

Supporting Information

Three-dimensional flower-like organic dye intercalated layered double hydroxide composite for efficient photocatalysis in heterogeneous flow chemistry

Jinpeng Guo, ^[a] Yuanfa He, ^[a] Jianyao Kou, ^[b] Jiachao Liu, ^[a] Zhuo Xin, ^{[b]} Troels Skrydstrup, ^{*[d]} Yuxing Huang^{*[a]}*

[a]School of Physics and Material Science, Nanchang University, Nanchang 330031 Jiangxi, P. R. China.

[b]School of Pharmacy and Institute for Advanced Study, Nanchang University, Nanchang 330031 Jiangxi, P. R. China.

[c]Carbon Dioxide Activation Center (CADIAC), Novo Nordisk Foundation CO₂ Research Center, Interdisciplinary Nanoscience Center, Department of Chemistry, Aarhus University, Gustav Wieds Vej 14, Aarhus C, Denmark.

*Corresponding authors.

E-mail addresses: zhuoxin2@gmail.com (Z. Xin), ts@chem.au.dk (T. Skrydstrup), yuxinghuang2@gmail.com (Y. Huang),

1. Experimental section

1.1. General experimental procedure for the desired product 3a-3g:

In a typical flow photocatalytic reaction, 26.2 mg RB@MgAl-LDH is dispersed in 15 mL ethanol, followed by aldehyde (0.2 mmol), malononitrile (0.4 mmol) in a 100 mL reservoir. It is then pumped with a peristaltic pump at 0.3mL/s and the fluid reach a steady state. After this, the reaction is irradiated for 3 to 10 min by a 4×3W blue LEDs. The solvent was removed on a rotary evaporator under reduced pressure and the crude product was purified by column chromatography isolation on silica gel (eluent petroleum ether: ethyl acetate = 6:1) to give the product.

1.2. General experimental procedure for the desired product 6a-6g:

In a typical flow photocatalytic reaction, 26.2 mg RB@MgAl-LDH is dispersed in 15 mL ethanol, N-phenyltetrahydroisoquinoline (0.2 mmol), nitromethane (2 mmol) in a 100 mL reservoir. It is then pumped with a peristaltic pump at 0.3mL/s and the fluid reach a steady state. After this, the reaction is irradiated for 3.5 h by 2×1W green LEDs. The solvent was removed on a rotary evaporator under reduced pressure and the crude product was purified by column chromatography isolation on silica gel (eluent petroleum ether: ethyl acetate = 10:1) to give the product.

1.3. General experimental procedure for the desired product 8a-8g:

In a typical flow photocatalytic reaction, 26.2 mg RB@MgAl-LDH is dispersed in 15 mL acetone, N-phenyltetrahydroisoquinoline (0.2 mmol), trifluoroacetic acid (2.3uL) and pyrrolidine (2.5uL) in a 100 reservoir. It is then pumped with a peristaltic pump at 0.3mL/s and the fluid reach a steady state. After this, the reaction is irradiated for 2 h by a 2×1 W green LEDs. The solvent was removed on a rotary evaporator under reduced pressure and the crude product was

purified by column chromatography isolation on silica gel (eluent petroleum ether: ethyl acetate = 10:1) to give the product.

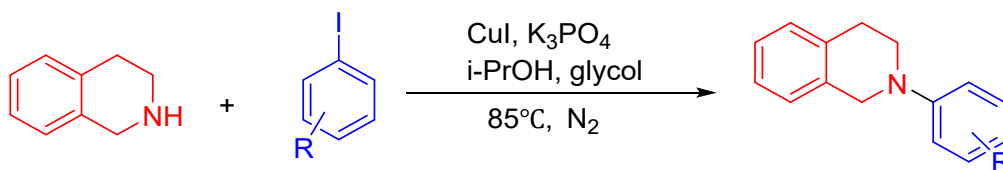
1.4. Cyclic test

Taking the reaction between *N*-phenyltetrahydroisoquinoline and nitromethane as an example, the cyclic catalytic performance was expressed through NMR yields. 26.2 mg RB@MgAl-LDH is dispersed in 15 mL ethanol, *N*-phenyltetrahydroisoquinoline (0.2 mmol), nitromethane (2 mmol) in a 100 mL reservoir. It is then pumped with a peristaltic pump at 0.3mL/s and the fluid reach a steady state. After this, the reaction is irradiated for 2 h by 2×1W green LEDs. After complete transformation, the catalyst was separated by centrifugation, and then washed three times with ethanol (8,000 r/min, 5 min). Lyophilized thoroughly before being used in the next round of stability experiments. Then, the yield of the target product was determined using ¹HNMR, using 1,3,5-trimethoxybenzene as the internal standard.

1.5. Gram-scale synthesis

524 mg RB@MgAl-LDH is dispersed in 40 mL ethanol, *N*-phenyltetrahydroisoquinoline (4 mmol), nitromethane (40 mmol) in a 100 mL reservoir. It is then pumped with a peristaltic pump at 0.3mL/s and the fluid reach a steady state. After this, the reaction is irradiated for 3.5 h by 2×1W green LEDs. The solvent was removed on a rotary evaporator under reduced pressure and the crude product was purified by column chromatography isolation on silica gel (eluent petroleum ether: ethyl acetate =10:1) to give the product.

1.6. Preparation of *N*-phenyl-1,2,3,4-tetrahydroisoquinoline and its derivatives



Scheme S1. Synthesis of *N*-phenyl-1,2,3,4-tetrahydroisoquinoline and its derivatives.

Typically, a mixture of Copper (I) iodide (1.0 mmol, 200 mg) and potassium phosphate (20.0 mmol, 4.25 g), isopropanol (10 mL), ethylene glycol (1.1 mL), iodobenzene (6 mmol), 1,2,3,4-tetrahydroisoquinoline (15 mmol) in a Schlenk tube was magnetically stirred at 85 °C under a 1 atm of nitrogen atmosphere for 24 h and then allowed to cool to room temperature. The liquid was transferred to a separatory funnel for extraction, and the upper organic solution was collected. The solvent was removed via rotary evaporation, and the remaining residue was purified via column chromatography on silica gel (hexane/ethyl acetate=20:1) to give the desired product.

1.7. Quantification of RB loading in RB@MgAl-LDH photocatalyst

A loading of 30 mg SDS@MgAl-LDH photocatalyst was loaded into the 1mg/mL RB solution, which was constantly stirred 12 h using a magnetic stir bar and stood still for 12 h. The mixture was then centrifuged (10,000 r/min, 5 min, 9 times) to separate the supernatant and the precipitate. The absorbance of the RB at its maximum absorption wavelength was recorded using UV-Vis spectroscopy, and its concentration was calculated based on the calibration curve with standard solutions. Then the quantification of RB loading was inferred. As shown in Fig S4, we can know quantification of RB loading in ethanol is better than water.

Then, RB@MgAl-LDH catalysts prepared in ethanol were used for all photocatalytic experiments.

1.7. Quantification of RB leakage in RB@MgAl-LDH photocatalyst

30 mg RB@MgAl-LDH photocatalyst was added into the 50 mL solution, which was constantly stirred 12 h using a magnetic stir bar. The mixture was then centrifuged (10,000 r/min, 5 min, 9 times) to separate the supernatant and the precipitate. The absorbance of the RB at its maximum absorption wavelength was recorded using UV-Vis spectroscopy, and its

concentration was calculated based on the calibration curve with standard solutions. Then the quantification of RB leakage was inferred. As shown in Fig S4, we can know that quantification of RB leakage in ethanol is better than water.

Then, In the homemade circulation flow reactors, 26.2 mg RB@MgAl-LDH is dispersed in 15 mL ethanol in a 100 mL reservoir. The fluid was pumped with a peristaltic pump at 0.3mL/s. To react for 3.5 h under 2×1W green LEDs irradiation. The absorbance of the RB at its maximum absorption wavelength was recorded using UV-Vis spectroscopy, and its concentration was calculated based on the calibration curve with standard solutions. It is inferred that the quantification of RB leakage is only 0.098 mg in aza-Henry CDC reaction.

2. Characterization data

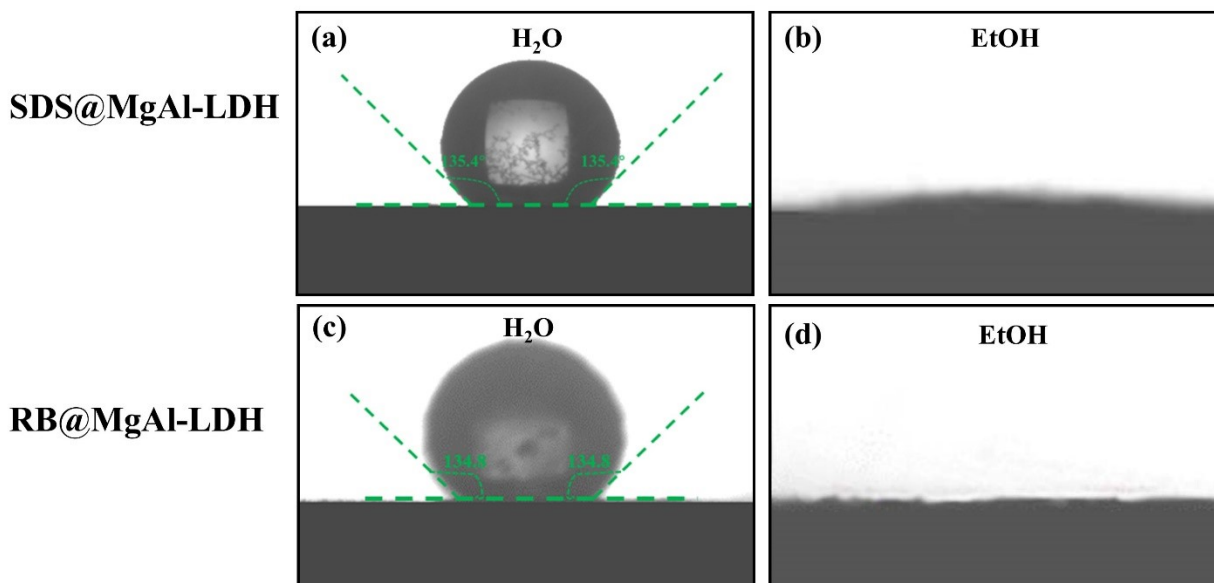


Figure S1. Contact angles of water (a, c) or ethanol (b, d) on SDS@MgAl-LDH (a, b) or RB@MgAl-LDH (c, d).

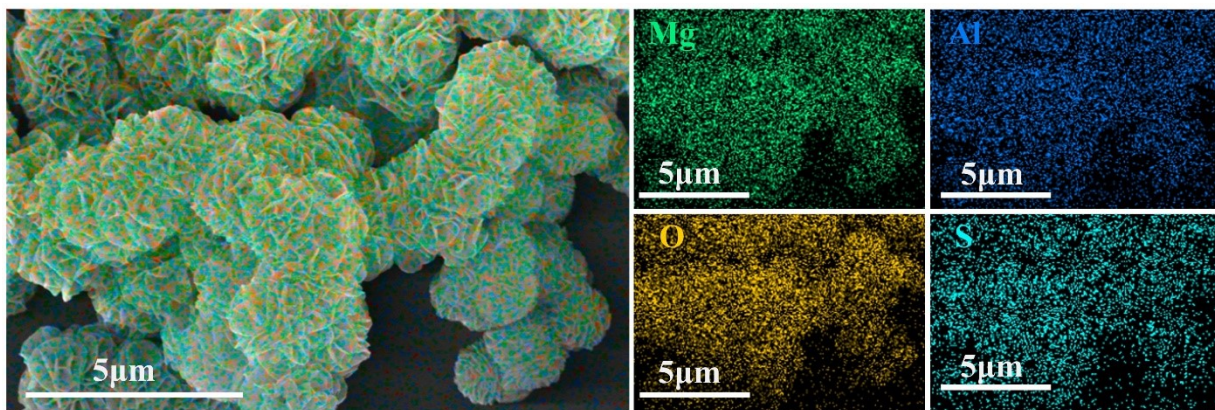


Figure S2. SEM and EDS spectrum of SDS@MgAl-LDH.

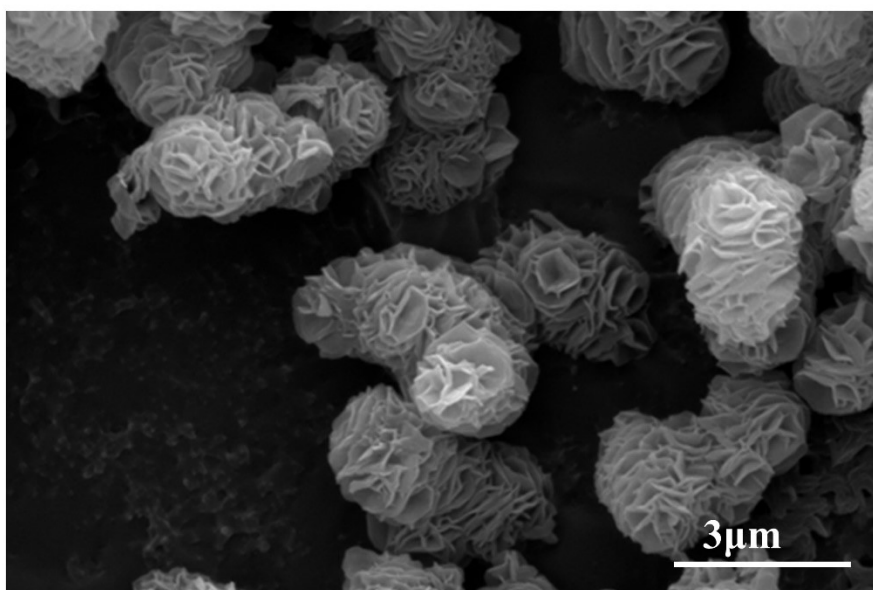


Figure S3. SEM spectrum of RB@MgAl-LDH after 8 cycles

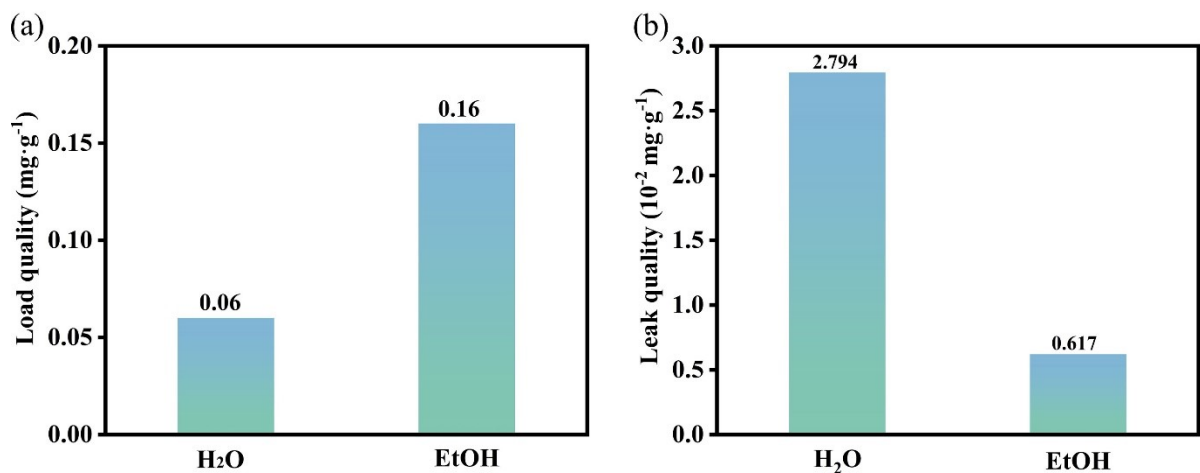


Figure S4. The load quality (a) or leak quality (b) of RB@MgAl-LDH.

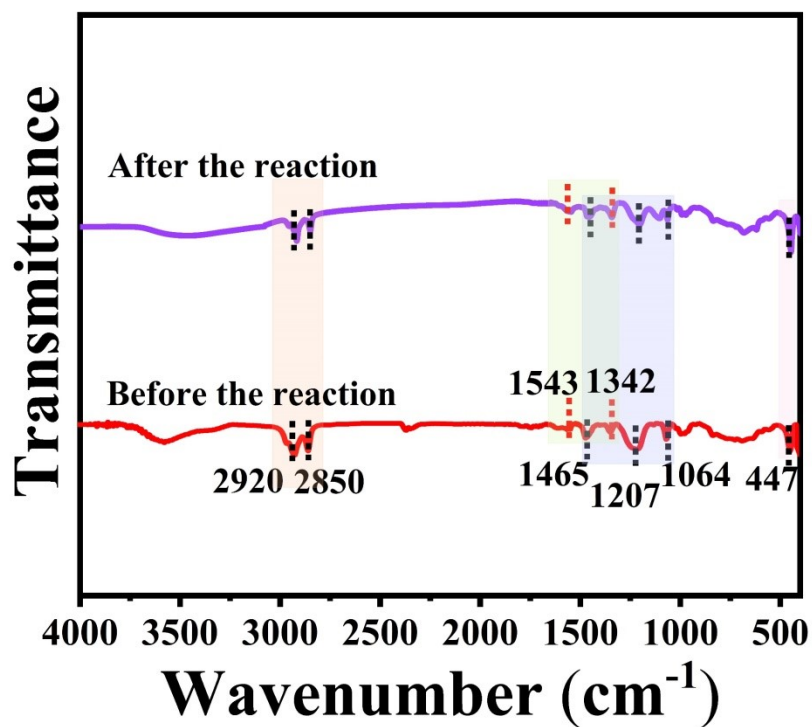


Figure S5. FT-IR spectra of RB@MgAl-LDH photocatalyst before and after the reaction.

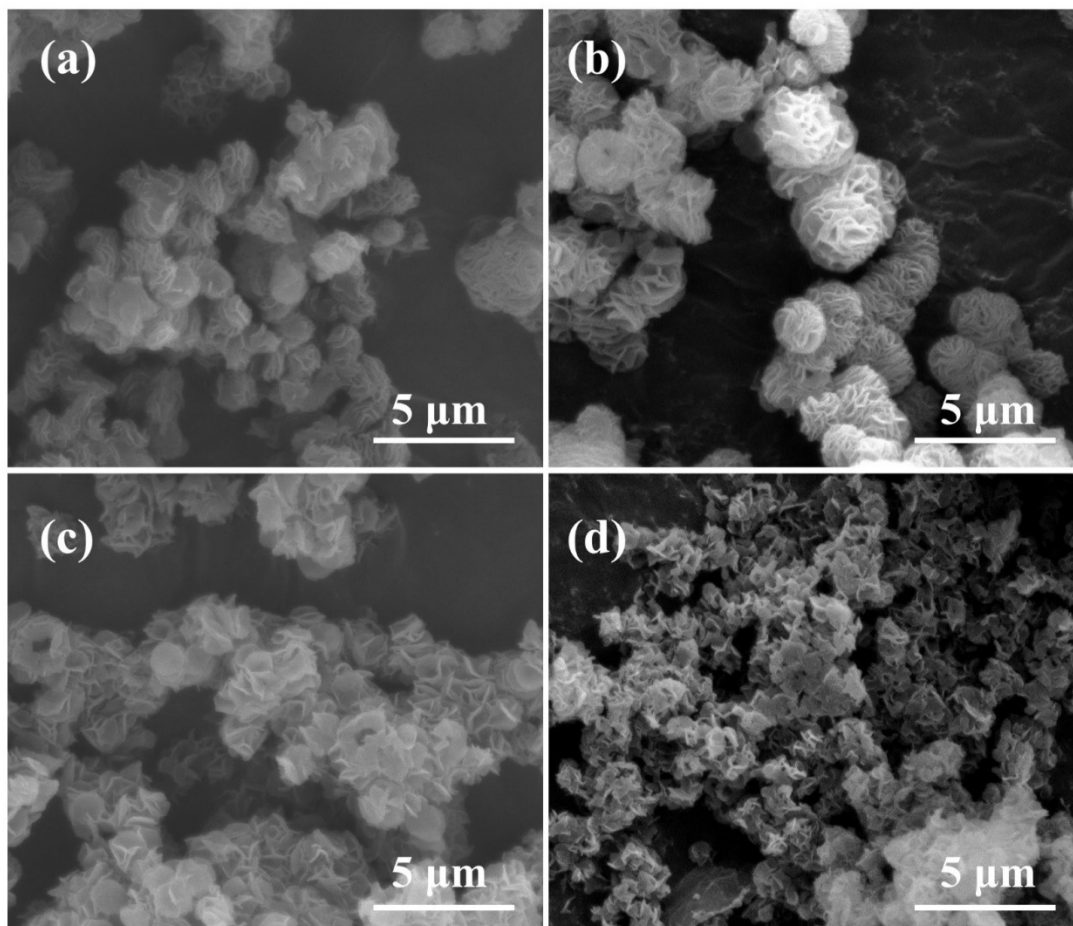
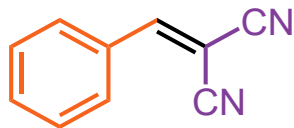
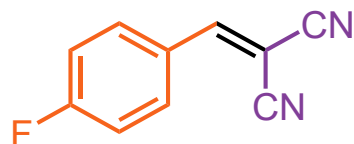


Figure S6. SEM of SDS@MgAl-LDH, (a)0.075mmol SDS, (b)0.15mmol SDS , (c)0.225mmol SDS , (d)0.3mmol SDS.

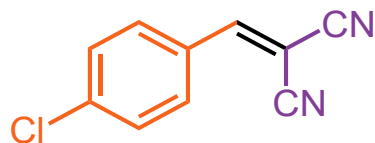
¹H and ¹³C NMR spectra of products



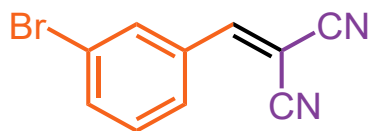
2-Benzylidenemalononitrile (3a) [1]: The product was obtained according to general procedure, as a white solid. (eluent petroleum ether: ethyl acetate = 6: 1). Yield: 95%. ¹H NMR (400 MHz, Chloroform-d): δ 7.91 (d, J = 7.7 Hz, 2H), 7.78 (s, 1H), 7.64 (t, J = 7.5 Hz, 1H), 7.55 (t, J = 7.7 Hz, 2H). ¹³C NMR (100 MHz, Chloroform-d): δ 159.90, 134.61, 130.92, 130.71, 129.62, 113.67, 112.51, 82.90.



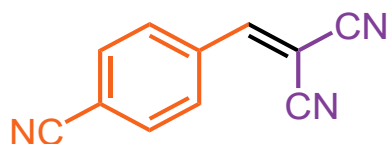
2-(4-Fluorobenzylidene)malononitrile (3b) [1]: The product was obtained according to general procedure, as a white solid. (eluent petroleum ether: ethyl acetate = 6: 1). Yield: 90%. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.94 (dd, J = 8.6, 5.2 Hz, 2H), 7.74 (s, 1H), 7.22 (t, J = 8.4 Hz, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 167.39, 164.80, 158.26, 133.57, 133.40 (d, J = 9.6 Hz), 127.34 (d, J = 3.3 Hz), 117.30, 117.08, 113.53, 112.46, 82.46, 82.43.



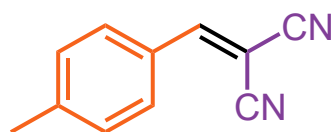
2-(4-Chlorobenzylidene)malononitrile(3c) [1]: The product was obtained according to general procedure, as a white solid. (eluent petroleum ether: ethyl acetate = 6: 1). Yield: 87%. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.86 (d, J = 8.3 Hz, 2H), 7.73 (s, 1H), 7.52 (d, J = 8.4 Hz, 2H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 158.25, 141.16, 131.83, 130.07, 129.26, 113.42, 112.32, 83.37.



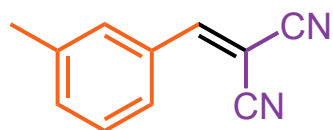
2-(3-Bromobenzylidene)malononitrile (3d): The product was obtained according to general procedure, as a White solid. (eluent petroleum ether: ethyl acetate = 6: 1). Yield:92%. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.97 (s, 1H), 7.89 (d, J = 8.0 Hz, 1H), 7.78 – 7.69 (m, 2H), 7.43 (t, J = 8.0 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 158.07, 137.24, 133.44, 132.55, 131.07, 128.61, 123.64, 113.16, 111.99, 84.68.



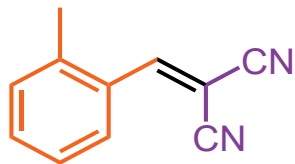
2-(4-cyanobenzylidene)malononitrile (3e) [2]: The product was obtained according to general procedure, as a White solid. (eluent petroleum ether: ethyl acetate = 6: 1). Yield:93%. ^1H NMR (400 MHz, Chloroform-*d*) δ 8.00 (d, J = 8.0 Hz, 2H), 7.84 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 157.37, 134.25, 133.14, 130.69, 117.27, 117.26, 112.75, 111.69, 86.88.



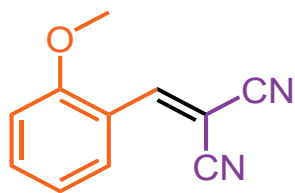
2-(4-methylbenzylidene)malononitrile (3f) [1]: The product was obtained according to general procedure, as a White solid. (eluent petroleum ether: ethyl acetate = 6: 1). Yield:91%. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.81 (dd, J = 8.3, 1.6 Hz, 2H), 7.72 (d, J = 1.5 Hz, 1H), 7.34 (d, J = 7.9 Hz, 2H), 2.46 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 159.76, 146.37, 130.90, 130.36, 128.45, 114.00, 112.85, 81.17, 22.00.



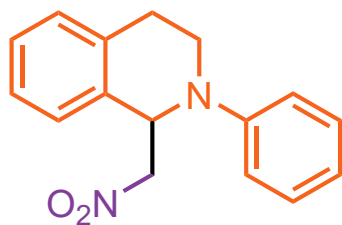
2-(3-methylbenzylidene)malononitrile (3g) [2]: The product was obtained according to general procedure, as a White solid. (eluent petroleum ether: ethyl acetate = 6: 1). Yield:90%. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.73 (d, *J* = 6.3 Hz, 2H), 7.68 (s, 1H), 7.44 (s, 2H), 2.43 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 160.14, 139.63, 135.55, 131.26, 130.94, 129.49, 127.90, 113.82, 112.63, 82.41, 21.25.



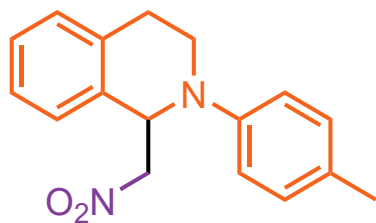
2-(2-methylbenzylidene)malononitrile (3h) [2]: The product was obtained according to general procedure, as a White solid. (eluent petroleum ether: ethyl acetate = 6: 1). Yield:88%. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.11 (s, 1H), 8.08 (d, *J* = 8.0 Hz, 1H), 7.49 (td, *J* = 7.6, 1.5 Hz, 1H), 7.39 – 7.30 (m, 2H), 2.45 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 158.17, 139.74, 134.15, 131.39, 129.90, 128.26, 127.03, 113.80, 112.45, 83.95, 19.78.



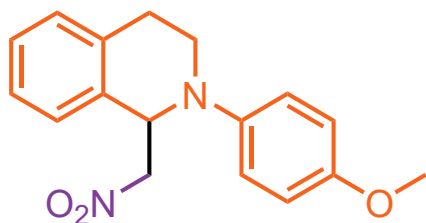
2-(2-methoxybenzylidene)malononitrile(3i) [3]: The product was obtained according to general procedure, as a White solid. (eluent petroleum ether: ethyl acetate = 6: 1). Yield: 82%. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.29 (s, 1H), 8.17 (dd, *J* = 8.0, 1.7 Hz, 1H), 7.59 (ddd, *J* = 8.7, 7.3, 1.7 Hz, 1H), 7.06 (t, *J* = 7.7 Hz, 1H), 6.99 (d, *J* = 8.5 Hz, 1H), 3.92 (d, *J* = 1.5 Hz, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 158.96, 154.47, 136.56, 128.82, 121.19, 120.15, 114.34, 113.02, 111.55, 81.29, 55.97.



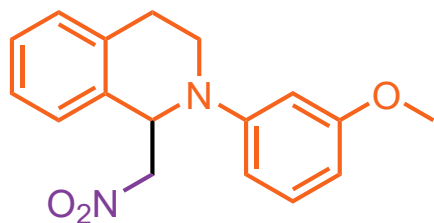
1-(nitromethyl)-2-phenyl-1,2,3,4-tetrahydroisoquinoline(6a) [4]:The product was obtained according to general procedure, as a Yellow oil. (eluent petroleum ether: ethyl acetate = 10: 1). Yield: 97%. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.30 – 7.13 (m, 6H), 7.05 – 6.92 (m, 2H), 6.85 (tt, J = 7.2, 1.1 Hz, 1H), 5.55 (t, J = 7.2 Hz, 1H), 4.88 (dd, J = 11.8, 7.8 Hz, 1H), 4.57 (dd, J = 11.8, 6.6 Hz, 1H), 3.72 – 3.57 (m, 2H), 3.09 (ddd, J = 16.4, 8.6, 5.7 Hz, 1H), 2.80 (dt, J = 16.3, 5.0 Hz, 1H)); ^{13}C NMR (100 MHz, Chloroform-*d*): δ 148.53, 135.39, 133.02, 129.61, 129.30, 128.22, 127.11, 126.80, 119.52, 115.20, 78.87, 58.30, 42.15, 26.54.



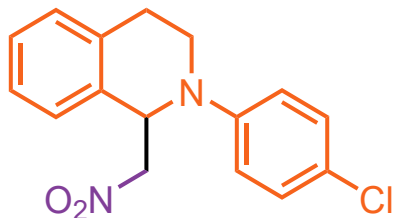
1-(nitromethyl)-2-(p-tolyl)-1,2,3,4-tetrahydroisoquinoline(6b) [4]:The product was obtained according to general procedure, as a yellow oil. (eluent petroleum ether: ethyl acetate = 10: 1). Yield: 95%. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.26 – 6.99 (m, 6H), 6.90 – 6.82 (m, 2H), 5.47 (dd, J = 8.0, 6.4 Hz, 1H), 4.82 (dd, J = 11.8, 8.1 Hz, 1H), 4.52 (dd, J = 11.8, 6.3 Hz, 1H), 3.67 – 3.49 (m, 2H), 3.03 (ddd, J = 15.5, 9.3, 5.8 Hz, 1H), 2.72 (dt, J = 16.4, 4.6 Hz, 1H), 2.24 (s, 3H). ^{13}C NMR (100 MHz, Chloroform-*d*): 146.49, 135.45, 133.05, 130.08, 129.38, 129.20, 128.09, 127.07, 126.72, 116.01, 78.92, 58.49, 42.40, 26.32, 20.46.



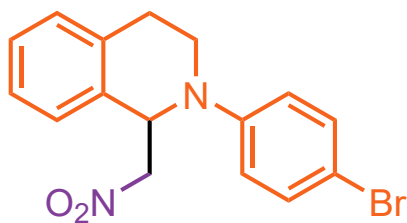
2-(4-methoxyphenyl)-1-(nitromethyl)-1,2,3,4-tetrahydroisoquinoline (6c) [4]: The product was obtained according to general procedure, as a Yellow oil. (eluent petroleum ether: ethyl acetate = 10: 1). Yield: 91%. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.29 – 7.11 (m, 4H), 6.97 – 6.88 (m, 2H), 6.82 (ddd, $J = 9.0, 3.5, 1.8$ Hz, 2H), 5.39 (dd, $J = 8.5, 5.7$ Hz, 1H), 4.83 (ddd, $J = 12.0, 8.6, 1.4$ Hz, 1H), 4.56 (ddd, $J = 12.0, 5.8, 1.3$ Hz, 1H), 3.75 (d, $J = 1.6$ Hz, 3H), 3.64 – 3.46 (m, 2H), 3.02 (ddd, $J = 16.3, 9.3, 6.7$ Hz, 1H), 2.70 (dt, $J = 16.5, 4.0$ Hz, 1H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 154.11, 143.19, 135.56, 133.02, 129.58, 128.02, 127.04, 126.74, 118.99, 114.84, 79.09, 55.71, 43.27, 25.93.



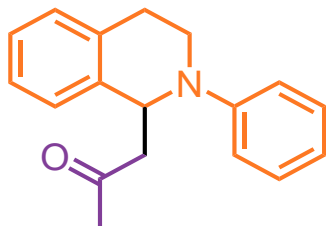
2-(3-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline(6d) [4]: The product was obtained according to general procedure, as a yellow oil. (eluent petroleum ether: ethyl acetate = 10: 1). Yield: 91%. ^1H NMR (400 MHz, Chloroform-*d*): δ 7.40 – 7.12 (m, 5H), 6.63 (dt, $J = 8.3, 2.1$ Hz, 1H), 6.58 (t, $J = 2.2$ Hz, 1H), 6.45 (dt, $J = 8.3, 2.0$ Hz, 1H), 5.58 (t, $J = 7.2$ Hz, 1H), 4.90 (ddd, $J = 11.8, 7.6, 1.6$ Hz, 1H), 4.59 (ddd, $J = 11.9, 6.8, 1.6$ Hz, 1H), 3.84 (d, $J = 1.6$ Hz, 3H), 3.73 – 3.58 (m, 2H), 3.13 (ddd, $J = 17.2, 8.3, 5.9$ Hz, 1H), 2.84 (dt, $J = 16.3, 5.2$ Hz, 1H). ^{13}C NMR (100 MHz, Chloroform-*d*): δ 160.99, 149.86, 135.34, 133.03, 130.35, 129.25, 128.27, 127.11, 126.82, 107.68, 104.24, 101.59, 78.90, 58.35, 55.31, 42.26, 26.73.



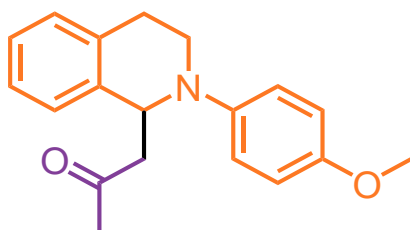
2-(4-chloro-phenyl)-1-nitromethyl-1,2,3,4-tetrahydroisoquinoline(6e) [4]: The product was obtained according to general procedure, as a yellow oil. (eluent petroleum ether: ethyl acetate = 10: 1). Yield: 80%. ^1H NMR (400 MHz, V) δ 7.47 – 7.20 (m, 6H), 7.05 – 6.96 (m, 2H), 5.60 (dd, $J = 8.1, 6.4$ Hz, 1H), 4.96 (ddd, $J = 11.9, 8.1, 0.8$ Hz, 1H), 4.68 (ddd, $J = 12.0, 6.4, 0.8$ Hz, 1H), 3.81 – 3.66 (m, 2H), 3.18 (ddd, $J = 15.2, 8.5, 6.2$ Hz, 1H), 2.89 (dt, $J = 16.4, 4.8$ Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 147.23, 135.20, 132.60, 129.46, 128.40, 127.11, 126.97, 124.55, 116.63, 78.81, 58.36, 53.58, 42.34, 26.29.



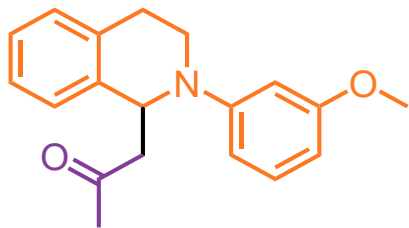
2-(4-bromophenyl)-1-(nitromethyl)-1,2,3,4-tetrahydroisoquinoline (3d) [5]: The product was obtained according to general procedure, as a yellow oil. (eluent petroleum ether: ethyl acetate = 10: 1). Yield: 83%. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.31 – 7.24 (m, 2H), 7.24 – 7.10 (m, 3H), 7.07 (d, $J = 7.4$ Hz, 1H), 6.78 (d, $J = 8.3$ Hz, 2H), 5.43 (t, $J = 7.4$ Hz, 1H), 4.82 – 4.72 (m, 1H), 4.50 (ddd, $J = 12.2, 6.4, 1.9$ Hz, 1H), 3.54 (d, $J = 5.9$ Hz, 2H), 3.00 (dt, $J = 15.4, 7.3$ Hz, 1H), 2.71 (dd, $J = 16.6, 5.2$ Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 147.51, 135.05, 132.45, 132.22, 129.29, 128.28, 126.97, 126.82, 116.77, 111.52, 78.60, 58.10, 42.06, 26.18.



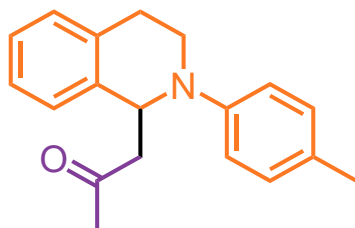
1-(1,2,3,4-tetrahydro-2-phenyl-1-isoquinolinyl)-2-propanone (8a) [6]: The product was obtained according to general procedure, as a White solid. (eluent petroleum ether: ethyl acetate = 10: 1). Yield: 93%. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.30 – 7.21 (m, 2H), 7.21 – 7.10 (m, 4H), 6.95 (d, J = 8.1 Hz, 2H), 6.79 (t, J = 7.3 Hz, 1H), 5.41 (t, J = 6.4 Hz, 1H), 3.66 (dt, J = 12.4, 5.3 Hz, 1H), 3.54 (ddd, J = 13.0, 9.1, 4.6 Hz, 1H), 3.06 (ddd, J = 15.3, 5.5, 3.6 Hz, 2H), 2.89 – 2.78 (m, 2H), 2.08 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 207.27, 148.88, 138.28, 134.43, 129.35, 128.68, 126.86, 126.81, 126.28, 118.27, 114.78, 54.79, 50.21, 42.06, 31.09, 27.21.



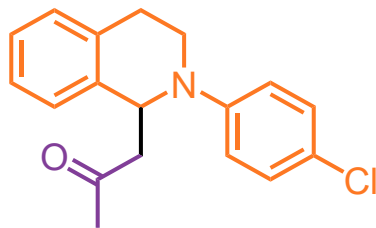
1-[1,2,3,4-tetrahydro-2-(4-methoxyphenyl)-1-isoquinolinyl]-2-propanone (8b) [6]: The product was obtained according to general procedure, as a White solid. (eluent petroleum ether: ethyl acetate = 10: 1). Yield: 85%. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.23 – 7.05 (m, 4H), 6.99 – 6.87 (m, 2H), 6.87 – 6.74 (m, 2H), 5.25 (dt, J = 17.7, 6.4 Hz, 1H), 3.86 – 3.62 (m, 3H), 3.62 – 3.37 (m, 2H), 3.02 (ddq, J = 17.7, 12.5, 6.3 Hz, 2H), 2.76 (dddd, J = 20.3, 16.0, 9.9, 5.2 Hz, 2H), 2.11 – 2.02 (m, 3H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 207.35, 153.27, 143.67, 138.26, 134.30, 128.92, 126.79, 126.60, 126.16, 118.38, 114.62, 55.95, 55.59, 49.96, 42.86, 30.86, 26.73.



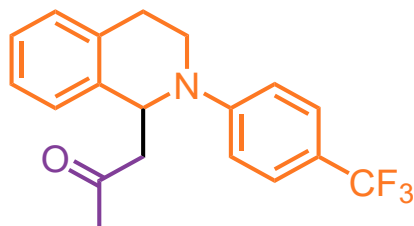
1-[1,2,3,4-tetrahydro-2-(3-methoxyphenyl)-1-isoquinolinyl]-2-propanone (8c) [7]: The product was obtained according to general procedure, as a White solid. (eluent petroleum ether: ethyl acetate = 10: 1). Yield: 80%. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.20 – 7.10 (m, 5H), 6.55 (dd, $J = 8.2, 2.3$ Hz, 1H), 6.50 (d, $J = 2.3$ Hz, 1H), 6.35 (dd, $J = 8.2, 2.3$ Hz, 1H), 5.40 (t, $J = 6.3$ Hz, 1H), 3.79 (d, $J = 1.1$ Hz, 3H), 3.64 (dt, $J = 11.5, 5.4$ Hz, 1H), 3.52 (ddd, $J = 12.9, 8.8, 4.7$ Hz, 1H), 3.12 – 3.04 (m, 1H), 3.04 – 3.00 (m, 1H), 2.83 (ddd, $J = 16.3, 6.0, 3.3$ Hz, 2H), 2.08 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 207.19, 160.81, 150.22, 138.25, 134.41, 130.01, 128.62, 126.82, 126.28, 107.37, 103.17, 100.94, 54.78, 50.28, 42.14, 31.09, 27.31.



1-(2-(p-tolyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)propan-2-one (8d) [8]: The product was obtained according to general procedure, as a White solid. (eluent petroleum ether: ethyl acetate = 10: 1). Yield: 88%. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.32 – 7.20 (m, 4H), 7.16 (d, $J = 8.1$ Hz, 2H), 6.99 (d, $J = 8.1$ Hz, 2H), 5.45 (t, $J = 6.4$ Hz, 1H), 3.80 – 3.52 (m, 2H), 3.14 (ddd, $J = 15.8, 9.6, 5.9$ Hz, 2H), 2.91 (dd, $J = 16.1, 6.8$ Hz, 2H), 2.36 (s, 3H), 2.18 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 207.37, 146.71, 138.24, 134.36, 129.82, 128.77, 126.82, 126.69, 126.18, 115.71, 55.18, 50.05, 42.21, 30.97, 26.95, 20.30.



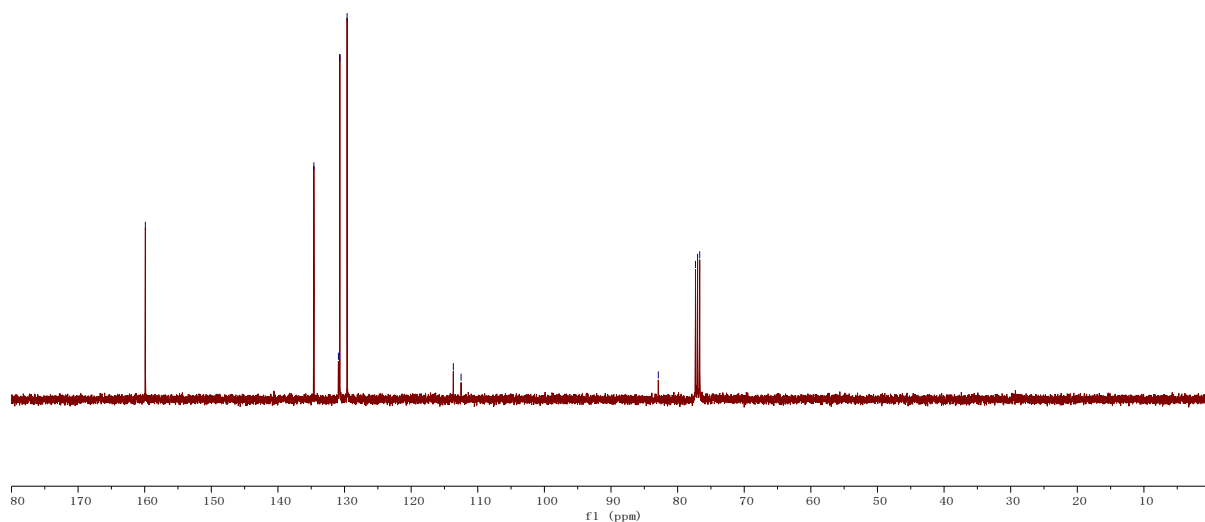
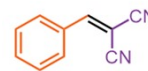
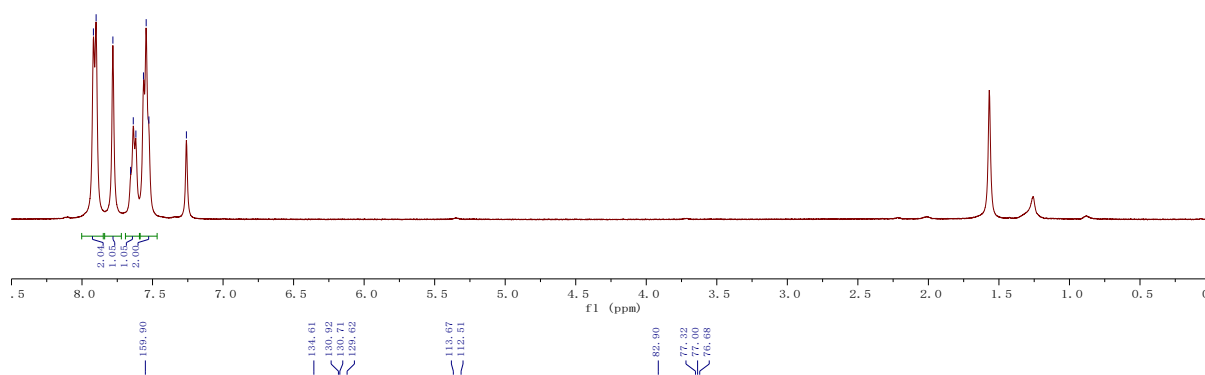
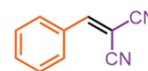
1-[2-(4-Chlorophenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl]propan-2-one (8e) [8]: The product was obtained according to general procedure, as a White solid. (eluent petroleum ether: ethyl acetate = 10: 1). Yield: 92%. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.17 (ddd, $J = 8.9, 6.0, 2.3$ Hz, 6H), 6.86 (d, $J = 8.8$ Hz, 2H), 5.34 (t, $J = 6.3$ Hz, 1H), 3.67 – 3.45 (m, 2H), 3.04 (ddd, $J = 16.2, 9.0, 5.6$ Hz, 2H), 2.88 – 2.77 (m, 2H), 2.08 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 207.03, 147.46, 137.92, 134.19, 129.12, 128.70, 126.95, 126.80, 126.40, 122.98, 115.82, 54.77, 50.14, 42.20, 31.10, 27.01.

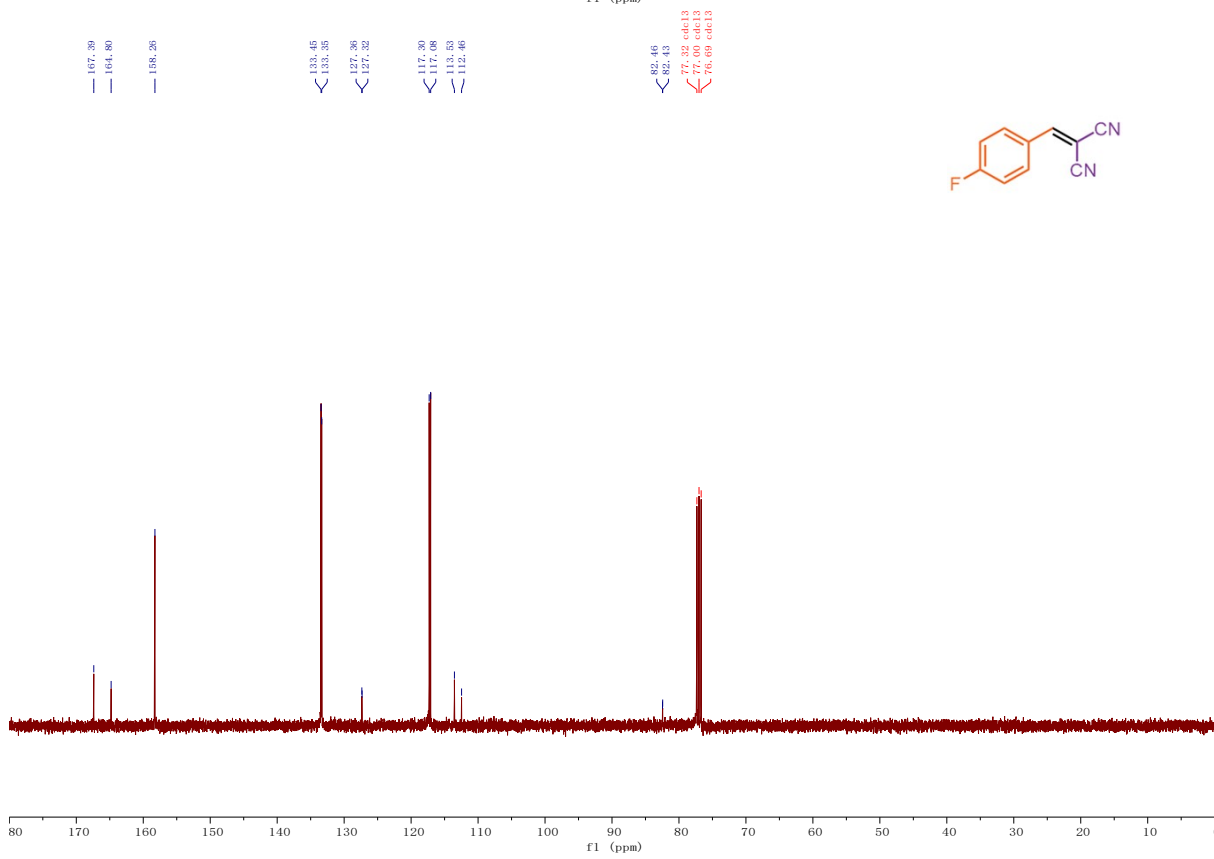
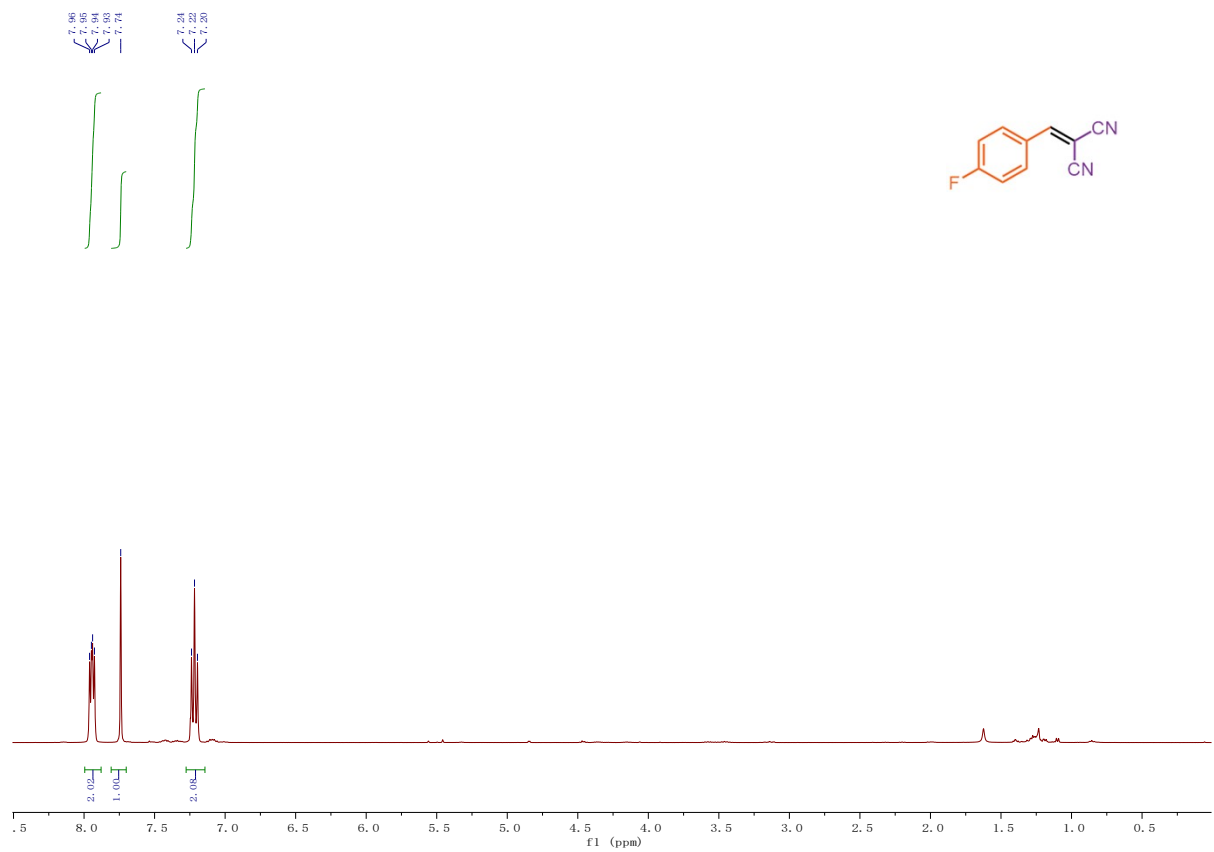


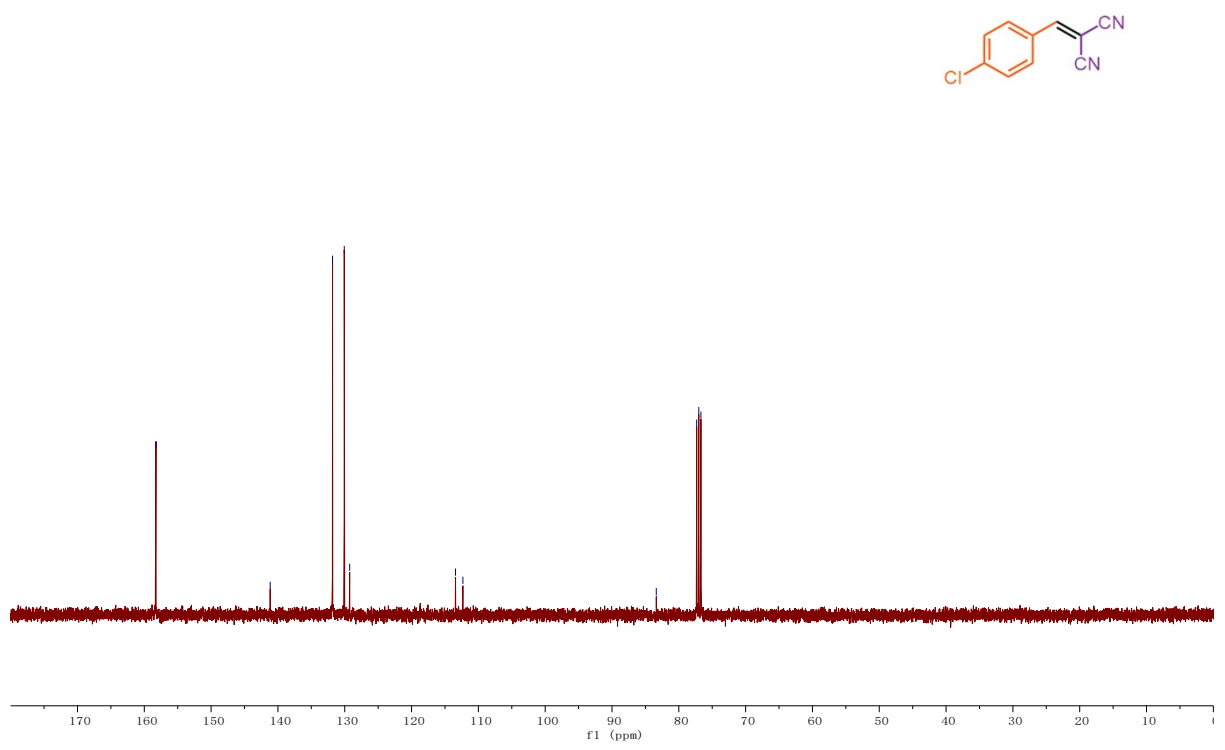
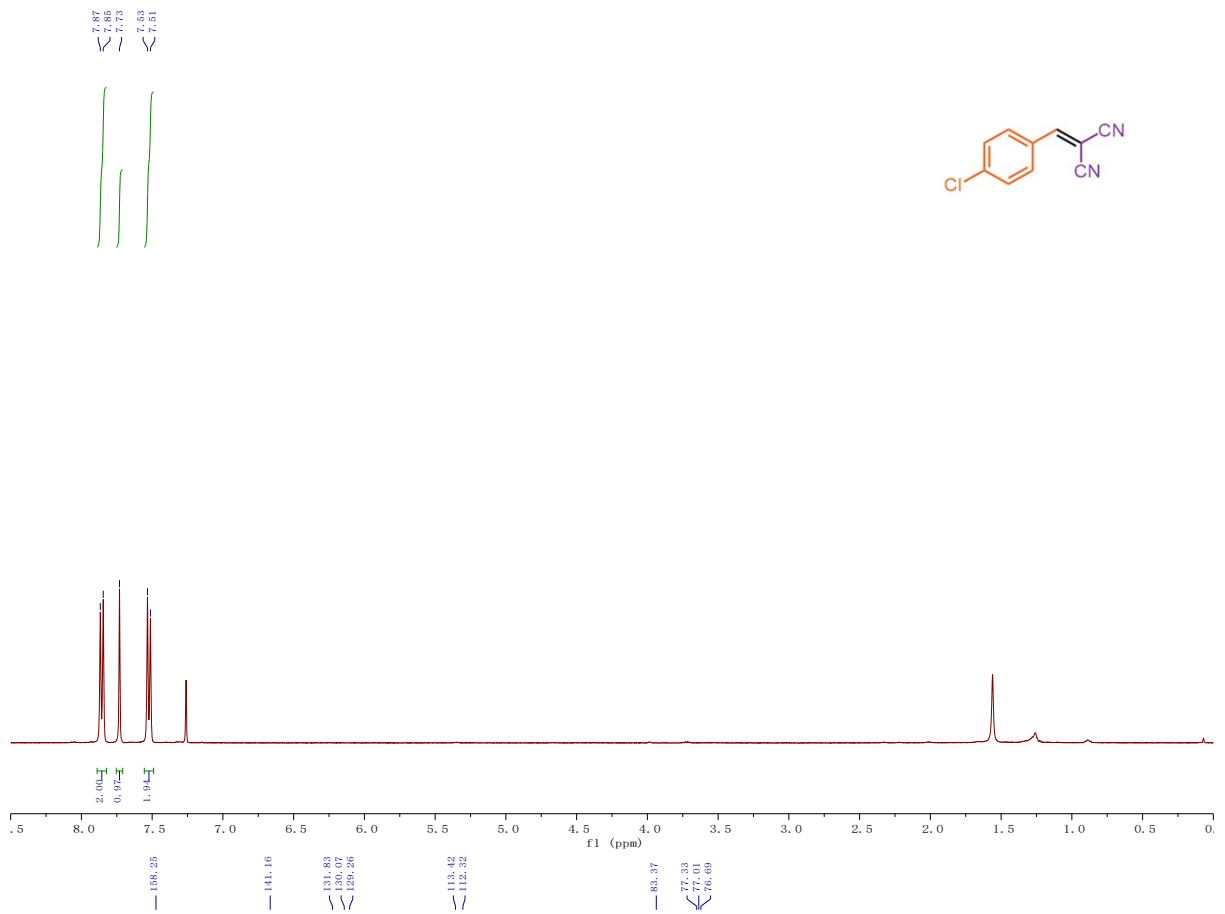
1-(2-(4-(trifluoromethyl)phenyl)-1,2,3,4-tetrahydroisoquinolin-1-yl)propan-2-one (8f) [6]: The product was obtained according to general procedure, as a White solid. (eluent petroleum ether: ethyl acetate = 10: 1). Yield: 88%. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.48 (d, $J = 8.6$ Hz, 2H), 7.28 – 7.14 (m, 4H), 6.95 (d, $J = 8.6$ Hz, 2H), 5.48 (dd, $J = 7.5, 5.0$ Hz, 1H), 3.62 (tdd, $J = 12.5, 9.9, 5.5$ Hz, 2H), 3.13 – 3.01 (m, 2H), 2.98 – 2.84 (m, 2H), 2.10 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 206.53, 150.41, 137.54, 133.97, 128.27, 126.96, 126.59, 126.43, 126.39, 126.33, 112.26, 53.88, 49.92, 41.92, 30.92, 27.15.

^1H NMR and ^{13}C NMR spectra

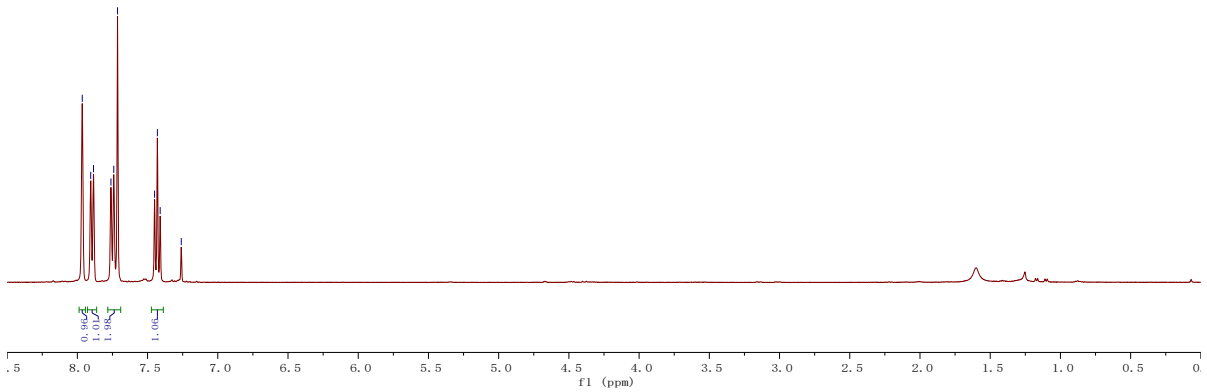
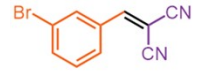
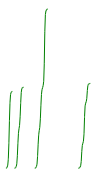
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7.53
7.26



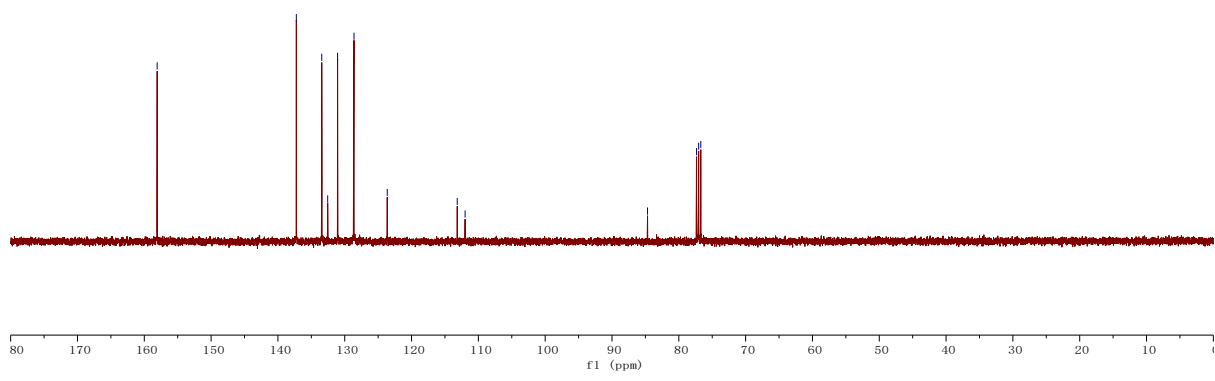
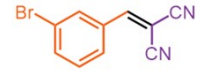


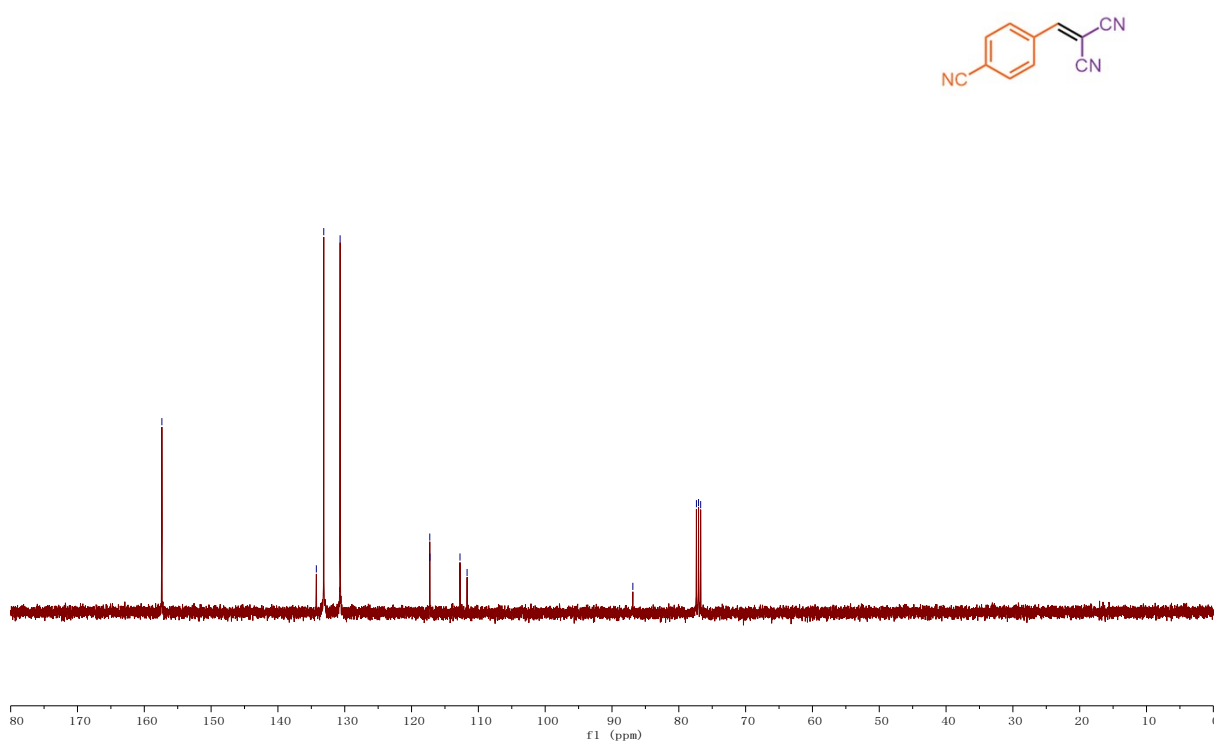
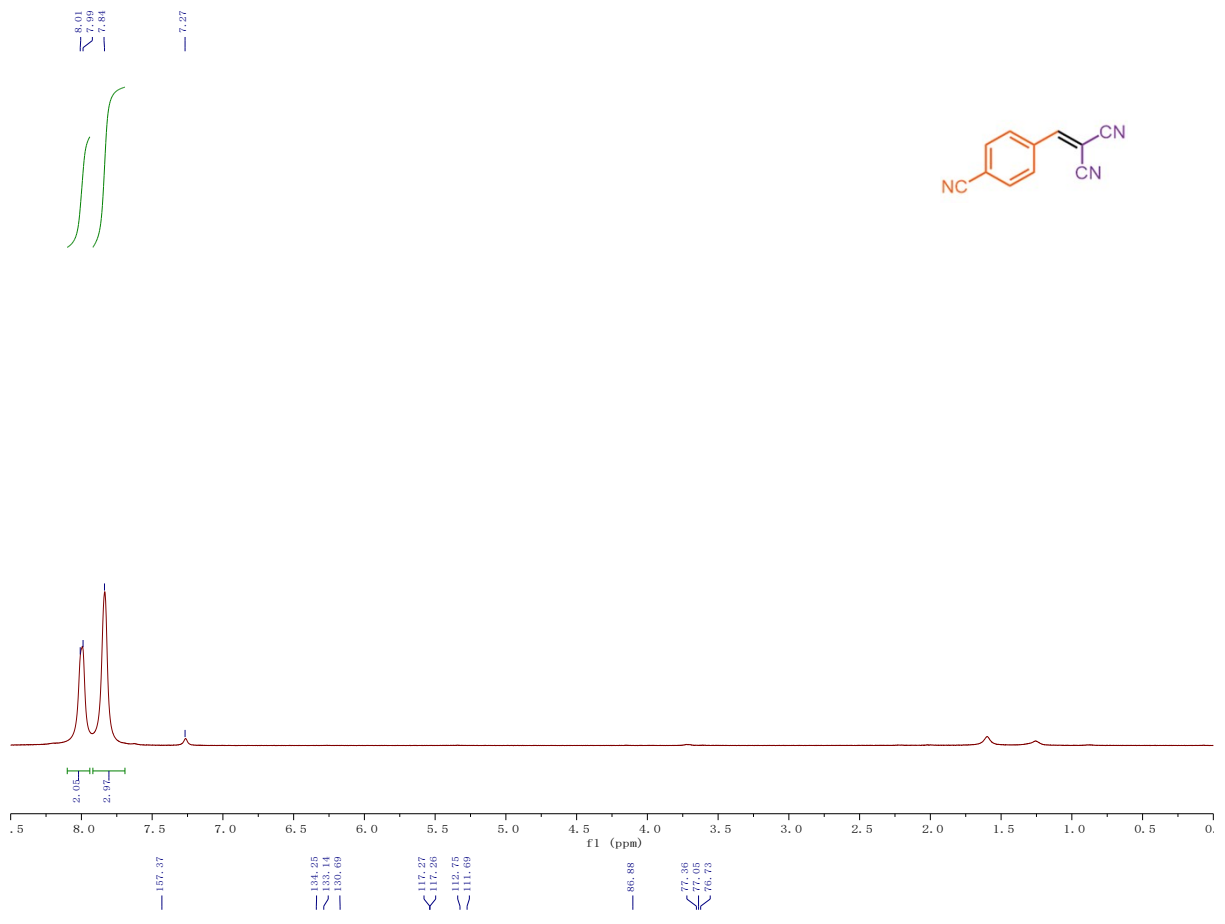


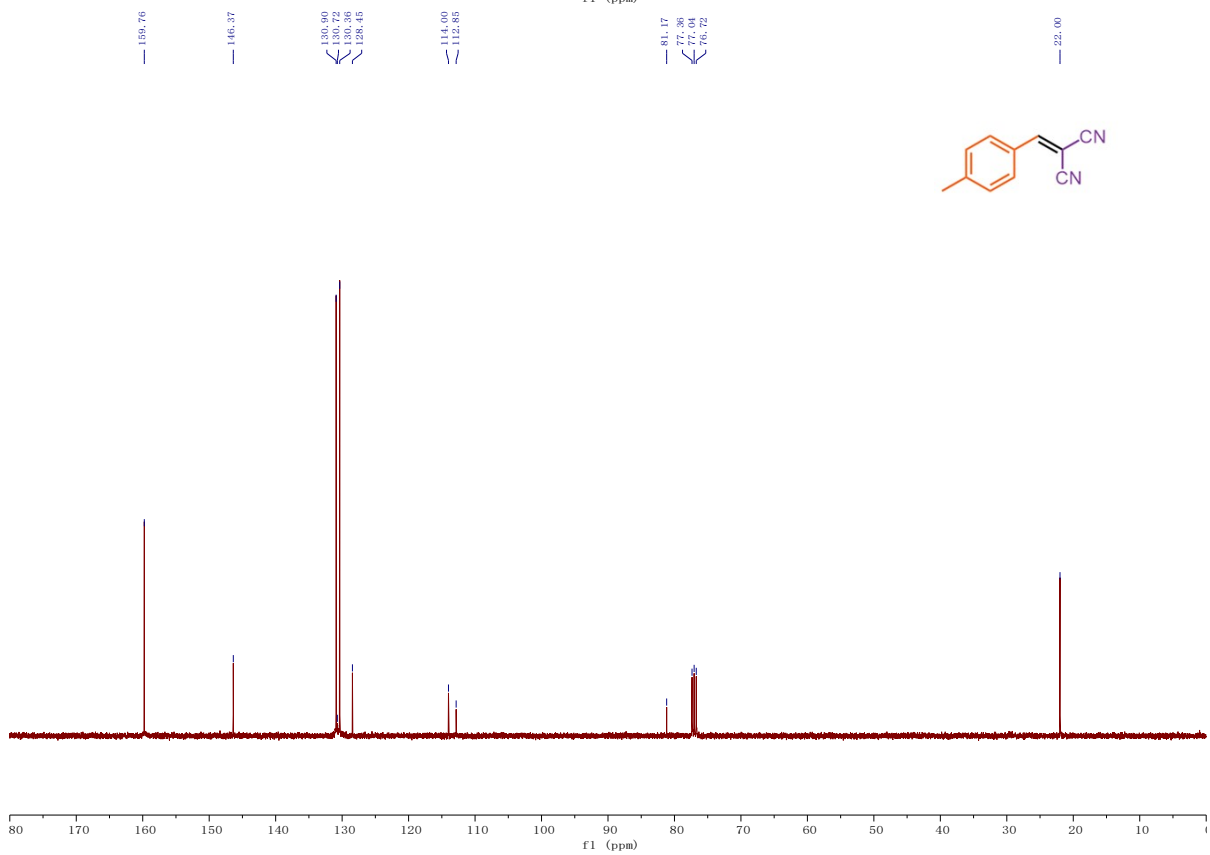
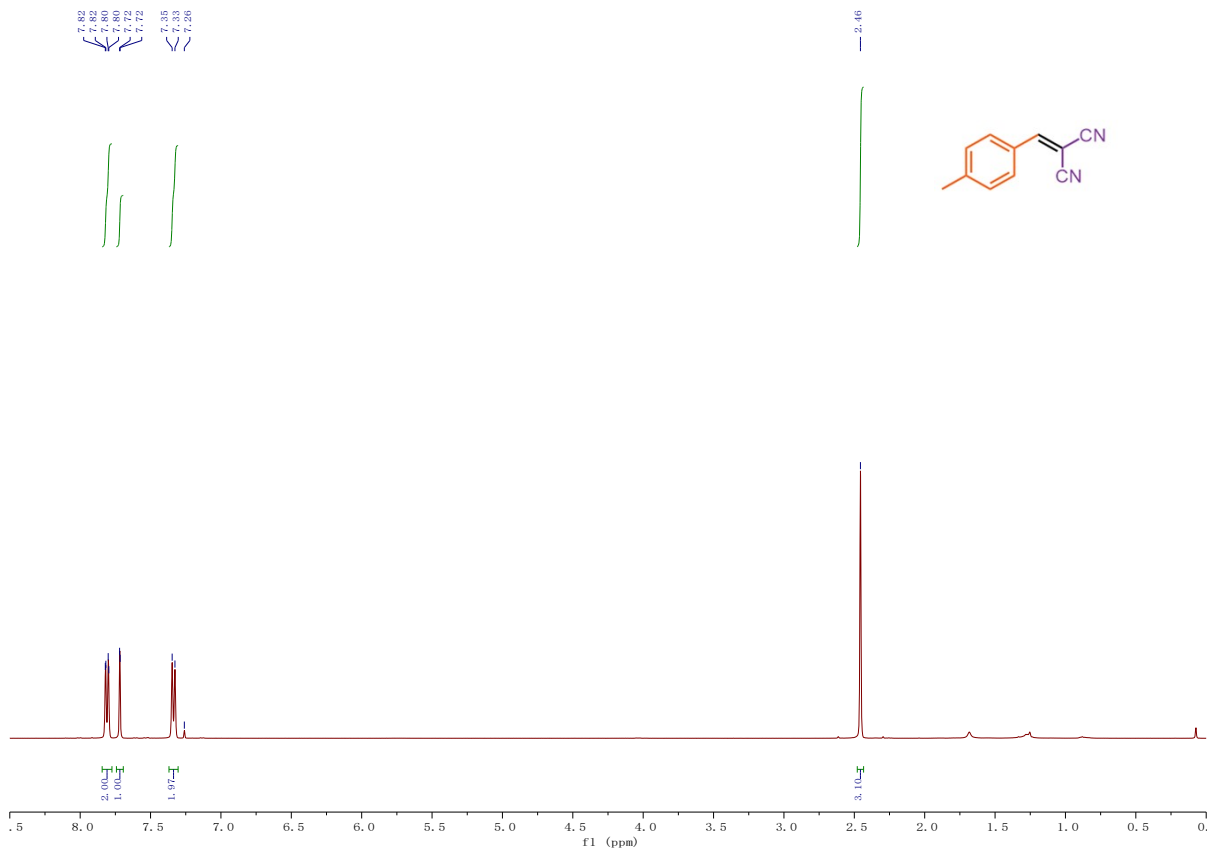
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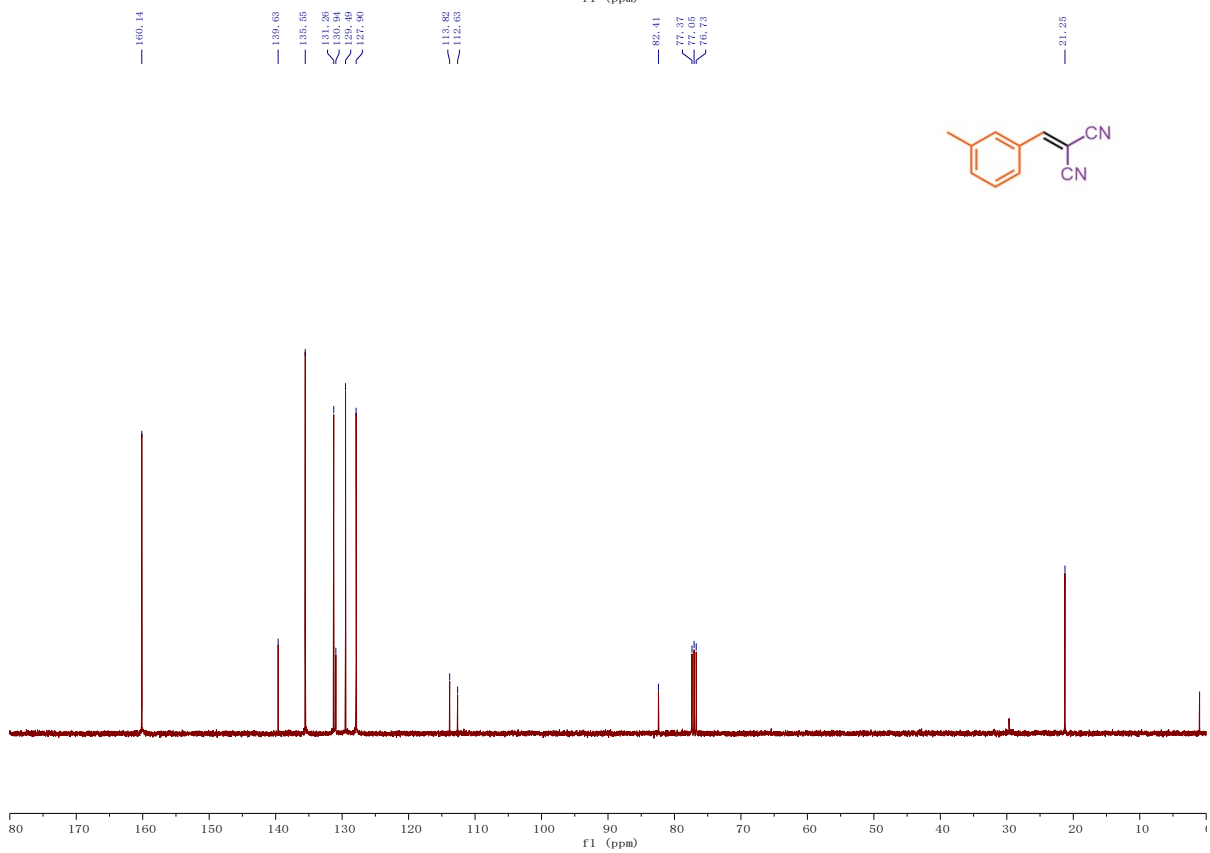
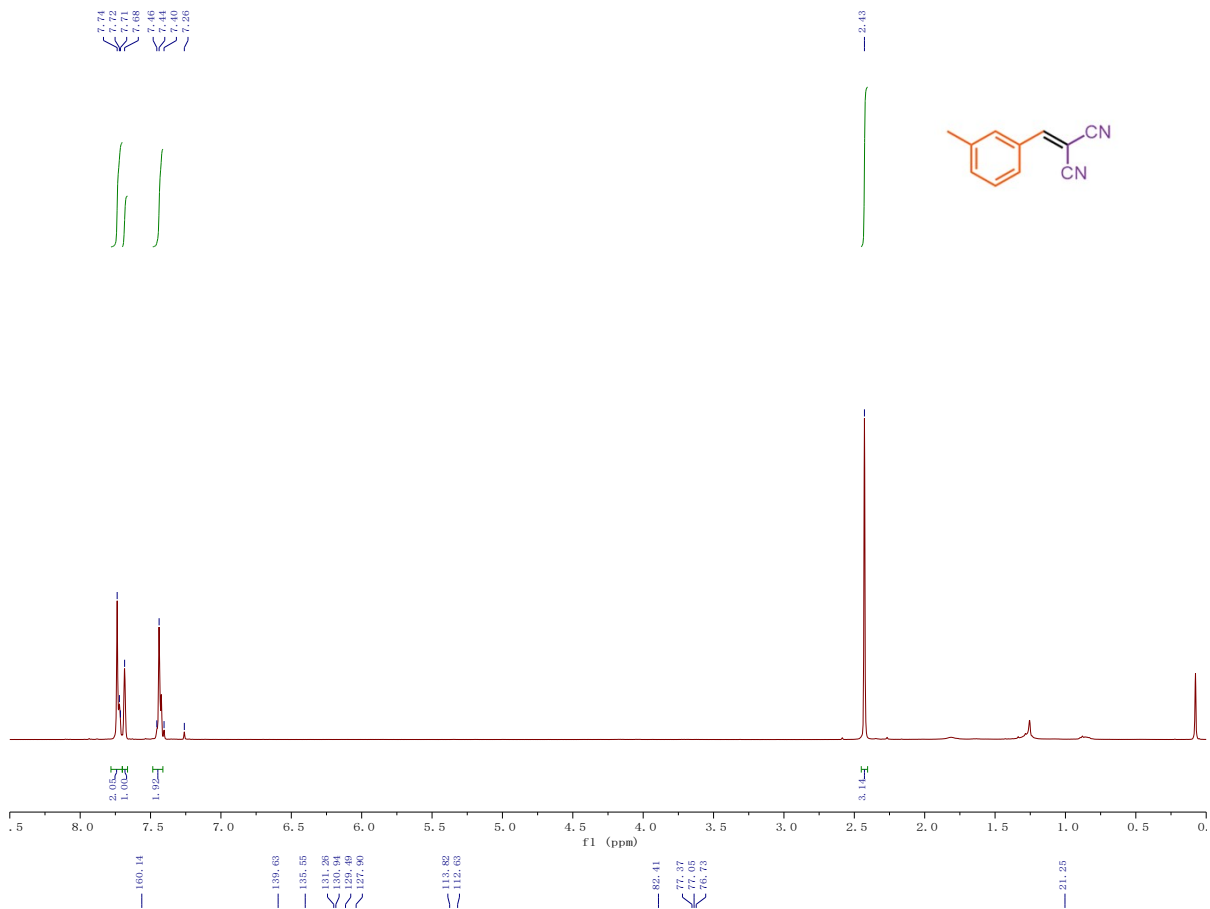


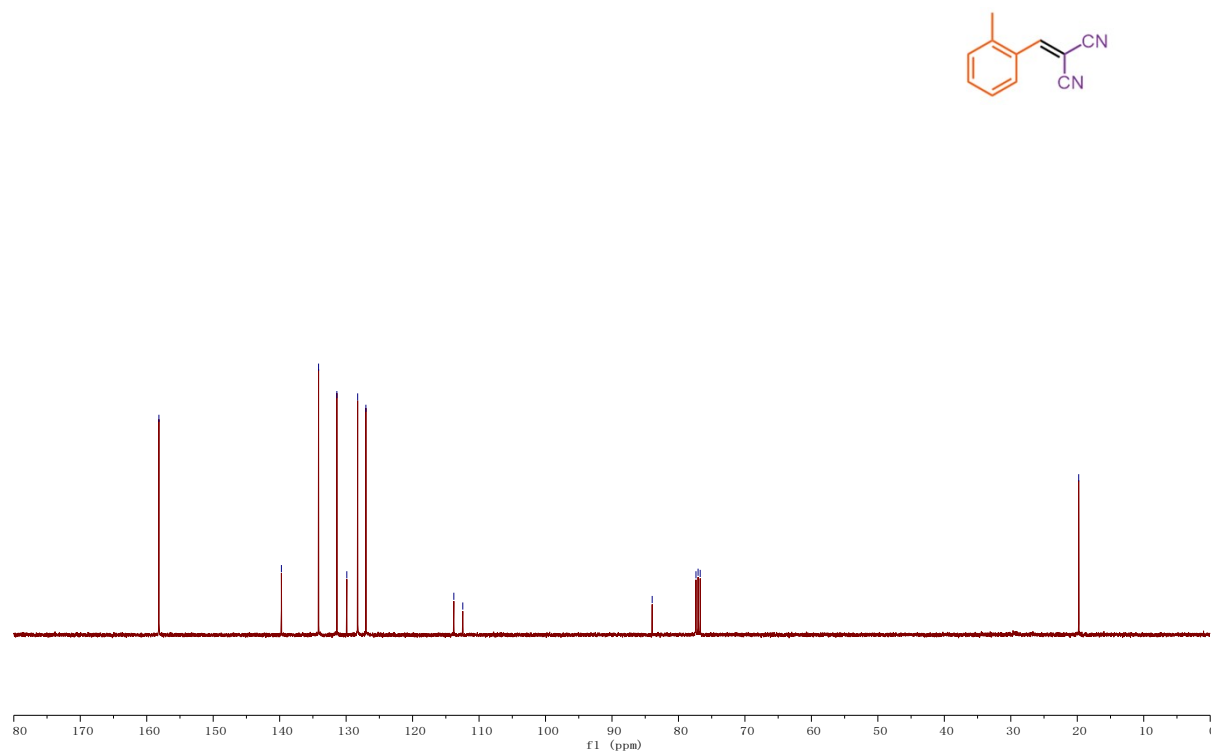
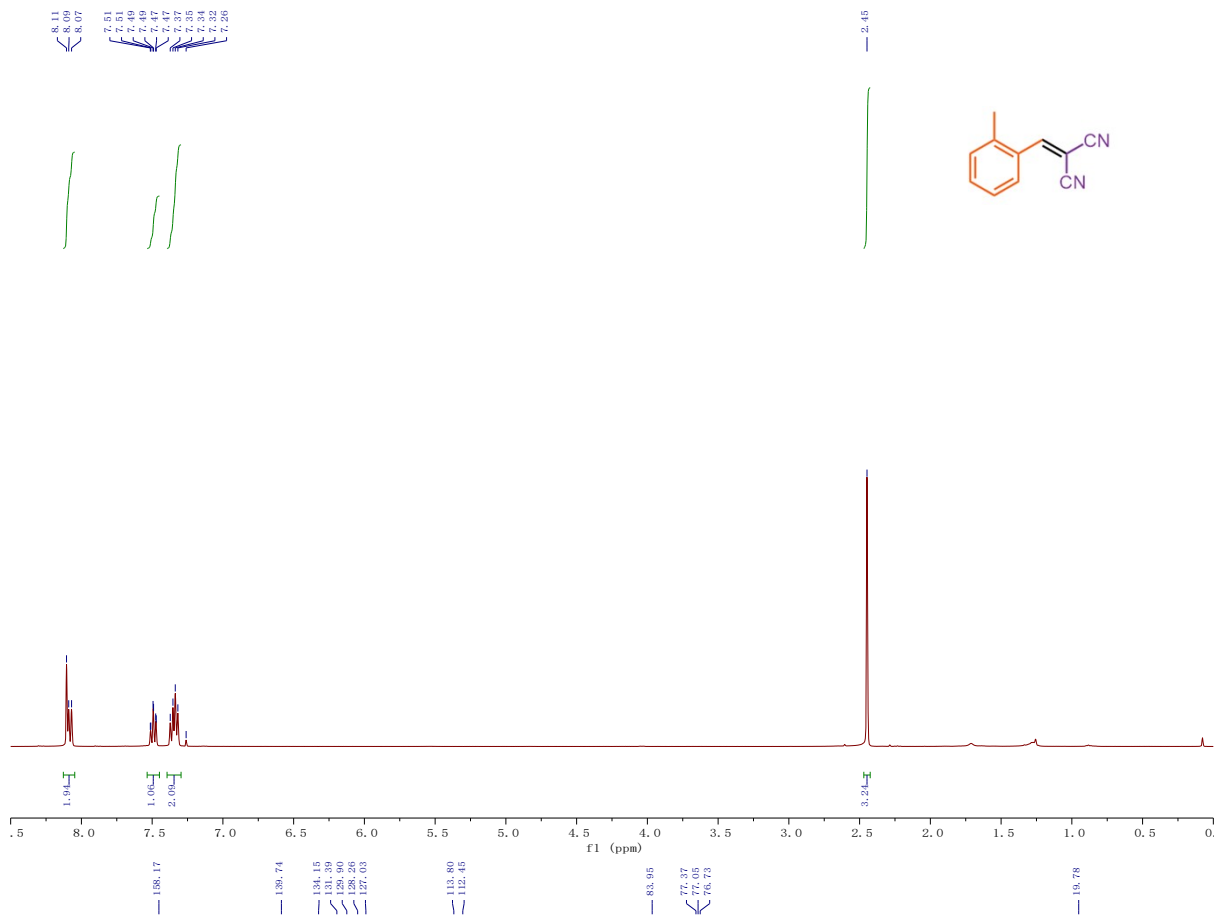
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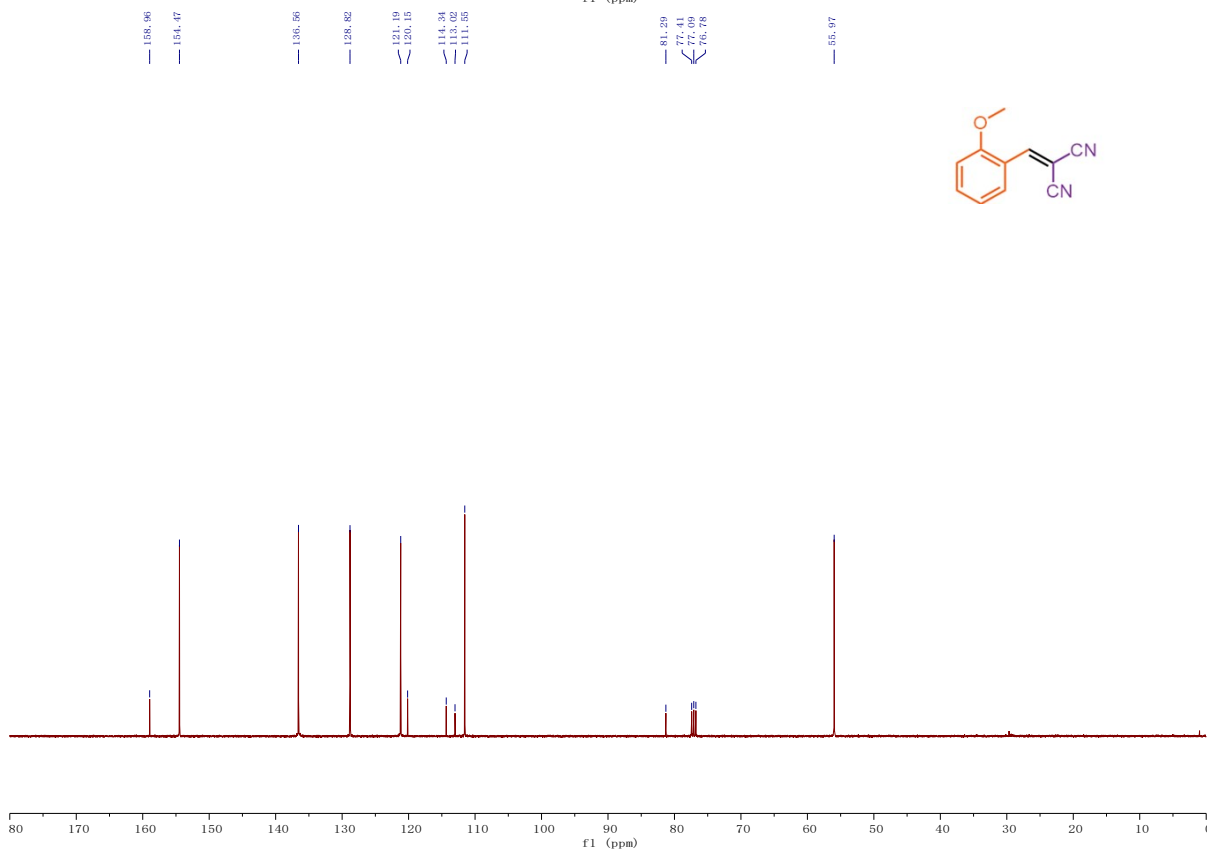
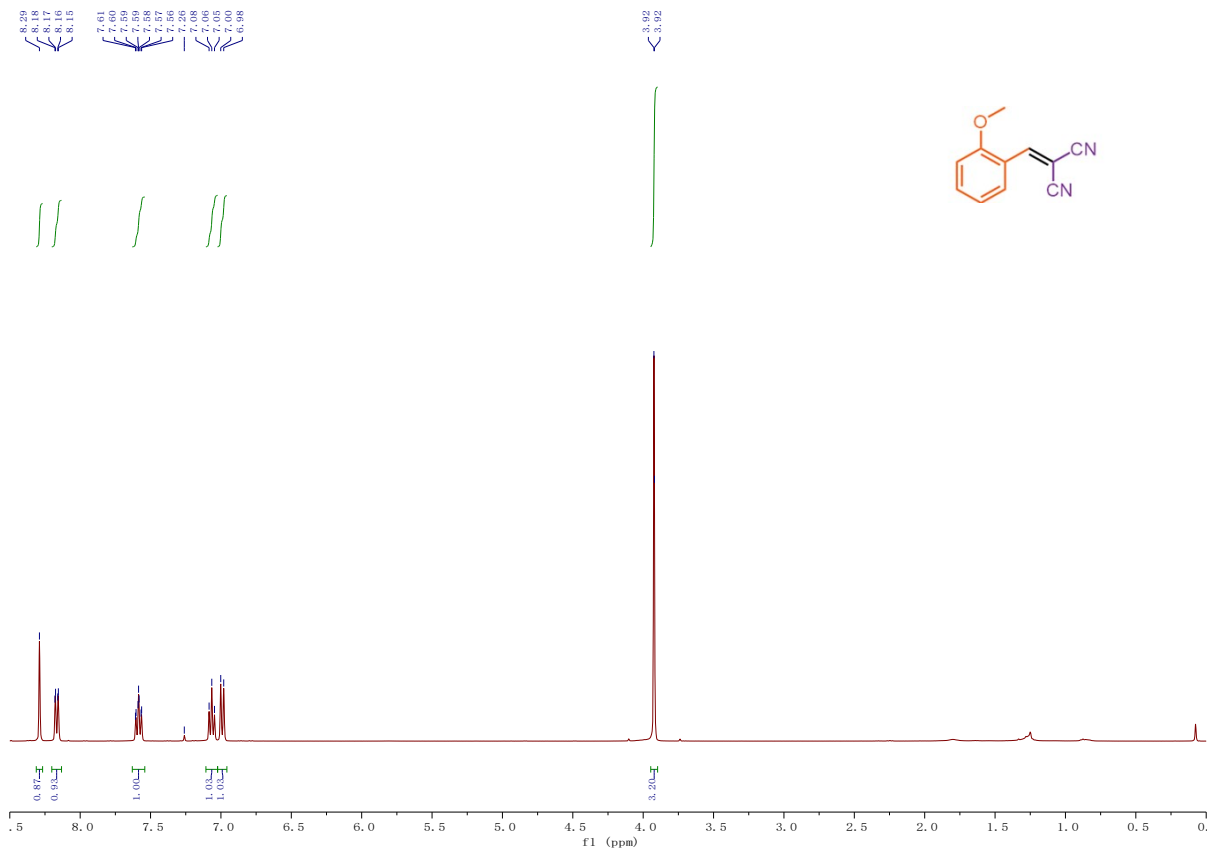


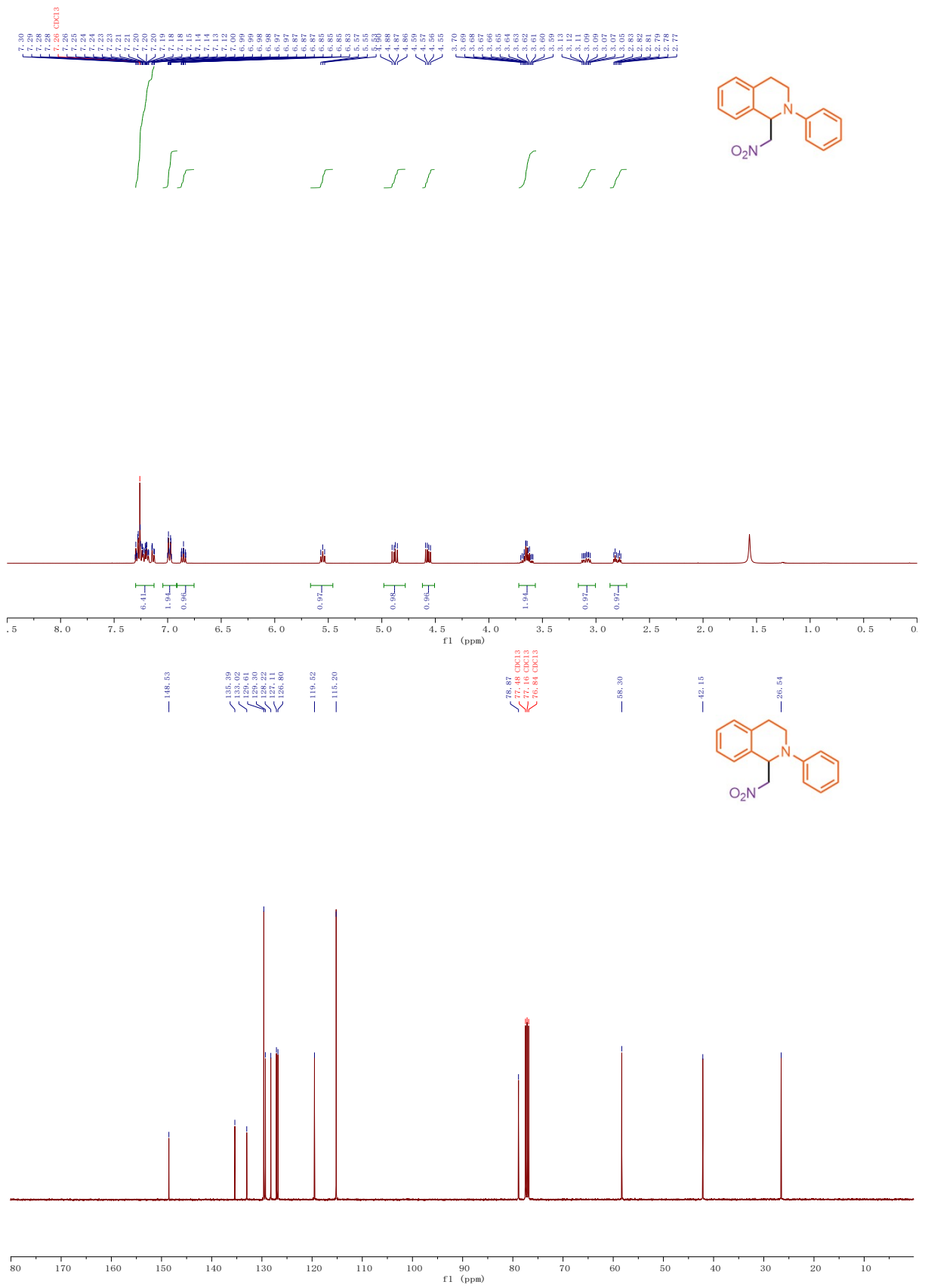


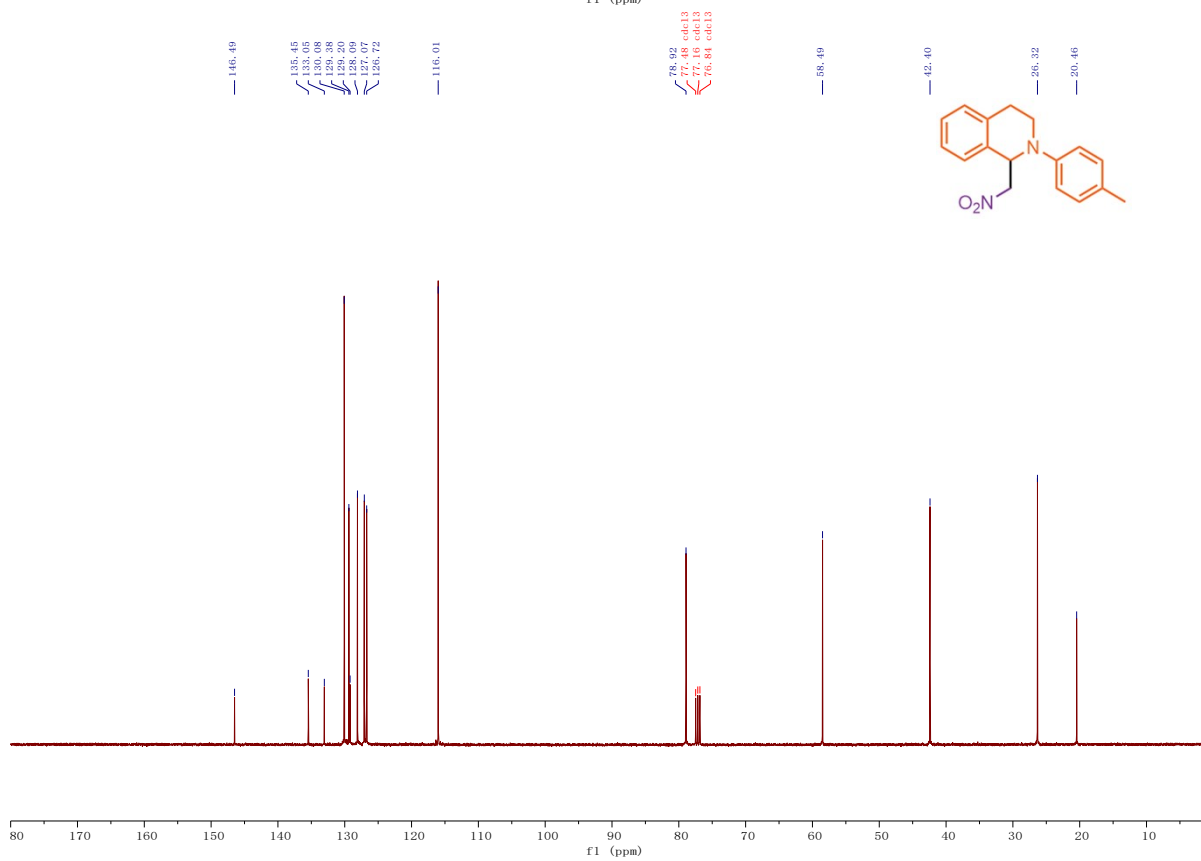
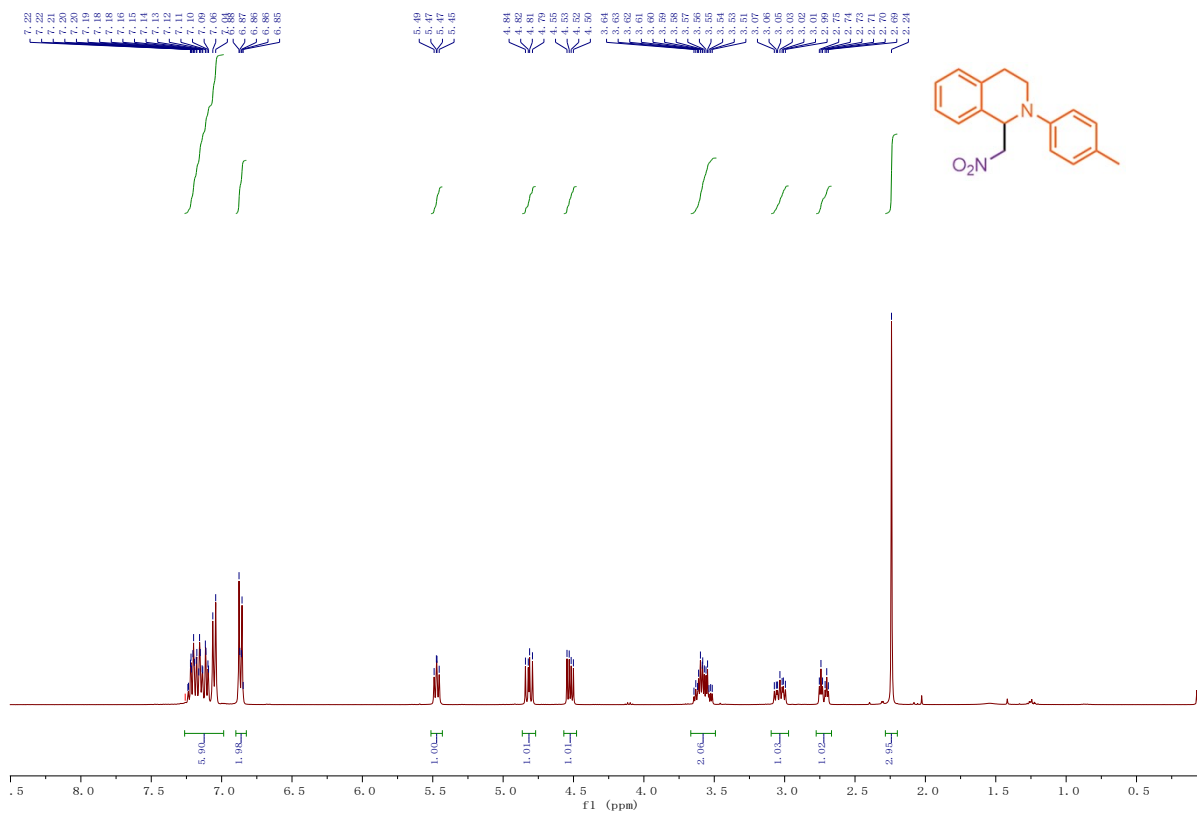


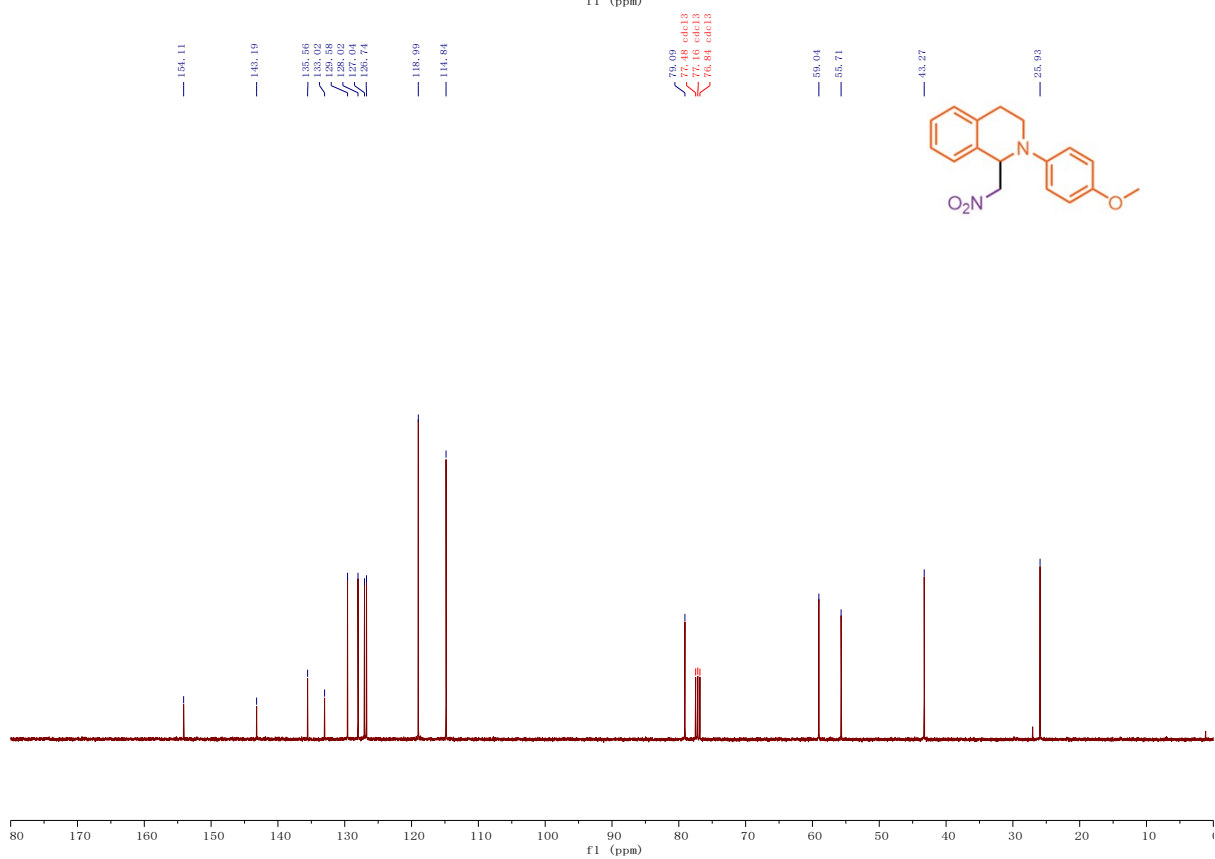
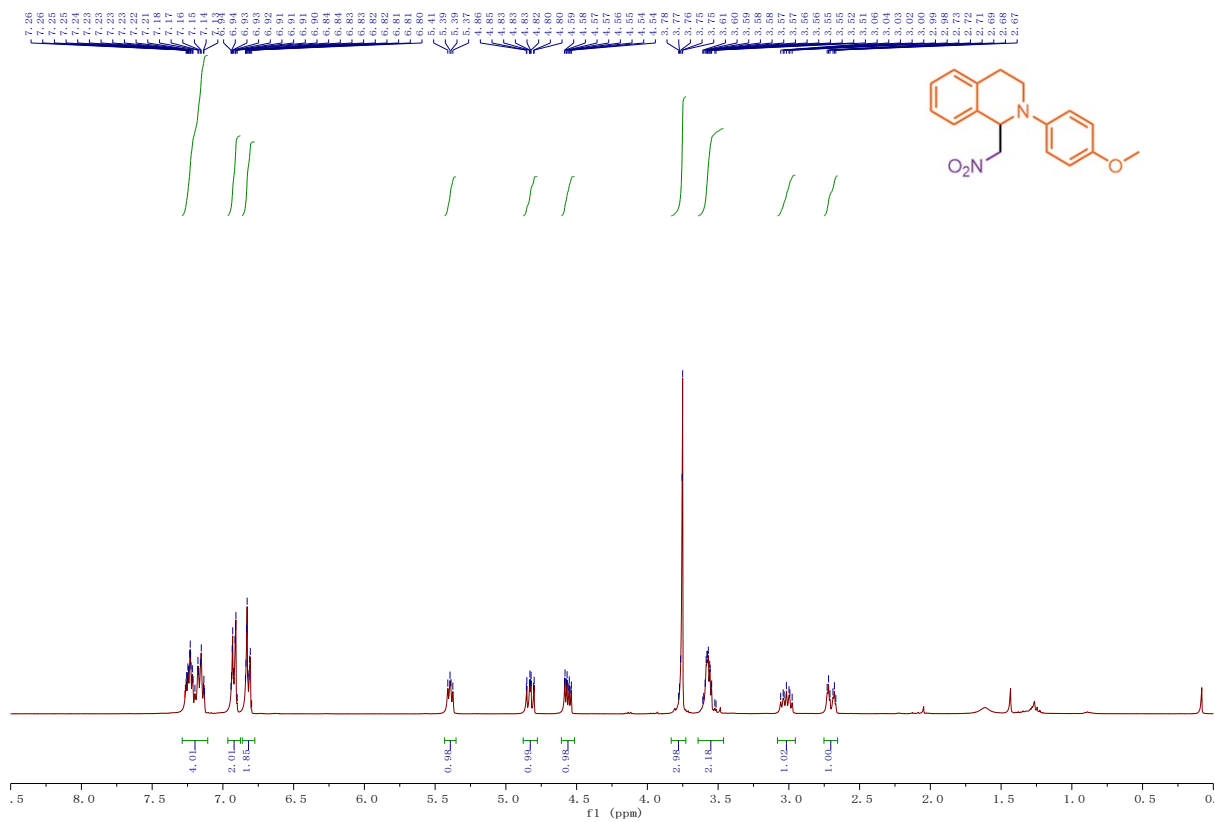


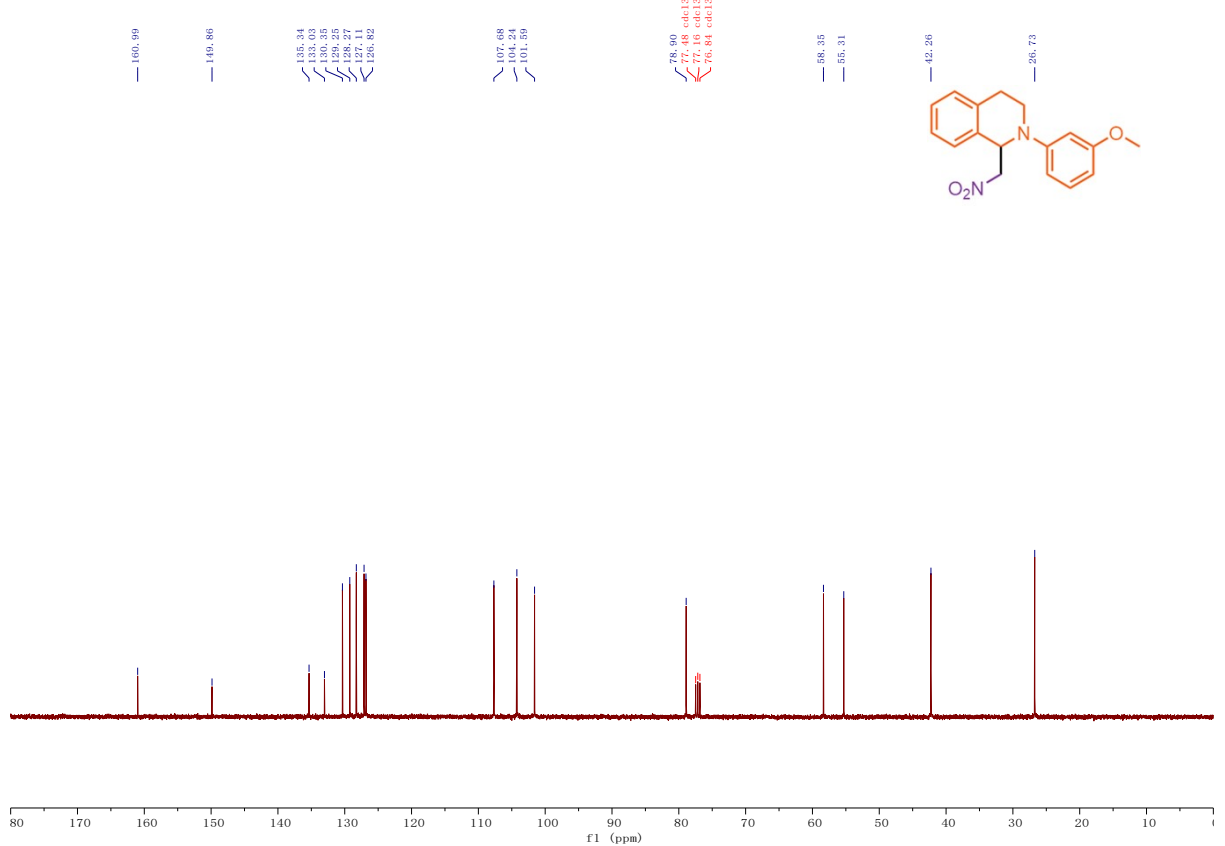
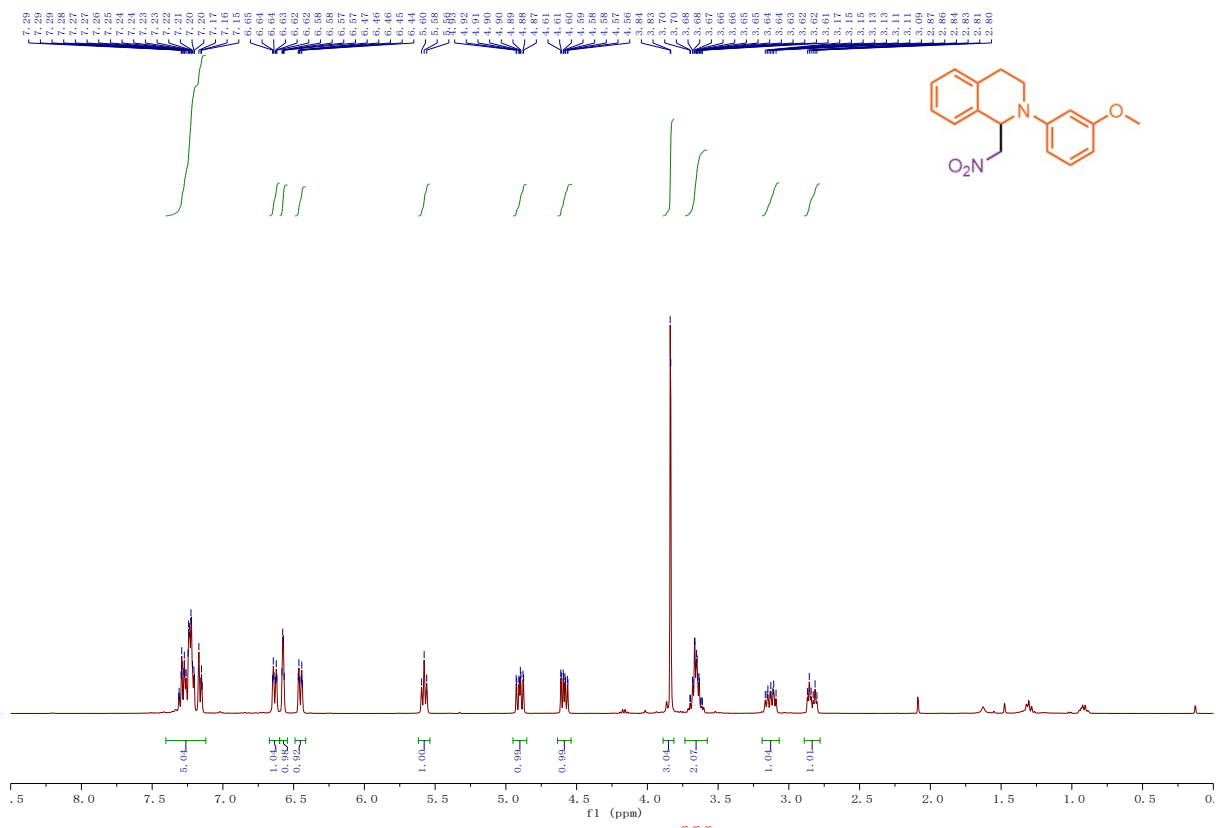


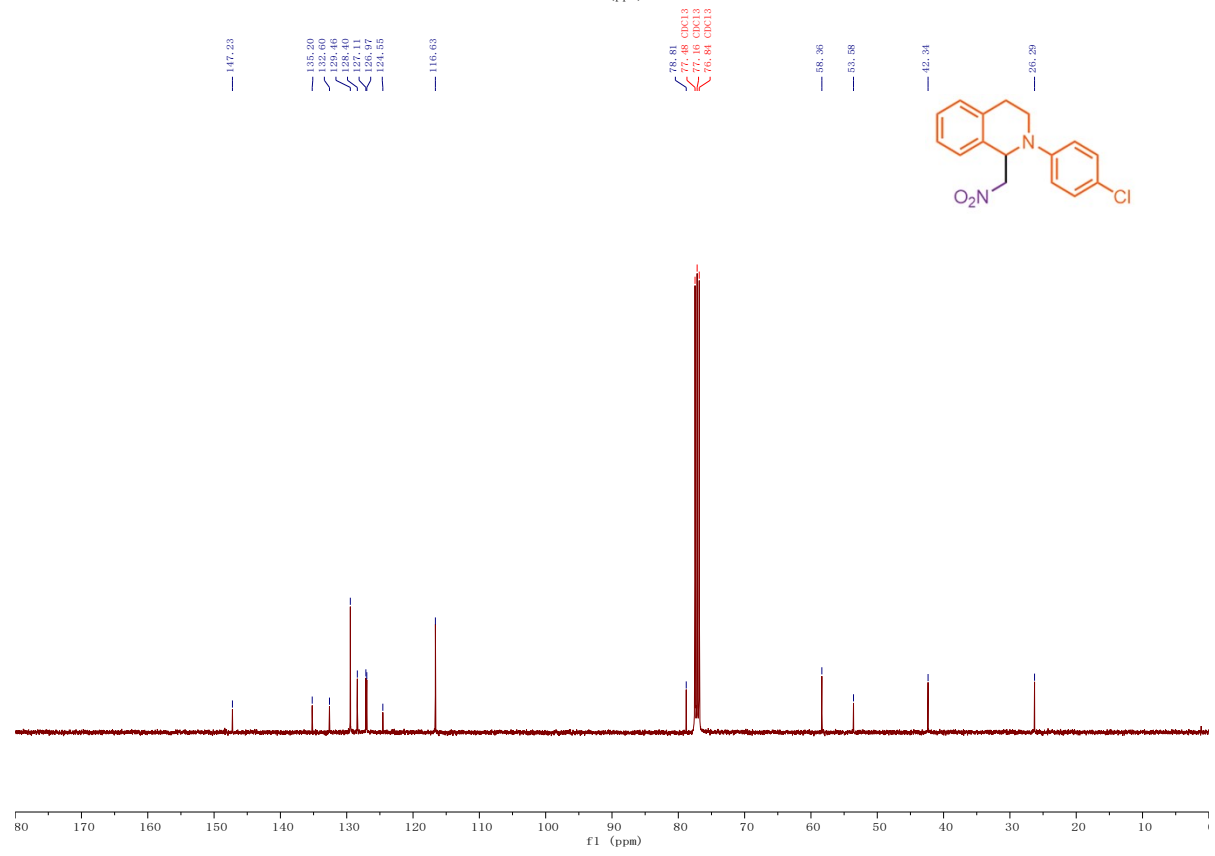
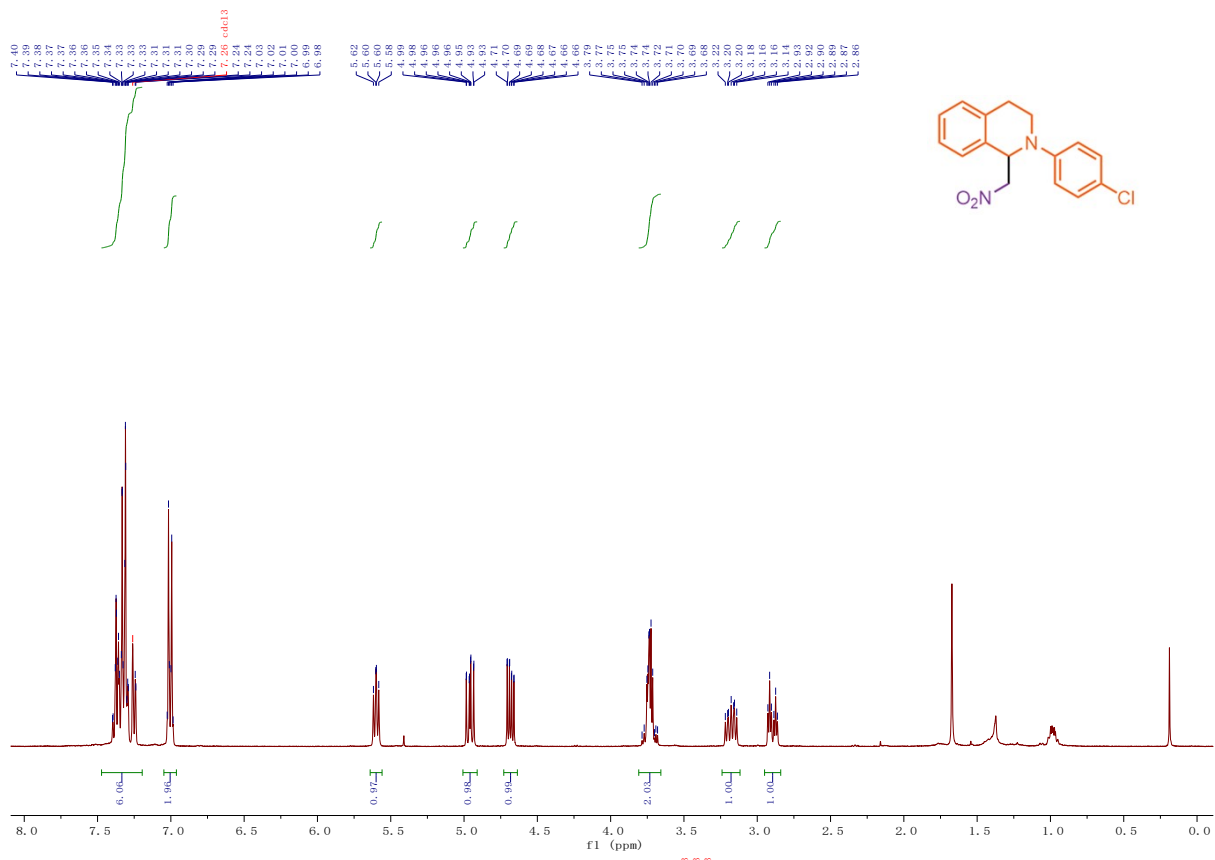


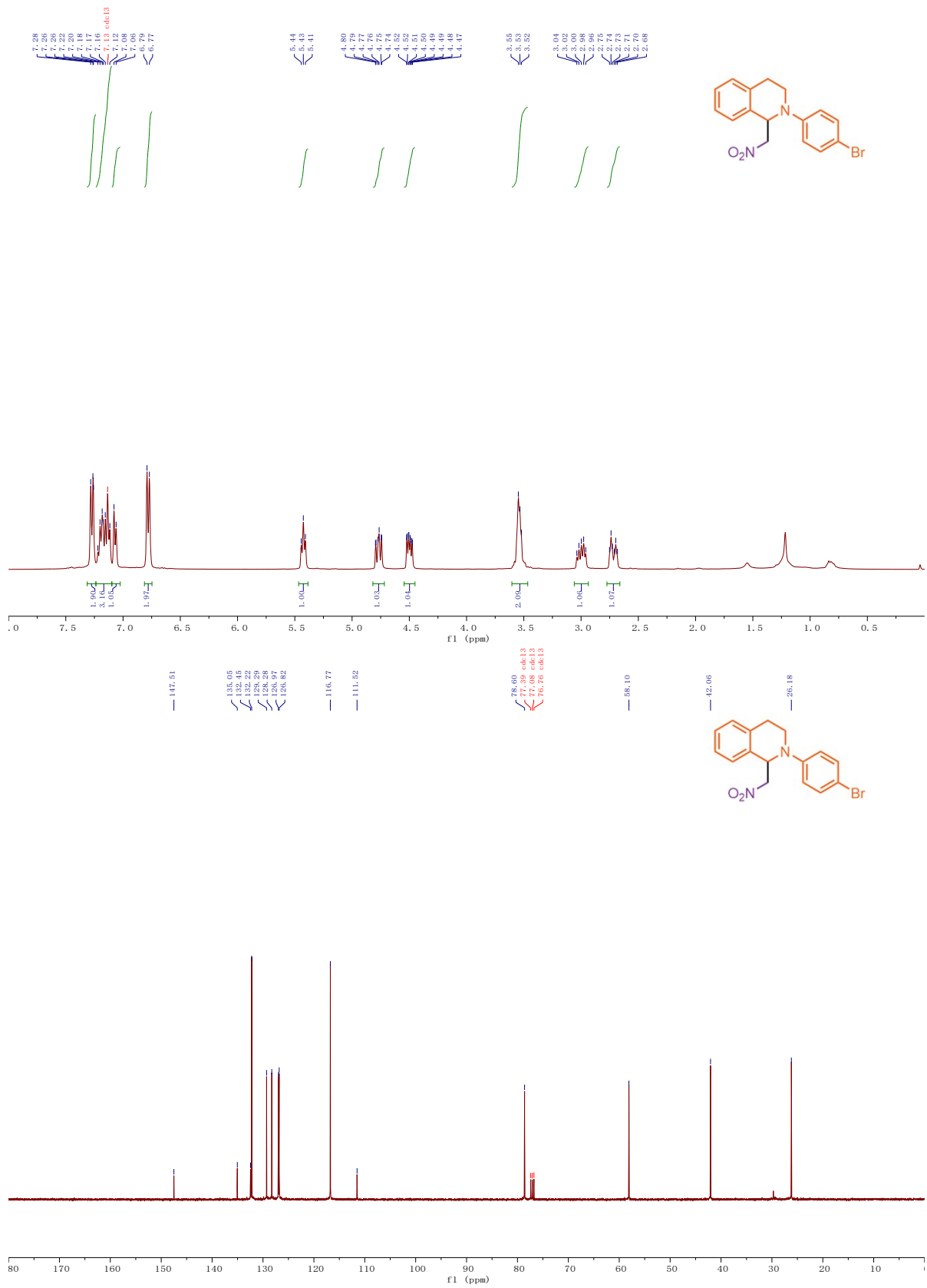


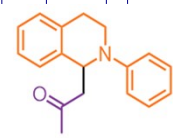
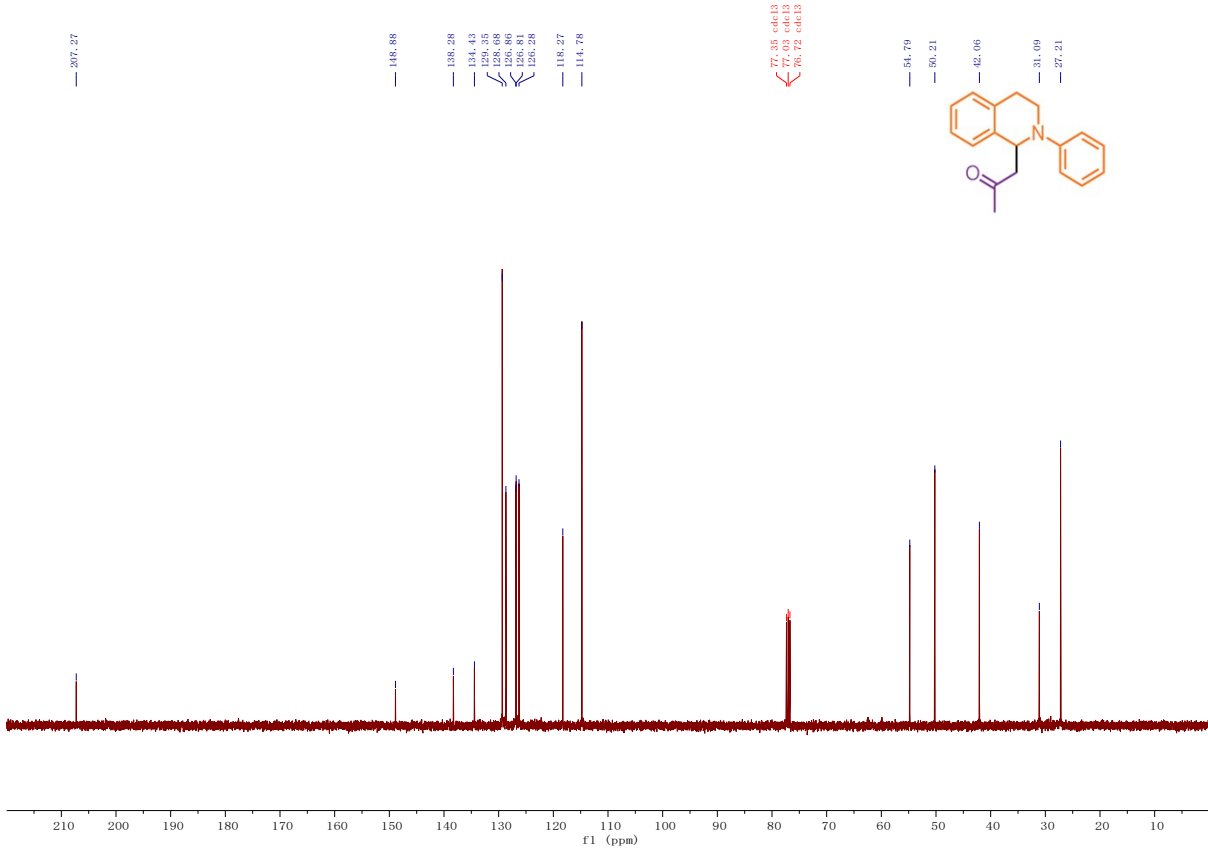
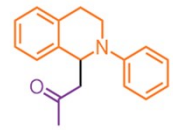
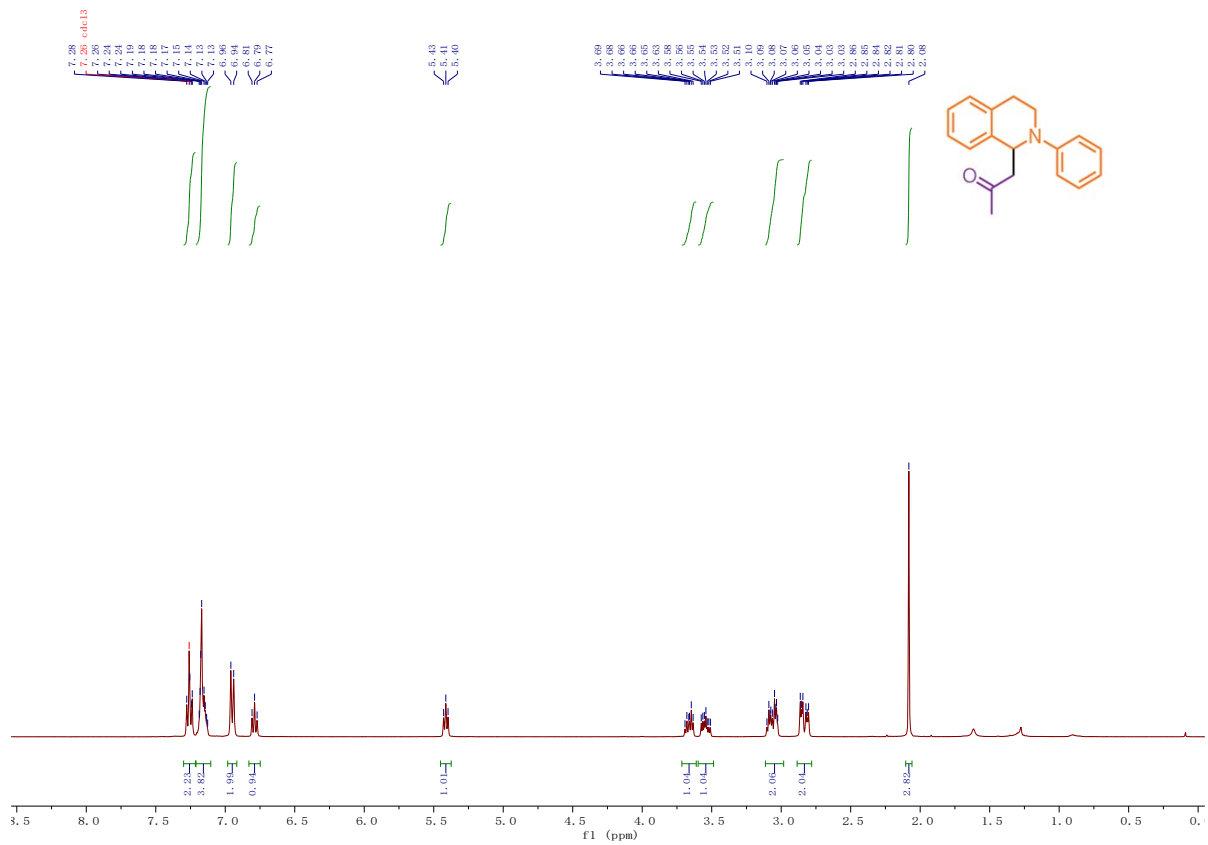


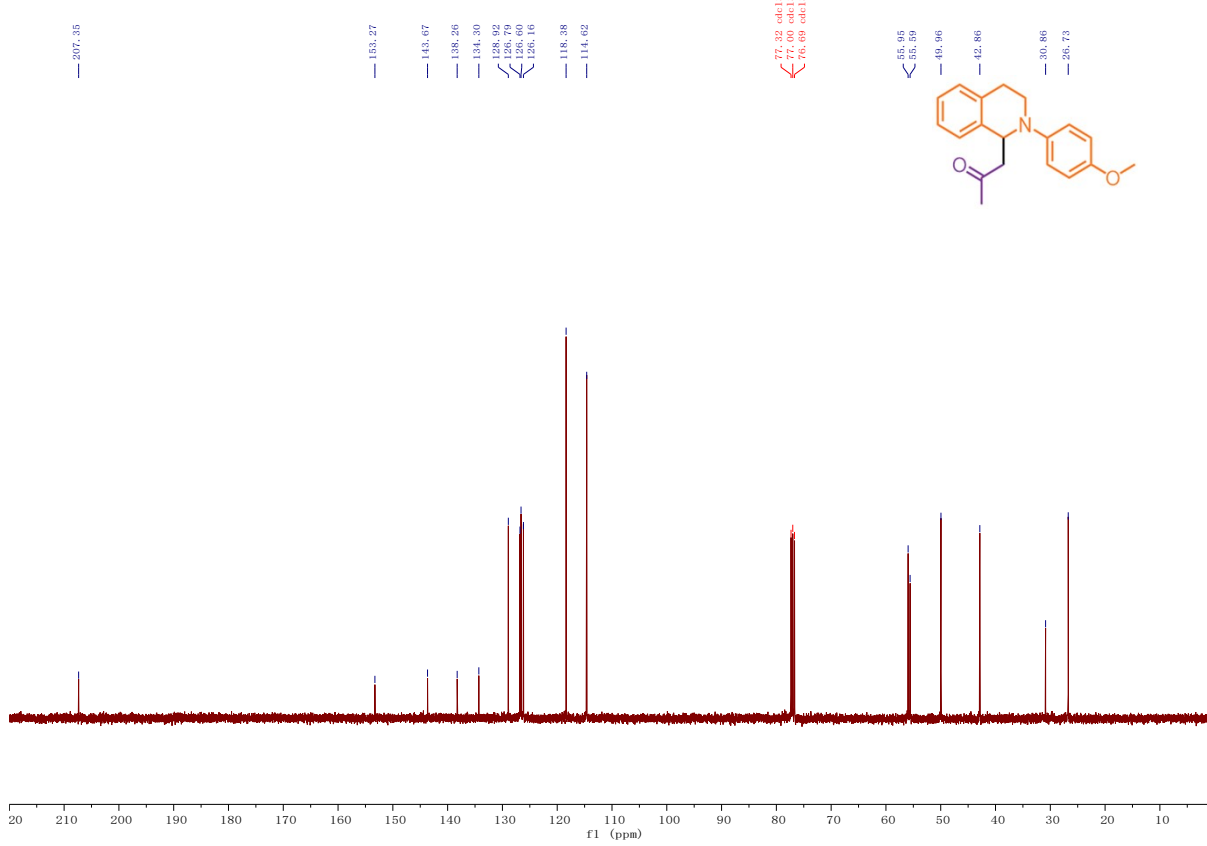
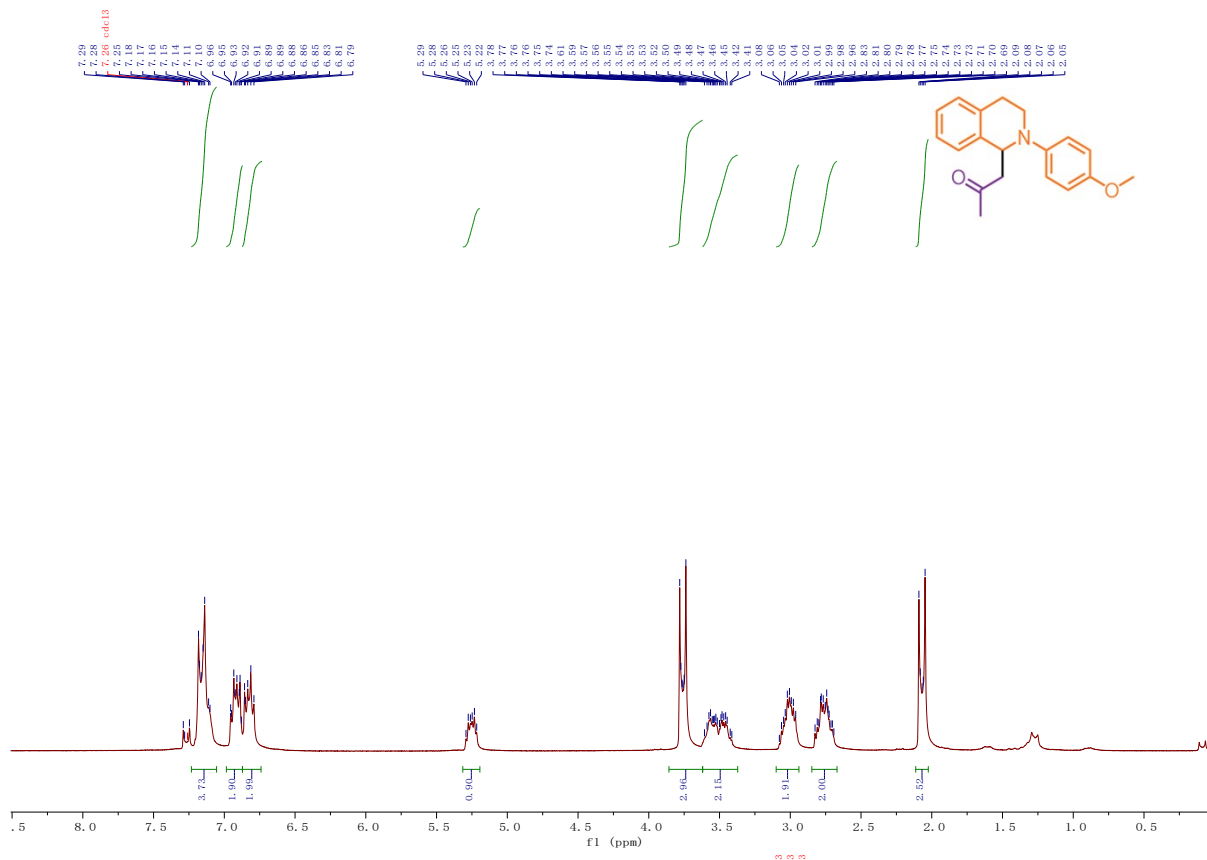


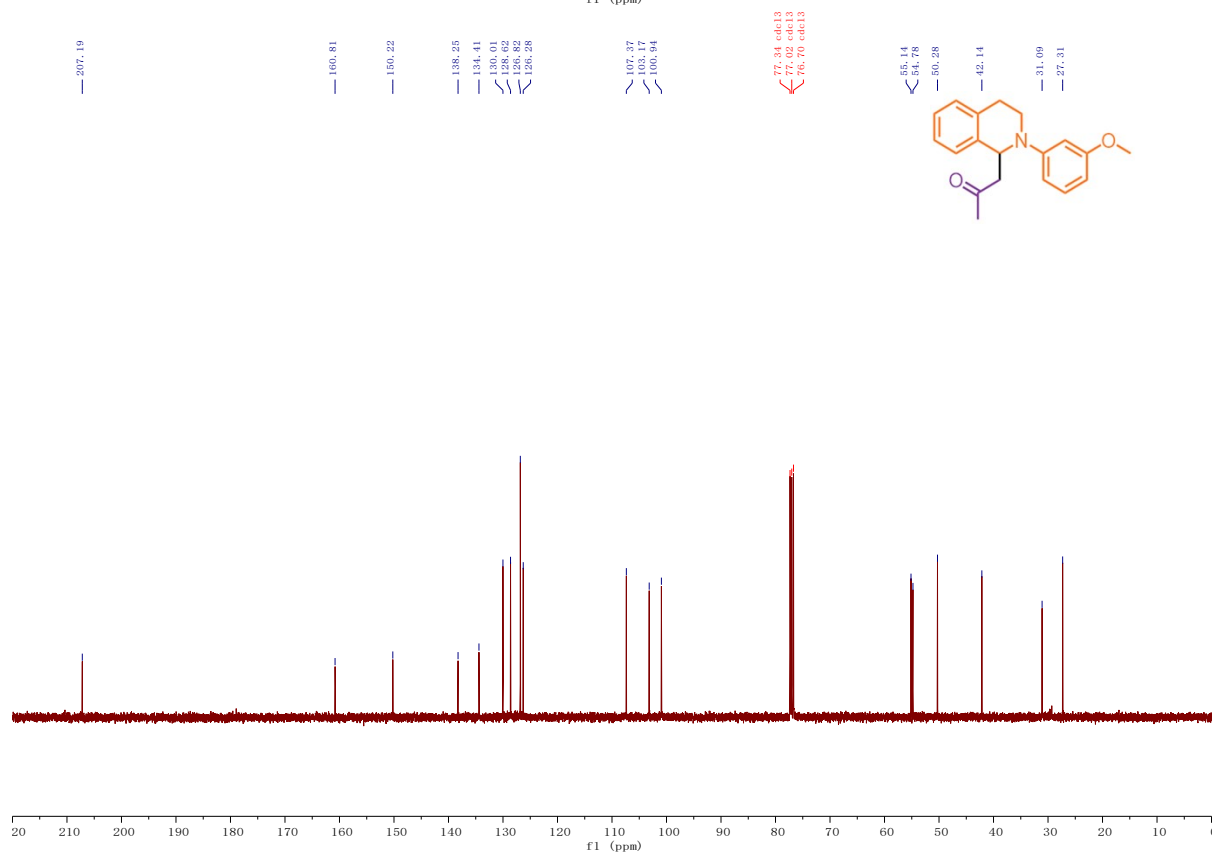
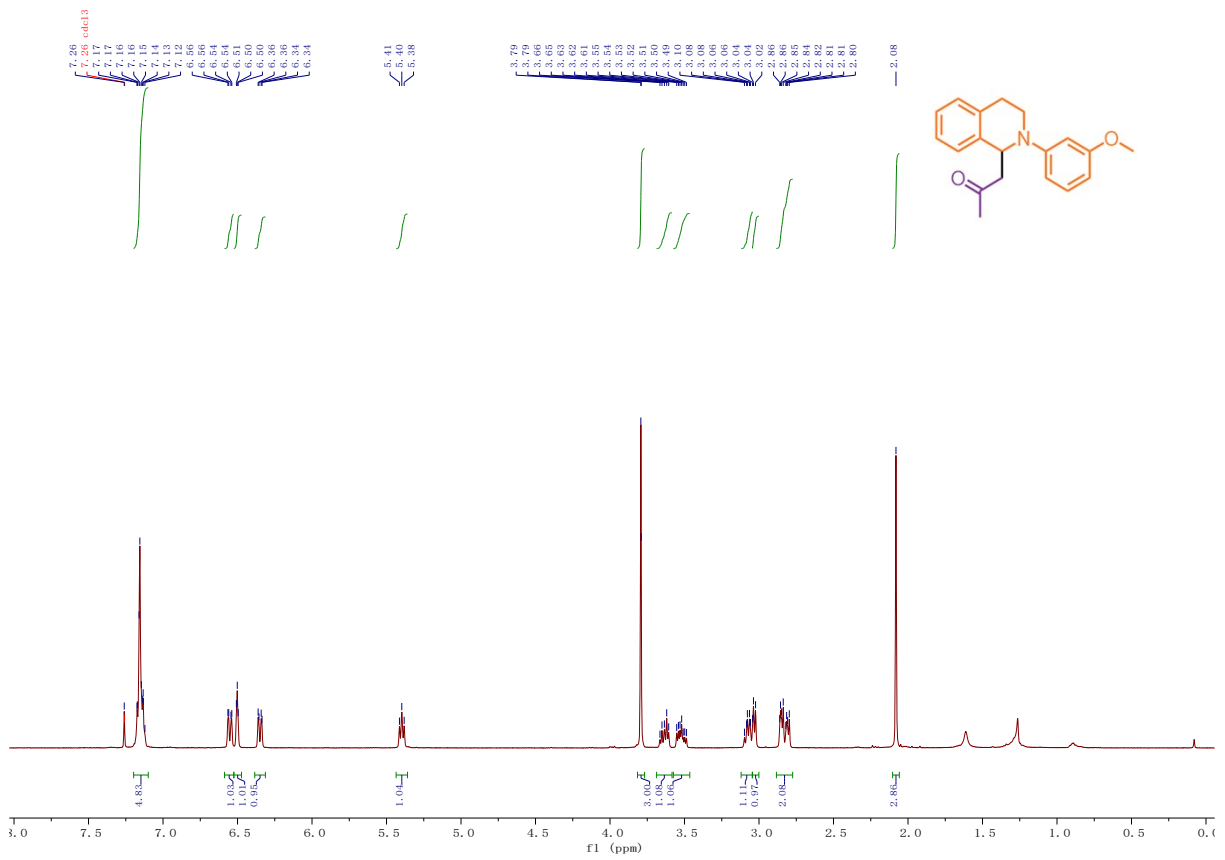


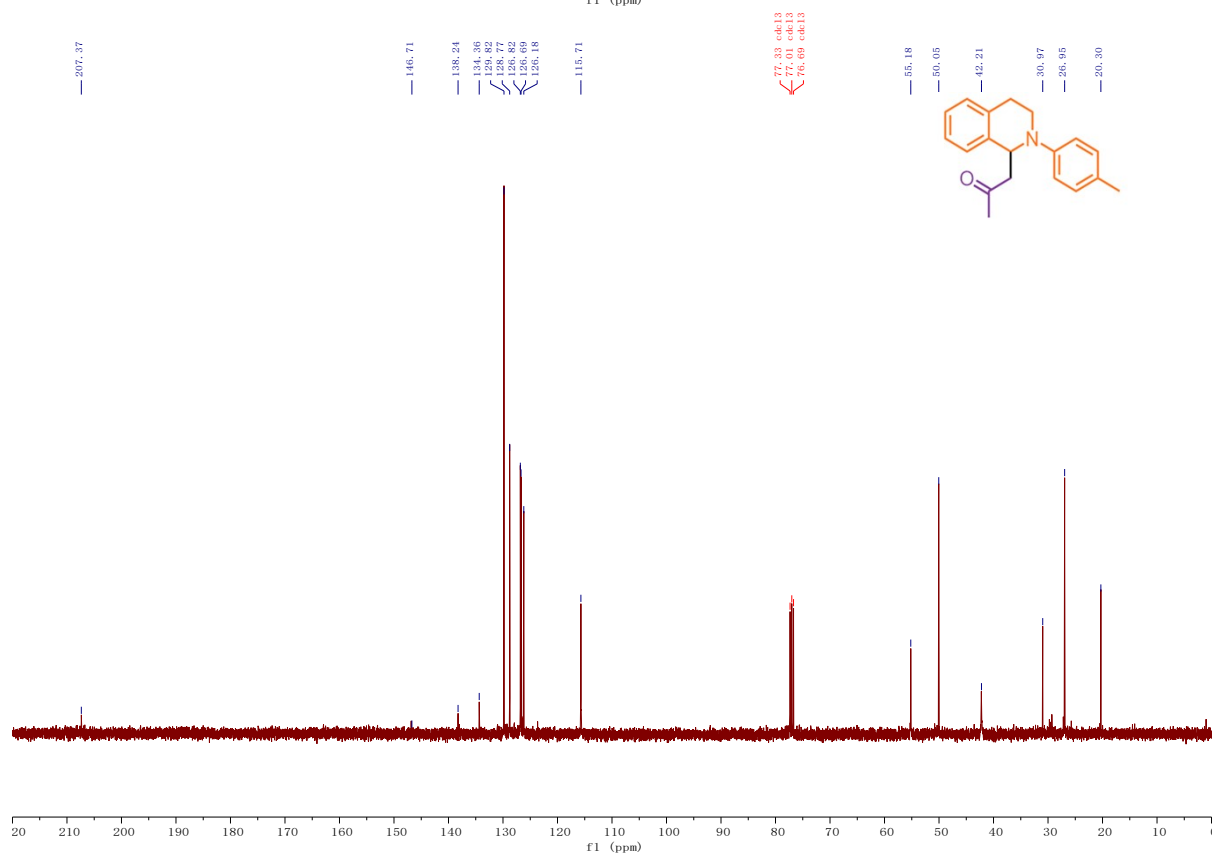
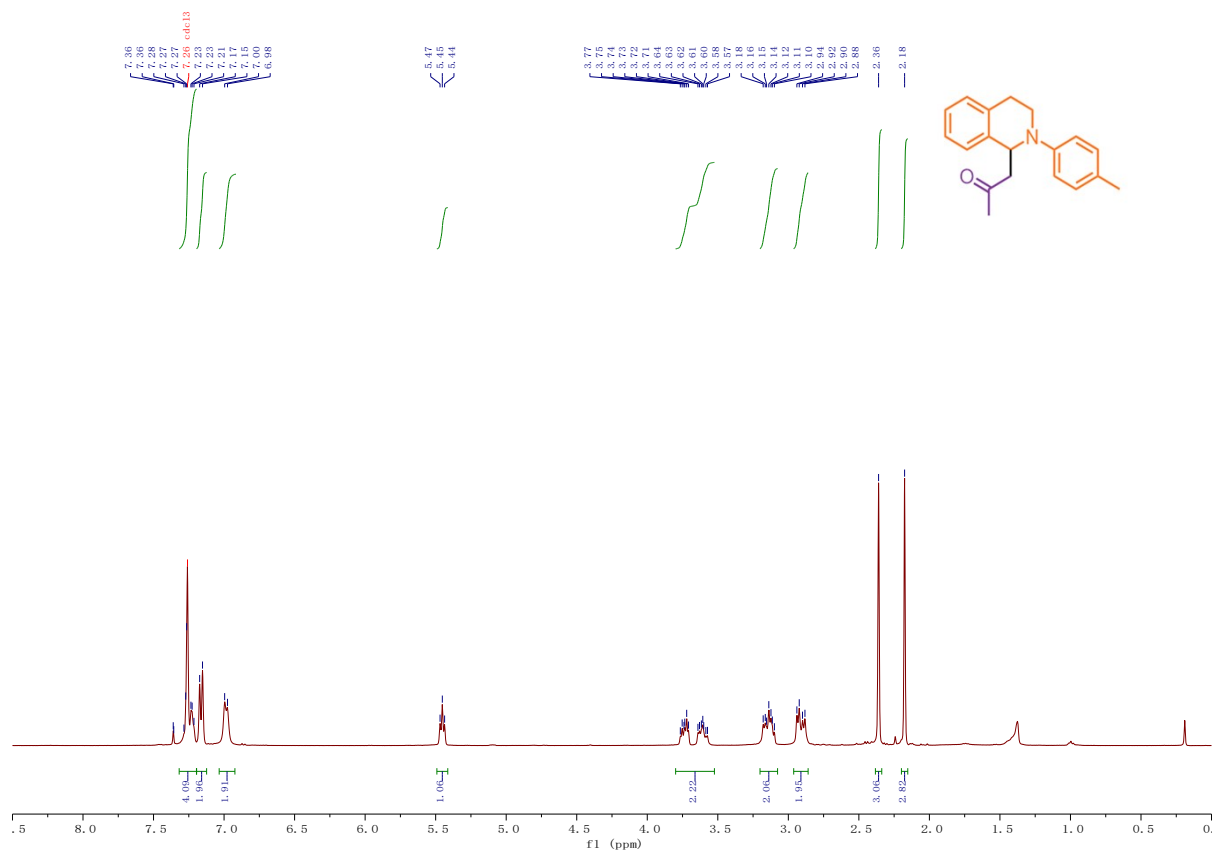


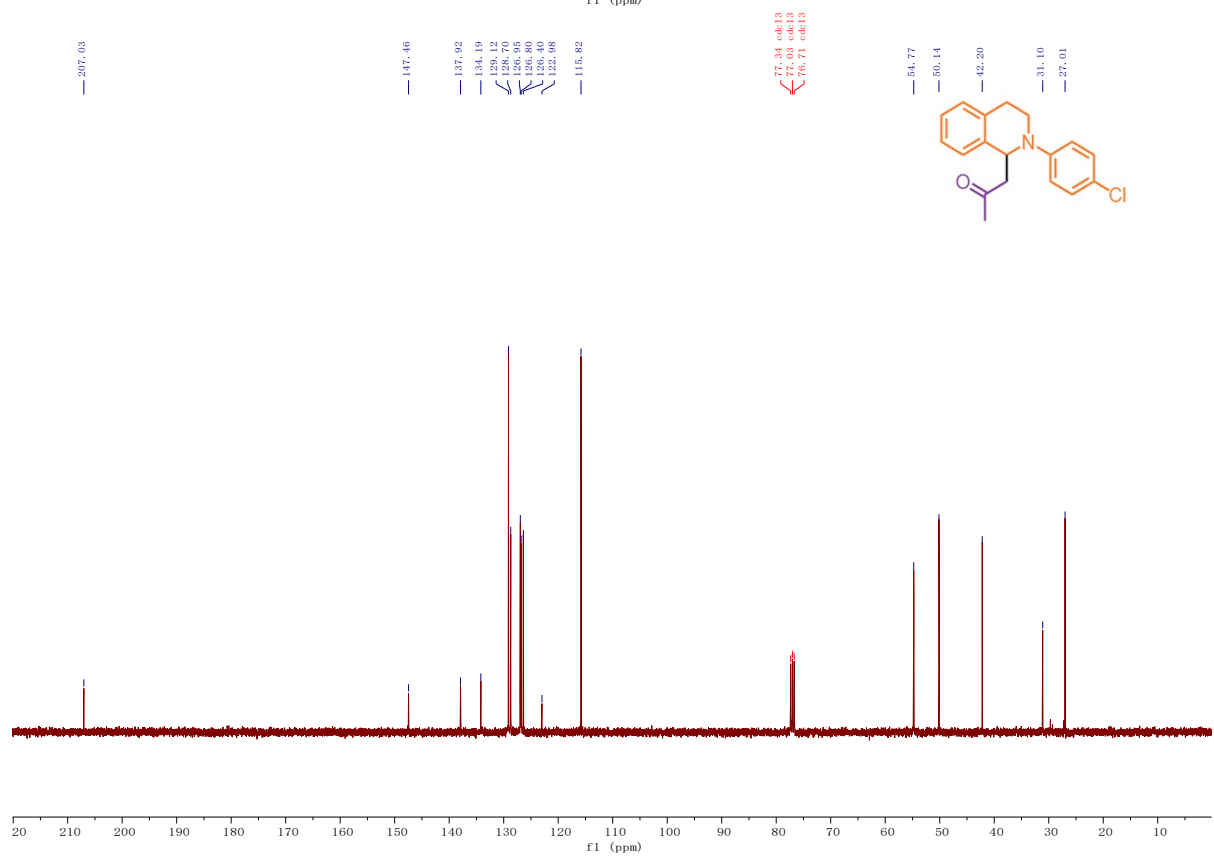
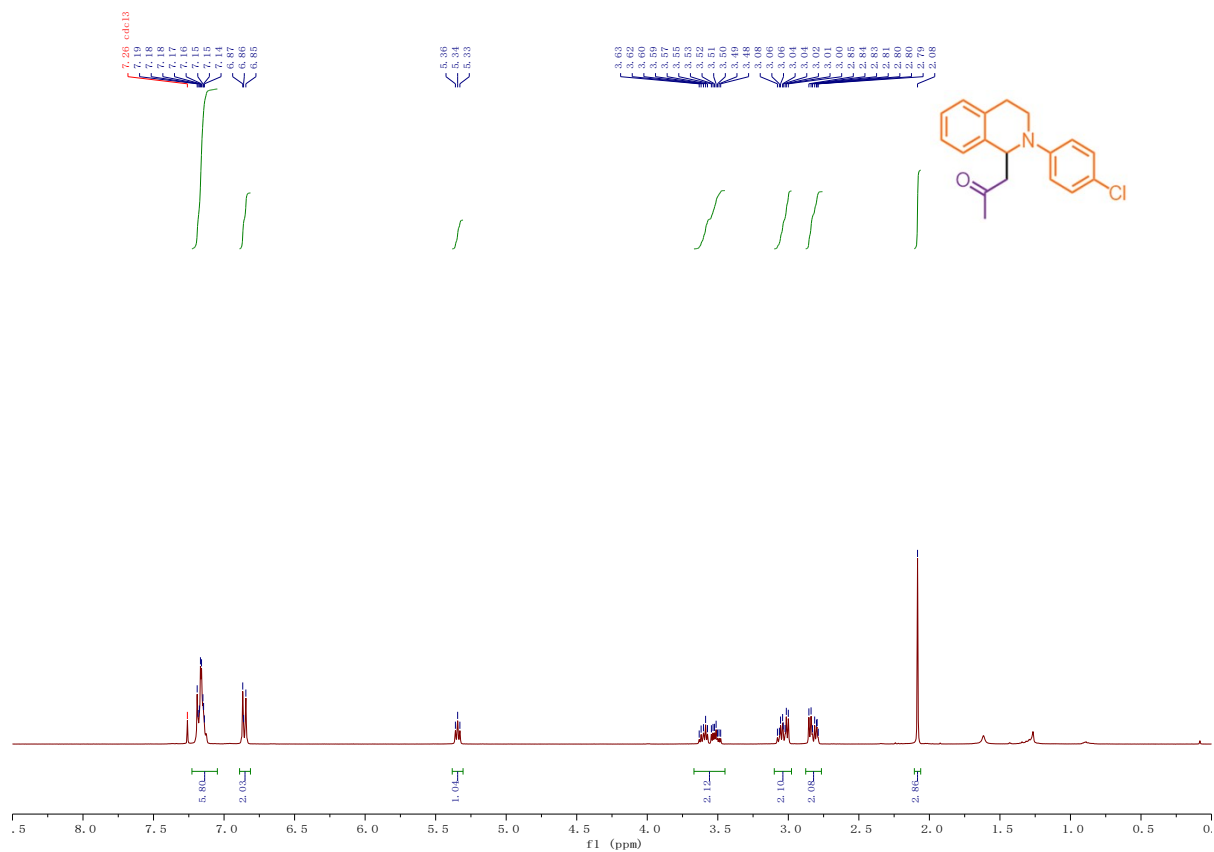


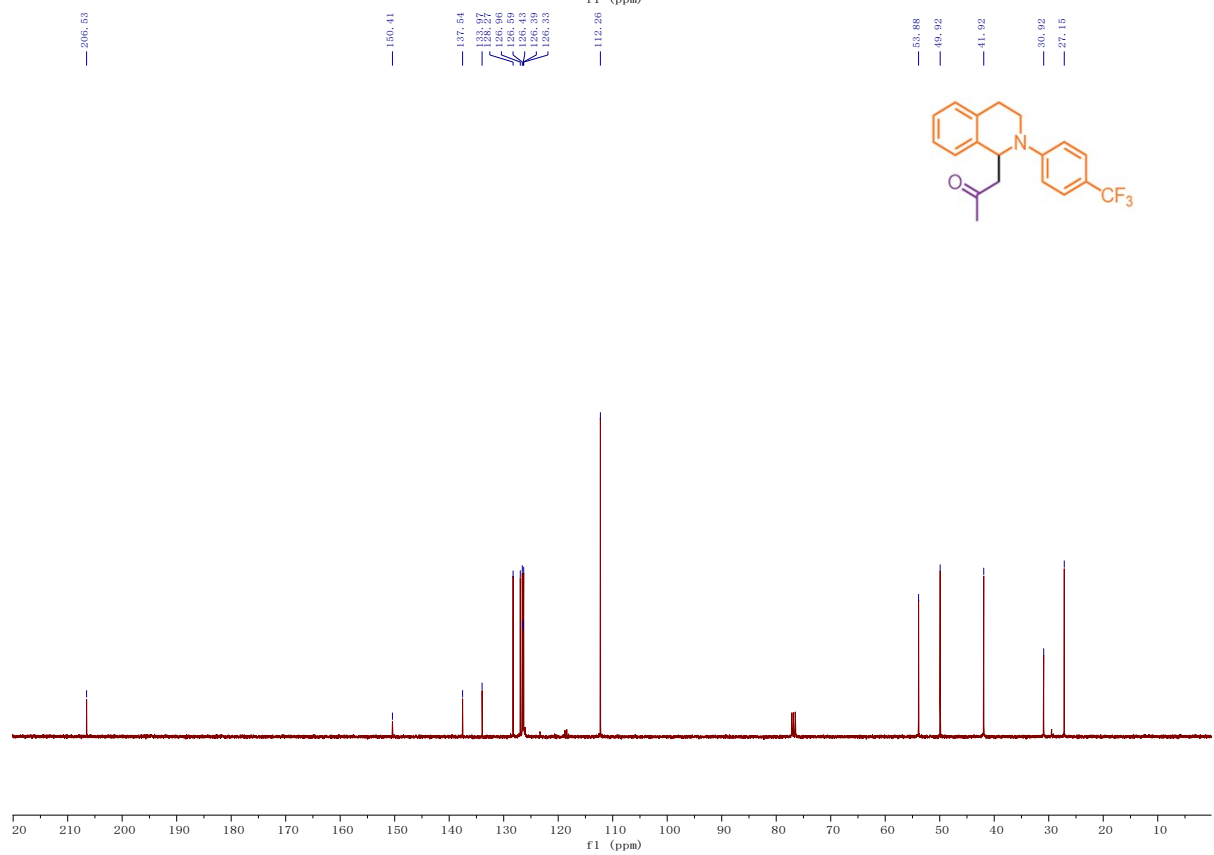
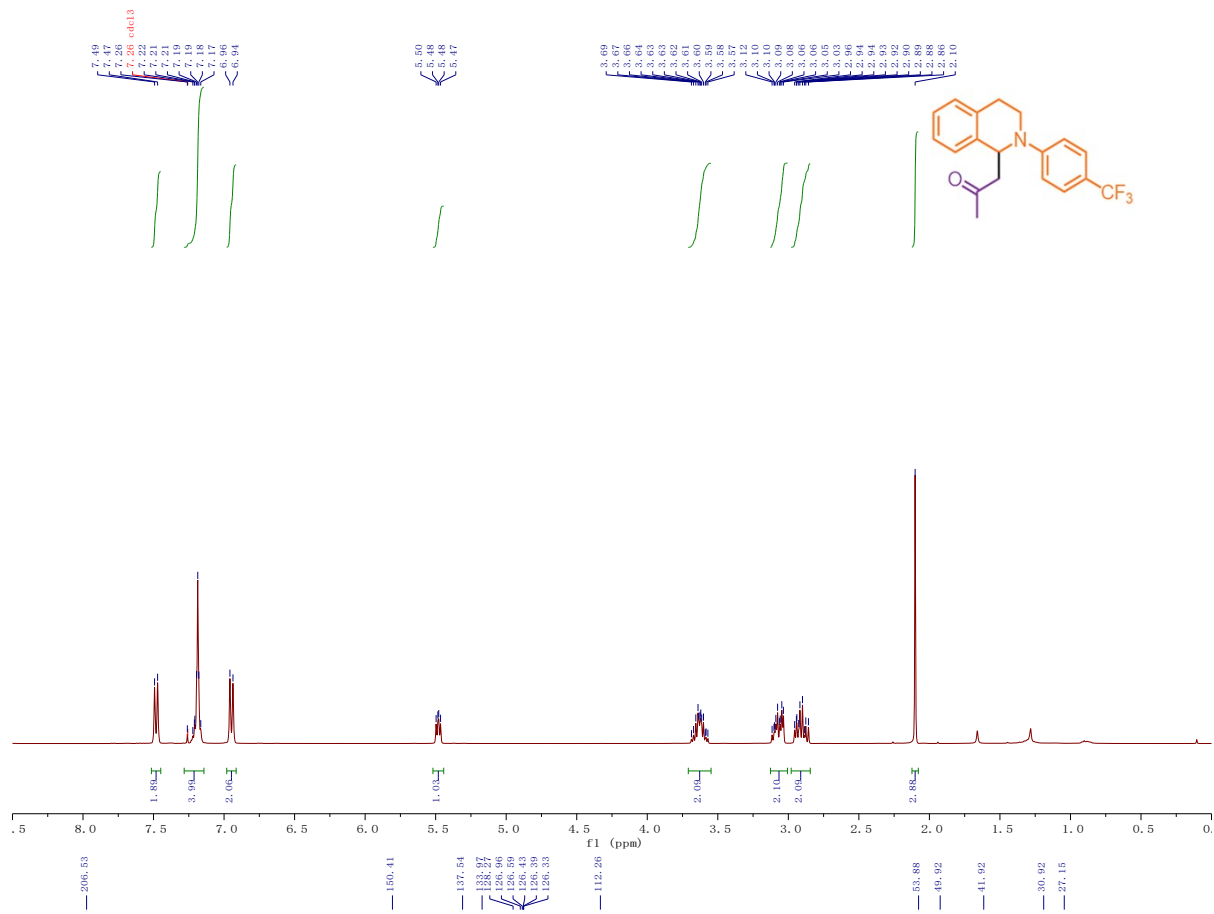












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