Supporting Information

Palladium-catalyzed domino Heck/ring opening of sulfolenes/desulfitative coupling: regio- and stereoselective synthesis of alkylated conjugated dienes

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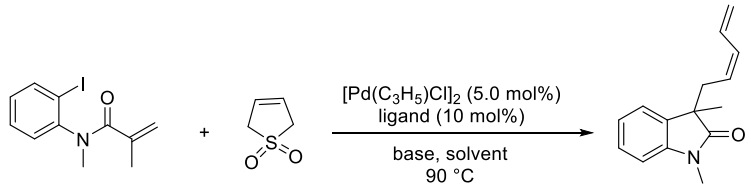
1. General considerations

All reactions were carried out under a nitrogen atmosphere. Materials were obtained from commercial suppliers or prepared according to standard procedures unless otherwise noted. Solvents were purified and dried according to standard methods prior to use. For product purification by flash column chromatography, silica gel (200~300 mesh) and light petroleum ether (bp. 60~90) are used. ^{1}H NMR spectra were recorded on a Bruker advance III 400 MHz in $CDCl_3$ and ^{13}C NMR spectra were recorded on 101 MHz in $CDCl_3$ using TMS as internal standard, Data for ^{1}H NMR are recorded as follows: chemical shift (δ, ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet or unresolved, br = broad singlet, coupling constant (s) in Hz, integration). Data for ^{13}C NMR is reported in terms of chemical shift (δ, ppm). High-resolution mass spectral analysis (HRMS) data were measured on a Bruker Apex II.

2. Preparation of substrates

Subatrates **1a**-**1i**[1],**1q**[2], **2a**-**2d**[3] were reported in the literatures.

3. Screening of palladium catalyst

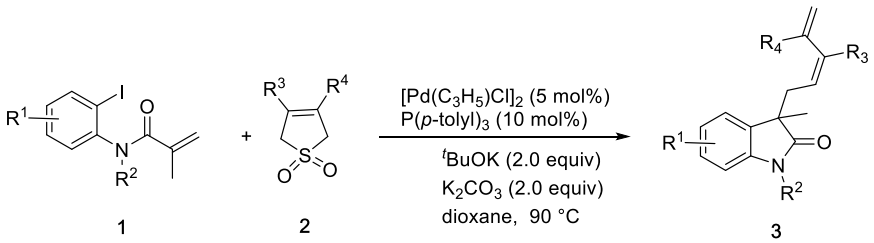


1a 2a 3a

entry	catalyst	ligand	base	solvent	yield (%)	Z/E Ratio
1	$Pd(OAc)_2$	P(p-tolyl)$_3$	^{t}BuOK/K_2CO_3	dioxane	41	>20:1
2	$Pd(TFA)_2$	P(p-tolyl)$_3$	^{t}BuOK/K_2CO_3	dioxane	29	18:1
3	$PdCl_2(PPh_3)_2$	P(p-tolyl)$_3$	^{t}BuOK/K_2CO_3	dioxane	46	>20:1
4	$Pd_2(dba)_3$	P(p-tolyl)$_3$	^{t}BuOK/K_2CO_3	dioxane	16	7:1
5	$[Pd(C_3H_5)Cl]_2$	P(p-tolyl)$_3$	^{t}BuOK/K_2CO_3	dioxane	71	>20:1

1a (0.2 mmol), **2a** (0.4 mmol), catalyst (5 mol%), ligand (10 mol%), base (0.4 mmol), solvent (2.0 mL, 0.1 M), 90 ℃, 12 h under argon atmosphere conditions.

4. Experiment procedure

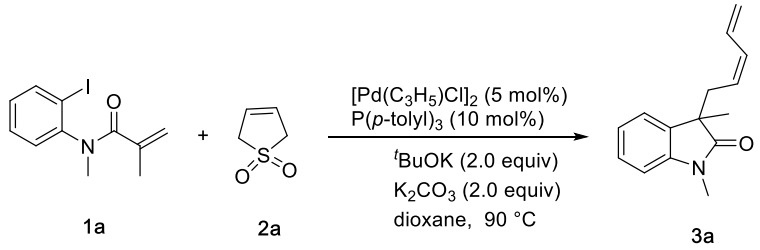


1 2 3

1 (0.2 mmol), **2** (0.4 mmol), $[Pd(C_3H_5)Cl]_2$ (5 mol%), P(*p*-tolyl)$_3$ (10 mol%), ^{t}BuOK (0.4 mmol), K_2CO_3 (0.4 mmol) were added to a sealed tube, dioxane (2.0 mL) were added via syringe. The mixture was flushed with N_2 and stirred at room temperature for 15 min firstly, and then was heated at 90 °C about for 12 h until completion (monitored by TLC). After

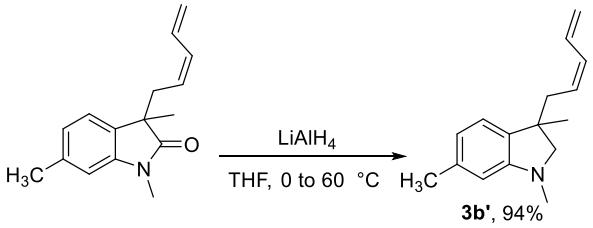
cooling at room temperature, the mixture was extracted with ethyl acetate, dried with anhydrous sodium sulfate and concentrated under reduced pressure. The residue was purified through silica gel chromatography to afford the products **3**.

5. Large-scale preparation of 3a



1a (2.0 mmol, 0.6 g), **2a** (4.0 mmol, 0.47 g), $[Pd(C_3H_5)Cl]_2$ (0.1 mmol, 36.4 mg), P(*p*-tolyl)$_3$ (0.2 mmol, 60.8 mg), ^{t}BuOK (4.0 mmol, 0.45 g), K_2CO_3 (4.0 mmol, 0.55 g) were added to a sealed tube, dioxane (40.0 mL) were added via syringe. The mixture was flushed with N_2 and stirred at room temperature for 15 min firstly, and then was heated at 90 °C about for 12 h until completion (monitored by TLC). After cooling at room temperature, the mixture was extracted with ethyl acetate, dried over anhydrous sodium sulfate and concentrated under reduced pressure. The residue was purified by silica gel chromatography to afford the product **3a** (0.29 g, 64%).

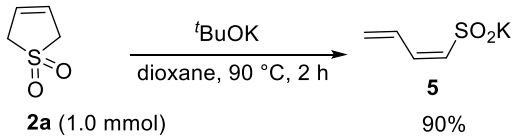
6. Synthetic transformation of 3b



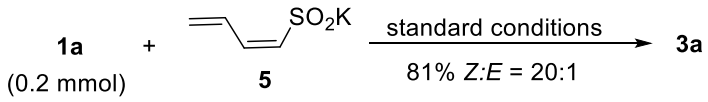
To a solution of **3b** (0.2 mmol) in 2.0 mL of THF was added a solution of $LiAlH_4$ (3.0 equiv.) in THF at 0 ºC. The ice bath was removed and the reaction was allowed to stir for about 3 h at 60 ºC. The reaction mixture was diluted with ice water (5.0 mL) and extracted with EtOAc (10 mL). The combined organic layers were washed with brine, dried over anhydrous sodium sulfate, and concentrated under reduced pressure. The crude product was purified by a silica gel column chromatography to afford **3b'** as the product.

3b': colorless oil; *Z:E* = 10:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.89 (d, *J* = 7.4 Hz, 1H), 6.67-6.55 (m, 1H), 6.54-6.49 (m, 1H), 6.31 (s, 1H), 6.11 (t, *J* = 11.1 Hz, 1H), 5.47 (dt, *J* = 10.9, 8.0 Hz, 1H), 5.19 (dd, *J* = 16.9, 2.0 Hz, 1H), 5.09 (dt, *J* = 10.0, 1.9 Hz, 1H), 3.18 (d, *J* = 8.7 Hz, 1H), 2.93 (d, *J* = 8.7 Hz, 1H), 2.72 (s, 3H), 2.46 (dd, *J* = 8.0, 1.5 Hz, 2H), 2.30 (s, 3H), 1.28 (d, *J* = 1.6 Hz, 3H). ^{13}C NMR (101 MHz, CDCl3) δ 152.5, 137.6, 135.0, 132.3, 131.5, 128.7, 122.0, 118.4, 117.5, 108.4, 68.0, 43.8, 37.8, 35.9, 24.9, 21.8. HRMS (ESI) calcd for $C_{16}H_{22}N$ $[M+H]^+$: 228.1747, found: 228.1750.

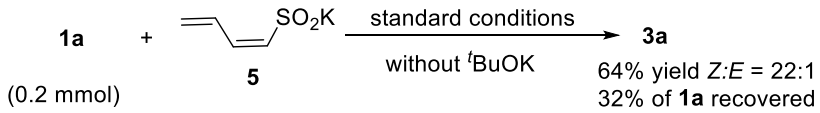
7. Mechanistic studies



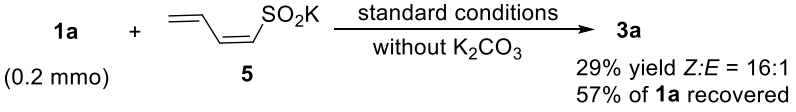
A solution of **2a** (118 mg, 1.0 mmol) in 10 mL of dioxane at 90 ºC was stirred. Then a mixture of ^{t}BuOK (224 mg, 2.0 mmol) in 4 mL of dioxane was added dropwise. During the addition, a mustard-yellow precipitate was observed. After stirring 2 h, the solid went to a pale yellow color. The solution was evaporated under vacuum and the residue was washed five times with dioxane (10 ml) and dried under vacuum to afford the potassium sulfinate **5** in 90 yield. ^{1}H NMR (400 MHz, D_2O) δ 7.03 (dddd, J = 16.8, 11.2, 10.1, 1.1 Hz, 1H), 6.46 (ddd, J = 11.3, 10.2, 0.8 Hz, 1H), 6.06-5.84 (m, 1H), 5.54-5.24 (m, 2H). ^{13}C NMR (101 MHz, D_2O) δ 144.3, 133.8, 131.1, 123.2. The results are consistent with the previous literature.[4]



1 (0.2 mmol), **5** (0.4 mmol), $[Pd(C_3H_5)Cl]_2$ (5 mol%), P(*p*-tolyl)$_3$ (10 mol%), ^{t}BuOK (0.4 mmol), K_2CO_3 (0.4 mmol) were added to a sealed tube, dioxane (2.0 mL) were added via syringe. The mixture was flushed with N_2 and stirred at room temperature for 15 min firstly, and then was heated at 90 °C about for 12 h until completion (monitored by TLC). After cooling at room temperature, the mixture was extracted with ethyl acetate, dried with anhydrous sodium sulfate and concentrated under reduced pressure. The residue was purified through silica gel chromatography to afford the products **3**.



1a (0.2 mmol), **5** (0.4 mmol), $[Pd(C_3H_5)Cl]_2$ (5 mol%), P(*p*-tolyl)$_3$ (10 mol%), K_2CO_3 (0.4 mmol) were added to a sealed tube, dioxane (2.0 mL) were added via syringe. The mixture was flushed with N_2 and stirred at room temperature for 15 min firstly, and then was heated at 90 °C about for 12 h until completion (monitored by TLC). After cooling at room temperature, the mixture was extracted with ethyl acetate, dried with anhydrous sodium sulfate and concentrated under reduced pressure. The residue was purified through silica gel chromatography to afford the products **3a.**

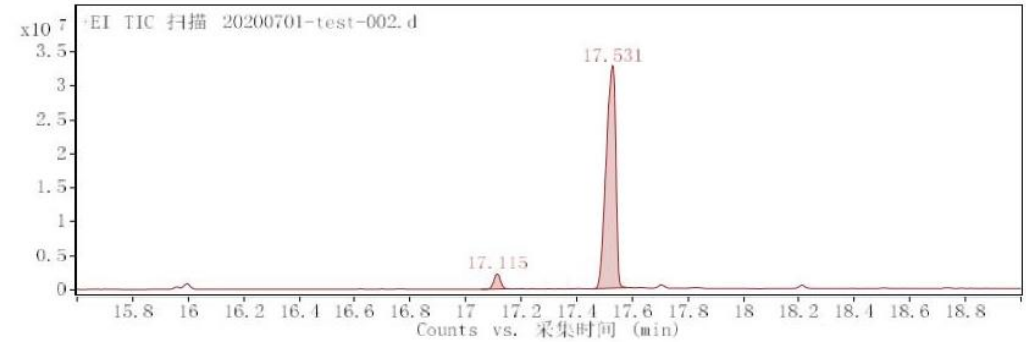


1a (0.2 mmol), **5** (0.4 mmol), $[Pd(C_3H_5)Cl]_2$ (5 mol%), P(*p*-tolyl)$_3$ (10 mol%), ^{t}BuOK (0.4 mmol) were added to a sealed tube, dioxane (2.0 mL) were added via syringe. The mixture was flushed with N_2 and stirred at room temperature for 15 min firstly, and then was heated at 90 °C about for 12 h until completion (monitored by TLC). After cooling at room temperature, the mixture was extracted with ethyl acetate, dried with anhydrous sodium sulfate and concentrated under reduced pressure. The residue was purified through silica gel chromatography to afford the products **3a**.

8. GC/MS experiment of 3a

用户色谱图

裂解电压　　碰撞能量　0　电离模式　EI

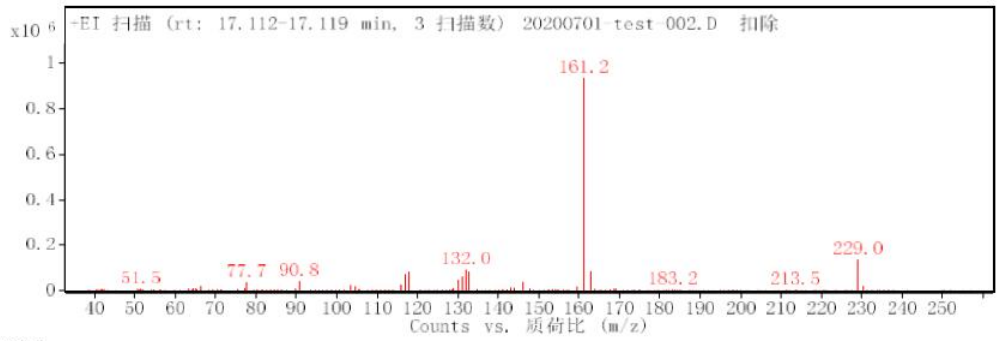


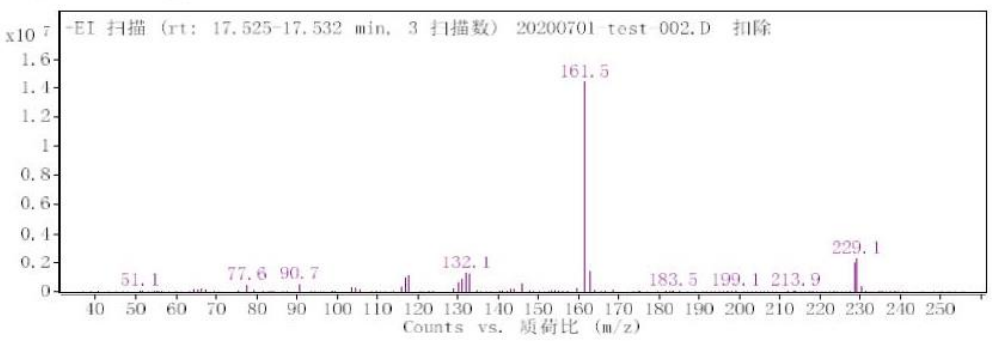
积分峰列表

峰	开始	保留时间	结束	峰高	面积	峰面积总和%
1	17.056	17.115	17.16	2181019.34	3703430.03	4.48
2	17.459	17.531	17.592	32782491.71	79040803.71	95.52

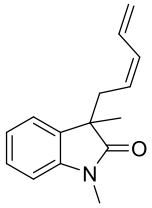
用户质谱图

碰撞能量　0　　电离模式　EI

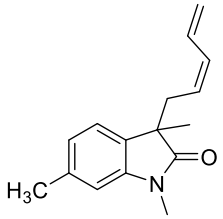




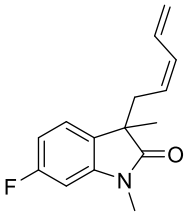
9. Spectra data



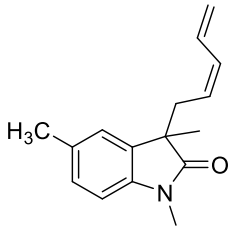
1,3-dimethyl-3-(penta-2,4-dien-1-yl)indolin-2-one (3a): 33 mg; 71% yield; yellow oil; *Z:E* = 21:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30-7.18 (m, 2H), 7.04 (td, *J* = 7.5, 1.0 Hz, 1H), 6.83 (d, *J* = 7.7 Hz, 1H), 6.58 (dddd, *J* = 16.5, 11.0, 10.1, 1.1 Hz, 1H), 5.95 (td, *J* = 11.0, 1.3 Hz, 1H), 5.18-5.04 (m, 3H), 3.20 (s, 3H), 2.76-2.57 (m, 2H), 1.39 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 180.1, 143.1, 133.4, 132.1, 131.8, 127.8, 125.6, 122.9, 122.3, 118.0, 107.9, 48.1, 36.0, 26.1, 22.7. HRMS (ESI) calcd for $C_{15}H_{18}NO$ $[M+H]^+$: 228.1383, found: 228.1385.



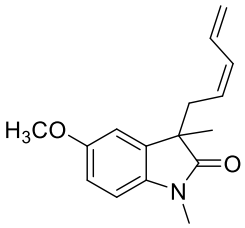
1,3,6-trimethyl-3-(penta-2,4-dien-1-yl)indolin-2-one (3b): 36 mg; 74% yield; yellow oil; *Z:E* = 10:1.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.07 (d, *J* = 7.5 Hz, 1H), 6.85 (d, *J* = 7.5 Hz, 1H), 6.73-6.49 (m, 2H), 5.95 (t, *J* = 10.9 Hz, 1H), 5.21-5.00 (m, 3H), 3.17 (s, 3H), 2.75-2.52 (m, 2H), 2.37 (s, 3H), 1.37 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 180.3, 143.1, 137.7, 132.0, 131.8, 130.4, 125.8, 122.7, 122.5, 117.9, 108.8, 47.8, 35.9, 256.0, 22.7, 21.7. HRMS (ESI) calcd for $C_{16}H_{20}NO$ $[M+H]^+$: 242.1539, found: 242.1542.



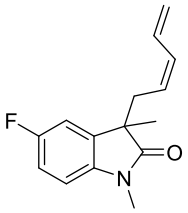
6-fluoro-1,3-dimethyl-3-(penta-2,4-dien-1-yl)indolin-2-one (3c): 37 mg; 76% yield; colorless oil; *Z:E* = 20:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.12 (dd, *J* = 8.2, 5.4 Hz, 1H), 6.71 (ddd, *J* = 9.6, 8.1, 2.3 Hz, 1H), 6.62-6.46 (m, 2H), 5.96 (ddd, *J* = 12.7, 10.9, 1.9 Hz, 1H), 5.21-5.01 (m, 3H), 3.18 (s, 3H), 2.76-2.55 (m, 2H), 1.38 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 180.3, 164.0, 144.5, 132.3, 131.6, 128.6, 128.5, 125.2, 123.8, 123. 7, 118.2, 108.3, 108.1, 96.8, 96.6, 47.8, 36.0, 26.2, 22.7. ^{19}F NMR (376 MHz, $CDCl_3$) δ -112.8. HRMS (ESI) calcd for $C_{15}H_{17}NFO$ $[M+H]^+$: 246.1289, found: 246.1291.



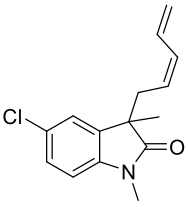
1,3,5-trimethyl-3-(penta-2,4-dien-1-yl)indolin-2-one (3d): 30 mg; 62% yield; colorless oil; *Z:E* = 13:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.12-6.95 (m, 2H), 6.71 (d, *J* = 7.8 Hz, 1H), 6.58 (dddd, *J* = 17.0, 11.3, 10.2, 1.2 Hz, 1H), 5.95 (t, *J* = 10.6 Hz, 1H), 5.21-4.98 (m, 3H), 3.17 (s, 3H), 2.78 -2.53 (m, 2H), 2.33 (s, 3H), 1.37 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 180.0, 140.7, 133.4, 132.0, 131.8, 131.7, 127.9, 125.7, 123.7, 117.8, 107.5, 48.1, 35.9, 26.1, 22.6, 21.0. HRMS (ESI) calcd for $C_{16}H_{20}NO$ $[M+H]^+$: 242.1539, found: 242.1542.



5-methoxy-1,3-dimethyl-3-(penta-2,4-dien-1-yl)indolin-2-one (3e): 24 mg; 46% yield; colorless oil; *Z:E* = 15:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.84-6.71 (m, 3H), 6.58 (dt, *J* = 16.6, 10.4 Hz, 1H), 5.96 (t, *J* = 11.0 Hz, 1H), 5.20-5.03 (m, 3H), 3.78 (s, 3H), 3.17 (s, 3H), 2.78-2.53 (m, 2H), 1.38 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 179.6, 155.8, 136.5, 134.7, 132.1, 131.7, 125.5, 118.0, 111.8, 110.5, 108.0, 55.7, 48.4, 35.8, 26.1, 22.6. HRMS (ESI) calcd for $C_{16}H_{20}NO_2$ $[M+H]^+$: 258.1489, found: 258.1491.

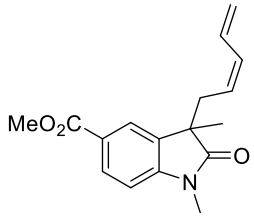


5-fluoro-1,3-dimethyl-3-(penta-2,4-dien-1-yl)indolin-2-one (3f): 34 mg; 69% yield; pale yellow oil; *Z:E* = 16:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.99-6.90 (m, 2H), 6.75 (dd, *J* = 9.2, 4.2 Hz, 1H), 6.56 (dtd, *J* = 17.0, 10.6, 1.3 Hz, 1H), 6.03-5.90 (m, 1H), 5.19-5.05 (m, 3H), 3.19 (s, 3H), 2.76-2.55 (m, 2H), 1.39 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 179.6, 160.3, 157.9, 139.0, 138.9, 135.0, 135.0, 132.4, 131.5, 125.0, 118.3, 113.9, 113.7, 111.2, 110.9, 108.2, 108.2, 48.5, 48.5, 35.8, 26.2, 22.5. ^{19}F NMR (376 MHz, $CDCl_3$) δ -120.9. HRMS (ESI) calcd for $C_{15}H_{17}NFO$ $[M+H]^+$: 246.1289, found: 246.1291.

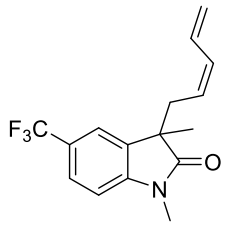


5-chloro-1,3-dimethyl-3-(penta-2,4-dien-1-yl)indolin-2-one (3g): 31 mg; 59% yield; yellow oil; *Z:E* = 17:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25-7.16 (m, 2H), 6.76 (t, *J* = 7.5 Hz,

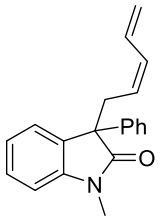
1H), 6.62-6.47 (m, 1H), 5.97 (t, *J* = 11.1 Hz, 1H), 5.21-5.02 (m, 3H), 3.18 (s, 3H), 2.76-2.56 (m, 2H), 1.38 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 179.4, 141.6, 135.0, 132.4, 131.4, 127.6, 127.5, 124.8, 123.3, 118.3, 108.7, 48.3, 35.7, 26.1, 22.4. HRMS (ESI) calcd for $C_{15}H_{17}NOCl$ $[M+H]^+$: 262.0993, found: 262.0996.



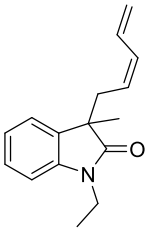
methyl 1,3-dimethyl-2-oxo-3-(penta-2,4-dien-1-yl)indoline-5-carboxylate (3h): 22 mg; 39% yield; yellow solid; mp = 89-91 °C; *Z:E* = 22:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (dd, *J* = 8.2, 1.7 Hz, 1H), 7.88 (d, *J* = 1.7 Hz, 1H), 6.87 (d, *J* = 8.2 Hz, 1H), 6.64-6.50 (m, 1H), 5.95 (t, *J* = 11.0 Hz, 1H), 5.20-4.98 (m, 3H), 3.91 (s, 3H), 3.23 (s, 3H), 2.81-2.59 (m, 2H), 1.42 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 180.4, 166. 9, 147.3, 133.3, 132.5, 131.6, 130.6, 124.9, 124.3, 124.1, 118.4, 107.4, 52.0, 48.1, 36.0, 26.3, 22.6. HRMS (ESI) calcd for $C_{17}H_{20}NO_3$ $[M+H]^+$: 286.1438, found: 286.1440.



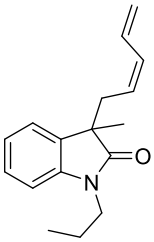
1,3-dimethyl-3-(penta-2,4-dien-1-yl)-5-(trifluoromethyl)indolin-2-one (3i): 30 mg; 51% yield; yellow solid; mp = 101-102 °C; *Z:E* = 18:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (dd, *J* = 8.2, 1.8 Hz, 1H), 7.43 (d, *J* = 1.9 Hz, 1H), 6.89 (d, *J* = 8.2 Hz, 1H), 6.52 (dtd, *J* = 16.8, 10.5, 1.2 Hz, 1H), 6.01-5.91 (m, 1H), 5.18-5.04 (m, 3H), 3.23 (s, 3H), 2.78-2.60 (m, 2H), 1.43 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 180.1, 146.2, 134.0, 132.8, 131.5, 125.7, 125. 7, 124.8, 124.6, 120.0, 120.0, 118.6, 107.6, 48.3, 35.9, 26.4, 22.5. ^{19}F NMR (376 MHz, $CDCl_3$) δ -61.4. HRMS (ESI) calcd for $C_{16}H_{17}NF_3O$ $[M+H]^+$: 296.1257, found: 296.1259.



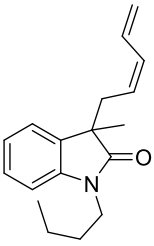
1-methyl-3-(penta-2,4-dien-1-yl)-3-phenylindolin-2-one (3j) : 28 mg; 48% yield; yellow oil; *Z:E* = 9:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (dt, *J* = 6.2, 1.4 Hz, 2H), 7.35-7.23 (m, 5H), 7.09 (td, *J* = 7.6, 1.0 Hz, 1H), 6.88 (d, *J* = 7.8 Hz, 1H), 6.63-6.49 (m, 1H), 5.98-5.86 (m, 1H), 5.16-5.02 (m, 3H), 3.26-3.08 (m, 5H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.0, 143.9, 139.5, 132.4, 131.9, 131.6, 128.6, 128.3, 127.4, 127.1, 125.5, 125.2, 122.6, 118.3, 108.3, 56.1, 35.9, 26.5. HRMS (ESI) calcd for $C_{20}H_{20}NO$ $[M+H]^+$: 290.1539, found: 290.1542.



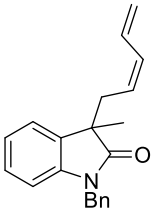
1-ethyl-3-methyl-3-(penta-2,4-dien-1-yl)indolin-2-one (3k): 32 mg; 67% yield; pale yellow oil; *Z:E* = 11:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28-7.16 (m, 2H), 7.03 (td, *J* = 7.5, 1.0 Hz, 1H), 6.84 (d, *J* = 7.8 Hz, 1H), 6.69-6.47 (m, 1H), 5.93 (t, *J* = 11.1 Hz, 1H), 5.18-4.98 (m, 3H), 3.89-3.73 (m, 1H), 3.66 (dq, *J* = 14.3, 7.2 Hz, 1H), 2.75 (ddd, *J* = 14.0, 8.3, 1.4 Hz, 1H), 2.60 (ddd, *J* = 13.9, 7.5, 1.5 Hz, 1H), 1.39 (s, 3H), 1.22 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 179.6, 142.2, 133.5, 132.1, 131.8, 127.7, 125.6, 122.9, 122.0, 117.9, 107.9, 47.9, 36.1, 34.4, 22.6, 12.7. HRMS (ESI) calcd for $C_{16}H_{20}NO$ $[M+H]^+$: 242.1539, found: 242.1542.



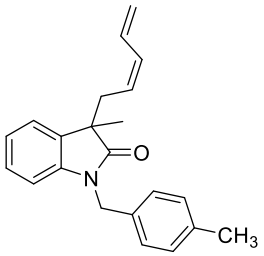
3-methyl-3-(penta-2,4-dien-1-yl)-1-propylindolin-2-one (3l): 32 mg; 62% yield; yellow oil; *Z:E* = 9:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26-7.16 (m, 2H), 7.02 (t, *J* = 7.4 Hz, 1H), 6.83 (d, *J* = 7.8 Hz, 1H), 6.67-6.52 (m, 1H), 5.97-5.86 (m, 1H), 3.77-3.66 (m, 1H), 3.58 (dt, *J* = 14.1, 7.2 Hz, 1H), 2.75 (ddd, *J* = 14.0, 8.2, 1.4 Hz, 1H), 2.61 (ddd, *J* = 14.0, 7.5, 1.6 Hz, 1H), 1.67 (h, *J* = 7.2 Hz, 2H), 1.39 (s, 3H), 0.93 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 179.9, 142.6, 133.4, 132.0, 131.8, 127.6, 125.6, 122.8, 122.0, 117.8, 108.1, 47.9, 41.2, 36.0, 22.9, 20.6, 11.2. HRMS (ESI) calcd for $C_{17}H_{22}NO$ $[M+H]^+$: 256.1696, found: 256.1698.



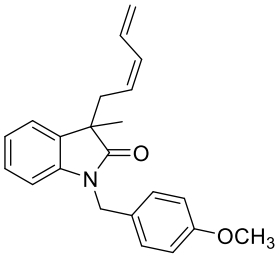
1-butyl-3-methyl-3-(penta-2,4-dien-1-yl)indolin-2-one (3m): 29 mg; 53% yield; yellow oil; *Z:E* = 7:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.18 (m, 2H), 7.02 (t, *J* = 7.4 Hz, 1H), 6.83 (d, *J* = 7.8 Hz, 1H), 6.67-6.51 (m, 1H), 5.93 (t, *J* = 11.0 Hz, 1H), 5.17-5.00 (m, 3H), 3.75 (dt, *J* = 14.3, 7.2 Hz, 1H), 3.61 (dt, *J* = 14.1, 7.2 Hz, 1H), 2.74 (ddd, *J* = 14.1, 8.4, 1.4 Hz, 1H), 2.64-2.51 (m, 1H), 1.61 (q, *J* = 7.4 Hz, 2H), 1.39 (s, 3H), 1.37-1.32 (m, 2H), 0.93 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 179.9, 142.6, 133.6, 132.1, 131.9, 127.7, 125.7, 122.9, 122.0, 117.9, 108.1, 48.0, 39.6, 36.1, 29.5, 22.9, 20.1, 13.7. HRMS (ESI) calcd for $C_{18}H_{24}NO$ $[M+H]^+$: 270.1852, found: 270.1855.



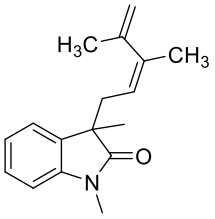
1-benzyl-3-methyl-3-(penta-2,4-dien-1-yl)indolin-2-one (3n): 27 mg; 45% yield; yellow oil; *Z:E* = 24:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32-7.19 (m, 6H), 7.13 (td, *J* = 7.7, 1.3 Hz, 1H), 7.01 (td, *J* = 7.5, 1.1 Hz, 1H), 6.73-6.56 (m, 2H), 5.95 (t, *J* = 11.0 Hz, 1H), 5.20-4.98 (m, 4H), 4.76 (d, *J* = 15.6 Hz, 1H), 2.85 (ddd, *J* = 14.0, 8.5, 1.3 Hz, 1H), 2.65 (ddd, *J* = 14.0, 7.3, 1.5 Hz, 1H), 1.46 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 180.2, 142.3, 136.0, 133.4, 132.3, 131.9, 128.6, 127.7, 127.5, 127.3, 125.7, 122.9, 122.4, 118.2, 109.0, 48.2, 43.6, 36.1, 23.2. HRMS (ESI) calcd for $C_{21}H_{22}NO$ $[M+H]^+$: 304.1696, found: 304.1699.



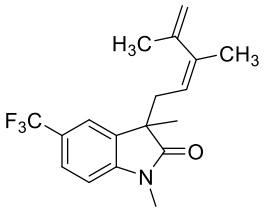
3-methyl-1-(4-methylbenzyl)-3-(penta-2,4-dien-1-yl)indolin-2-one (3o): 36 mg; 56% yield; yellow oil; *Z:E* = 6:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21-7.14 (m, 3H), 7.10 (td, *J* = 8.0, 1.6 Hz, 3H), 6.99 (td, *J* = 7.5, 1.0 Hz, 1H), 6.70 (d, *J* = 8.0 Hz, 1H), 6.68-6.58 (m, 1H), 5.95 (t, *J* = 11.1 Hz, 1H), 5.20-5.07 (m, 3H), 4.98 (d, *J* = 15.4 Hz, 1H), 4.71 (d, *J* = 15.5 Hz, 1H), 2.84 (ddd, *J* = 13.9, 8.5, 1.3 Hz, 1H), 2.64 (ddd, *J* = 14.0, 7.2, 1.5 Hz, 1H), 2.29 (s, 3H), 1.44 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 180.1, 142.3, 137.1, 133.3, 132.9, 132.3, 131.9, 129.4, 129.2, 127.7, 127.3, 125.8, 122.8, 122.3, 118.1, 109.0, 48.2, 43.4, 36.1, 23.2, 21.0. HRMS (ESI) calcd for $C_{22}H_{24}NO$ $[M+H]^+$: 318.1852, found: 318.1855.



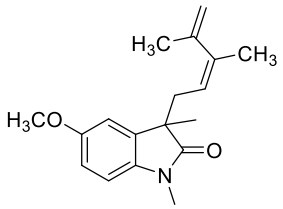
1-(4-methoxybenzyl)-3-methyl-3-(penta-2,4-dien-1-yl)indolin-2-one (3p): 41 mg; 61% yield; yellow oil; *Z:E* = 10:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (dd, *J* = 8.2, 3.8 Hz, 3H), 7.13 (tt, *J* = 7.7, 1.2 Hz, 1H), 7.00 (t, *J* = 7.4 Hz, 1H), 6.81 (d, *J* = 8.2 Hz, 2H), 6.76-6.68 (m, 1H), 6.68-6.58 (m, 1H), 5.94 (t, *J* = 10.9 Hz, 1H), 5.18-5.04 (m, 3H), 4.99-4.84 (m, 1H), 4.68 (d, *J* = 15.4 Hz, 1H), 3.75 (d, *J* = 1.0 Hz, 3H), 2.90-2.79 (m, 1H), 2.63 (dd, *J* = 14.1, 7.2 Hz, 1H), 1.44 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 180.1, 158.9, 142.2, 133.3, 132.2, 131.9, 128.7, 128.5, 128.0, 127.6, 125.7, 122.8, 122.3, 118.1, 114.1, 113.9, 108.9, 55.1, 48.1, 43.0, 36.1, 23.1. HRMS (ESI) calcd for $C_{22}H_{24}NO_2$ $[M+H]^+$: 334.1802, found: 334.1805.



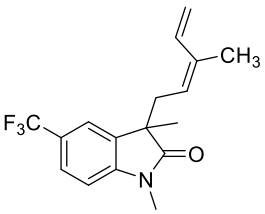
3-(3,4-dimethylpenta-2,4-dien-1-yl)-1,3-dimethylindolin-2-one (3q): 29 mg; 57% yield; colorless oil; *Z:E* = 32:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 (td, *J* = 7.7, 1.4 Hz, 1H), 7.15 (dd, *J* = 7.4, 1.3 Hz, 1H), 7.04 (td, *J* = 7.5, 1.0 Hz, 1H), 6.83 (dt, *J* = 7.7, 0.8 Hz, 1H), 4.90-4.81 (m, 2H), 4.53 (dt, *J* = 1.9, 1.0 Hz, 1H), 3.21 (s, 3H), 2.56 (dq, *J* = 7.2, 1.2 Hz, 2H), 1.64 (p, *J* = 1.3 Hz, 6H), 1.34 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 180.5, 144.8, 143.1, 141.6, 134.0, 127.5, 122.9, 122.2, 118.9, 112.7, 107.7, 48.2, 37.3, 26.1, 23.1, 22.6, 21.9. HRMS (ESI) calcd for $C_{17}H_{22}NO$ $[M+H]^+$: 256.1696, found: 256.1699.



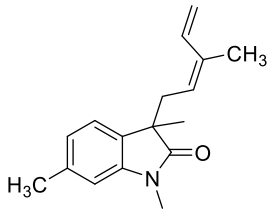
3-(3,4-dimethylpenta-2,4-dien-1-yl)-1,3-dimethyl-5-(trifluoromethyl)indolin-2-one (3r): 41 mg; 63% yield; yellow oil; *Z:E* = 30:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59-7.49 (m, 1H), 7.36 (d, *J* = 1.9 Hz, 1H), 6.89 (d, *J* = 8.1 Hz, 1H), 4.91-4.77 (m, 2H), 4.54-4.44 (m, 1H), 3.25 (s, 3H), 2.60 (h, *J* = 7.1 Hz, 2H), 1.67-1.62 (m, 3H), 1.59 (s, 3H), 1.38 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 180.4, 146.1, 144.6, 142.5, 134.5, 125.4, 125.4, 120.1, 120.0, 118.1, 112.9, 107.4, 48.3, 37.2, 26.3, 23.1, 22.5, 21.7. ^{19}F NMR (376 MHz, $CDCl_3$) δ -61.4. HRMS (ESI) calcd for $C_{18}H_{20}NNaF_3O$ $[M+Na]^+$: 346.1389, found: 346.1386.



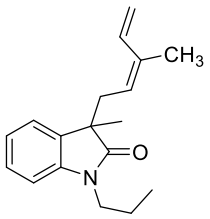
3-(3,4-dimethylpenta-2,4-dien-1-yl)-5-methoxy-1,3-dimethylindolin-2-one (3s): 27 mg; 48% yield; pale yellow oil; *Z:E* = 22:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.81-6.68 (m, 3H), 4.92-4.78 (m, 2H), 4.55 (dd, *J* = 2.6, 1.1 Hz, 1H), 3.80 (s, 3H), 3.19 (s, 3H), 2.55 (dd, *J* = 7.3, 1.4 Hz, 2H), 1.72-1.58 (m, 6H), 1.33 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 180.2, 155.8, 144.9, 141.6, 136.7, 135.4, 118.9, 112.8, 111.5, 110.6, 107.9, 55.7, 48.6, 37.3, 26.2, 23.1, 22.7, 21.9. HRMS (ESI) calcd for $C_{18}H_{24}NO_2$ $[M+H]^+$: 286.1802, found: 286.1800.



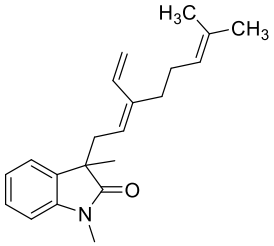
1,3-dimethyl-3-(3-methylpenta-2,4-dien-1-yl)-5-(trifluoromethyl)indolin-2-one (3t): 38 mg, 61% yield, yellow oil; *Z:E* = 14:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (dd, *J* = 8.1, 1.8 Hz, 1H), 7.40 (d, *J* = 1.9 Hz, 1H), 6.89 (d, *J* = 8.2 Hz, 1H), 6.61 (dd, *J* = 17.2, 10.7 Hz, 1H), 5.23-4.96 (m, 3H), 3.23 (s, 3H), 2.66 (qd, *J* = 14.3, 8.0 Hz, 2H), 1.72-1.68 (m, 3H), 1.41 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 180.2, 146.1, 136.0, 134.1, 132.9, 125.6, 125.5, 125.5, 124.6, 123.0, 120.1, 120.1, 114.7, 107.5, 48.3, 35.5, 26.3, 22.3, 19.8. ^{19}F NMR (376 MHz, $CDCl_3$) δ -61.4. HRMS (ESI) calcd for $C_{17}H_{18}NNaF_3O$ $[M+Na]^+$: 332.1233, found: 332.1230.



1,3,6-trimethyl-3-(3-methylpenta-2,4-dien-1-yl)indolin-2-one (3u): 26 mg; 50% yield; pale yellow oil; *Z:E* = 24:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.06 (d, *J* = 7.5 Hz, 1H), 6.88-6.80 (m, 1H), 6.77-6.60 (m, 2H), 5.21-5.02 (m, 3H), 3.18 (s, 3H), 2.71-2.52 (m, 2H), 2.38 (s, 3H), 1.71 (d, *J* = 1.1 Hz, 3H), 1.35 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 180.6, 143.1, 137.7, 135.1, 133.4, 130.7, 124.3, 122.8, 122.7, 114.1, 108.8, 48.0, 35.6, 26.1, 22.7, 21.7, 19.9. HRMS (ESI) calcd for $C_{17}H_{22}NO$ $[M+H]^+$: 256.1696, found: 256.1699.

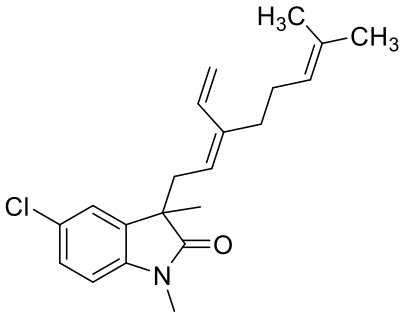


3-methyl-3-(3-methylpenta-2,4-dien-1-yl)-1-propylindolin-2-one (3v): 23 mg; 43% yield; yellow oil; *Z:E* = 18:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (dtd, *J* = 15.4, 7.5, 1.3 Hz, 2H), 7.04 (qd, *J* = 7.4, 1.0 Hz, 1H), 6.84 (dd, *J* = 11.0, 7.7 Hz, 1H), 6.72 (ddd, *J* = 17.2, 10.8, 0.9 Hz, 1H), 5.19-4.97 (m, 3H), 3.76 (dt, *J* = 14.4, 7.3 Hz, 1H), 3.54 (dt, *J* = 14.1, 7.0 Hz, 1H), 2.78-2.70 (m, 1H), 2.58 (ddd, *J* = 14.4, 7.3, 1.3 Hz, 1H), 1.73-1.63 (m, 5H), 1.38 (s, 3H), 0.92 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 180.1, 142.7, 135.1, 133.7, 133.4, 127.6, 127.5, 124.2, 123.0, 122.0, 114.1, 108.3, 108.1, 48.2, 41.3, 35.8, 22.9, 20.7, 19.8, 11.3. HRMS (ESI) calcd for $C_{18}H_{24}NO$ $[M+H]^+$: 270.1852, found: 270.1855.

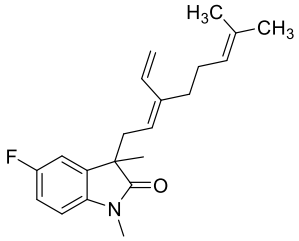


1,3-dimethyl-3-(7-methyl-3-vinylocta-2,6-dien-1-yl)indolin-2-one (3w): 22 mg; 36% yield; yellow oil; *Z:E* = 12:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28-7.23 (m, 1H), 7.19 (dd, *J* = 7.4, 1.3 Hz, 1H), 7.04 (t, *J* = 7.5 Hz, 1H), 6.82 (d, *J* = 7.6 Hz, 1H), 6.60 (dd, *J* = 17.4, 11.0 Hz, 1H), 5.19 (d, *J* = 17.4 Hz, 1H), 5.10-4.97 (m, 3H), 3.19 (s, 3H), 2.63 (d, *J* = 8.0 Hz, 2H),

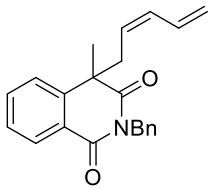
2.07 (t, *J* = 7.7 Hz, 2H), 1.94 (q, *J* = 6.6, 5.9 Hz, 2H), 1.65 (d, *J* = 1.5 Hz, 3H), 1.53 (s, 3H), 1.38 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.1, 139.3, 133.6, 132.4, 131.5, 127.7, 124.2, 123.6, 123.0, 122.3, 113.9, 107.8, 48.4, 35.7, 33.4, 27.6, 26.1, 25.7, 22.3, 17.6. HRMS (ESI) calcd for $C_{21}H_{28}NO$ $[M+H]^+$: 310.2165, found: 310.2168.



5-chloro-1,3-dimethyl-3-(7-methyl-3-vinylocta-2,6-dien-1-yl)indolin-2-one (3x): 21 mg; 30% yield; yellow oil; *Z:E* = 7:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 (dd, *J* = 8.2, 2.1 Hz, 1H), 7.16 (d, *J* = 2.1 Hz, 1H), 6.73 (d, *J* = 8.2 Hz, 1H), 6.56 (ddd, *J* = 17.4, 11.1, 1.0 Hz, 1H), 5.21 (dt, *J* = 17.4, 1.1 Hz, 1H), 5.12-4.97 (m, 3H), 3.17 (s, 3H), 2.69-2.56 (m, 2H), 2.12-2.02 (m, 2H), 2.00-1.89 (m, 2H), 1.66 (d, *J* = 1.5 Hz, 3H), 1.54 (d, *J* = 1.3 Hz, 3H), 1.38 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 179.9, 141.7, 139.9, 135.3, 132.2, 131.7, 127.7, 127.7, 124.2, 123.7, 122.9, 114.3, 108.7, 48.8, 35.6, 33.5, 27.7, 26.3, 25.7, 22.2, 17.7. HRMS (ESI) calcd for $C_{21}H_{27}NClO$ $[M+H]^+$: 344.1776, found: 344.1778.

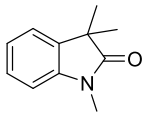


5-fluoro-1,3-dimethyl-3-(7-methyl-3-vinylocta-2,6-dien-1-yl)indolin-2-one (3y): 29 mg; 44% yield; yellow oil; *Z:E* = 8:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.98-6.90 (m, 2H), 6.73 (ddd, *J* = 7.8, 4.0, 1.2 Hz, 1H), 6.57 (ddd, *J* = 17.5, 11.0, 0.9 Hz, 1H), 5.21 (dt, *J* = 17.5, 1.0 Hz, 1H), 5.12-4.96 (m, 3H), 3.18 (s, 3H), 2.62 (dd, *J* = 8.0, 3.9 Hz, 2H), 2.08 (t, *J* = 7.7 Hz, 2H), 1.99-1.88 (m, 2H), 1.65 (d, *J* = 1.7 Hz, 3H), 1.54 (d, *J* = 1.3 Hz, 3H), 1.38 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 180.0, 139.7, 139.0, 132.2, 131.6, 124.1, 123.0, 114.2, 113.9, 113.7, 111.4, 111.2, 108.2, 108.1, 48.8, 35.6, 33.4, 27.6, 26.2, 25.7, 22.2, 17.6. ^{19}F NMR (376 MHz, $CDCl_3$) δ -121.0. HRMS (ESI) calcd for $C_{21}H_{27}NFO$ $[M+H]^+$: 328.2071, found: 328.2069.



2-benzyl-4-methyl-4-(penta-2,4-dien-1-yl)isoquinoline-1,3(*2H*,*4H*)-dione (3z): 41 mg; 62% yield; yellow oil; *Z:E* = 11:1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (dt, *J* = 7.9, 1.9 Hz, 1H), 7.63 (td, *J* = 7.6, 1.5 Hz, 1H), 7.51-7.34 (m, 4H), 7.24 (ddd, *J* = 13.4, 7.8, 6.0 Hz, 3H), 6.40 (dt, *J* = 16.9, 10.7 Hz, 1H), 5.82 (t, *J* = 11.0 Hz, 1H), 5.29-4.98 (m, 4H), 4.75 (q, *J* =

8.9 Hz, 1H), 3.16 (dd, *J* = 13.9, 8.8 Hz, 1H), 2.71-2.52 (m, 1H), 1.70 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.8, 164.1, 142.7, 137.1, 134.0, 132.9, 131.3, 129.0, 128.8, 128.3, 127.4, 127.3, 125.4, 124.6, 118.9, 47.9, 43.7, 41.7, 27.5. HRMS (ESI) calcd for $C_{22}H_{22}NO_2$ $[M+H]^+$: 332.1645, found: 332.1648.



1,3,3-trimethylindolin-2-one (6): 27 mg; 77% yield; oil. ^{11}H NMR (400 MHz, $CDCl_3$) δ 7.31-7.16 (m, 2H), 7.07 (td, *J* = 7.5, 1.0 Hz, 1H), 6.85 (d, *J* = 7.7 Hz, 1H), 3.22 (s, 3H), 1.37 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 181.4, 142.7, 135.9, 127.7, 122.5, 122.3, 108.0, 44.2, 26.2, 24.4.

10. References

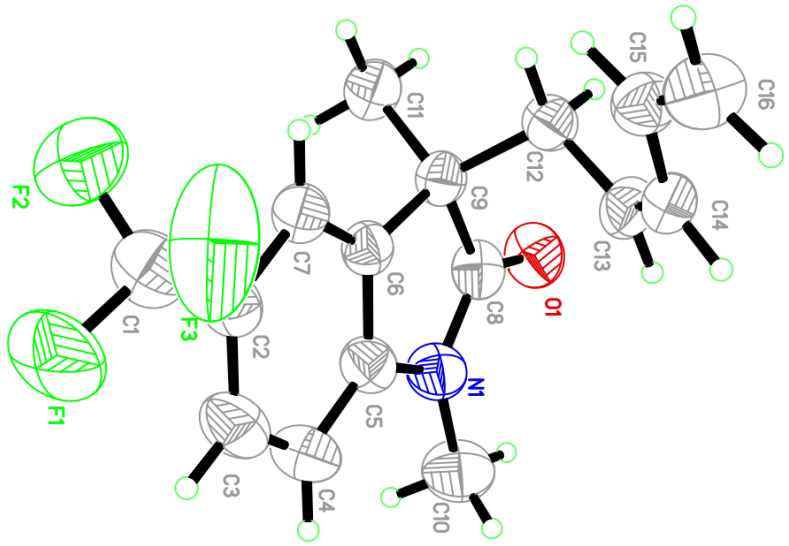
(1) (a) Liu, X.; Ma, X.; Huang, Y.; Gu, Z. *Org. Lett.* **2013**, *15*, 4814. (b) Wei, W.; Wen, J.; Yang, D.; Guo, M.; Tian, L.; You, J.; Wang, H. *RSC Adv.* **2014**, *4*, 48535. (c) Tang, X.; Thomoson, C. S.; Dolbier, W. R. *Org. Lett.* **2014**, *16*, 4594.

(2) Yang, X.; Lu, H.; Zhu, X.; Zhou, L.; Deng, G.; Yang, Y.; Liang, Y. *Org. Lett.* **2019**, *21*, 7284.

(3) (a) Dang, H. T.; Nguyen, V. T.; Nguyen, V. D.; Arman, H. D.;Larionov, O. V. *Org. Biomol. Chem.* **2018**, *16*, 3605. (b) Nguyen, V. T.; Dang, H. T.; Pham, H. H.; Nguyen, V. D.; Flores-Hansen, C.; Arman, H. D.; Larionov, O. V. *J. Am. Chem. Soc.* **2018**, *140*, 8434.

(4) Gamero-Melo, P.; Villanueva-García, M.; Robles, J.; Contreras, R.; Paz-Sandoval, M. A.; *J. Organomet. Chem.* **2005**, *690*, 1379.

11. Crystallographic data of 3i



Structure of 3i **CCDC:** 1975373

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	alpha=90	beta=90	gamma=90
Temperature:	293 K		
	Calculated		Reported
Volume	3100.2(3)		3100.2(3)
Space group	p b c a		p b c a
Hall group	-p 2ac 2ab		-p 2ac 2ab
Moiety formula	C16 H16 F3 N O		
Sum formula	C16 H16 F3 N O		C16 H16 F3 N O
Mr	295.30		295.30
Dx,g cm-3	1.265		1.265
Z	8		8
Mu (mm-1)	0.882		0.882
F000	1232.0		1232.0
F000’	1236.53		
h,k,lmax	18,14,19		19,14,19
Nref	2780		2779
Tmin,Tmax	0.900,0.908		0.923,1.000
Tmin’	0.900		

Correction method = # Reported T Limits: Tmin = 0.923 Tmax = 1.000

AbsCorr = MULTI-SCAN

Data completeness = 1.000	Theta (max) = 67.232
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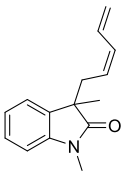
S = 1.028	Npar = 193

12. NMR spectra



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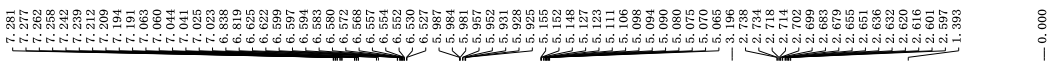

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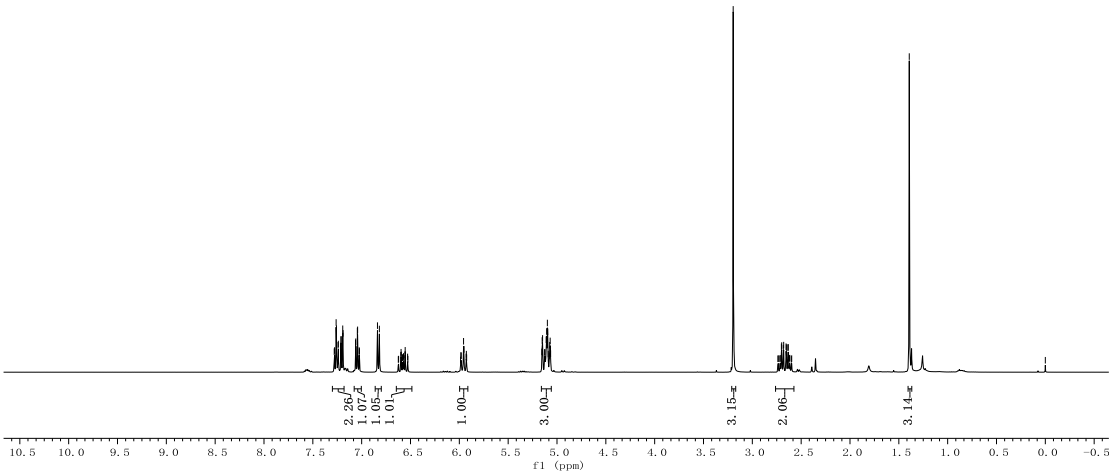

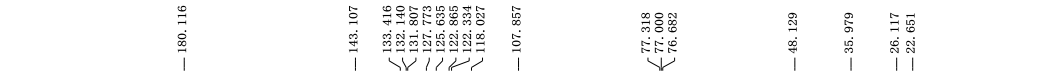

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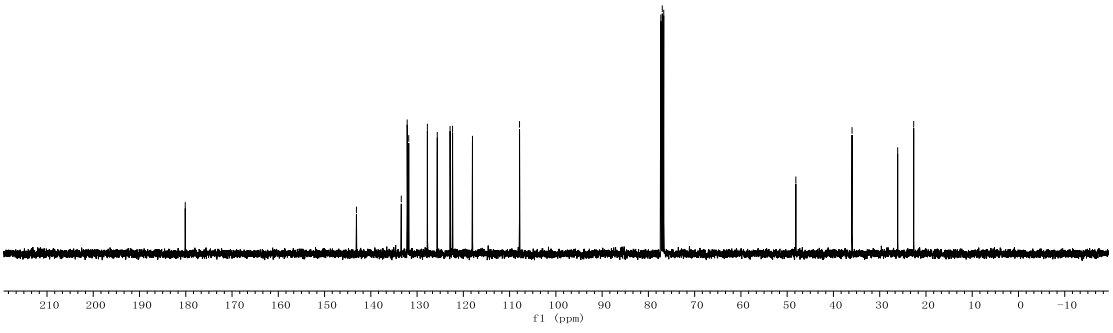

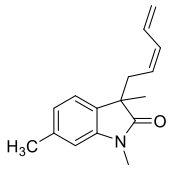

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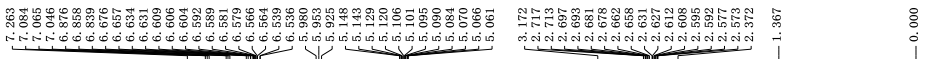

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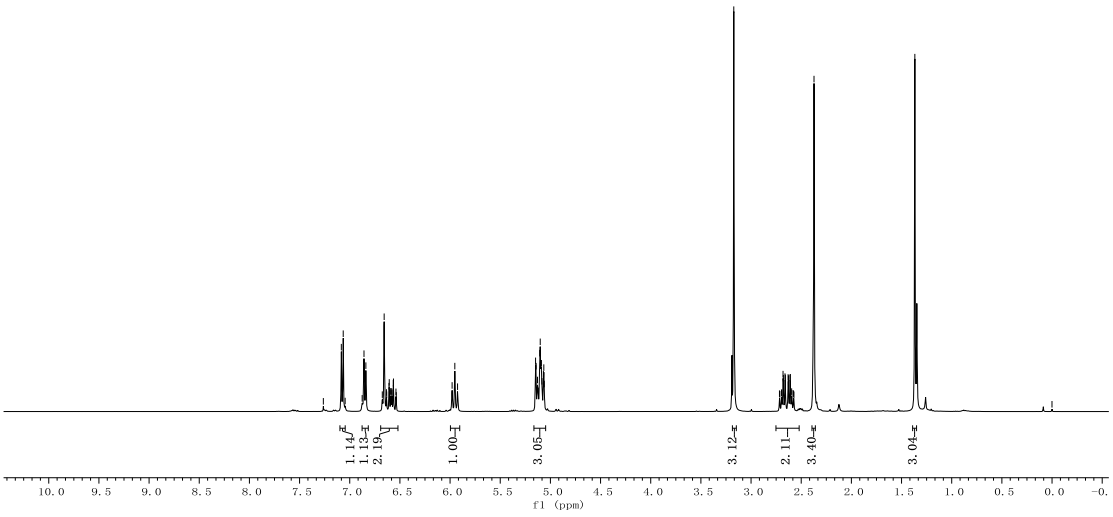

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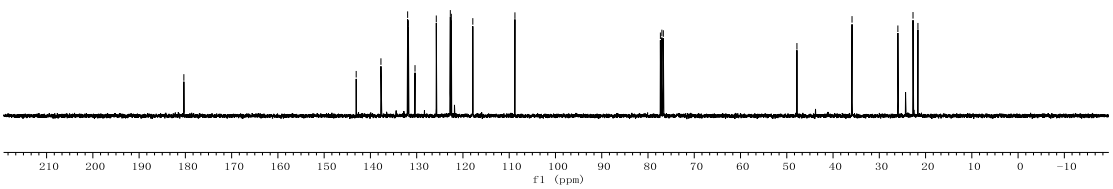

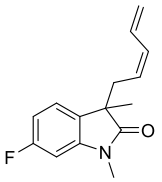

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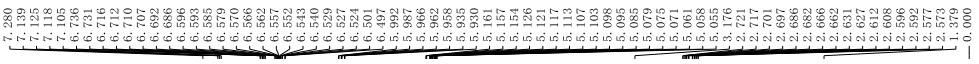

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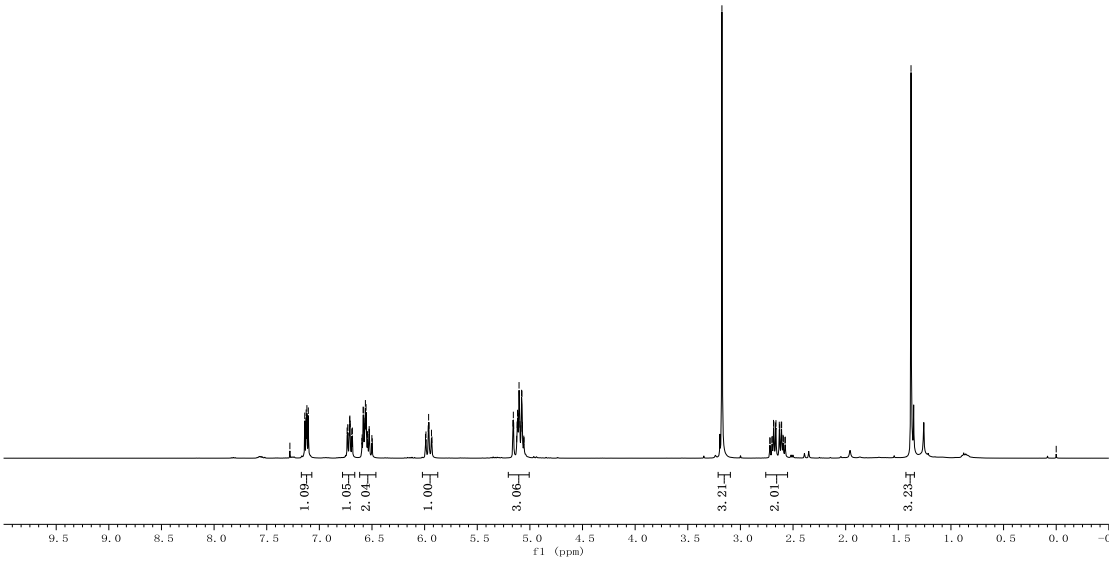

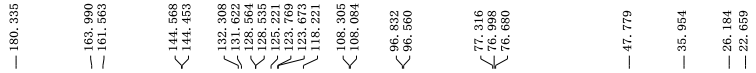

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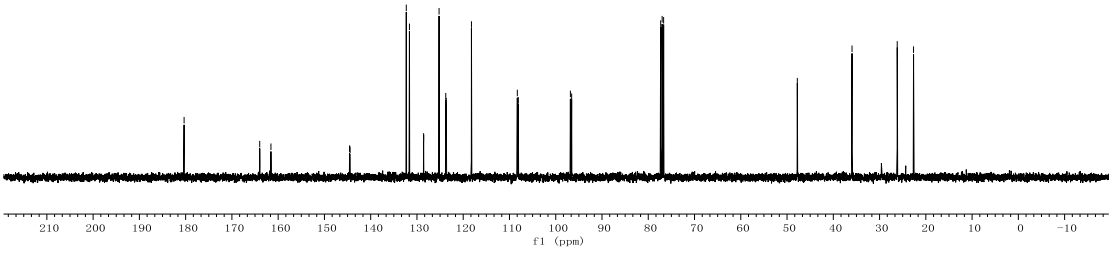

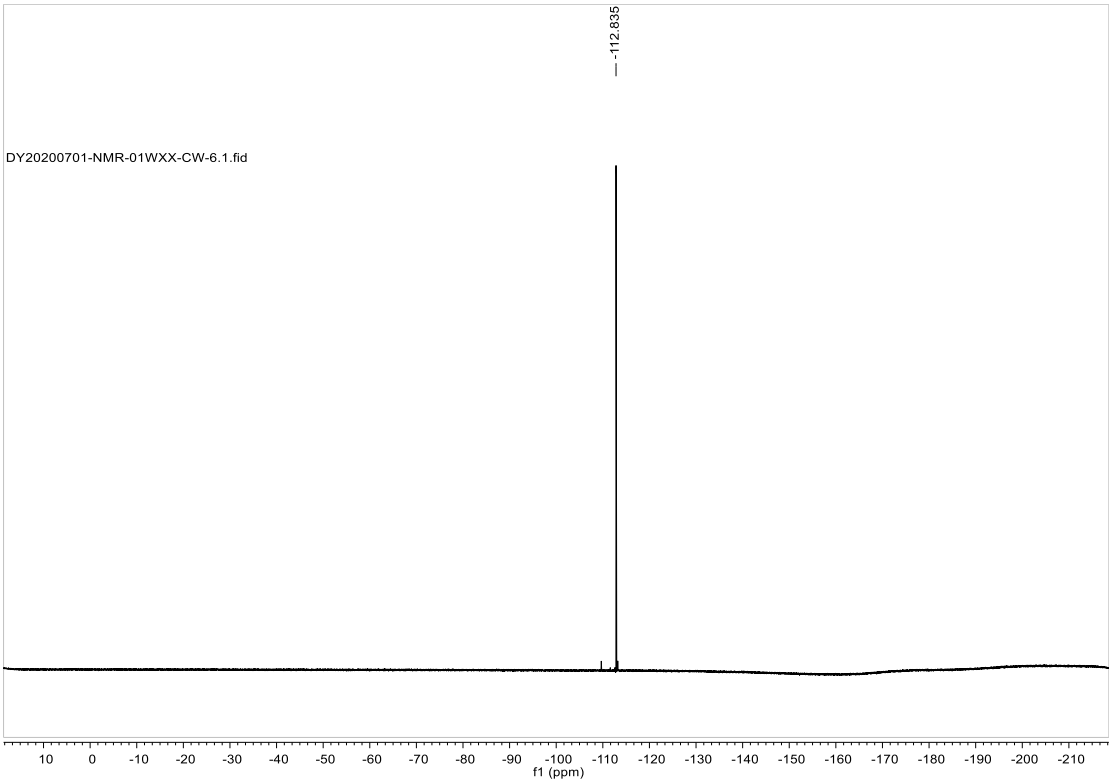

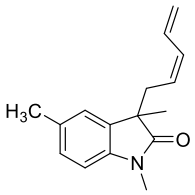

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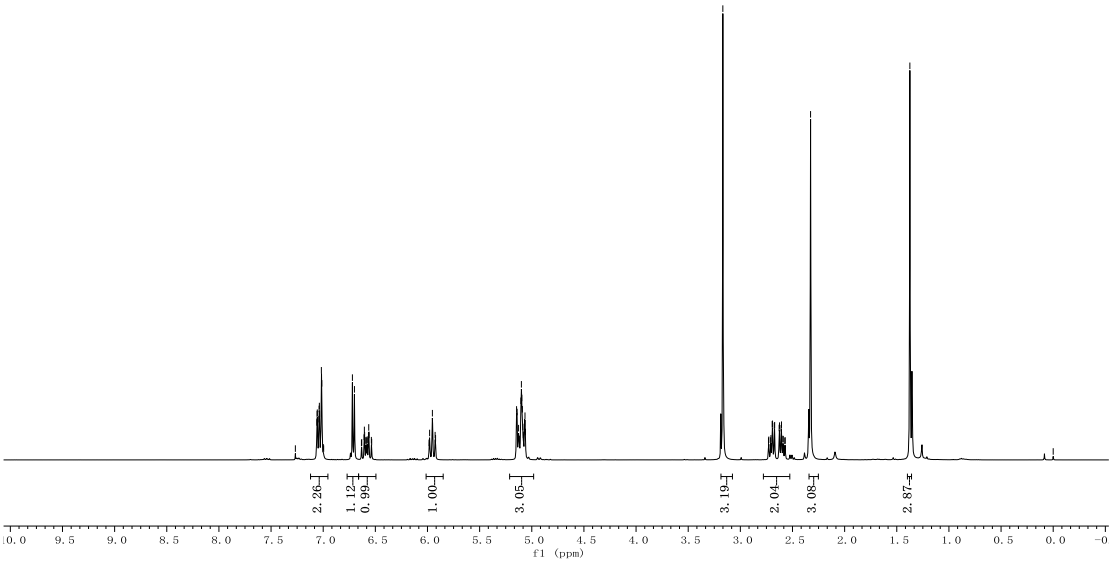

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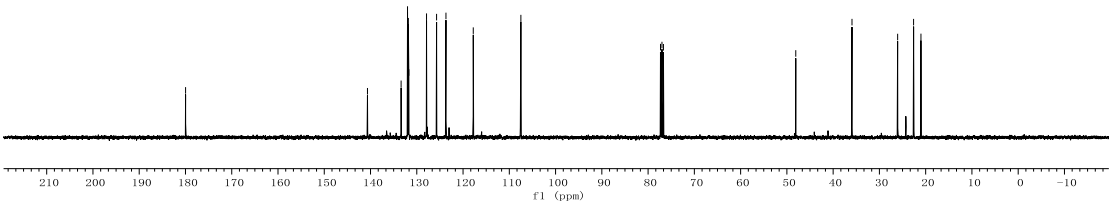

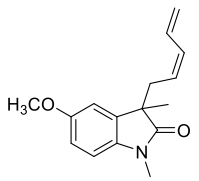

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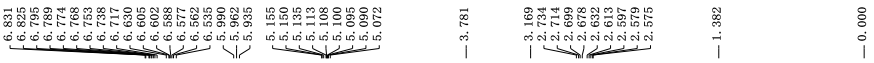

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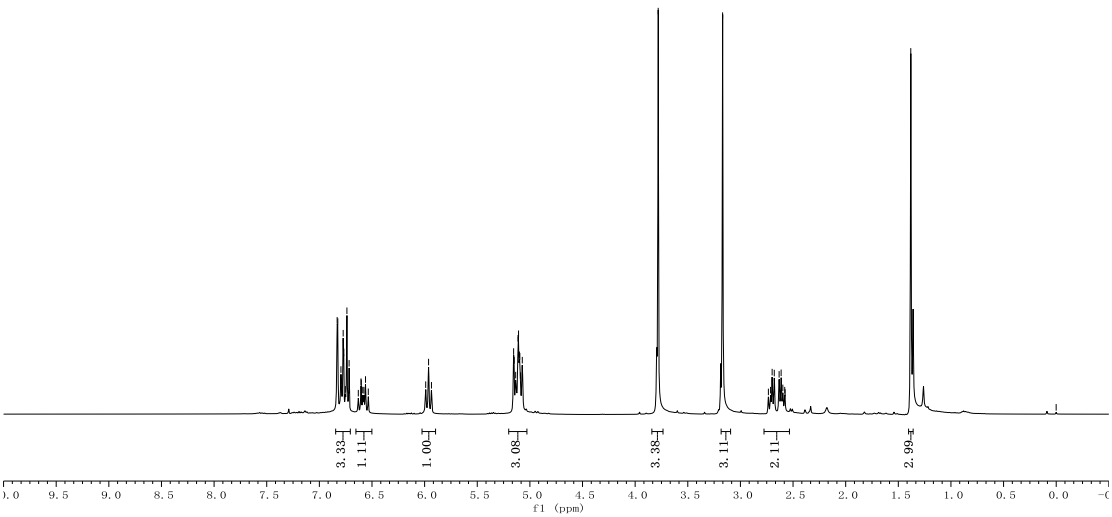

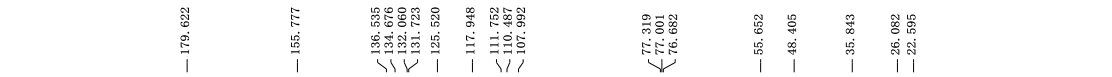

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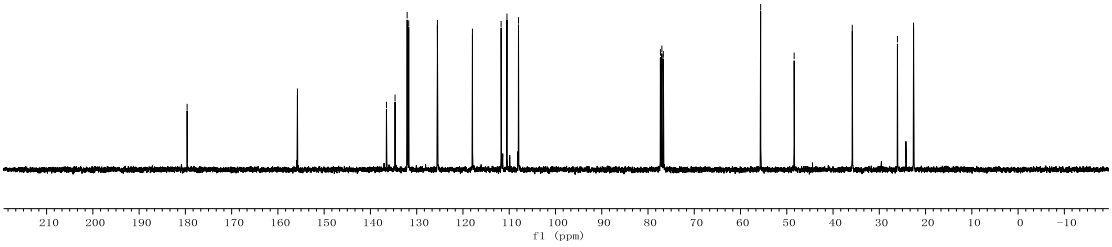

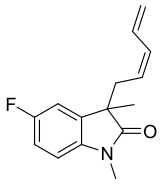

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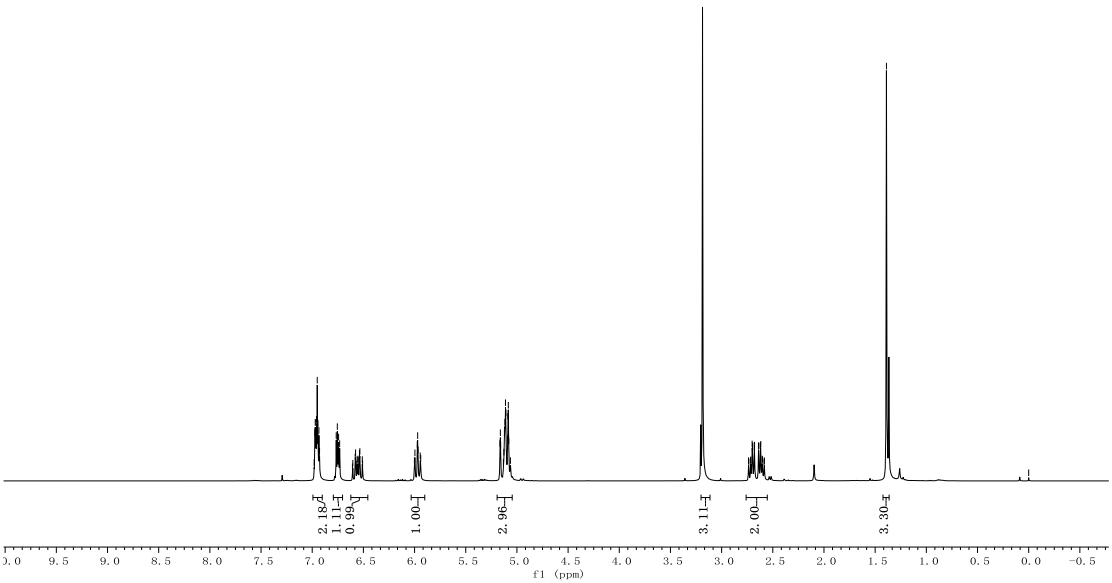

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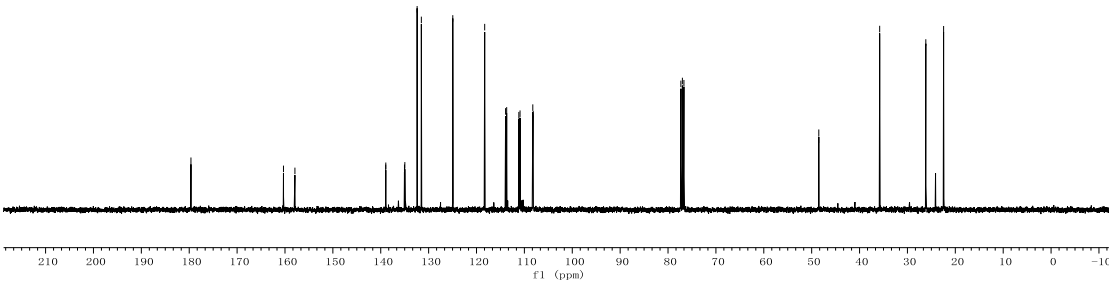

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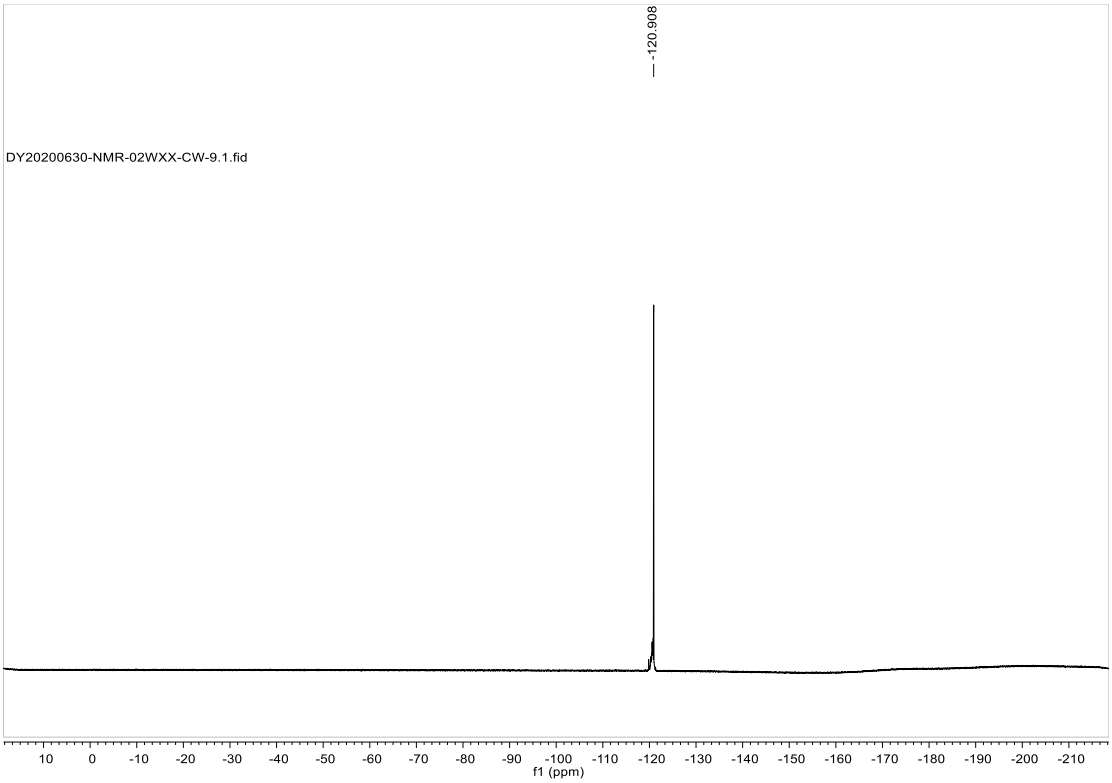

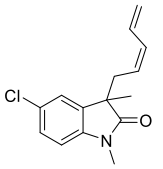

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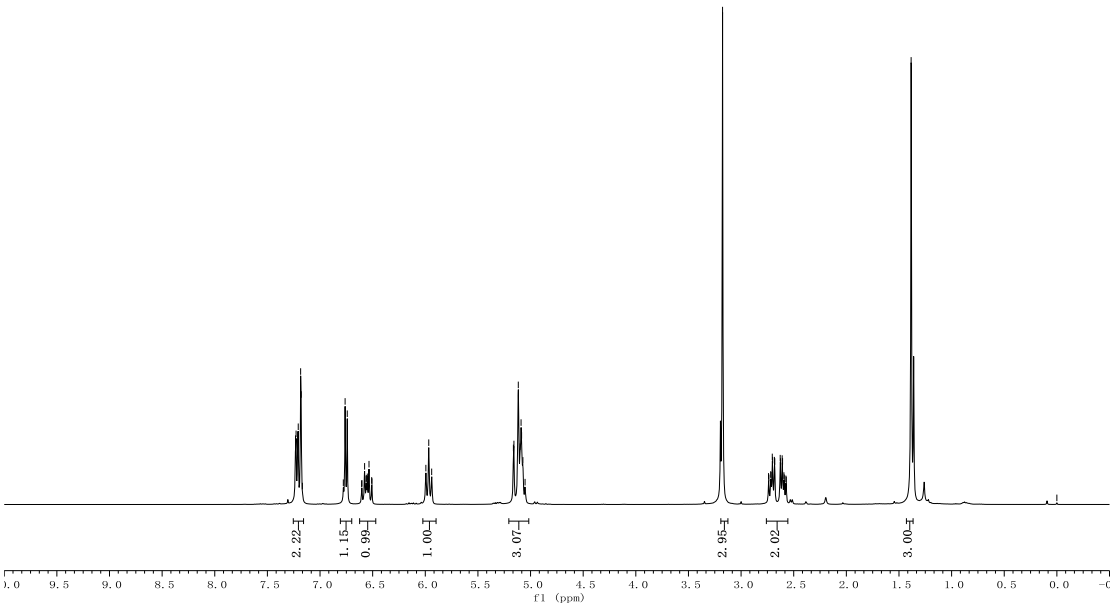

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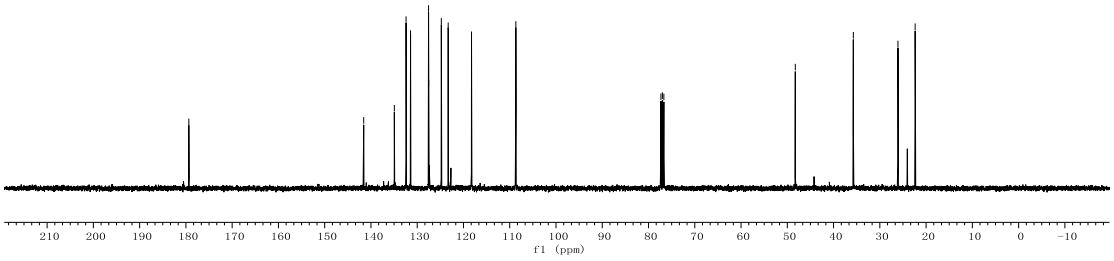

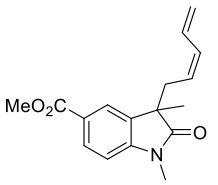

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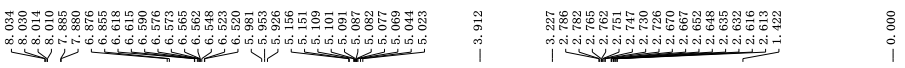
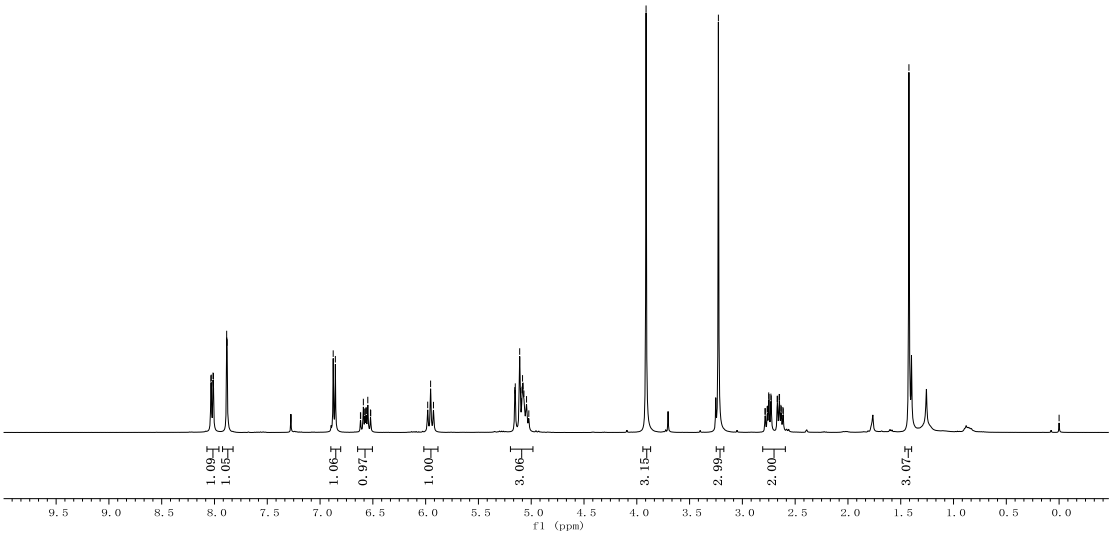
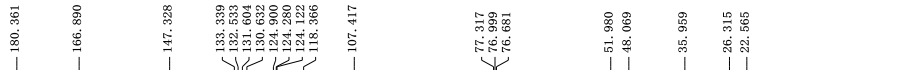
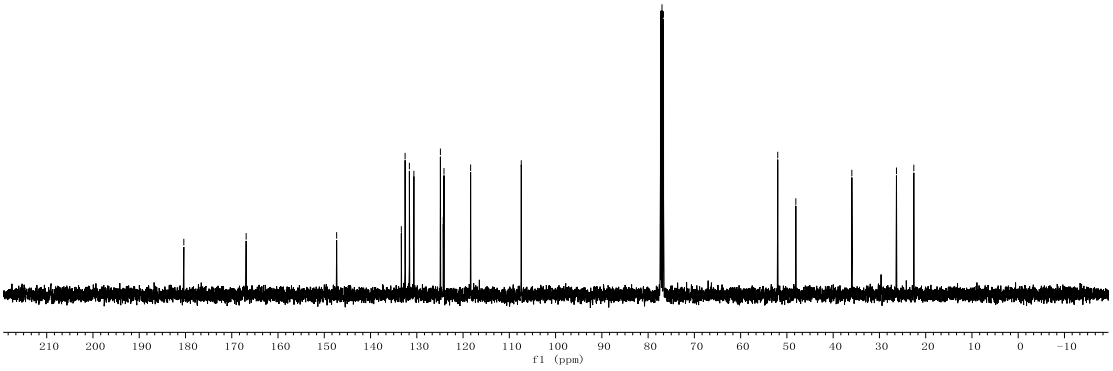


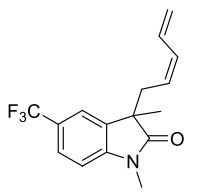
20191107FCO011WXX-CW-2.1.fid



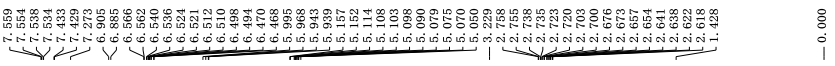


20191108FCO002WXX-CW-2.2.fid

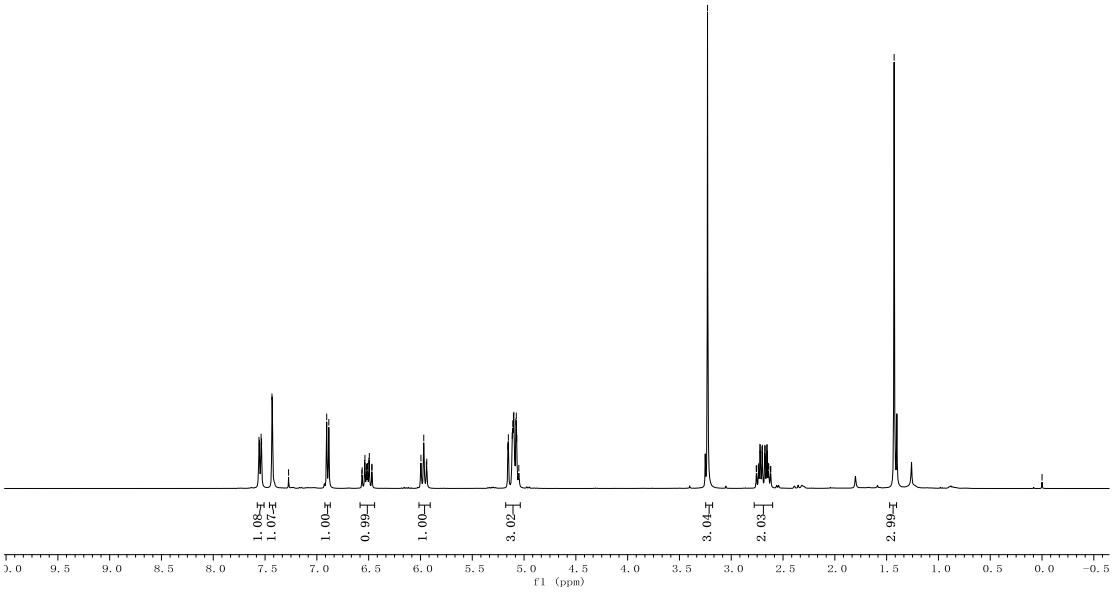


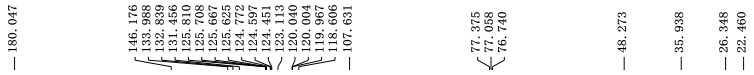


3i (*Z*:*E* = 18:1)

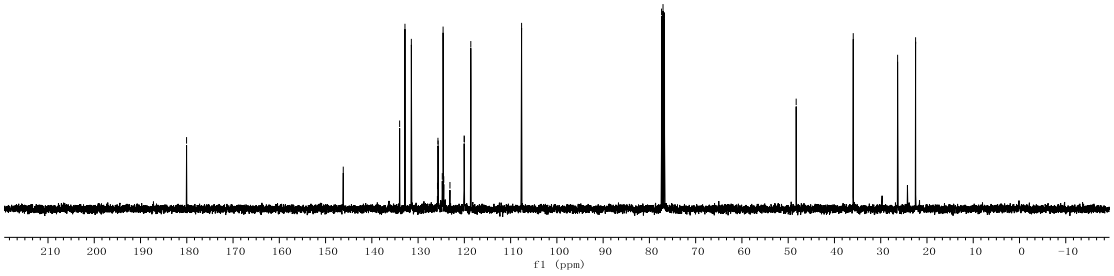


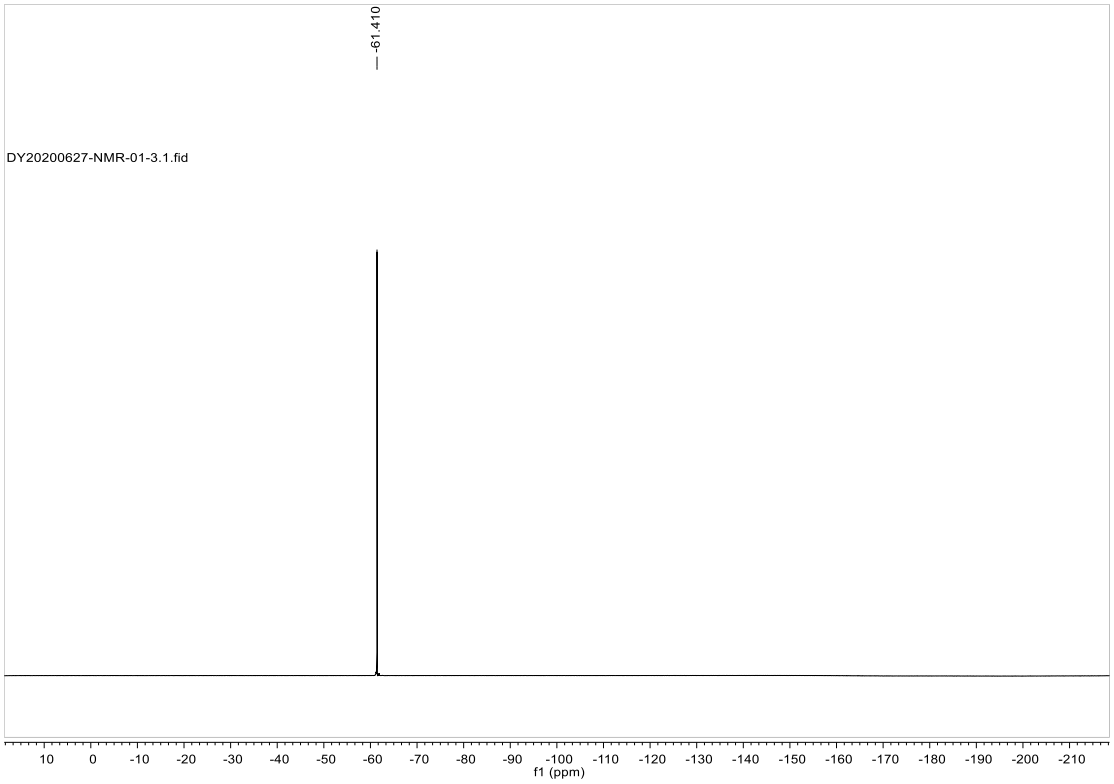
20191107FCO011WXX-CW-3.1.fid

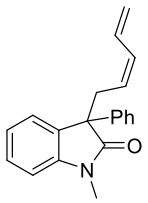




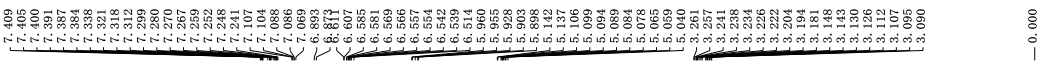
20191108FCO002WXX-CW-3.2.fid



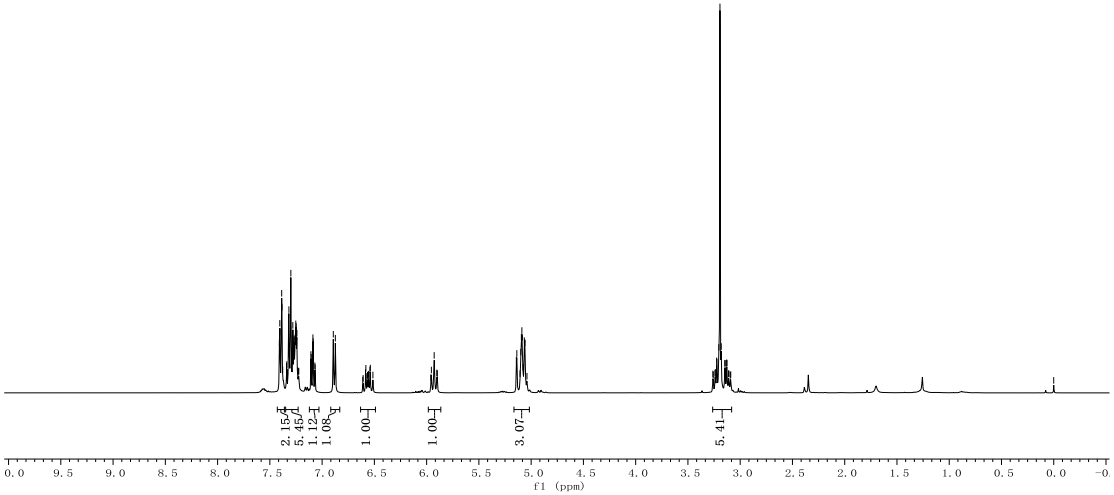




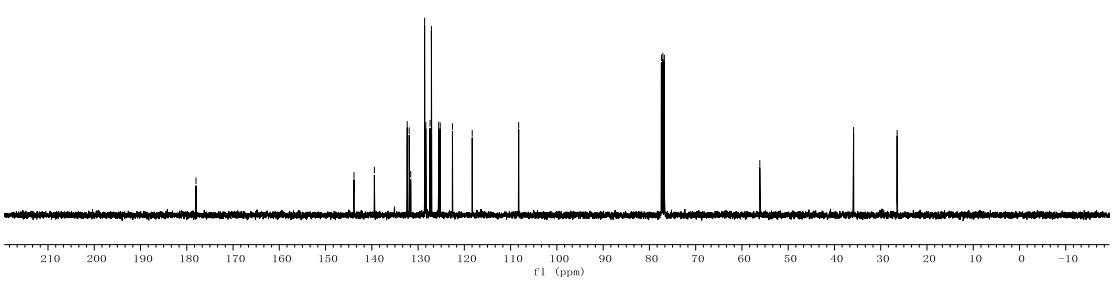
3j (*Z*:*E* = 9:1)

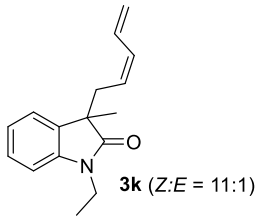


20191107FC0011WXX-CW-16.1.fid

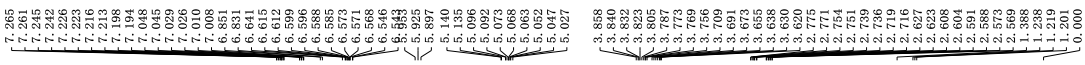


20191108FC0002WXX-CW-16.2.fid

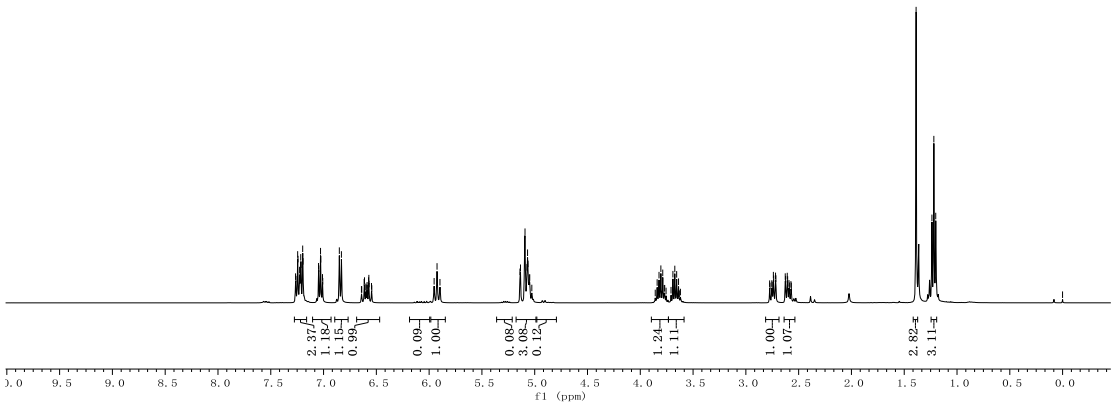




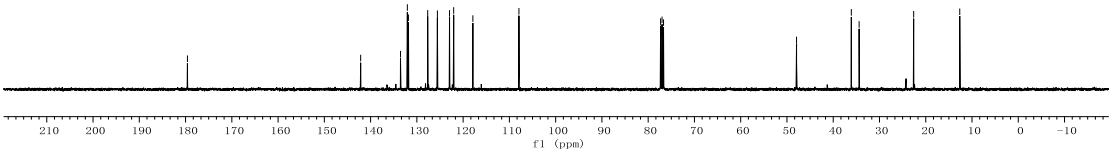
3k (*Z*:*E* = 11:1)

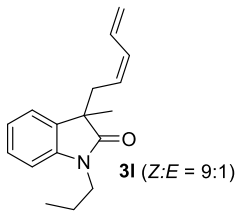


wxx1-cw-11

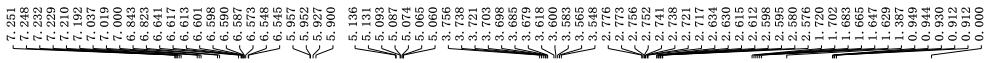


wxx1-cw-11

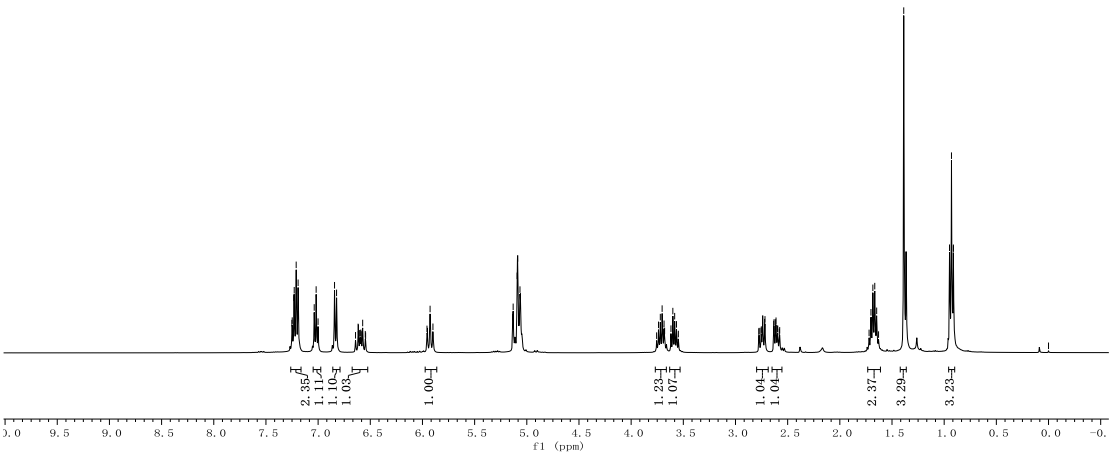




3l (*Z*:*E* = 9:1)

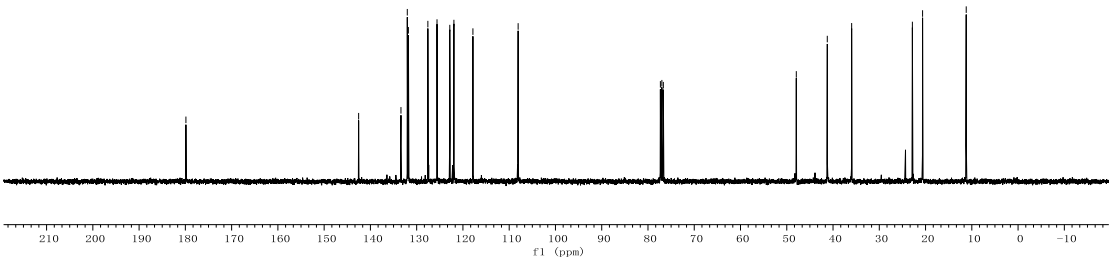


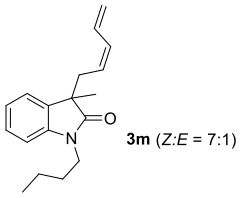
wxx1-cw-12



179.882
142.546
133.396
132.028
131.790
127.582
125.604
122.840
121.950
117.843
108.068
77.318
77.000
76.681
47.940
41.243
35.974
22.848
20.643
11.211

20191106FCO003WXX-CW-12.2.fid

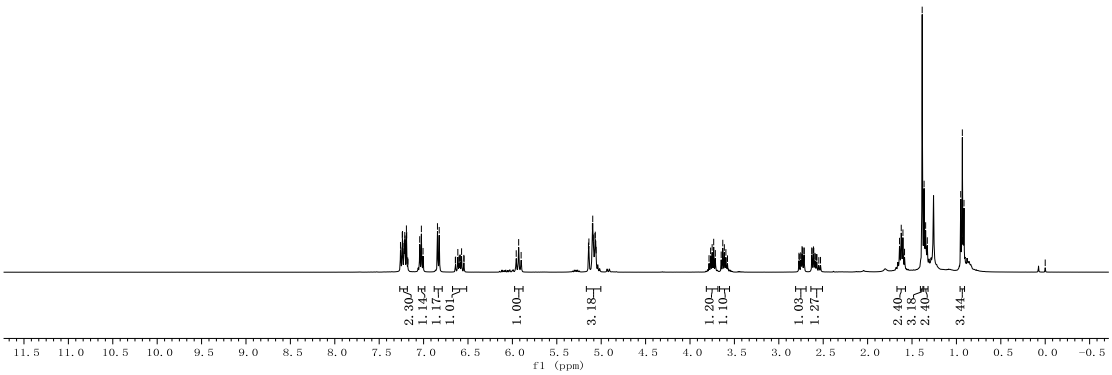




3m (*Z*:*E* = 7:1)

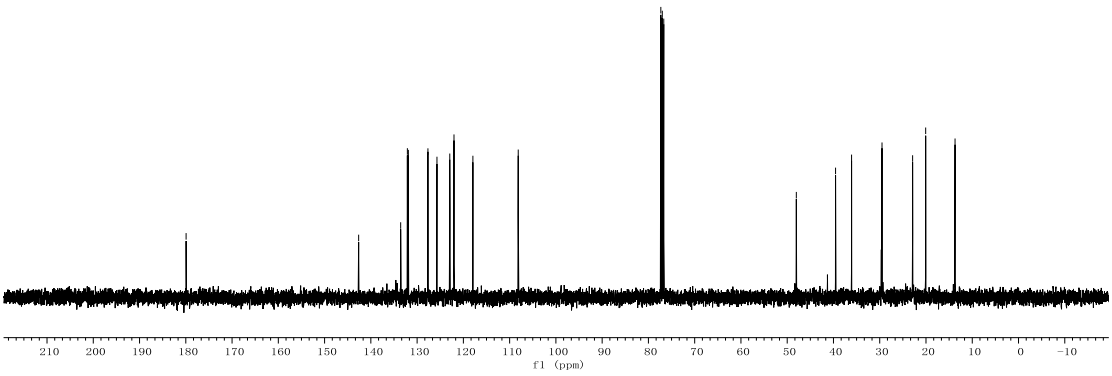
7.260
7.256
7.240
7.237
7.221
7.218
7.212
7.209
7.193
7.190
7.179
7.176
7.043
7.025
7.006
6.844
6.824
6.642
6.640
6.614
6.601
6.598
6.589
6.586
6.572
6.547
6.544
5.957
5.929
5.902
5.144
5.138
5.099
5.095
5.089
5.085
5.071
5.067
5.061
5.057
3.786
3.768
3.751
3.733
3.715
3.649
3.631
3.613
3.596
3.578
3.573
2.770
2.752
2.749
2.738
2.735
2.717
2.714
2.629
2.625
2.610
2.606
2.593
2.590
2.575
2.571
2.549
2.530
1.640
1.621
1.602
1.584
1.385
1.364
1.347
1.328
0.951
0.933
0.915
0.000

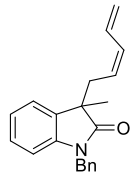
wxx1-cw-13



179.934
142.638
133.551
132.104
131.890
127.663
125.710
122.940
122.027
117.942
108.141
77.318
77.000
76.682
48.029
39.547
36.087
29.508
22.891
20.067
13.719

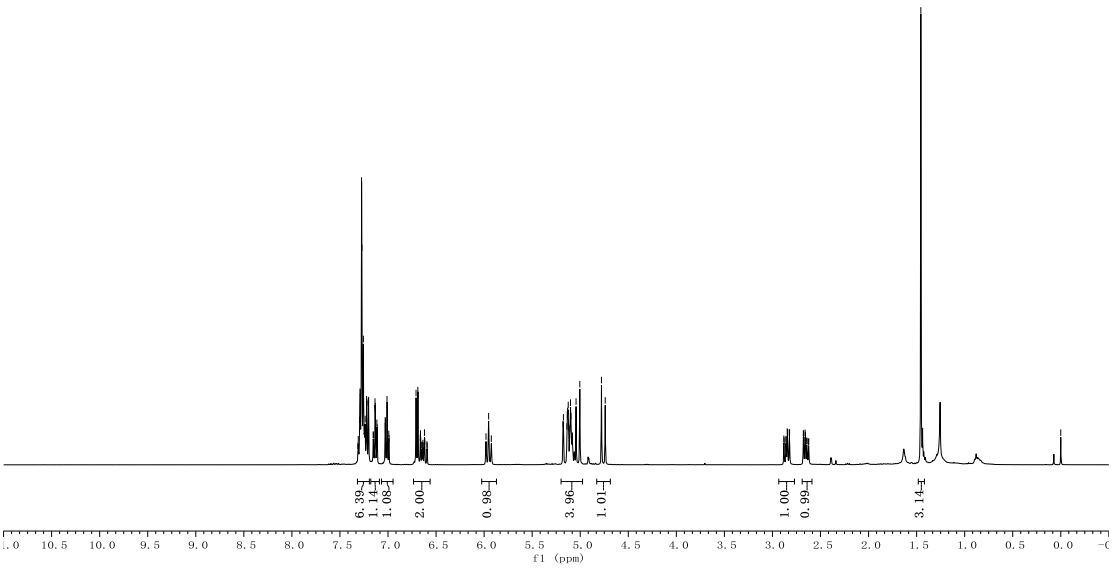
20191031FCO002WXX-CW-13-C13.4.fid

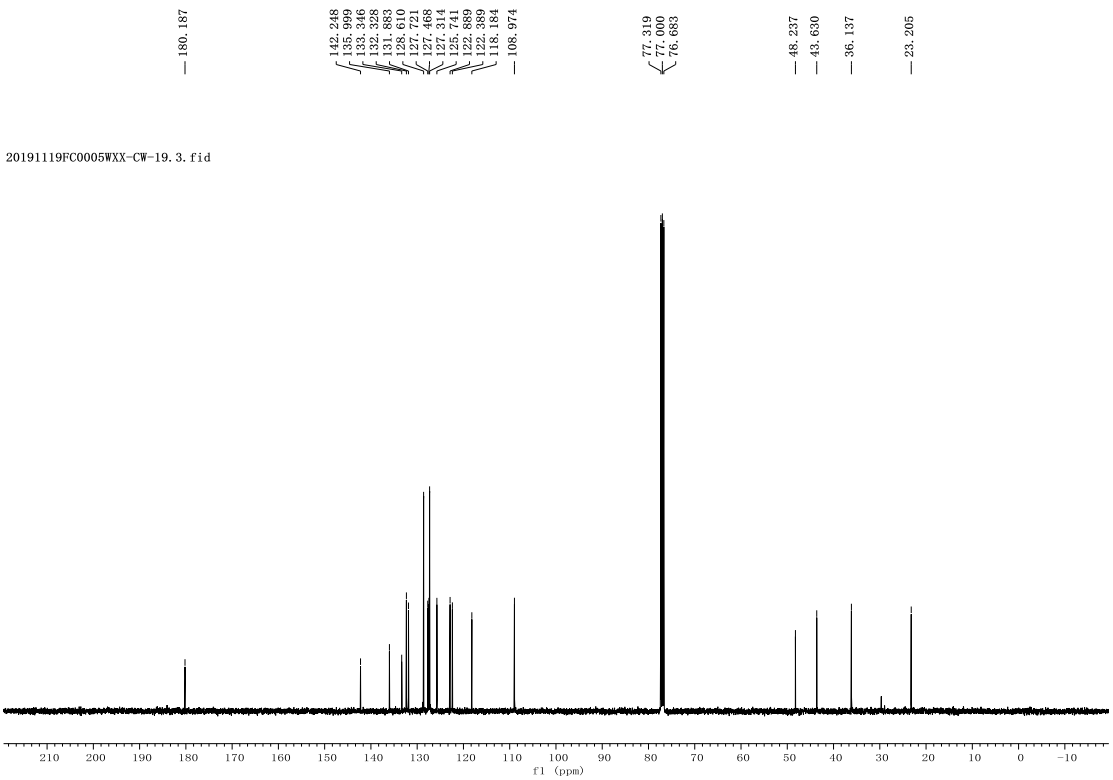


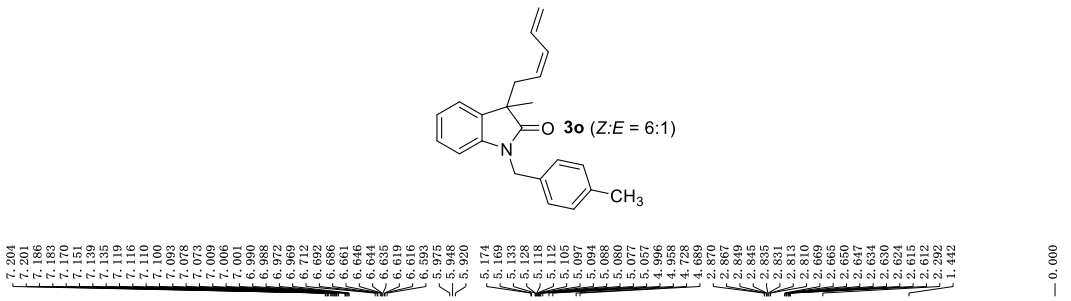


3n (*Z*:*E* = 24:1)

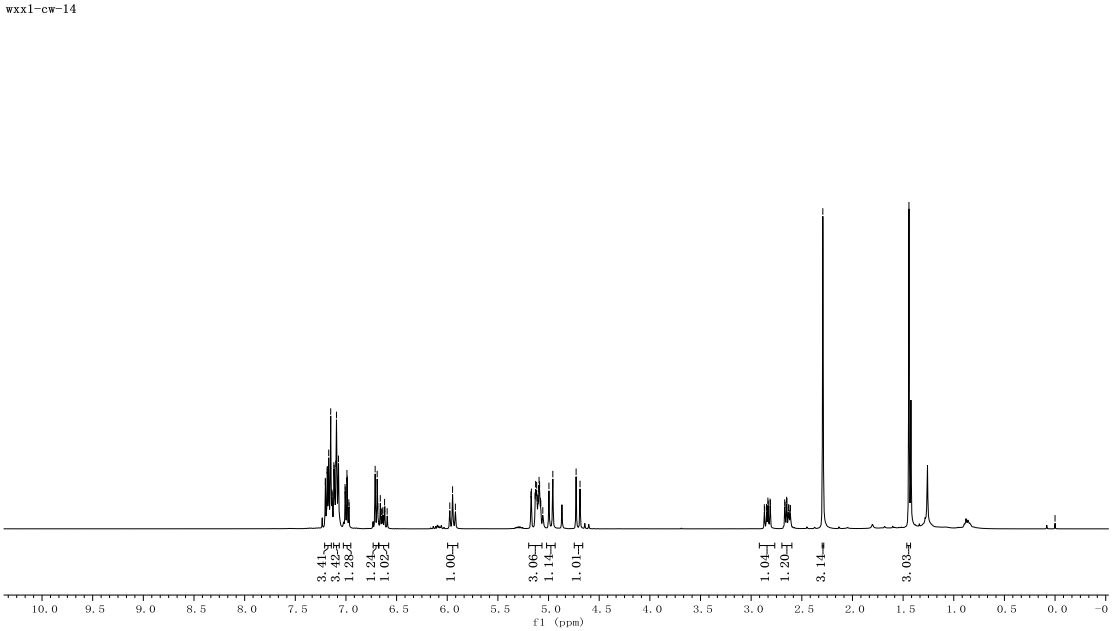
20191119FCO005WXX-CW-19.1.fid



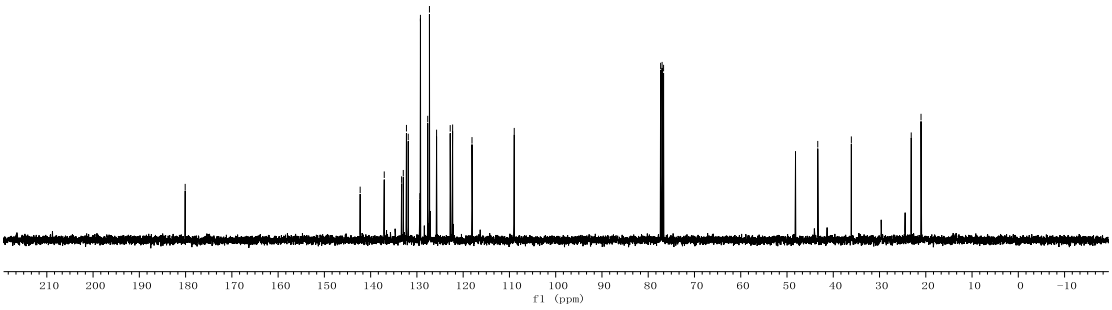

180.187
142.248
135.999
133.346
132.328
131.883
128.610
127.721
127.468
127.314
125.741
122.889
122.389
118.184
108.974
77.319
77.000
76.683
48.237
43.630
36.137
23.205
20191119FCO005WXX-CW-19.3.fid
f1 (ppm)

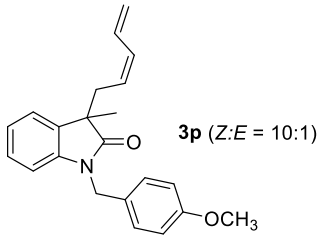

O
N
CH3
7.204
7.201
7.186
7.183
7.170
7.151
7.139
7.135
7.119
7.116
7.110
7.100
7.093
7.078
7.073
7.009
7.006
7.001
6.990
6.988
6.972
6.969
6.712
6.692
6.686
6.661
6.646
6.644
6.635
6.619
6.616
6.593
5.975
5.948
5.920
5.174
5.169
5.133
5.128
5.118
5.112
5.105
5.097
5.094
5.088
5.080
5.077
5.057
4.996
4.958
4.728
4.689
2.870
2.867
2.849
2.845
2.835
2.831
2.813
2.810
2.669
2.665
2.650
2.647
2.634
2.630
2.624
2.615
2.612
2.492
1.442
0.000

3o (*Z*:*E* = 6:1)

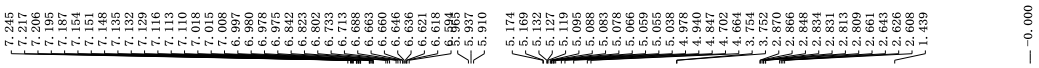

wxx1-cw-14
3.41
3.42
1.28
1.24
1.02
1.00
3.06
1.14
1.01
1.04
1.20
3.14
3.03
f1 (ppm)

20191031fc0002wxx-cw-14-C13.2.fid

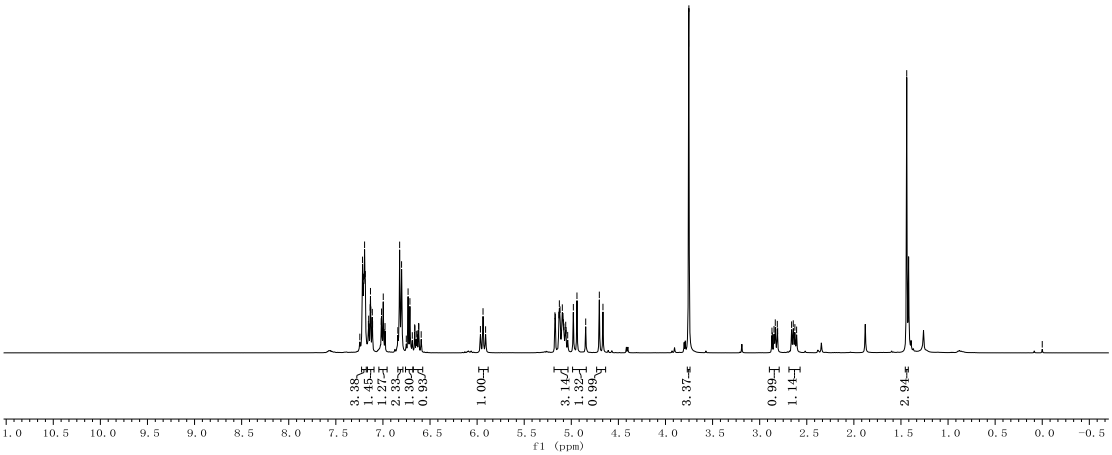


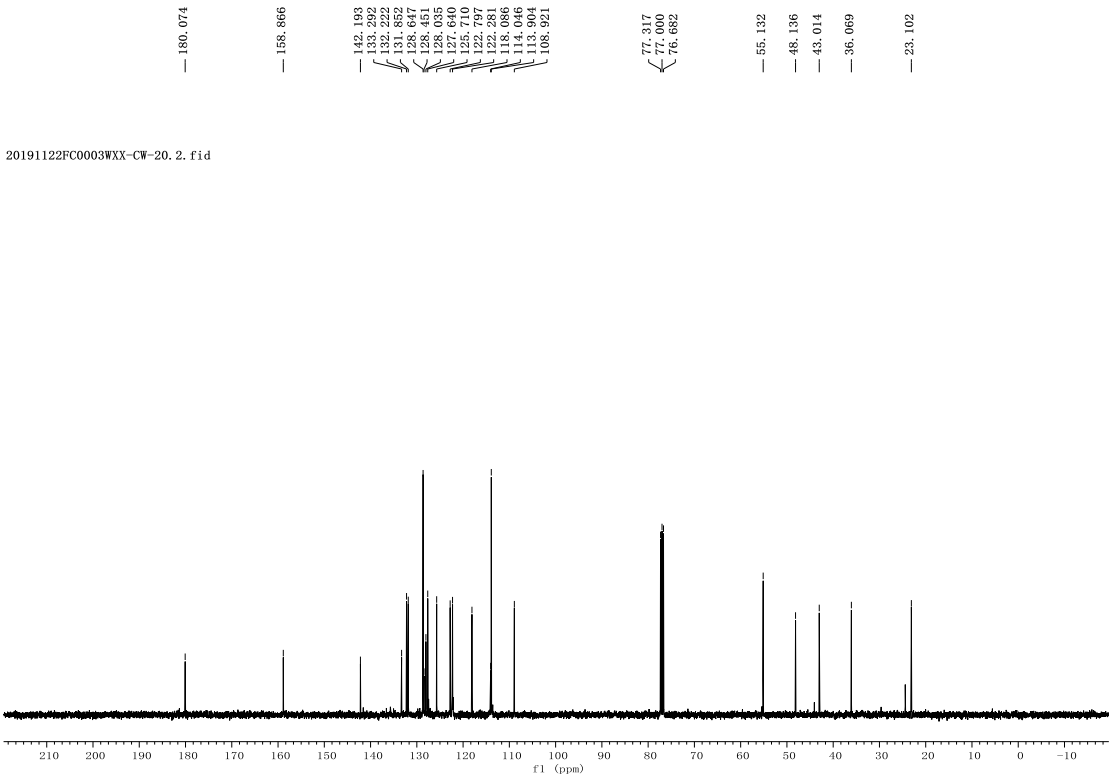


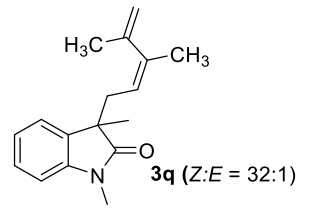
3p (*Z*:*E* = 10:1)



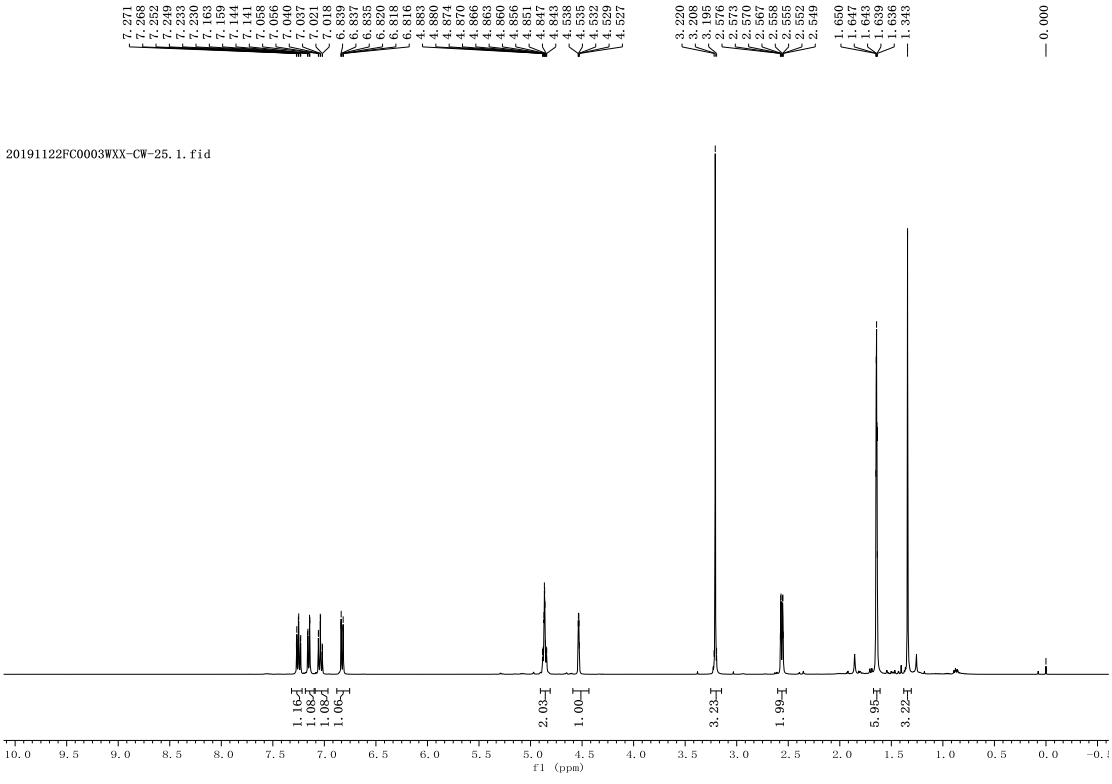
20191122FC0003WXX-CW-20.1.fid

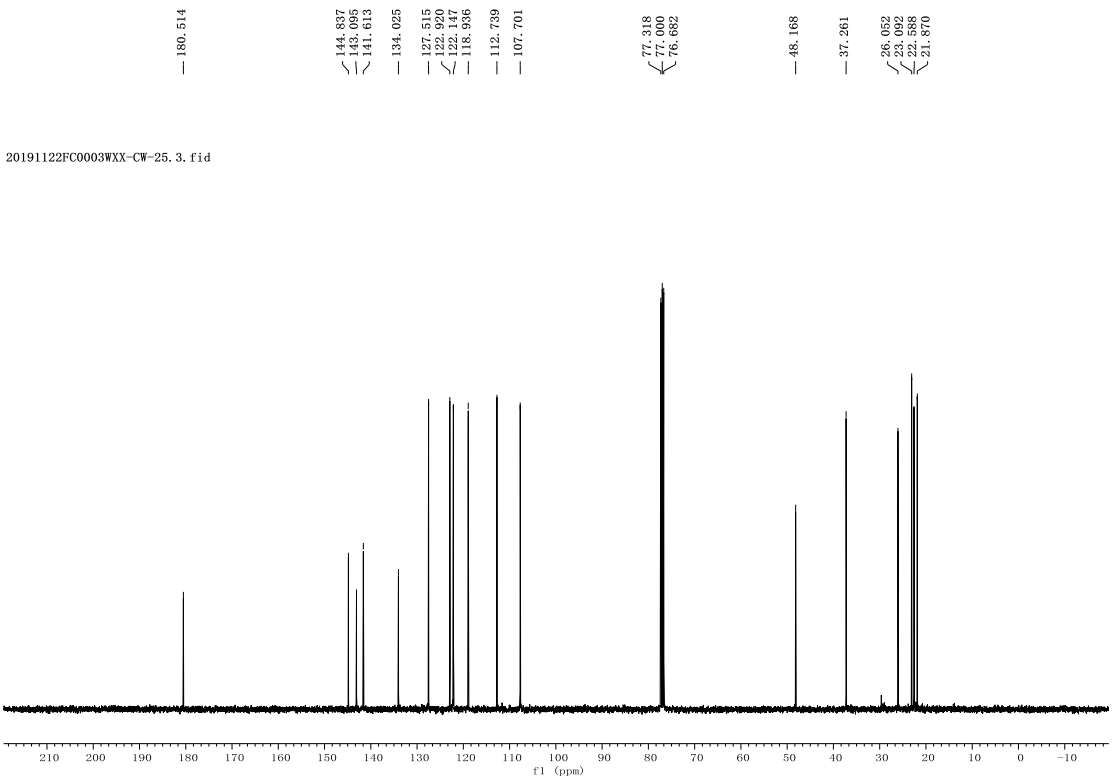


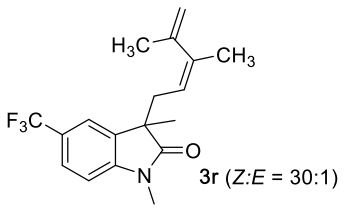

180.074
158.866
142.193
133.292
132.222
131.852
128.647
128.451
128.035
127.640
125.710
122.797
122.281
118.086
114.046
113.994
108.921
77.317
77.000
76.682
55.132
48.136
43.014
36.069
23.102
20191122FCO003WXX-CW-20.2.fid
f1 (ppm)


H3C
CH3
O
N

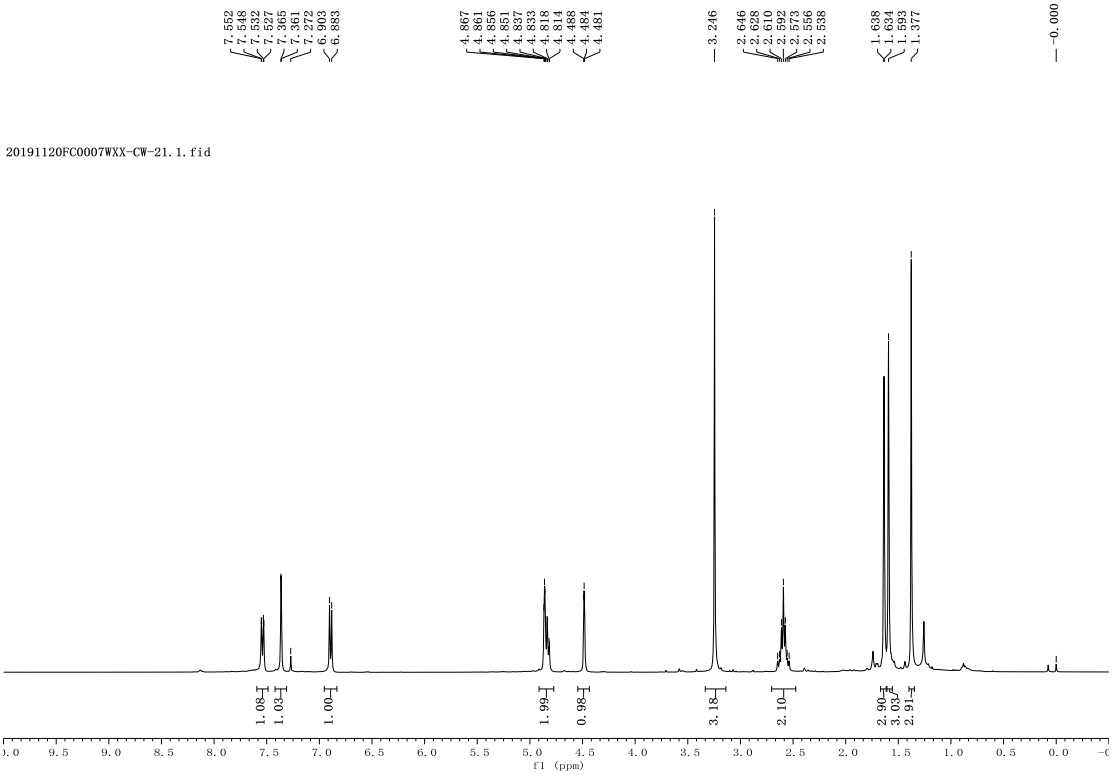
3q (*Z*:*E* = 32:1)


7.271
7.268
7.252
7.249
7.233
7.230
7.163
7.159
7.144
7.141
7.058
7.056
7.040
7.037
7.021
7.018
6.839
6.837
6.835
6.820
6.818
6.816
4.883
4.880
4.874
4.870
4.866
4.863
4.860
4.856
4.851
4.847
4.843
4.538
4.535
4.532
4.529
4.527
3.220
3.208
3.195
2.576
2.573
2.570
2.567
2.558
2.555
2.552
2.549
1.650
1.647
1.643
1.639
1.636
1.343
0.000
20191122FCO003WXX-CW-25.1.fid
1.16
1.08
1.08
1.06
2.03
1.00
3.23
1.99
5.95
3.22
f1 (ppm)

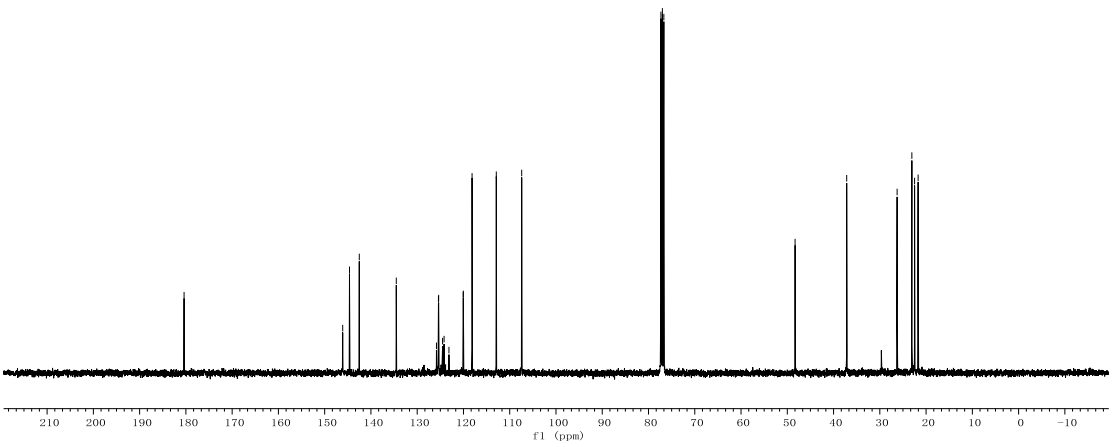

180.514
144.837
143.095
141.613
134.025
127.515
122.920
122.147
118.936
112.739
107.701
77.318
77.000
76.682
48.168
37.261
26.052
23.092
22.588
21.870
20191122FC0003WXX-CW-25.3.fid
f1 (ppm)

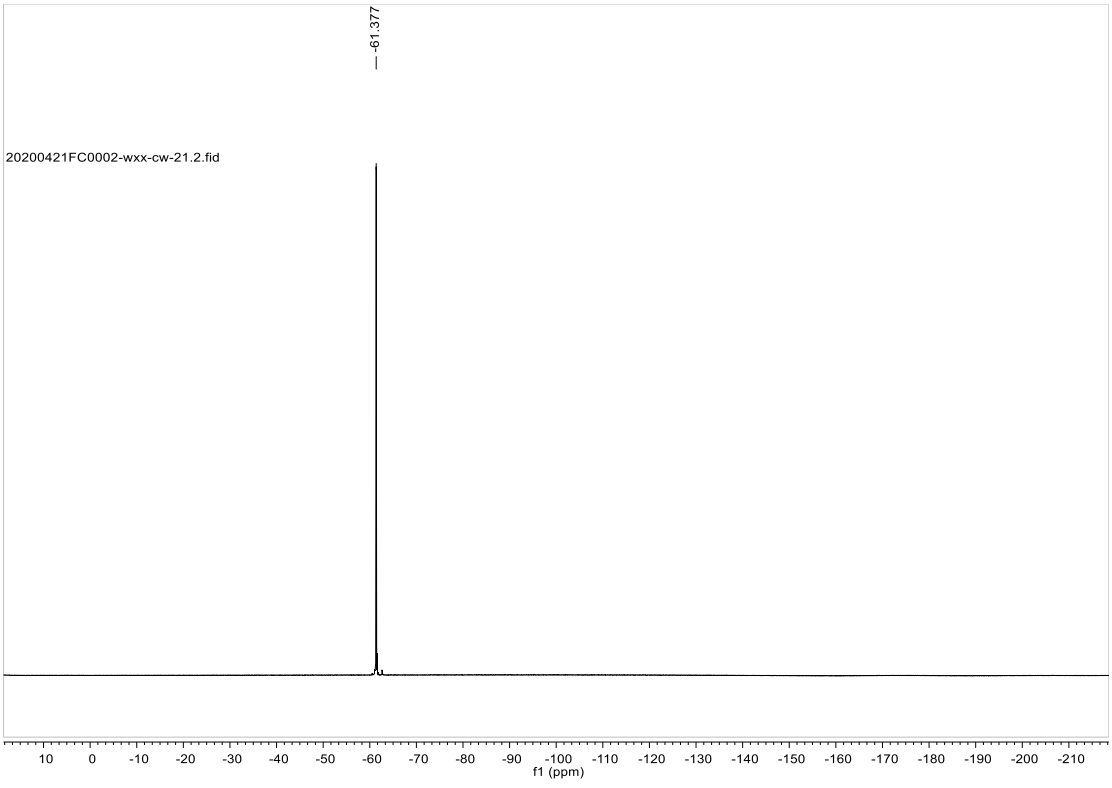

H3C
CH3
F3C
O
N

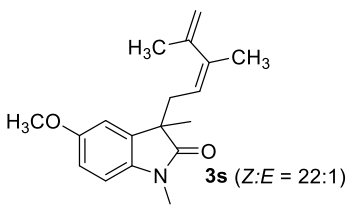
3r (*Z*:*E* = 30:1)


7.552
7.548
7.532
7.527
7.365
7.361
7.272
6.903
6.883
4.867
4.861
4.856
4.851
4.837
4.833
4.818
4.814
4.488
4.484
4.481
3.246
2.646
2.628
2.610
2.592
2.573
2.556
2.538
1.638
1.634
1.593
1.377
0.000
20191120FC0007WXX-CW-21.1.fid
1.08
1.03
1.00
1.99
0.98
3.18
2.10
2.90
3.03
2.91
f1 (ppm)

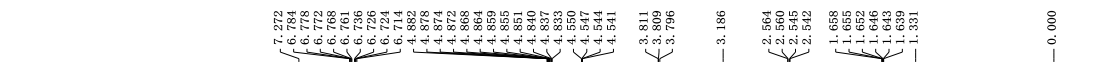
20191120FC0007WXX-CW-21.2.fid



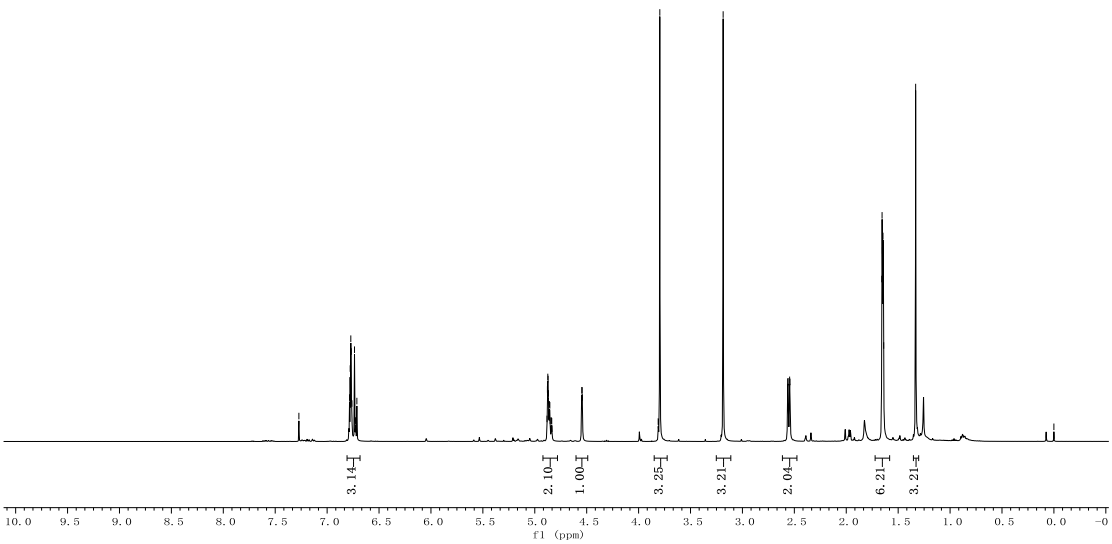


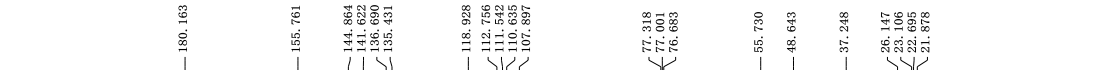


3s (*Z*:*E* = 22:1)

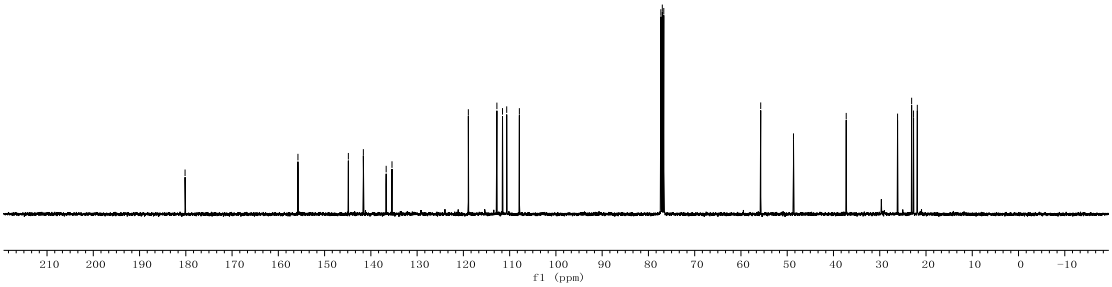


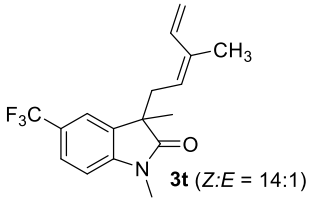
20191122FCO003WXX-CW-26.1.fid



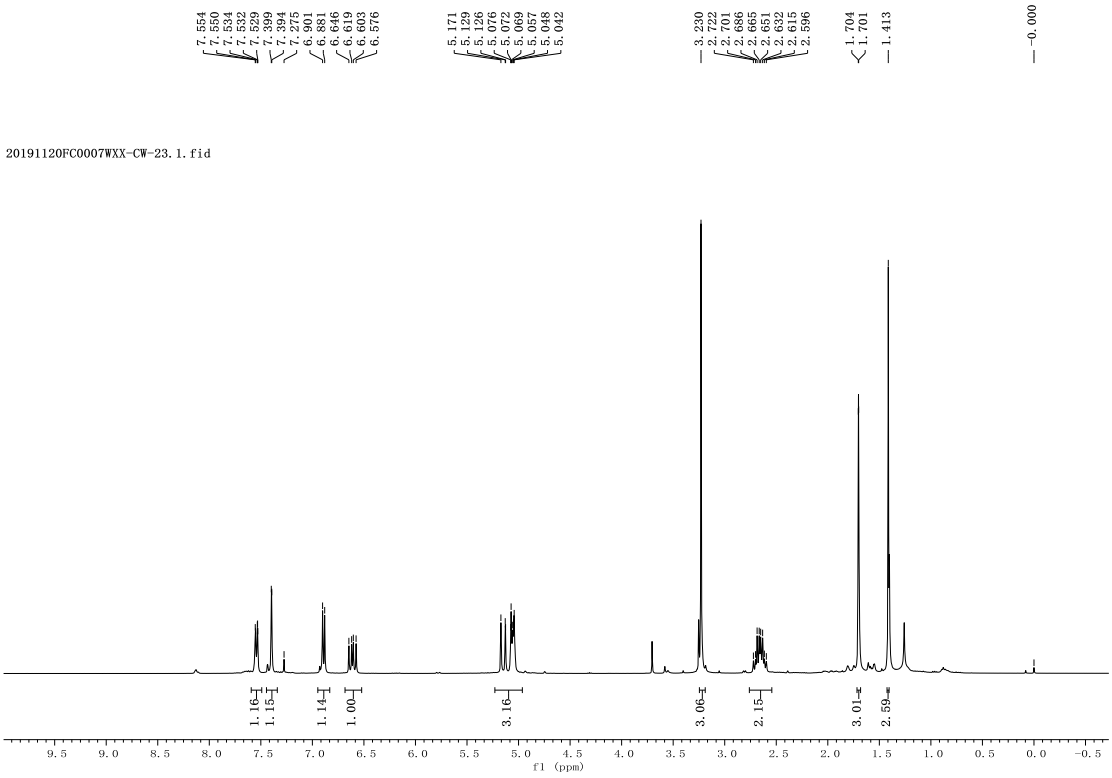


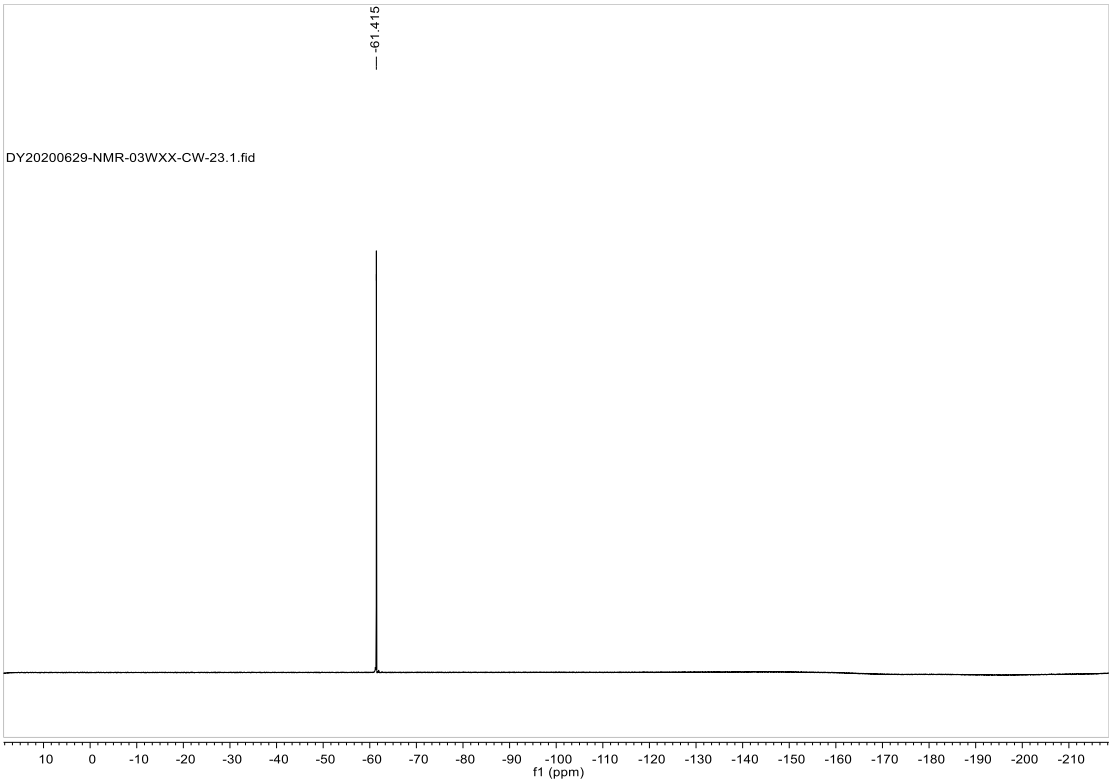
20191122FCO003WXX-CW-26.3.fid

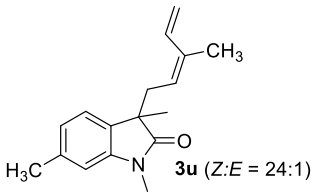




3t (*Z*:*E* = 14:1)


7.554
7.550
7.534
7.532
7.529
7.399
7.394
7.275
6.681
6.651
6.646
6.619
6.603
6.576
5.171
5.129
5.126
5.076
5.072
5.069
5.057
5.048
5.042
3.230
2.722
2.701
2.686
2.665
2.651
2.632
2.615
2.596
1.704
1.701
1.413
-0.000
20191120FCO007WXX-CW-23.1.fid
1.16
1.15
1.14
1.00
3.16
3.06
2.15
3.01
2.59
f1 (ppm)
180.159
146.054
136.025
134.047
132.885
125.762
125.598
125.557
125.516
125.476
124.635
124.312
123.063
122.961
120.128
120.091
120.055
120.018
114.667
107.695
107.491
77.317
77.000
76.682
48.300
35.483
26.282
22.249
19.626
20191120FCO007WXX-CW-23.2.fid
f1 (ppm)

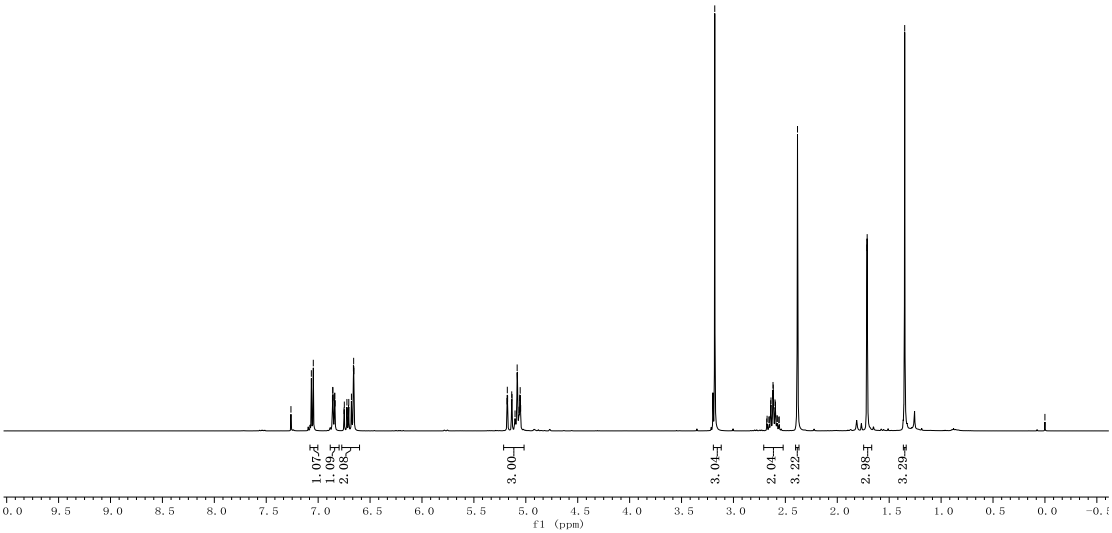




3u (*Z*:*E* = 24:1)

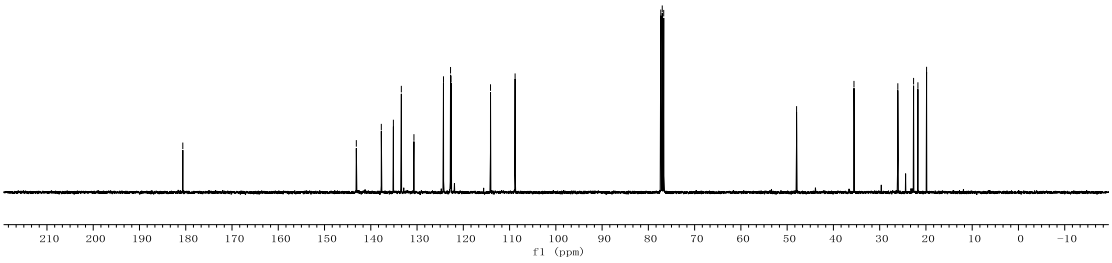
7.262
7.065
7.047
6.862
6.860
6.858
6.856
6.843
6.841
6.839
6.837
6.751
6.748
6.723
6.720
6.707
6.705
6.679
6.660
6.658
6.656
5.181
5.178
5.175
5.138
5.136
5.133
5.104
5.083
5.080
5.065
5.058
5.054
5.050
3.180
2.680
2.676
2.658
2.655
2.642
2.639
2.622
2.619
2.616
2.613
2.599
2.595
2.581
2.578
2.563
2.559
2.383
1.715
1.712
1.351
0.000

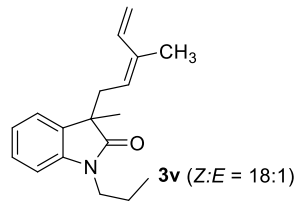
20191126FCO006WXX-CW-27.1.fid



180.635
143.128
137.725
135.114
133.397
130.674
124.287
122.764
122.653
114.120
108.807
77.316
76.999
76.681
47.962
35.553
26.078
22.671
21.735
19.861

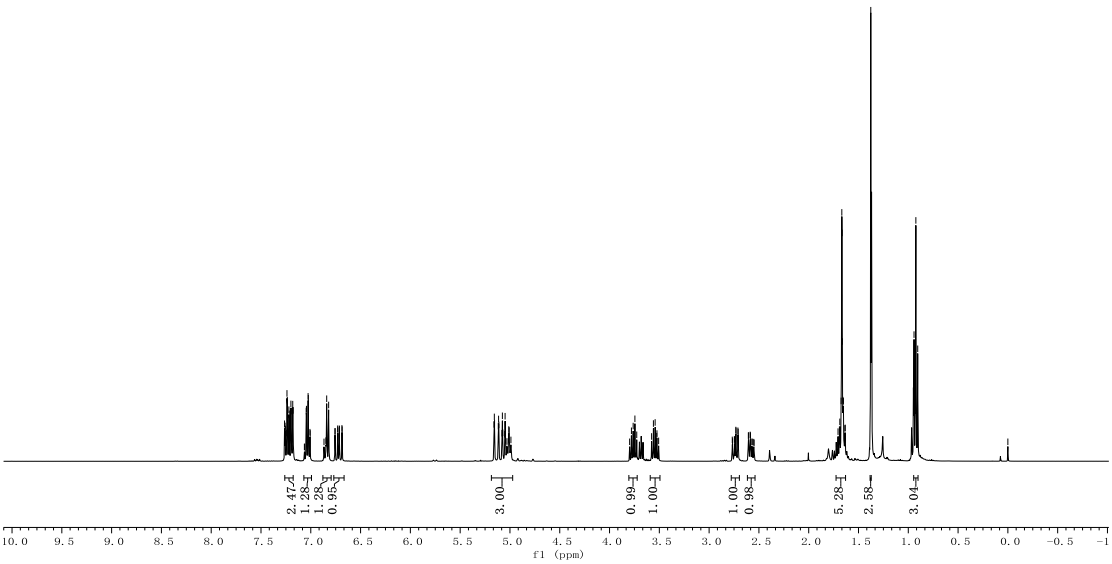
20191127FC0003WXX-CW-27.1.fid





3v (*Z*:*E* = 18:1)

20191126FC0006WXX-CW-28.1.fid



180.132
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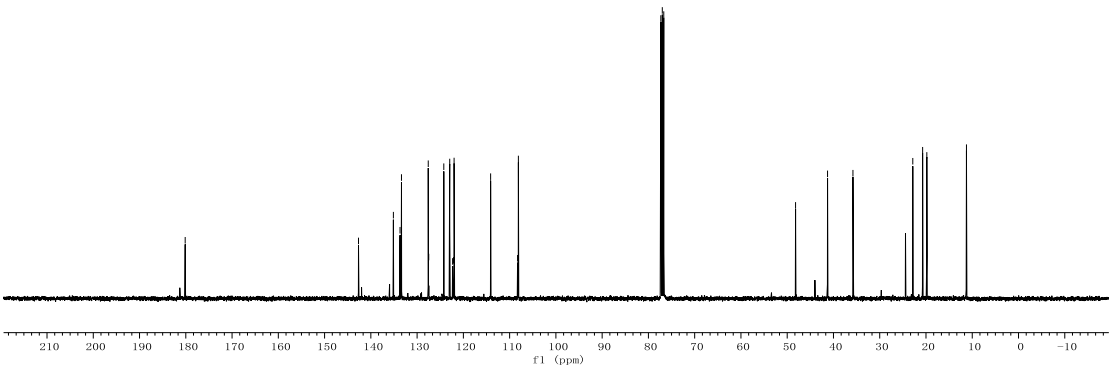
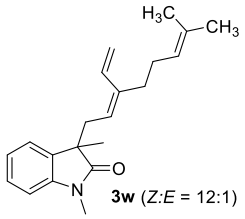
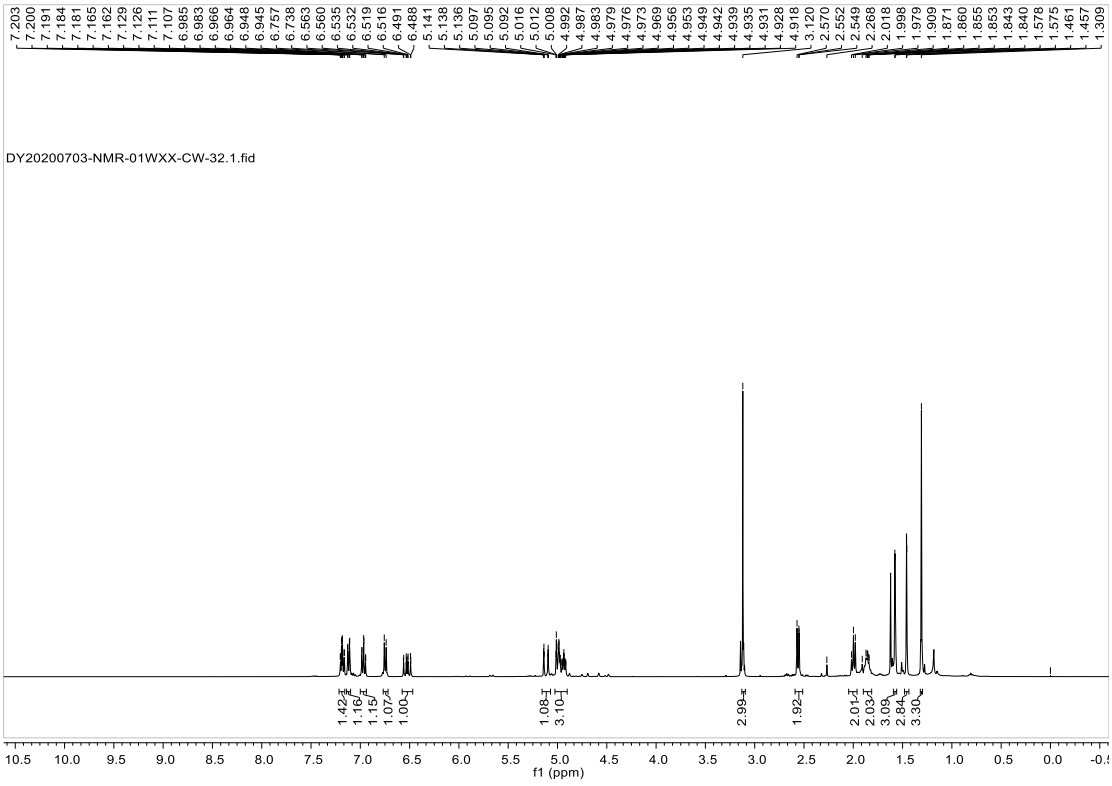
135.126
133.690
133.378
127.592
127.488
124.229
122.949
122.331
122.125
122.005
114.109
108.287
108.109
77.318
77.000
76.683
48.193
41.280
35.789
22.851
20.726
19.819
11.249

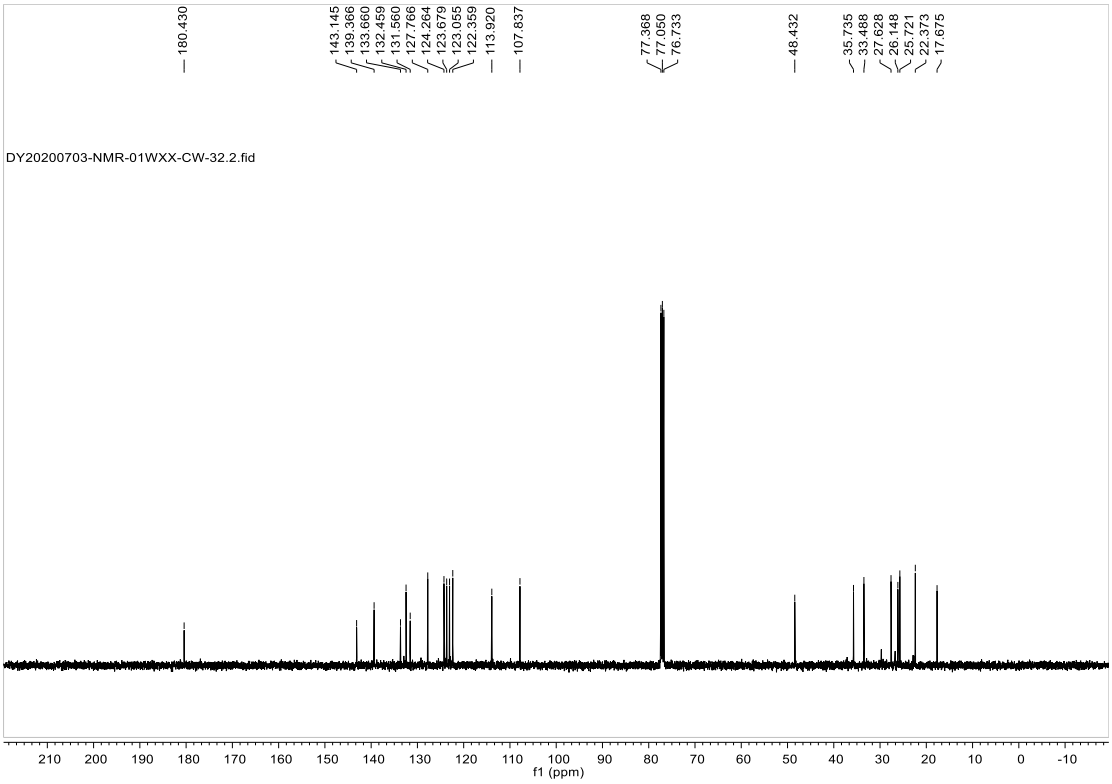
20191127FC0003WXX-CW-28.1.fid

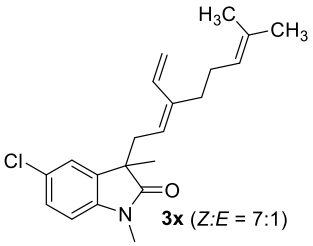




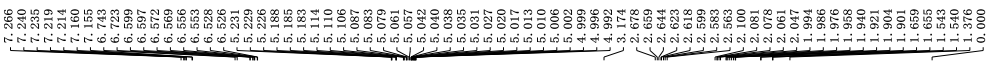
3w (*Z*:*E* = 12:1)



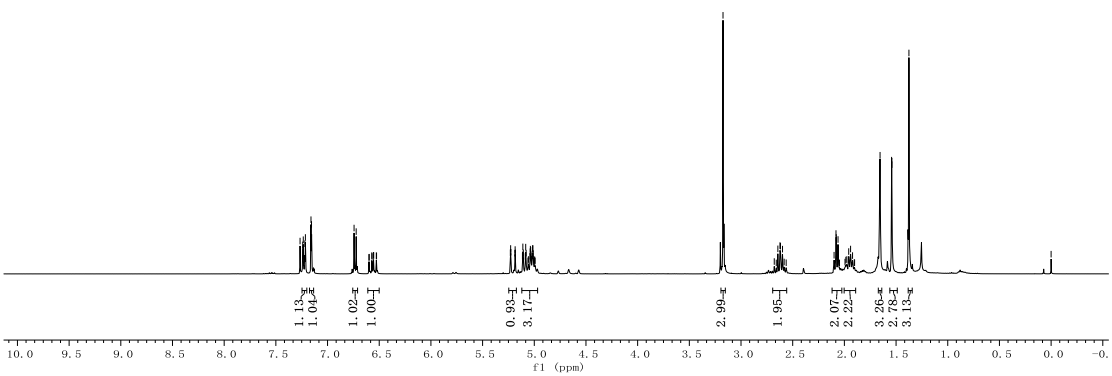




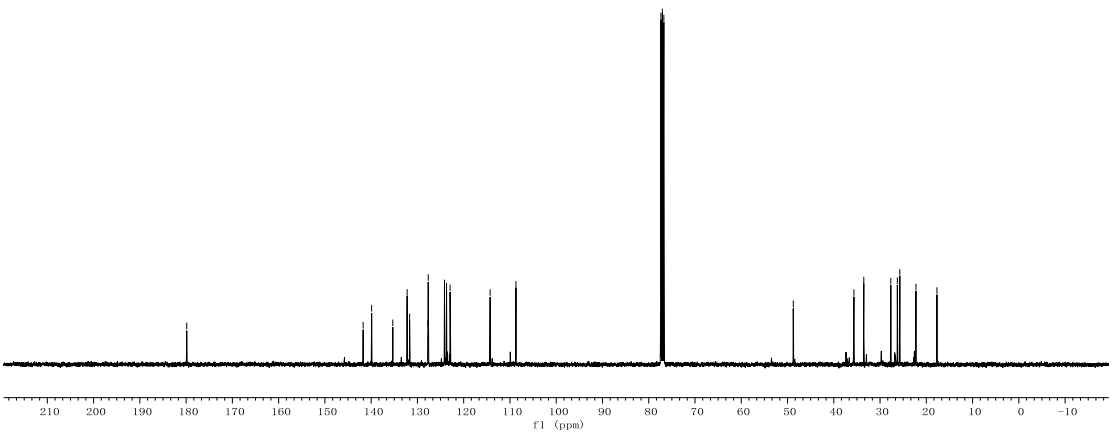
3x (*Z*:*E* = 7:1)

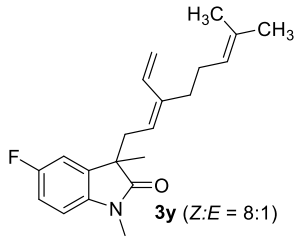


20191126FC0006WXX-CW-31.1.fid

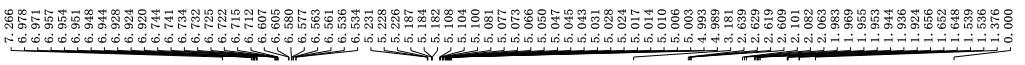


20191127FCO003WXX-CW-31.1.fid

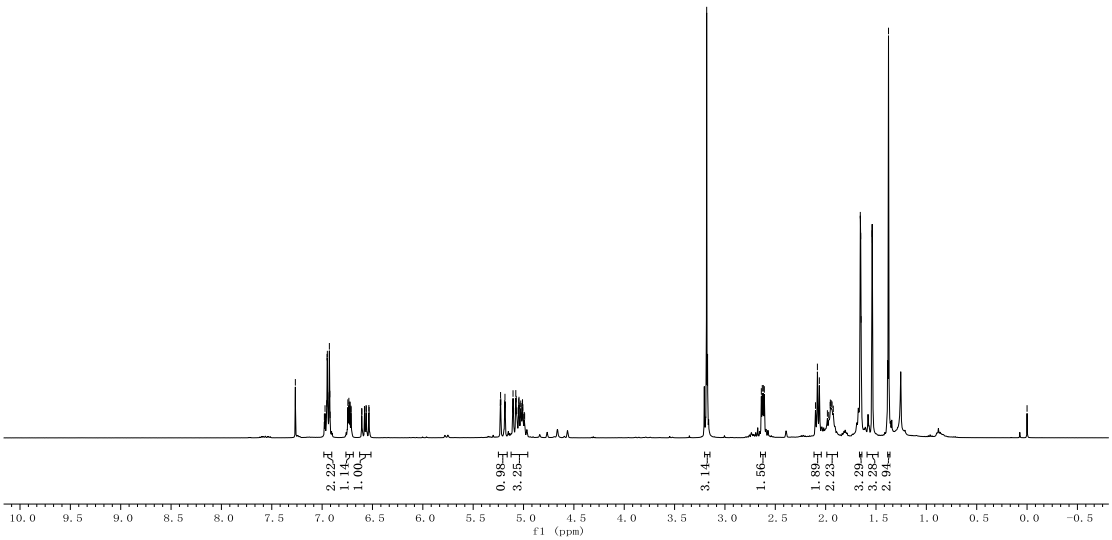


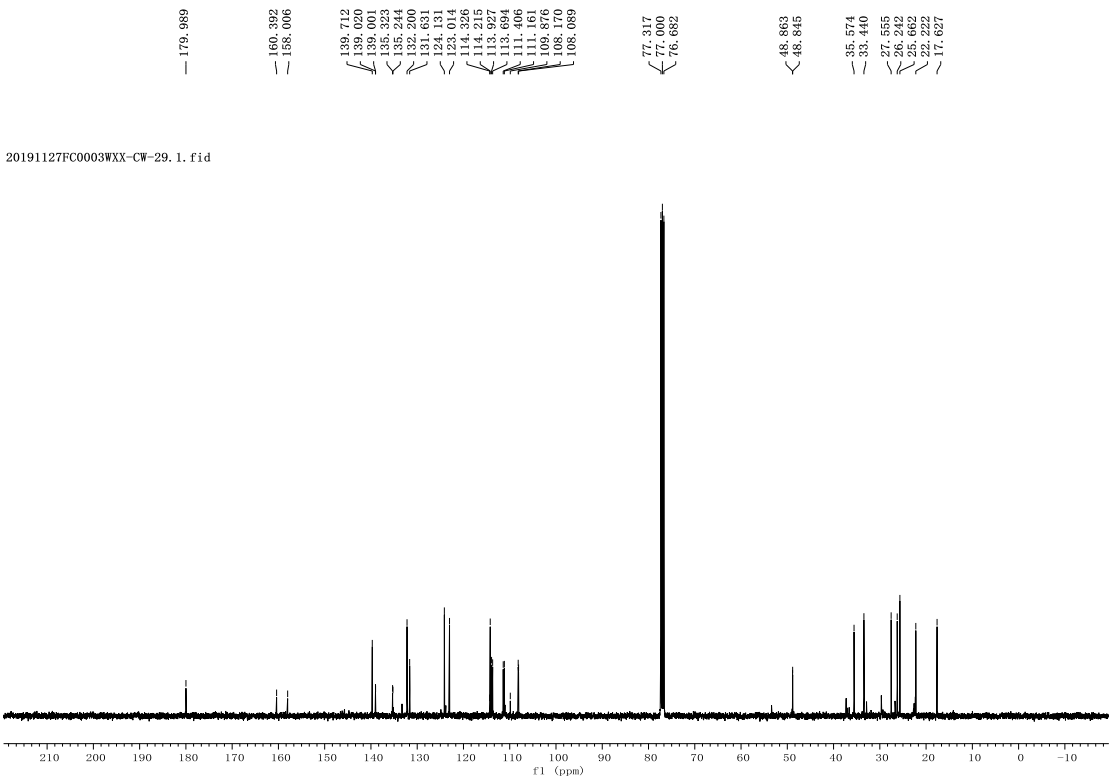


3y (*Z*:*E* = 8:1)



20191126FCO006WXX-CW-29.1.fid

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158.006
139.712
139.020
139.001
135.323
135.244
132.200
131.631
124.131
123.014
114.326
114.215
113.927
113.694
111.496
111.161
109.876
108.170
108.089
77.317
77.000
76.682
48.863
48.845
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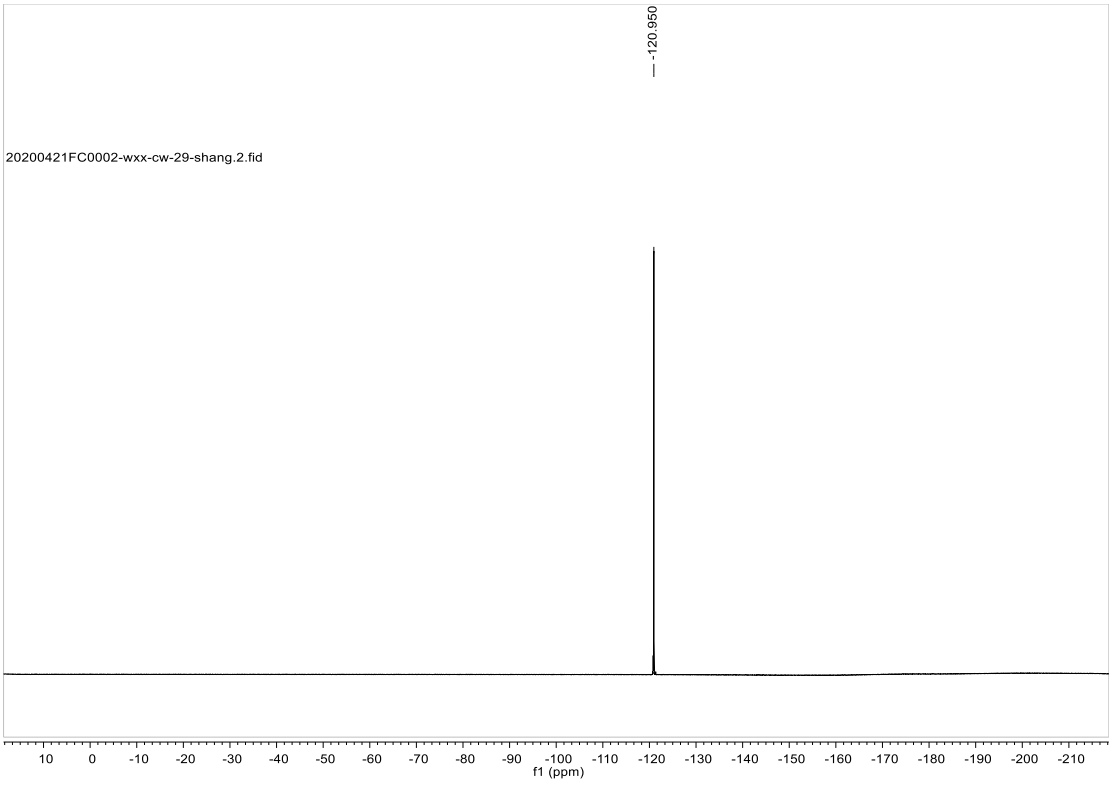
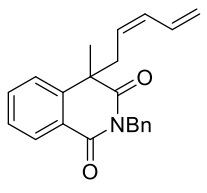
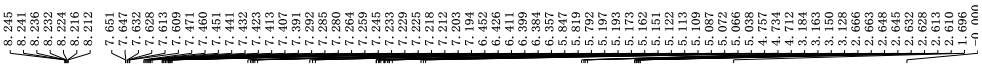
33.440
27.555
26.242
25.662
22.222
17.627
20191127FC0003WXX-CW-29.1.fid
f1 (ppm)


-120.950
20200421FC0002-wxx-cw-29-shang.2.fid
f1 (ppm)


O
NBn
O

3z (*Z*:*E* = 11:1)



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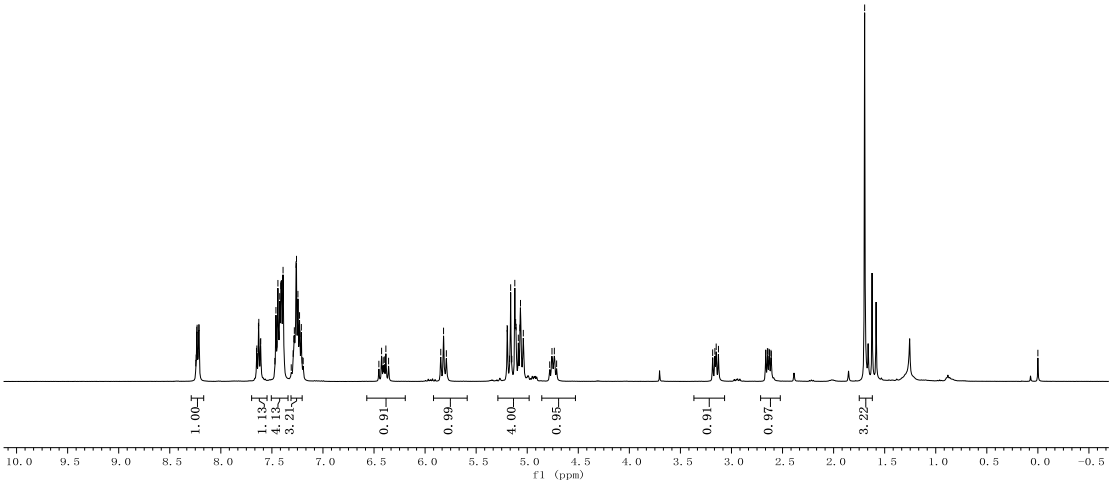
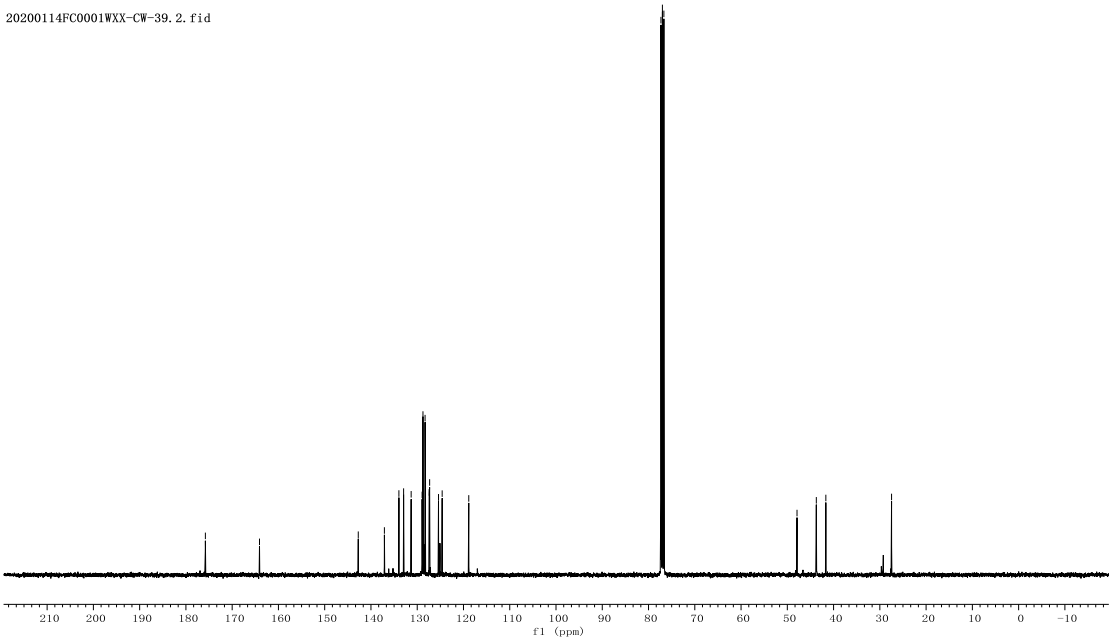
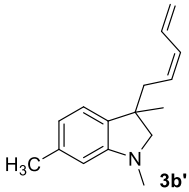
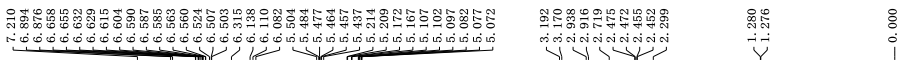


20200114FC0001WXX-CW-39.2.fid

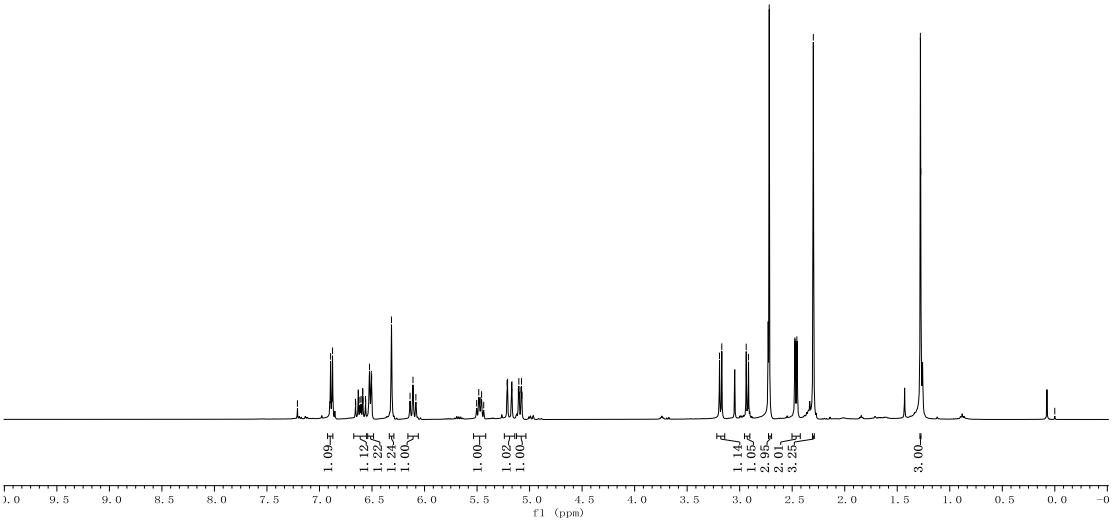




3b' (*Z*:*E* = 10:1)



20191213FCO004WXX-CW-8-HUANYUAN.2.fid



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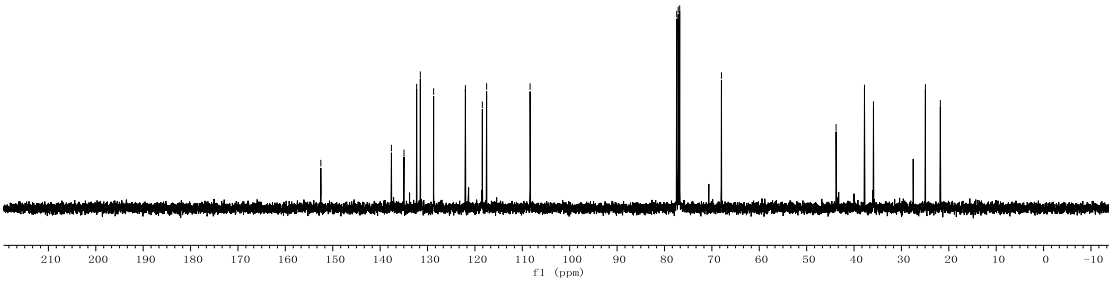
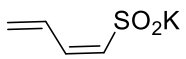
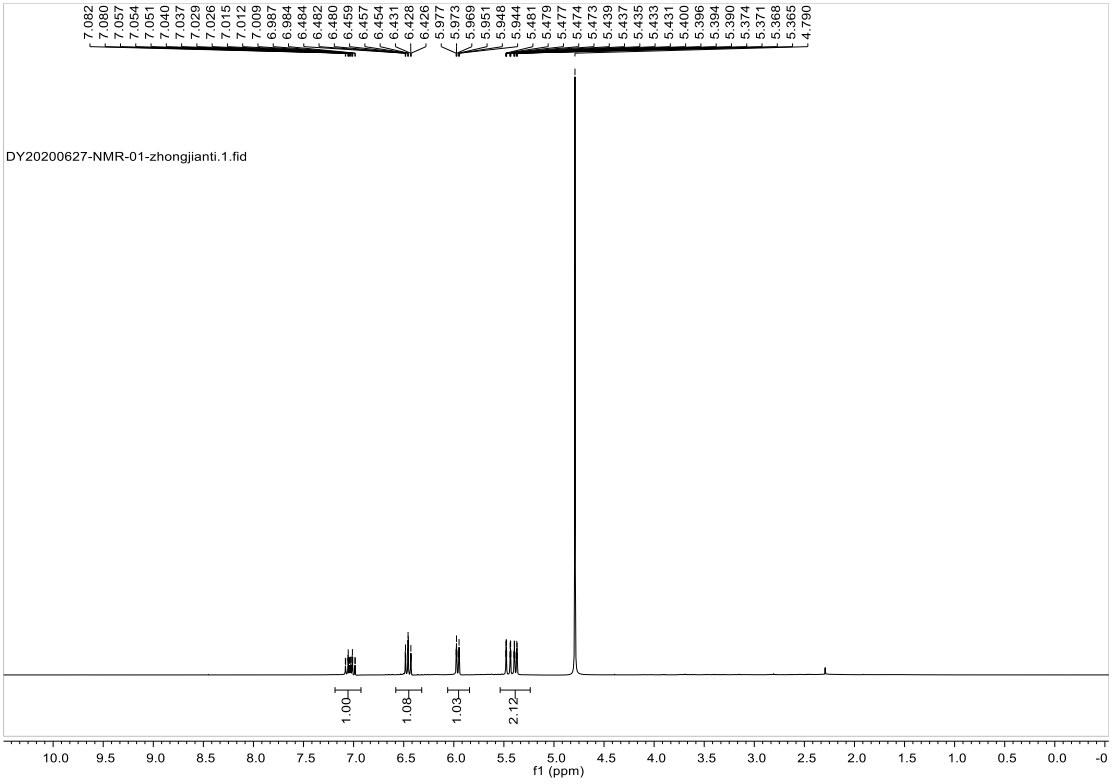



SO2K

5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7.057
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7.040
7.037
7.029
7.026
7.015
7.012
7.009
6.987
6.984
6.484
6.482
6.480
6.459
6.457
6.454
6.431
6.428
6.426
5.977
5.973
5.969
5.951
5.948
5.944
5.481
5.479
5.477
5.474
5.473
5.439
5.437
5.435
5.433
5.431
5.400
5.396
5.394
5.390
5.374
5.371
5.368
5.365
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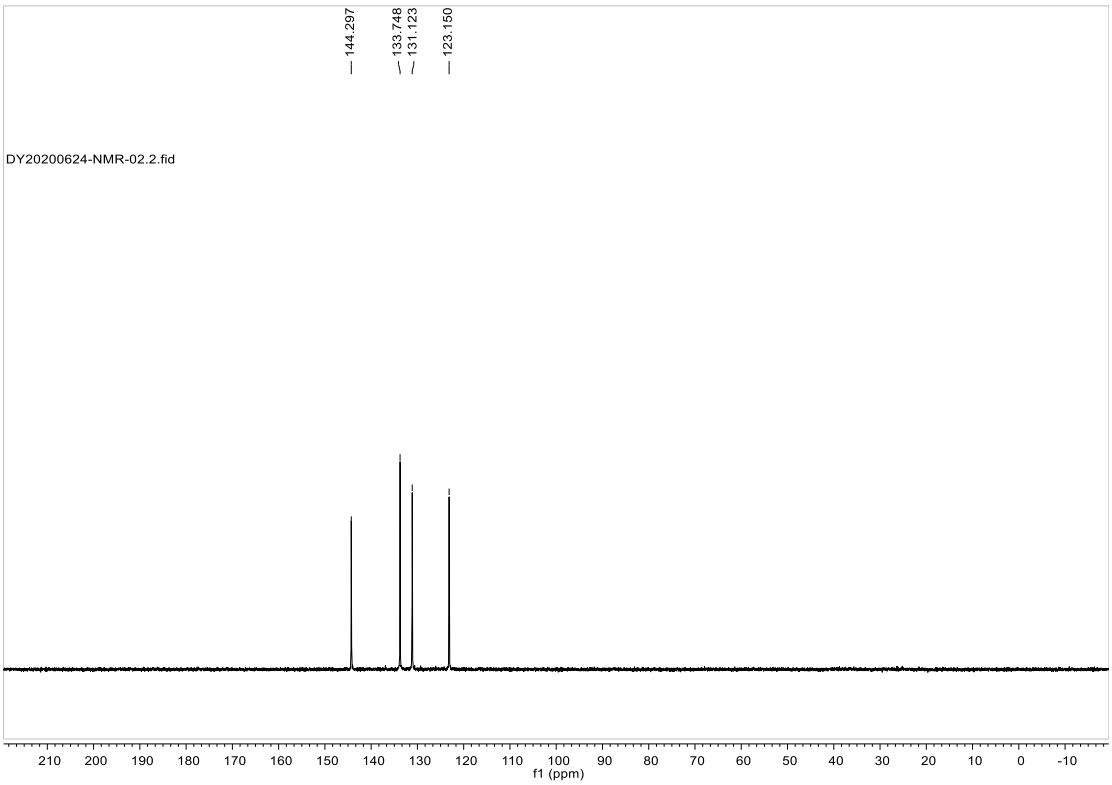
DY20200627-NMR-01-zhongjianti.1.fid
1.00
1.08
1.03
2.12
f1 (ppm)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131.123
123.150
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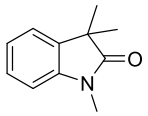
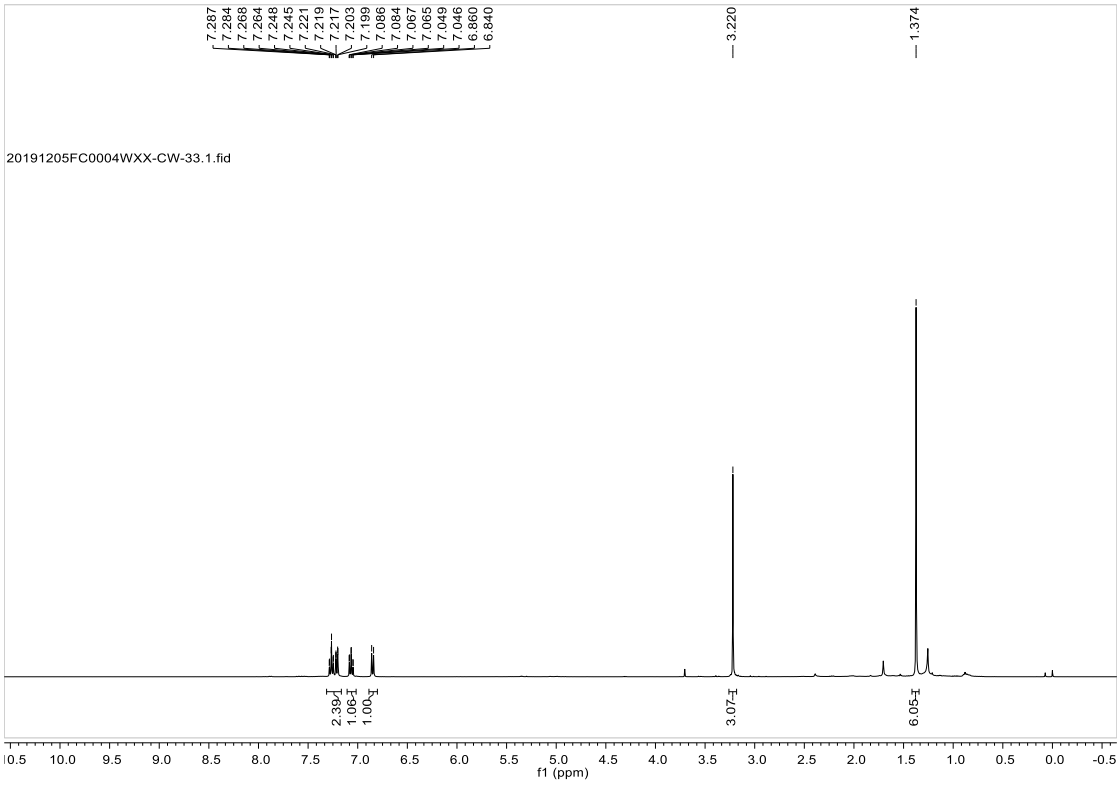
f1 (ppm)


O
N

6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7.248
7.245
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7.219
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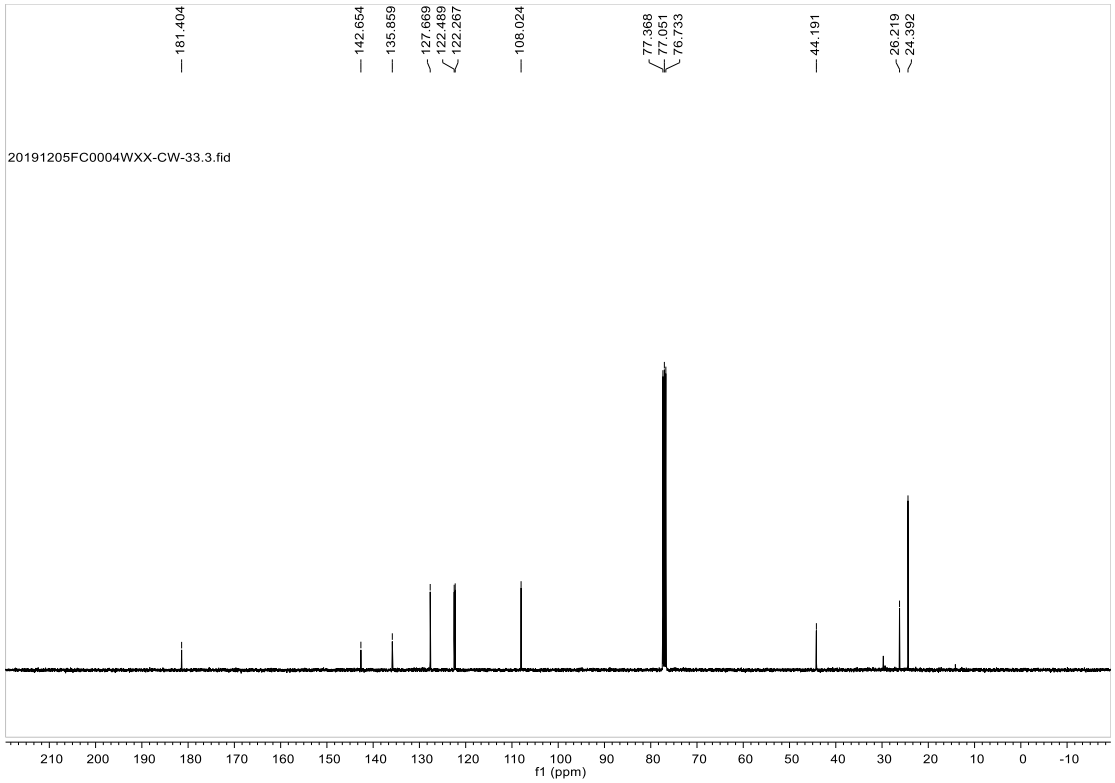
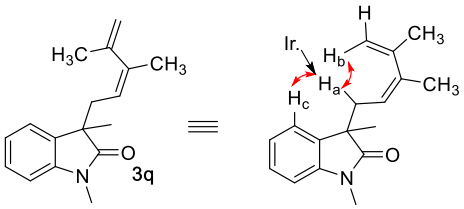
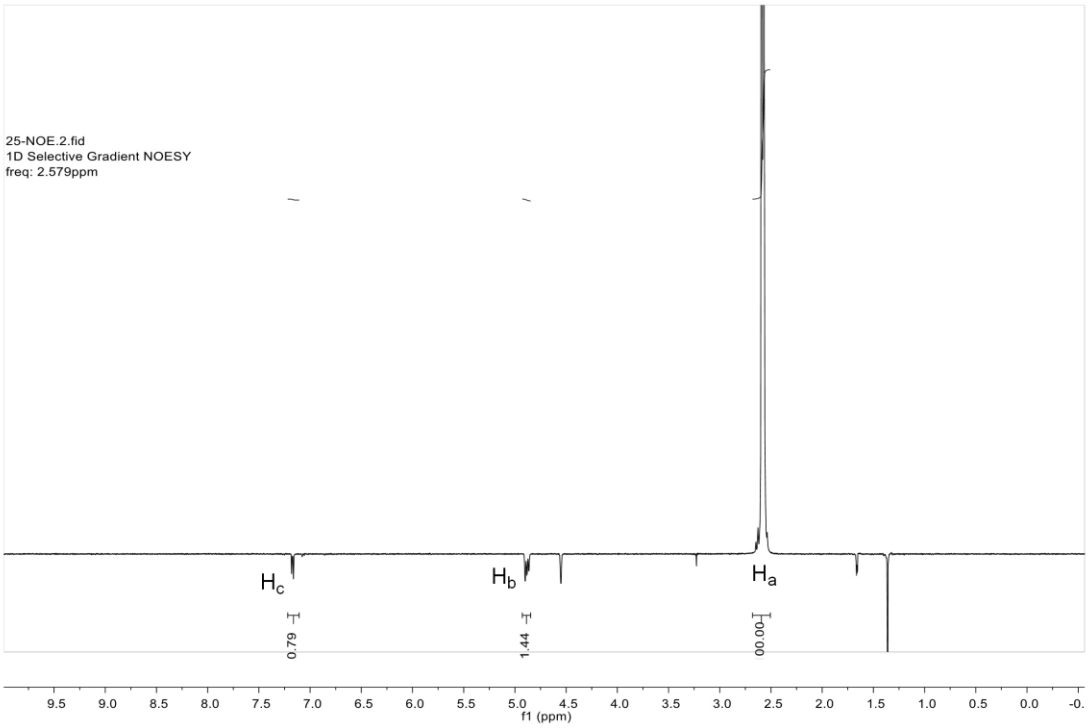
7.203
7.199
7.086
7.084
7.067
7.065
7.049
7.046
6.860
6.840
3.220
1.374
20191205FC0004WXX-CW-33.1.fid
2.39
1.06
1.00
3.07
6.05
f1 (ppm)


181.404
142.654
135.859
127.669
122.489
122.267
108.024
77.368
77.051
76.733
44.191
26.219
24.392
20191205FC0004WXX-CW-33.3.fid
f1 (ppm)



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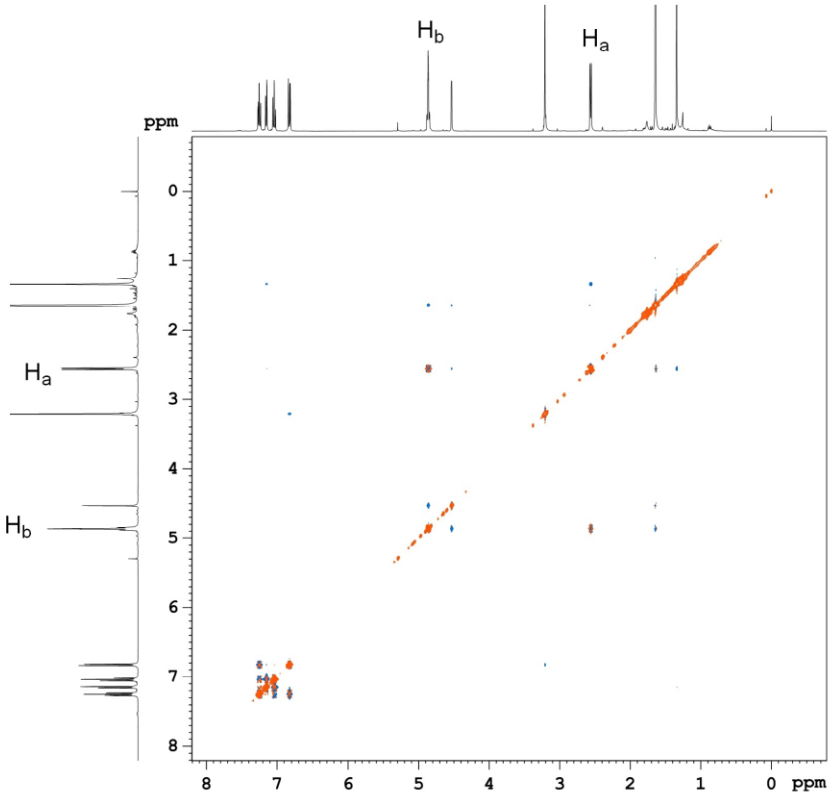
Current Data Parameters
NAME            CW-25-NOE
EXPNO                   2
PROCNO                  1

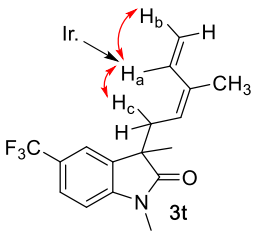
F2 - Acquisition Parameters
Date_          20200417
Time                4.24 h
INSTRUM             spect
PROBHD    Z116098_0503 (
PULPROG      noesygpphpp
TD                  2048
SOLVENT             CDCl3
NS                     8
DS                    32
SWH              3597.122 Hz
FIDRES           1.756407 Hz
AQ               0.2846720 sec
RG                  25.39
DW                139.000 usec
DE                  6.50 usec
TE                294.3 K
D0             0.00012627 sec
D1             1.97132802 sec
D8             0.30000001 sec
D11            0.03000000 sec
D12            0.00002000 sec
D16            0.00020000 sec
IN0            0.00027800 sec
Thav                    1
SFO1         400.1314924 MHz
NUC1                 1H
P1                 10.00 usec
P2                 20.00 usec
P17              2500.00 usec
PLW1          15.02099991 W
PLW10          1.66900003 W
GPNAM[1]       SMSQ10.100
GPZ1               40.00 %
P16              1000.00 usec

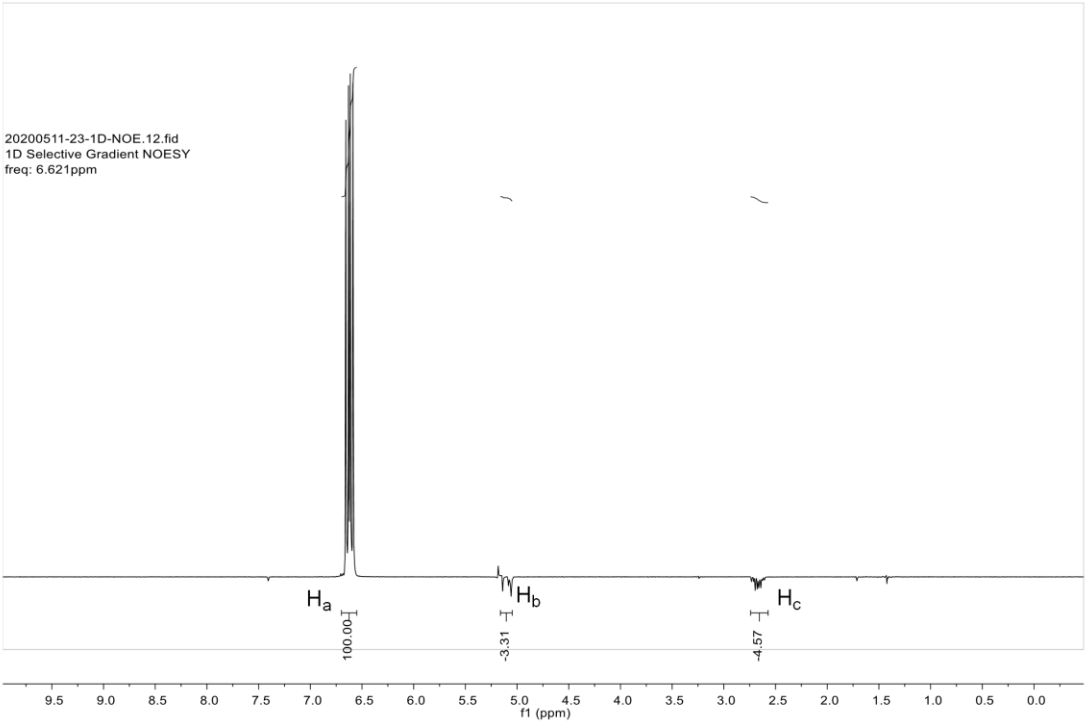
F1 - Acquisition parameters
TD                    256
SFO1           400.1315 MHz
FIDRES         28.102518 Hz
SW                8.990 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI                   1024
SF           400.1300076 MHz
WDW                 QSINE
SSB                     2
LB                      0 Hz
GB                      0
PC                   1.00

F1 - Processing parameters
SI                   1024
MC2          States-TPPI
SF           400.1300076 MHz
WDW                 QSINE
SSB                     2
LB                      0 Hz
GB                      0
```







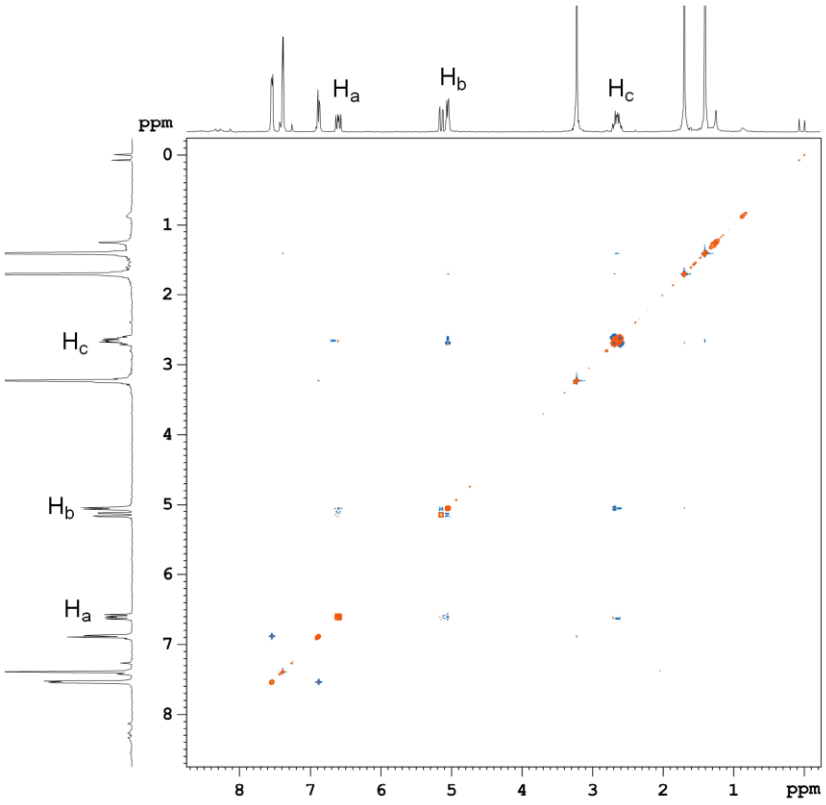

```
Current Data Parameters
NAME            cw-23-NOE
EXPNO                   2
PROCNO                  1

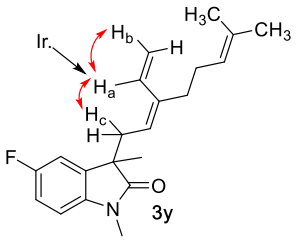
F2 - Acquisition Parameters
Date_          20200423
Time               9.25 h
INSTRUM            spect
PROBHD   Z116098_0503 (
PULPROG     noesygpphpp
TD                 2048
SOLVENT            CDCl3
NS                   16
DS                   32
SWH             3597.122 Hz
FIDRES          1.756407 Hz
AQ              0.2846720 sec
RG                  25.39
DW                139.000 usec
DE                  6.50 usec
TE                294.2 K
D0             0.00012627 sec
D1              1.97132802 sec
D8              0.30000001 sec
D11             0.03000000 sec
D12             0.00002000 sec
D16             0.00020000 sec
IN0             0.00027800 sec
TDav                    1
SFO1         400.1317090 MHz
NUC1                   1H
P1                 10.00 usec
P2                 20.00 usec
P17              2500.00 usec
PLW1          15.02099991 W
PLW10          1.66900003 W
GPNAM[1]      SMSQ10.100
GPZ1               40.00 %
P16              1000.00 usec

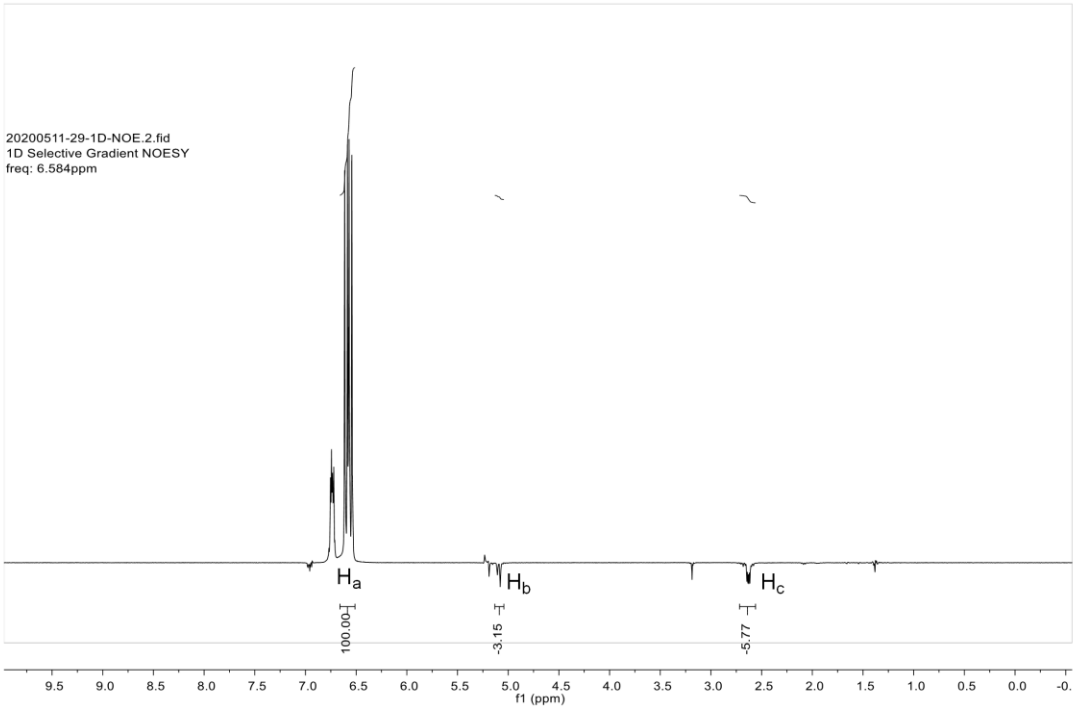
F1 - Acquisition parameters
TD                    256
SFO1            400.1317 MHz
FIDRES         28.102518 Hz
SW                 8.990 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI                   1024
SF            400.1300062 MHz
WDW                  QSINE
SSB                     2
LB                      0 Hz
GB                      0
PC                   1.00

F1 - Processing parameters
SI                   1024
MC2          States-TPPI
SF            400.1300062 MHz
WDW                  QSINE
SSB                     2
LB                      0 Hz
GB                      0
```








```
Current Data Parameters
NAME          cw-29-NOE
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20200422
Time              11.47 h
INSTRUM             spect
PROBHD   Z116098_0503 (
PULPROG      noesygpphpp
TD                  2048
SOLVENT            CDCl3
NS                    16
DS                    32
SWH             3267.974 Hz
FIDRES          1.595690 Hz
AQ              0.3133440 sec
RG                 10.03
DW                153.000 usec
DE                  6.50 usec
TE                294.5 K
D0             0.00014027 sec
D1             1.94265604 sec
D8             0.30000001 sec
D11            0.03000000 sec
D12            0.00002000 sec
D16            0.00020000 sec
IN0            0.00030600 sec
TDav                  1
SFO1          400.1315211 MHz
NUC1                  1H
P1                10.00 usec
P2                20.00 usec
P17             2500.00 usec
PLW1           15.02099991 W
PLW10           1.66900003 W
GPNAM[1]      SMSQ10.100
GPZ1             40.00 %
P16            1000.00 usec

F1 - Acquisition parameters
TD                   256
SFO1          400.1315 MHz
FIDRES         25.531046 Hz
SW                 8.167 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI                  1024
SF            400.1300352 MHz
WDW                 QSINE
SSB                     2
LB                      0 Hz
GB                      0
PC                   1.00

F1 - Processing parameters
SI                  1024
MC2          States-TPPI
SF            400.1300352 MHz
WDW                 QSINE
SSB                     2
LB                      0 Hz
GB                      0
```