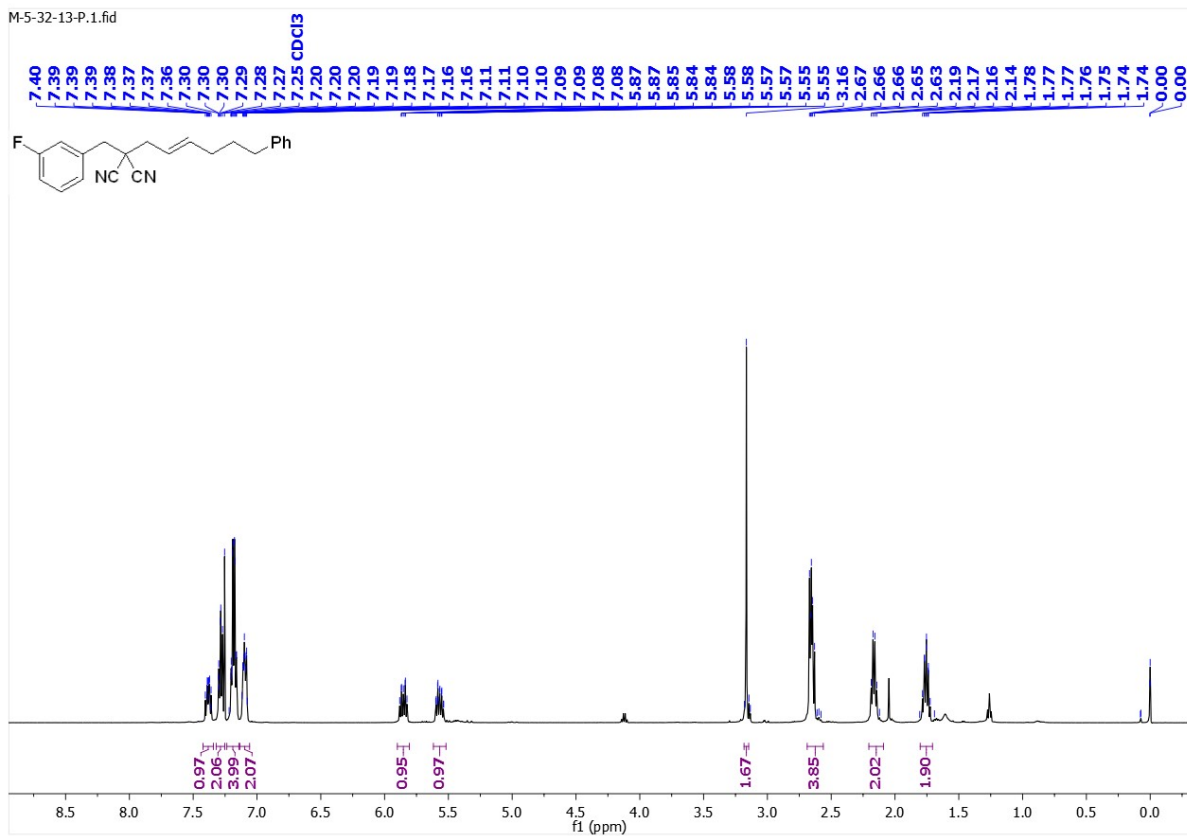
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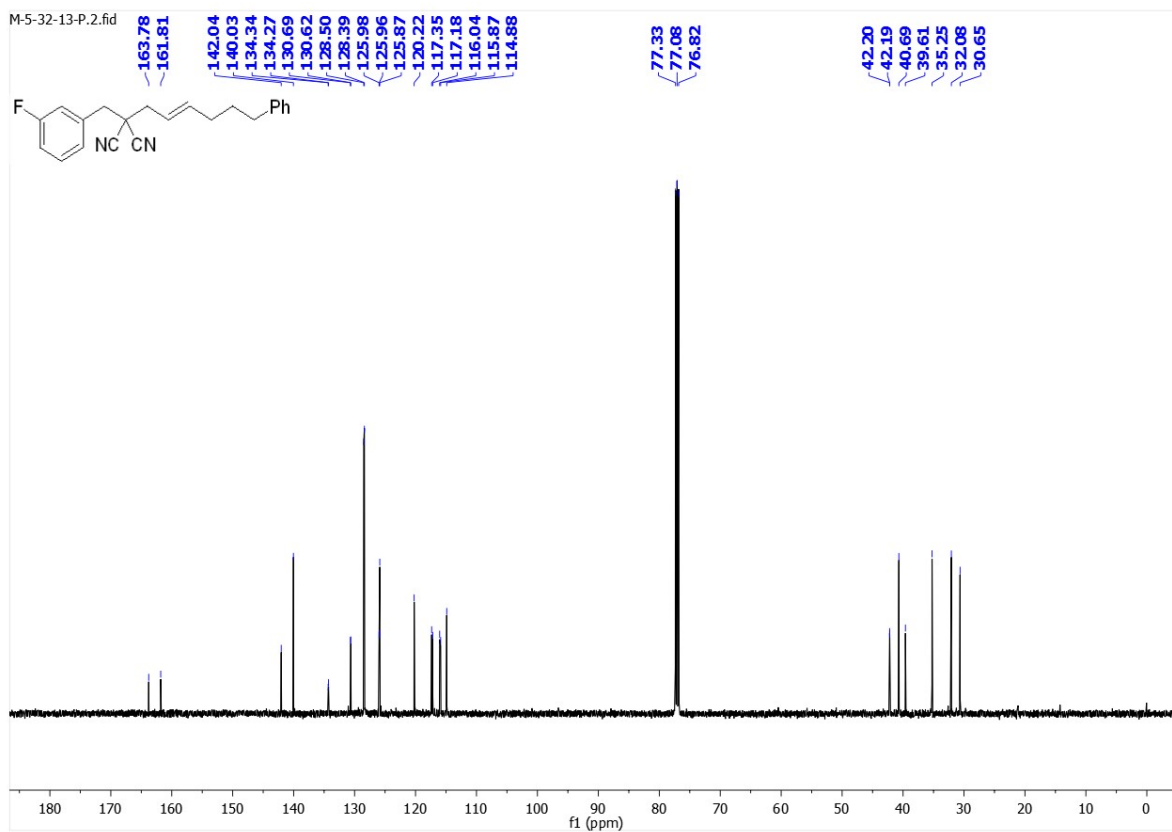
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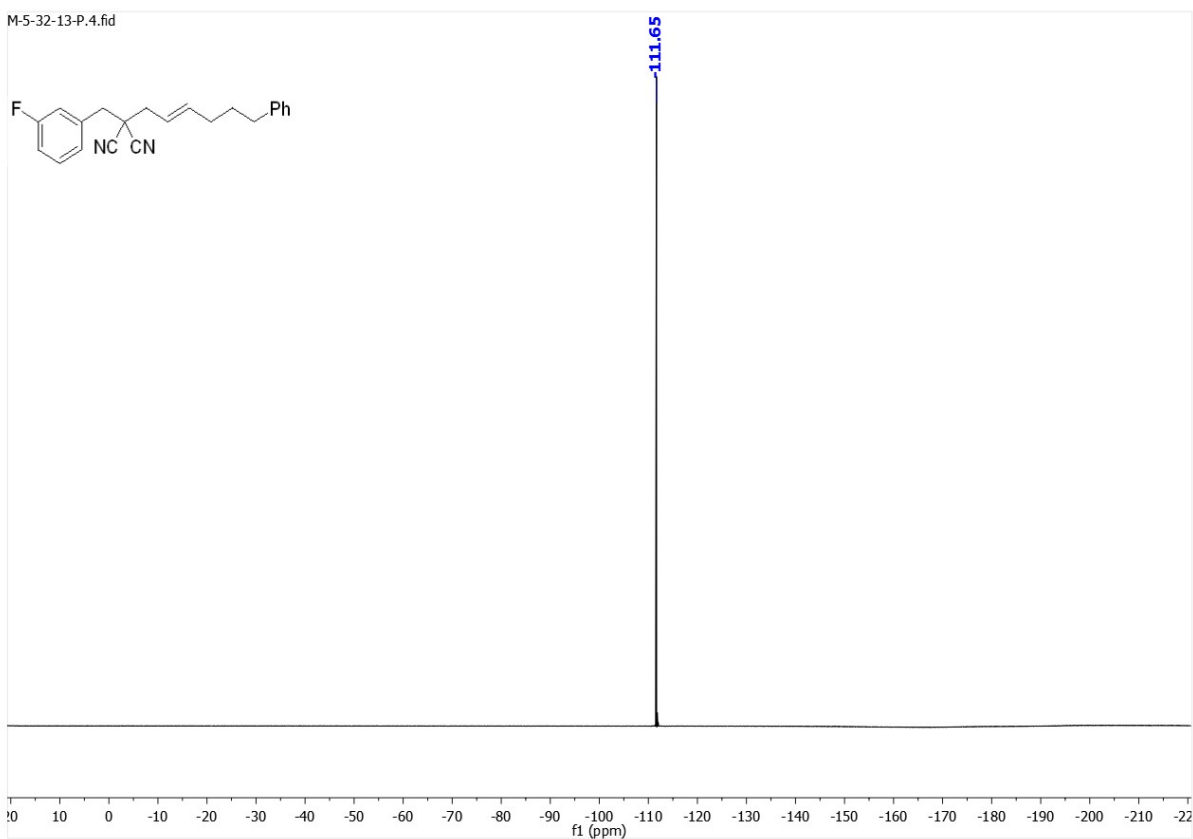
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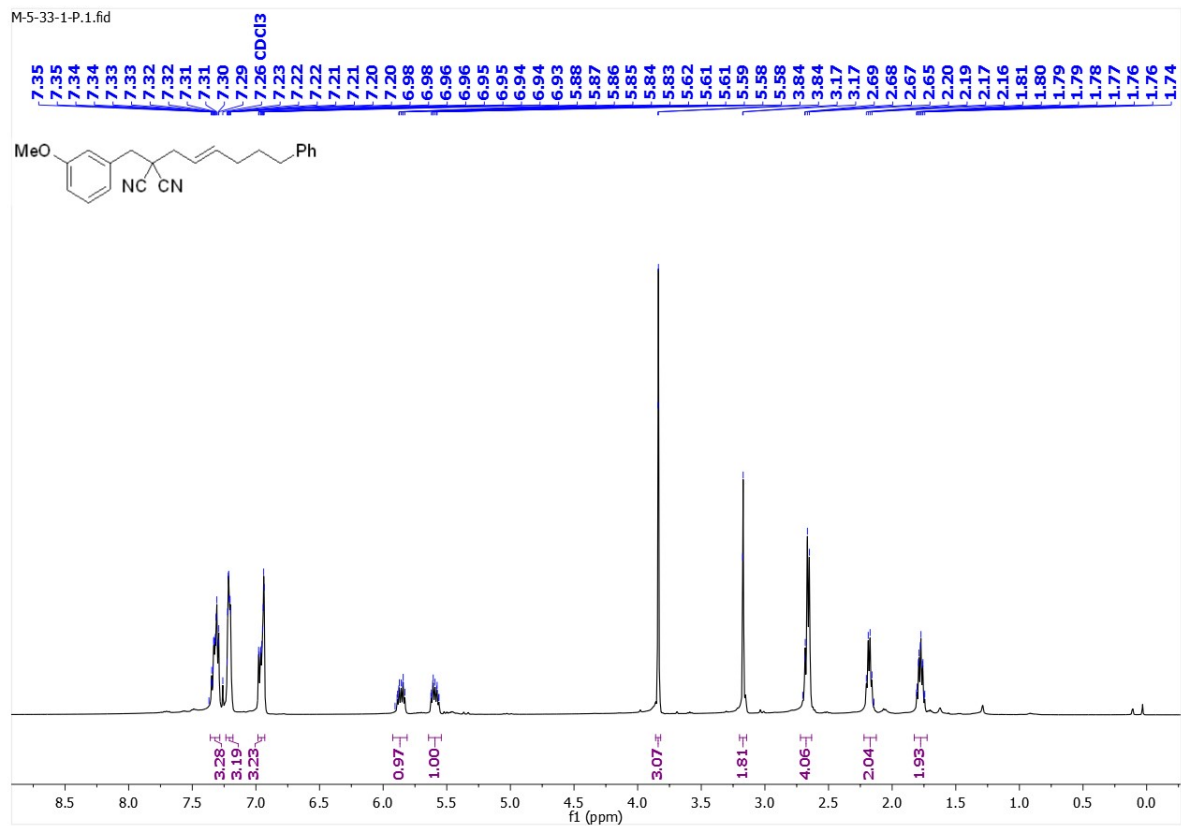
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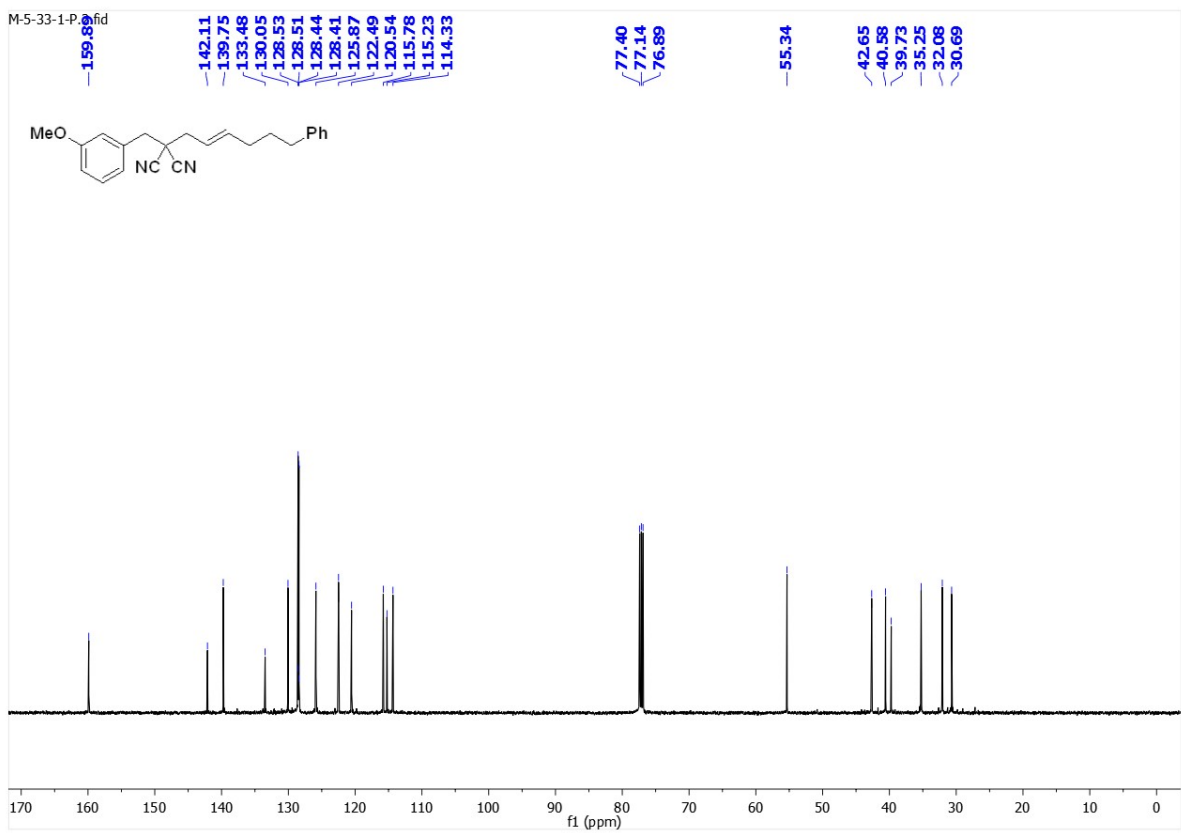
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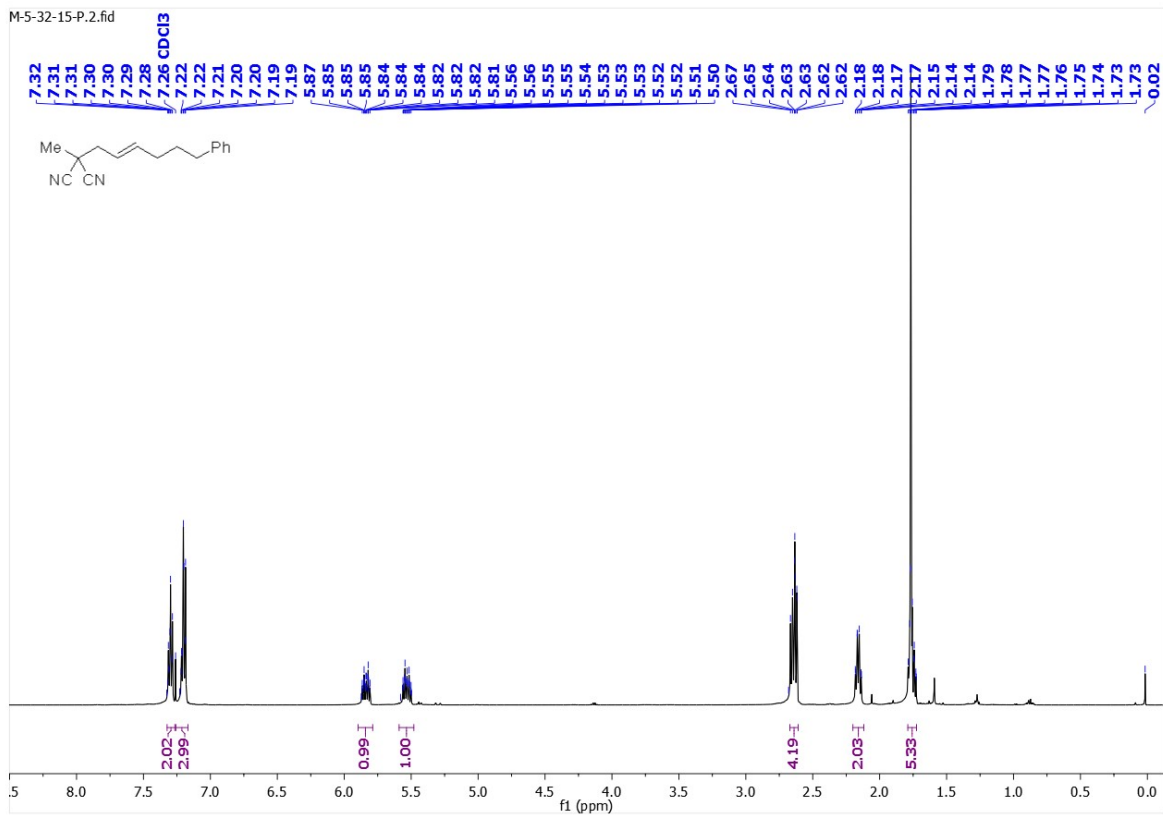
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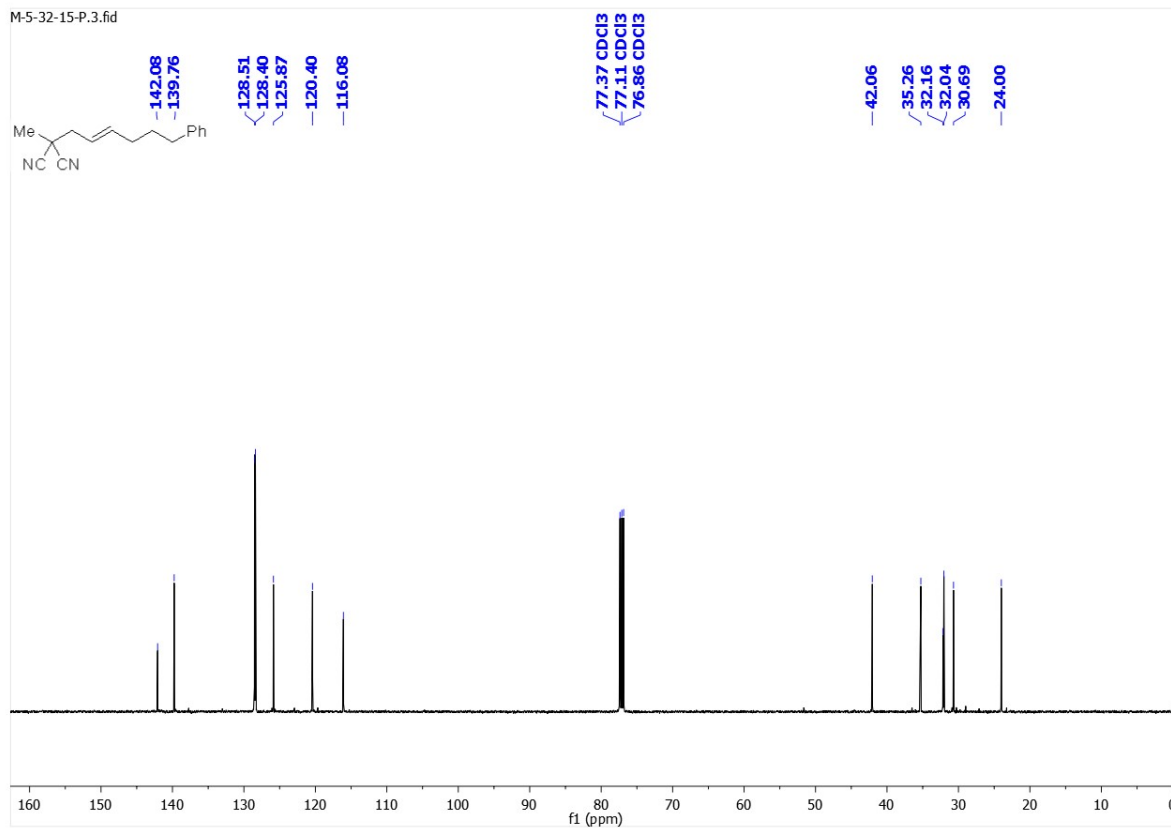
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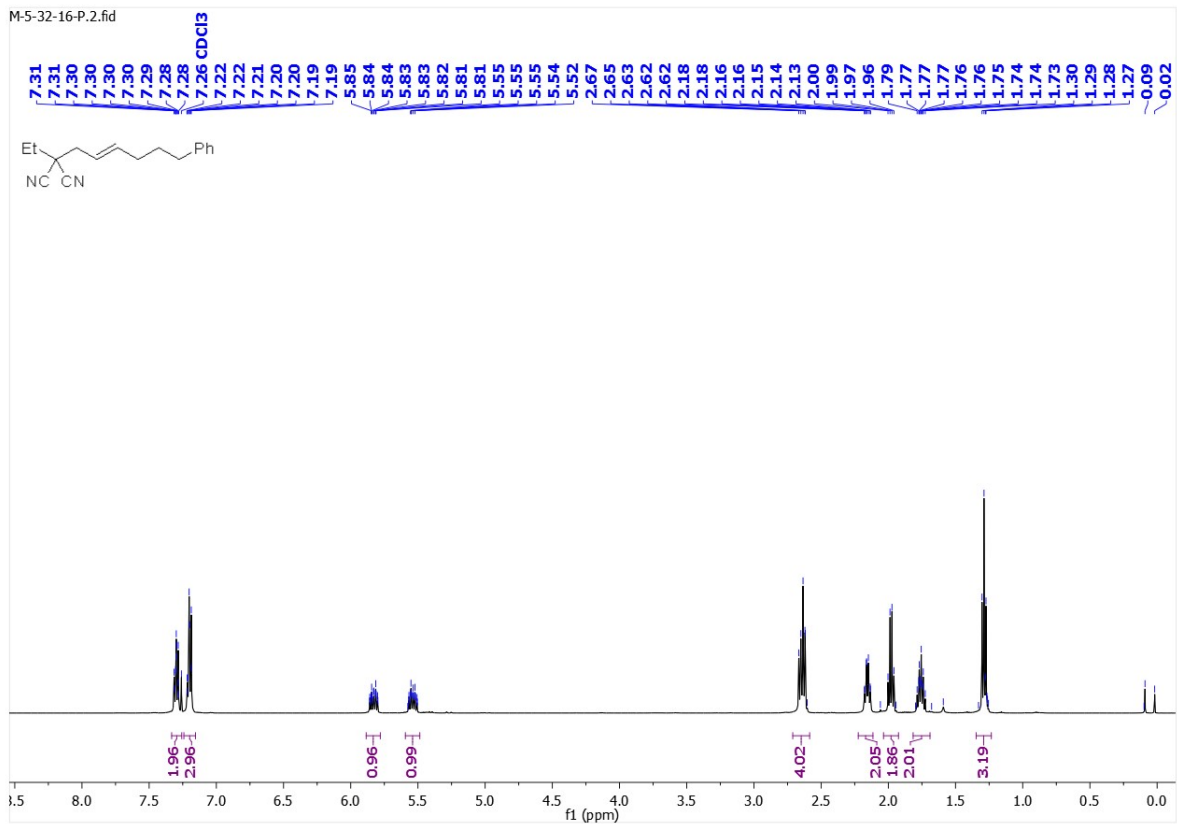
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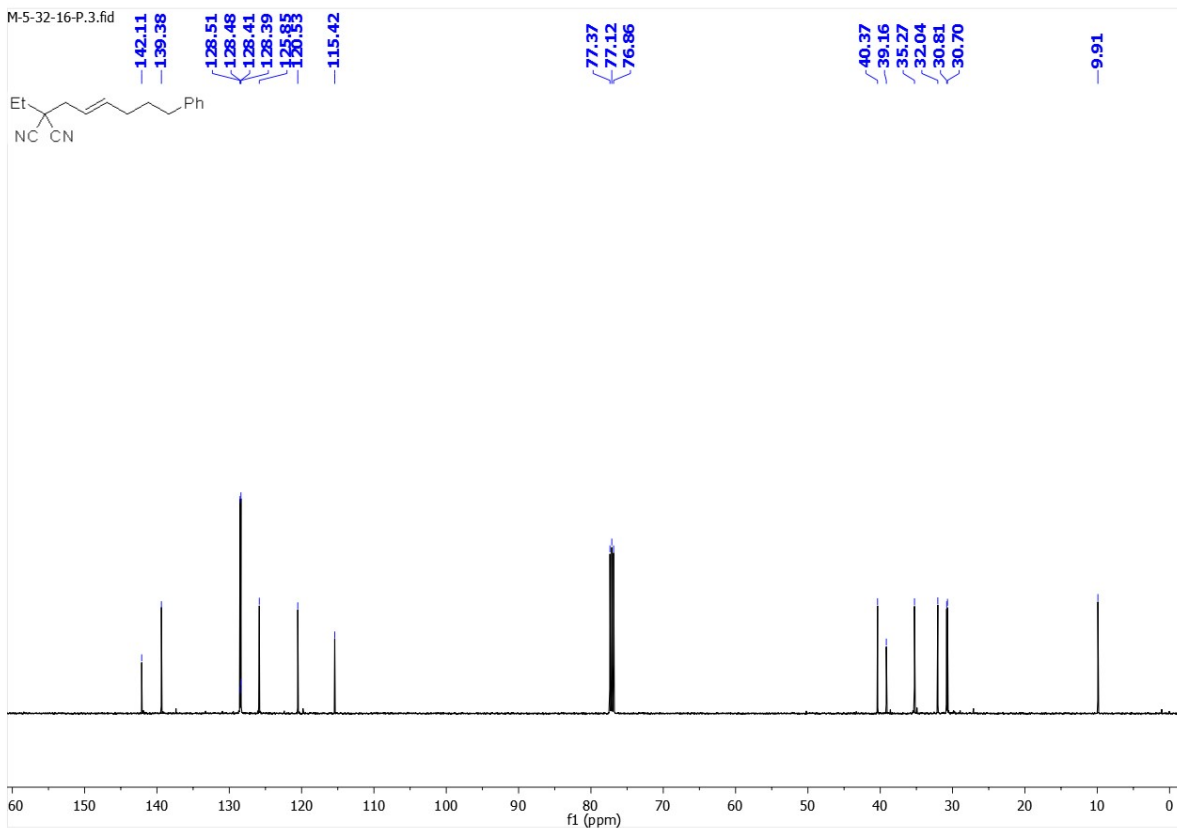
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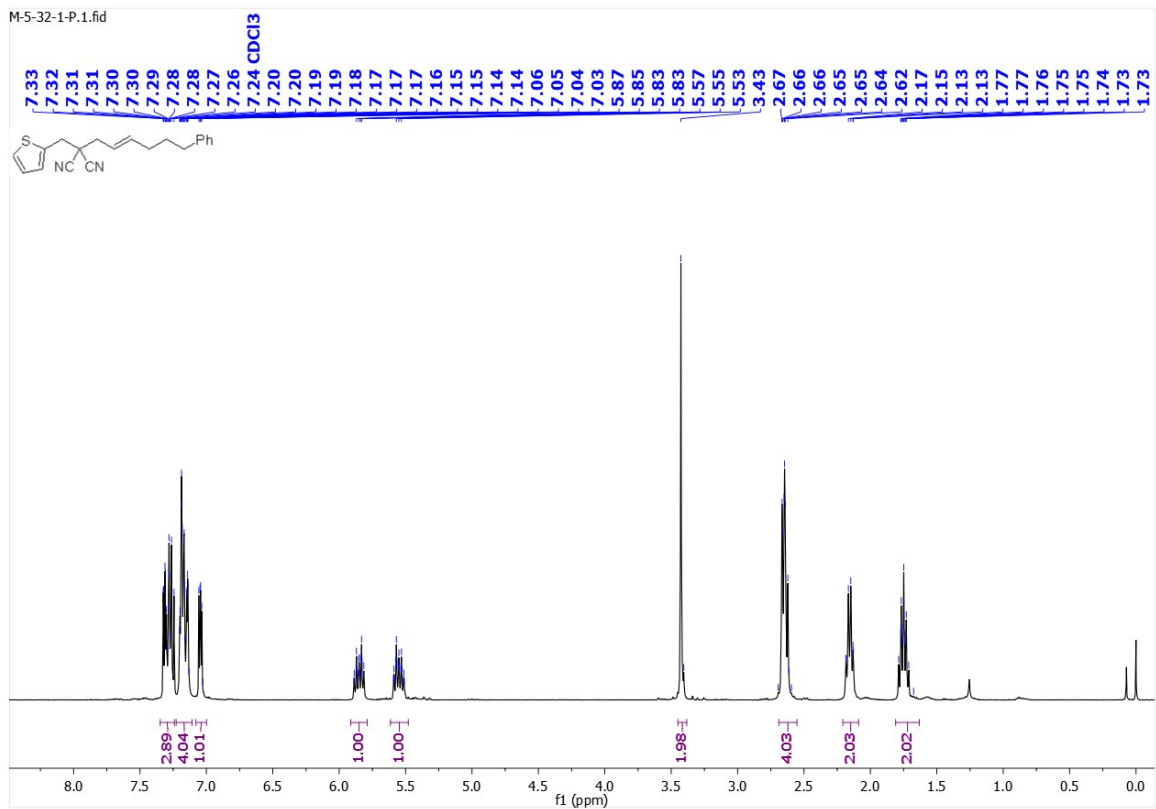
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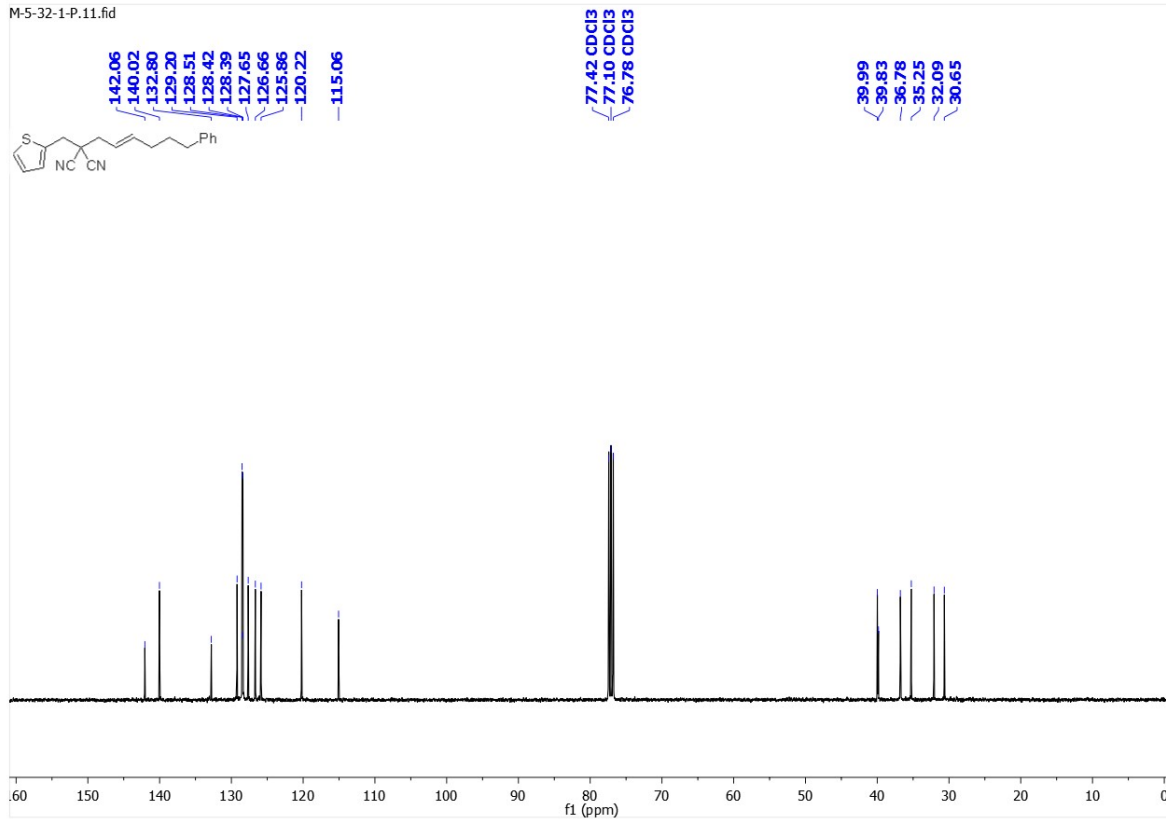
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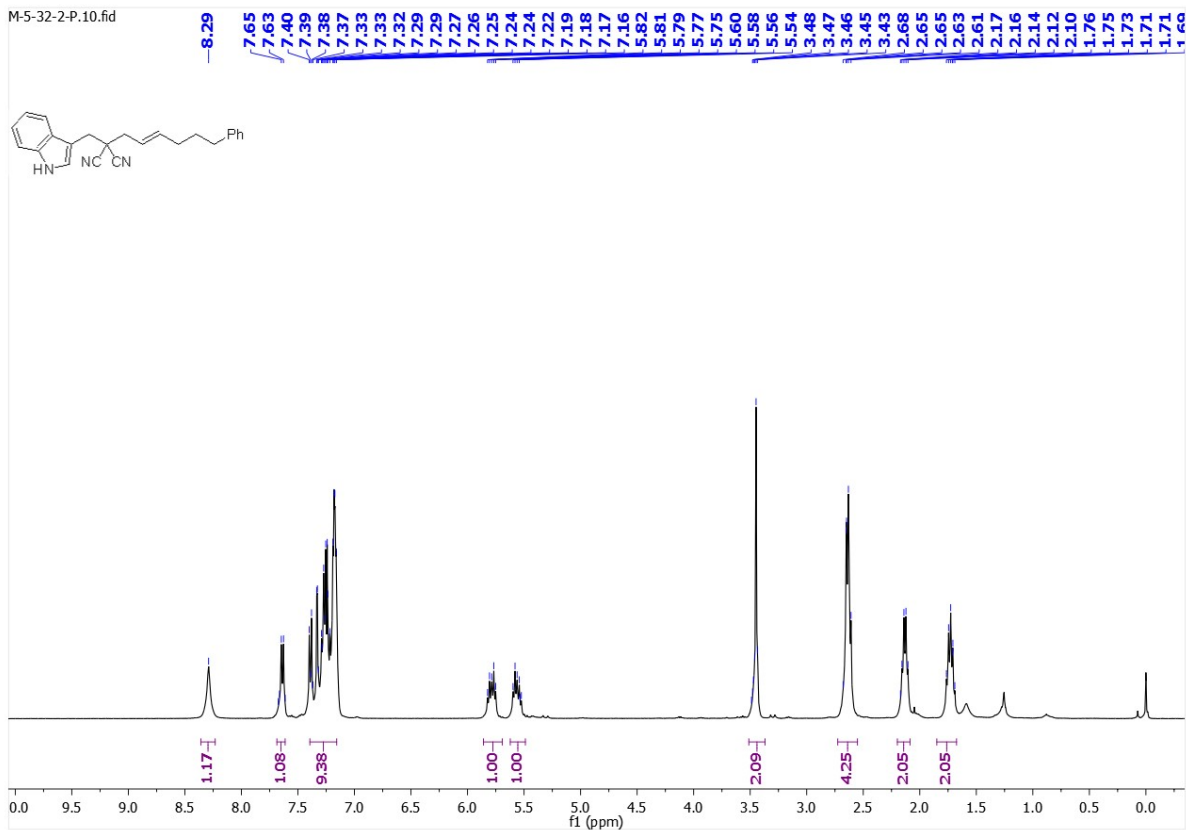
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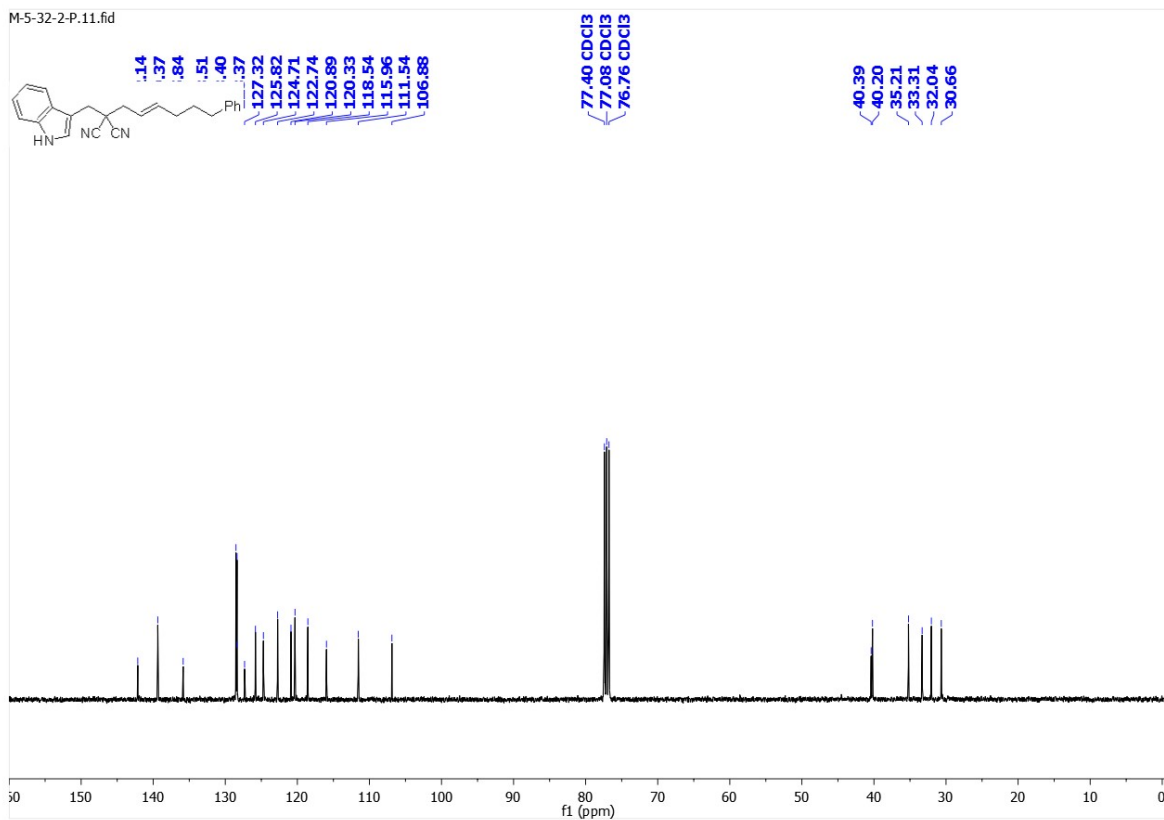
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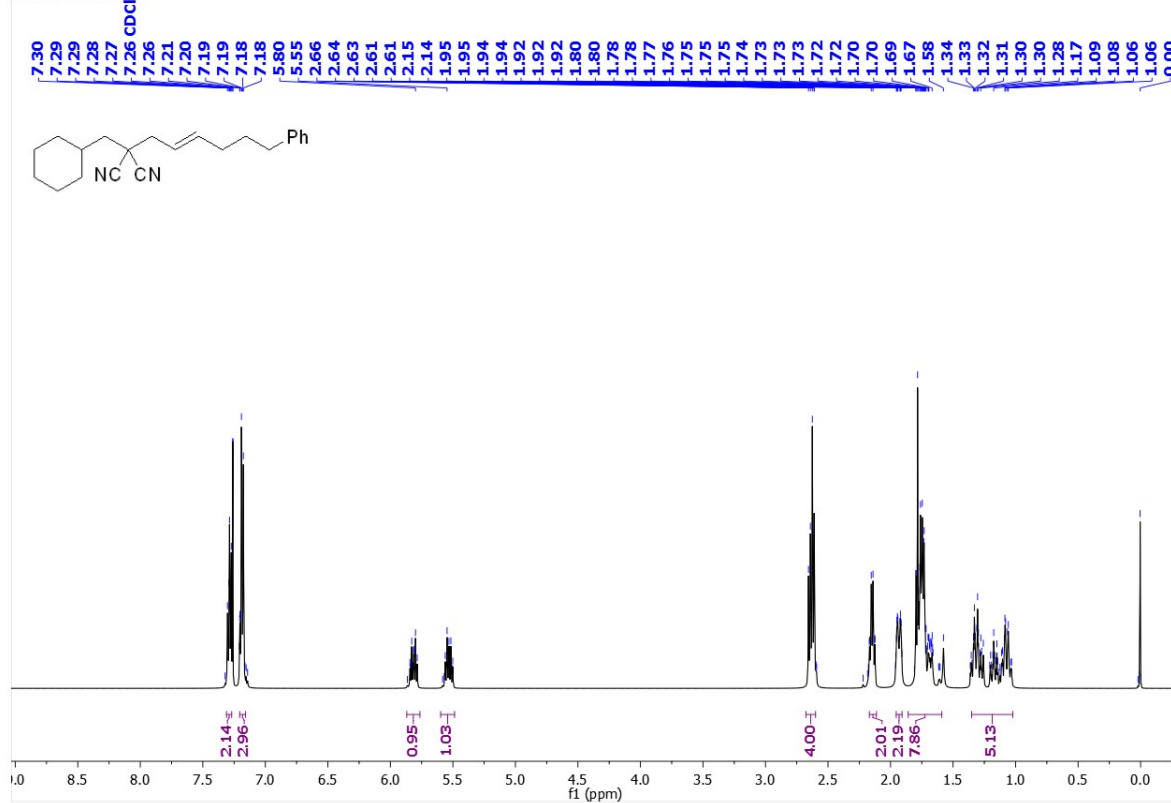
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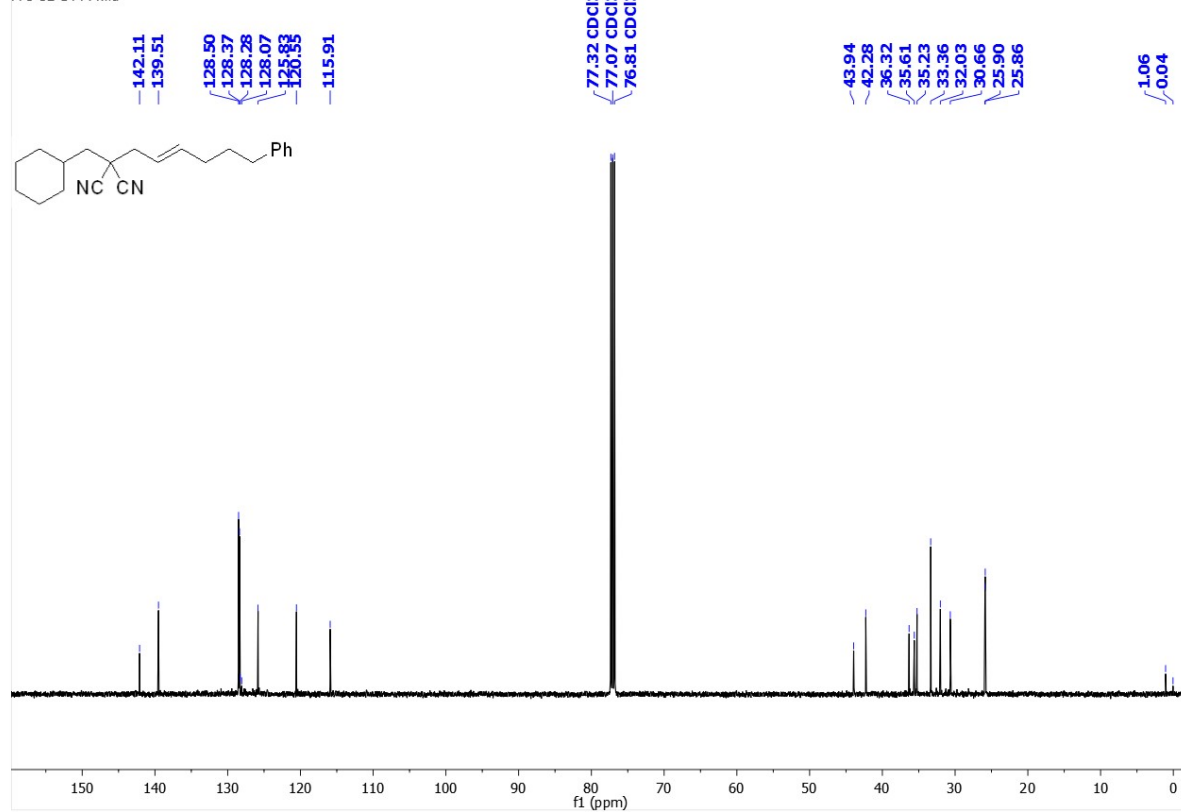
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M-5-32-14-P.3.fid



M-5-32-14-P.4.fid



- 1 Fan, L.-F., Xie, P.-P., Wang, P.-S., Hong, X. & Gong, L.-Z. Platinum-Catalyzed Allylic C–H Alkylation with Malononitriles. *CCS Chemistry* **4**, 1366-1375 (2021).
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