Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

A Highly Efficient Method Access to Unprotected C-3 Bifunctional Quaternary 3-Allyl-3-(amino)oxindoles

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Supporting Information

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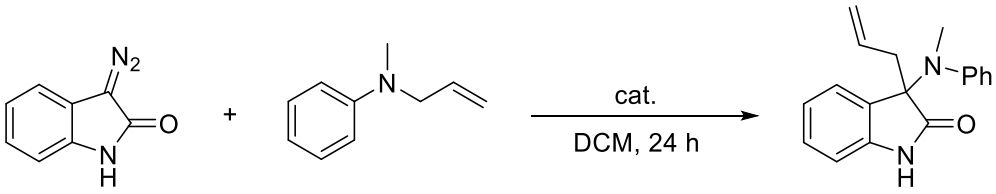
(A) General Information

Chemicals and solvents were purchased from commercial suppliers and used as received unless noted. All products were purified by flash chromatography on silica gel. The chemical yields referred are isolated products. ^{1}H NMR and ^{13}C NMR spectra were recorded on 400 MHz Bruker spectrometers. Chemical shifts of ^{1}H were reported in part per million relative to the $CDCl_3$ residual peak (δ 7.26). Chemical shifts of ^{13}C NMR were reported relative to $CDCl_3$ (δ 77.16). The used abbreviations are as follows: s (singlet), d (doublet), t (triplet), quart. (quartet), quint. (quintet), m (multiplet), br (broad). Multiplets which arise from accidental equality of coupling constants of magnetically non-equivalent protons are marked as virtual (*virt.*). High resolution mass spectra (HRMS) data were measured on a ESI-microTOF Ⅱ. Melting points were measured on a SGW® X-4B and are not corrected. Reactions were monitored by TLC analysis using silica gel 60 Å F-254 thin layer plates and compounds were visualized with a UV light at 254 nm or 365 nm. Further visualization was achieved by staining with iodine, or $KMnO_4$ followed by heating on a hot plate. Flash column chromatography was performed on silica gel 60 Å, 10–40 μm.

The catalyst $Rh_2(esp)_2$ was prepared following the literature procedure.[1]

(B) Reaction Condition Optimization

B-1 Screening of catalysts and ligands



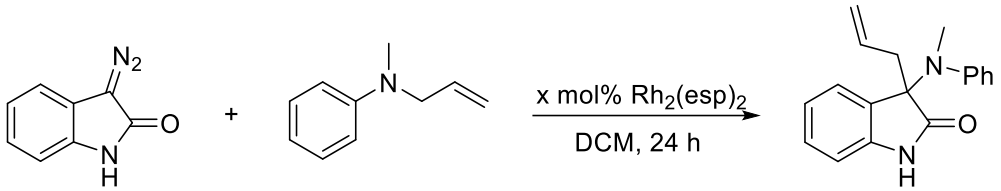
1a **2a** **3a**

Entry [a]	Catalyst (10 mol%)	Solvent	T (℃)	Yield(%) [b]
1	I_2	DCM	r.t.	0
2	$Fe(acac)_2$	DCM	r.t.	0
3 [c]	$Fe(acac)_2$ +1,10-Phen	DCM	r.t.	0
4	$Cu(OAc)_2$	DCM	r.t.	27
5 [d]	$Cu(OAc)_2$ +1,10-Phen	DCM	r.t.	0
6	$Cu(acac)_2$	DCM	r.t.	0
7	$Cu(OTf)_2$	DCM	r.t.	29
8 [e]	$Cu(OTf)_2$ +1,10-Phen	DCM	r.t.	0
9	$Cu_2(OAc)_4$	DCM	r.t.	19
10	Hemin	DCM	r.t.	<5
11	FePcCl	DCM	r.t.	0
12 [f]	$Rh_2(Ph_3CCOO)_4$	DCM	r.t.	50

13 [f]	$Rh_2(OAc)_4$	DCM	r.t.	60
14 [f]	$Rh_2(esp)_2$	DCM	r.t.	78
15 [g]	$Rh_2(esp)_2$	DCM	r.t.	76
14 [h]	$Rh_2(esp)_2$	DCM	r.t.	69

a. Reaction conditions: 0.2 mmol **1a**, 0.2 mmol **2a** and 10 mol% catalyst in DCM (2.0 mL) at room temperature was stirred for 24 h. *b*. Isolated yield. *c*. 10 mol% $Cu_2(OAc)_4$ and 12 mol% 1,10-phen were used. *d*. 10 mol% $Cu(OAc)_2$ and 12 mol% 1,10-phen were used. *e*. 10 mol% $Cu(OTf)_2$ and 12 mol% 1,10-phen were used. *f*. 5 mol% $Rh_2(esp)_2$ was used. *g*. 1 mol% $Rh_2(esp)_2$ was used. *h*. 0.5 mol% $Rh_2(esp)_2$ was used.

B-2 Screening of catalyst loading

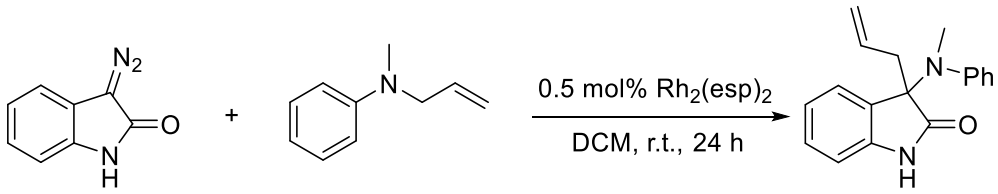


1a **2a** **3a**

Entry [a]	x mol%	Solvent	T (℃)	Yield(%) [b]
1	5.0	DCM	r.t.	78
2	1.0	DCM	r.t.	76
3	0.5	DCM	r.t.	69
4	0.2	DCM	r.t.	20
5 [c]	0.5	DCM	r.t.	71

a. Reaction conditions: 0.2 mmol **1a**, 1.2 equiv. **2a** and x mol% $Rh_2(esp)_2$ in DCM (2.0 mL) at room temperature was stirred for 24 h. *b*. Isolated yield. *c*. 0.2 mmol **1a**, 1.2 eq. **2a** used.

B-3 Screening of solvents



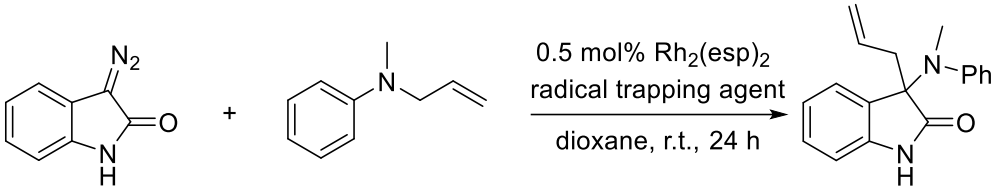
1a **2a** **3a**

Entry [a]	Catalyst (0.5 mol%)	Solvent	T (℃)	Yield(%) [b]
1	$Rh_2(esp)_2$	DCM	r.t.	73
2	$Rh_2(esp)_2$	DMF	r.t.	21
3	$Rh_2(esp)_2$	H_2O	r.t.	10
4	$Rh_2(esp)_2$	THF	r.t.	82
5	$Rh_2(esp)_2$	Toluene	r.t.	80
6	$Rh_2(esp)_2$	$PhCF_3$	r.t.	85
7	$Rh_2(esp)_2$	DCE	r.t.	77
8	$Rh_2(esp)_2$	neat	r.t.	5
9	$Rh_2(esp)_2$	1,4-dixoane	r.t.	90

a. Reaction conditions: 0.2 mmol **1a** and 0.5 mol% $Rh_2(esp)_2$ in solvent (2.0 mL) at room temperature, then **2a** (1.2 equiv.) was added in three portions over 30 min, and the mixture was stirred for 24 h. *b*. Isolated yield.

(C) Mechanistic studies

Mechanistic experiments – addition of radical trapping agents (TEMPO and BHT)



1a **2a** **3a**

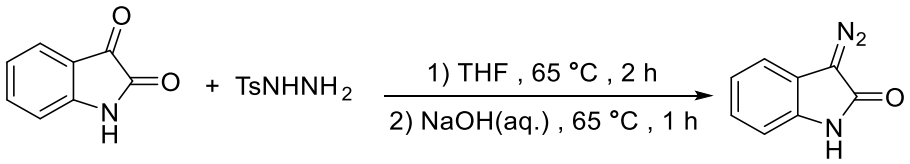
Entry [a]	conditions	Yield(%) [b]
1	**No trapping agent**	90
2 [c]	1.0 eq. TEMPO	85
3 [d]	1.0 eq. BHT	88

a. Reaction conditions: 0.2 mmol **1a**, 0.24 mmol **2a** and 0.5 mol% $Rh_2(esp)_2$ in dioxane (2.0 mL) at room temperature was stirred for 24 h. *b*. Assay of intact 1,3,5-trimethoxybenzene as a function of the NMR yield of **3a**. *c*. 0.2 mmol **1a**, 0.2 mmol **2a**, 1.0 eq. TEMPO and 0.5 mol% $Rh_2(esp)_2$ in dioxane (2.0 mL) at room temperature was stirred for 24 h. *d*. 0.2 mmol **1a**, 0.2 mmol **2a**, 1.0 eq. BHT and 0.5 mol% $Rh_2(esp)_2$ in dioxane (2.0 mL) at room temperature was stirred for 24 h.

(D) General synthetic procedures for preparation of 1 and 2

D-1. Procedures for preparation of diazo compounds

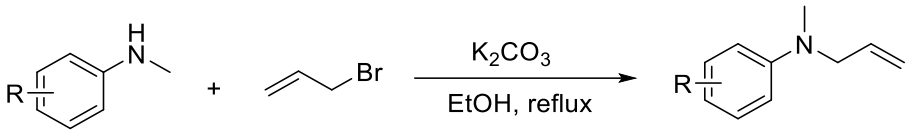
Diazo compounds were prepared via diazo transfer with $TsNHNH_2$ according to the literature procedure. [2]



A mixture of isatin (10 mmol) and $TsNHNH_2$ (1.1 eq.) in THF (50 mL) was stirred at 65 ℃ for 2 h, then cooled at room temperature. The mixture solvent was concentrated under reduced pressure, and the pure tosylhydrazone was precipitated from MeOH (30 mL) solution. Then tosylhydrazone was dissolved in aq. NaOH (0.2 N) solution, and the mixture was stirred at 65 ℃ for 2 h. Water (30 mL) and EtOAc (30 mL) were added and the organic layer was separated. The collected organic layers were washed with brine (10 mL), dried over Na_2SO_4, and concentrated under reduced pressure to give the crude products without further purification. The observed characterization data are consistent with those previously reported.[2]

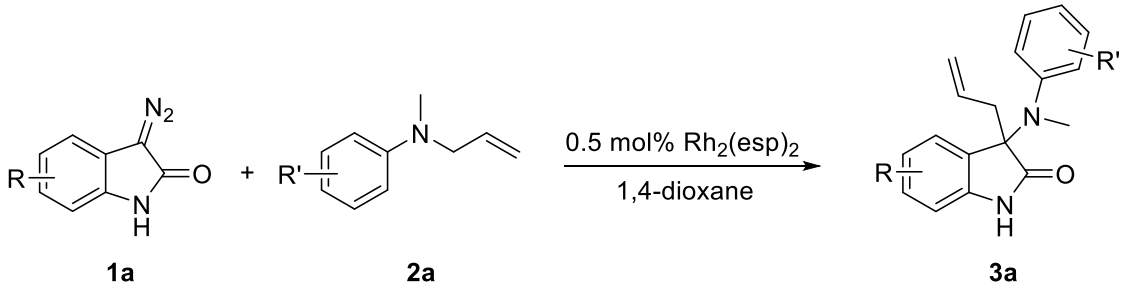
D-2. Procedures for preparation of N-allyl-N-methylanilines

Those compounds were prepared according to the literature procedure.[3]



50 mL flask equipped with a stir-bar was charged with substituted anilines (1.0 equiv., 5 mmol) and K_2CO_3 (1.5 equiv., 7.5 mmol). 20 mL of EtOH was added to the flask and the solution was stirred under room temperature. Followed alkyl bromides (1.2 equiv.) was added. The reaction mixture was stirred at reflux and monitored by TLC. Upon completion the reaction mixture was quenched by water (20 mL) and extracted by ethyl acetate (3×30 mL). Combined organic phase were dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The residue was then purified by chromatography on silica gel with a mixture eluent of petroleum ether, ethyl acetate. The observed characterization data are consistent with those previously reported.[3]

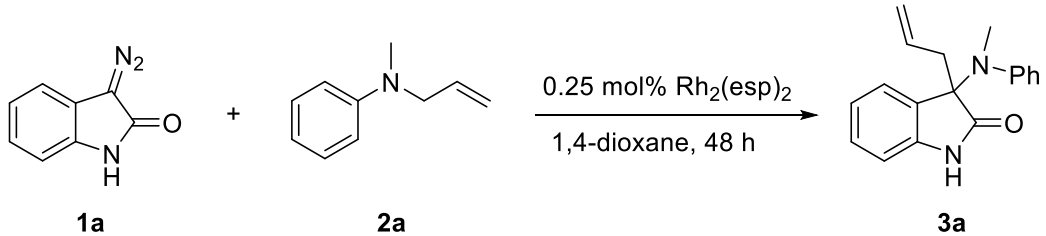
(E) General procedure for [2,3]-sigmatropic rearrangement reactions



1a **2a** **3a**

N-allyl-*N*-methylanilines (0.24 mmol, 1.2 eq.) was added into 2.0 mL 1,4-dioxane solution of 0.5 mol% $Rh_2(esp)_2$ (0.8 mg, 1.0 μmol), followed by adding 0.2 mmol diazo reagent in three portions over 30 min. Then the reaction vial was stirred for 24 hours under room temperature. After completing the reaction confirmed by TLC, the mixture was subjected to a short silica gel column directly using PE/EA as elute to give the corresponding product.

(F) Scale-up synthesis of 3,3-disubstituted oxindoles

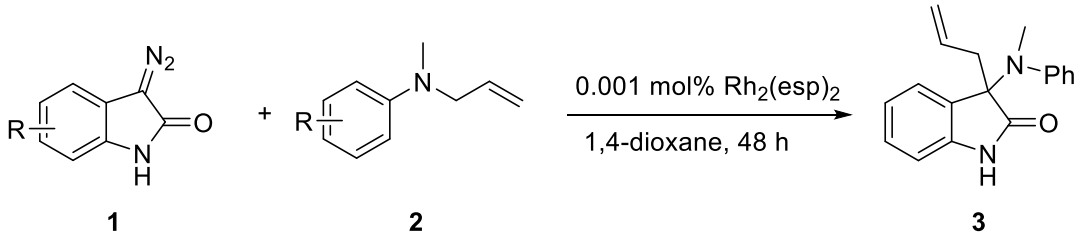


1a **2a** **3a**

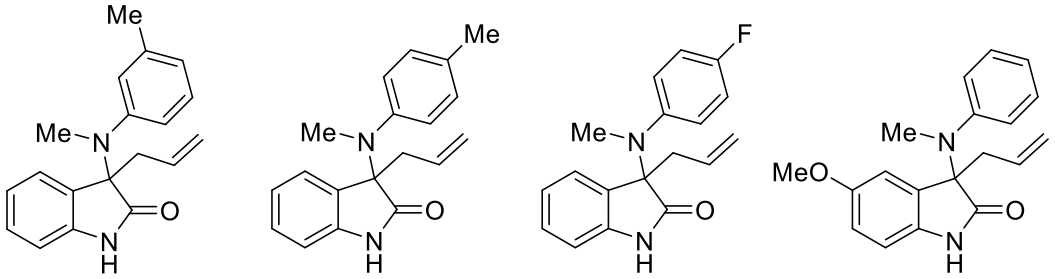
A 100 mL round-bottomed flask was charged with a stir bar. *N*-allyl-*N*-methylaniline (12 mmol, 1.77 g, 1.2 eq.) was added into 50 mL 1,4-dioxane solution of 0.25 mol% $Rh_2(esp)_2$ (19 mg, 0.025 mmol), followed by adding 3-diazooxindole (10 mmol, 1.59 g) in five portions over 30 min. Then

the reaction vial was stirred for 48 hours under 30 ℃. After completing the reaction confirmed by TLC, the organic layer was further concentrated *in vacuo*. The mixture was subjected to silica gel, and eluted by petroleum ether to give the desired product 2.23g, the yield up to 80%.

(G) Reactions with low catalyst loading for TON calculation



1 **2** **3**



TON: 77000 TON: 87000 TON: 90000 TON: 85000

S-1: Preparation of low concentration $Rh_2(esp)_2$ solution

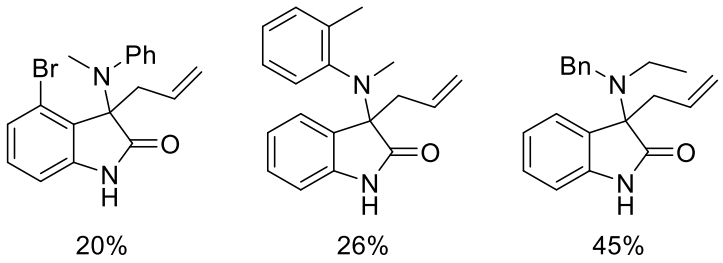
8.0 mg $Rh_2(esp)_2$ and 30 mL EtOAc were added into a 50 mL round bottom flask, then the flask was sealed with rubber stopper. The flask was sonicated for 10 minutes until the catalyst was completely dissolved.

S-2: The procedure of TON experiments

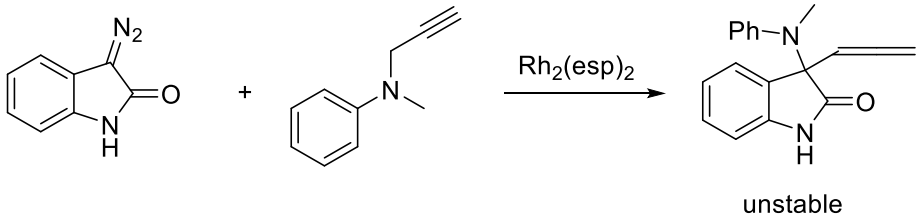
The 5 mL round bottom flask was equipment with 563 ul above $Rh_2(esp)_2$ solution, and the solution was concentrated *in vacuo*. 0.24 mmol substrate **2** and 2.0 mL 1,4-dioxane were added and then sonicated for 10 minutes until the catalyst was completely dissolved, followed by adding 0.2 mmol diazo reagent in three portions over 30 min. Then the reaction vial was stirred for 96 hours under room temperature. After that mestrimethoxybenzene as internal standards was added. The organic solution was further concentrated *in vacuo*, and the yields confirmed by HNMR.

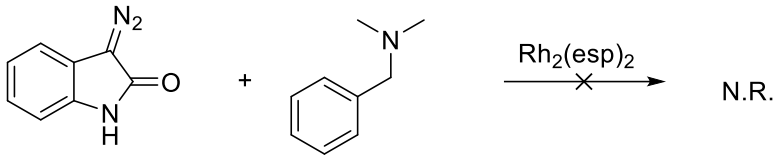
(H) Examples of low yield products and filed examples

The following examples were synthesis according to modified *General procedure for [2,3]-sigmatropic rearrangement reactions*. 1.5 equiv. **2** were used and the reactions performed at 40℃ for 24 hours.



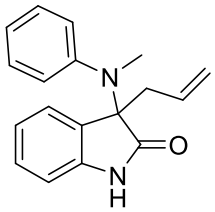
Filed examples: (*The following 2 reactions were performed according to the General Procedure shown in* **Section E**)





(I) Analytical data of products

3-allyl-3-(methyl(phenyl)amino)indolin-2-one (3a)



A pale yellow solid, 90% yield.

M.P.: 103.1℃~105.5℃.

TLC: R_f = 0.61 (Hexane/EtOAc = 3:1).

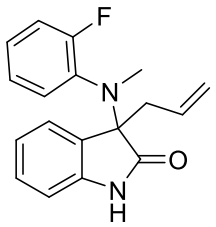
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.93 (s, 1H), 7.28 – 7.15 (m, 6H), 7.11 – 7.04 (m, 1H), 7.05 – 6.99 (m, 1H), 6.89 – 6.77 (m, 1H), 5.36 – 5.21 (m, 1H), 5.00 – 4.87 (m, 1H), 4.84 (ddd, *J* = 10.2, 2.0, 0.9 Hz, 1H), 2.81 (s, 3H), 2.77 – 2.66 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 180.2, 149.3, 140.8, 131.5, 130.3, 128.8, 128.3, 127.5, 125.0, 125.0, 122.5, 119.7, 110.0, 70.7, 41.9, 38.6.

IR(KBr/cm^{-1}): 3431.2, 3150.1, 3072.8, 1701.4, 1618.6, 1472.3, 1291.9, 1198.4, 763.4, 696.3.

HRMS (ESI+): *m/z* calcd for $C_{18}H_{18}N_2ONa^+$ $[M+Na]^+$: 301.1311, found: 301.1311.

3-allyl-3-((2-fluorophenyl)(methyl)amino)indolin-2-one (3b)



A pale yellow solid, 98% yield.

TLC: R_f = 0.63 (Hexane/EtOAc = 3:1).

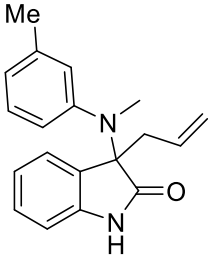
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.03 (br s, 1H), 7.59 – 7.47 (m, 1H), 7.30 – 7.22 (m, 1H), 7.14 – 7.07 (m, 1H), 7.04 – 6.97 (m, 1H), 6.98 – 6.88 (m, 3H), 6.79 – 6.74 (m, 1H), 5.28 – 5.14 (m, 1H), 4.81 (dd, *J* = 17.1, 1.8 Hz, 1H), 4.75 (dd, *J* = 10.1, 1.8 Hz, 1H), 2.69 – 2.62 (m, 4H), 2.58 – 2.50 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 180.52, 159.53 (d, *J* = 246.9 Hz), 140.81, 136.78 (d, *J* = 10.6 Hz), 131.32, 130.48, 130.34, 128.84, 126.85 (d, *J* = 8.1 Hz), 125.03, 124.00 (d, *J* = 3.9 Hz), 122.73, 116.09 (d, *J* = 21.9 Hz), 109.92, 70.53, 40.50 (d, *J* = 1.7 Hz), 38.53 (d, *J* = 1.6 Hz).

^{19}F NMR (376 MHz, $CDCl_3$) δ -118.8.

HRMS (ESI+): *m/z* calcd for $C_{18}H_{17}FN_2ONa^+$ $[M+Na]^+$: 319.1217, found: 319.1218.

3-allyl-3-(methyl(m-tolyl)amino)indolin-2-one (3c)



A pale yellow oil, 94 % yield.

TLC: R_f = 0.51 (Hexane/EtOAc = 3:1).

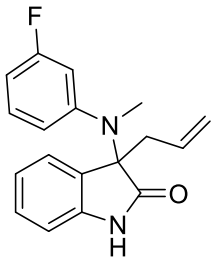
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.29 – 9.03 (m, 1H), 7.15 – 7.07 (m, 2H), 7.06 – 6.90 (m, 4H), 6.85 – 6.79 (m, 1H), 6.79 – 6.71 (m, 1H), 5.29 – 5.12 (m, 1H), 4.91 – 4.79 (m, 1H), 4.79 – 4.71 (m, 1H), 2.69 (s, 3H), 2.67 – 2.56 (m, 2H), 2.19 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 180.4, 149.1, 141.0, 137.9, 131.5, 130.2, 128.7, 128.3, 128.0, 125.7, 124.9, 124.3, 122.4, 119.5, 110.1, 70.7, 41.9, 38.5, 21.5.

IR(KBr/cm^{-1}): 3242.5, 3081.1, 2921.8, 1713.6, 1481.4, 1470.9, 1325.1, 1226.5, 751.1, 707.4.

HRMS (ESI+): *m/z* calcd for $C_{19}H_{20}N_2ONa^+$ $[M+Na]^+$: 315.1468, found: 315.1468.

3-allyl-3-((3-fluorophenyl)(methyl)amino)indolin-2-one (3d)



A pale yellow solid, 93% yield.

TLC: R_f = 0.60 (Hexane/EtOAc = 3:1).

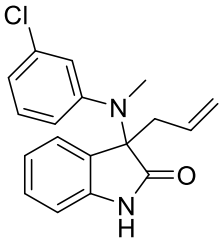
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.14 (s, 1H), 7.23 – 7.18 (m, 2H), 7.16 – 7.09 (m, 1H), 7.05 – 7.00 (m, 1H), 6.98 – 6.91 (m, 2H), 6.87 – 6.83 (m, 1H), 6.78 – 6.72 (m, 1H), 5.41 – 5.25 (m, 1H), 4.95 (dd, *J* = 17.1, 1.7 Hz, 1H), 4.87 (dd, *J* = 10.2, 1.7 Hz, 1H), 2.84 (s, 3H), 2.78 – 2.70 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 180.1, 162.7 (d, *J* = 245.4 Hz), 151.3 (d, *J* = 9.1 Hz), 140.7, 131.2, 130.1, 129.2 (d, *J* = 9.5 Hz), 129.0, 124.7, 122.7, 122.3 (d, *J* = 2.8 Hz), 119.9, 113.6 (d, *J* = 21.7 Hz), 111.3 (d, *J* = 21.2 Hz), 110.3, 70.5, 41.9, 38.5.

^{19}F NMR (376 MHz, $CDCl_3$) δ -112.8.

HRMS (ESI+): *m/z* calcd for $C_{18}H_{17}FN_2ONa^+$ $[M+Na]^+$: 319.1217, found: 319.1217.

3-allyl-3-((3-chlorophenyl)(methyl)amino)indolin-2-one (3e)



A pale yellow solid, 96 % yield.

M.P.: 102.5℃~105.3℃.

TLC: R_f = 0.45 (Hexane/EtOAc = 3:1).

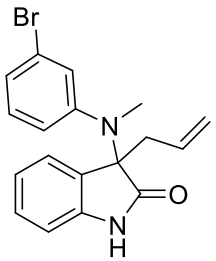
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.38 (s, 1H), 7.27 – 7.16 (m, 3H), 7.14 – 7.07 (m, 2H), 7.07 – 6.98 (m, 2H), 6.90 – 6.83 (m, 1H), 5.38 – 5.23 (m, 1H), 4.99 – 4.89 (m, 1H), 4.89 – 4.82 (m, 1H), 2.81 (s, 3H), 2.74 – 2.68 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 179.9, 150.7, 140.7, 140.7, 133.8, 131.1, 130.0, 129.2, 129.0, 127.1, 125.1, 124.8, 122.7, 119.9, 110.3, 110.2, 70.4, 41.8, 38.6.

IR(KBr/cm^{-1}): 3287.8, 3061.6, 2944.9, 1717.9, 1678.5, 1473.45, 1324.9, 1191.2, 756.1, 695.5.

HRMS (ESI+): *m/z* calcd for $C_{18}H_{17}ClN_2ONa^+$ $[M+Na]^+$: 335.0922, found: 335.0922.

3-allyl-3-((3-bromophenyl)(methyl)amino)indolin-2-one (3f)



A white solid, 93% yield.

TLC: R_f = 0. (Hexane/EtOAc = 3:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.11 (br s, 1H), 7.35 – 7.29 (m, 1H), 7.17 – 7.04 (m, 4H), 7.01 – 6.92 (m, 2H), 6.82 – 6.75 (m, 1H), 5.29 – 5.15 (m, 1H), 4.86 (dd, *J* = 17.0, 1.8 Hz, 1H), 4.79 (dd, *J* = 10.1, 1.9 Hz, 1H), 2.73 (s, 3H), 2.67 – 2.59 (m, 2H).

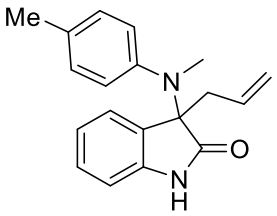
^{13}C NMR (101 MHz, $CDCl_3$) δ 180.0, 150.8, 140.7, 131.1, 130.1, 130.0, 129.6, 129.0, 127.7, 125.6, 124.8, 122.7, 121.9, 119.9, 110.3, 110.3, 70.5, 41.8, 38.6.

IR(KBr/cm^{-1}): 3242.8, 2921.3, 1714.6, 1618.2, 1471.5, 1187.2, 752.5, 700.0.

HRMS (ESI+): *m/z* calcd for $C_{18}H_{17}{}^{79}BrN_2ONa^+$ $[M+Na]^+$: 379.0416, found: 379.0416.

HRMS (ESI+): *m/z* calcd for $C_{18}H_{17}{}^{81}BrN_2ONa^+$ $[M+Na]^+$: 381.0396, found: 381.0396.

3-allyl-3-(methyl(p-tolyl)amino)indolin-2-one (3g)



A pale yellow oil, 98% yield.

TLC: R_f = 0.49 (Hexane/EtOAc = 3:1).

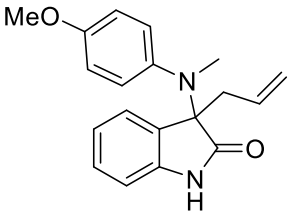
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.98 (s, 1H), 7.25 – 7.18 (m, 2H), 7.18 – 7.11 (m, 2H), 7.06 – 6.98 (m, 3H), 6.86 – 6.79 (m, 1H), 5.34 – 5.18 (m, 1H), 4.97 – 4.87 (m, 1H), 4.82 (dd, *J* = 10.1, 1.9 Hz, 1H), 2.75 (s, 3H), 2.74 – 2.61 (m, 2H), 2.27 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 180.3, 146.6, 141.0, 134.7, 131.5, 130.2, 128.9, 128.7, 127.6, 125.0, 122.4, 119.5, 110.0, 70.8, 41.9, 38.6, 21.0.

IR(KBr/cm^{-1}): 3243.9, 3082.2, 3026.7, 1717.3, 1617.6, 1510.1, 1469.8, 1225.9, 1185.9, 751.5,

HRMS (ESI+): *m/z* calcd for $C_{19}H_{20}N_2ONa^+$ $[M+Na]^+$: 315.1468, found: 315.1468.

3-allyl-3-((4-methoxyphenyl)(methyl)amino)indolin-2-one (3h)



A pale yellow solid, 91% yield.

TLC: R_f = 0.50 (Hexane/EtOAc = 3:1).

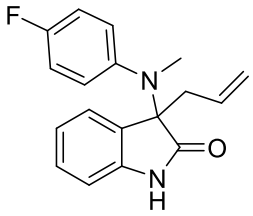
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.10 (s, 1H), 7.19 – 7.07 (m, 4H), 6.97 – 6.91 (m, 1H), 6.74 (d, *J* = 7.7 Hz, 1H), 6.69 – 6.63 (m, 2H), 5.24 – 5.11 (m, 1H), 4.87 – 4.80 (m, 1H), 4.73 (dd, *J* = 10.2, 2.0 Hz, 1H), 3.66 (s, 3H), 2.64 (s, 3H), 2.63 – 2.52 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 180.3, 157.1, 142.0, 140.9, 131.5, 130.2, 129.1, 128.7, 125.0, 122.4, 119.4, 113.3, 109.9, 70.9, 55.3, 41.7, 38.8.

IR(KBr/cm^{-1}): 3246.9, 3251.7, 1713.1, 1617.5, 1508.0, 1470.9, 1243.6, 1181.9, 1035.8, 752.2.

HRMS (ESI+): *m/z* calcd for $C_{19}H_{20}N_2O_2Na^+$ $[M+Na]^+$: 331.1417, found: 331.1417.

3-allyl-3-((4-fluorophenyl)(methyl)amino)indolin-2-one (3i)



A pale yellow oil, 95 % yield.

TLC: R_f = 0.48(Hexane/EtOAc = 3:1).

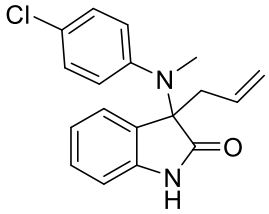
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.18 – 8.65 (m, 1H), 7.29 – 7.17 (m, 4H), 7.09 – 7.00 (m, 1H), 6.93 – 6.85 (m, 2H), 6.85 – 6.78 (m, 1H), 5.35 – 5.21 (m, 1H), 4.97 – 4.88 (m, 1H), 4.88 – 4.81 (m, 1H), 2.75 (s, 3H), 2.70 – 2.64 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 180.2, δ 160.3 (d, *J* = 244.1 Hz), 145.2 (d, *J* = 3.0 Hz), 140.9 (d, *J* = 2.3 Hz), 131.3, 130.1, 129.5, 129.5, 128.9, 125.0, 122.6, 119.7, 115.1, 114.8, 110.1 (d, *J* = 2.1 Hz), 70.7, 41.6, 38.8.

IR(KBr/cm^{-1}): 3244.3, 3081.1, 2951.1, 1713.3, 1505.2, 1470.8, 1220.6, 1187.4, 752.2, 672.2.

HRMS (ESI+): *m/z* calcd for $C_{18}H_{17}FN_2ONa^+$ $[M+Na]^+$: 319.1217, found: 319.1217.

3-allyl-3-((4-chlorophenyl)(methyl)amino)indolin-2-one (3j)



A pale yellow oil, 90 % yield.

TLC: R_f = 0.57 (Hexane/EtOAc = 3:1).

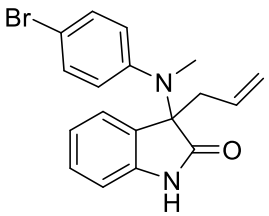
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.22 – 9.04 (m, 1H), 7.16 – 7.11 (m, 2H), 7.11 – 7.03 (m, 4H), 6.98 – 6.91 (m, 1H), 6.80 – 6.71 (m, 1H), 5.27 – 5.13 (m, 1H), 4.90 – 4.80 (m, 1H), 4.80 – 4.73 (m, 1H), 2.70 (s, 3H), 2.64 – 2.59 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 180.1, 147.9, 140.8, 131.2, 130.3, 130.0, 129.0, 128.7, 128.4, 124.8, 122.7, 119.8, 110.2, 70.6, 41.8, 38.6.

IR(KBr/cm^{-1}): 3245.9, 3083.1, 2924.1, 1713.7, 1618.8, 1487.9, 1187.4, 1095.5, 752.3, 654.9.

HRMS (ESI+): *m/z* calcd for $C_{18}H_{17}ClN_2ONa^+$ $[M+Na]^+$: 335.0922, found: 335.0922.

3-allyl-3-((4-bromophenyl)(methyl)amino)indolin-2-one (3k)



A pale yellow oil, 84 % yield.

TLC: R_f = 0.57 (Hexane/EtOAc = 3:1).

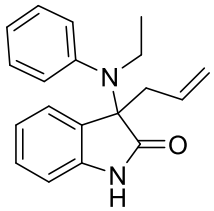
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.12 (s, 1H), 7.35 – 7.30 (m, 2H), 7.26 – 7.19 (m, 2H), 7.15 – 7.09 (m, 2H), 7.08 – 7.00 (m, 1H), 6.88 – 6.82 (m, 1H), 5.31 (ddt, *J* = 17.1, 10.0, 7.2 Hz, 1H), 4.96 (dq, *J* = 17.1, 1.4 Hz, 1H), 4.90 – 4.84 (m, 1H), 2.81 (s, 3H), 2.72 (d, *J* = 7.2 Hz, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 180.0, 148.4, 140.7, 131.4, 131.2, 130.0, 129.0, 128.9, 124.8, 122.7, 119.9, 118.1, 110.2, 70.5, 41.8, 38.6.

HRMS (ESI+): *m/z* calcd for $C_{18}H_{17}{}^{79}BrN_2ONa^+$ $[M+Na]^+$: 379.0416, found: 379.0416.

HRMS (ESI+): *m/z* calcd for $C_{18}H_{17}{}^{81}BrN_2ONa^+$ $[M+Na]^+$: 381.0396, found: 381.0396.

3-allyl-3-(ethyl(phenyl)amino)indolin-2-one (3l)



A pale yellow solid, 56% yield.

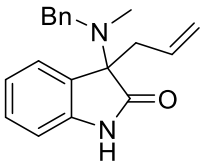
TLC: R_f = 0.61 (Hexane/EtOAc = 3:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.89 (s, 1H), 7.44 – 7.35 (m, 2H), 7.34 – 7.29 (m, 1H), 7.29 – 7.22 (m, 2H), 7.22 – 7.10 (m, 2H), 7.07 – 6.99 (m, 1H), 6.85 – 6.80 (m, 1H), 5.31 – 5.16 (m, 1H), 4.87 (d, *J* = 17.0 Hz, 1H), 4.79 (dd, *J* = 10.0, 2.8 Hz, 1H), 3.17 – 3.04 (m, 1H), 2.97 – 2.83 (m, 1H), 2.62 – 2.48 (m, 2H), 0.83 – 0.75 (m, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 180.8, 146.2, 140.9, 131.5, 131.4, 130.5, 128.6, 128.3, 125.9, 124.9, 122.5, 119.4, 109.8, 71.1, 44.4, 42.0, 14.5.

HRMS (ESI+): *m/z* calcd for $C_{19}H_{20}N_2ONa^+$ $[M+Na]^+$: 315.1468, found: 315.1468.

3-allyl-3-(benzyl(methyl)amino)indolin-2-one (3m)



A white solid, 60% yield.

TLC: R_f = 0.62 (Hexane/EtOAc = 3:1).

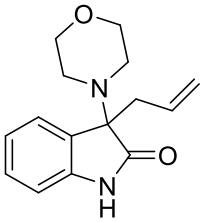
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.72 (br s, 1H), 7.38 – 7.29 (m, 1H), 7.25 – 7.09 (m, 6H), 7.04 – 6.94 (m, 1H), 6.86 – 6.79 (m, 1H), 5.57 – 5.43 (m, 1H), 5.00 – 4.92 (m, 1H), 4.92 – 4.86 (m, 1H), 3.52 (q, *J* = 13.4 Hz, 2H), 2.86 – 2.74 (m, 1H), 2.74 – 2.64 (m, 1H), 2.24 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 180.2, 140.9, 139.9, 131.8, 131.0, 128.8, 128.6, 128.3, 127.0, 124.8, 122.8, 119.4, 110.0, 70.3, 55.6, 40.0, 35.1.

IR(KBr/cm^{-1}): 3187.7, 3081.8, 2952.0, 1711.3, 1615.3, 1474.6, 1287.9, 1249.6, 1198.4, 749.9..

HRMS (ESI+): *m/z* calcd for $C_{19}H_{20}N_2ONa^+$ $[M+Na]^+$: 315.1468, found: 315.1468.

3-allyl-3-morpholinoindolin-2-one (3n)



A pale yellow oil, 51% yield.

TLC: R_f = 0.51 (Hexane/EtOAc = 3:1).

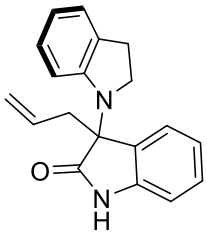
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.07 (br s, 1H), 7.26 – 7.19 (m, 1H), 7.19 – 7.10 (m, 1H), 7.01 – 6.92 (m, 1H), 6.85 – 6.74 (m, 1H), 5.51 – 5.35 (m, 1H), 4.98 – 4.90 (m, 1H), 4.90 – 4.84 (m, 1H), 3.65 – 3.52 (m, 5H), 2.74 – 2.54 (m, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 179.6, 141.1, 131.3, 129.5, 128.9, 124.9, 122.6, 119.5, 110.1, 70.3, 67.4, 47.3, 38.7.

IR(KBr/cm^{-1}): 3427.2, 3197.9, 2971.0, 1712.9, 1673.3, 1472.3, 1112.7, 920.6, 758.2, 725.8.

HRMS (ESI+): *m/z* calcd for $C_{15}H_{18}N_2O_2Na^+$ $[M+Na]^+$: 281.1260, found: 281.1260.

3'-allyl-[1,3'-biindolin]-2'-one (3o)



A pale yellow solid, 55% yield.

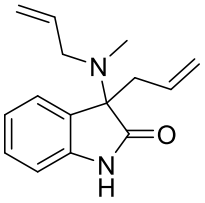
TLC: R_f = 0.52 (Hexane/EtOAc = 3:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.80 (br s, 1H), 7.23 – 7.19 (m, 1H), 7.18 – 7.12 (m, 1H), 6.99 – 6.88 (m, 2H), 6.80 – 6.73 (m, 1H), 6.63 – 6.55 (m, 1H), 6.54 – 6.45 (m, 1H), 5.63 – 5.55 (m, 1H), 5.53 – 5.38 (m, 1H), 5.03 – 4.96 (m, 1H), 4.96 – 4.91 (m, 1H), 4.12 – 4.01 (m, 1H), 3.87 – 3.78 (m, 1H), 3.00 – 2.86 (m, 3H), 2.84 – 2.73 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 179.9, 149.9, 140.2, 131.0, 130.8, 130.4, 129.1, 127.2, 124.6, 124.6, 123.0, 120.3, 118.9, 110.5, 108.9, 67.0, 50.3, 42.5, 28.3.

HRMS (ESI+): *m/z* calcd for $C_{19}H_{18}N_2ONa^+$ $[M+Na]^+$: 313.1311, found: 313.1311.

3-allyl-3-(allyl(methyl)amino)indolin-2-one (3p)

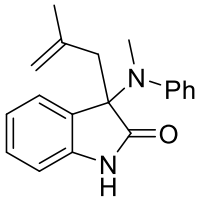


A pale yellow oil, 61% yield.

TLC: R_f = 0.56 (Hexane/EtOAc = 3:1).

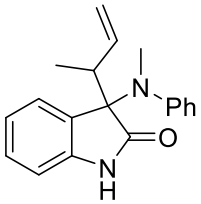
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.96 (br s, 1H), 7.27 – 7.21 (m, 1H), 7.18 – 7.10 (m, 1H), 7.03 – 6.93 (m, 1H), 6.84 – 6.78 (m, 1H), 5.75 – 5.63 (m, 1H), 5.48 – 5.35 (m, 1H), 5.09 – 5.00 (m, 1H), 5.00 – 4.89 (m, 2H), 4.89 – 4.83 (m, 1H), 3.05 (qd, *J* = 13.6, 6.3 Hz, 2H), 2.77 – 2.70 (m, 1H), 2.70 – 2.60 (m, 1H), 2.32 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 180.3, 140.9, 136.4, 131.6, 130.4, 128.8, 124.8, 122.6, 119.4, 117.2, 110.1, 70.3, 54.8, 40.1, 35.3.
HRMS (ESI+): *m/z* calcd for $C_{15}H_{18}N_2ONa^+$ $[M+Na]^+$: 265.1311, found: 265.1311.

3-(methyl(phenyl)amino)-3-(2-methylallyl)indolin-2-one (3q)



A pale yellow oil, 60% yield.
TLC: R_f = 0.52 (Hexane/EtOAc = 3:1).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.95 (s, 1H), 7.34 – 7.18 (m, 6H), 7.18 – 7.09 (m, 1H), 7.07 – 6.99 (m, 1H), 6.87 – 6.80 (m, 1H), 4.62 – 4.59 (m, 1H), 4.58 – 4.54 (m, 1H), 2.89 (d, *J* = 12.3 Hz, 1H), 2.79 (s, 3H), 2.60 (d, *J* = 12.3 Hz, 1H), 1.30 – 1.28 (m, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 180.2, 149.3, 141.3, 139.5, 130.4, 128.9, 128.3, 128.0, 125.6, 125.2, 122.3, 116.1, 110.0, 71.0, 45.3, 38.6, 24.0.
HRMS (ESI+): *m/z* calcd for $C_{19}H_{20}N_2ONa^+$ $[M+Na]^+$: 315.1468, found: 315.1468.

3-(but-3-en-2-yl)-3-(methyl(phenyl)amino)indolin-2-one (3r)



A pale yellow oil, 66% yield. (d.r.=1.0:0.46)
TLC: R_f = 0.51 (Hexane/EtOAc = 3:1).

Minor diastereoisomer

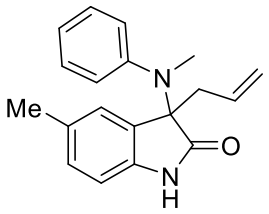
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.45 (s, 1H), 7.21 – 7.05 (m, 5H), 7.00 – 6.92 (m, 2H), 6.72 – 6.68 (m, 1H), 6.44 – 6.33 (m, 1H), 5.30 – 5.18 (m, 2H), 5.06 – 4.99 (m, 1H), 3.38 – 3.28 (m, 1H), 3.07 (s, 3H), 1.31 (s, 3H).

Major diastereoisomer

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.45 (s, 1H), 7.36 – 7.30 (m, 1H), 7.21 – 7.05 (m, 5H), 7.00 – 6.92 (m, 2H), 6.72 – 6.68 (m, 1H), 5.30 – 5.18 (m, 1H), 5.13 – 5.07 (m, 1H), 4.89 – 4.81 (m, 1H), 3.26 – 3.16 (m, 1H), 3.04 (s, 3H), 1.29 (s, 3H).

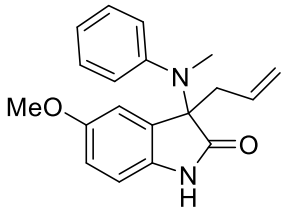
^{13}C NMR (101 MHz, $CDCl_3$) δ 180.7, 180.3, 150.6, 141.0, 140.8, 138.1, 138.0, 128.9, 128.9, 128.6, 128.4, 128.2, 127.6, 127.4, 126.6, 126.4, 124.8, 124.7, 121.9, 121.9, 117.2, 116.5, 109.7, 109.5, 72.9, 72.8, 42.3, 39.6, 38.8, 38.4, 14.3, 13.8.
HRMS (ESI+): *m/z* calcd for $C_{19}H_{20}N_2ONa^+$ $[M+Na]^+$: 315.1468, found: 315.1467.

3-allyl-5-methyl-3-(methyl(phenyl)amino)indolin-2-one (3s)



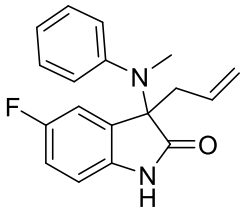
A white solid, 72% yield.
TLC: R_f = 0.60 (Hexane/EtOAc = 3:1).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.75 (s, 1H), 7.23 – 7.10 (m, 4H), 7.04 – 6.97 (m, 1H), 6.97 – 6.94 (m, 1H), 6.94 – 6.88 (m, 1H), 6.63 (d, *J* = 7.8 Hz, 1H), 5.26 – 5.12 (m, 1H), 4.85 (dd, *J* = 17.1, 1.8 Hz, 1H), 4.75 (dd, *J* = 10.1, 1.8 Hz, 1H), 2.71 (s, 3H), 2.64 – 2.58 (m, 2H), 2.24 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 180.1, 149.3, 138.4, 132.0, 131.6, 130.4, 129.1, 128.3, 127.5, 125.6, 124.9, 119.5, 109.7, 70.7, 42.0, 38.6, 21.3.
HRMS (ESI+): *m/z* calcd for $C_{19}H_{20}N_2ONa^+$ $[M+Na]^+$: 315.1468, found: 315.1468.

3-allyl-5-methoxy-3-(methyl(phenyl)amino)indolin-2-one (3t)



A pale yellow solid, 90% yield.
M.P.: 135.6℃~137.1℃.
TLC: R_f = 0.31 (Hexane/EtOAc = 3:1).
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.22 (s, 1H), 7.34 – 7.16 (m, 4H), 7.13 – 7.04 (m, 1H), 6.83 – 6.79 (m, 1H), 6.79 – 6.71 (m, 2H), 5.36 – 5.20 (m, 1H), 4.94 (dd, *J* = 17.1, 1.8 Hz, 1H), 4.89 – 4.79 (m, 1H), 3.76 (s, 3H), 2.79 (s, 3H), 2.77 – 2.60 (m, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 180.2, 155.8, 149.1, 134.3, 131.5, 131.4, 128.3, 127.4, 125.0, 119.7, 113.4, 111.7, 110.5, 71.1, 55.8, 42.1, 38.6.
IR(KBr/cm^{-1}): 3285.4, 3069.9, 2946.8, 1718.9, 1677.4, 1489.3, 1294.4, 1200.8, 701.1, 620.8.
HRMS (ESI+): *m/z* calcd for $C_{19}H_{20}N_2O_2Na^+$ $[M+Na]^+$: 331.1417, found: 331.1417.

3-allyl-5-fluoro-3-(methyl(phenyl)amino)indolin-2-one (3u)



A white solid, 81 % yield.

M.P.: 140.2℃~142.7℃.

TLC: R_f = 0.37 (Hexane/EtOAc = 3:1).

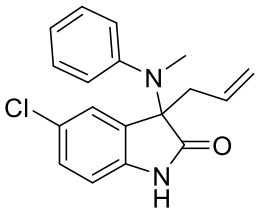
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.01 – 8.68 (m, 1H), 7.28 – 7.19 (m, 4H), 7.12 – 7.06 (m, 1H), 7.00 – 6.95 (m, 1H), 6.94 – 6.85 (m, 1H), 6.81 – 6.72 (m, 1H), 5.40 – 5.19 (m, 1H), 4.99 – 4.91 (m, 1H), 4.90 – 4.85 (m, 1H), 2.81 (s, 3H), 2.76 – 2.60 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 180.1, 159.1 (d, J = 240.9 Hz), 148.9, 136.5, 132.1 (d, J = 7.5 Hz), 130.9, 128.4, 127.4, 125.2, 120.0, 115.1 (d, J = 23.7 Hz), 112.7 (d, J = 24.5 Hz), 110.4 (d, J = 7.8 Hz), 71.0, 71.0, 41.8, 38.6.

IR(KBr/cm^{-1}): 3249.9, 2945.6, 2848.2, 1718.7, 1677.8, 1486.7, 1192.1, 1170.9, 697.8.

HRMS (ESI+): *m/z* calcd for $C_{18}H_{17}FN_2ONa^+$ $[M+Na]^+$: 319.1217, found: 319.1218.

3-allyl-5-chloro-3-(methyl(phenyl)amino)indolin-2-one (3v)



A pale yellow solid, 78 % yield.

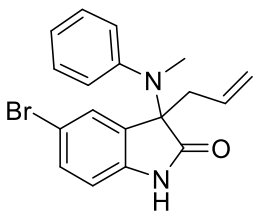
TLC: R_f = 0.36 (Hexane/EtOAc = 3:1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 10.51 (s, 1H), 7.26 – 7.21 (m, 1H), 7.20 – 7.11 (m, 3H), 7.09 – 7.03 (m, 2H), 7.01 – 6.95 (m, 1H), 6.72 (d, J = 8.2 Hz, 1H), 5.35 – 5.10 (m, 1H), 4.97 – 4.76 (m, 2H), 2.74 (s, 3H), 2.72 – 2.66 (m, 1H), 2.52 – 2.44 (m, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 178.3, 149.7, 140.9, 132.7, 131.9, 128.9, 128.7, 126.3, 126.2, 124.9, 124.4, 120.0, 111.3, 69.8, 41.4, 38.4.

HRMS (ESI+): *m/z* calcd for $C_{18}H_{17}ClN_2ONa^+$ $[M+Na]^+$: 335.0922, found: 335.0922.

3-allyl-5-bromo-3-(methyl(phenyl)amino)indolin-2-one (3w)



A white solid, 84% yield.

TLC: R_f = 0.42 (Hexane/EtOAc = 3:1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 10.55 (s, 1H), 7.41 – 7.36 (m, 1H), 7.36 – 7.31 (m, 1H), 7.22 –

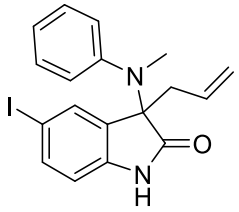
7.16 (m, 2H), 7.13 – 7.06 (m, 2H), 7.05 – 6.98 (m, 1H), 6.72 (d, *J* = 8.2 Hz, 1H), 5.37 – 5.20 (m, 1H), 4.96 – 4.85 (m, 2H), 2.76 (s, 3H), 2.74 – 2.70 (m, 1H), 2.53 – 2.49 (m, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 178.2, 149.7, 141.4, 133.1, 131.9, 131.8, 128.7, 127.6, 126.3, 124.4, 120.0, 113.9, 111.8, 69.8, 41.4, 38.4.

HRMS (ESI+): *m/z* calcd for $C_{18}H_{17}{}^{79}BrN_2ONa^+$ $[M+Na]^+$: 379.0416, found: 379.0416.

HRMS (ESI+): *m/z* calcd for $C_{18}H_{17}{}^{81}BrN_2ONa^+$ $[M+Na]^+$: 381.0396, found: 381.0396.

3-allyl-5-iodo-3-(methyl(phenyl)amino)indolin-2-one (3x)



A pale yellow solid, 80 % yield.

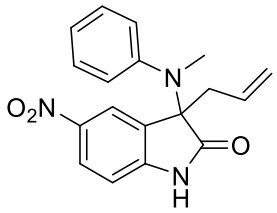
TLC: R_f = 0.41 (Hexane/EtOAc = 3:1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 10.52 (s, 1H), 7.53 – 7.47 (m, 2H), 7.25 – 7.15 (m, 2H), 7.11 (dt, *J* = 8.2, 1.7 Hz, 2H), 7.05 – 6.99 (m, 1H), 6.61 (d, *J* = 7.9 Hz, 1H), 5.34 – 5.20 (m, 1H), 4.96 – 4.84 (m, 2H), 2.75 (s, 3H), 2.71 – 2.66 (m, 1H), 2.51 – 2.44 (m, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 177.9, 149.6, 141.9, 137.5, 133.3, 133.1, 131.8, 128.6, 126.5, 124.4, 119.9, 112.3, 84.8, 69.6, 41.5, 38.5.

HRMS (ESI+): *m/z* calcd for $C_{18}H_{17}IN_2ONa^+$ $[M+Na]^+$: 427.0278, found: 427.0279.

3-allyl-3-(methyl(phenyl)amino)-5-nitroindolin-2-one (3y)



A pale yellow solid, 65% yield.

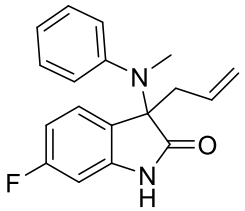
TLC: R_f = 0.43 (Hexane/EtOAc = 3:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.42 – 9.22 (m, 1H), 8.19 (d, *J* = 7.9 Hz, 2H), 7.30 – 7.20 (m, 4H), 7.16 – 7.10 (m, 1H), 6.98 – 6.91 (m, 1H), 5.38 – 5.23 (m, 1H), 4.99 – 4.93 (m, 1H), 4.93 – 4.89 (m, 1H), 2.83 (s, 3H), 2.79 – 2.67 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 180.7, 148.6, 146.5, 146.5, 143.6, 131.7, 130.3, 128.7, 127.7, 126.0, 125.8, 120.9, 120.9, 109.8, 70.7, 41.6, 38.8.

HRMS (ESI+): *m/z* calcd for $C_{18}H_{17}N_3O_3Na^+$ $[M+Na]^+$: 346.1162, found: 346.1163.

3-allyl-6-fluoro-3-(methyl(phenyl)amino)indolin-2-one (3z)



A white solid, 82% yield.

M.P.: 139.5℃~141.7℃.

TLC: R_f = 0.51 (Hexane/EtOAc = 3:1).

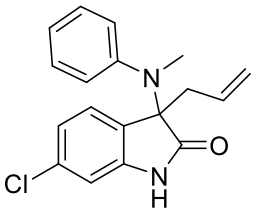
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.38 – 9.12 (m, 1H), 7.25 – 7.18 (m, 4H), 7.18 – 7.11 (m, 1H), 7.11 – 7.05 (m, 1H), 6.75 – 6.67 (m, 1H), 6.63 – 6.55 (m, 1H), 5.36 – 5.22 (m, 1H), 4.97 – 4.89 (m, 1H), 4.89 – 4.83 (m, 1H), 2.80 (s, 3H), 2.76 – 2.64 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 180.7, 163.1 (d, J = 245.6 Hz), 149.1, 142.2 (d, J = 12.1 Hz), 131.2, 128.4, 127.5, 126.1 (d, J = 9.6 Hz), 125.6 (d, J = 3.0 Hz), 125.2, 120.0, 108.9 (d, J = 22.3 Hz), 98.7 (d, J = 27.2 Hz), 70.3, 41.9, 38.5.

IR(KBr/cm^{-1}): 3169.0, 3062.4, 2955.9, 1704.3, 1618.7, 1491.0, 1141.5, 1120.4, 852.3, 698.8.

HRMS (ESI+): *m/z* calcd for $C_{18}H_{17}FN_2ONa^+$ [M+Na]$^+$: 319.1217, found: 319.1217.

3-allyl-6-chloro-3-(methyl(phenyl)amino)indolin-2-one (3aa)



A pale yellow solid, 50% yield.

M.P.: 155.4℃~156.9℃.

TLC: R_f = 0.47 (Hexane/EtOAc = 3:1).

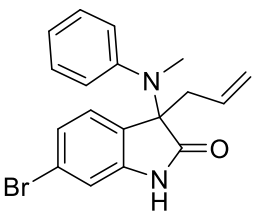
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.21 – 8.81 (m, 1H), 7.26 – 7.18 (m, 4H), 7.16 – 7.07 (m, 2H), 7.04 – 6.98 (m, 1H), 6.87 – 6.83 (m, 1H), 5.35 – 5.23 (m, 1H), 4.97 – 4.90 (m, 1H), 4.90 – 4.84 (m, 1H), 2.80 (s, 3H), 2.74 – 2.64 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 180.2, 149.0, 141.9, 134.4, 131.1, 128.7, 128.5, 127.5, 126.0, 125.3, 122.6, 120.1, 110.7, 70.4, 41.8, 38.6.

IR(KBr/cm^{-1}): 3241.6, 3077.8, 2948.8, 1725.3, 1683.1, 1615.3, 1487.5, 1320.7, 698.2, 629.8.

HRMS (ESI+): *m/z* calcd for $C_{18}H_{17}ClN_2ONa^+$ [M+Na]$^+$: 335.0922, found: 335.0922.

3-allyl-6-bromo-3-(methyl(phenyl)amino)indolin-2-one (3ab)



A pale yellow solid, 74% yield.

M.P.: 153.1℃~155.8℃.

TLC: R_f = 0.49 (Hexane/EtOAc = 3:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.05 (s, 1H), 7.25 – 7.19 (m, 4H), 7.19 – 7.14 (m, 1H), 7.13 – 7.05 (m, 2H), 7.04 – 6.99 (m, 1H), 5.36 – 5.21 (m, 1H), 4.98 – 4.91 (m, 1H), 4.91 – 4.85 (m, 1H), 2.79 (s, 3H), 2.73 – 2.65 (m, 2H).

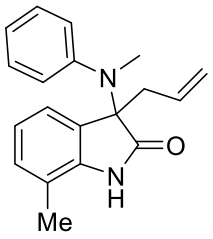
^{13}C NMR (101 MHz, $CDCl_3$) δ 180.1, 149.0, 142.0, 131.0, 129.3, 128.5, 127.5, 126.3, 125.5, 125.3, 122.2, 120.1, 113.5, 70.5, 41.8, 38.7.

IR(KBr/cm^{-1}): 3432.4, 3207.4, 2924.1, 1724.3, 1681.7, 1610.1, 1320.6, 1229.9, 773.1, 698.8.

HRMS (ESI+): *m/z* calcd for $C_{18}H_{17}{}^{79}BrN_2ONa^+$ $[M+Na]^+$: 379.0416, found: 379.0416.

HRMS (ESI+): *m/z* calcd for $C_{18}H_{17}{}^{81}BrN_2ONa^+$ $[M+Na]^+$: 381.0396, found: 381.0396.

3-allyl-7-methyl-3-(methyl(phenyl)amino)indolin-2-one (3ac)



A pale yellow oil, 95% yield.

TLC: R_f = 0.51(Hexane/EtOAc = 3:1).

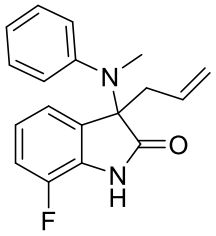
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.85 – 9.55 (m, 1H), 7.30 – 7.23 (m, 2H), 7.23 – 7.15 (m, 2H), 7.12 – 7.06 (m, 1H), 7.06 – 6.99 (m, 2H), 6.98 – 6.90 (m, 1H), 5.32 – 5.20 (m, 1H), 4.92 (dd, *J* = 16.9, 1.9 Hz, 1H), 4.83 (dd, *J* = 10.1, 1.9 Hz, 1H), 2.78 (s, 3H), 2.75 – 2.62 (m, 2H), 2.28 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 180.8, 149.3, 139.6, 131.6, 130.1, 129.8, 128.2, 127.5, 124.9, 122.4, 122.3, 119.4, 119.3, 71.0, 42.1, 38.7, 16.5.

IR(KBr/cm^{-1}): 3188.1, 3059.5, 2949.3, 1709.6, 1626.4, 1600.2, 1490.2, 1461.5, 747.1, 700.6.

HRMS (ESI+): *m/z* calcd for $C_{19}H_{20}N_2ONa^+$ $[M+Na]^+$: 315.1468, found: 315.1468.

3-allyl-7-fluoro-3-(methyl(phenyl)amino)indolin-2-one (3ad)



A white solid, 99% yield.

TLC: R_f = 0.67 (Hexane/EtOAc = 3:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.68 (br s, 1H), 7.20 – 7.08 (m, 4H), 7.03 – 6.94 (m, 2H), 6.93 – 6.86 (m, 2H), 5.20 (ddt, *J* = 17.1, 10.1, 7.2 Hz, 1H), 4.86 (dd, *J* = 17.1, 1.9 Hz, 1H), 4.79 (dd, *J* = 10.1, 1.9 Hz, 1H), 2.74 (s, 3H), 2.69 – 2.58 (m, 2H).

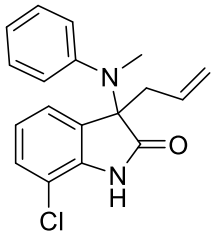
^{13}C NMR (101 MHz, $CDCl_3$) δ 179.3, 149.0, 147.0 (d, *J* = 244.7 Hz), 133.3 (d, *J* = 3.3 Hz), 131.0, 128.0 (d, *J* = 12.3 Hz), 127.9, 127.6, 125.3, 123.1 (d, *J* = 5.6 Hz), 120.7 (d, *J* = 12.3 Hz), 120.1, 115.8 (d, *J* = 17.2 Hz), 71.1, 41.8, 38.6.

^{19}F NMR (376 MHz, $CDCl_3$) δ -134.0.

IR(KBr/cm^{-1}): 3431.2, 3217.2, 3082.9, 1721.0, 1642.1, 1599.1, 1493.1, 1197.8, 774.7, 701.8.

HRMS (ESI+): *m/z* calcd for $C_{18}H_{17}FN_2ONa^+$ $[M+Na]^+$: 319.1217, found: 319.1217.

3-allyl-7-chloro-3-(methyl(phenyl)amino)indolin-2-one (3ae)



A white solid, 84% yield.

TLC: R_f = 0.65 (Hexane/EtOAc = 3:1).

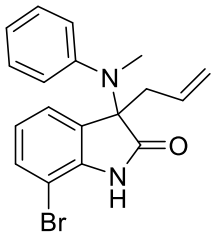
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.77 (s, 1H), 7.30 – 7.25 (m, 2H), 7.24 – 7.17 (m, 3H), 7.16 – 7.12 (m, 1H), 7.11 – 7.06 (m, 1H), 7.01 – 6.94 (m, 1H), 5.35 – 5.22 (m, 1H), 4.97 – 4.90 (m, 1H), 4.90 – 4.85 (m, 1H), 2.79 (s, 3H), 2.71 – 2.66 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 179.2, 149.0, 138.4, 132.1, 131.0, 128.7, 128.4, 127.8, 125.3, 123.3, 123.2, 120.1, 115.1, 71.7, 41.8, 38.8.

IR(KBr/cm^{-1}): 3414.0, 3194.9, 2922.4, 1720.3, 1618.1, 1475.8, 1315.9, 1174.8, 1113.6, 772.1, 701.1.

HRMS (ESI+): *m/z* calcd for $C_{18}H_{17}ClN_2ONa^+$ $[M+Na]^+$: 335.0922, found: 335.0922.

3-allyl-7-bromo-3-(methyl(phenyl)amino)indolin-2-one (3af)



A pale yellow oil, 84% yield.

TLC: R_f = 0.62 (Hexane/EtOAc = 3:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 (s, 1H), 7.34 – 7.26 (m, 3H), 7.26 – 7.15 (m, 3H), 7.13 – 7.06 (m, 1H), 6.96 – 6.88 (m, 1H), 5.37 – 5.24 (m, 1H), 4.97 – 4.90 (m, 1H), 4.90 – 4.85 (m, 1H), 2.78 (s, 3H), 2.71 – 2.64 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 179.0, 149.0, 140.1, 132.1, 131.5, 131.0, 128.4, 127.8, 125.3, 123.8, 123.7, 120.1, 103.1, 71.9, 41.9, 38.9.

IR(KBr/cm^{-1}): 3241.7, 3196.3, 3079.2, 1720.5, 1615.3, 1491.9, 1471.7, 1315.2, 1173.9, 1108.5, 772.9, 737.2, 701.8.

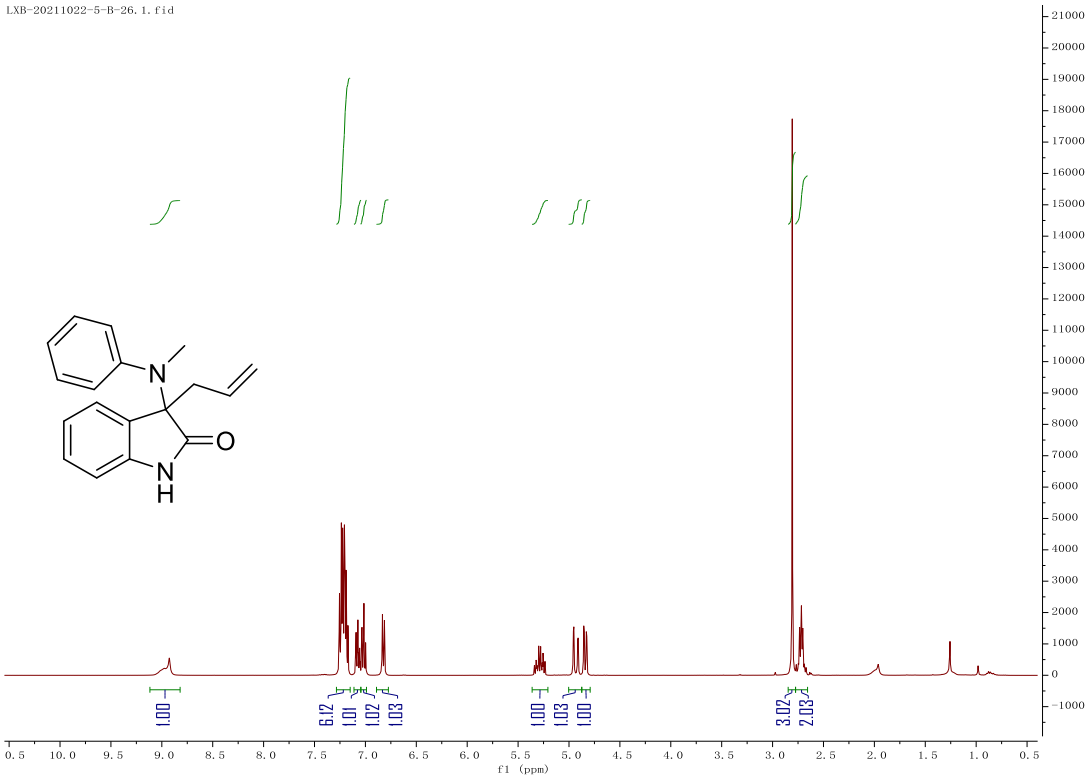
HRMS (ESI+): *m/z* calcd for $C_{18}H_{17}{}^{79}BrN_2ONa^+$ $[M+Na]^+$: 379.0416, found: 379.0416.

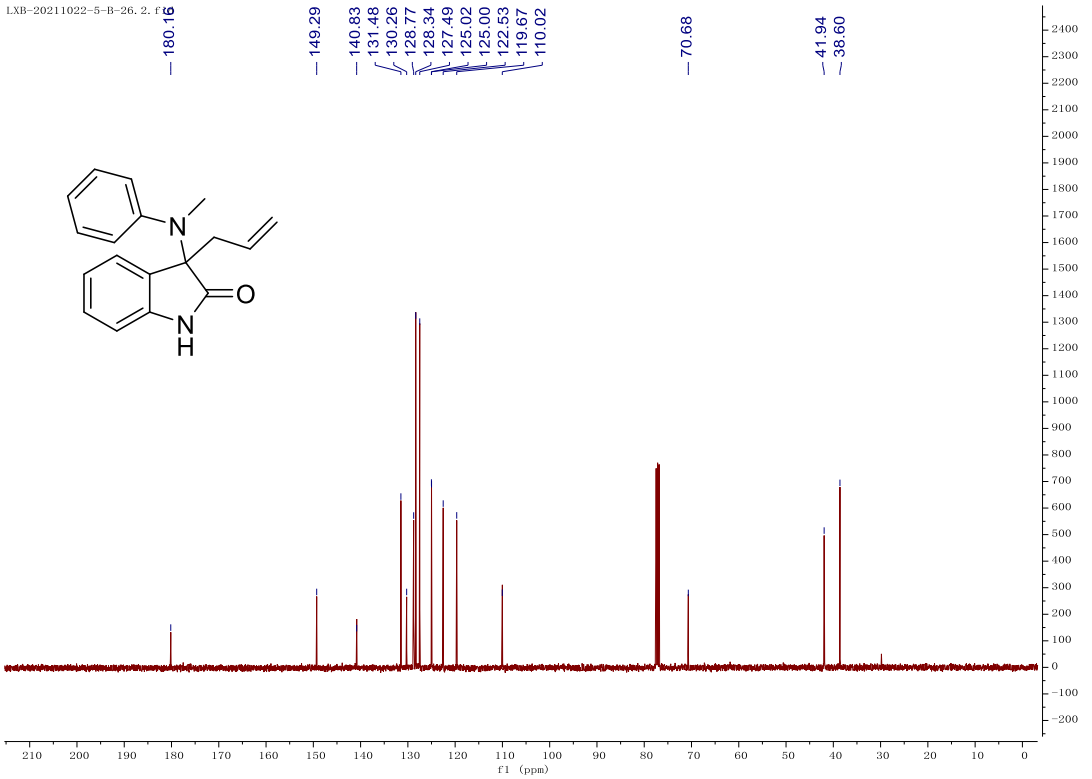
HRMS (ESI+): *m/z* calcd for $C_{18}H_{17}{}^{81}BrN_2ONa^+$ $[M+Na]^+$: 381.0396, found: 381.0396.

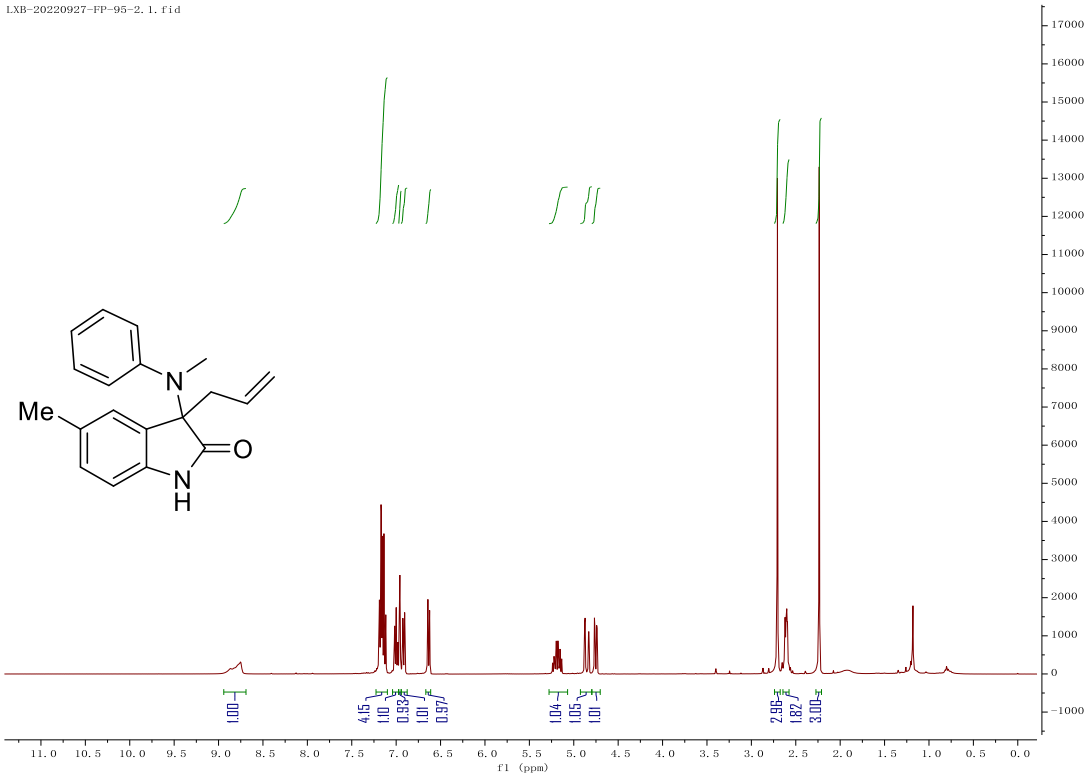
(J) References

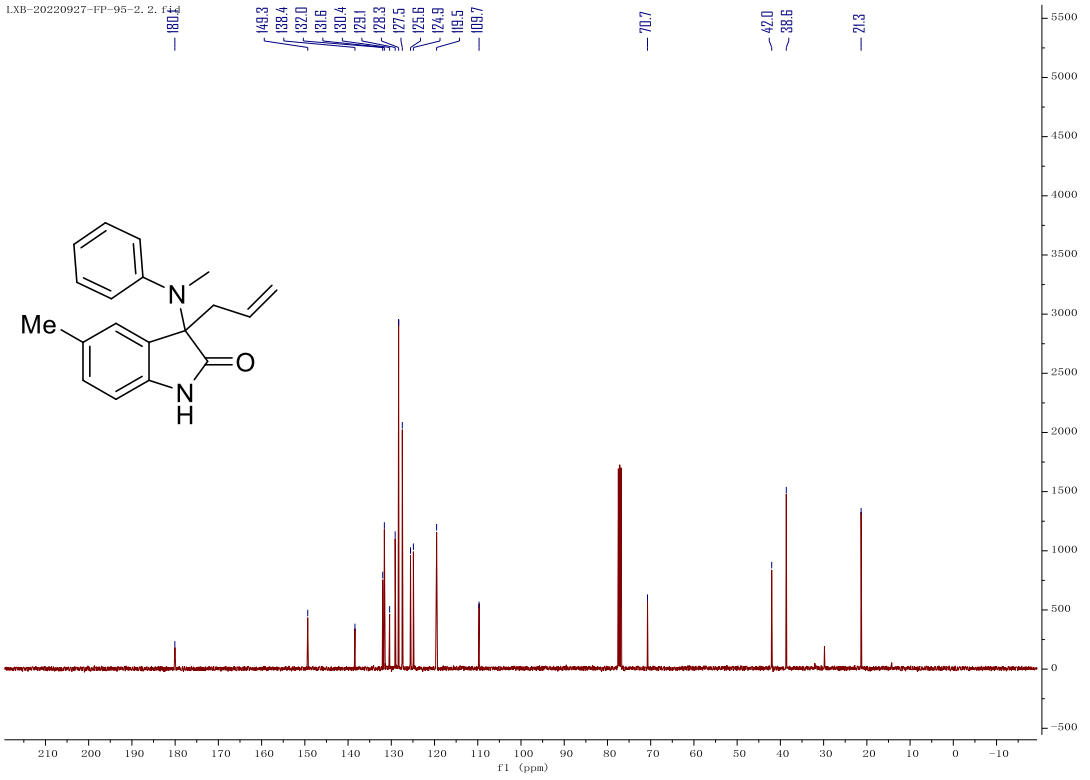
1. C. G. Espino, K. W. Fiori, M. Kim, J. Du Bois, *J. Am. Chem. Soc.*, **2004**, *126*, 15378.
2. B. Ma, P. Wu, X. Wang, Z. Wang, H.-X. Lin, H.-X. Dai, *Angew. Chem. Int. Ed.*, **2019**, *58*, 13335.
3. (a) M. Wang, F. Xiao, Y. Bai, X. Hu, *Synthetic Commun.*, **2015**, *45*, 2259; (b) L. A. Körte, S. Blomeyer, S. Heidemeyer, A. Mix, B. Neumann, N. W. Mitzel, *Chem. Commun.*, **2016**, *52*, 9949. (c) F. Li, F. He, R. M. Koenigs, *Synthesis* **2019**, *51*, 4348;

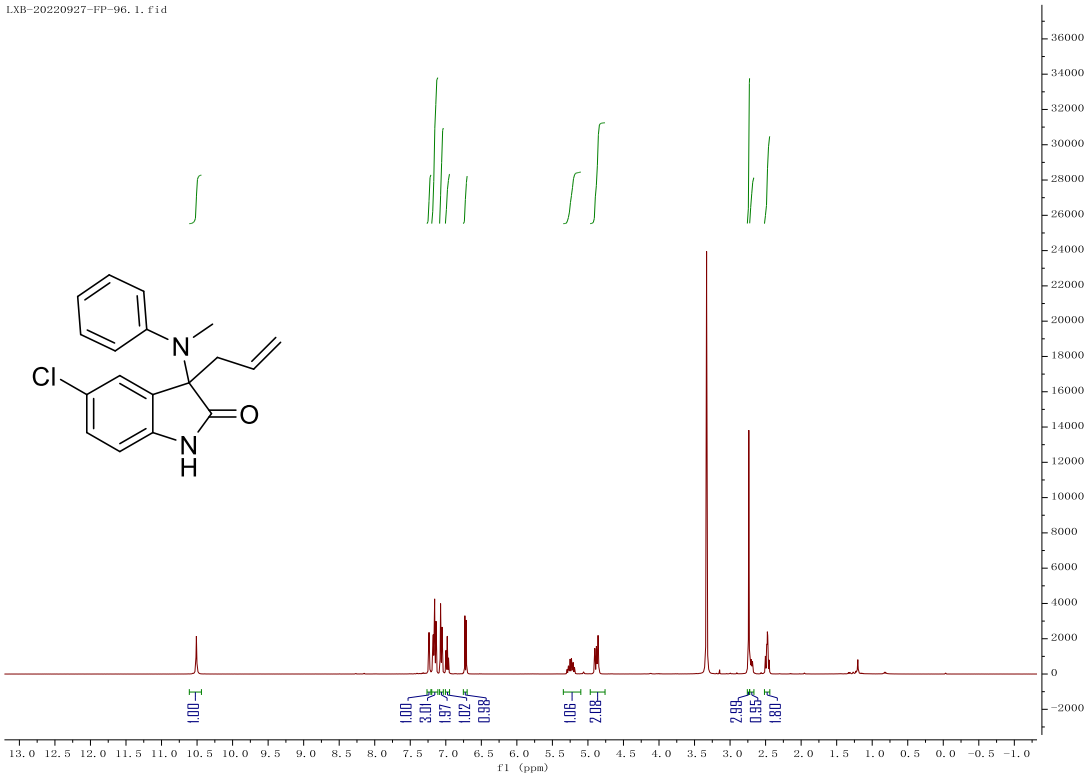
(K) Copies of NMR spectra (^{1}H, ^{13}C, ^{19}F)

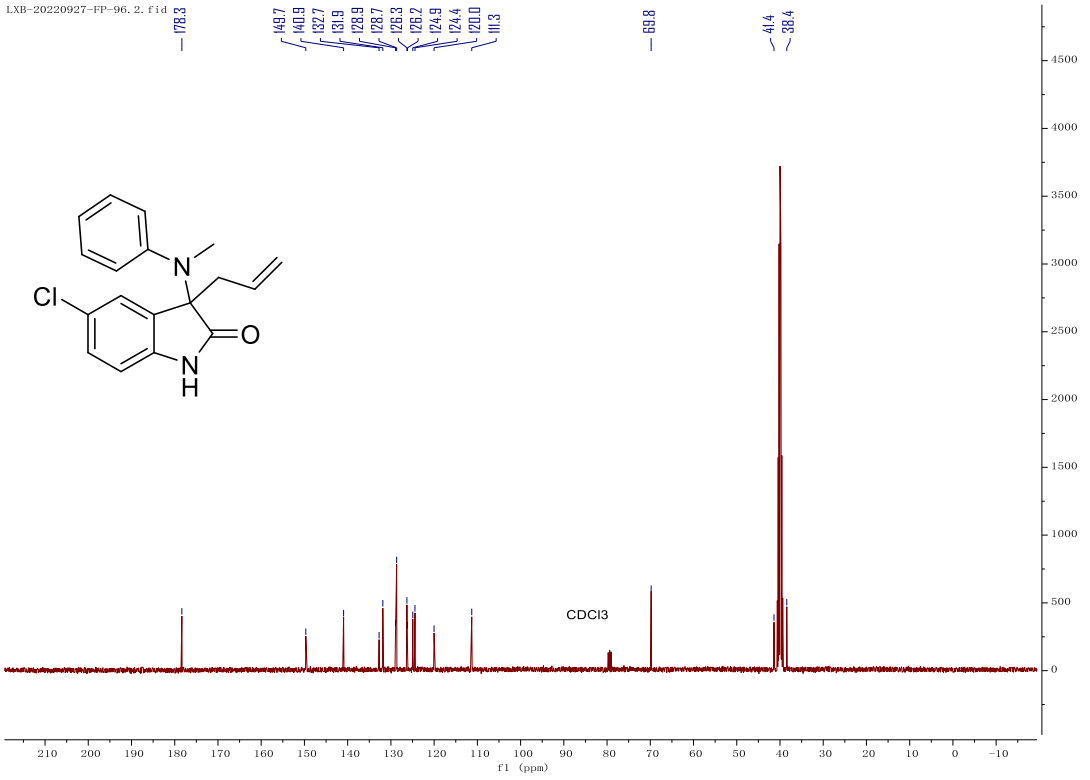


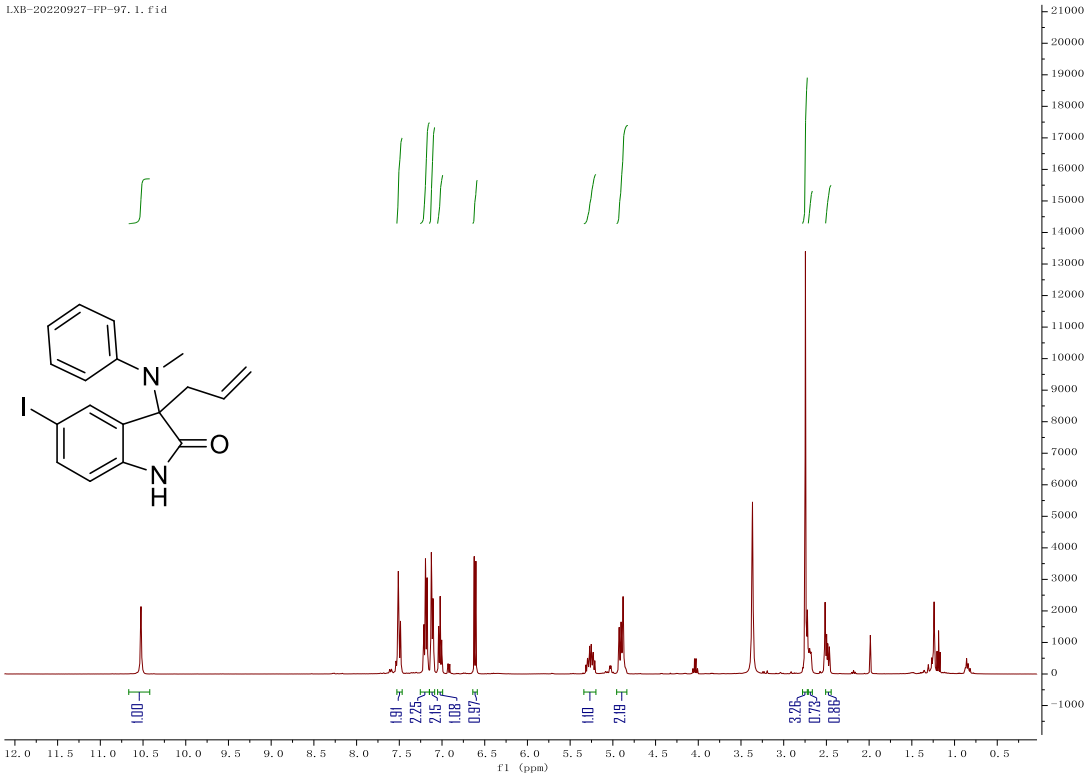


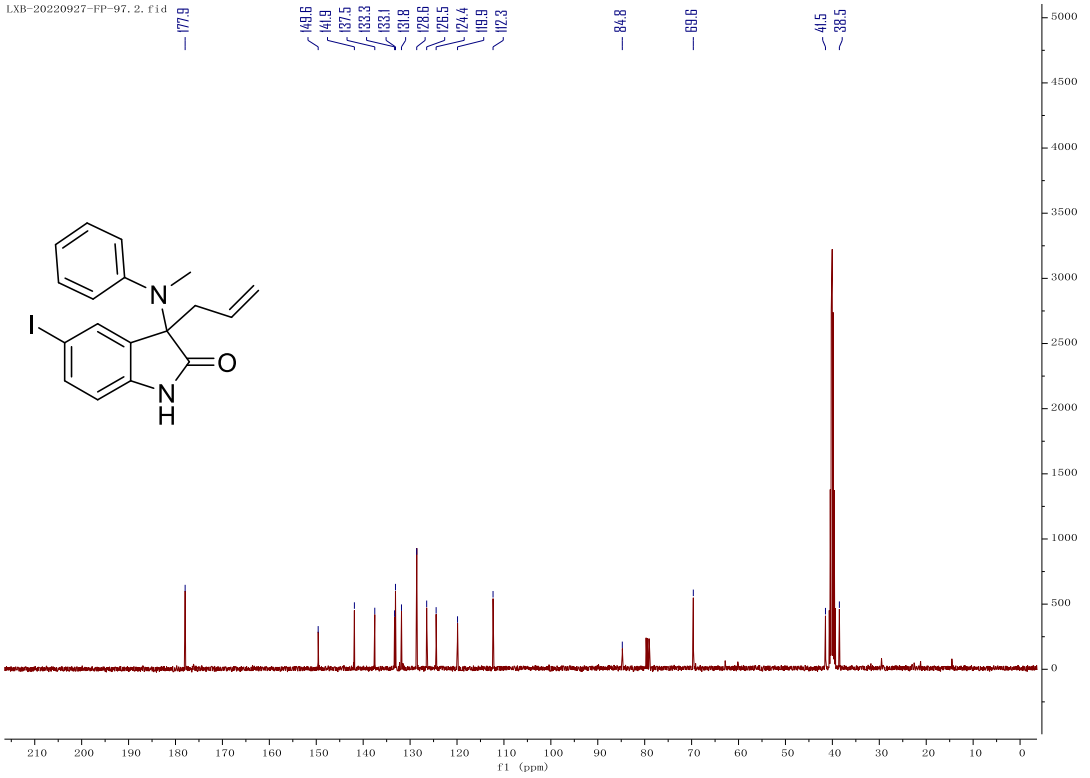

LXB-20220927-FP-95-2.1.fid
Me
N
O
N
H
1.00
4.15
1.10
0.93
1.01
0.97
1.04
1.05
1.01
2.96
1.82
3.00
f1 (ppm)

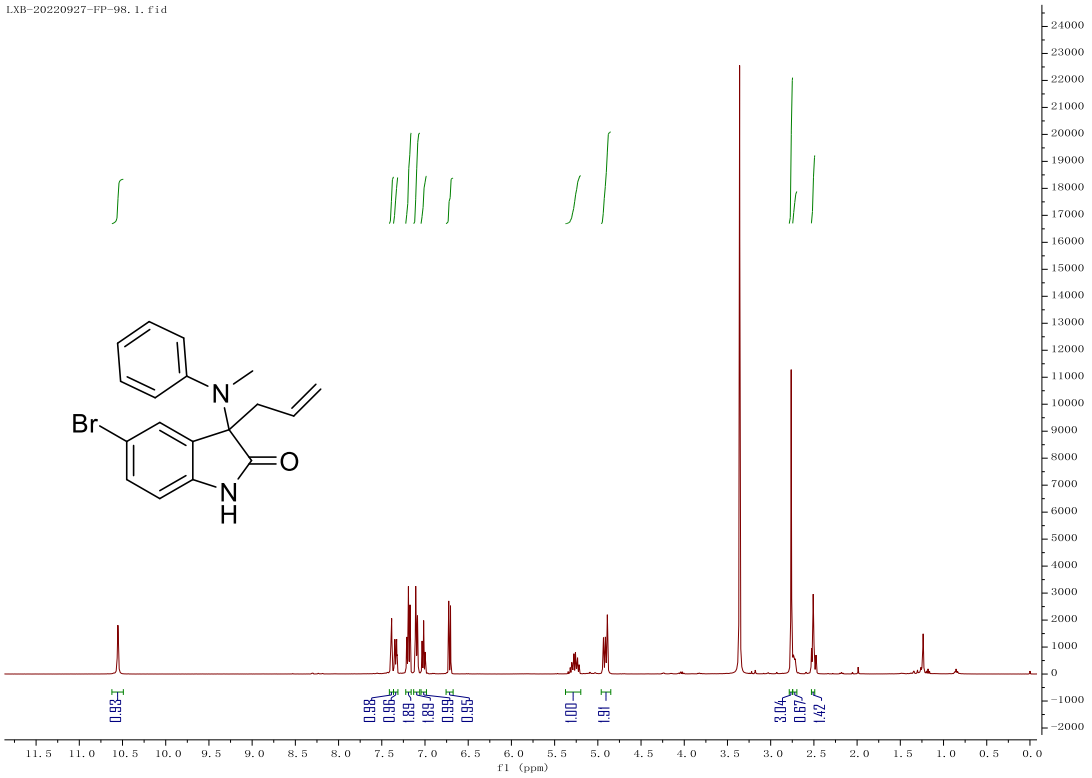

LXB-20220927-FP-95-2.2.fid
180.4
149.3
138.4
132.0
131.6
130.4
129.1
128.3
127.5
125.6
124.9
119.5
109.7
70.7
42.0
38.6
21.3
Me
N
O
N
H
f1 (ppm)

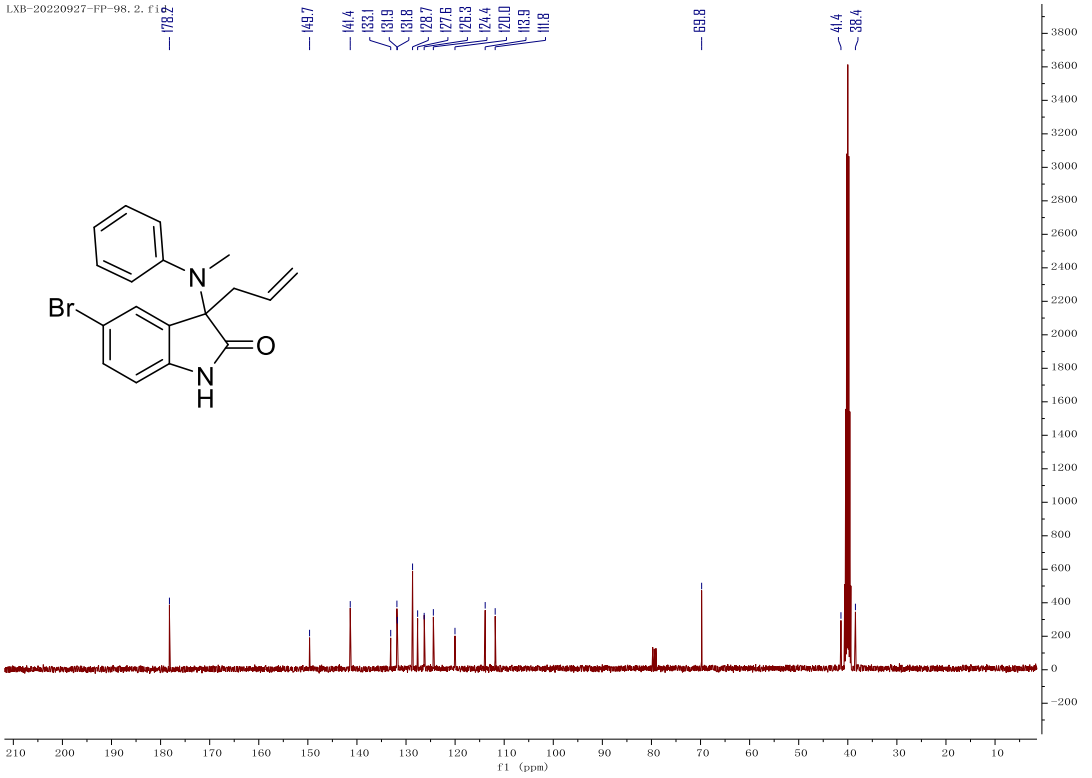

LXB-20220927-FP-96.1.fid
Cl
N
O
N
H
1.00
3.01
1.97
1.02
0.98
1.06
2.08
2.99
0.95
1.80
f1 (ppm)

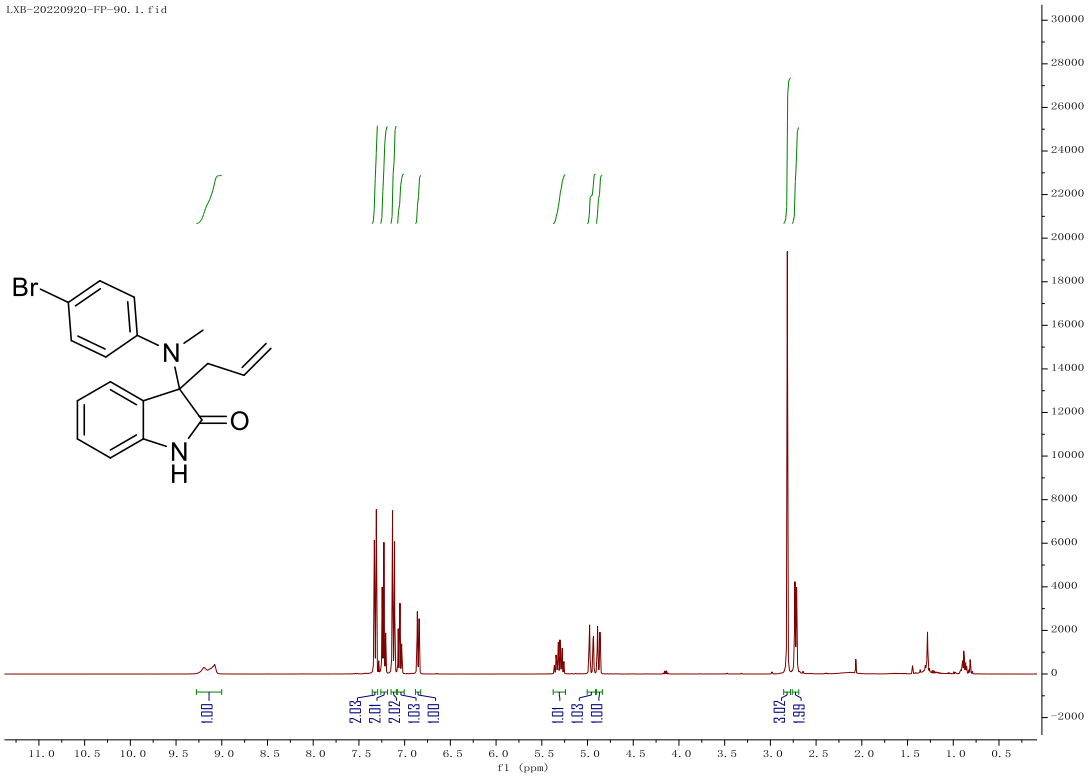

LXB-20220927-FP-96.2.fid
178.3
149.7
140.9
132.7
131.9
128.9
128.7
126.3
126.2
124.9
124.4
120.0
111.3
69.8
41.4
38.4
Cl
N
O
N
H
CDCl3
f1 (ppm)

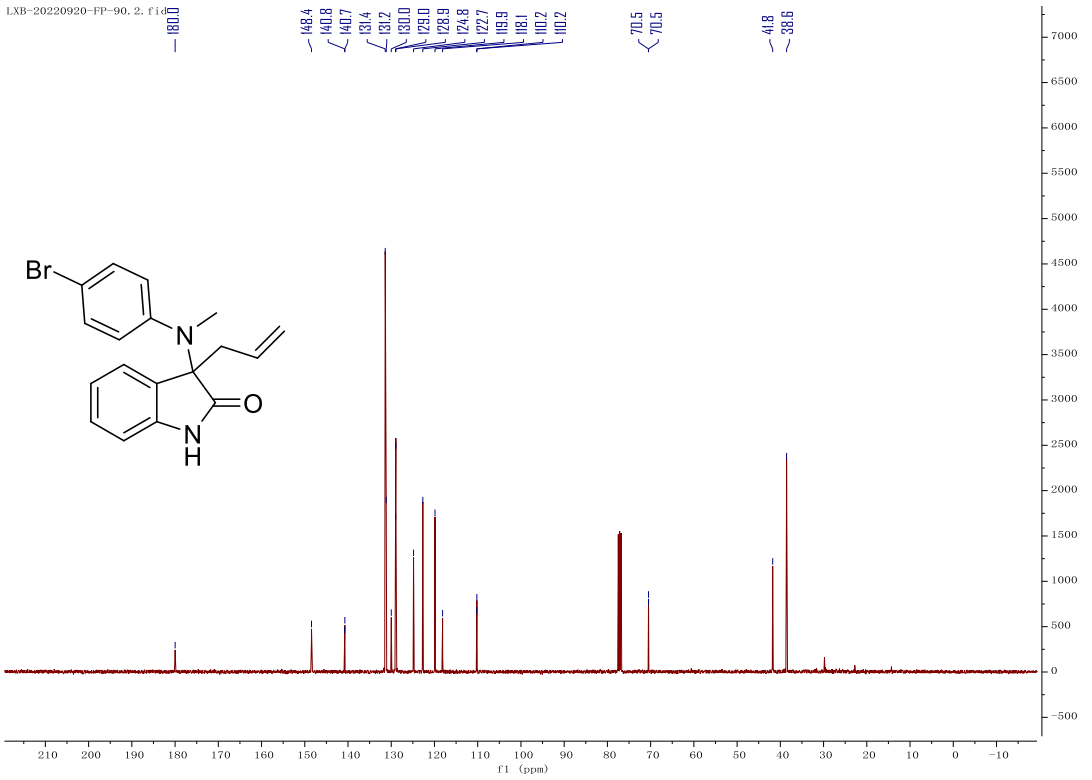

LXB-20220927-FP-97.1.fid
I
N
O
N
H
1.00
1.91
2.25
2.15
1.08
0.97
1.10
2.19
3.26
0.73
0.86
f1 (ppm)

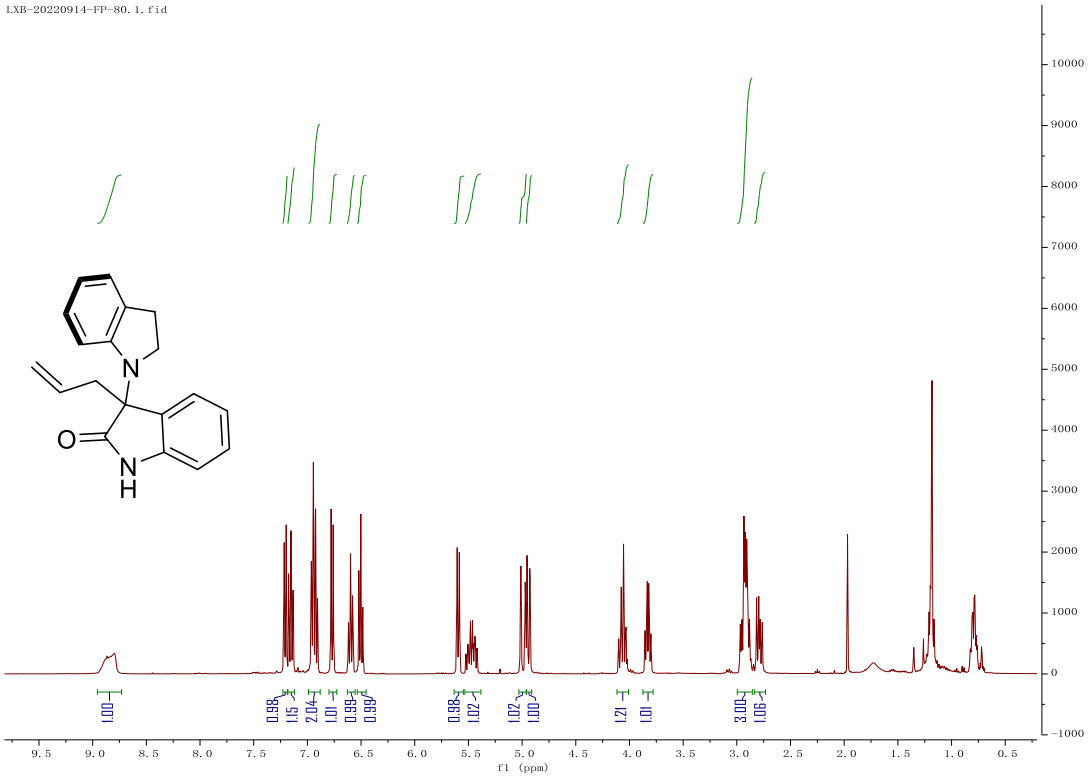

LXB-20220927-FP-97.2.fid
177.9
149.6
141.9
137.5
133.3
133.1
131.8
128.6
126.5
124.4
119.9
112.3
84.8
69.6
41.5
38.5
I
N
O
N
H
f1 (ppm)

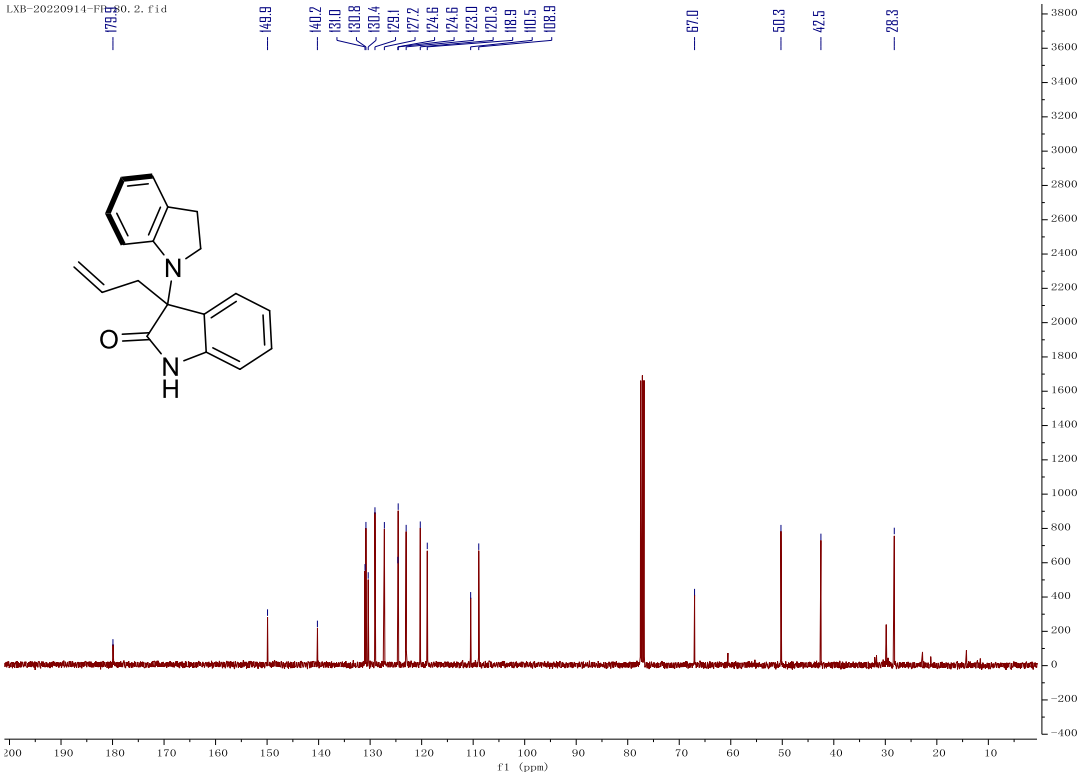

LXB-20220927-FP-98.1.fid
Br
N
O
N
H
0.93
0.98
0.96
1.89
1.89
0.99
0.95
1.00
1.91
3.04
0.67
1.42
f1 (ppm)

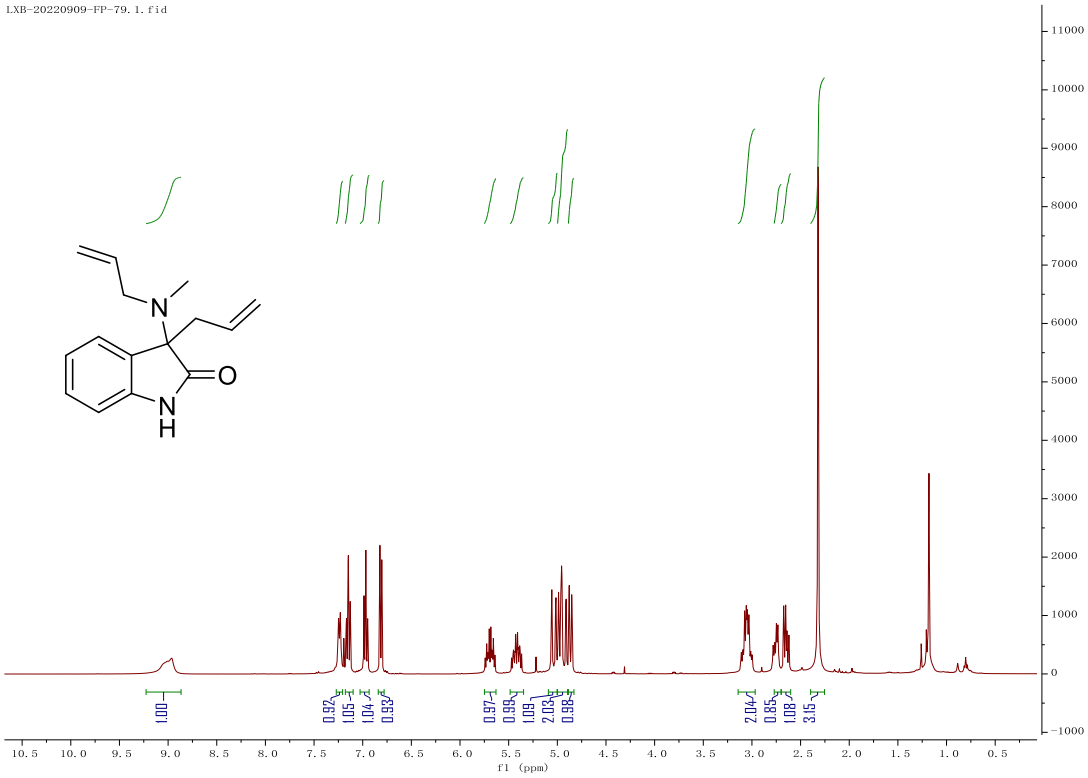

LXB-20220927-FP-98.2.fid
178.2
149.7
141.4
133.1
131.9
131.8
128.7
127.6
126.3
124.4
120.0
113.9
111.8
69.8
41.4
38.4
Br
N
O
N
H
f1 (ppm)

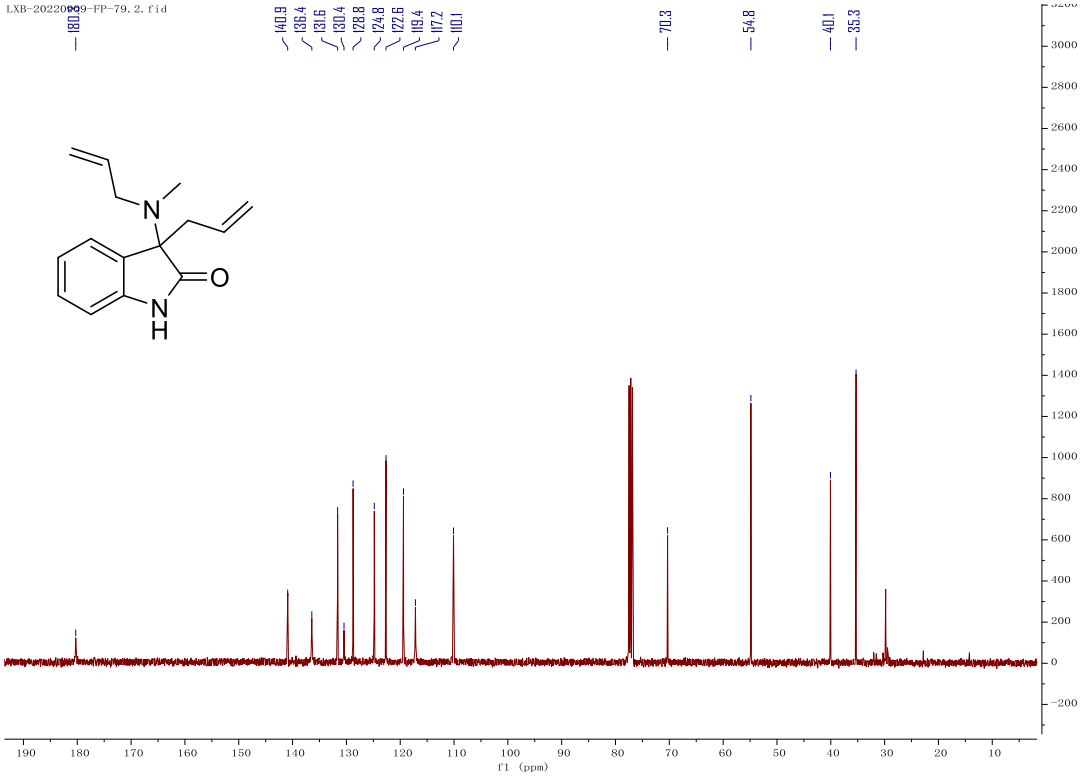

LXB-20220920-FP-90.1.fid
Br
N
O
N
H
1.00
2.03
2.01
2.02
1.03
1.00
1.01
1.03
1.00
3.02
1.99
f1 (ppm)

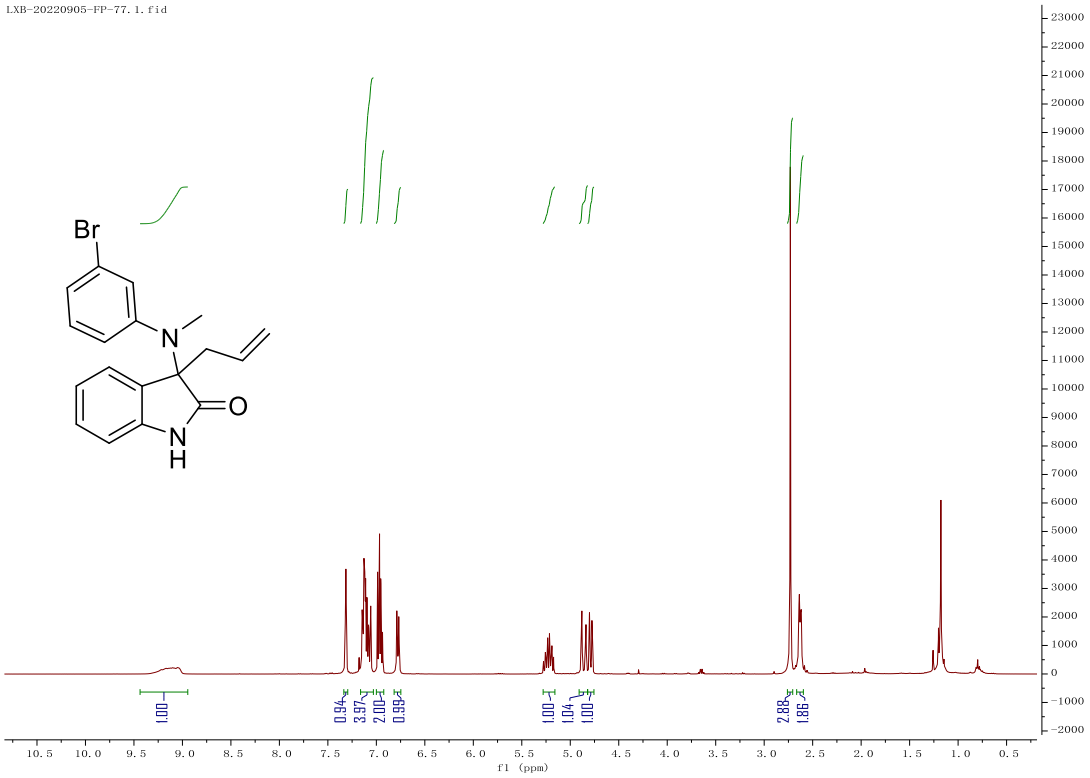

LXB-20220920-FP-90.2.fid
180.0
148.4
140.8
140.7
131.4
131.2
130.0
129.0
128.9
124.8
122.7
119.9
118.1
110.2
110.2
70.5
70.5
41.8
38.6
Br
N
O
N
H
f1 (ppm)

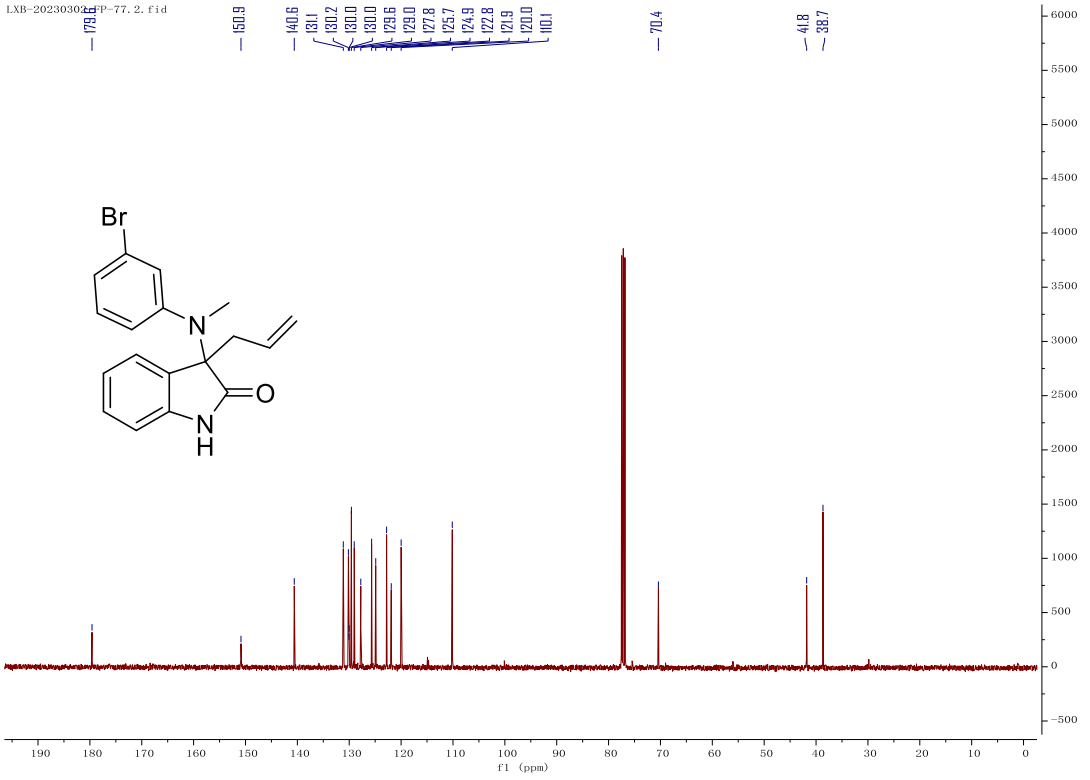

LXB-20220914-FP-80.1.fid
1.00
0.98
1.15
2.04
1.01
0.99
0.99
0.98
1.02
1.02
1.00
1.21
1.01
3.06
1.06
f1 (ppm)

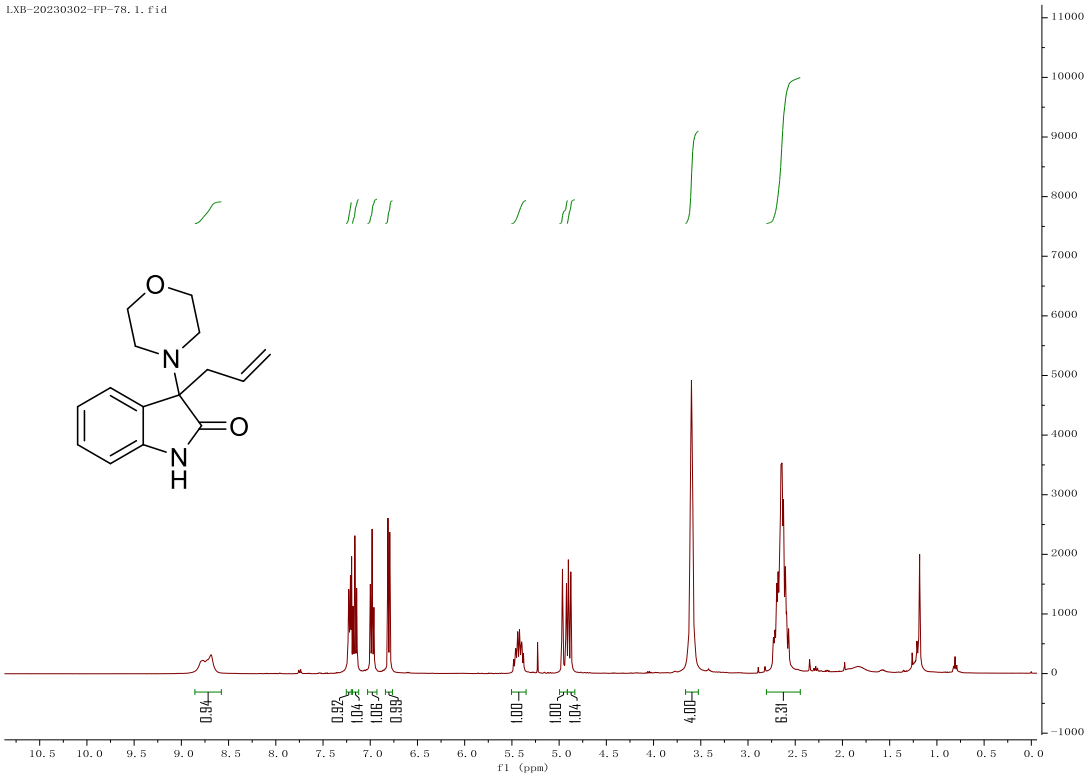

LXB-20220914-FP-80.2.fid
179.5
149.9
140.2
131.0
130.8
130.4
129.1
127.2
124.6
124.6
123.0
120.3
118.9
110.5
108.9
67.0
50.3
42.5
28.3
f1 (ppm)

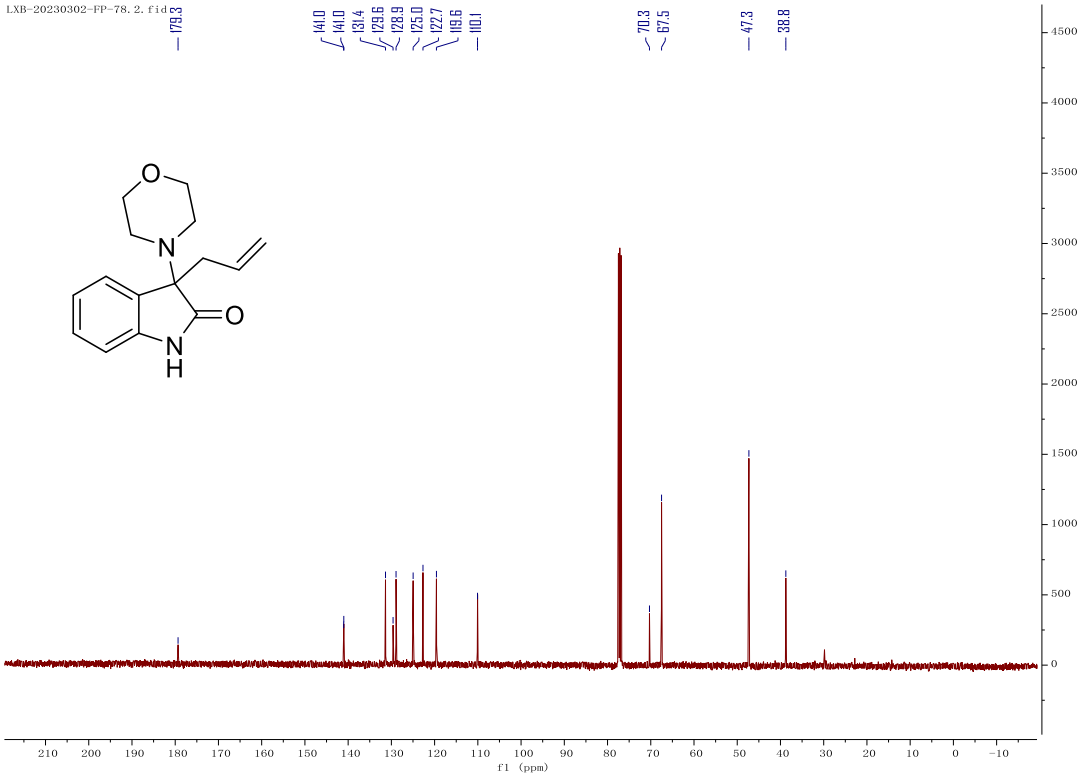

LXB-20220909-FP-79.1.fid
1.00
0.92
1.05
1.04
0.93
0.97
0.99
1.09
2.03
0.98
2.04
0.85
1.08
3.15
f1 (ppm)

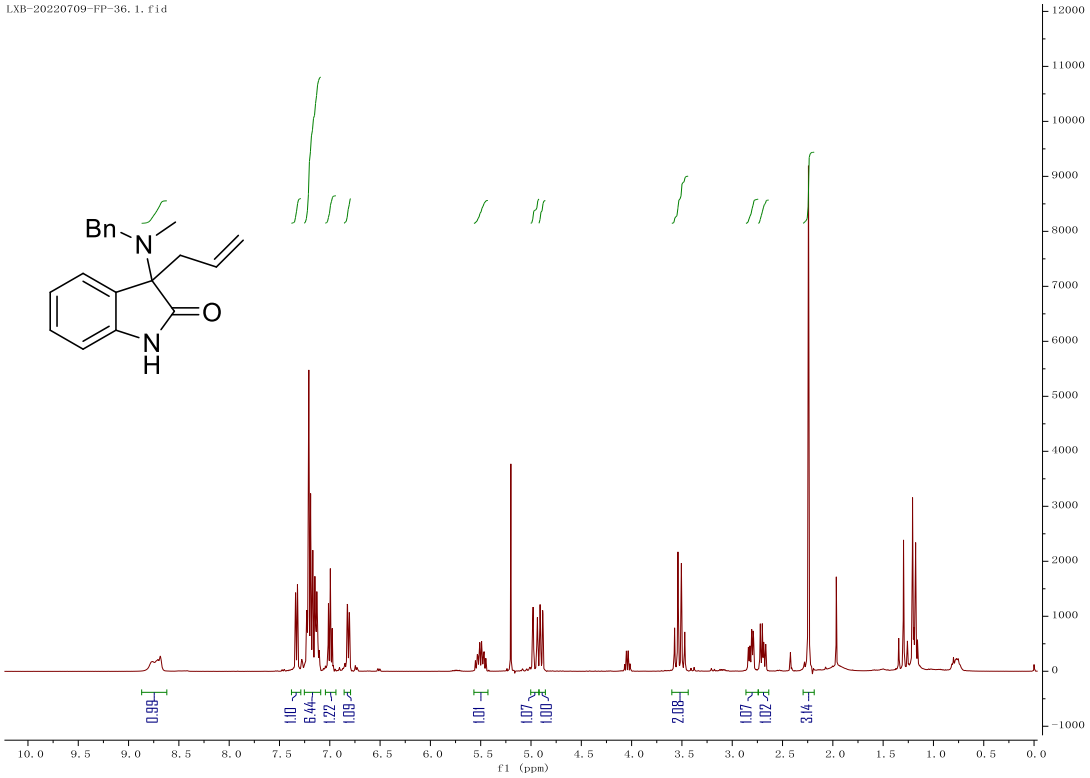

LXB-20220909-FP-79.2.fid
180.0
140.9
136.4
131.6
130.4
128.8
124.8
122.6
119.4
117.2
110.1
70.3
54.8
40.1
35.3
f1 (ppm)

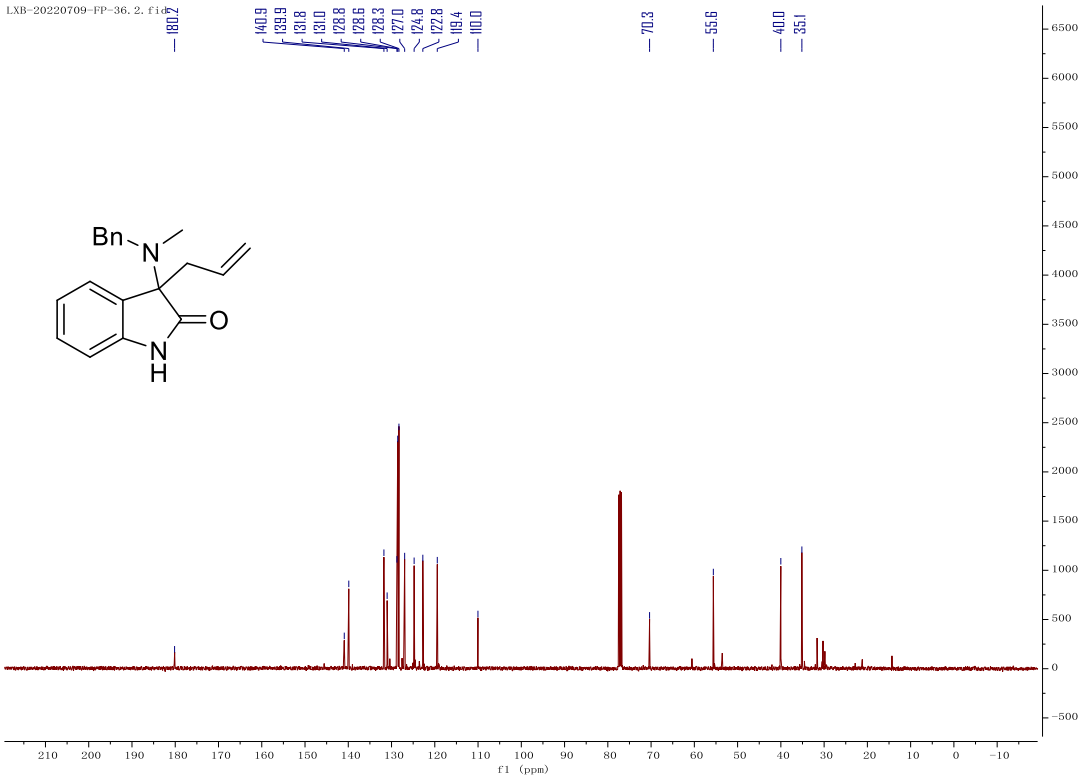

LXB-20220905-FP-77.1.fid
Br
N
O
N
H
1.00
0.94
3.97
2.00
0.99
1.00
1.04
1.00
2.88
1.86
f1 (ppm)

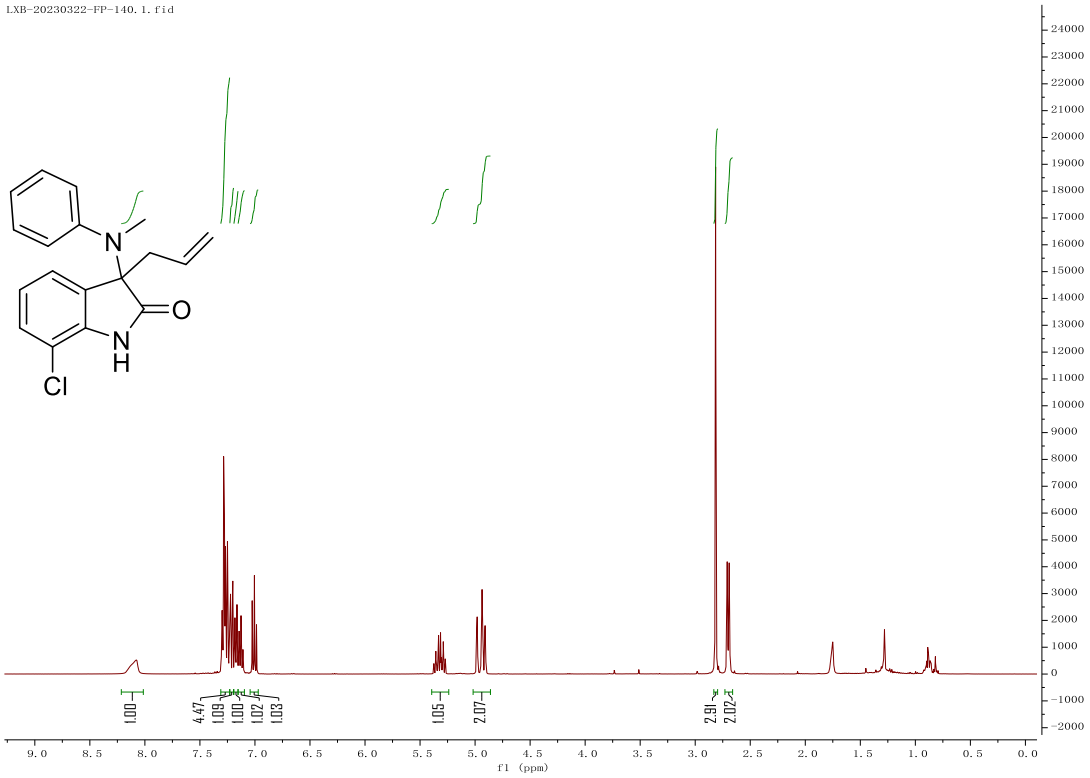

LXB-20230302-FP-77.2.fid
179.6
150.9
140.6
131.1
130.2
130.0
130.0
129.6
129.0
127.8
125.7
124.9
122.8
121.9
120.0
110.1
70.4
41.8
38.7
Br
N
O
N
H
f1 (ppm)

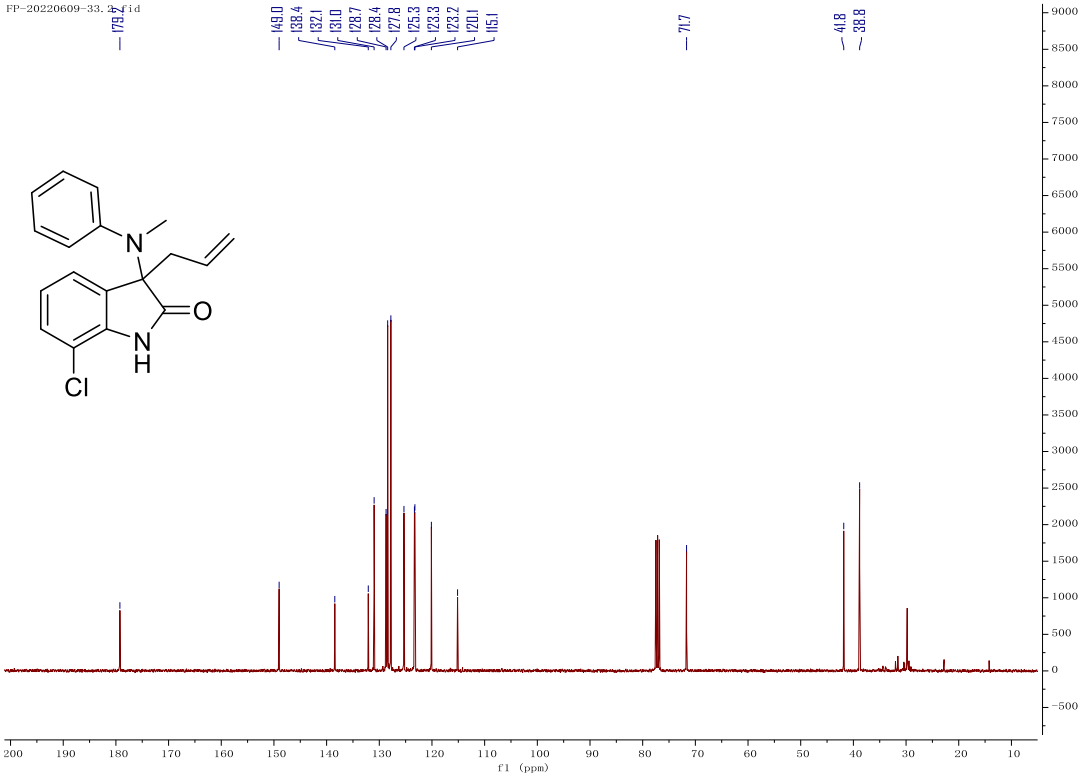

LXB-20230302-FP-78.1.fid
0.94
0.92
1.04
1.06
0.99
1.00
1.00
1.04
4.00
6.31
f1 (ppm)

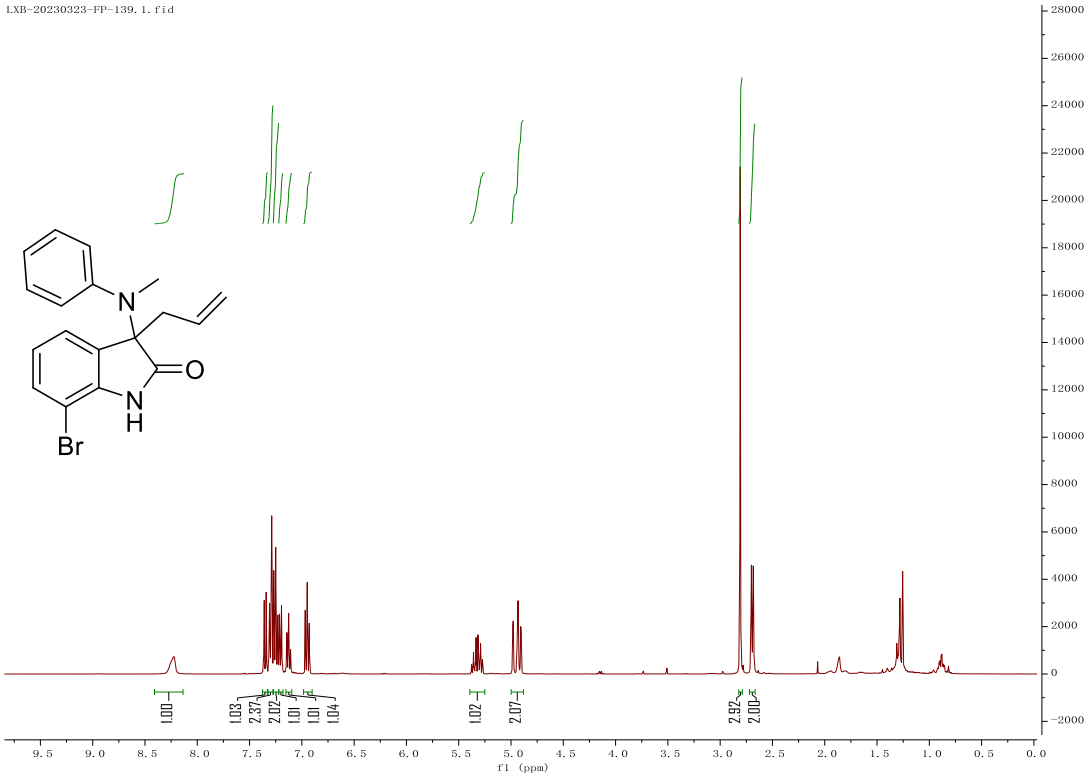

LXB-20230302-FP-78.2.fid
179.3
141.0
141.0
131.4
129.6
128.9
125.0
122.7
119.6
110.1
70.3
67.5
47.3
38.8
f1 (ppm)

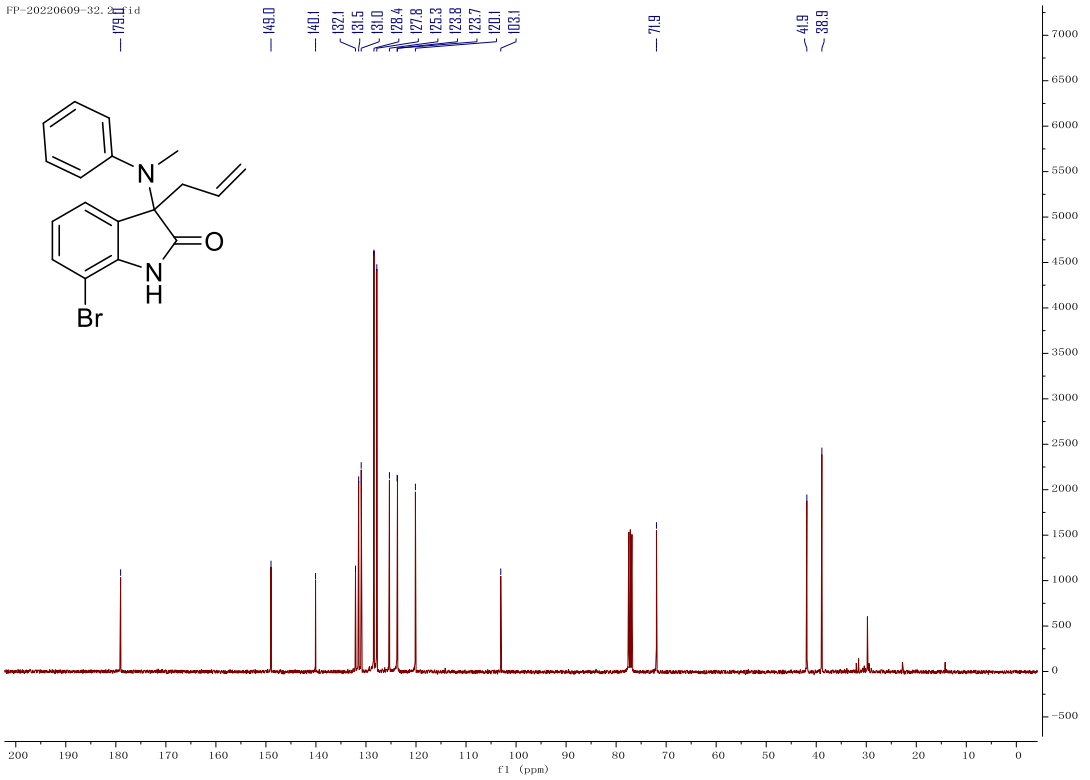

LXB-20220709-FP-36.1.fid
Bn
N
O
N
H
0.99
1.10
6.44
1.22
1.09
1.01
1.07
1.00
2.08
1.07
1.02
3.14
f1 (ppm)

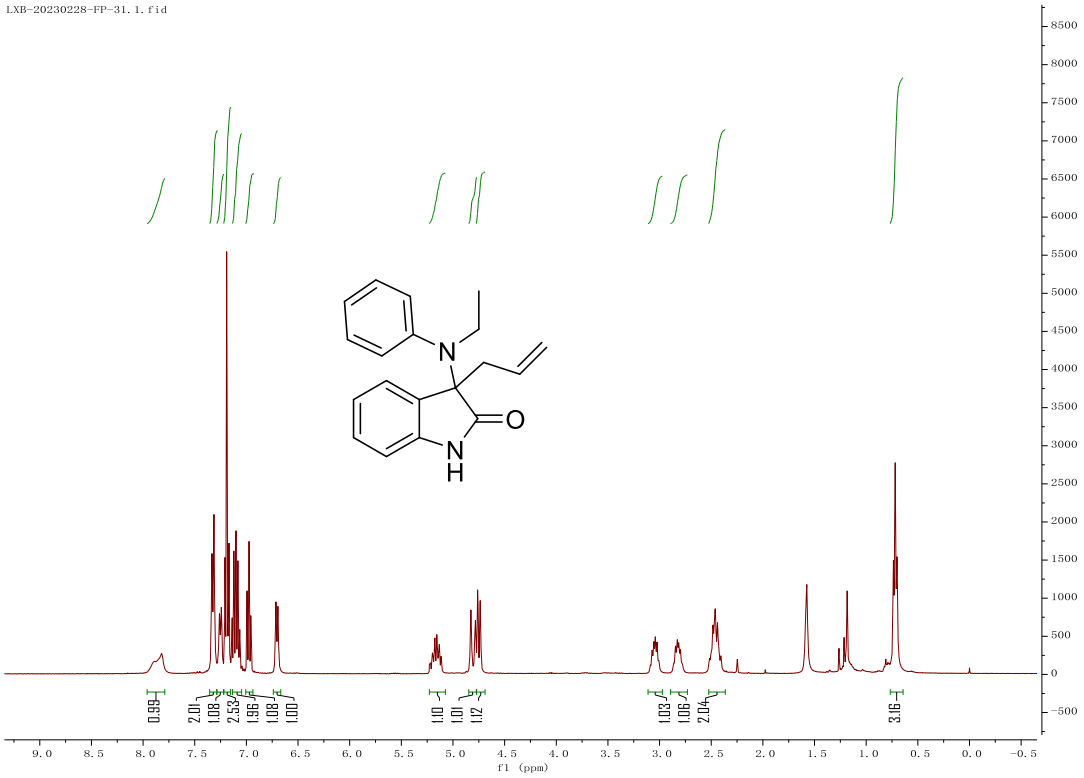

LXB-20220709-FP-36.2.fid
180.2
140.9
139.9
131.8
131.0
128.8
128.6
128.3
127.0
124.8
122.8
119.4
110.0
70.3
55.6
40.0
35.1
Bn
N
O
N
H
f1 (ppm)

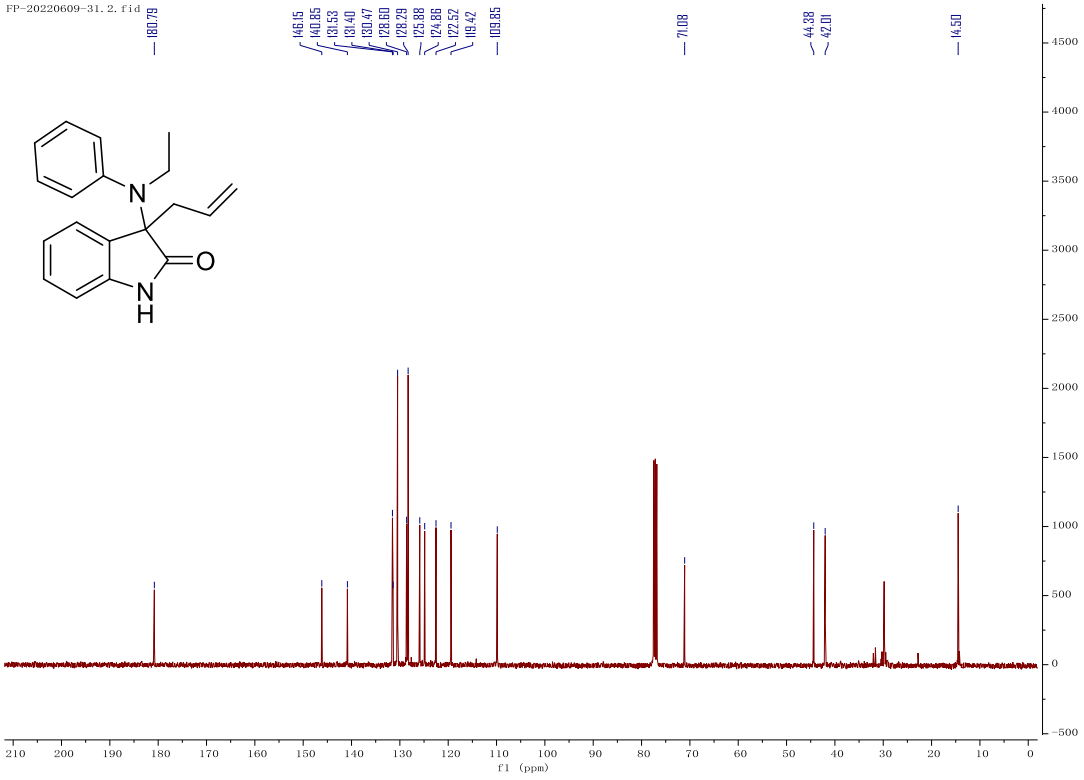

LXB-20230322-FP-140.1.fid
N
O
N
H
Cl
1.00
4.47
1.09
1.00
1.02
1.03
1.05
2.07
2.91
2.02
f1 (ppm)

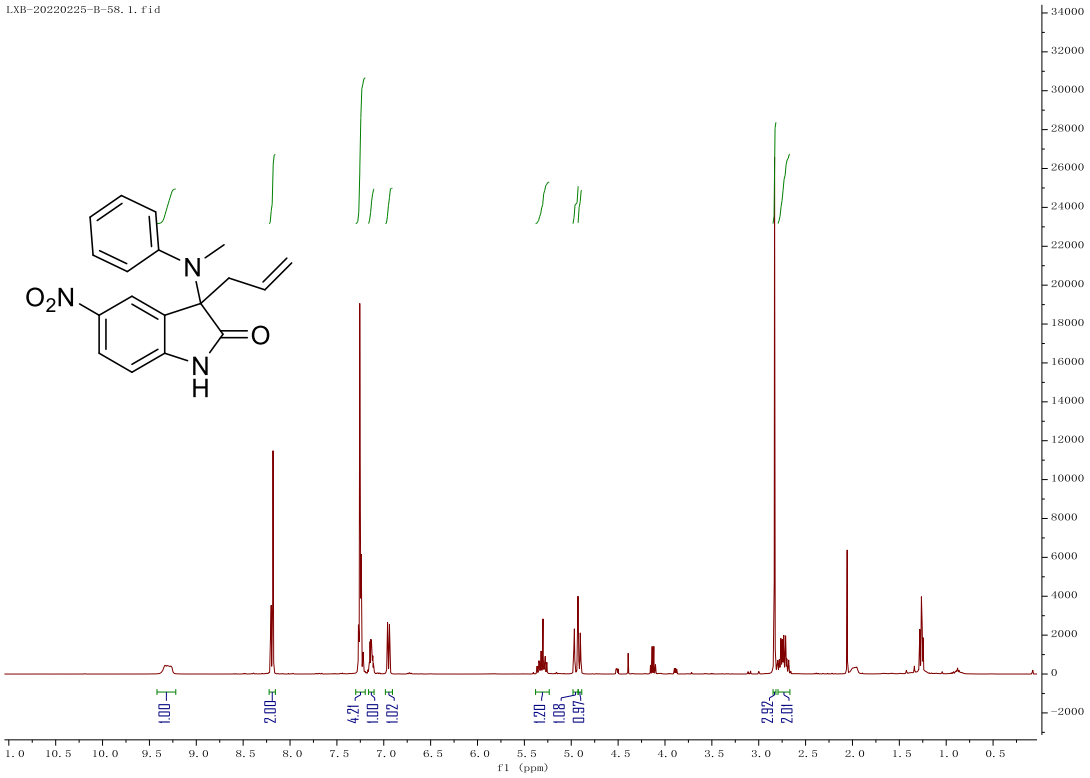

FP-20220609-33.3.fid
179.2
149.0
138.4
132.1
131.0
128.7
128.4
127.8
125.3
123.3
123.2
120.1
115.1
71.7
41.8
38.8
N
O
N
H
Cl
f1 (ppm)

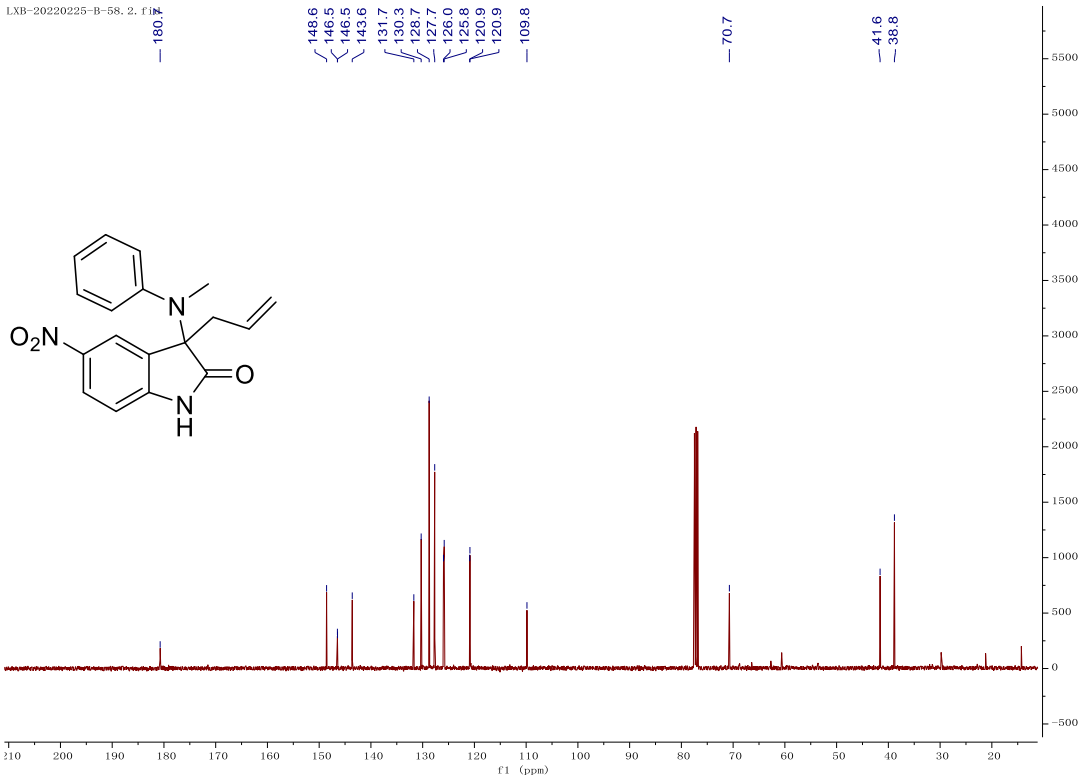

LXB-20230323-FP-139.1.fid
N
O
N
H
Br
1.00
1.03
2.37
2.02
1.01
1.01
1.04
1.02
2.07
2.92
2.00
f1 (ppm)

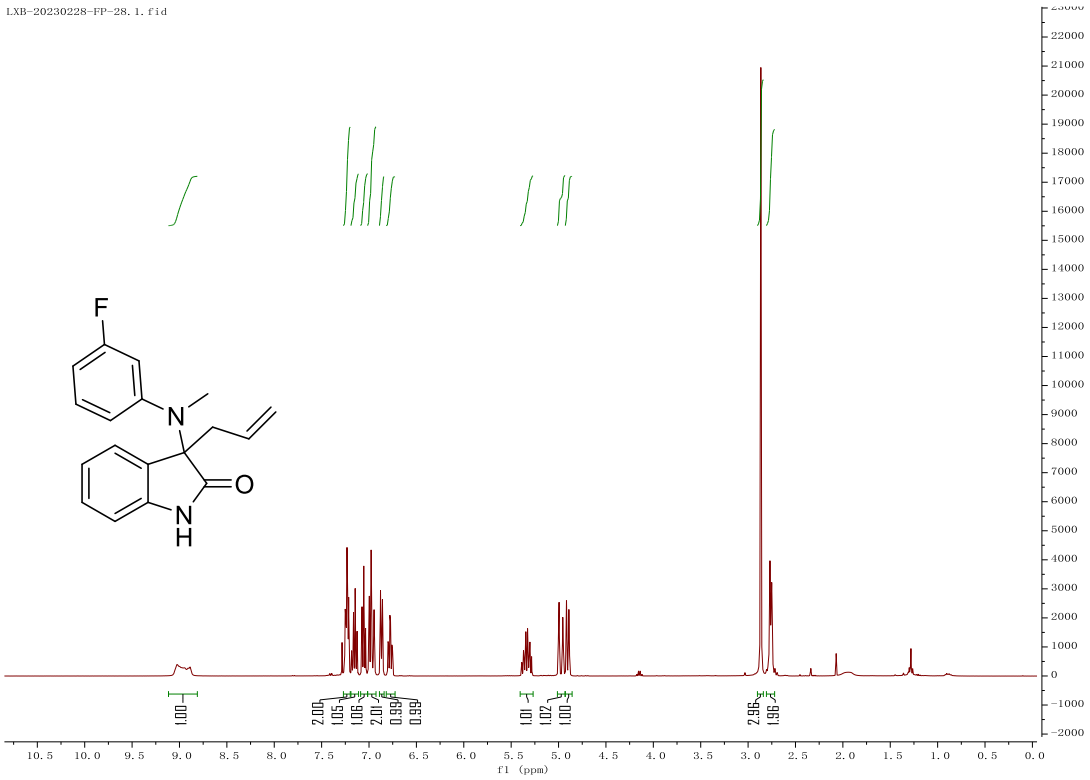

FP-20220609-32.2.fid
179.4
149.0
140.1
132.1
131.5
131.0
128.4
127.8
125.3
123.8
123.7
120.1
103.1
71.9
41.9
38.9
N
O
N
H
Br
f1 (ppm)

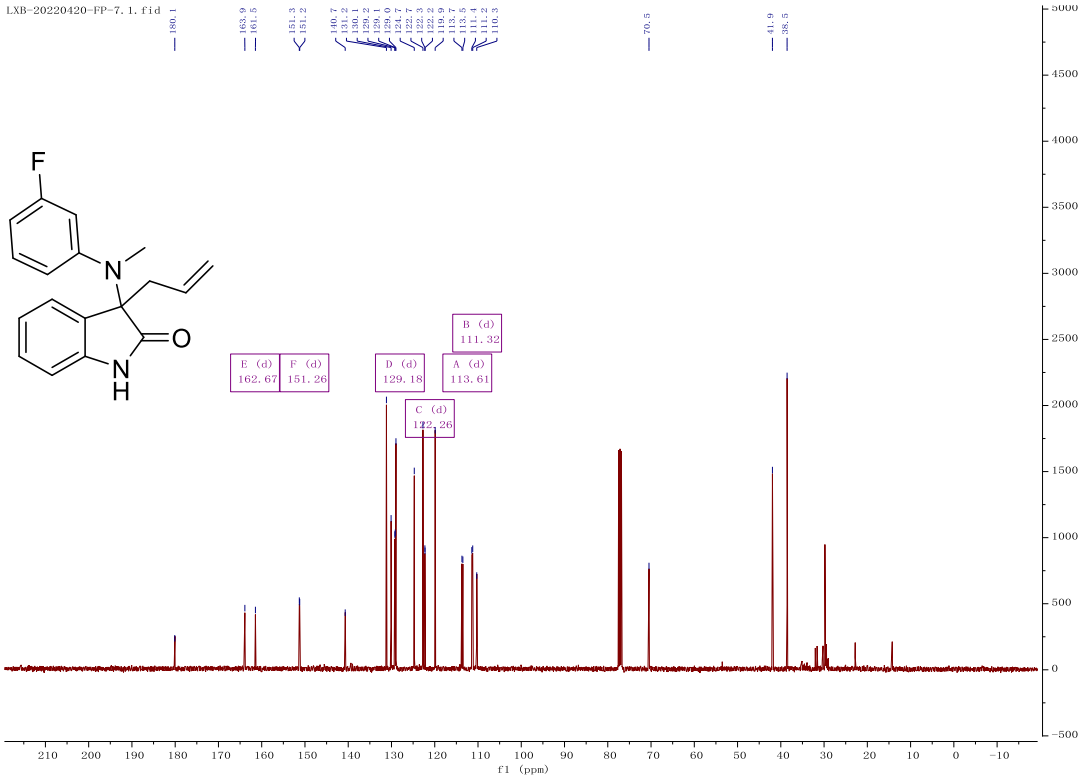

LXB-20230228-FP-31.1.fid
0.99
2.01
1.08
2.52
1.96
1.08
1.00
1.10
1.01
1.12
1.03
1.06
2.04
3.16
f1 (ppm)

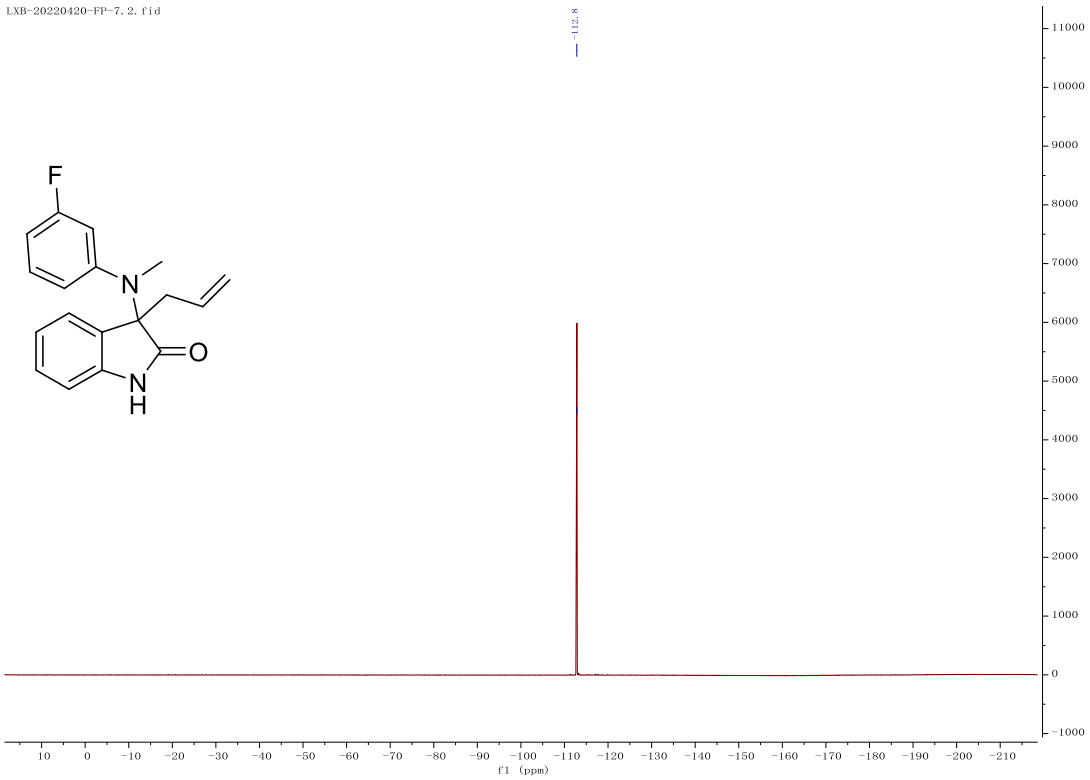

FP-20220609-31.2.fid
180.79
146.15
140.85
131.53
131.40
130.47
128.60
128.29
125.88
124.86
122.52
119.42
109.85
71.08
44.38
42.01
14.50
f1 (ppm)

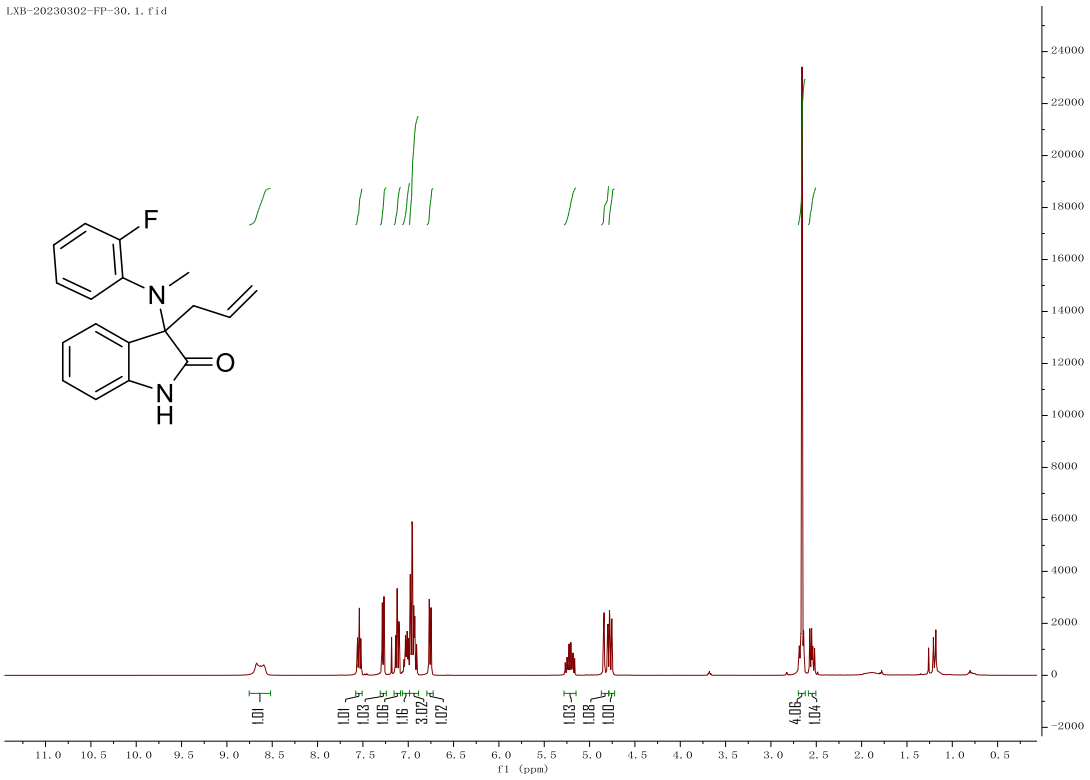

LXB-20220225-B-58.1.fid
1.00
2.00
4.21
1.00
1.02
1.20
1.08
0.97
2.92
2.01
f1 (ppm)

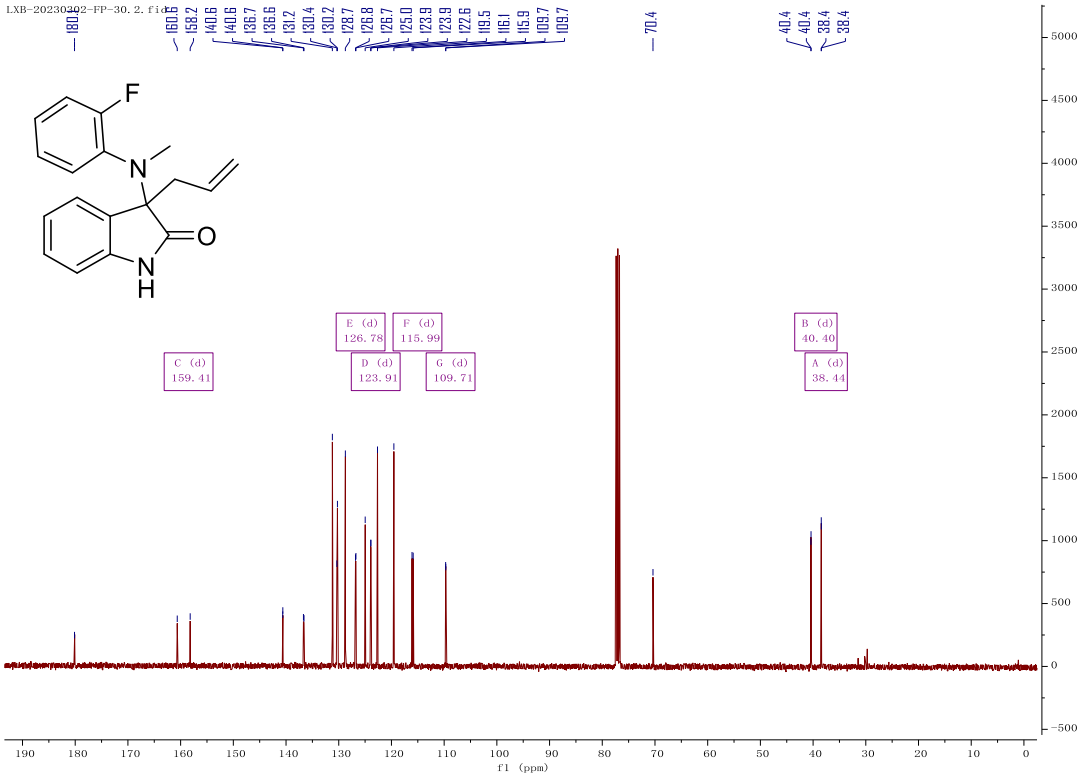

LXB-20220225-B-58.2.fid
180.4
148.6
146.5
146.5
143.6
131.7
130.3
128.7
127.7
126.0
125.8
120.9
120.9
109.8
70.7
41.6
38.8
f1 (ppm)

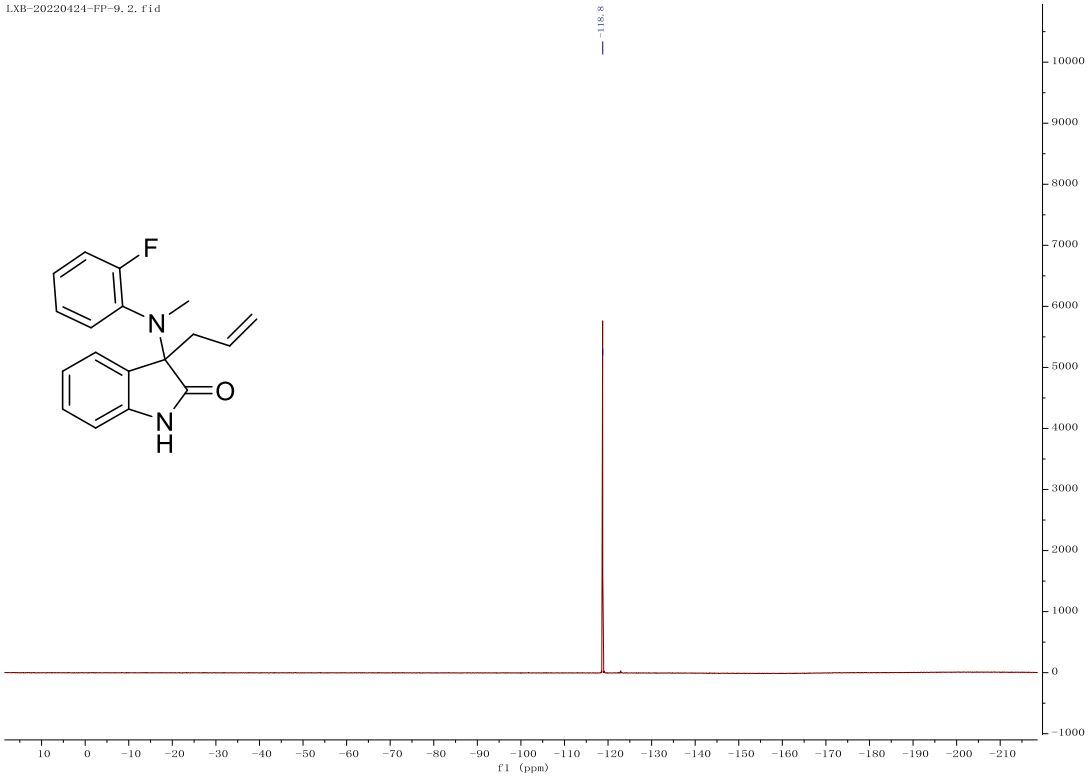

LXB-20230228-FP-28.1.fid
F
N
O
N
H
1.00
2.00
1.05
1.06
2.01
0.99
0.99
1.01
1.02
1.00
2.96
1.96
f1 (ppm)

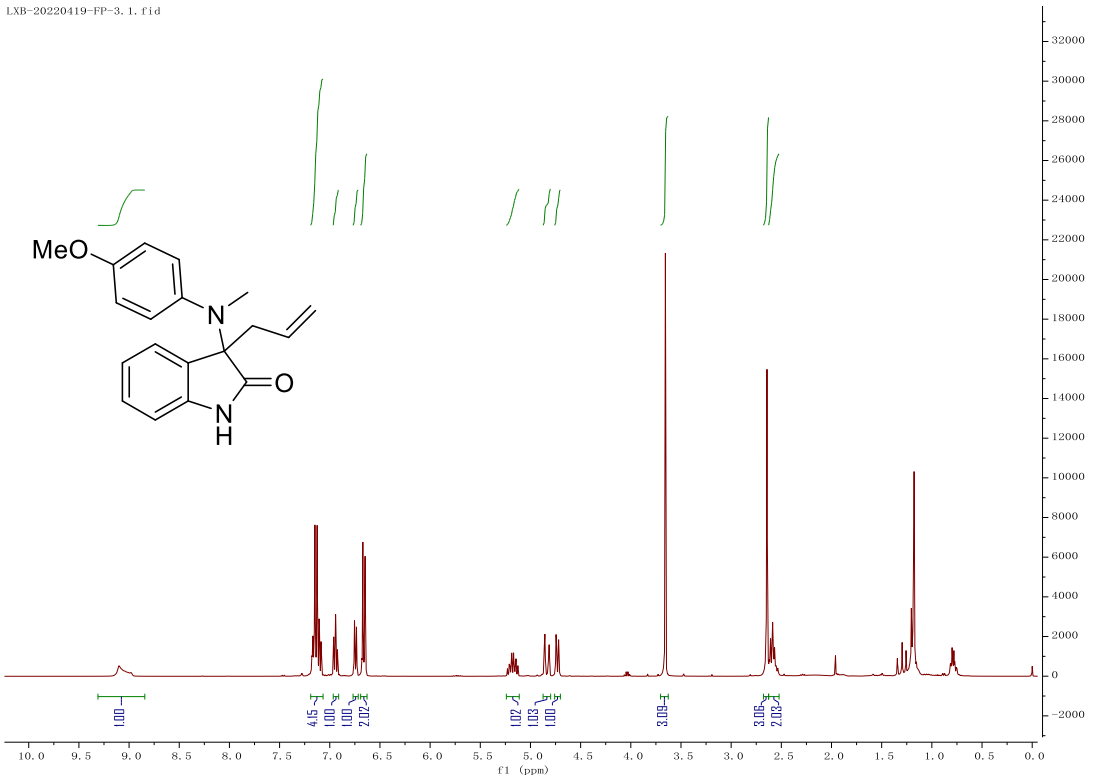

LXB-20220420-FP-7.1.fid
180.1
163.9
161.5
151.3
151.2
140.7
131.2
130.1
129.2
129.1
129.0
124.7
122.7
122.3
122.2
119.9
113.7
113.5
111.4
111.2
110.3
70.5
41.9
38.5
F
N
O
N
H
E (d)
162.67
F (d)
151.26
D (d)
129.18
C (d)
122.26
A (d)
113.61
B (d)
111.32
f1 (ppm)

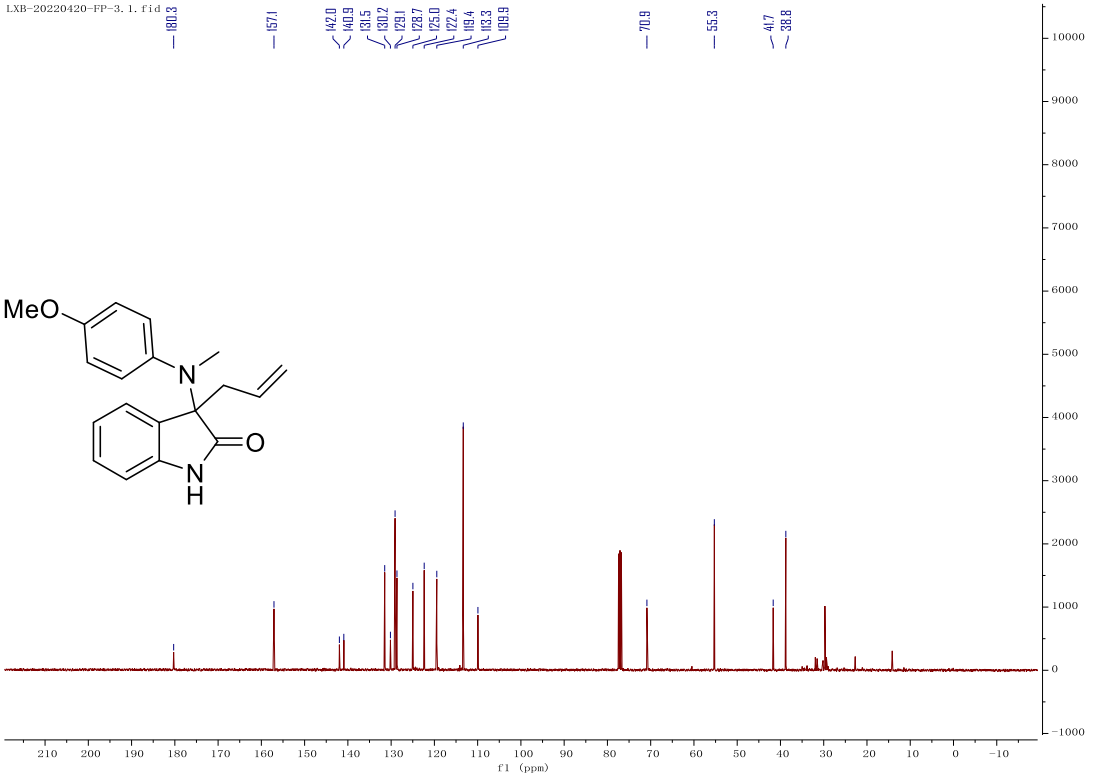

LXB-20220420-FP-7.2.fid
-112.8
F
N
O
N
H
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

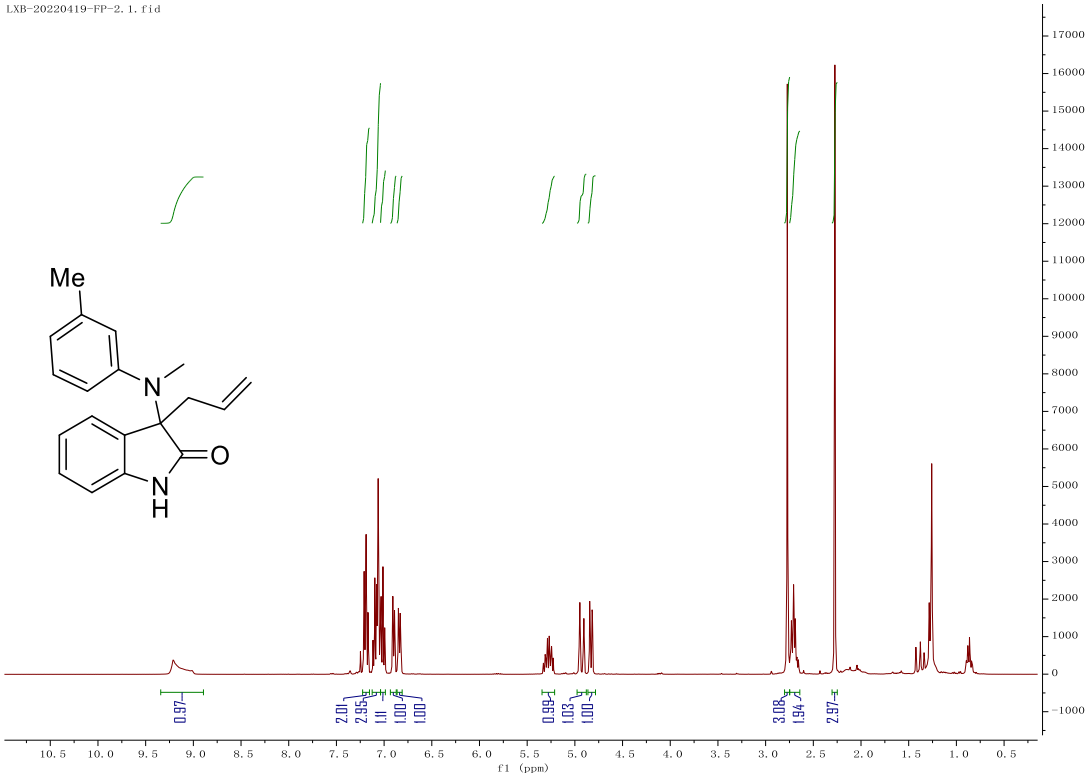

LXB-20230302-FP-30.1.fid
F
N
O
N
H
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
1.01
1.01
1.03
1.06
1.16
3.02
1.02
1.03
1.08
1.00
4.06
1.04
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

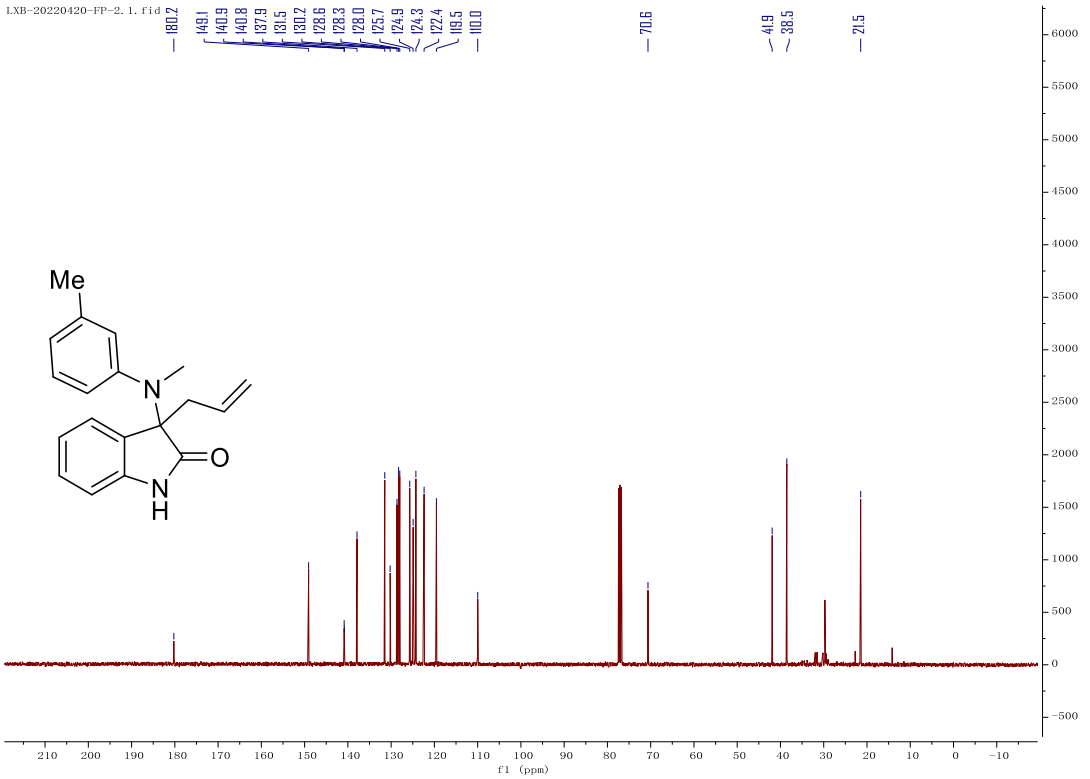

LXB-20230202-FP-30.2.fid
180.0
160.6
158.2
140.6
140.6
136.7
136.6
131.2
130.4
130.2
128.7
126.8
126.7
125.0
123.9
123.9
122.6
119.5
116.1
115.9
109.7
109.7
70.4
40.4
40.4
38.4
38.4
C (d)
159.41
E (d)
126.78
D (d)
123.91
F (d)
115.99
G (d)
109.71
B (d)
40.40
A (d)
38.44
F
N
O
N
H
f1 (ppm)

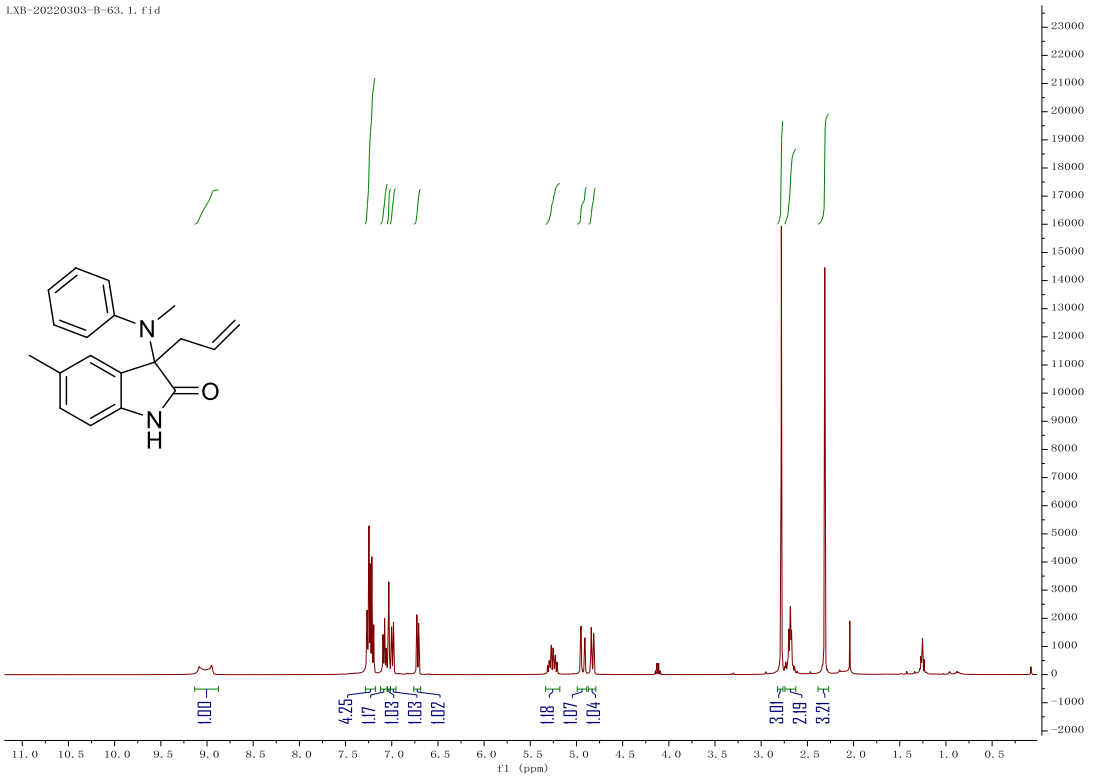

LXB-20220424-FP-9.2.fid
-118.8
F
N
O
N
H
f1 (ppm)

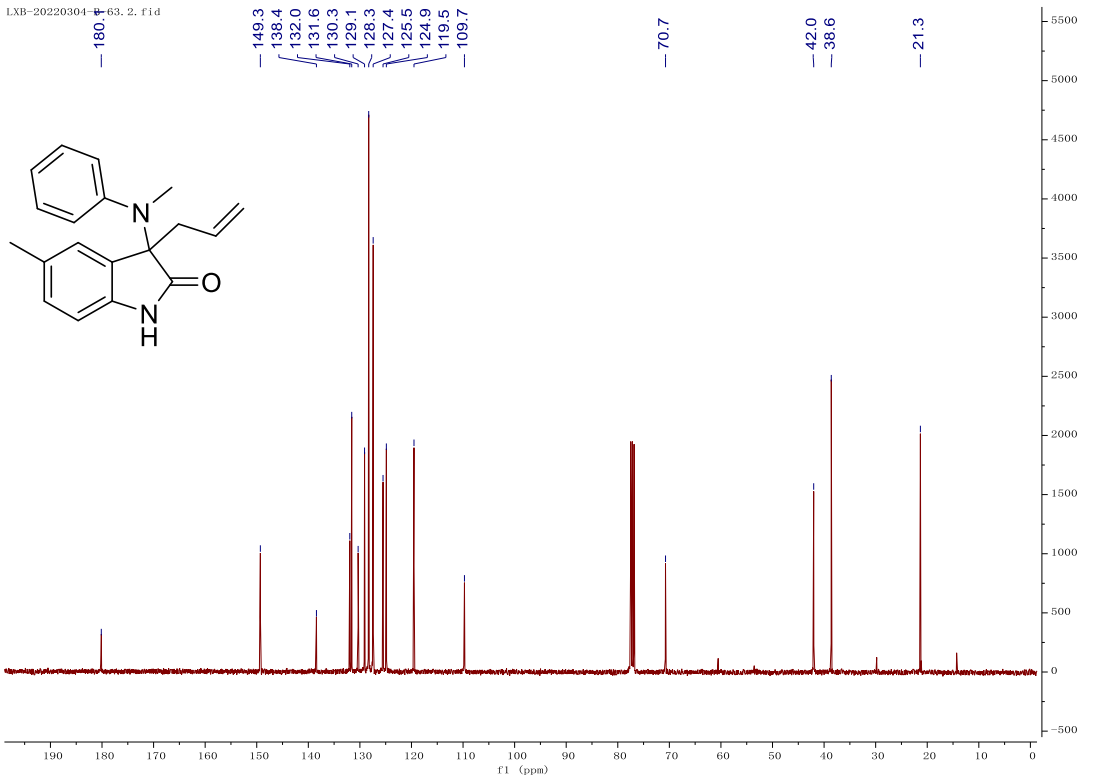

LXB-20220419-FP-3.1.fid
MeO
N
O
N
H
1.00
4.15
1.00
1.00
2.02
1.02
1.03
1.00
3.09
3.06
2.03
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
32000 30000 28000 26000 24000 22000 20000 18000 16000 14000 12000 10000 8000 6000 4000 2000 0 −2000

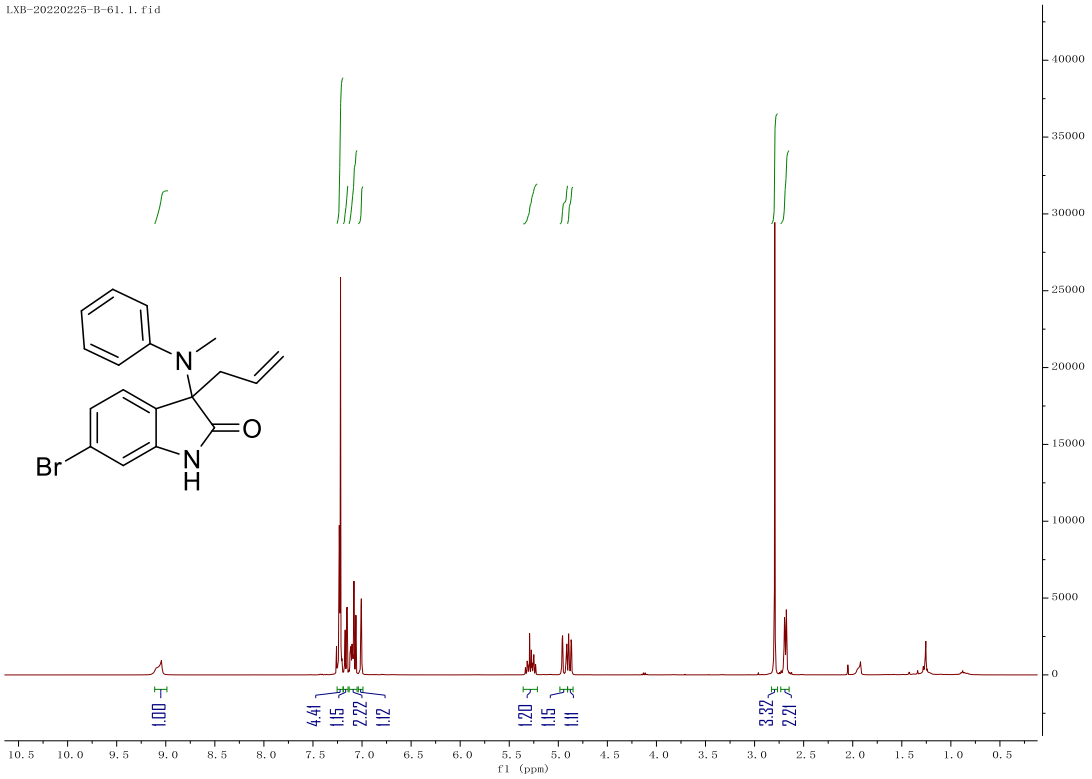

LXB-20220420-FP-3.1.fid
180.3
157.1
142.0
140.9
131.5
130.2
129.1
128.7
125.0
122.4
119.4
113.3
109.9
70.9
55.3
41.7
38.8
MeO
N
O
N
H
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)
10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 −1000

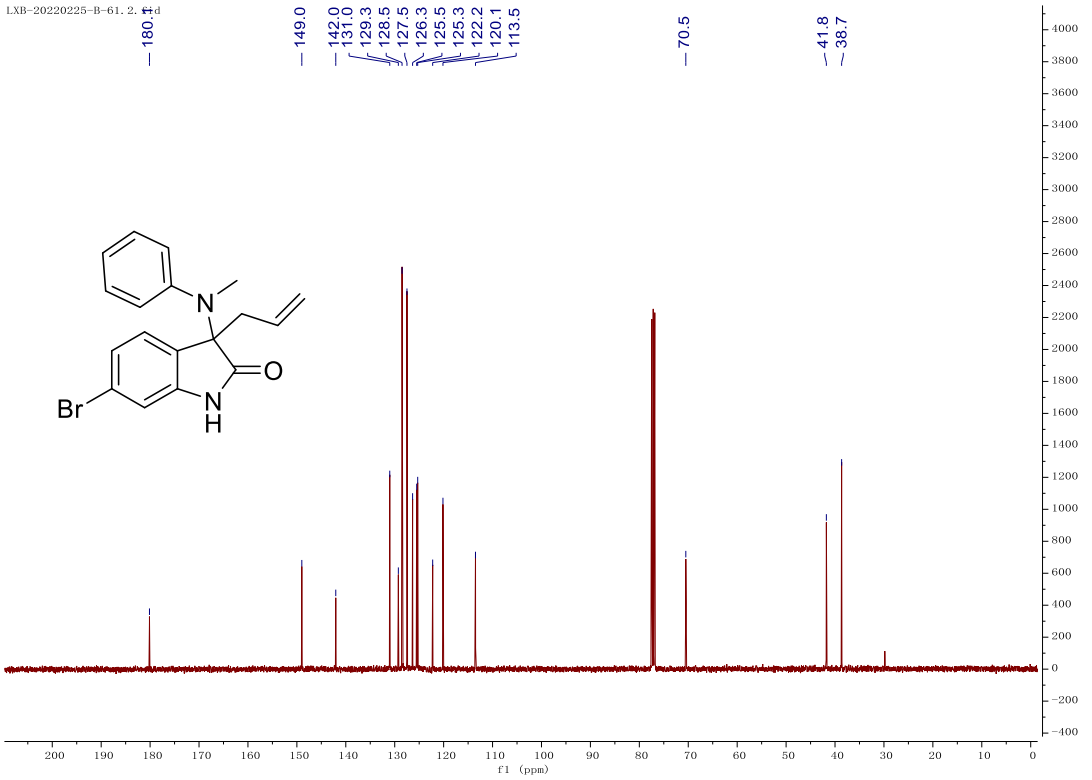

LXB-20220419-FP-2.1.fid
Me
N
O
N
H
0.97
2.01
2.95
1.11
1.00
1.00
0.99
1.03
1.00
3.08
1.94
2.97
f1 (ppm)

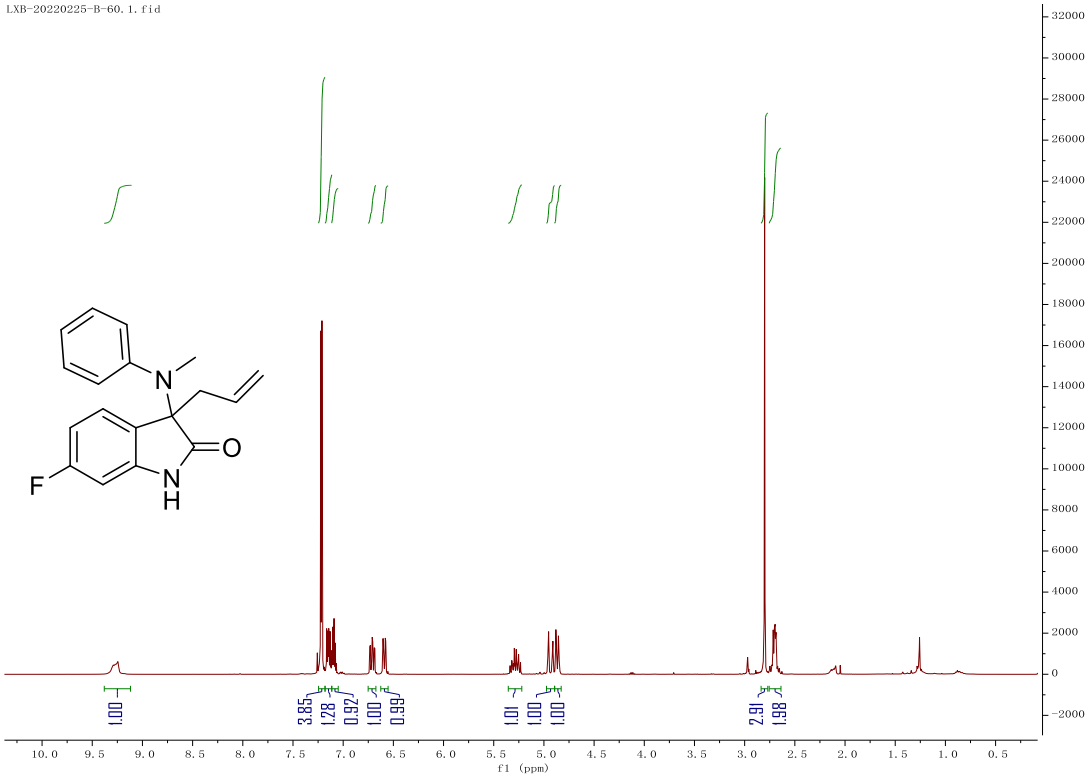

LXB-20220420-FP-2.1.fid
180.2
149.1
140.9
140.8
137.9
131.5
130.2
128.6
128.3
128.0
125.7
124.9
124.3
122.4
119.5
110.0
70.6
41.9
38.5
21.5
Me
N
O
N
H
f1 (ppm)

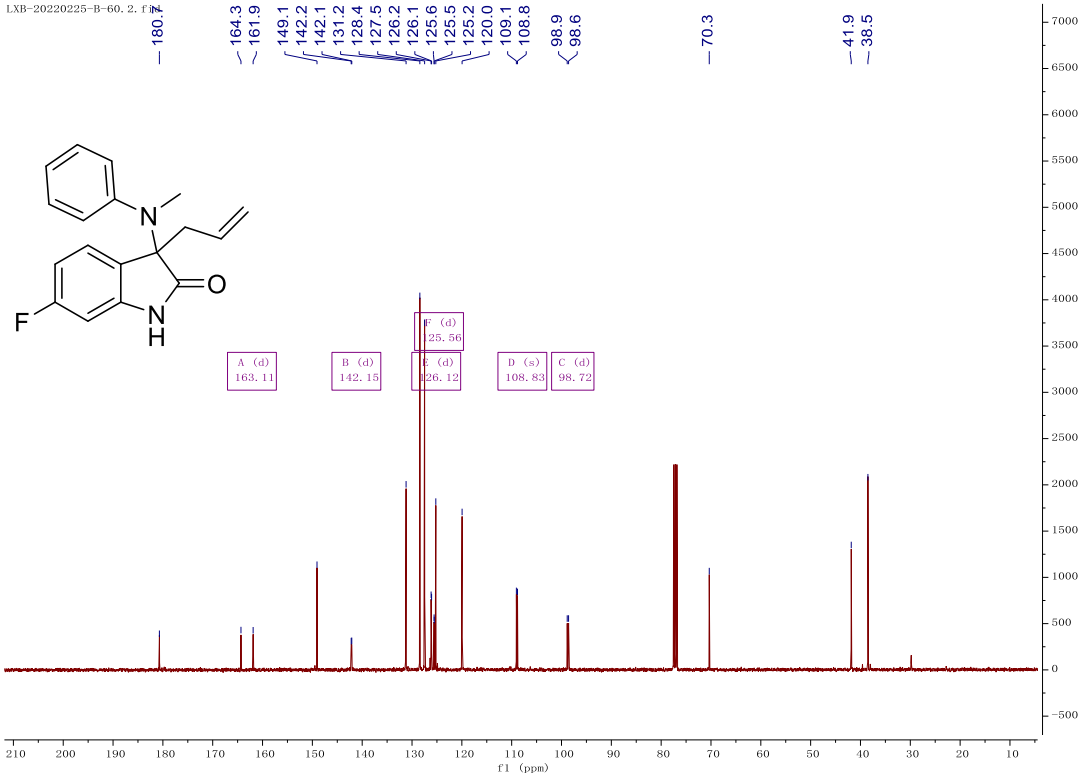

LXB-20220303-B-63.1.fid
1.00
4.25
1.17
1.03
1.03
1.02
1.18
1.07
1.04
3.01
2.19
3.21
f1 (ppm)

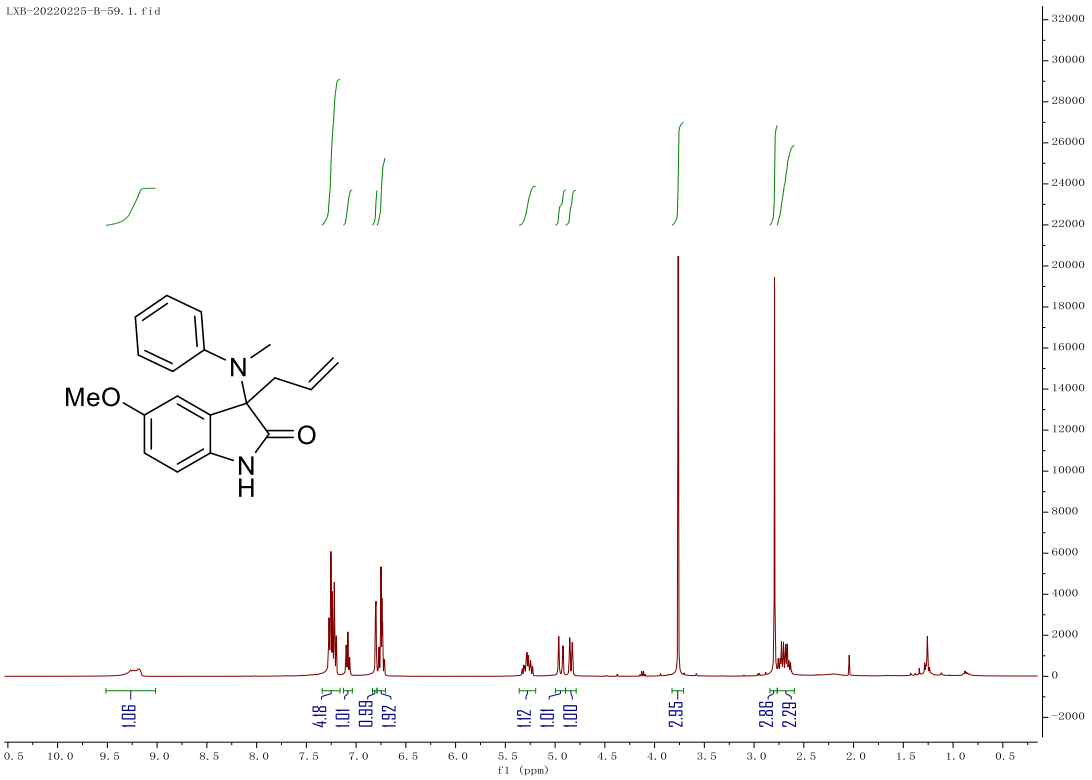

LXB-20220304-B-63.2.fid
180.3
149.3
138.4
132.0
131.6
130.3
129.1
128.3
127.4
125.5
124.9
119.5
109.7
70.7
42.0
38.6
21.3
f1 (ppm)

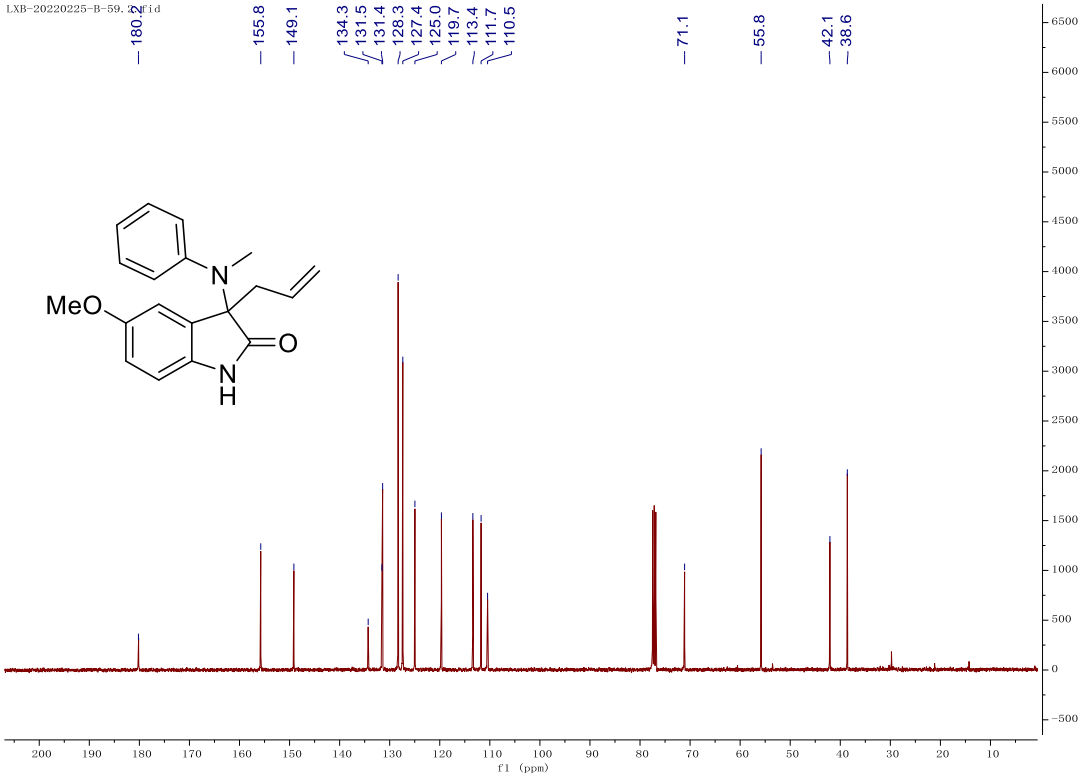

LXB-20220225-B-61.1.fid
Br
N
O
N
H
1.00
4.41
1.15
2.22
1.12
1.20
1.15
1.11
3.32
2.21
f1 (ppm)

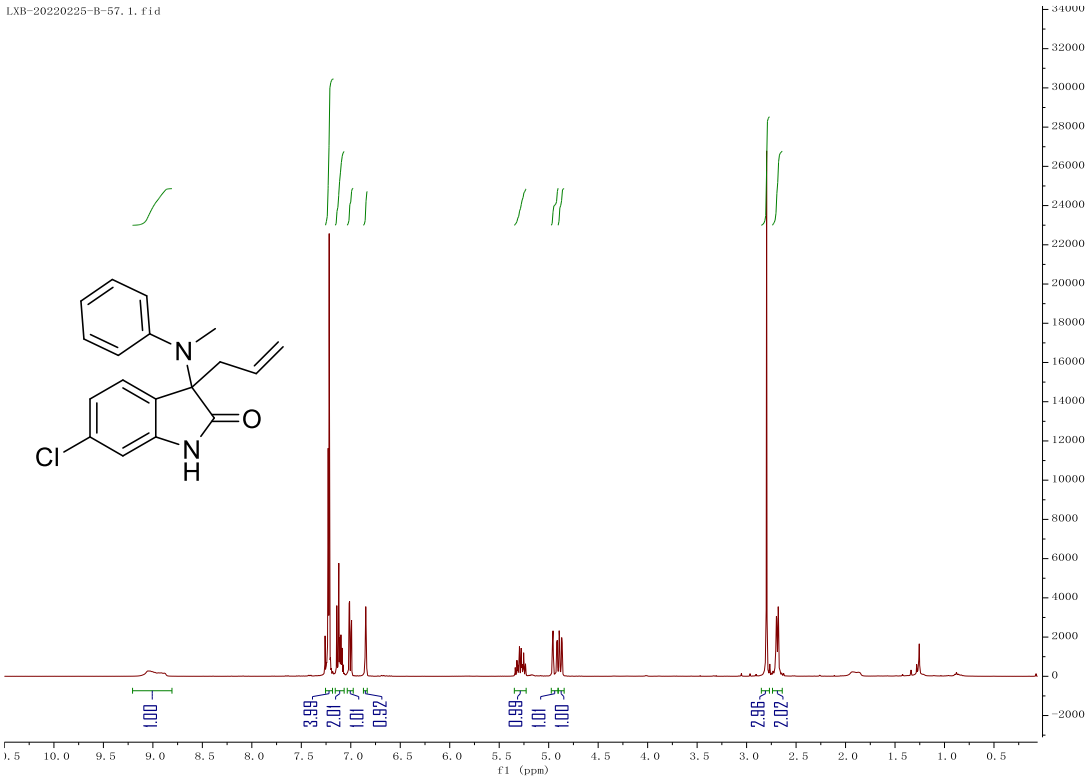

LXB-20220225-B-61.2.fid
180.1
149.0
142.0
131.0
129.3
128.5
127.5
126.3
125.5
125.3
122.2
120.1
113.5
70.5
41.8
38.7
Br
N
O
N
H
f1 (ppm)

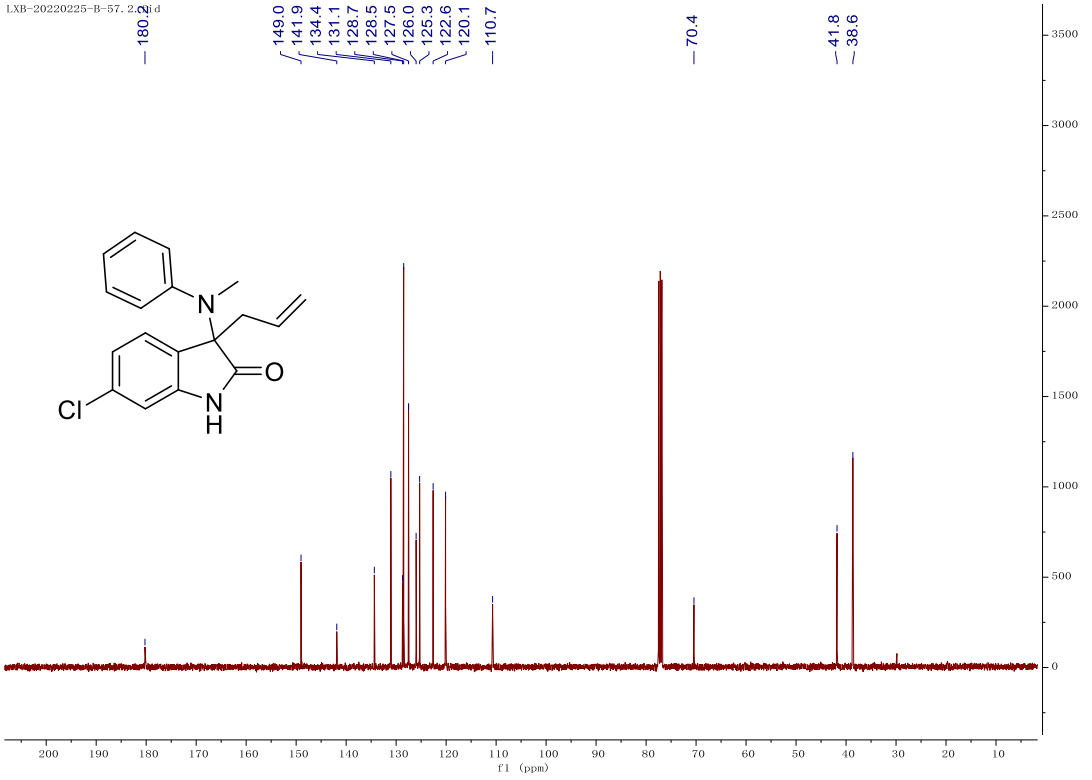

LXB-20220225-B-60.1.fid
N
O
F
N
H
1.00
3.85
1.28
0.92
1.00
0.99
1.01
1.00
1.00
2.91
1.98
f1 (ppm)

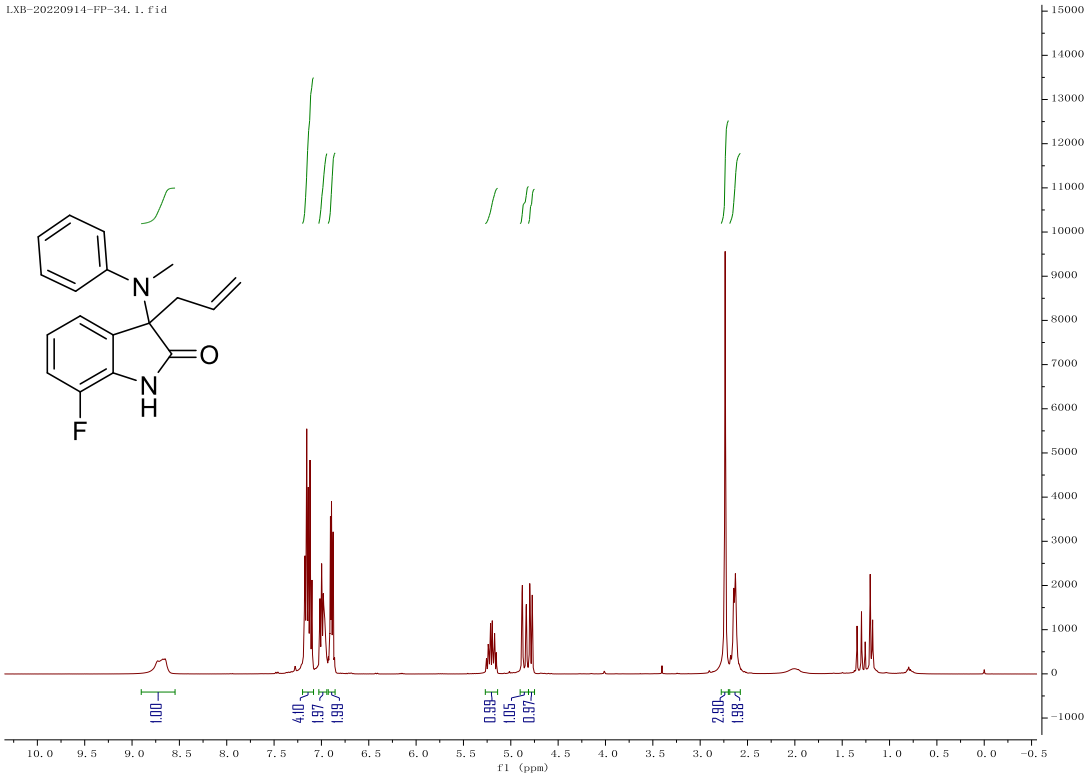

LXB-20220225-B-60.2.fid
180.7
164.3
161.9
149.1
142.2
142.1
131.2
128.4
127.5
126.2
126.1
125.6
125.5
125.2
120.0
109.1
108.8
98.9
98.6
70.3
41.9
38.5
N
O
F
N
H
A (d)
163.11
B (d)
142.15
F (d)
125.56
E (d)
126.12
D (s)
108.83
C (d)
98.72
f1 (ppm)

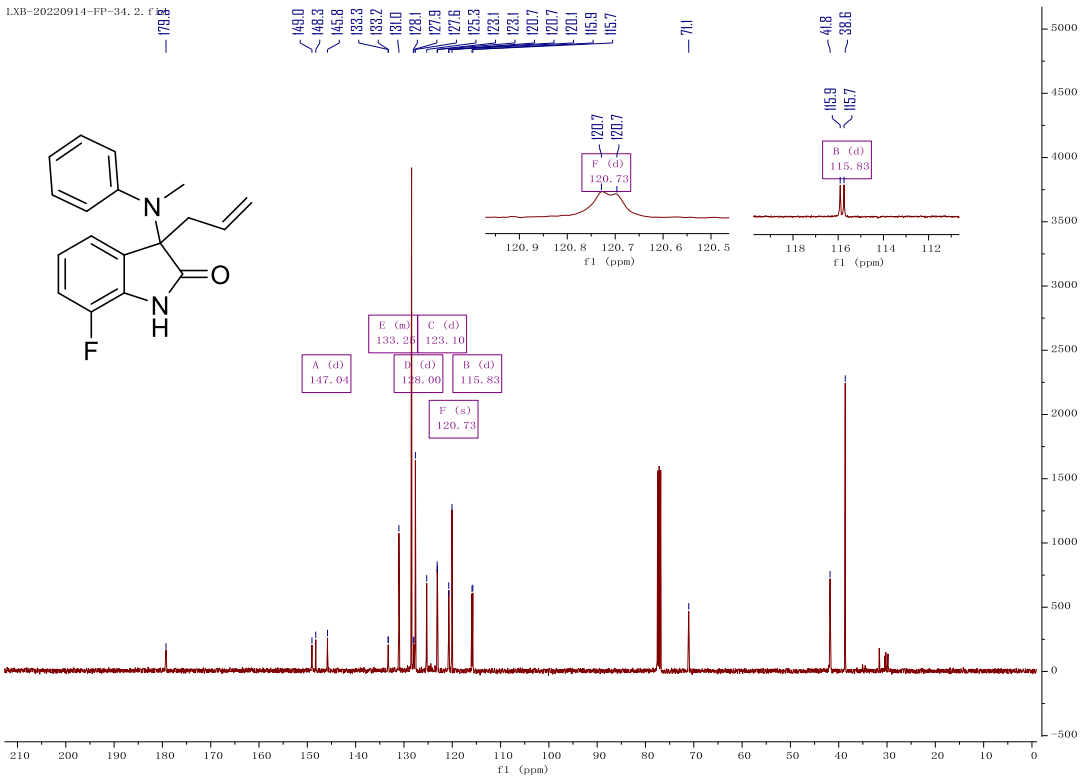

LXB-20220225-B-59.1.fid
MeO
N
O
N
H
1.06
4.18
1.01
0.95
1.92
1.12
1.01
1.00
2.95
2.86
2.29
f1 (ppm)

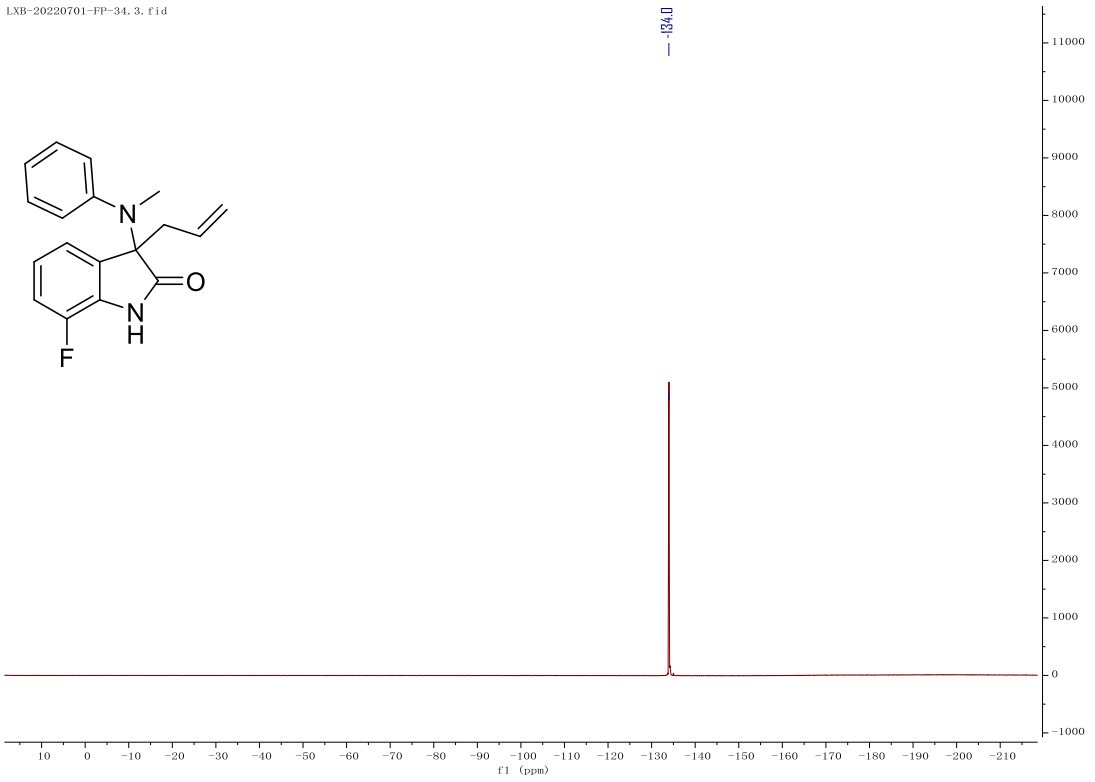

LXB-20220225-B-59.2.fid
180.2
155.8
149.1
134.3
131.5
131.4
128.3
127.4
125.0
119.7
113.4
111.7
110.5
71.1
55.8
42.1
38.6
MeO
N
O
N
H
f1 (ppm)

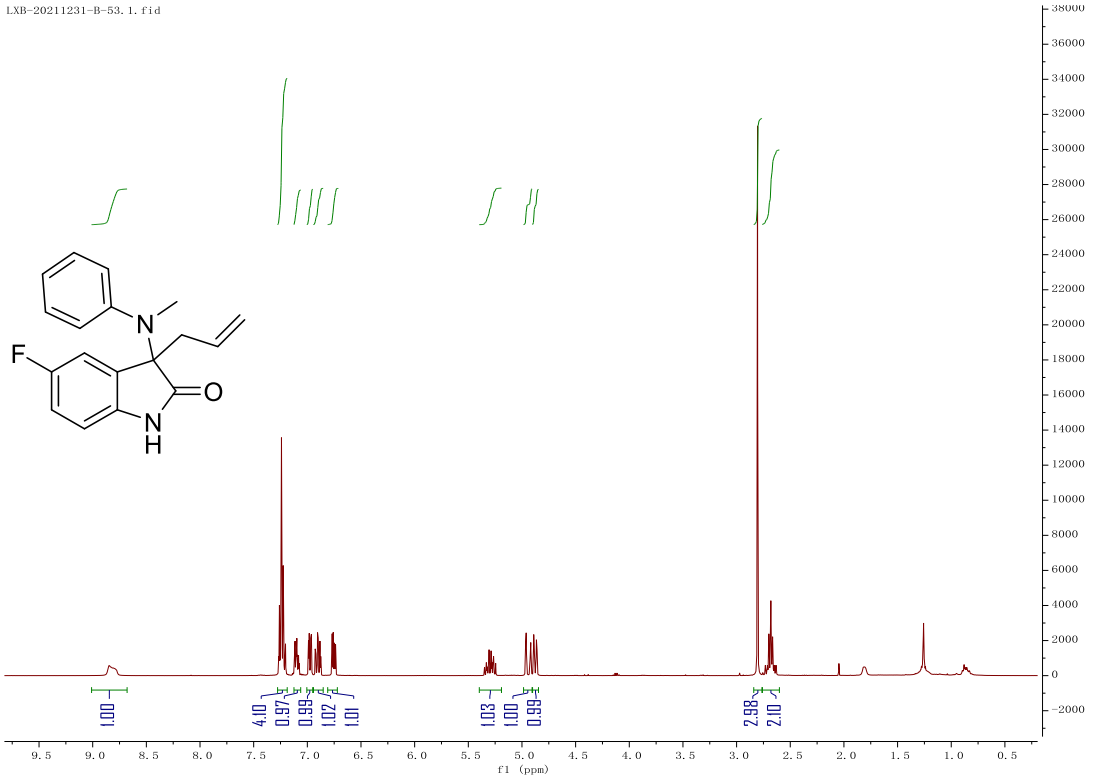

LXB-20220225-B-57.1.fid
Cl
N
O
N
H
1.00
3.99
2.01
1.01
0.92
0.99
1.01
1.00
2.96
2.02
f1 (ppm)

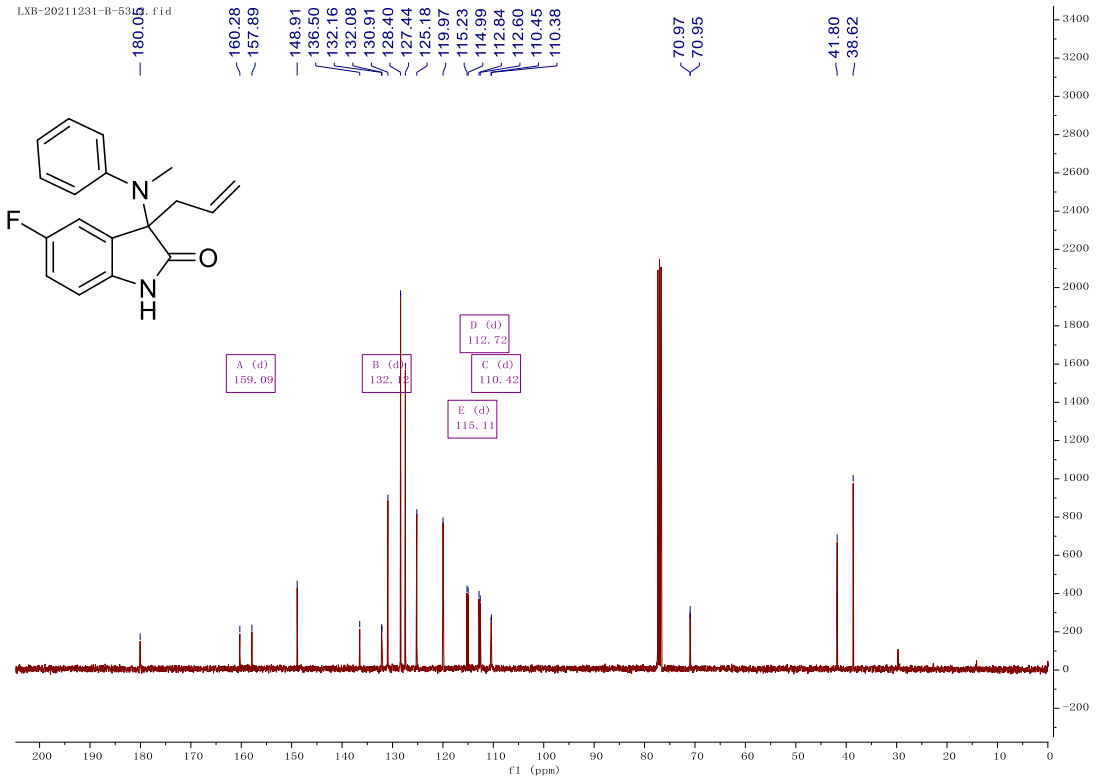

LXB-20220225-B-57.2.fid
180.2
149.0
141.9
134.4
131.1
128.7
128.5
127.5
126.0
125.3
122.6
120.1
110.7
70.4
41.8
38.6
Cl
N
O
N
H
f1 (ppm)

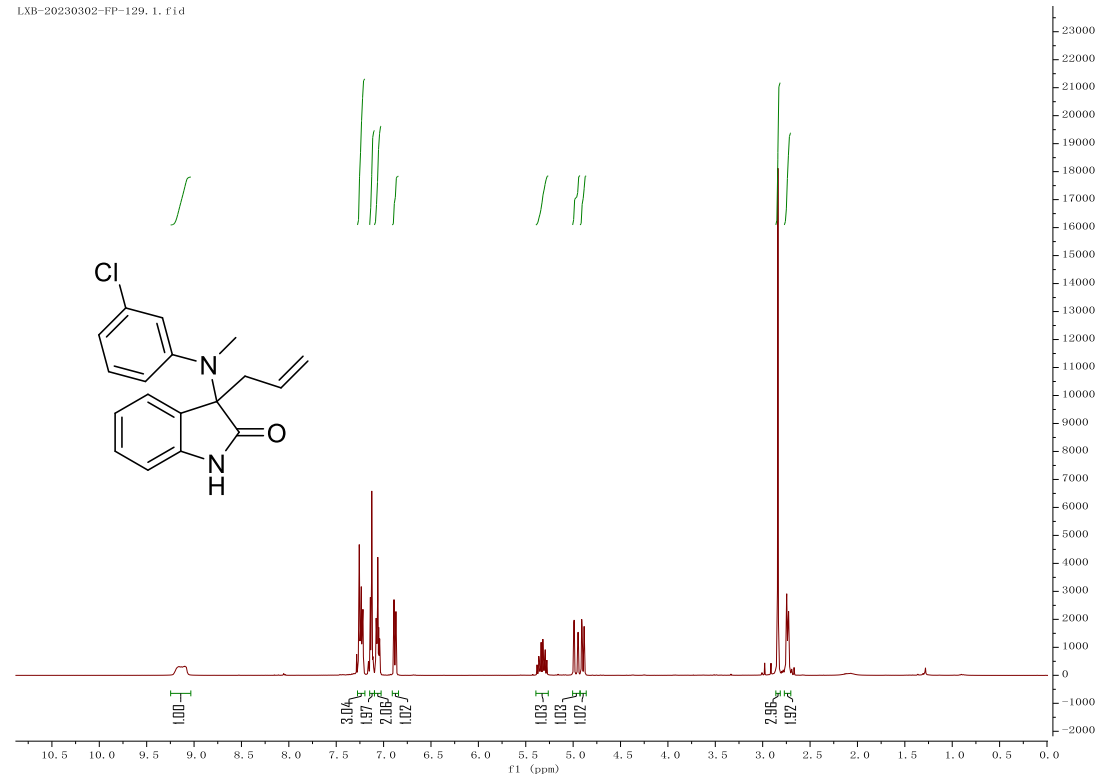

LXB-20220914-FP-34.1.fid
1.00
4.10
1.97
1.99
0.99
1.05
0.97
2.90
1.98
f1 (ppm)

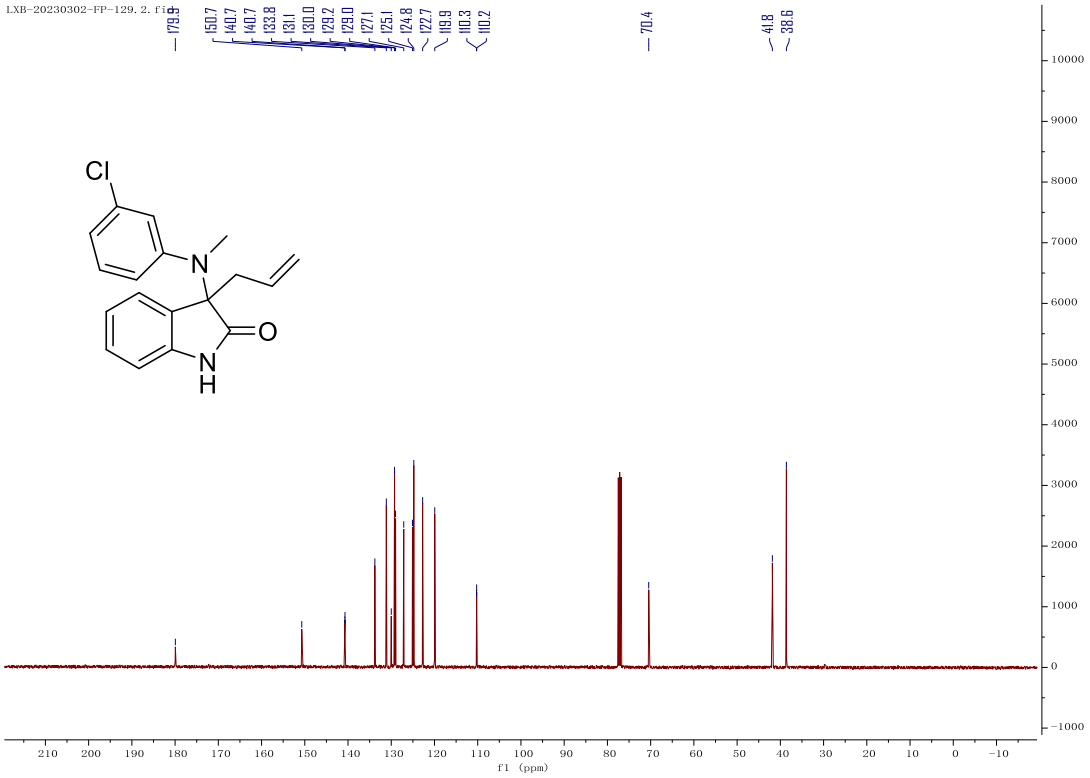

LXB-20220914-FP-34.2.fid
179.8
149.0
148.3
145.8
133.3
133.2
131.0
128.1
127.9
127.6
125.3
123.1
123.1
120.7
120.7
120.1
115.9
115.7
71.1
41.8
38.6
120.7
120.7
F (d)
120.73
115.9
115.7
B (d)
115.83
A (d)
147.04
E (m)
133.25
C (d)
123.10
D (d)
128.00
B (d)
115.83
F (s)
120.73
f1 (ppm)

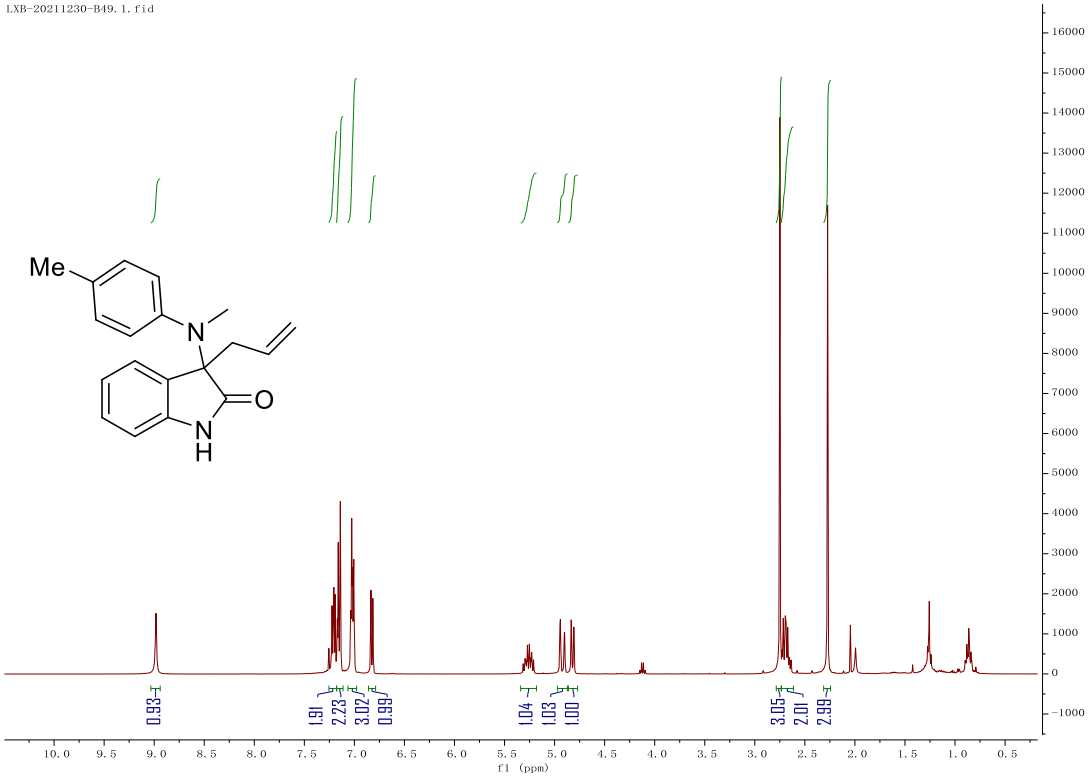

LXB-20220701-FP-34.3.fid
-134.0
f1 (ppm)

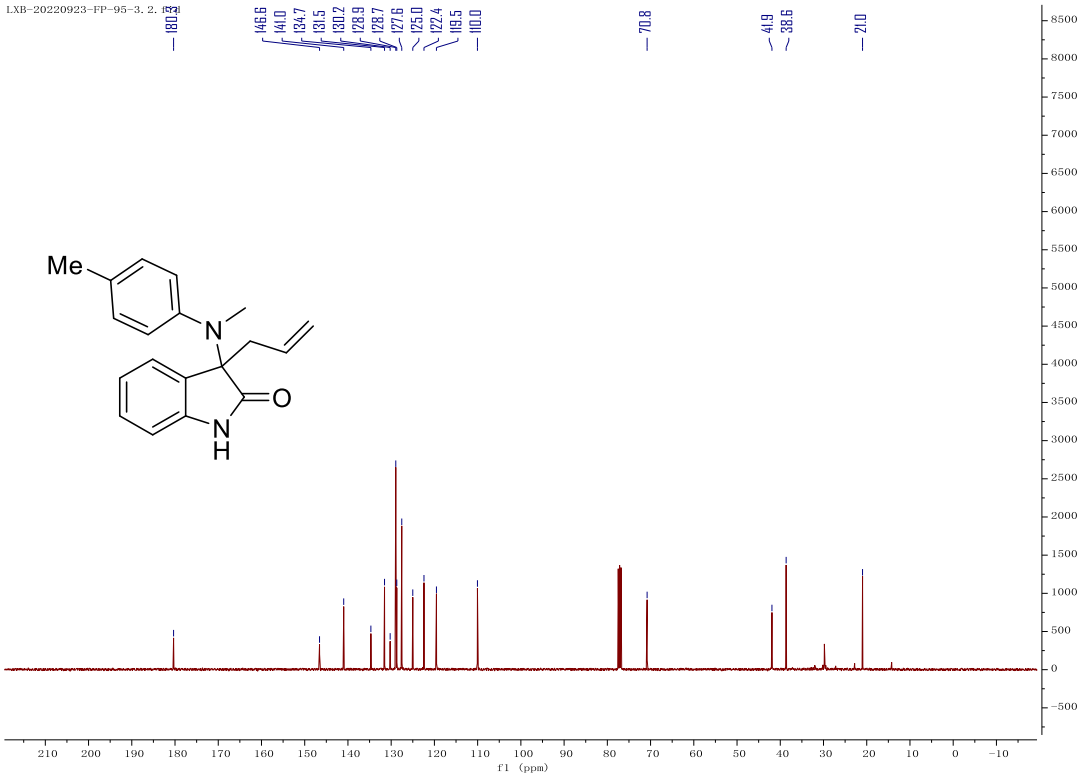

LXB-20211231-B-53.1.fid
1.00
4.10
0.97
0.99
1.02
1.01
1.03
1.00
0.99
2.98
2.10
f1 (ppm)

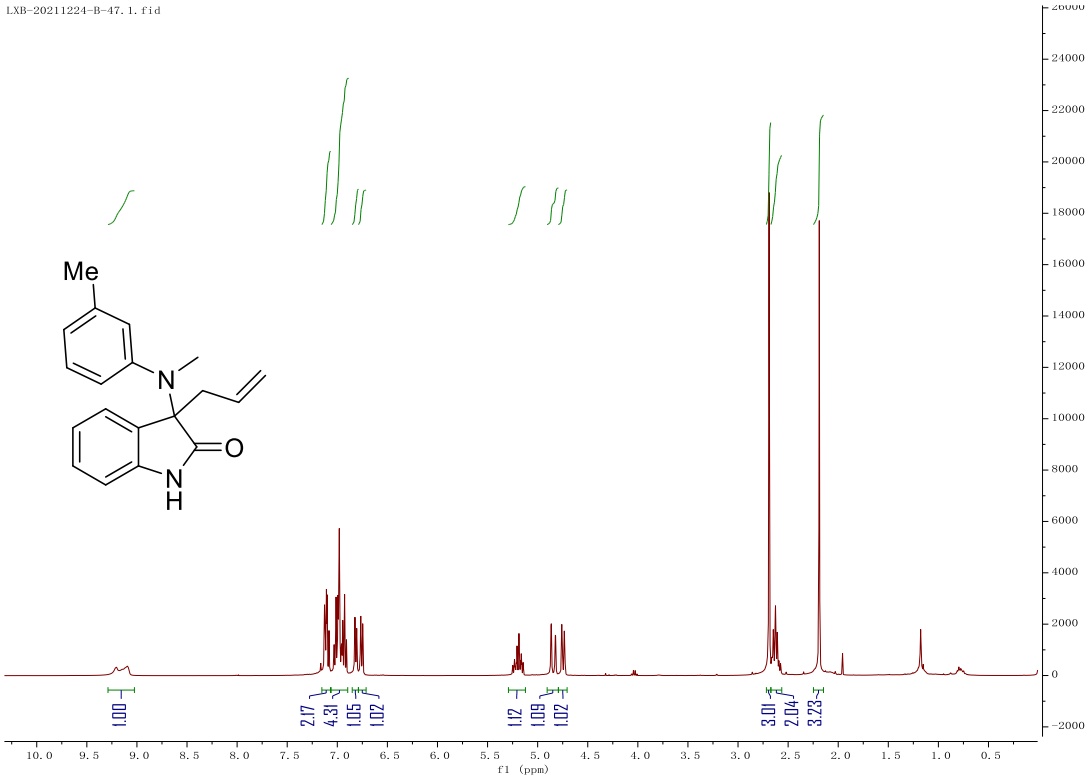

LXB-20211231-B-53(2).fid
180.03
160.28
157.89
148.91
136.50
132.16
132.08
130.91
128.40
127.44
125.18
119.97
115.23
114.99
112.84
112.60
110.45
110.38
70.97
70.95
41.80
38.62
A (d)
159.09
B (d)
132.12
C (d)
110.42
D (d)
112.72
E (d)
115.11
F
N
O
N
H
f1 (ppm)

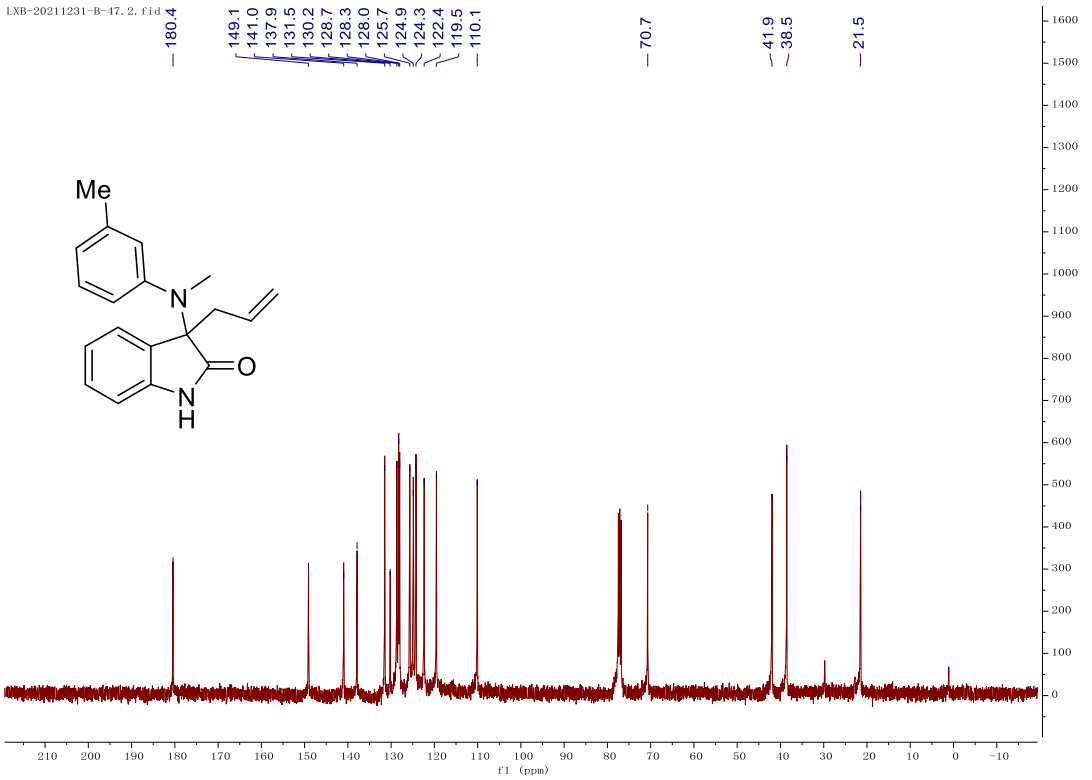

LXB-20230302-FP-129.1.fid
Cl
N
O
N
H
1.00
3.04
1.97
2.06
1.02
1.03
1.03
1.02
2.96
1.92
f1 (ppm)

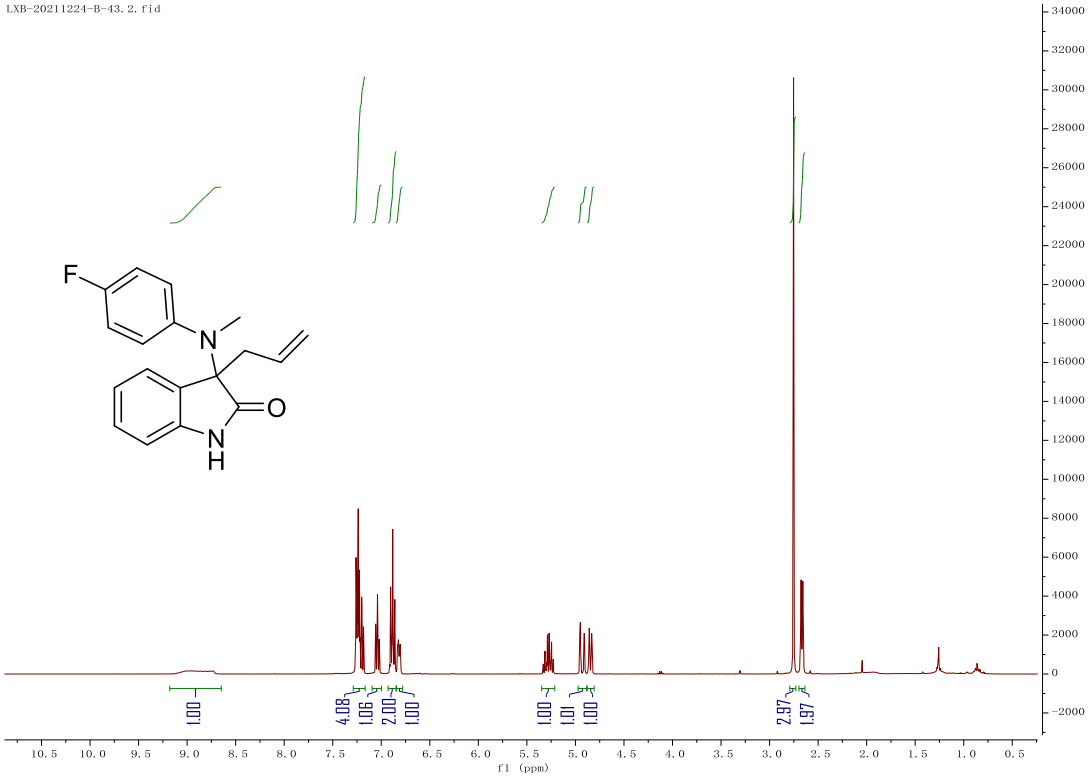

LXB-20230302-FP-129.2.fid
179.9
150.7
140.7
140.7
133.8
131.1
130.0
129.2
129.0
127.1
125.1
124.8
122.7
119.9
110.3
110.2
70.4
41.8
38.6
Cl
N
O
N
H
f1 (ppm)

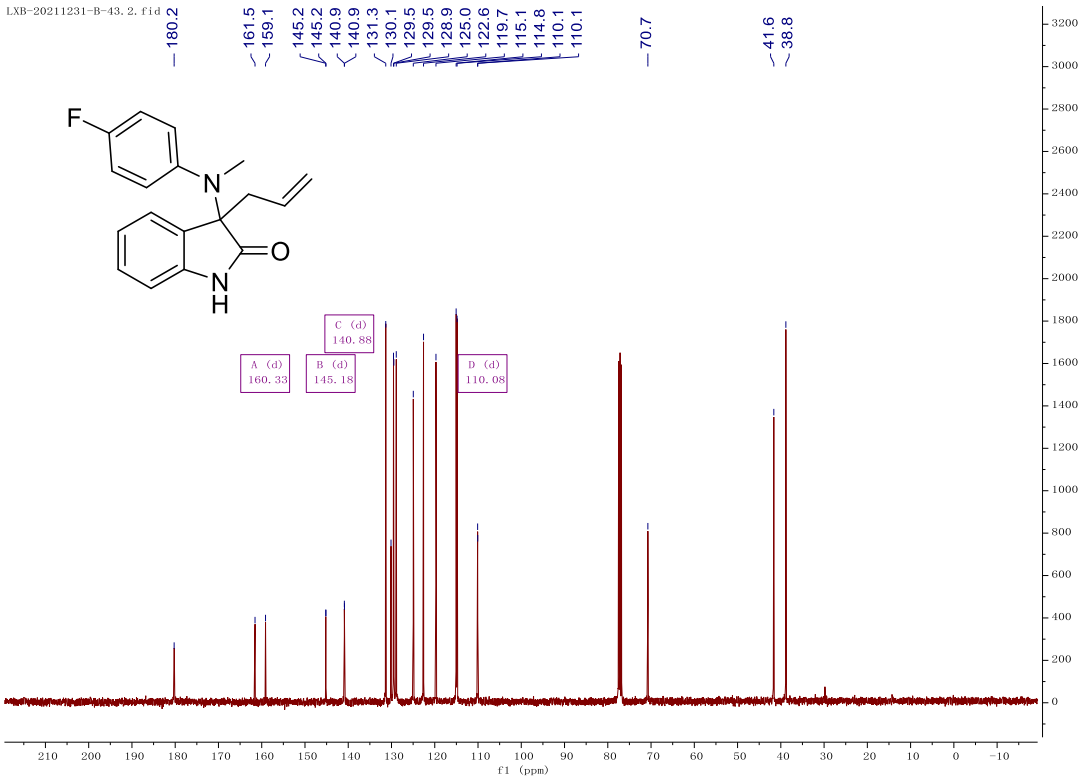

LXB-20211230-B49.1.fid
Me
N
O
N
H
0.93
1.91
2.23
3.02
0.99
1.04
1.03
1.00
3.05
2.01
2.99
f1 (ppm)

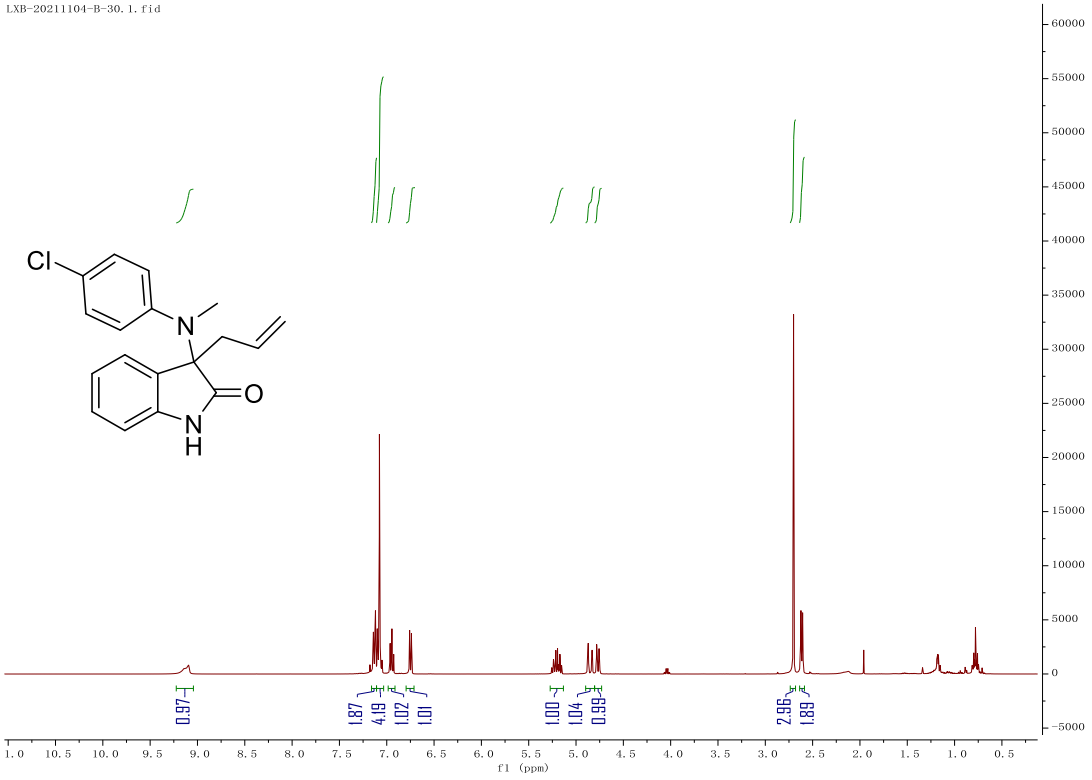

LXB-20220923-FP-95-3.2.
180.2
146.6
141.0
134.7
131.5
130.2
128.9
128.7
127.6
125.0
122.4
119.5
110.0
70.8
41.9
38.6
21.0
Me
N
O
N
H
f1 (ppm)

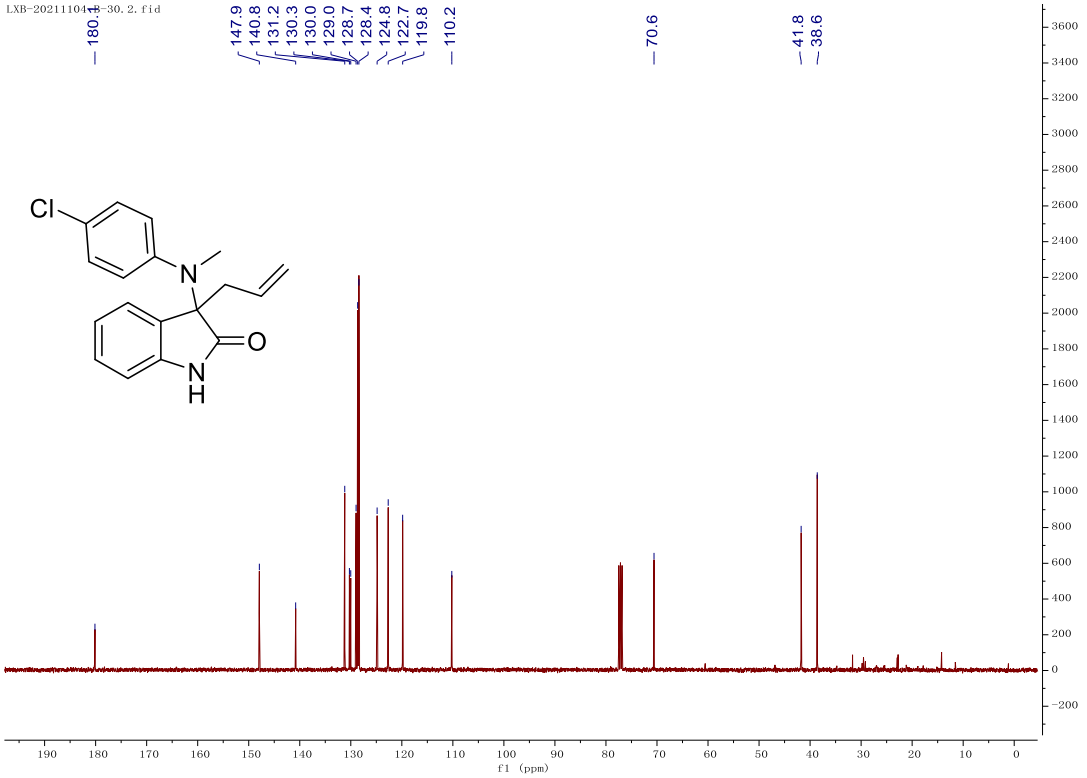

LXB-20211224-B-47.1.fid
Me
N
O
N
H
1.00
2.17
4.31
1.05
1.02
1.12
1.09
1.02
3.01
2.04
3.23
f1 (ppm)

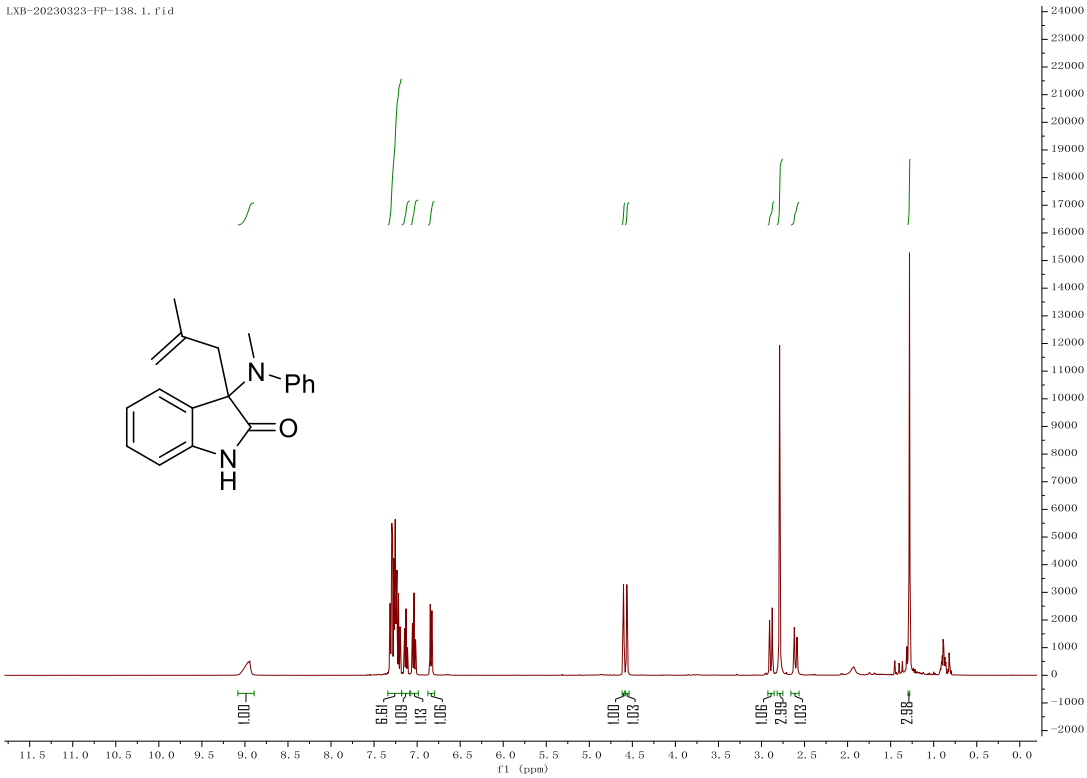

LXB-20211231-B-47.2.fid
180.4
149.1
141.0
137.9
131.5
130.2
128.7
128.3
128.0
125.7
124.9
124.3
122.4
119.5
110.1
70.7
41.9
38.5
21.5
Me
N
O
N
H
f1 (ppm)

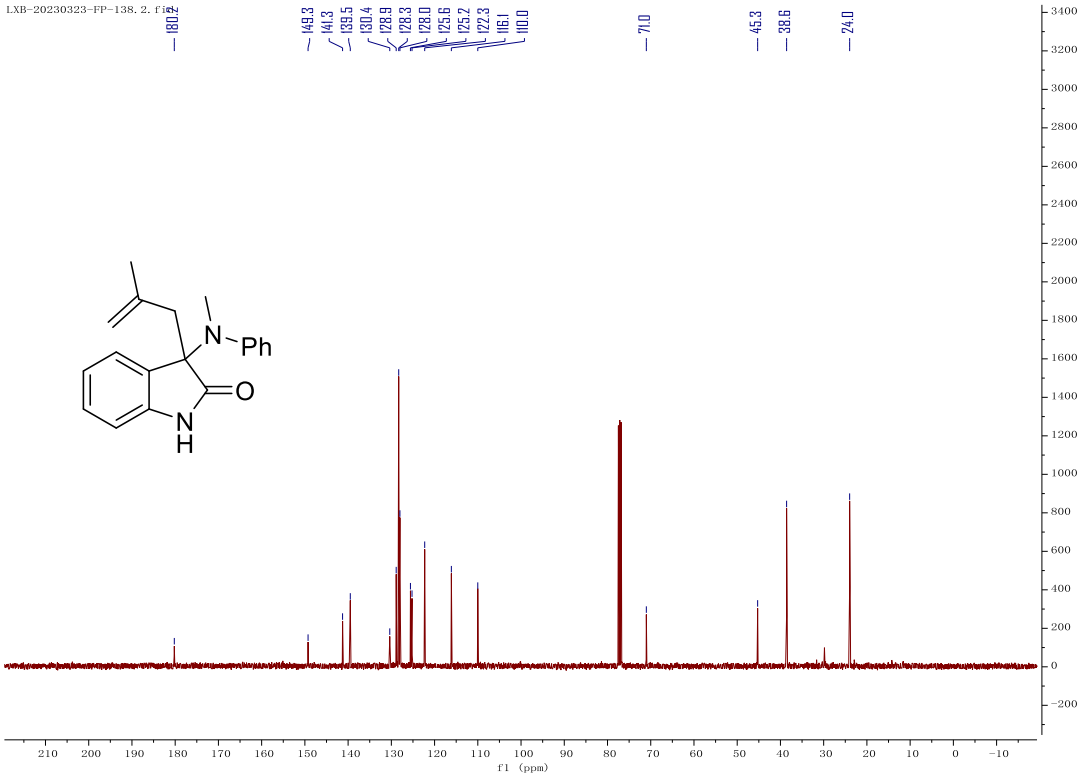

LXB-20211224-B-43.2.fid
F
N
O
N
H
1.00
4.08
1.06
2.00
1.00
1.00
1.01
1.00
2.97
1.97
f1 (ppm)

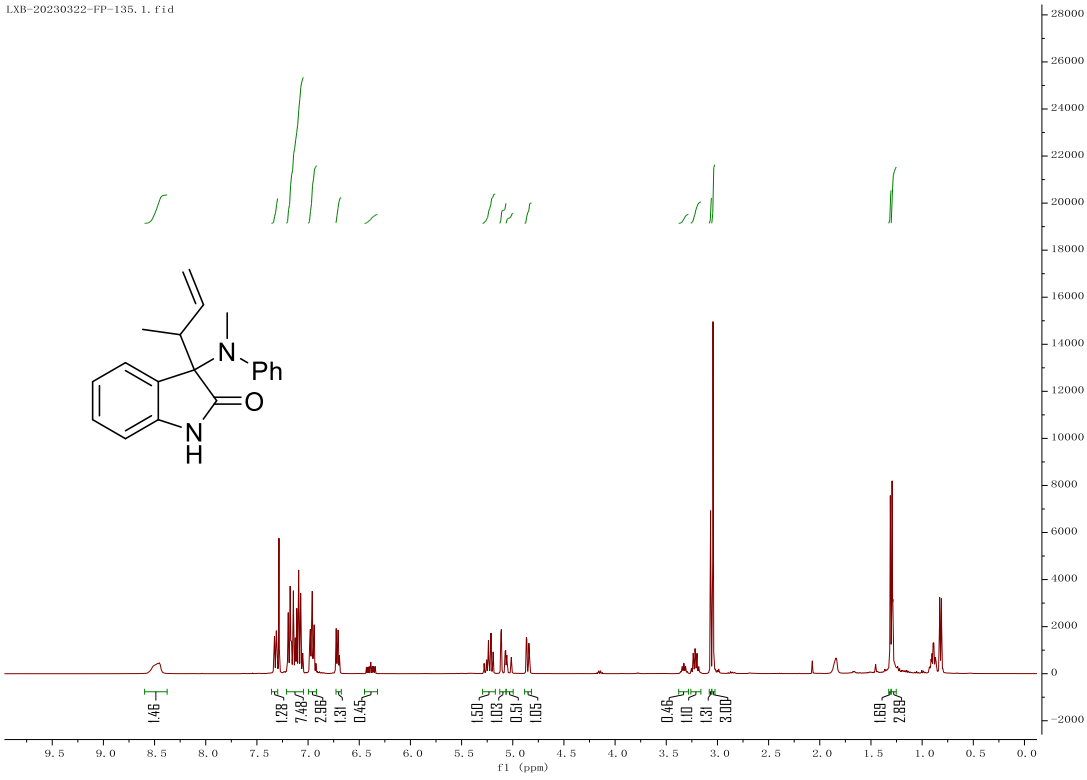

LXB-20211231-B-43.2.fid
180.2
161.5
159.1
145.2
145.2
140.9
140.9
131.3
130.1
129.5
129.5
128.9
125.0
122.6
119.7
115.1
114.8
110.1
110.1
70.7
41.6
38.8
F
N
O
N
H
A (d) 160.33
B (d) 145.18
C (d) 140.88
D (d) 110.08
f1 (ppm)

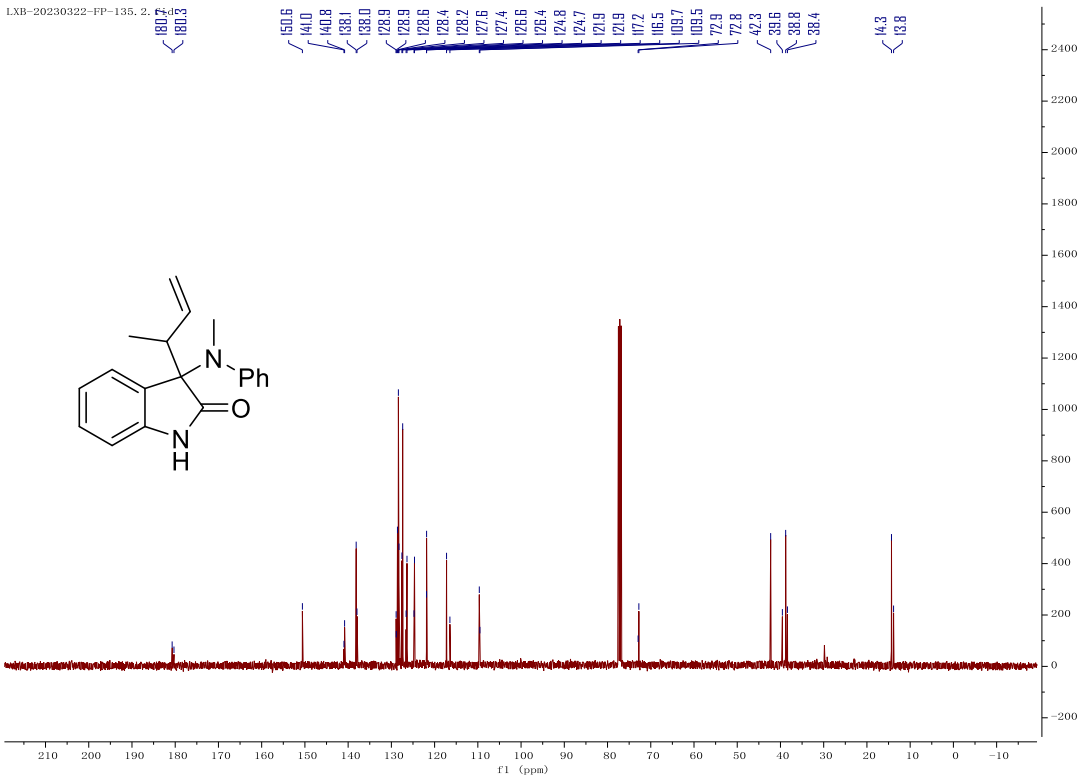

LXB-20211104-B-30.1.fid
Cl
N
O
N
H
0.97
1.87
4.19
1.02
1.01
1.00
1.04
0.99
2.96
1.89
f1 (ppm)


LXB-20211104-30.2.fid
180.1
147.9
140.8
131.2
130.3
130.0
129.0
128.7
128.4
124.8
122.7
119.8
110.2
70.6
41.8
38.6
Cl
N
O
N
H
f1 (ppm)


LXB-20230323-FP-138.1.fid
N
Ph
O
N
H
1.00
6.61
1.09
1.13
1.06
1.00
1.03
1.06
2.99
1.03
2.98
f1 (ppm)


LXB-20230323-FP-138.2.fid
180.2
149.3
141.3
139.5
130.4
128.9
128.3
128.0
125.6
125.2
122.3
116.1
110.0
71.0
45.3
38.6
24.0
N
Ph
O
N
H
f1 (ppm)


LXB-20230322-FP-135.1.fid
1.46
1.28
7.48
2.96
1.31
0.45
1.50
1.03
0.51
1.05
0.46
1.10
1.31
3.00
1.69
2.89
N
Ph
O
N
H
f1 (ppm)


LXB-20230322-FP-135.2.fid
180.7
180.3
150.6
141.0
140.8
138.1
138.0
128.9
128.9
128.6
128.4
128.2
127.6
127.4
126.6
126.4
124.8
124.7
121.9
121.9
117.2
116.5
109.7
109.5
77.9
72.8
42.3
39.6
38.8
38.4
14.3
13.8
N
Ph
O
N
H
f1 (ppm)