Palladium-Catalyzed Csp^2–H Carbonylation of Aromatic Oximes: Selective Access to Benzo[d][1,2]oxazin-1-ones and 3-Methyleneisoindolin-1-ones*

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Supporting Information

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General Information

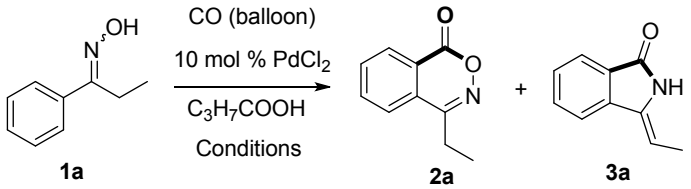
Solvents and all reagents were used as received. ^{1}H NMR and ^{13}C NMR spectra were recorded in $CDCl_3$ or DMSO-d6 at 400 MHz NMR spectrometer. The chemical shifts (d) were referenced to TMS. GC-MS was obtained using electron ionization (EI). IR spectra were obtained as potassium bromide pellets or as liquid films between two potassium bromide pellets with a Brucker Vector 22 spectrometer. Melting points were measured with a micro melting point apparatus. TLC was performed using commercially prepared 100-400 mesh silica gel plates (GF_{254}), and visualization was effected at 254 nm. High-resolution mass spectra (ESI) were obtained with a LCMS-IT-TOF mass spectrometer.

General Procedure for Oximes

To a solution of aromatic ketones or aromatic aldehydes (3.0 mmol) in the mixture of C_2H_5OH/H_2O (v/v = 1:1) was added hydroxylamine hydrochloride (3.6 mmol), NaOAc (4.5 mmol) in one portion, and the reaction mixture was stirred at 100 ^{o}C (when the substrates are aromatic ketones) or at room temperature (when the substrates are aromatic aldehydes) for 6-8 h. Upon completion of the reaction as indicated by TLC, the reaction mixture was diluted with water, extracted with ethyl acetate, and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure to give oximes.

The Optimization of Reaction Conditions

Table S1. Optimization of Reaction Conditions to 2a and 3a[a]

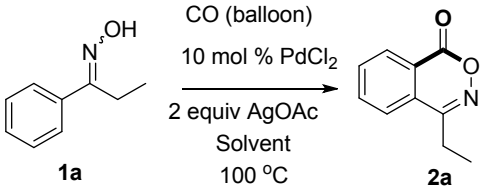


Entry	Oxidant	Base	Temp (^{o}C)	Yield (%)	
				2a	3a
1	-	-	100	28	<5
2	Ag_2O	-	100	37	nd
3	$AgNO_3$	-	100	33	nd
4	AgCl	-	100	56	nd
5	AgOAc	-	100	66	nd
6	$CuCl_2$	-	100	nd	nd
7	$Cu(OAc)_2$	-	100	nd	nd
8	DDQ	-	100	nd	nd
9	BQ	-	100	nd	nd

10	$PhI(OAc)_2$	-	100	nd	nd
11	-	-	120	nd	40
12	-	Li_2CO_3	120	nd	38
13	-	Na_2CO_3	120	nd	43
14	-	K_2CO_3	120	nd	80 (73)
15	-	Cs_2CO_3	120	nd	64
16	-	NaOAc	120	nd	37
17	-	KOAc	120	nd	32

[a] Reaction conditions: unless otherwise noted, all reactions were performed with **1a** (0.5 mmol), $PdCl_2$ (10 mol %), oxidant (1.0 mmol) or base (0.25 mmol) in C_3H_7COOH (2.0 mL) for 48 h. Determined by GC using dodecane as the internal standard. Data in parentheses is the yield of isolated product.

Table S2. Optimization of Different Solvents for 2a[a]

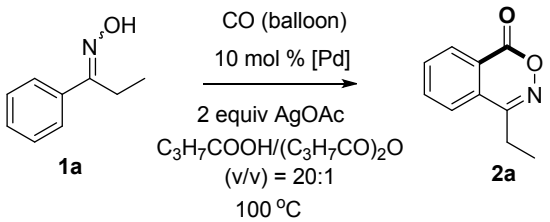


1a **2a**

Entry	Solvent	Yield (%)
1	DMF	10
2	toluene	<5
3	1,4-dioxane	nd
4[b]	DCE	nd
5[b]	DCM	nd
6[b]	CH_3CN	<5
7	C_3H_7COOH	66
8	$C_3H_7COOH/(C_3H_7CO)_2O$ (v/v) = 20:1	79 (74)

[a] Reaction conditions: unless otherwise noted, all reactions were performed with **1a** (0.5 mmol), $PdCl_2$ (10 mol %), AgOAc (1.0 mmol) in solvent (2.0 mL) at 100 °C for 48 h. Determined by GC using dodecane as the internal standard. Data in parentheses is the yield of isolated product. [b] The reaction was carried out in 25 mL Schlenk-type sealed tube.

Table S3. Optimization of Different Pd-catalysts for 2a[a]



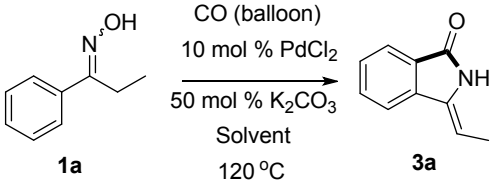
1a **2a**

Entry	[Pd]	Yield (%)
1	$Pd(OAc)_2$	22
2	$Pd(PPh_3)_2Cl_2$	<10
3	$Pd(TFA)_2$	19

4	$Pd(PPh_3)_4$	nd
5	Pd_2dba_3	<5
6	$PdCl_2$	79 (74)

[a] Reaction conditions: unless otherwise noted, all reactions were performed with **1a** (0.5 mmol), Pd-catalyst (10 mol %), AgOAc (1.0 mmol) in a mixture of $C_3H_7COOH/(C_3H_7CO)_2O$ (v/v) = 20:1 (2.0 mL) 100 °C for 48 h. Determined by GC using dodecane as the internal standard. Data in parentheses is the yield of isolated product.

Table S4. Optimization of Different Solvents for 3a[a]

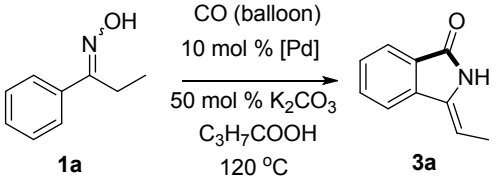


1a **3a**

Entry	Solvent	Yield (%)
1[b]	HOAc	nd
2[b]	CH_3CN	nd
3[b]	toluene	61
4	DMF	nd
5	DMSO	nd
6	C_3H_7COOH	80 (73)

[a] Reaction conditions: unless otherwise noted, all reactions were performed with **1a** (0.5 mmol), $PdCl_2$ (10 mol %), K_2CO_3 (0.25 mmol) in solvent (2.0 mL) at 120 °C for 48 h. Determined by GC using dodecane as the internal standard. Data in parentheses is the yield of isolated product. [b] The reaction was carried out in 25 mL Schlenk-type sealed tube.

Table S5. Optimization of Different Pd-catalysts for 3a[a]



1a **3a**

Entry	[Pd]	Yield (%)
1	$Pd(OAc)_2$	32
2	$Pd(PPh_3)_2Cl_2$	nd
3	$Pd(TFA)_2$	28
4	$Pd(PPh_3)_4$	nd
5	Pd_2dba_3	15
6	$PdCl_2$	80 (73)

[a] Reaction conditions: unless otherwise noted, all reactions were performed with **1a** (0.5 mmol), Pd-catalyst (10 mol %), K_2CO_3 (0.25 mmol) in C_3H_7COOH (2.0 mL) at 120 °C for 48 h. Determined by GC using dodecane as the internal standard. Data in parentheses is the yield of isolated product.

General Procedure for Benzo[d][1,2]oxazin-1-ones 2

Under air atmosphere, oxime **1** (0.5 mmol), $PdCl_2$ (10 mol %), AgOAc (1.0 mmol), and 2.0 mL of *n*-C_3H_7COOH/(*n*-$C_3H_7CO)_2O$ (v/v = 20:1) were added to a tube equipped with magnetic stirrer bar. Then the tube was charged with CO (1 atm), and was stirred at 100 °C (oil bath temperature). After the reaction was finished (monitored by TLC), the crude product was cooled to room temperature and quenched with aqueous $NaHCO_3$, and the crude product was extracted with ethyl acetate. The organic extracts were concentrated in vacuum, and the resulting residue was purified by column chromatography on silica gel with light petroleum ether/ethyl acetate as eluent to afford the desired product **2**.

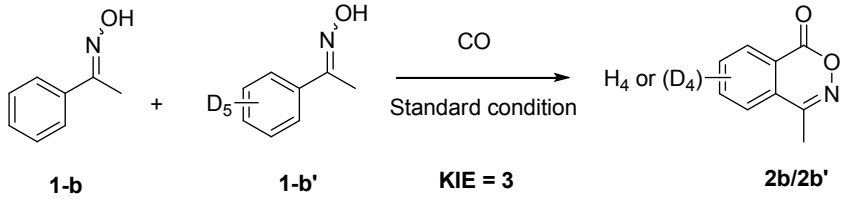
General Procedure for the Preparation of 3

Oxime **1** (0.5 mmol), $PdCl_2$ (10 mol %), K_2CO_3 (0.25 mmol), and 2.0 mL of *n*-C_3H_7COOH were added to a tube equipped with magnetic stirrer bar. Then the tube was charged with CO (1 atm) , and was stirred at 120 °C (oil bath temperature). After the reaction was finished (monitored by TLC), the crude product was cooled to room temperature and quenched with aqueous NaOH, and the crude product was extracted with ethyl acetate. The organic extracts were concentrated in vacuum, and the resulting residue was purified by column chromatography on silica gel with light petroleum ether/ethyl acetate as eluent to afford the desired product **3**.

General Procedure for the Preparation of 4

Compound **3a** (0.3 mmol), 4-iodopyridine (1.5 mmol), CuI (0.06 mmol), K_2CO_3 (0.9 mmol), DMF (2 mL), were added to a 25-mL Schlenk-type sealed tube equipped with a magnetic stirrer and septum. The tube was evacuated and purged with nitrogen gas three times. And the reaction mixture was stirred at 150 °C for 48 h. Upon completion of the reaction as indicated by TLC, the reaction mixture was diluted with water, extracted with ethyl acetate, and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the resulting residue was purified by column chromatography on silica gel with light petroleum ether/ethyl acetate as eluent to afford the desired product **4**.

The KIE Experiments for the Synthesis of 2b/2b'



Under air atmosphere, oxime **1-b** (0.25 mmol), oxime **1-b'** (0.25 mmol), $PdCl_2$ (10 mol %), AgOAc (1.0 mmol), and 2.0 mL of *n*-C_3H_7COOH/(*n*-$BuO)_2O$ (v/v = 20:1) were added to a tube

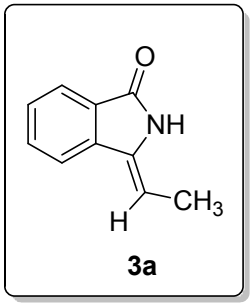
equipped with magnetic stirrer bar. Then the tube was charged with CO (1 atm), and was stirred at 100 °C (oil bath temperature) for 12 h. The crude product was cooled to room temperature and quenched with aqueous $NaHCO_3$, then extracted with ethyl acetate. The organic extracts were concentrated in vacuum, and the resulting residue was purified by column chromatography on silica gel with light petroleum ether/ethyl acetate as eluent to afford the desired products **2b/2b'**, detected by ^{1}HNMR.

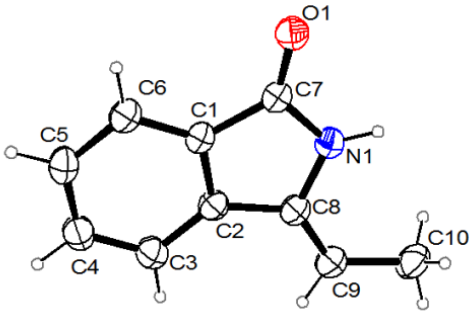
Compounds **2b/2b'**, ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.38 (d, *J* = 7.7 Hz, 1H), 7.94 (t, *J* = 7.6 Hz, 1H), 7.86 (t, *J* = 7.5 Hz, 1H), **7.69 (d, *J* = 6.7 Hz, 1.5H)**, 2.58 (s, 6H).

Compound **2b,** ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.35 (d, *J* = 7.7 Hz, 2H), 7.94 (t, *J* = 7.6 Hz, 2H), 7.85 (t, *J* = 7.6 Hz, 2H), **7.69 (d, *J* = 7.8 Hz, 2H)**, 2.57 (s, 6H).

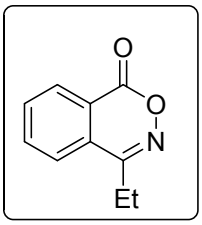
KIE = K_H/K_D = 1.5/(2-1.5) = 3.

The X-ray Crystal Structure of (*Z*)-3-Ethylideneisoindolin-1-one 3a

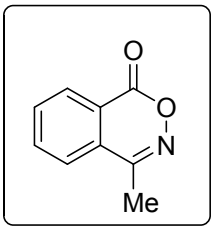




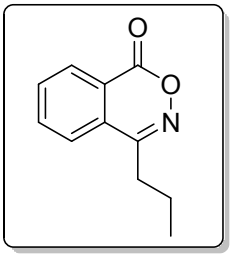
Analytical Data for All Compounds



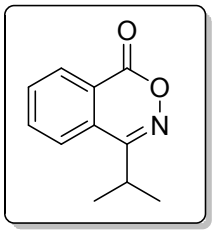
4-Ethyl-1*H*-benzo[d][1,2]oxazin-1-one (2a). Pale yellow solid, m.p. 115-116 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.40 (d, *J* = 7.8 Hz, 1H), 7.93 (t, *J* = 7.7 Hz, 1H), 7.85 (t, *J* = 7.6 Hz, 1H), 7.73 (d, *J* = 7.9 Hz, 1H), 3.00 (q, *J* = 7.4 Hz, 2H), 1.40 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.8, 156.5, 135.3, 133.4, 128.9, 126.9, 124.7, 122.7, 24.2, 11.7. IR (KBr): 3054, 2922, 2851, 1726, 1643, 1559, 1455, 1283, 1249, 1089, 1024, 911, 758, 688 cm^{-1}. ESI-HRMS calcd for $C_{10}H_{10}NO_2$ $[M+H]^+$ 176.0706, found 176.0703.



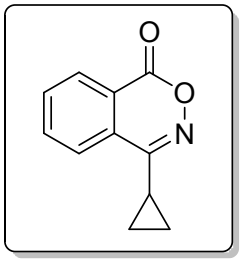
4-Methyl-1*H*-benzo[d][1,2]oxazin-1-one (2b). Pale yellow solid, m.p. 158-159 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.35 (d, *J* = 7.7 Hz, 1H), 7.94 (t, *J* = 7.6 Hz, 1H), 7.85 (t, *J* = 7.6 Hz, 1H), 7.69 (d, *J* = 7.8 Hz, 1H), 2.57 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.7, 153.2, 135.5, 133.6, 128.6, 127.4, 125.2, 122.3, 16.6. IR (KBr): 3064, 2925, 2855, 1695, 1609, 1556, 1433, 1379, 1252, 1090, 1012, 899, 759, 683 cm^{-1}. ESI-HRMS calcd for $C_9H_8NO_2$ $[M+H]^+$ 162.0550, found 162.0550.



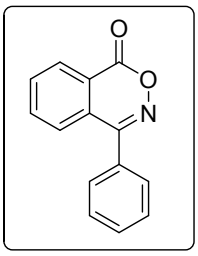
4-Propyl-1*H*-benzo[d][1,2]oxazin-1-one (2c). Pale yellow solid, m.p. 64-65 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.39 (d, *J* = 7.8 Hz, 1H), 7.93 (t, *J* = 7.6 Hz, 1H), 7.84 (t, *J* = 7.6 Hz, 1H), 7.72 (d, *J* = 7.9 Hz, 1H), 2.92 (t, *J* = 8.0 Hz, 2H), 1.88-1.78 (m, 2H), 1.07 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.8, 155.7, 135.3, 133.4, 128.8, 126.9, 124.9, 122.7, 32.4, 20.8, 13.8. IR (KBr): 3054, 2969, 2877, 1740, 1602, 1566, 1461, 1251, 1102, 1023, 916, 739, 685 cm^{-1}. ESI-HRMS calcd for $C_{11}H_{12}NO_2$ $[M+H]^+$ 190.0863, found 190.0866.



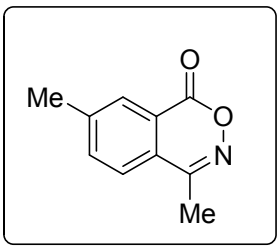
4-Isopropyl-1*H*-benzo[d][1,2]oxazin-1-one(2d).[1] Pale yellow solid, m.p. 79-80°C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.38 (d, *J* = 7.8 Hz, 1H), 7.93 (t, *J* = 7.6 Hz, 1H), 7.82 (t, *J* = 7.6 Hz, 1H), 7.78 (d, *J* = 8.0 Hz, 1H), 3.61-3.30 (m, 1H), 1.41 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.7, 159.2, 135.3, 133.2, 128.9, 126.5, 124.5, 122.7, 29.8, 20.7. IR (KBr): 3074, 2978, 2875, 1727, 1598, 1461, 1384, 1281, 1105, 1022, 912, 738, 691 cm^{-1}. MS (EI) m/z: 189, 174, 159, 130, 115, 77.



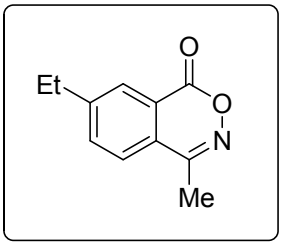
4-Cyclopropyl-1*H*-benzo[d][1,2]oxazin-1-one (2e). Pale yellow solid, m.p. 94-95 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.33 (d, *J* = 7.7 Hz, 1H), 8.03 (d, *J* = 7.8 Hz, 1H), 7.95 (t, *J* = 7.5 Hz, 1H), 7.84 (t, *J* = 7.4 Hz, 1H), 2.20-2.10 (m, 1H), 1.08 (d, *J* = 9.6 Hz, 4H).^{13}C NMR (100 MHz, $CDCl_3$) δ 163.8, 156.0, 135.4, 133.4, 128.5, 128.0, 125.0, 122.0, 10.6, 6.1. IR (KBr): 3012, 2924, 2853, 1734, 1599, 1557, 1454, 1390, 1280, 1058, 1022, 732, 688 cm^{-1}. ESI-HRMS calcd for $C_{11}H_{10}NO_2$ $[M+H]^+$ 188.0706, found 188.0710.



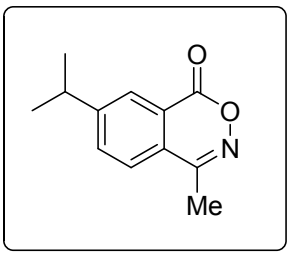
4-Phenyl-1*H*-benzo[d][1,2]oxazin-1-one (2f). Pale yellow solid, m.p. 149-150 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50-8.42 (m, 1H), 7.90-7.83 (m, 2H), 7.62-7.54 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.5, 157.2, 135.3, 133.8, 131.1, 130.4, 129.3, 128.9, 128.8, 127.5, 127.3, 122.9. IR (KBr): 3059, 2924, 2853, 1734, 1603, 1450, 1386, 1346, 1284,1245, 1109, 1054, 907, 692 cm^{-1}. ESI-HRMS calcd for $C_{14}H_{10}NO_2$ $[M+H]^+$ 224.0706, found 224.0711.



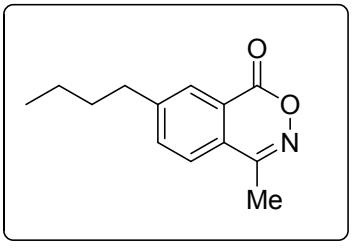
4,7-Dimethyl-1*H*-benzo[d][1,2]oxazin-1-one (2g). Pale yellow solid, m.p. 120-121 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (s, 1H), 7.70 (d, *J* = 8.0 Hz, 1H), 7.55 (d, *J* = 8.0 Hz, 1H), 2.51 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.0, 153.1, 144.8, 136.4, 128.3, 125.2, 124.9, 122.0, 21.7, 16.5. IR (KBr): 3079, 2925, 2855, 1728, 1610, 1441, 1382, 1336, 1280, 1100, 1048, 836, 743 cm^{-1}. ESI-HRMS calcd for $C_{10}H_{10}NO_2$ $[M+H]^+$ 176.0706, found 176.0713.



7-Ethyl-4-methyl-1*H*-benzo[d][1,2]oxazin-1-one (2h). Pale yellow solid, m.p. 117-118 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (s, 1H), 7.73 (d, *J* = 7.8 Hz, 1H), 7.58 (d, *J* = 7.8 Hz, 1H), 2.80 (q, *J* = 7.2 Hz, 2H), 2.51 (s, 3H), 1.27 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.1, 153.1, 150.9, 135.4, 127.1, 125.3, 125.1, 122.1, 28.9, 16.5, 14.8. IR (KBr): 3053, 2968, 2896, 1726, 1607, 1561, 1501, 1436, 1340, 1247, 1105, 1021, 903, 857, 755 cm^{-1}.ESI-HRMS calcd for $C_{11}H_{12}NO_2$ $[M+H]^+$ 190.0863, found 190.0861.

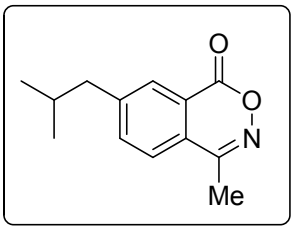


7-Isopropyl-4-methyl-1*H*-benzo[d][1,2]oxazin-1-one (2i). Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (s, 1H), 7.76 (d, *J* = 8.0 Hz, 1H), 7.60 (d, *J* = 8.0 Hz, 1H), 3.09-3.00 (m, 1H), 2.50 (s, 3H), 1.27 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.1, 155.5, 153.0, 134.2, 125.7, 125.4, 125.2, 122.1, 34.3, 23.3, 16.4. IR (KBr): 3073, 2966, 2876, 1725, 1609, 1562, 1501, 1433, 1340, 1247, 1102, 1011, 903, 858, 755 cm^{-1}. ESI-HRMS calcd for $C_{12}H_{14}NO_2$ $[M+H]^+$ 204.1019, found 204.1015.

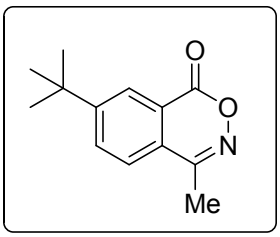


7-Butyl-4-methyl-1*H*-benzo[d][1,2]oxazin-1-one (2j). Pale yellow solid, m.p. 69-70 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (s, 1H), 7.71 (d, *J* = 8.1 Hz, 1H), 7.58 (d, *J* = 8.1 Hz, 1H), 2.76 (t, *J* = 7.7 Hz, 2H), 2.51 (s, 3H), 1.67-1.58 (m, 2H), 1.32 (dt, *J* = 14.5, 7.4 Hz, 2H), 0.89 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.1, 153.1, 149.7, 135.8, 127.7, 125.2, 125.1, 122.0, 35.6, 32.9, 22.1, 16.5, 13.7. IR (KBr): 3056, 2958, 2865, 1734,

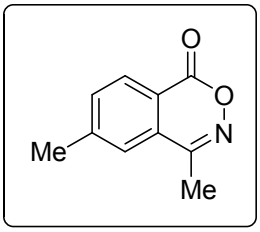
1611, 1562, 1430, 1379, 1335, 1290, 1102, 1052, 912, 839, 783, 752, 698 cm^{-1}; ESI-HRMS calcd for $C_{13}H_{16}NO_2$ $[M+H]^+$ 218.1176, found 218.1173.



7-Isobutyl-4-methyl-1*H*-benzo[d][1,2]oxazin-1-one (2k). Pale yellow oil. ^1H NMR (400 MHz, $CDCl_3$) δ 8.05 (s, 1H), 7.67 (d, *J* = 8.0 Hz, 1H), 7.58 (d, *J* = 8.1 Hz, 1H), 2.61 (d, *J* = 7.1 Hz, 2H), 2.50 (s, 3H), 1.90 (m, 1H), 0.87 (s, 3H), 0.85 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.0, 153.0, 148.5, 136.4, 128.3, 125.1, 125.0, 121.9, 45.1, 29.9, 22.0, 16.4. IR (KBr): 3056, 2959, 2871, 1737, 1610, 1562, 1504, 1461, 1431, 1172, 1101, 1051, 913, 849, 785, 697, 613 cm^{-1}; ESI-HRMS calcd for $C_{13}H_{16}NO_2$ $[M+H]^+$ 218.1176, found 218.1175.

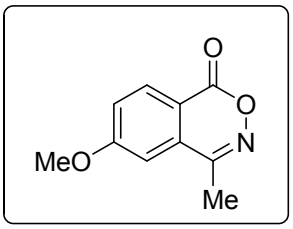


7-(*tert*-Butyl)-4-methyl-1*H*-benzo[d][1,2]oxazin-1-one (2l). Pale yellow solid, m.p. 157-158 °C ^1H NMR (400 MHz, $CDCl_3$) δ 8.35 (s, 1H), 7.95 (d, *J* = 8.3 Hz, 1H), 7.63 (d, *J* = 8.3 Hz, 1H), 2.54 (s, 3H), 1.38 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.4, 158.0, 153.1, 133.0, 125.1, 125.0, 124.9, 122.0, 35.6, 30.91, 16.6. IR (KBr): 3059, 2922, 2855, 1722, 1610, 1445, 1389, 1346, 1282, 1105, 1038, 838, 749 cm^{-1}. ESI-HRMS calcd for $C_{13}H_{16}NO_2$ $[M+H]^+$ 218.1176, found 218.1173.

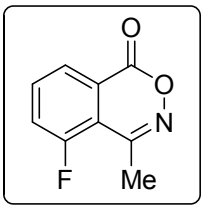


4,6-Dimethyl-1*H*-benzo[d][1,2]oxazin-1-one (2n). Pale yellow solid, m.p. 131-132 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 8.20 (d, *J* = 8.0 Hz, 1H), 7.62 (d, *J* = 8.0 Hz, 1H), 7.44 (s, 1H), 2.56 (s, 3H), 2.53 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.8, 153.1, 146.9, 134.7, 128.5, 127.5, 125.2, 119.7, 22.1, 16.6. IR (KBr): 3056, 2924, 2854,

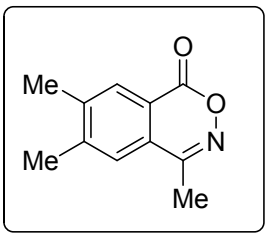
1734, 1598, 1501, 1437, 1380, 1338, 1289, 1092, 1010, 898, 771, 687 cm^{-1}. ESI-HRMS calcd for $C_{10}H_{10}NO_2$ $[M+H]^+$ 176.0706, found 176.0704.



6-Methoxy-4-methyl-1*H*-benzo[d][1,2]oxazin-1-one (2o). White solid, m.p. 140-141 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 8.28 (d, *J* = 8.8 Hz, 1H), 7.32 (d, *J* = 8.8 Hz, 1H), 7.01 (s, 1H), 3.97 (s, 3H), 2.53 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.1, 163.6, 153.0, 131.1, 129.6, 120.3, 115.2, 108.7, 56.0, 16.8. IR (KBr): 3029, 2923, 2851, 1717, 1647, 1612, 1486, 1384, 1265, 1097, 1026, 824, 758 cm^{-1}. ESI-HRMS calcd for $C_{10}H_{10}NO_3$ $[M+H]^+$ 192.0655, found 192.0654.

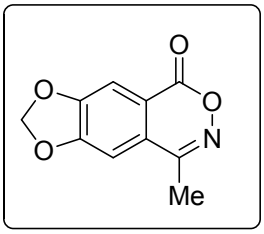


5-Fluoro-4-methyl-1*H*-benzo[d][1,2]oxazin-1-one (2p). Pale yellow solid, m.p. 124-125 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 8.22 (d, *J* = 7.8 Hz, 1H), 7.85 (td, *J* = 8.0, 5.0 Hz, 1H), 7.69-7.57 (m, 1H), 2.68 (d, J_{F-H}= 6.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.3, 159.4, 156.8, 150.6, 135.2, 124.9, 123.7, 123.0, 116.2, 20.3. IR (KBr): 3054, 2923, 2851, 1742, 1605, 1464, 1384, 1287, 1100, 1012, 891, 761 cm^{-1}. ESI-HRMS calcd for $C_9H_6FNNaO_2$ $[M+Na]^+$ 202.0275, found 202.0272.

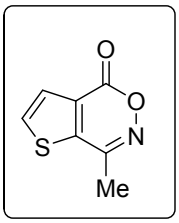


4,6,7-Trimethyl-1*H*-benzo[d][1,2]oxazin-1-one (2q). Pale yellow solid, m.p. 181-182 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 8.04 (s, 1H), 7.38 (s, 1H), 2.51 (s, 3H), 2.45 (s, 3H), 2.42 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.1, 153.0, 145.8, 143.8, 128.8, 125.8, 125.4, 120.0, 20.5, 20.1, 16.6. IR (KBr): 3066, 2925, 2856, 1730, 1605, 1566, 1500, 1436, 1383, 1342, 1258, 1103, 1020, 978, 902, 824, 769 cm^{-1}. ESI-HRMS calcd for $C_{11}H_{12}NO_2$ $[M+H]^+$

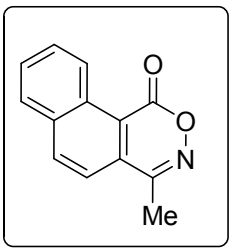
190.0863, found 190.0861.



8-Methyl-5*H*-[1,3]dioxolo[4',5':4,5]benzo[1,2-d][1,2]oxazin-5-one (2r). Pale yellow solid, m.p. 188-189 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (s, 1H), 7.00 (s, 1H), 6.21 (s, 2H), 2.50 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.7, 153.9, 152.3, 152.1, 124.5, 118.5, 107.0, 103.9, 103.1, 17.0. IR (KBr): 3058, 2921, 2851, 1728, 1640, 1607, 1493, 1423, 1319, 1271, 1079, 1032, 987, 910, 741 cm^{-1}. ESI-HRMS calcd for $C_{10}H_8NO_4$ $[M+H]^+$ 206.0448, found 206.0447.

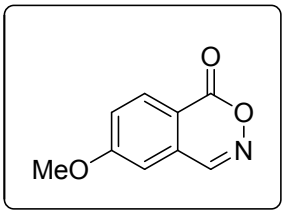


7-Methyl-4*H*-thieno[2,3-d][1,2]oxazin-4-one (2s).[1] Pale yellow solid, m.p. 88-89 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 5.1 Hz, 1H), 7.73 (d, *J* = 5.0 Hz, 1H), 2.58 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.1, 149.6, 139.9, 132.4, 129.4, 126.4, 17.7. IR (KBr): 3057, 2924, 2854, 1728, 1433, 1380, 1251, 1090, 1011, 899, 759, 682 cm^{-1}; MS (EI) m/z: 167, 151, 125, 109, 84.

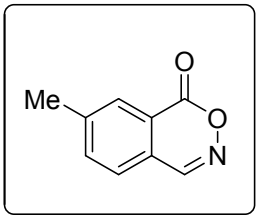


4-Methyl-1*H*-naphtho[2,1-d][1,2]oxazin-1-one (2t). Pale yellow solid, m.p. 162-163 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.75 (d, *J* = 8.6 Hz, 1H), 8.34 (d, *J* = 8.7 Hz, 1H), 7.99 (d, *J* = 8.0 Hz, 1H), 7.84 (t, *J* = 7.7 Hz, 1H), 7.77 (t, *J* = 7.3 Hz, 1H), 7.66 (d, *J* = 8.6 Hz, 1H), 2.67 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.8, 153.1, 137.4, 135.2, 130.3, 130.0, 129.3, 128.7, 128.2, 128.1, 120.5, 117.7, 17.5. IR (KBr): 3055, 2923, 2852, 1722, 1635, 1509,

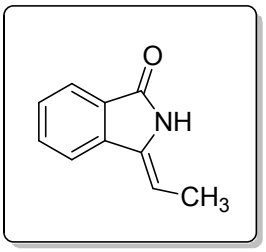
1468, 1429, 1386, 1265, 1107, 1025, 988, 938, 826, 755 cm^{-1}; ESI-HRMS calcd for $C_{13}H_{10}NO_2$ $[M+H]^+$ 212.0706, found 212.0705.



6-Methoxy-1*H*-benzo[d][1,2]oxazin-1-one (2u). Pale yellow solid, m.p. 153-154 °C. ^1H NMR (400 MHz, DMSO-d_6) δ 11.15 (s, 1H), 7.73 (d, *J* = 7.9 Hz, 1H), 7.29 (d, *J* = 10.0 Hz, 2H), 3.91 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 168.8, 168.7, 164.3, 135.2, 124.7, 124.5, 119.8, 107.8, 56.2. IR (KBr): 3056, 2923, 2851, 1717, 1647, 1612, 1486, 1384, 1265, 1097, 1026, 828, 758, 693 cm^{-1}. ESI-HRMS calcd for $C_9H_7NNaO_3$ $[M+Na]^+$ 200.0318, found 200.0320.

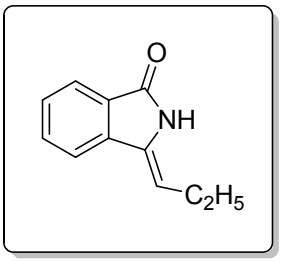


7-Methyl-1*H*-benzo[d][1,2]oxazin-1-one (2v). Yellow solid, m.p. 187-188 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 7.6 Hz, 2H), 7.66 (s, 1H), 7.54 (d, *J* = 7.5 Hz, 1H), 2.52 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.1, 168.0, 145.7, 134.9, 133.0, 130.0, 124.1, 123.5, 22.0. IR (KBr): 3065, 2925, 2855, 1718, 1612, 1541, 1380, 1333, 1280, 1105, 1028, 838, 741 cm^{-1}. ESI-HRMS calcd for $C_9H_7NNaO_2$ $[M+Na]^+$ 184.0369, found 184.0362.

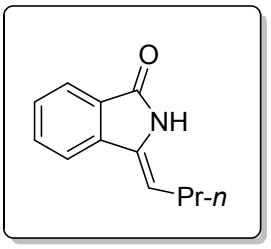


(*Z*)-3-Ethylideneisoindolin-1-one (3a). Yellow solid, m.p. 107-108 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 9.76 (s, 1H), 7.85 (d, *J* = 7.4 Hz, 1H), 7.63 (d, *J* = 7.6 Hz, 1H), 7.55 (t, *J* = 7.3 Hz, 1H), 7.44 (t, *J* = 7.2 Hz, 1H), 5.69 (q, *J* = 7.1 Hz, 1H), 2.06 (d, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.5, 137.6, 134.4, 131.8, 129.6, 128.3, 123.2, 119.3, 103.3, 12.6. IR (KBr): 3458, 3054, 2922, 2852, 1690, 1645, 1466, 1383, 1266, 1093, 802, 753 cm^{-1}.

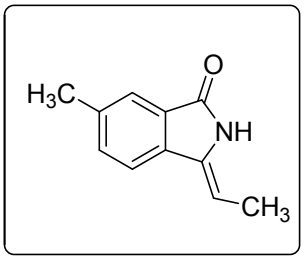
ESI-HRMS calcd for $C_{10}H_{10}NO$ $[M+H]^+$ 160.0757, found 160.0756.



(*Z*)-3-Propylideneisoindolin-1-one (3b). Yellow solid, m.p. 93-94 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.19 (s, 1H), 7.85 (d, *J* = 7.6 Hz, 1H), 7.65 (d, *J* = 7.7 Hz, 1H), 7.56 (t, *J* = 7.5 Hz, 1H), 7.45 (t, *J* = 7.4 Hz, 1H), 5.63 (t, *J* = 7.7 Hz, 1H), 2.43 (m, 2H), 1.19 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.2, 137.7, 132.9, 131.9, 129.5, 128.4, 123.3, 119.4, 110.3, 20.7, 14.1. IR (KBr): 3451, 3055, 2927, 2857, 1691, 1616, 1470, 1381, 1268, 1093, 753 cm^{-1}. ESI-HRMS calcd for $C_{11}H_{12}NO$ $[M+H]^+$ 174.0913, found 174.0906.

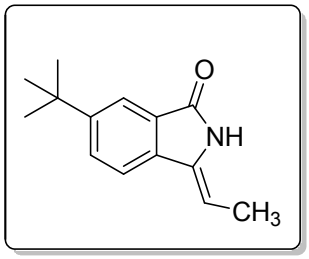


(*Z*)-3-Butylideneisoindolin-1-one (3c). Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.96 (s, 1H), 7.85 (d, *J* = 7.6 Hz, 1H), 7.65 (d, *J* = 7.7 Hz, 1H), 7.56 (t, *J* = 7.5 Hz, 1H), 7.45 (t, *J* = 7.4 Hz, 1H), 5.64 (t, *J* = 7.8 Hz, 1H), 2.37 (q, *J* = 7.4 Hz, 2H), 1.58 (m, 2H), 1.02 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.0, 137.7, 133.6, 131.9, 129.6, 128.4, 123.3, 119.5, 108.3, 29.2, 22.8, 13.8. IR (KBr): 3470, 3045, 2926, 2854, 1690, 1640, 1467, 1386, 1266, 1099, 745 cm^{-1}. ESI-HRMS calcd for $C_{12}H_{13}NNaO$ $[M+Na]^+$ 210.0889, found 210.0887.

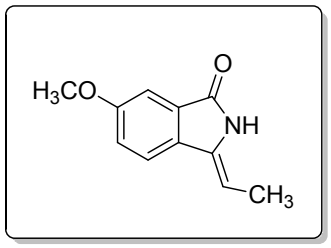


(*Z*)-3-Ethylidene-6-methylisoindolin-1-one (3d). Pale yellow solid, m.p. 223-224 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.63 (s, 1H), 7.65 (s, 1H), 7.51 (d, *J* = 7.9 Hz, 1H), 7.36 (d, *J* = 7.9 Hz, 1H), 5.62 (q, *J* = 7.3 Hz, 1H), 2.45 (s, 3H), 2.03 (d, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.5, 138.5, 135.2, 134.4, 133.0, 129.8, 123.3,

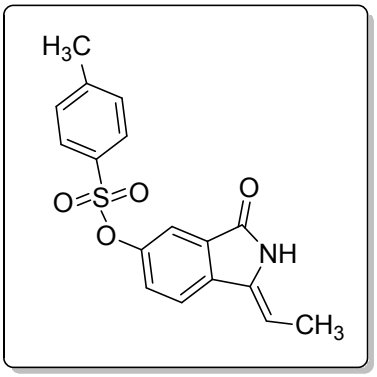
119.1, 102.4, 21.5, 12.6. IR (KBr): 3466, 3068, 2922, 2853, 1696, 1635, 1456, 1385, 1270, 1022, 824, 753 cm^{-1}. ESI-HRMS calcd for $C_{11}H_{11}NNaO$ $[M+Na]^+$ 196.0733, found 196.0735.



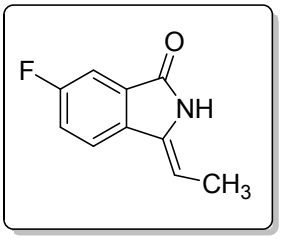
(*Z*)-6-(*tert*-Butyl)-3-ethylideneisoindolin-1-one (3e). Pale yellow solid, m.p. 188-189 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 8.99 (s, 1H), 7.86 (s, 1H), 7.62 (d, *J* = 8.1 Hz, 1H), 7.55 (d, *J* = 8.1 Hz, 1H), 5.63 (q, *J* = 7.3 Hz, 1H), 2.01 (d, *J* = 7.3 Hz, 3H), 1.37 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.5, 152.2, 135.2, 134.2, 129.6, 129.4, 119.8, 119.1, 102.1, 35.1, 31.4, 12.6. IR (KBr): 3466, 3038, 2926, 2857, 1690, 1635, 1457, 1385, 1270, 1022, 825, 756 cm^{-1}. ESI-HRMS calcd for $C_{14}H_{17}NNaO$ $[M+Na]^+$ 238.1202, found 238.1200.



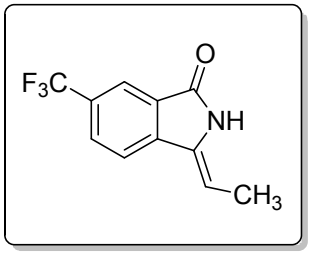
(*Z*)-3-Ethylidene-6-methoxyisoindolin-1-one (3f). White solid, m.p. 196-197 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 8.99 (s, 1H), 7.52 (d, *J* = 8.4 Hz, 1H), 7.30 (s, 1H), 7.12 (d, *J* = 8.4 Hz, 1H), 5.55 (q, *J* = 7.3 Hz, 1H), 3.88 (s, 3H), 1.99 (d, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.9, 160.4, 134.0, 130.9, 130.6, 120.8, 120.6, 105.5, 101.5, 55.7, 12.5. IR (KBr): 3450, 3058, 2922, 2845, 1685, 1545, 1512, 1411, 1242, 1178, 1031, 827, 753 cm^{-1}. ESI-HRMS calcd for $C_{11}H_{11}NNaO_2$ $[M+Na]^+$ 212.0682, found 212.0684.



(*Z*)-1-Ethylidene-3-oxoisoindolin-5-yl 4-methylbenzenesulfonate (3g). Yellow solid, m.p. 193-194 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.32 (s, 1H), 7.71 (d, *J* = 8.1 Hz, 2H), 7.56 (d, *J* = 8.4 Hz, 1H), 7.39 (s, 1H), 7.31 (d, *J* = 8.0 Hz, 3H), 5.67 (q, *J* = 7.3 Hz, 1H), 2.44 (s, 3H), 2.00 (d, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.7, 149.7, 145.7, 136.0, 133.4, 132.0, 130.7, 129.9, 128.5, 126.6, 120.8, 117.1, 104.5, 21.7, 12.7. IR (KBr): 3455, 3064, 2923, 2853, 1690, 1647, 1550, 1473, 1374, 1267, 1093, 810, 749 cm^{-1}. ESI-HRMS calcd for $C_{17}H_{15}NNaO_4S$ $[M+Na]^+$ 352.0619, found 352.0622.

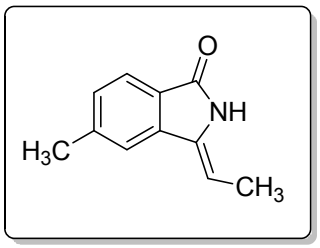


(*Z*)-3-Ethylidene-6-fluoroisoindolin-1-one (3h). Pale yellow solid, m.p. 205-206 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.69 (s, 1H), 7.90 (dd, *J* = 8.1, 4.6 Hz, 1H), 7.46 (t, *J* = 9.1 Hz, 2H), 5.76 (q, *J* = 7.4 Hz, 1H), 1.91 (d, *J* = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 167.1, 164.0, 161.6, 134.0, 131.7, 122.7, 120.0, 109.2, 103.6, 12.9. IR (KBr): 3466, 3054, 2921, 2851, 1690, 1640, 1483, 1387, 1267, 1093, 746 cm^{-1}. ESI-HRMS calcd for $C_{10}H_8FNNaO$ $[M+Na]^+$ 200.0482, found 200.0480.

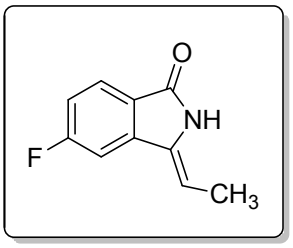


(*Z*)-3-Ethylidene-6-(trifluoromethyl)isoindolin-1-one (3i). Pale yellow solid, m.p. 238-239 °C. ^{1}H NMR (400 MHz, DMSO-d6) δ 10.86 (s, 1H), 8.08 (d, *J* = 8.2 Hz, 1H), 7.96 (d, *J* = 7.6 Hz, 2H), 5.98 (q, *J* = 7.4 Hz, 1H), 1.96

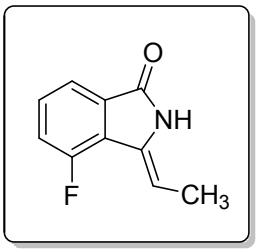
(d, *J* = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, DMSO-d6) δ 166.8, 141.0, 133.9, 130.2, 129.5, 129.0, 125.8, 121.7, 119.9, 106.7, 13.1. IR (KBr): 3442, 3054, 2922, 2851, 1701, 1646, 1385, 1322, 1271, 1118, 1025, 998, 827, 762 cm^{-1}. ESI-HRMS calcd for $C_{11}H_8F_3NNaO$ $[M+Na]^+$ 250.0450, found 250.0450.



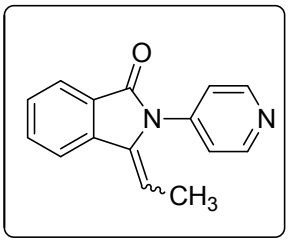
(*Z*)-3-Ethylidene-5-methylisoindolin-1-one (3j). Pale yellow solid, m.p.176-177 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.53 (s, 1H), 7.72 (d, *J* = 7.8 Hz, 1H), 7.42 (s, 1H), 7.25 (d, *J* = 7.1 Hz, 1H), 5.64 (q, *J* = 7.4 Hz, 1H), 2.46 (s, 3H), 2.03 (d, *J* = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.5, 142.5, 138.0, 134.5, 129.5, 127.2, 123.0, 119.7, 102.7, 22.0, 12.6. IR (KBr): 3455, 3058, 2925, 2855, 1687, 1618, 1475, 1360, 1269, 1172, 1045, 893, 749 cm^{-1}. ESI-HRMS calcd for $C_{11}H_{11}NNaO$ $[M+Na]^+$ 196.0733, found 196.0735.



(*Z*)-3-Ethylidene-5-fluoroisoindolin-1-one (3k). Pale yellow solid, m.p.166-177 °C. ^{1}H NMR (400 MHz, DMSO-d_6) 10.58 (s, 1H), 7.72 (dd, *J* = 11.7, 5.4 Hz, 2H), 7.29 (t, *J* = 8.9 Hz, 1H), 5.82 (q, *J* = 7.3 Hz, 1H), 1.92 (d, *J* = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 167.3, 164.1, 140.4, 134.1, 126.2, 125.4, 116.7, 107.4, 104.6, 12.9. IR (KBr): 3456, 3053, 2925, 2853, 1690, 1617, 1475, 1383, 1268, 1187, 1093, 864, 748 cm^{-1}. ESI-HRMS calcd for $C_{10}H_8FNNaO$ $[M+Na]^+$ 200.0482, found 200.0480.



(*Z*)-3-Ethylidene-4-fluoroisoindolin-1-one (3l). Pale yellow solid, m.p.191-192 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.76 (s, 1H), 7.66 (d, *J* = 7.4 Hz, 1H), 7.42-7.37 (m, 1H), 7.23 (d, *J* = 9.0 Hz, 1H), 5.96 (q, *J* = 7.4 Hz, 1H), 2.06 (d, *J* = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.4, 158.9, 156.3, 132.6, 131.5, 129.5, 124.0, 119.4, 109.6, 13.2. IR (KBr): 3452, 3052, 2925, 2854, 1695, 1653, 1484, 1380, 1258, 1096, 1059, 755 cm^{-1}. ESI-HRMS calcd for $C_{10}H_8FNNaO$ $[M+Na]^+$ 200.0482, found 200.0480.

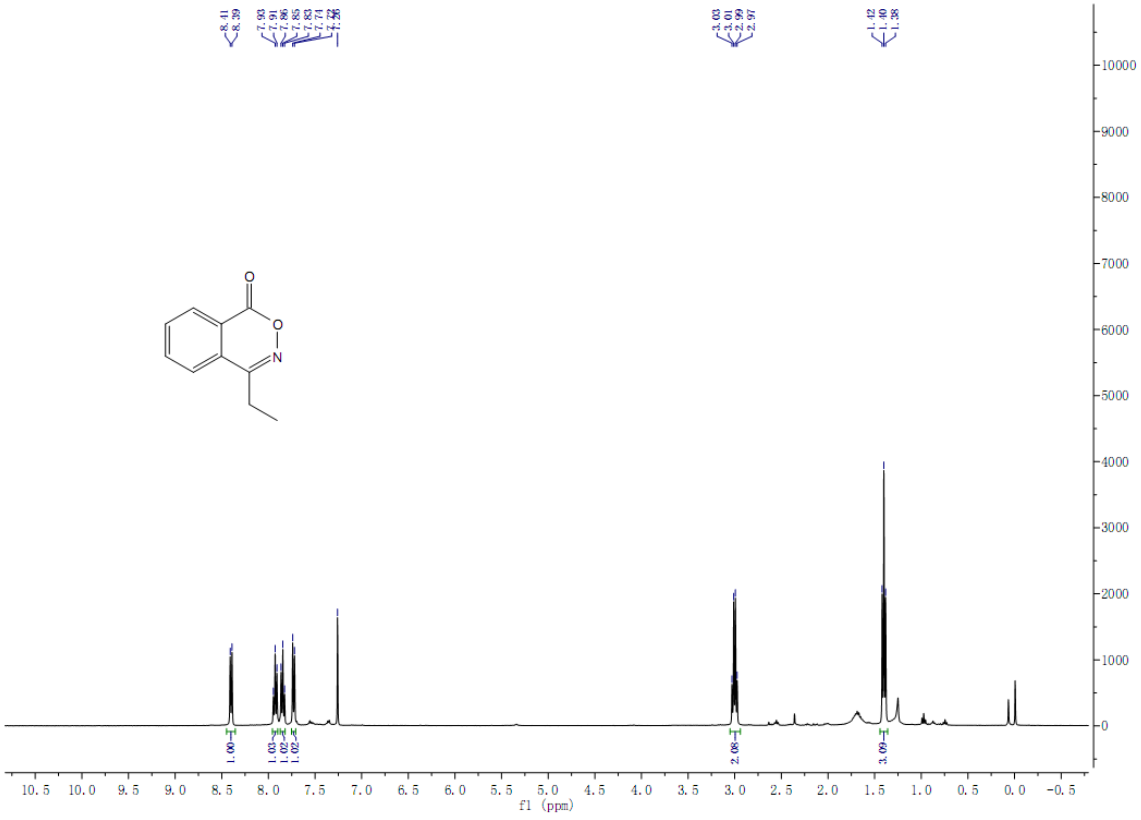


3-Ethylidene-2-(pyridin-4-yl)isoindolin-1-one(4). Pale yellow solid, m.p. 127-135 °C. *Z:E* = 3:2. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.72 (dd, *J* = 11.0, 4.6 Hz, 3.4H), 8.00-7.79 (m, 3H), 7.70-7.61 (m, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.48 (t, *J* = 7.5 Hz, 1H), 7.34 (s, 3H), 5.83 (q, *J* = 7.7 Hz, 0.6H), **5.59 (q, *J* = 7.6 Hz, 1H), 2.19 (d, *J* = 7.6 Hz, 3H)**, 1.50 (d, *J* = 7.7 Hz, 1.8H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.1, 165.3, 151.0, 150.5, 144.5, 142.7, 138.2, 136.0, 135.5, 134.6, 132.7, 132.6, 129.3, 129.0, 128.7, 127.1, 124.0, 123.8, 123.6, 122.9, 122.4, 119.0, 108.1, 105.2, 13.9, 12.9. IR (KBr): 3466, 3054, 2921, 2851, 1690, 1644, 1453, 1283, 1187, 1093, 746 cm^{-1}. MS (EI) m/z: 236, 207, 152, 115, 78.

References

[1] T. G. Chun, K. S. Kim, S. Lee, T. Jeong, H. Lee, Y. H.Kim, W. S. Lee, *Syn. Commun.* **2004**, *34*, 1301.

NMR Spectra for All Compounds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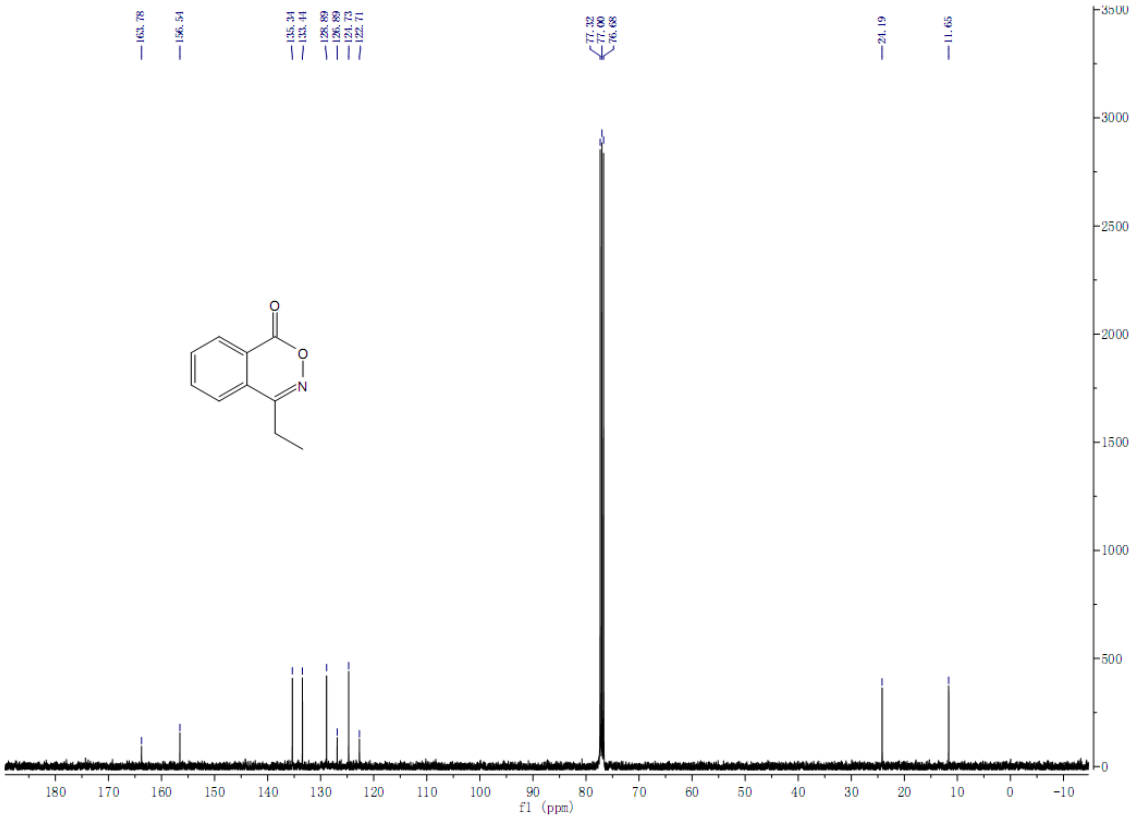
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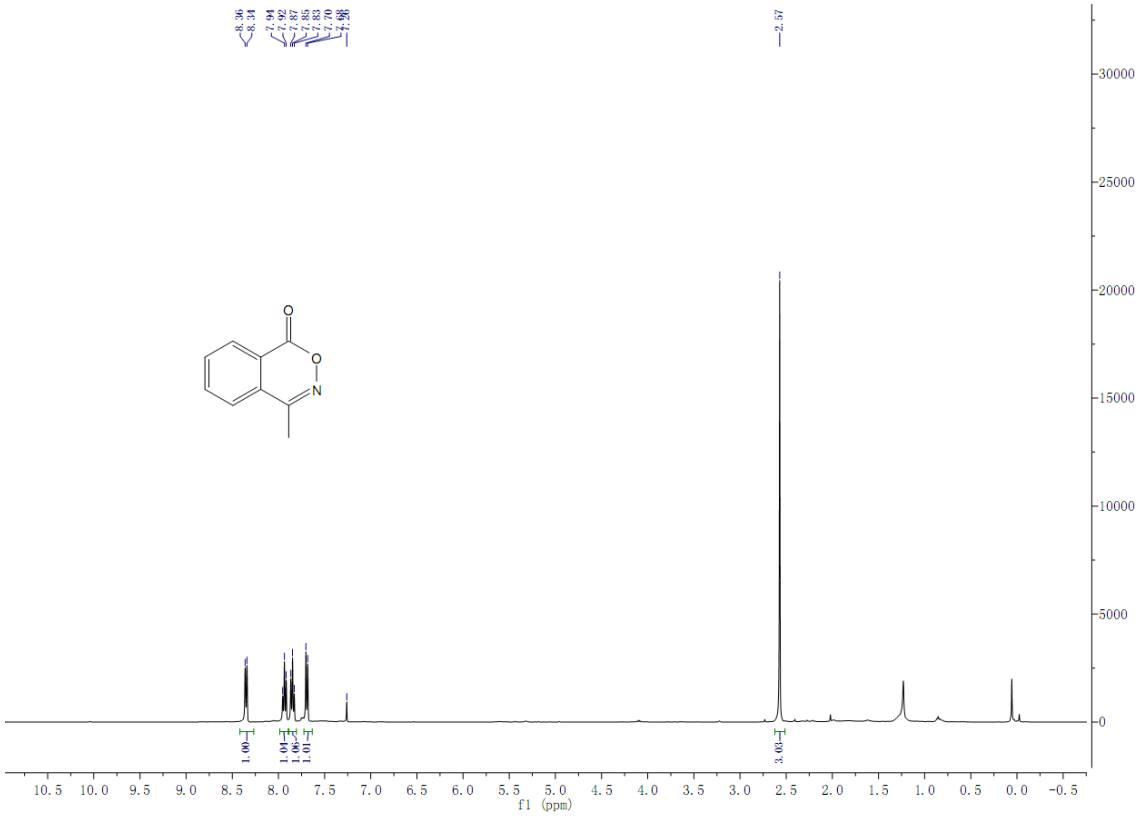
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1.06
1.01
3.03
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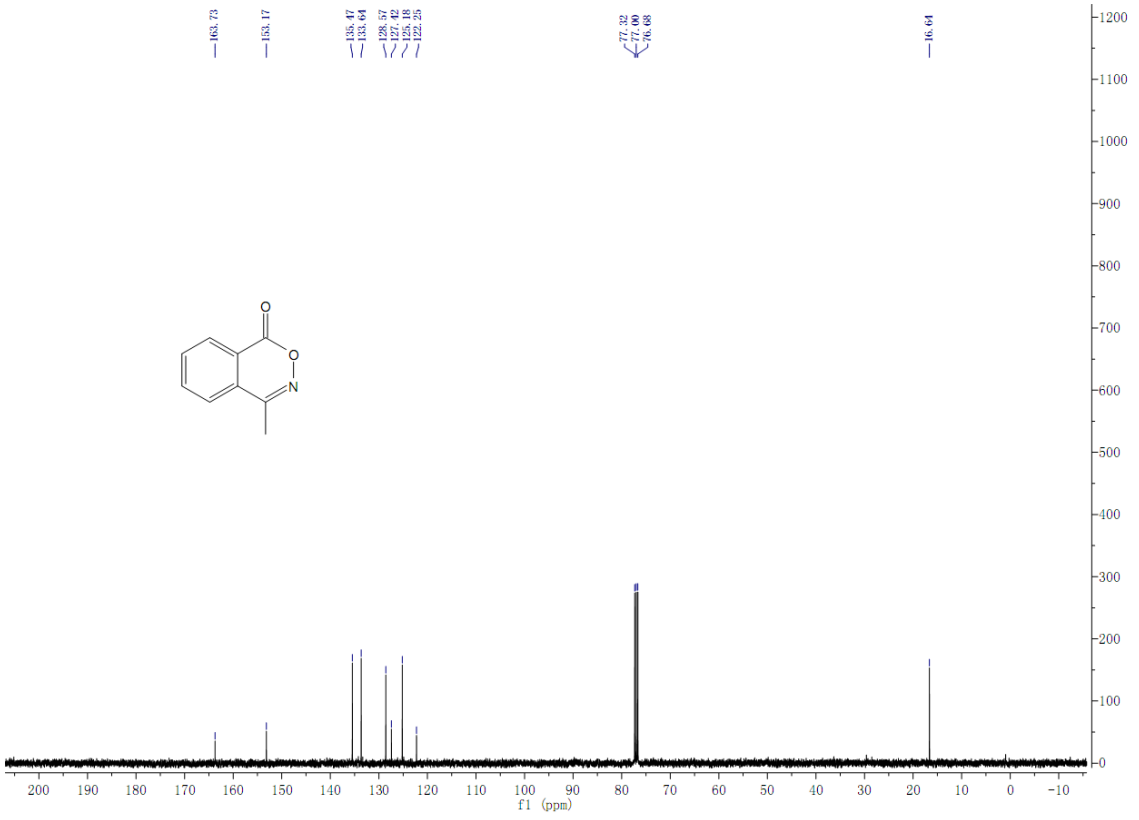
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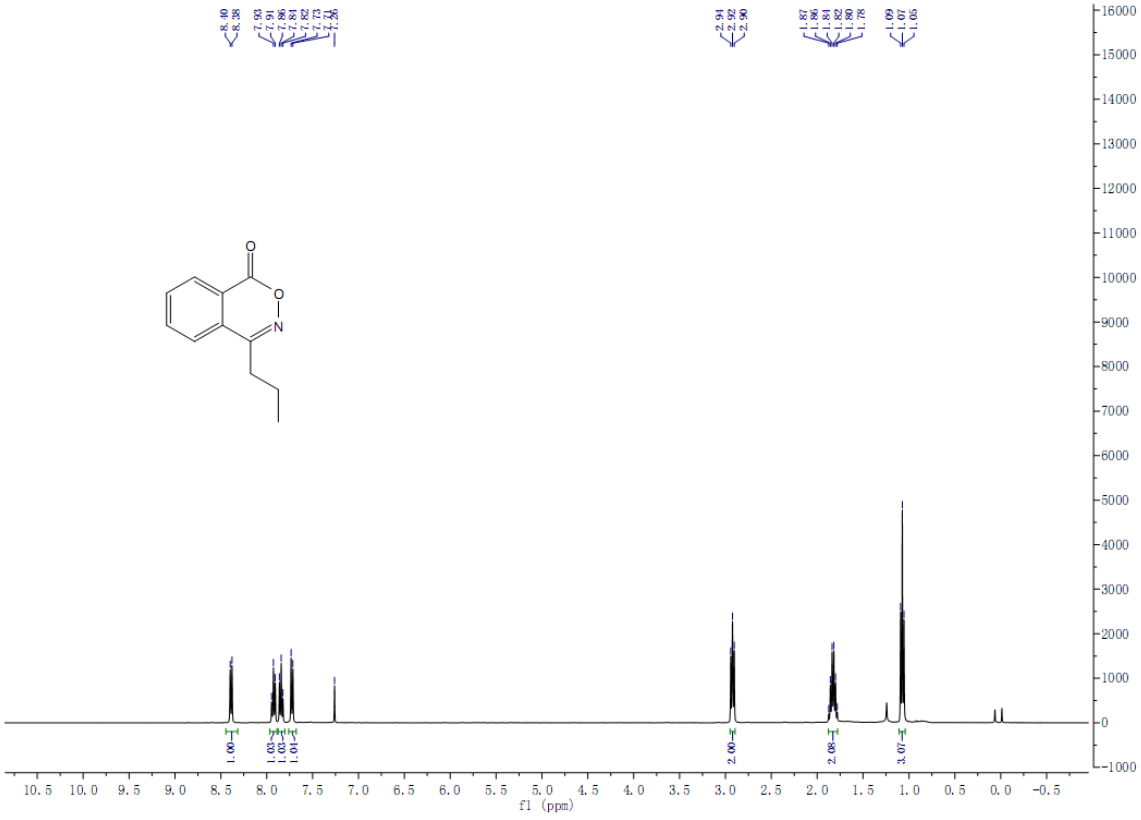
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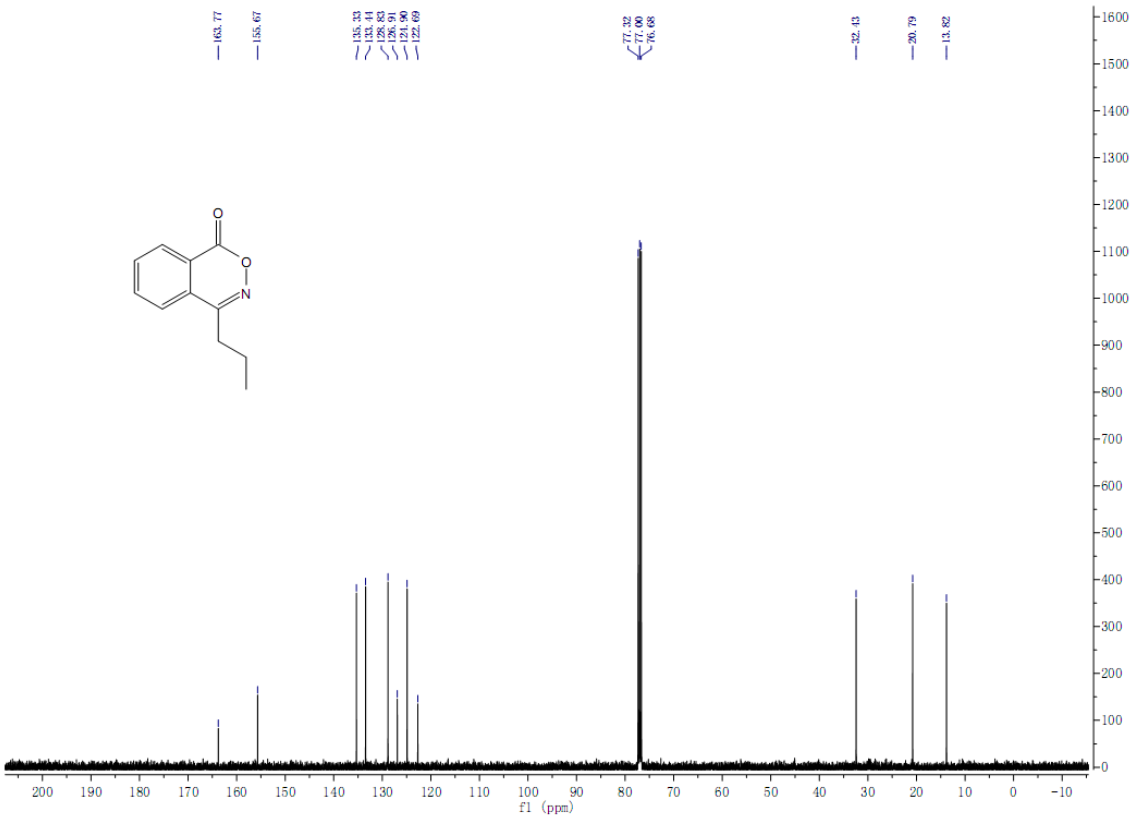
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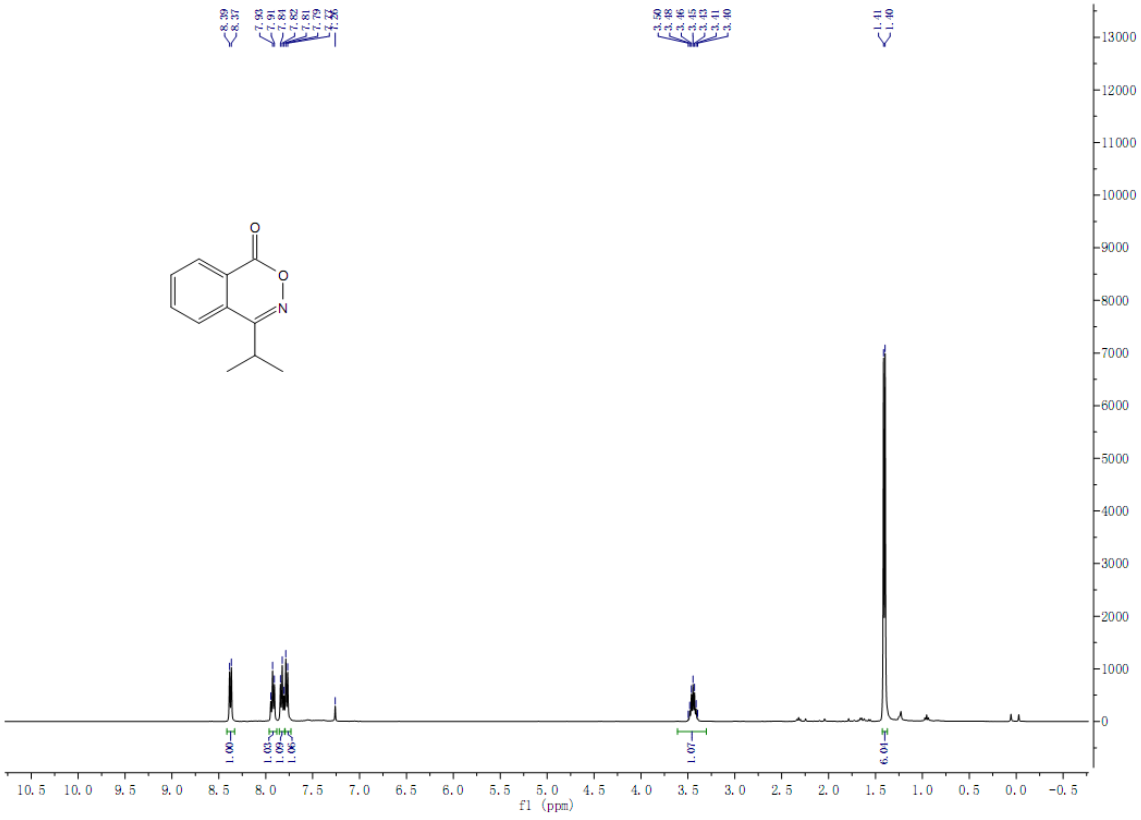
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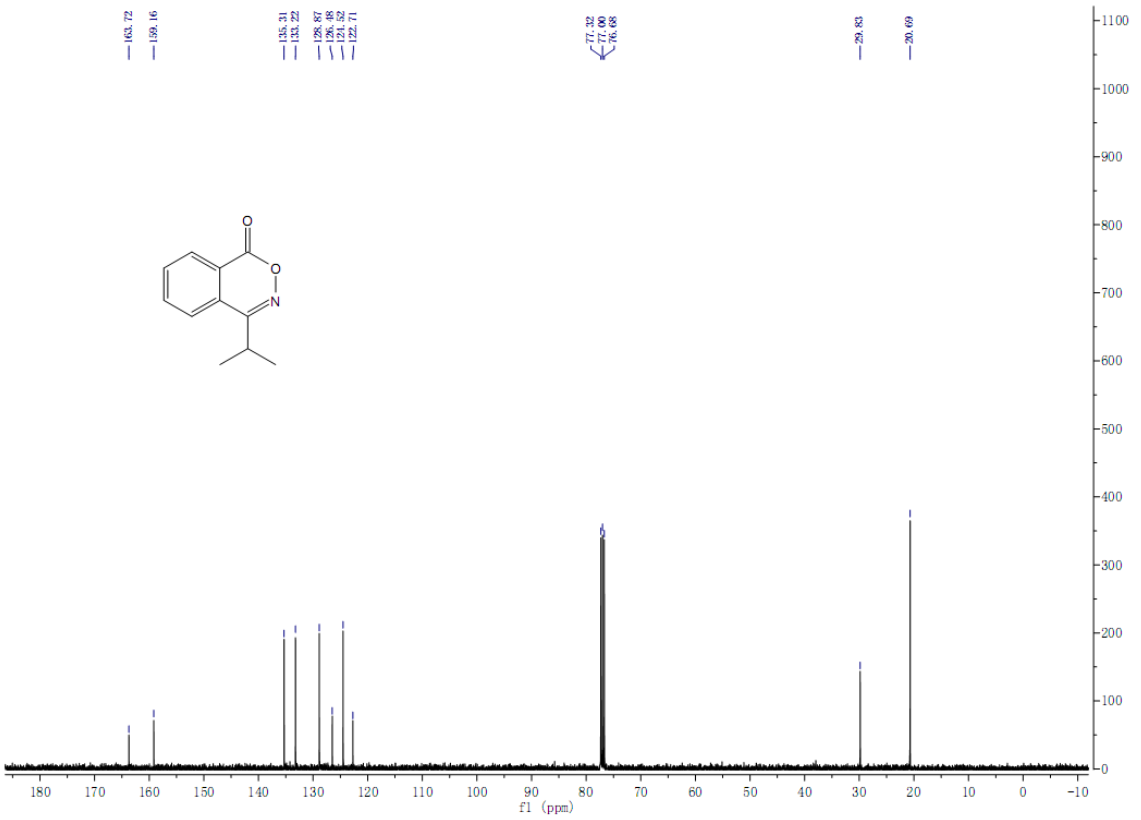
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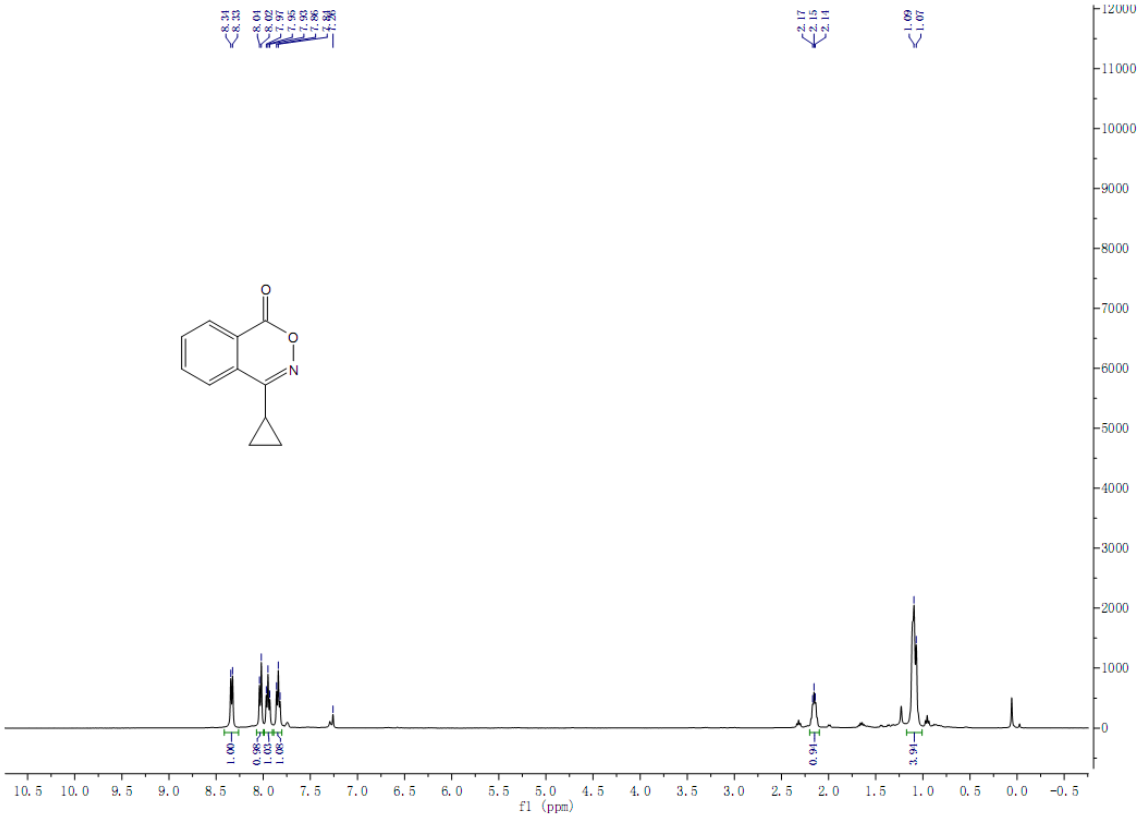
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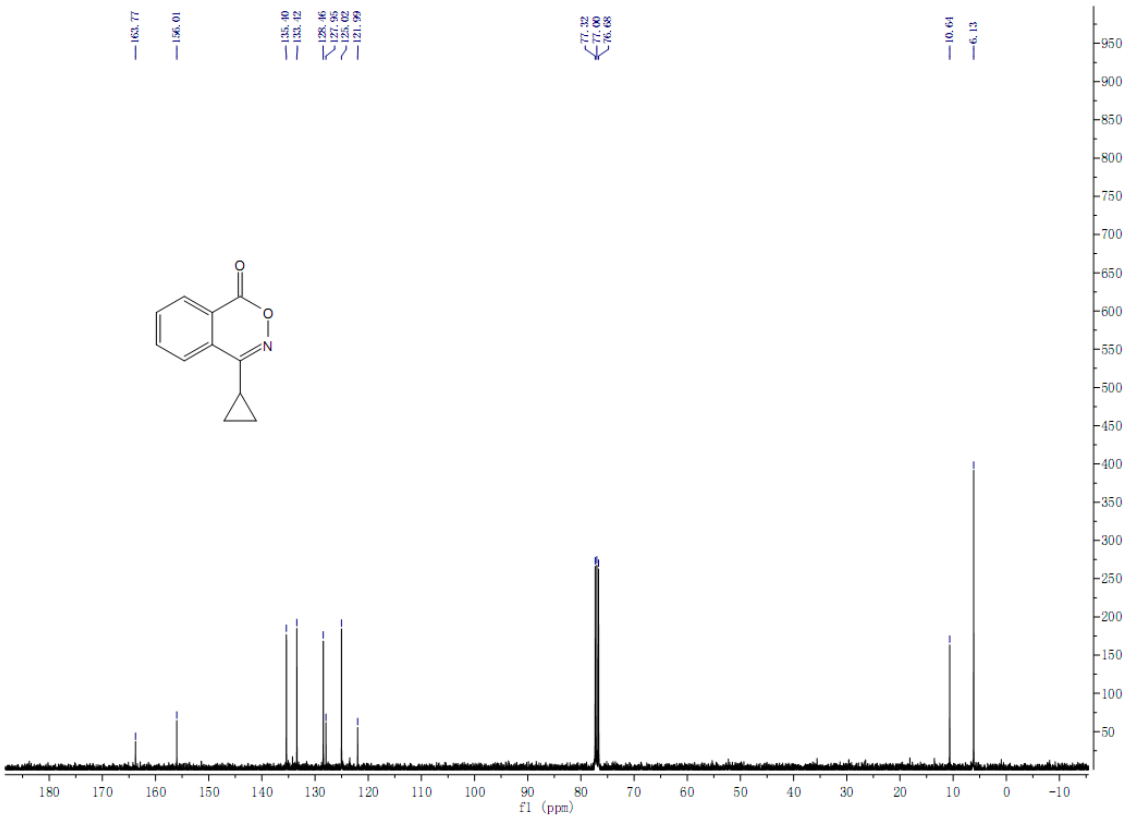
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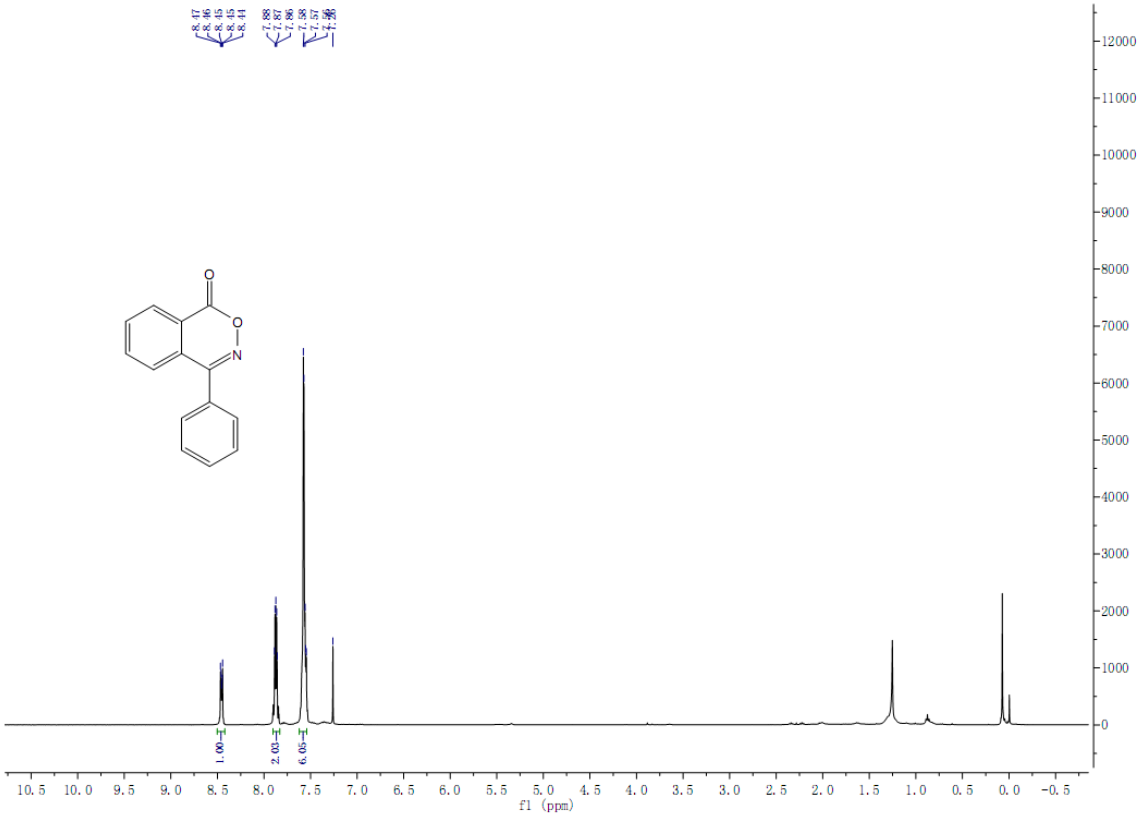
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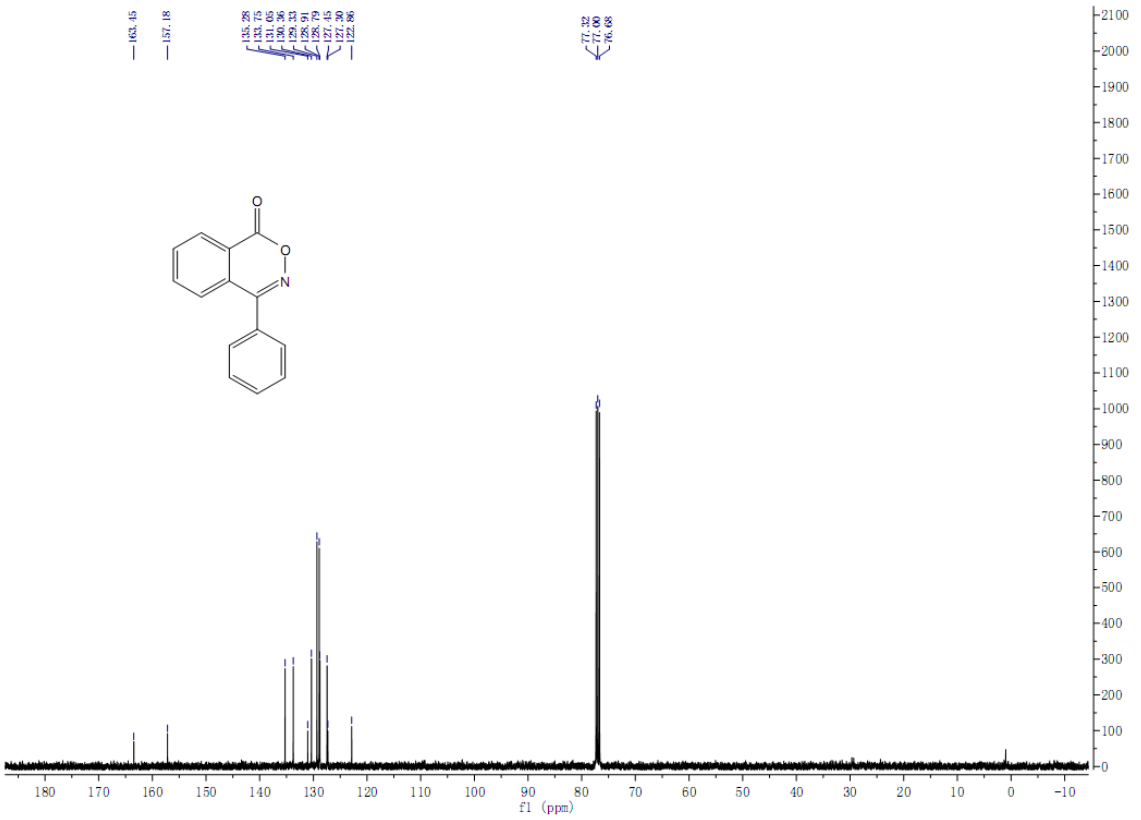
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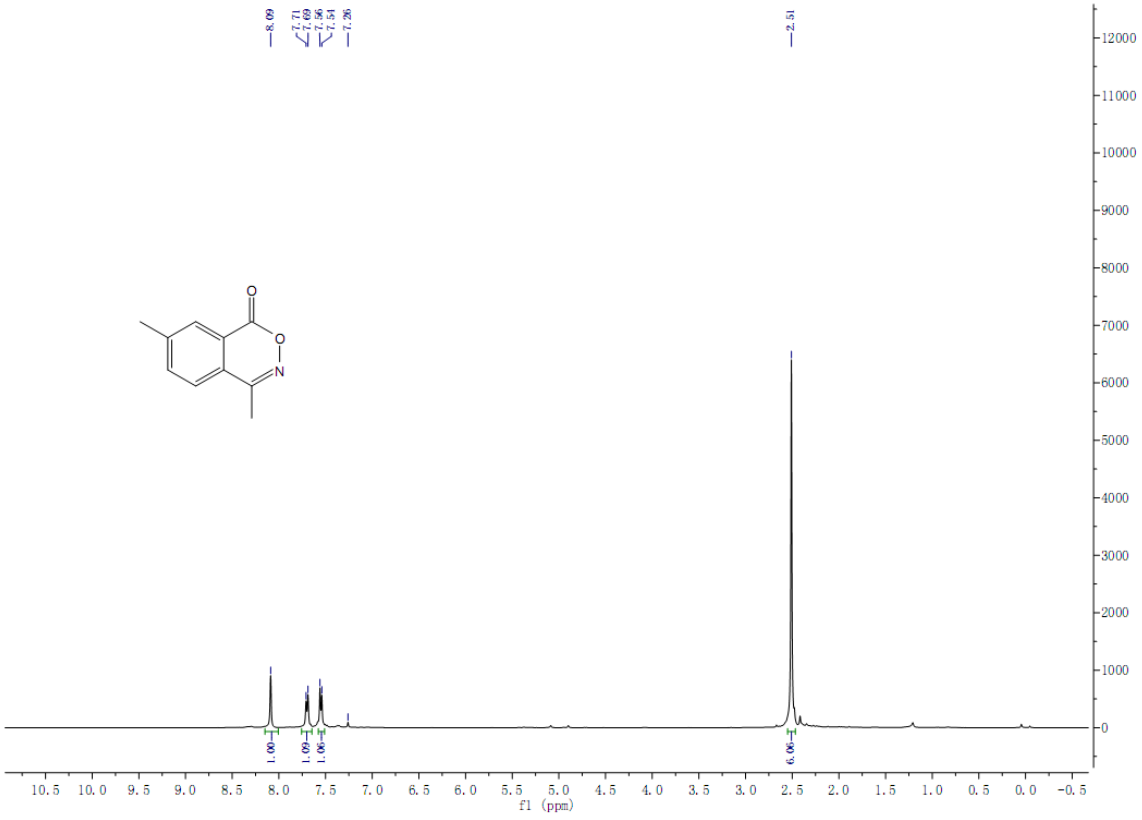
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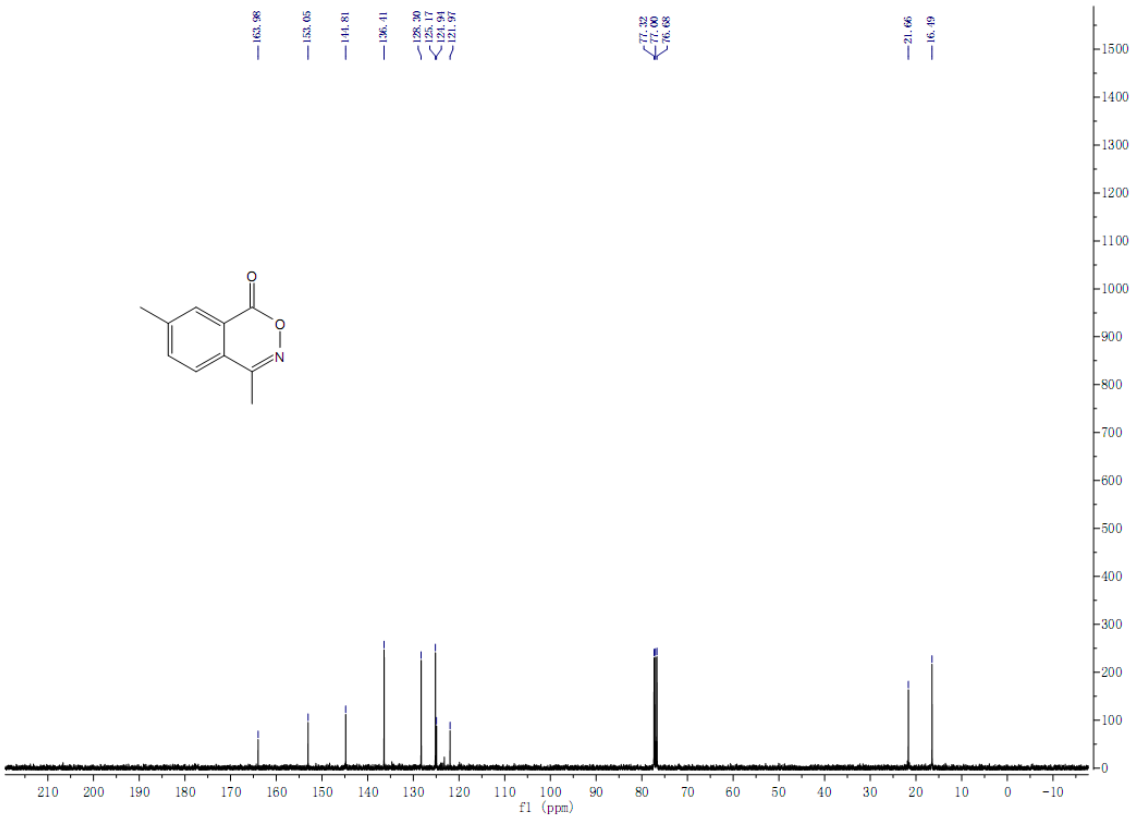
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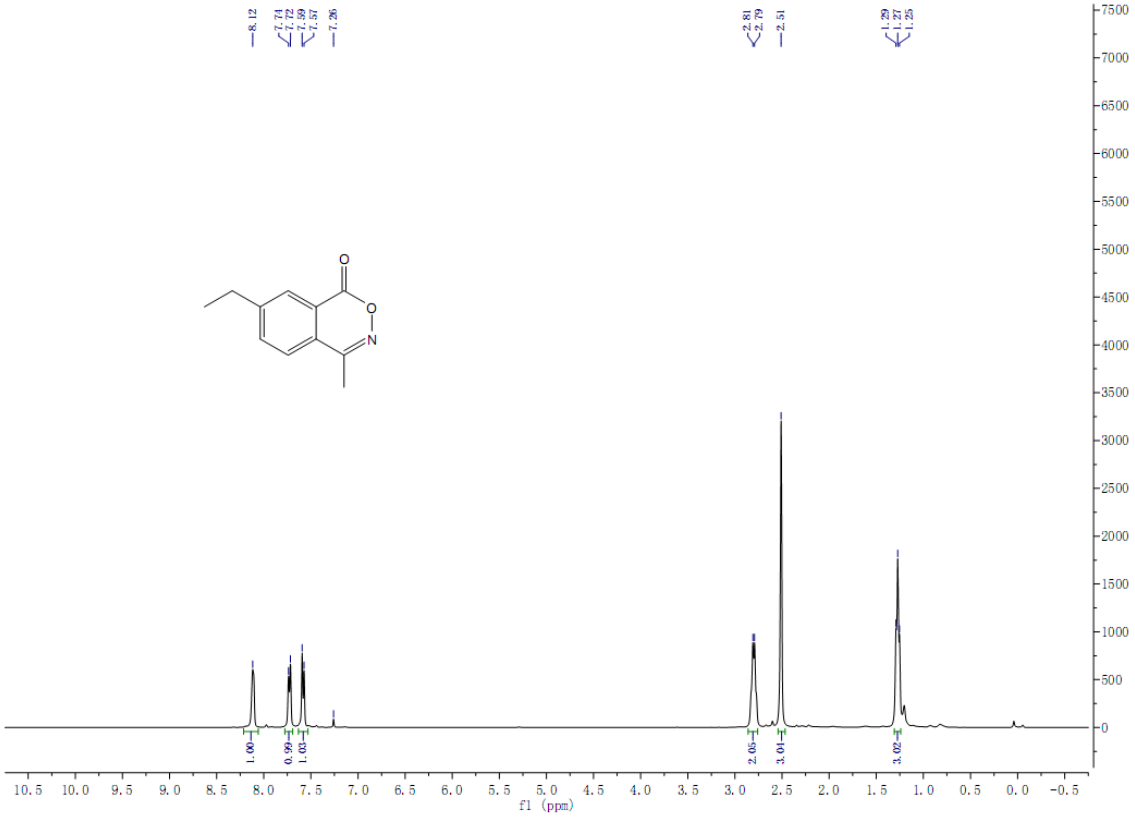
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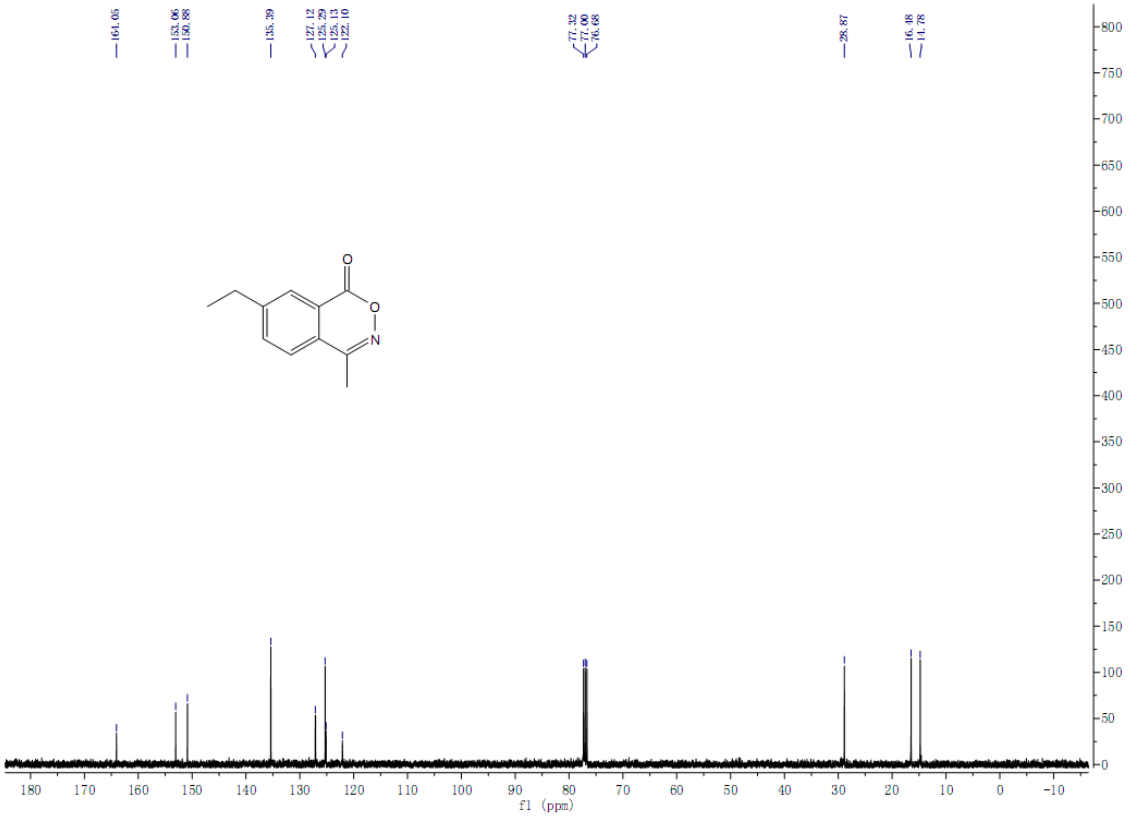
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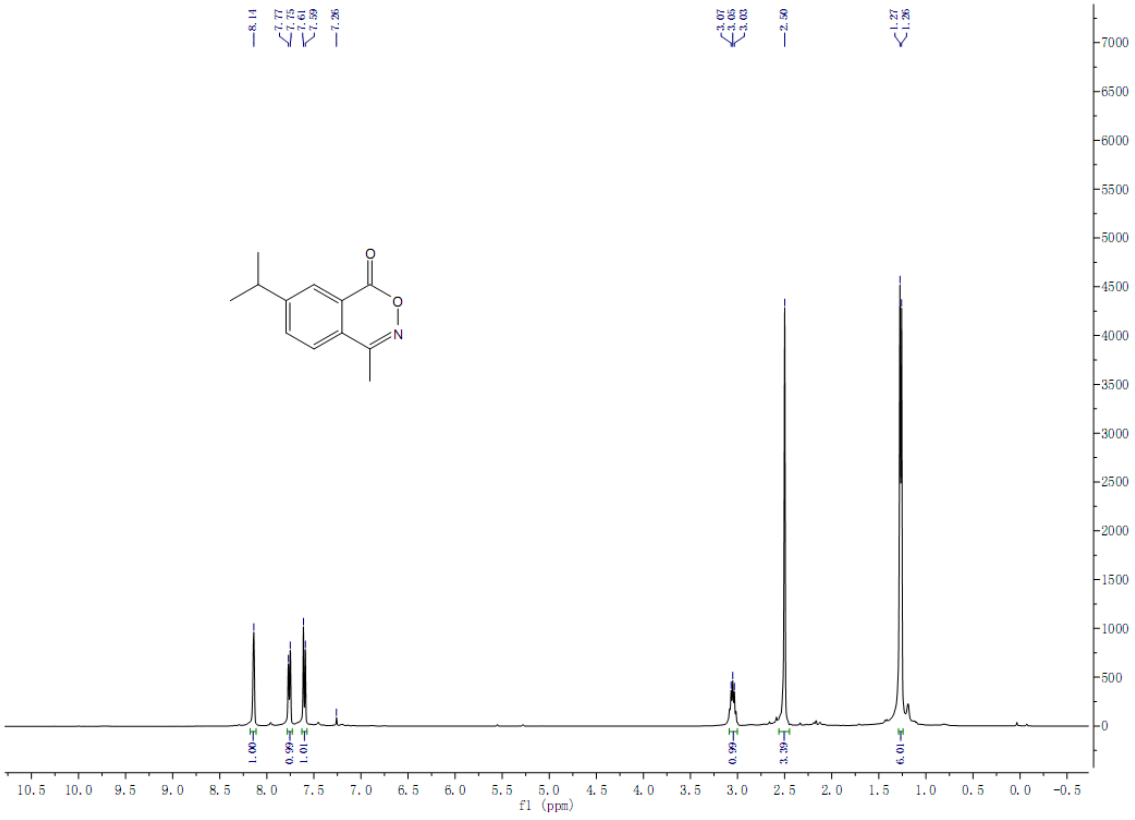
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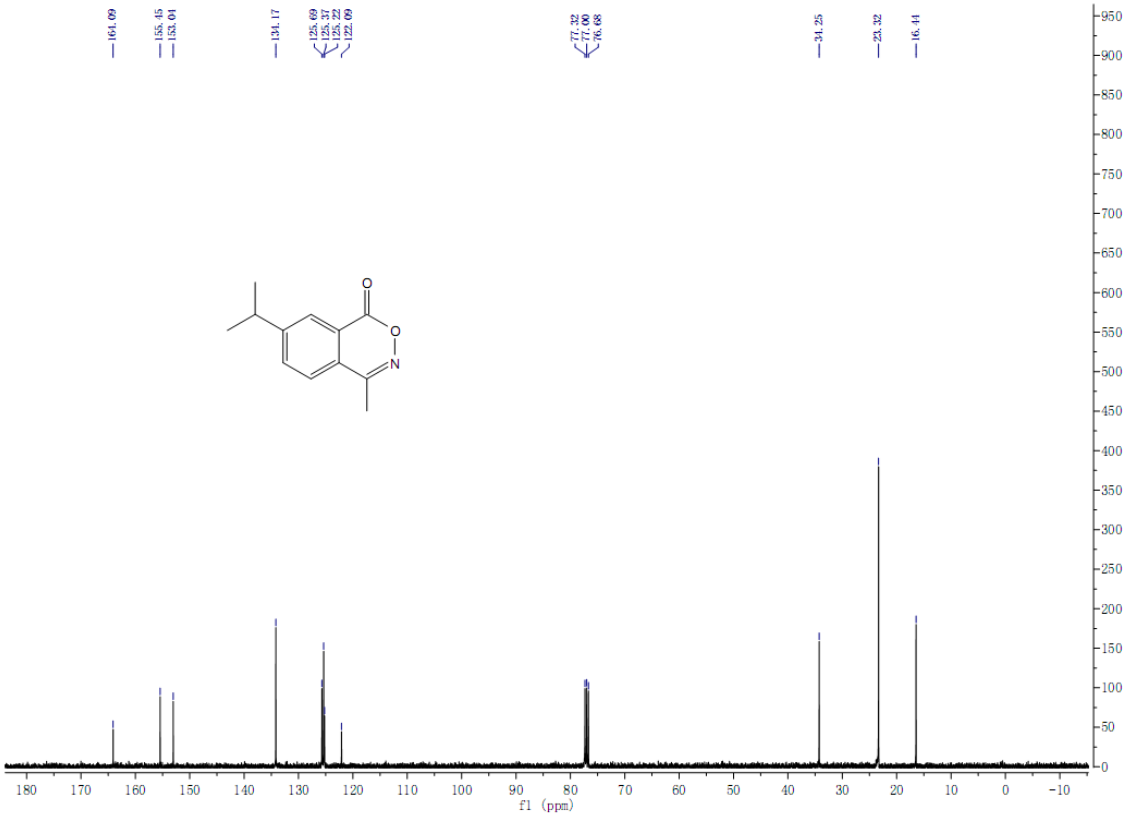
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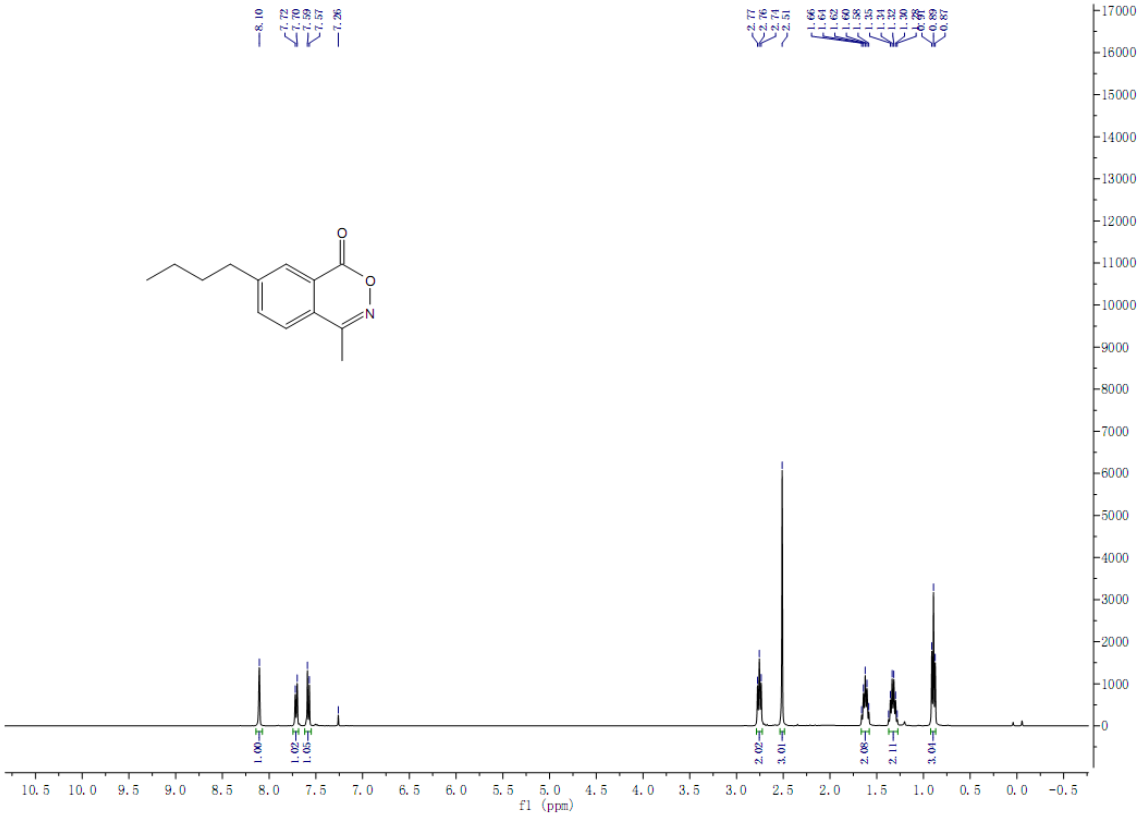
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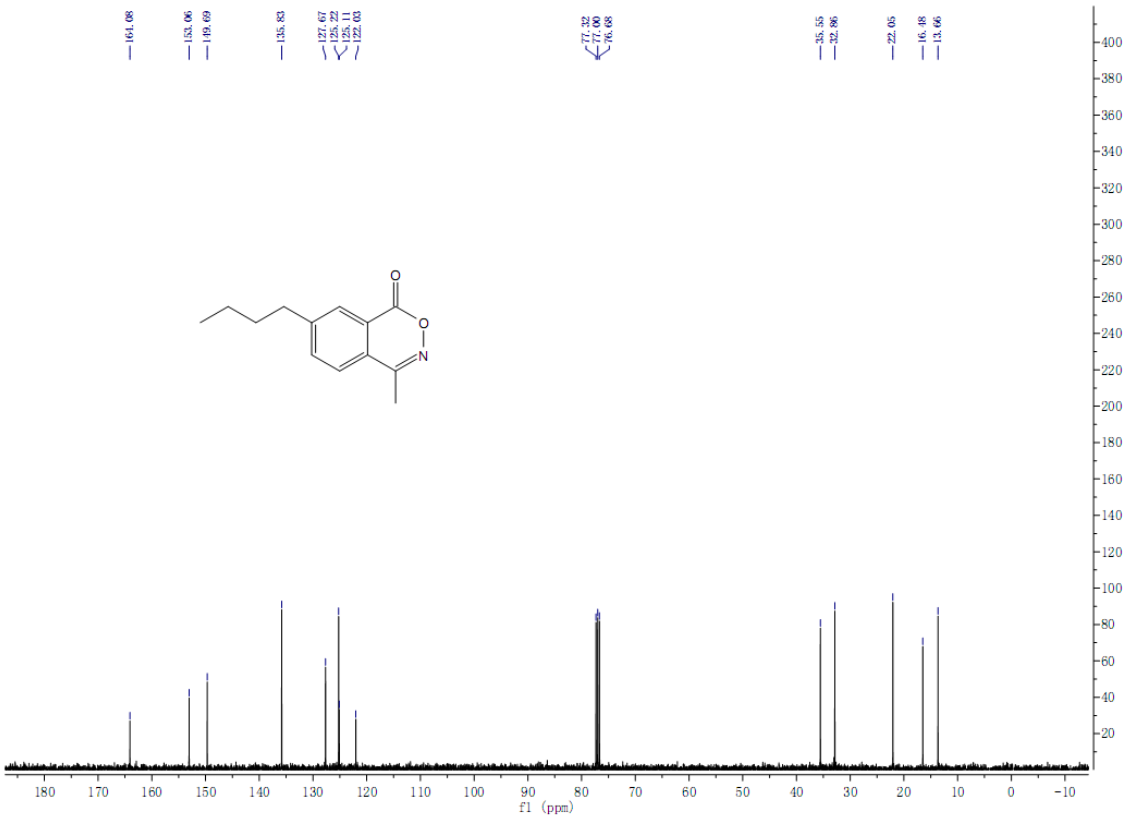
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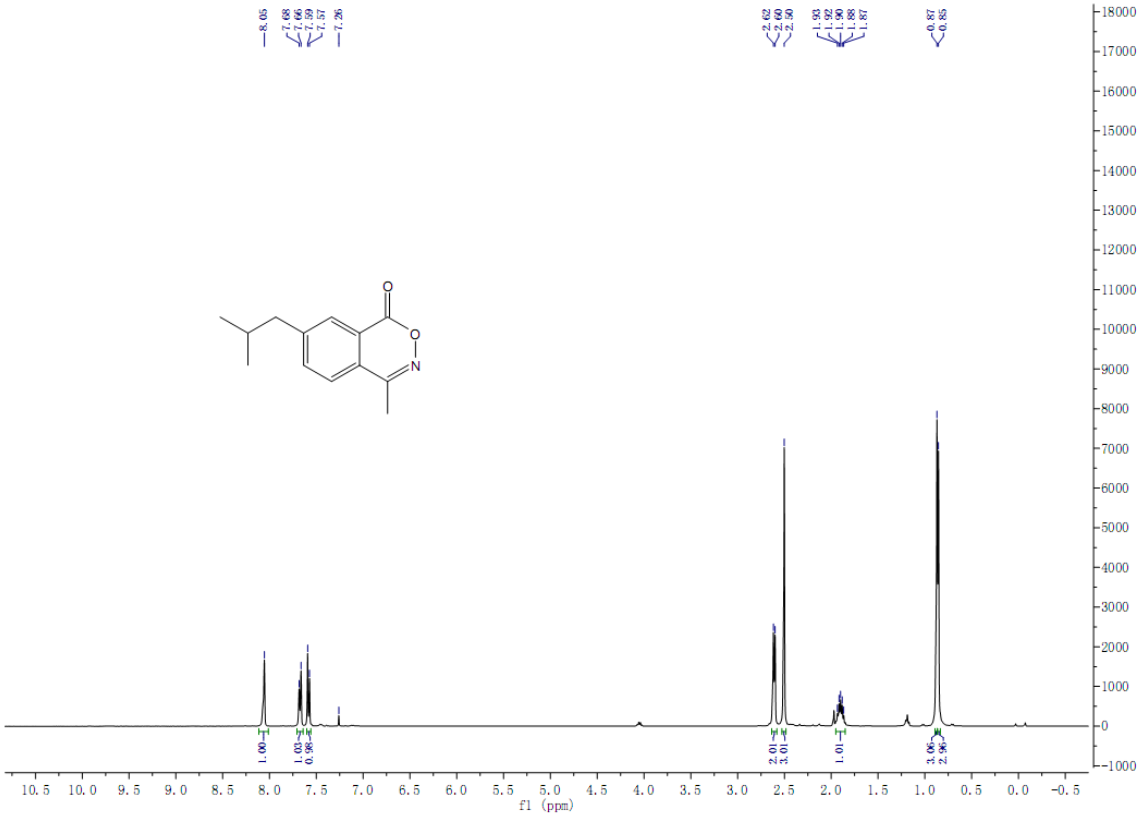
122.03
77.32
77.00
76.68
35.55
32.86
22.05
16.48
13.66
O
O
N
f1 (ppm)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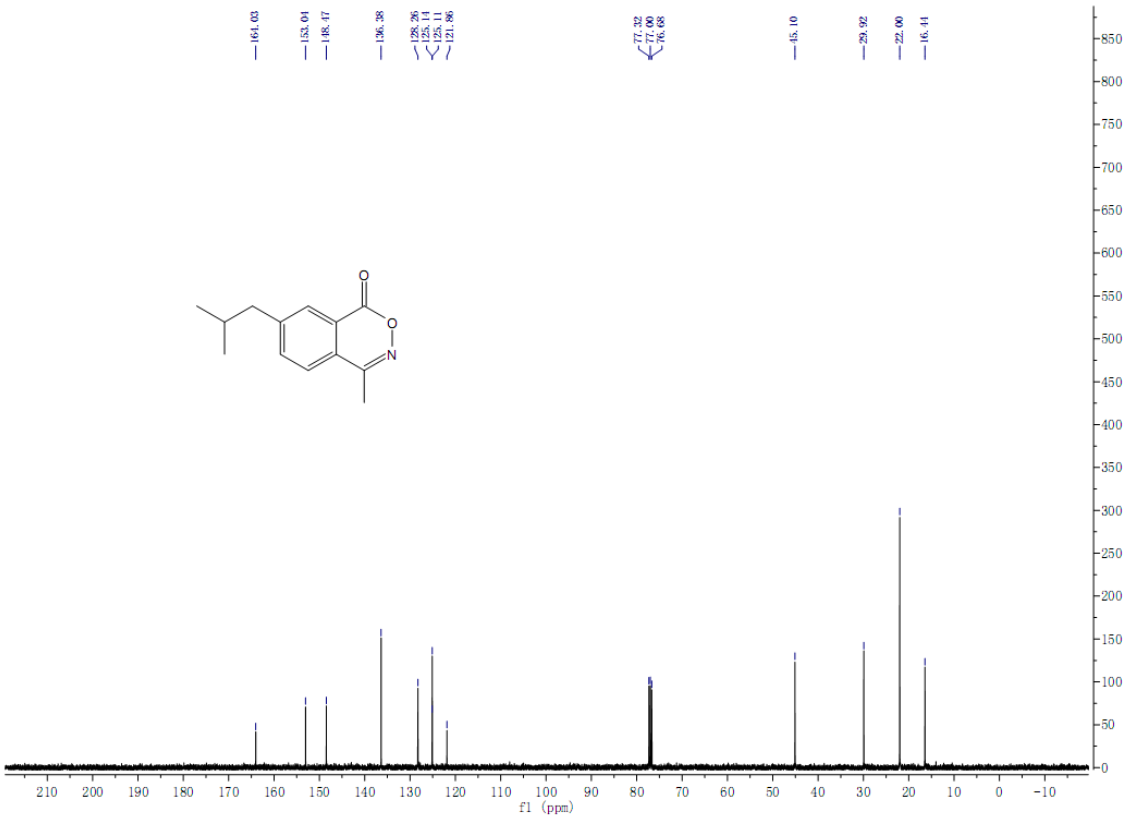
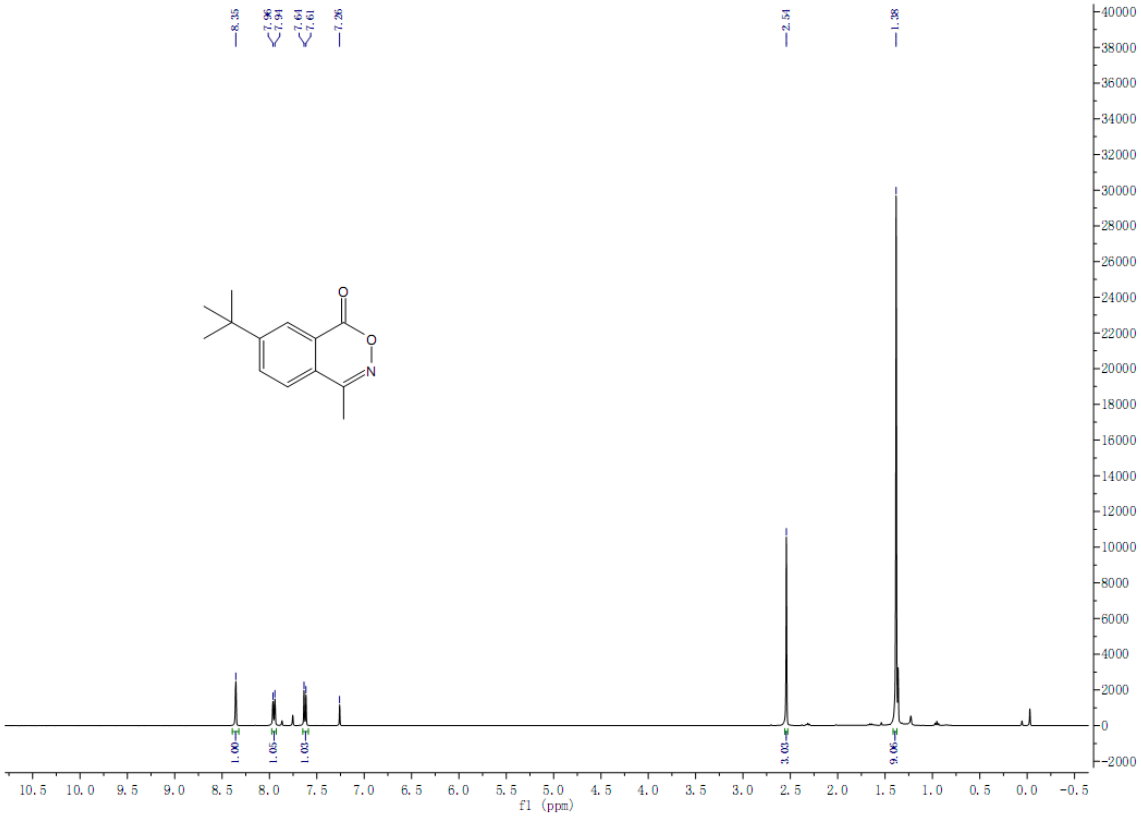
7.68
7.66
7.59
7.57
7.26
2.62
2.60
2.50
1.93
1.92
1.90
1.88
1.87
0.87
0.85
O
O
N
1.00
1.03
0.98
2.01
3.01
1.01
3.06
2.96
f1 (ppm)


164.03
153.04
148.47
136.38
128.26
125.14
125.11
121.86
77.32
77.00
76.68
45.10
29.92
22.00
16.44
O
O
N
f1 (ppm)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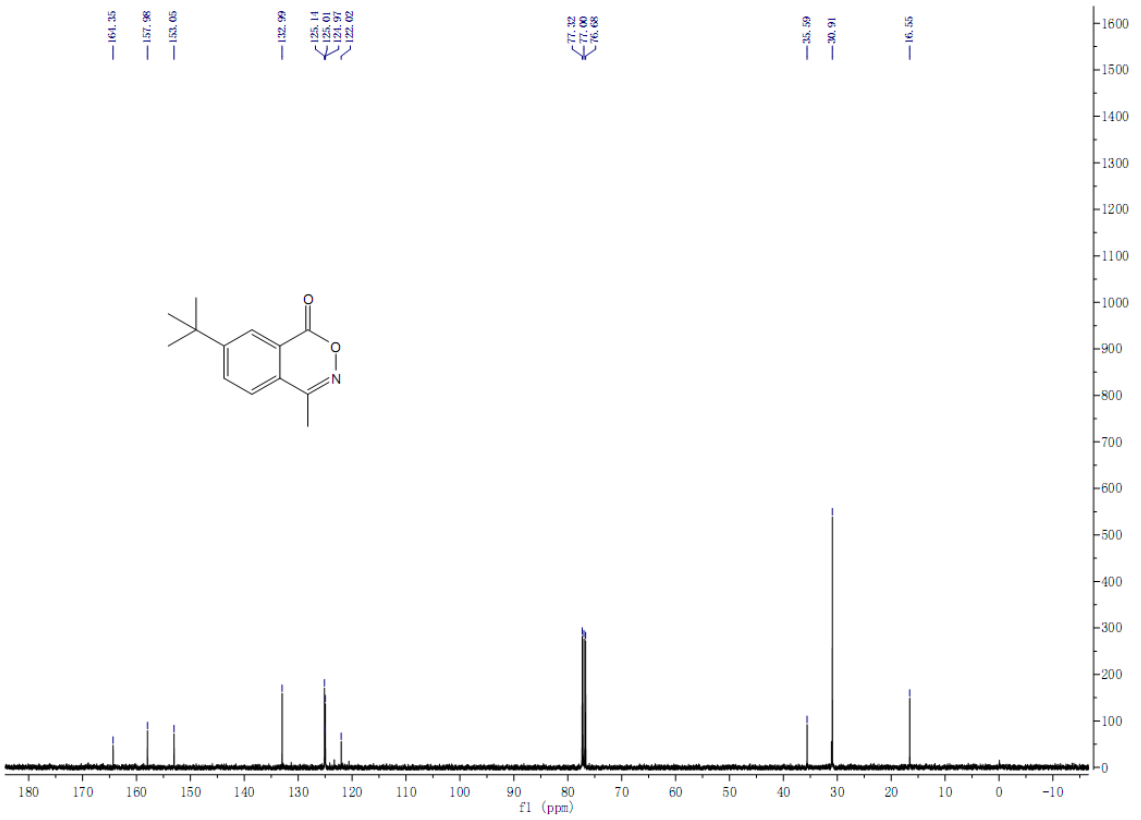
7.96
7.94
7.64
7.61
7.26
2.54
1.38
1.00
1.05
1.03
3.03
9.06
f1 (ppm)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157.98
153.05
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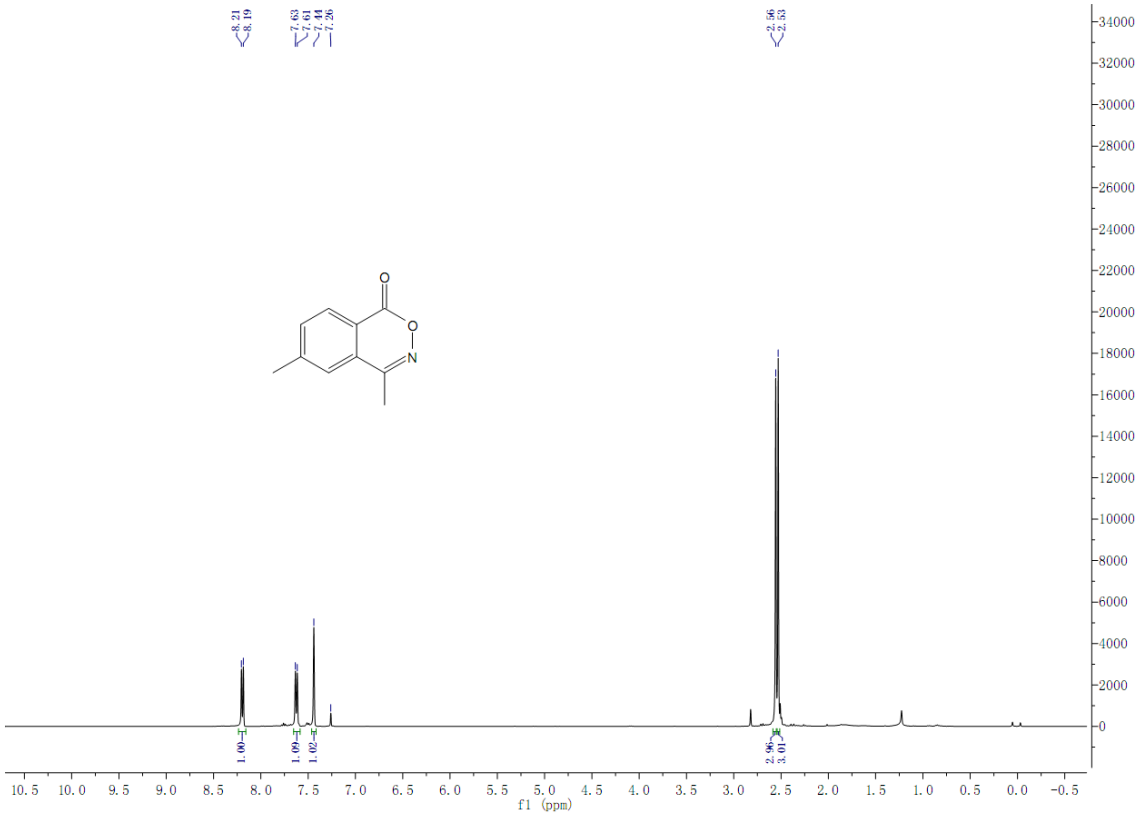
125.14
125.01
124.97
122.02
77.32
77.00
76.68
35.59
30.91
16.55
f1 (ppm)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8.19
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7.61
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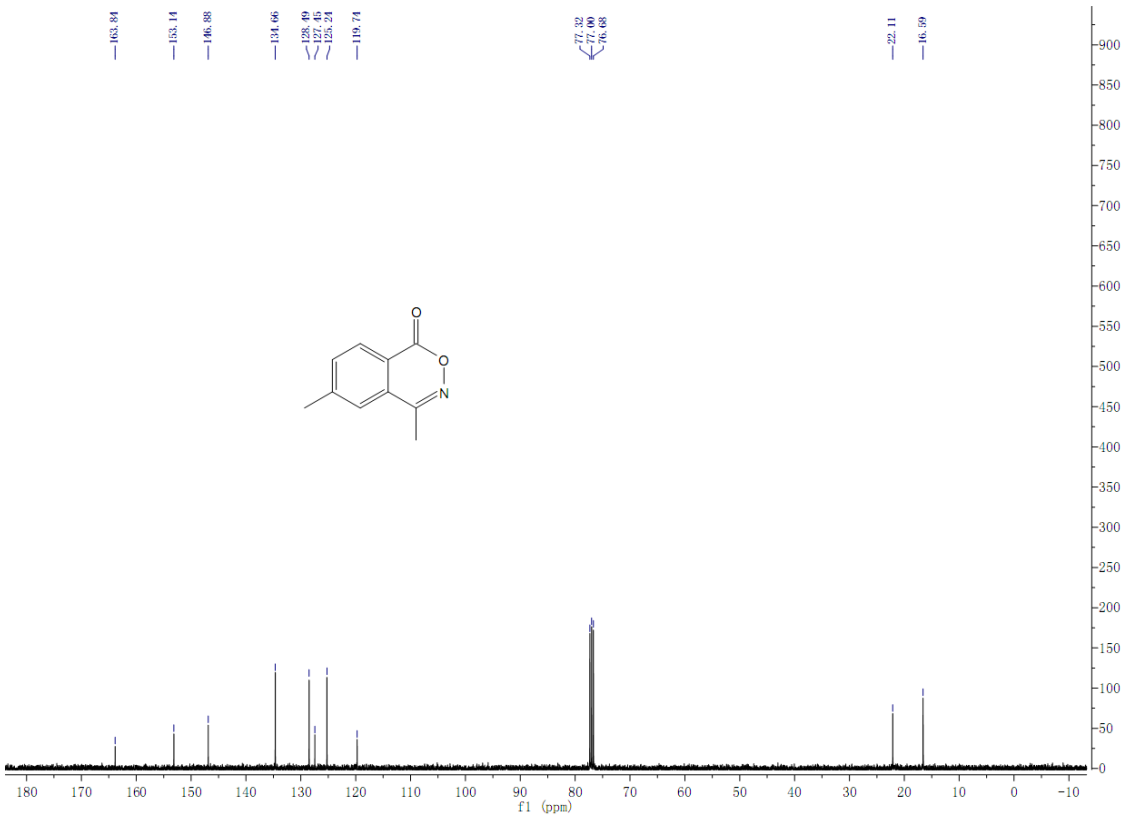
7.26
2.56
2.53
1.00
1.09
1.02
2.96
3.01
f1 (ppm)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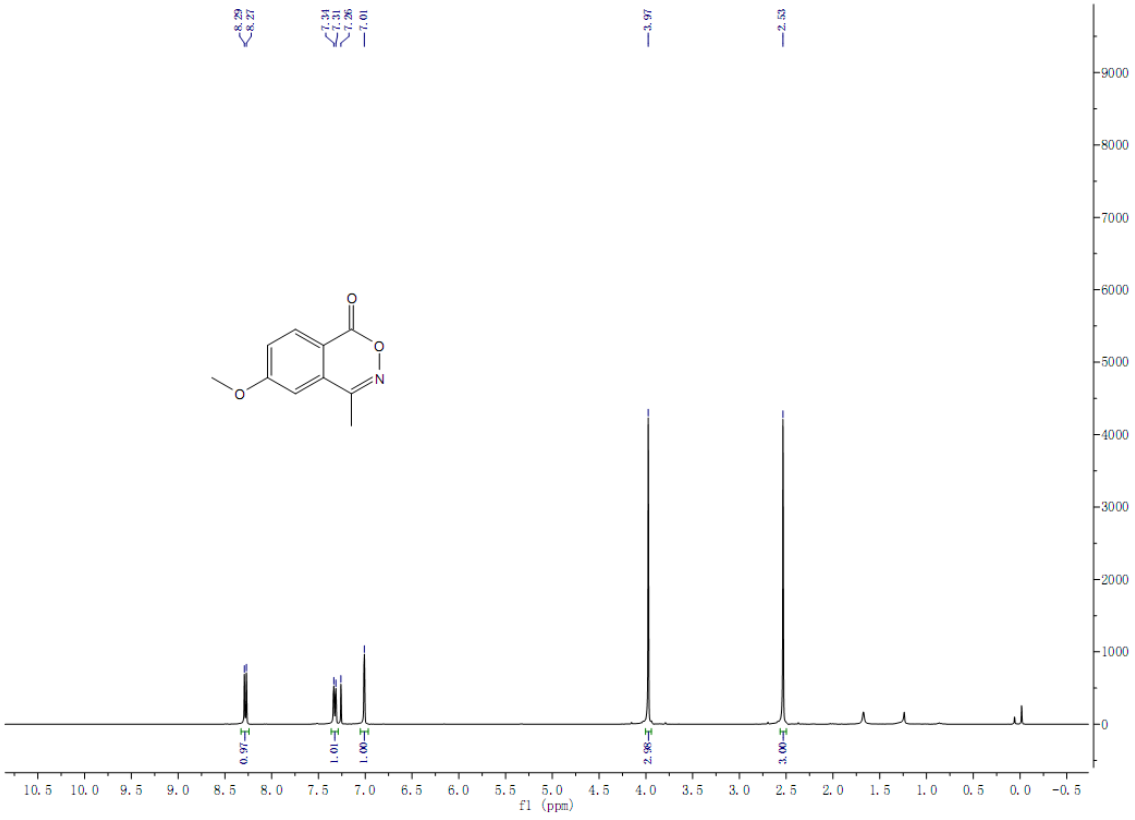
127.45
125.24
119.74
77.32
77.00
76.68
22.11
16.59
f1 (ppm)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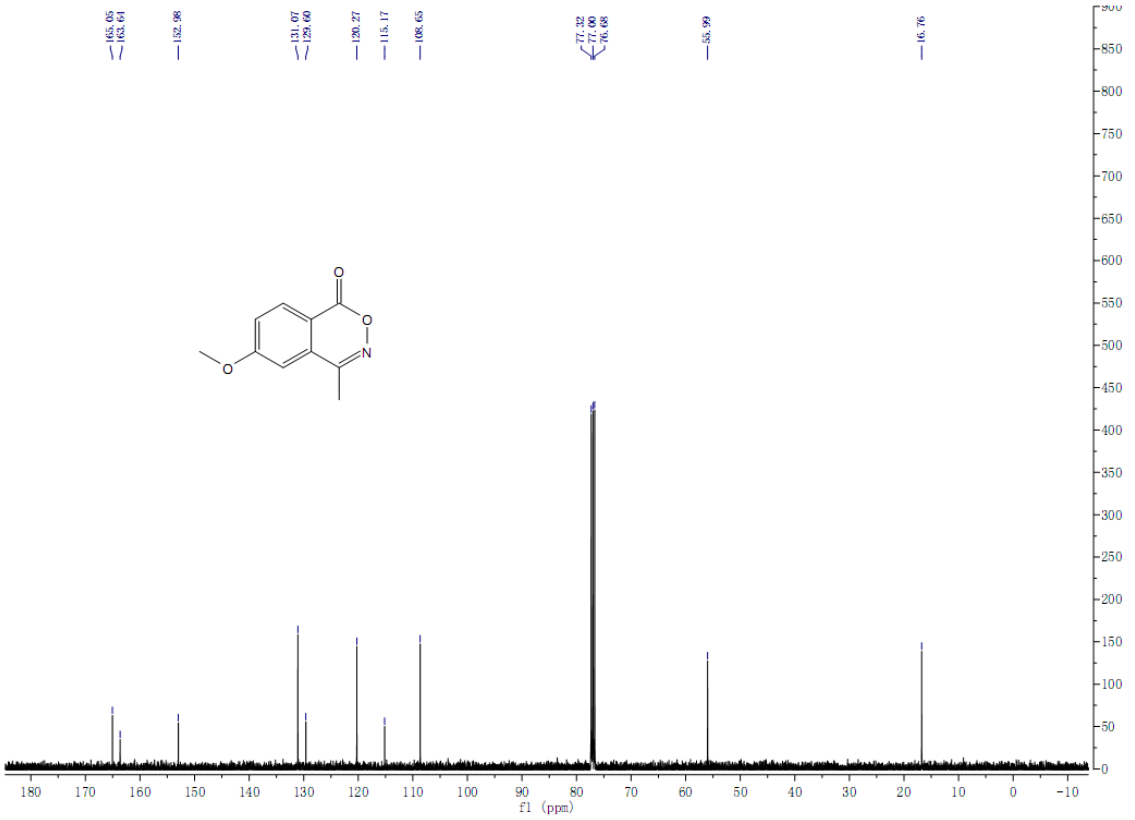
8.27
7.34
7.31
7.26
7.01
3.97
2.53
0.97
1.01
1.00
2.98
3.00
f1 (ppm)


165.65
163.64
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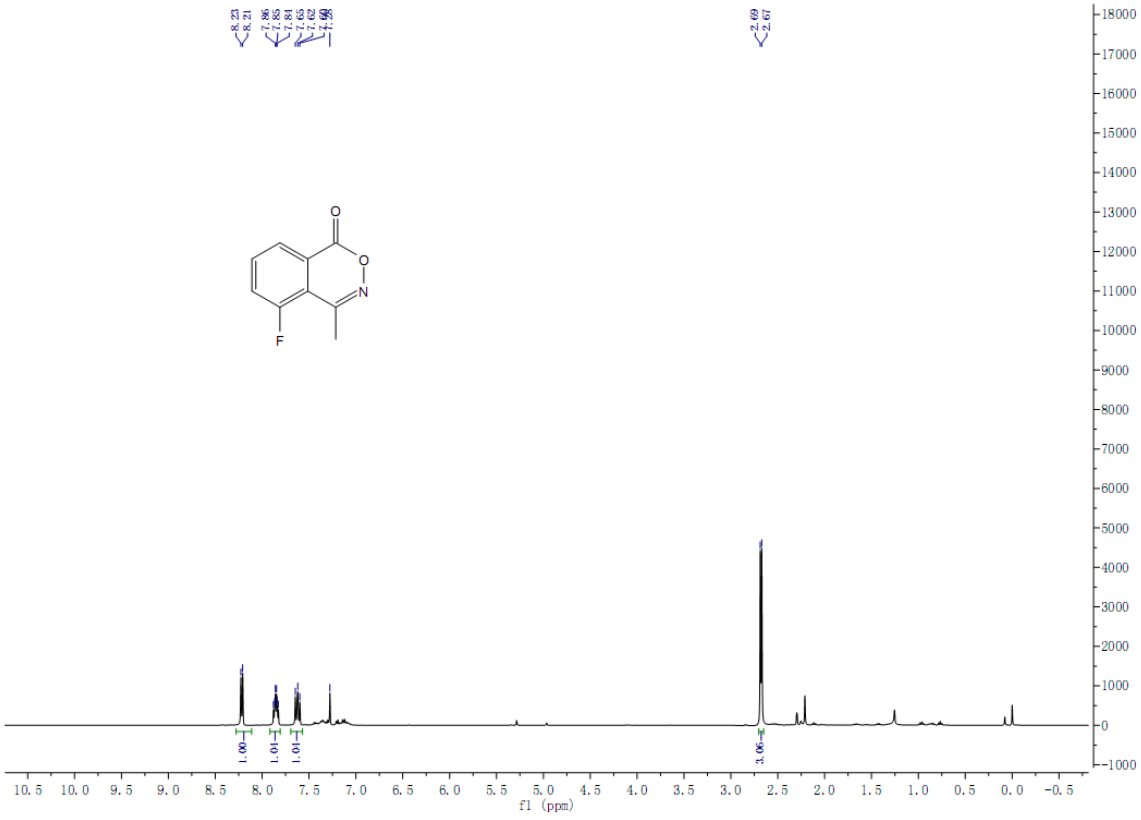
131.07
129.60
120.27
115.17
106.65
77.32
77.00
76.68
55.99
16.76
f1 (ppm)


8.23
8.21
7.86
7.85
7.83
7.65
7.62
7.60
2.69
2.67
O
O
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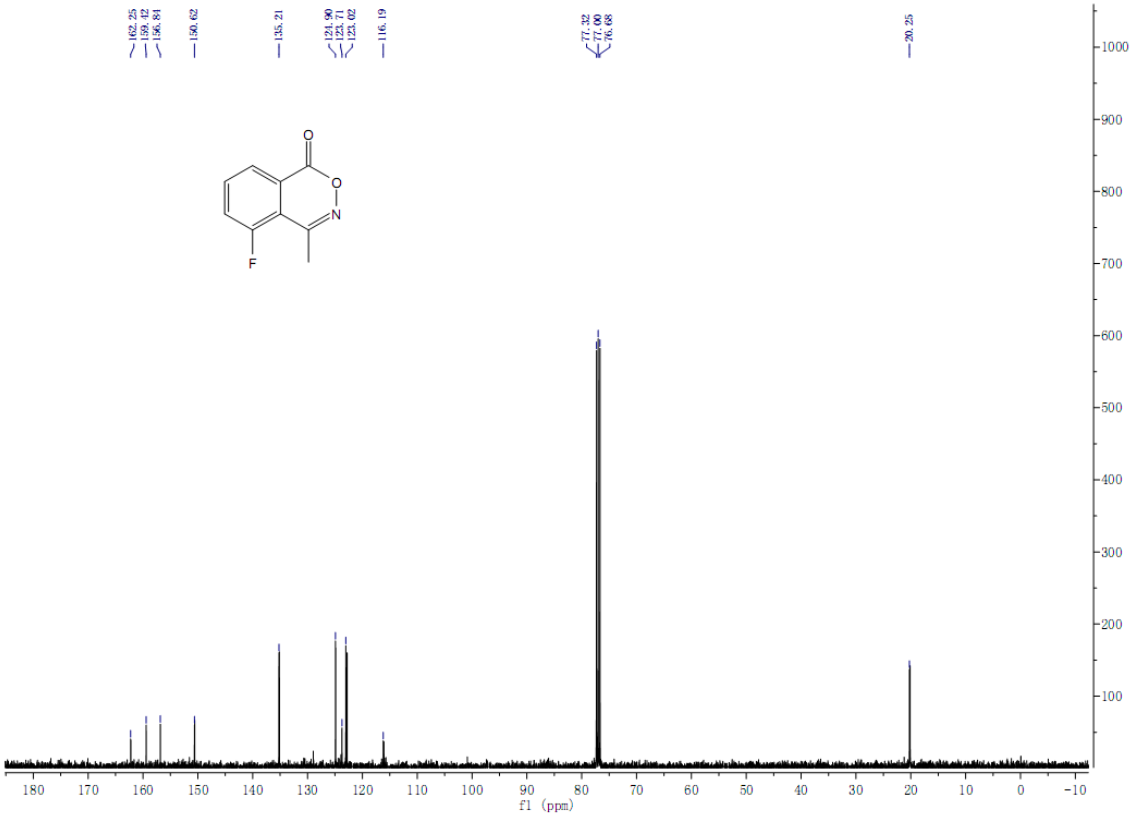
F
1.00
1.01
1.01
3.06
f1 (ppm)


162.25
159.42
156.84
150.62
135.21
124.90
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123.02
116.19
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77.00
76.68
20.25
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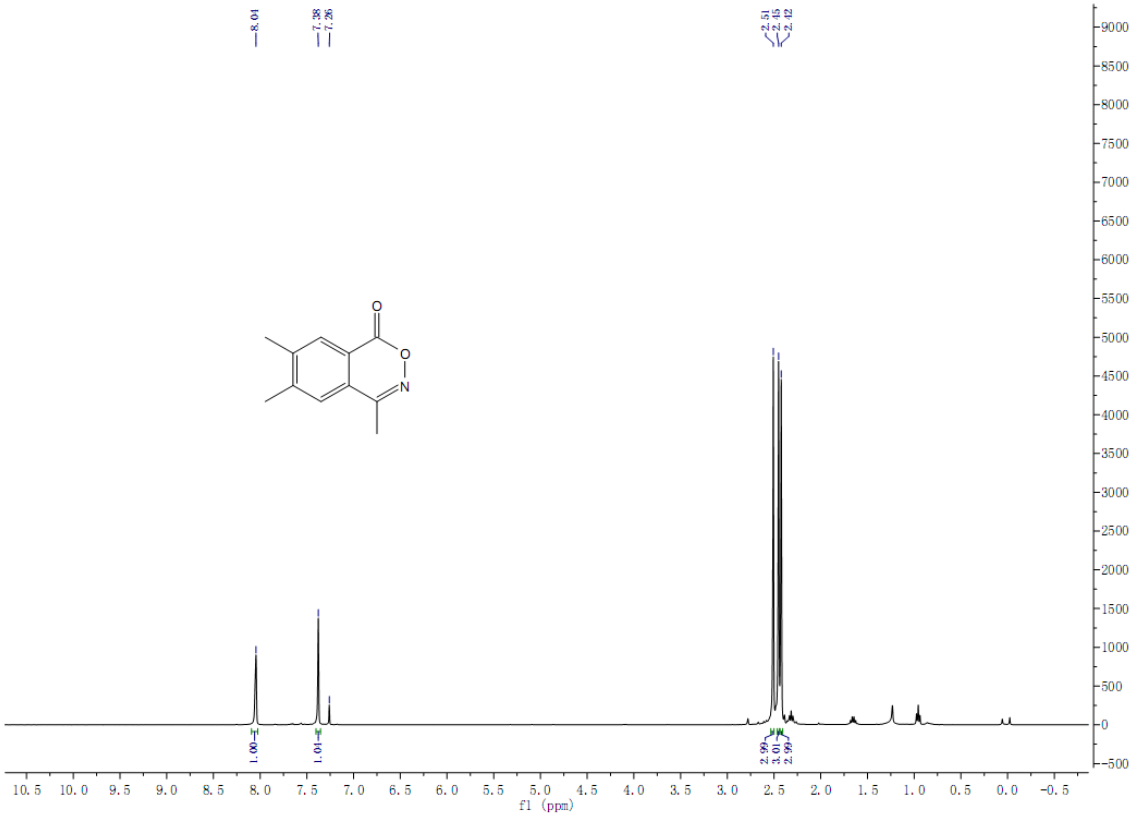
O
N
F
f1 (ppm)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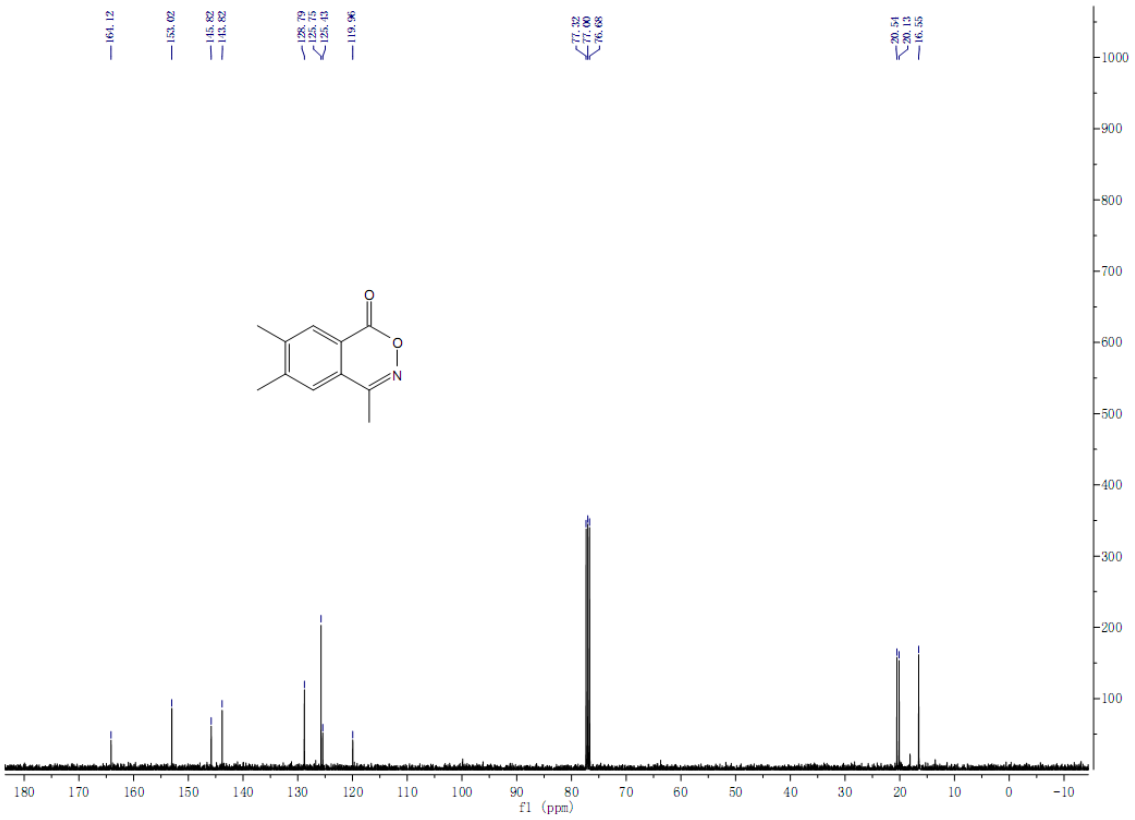
7.38
7.26
2.51
2.45
2.42
1.00
1.04
2.99
3.01
2.99
f1 (ppm)


164.12
153.02
145.82
145.82
128.79
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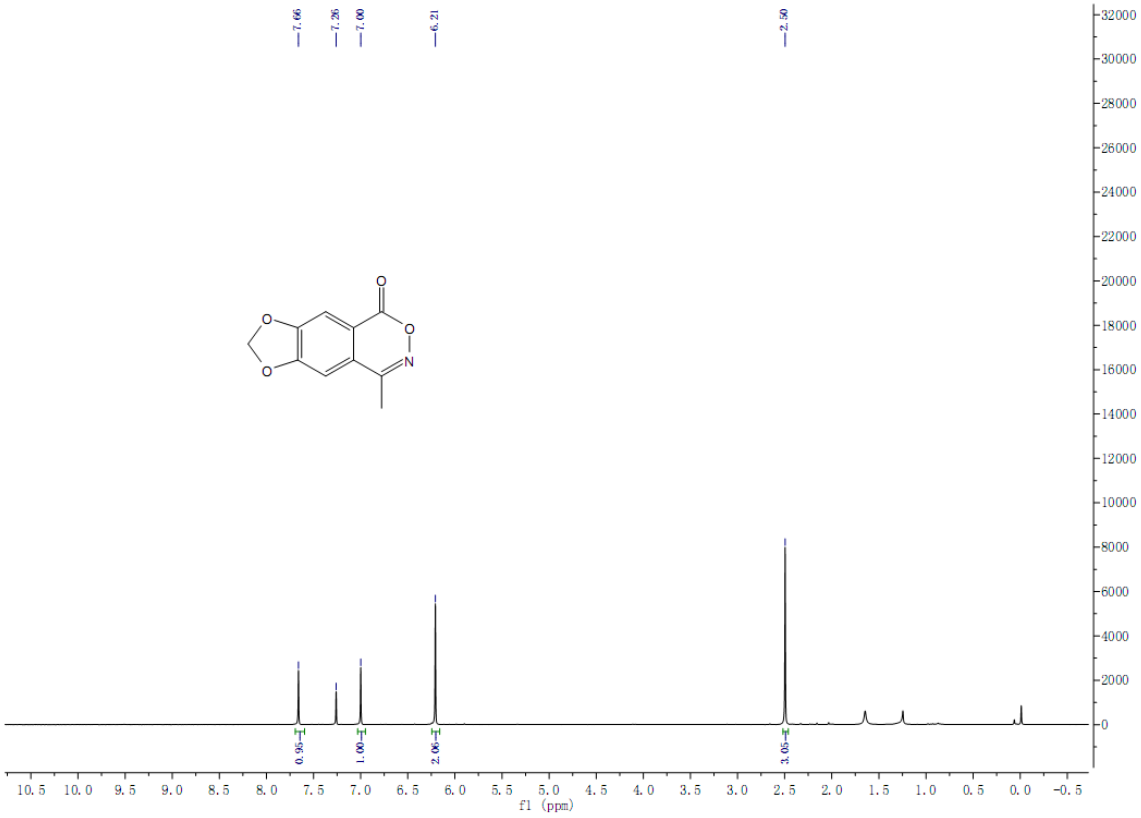
125.43
119.96
77.32
77.00
76.68
20.54
20.13
16.55
f1 (ppm)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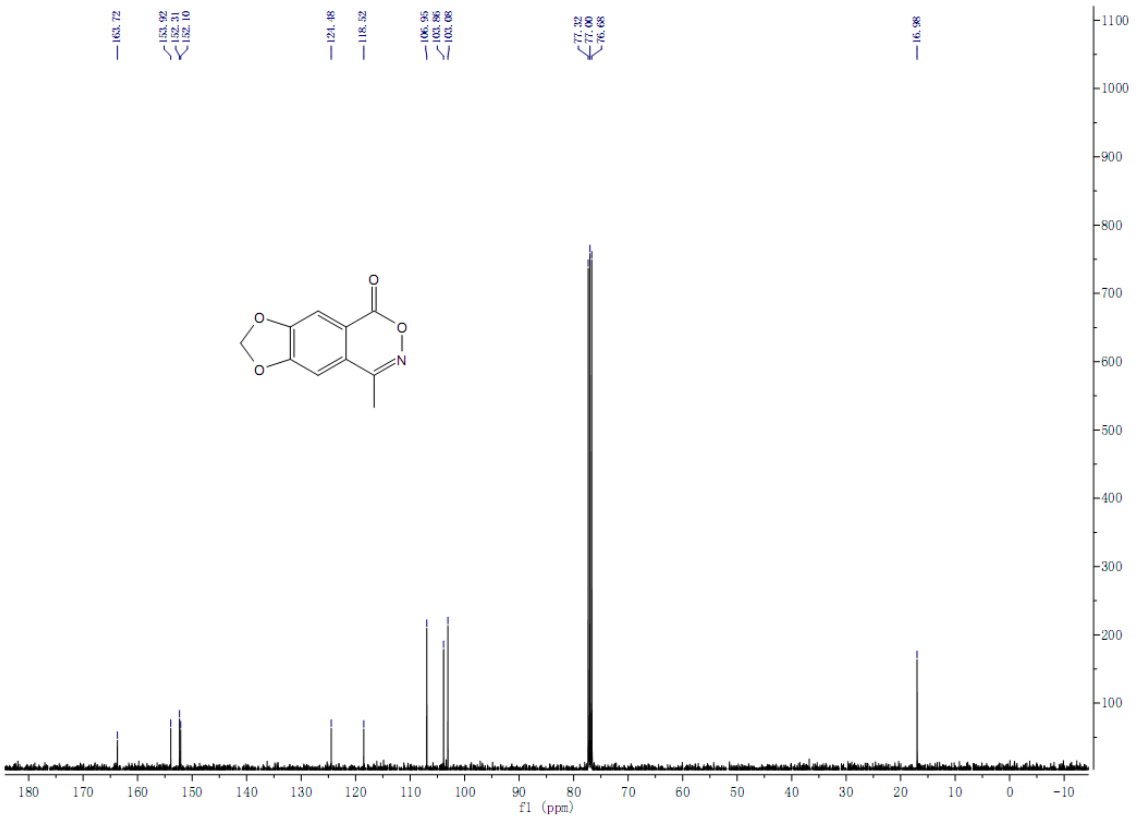
7.26
7.00
6.21
2.50
0.95
1.00
2.06
3.05
f1 (ppm)


163.72
153.92
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152.10
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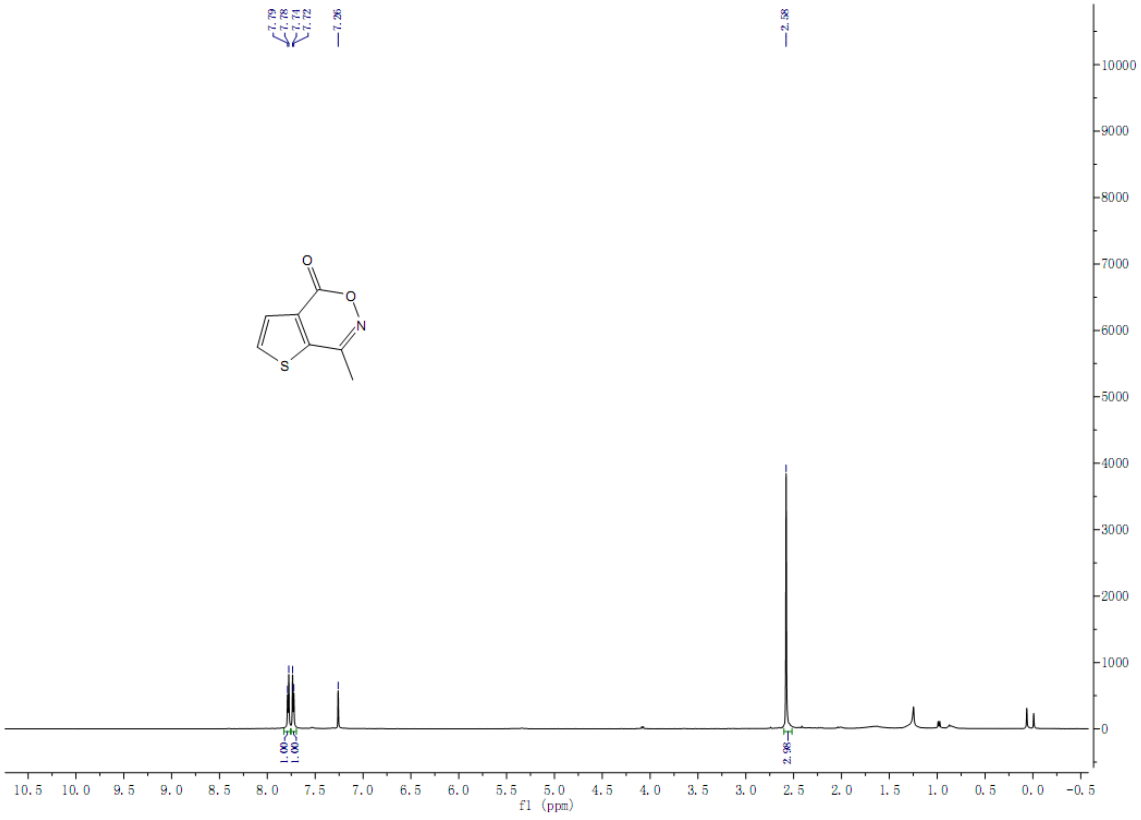
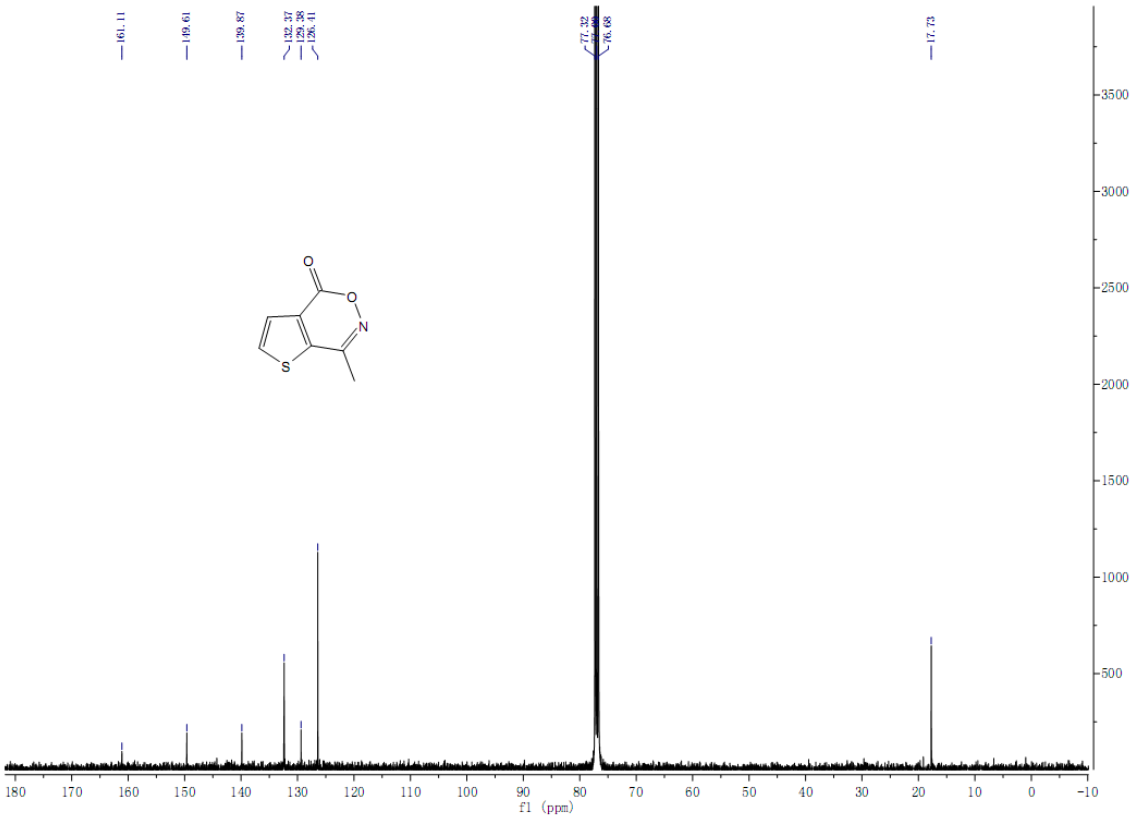
118.52
106.95
103.86
103.68
77.32
77.00
76.68
16.98
f1 (ppm)


7.79
7.78
7.74
7.72
7.26
2.58
O
O
N
S
1.00
1.00
2.98
f1 (ppm)


161.11
149.61
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132.37
129.38
126.41
77.32
76.68
17.73
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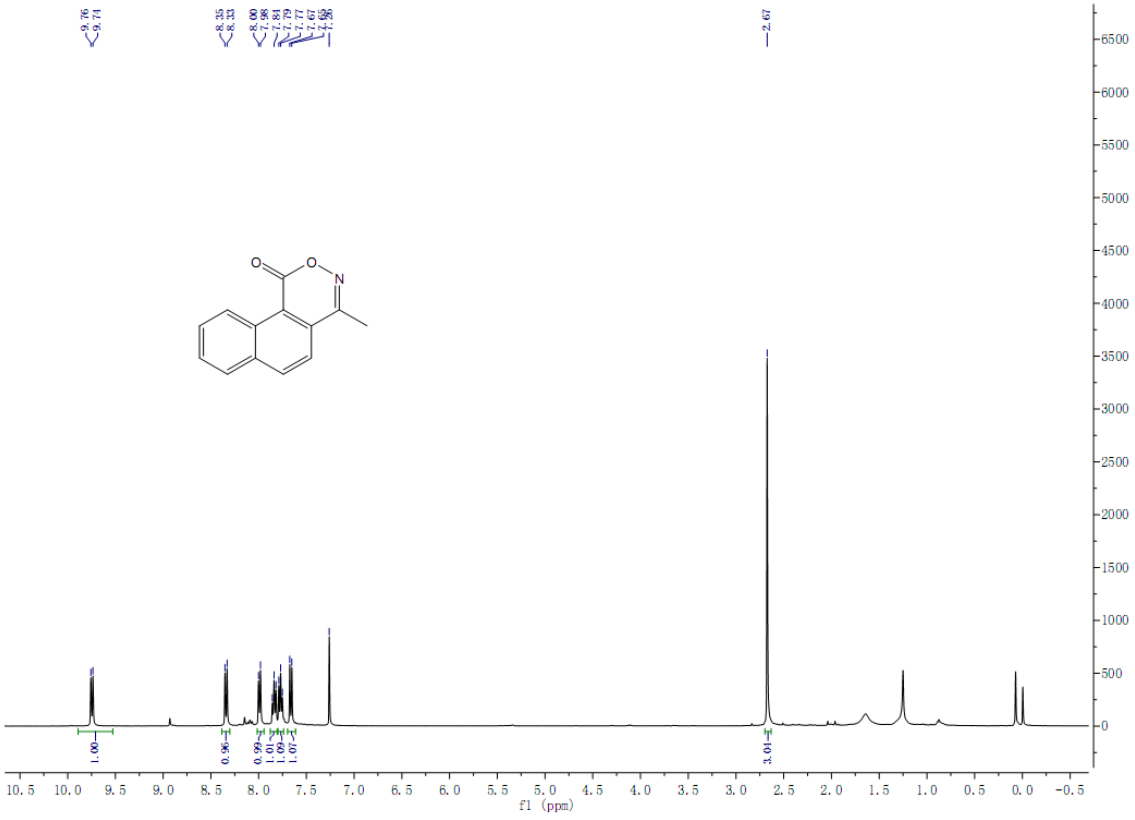
O
N
S
f1 (ppm)


9.76
9.74
8.35
8.33
8.00
7.98
7.94
7.79
7.77
7.67
7.66
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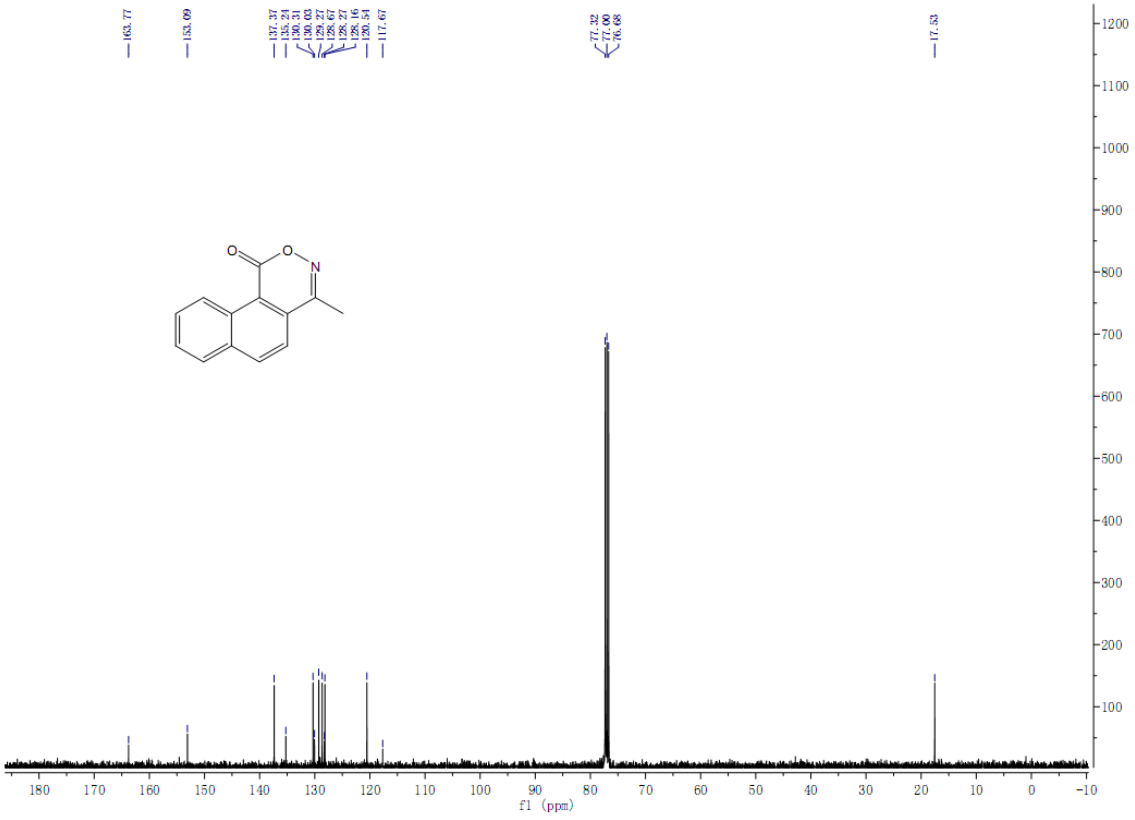
O
N
1.00
0.96
0.99
1.01
1.09
1.07
3.04
f1 (ppm)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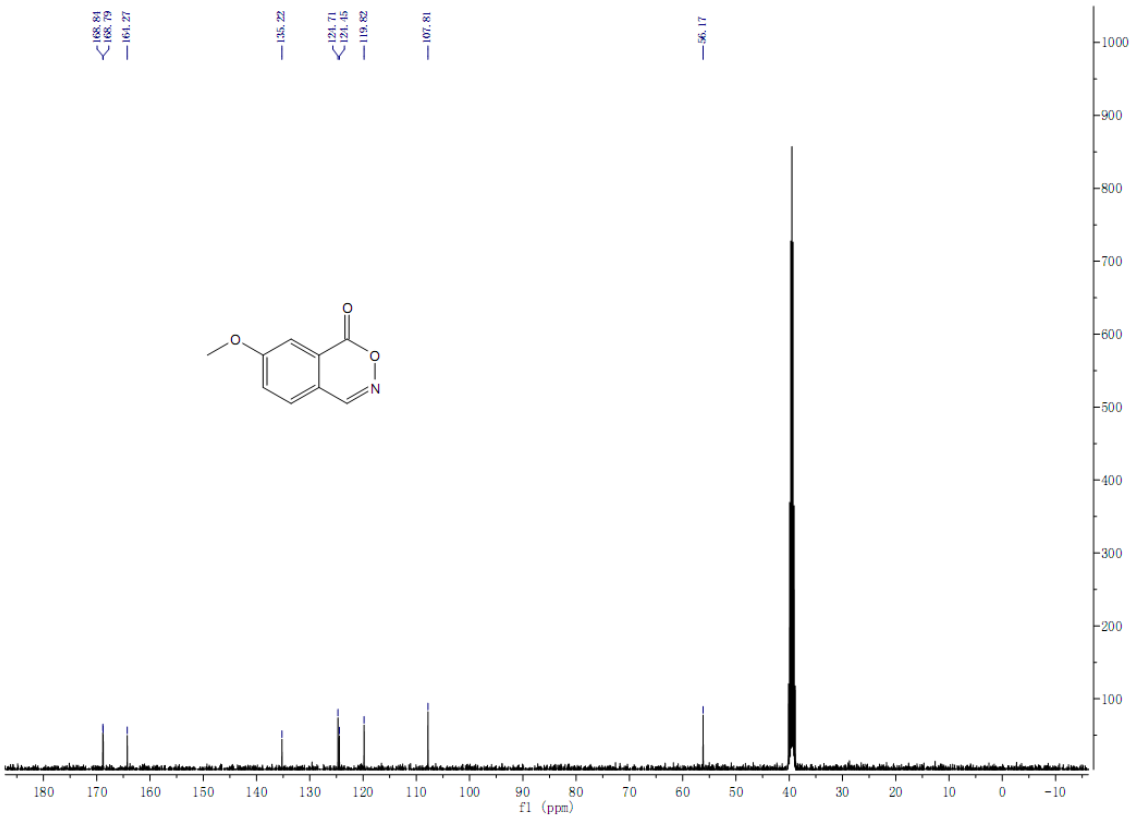
130.03
128.27
128.07
128.27
128.16
120.54
117.67
77.32
77.00
76.68
17.53
O
O
N
f1 (ppm)


11.15
7.74
7.72
7.30
7.28
3.91
1.00
1.07
2.04
3.05
f1 (ppm)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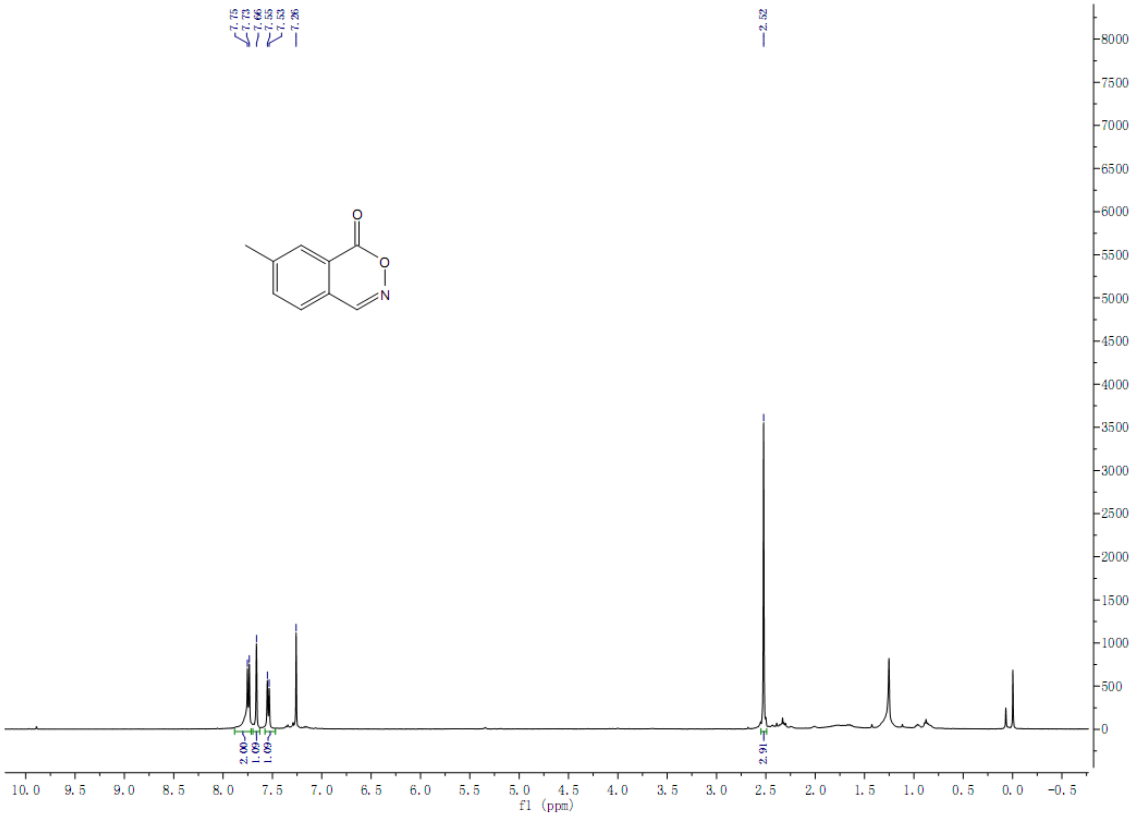
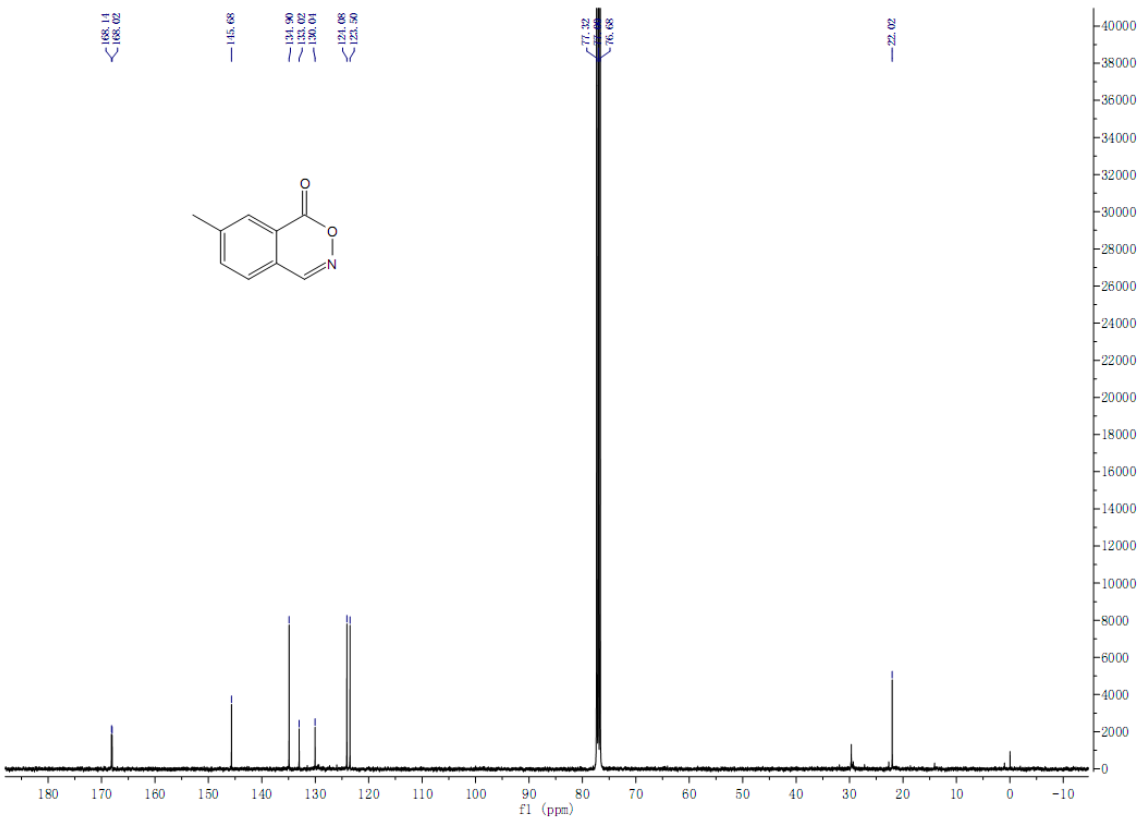
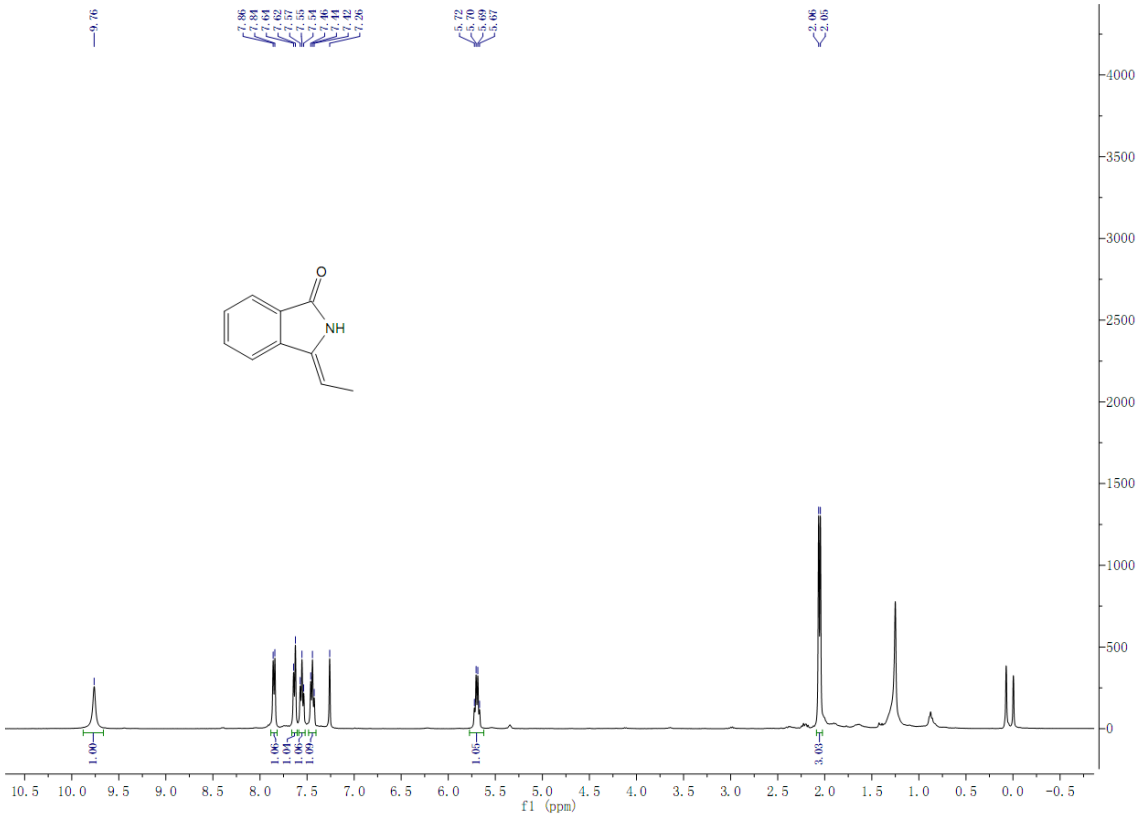
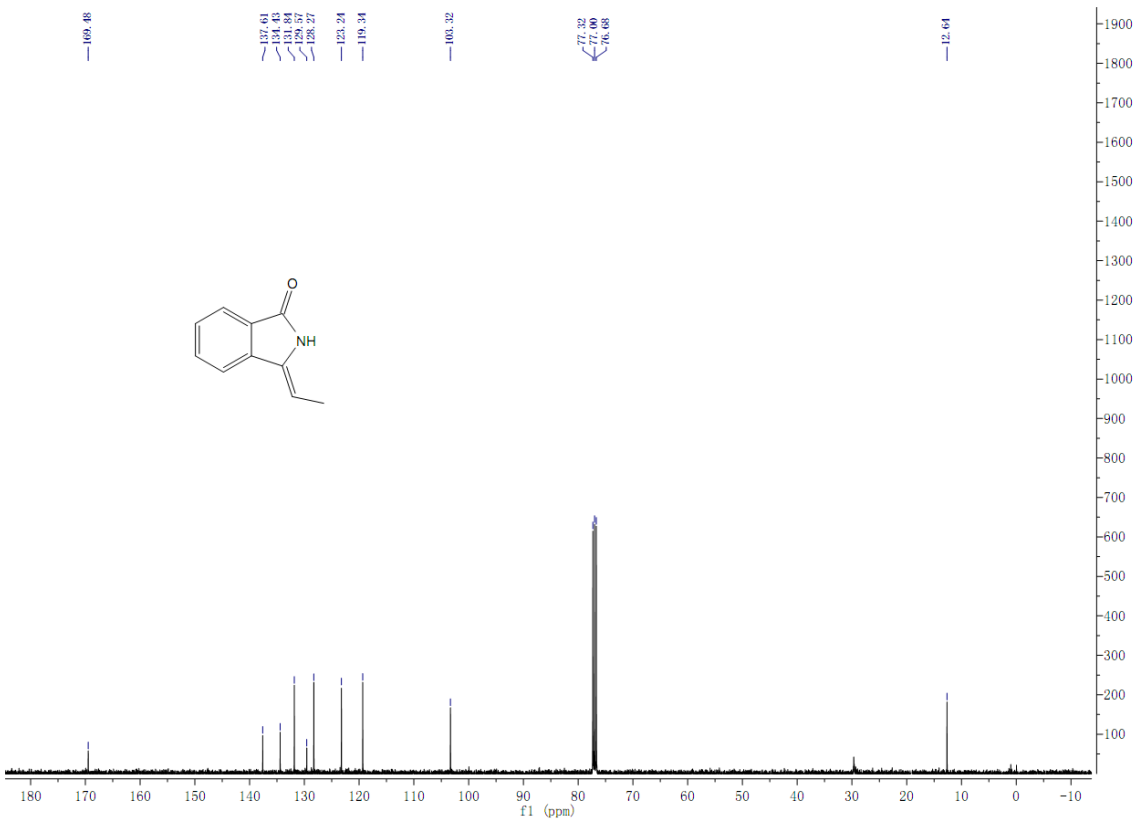
168.79
161.27
135.22
124.71
124.45
119.82
107.81
56.17
f1 (ppm)


7.75
7.73
7.66
7.55
7.53
7.26
2.52
O
O
N
2.00
1.09
1.09
1.09
2.91
f1 (ppm)


168.14
168.02
145.68
134.90
133.02
130.04
124.08
123.50
77.32
77.00
76.68
22.02
O
O
N
f1 (ppm)


9.76
7.86
7.84
7.64
7.62
7.55
7.54
7.46
7.44
7.42
7.26
5.72
5.70
5.69
5.67
2.06
2.05
O
NH
1.00
1.08
1.04
1.06
1.09
1.05
3.03
f1 (ppm)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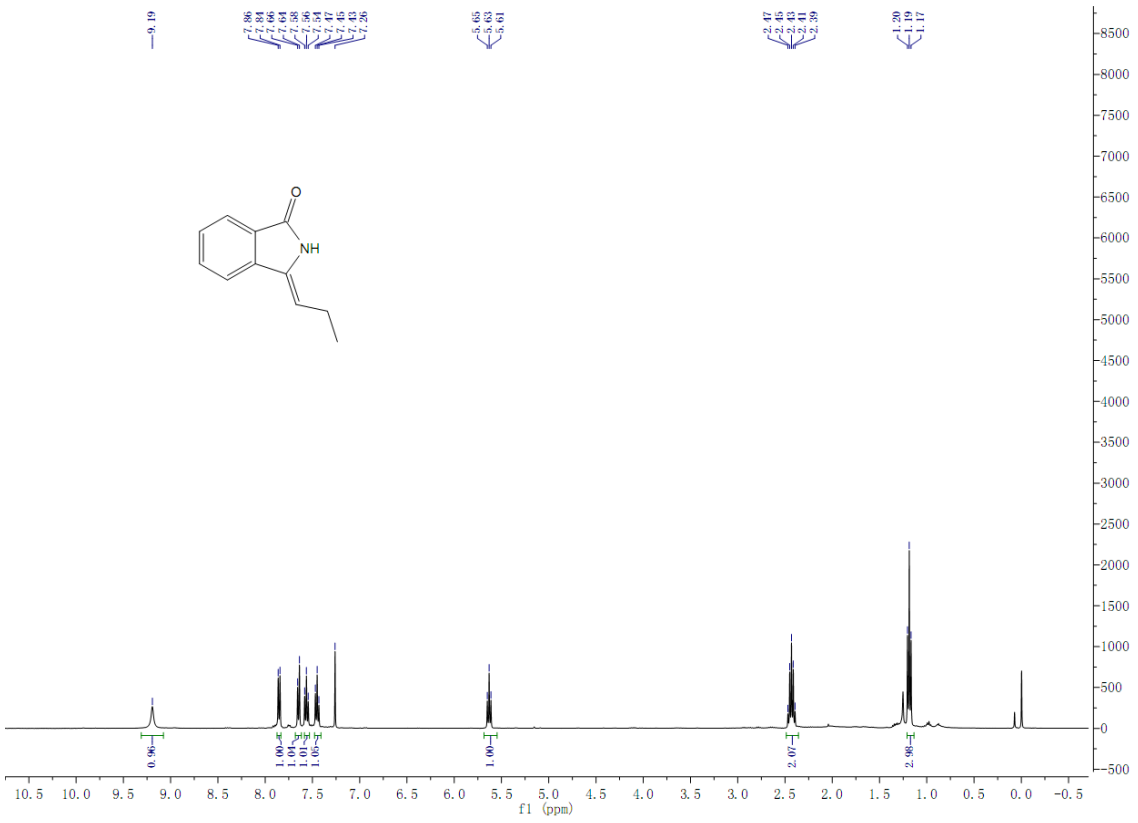
119.34
103.32
77.32
77.00
76.68
12.64
O
NH
f1 (ppm)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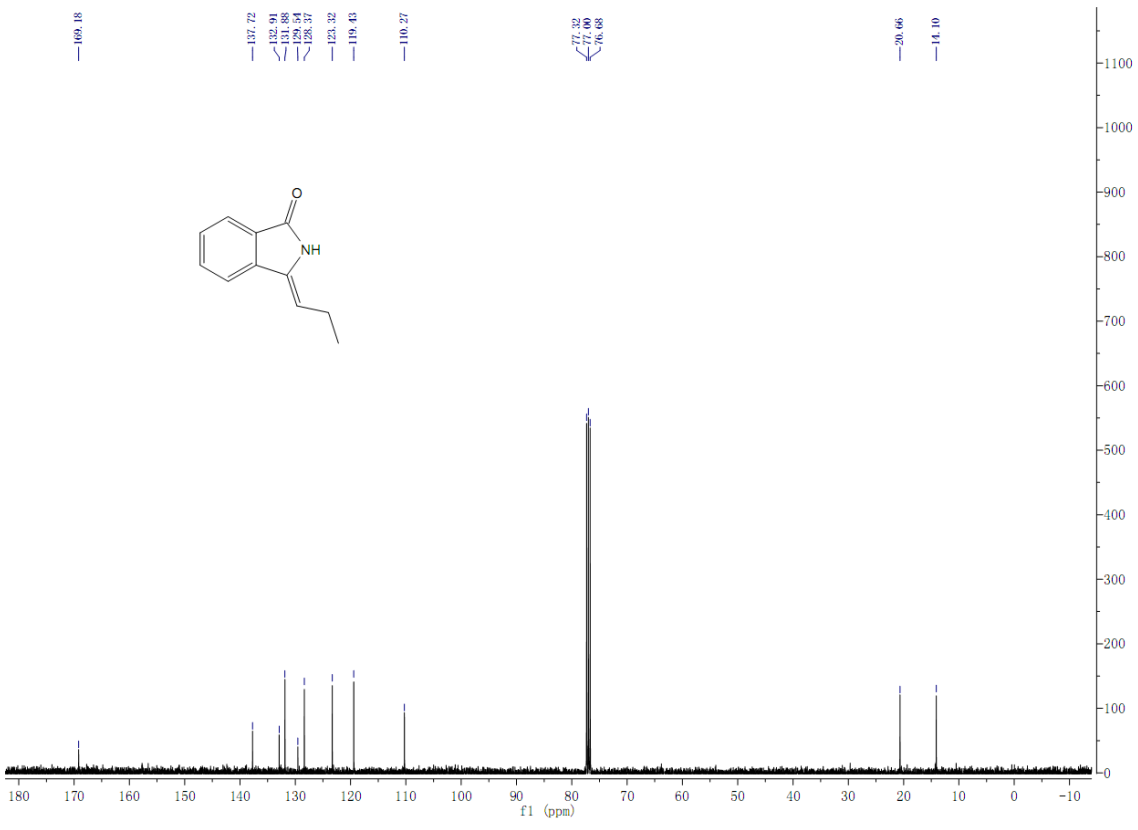
7.64
7.58
7.56
7.54
7.47
7.45
7.43
7.26
5.65
5.63
5.61
2.47
2.45
2.43
2.41
2.39
1.20
1.19
1.17
O
NH
0.96
1.00
1.01
1.05
1.00
2.07
2.98
f1 (ppm)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132.91
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129.54
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119.43
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77.00
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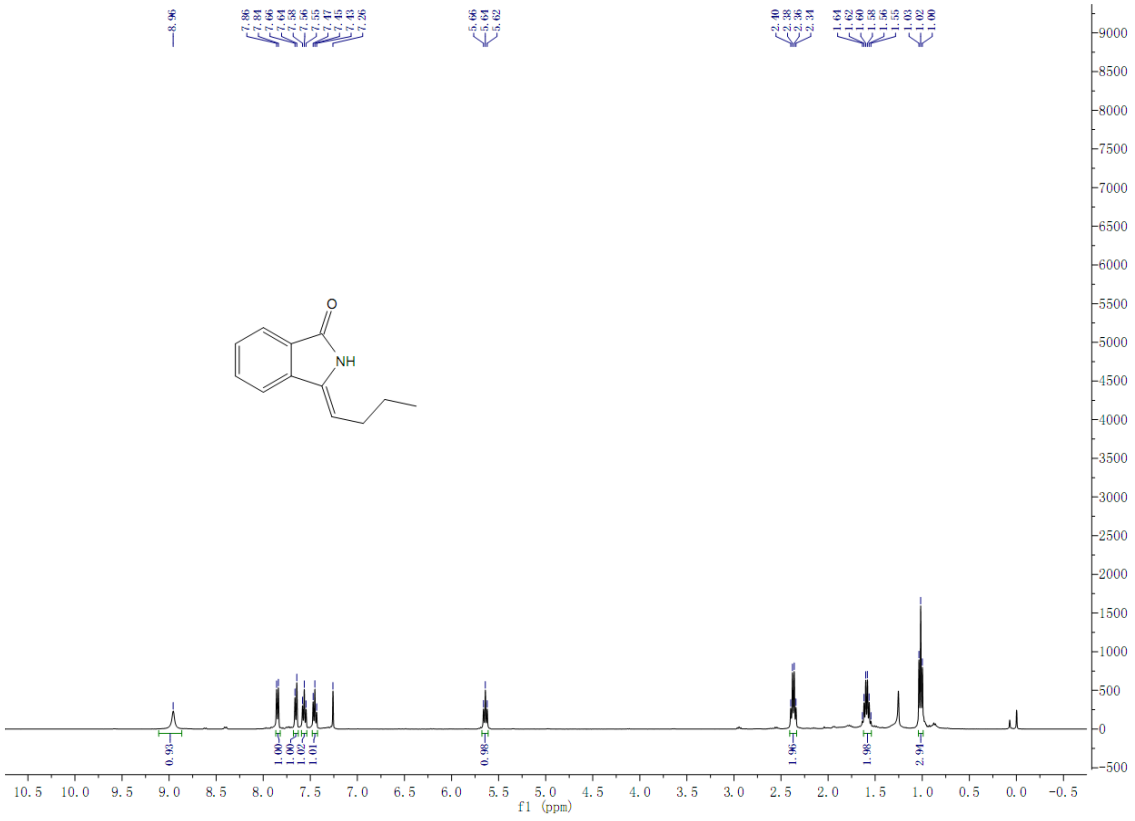
20.66
14.10
O
NH
f1 (ppm)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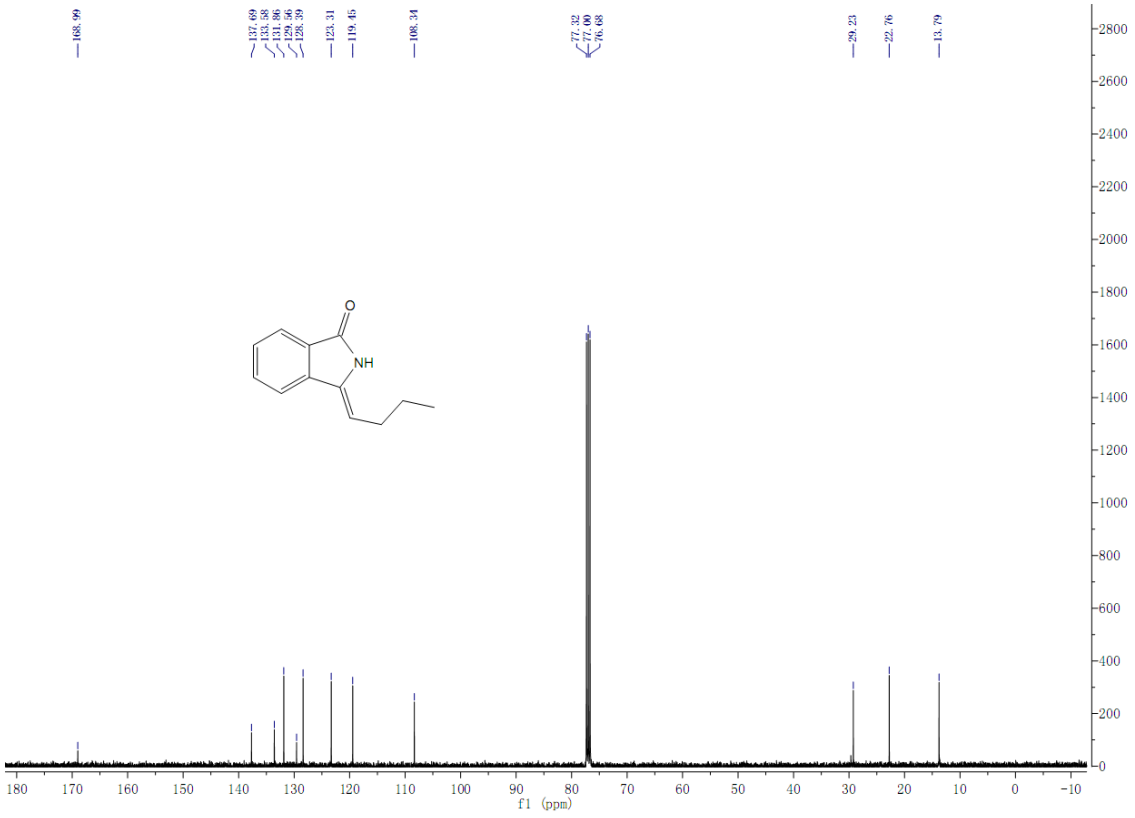
7.86
7.84
7.66
7.64
7.58
7.56
7.55
7.47
7.45
7.43
7.26
5.66
5.64
5.62
2.40
2.38
2.36
2.34
1.64
1.62
1.60
1.58
1.56
1.55
1.03
1.02
1.00
O
NH
0.93
1.00
1.00
1.02
1.01
0.98
1.96
1.98
2.94
f1 (ppm)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129.56
128.39
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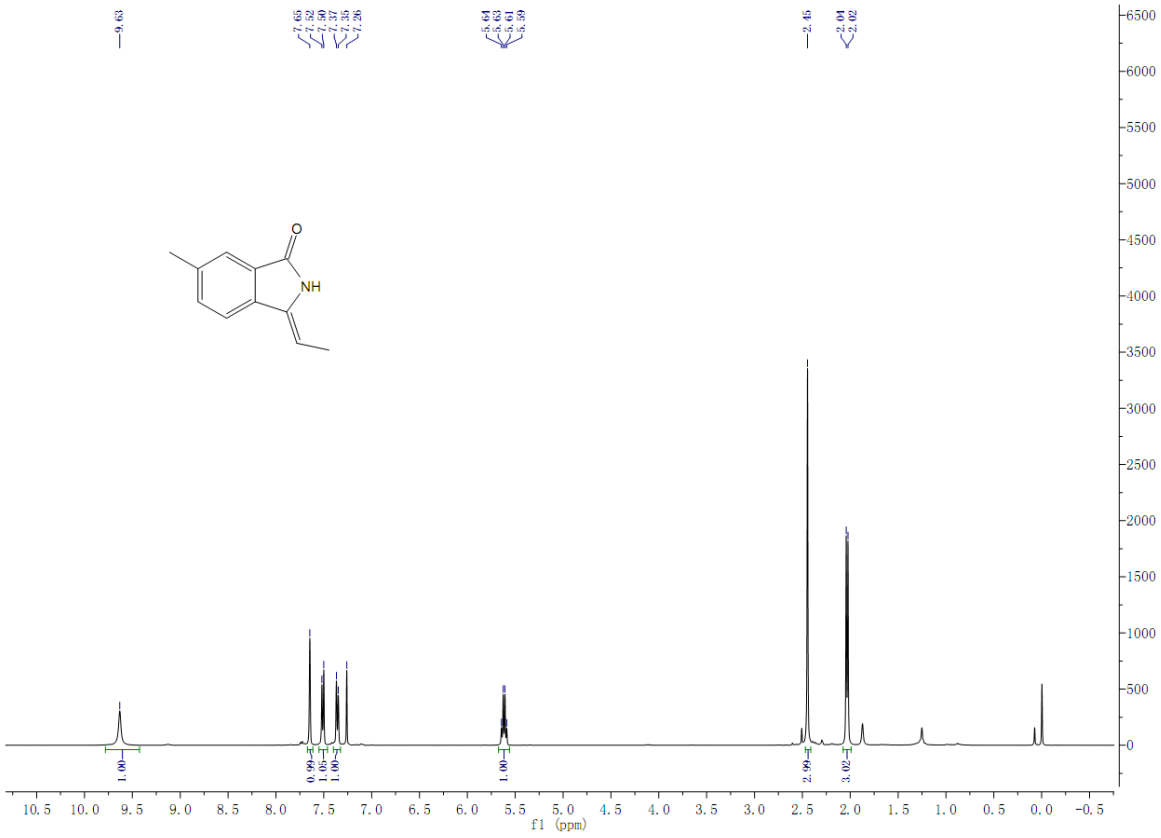
108.34
77.32
77.00
76.68
29.23
22.76
13.79
O
NH
f1 (ppm)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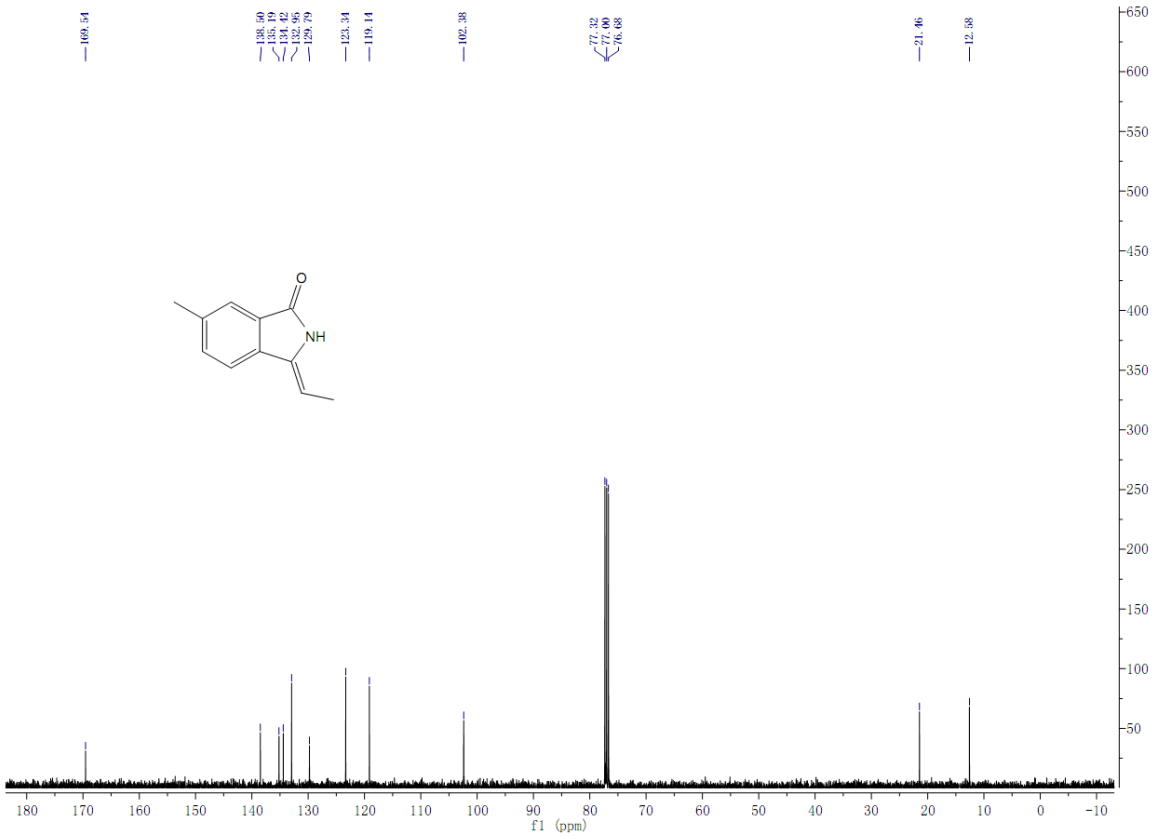
7.65
7.52
7.50
7.37
7.35
7.36
5.64
5.63
5.61
5.59
2.45
2.04
2.02
O
NH
1.00
0.99
1.05
1.00
1.00
2.99
3.02
f1 (ppm)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138.50
135.19
134.42
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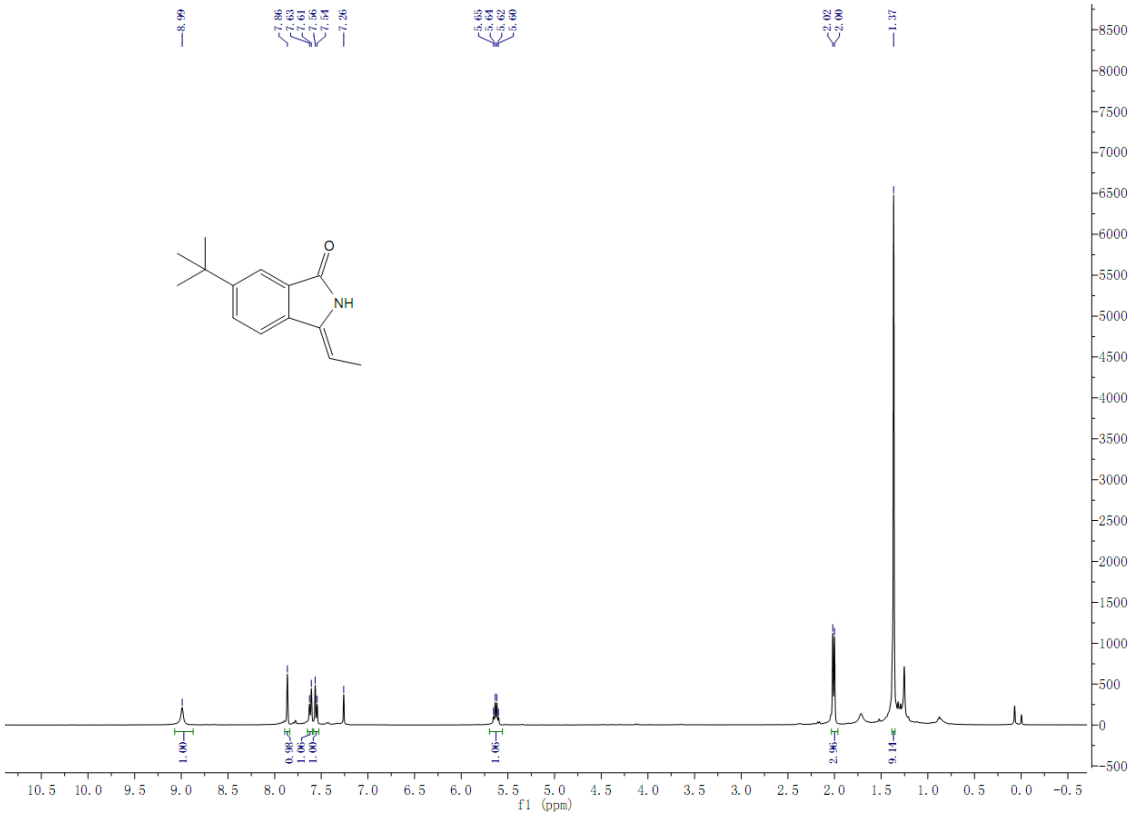
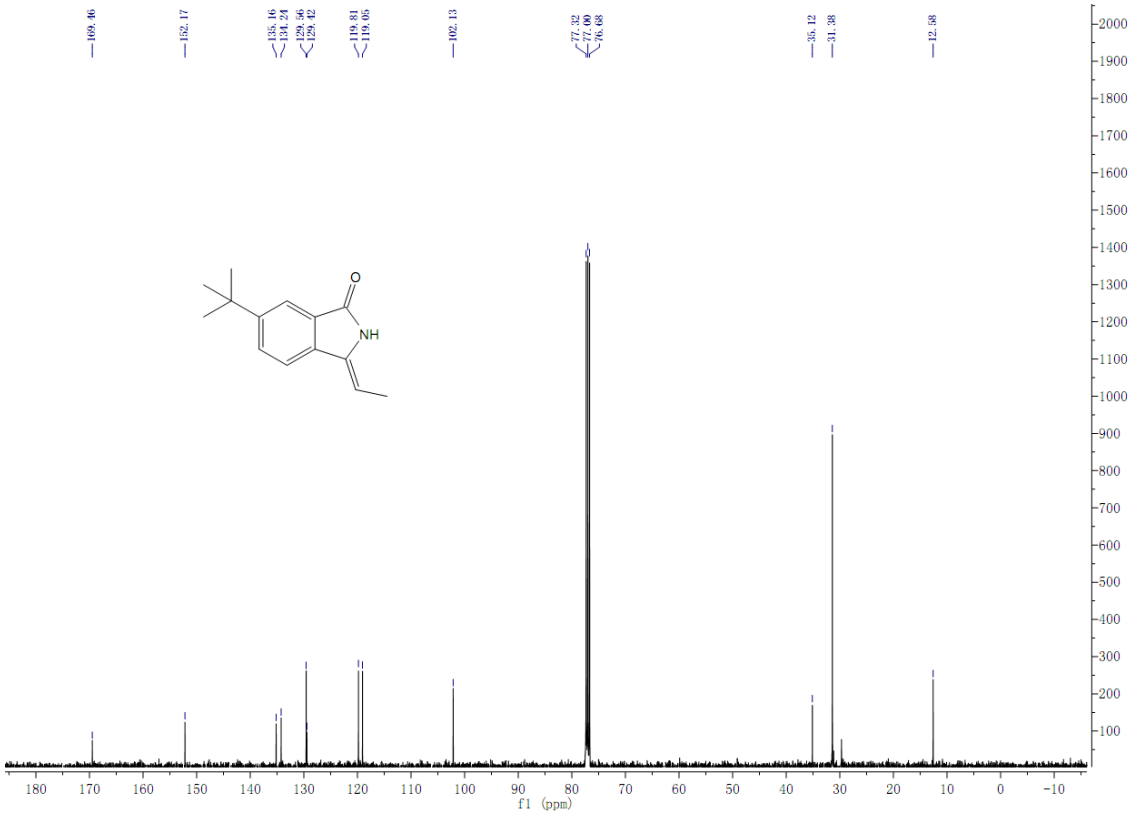
119.14
102.38
77.32
77.00
76.68
21.46
12.58
O
NH
f1 (ppm)


8.99
7.66
7.63
7.61
7.56
7.54
7.26
5.65
5.64
5.62
5.60
2.02
2.00
1.37
O
NH
1.00
0.98
1.06
1.00
1.06
2.96
9.14
f1 (ppm)


169.46
152.17
135.16
134.24
129.56
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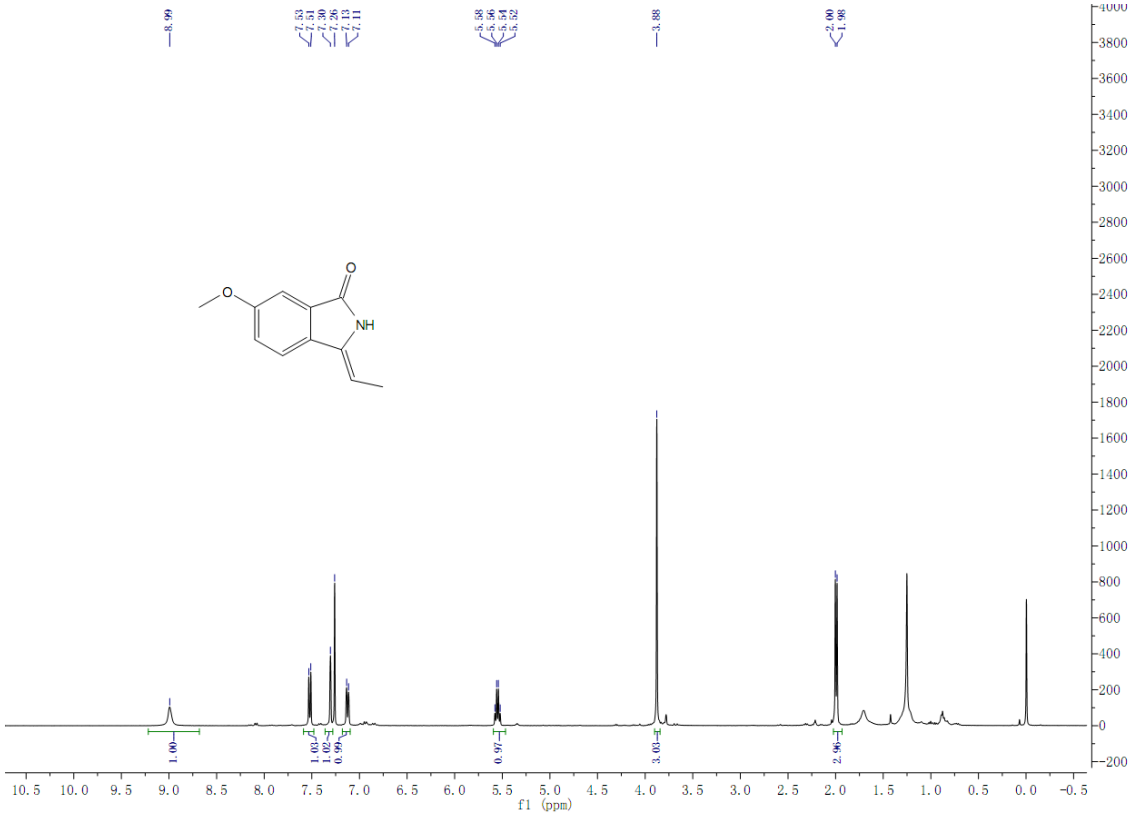
102.13
77.32
77.00
76.68
35.12
31.38
12.58
O
NH
f1 (ppm)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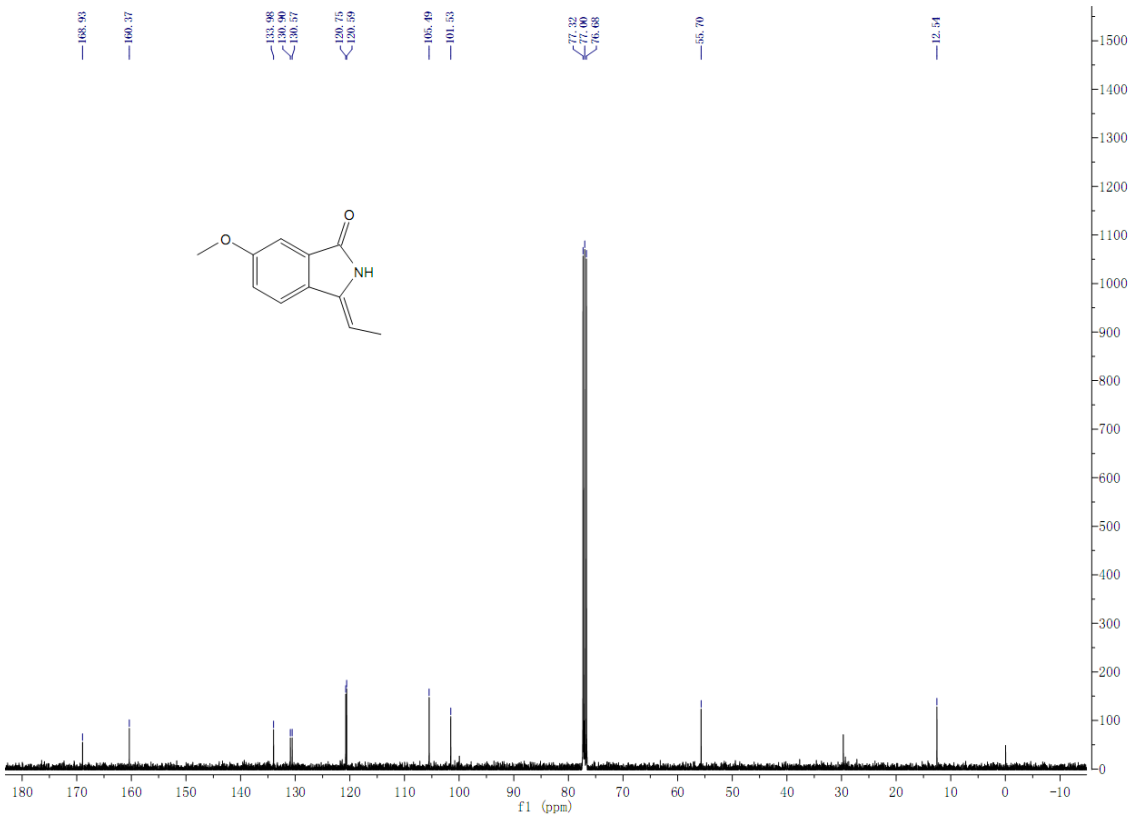
7.53
7.51
7.30
7.26
7.15
7.11
5.58
5.56
5.54
5.52
3.88
2.00
1.98
O
NH
1.00
1.03
1.02
0.99
0.97
3.03
2.96
f1 (ppm)


168.93
160.37
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130.90
130.57
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105.49
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77.00
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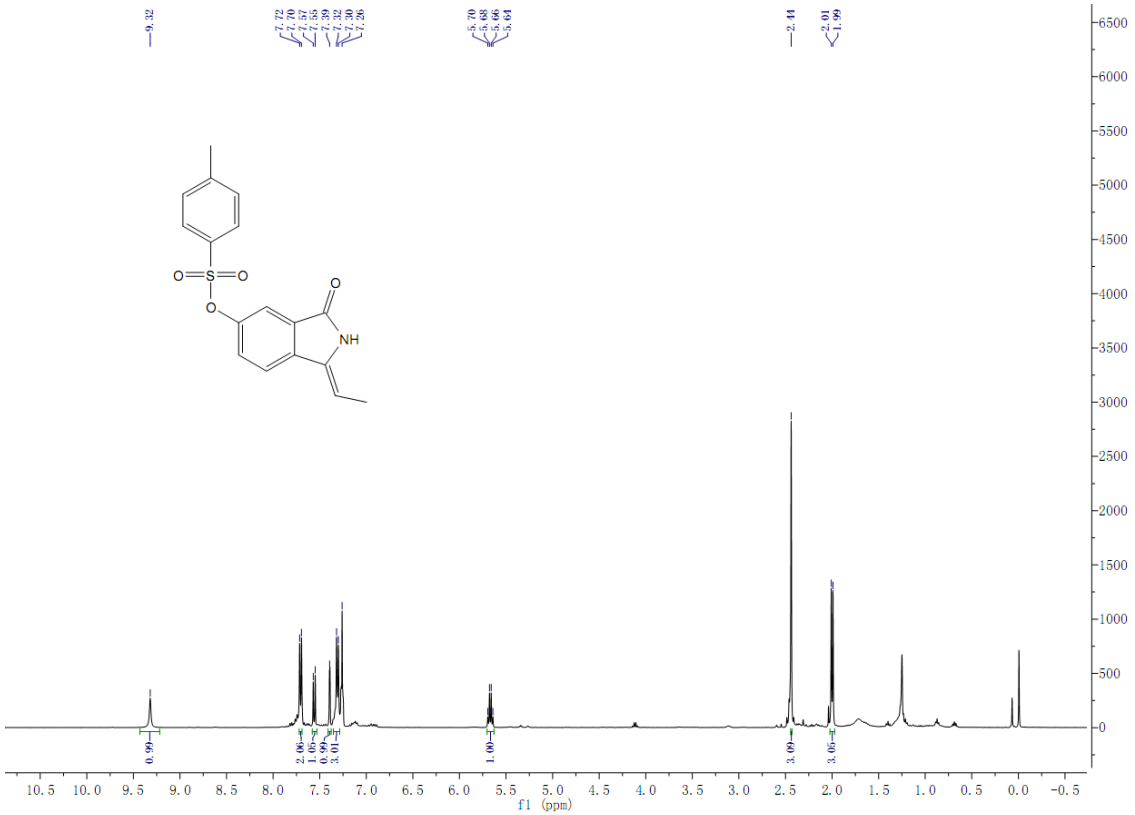
55.70
12.54
O
NH
f1 (ppm)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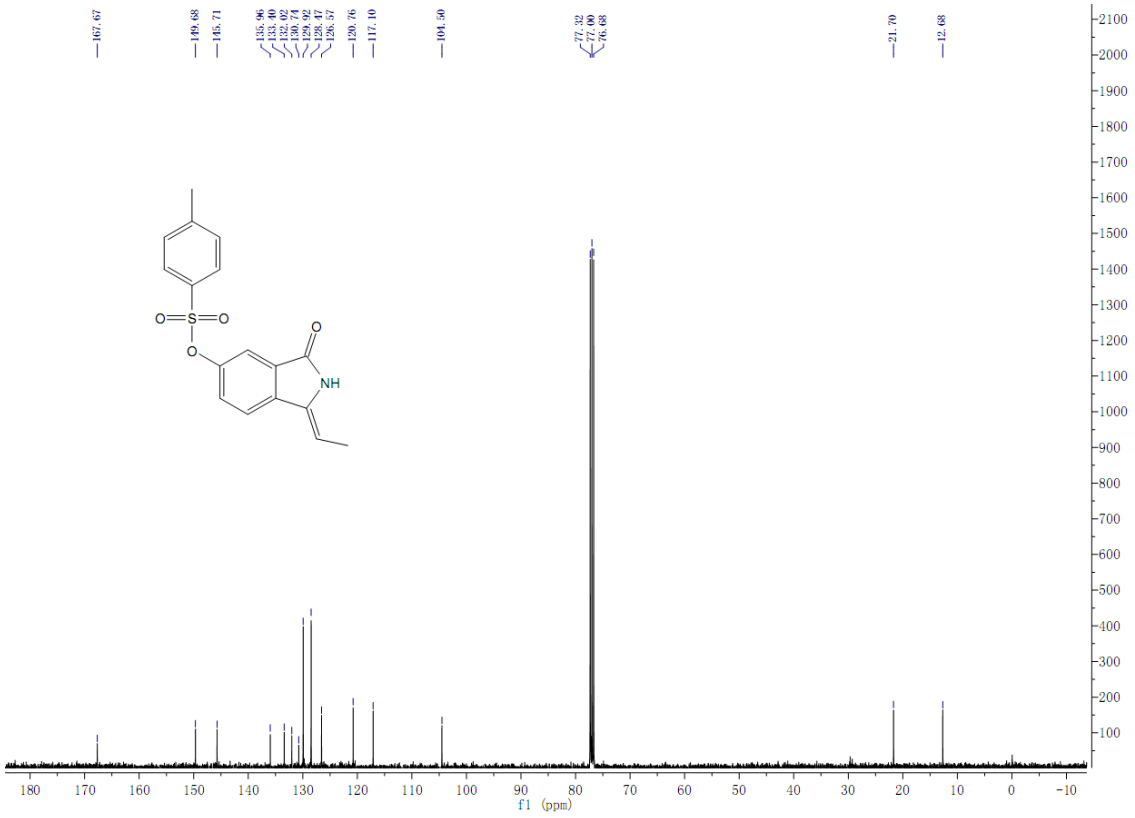
7.70
7.57
7.55
7.39
7.32
7.30
7.26
5.70
5.68
5.66
5.64
2.44
2.01
1.99
O=S=O
O
O
NH
f1 (ppm)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149.68
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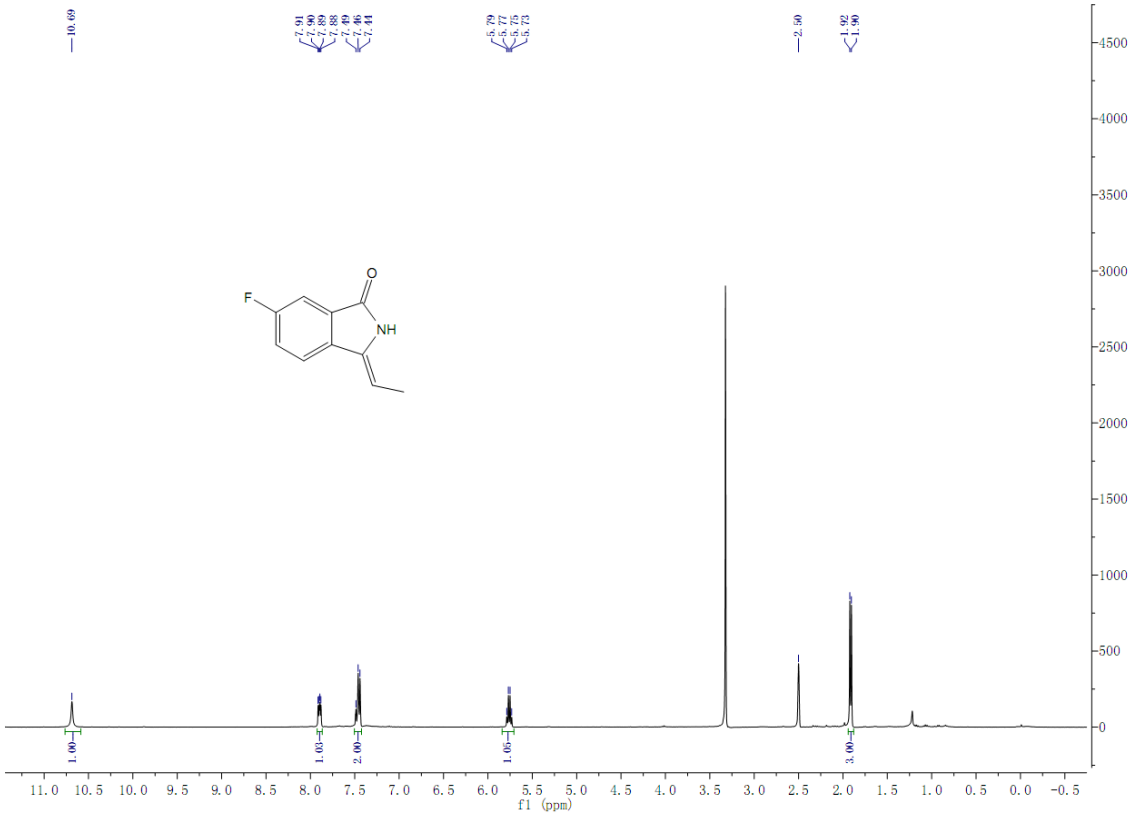
130.74
129.92
128.47
126.57
120.76
117.10
104.50
77.32
77.00
76.68
21.70
12.68
O=S=O
O
O
NH
f1 (ppm)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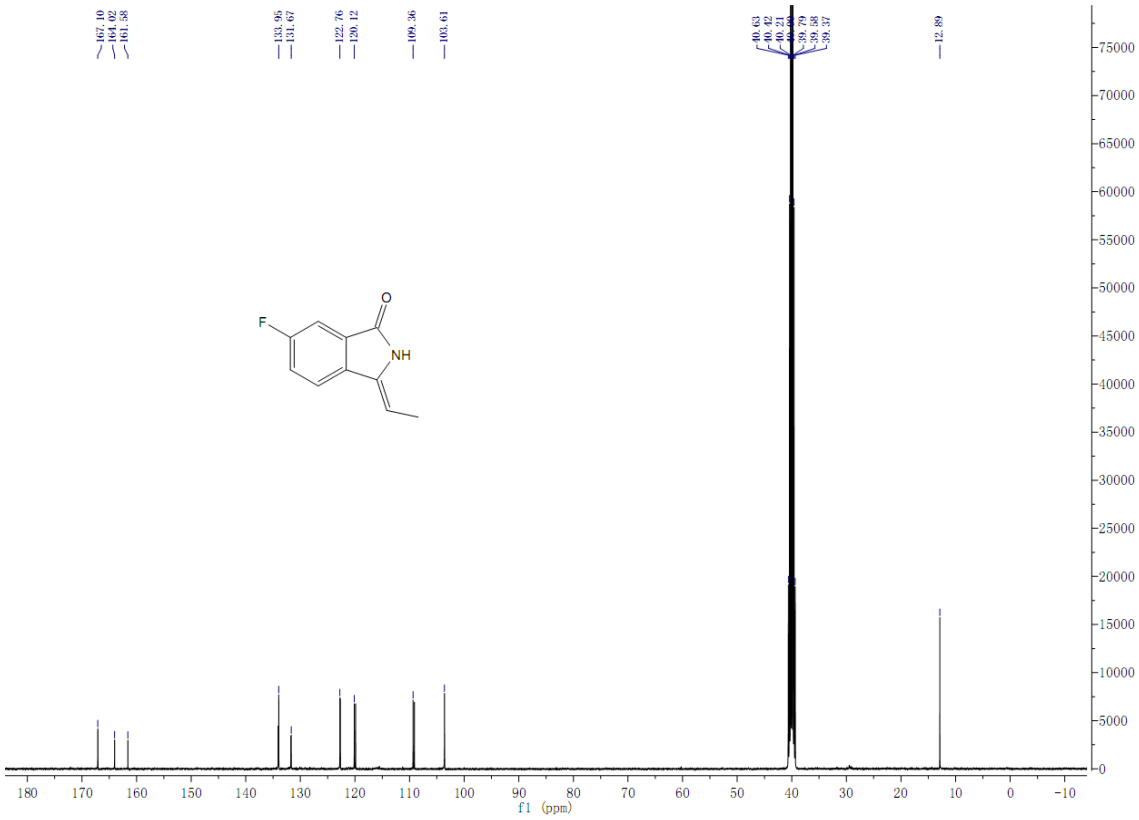
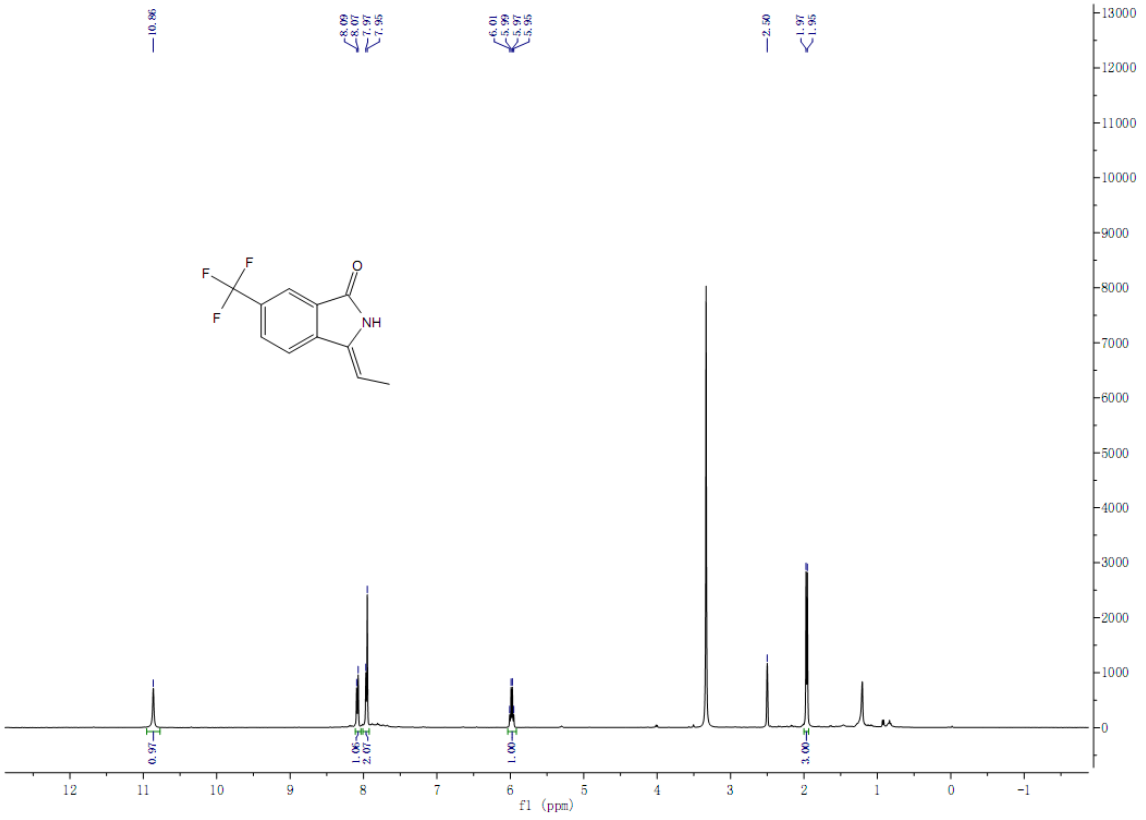
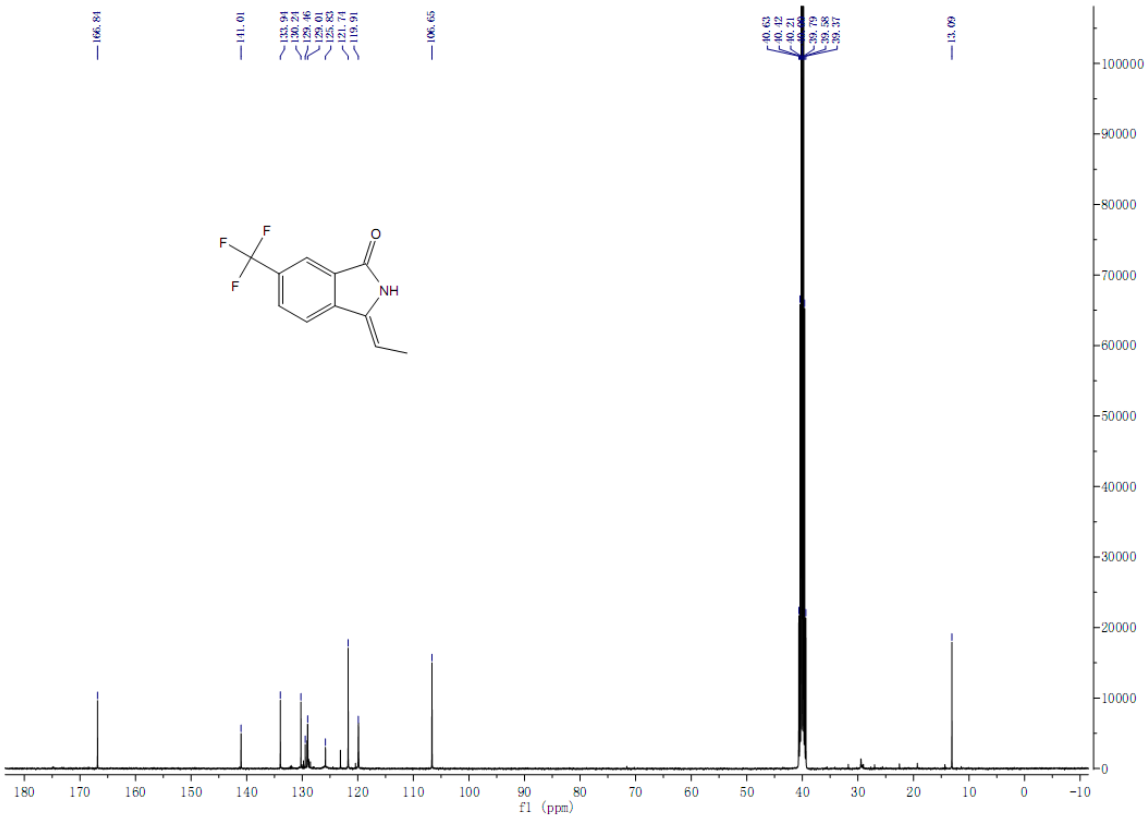
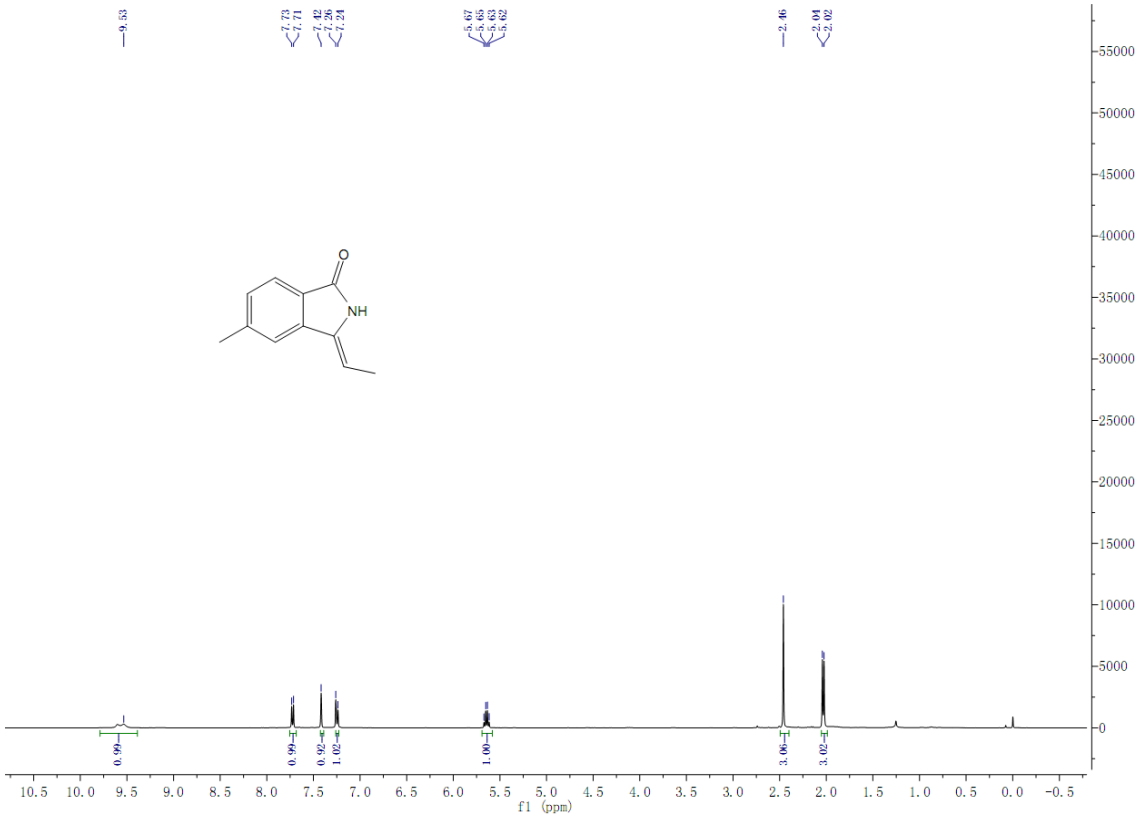
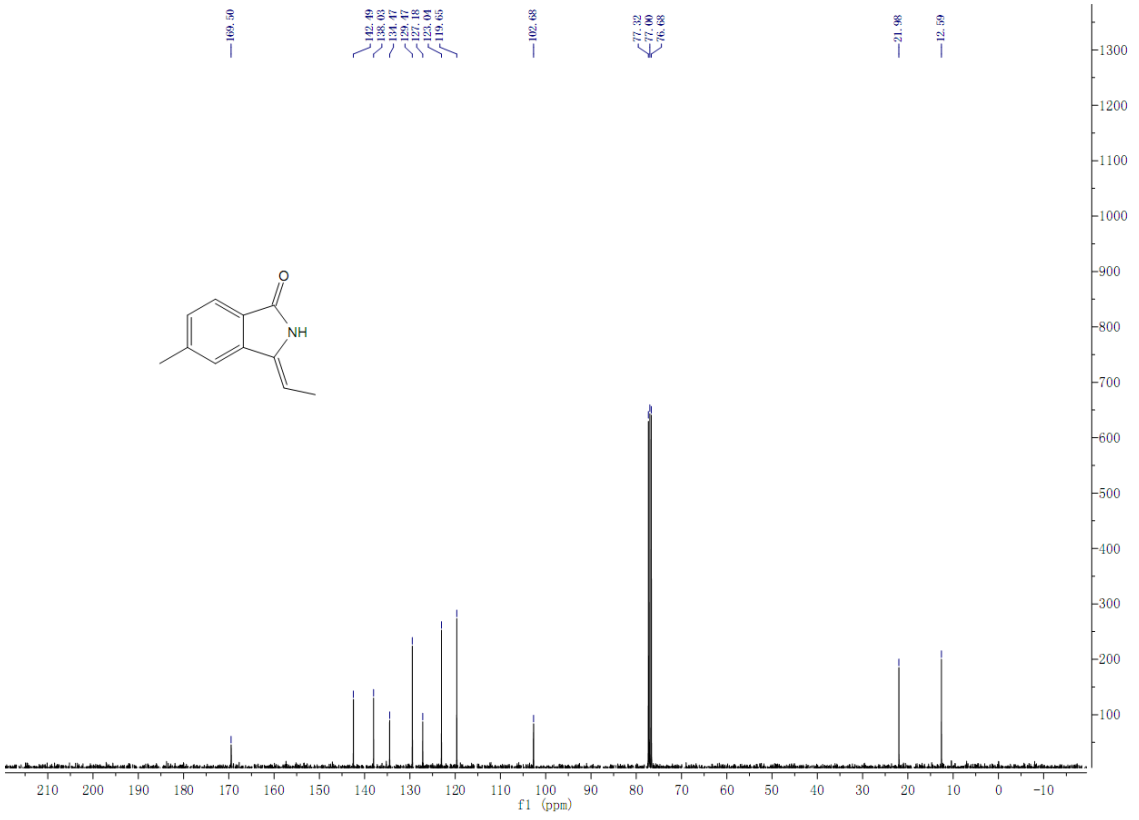
7.46
7.44
5.79
5.77
5.75
5.73
2.50
1.92
1.90
F
O
NH
1.00
1.03
2.00
1.05
3.00
f1 (ppm)


167.10
164.02
161.58
133.95
131.67
122.76
120.12
109.36
103.61
40.63
40.42
40.21
39.79
39.58
39.37
12.89
F
O
NH
f1 (ppm)


10.86
8.09
8.07
7.97
7.95
6.01
5.99
5.97
5.95
2.50
1.97
1.95
F
F
F
O
NH
0.97
1.06
2.07
1.00
3.00
f1 (ppm)


166.84
141.01
133.94
130.21
129.46
128.01
125.83
121.74
119.91
106.65
40.63
40.42
40.21
39.79
39.58
39.37
13.09
F
F
F
O
NH
f1 (ppm)


9.53
7.73
7.71
7.42
7.26
7.24
5.67
5.65
5.63
5.62
2.46
2.04
2.02
O
NH
0.99
0.99
0.92
1.02
1.00
3.06
3.02
f1 (ppm)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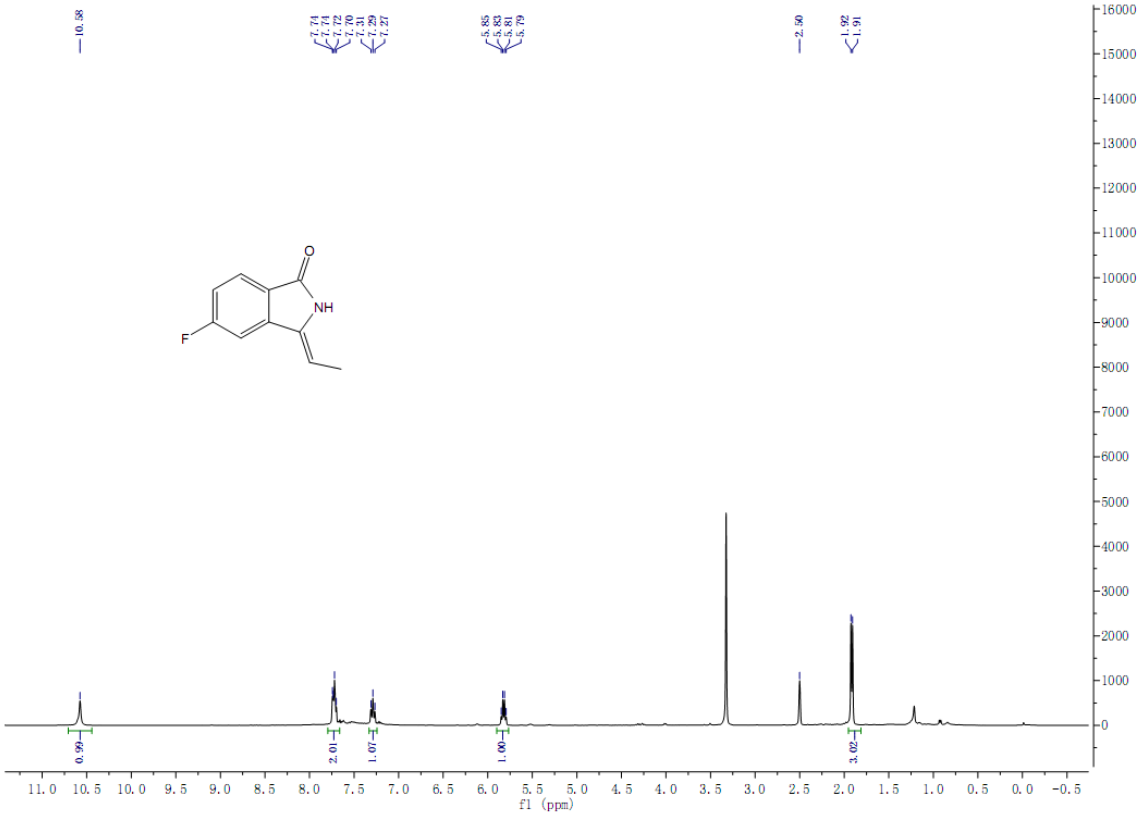
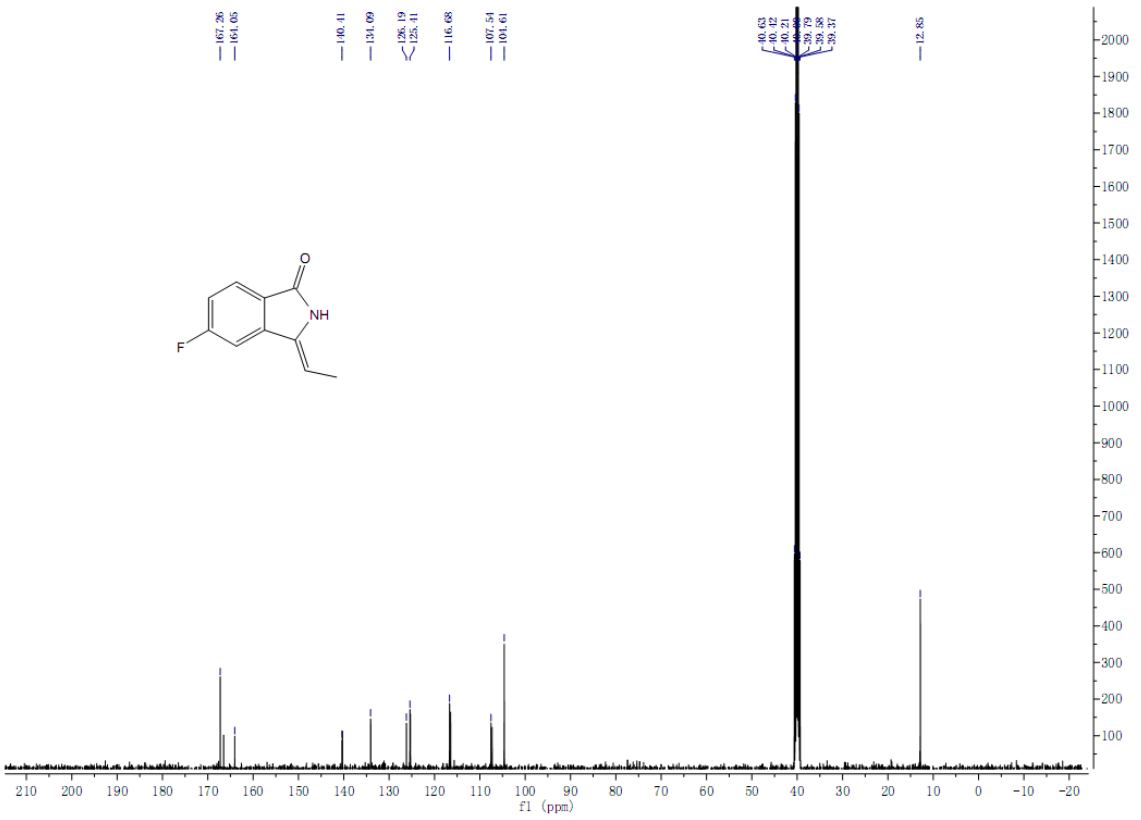
142.49
138.03
134.47
129.47
122.18
123.04
119.65
102.68
77.32
77.00
76.68
21.98
12.59
O
NH
f1 (ppm)


10.58
7.74
7.74
7.72
7.70
7.31
7.29
7.27
5.85
5.83
5.81
5.79
2.50
1.92
1.91
O
NH
F
0.99
2.01
1.07
1.00
3.02
f1 (ppm)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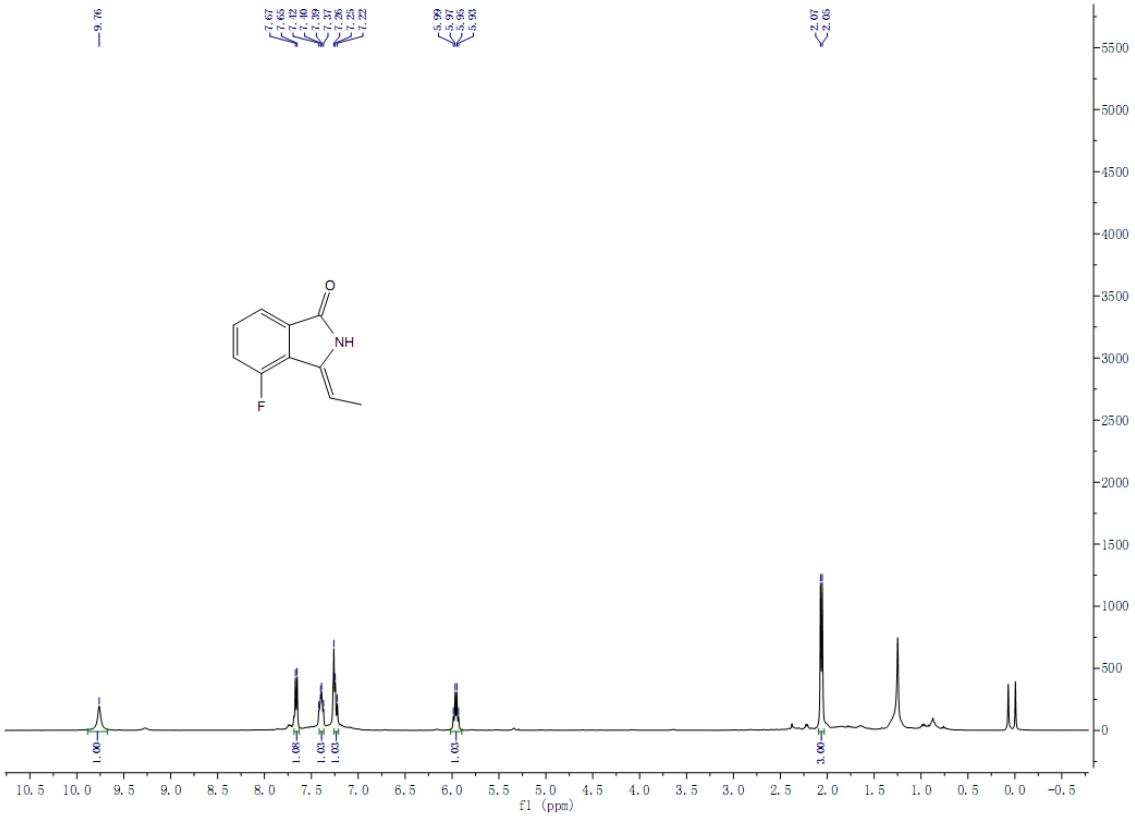
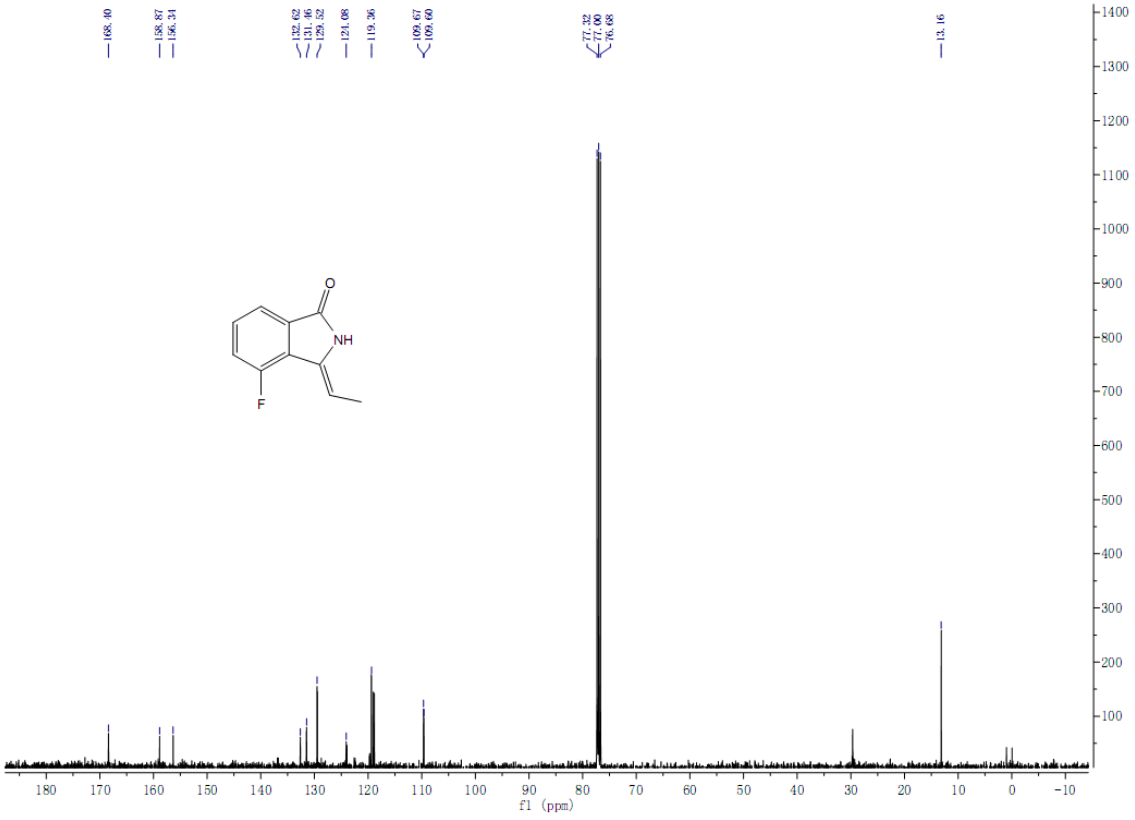
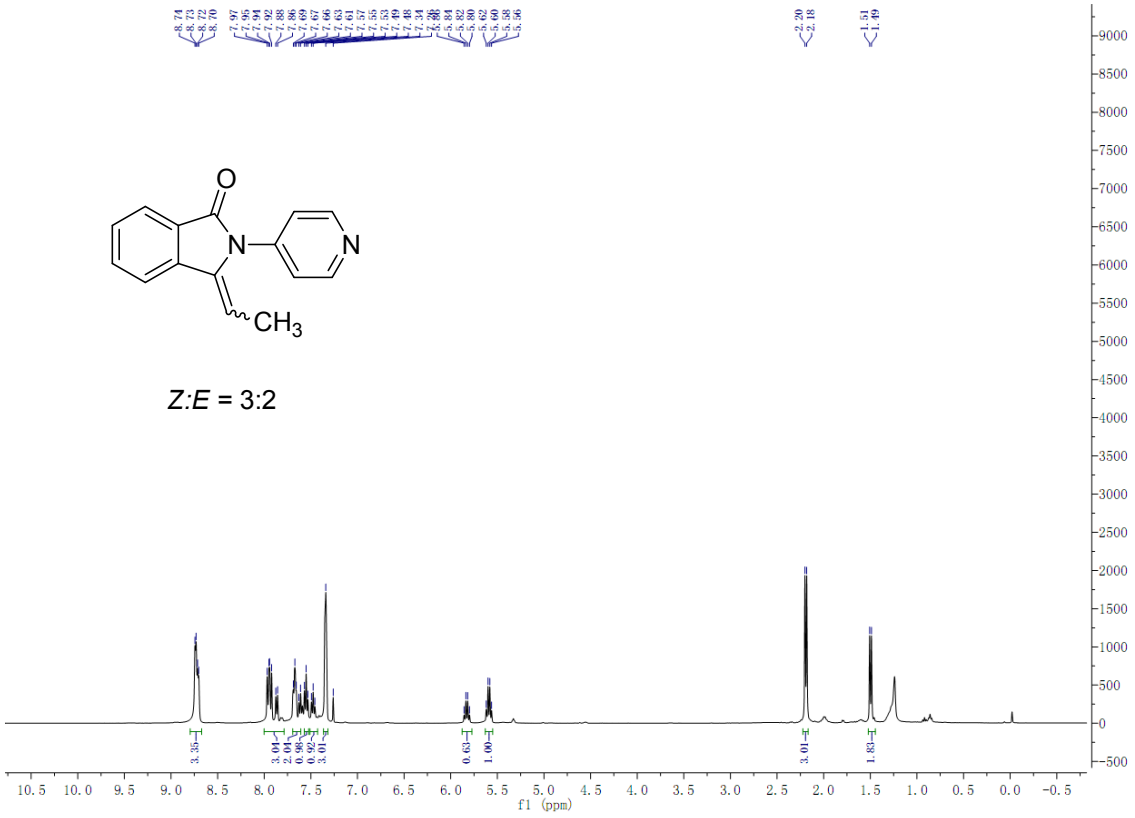
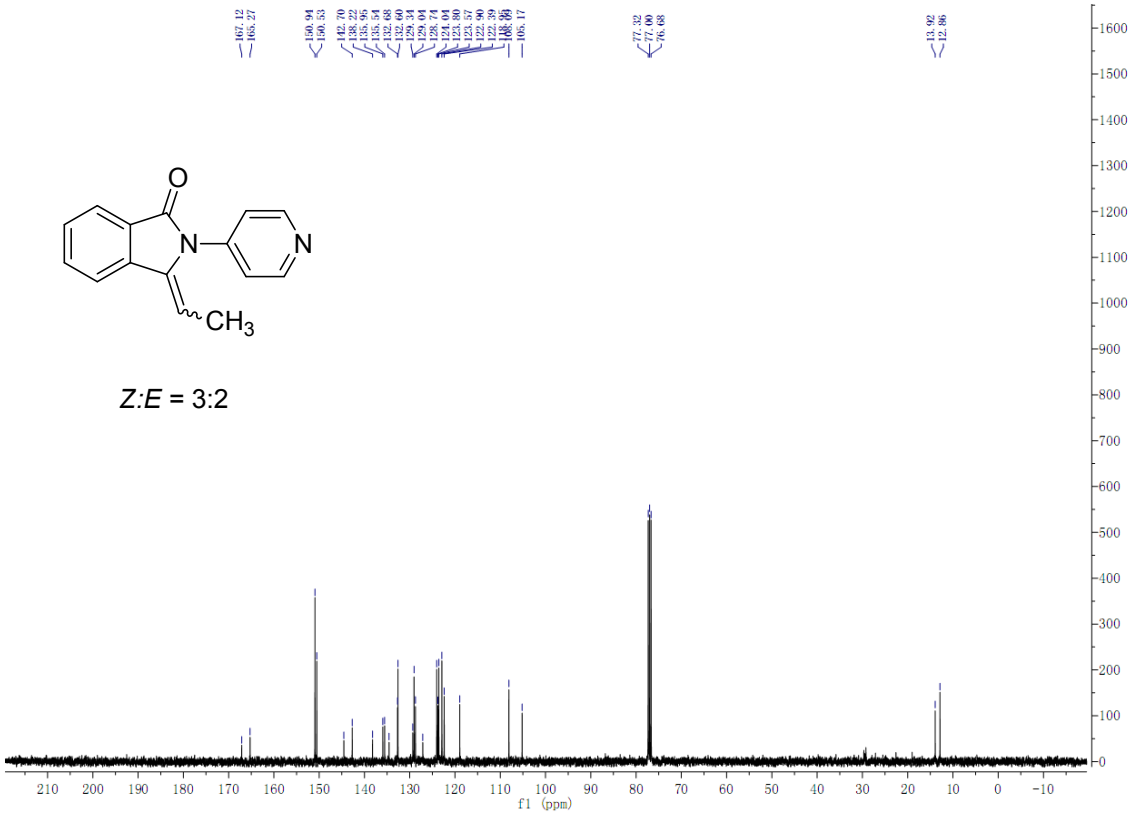
164.05
140.41
134.09
126.19
125.41
116.68
107.54
104.61
40.63
40.42
40.21
40.00
39.79
39.58
39.37
12.85
O
NH
F
f1 (ppm)


9.76
7.67
7.63
7.42
7.40
7.39
7.37
7.26
7.25
7.22
5.99
5.97
5.95
5.93
2.07
2.05
O
NH
F
1.00
1.08
1.03
1.03
1.03
3.00
f1 (ppm)


168.40
158.87
156.34
132.62
131.96
129.52
124.08
119.36
109.67
109.60
77.32
77.00
76.68
13.16
O
NH
F
f1 (ppm)

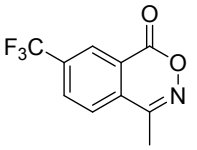


Z:*E* = 3:2



Z:*E* = 3:2

MS Spectra for 2m



2m

Chemical Formula: $C_{10}H_6F_3NO_2$
Molecular Weight: 229.1553

The ^{1}HNMR for 2b/2b'