Electronic Supplementary Material (ESI) for Green Chemistry.

Supporting Information

Synthesis of renewable isoindolines from Bio-based furfurals

Feng Xu [a,c], Zao Li [a], Li-long Zhang [a], Shengqi Liu [a], Hu Li*[a], Yuhe Liao*[b], Song Yang*[a]

a. National Key Laboratory of Green Pesticide, Key Laboratory of Green Pesticide & Agricultural Bioengineering, Ministry of Education, State-Local Joint Laboratory for Comprehensive Utilization of Biomass, Center for R&D of Fine Chemicals, Guizhou University, Guiyang, Guizhou 550025, China. E-mail: hli13@gzu.edu.cn (HL), jhzx.msm@gmail.com (SY).

b. Guangzhou Institute of Energy Conversion, Chinese Academy of Sciences, Guangzhou, 510640, China. E-mail: liaoyh@ms.giec.ac.cn.

c. Department of Chemistry and Materials Engineering, Guiyang University, Guiyang, Guizhou 550025, China.

Table of Contents

1. Experimental Procedures S3
2. Results and Discussion S8
3. X-Ray Crystal Structure Data S20
4. Spectra Data of Products S24
5. NMR Spectroscopy S35
6. References S81

1. Experimental Procedures

1.1. Chemicals

Chemical	Chemical Linear Formula	Manufacturer
Furfural	$C_5H_4O_2$	Sigma-Aldrich
5-Methyl furfural	$C_6H_6O_2$	Sigma-Aldrich
2-Acetylfuran	$C_6H_6O_2$	Sigma-Aldrich
5-Bromo-2-furaldehyde	$C_5H_3BrO_2$	Sigma-Aldrich
Acryloyl chloride	CH_2=CHCOCl	Sigma-Aldrich
Methacryloyl chloride	CH_2=CH(CH_2)COCl	Alfa Aesar
Palladium 10% on Carbon (wetted with ca. 55% Water)	Pd/C	Alfa Aesar
Ammonium formate	HCO_2NH_4	Sigma-Aldrich
Zinc nitrate hexahydrate	$Zn(NO_3)_2 \cdot 6H_2O$	Sigma-Aldrich
Bismuth (III) nitrate pentahydrate	$Bi(NO_3)_3 \cdot 5H_2O$	Sigma-Aldrich
Aluminium chloride	$AlCl_3$	Alfa Aesar
Zirconium tetrachloride	$ZrCl_4$	Alfa Aesar
Tin tetrachloride	$SnCl_4$	Alfa Aesar
Zinc chloride	$ZnCl_2$	Alfa Aesar
Trimesic acid	$C_6H_3(CO_2H)_3$	Sigma-Aldrich
Stearic acid	$CH_3(CH_2)_{16}COOH$	Sigma-Aldrich
1-Methylimidazole	$C_4H_6N_2$	Sigma-Aldrich
Concentrated sulfuric acid	H_2SO_4	Sigma-Aldrich
Phosphoric acid	H_3PO_4	Sigma-Aldrich
Hydrochloric acid	HCl	Sigma-Aldrich
Amberlyst-15		Sigma-Aldrich
Methanesulfonic acid	CH_3SO_3H	Acros
P-toluenesulfonic acid	*p*-$CH_3C_6H_4SO_3H$	Acros
Sodium hydroxide	NaOH	Macron
Potassium hydroxide	KOH	Macron
Aniline	C_6H_7N	Sigma-Aldrich
Toluene	$C_6H_5CH_3$	Sigma-Aldrich
Tetrahydrofuran	C_4H_8O	Sigma-Aldrich
N,*N*-Dimethylformamide	C_3H_7NO	Sigma-Aldrich
Dimethyl sulfoxide	CH_3SOCH_3	Sigma-Aldrich

1.2. General Information and Methods

Unless otherwise noted, all materials were used as received from commercial sources without further purification. ^{1}H NMR spectra were recorded on a Bruker DRX-400 (400 MHz) spectrometer and chemical shifts were reported in ppm. The peak information was described as: br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet; coupling constants in Hz. ^{13}C NMR spectra were recorded on a Bruker DRX-400 (101 MHz) spectrometer with complete proton decoupling. Wide-angle X-ray diffraction patterns (XRD) were measured on Rigaku Ultima IV with Cu Kα radiation (λ=1.5418 Å, 40 kV, 40 mA, 5- 85^o) as the X-ray source to analyze the crystal structure of particles. The morphology of samples was observed by SEM (NovaTM NanoSEM). All the samples were dispersed in methanol

solution before placing on a silica wafer. The samples before SEM observation were kept under vacuum and subjected to Pt coating for 120 seconds by sputter at the current of 20 mA. TEM measurements were performed at 200kV with an aberration-corrected JEM-ARM200F (JEOL, Corrector: CEOS). The element distribution is obtained by electron dispersive spectroscopy (EDS) connected with TEM, the operating voltage is 10 kV. In the thermogravimetry/differential thermal analysis thermoanalyzer (TG-DTA) measurement, the samples are treated under N_2 conditions. The heating rate is 5 °C /min and the goal temperature was 800 °C. Fourier transform infrared (FT-IR) spectra were measured with Perkin Elmer Spectrum 100 at a resolution of 4 cm^{-1}. Samples for FT-IR measurement were prepared by mixing vacuum-dried particles with potassium bromide (KBr) in an appropriate ratio (KBr/sample < 1:100, w/w) and pressing into translucent discs. X-ray photoelectron spectroscopy (XPS) spectra were recorded on a Thermo Scientific K_α X-ray photoelectron spectrometer to identify the states of Zn, C, and O. Before measurement, the spectra were calibrated with reference to C*1S* at binding energy 284.16 eV. BET surface areas (BET-SA) of the catalysts were determined on NOVA 4200e instrument by N_2-physisorption at -196 °C. Prior to the measurements, the known amount of catalyst was evacuated for 2 h at 220 °C to remove physically adsorbed water. High-resolution mass spectra (HRMS) were determined with a LC-MS system equipped with an Agilent 1260 Infinity Liquid Chromatography instrument and an Agilent 6545 Q-TOF Mass Spectrometer using ESI ion sources, as specified. Low-resolution mass spectra (LRMS) measurements were recorded on an Agilent 6890N/5973 Network GC System equipped with an HP-5MS column.

1.2. DFT Calculations

The DFT computations with the local meta–GGA exchange-correlation functional M06l were performed by using the Gaussian 16 program.[S1] The accuracy of the M06L functional in the prediction of geometry, vibrational frequency, thermochemistry, etc. of metal−organic frameworks was validated in previous work.[S2] All the geometry optimization calculations were fully relaxed with 6-31G(d) basis sets for all elements.[S3] To verify and confirm the transition states, frequency calculations were performed at the same level of theory to classify the stationary points along the reaction coordinates. For further refining the electronic energy, the single point energy calculations were carried out by using the same density functional with 6-311+G(d, p). The bulk solvent effect of DMSO was considered by the self-consistent reaction field (SCRF) method using SMD solvent model.[S4]

1.3. Catalyst Preparation

Zn-BTC: The zinc nitrate hexahydrate (291.4 mg) and trimesic acid (BTC) (383.9 mg) were mixed in the pre-mixed 10 mL N,N-dimethylformamide: toluene = 9:1 (v/v) solution. The mixture is then purged into a 100 mL Teflon-lined autoclave. After being tightly sealed, the autoclave was heated in the oven for 12 h at 130 °C. After cooling down to room temperature, the obtained precipitate was collected by centrifuge and washed with N,N-dimethylformamide, and ethanol twice respectively. The resulting precipitate was dried overnight to obtain the final white Zn-BTC crystalline powder.

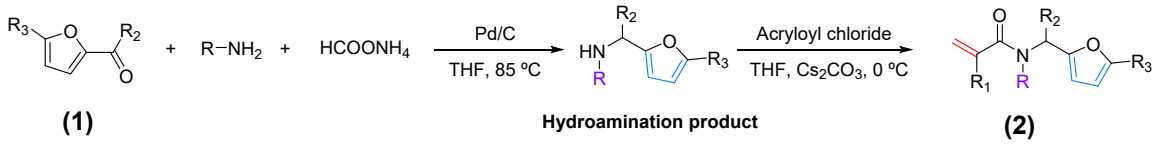
Zn-BTC-SA*:* Stearic acid grafted Zn-BTC was synthesized by a green method.[S5] In general, the zinc nitrate hexahydrate (291.4 mg) as the metal precursor, trimesic acid (BTC) as the organic linker (383.9 mg), and 10% of stearic acid (according to the weight of the BTC linker) were ground together for about 30 min at room temperature. Then, the powder was moved into a 100 mL Teflon-lined autoclave at 130 °C for 12 h. After cooling to room temperature, the obtained white solid was washed with 70 °C ethanol and dried for 24 h at 100 °C under vacuum, the catalyst was referred to as Zn-BTC-SA.

[Hmim]HSO_4*:* N-methylimidazole (7.9 mL, 0.1 mmol) was added to a 250 mL three-necked flask, and placed in an ice-water bath to cool to 0-5 °C. Under vigorous stirring, a mixed solution of 10.2 g of 98% concentrated sulfuric acid and 10 mL of water was added within 30 min, followed by stirring at room temperature for 2 h. After the reaction, the reaction solution was evaporated under reduced pressure at 75 °C to remove water to obtain a colorless and transparent ionic liquid [Hmim]HSO_4 18.0 g with a yield of 100%. ^{1}H NMR (400 MHz, DMSO-d_6), δ:3.70(s, 3H, CH_3), 7.21(bs, 1H, CH), 8.02(bs, 2H, CHCH), 8.32(bs, 1H, NH), 12.74(bs, 1H, HSO_4); IR (liquid), σ /cm^{-1}:3327(m), 3050(m), 2880(m), 665(w), 1589(w), 1439(w), 1337(m),1220(s), 1087(s), 1053(s), 891(s), 567(s).
Recycled [Hmim]HSO_4**:** ^{1}H NMR (400 MHz, DMSO-d_6), δ: 3.70(s, 3H, CH_3), 7.21(bs, 1H, CH), 8.02(bs, 2H, CHCH), 8.32(bs, 1H, NH), 12.74(bs, 1H, HSO_4); IR (liquid), σ /cm^{-1}:3327(m), 3050(m), 2880(m), 665(w), 1589(w), 1439(w), 1337(m),1220(s), 1087(s), 1053(s), 891(s), 567(s).

Zn-BTC-SA/[Hmim]HSO_4: The Zn-BTC-SA@[Hmim]HSO_4 composite was prepared by using wet impregnation, according to the reports with modifications.[S6] In detail, 0.15 g of [Hmim]HSO_4 was dissolved in 10 mL methanol and stirred for 1 h at room temperature. Then, activated Zn-BTC-SA (0.30 g) (to remove moisture, Zn-BTC-SA was evacuated at 100 °C under vacuum overnight.) was added to the solution and the

mixture was stirred at 30 °C under an open atmosphere until the methanol was evaporated. After methanol was completely evaporated, the sample was dried in an oven at 80 °C overnight to obtain the white Zn-BTC-SA/[Hmim]HSO_4 powder.

1.4. Preparation of Starting Materials

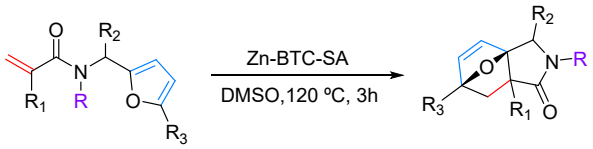


(1) **Hydroamination product** **(2)**

General Procedure for the Synthesis of (**2**): Furfural (12.5 mmol) was added to a stirred solution of the corresponding amines (13.75 mmol), $HCOONH_4$ (18.57 mmol) and Pd/C (100 mg) in anhydrous THF (25 mL) under N_2. The resulting mixture was stirred at 85 °C until the furfural was consumed as determined by GC-MS. After the reaction, the Pd/C was recycled by filtering. Deionized water (50 mL) was added to the filtrate and the mixture was extracted with EtOAc (3 × 50 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and filtered, the solvents were removed under reduced pressure to give the hydroamination product.

Next, acryloyl chloride (13.75 mmol) was added to a stirred solution of the hydroamination product (12.5 mmol) and Cs_2CO_3 (15.6 mmol) in THF. The reaction mixture was stirred at 0 °C until the hydroamination product was consumed as determined by GC−MS. After the reaction, the reaction mixture was diluted by the addition of deionized water (30 mL) and the aqueous layer was extracted with EtOAc (3 × 30 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and filtered, the solvents were removed under reduced pressure. The residue was purified by silica gel flash column chromatography using mixtures of petroleum ether and EtOAc as eluents to obtain the corresponding product (**2**).

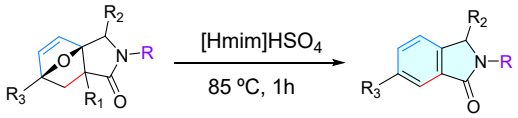
1.5. Intramolecular Diels-Alder Cycloaddition Reaction



(2) **(3)**

A glass pressure tube (reactor) was loaded with 2 wt% Zn-BTC-SA, 3 mL solvent, and 0.3 mmol (**2**). The resulting mixture was stirred at 120 °C for 3 h until the (**2**) was consumed. After the reaction, the catalyst was removed by filtering. Deionized water (10 mL) was added to the filtrate and the mixture was extracted with EtOAc (3 × 10 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and filtered, and the solvents were removed under reduced pressure to obtain DA cycloaddition products (**3**). Further purification was required by flash chromatography (silica gel, EtOAc/petroleum ether) depending on the reaction situation. The reaction was monitored by TLC.

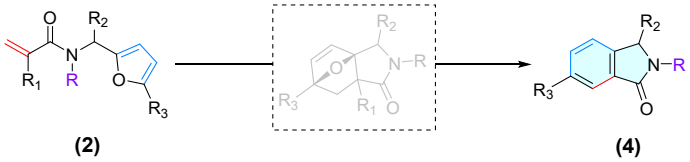
1.6. Dehydrative Aromatization



(3) **(4)**

In a 15 mL glass pressure tube (reactor) was placed DA cycloaddition product (**3**) (1 mmol) in 2 mL of [Hmim]HSO_4 ionic liquid. The resulting mixture was stirred at 85 °C for 1 h until the (**3**) was consumed. After the reaction, deionized water (10 mL) was added and the mixture was extracted with EtOAc (3 × 10 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and filtered, and the solvents were removed under reduced pressure to obtain dehydration products (**4**). Further purification was required by flash chromatography (silica gel, EtOAc/petroleum ether) depending on the reaction situation. The reaction was monitored by TLC.

1.7. DA Cycloaddition/Aromatization Single-step Conversion



(2) **(4)**

Physically combined Zn-BTC-SA + [Hmim]HSO_4:

0.3 mmol (**2**), pre-mixed well Zn-BTC-SA (2 wt%) + [Hmim]HSO_4 (2 mL) was charged into a 15 mL glass pressure tube (reactor). The resulting mixture was stirred at 120 °C for 3 h until the (**2**) was consumed. After the reaction, the catalyst Zn -BTC-SA was removed by filtering. Deionized water (10 mL) was added to the filtrate and the mixture was extracted with EtOAc (3 × 10 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and filtered, the solvents were removed under reduced pressure to obtain products (**4**). Further purification was required by flash chromatography (silica gel, EtOAc/petroleum ether) depending on the reaction situation. The reaction was monitored by TLC.

[Hmim]HSO_4:

0.3 mmol (**2**) and 2 mL [Hmim]HSO_4 were charged into a 15 mL glass pressure tube (reactor). The resulting mixture was stirred at 120 °C for 8 h until the (**2**) was consumed. After the reaction, Deionized water (10 mL) was added and the mixture was extracted with EtOAc (3 × 10 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and filtered, the solvents were removed under reduced pressure to obtain products (**4**). Further purification was required by flash chromatography (silica gel, EtOAc/petroleum ether) depending on the reaction situation. The reaction was monitored by TLC.

Zn-BTC-SA/[Hmim]HSO_4:

A glass pressure tube (reactor) was loaded with 2 wt% Zn-BTC-SA/[Hmim]HSO_4, 0.3 mmoles (**2**), 3 mL DMSO. The resulting mixture was stirred at 120 °C for 3 h until the (**2**) was consumed. After the reaction, the catalyst Zn-BTC-SA/[Hmim]HSO_4 was removed by filtering. Deionized water (10 mL) was added to the filtrate and the mixture was extracted with EtOAc (3×10 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and filtered, the solvents were removed under reduced pressure to obtain products (**4**). Further purification was required by flash chromatography (silica gel, EtOAc/petroleum ether) depending on the reaction situation. The reaction was monitored by TLC.

1.8. Recycling Tests

Zn -BTC-SA: Recycling tests of Zn-BTC-SA were conducted at optimized reaction conditions (2 wt% catalyst, 3mL DMSO, 0.3 mmol (**2a**), at 120 °C for 3 h) to verify the recyclability of Zn-BTC-SA. The used catalyst after the reaction was collected by centrifugation and regenerated by methanol washing for 3 times. The collected residue was directly used for the next reaction.

[Hmim]HSO_4: Recycling tests of [Hmim]HSO_4 were conducted at optimized reaction conditions (2 mL [Hmim]HSO_4, 1 mmol (**3**), at 85 °C for 1 h) to verify the recyclability of [Hmim]HSO_4. After the completion of the reaction, appropriate amounts of deionized water and ethyl acetate were added to the reaction solution for extraction, and the water phase was collected and evaporated under reduced pressure at 75 °C to remove water to obtain the ionic liquid, and it is used directly for the next reaction.

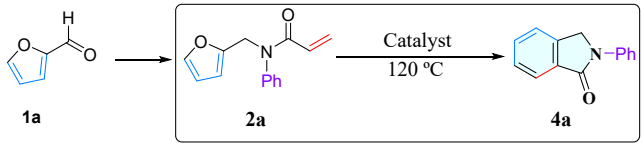
Zn-BTC-SA/[Hmim]HSO_4: Recycling tests of Zn-BTC-SA/[Hmim]HSO_4 were conducted under optimized reaction conditions (2 wt% catalyst, 3 mL DMSO, 0.3 mmol (**2a**), at 120 °C for 3 h) to verify the recyclability of Zn-BTC-SA/[Hmim]HSO_4. The used catalyst after the reaction was collected by centrifugation and regenerated by methanol washing 3 times. The collected catalyst residue was directly used for the next reaction.

1.9. Large-scale Reaction

Two-step Conversion Process

Intramolecular DA cycloaddition: 12 mmol (**2a**), 2 wt% Zn-BTC-SA, and 25 mL DMSO were charged into a 50 mL round bottom flask. The resulting mixture was stirred at 120 °C for 3 h. After the reaction, the catalyst Zn-BTC-SA was removed by filtering. Deionized water (50 mL) was added to the filtrate and the mixture was extracted with EtOAc (3 × 50 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and filtered, the solvents were removed under reduced pressure to obtain DA cycloaddition products (**3a**) (yield: 96%).
Aromatization reaction: 12 mmol (**3a**) and 10 mL [Hmim]HSO_4 were charged into a 25 mL round bottom flask. The resulting mixture was stirred at 85 °C for 2 h. After the reaction, the reaction system is cooled to room temperature and the product was precipitated directly. Deionized water (30 mL) was added and the mixture was filtered to obtain the product (**4a**) with a yield of 96%.

Single-step Conversion Process



Physically combining Zn-BTC-SA + [Hmim]HSO_4:

12 mmol (**2a**), pre-mixed well Zn-BTC-SA (2 wt%) + [Hmim]HSO_4 (10 mL) was charged into a 25 mL round bottom flask. The resulting mixture was stirred at 120 °C for 7 h. After the reaction, the catalyst Zn-BTC-SA was removed by hot filtering. The filtrate was cooled to room temperature and the product was precipitated directly. Deionized water (30 mL) was added and the mixture was filtered to obtain the product (**4a**) with a yield of 90%. The reaction was monitored by TLC.

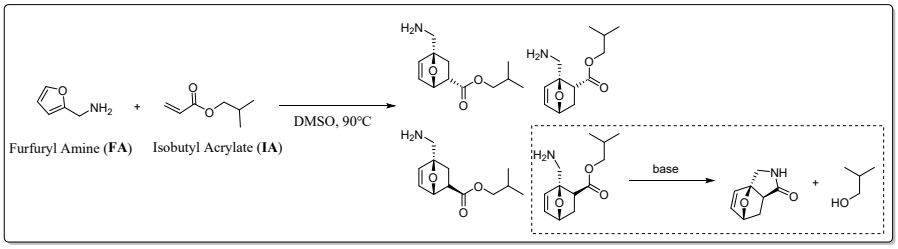
[Hmim]HSO_4:

12 mmol (**2a**) and 10 mL [Hmim]HSO_4 were charged into a 25 mL round bottom flask. The resulting mixture was stirred at 120 °C for 12 h. After the reaction, the reaction system is cooled to room temperature and the product was precipitated directly. Deionized water (30 mL) was added and the mixture was filtered to obtain the product (**4a**) with a yield of 83%. The reaction was monitored by TLC.

Zn-BTC-SA/[Hmim]HSO_4:

12 mmol (**2a**), Zn-BTC-SA/[Hmim]HSO_4 (2 wt%), and 25 mL DMSO were charged into a 50 mL round bottom flask. The resulting mixture was stirred at 120 °C for 5 h. After the reaction, the catalyst was removed by filtering. Deionized water (50 mL) was added to the filtrate, and the mixture was extracted with EtOAc (3 × 50 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and filtered, the solvents were removed under reduced pressure to obtain the product (**4a**) (yield: 93%). The reaction was monitored by TLC.

2. Results and Discussion



Scheme S1. Intermolecular DA reaction between **FA** and **IA**.

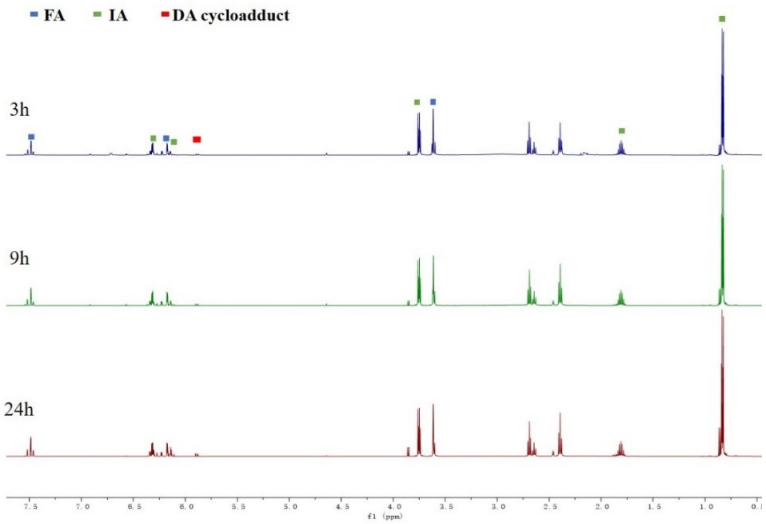


Figure S1. ^{1}H NMR spectra as a function of time for the intermolecular DA cycloaddition in DMSO-d_6 without catalyst at 90 °C

Table S1. Screening of intermolecular DA reaction conditions

entry	solvent	catalyst	T(℃)	DA adduct yield (%) [a]
1	DMSO	-	70	3
2	DMSO	-	80	5
3	DMSO	-	90	8
4	DMSO	$ZnCl_2$	90	9
5	DMSO	$AlCl_3$	90	8
6	DMSO	$Hf(OTf)_4$	90	6
7	DMSO	$SnCl_4$	90	7
8	DMSO	$Bi(NO_3)_3{\cdot}5H_20$	90	5
9	DMSO	$ZrCl_4$	90	7
10	DMSO	$Sc(OTf)_3$	90	7
11	Toluene	$ZnCl_2$	90	6
12	Acetonitrile	$ZnCl_2$	90	7
13	Acetone	$ZnCl_2$	90	7
14	Methanol	$ZnCl_2$	90	5
15	DMSO	$ZnCl_2$	100	9
16	DMSO	$ZnCl_2$	90	11[b]

Reaction conditions: **FA** (1.5 mmol), **IA** (1.5 mmol), solvent (2 mL), catalyst (0.3 eq), 24 h. [a] ^{1}H NMR yields using mesitylene as internal standard. [b] Reaction time 60 h.

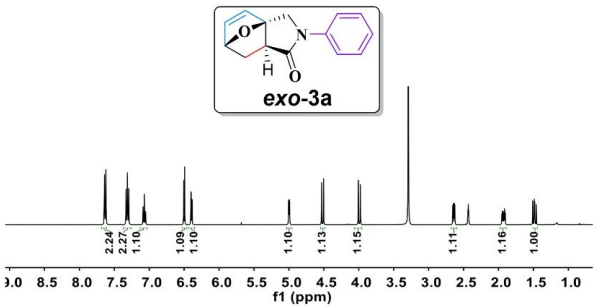


Figure S2. ^{1}H NMR spectrum of *exo*-**3a** after direct extraction.

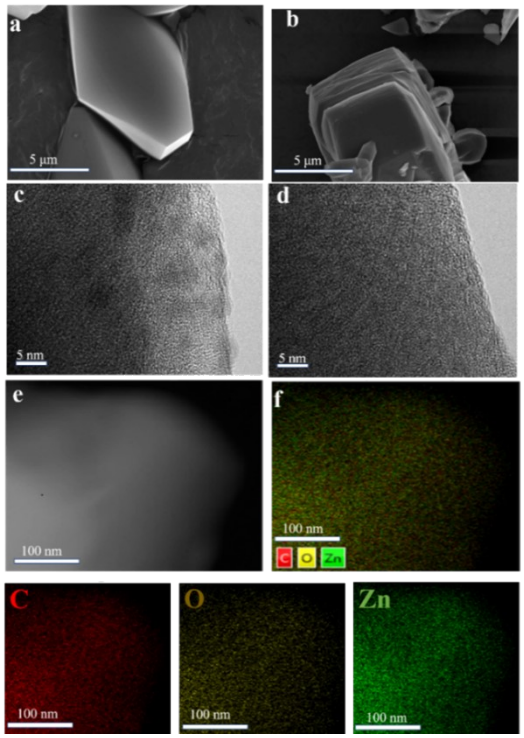


Figure S3. SEM images of Zn-BTC (a) and Zn-BTC-SA (b). HR-TEM images of Zn-BTC (c) and Zn-BTC-SA (d). STEM image (f) as well as C, O, and Zn elemental mappings of Zn-BTC-SA

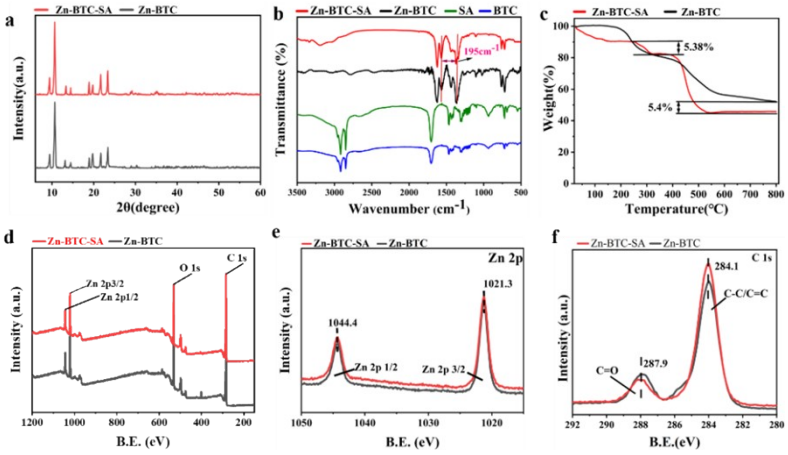


Figure S4. a) Powder XRD patterns, b) FT-IR spectra, c) TGA curves, d) XPS Full-spectrum, e) Zn *2p* XPS peaks, and f) C *1s* XPS peaks of Zn-BTC and Zn-BTC-SA.

The structural integrity of Zn-BTC and Zn-BTC-SA was examined by powder XRD, as shown in **Figure S4 a**. The peaks of Zn-BTC-SA were in good agreement with the characteristic peaks of pristine Zn-BTC, which is in agreement with reported in the literature.[S7] FT-IR spectra of Zn-BTC and Zn-BTC-SA in **Figure S4 b** show the characteristic signals of Zn-O bonds at 728 cm^{-1}, and the transmittance at 1370 and 1565-1630 cm^{-1} for both Zn-BTC and Zn-BTC-SA reveals the symmetric and asymmetric stretching vibrations of the $-CO_2^-$ group, respectively. A splitting of approximately 195 cm^{-1} between these two peaks further indicates that the coordination mode of BTC/SA to Zn is bridging rather than monodentate and bidentate modes.[S8] The FT-IR spectra of Zn-BTC and Zn-BTC-SA exhibit no absorption peak at approximately 2850 and 2920 cm^{-1}, which shows that the -COOH groups of BTC/SA molecules are completely coordinated to Zn^{2+}. TG curves of Zn-BTC-SA, and Zn-BTC present well-resolved weight loss steps (**Figure S4 c**). In the Zn-BTC, the first one before 400 °C weight loss mainly corresponds to the removal of adsorbate H_2O from different sites. The second one above 400 °C results from the fully burned with the decomposition of MOF structure. In the case of Zn-BTC-SA, the weight loss in the range of 25-150 °C is mainly due to the loss of water adsorbate. As Zn-BTC-SA changes into a layered structure after stearic acid is added, the physical adsorption capacity of the Zn-BTC-SA to water is reduced, and rapid dehydration occurs at this stage. The second weight loss observed in the temperature range of 150-320 °C mainly corresponds to the removal of the aliphatic chain of stearic acid.[S5] The final weight loss above 400 °C was attributed to the complete collapse of the Zn-BTC-SA framework as the organic linker (BTC) fully combusts.

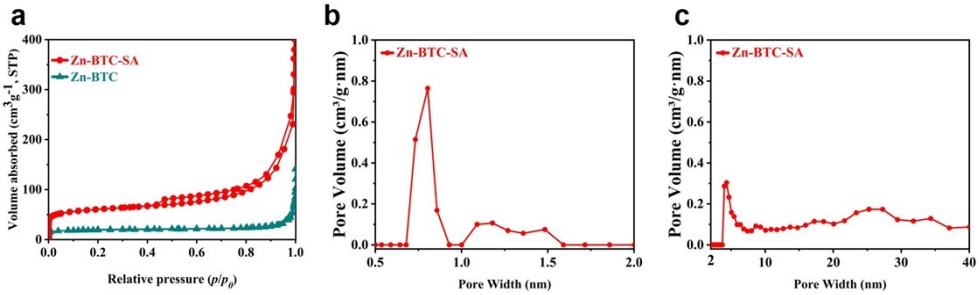


Figure S5. a) Adsorption and desorption isotherm of N_2, b) micropore size distribution, and c) mesopore size distribution of Zn-BTC-SA.

Table S2. Textural properties of Zn-MOF obtained by nitrogen adsorption-desorption experiments.

Samples	BET surface area ($m^2 \cdot g^{-1}$)	Pore volume [a] ($cm^3 \cdot g^{-1}$)	Pore size [b] (nm)	Ref
Zn-BTC-SA	174.66	0.47	2.65	This work
Zn-BTC	61.45	0.10	6.05	This work
Zn-BTC-A	12.64	0.13	5.24	S9
Zn-BTC-S	35.08	0.79	9.04	S9
Zn-BTC-D (No cyclohexan)	49.65	0.86	8.40	S9
Zn-BTC-D	55.05	1.54	13.55	S9

[a] Total pore volume

[b] Average pore diameter calculated using BJH method

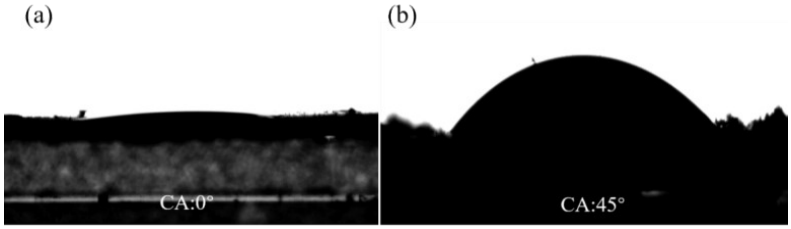


Figure S6. Contact angle (CA) of a water droplet on the surface of Zn-BTC (a) and Zn-BTC-SA (b).

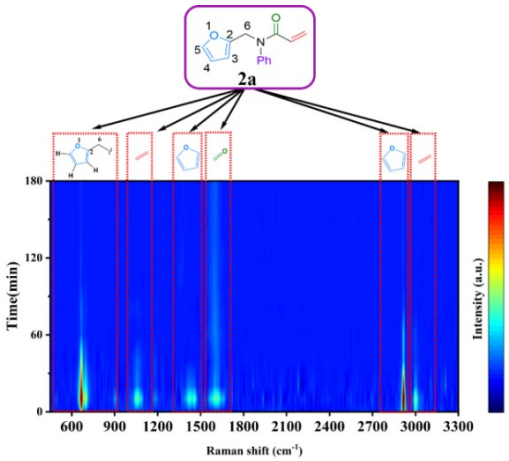


Figure S7. *In-situ* Raman spectra of the liquid mixtures resulting from intramolecular DA cycloaddition of **2a** after varying time.

To obtain more information on the reaction process of the intramolecular DA cycloaddition of **2a**, in-situ Raman spectra of the liquid mixtures at different reaction times were recorded to study the benchmark reaction using Zn-BTC-SA as the catalyst. Besides the bands of benzene in the catalyst and the used solvent (DMSO), **2a** shows several characteristic bands, including the terminal olefinic C-H stretching at 3001 cm^{-1} and C=C stretching at 1054 cm^{-1}, strong C-H/C=C bond stretching, C-H symmetric waggle, and O1–C2–C6 waggle of furan ring at 2910, 1443, 671, and 659 cm^{-1}, respectively, and C=O stretching band at 1607 cm^{-1} (Figure 3).[S10] Initially, the absorption peaks of diene and dienophile were enhanced after mixing the substrate with Zn-BTC-SA, demonstrating that the catalyst can rapidly contact/combine with the substrate **2a**. With the progress of the reaction, the absorption peaks of the furan ring and terminal olefin disappeared almost in 60 min. Simultaneously, the C=O stretching band was observed to have a slight shift toward the high wavenumber due to the disappearance of its conjugated system after undergoing the DA cycloaddition.

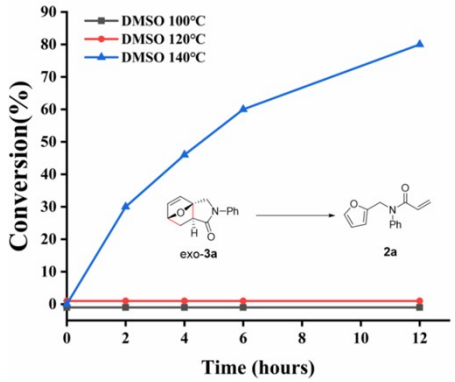


Figure S8. Temperature stability of *exo*-**3a** with respect to the retro-Diels-Alder reaction giving **2a**.
These experiments were run in an NMR tube with periodic quantitative ^{1}H NMR analysis used to determine conversion.

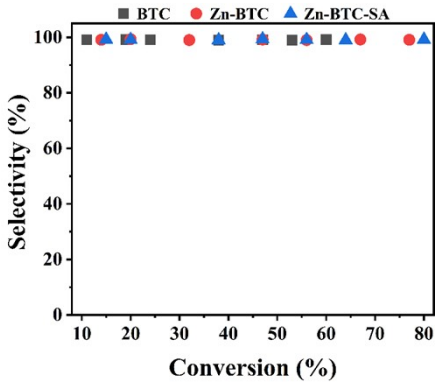


Figure S9. Selectivity of *exo*-**3a** as a function of the conversion under different catalyst conditions.

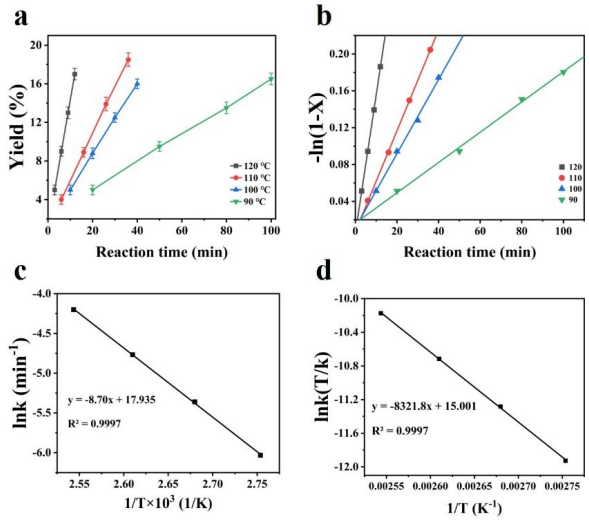


Figure S10. (a) Kinetics of DA cycloaddition process catalyzed by Zn-BTC-SA at different temperatures (reaction conditions: 0.5 mmol of **2a**, 3 mL of DMSO, Zn-BTC-SA dosage of 2 wt %); (b) Relationship between -ln(1-X) and t at different reaction temperatures; (c) Arrhenius plot lnk versus 1/T × 103 (1/K). (d) Eyring-Polanyi plot between ln (k/T) and 1/T.

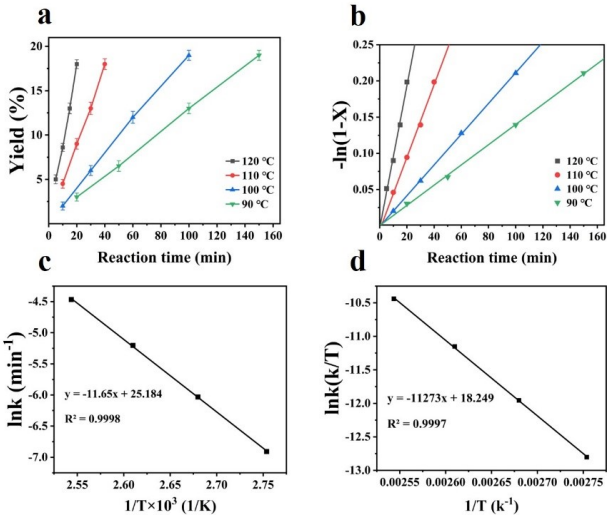


Figure S11. (a) Kinetics of DA cycloaddition process catalyzed by Zn-BTC at different temperatures (reaction conditions: 0.5 mmol of **2a**, 3 mL of DMSO, Zn-BTC dosage of 2 wt %); (b) Relationship between -ln(1-X) and t at different reaction temperatures; (c) Arrhenius plot lnk versus $1/T \times 10^3$ (1/K). (d) Eyring-Polanyi plot between ln (k/T) and 1/T.

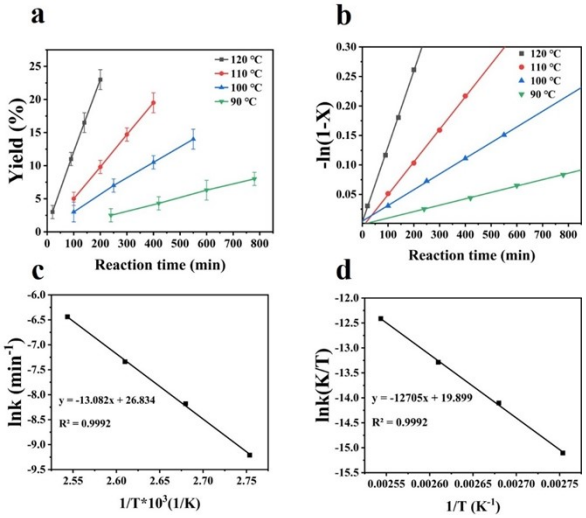


Figure S12. (a) Kinetics of DA cycloaddition process at different temperatures without catalyst (reaction conditions: 0.5 mmol of **2a**, 3 mL of DMSO); (b) Relationship between -ln(1-X) and t at different reaction temperatures; (c) Arrhenius plot lnk versus $1/T \times 10^3$ (1/K). (d) Eyring-Polanyi plot between ln (k/T) and 1/T.

The activation energy can be calculated by the Arrhenius equation:

$$\ln k = \ln A - \frac{Ea}{RT}$$

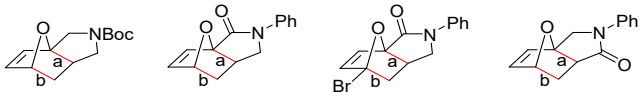
where Ea is the activation energy in kJ mol^{-1}(or kcal/mol), A is the frequency factor in min^{-1}, R is the universal molar gas constant equal to 8.314 J K^{-1} mol^{-1} and T is the temperature (K).

The thermodynamic properties of the reaction were studied by the Eyring-Polanyi equation:

$$\ln\left(\frac{k}{T}\right) = -\frac{\Delta H}{RT} + \ln\left(\frac{k_b}{h}\right) + \frac{\Delta S}{R}$$

where k is the rate constant at temperature T and ΔH and ΔS are the variations in the enthalpy and entropy of the reaction system, respectively. k_b, h and R are the Boltzmann, Planck, and universal gas constants, respectively. ΔH and ΔS were calculated from the slope and y-intercept of the Eyring-Polanyi plot for the reaction.

Table S3. Typical intramolecular DA cycloadduct C–C bond distances



A B C 3a

Cycloadduct	C–C bond length a	C–C bond length b	Ref.
A	1.576	1.572	S11
B	1.558	1.571	S11
C	1.558	1.565	S11
3a	1.538	1.541	This work

All distances in angstroms

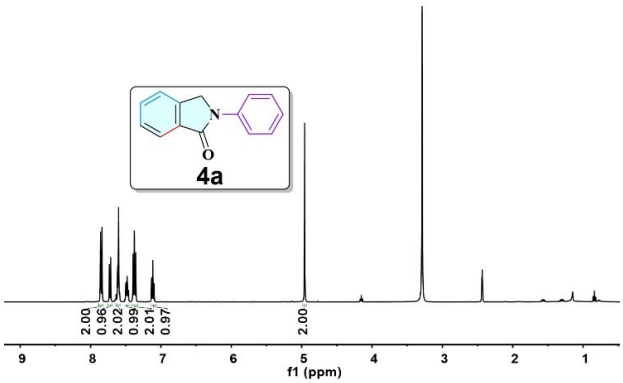


Figure S13. ^{1}H NMR spectrum of **4a** after direct extraction.

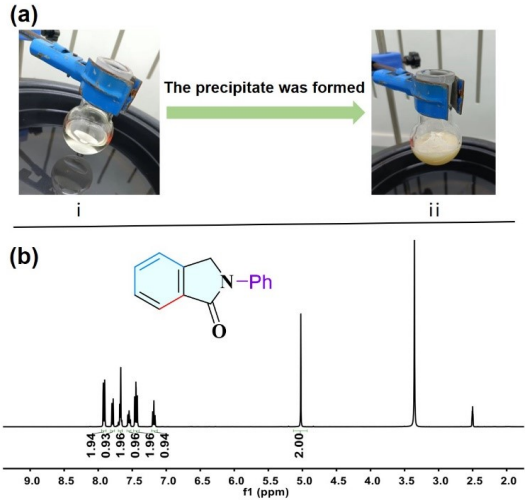
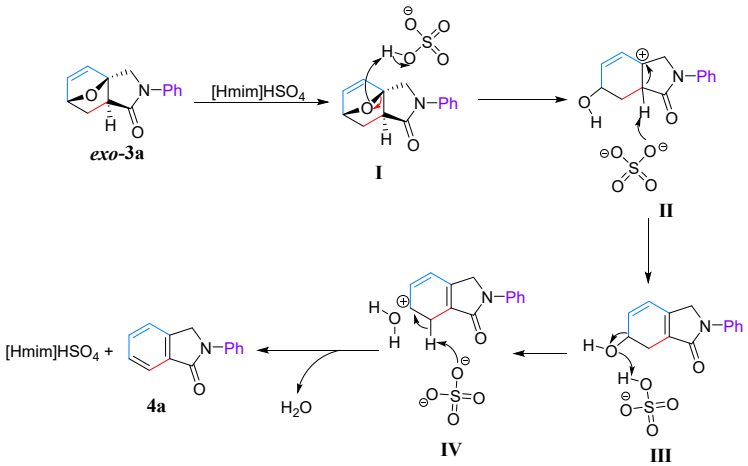


Figure. S14. a) The experimental phenomenon of after the completion of scale-up dehydration reaction: (i) Reaction just completed; (ii) The reaction is completely cooled; b) ^{1}H NMR spectrum of **4a** after filtration.



Scheme S2. The possible mechanism of *exo*-**3a**-to-**4a** dehydration

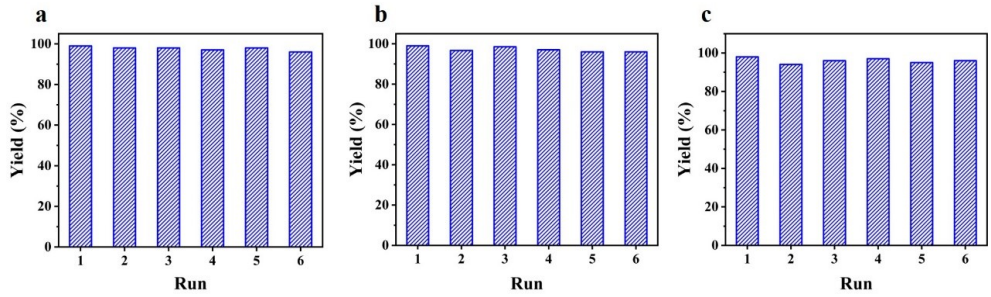


Figure S15. (**a**) Recycling test of Zn-BTC-SA; Reaction conditions: 2 wt% Zn-BTC-SA, 3 mL DMSO, 0.3 mmol (**2a**), 120 °C and 3 h. (**b**): Recycling test of $[Hmim]HSO_4$; Reaction conditions: 2 mL $[Hmim]HSO_4$, 1 mmol (**3a**), 85 °C, and 1 h. (**c**) Recycling test of Zn-BTC-SA/$[Hmim]HSO_4$; Reaction conditions: 2 wt% Zn-BTC-SA@$[Hmim]HSO_4$, 3 mL DMSO, 0.3 mmol (**2a**), 120 °C and 3 h.

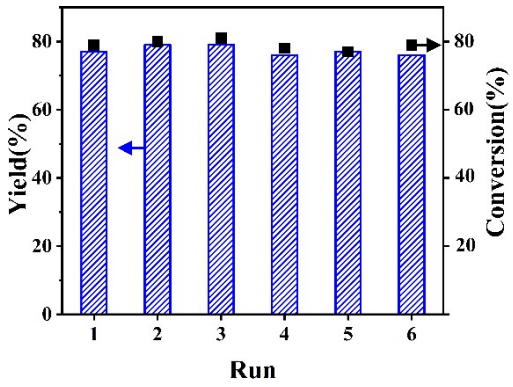


Figure S16. Recycling tests of DA cycloaddition with low conversion rate

Reaction conditions: 1 wt% Zn-BTC-SA, 3 mL DMSO, 0.3 mmol (**2a**), 120 °C, and 3 h.

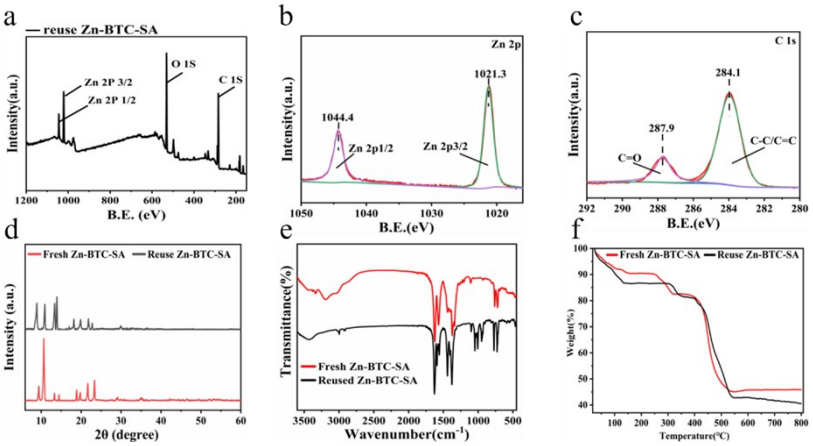


Figure S17. XPS, XRD, FT-IR, and TG analysis of reused Zn-BTC-SA.

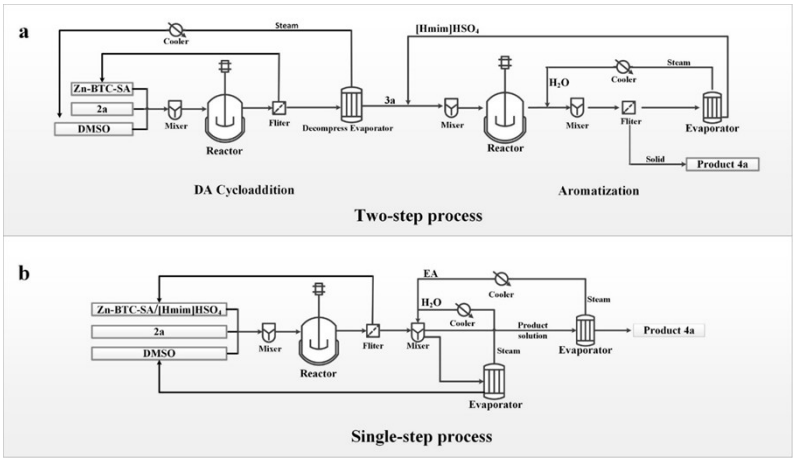


Figure S18. A conceptual process for the production process of renewable isoindolinone. a) Two-step series reaction DA cycloaddition with Zn-BTC-SA and aromatization with [Hmim]HSO_4, and b) One-step method of DA addition and aromatization catalyzed by Zn-BTC-SA/[Hmim]HSO_4.

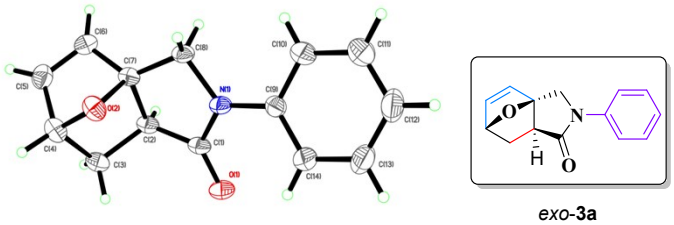
Lab scale trial process:

a) ***Two-step process:*** 12 mmol (**2a**), 2 wt% Zn-BTC-SA, and 25 mL DMSO were charged into a 50 mL round bottom flask and then heated at 120 °C for 3 h. After the reaction was completed, it was cooled to room temperature, and the catalyst was recovered by filtration. The filtrate was distilled under reduced pressure at 80 °C by an oil pump to recover the organic solvent DMSO, and a viscous DA cyclic adduct (**3a**) was obtained. Then 10 mL [Hmim]HSO_4 was added, and the two were mixed evenly at room temperature, and then placed in the preheated 85 °C oil bath. After the reaction, the system was cooled to room temperature and the product was precipitated. The pure final product isoindolinone (**4a**) was obtained with a yield of 90% by adding an appropriate amount of deionized water to dilute the [Hmim]HSO_4 and filter directly. The filtrate was further evaporated under reduced pressure to remove water and recover the [Hmim]HSO_4.

b) ***One-step process:*** 12 mmol (**2a**), 2 wt% Zn-BTC-SA/[Hmim]HSO_4, and 25 mL DMSO were charged into a 50 mL round bottom flask and then heated at 120 °C for 5 h. After the reaction, the catalyst was removed by filtering. Deionized water (50 mL) was added to the filtrate and the mixture was extracted with EtOAc (50 mL). The organic layer solvents were removed under reduced pressure to obtain the product (**4a**) to recover the EtOAc. The aqueous phase was further evaporated under reduced pressure to remove water and recover DMSO.

3. X-Ray Crystal Structure Data

3.1. X-ray Ellipsoid Plots of *exo*-3a (CCDC 2165681)



exo-**3a**

Figure S19. Molecular structure of *exo*-**3a** in the crystal.

X-ray crystallographic data for (*exo*-3a): $C_{14}H_{13}NO_2$, Fw = 227.25, colourless block, 0.24 x 0.12 x 0.07 mm^3, Orthorhombic, P*bca* (no. 61), a = 8.6581(15) Å, b = 12.1447(18) Å, c = 22.107(3) Å, V = 2324.6(6) $Å^3$, Z =8, Dx = 1.299 g/cm^3, μ =0.705 mm^{-1}. CCDC **2165681** contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via https://ccdc.cam.ac.uk/mystructures/.

Table S4 Crystal structure data for *exo*-**3a**.

Identification code	*exo*-**3a**
Empirical formula	$C_{14}H_{13}NO_2$
Formula weight	227.25
Temperature (K)	293(2)
Wavelength (Å)	1.54178
Crystal system	Orthorhombic
Space group	P*bca*
Unit cell dimensions	a = 8.6581(15) Å b = 12.1447(18) Å c = 22.107(3) Å a= 90° β= 90° γ= 90°
Volume $(Å)^3$	2324.6(6)
Z	8
Density (calculated) (mg/m^3)	1.299
Absorption coefficient (mm^{-1})	0.705
F(000)	960
Crystal size (mm)	0.24 x 0.12 x 0.07
Theta range for data collection (°)	4.00 to 66.04.
Index ranges	-10<=h<=7, -8<=k<=14, -26<=l<=25
Reflections collected	4619
Independent reflections	2034 [R(int) = 0.0527]
Completeness to theta = 66.04°	100.00%
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9523 and 0.8491
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2034 / 0 / 155
Goodness-of-fit on F^2	1
Final R indices [I>2sigma(I)]	R1 = 0.0577, wR2 = 0.1195
R indices (all data)	R1 = 0.1112, wR2 = 0.1568
Extinction coefficient	0.0046(3)
Largest diff. peak and hole $(e.Å^{-3})$	0.182 and -0.215

3.2. X-ray Ellipsoid Plots of *exo*-3c (CCDC 2179840)

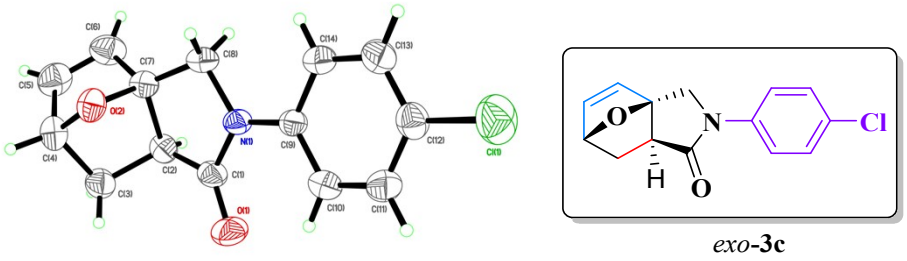


Figure S20. Molecular structure of *exo*-**3c** in the crystal.

X-ray crystallographic data for (*exo*-3c): $C_{14}H_{12}ClNO_2$, Fw = 261.70, colourless block, 0.41 x 0.40 x 0.12 mm^3, Monoclinic, P2(1)/c, a = 13.6538(11) Å, b = 9.0781(8) Å, c = 10.6852(9) Å, 1255.83(18) $Å^3$, Z=4, Dx = 1.384 g/cm^3, μ = 0.297 mm^{-1}. CCDC **2179840** contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via https://ccdc.cam.ac.uk/mystructures/.

Table S5. Crystal structure data for *exo*-**3c**.

Identification code	*exo*-**3c**
Empirical formula	$C_{14}H_{12}ClNO_2$
Formula weight	261.70
Temperature (K)	298(2)
Wavelength (Å)	0.71073
Crystal system	Monoclinic
space group	*P2(1)/c*
Unit cell dimensions	a=13.6538(11) Å b=9.0781(8) Å c=10.6852(9) Å α=90° β=108.522(4)° γ=90°
Volume ($Å^{-3}$)	1255.83(18)
Z	4
Calculated density (mg/m^3)	1.384
Absorption coefficient (mm^{-1})	0.297
F(000)	544
Crystal size (mm)	0.41 x 0.40 x 0.12
Theta range for data collection (°)	2.74 to 25.02
Limiting indices	-16<=h<=16, -10<=k<=10, -7<=l<=12
Reflections collected / unique	5847 / 2215 [R(int) = 0.0499]
Completeness to theta = 25.02	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9653 and 0.8881
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2215 / 0 / 163
Goodness-of-fit on F^2	1.079
Final R indices [I>2sigma(I)]	R1 = 0.0481, wR2 = 0.0946
R indices (all data)	R1 = 0.0832, wR2 = 0.1024
Largest diff. peak and hole ($e.A^{-3}$)	0.164 and -0.302

3.3. X-ray ellipsoid plots of *exo*-3l (CCDC 2209873)

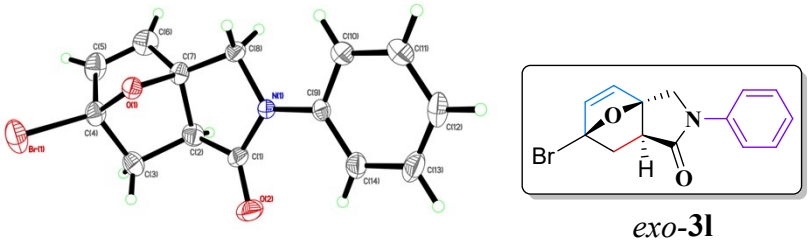


Figure S21. Molecular structure of *exo*-**3l** in the crystal.

X-ray crystallographic data for (*exo*-3l): $C_{14}H_{12}BrNO_2$, Fw = 306.16, colourless block, 0.40 x 0.37 x 0.30 mm^3, Orthorhombic, P2(1)2(1)2(1), a = 5.4795(4) Å, b = 11.4206(9) Å, c = 19.3592(16) Å, V = 1211.48(16) $Å^3$, Z = 4, Dx = 1.679 g/cm^3, μ = 3.385 mm^{-1}. CCDC **2209873** contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via https://ccdc.cam.ac.uk/mystructures/.

Table S6. Crystal structure data for *exo*-**3l**.

Identification code	*exo*-**3l**
Empirical formula	$C_{14}H_{12}BrNO_2$
Formula weight	306.16
Temperature (K)	298(2)
Wavelength (Å)	0.71073
Crystal system	Orthorhombic
Space group	*P2(1)2(1)2(1)*
Unit cell dimensions	a = 5.4795(4) Å b = 11.4206(9) Å c = 19.3592(16) Å α= 90° β= 90° γ = 90°
Volume ($Å^3$)	1211.48(16)
Z	4
Density (calculated) (mg/m^3)	1.679
Absorption coefficient (mm^{-1})	3.385
F(000)	616
Crystal size (mm)	0.400 x 0.370 x 0.030
Theta range for data collection (°)	2.76 to 25.01
Index ranges	-5<=h<=6, -13<=k<=13, -19<=l<=23
Reflections collected	5833
Independent reflections	2136 [R(int) = 0.0566]
Completeness to theta = 66.032°	99.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.4299 and 0.3445
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	2136 / 0 / 164
Goodness-of-fit on F^2	1.013
Final R indices [I>2sigma(I)]	R1 = 0.0413, wR2 = 0.0881
R indices (all data)	R1 = 0.0543, wR2 = 0.0922
Extinction coefficient	n/a
Largest diff. peak and hole ($e.Å^{-3}$)	0.379 and -0.482

3.4 X-ray ellipsoid plots of 4a (CCDC 2165682)

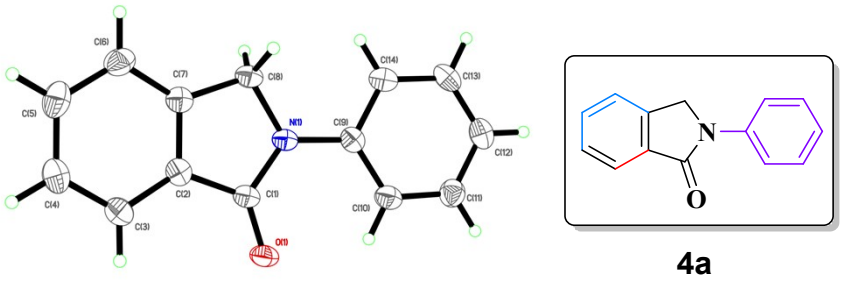


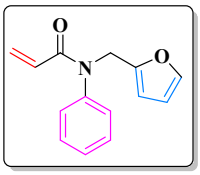
Figure S22. Molecular structure of **4a** in the crystal.

X-ray crystallographic data for (4a): $C_{14}H_{11}NO$, Fw = 209.24, colourless block, 0.400 x 0.320 x 0.030 mm^3, Monoclinic, P21/n, a = 5.9303(7) Å, b = 7.6388(9) Å, c = 23.149(3) Å, V = 1045.4(2) $Å^3$, Z =4, Dx = 1.329 g/cm^3, μ = 0.667 mm^{-1}. CCDC **2165682** contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via https://ccdc.cam.ac.uk/mystructures/.

Table S7. Crystal structure data for **4a**.

Identification code	4a
Empirical formula	$C_{14}H_{11}NO$
Formula weight	209.24
Temperature (K)	293(2)
Wavelength (Å)	1.54178
Crystal system	Monoclinic
Space group	*P21/n*
Unit cell dimensions	a = 5.9303(7) Å b = 7.6388(9) Å c = 23.149(3) Å α= 90° β= 94.506(3)° γ = 90°.
Volume ($Å^3$)	1045.4(2)
Z	4
Density (calculated) (mg/m^3)	1.329
Absorption coefficient (mm^{-1})	0.667
F(000)	440
Crystal size (mm)	0.400 x 0.320 x 0.030
Theta range for data collection (°)	6.102 to 66.032.
Index ranges	-7<=h<=6, -9<=k<=9, -3<=l<=27
Reflections collected	1776
Independent reflections	1776 [R(int) = 0.0428]
Completeness to theta = 66.032°	98.20%
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9803 and 0.7763
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1776 / 0 / 146
Goodness-of-fit on F^2	1.395
Final R indices [I>2sigma(I)]	R1 = 0.0905, wR2 = 0.2340
R indices (all data)	R1 = 0.1104, wR2 = 0.2474
Extinction coefficient	n/a
Largest diff. peak and hole ($e.Å^{-3}$)	0.282 and -0.260

4. Spectra Data of Products

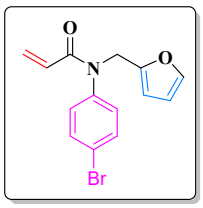


N-(furan-2-ylmethyl)-N-phenylacrylamide (2a): Following the general procedure, compound **2a** was obtained as a brown oil (2.64 g, 93% yield).

R_f = 0.73 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.29 (ddd, *J* = 8.7, 5.4, 2.3 Hz, 2H), 7.26 – 7.23 (m, 1H), 7.19 (s, 1H), 7.04 – 6.97 (m, 2H), 6.34 (dd, *J* = 16.8, 2.0 Hz, 1H), 6.19 (dd, *J* = 3.1, 1.9 Hz, 1H), 6.10 (d, *J* = 3.1 Hz, 1H), 5.93 (dd, *J* = 16.7, 10.3 Hz, 1H), 5.46 (dd, *J* = 10.3, 1.8 Hz, 1H), 4.87 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 164.63, 150.87, 142.99, 141.58, 129.94, 129.02, 128.52, 128.44, 128.33, 110.92, 109.11,45.40.

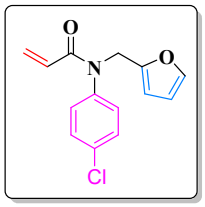


N-(4-bromophenyl)-N-(furan-2-ylmethyl)acrylamide (2b): Following the general procedure, compound **2b** was obtained a slight yellow oil (3.40 g, 89% yield).

R_f = 0.71 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.65 – 7.59 (m, 2H), 7.56 (d, *J* = 1.8 Hz, 1H), 7.15 – 7.07 (m, 2H), 6.34 (dd, *J* = 3.2, 1.9 Hz, 1H), 6.27 – 6.16 (m, 2H), 5.99 (s, 1H), 5.69 – 5.57 (m, 1H), 4.92 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 164.58, 150.61, 143.16, 140.89, 132.87, 130.69, 128.96, 121.59, 121.26, 110.95, 109.32, 45.23.

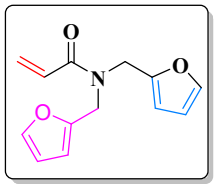


N-(4-chlorophenyl)-N-(furan-2-ylmethyl)acrylamide (2c): Following the general procedure, compound **2c** was obtained a brown oil (2.90 g, 87% yield).

R_f = 0.64 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.55 (s, 1H), 7.52 – 7.39 (m, 2H), 7.16 (d, *J* = 8.6 Hz, 2H), 6.33 (q, *J* = 1.5 Hz, 1H), 6.27 – 6.16 (m, 2H), 5.99 (s, 1H), 5.69 – 5.58 (m, 1H), 4.97 – 4.91 (m, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 164.61, 150.63, 143.13, 140.47, 132.81, 130.35, 129.91, 128.95, 128.78, 110.93, 109.31, 45.28.

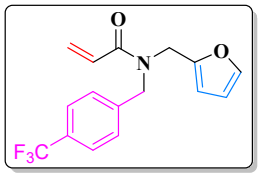


N,N-bis(furan-2-ylmethyl)acrylamide (2d): Following the general procedure, compound **2d** was obtained a slight yellow to brown oil (2.54 g, 88% yield).

R_f = 0.54 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.58 – 7.44 (m, 2H), 6.84 (dd, *J* = 16.6, 10.3 Hz, 1H), 6.33 (q, *J* = 2.7 Hz, 2H), 6.24 (d, *J* = 2.8 Hz, 2H), 6.15 (dd, *J* = 16.6, 2.4 Hz, 1H), 5.69 (dd, *J* = 10.4, 2.4 Hz, 1H), 4.51 (d, *J* = 19.3 Hz, 4H).

^{13}C NMR (101 MHz, DMSO-d6) δ 165.76, 151.05, 151.01, 143.42, 143.02, 128.82, 128.56, 110.96, 109.02, 108.62, 43.77, 41.73.

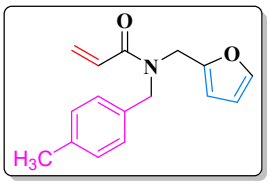


N-(furan-2-ylmethyl)-N-(4-(trifluoromethyl)benzyl)acrylamide (2e): Following the general procedure, compound **2e** was obtained a slight yellow to brown oil (3.52 g, 91% yield).

R_f = 0.53 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.76 – 7.51 (m, 3H), 7.37 (dd, *J* = 30.4, 8.0 Hz, 2H), 7.07 – 6.62 (m, 1H), 6.43 – 6.28 (m, 2H), 6.25 (dd, *J* = 16.5, 2.7 Hz, 1H), 5.75 (ddd, *J* = 37.9, 10.3, 2.4 Hz, 1H), 4.84 – 4.49 (m, 4H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 166.26, 150.95, 143.52, 143.08, 131.11, 129.96, 128.93, 128.58, 127.51, 125.65, 110.96, 109.00, 48.96, 44.51.

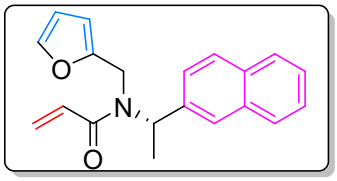


N-(furan-2-ylmethyl)-N-(4-methylbenzyl)acrylamide (2f): Following the general procedure, compound **2f** was obtained a slight yellow oil (2.87 g, 90% yield).

R_f = 0.58 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.60 (dd, *J* = 18.5, 1.8 Hz, 1H), 7.19 – 7.00 (m, 4H), 6.99 – 6.62 (m, 1H), 6.44 – 6.36 (m, 1H), 6.36 – 6.29 (m, 1H), 6.24 (ddd, *J* = 13.1, 7.0, 3.5 Hz, 1H), 5.73 (ddd, *J* = 30.6, 10.3, 2.4 Hz, 1H), 4.60 (s, 2H), 4.53 (s, 2H), 2.28 (s, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 166.00, 151.08, 143.48, 136.71, 134.84, 129.68, 129.46, 128.23, 126.88, 110.98, 108.81, 48.35, 43.68, 21.13.

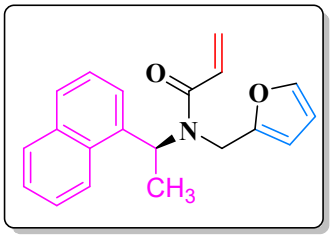


(S)-N-(furan-2-ylmethyl)-N-(1-(naphthalen-2-yl)ethyl)acrylamide (2g): Following the general procedure, compound **2g** was obtained a brown oil (3.32 g, 87% yield).

R_f = 0.61 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.88 (ddd, *J* = 22.5, 11.6, 6.3 Hz, 4H), 7.51 (tt, *J* = 6.0, 2.3 Hz, 3H), 7.36 (d, *J* = 8.6 Hz, 1H), 6.83 (dd, *J* = 16.5, 10.4 Hz, 1H), 6.41 – 6.16 (m, 2H), 6.18 – 6.05 (m, 1H), 6.07 – 5.86 (m, 1H), 5.82 – 5.63 (m, 1H), 4.46 (dd, *J* = 145.0, 17.9 Hz, 2H), 1.57 (d, *J* = 6.2 Hz, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 166.25, 152.45, 142.76, 142.12, 139.00, 137.09, 133.30, 132.63, 129.45, 128.40, 127.86, 126.62, 126.39, 125.80, 125.09, 111.01, 107.87, 60.23, 47.56, 17.12.

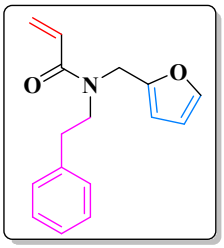


(S)-N-(furan-2-ylmethyl)-N-(1-(naphthalen-1-yl)ethyl)acrylamide (2h): Following the general procedure, compound **2h** was obtained a brown oil (3.40 g, 89% yield).

R_f = 0.63 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.92 (t, *J* = 6.0 Hz, 3H), 7.68 (d, *J* = 7.3 Hz, 1H), 7.59 – 7.48 (m, 4H), 7.33 (d, *J* = 1.8 Hz, 1H), 6.77 (dd, *J* = 16.6, 10.2 Hz, 1H), 6.59 (d, *J* = 7.1 Hz, 1H), 6.45 – 6.29 (m, 1H), 6.21 – 6.01 (m, 1H), 5.84 – 5.69 (m, 1H), 5.51 (d, *J* = 3.2 Hz, 1H), 4.27 (dd, *J* = 122.5, 17.6 Hz, 2H), 1.54 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 165.65, 151.93, 142.42, 135.91, 133.78, 132.08, 129.22, 129.06, 128.72, 126.94, 126.22, 125.83, 125.78, 125.61, 123.52, 110.63, 107.19, 48.31, 43.99, 17.16.

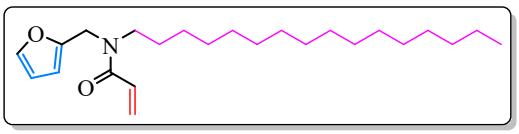


N-(furan-2-ylmethyl)-N-phenethylacrylamide (2i): Following the general procedure, compound **2i** was obtained a brown oil (2.94 g, 92% yield).

R_f = 0.46 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.62 (dd, *J* = 8.7, 1.7 Hz, 1H), 7.34 – 7.13 (m, 5H), 6.77 (ddd, *J* = 108.6, 16.6, 10.3 Hz, 1H), 6.46 – 6.34 (m, 2H), 6.26 – 6.07 (m, 1H), 5.77 – 5.58 (m, 1H), 4.60 (d, *J* = 10.3 Hz, 2H), 3.54 (q, *J* = 8.9, 8.2 Hz, 2H), 2.77 – 2.66 (m, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 165.57, 143.47, 143.05, 139.57, 138.80, 129.34, 128.88, 128.09, 126.86, 111.03, 108.64, 48.74, 41.67, 35.21.

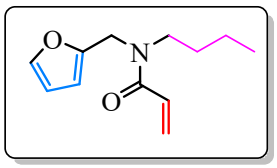


N-(furan-2-ylmethyl)-N-hexadecylacrylamide (2j): Following the general procedure, compound **2j** was obtained a colorless oil (4.08 g, 87% yield)**.**

R_f = 0.68 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.65 – 7.53 (m, 1H), 6.80 (ddd, *J* = 59.2, 16.6, 10.4 Hz, 1H), 6.45 – 6.26 (m, 2H), 6.16 (ddd, *J* = 16.7, 12.0, 2.5 Hz, 1H), 5.69 (ddd, *J* = 9.9, 7.0, 2.5 Hz, 1H), 4.58 (d, *J* = 17.9 Hz, 2H), 1.50 – 1.33 (m, 2H), 1.23 (d, *J* = 7.2 Hz, 28H), 0.93 – 0.80 (m, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 165.43, 151.66, 142.85, 133.47, 128.21, 108.80, 108.42, 47.23, 44.39, 31.76, 29.49, 27.38, 26.43, 22.56, 14.41.

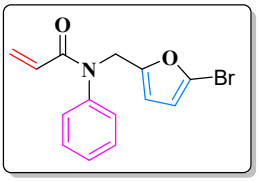


N-butyl-N-(furan-2-ylmethyl)acrylamide (2k): Following the general procedure, compound **2k** was obtained a brown oil (2.33 g, 90% yield).

R_f = 0.63 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.60 (d, *J* = 15.5 Hz, 1H), 6.82 (ddd, *J* = 57.5, 16.6, 10.4 Hz, 1H), 6.48 – 6.30 (m, 2H), 6.26 – 6.11 (m, 1H), 5.70 (td, *J* = 10.2, 2.5 Hz, 1H), 4.60 (d, *J* = 15.5 Hz, 2H), 3.42 – 3.36 (m, 2H), 3.31 (s, 2H), 1.49 – 1.33 (m, 2H), 1.23 (hd, *J* = 7.2, 3.1 Hz, 2H), 0.86 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 165.68, 151.64, 142.88, 129.03, 127.90, 108.82, 108.45, 46.99, 44.37, 29.56, 19.73, 14.08.

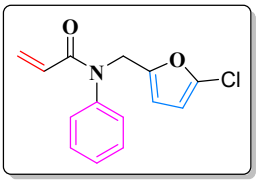


N-((5-bromofuran-2-yl)methyl)-N-phenylacrylamide (2l): Following the general procedure, compound **2l** was obtained a brown oil (3.52 g, 92% yield).

R_f = 0.59 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.49 – 7.39 (m, 2H), 7.39 – 7.29 (m, 1H), 7.18 – 7.10 (m, 2H), 6.42 (d, *J* = 3.3 Hz, 1H), 6.25 (dd, *J* = 2.8, 1.8 Hz, 1H), 6.20 (d, *J* = 2.3 Hz, 1H), 5.96 (s, 1H), 5.61 (dd, *J* = 9.8, 2.1 Hz, 1H), 4.92 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d6) δ164.66,153.35, 141.38, 130.02, 128.89, 128.63, 128.54, 128.43, 120.90, 112.82, 112.17, 45.38.

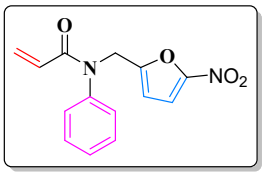


N-((5-chlorofuran-2-yl)methyl)-N-phenylacrylamide (2m): Following the general procedure, compound **2m** was obtained a brown oil (2.94 g, 90% yield).

R_f = 0.58 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d6) δ 7.43 (dd, J = 8.2, 6.6 Hz, 2H), 7.39 – 7.32 (m, 1H), 7.22 – 7.12 (m, 2H), 6.31 (d, J = 3.3 Hz, 1H), 6.27 (d, J = 3.4 Hz, 1H), 6.22 (dd, J = 16.8, 2.3 Hz, 1H), 5.65 – 5.50 (m, 2H), 4.91 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d6) δ 164.66, 151.15, 141.36, 134.47, 130.01, 128.87, 128.53, 128.42, 119.95, 111.85, 107.92, 40.89.

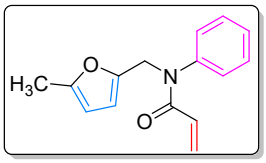


N-((5-nitrofuran-2-yl)methyl)-N-phenylacrylamide (2n): Following the general procedure, compound **2n** was obtained a yellow oil (3.12 g, 93% yield).

R_f = 0.56 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.55 (d, J = 3.7 Hz, 1H), 7.44 – 7.37 (m, 2H), 7.36 – 7.30 (m, 1H), 7.25 – 7.19 (m, 2H), 6.63 (d, J = 3.8 Hz, 1H), 6.19 (dd, J = 16.8, 2.2 Hz, 1H), 6.03 – 5.86 (m, 1H), 5.60 (dd, J = 10.3, 2.3 Hz, 1H), 5.01 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d6) δ 164.94, 155.68, 155.68, 151.58, 141.35, 130.19, 129.23, 129.08, 128.54, 114.42, 113.02, 45.96.

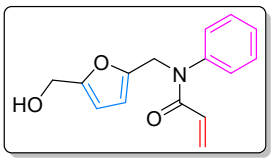


N-((5-methylfuran-2-yl)methyl)-N-phenylacrylamide(2o): Following the general procedure, compound **2o** was obtained a brown oil (2.87 g, 95% yield).

R_f = 0.66 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.49 – 7.37 (m, 2H), 7.37 – 7.26 (m, 1H), 7.17 – 7.09 (m, 2H), 6.21 (dd, *J* = 16.8, 2.3 Hz, 1H), 6.09 – 5.85 (m, 3H), 5.58 (dd, *J* = 10.3, 2.3 Hz, 1H), 4.87 (s, 2H), 2.17 (s, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 164.51, 151.56, 149.02, 141.58, 129.88, 129.07, 128.60, 128.35, 128.28, 109.93, 106.81, 45.43, 13.71.

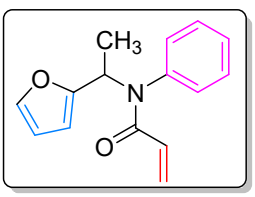


N-((5-(hydroxymethyl)furan-2-yl)methyl)-N-phenylacrylamide (2p): The product containing the ester group was obtained by a general procedure, which was further hydrolyzed under acidic conditions to give the compound **2p** with a brown oil (2.10 g, 65% yield).

R_f = 0.45 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.39 – 7.31 (m, 2H), 7.20 – 7.14 (m, 2H), 7.11 (tt, *J* = 7.4, 1.4 Hz, 1H), 6.40 – 6.29 (m, 2H), 6.26 (d, *J* = 8.4 Hz, 1H), 6.09 (dd, *J* = 9.9, 1.7 Hz, 1H), 6.02 (dd, *J* = 10.0, 1.7 Hz, 1H), 4.85 (s, 2H), 4.62 (d, *J* = 6.8 Hz, 2H), 3.98 (t, *J* = 6.8 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 166.96, 154.19, 151.00, 141.04, 129.41, 128.45, 127.91, 124.54, 122.05, 108.74, 107.81, 57.23, 39.40.

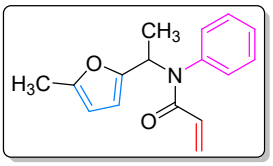


N-(1-(furan-2-yl)ethyl)-N-phenylacrylamide (2q): Following the general procedure, compound **2q** was obtained a brown oil (2.20 g, 72% yield).

R_f = 0.65 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.39 – 7.31 (m, 3H), 7.32 – 7.25 (m, 2H), 7.10 (tt, *J* = 7.2, 1.5 Hz, 1H), 6.45 (dd, *J* = 7.8, 1.4 Hz, 1H), 6.41 – 6.30 (m, 2H), 6.10 (dd, *J* = 10.1, 1.8 Hz, 1H), 6.02 (dd, *J* = 9.9, 1.6 Hz, 1H), 5.51 (q, *J* = 6.2 Hz, 1H), 1.53 (d, *J* = 6.0 Hz, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 166.58, 155.30, 142.33, 140.84, 129.36, 129.03, 128.77, 126.60, 124.16, 109.65, 107.84, 49.27, 17.18.

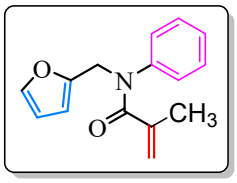


N-(1-(5-methylfuran-2-yl)ethyl)-N-phenylacrylamide (2r): Following the general procedure, compound **2r** was obtained a brown oil (2.23 g, 70% yield).

R_f = 0.62 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.40 – 7.32 (m, 4H), 7.14 – 7.07 (m, 1H), 6.37 (t, *J* = 9.9 Hz, 1H), 6.23 (d, *J* = 8.4 Hz, 1H), 6.08 (dd, *J* = 9.9, 1.8 Hz, 1H), 6.01 (dd, *J* = 9.9, 1.8 Hz, 1H), 5.93 (dq, *J* = 8.3, 0.8 Hz, 1H), 5.69 (q, *J* = 6.2 Hz, 1H), 2.23 (d, *J* = 0.7 Hz, 3H), 1.53 (d, *J* = 6.2 Hz, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 166.56, 153.62, 151.88, 141.17, 129.49, 128.98, 128.77, 126.54, 124.08, 107.41, 106.64, 51.51, 17.23, 13.55.

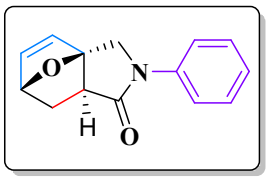


N-(furan-2-ylmethyl)-N-phenylmethacrylamide (2s). Following the general procedure, compound **2s** was obtained a brown oil (2.71 g, 90% yield).

R_f = 0.63 (Petroleum ether/EtOAc, v/v = 3:1).

^{1}H NMR (400 MHz, DMSO-d6) δ 7.55 (d, J = 1.6 Hz, 1H), 7.33 (t, *J* = 7.5 Hz, 2H), 7.24 (t, *J* = 7.4 Hz, 1H), 7.17 – 7.10 (m, 2H), 6.34 (dd, *J* = 3.2, 1.9 Hz, 1H), 6.18 (d, *J* = 3.1 Hz, 1H), 5.02 (t, *J* = 1.6 Hz, 1H), 4.91 (s, 2H), 4.87 (s, 1H), 1.72 (t, *J* = 1.2 Hz, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 170.95, 151.00, 143.04, 142.93, 140.77, 129.49, 127.74, 127.50, 119.13, 110.93, 108.90, 45.73, 20.52.

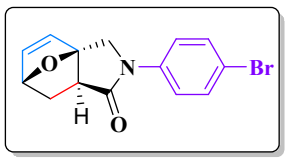


(3aR,6R,7aS)-2-phenyl-2,3,7,7a-tetrahydro-3a,6-epoxyisoindol-1(6H)-one (3a): Following the general procedure, compound **3a** was obtained as a white solid (67 mg, 99%).

R_f = 0.28 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.68 – 7.61 (m, 2H), 7.37 – 7.26 (m, 2H), 7.07 (t, *J* = 7.4 Hz, 1H), 6.50 (d, *J* = 5.8 Hz, 1H), 6.39 (dd, *J* = 5.8, 1.7 Hz, 1H), 5.00 (dd, *J* = 4.5, 1.7 Hz, 1H), 4.52 (d, *J* = 11.6 Hz, 1H), 3.99 (d, *J* = 11.6 Hz, 1H), 2.64 (dd, *J* = 8.8, 3.4 Hz, 1H), 1.98 – 1.89 (m, 1H), 1.48 (dd, *J* = 11.6, 8.8 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 173.97, 140.10, 137.41, 133.93, 129.23, 124.41, 119.79, 88.46, 78.95, 50.47, 48.67, 29.22.

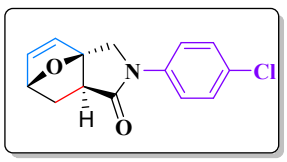


(3aR,6R,7aS)-2-(4-bromophenyl)-2,3,7,7a-tetrahydro-3a,6-epoxyisoindol-1(6H)-one (3b): Following the general procedure, compound **3b** was obtained as a white solid (90 mg, 98%).

R_f = 0.24 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.69 (d, *J* = 9.0 Hz, 2H), 7.56 (d, *J* = 9.0 Hz, 2H), 6.64 – 6.45 (m, 2H), 5.06 (dd, *J* = 4.6, 1.7 Hz, 1H), 4.56 (d, *J* = 11.5 Hz, 1H), 4.06 (d, *J* = 11.5 Hz, 1H), 2.72 (dd, *J* = 8.8, 3.4 Hz, 1H), 2.06 – 1.93 (m, 1H), 1.55 (dd, *J* = 11.7, 8.8 Hz, 1H).

^{13}C NMR (151 MHz, DMSO-d_6) δ 174.23, 139.38, 137.49, 133.81, 132.02, 121.60, 116.25, 88.33, 78.97, 50.39, 48.68, 29.26.

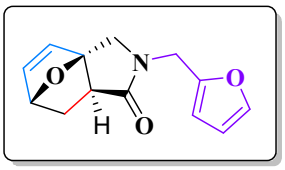


(3aR,6R,7aS)-2-(4-chlorophenyl)-2,3,7,7a-tetrahydro-3a,6-epoxyisoindol-1(6H)-one (3c): Following the general procedure, compound **3c** was obtained as a white solid (77 mg, 98%).

R_f = 0.24 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.74 (d, *J* = 9.0 Hz, 2H), 7.42 (d, *J* = 9.0 Hz, 2H), 6.55 (d, *J* = 5.8 Hz, 1H), 6.45 (dd, *J* = 5.8, 1.7 Hz, 1H), 5.06 (dd, *J* = 4.5, 1.6 Hz, 1H), 4.56 (d, *J* = 11.6 Hz, 1H), 4.06 (d, *J* = 11.6 Hz, 1H), 2.71 (dd, *J* = 8.8, 3.4 Hz, 1H), 1.99 (dt, *J* = 11.6, 4.4 Hz, 1H), 1.55 (dd, *J* = 11.7, 8.8 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 174.19, 138.95, 137.48, 133.80, 129.09, 128.16, 121.25, 88.35, 78.98, 50.45, 48.65, 29.25.

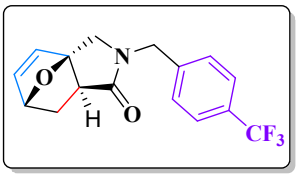


(3aR,6R,7aS)-2-(furan-2-ylmethyl)-2,3,7,7a-tetrahydro-3a,6-epoxyisoindol-1(6H)-one (3d): Following the general procedure, compound **3d** was obtained as a white solid (66 mg, 95%).

R_f = 0.29 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.55 (dd, *J* = 1.9, 0.9 Hz, 1H), 6.43 (d, *J* = 5.8 Hz, 1H), 6.37 – 6.28 (m, 2H), 6.26 (dd, *J* = 3.2, 0.9 Hz, 1H), 4.94 (dd, *J* = 4.5, 1.7 Hz, 1H), 4.43 (d, *J* = 15.6 Hz, 1H), 4.30 (d, *J* = 15.7 Hz, 1H), 3.93 (d, *J* = 11.5 Hz, 1H), 3.48 (d, *J* = 11.6 Hz, 1H), 2.37 (dd, *J* = 8.9, 3.6 Hz, 1H), 1.79 (ddd, *J* = 11.6, 4.6, 3.6 Hz, 1H), 1.38 (dd, *J* = 11.6, 8.9 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 173.55, 150.59, 143.27, 136.96, 134.23, 110.95, 108.48, 89.39, 78.67, 48.99, 47.12, 28.25.

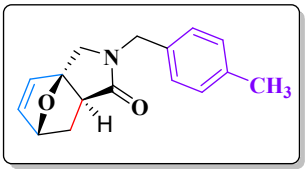


(3aR,6R,7aS)-2-(4-(trifluoromethyl)benzyl)-2,3,7,7a-tetrahydro-3a,6-epoxyisoindol-1(6H)-one (3e): Following the general procedure, compound **3e** was obtained as a white solid (89 mg, 96%).

R_f = 0.27 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.76 (d, *J* = 8.0 Hz, 2H), 7.51 (d, *J* = 8.0 Hz, 2H), 6.58 – 6.42 (m, 2H), 5.09 (dd, *J* = 4.5, 1.7 Hz, 1H), 4.58 (d, *J* = 2.8 Hz, 2H), 4.07 (d, *J* = 11.6 Hz, 1H), 3.55 (d, *J* = 11.7 Hz, 1H), 2.56 (dd, *J* = 3.4, 1.2 Hz, 1H), 1.96 (ddd, *J* = 11.5, 4.6, 3.6 Hz, 1H), 1.53 (dd, *J* = 11.6, 8.9 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 174.25, 142.33, 137.02, 134.13, 128.55, 125.92, 125.88, 89.57, 78.74, 49.01, 47.03, 45.45, 40.83, 28.34.

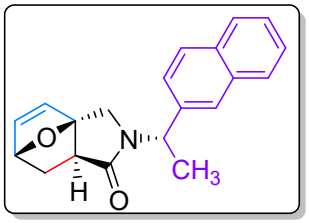


(3aR,6R,7aS)-2-(4-methylbenzyl)-2,3,7,7a-tetrahydro-3a,6-epoxyisoindol-1(6H)-one (3f): Following the general procedure, compound **3f** was obtained as a white solid (74 mg, 96%).

Rf = 0.32 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.13 – 6.96 (m, 4H), 6.46 – 6.21 (m, 2H), 4.92 (dd, *J* = 4.5, 1.7 Hz, 1H), 4.29 (s, 2H), 3.83 (d, *J* = 11.6 Hz, 1H), 3.35 (d, *J* = 11.6 Hz, 1H), 2.37 (dd, *J* = 8.8, 3.5 Hz, 1H), 2.19 (s, 3H), 1.85 – 1.75 (m, 1H), 1.36 (dd, *J* = 11.6, 8.9 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 173.82, 136.93, 136.80, 134.23, 134.21, 129.59, 127.95, 89.45, 78.71, 48.70, 47.21, 45.58, 28.30, 21.14.

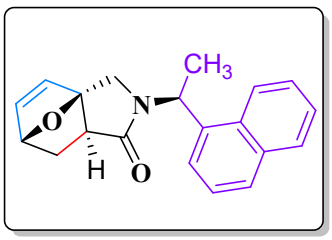


(3aR,6R,7aS)-2-((S)-1-(naphthalen-1-yl)ethyl)-2,3,7,7a-tetrahydro-3a,6-epoxyisoindol-1(6H)-one (3g): Following the general procedure, compound **3g** was obtained as a white solid (91 mg, 99%).

R_f = 0.24 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 8.12 – 7.89 (m, 3H), 7.68 (d, *J* = 7.1 Hz, 1H), 7.61 – 7.46 (m, 3H), 6.30 (d, *J* = 1.6 Hz, 2H), 6.00 (d, *J* = 6.9 Hz, 1H), 4.99 (dd, *J* = 4.5, 1.3 Hz, 1H), 3.58 (d, *J* = 11.8 Hz, 1H), 3.05 (d, *J* = 11.8 Hz, 1H), 2.32 (dd, *J* = 8.8, 3.4 Hz, 1H), 1.98 – 1.83 (m, 1H), 1.55 (d, *J* = 6.9 Hz, 3H), 1.42 (dd, *J* = 11.6, 8.8 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 172.80, 136.91, 135.78, 134.08, 133.90, 131.58, 129.24, 128.82, 127.17, 126.38, 125.79, 124.82, 123.38, 89.09, 78.61, 47.40, 45.19, 44.53, 28.29, 16.23.

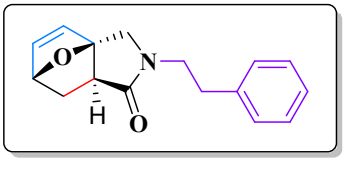


(3aR,6R,7aS)-2-((S)-1-(naphthalen-1-yl)ethyl)-2,3,7,7a-tetrahydro-3a,6-epoxyisoindol-1(6H)-one (3h): Following the general procedure, compound **3h** was obtained as a white solid (91 mg, 99%).

Rf = 0.25 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR 1H NMR (400 MHz, DMSO-d6) δ 7.99 – 7.81 (m, 4H), 7.58 – 7.48 (m, 2H), 7.46 (dd, J = 8.5, 1.8 Hz, 1H), 6.47 – 6.28 (m, 2H), 5.47 (q, J = 7.0 Hz, 1H), 5.00 (dd, J = 4.6, 1.7 Hz, 1H), 3.65 (d, J = 11.7 Hz, 1H), 3.56 (d, J = 11.7 Hz, 1H), 2.46 (dd, J = 8.8, 3.4 Hz, 1H), 1.94 – 1.84 (m, 1H), 1.53 (d, J = 7.1 Hz, 3H), 1.44 (dd, J = 11.5, 8.8 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 173.48, 138.45, 136.96, 134.24, 133.41, 132.72, 128.65, 128.39, 127.90, 126.71, 126.50, 126.15, 125.69, 89.30, 78.66, 49.27, 47.55, 44.79, 28.33, 16.73.

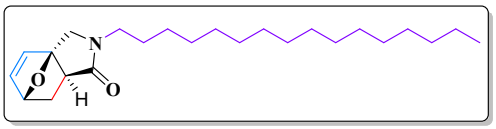


(3aR,6R,7aS)-2-phenethyl-2,3,7,7a-tetrahydro-3a,6-epoxyisoindol-1(6H)-one (3i): Following the general procedure, compound **3i** was obtained as a white solid (73 mg, 95%).

R_f = 0.30 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.43 – 7.01 (m, 5H), 6.43 (dd, *J* = 43.6, 5.7 Hz, 2H), 4.98 (d, *J* = 4.5 Hz, 1H), 3.99 (d, *J* = 11.6 Hz, 1H), 3.56 (dd, *J* = 16.0, 12.5 Hz, 2H), 3.37 – 3.27 (m, 1H), 2.77 (t, *J* = 7.5 Hz, 2H), 2.40 – 2.28 (m, 1H), 1.83 (d, *J* = 11.5 Hz, 1H), 1.40 (dd, *J* = 11.6, 8.8 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 173.62, 139.45, 137.01, 134.21, 129.10, 128.84, 126.68, 89.50, 78.69, 49.27, 47.29, 43.97, 33.57, 28.20.

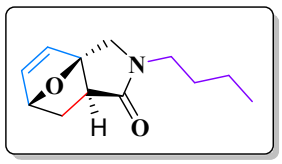


(3aR,6R,7aS)-2-hexadecyl-2,3,7,7a-tetrahydro-3a,6-epoxyisoindol-1(6H)-one (3j): Following the general procedure, compound **3j** was obtained as a white solid (106 mg, 94%).

R_f = 0.29 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d6) δ 6.56 – 6.33 (m, 2H), 5.07 – 4.90 (m, 1H), 4.00 (d, J = 11.7 Hz, 1H), 3.51 (d, J = 11.6 Hz, 1H), 3.18 (ddd, J = 44.9, 13.6, 6.7 Hz, 2H), 2.35 (dd, J = 8.8, 3.5 Hz, 1H), 1.89 – 1.75 (m, 1H), 1.44 – 1.39 (m, 2H), 1.23 (s, 28H), 0.85 (t, J = 6.6 Hz, 3H).

^{13}C NMR (101 MHz, DMSO-d6) δ 173.38, 136.89, 134.16, 89.42, 78.66, 60.17, 48.79, 47.27, 42.04, 40.86, 31.86, 29.65, 29.60, 29.56, 29.30, 29.25, 28.21, 27.24, 26.60, 22.62, 21.17, 21.13, 14.48, 14.28.

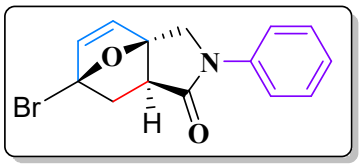


(3aR,6R,7aS)-2-butyl-2,3,7,7a-tetrahydro-3a,6-epoxyisoindol-1(6H)-one (3k): Following the general procedure, compound **3k** was obtained as a white solid (58 mg, 94%).

R_f = 0.35 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 6.50 (d, *J* = 5.8 Hz, 1H), 6.43 – 6.27 (m, 1H), 4.99 (dd, *J* = 4.5, 1.7 Hz, 1H), 4.02 (d, *J* = 11.7 Hz, 1H), 3.54 (d, *J* = 11.7 Hz, 1H), 3.31 – 3.03 (m, 2H), 2.38 (dd, *J* = 8.9, 3.6 Hz, 1H), 1.87 – 1.78 (m, 1H), 1.51 – 1.35 (m, 3H), 1.30 – 1.14 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 173.54, 136.92, 134.29, 89.50, 78.66, 48.78, 47.30, 41.70, 29.30, 28.24, 19.73, 14.07.

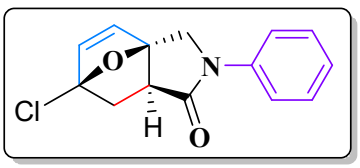


(3aR,6S,7aS)-6-bromo-2-phenyl-2,3,7,7a-tetrahydro-3a,6-epoxyisoindol-1(6H)-one (3l): Following the general procedure, compound **3l** was obtained as a white solid (90 mg, 98%).

R_f = 0.30 (Petroleum ether/EtOAc, v/v = 3/1)

^{1}H NMR (400 MHz, DMSO-d6) δ 7.81 – 7.56 (m, 2H), 7.53 – 7.27 (m, 2H), 7.23 – 7.08 (m, 1H), 6.74 (dd, J = 5.8, 2.0 Hz, 1H), 6.63 (dd, J = 5.8, 2.0 Hz, 1H), 4.62 (dd, J = 11.9, 2.1 Hz, 1H), 4.18 (dd, J = 12.0, 2.2 Hz, 1H), 3.10 – 2.90 (m, 1H), 2.33 (tt, J = 7.9, 3.9 Hz, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 172.41, 140.67, 139.66, 135.49, 129.31, 124.78, 119.98, 90.00, 88.26, 51.53, 50.13, 27.90.

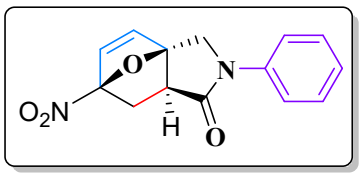


(3aR,6S,7aS)-6-chloro-2-phenyl-2,3,7,7a-tetrahydro-3a,6-epoxyisoindol-1(6H)-one (3m): Following the general procedure, compound **3m** was obtained as a white solid (76.9 mg, 98%).

R_f = 0.30 (Petroleum ether/EtOAc, v/v = 3/1)

^{1}H NMR (400 MHz, DMSO-d6) δ 7.79 – 7.68 (m, 2H), 7.50 – 7.39 (m, 2H), 7.25 – 7.15 (m, 1H), 6.84 (d, J = 5.6 Hz, 1H), 6.59 (d, J = 5.6 Hz, 1H), 4.62 (d, J = 11.9 Hz, 1H), 4.17 (d, J = 11.9 Hz, 1H), 3.07 (dd, J = 8.5, 3.7 Hz, 1H), 2.31 (dd, J = 11.7, 8.5 Hz, 1H), 2.22 (dd, J = 11.7, 3.8 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 172.31, 141.09, 133.81, 129.43, 125.94, 124.46, 120.79, 105.31, 90.73, 53.66, 49.45, 38.65.

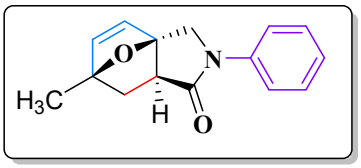


(3aR,6R,7aS)-6-nitro-2-phenyl-2,3,7,7a-tetrahydro-3a,6-epoxyisoindol-1(6H)-one (3n): Following the general procedure, compound **3n** was obtained as a white solid (80.0 mg, 98%).

R_f = 0.28 (Petroleum ether/EtOAc, v/v = 3/1)

^{1}H NMR (400 MHz, DMSO-d6) δ 7.71 (d, J = 8.2 Hz, 2H), 7.41 (t, J = 7.9 Hz, 2H), 7.18 (t, J = 7.4 Hz, 1H), 6.95 (dd, J = 39.9, 5.7 Hz, 2H), 4.66 (d, J = 12.3 Hz, 1H), 4.27 (d, J = 12.2 Hz, 1H), 3.44 – 3.04 (m, 2H), 2.46 – 2.31 (m, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 171.82, 139.52, 137.43, 134.44, 129.35, 124.93, 120.04, 112.56, 88.78, 50.78, 49.99, 34.04.

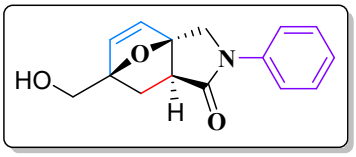


(3aR,6R,7aS)-6-methyl-2-phenyl-2,3,7,7a-tetrahydro-3a,6-epoxyisoindol-1(6H)-one (3o): Following the general procedure, compound **3o** was obtained as a yellowish solid (69mg, 95%).

R_f = 0.33 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.71 (dd, *J* = 8.7, 1.2 Hz, 2H), 7.38 (dd, *J* = 8.6, 7.3 Hz, 2H), 7.18 – 7.09 (m, 1H), 6.56 (d, *J* = 5.6 Hz, 1H), 6.33 (d, *J* = 5.6 Hz, 1H), 4.53 (d, *J* = 11.5 Hz, 1H), 4.03 (d, *J* = 11.5 Hz, 1H), 2.81 (dd, *J* = 8.5, 3.6 Hz, 1H), 1.75 (dd, *J* = 11.6, 3.7 Hz, 1H), 1.72 – 1.64 (m, 1H), 1.56 (s, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 174.01, 140.51, 140.11, 134.48, 129.23, 124.37, 119.71, 88.15, 87.28, 51.91, 50.66, 35.42, 19.04.

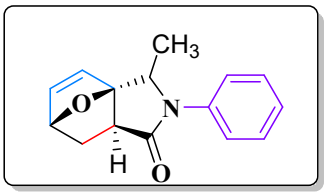


((3aR,6R,7aS)-1-oxo-2-phenyl-2,3,7,7a-tetrahydro-3a,6-epoxyisoindol-6(1H)-yl)methyl acrylate (3p): Following the general procedure, compound **3p** was obtained as a white solid (72.6mg, 94%).

R_f = 0.30 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.39 – 7.31 (m, 2H), 7.22 – 7.15 (m, 1H), 7.16 – 7.09 (m, 2H), 6.78 – 6.68 (m, 2H), 4.27 (d, J = 11.9 Hz, 1H), 4.17 – 4.08 (m, 2H), 3.79 (dd, J = 11.2, 7.0 Hz, 1H), 3.70 (dd, J = 11.1, 6.9 Hz, 1H), 2.59 (dd, J = 6.7, 3.4 Hz, 1H), 2.43 (dd, J = 12.9, 3.4 Hz, 1H), 2.23 (dd, J = 12.9, 6.7 Hz, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 172.49, 141.09, 133.08, 129.41, 126.05, 124.47, 120.80, 92.25, 90.84, 64.21, 53.56, 52.56, 33.67.

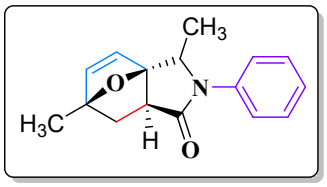


(3R,3aR,6R,7aS)-3-methyl-2-phenyl-2,3,7,7a-tetrahydro-3a,6-epoxyisoindol-1(6H)-one (3q): Following the general procedure, compound **3q** was obtained as a white solid (71.7mg, 99%).

R_f = 0.34 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.37 – 7.26 (m, 4H), 7.22 – 7.14 (m, 1H), 6.30 (dd, *J* = 6.6, 4.2 Hz, 1H), 6.03 (d, *J* = 6.6 Hz, 1H), 5.43 (td, *J* = 4.7, 3.7 Hz, 1H), 4.42 (q, *J* = 5.1 Hz, 1H), 3.17 (dd, *J* = 6.8, 3.3 Hz, 1H), 2.31 (dt, *J* = 13.2, 3.4 Hz, 1H), 2.11 (ddd, *J* = 13.2, 6.6, 4.9 Hz, 1H), 1.14 (d, *J* = 5.1 Hz, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 172.91, 139.68, 134.55, 134.12, 129.40, 126.55, 122.30, 96.29, 76.13, 55.41, 48.80, 30.34, 15.47.

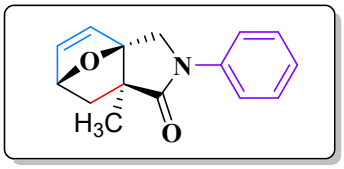


(3R,3aR,6R,7aS)-3,6-dimethyl-2-phenyl-2,3,7,7a-tetrahydro-3a,6-epoxyisoindol-1(6H)-one (3r): Following the general procedure, compound **3r** was obtained as a white solid (67 mg, 92%).

R_f = 0.31 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.44 – 7.37 (m, 2H), 7.35 – 7.27 (m, 2H), 7.18 (ddt, *J* = 8.8, 6.9, 1.5 Hz, 1H), 6.62 (d, *J* = 6.6 Hz, 1H), 5.81 (dq, *J* = 6.6, 0.9 Hz, 1H), 4.47 (q, *J* = 5.2 Hz, 1H), 2.66 (dd, *J* = 6.6, 3.5 Hz, 1H), 2.26 (dd, *J* = 13.0, 3.5 Hz, 1H), 2.14 (dd, *J* = 13.0, 6.8 Hz, 1H), 1.36 (d, *J* = 1.1 Hz, 3H), 1.17 (d, *J* = 5.3 Hz, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 173.75, 141.66, 139.68, 131.78, 129.46, 126.51, 122.33, 97.42, 90.19, 55.77, 50.82, 37.94, 26.07, 15.52.

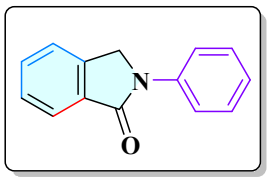


(3aR,6R,7aS)-7a-methyl-2-phenyl-2,3,7,7a-tetrahydro-3a,6-epoxyisoindol-1(6H)-one (3s): Following the general procedure, compound **3s** was obtained as a white solid (67 mg, 92%).

R_f = 0.31 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.79 – 7.65 (m, 2H), 7.42 (dd, *J* = 8.7, 7.3 Hz, 2H), 7.18 (d, *J* = 7.4 Hz, 1H), 6.66 – 6.50 (m, 2H), 5.01 (dd, *J* = 4.8, 1.7 Hz, 1H), 4.58 (d, *J* = 11.8 Hz, 1H), 4.07 (d, *J* = 11.8 Hz, 1H), 2.32 (dd, *J* = 11.6, 4.8 Hz, 1H), 1.14 (d, *J* = 11.6 Hz, 1H), 1.06 (s, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 177.35, 140.19, 138.03, 132.10, 129.22, 124.39, 119.82, 90.42, 78.74, 53.51, 49.05, 37.00, 20.73.



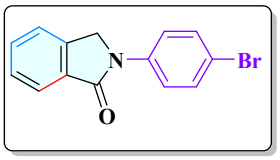
2-phenylisoindolin-1-one (4a): Following the general procedure, compound **4a** was obtained as a white solid (207 mg, 99%).

R_f = 0.57 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.88 – 7.82 (m, 2H), 7.72 (d, *J* = 7.6 Hz, 1H), 7.64 – 7.58 (m, 2H), 7.48 (td, *J* = 6.9, 6.0, 2.4 Hz, 1H), 7.38 (td, *J* = 7.6, 1.9 Hz, 2H), 7.12 (t, *J* = 7.4 Hz, 1H), 4.96 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 167.13, 141.51, 139.97, 132.92, 132.72, 129.45, 128.66, 124.58, 123.80, 123.71, 119.79, 50.88.

HRMS (ESI-TOF): calcd for $C_{14}H_{11}NO + Na^+$: 232.0739 $[M+Na]^+$; found: 232.0733.



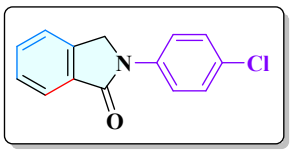
2-(4-bromophenyl)isoindolin-1-one (4b): Following the general procedure, compound **4b** was obtained as a white solid (282 mg, 98%).

R_f = 0.54 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.90 (d, *J* = 9.0 Hz, 2H), 7.79 (d, *J* = 7.6 Hz, 1H), 7.73 – 7.59 (m, 4H), 7.55 (td, *J* = 7.2, 6.8, 1.7 Hz, 1H), 5.01 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 167.24, 141.42, 139.31, 132.94, 132.62, 132.21, 128.73, 123.83, 123.80, 121.53, 116.43, 50.82.

HRMS (ESI-TOF): calcd for $C_{14}H_{10}BrNO + Na^+$: 309.9844 $[M+Na]^+$; found 309.9840.



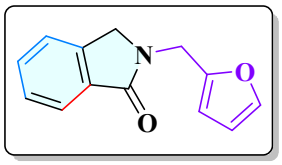
2-(4-chlorophenyl)isoindolin-1-one (4c): Following the general procedure, compound **4c** was obtained as a white solid (239 mg, 98%).

R_f = 0.53 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.99 (d, *J* = 8.5 Hz, 2H), 7.84 (d, *J* = 7.6 Hz, 1H), 7.73 (d, *J* = 7.7 Hz, 2H), 7.65 – 7.46 (m, 3H), 5.06 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 167.20, 141.42, 138.85, 132.90, 132.61, 132.57, 129.28, 128.70, 128.29, 123.78, 121.19, 50.85.

HRMS (ESI-TOF): calcd for $C_{14}H_{10}ClNO + Na^+$: 266.0349 $[M+Na]^+$; found 266.0341.



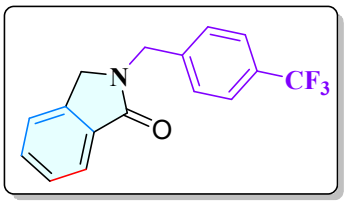
2-(furan-2-ylmethyl)isoindolin-1-one (4d): Following the general procedure, compound **4d** was obtained as a slight yellow solid (209 mg, 98%).

R_f = 0.49 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.71 (dt, *J* = 7.5, 1.1 Hz, 1H), 7.65 – 7.56 (m, 3H), 7.54 – 7.45 (m, 1H), 6.48 – 6.36 (m, 2H), 4.74 (s, 2H), 4.40 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 167.49, 150.95, 143.40, 142.18, 132.36, 131.98, 128.41, 124.03, 123.35, 111.04, 108.75, 49.78, 38.80.

HRMS (ESI-TOF): calcd for $C_{13}H_{11}NO_2 + Na^+$: 236.0687 $[M+Na]^+$; found 236.0685.



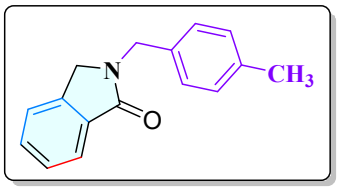
2-(4-(trifluoromethyl)benzyl)isoindolin-1-one (4e): Following the general procedure, compound **4e** was obtained as a white solid (285 mg, 98%).

R_f = 0.49 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d6) δ 7.72 (dd, J = 13.4, 7.7 Hz, 3H), 7.63 – 7.54 (m, 2H), 7.49 (d, J = 8.2 Hz, 3H), 4.83 (s, 2H), 4.41 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 168.09, 142.86, 142.33, 132.27, 132.03, 128.82, 128.44, 126.04, 126.01, 125.97, 124.03, 123.43, 49.93, 45.47.

HRMS (ESI-TOF): calcd for $C_{16}H_{12}F_3NO + Na^+$: 314.0769 $[M+Na]^+$; found 314.0763.



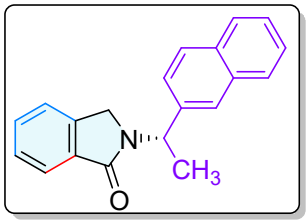
2-(4-methylbenzyl)isoindolin-1-one (4f): Following the general procedure, compound **4f** was obtained as a white solid (230 mg, 97%).

R_f = 0.42 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.73 (d, *J* = 7.5 Hz, 1H), 7.60 – 7.45 (m, 3H), 7.15 (s, 4H), 4.67 (s, 2H), 4.32 (s, 2H), 2.26 (d, *J* = 1.8 Hz, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 167.75, 142.18, 136.98, 134.89, 132.57, 131.83, 129.70, 128.37, 128.18, 123.97, 123.34, 49.56, 45.51, 21.13.

HRMS (ESI-TOF): calcd for $C_{16}H_{15}NO + Na^+$: 260.1051 $[M+Na]^+$; found 260.1049.



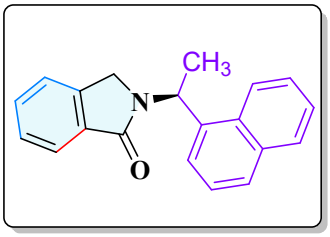
(S)-2-(1-(naphthalen-2-yl)ethyl)isoindolin-1-one (4g): Following the general procedure, compound **4g** was obtained as a white solid (297 mg, 97%).

TLC: R_f = 0.47 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.96 – 7.90 (m, 1H), 7.88 (dd, *J* = 7.7, 2.0 Hz, 3H), 7.72 (dt, *J* = 7.5, 1.0 Hz, 1H), 7.61 – 7.41 (m, 6H), 5.70 (q, *J* = 7.1 Hz, 1H), 4.56 (d, *J* = 17.7 Hz, 1H), 4.10 (d, *J* = 17.7 Hz, 1H), 1.74 (d, *J* = 7.1 Hz, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 167.54, 142.30, 139.32, 133.35, 132.72, 132.71, 131.83, 128.72, 128.39, 127.90, 126.75, 126.51, 125.99, 125.99, 125.41, 124.03, 123.31, 49.72, 46.28, 18.16.

HRMS (ESI-TOF): calcd for $C_{20}H_{17}NO+ Na^+$: 310.1208 $[M+Na]^+$; found 310.1207.



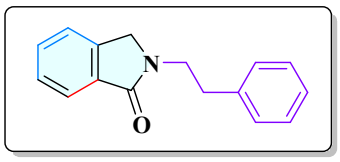
(S)-2-(1-(naphthalen-1-yl)ethyl)isoindolin-1-one (4h): Following the general procedure, compound **4h** was obtained as a white solid (297 mg, 97%).

R_f = 0.41(Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 8.15 – 8.06 (m, 1H), 7.99 – 7.90 (m, 2H), 7.77 – 7.70 (m, 2H), 7.58 (dd, *J* = 8.2, 7.3 Hz, 1H), 7.54 – 7.38 (m, 5H), 6.24 (d, *J* = 6.9 Hz, 1H), 4.47 (d, *J* = 17.8 Hz, 1H), 3.51 (d, *J* = 17.9 Hz, 1H), 1.75 (d, *J* = 6.9 Hz, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 166.87, 142.15, 136.34, 133.91, 132.53, 131.76, 131.38, 129.25, 128.95, 128.37, 127.15, 126.37, 125.82, 124.66, 124.01, 123.32, 123.26, 45.94, 45.74, 17.91.

HRMS (ESI-TOF): calcd for $C_{20}H_{17}NO + Na^+$: 310.1208 $[M+Na]^+$; found 310.1203.



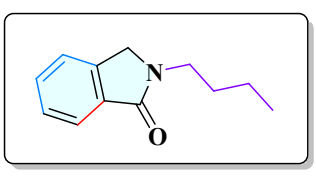
2-phenethylisoindolin-1-one (4i): Following the general procedure, compound **4i** was obtained as a white solid (233 mg, 98%).

R_f = 0.40 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.65 (d, *J* = 7.4 Hz, 1H), 7.56 (dd, *J* = 6.4, 1.3 Hz, 2H), 7.46 (ddd, *J* = 8.1, 6.4, 2.1 Hz, 1H), 7.30 – 7.22 (m, 4H), 7.22 – 7.12 (m, 1H), 4.39 (s, 2H), 3.77 (t, *J* = 7.3 Hz, 2H), 2.92 (t, *J* = 7.3 Hz, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 167.67, 142.21, 139.46, 132.82, 131.68, 129.05, 128.88, 128.26, 126.73, 123.77, 123.13, 50.12, 43.57, 34.37.

HRMS (ESI-TOF): calcd for $C_{16}H_{15}NO + Na^+$: 260.1051 $[M+Na]^+$; found 260.1050.



2-butylisoindolin-1-one (4k): Following the general procedure, compound **4k** was obtained as a white

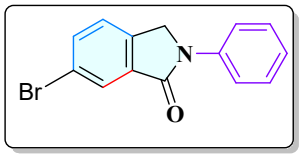
solid (184 mg, 97%).

R_f = 0.57 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.67 (dd, *J* = 7.4, 1.1 Hz, 1H), 7.57 (d, *J* = 4.0 Hz, 2H), 7.48 (dd, *J* = 7.8, 3.8 Hz, 1H), 4.45 (s, 2H), 3.51 (t, *J* = 7.2 Hz, 2H), 1.62 – 1.52 (m, 2H), 1.27 (q, *J* = 7.5 Hz, 2H), 0.90 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 167.62, 142.24, 132.98, 131.56, 128.22, 123.78, 123.09, 49.72, 41.58, 30.28, 19.97, 14.04.

HRMS (ESI-TOF): calcd for $C_{12}H_{15}NO + Na^+$: 212.1051 $[M+Na]^+$; found 212.1053.



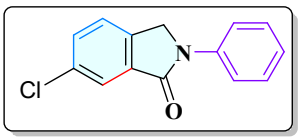
6-bromo-2-phenylisoindolin-1-one (4l): Following the general procedure, compound **4l** was obtained as a white solid (279 mg, 97%).

R_f = 0.53 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d6) δ 7.92 (d, J = 1.9 Hz, 1H), 7.91 (t, J = 0.9 Hz, 1H), 7.90 – 7.85 (m, 2H), 7.66 (dd, J = 8.0, 0.7 Hz, 1H), 7.49 – 7.43 (m, 2H), 7.21 (td, J = 7.3, 1.2 Hz, 1H), 5.02 (d, J = 0.8 Hz, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 165.68, 140.69, 135.43, 135.20, 130.66, 129.51, 126.28, 126.18, 124.93, 121.71, 119.94, 50.80.

HRMS (ESI-TOF): calcd for $C_{14}H_{10}BrNO + Na^+$: 309.9843 $[M+Na]^+$; found 309.9840.



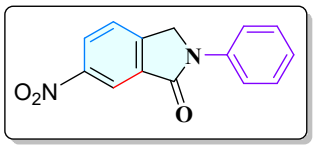
6-chloro-2-phenylisoindolin-1-one (4m): Following the general procedure, compound **4m** was obtained as a white solid (230 mg, 98%).

R_f = 0.54 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d6) δ 7.93 – 7.87 (m, 2H), 7.83 – 7.75 (m, 2H), 7.73 (dd, J = 4.3, 1.3 Hz, 1H), 7.51 – 7.47 (m, 1H), 7.46 – 7.43 (m, 1H), 7.24 – 7.17 (m, 1H), 5.04 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 165.78, 139.66, 134.90, 133.54, 132.66, 130.65, 129.50, 127.98, 125.86, 123.33, 119.94, 50.76.

HRMS (ESI-TOF): calcd for $C_{14}H_{10}ClNO + Na^+$: 266.0349 $[M+Na]^+$; found 266.0347.



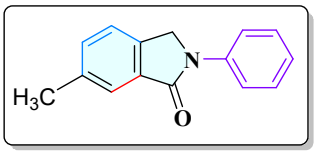
6-nitro-2-phenylisoindolin-1-one (4n): Following the general procedure, compound **4n** was obtained as a white solid (247 mg, 97%).

R_f = 0.52 (Petroleum ether/EtOAc, v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d6) δ 7.91 – 7.89 (m, 2H), 7.88 – 7.83 (m, 2H), 7.64 (dd, J = 8.0, 0.8 Hz, 1H), 7.47 – 7.42 (m, 2H), 7.19 (t, J = 7.4 Hz, 1H), 5.00 (s, 2H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 165.85, 140.27, 134.90, 133.13, 132.66, 130.60, 129.50, 128.06, 125.96, 123.33, 119.75, 50.82.

HRMS (ESI-TOF): calcd for $C_{14}H_{10}ClNO + Na^+$: 277.0589 $[M+Na]^+$; found 277.0588.



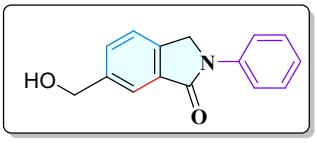
6-methyl-2-phenylisoindolin-1-one (4o): Following the general procedure, compound **4o** was obtained as a white solid (219 mg, 98%).

R_f = 0.57 (Petroleum ether/EtOAc v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.91 (d, *J* = 8.2 Hz, 2H), 7.71 – 7.35 (m, 5H), 7.17 (s, 1H), 4.97 (s, 2H), 2.43 (s, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ167.23, 140.04, 138.72, 134.48, 133.66, 133.06, 129.44, 129.22, 124.51, 123.75, 123.54, 50.64, 21.33.

HRMS (ESI-TOF): calcd for $C_{15}H_{13}NO + Na^+$: 246.0895 $[M+Na]^+$; found 246.0893.



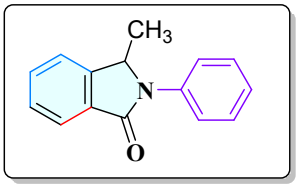
6-(hydroxymethyl)-2-phenylisoindolin-1-one (4p): Following the general procedure, compound **4p** was obtained as a white solid (232 mg, 97%).

R_f = 0.52 (Petroleum ether/EtOAc v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.97 (dt, *J* = 2.1, 1.1 Hz, 1H), 7.46 – 7.33 (m, 4H), 7.22 – 7.15 (m, 2H), 7.11 (tt, *J* = 7.5, 1.4 Hz, 1H), 4.83 (d, *J* = 0.8 Hz, 2H), 4.78 (dt, *J* = 6.8, 0.9 Hz, 2H), 3.18 – 3.12 (m, 1H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 167.47, 143.65, 141.66, 139.98, 131.07, 129.30, 129.02, 126.78, 124.47, 122.68, 121.63, 63.76, 48.45.

HRMS (ESI-TOF): calcd for $C_{15}H_{13}NO_2$ + Na^+: 262.0844 $[M+Na]^+$; found 262.0841.



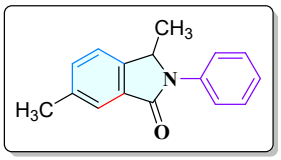
3-methyl-2-phenylisoindolin-1-one (4q): Following the general procedure, compound **4q** was obtained as a white solid (216 mg, 97%).

R_f = 0.57 (Petroleum ether/EtOAc v/v = 3/1).

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.80 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.60 – 7.48 (m, 4H), 7.42 – 7.32 (m, 3H), 7.11 (tt, *J* = 7.5, 1.5 Hz, 1H), 5.25 (qd, *J* = 6.2, 0.9 Hz, 1H), 1.50 (d, *J* = 6.2 Hz, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 166.47, 145.18, 140.33, 134.92, 130.57, 129.47, 126.52, 126.42, 125.84, 125.07, 123.87, 58.84, 17.87.

HRMS (ESI-TOF): calcd for $C_{15}H_{13}NO$ + Na^+: 246.0895 $[M+Na]^+$; found 246.0895.



3,6-dimethyl-2-phenylisoindolin-1-one (4r): Following the general procedure, compound **4r** was obtained as a white solid (230 mg, 97%).

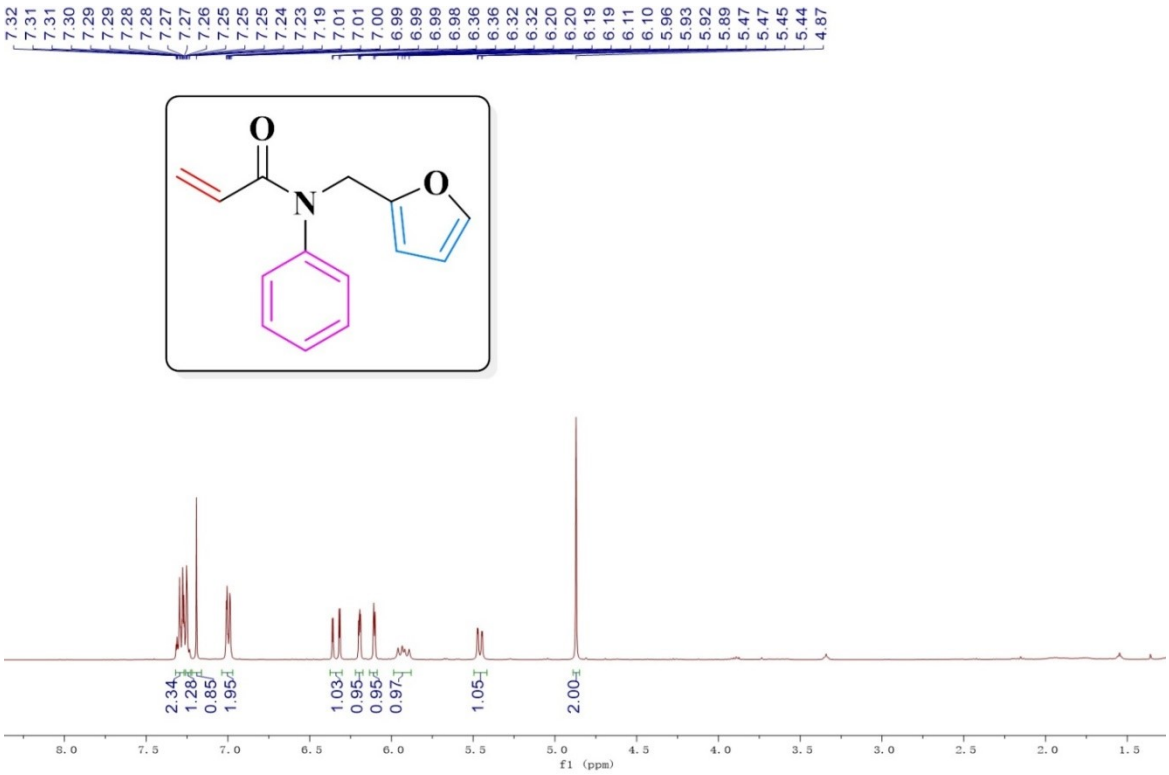
R_f = 0.55 (Petroleum ether/EtOAc v/v = 3/1).

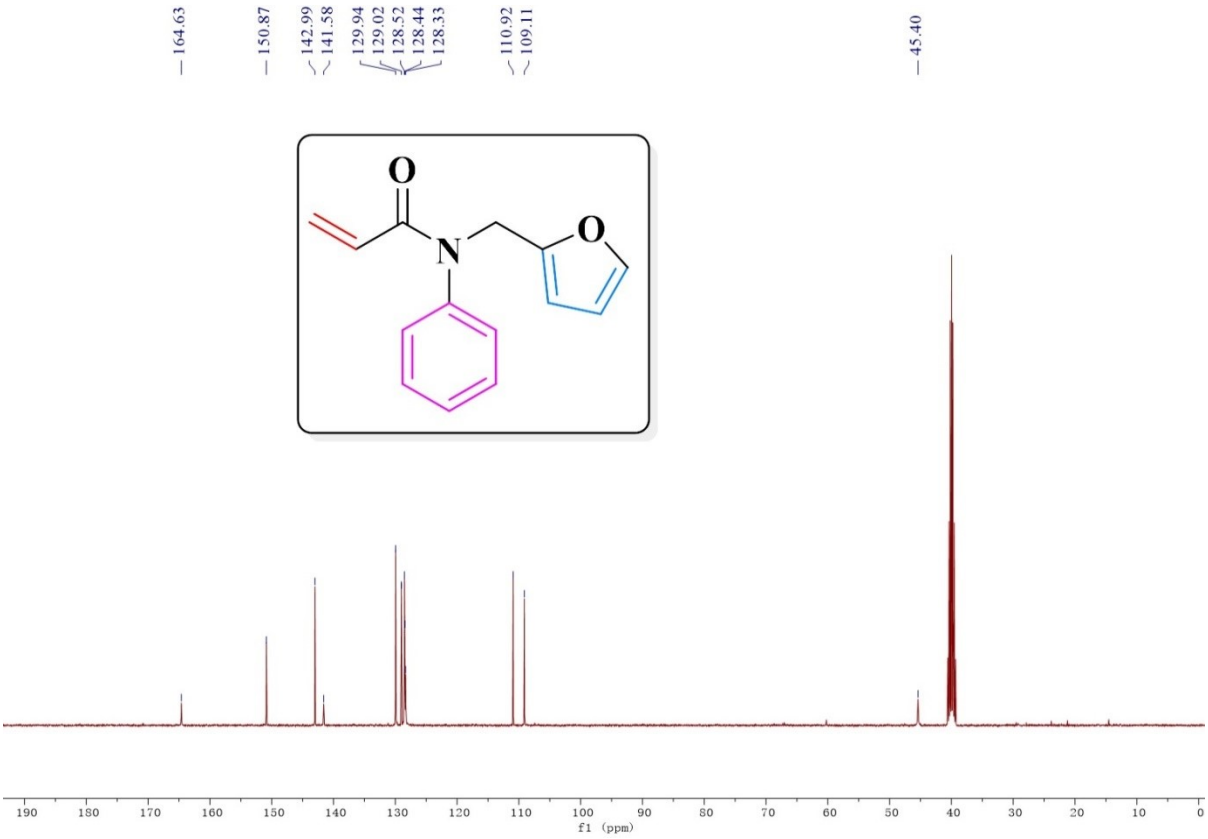
^{1}H NMR (400 MHz, DMSO-d_6) δ 7.77 – 7.72 (m, 1H), 7.60 – 7.53 (m, 2H), 7.49 (dd, *J* = 8.4, 1.0 Hz, 1H), 7.40 – 7.32 (m, 2H), 7.18 – 7.07 (m, 2H), 5.26 (qd, *J* = 6.2, 1.0 Hz, 1H), 2.41 (s, 2H), 1.49 (d, *J* = 6.2 Hz, 3H).

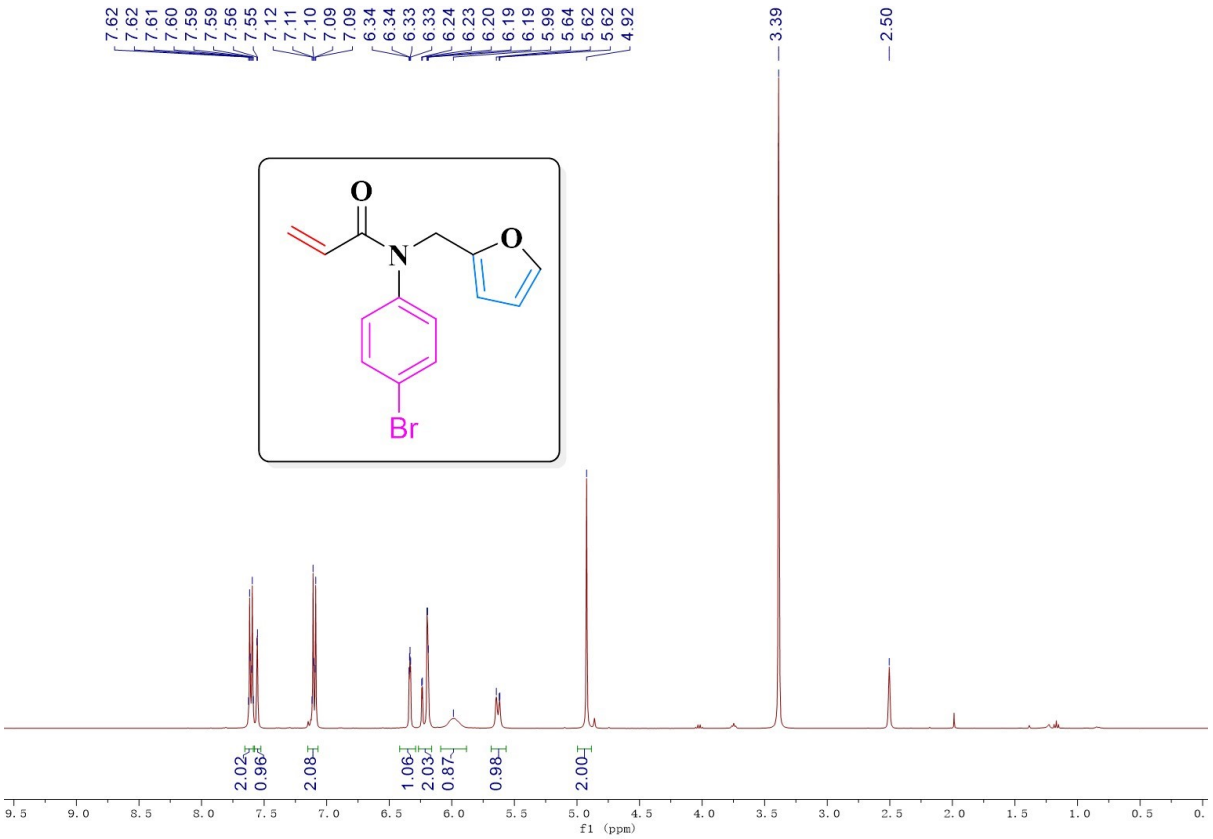
^{13}C NMR (101 MHz, DMSO-d_6) δ 166.82, 144.60, 140.33, 136.86, 132.94, 130.57, 129.47, 126.50, 125.25, 125.07, 58.84, 20.76, 17.86.

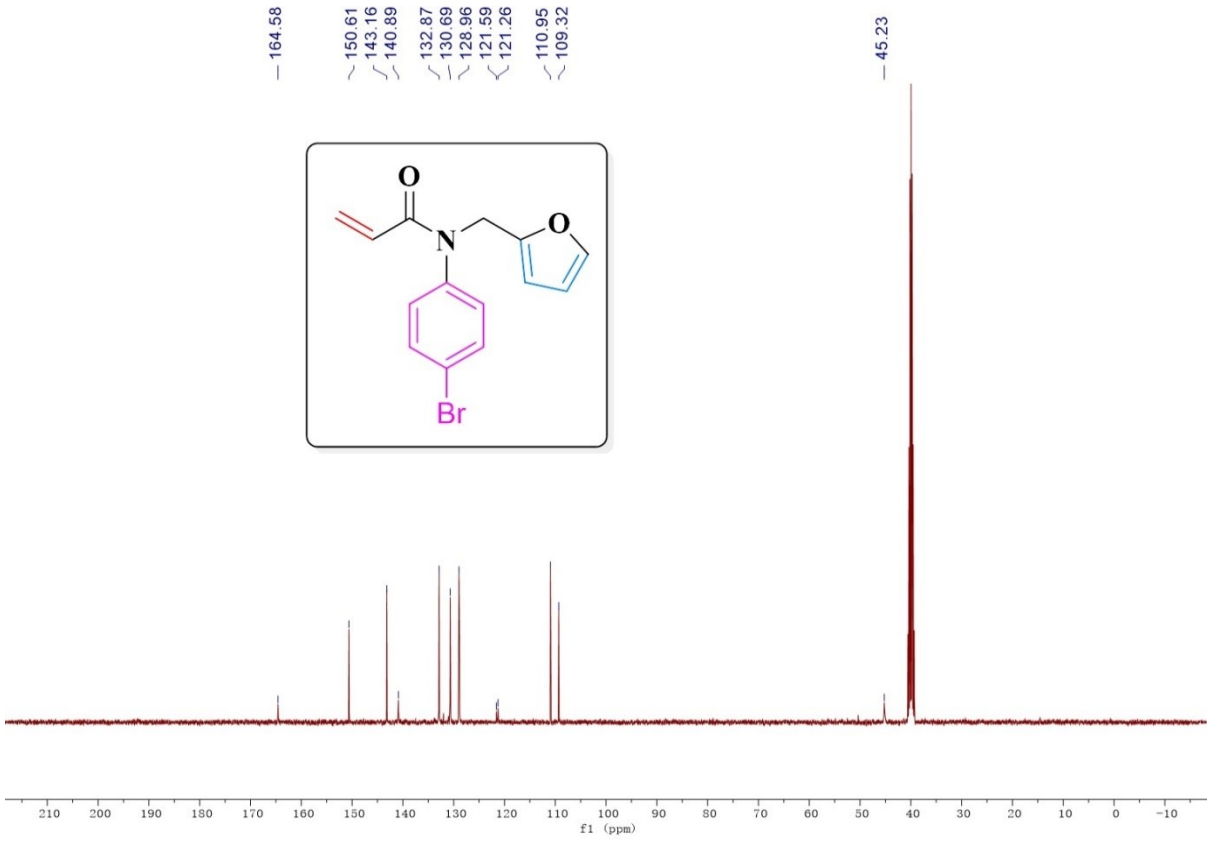
HRMS (ESI-TOF): calcd for $C_{16}H_{15}NO$ + Na^+: 260.1051 $[M+Na]^+$; found 260.1050.

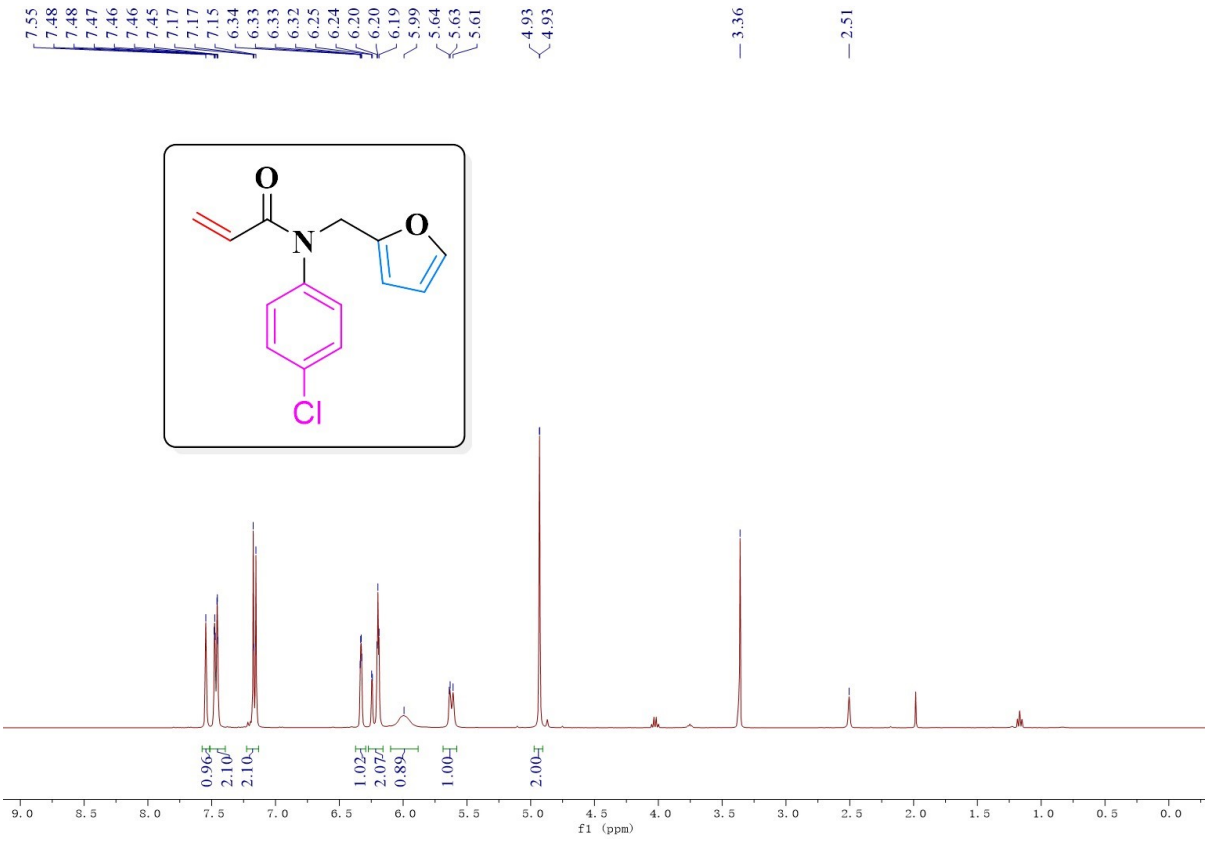
5. NMR Spectroscopy

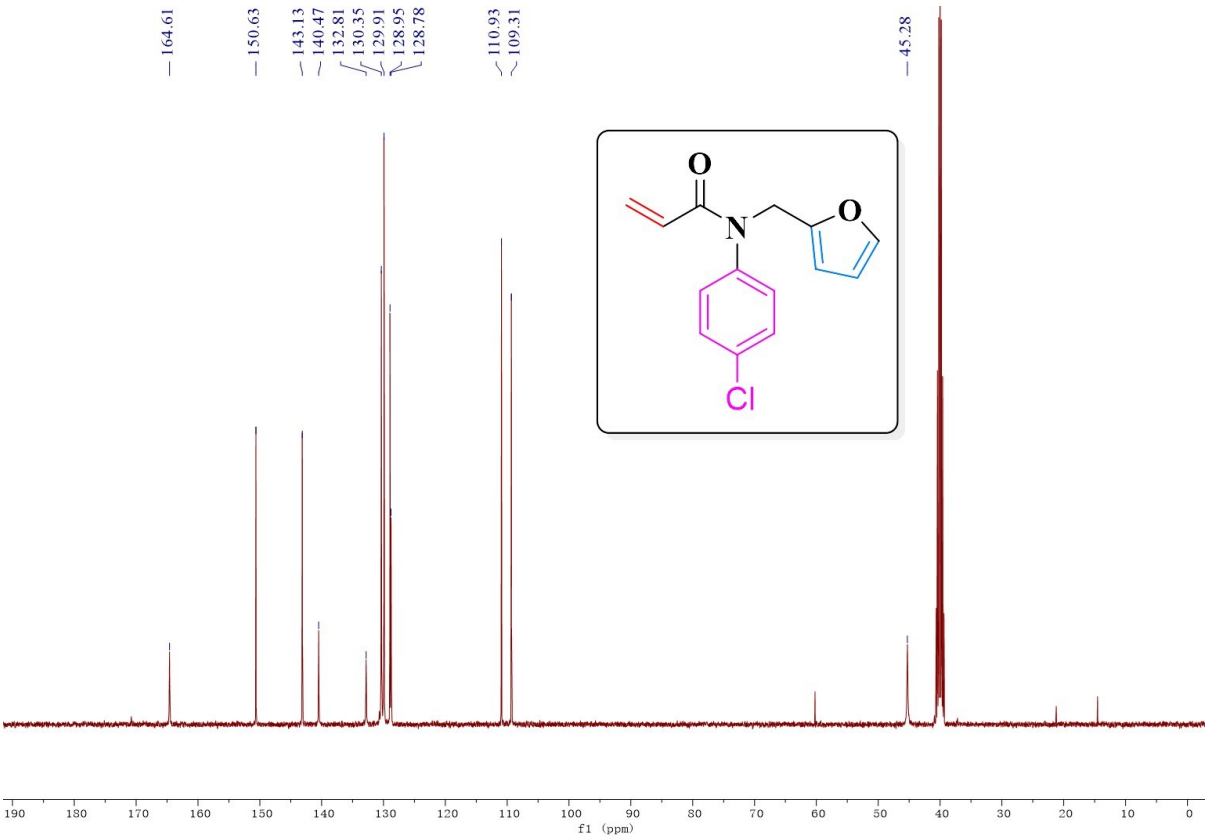


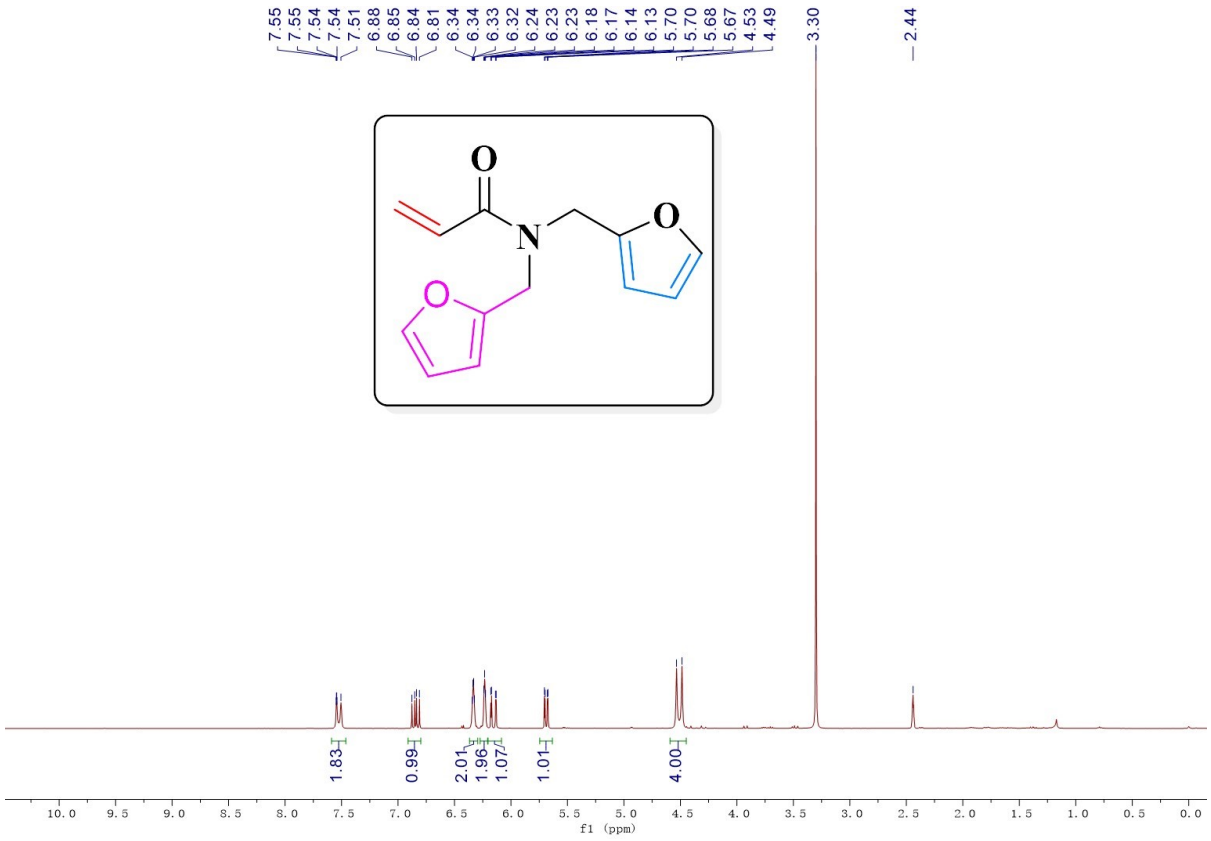


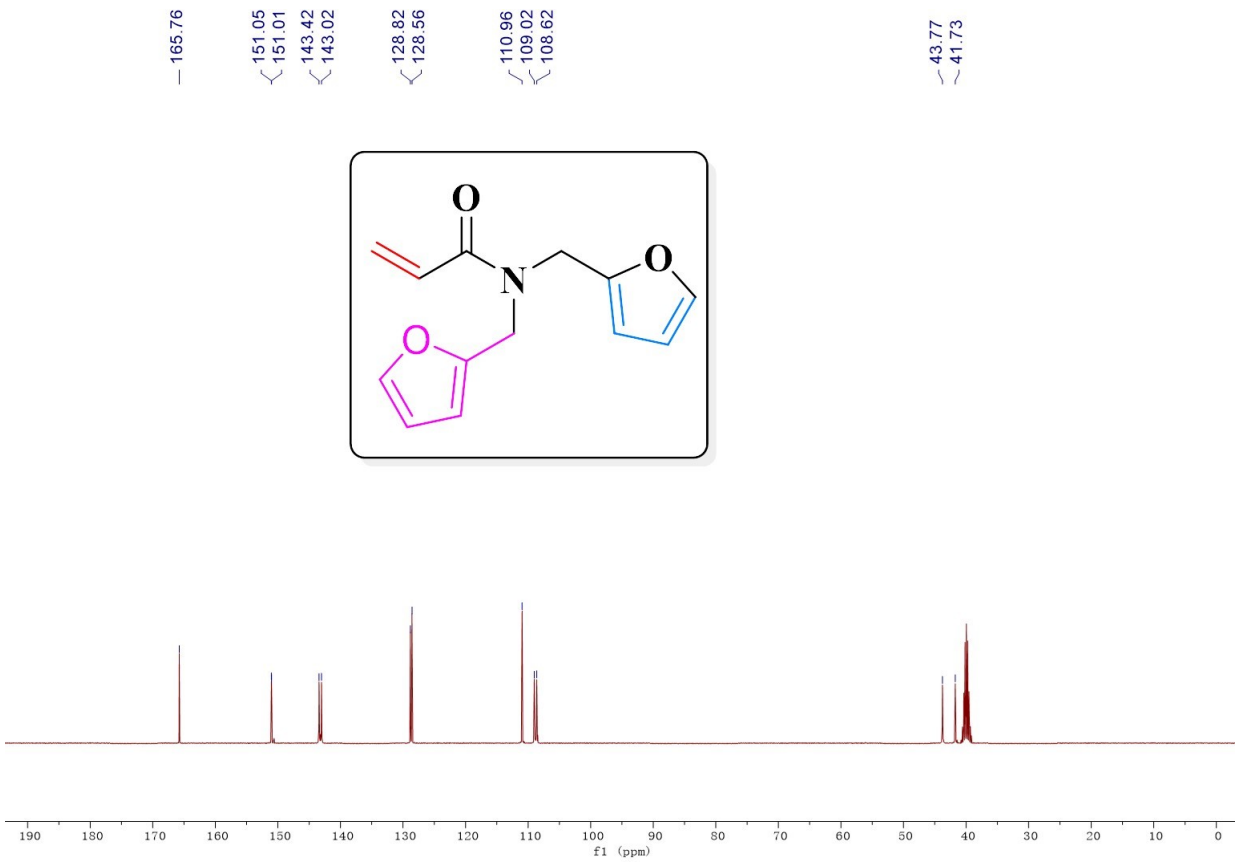

7.62
7.62
7.61
7.60
7.59
7.59
7.56
7.55
7.12
7.11
7.10
7.09
7.09
6.34
6.34
6.33
6.33
6.24
6.23
6.20
6.19
6.19
5.99
5.64
5.62
5.62
4.92
3.39
2.50
O
N
O
Br
2.02
0.96
2.08
1.06
2.03
0.87
0.98
2.00
f1 (ppm)

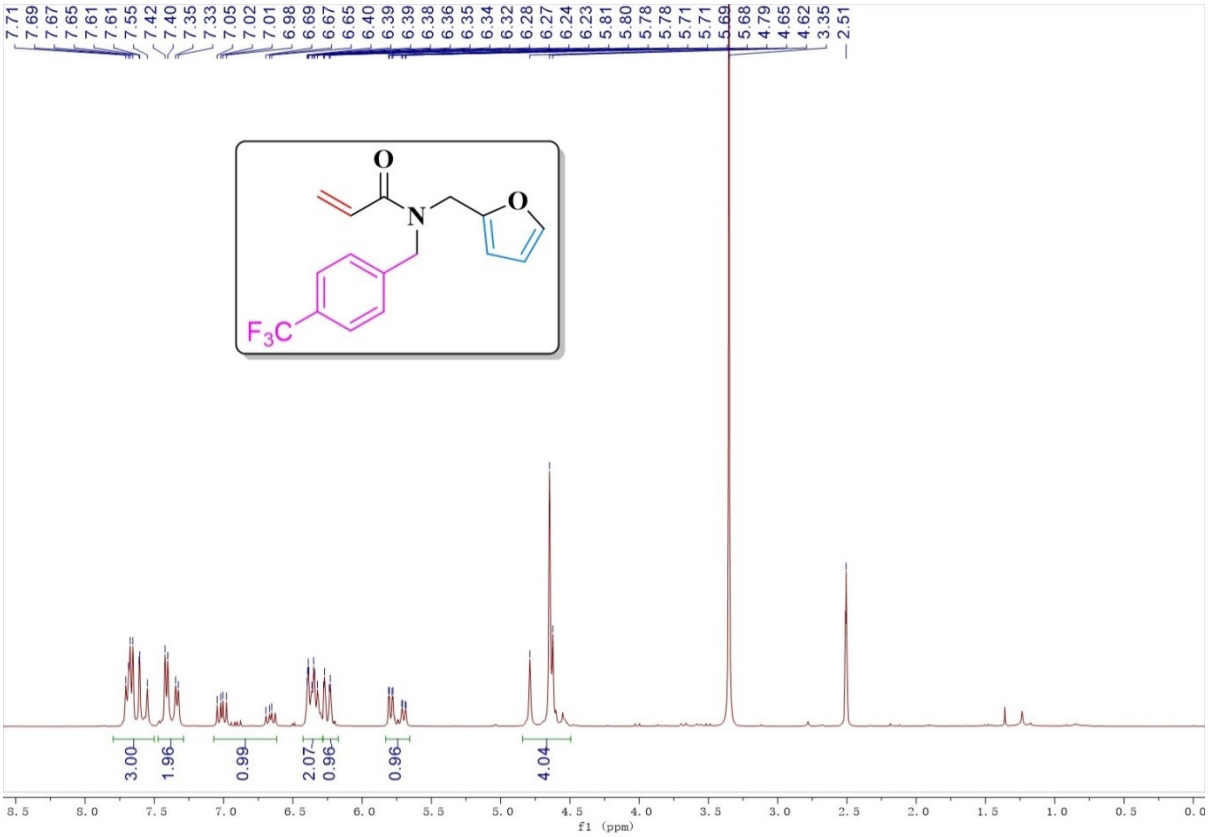

164.58
150.61
143.16
140.89
132.87
130.69
128.96
121.59
121.26
110.95
109.32
45.23
O
N
O
Br
f1 (ppm)

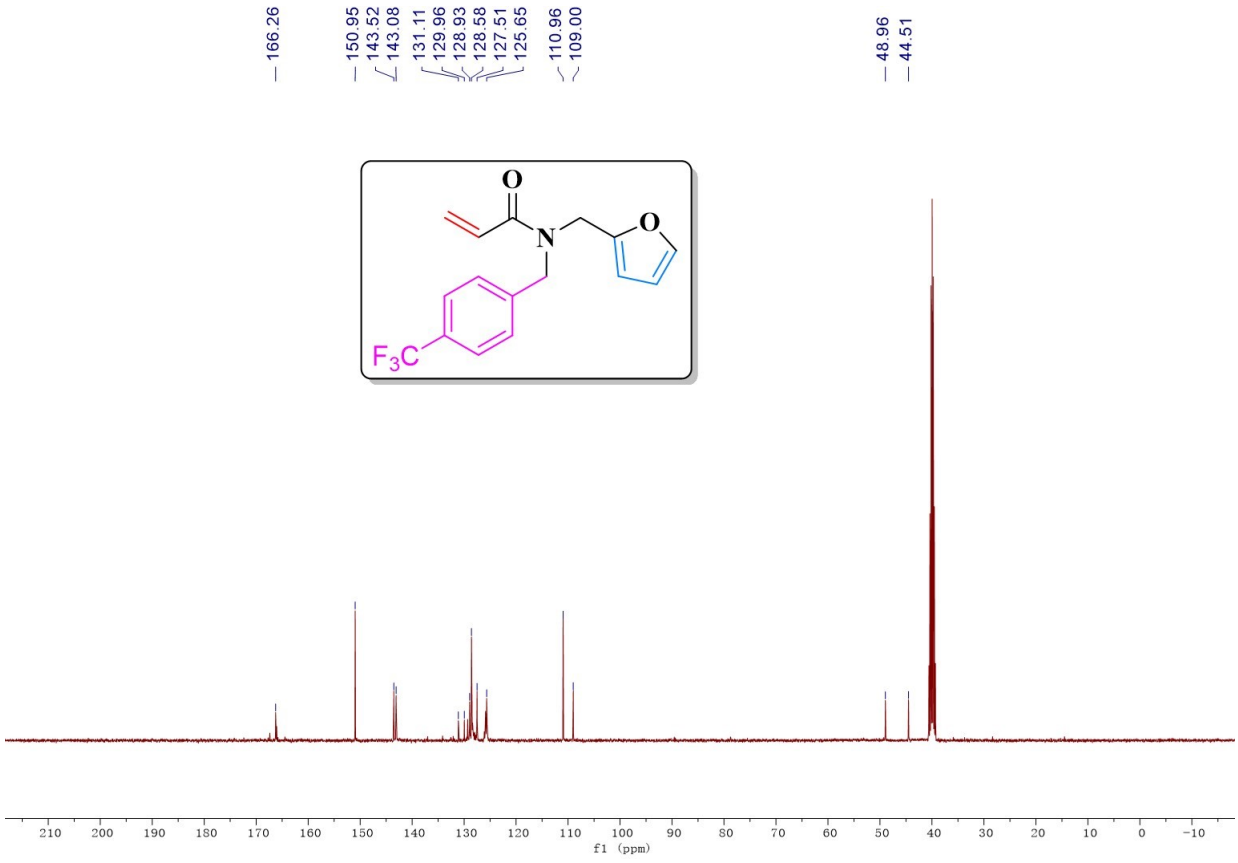

7.55
7.48
7.48
7.47
7.46
7.46
7.45
7.17
7.17
7.15
6.34
6.33
6.33
6.32
6.25
6.24
6.20
6.20
6.19
5.99
5.64
5.63
5.61
4.93
4.93
3.36
2.51
O
N
O
Cl
0.96
2.10
2.10
1.02
2.07
0.89
1.00
2.00
f1 (ppm)

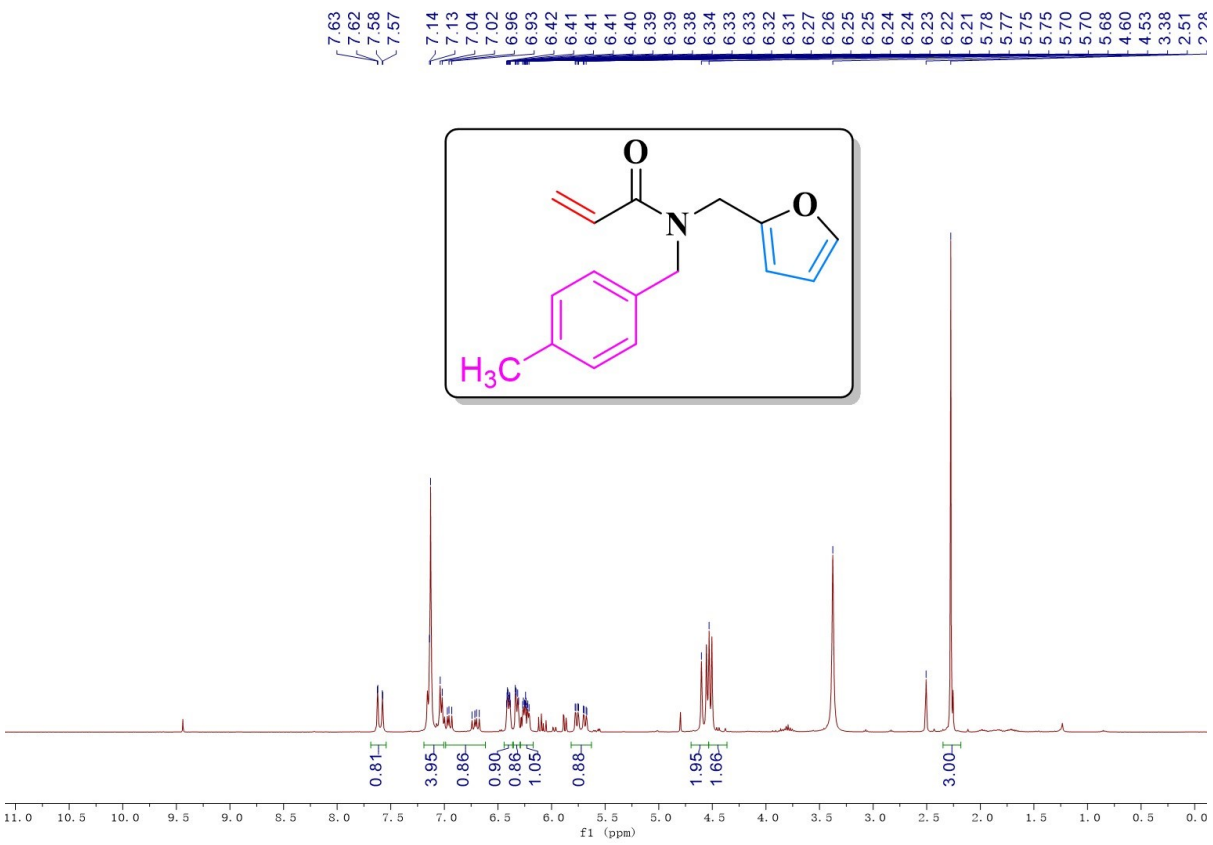

164.61
150.63
143.13
140.47
132.81
130.35
129.91
128.95
128.78
110.93
109.31
45.28
O
N
O
Cl
f1 (ppm)

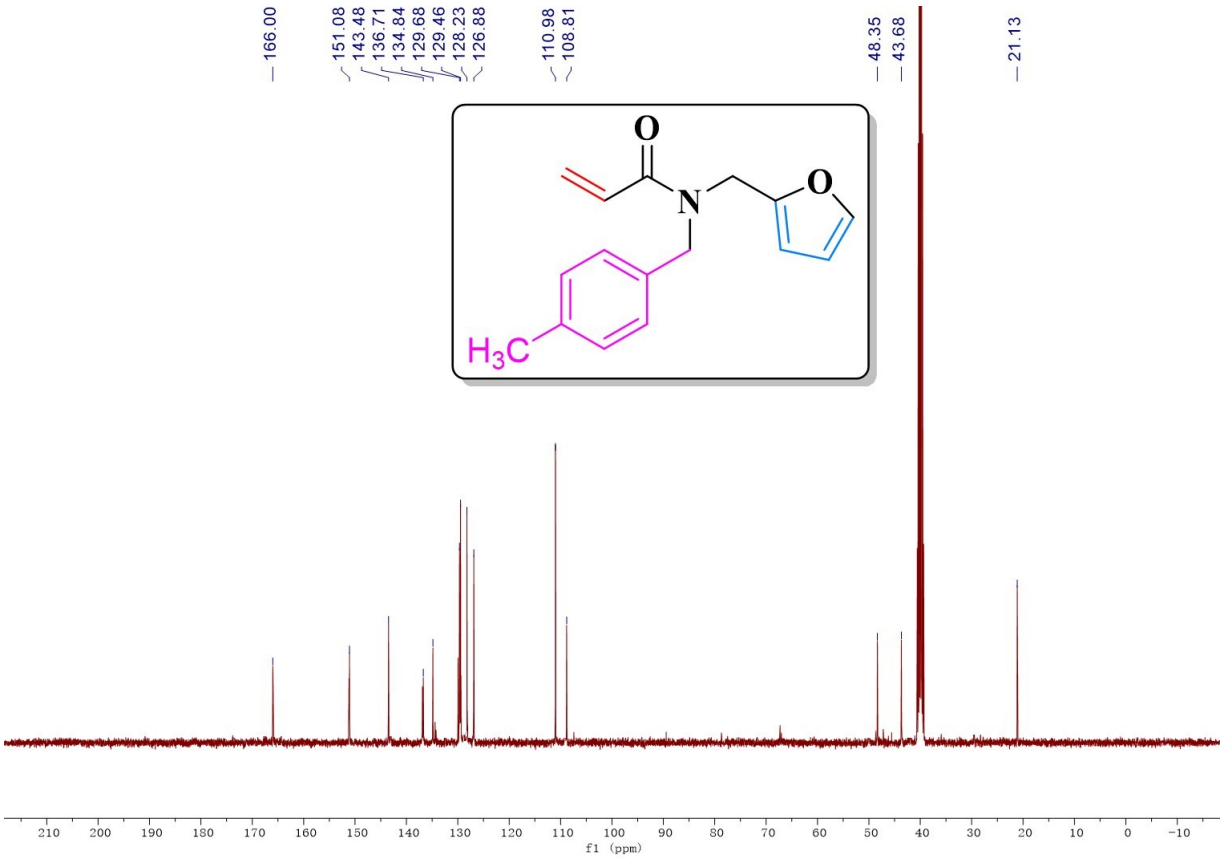

7.55
7.55
7.54
7.54
7.51
6.88
6.85
6.84
6.81
6.34
6.34
6.33
6.32
6.24
6.23
6.23
6.18
6.17
6.14
6.13
5.70
5.70
5.68
5.67
4.53
4.49
3.30
2.44
O
N
O
O
1.83
0.99
2.01
1.96
1.07
1.01
4.00
f1 (ppm)

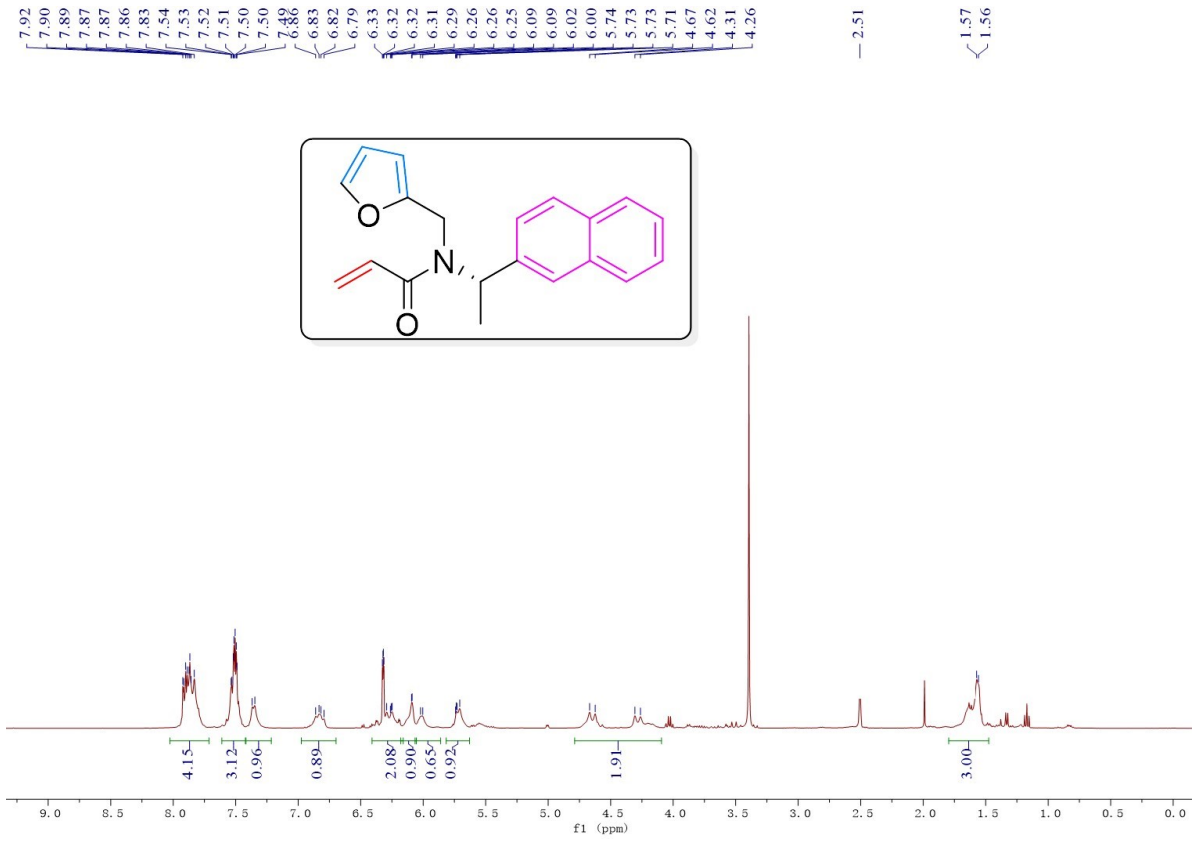

165.76
151.05
151.01
143.42
143.02
128.82
128.56
110.96
109.02
108.62
43.77
41.73
O
N
O
O
f1 (ppm)

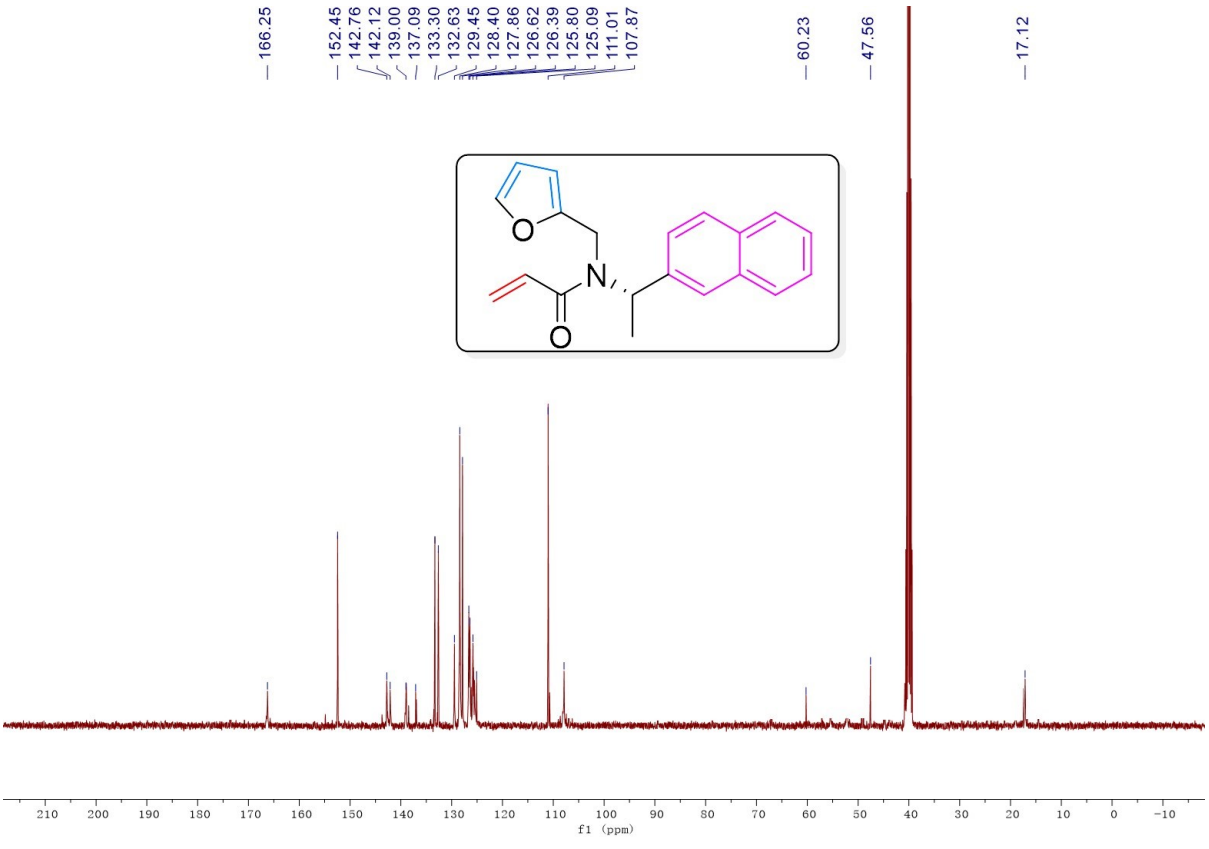

7.71
7.69
7.67
7.65
7.61
7.61
7.55
7.42
7.40
7.35
7.33
7.05
7.02
7.01
6.98
6.69
6.67
6.65
6.40
6.39
6.39
6.38
6.36
6.35
6.34
6.32
6.28
6.27
6.24
6.23
5.81
5.80
5.78
5.78
5.71
5.71
5.69
5.68
4.79
4.65
4.62
3.35
2.51
O
N
O
F3C
3.00
1.96
0.99
2.07
0.96
0.96
4.04
f1 (ppm)


166.26
150.95
143.52
143.08
131.11
129.96
128.93
128.58
127.51
125.65
110.96
109.00
48.96
44.51
O
N
O
F3C
f1 (ppm)

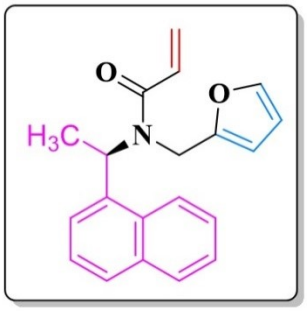

7.63
7.62
7.58
7.57
7.14
7.13
7.04
7.02
6.96
6.93
6.42
6.41
6.41
6.41
6.40
6.39
6.39
6.38
6.34
6.33
6.33
6.32
6.31
6.27
6.26
6.25
6.25
6.24
6.24
6.23
6.22
6.21
5.78
5.77
5.75
5.75
5.70
5.70
5.68
4.60
4.53
3.38
2.51
O
N
O
H3C
0.81
3.95
0.86
0.90
0.86
1.05
0.88
1.95
1.66
3.00
f1 (ppm)

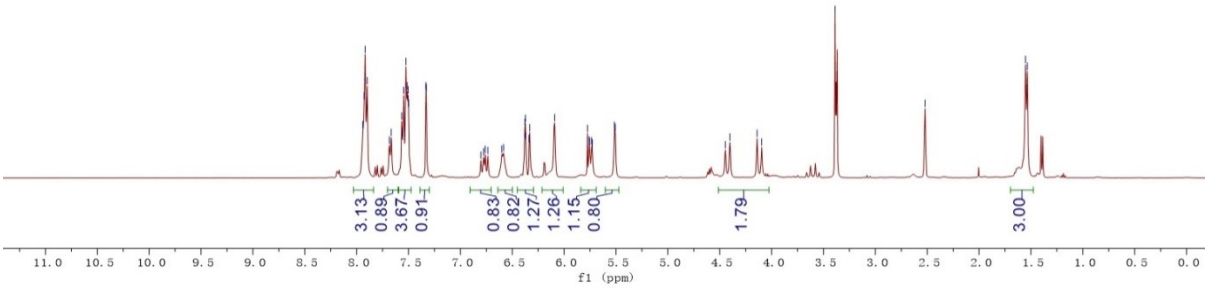

166.00
151.08
143.48
136.71
134.84
129.68
129.46
128.23
126.88
110.98
108.81
48.35
43.68
21.13
O
N
O
H3C
f1 (ppm)

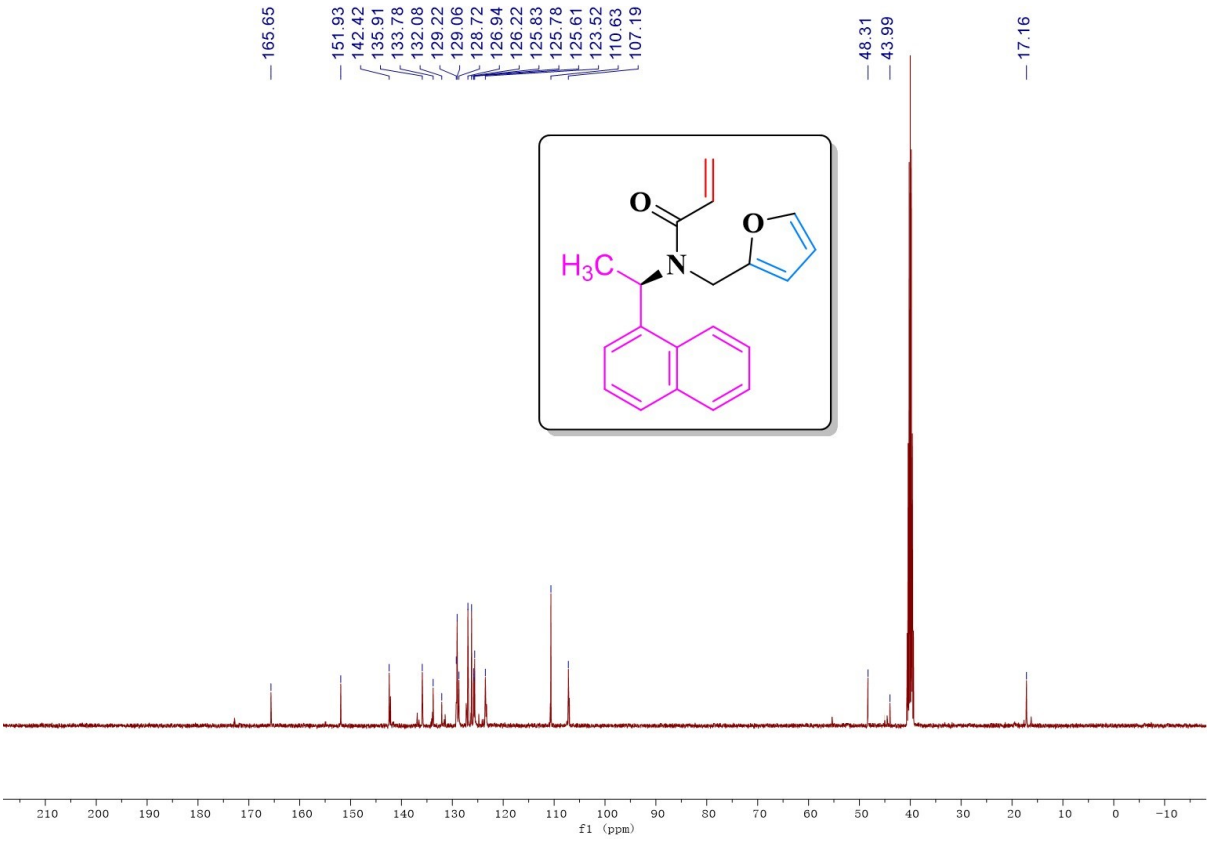

7.92
7.90
7.89
7.87
7.87
7.86
7.83
7.54
7.53
7.52
7.51
7.50
7.50
7.49
6.86
6.83
6.82
6.79
6.33
6.32
6.32
6.31
6.29
6.26
6.26
6.25
6.09
6.09
6.02
6.00
5.74
5.73
5.73
5.71
4.67
4.62
4.31
4.26
2.51
1.57
1.56
O
N
O
4.15
3.12
0.96
0.89
2.08
0.90
0.65
0.92
1.91
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

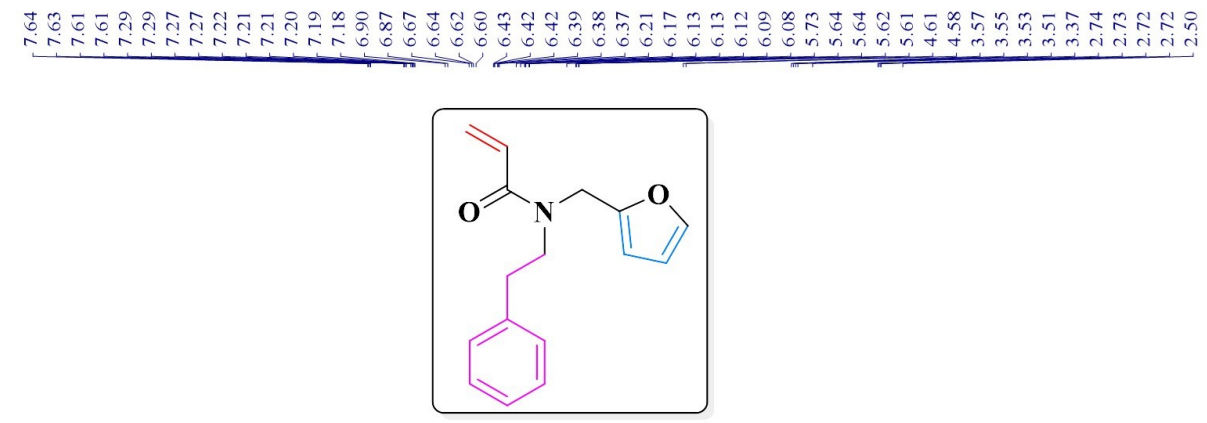

166.25
152.45
142.76
142.12
139.00
137.09
133.30
132.63
129.45
128.40
127.86
126.62
126.39
125.80
125.09
111.01
107.87
60.23
47.56
17.12
O
N
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

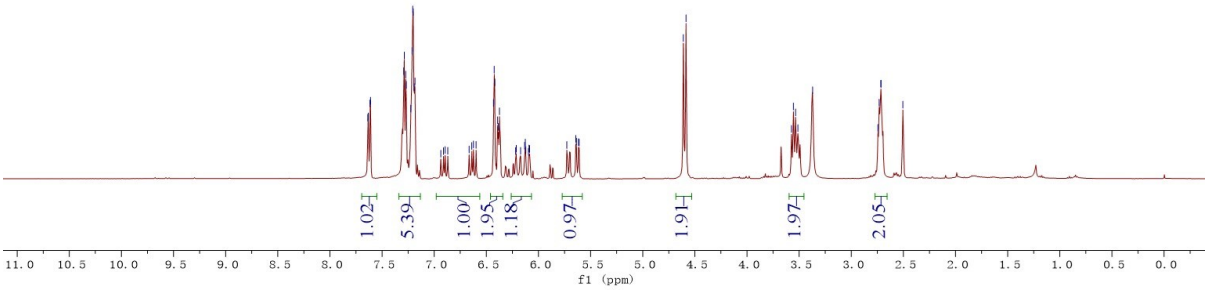

7.94
7.93
7.92
7.90
7.68
7.67
7.57
7.56
7.55
7.53
7.51
7.50
7.50
7.33
7.33
6.80
6.78
6.76
6.74
6.60
6.58
6.38
6.37
6.37
6.34
6.33
6.09
5.78
5.76
5.75
5.73
5.73
5.52
5.51
4.45
4.40
4.14
4.10
3.39
2.52
1.55
1.54

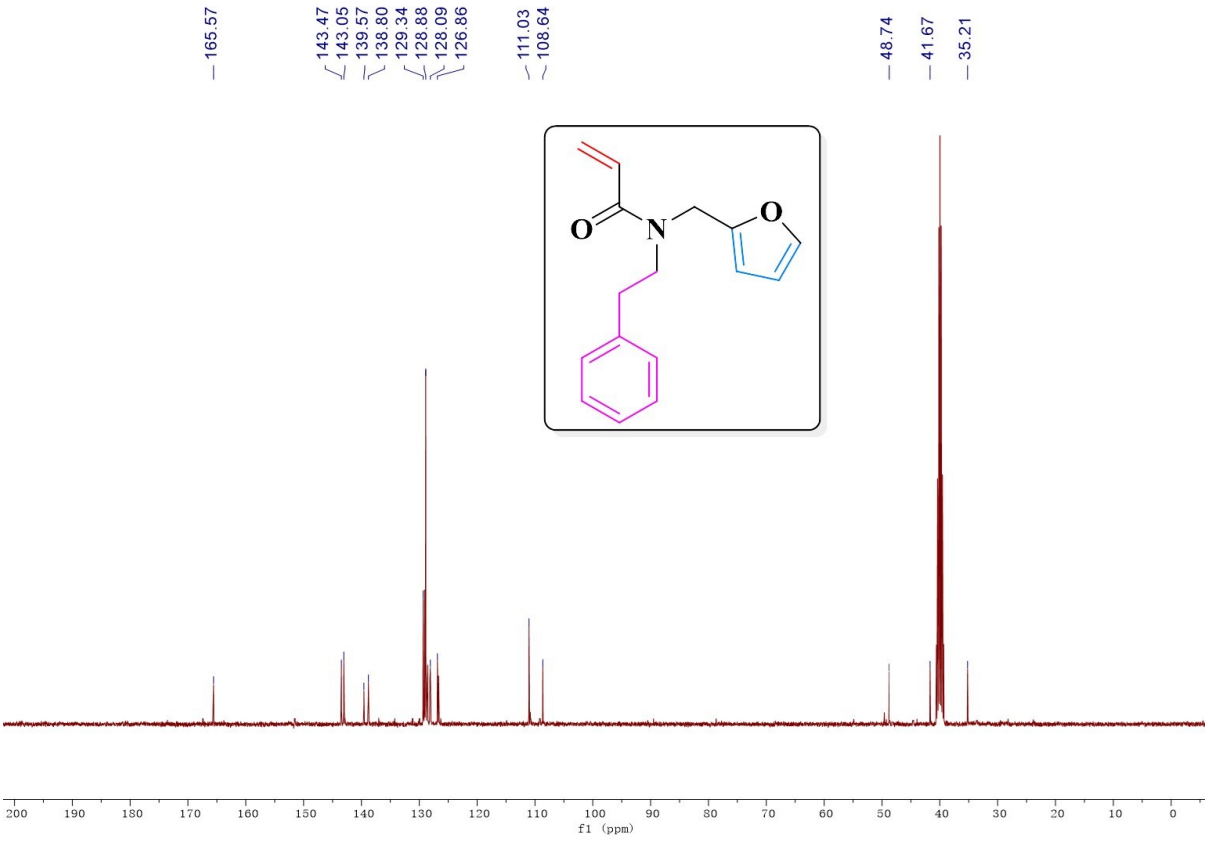

O
O
N
H3C

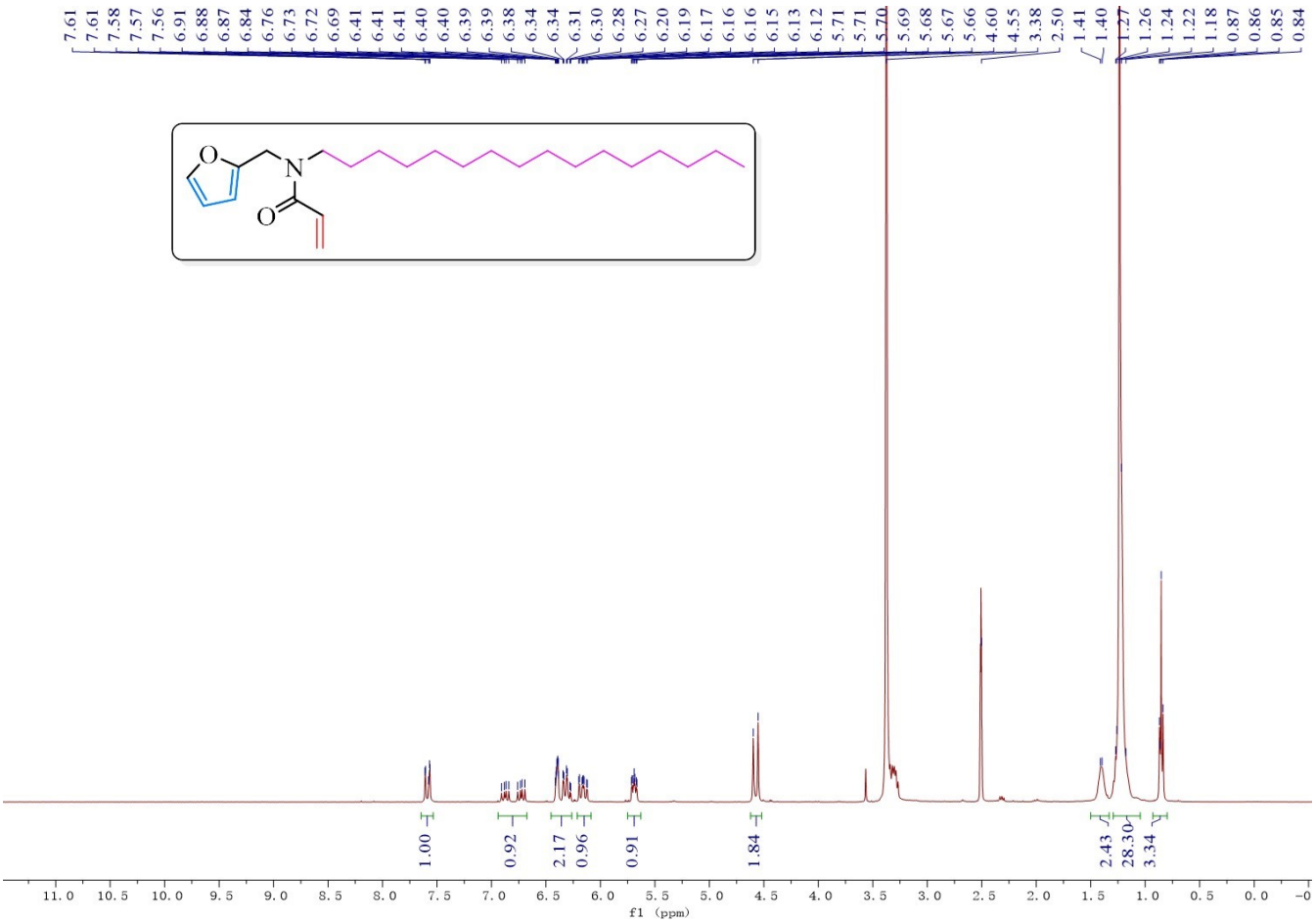

3.13
0.89
3.67
0.91
0.83
0.82
1.27
1.26
1.15
0.80
1.79
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

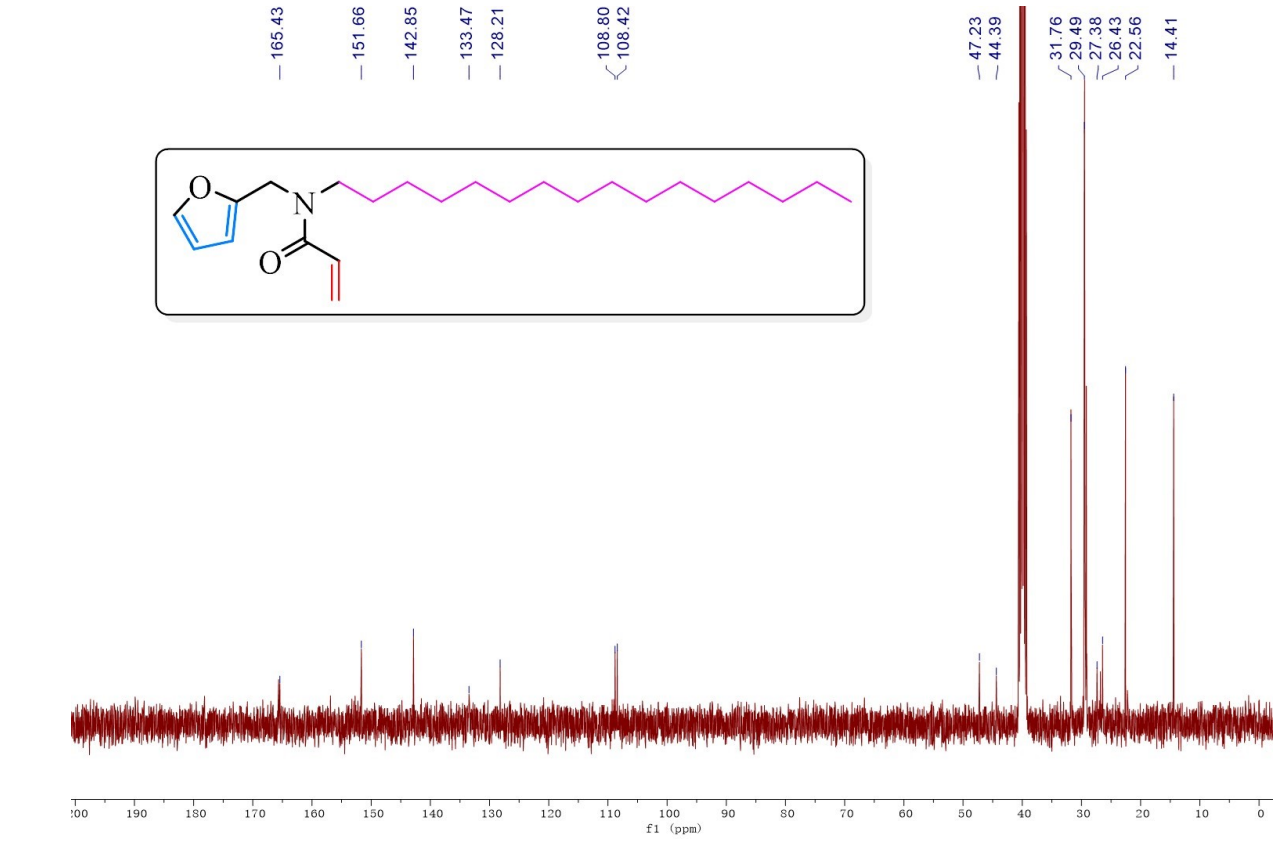

165.65
151.93
142.42
135.91
133.78
132.08
129.22
129.06
128.72
126.94
126.22
125.83
125.78
125.61
123.52
110.63
107.19
48.31
43.99
17.16
O
O
N
H3C
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

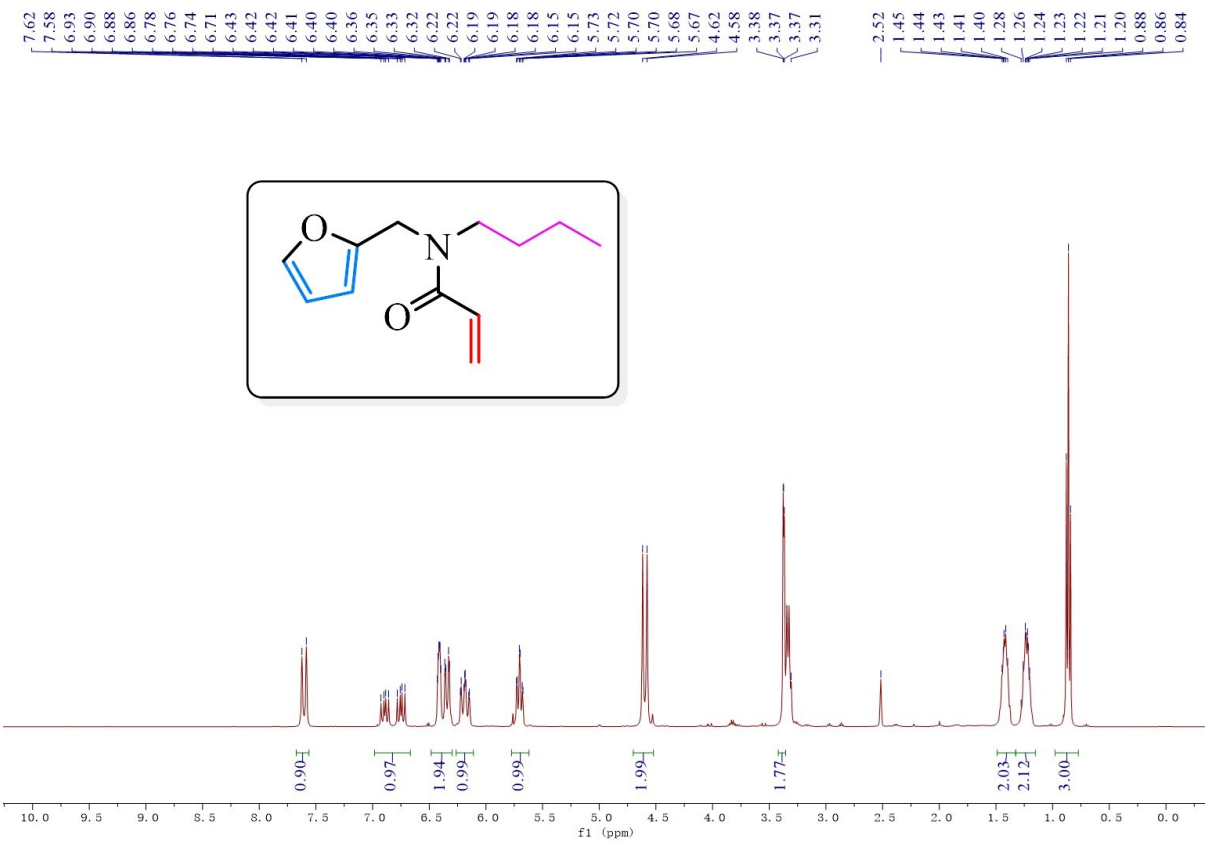

7.64
7.63
7.61
7.61
7.29
7.29
7.27
7.27
7.22
7.21
7.21
7.20
7.19
7.18
6.90
6.87
6.67
6.64
6.62
6.60
6.43
6.42
6.42
6.39
6.38
6.37
6.21
6.17
6.13
6.13
6.12
6.09
6.08
5.73
5.64
5.64
5.62
5.61
4.61
4.58
3.57
3.55
3.53
3.51
3.37
2.74
2.73
2.72
2.72
2.50
O
N
O

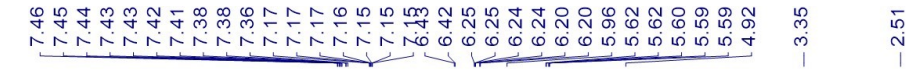

1.02
5.39
1.00
1.95
1.18
0.97
1.91
1.97
2.05
f1 (ppm)

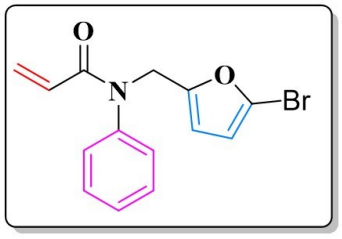

165.57
143.47
143.05
139.57
138.80
129.34
128.88
128.09
126.86
111.03
108.64
48.74
41.67
35.21
O
N
O
f1 (ppm)

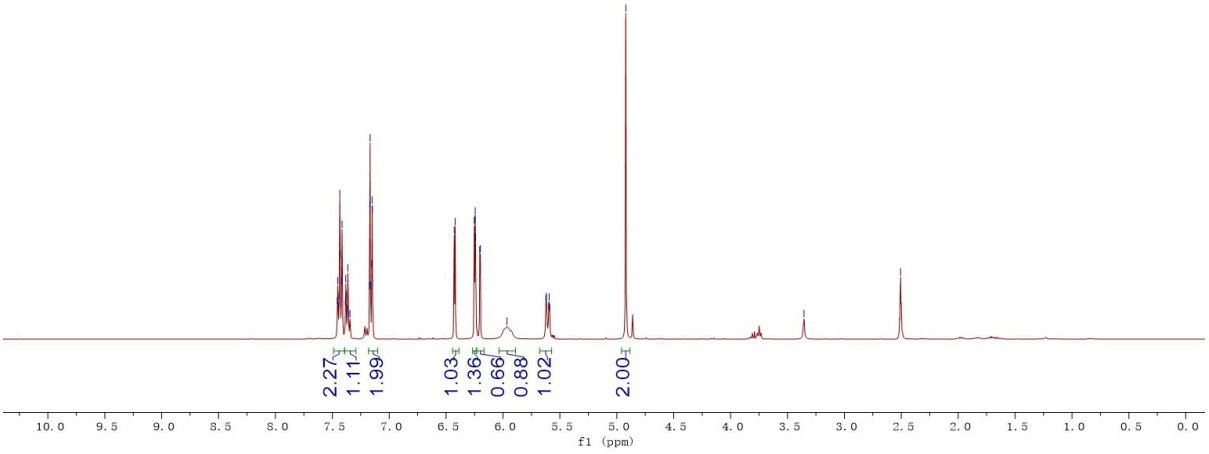

7.61 7.61 7.58 7.57 7.56 6.91 6.88 6.87 6.84 6.76 6.73 6.72 6.69 6.41 6.41 6.41 6.40 6.40 6.39 6.39 6.38 6.34 6.34 6.31 6.30 6.28 6.27 6.20 6.19 6.17 6.16 6.16 6.15 6.13 6.12 5.71 5.71 5.70 5.69 5.68 5.67 5.66 4.60 4.55 3.38 2.50 1.41 1.40 1.27 1.26 1.24 1.22 1.18 0.87 0.86 0.85 0.84
O
N
O
1.00
0.92
2.17
0.96
0.91
1.84
2.43
28.30
3.34
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 0.0 −0
f1 (ppm)


165.43
151.66
142.85
133.47
128.21
108.80
108.42
47.23
44.39
31.76
29.49
27.38
26.43
22.56
14.41
O
N
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

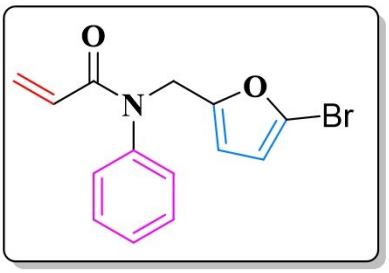

7.62
7.58
6.93
6.90
6.88
6.86
6.78
6.76
6.74
6.71
6.43
6.42
6.42
6.41
6.40
6.40
6.36
6.35
6.33
6.32
6.22
6.22
6.19
6.19
6.18
6.18
6.15
5.73
5.72
5.70
5.70
5.68
5.67
4.62
4.58
3.38
3.37
3.37
3.31
2.52
1.45
1.44
1.43
1.41
1.40
1.28
1.26
1.24
1.23
1.22
1.21
1.20
0.88
0.86
0.84
O
N
O
0.90
0.97
1.94
0.99
0.99
1.99
1.77
2.03
2.12
3.00
f1 (ppm)
165.68
151.64
142.88
129.03
127.90
108.82
108.45
46.99
44.37
29.56
19.73
14.08
O
N
O
f1 (ppm)

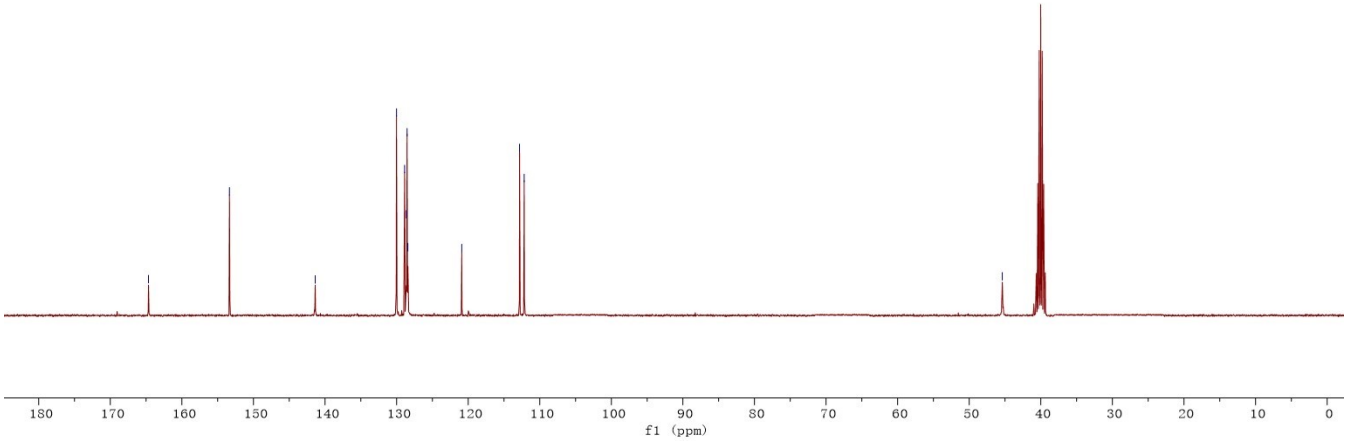

7.46
7.45
7.44
7.43
7.43
7.42
7.41
7.38
7.38
7.36
7.17
7.17
7.17
7.16
7.15
7.15
7.15
7.13
6.42
6.25
6.25
6.24
6.24
6.20
6.20
5.96
5.62
5.62
5.60
5.59
5.59
4.92
3.35
2.51

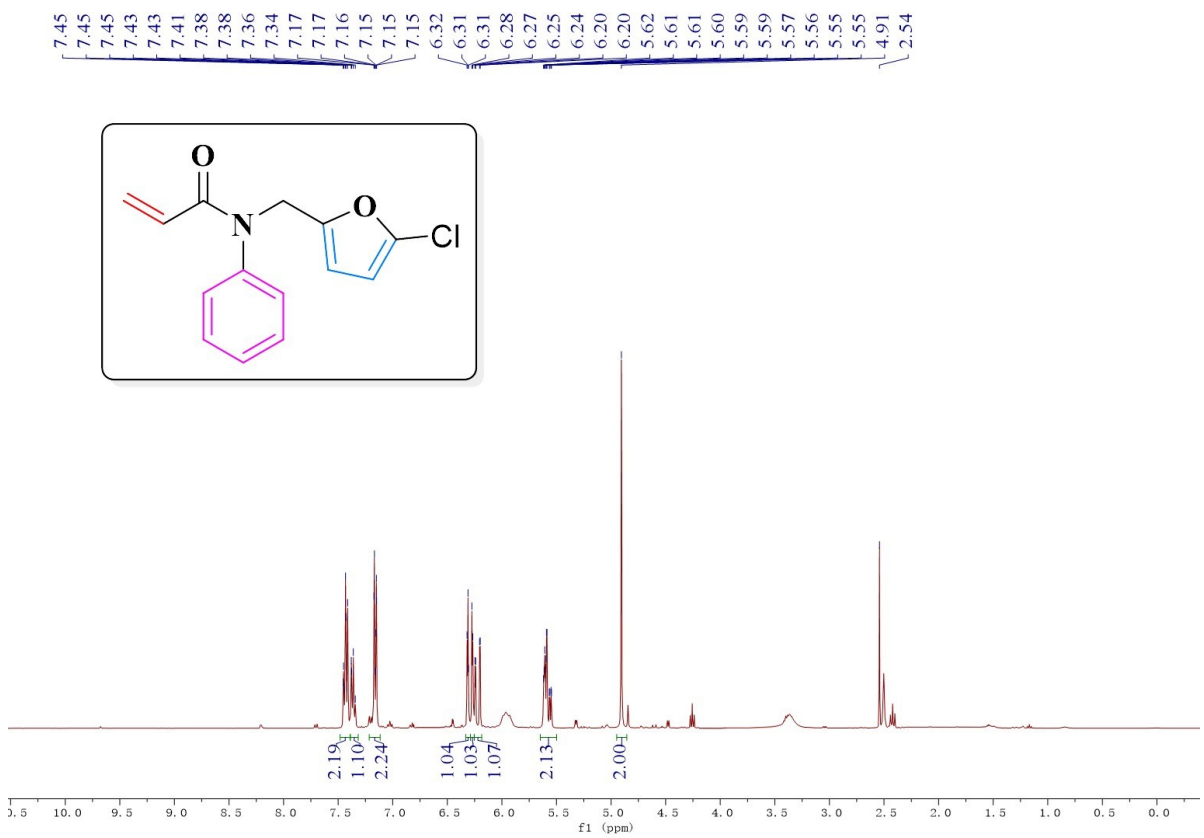

O
N
O
Br

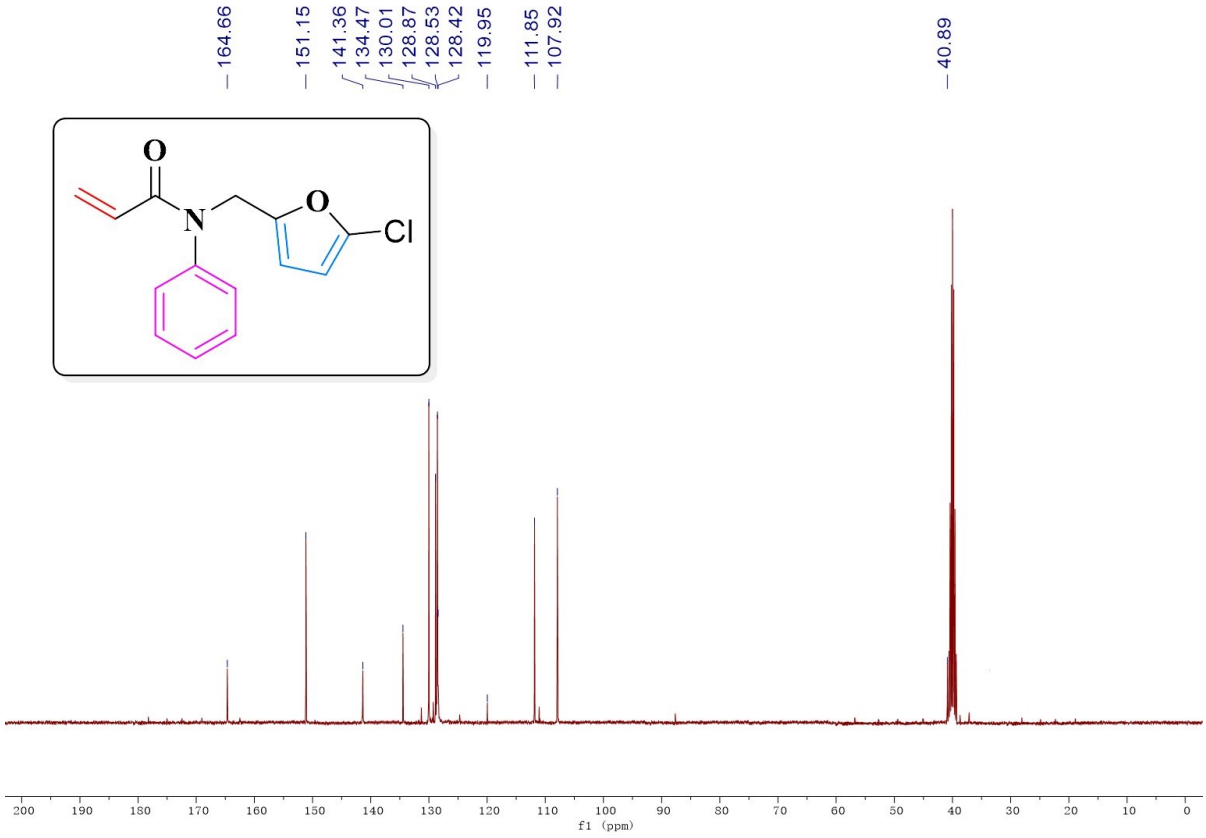

2.27
1.11
1.99
1.03
1.36
0.66
0.88
1.02
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

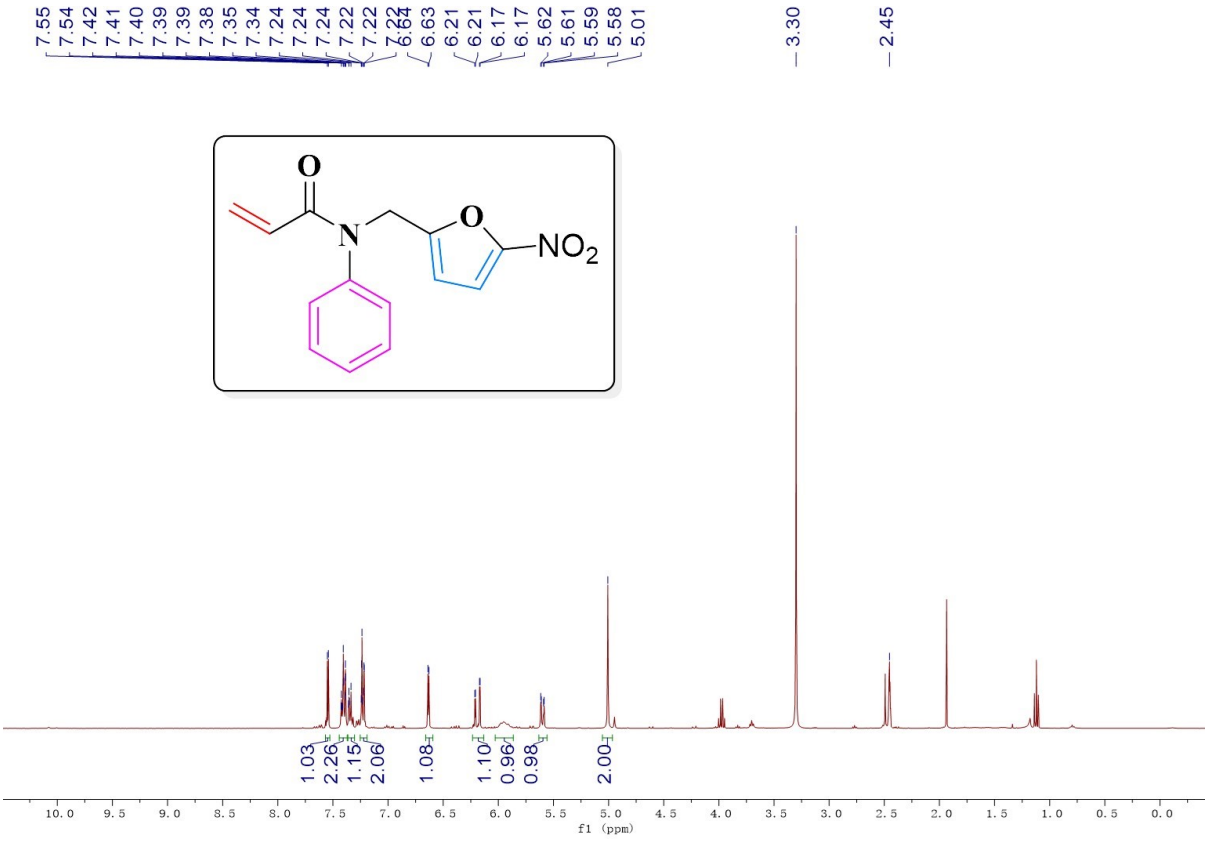

164.66
153.35
141.38
130.02
128.89
128.63
128.54
128.43
120.90
112.82
112.17
45.38

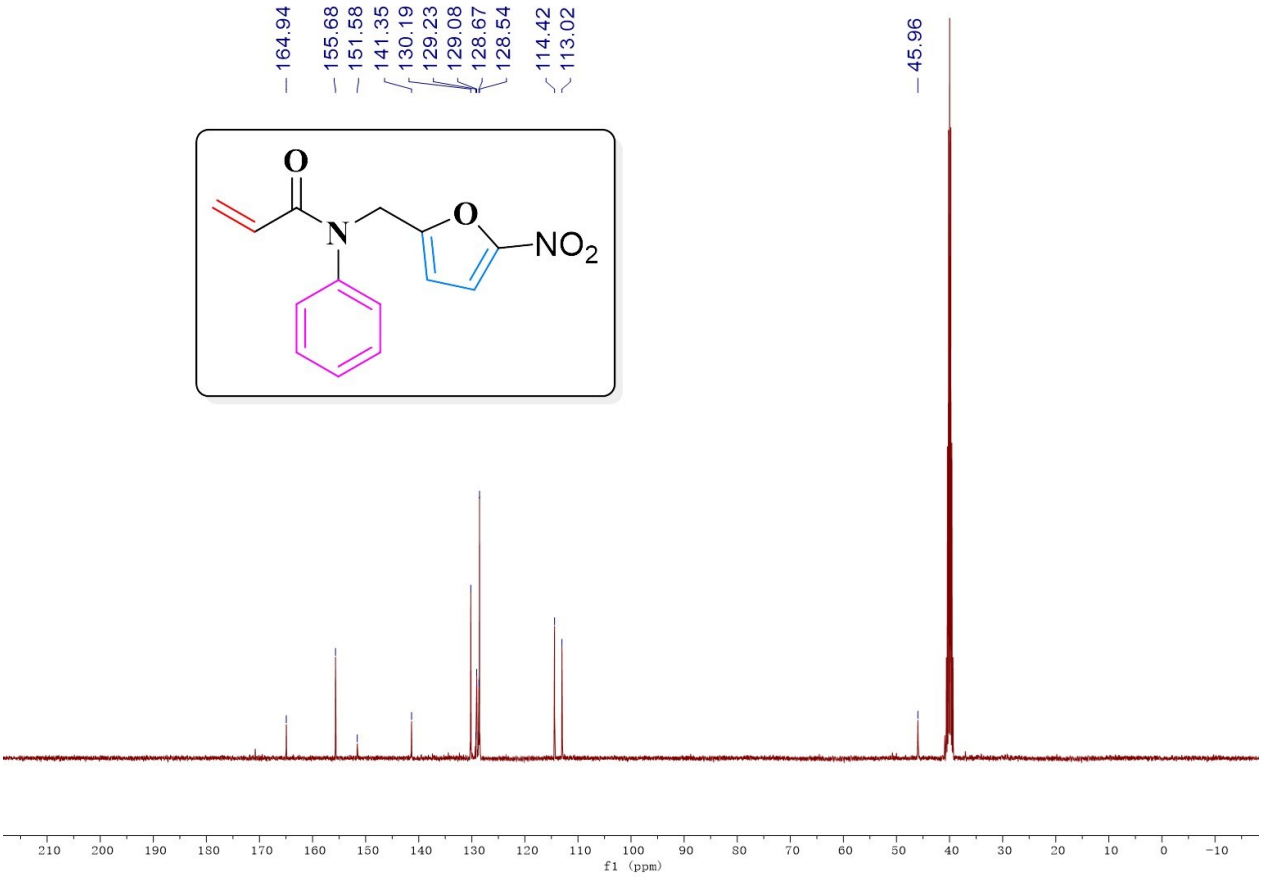

O
N
O
Br

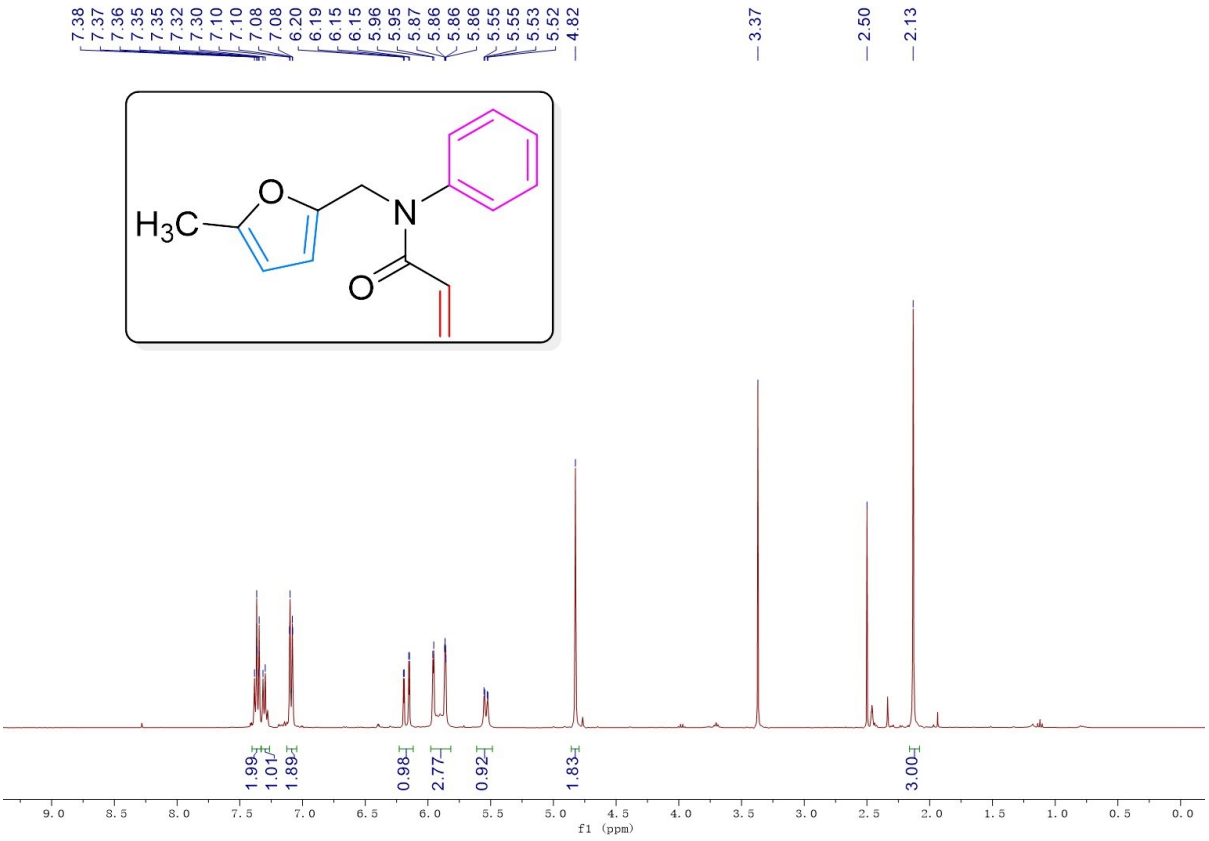

180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

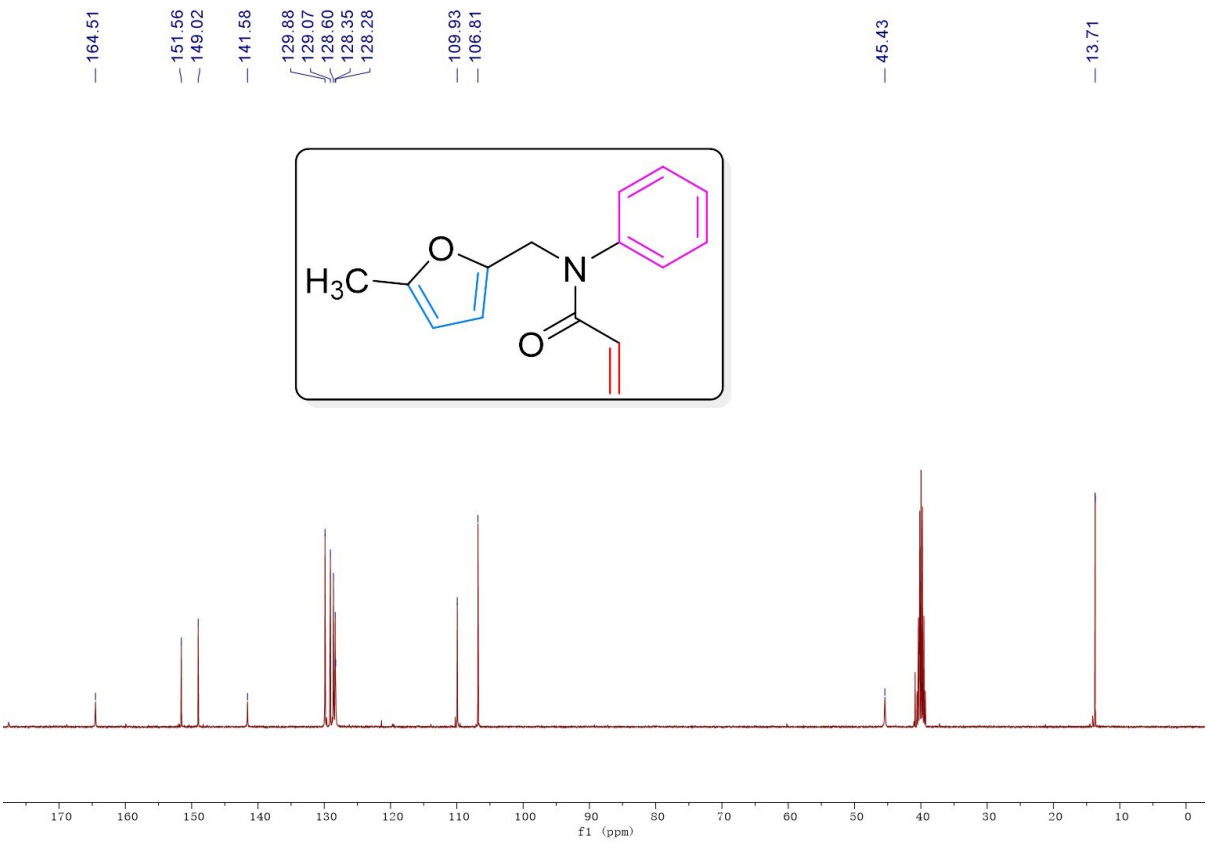

7.45
7.45
7.45
7.43
7.43
7.41
7.38
7.38
7.36
7.34
7.17
7.17
7.16
7.15
7.15
7.15
6.32
6.31
6.31
6.28
6.27
6.25
6.24
6.20
6.20
5.62
5.61
5.61
5.60
5.59
5.59
5.57
5.56
5.55
5.55
4.91
2.54
O
N
O
Cl
2.19
1.10
2.24
1.04
1.03
1.07
2.13
2.00
f1 (ppm)

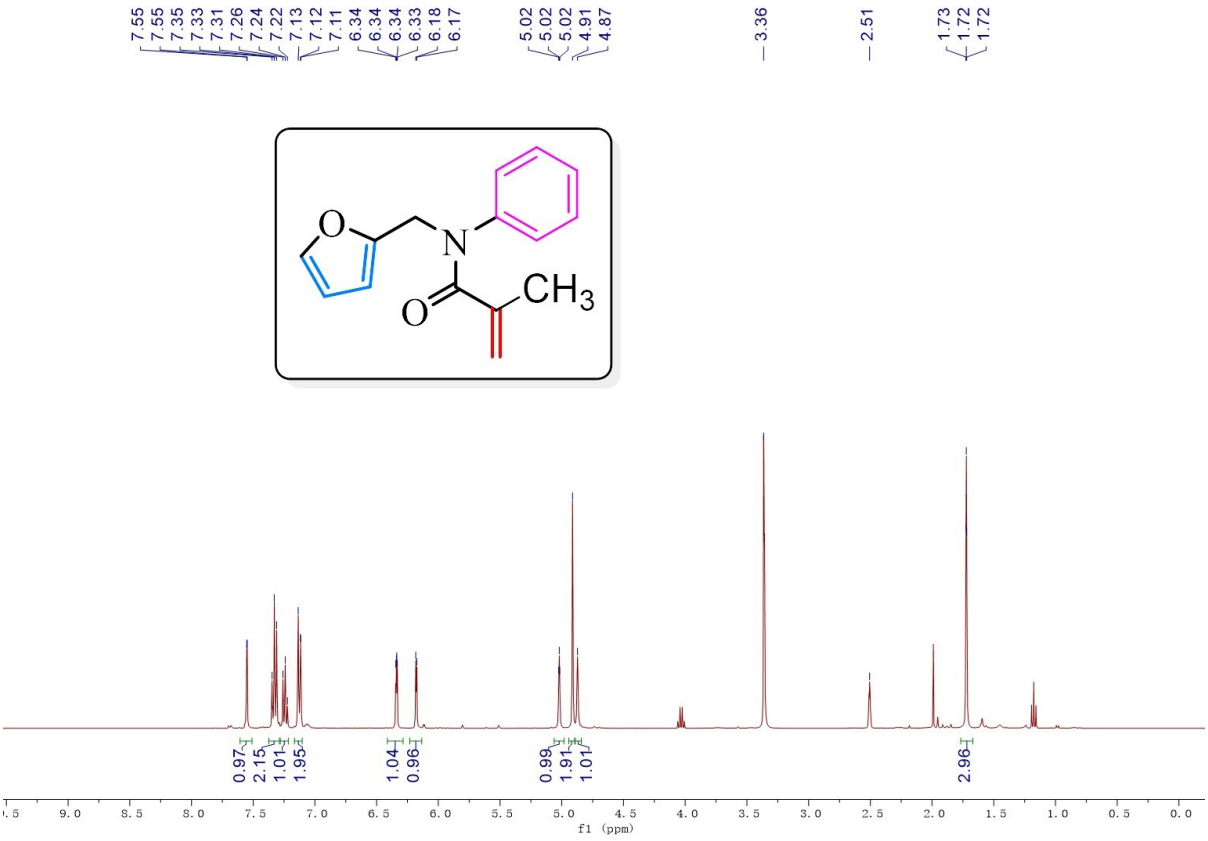

164.66
151.15
141.36
134.47
130.01
128.87
128.53
128.42
119.95
111.85
107.92
40.89
O
N
O
Cl
f1 (ppm)

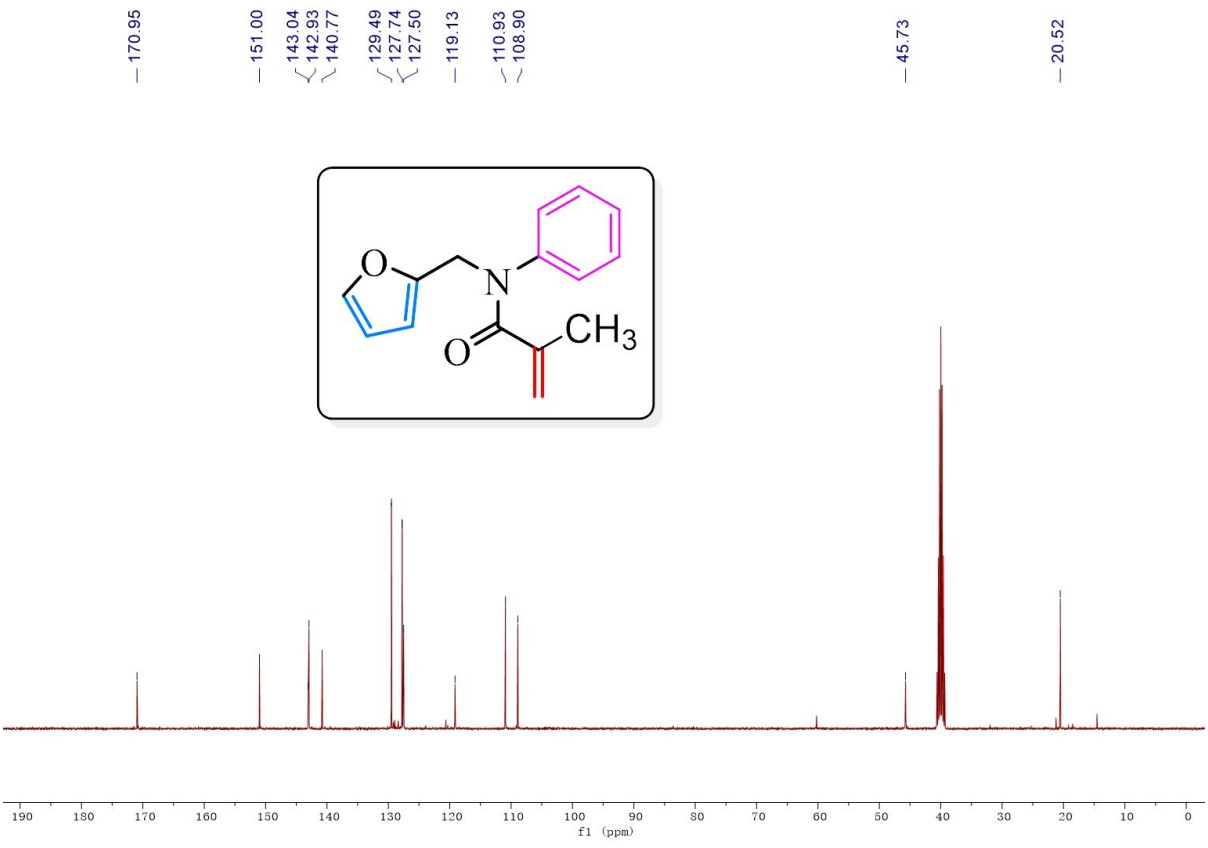

7.55
7.54
7.42
7.41
7.40
7.39
7.39
7.38
7.35
7.34
7.24
7.24
7.24
7.22
7.22
6.64
6.63
6.21
6.21
6.17
6.17
5.62
5.61
5.59
5.58
5.01
3.30
2.45
O
N
O
NO2
1.03
2.26
1.15
2.06
1.08
1.10
0.96
0.98
2.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

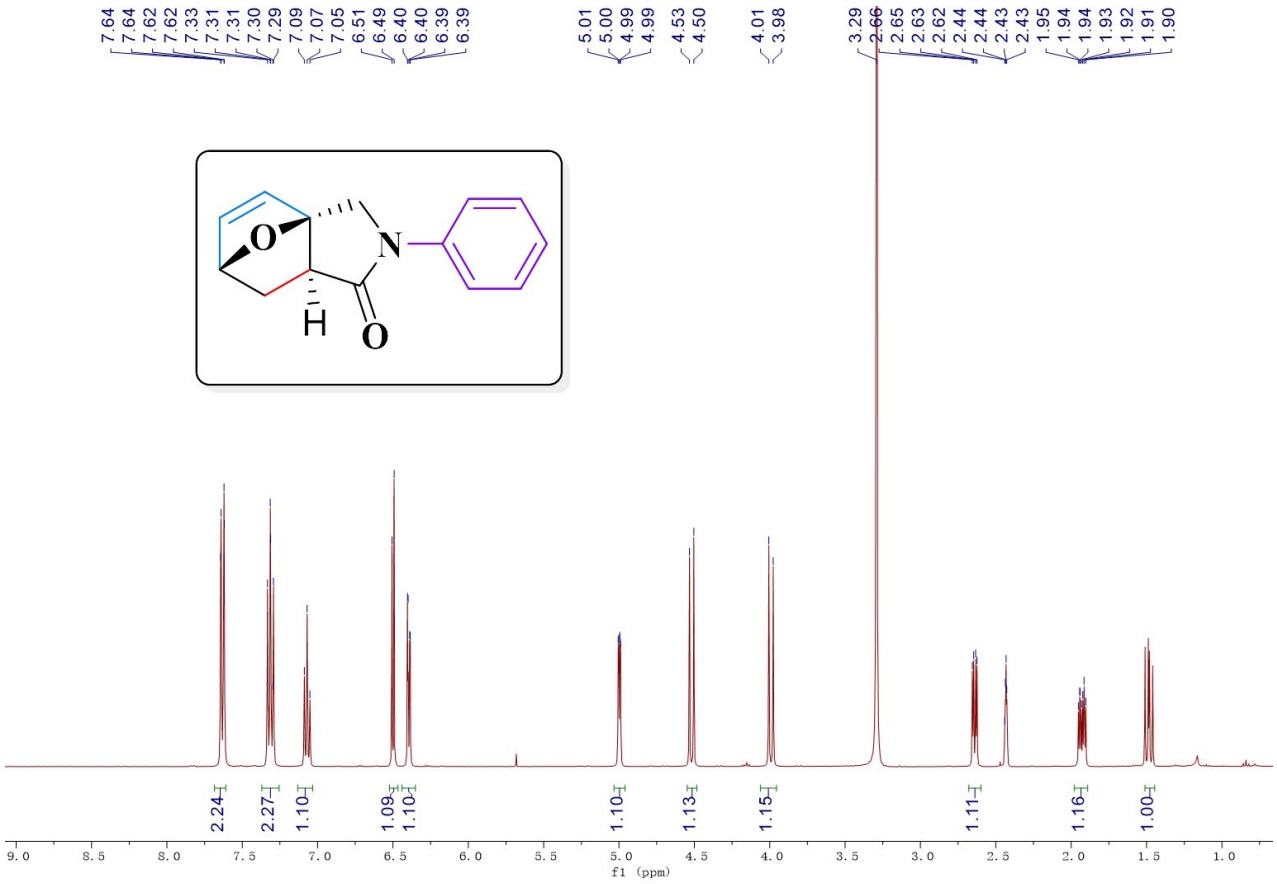

164.94
155.68
151.58
141.35
130.19
129.23
129.08
128.67
128.54
114.42
113.02
45.96
O
N
O
NO2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

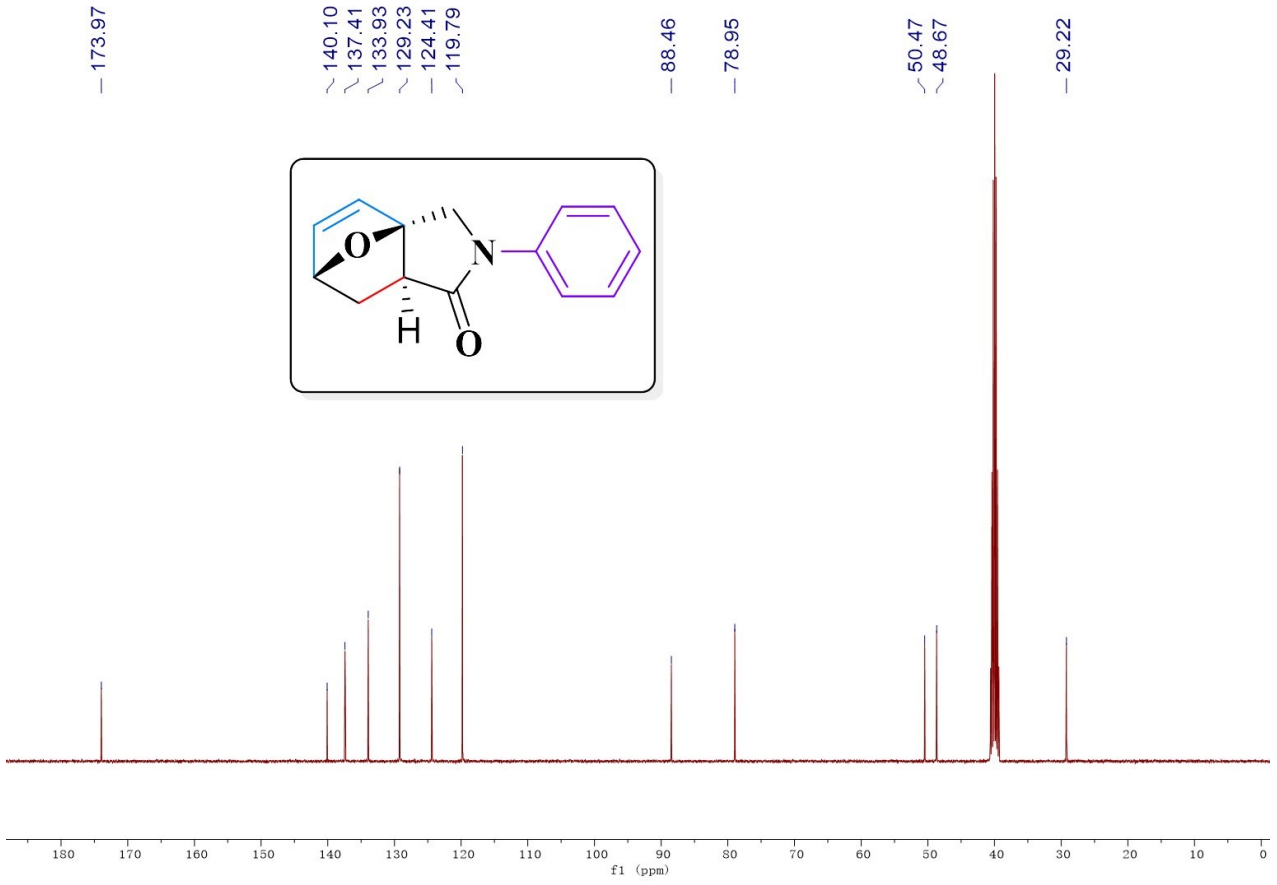

7.38
7.37
7.36
7.35
7.35
7.32
7.30
7.10
7.10
7.08
7.08
6.20
6.19
6.15
6.15
5.96
5.95
5.87
5.86
5.86
5.86
5.55
5.53
5.52
4.82
3.37
2.50
2.13
O
N
O
H3C
1.99
1.01
1.89
0.98
2.77
0.92
1.83
3.00
f1 (ppm)

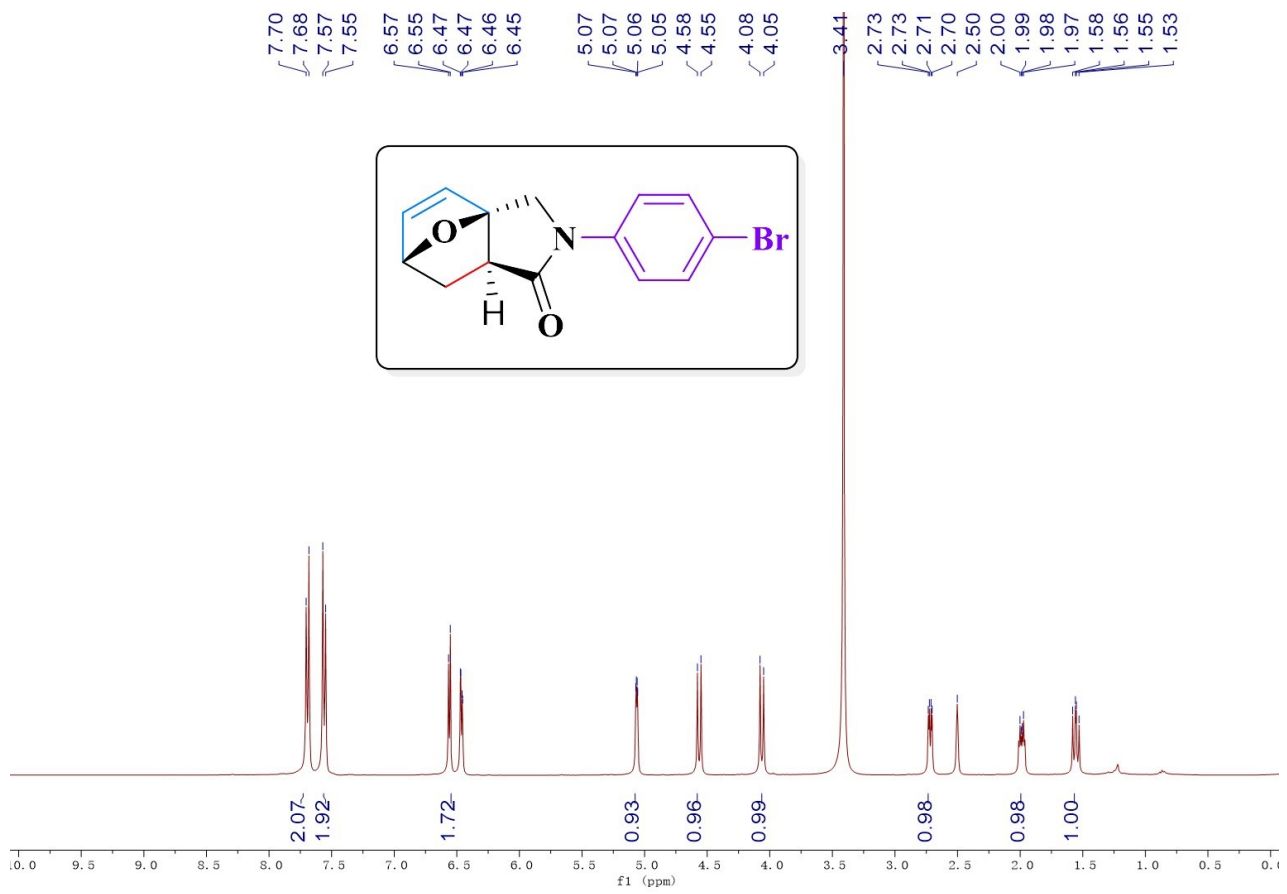

164.51
151.56
149.02
141.58
129.88
129.07
128.60
128.35
128.28
109.93
106.81
45.43
13.71
O
N
O
H3C
f1 (ppm)

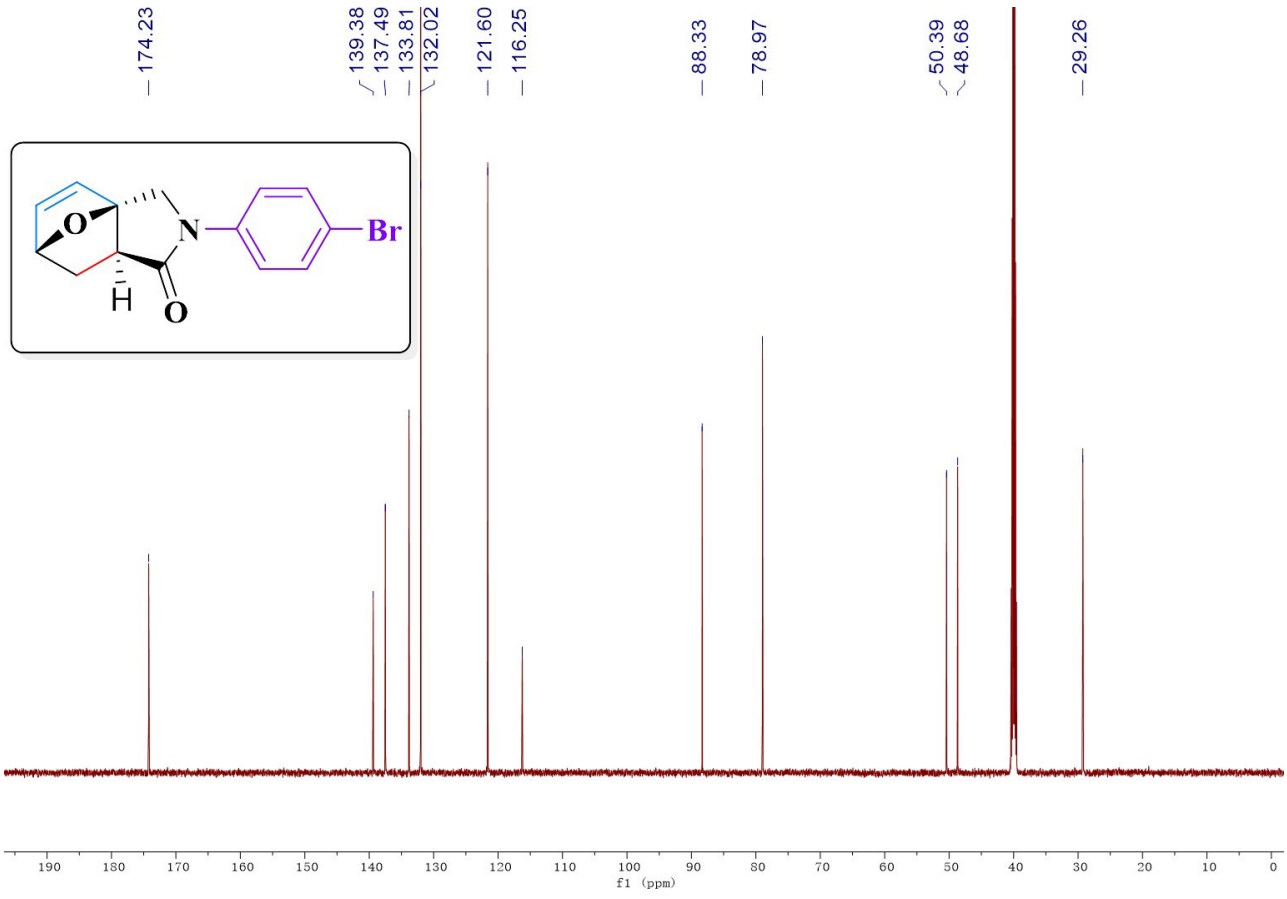

7.55
7.55
7.35
7.33
7.31
7.26
7.24
7.22
7.13
7.12
7.11
6.34
6.34
6.34
6.33
6.18
6.17
5.02
5.02
5.02
4.91
4.87
3.36
2.51
1.73
1.72
1.72
O
N
O
CH3
0.97
2.15
1.01
1.95
1.04
0.96
0.99
1.91
1.01
2.96
f1 (ppm)

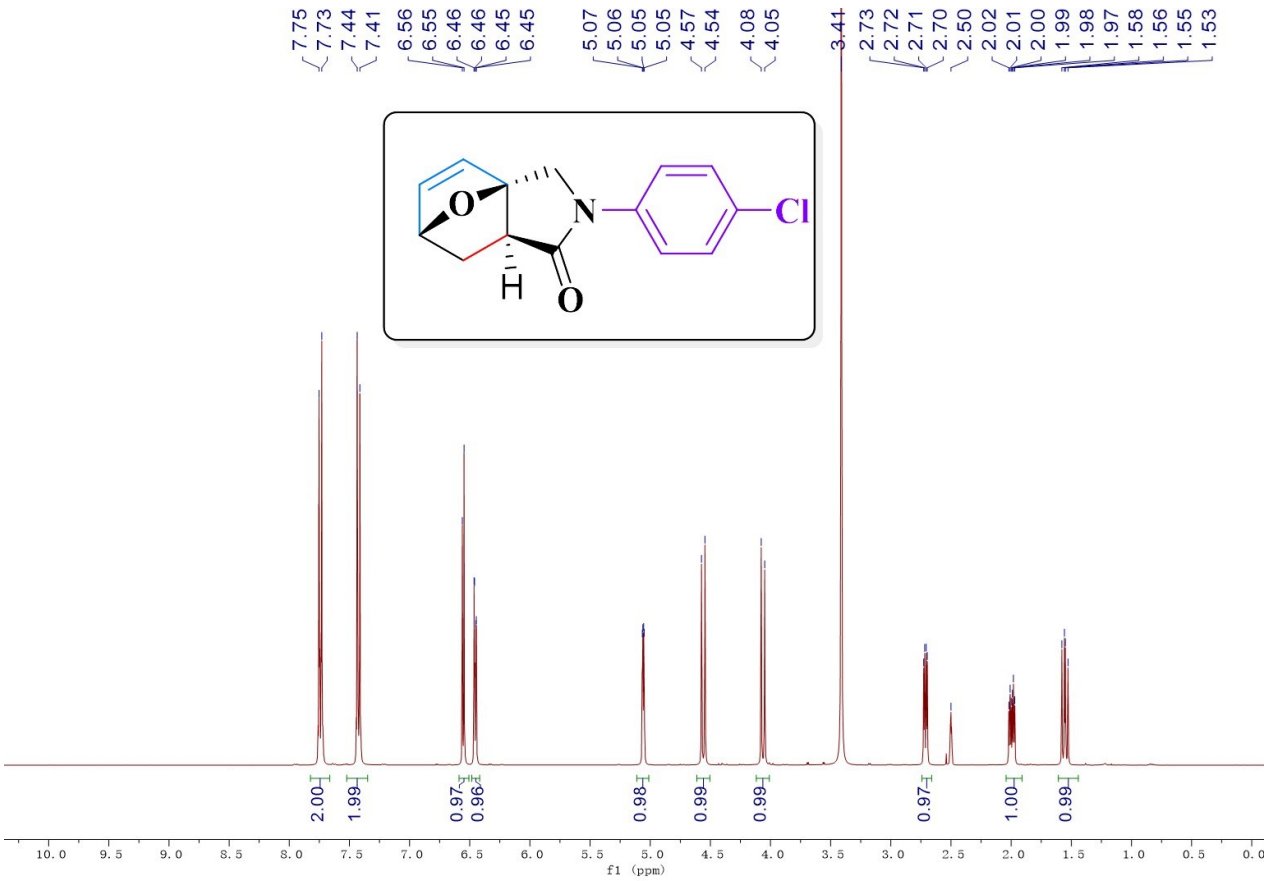

170.95
151.00
143.04
142.93
140.77
129.49
127.74
127.50
119.13
110.93
108.90
45.73
20.52
O
N
O
CH3
f1 (ppm)

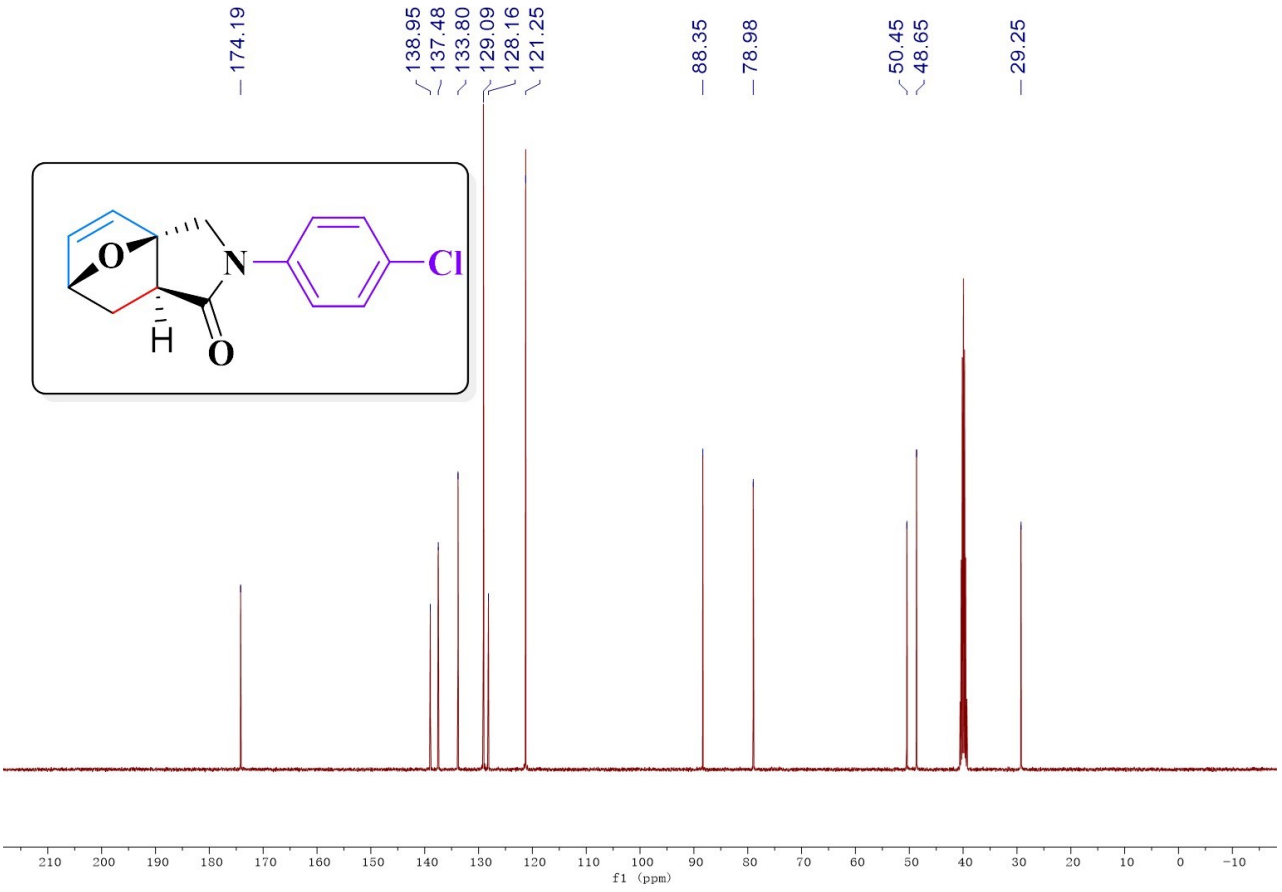

7.64
7.64
7.62
7.62
7.33
7.31
7.31
7.30
7.29
7.09
7.07
7.05
6.51
6.49
6.40
6.40
6.39
6.39
5.01
5.00
4.99
4.99
4.53
4.50
4.01
3.98
3.29
2.66
2.65
2.63
2.62
2.44
2.44
2.43
2.43
1.95
1.94
1.94
1.93
1.92
1.91
1.90
O
N
H
O
2.24
2.27
1.10
1.09
1.10
1.10
1.13
1.15
1.11
1.16
1.00
f1 (ppm)

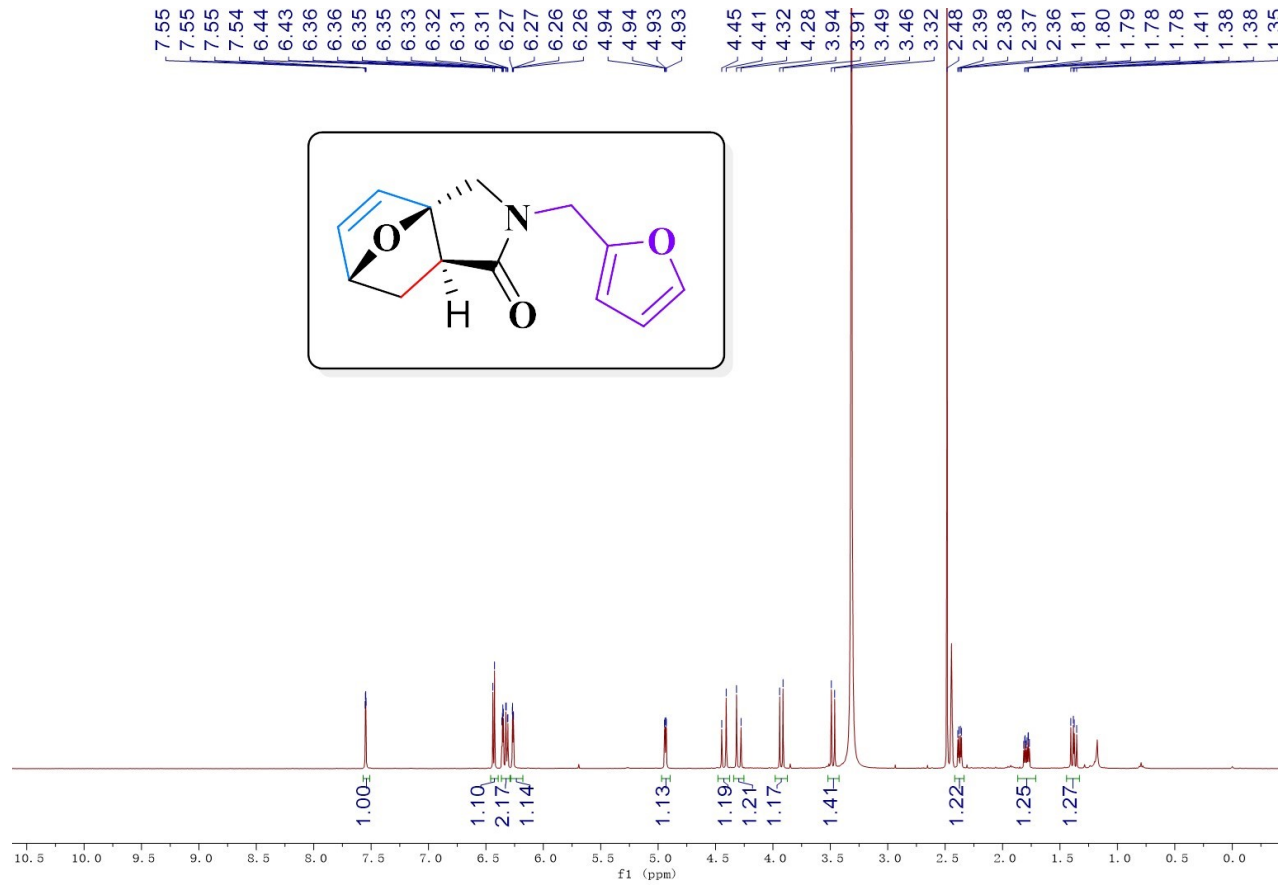

173.97
140.10
137.41
133.93
129.23
124.41
119.79
88.46
78.95
50.47
48.67
29.22
O
N
H
O
f1 (ppm)

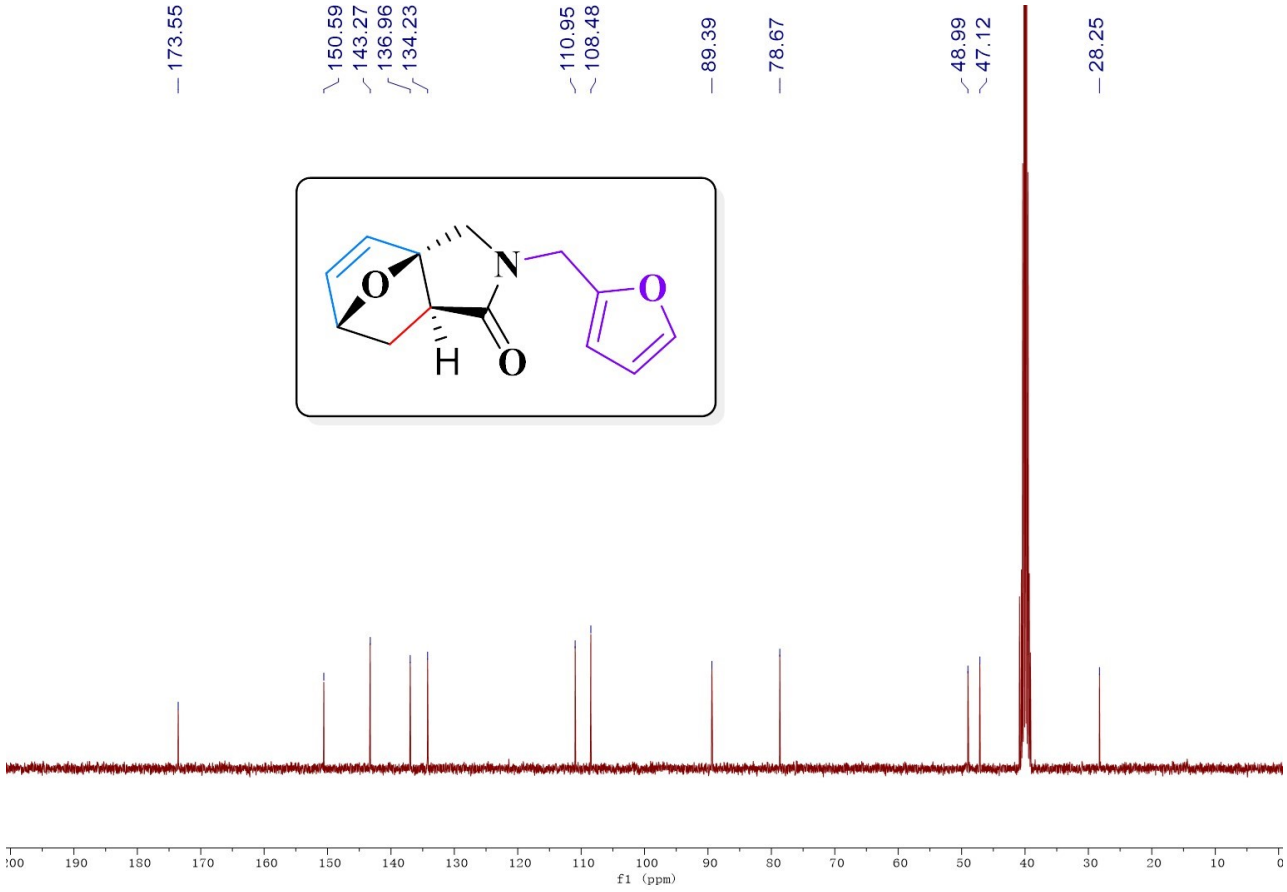

7.70
7.68
7.57
7.55
6.57
6.55
6.47
6.47
6.46
6.45
5.07
5.07
5.06
5.05
4.58
4.55
4.08
4.05
3.41
2.73
2.73
2.71
2.70
2.50
2.00
1.99
1.98
1.97
1.58
1.56
1.55
1.53
Br
O
N
H
O
2.07
1.92
1.72
0.93
0.96
0.99
0.98
0.98
1.00
f1 (ppm)

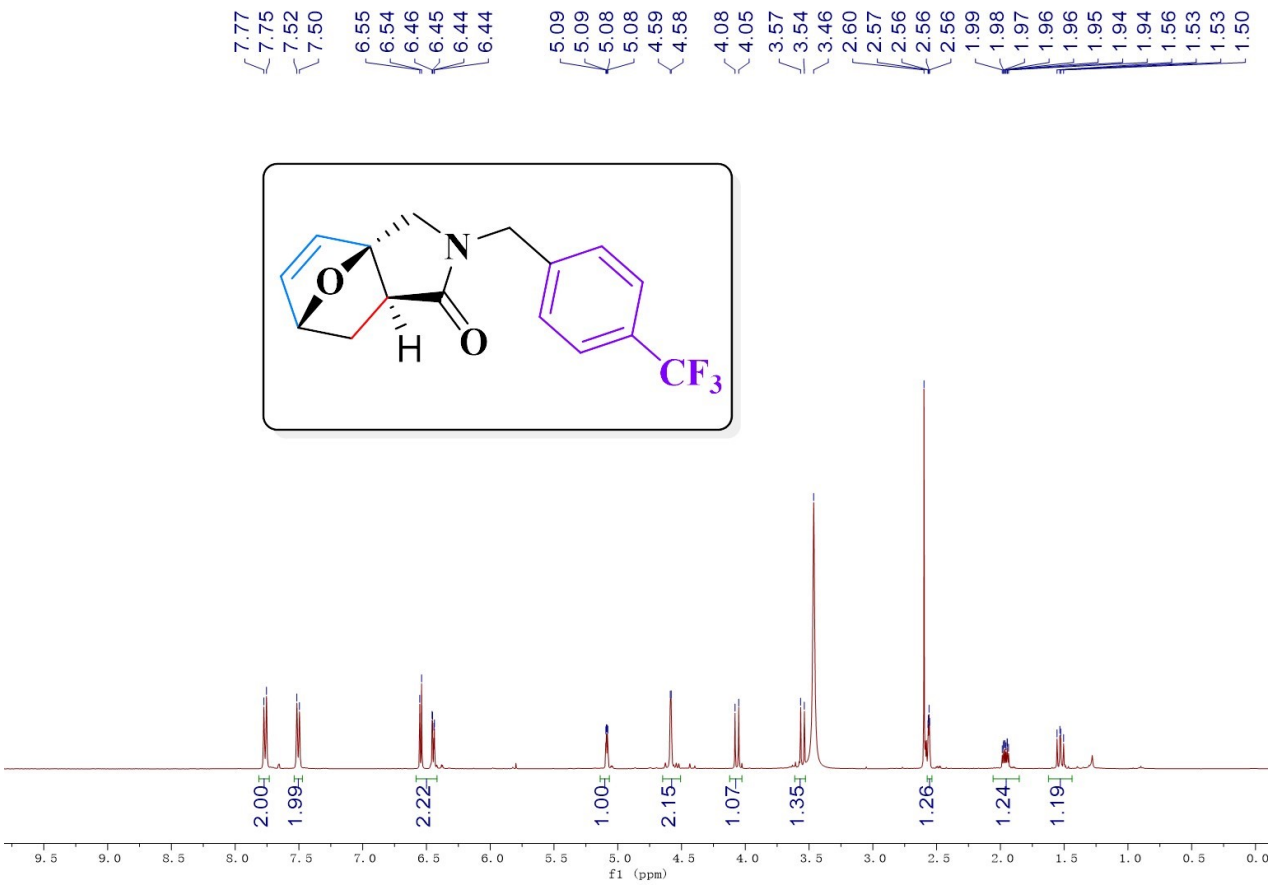

174.23
139.38
137.49
133.81
132.02
121.60
116.25
88.33
78.97
50.39
48.68
29.26
Br
O
N
H
O
f1 (ppm)

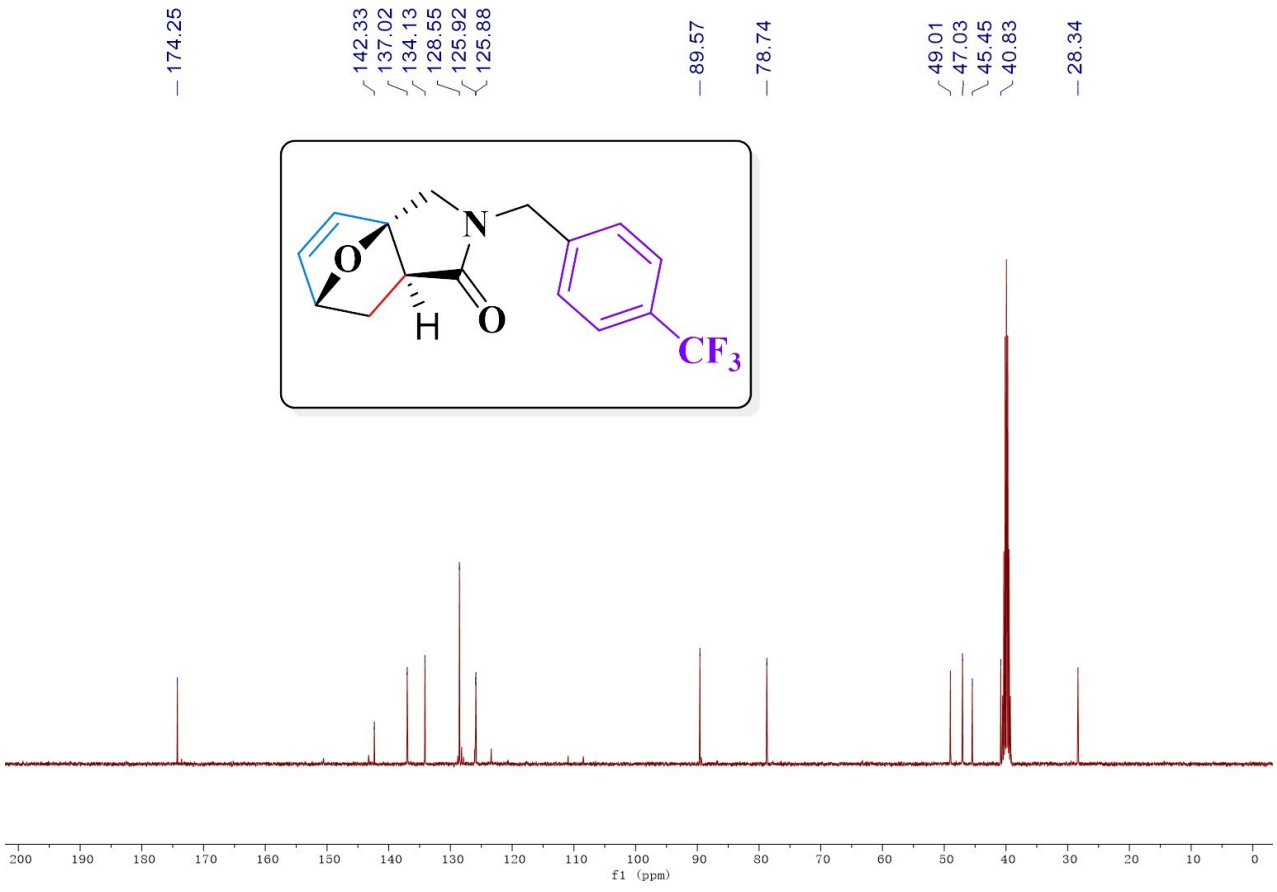

7.75
7.73
7.44
7.41
6.56
6.55
6.46
6.46
6.45
6.45
5.07
5.06
5.05
5.05
4.57
4.54
4.08
4.05
3.41
2.73
2.72
2.71
2.70
2.50
2.02
2.01
2.00
1.99
1.98
1.97
1.58
1.56
1.55
1.53
2.00
1.99
0.97
0.96
0.98
0.99
0.99
0.97
1.00
0.99
O
N
Cl
H
O
f1 (ppm)

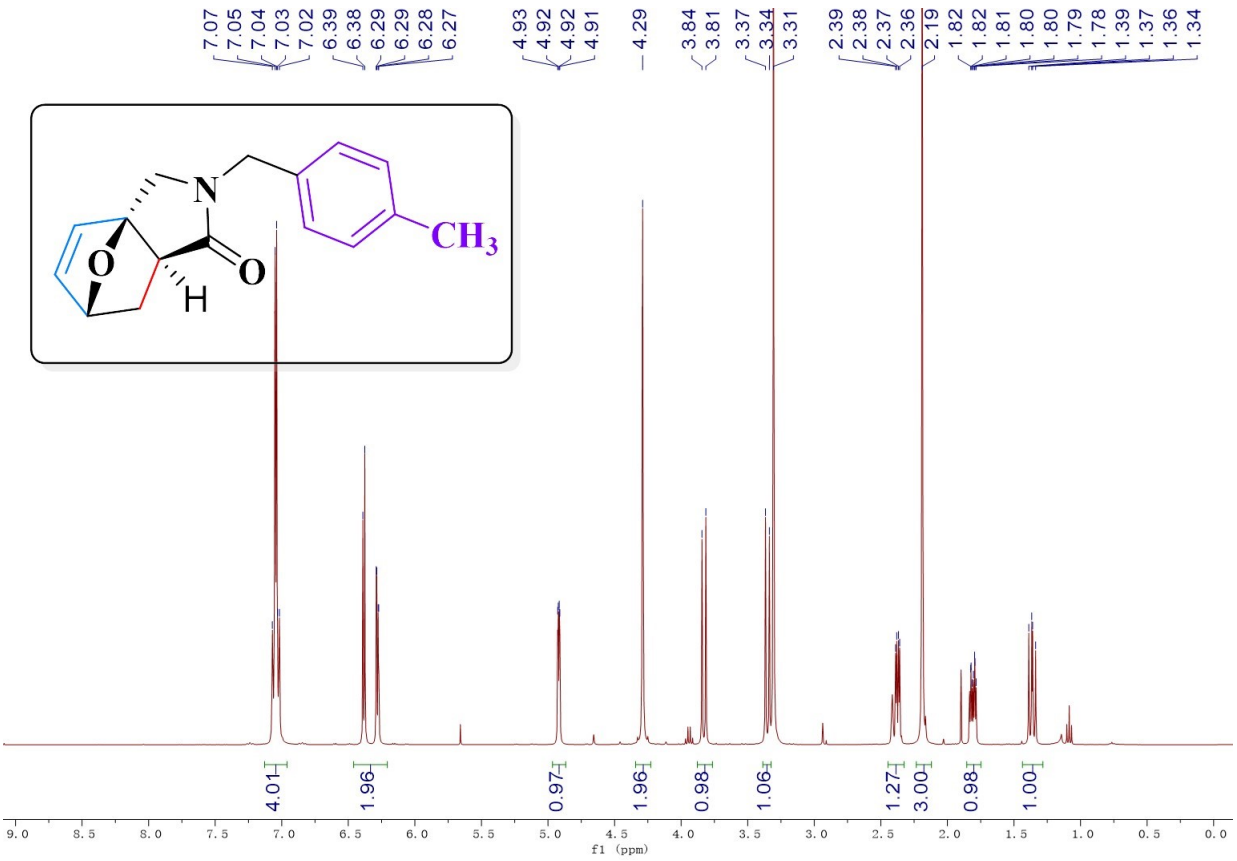

174.19
138.95
137.48
133.80
129.09
128.16
121.25
88.35
78.98
50.45
48.65
29.25
O
N
Cl
H
O
f1 (ppm)

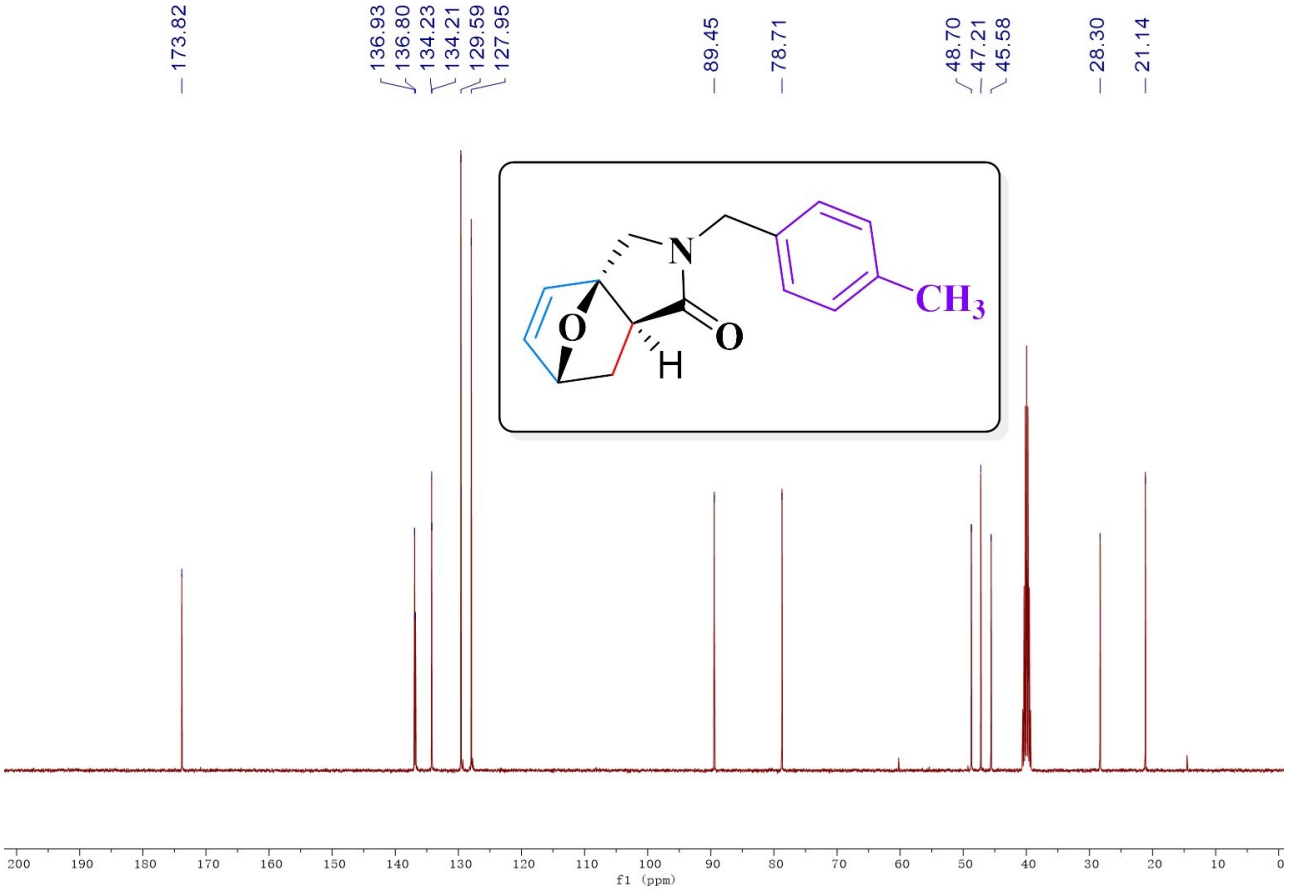

7.55
7.55
7.55
7.54
6.44
6.43
6.36
6.36
6.35
6.35
6.33
6.32
6.31
6.31
6.27
6.27
6.26
6.26
4.94
4.94
4.93
4.93
4.45
4.41
4.32
4.28
3.94
3.91
3.49
3.46
3.32
2.48
2.39
2.38
2.37
2.36
1.81
1.80
1.79
1.78
1.78
1.41
1.38
1.38
O
N
O
H
O
1.00
1.10
2.17
1.14
1.13
1.19
1.21
1.17
1.41
1.22
1.25
1.27
f1 (ppm)

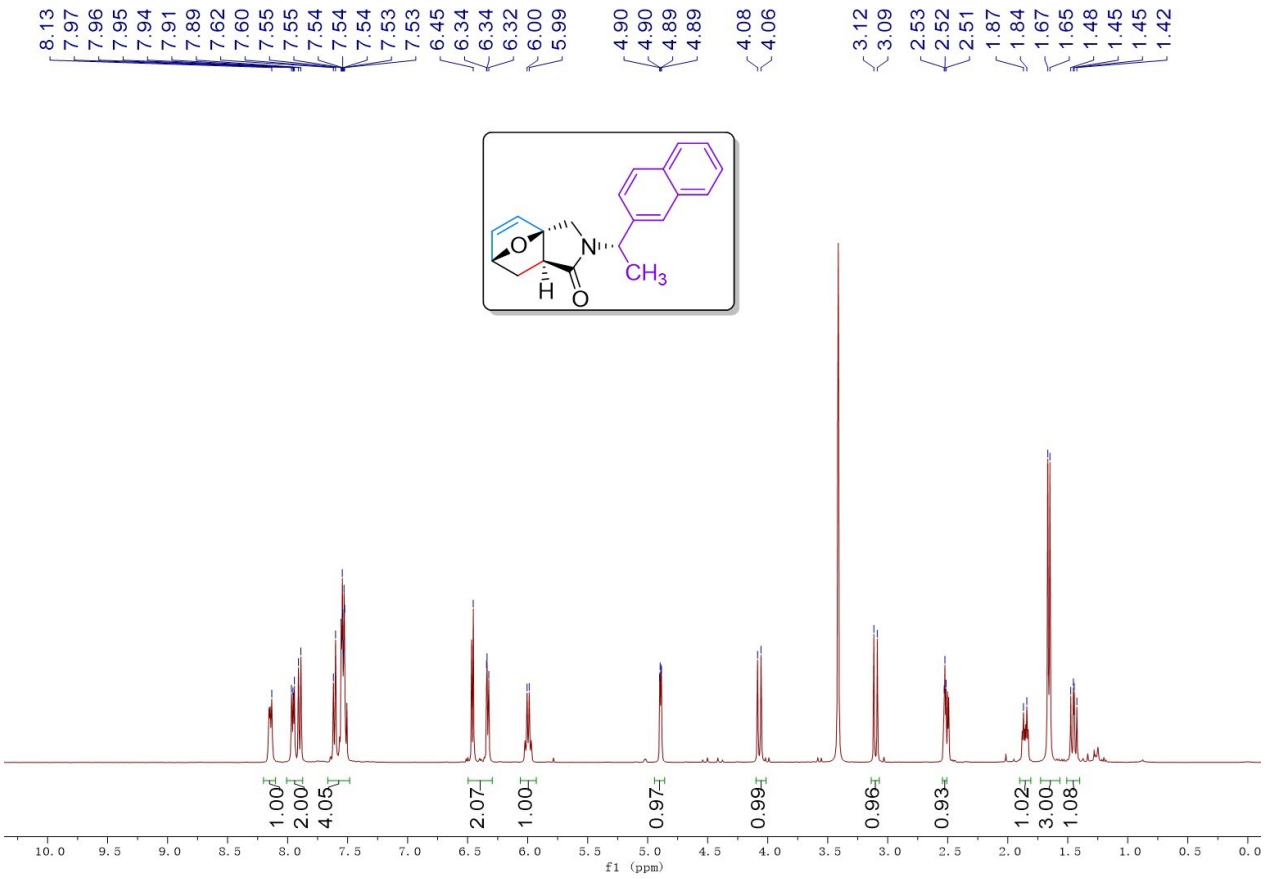

173.55
150.59
143.27
136.96
134.23
110.95
108.48
89.39
78.67
48.99
47.12
28.25
O
N
O
H
O
f1 (ppm)

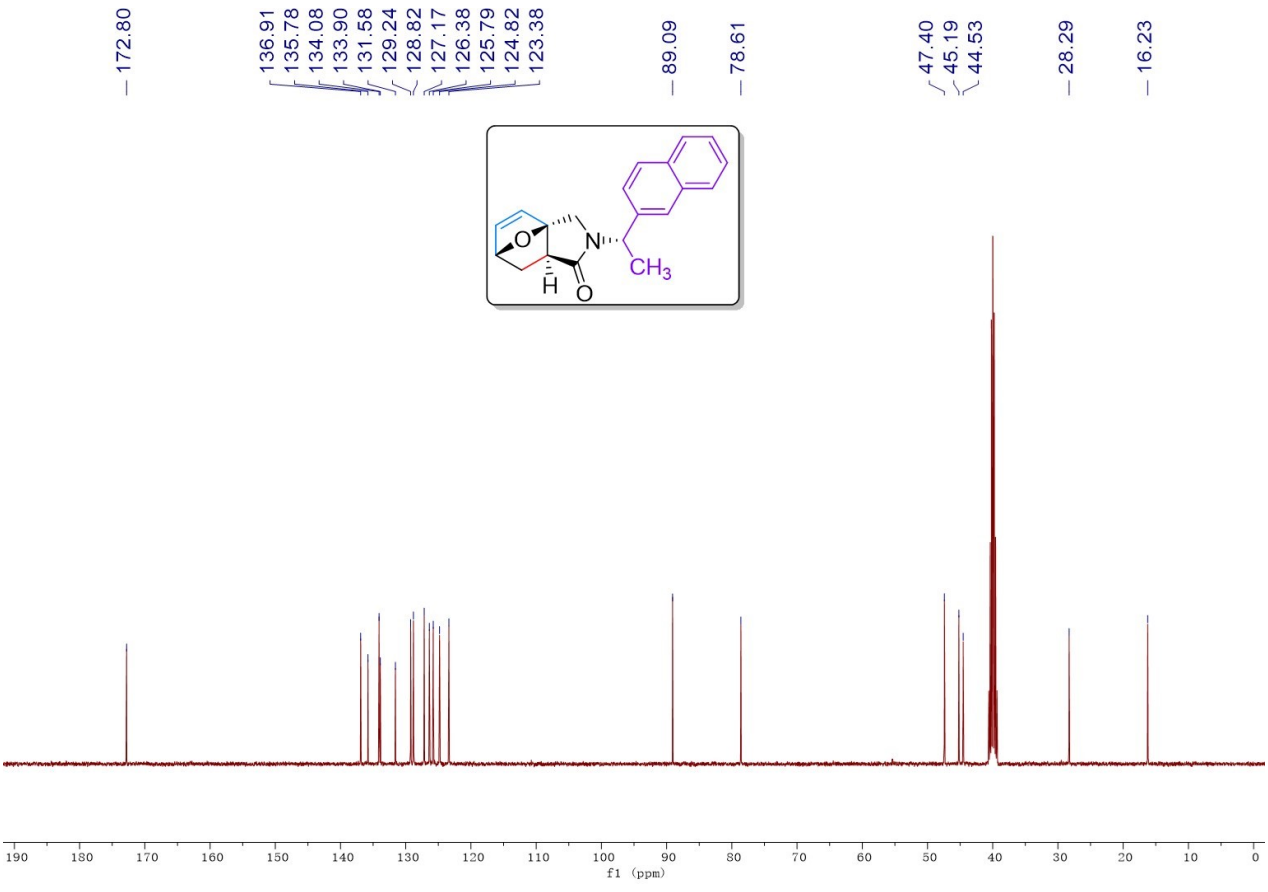

7.77
7.75
7.52
7.50
6.55
6.54
6.46
6.45
6.44
6.44
5.09
5.09
5.08
5.08
4.59
4.58
4.08
4.05
3.57
3.54
3.46
2.60
2.57
2.56
2.56
2.56
1.99
1.98
1.97
1.96
1.96
1.95
1.94
1.94
1.56
1.53
1.53
1.50
N
O
H
O
CF3
2.00
1.99
2.22
1.00
2.15
1.07
1.35
1.26
1.24
1.19
f1 (ppm)

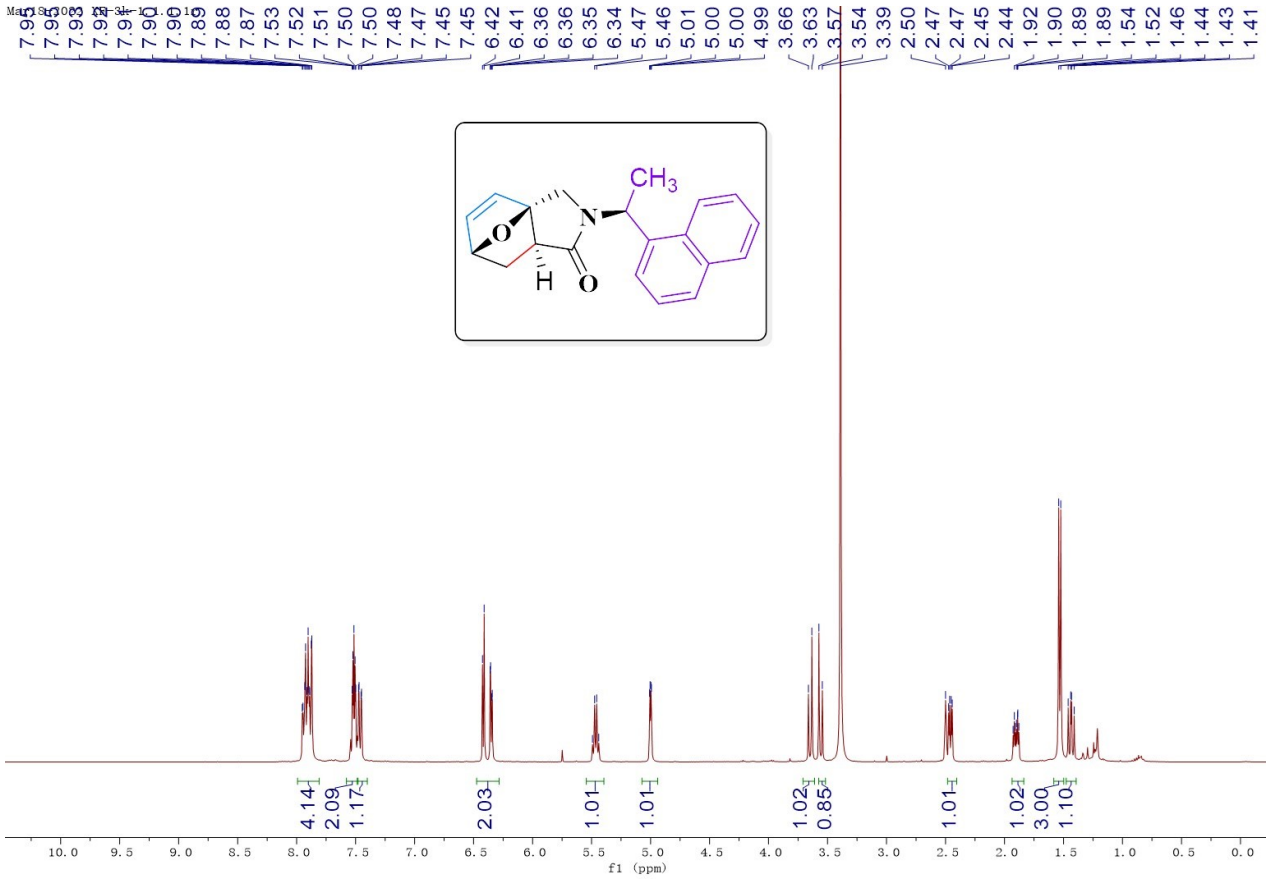

174.25
142.33
137.02
134.13
128.55
125.92
125.88
89.57
78.74
49.01
47.03
45.45
40.83
28.34
N
O
H
O
CF3
f1 (ppm)

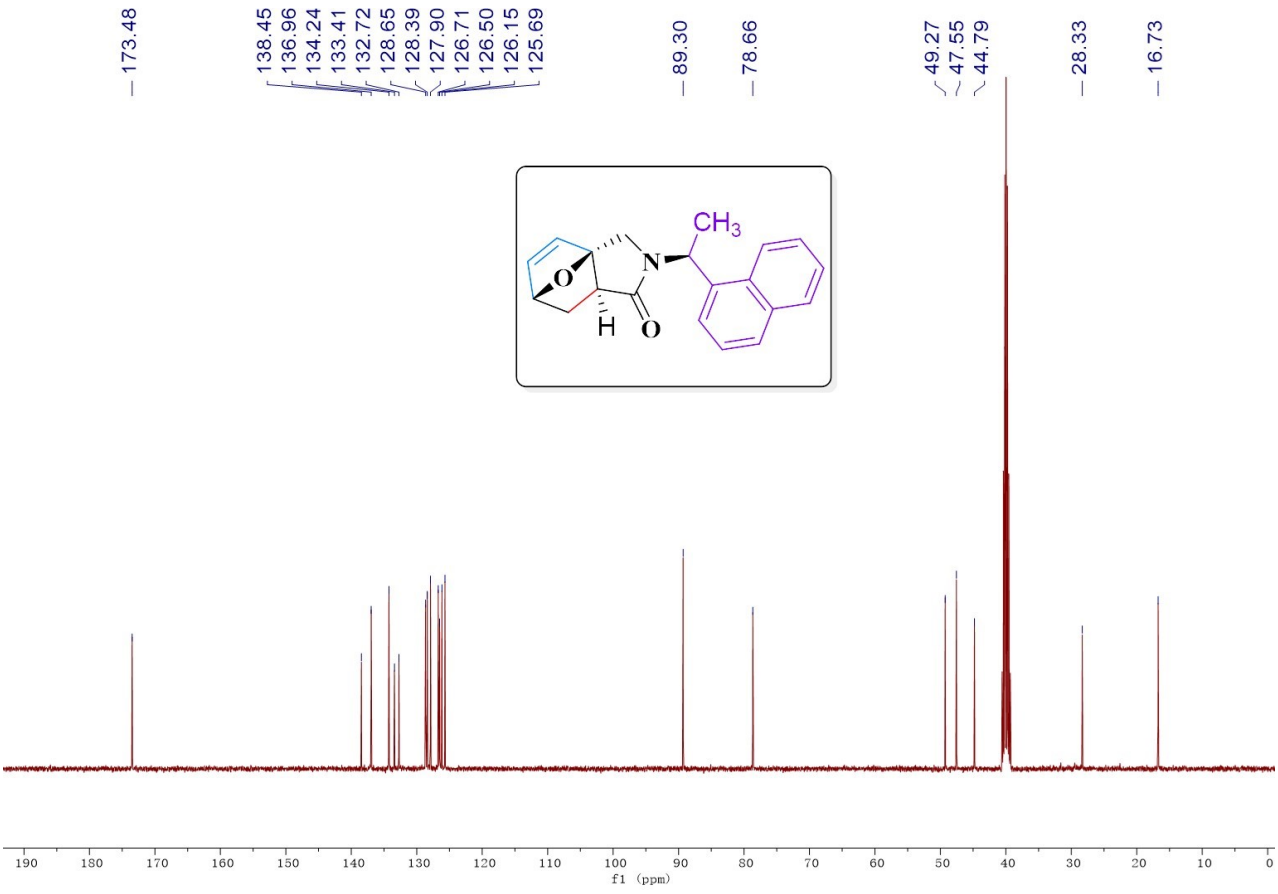

7.07
7.05
7.04
7.03
7.02
6.39
6.38
6.29
6.29
6.28
6.27
4.93
4.92
4.92
4.91
4.29
3.84
3.81
3.37
3.34
3.31
2.39
2.38
2.37
2.36
2.19
1.82
1.81
1.80
1.80
1.79
1.78
1.39
1.37
1.36
1.34
CH3
N
O
O
H
4.01
1.96
0.97
1.96
0.98
1.06
1.27
3.00
0.98
1.00
f1 (ppm)

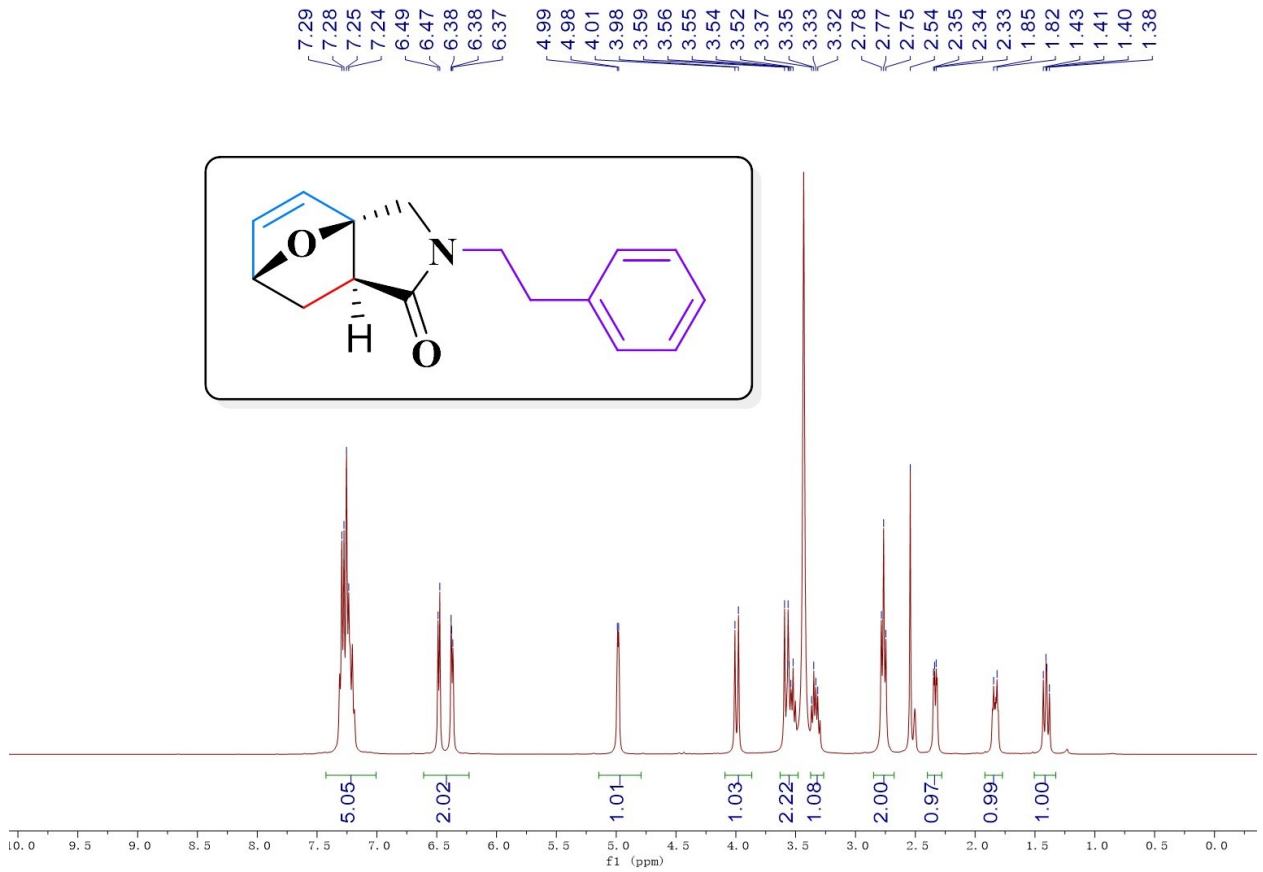

173.82
136.93
136.80
134.23
134.21
129.59
127.95
89.45
78.71
48.70
47.21
45.58
28.30
21.14
CH3
N
O
O
H
f1 (ppm)

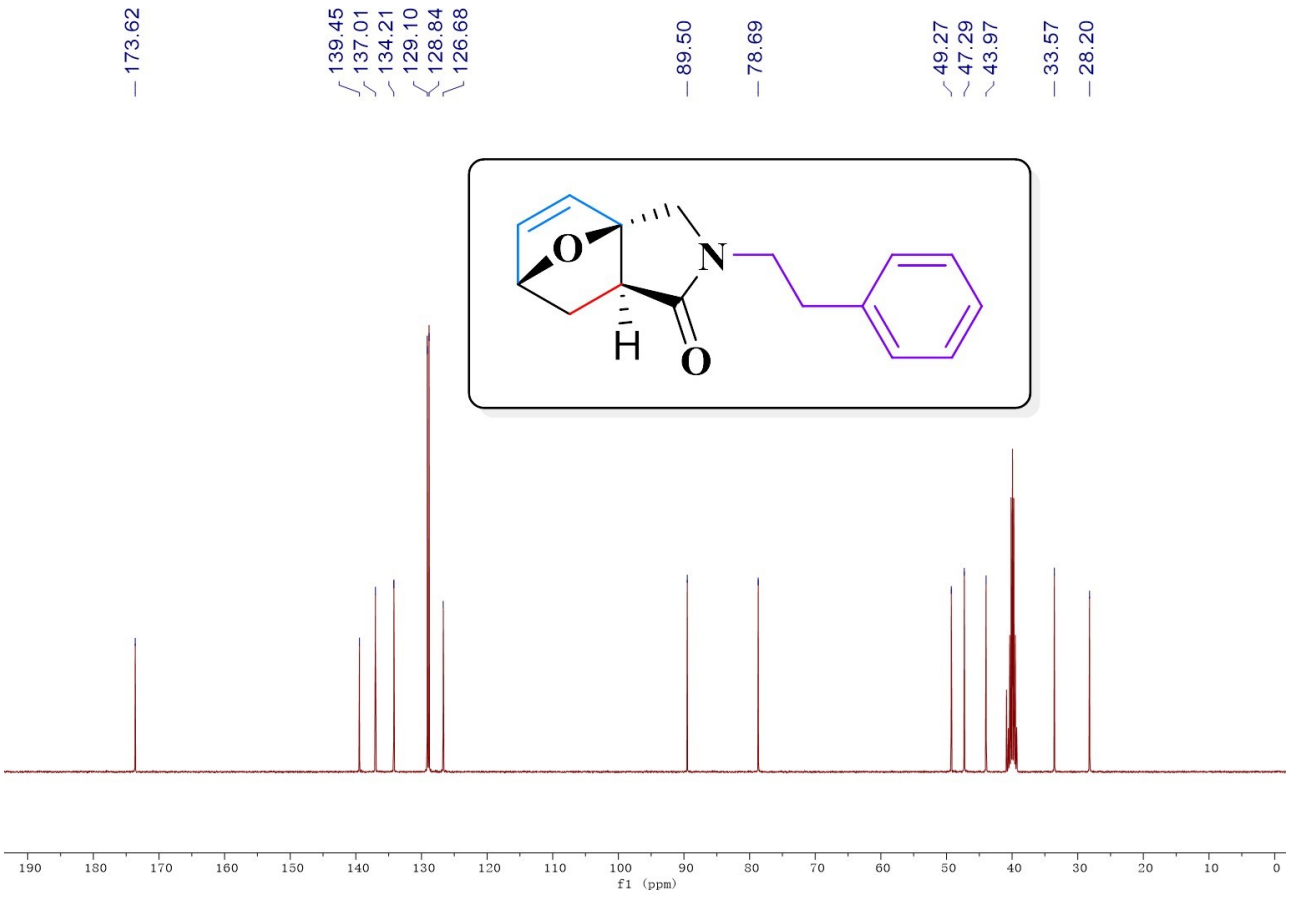

8.13
7.97
7.96
7.95
7.94
7.91
7.89
7.62
7.60
7.55
7.55
7.54
7.54
7.54
7.53
7.53
6.45
6.34
6.34
6.32
6.00
5.99
4.90
4.90
4.89
4.89
4.08
4.06
3.12
3.09
2.53
2.52
2.51
1.87
1.84
1.67
1.65
1.48
1.45
1.45
1.42
O
N
CH3
H
O
1.00
2.00
4.05
2.07
1.00
0.97
0.99
0.96
0.93
1.02
3.00
1.08
f1 (ppm)

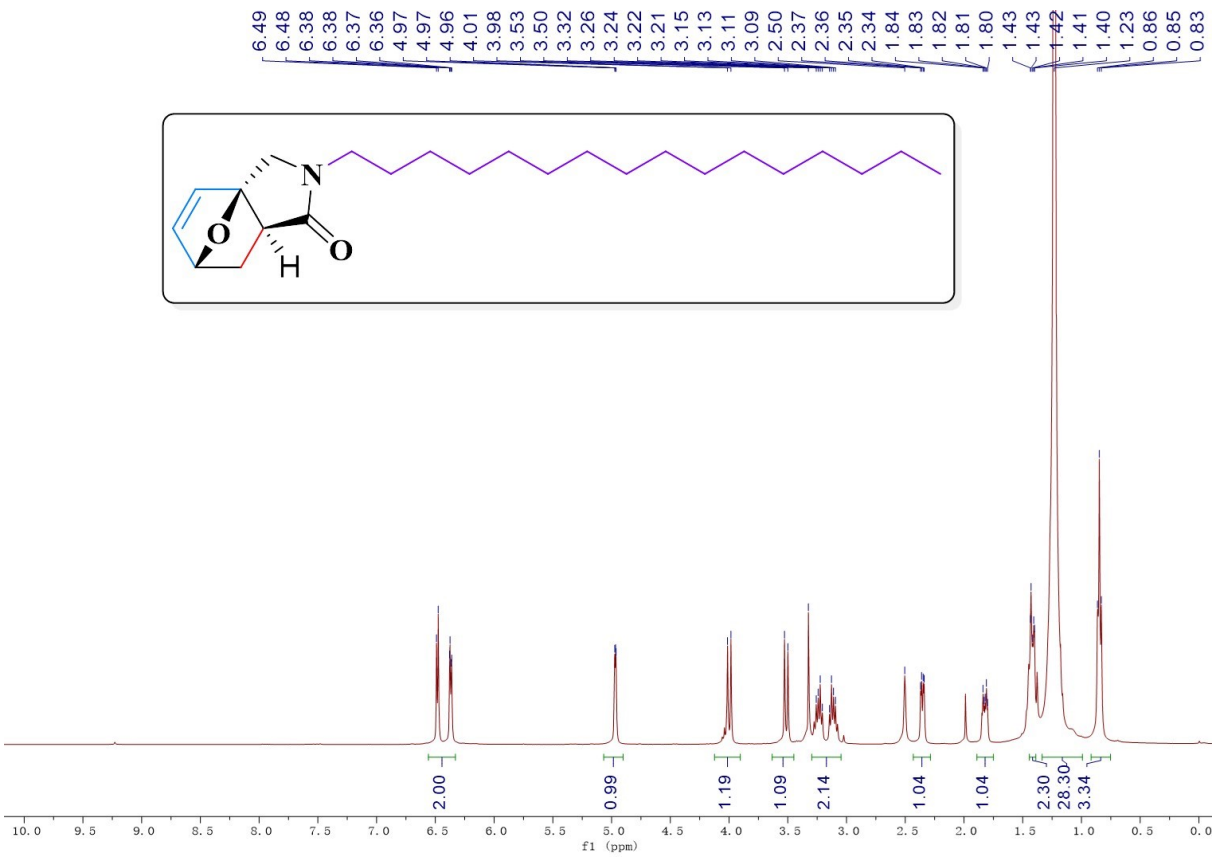

172.80
136.91
135.78
134.08
133.90
131.58
129.24
128.82
127.17
126.38
125.79
124.82
123.38
89.09
78.61
47.40
45.19
44.53
28.29
16.23
O
N
CH3
H
O
f1 (ppm)

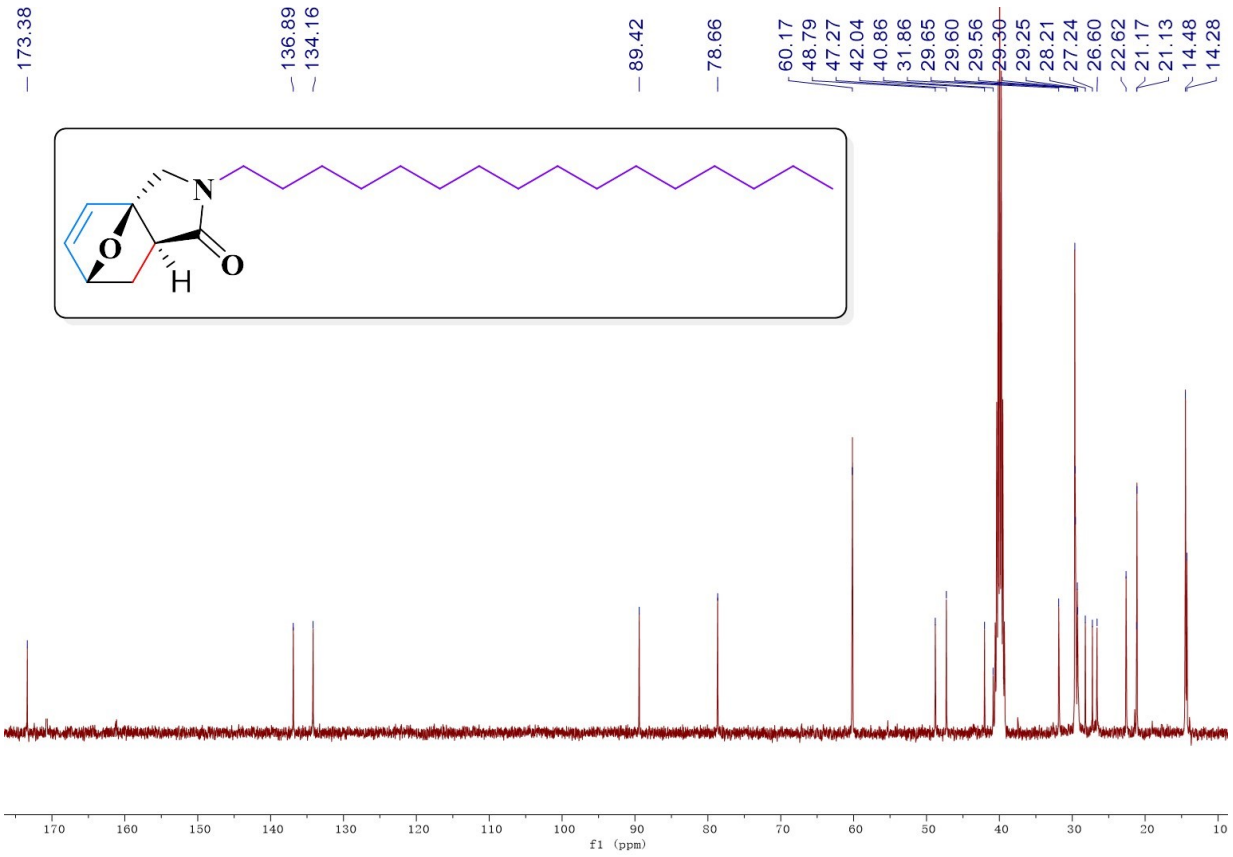

7.95 7.93 7.93 7.90 7.90 7.89 7.88 7.87 7.53 7.52 7.51 7.50 7.50 7.48 7.47 7.45 7.45
6.42 6.41 6.36 6.36 6.35 6.34 5.47 5.46 5.01 5.00 5.00 4.99
3.66 3.63 3.57 3.54 3.39
2.50 2.47 2.47 2.45 2.44
1.92 1.90 1.89 1.89 1.54 1.52 1.46 1.44 1.43 1.41
CH3
N
O
H O
4.14 2.09 1.17 2.03 1.01 1.01 1.02 0.85 1.01 1.02 3.00 1.10
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)

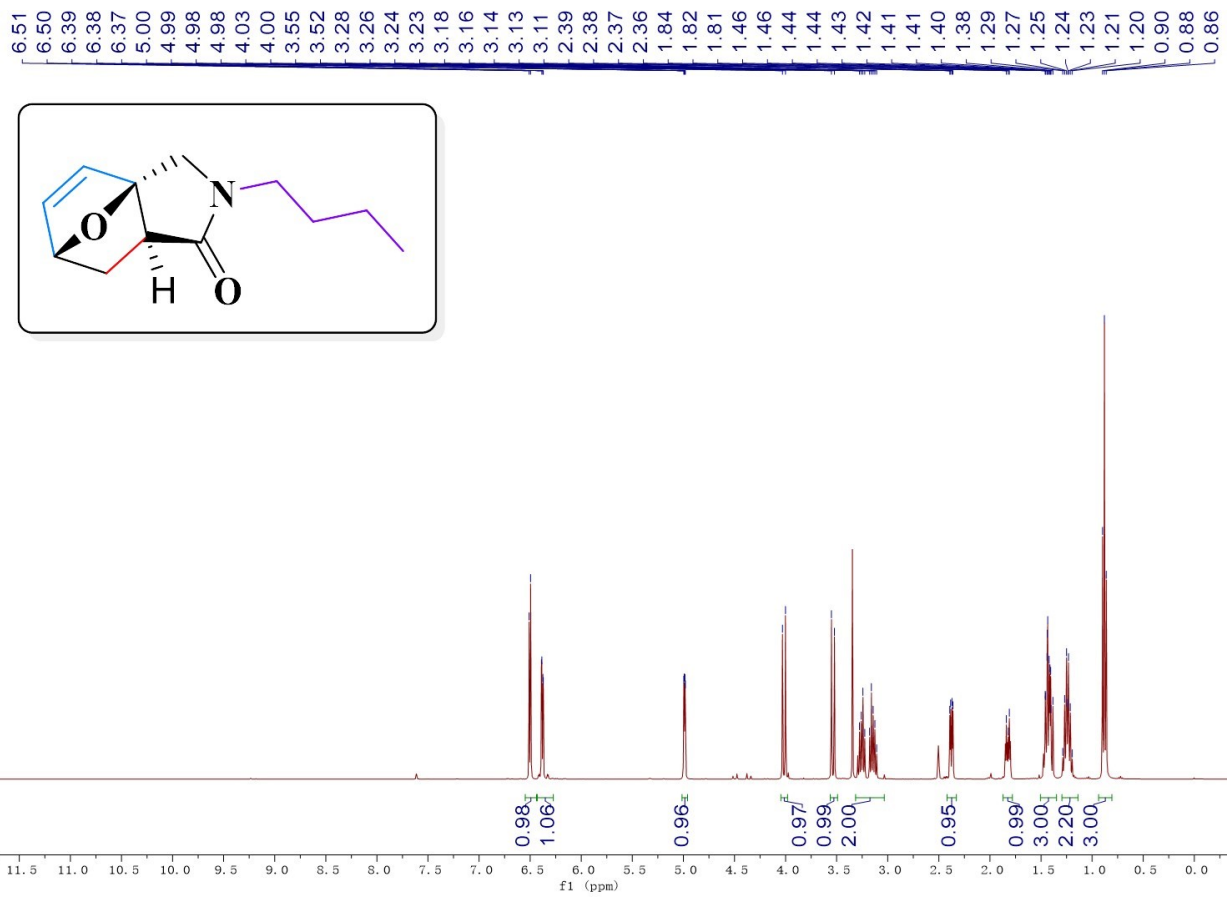

173.48
138.45 136.96 134.24 133.41 132.72 128.65 128.39 127.90 126.71 126.50 126.15 125.69
89.30
78.66
49.27 47.55 44.79
28.33
16.73
CH3
N
O
H O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

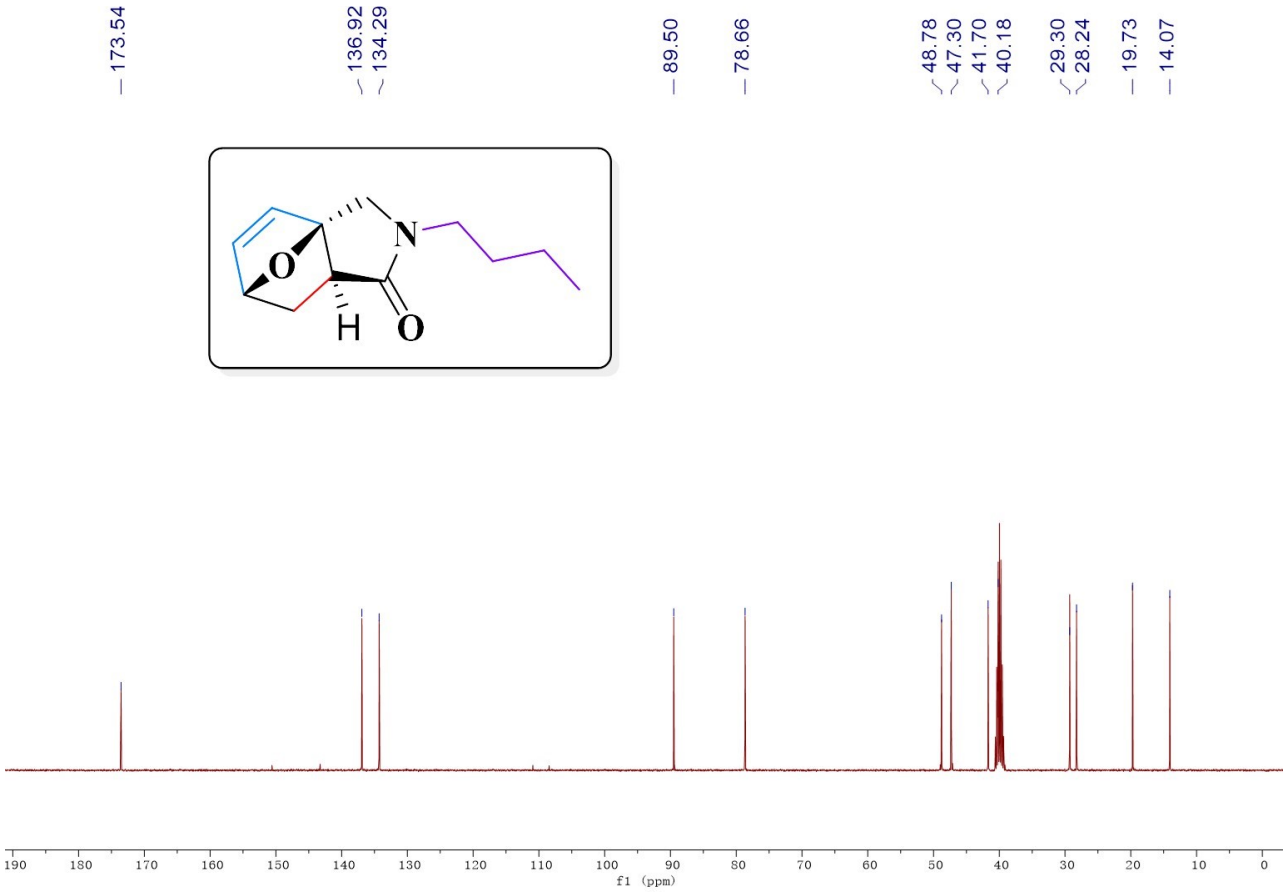

7.29
7.28
7.25
7.24
6.49
6.47
6.38
6.38
6.37
4.99
4.98
4.01
3.98
3.59
3.56
3.55
3.54
3.52
3.37
3.35
3.33
3.32
2.78
2.77
2.75
2.54
2.35
2.34
2.33
1.85
1.82
1.43
1.41
1.40
1.38
O
N
H
O
5.05
2.02
1.01
1.03
2.22
1.08
2.00
0.97
0.99
1.00
f1 (ppm)


173.62
139.45
137.01
134.21
129.10
128.84
126.68
89.50
78.69
49.27
47.29
43.97
33.57
28.20
O
N
H
O
f1 (ppm)

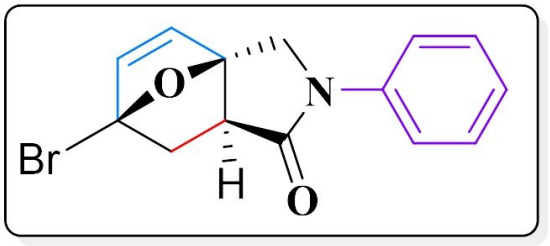

6.49
6.48
6.38
6.38
6.37
6.36
4.97
4.97
4.96
4.01
3.98
3.53
3.50
3.32
3.26
3.24
3.22
3.21
3.15
3.13
3.11
3.09
2.50
2.37
2.36
2.35
2.34
1.84
1.83
1.82
1.81
1.80
1.43
1.43
1.42
1.41
1.40
1.23
0.86
0.85
0.83
N
O
O
H
2.00
0.99
1.19
1.09
2.14
1.04
1.04
2.30
28.30
3.34
f1 (ppm)

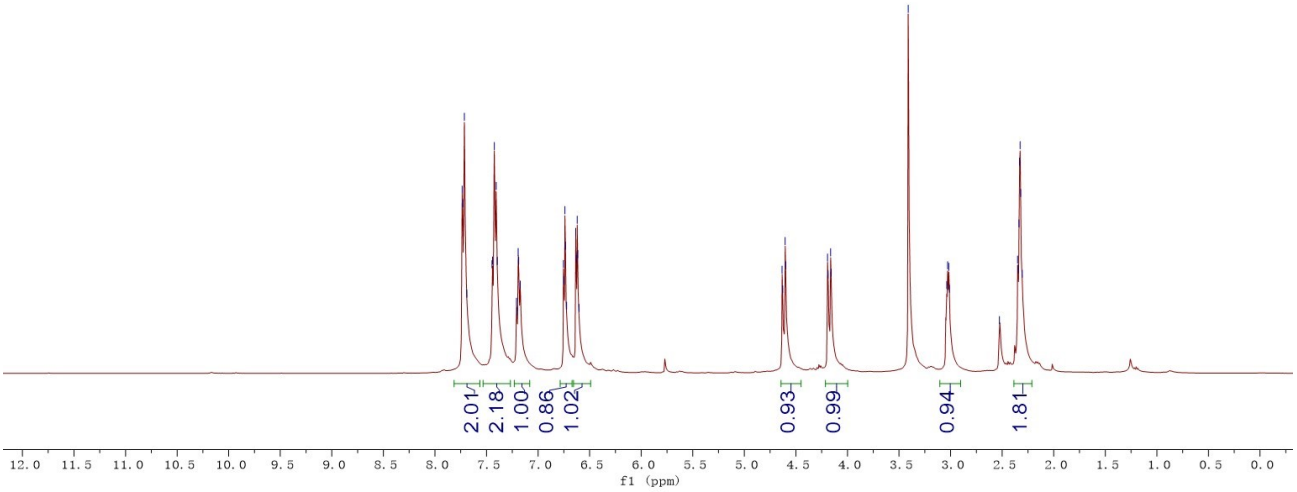

173.38
136.89
134.16
89.42
78.66
60.17
48.79
47.27
42.04
40.86
31.86
29.65
29.60
29.56
29.30
29.25
28.21
27.24
26.60
22.62
21.17
21.13
14.48
14.28
N
O
O
H
f1 (ppm)

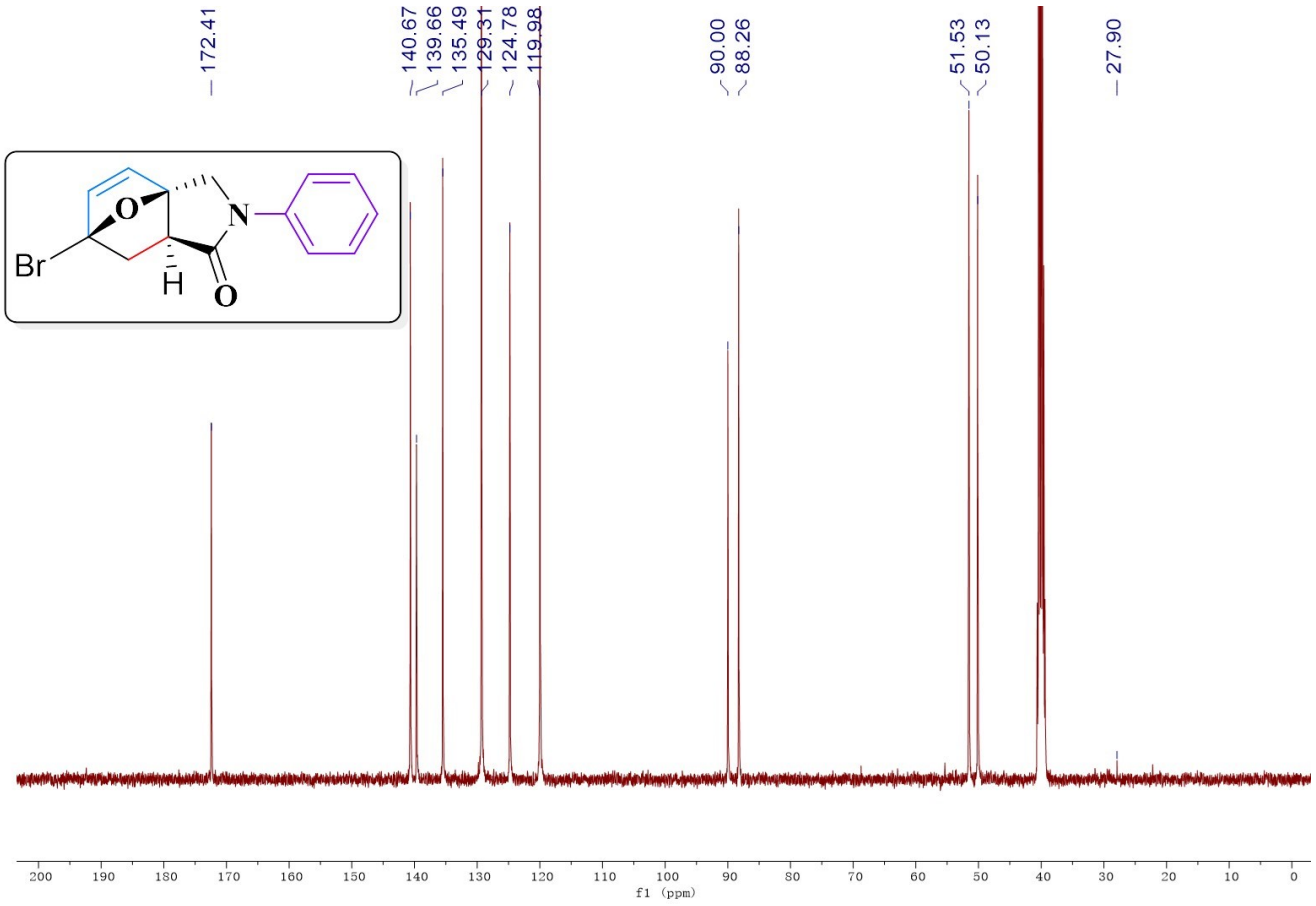

6.51 6.50 6.39 6.38 6.37 5.00 4.99 4.98 4.98 4.03 4.00 3.55 3.52 3.28 3.26 3.24 3.23 3.18 3.16 3.14 3.13 3.11 2.39 2.38 2.37 2.36 1.84 1.82 1.81 1.46 1.46 1.44 1.44 1.43 1.42 1.41 1.41 1.40 1.38 1.29 1.27 1.25 1.24 1.23 1.21 1.20 0.90 0.88 0.86
O
N
H
O
0.98 1.06 0.96 0.97 0.99 2.00 0.95 0.99 3.00 2.20 3.00
12.0 11.5 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 0.0
f1 (ppm)

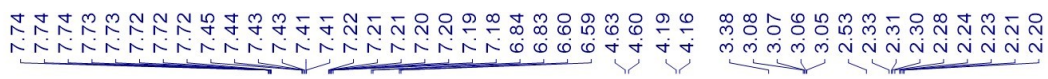

173.54
136.92
134.29
89.50
78.66
48.78
47.30
41.70
40.18
29.30
28.24
19.73
14.07
O
N
H
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

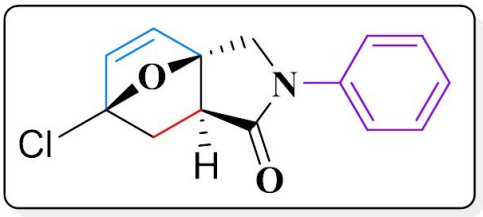

7.74
7.73
7.71
7.69
7.45
7.44
7.42
7.41
7.40
7.21
7.20
7.19
7.19
7.17
6.75
6.75
6.74
6.73
6.72
6.63
6.63
6.62
6.62
6.60
4.63
4.63
4.60
4.60
4.19
4.19
4.16
4.16
3.41
3.04
3.03
3.02
3.01
2.53
2.35
2.34
2.34
2.33
2.33
2.32
2.31

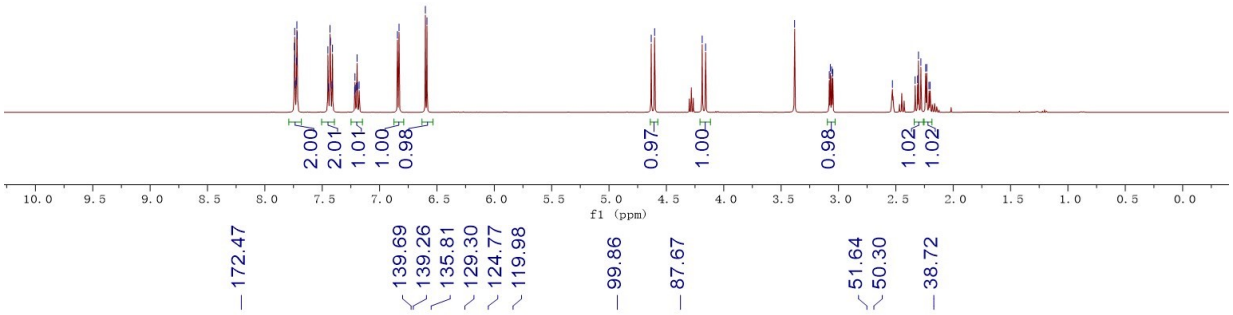

Br
O
N
H
O


2.01
2.18
1.00
0.86
1.02
0.93
0.99
0.94
1.81
12.0 11.5 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 0.0
f1 (ppm)

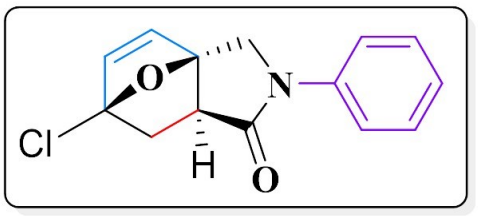

172.41
140.67
139.66
135.49
129.31
124.78
119.98
90.00
88.26
51.53
50.13
27.90
Br
O
N
H
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

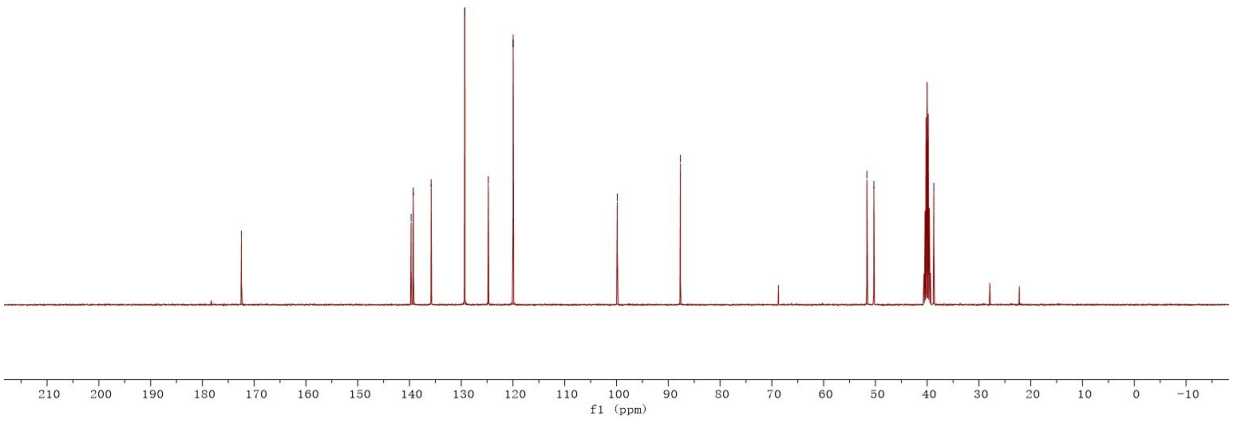

7.74
7.74
7.74
7.73
7.73
7.72
7.72
7.72
7.45
7.44
7.43
7.43
7.41
7.41
7.22
7.21
7.21
7.20
7.20
7.19
7.18
6.84
6.83
6.60
6.59
4.63
4.60
4.19
4.16
3.38
3.08
3.07
3.06
3.05
2.53
2.33
2.31
2.30
2.28
2.24
2.23
2.21
2.20

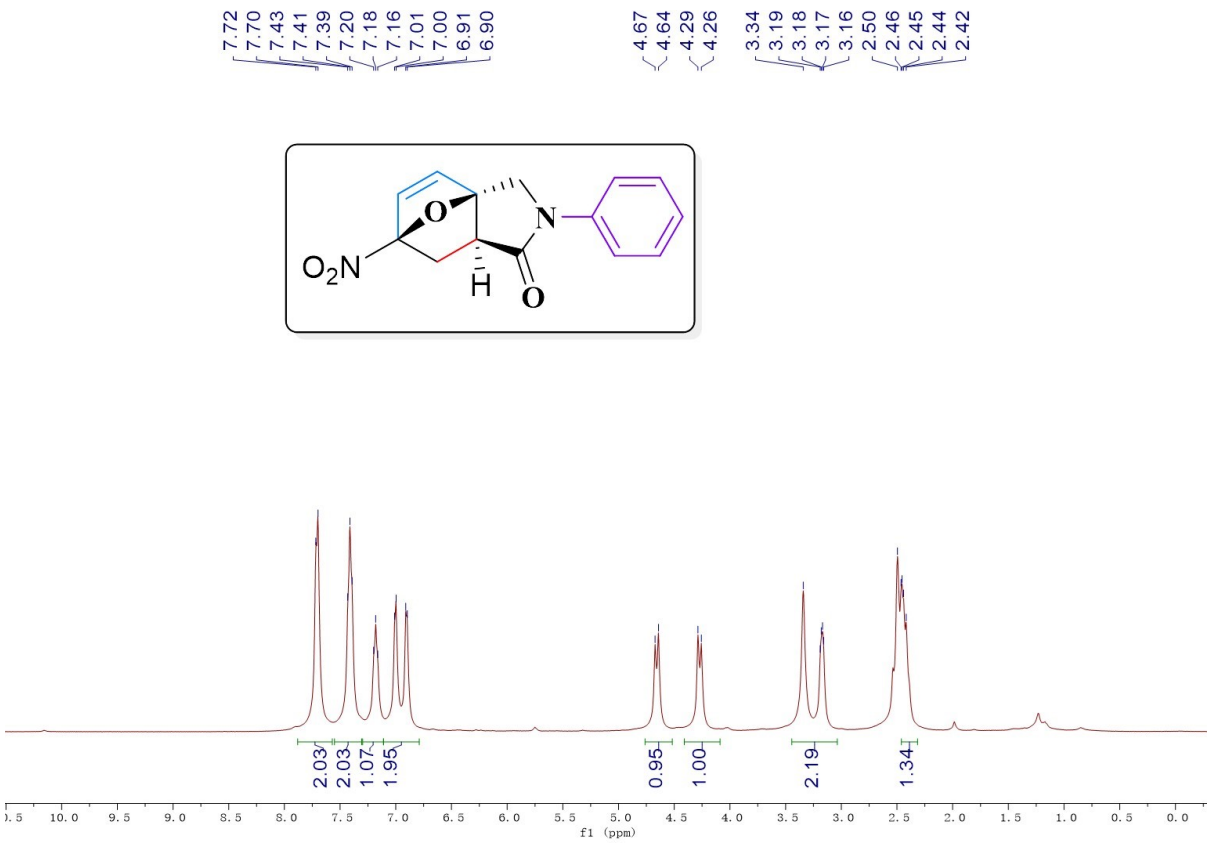

O
N
Cl
H
O

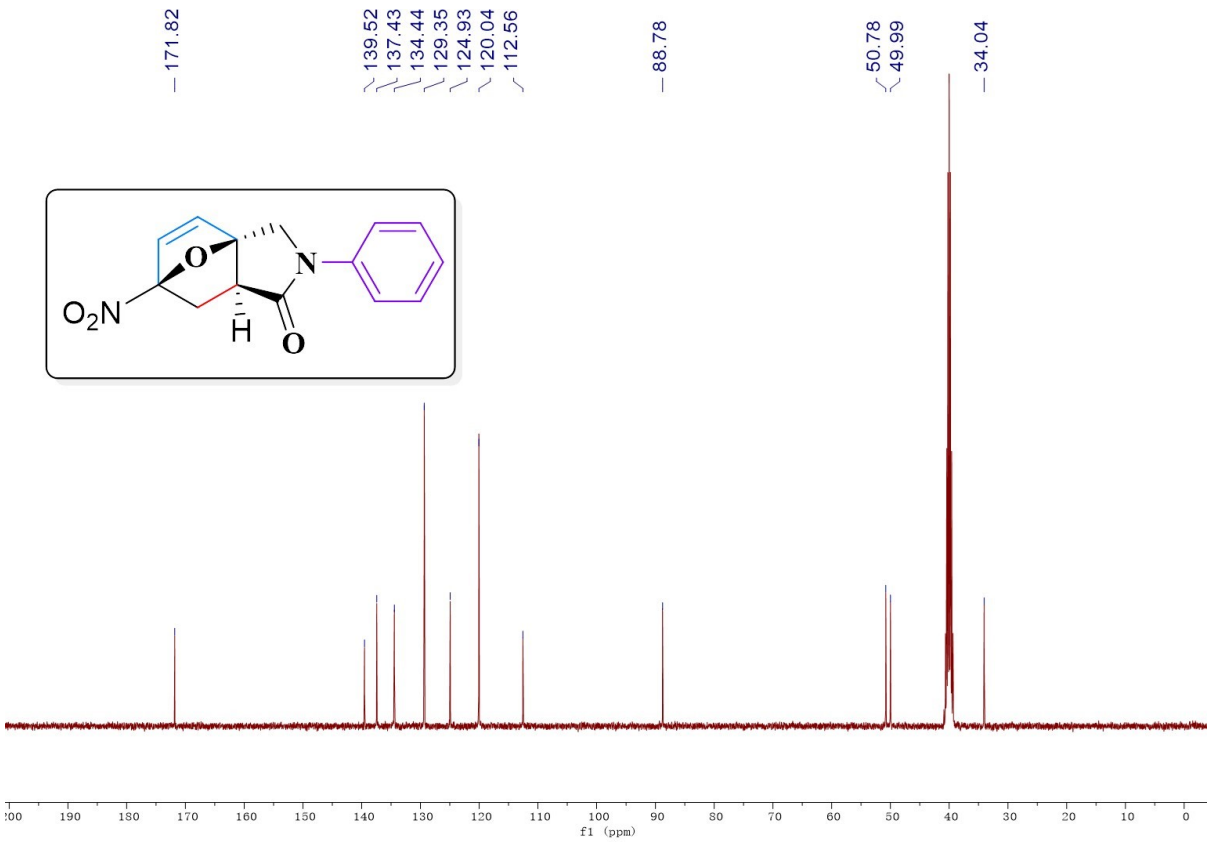

2.00
2.01
1.01
1.00
0.98
0.97
1.00
0.98
1.02
1.02
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

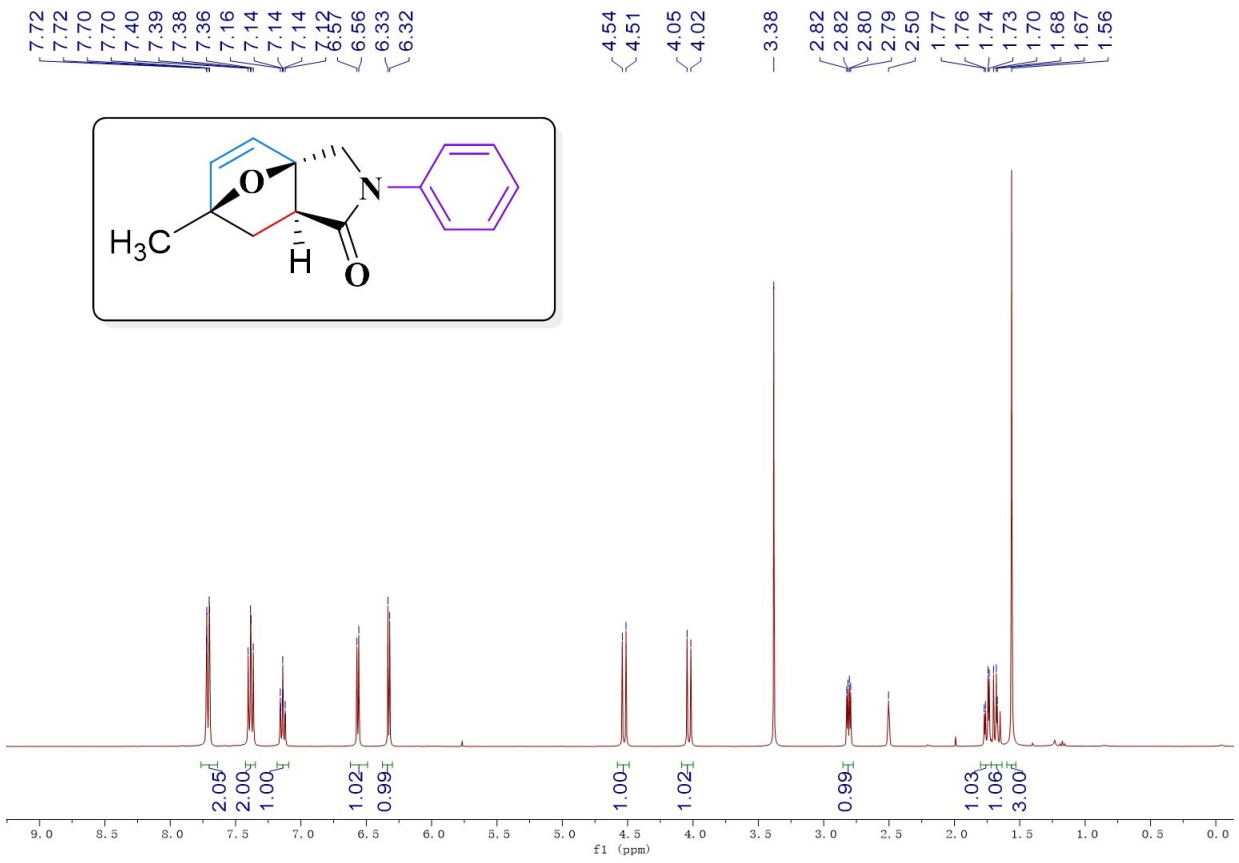
172.47
139.69
139.26
135.81
129.30
124.77
119.98
99.86
87.67
51.64
50.30
38.72

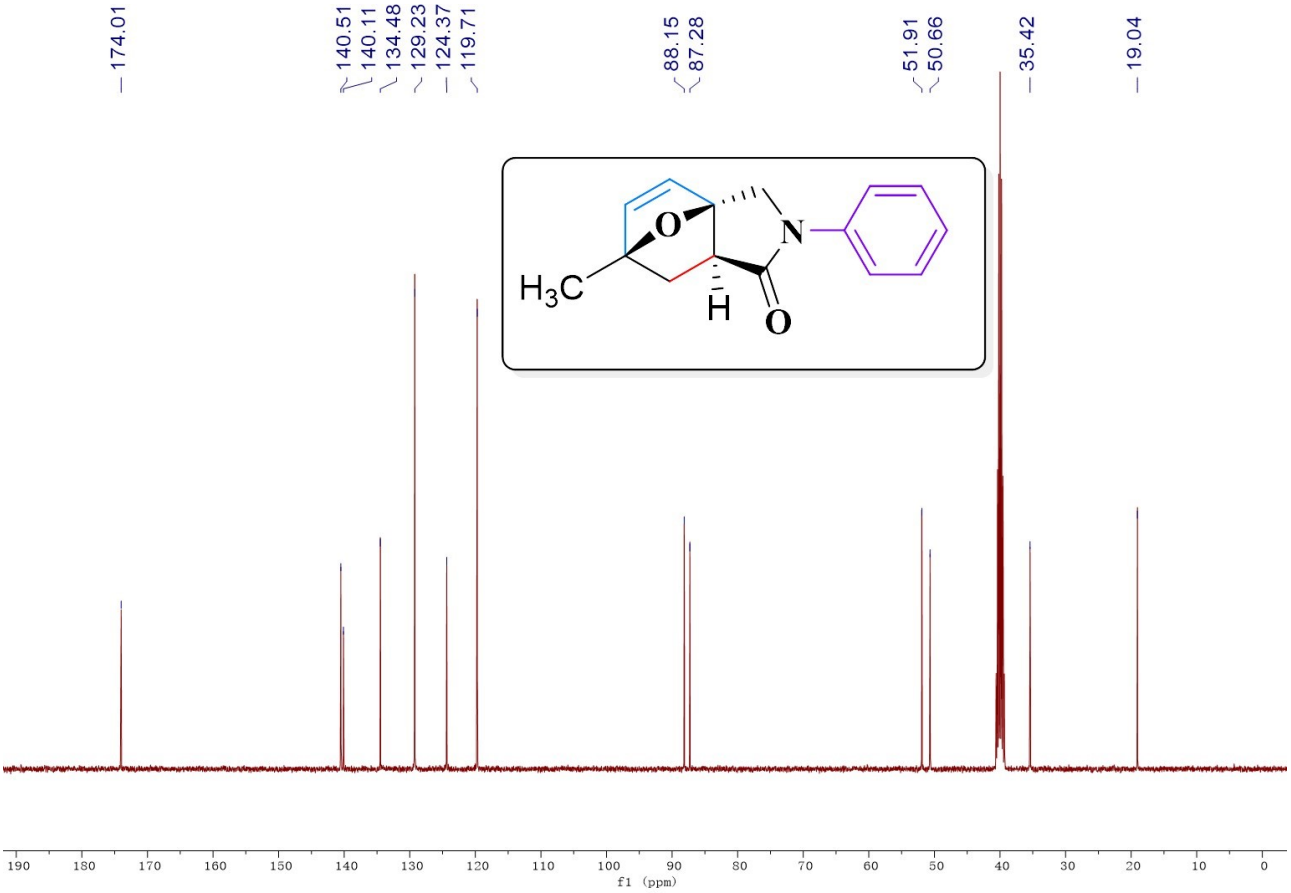

O
N
Cl
H
O

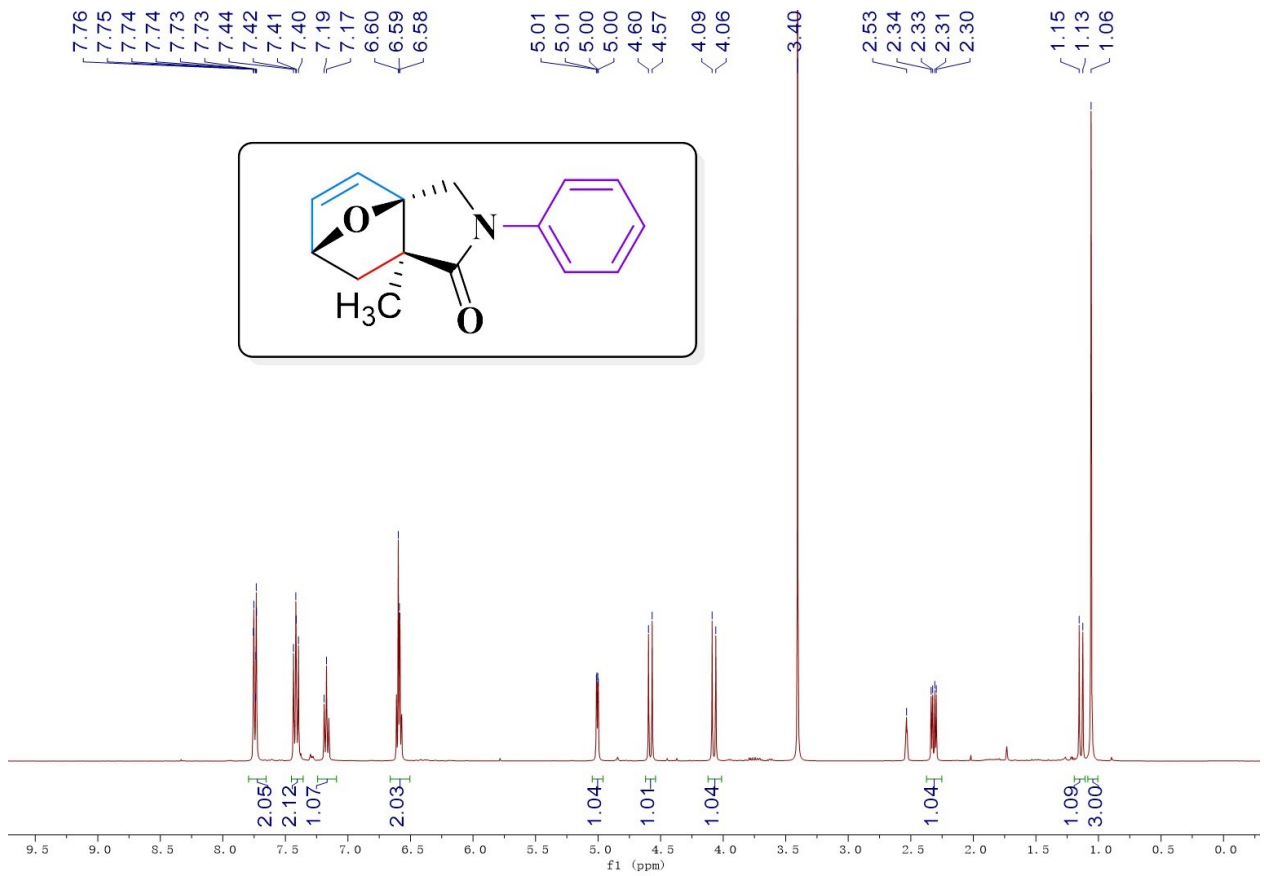

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

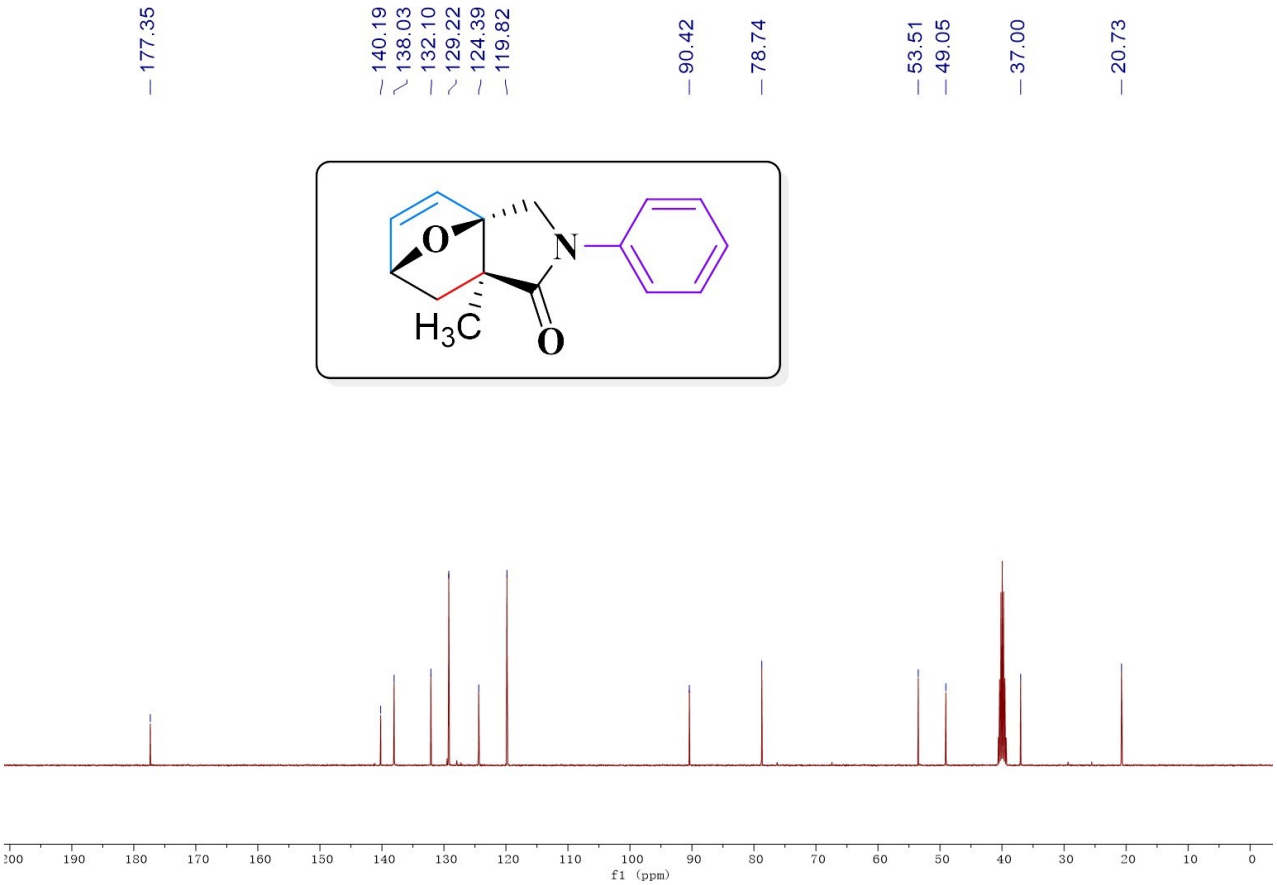

7.72
7.70
7.43
7.41
7.39
7.20
7.18
7.16
7.01
7.00
6.91
6.90
4.67
4.64
4.29
4.26
3.34
3.19
3.18
3.17
3.16
2.50
2.46
2.45
2.44
2.42
O2N
O
N
H
O
2.03
2.03
1.07
1.95
0.95
1.00
2.19
1.34
f1 (ppm)

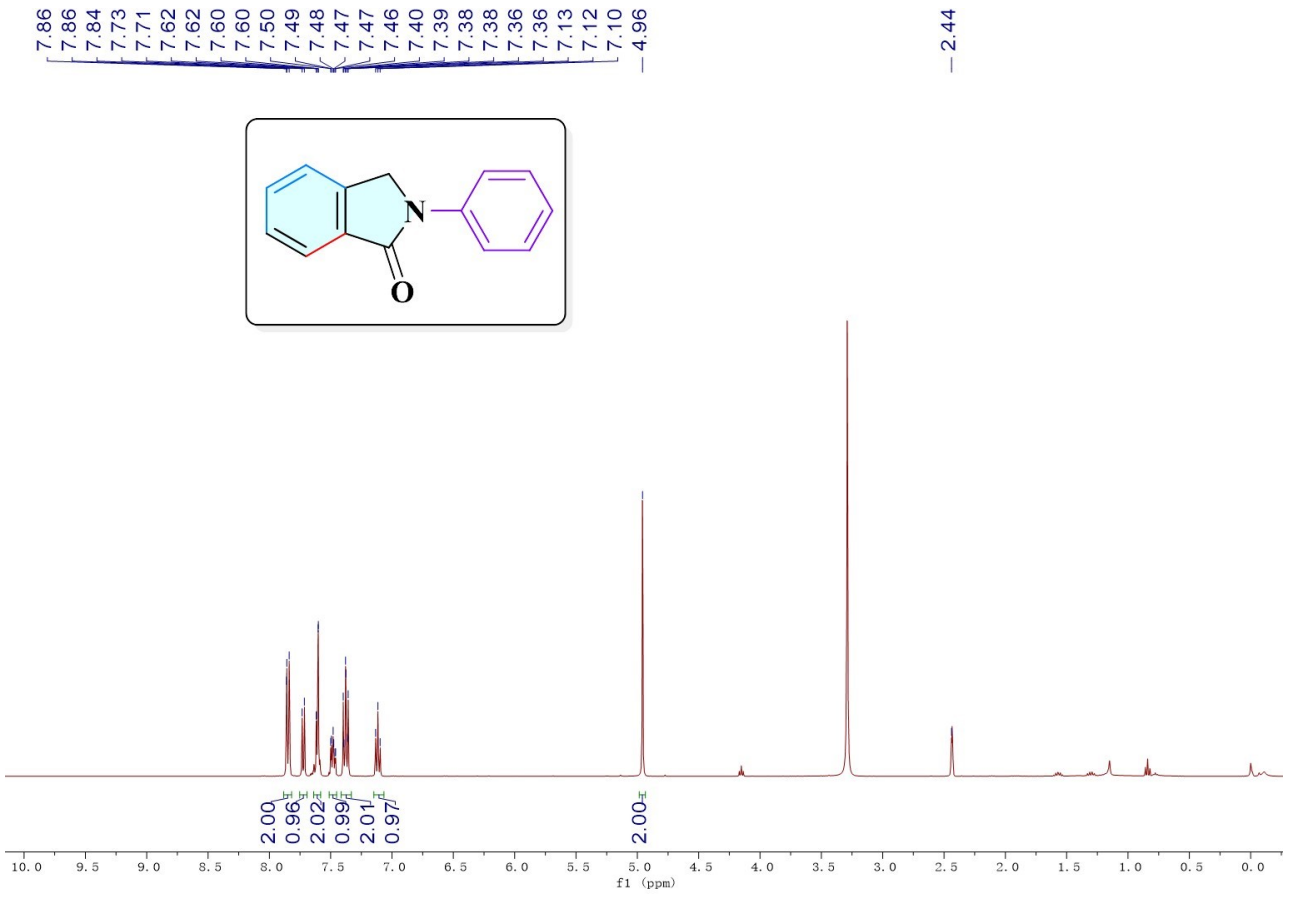

171.82
139.52
137.43
134.44
129.35
124.93
120.04
112.56
88.78
50.78
49.99
34.04
O2N
O
N
H
O
f1 (ppm)

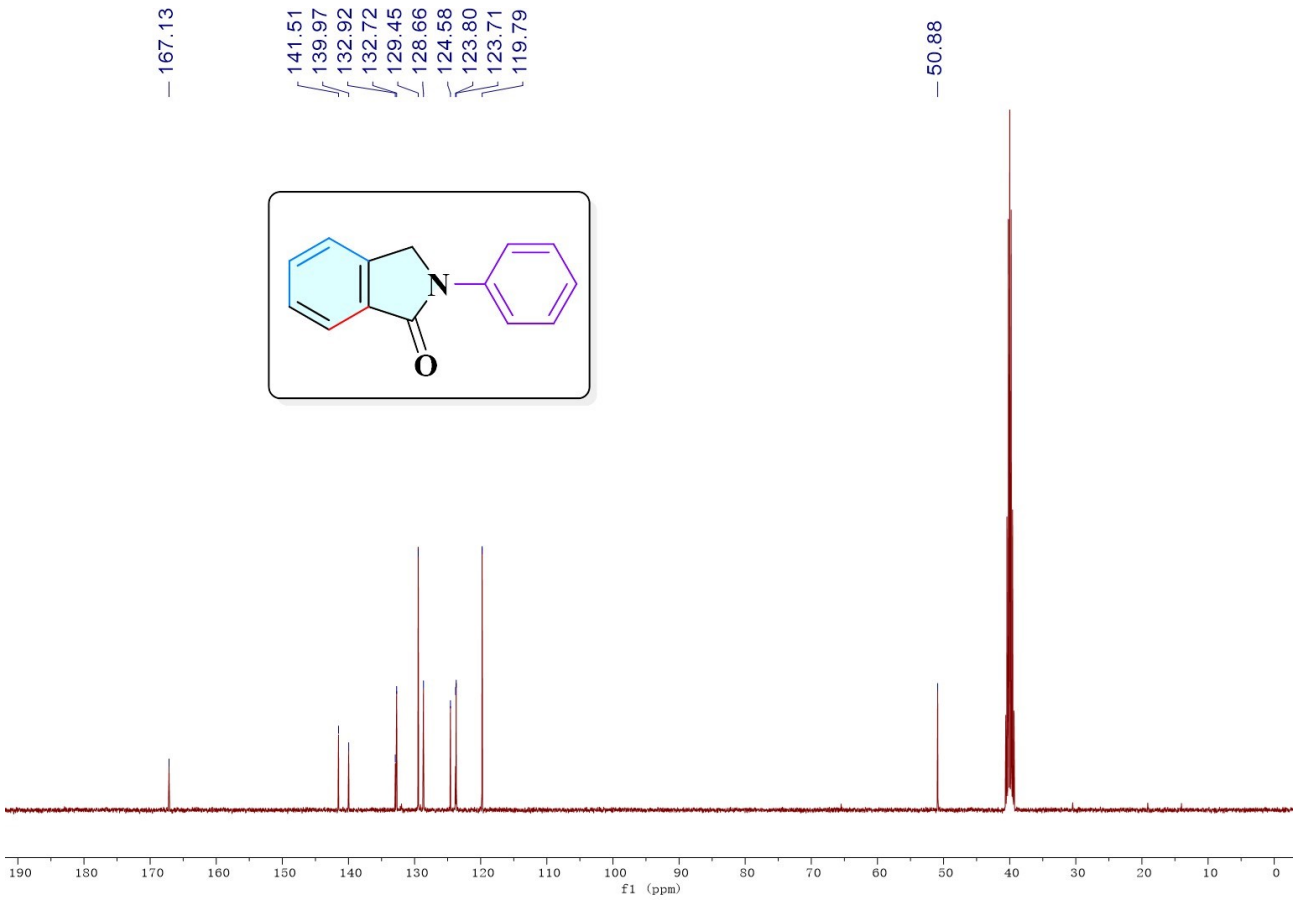

7.72
7.72
7.70
7.70
7.40
7.39
7.38
7.36
7.16
7.14
7.14
7.12
6.57
6.56
6.33
6.32
4.54
4.51
4.05
4.02
3.38
2.82
2.82
2.80
2.79
2.50
1.77
1.76
1.74
1.73
1.70
1.68
1.67
1.56
O
N
H3C
H
O
2.05
2.00
1.00
1.02
0.99
1.00
1.02
0.99
1.03
1.06
3.00
f1 (ppm)

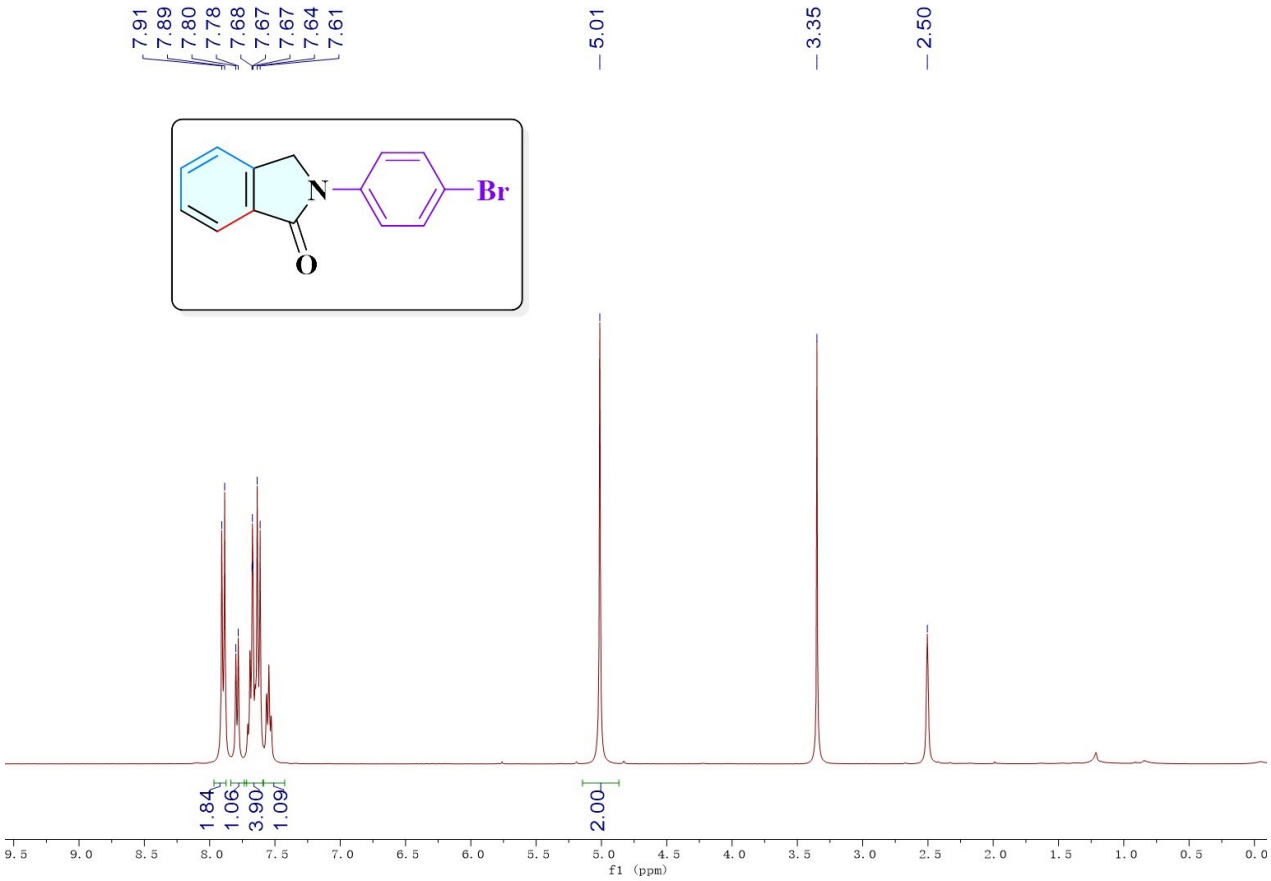

174.01
140.51
140.11
134.48
129.23
124.37
119.71
88.15
87.28
51.91
50.66
35.42
19.04
O
N
H3C
H
O
f1 (ppm)

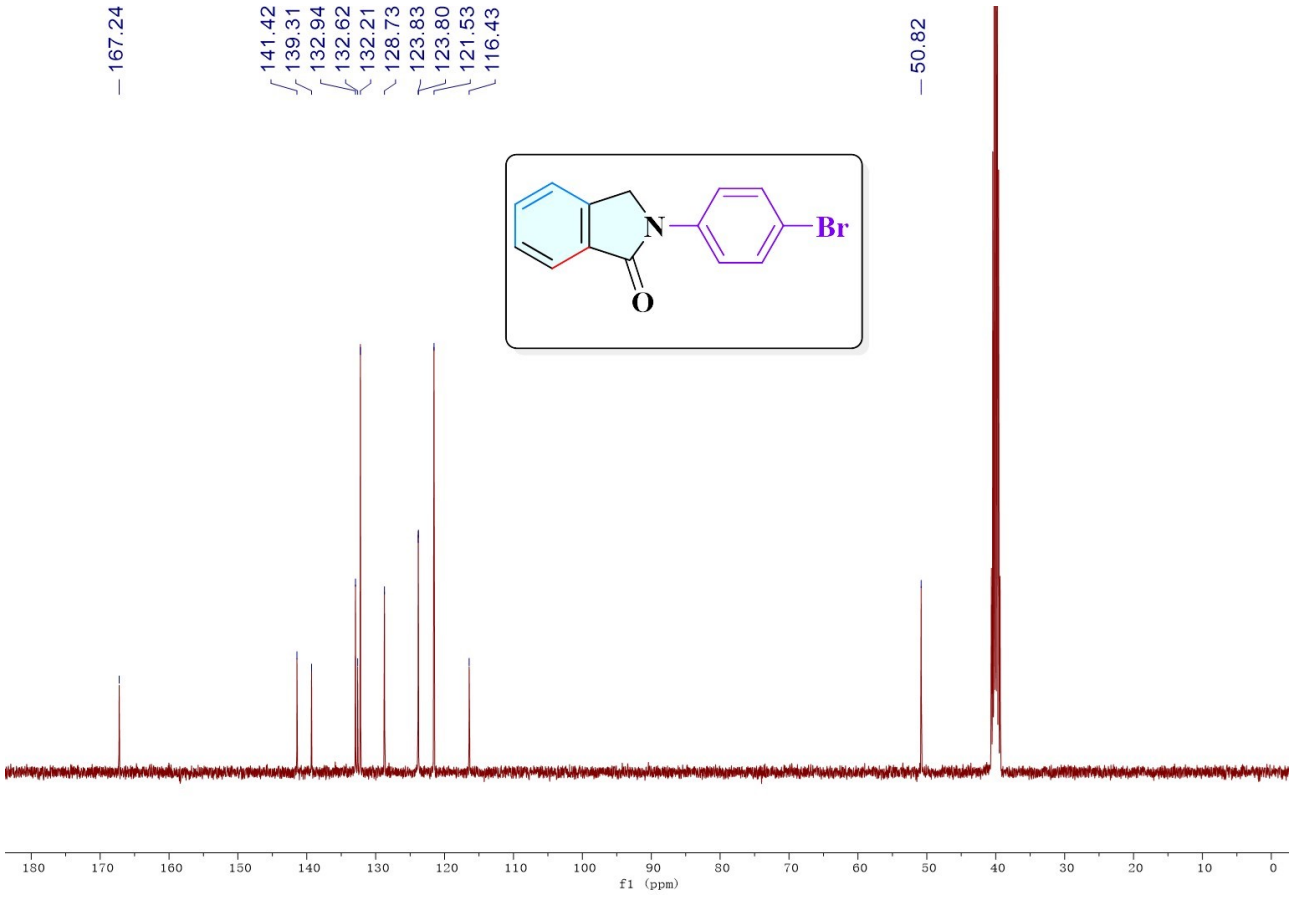

7.76
7.75
7.74
7.74
7.73
7.73
7.44
7.42
7.41
7.40
7.19
7.17
6.60
6.59
6.58
5.01
5.01
5.00
5.00
4.60
4.57
4.09
4.06
3.40
2.53
2.34
2.33
2.31
2.30
1.15
1.13
1.06
O
N
O
H3C
2.05
2.12
1.07
2.03
1.04
1.01
1.04
1.04
1.09
3.00
f1 (ppm)

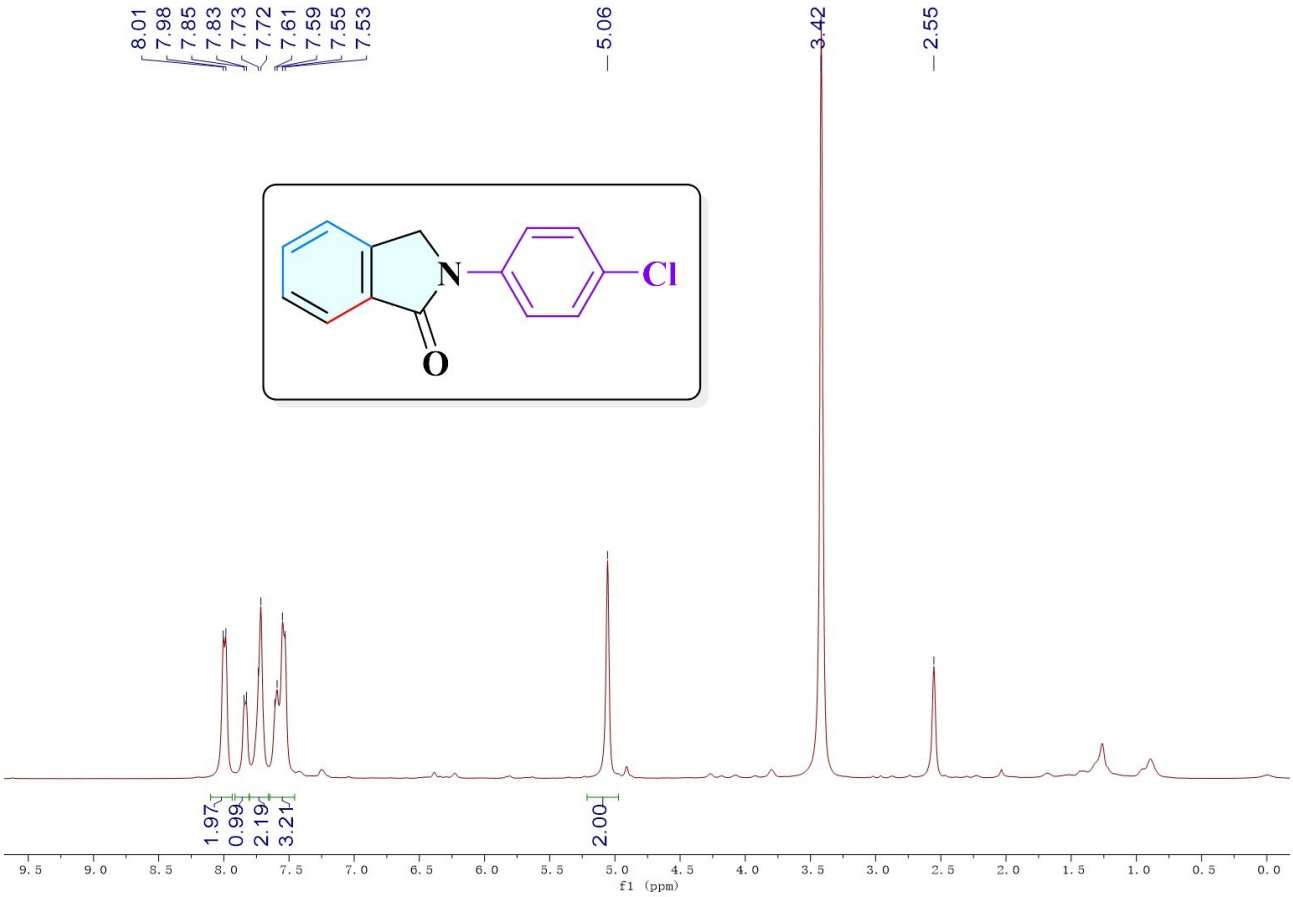

177.35
140.19
138.03
132.10
129.22
124.39
119.82
90.42
78.74
53.51
49.05
37.00
20.73
O
N
O
H3C
f1 (ppm)

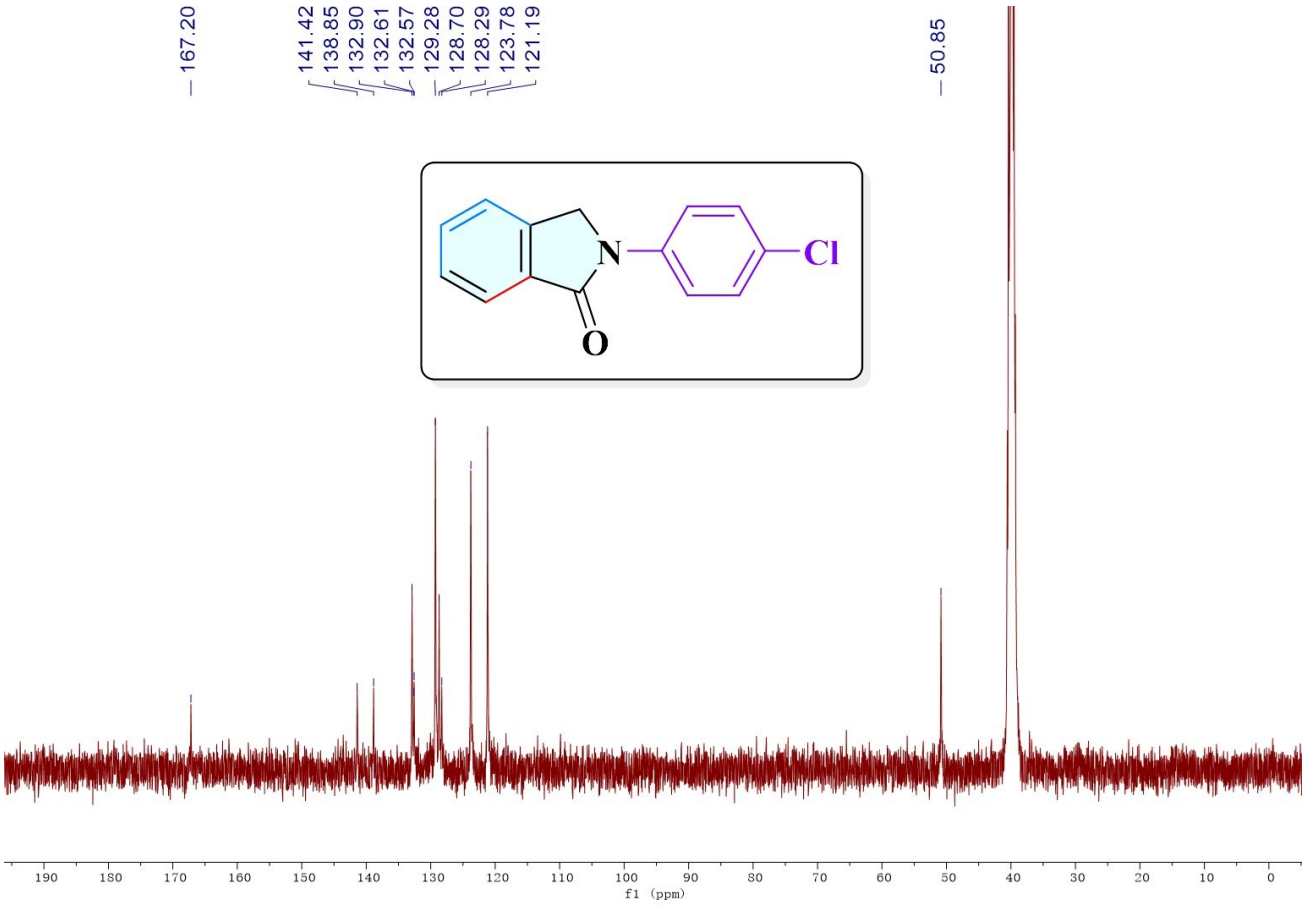

7.86
7.86
7.84
7.73
7.71
7.62
7.62
7.60
7.60
7.50
7.49
7.48
7.47
7.47
7.46
7.40
7.39
7.38
7.38
7.36
7.36
7.13
7.12
7.10
4.96
2.44
N
O
2.00
0.96
2.02
0.99
2.01
0.97
2.00
f1 (ppm)

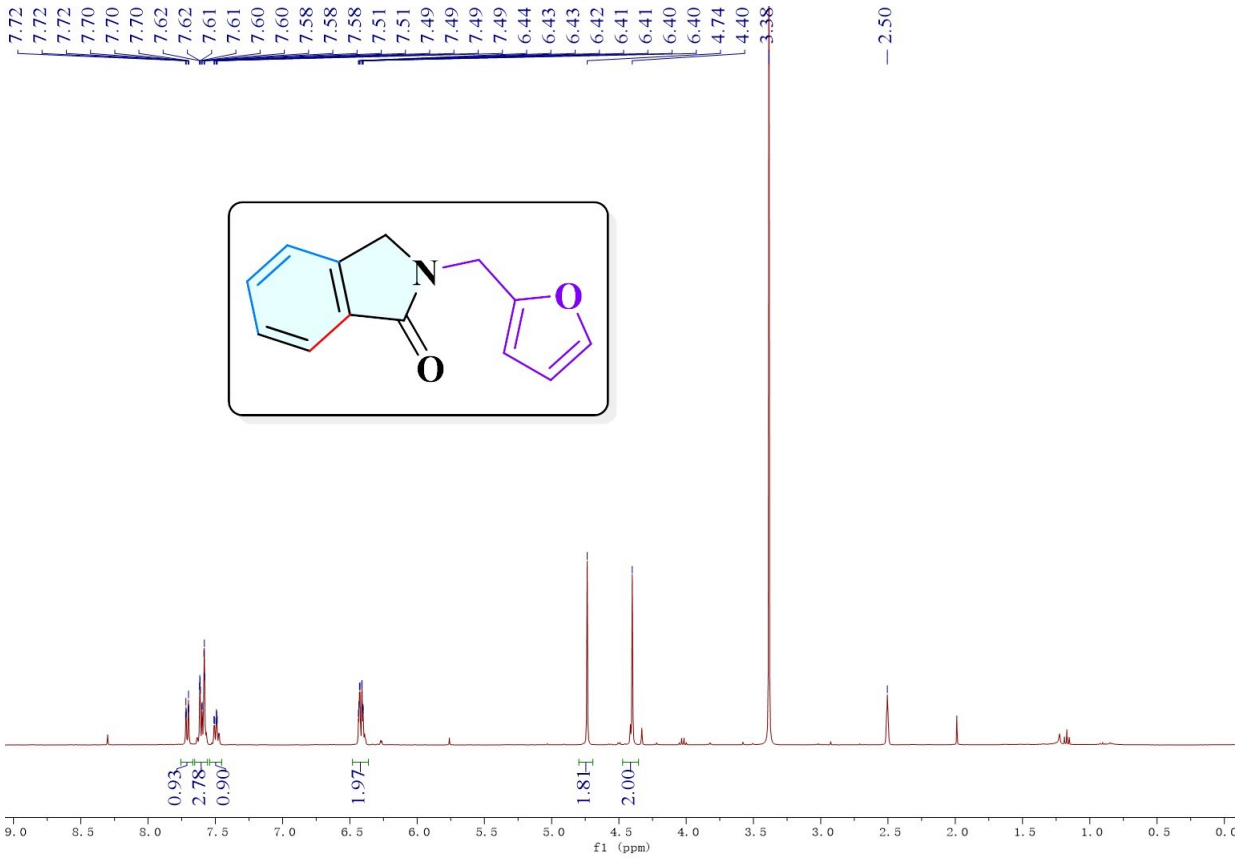

167.13
141.51
139.97
132.92
132.72
129.45
128.66
124.58
123.80
123.71
119.79
50.88
N
O
f1 (ppm)

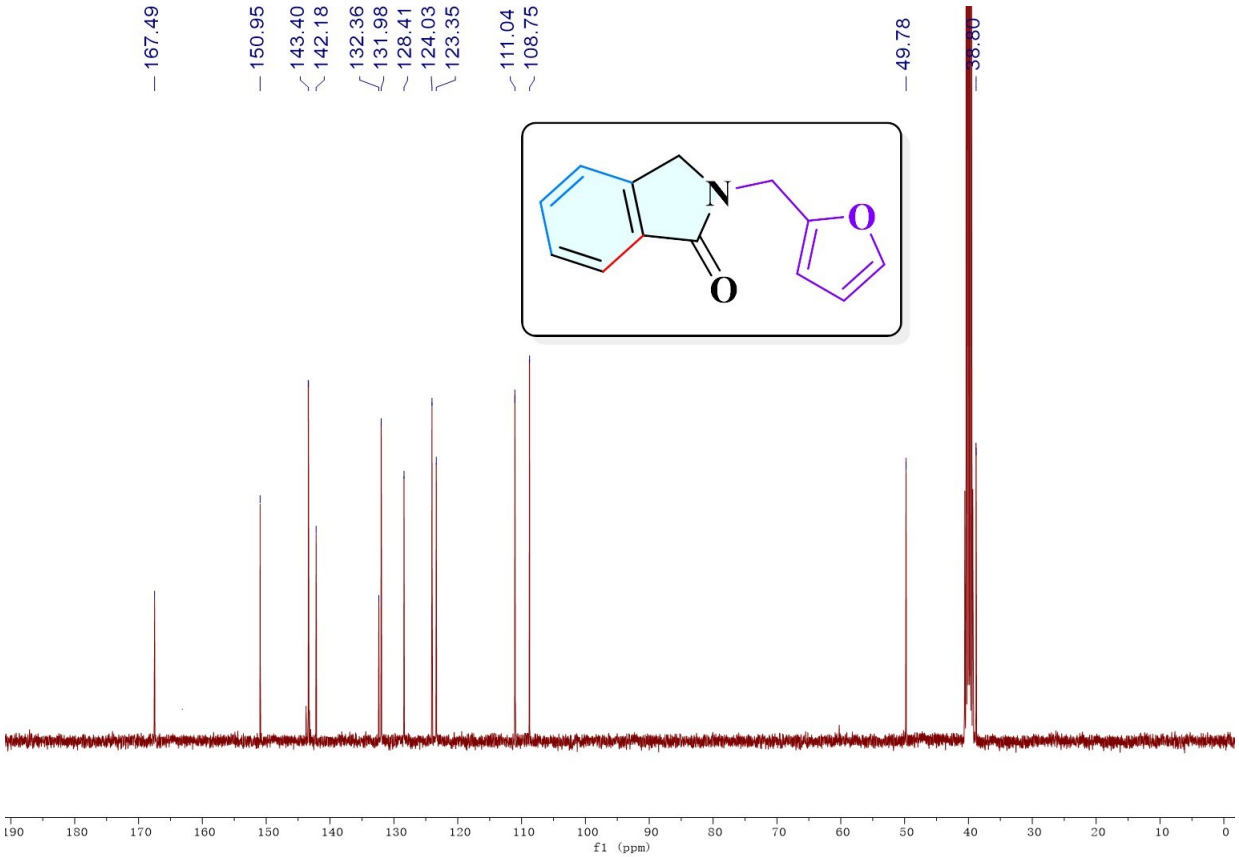

7.91
7.89
7.80
7.78
7.68
7.67
7.67
7.64
7.61
5.01
3.35
2.50
Br
N
O
1.84
1.06
3.90
1.09
2.00
f1 (ppm)

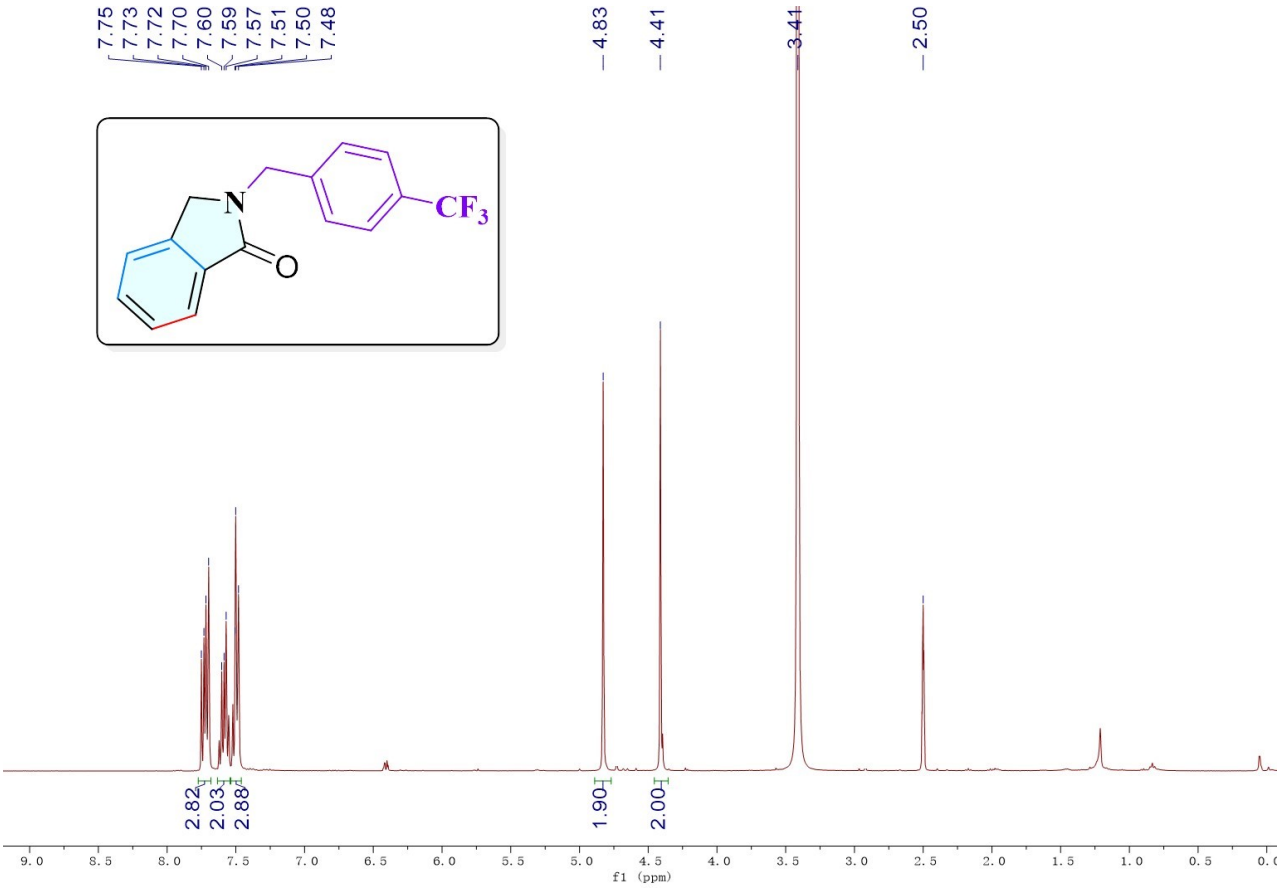

167.24
141.42
139.31
132.94
132.62
132.21
128.73
123.83
123.80
121.53
116.43
50.82
Br
N
O
f1 (ppm)

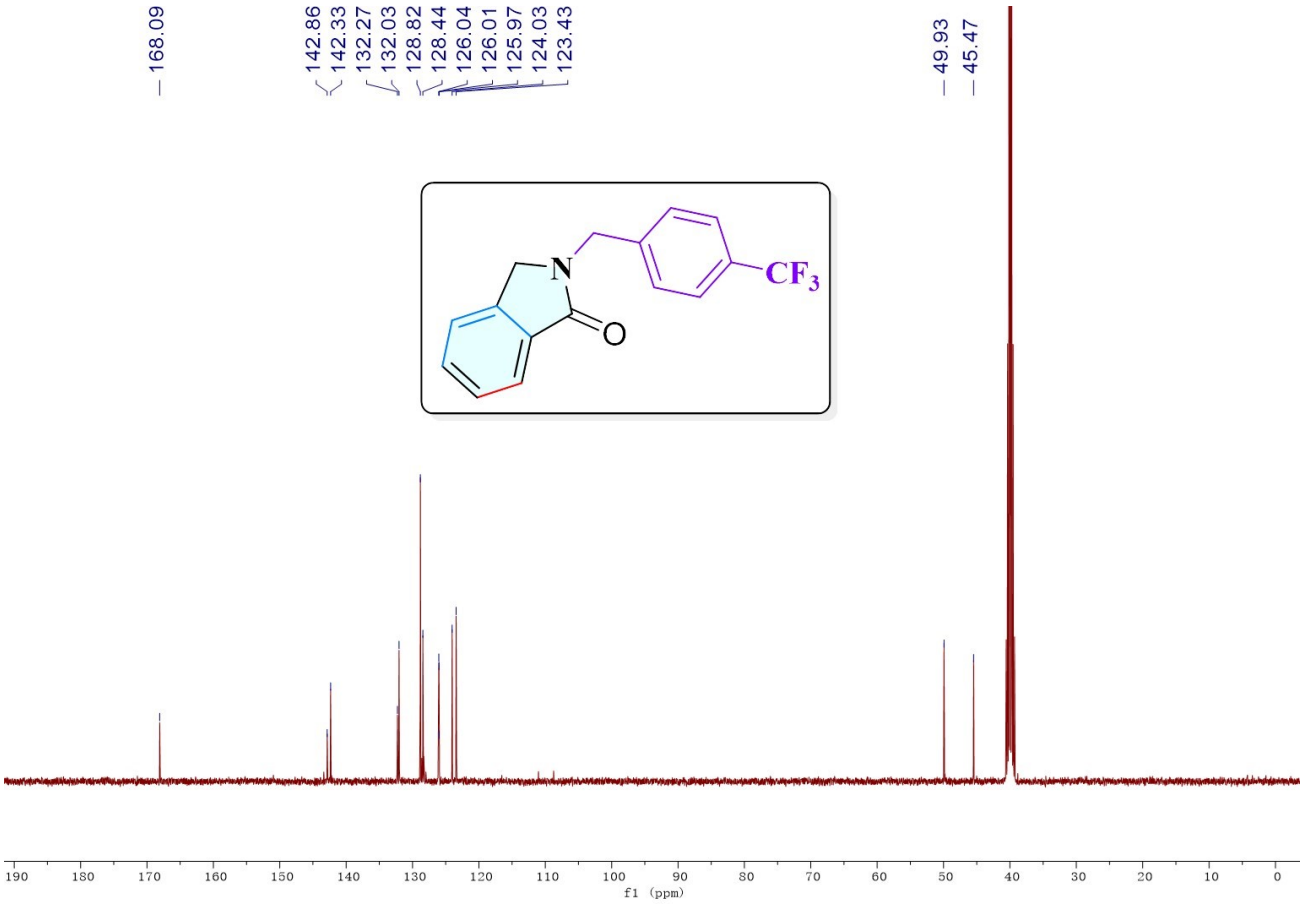

8.01
7.98
7.85
7.83
7.73
7.72
7.61
7.59
7.55
7.53
5.06
3.42
2.55
N
Cl
O
1.97
0.99
2.19
3.21
2.00
f1 (ppm)

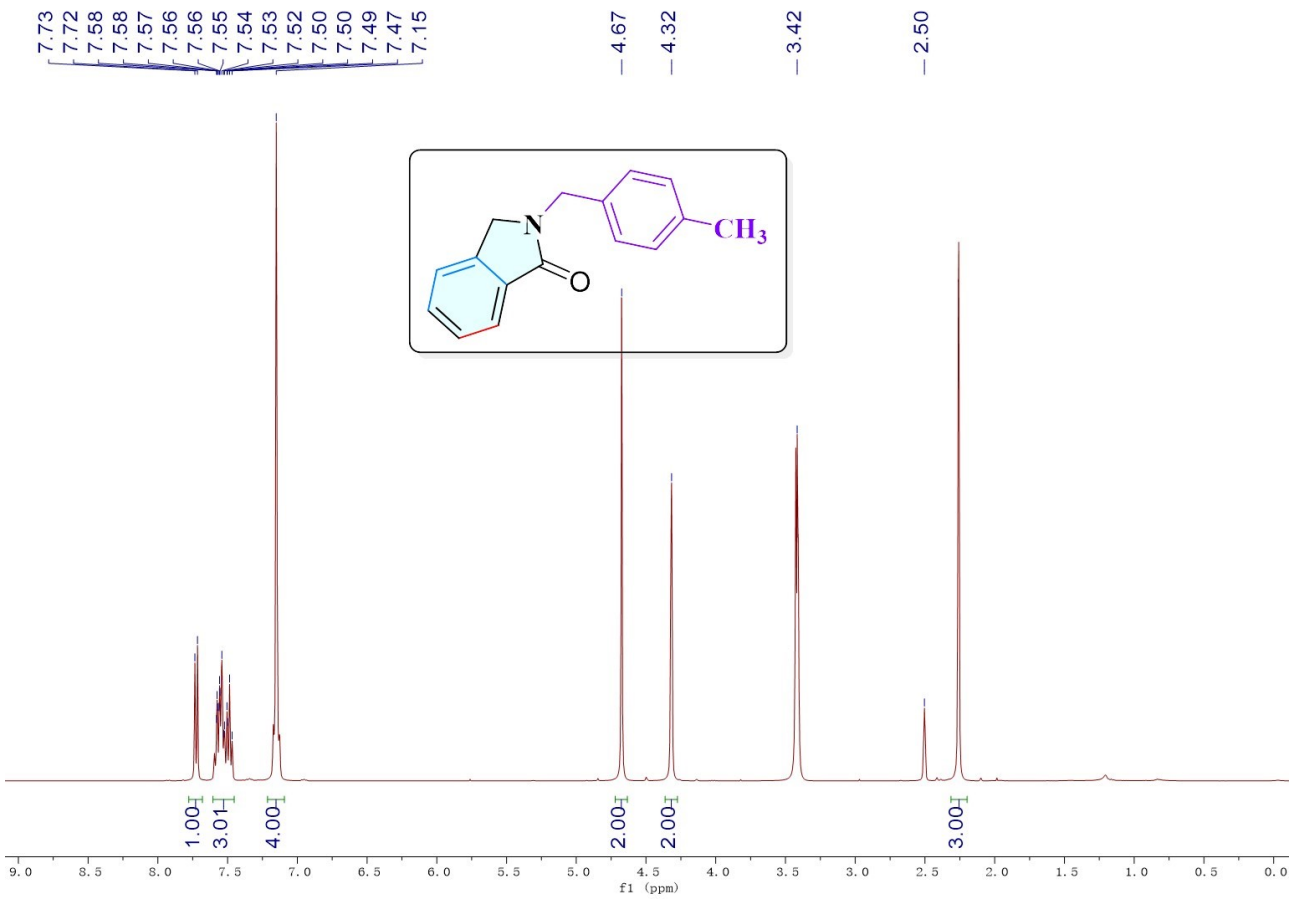

167.20
141.42
138.85
132.90
132.61
132.57
129.28
128.70
128.29
123.78
121.19
50.85
N
Cl
O
f1 (ppm)

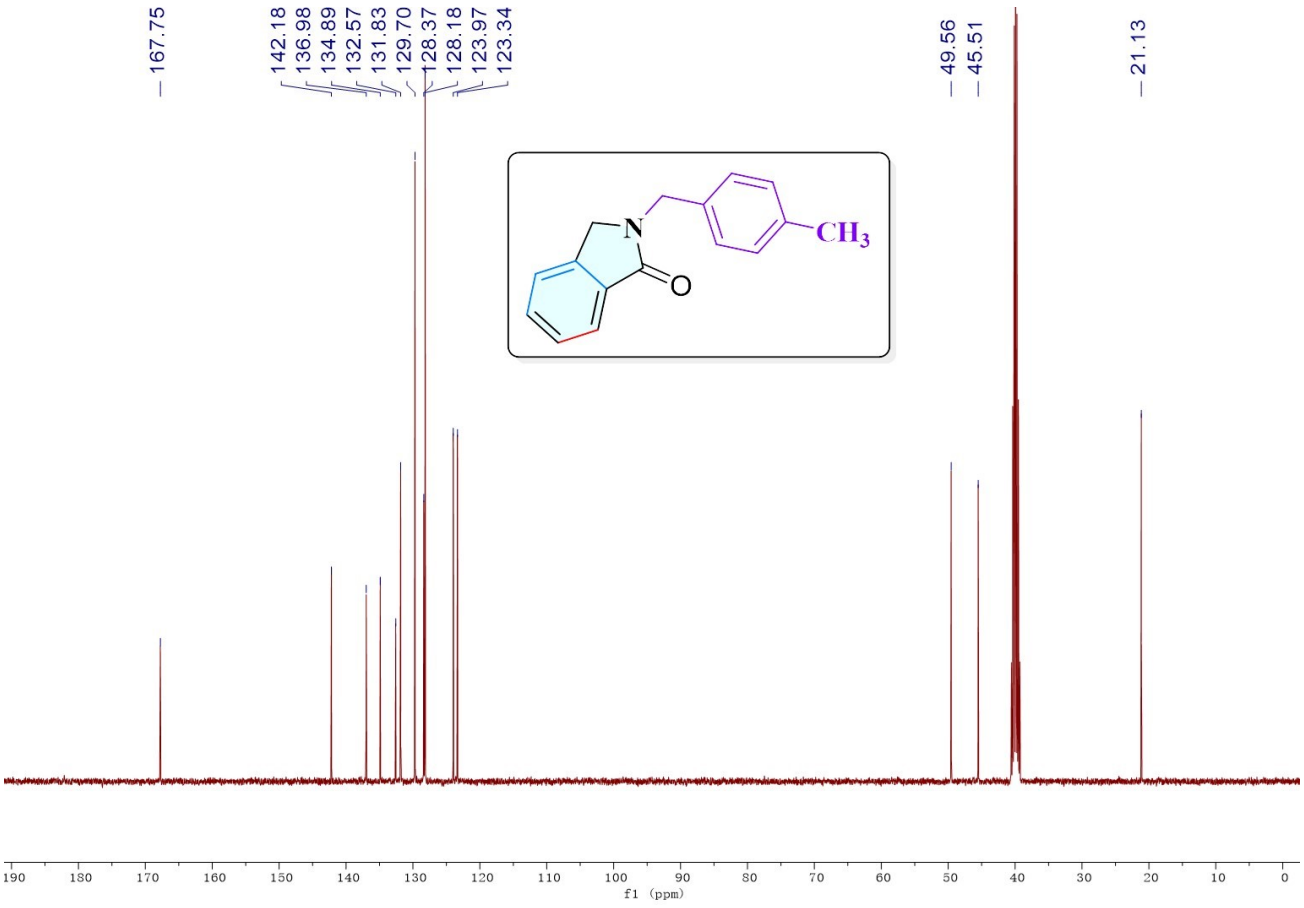

7.72
7.72
7.72
7.70
7.70
7.70
7.62
7.62
7.61
7.61
7.60
7.60
7.58
7.58
7.58
7.51
7.51
7.49
7.49
7.49
7.49
6.44
6.43
6.43
6.42
6.41
6.41
6.40
6.40
4.74
4.40
3.38
2.50
N
O
O
0.93
2.78
0.90
1.97
1.81
2.00
f1 (ppm)

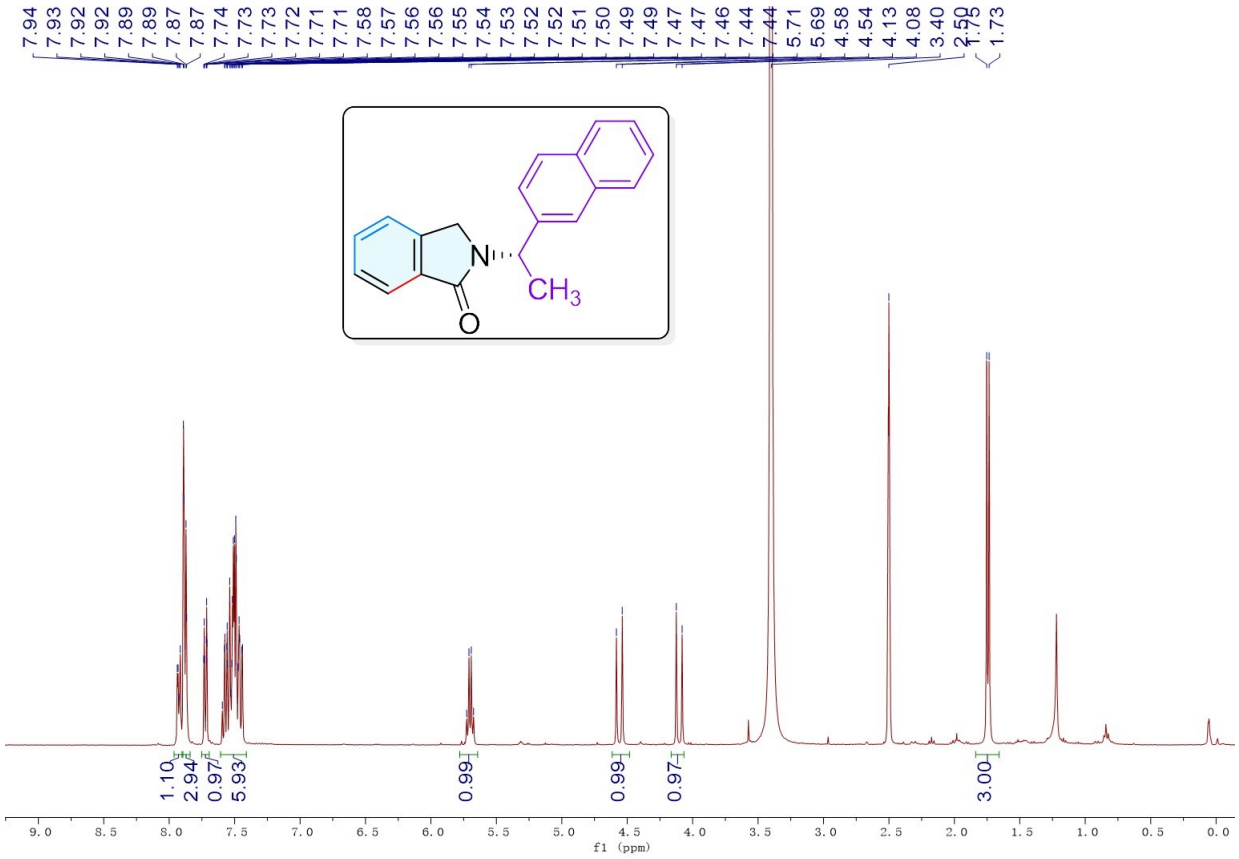

167.49
150.95
143.40
142.18
132.36
131.98
128.41
124.03
123.35
111.04
108.75
49.78
38.80
N
O
O
f1 (ppm)

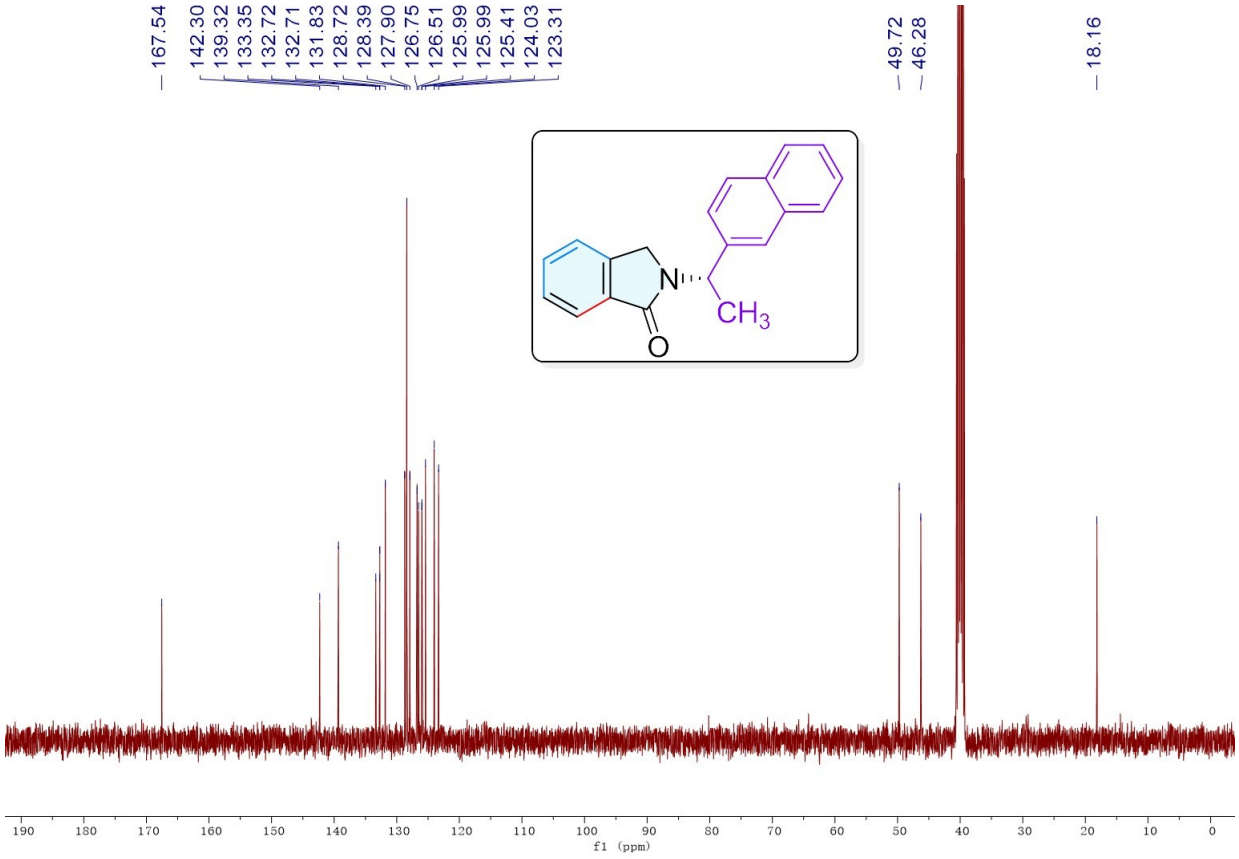

7.75
7.73
7.72
7.70
7.60
7.59
7.57
7.51
7.50
7.48
4.83
4.41
3.41
2.50
N
O
CF3
2.82
2.03
2.88
1.90
2.00
f1 (ppm)

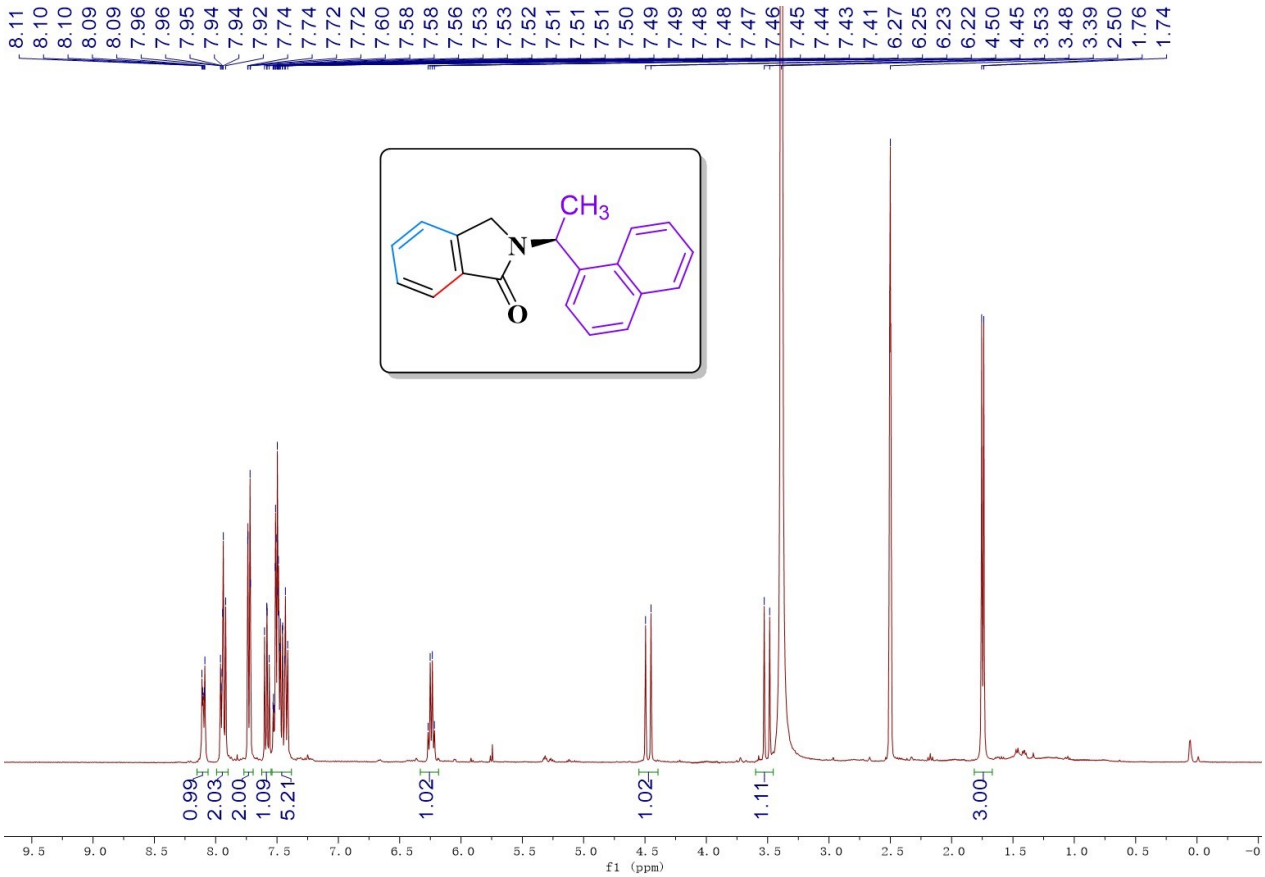

168.09
142.86
142.33
132.27
132.03
128.82
128.44
126.04
126.01
125.97
124.03
123.43
49.93
45.47
N
O
CF3
f1 (ppm)

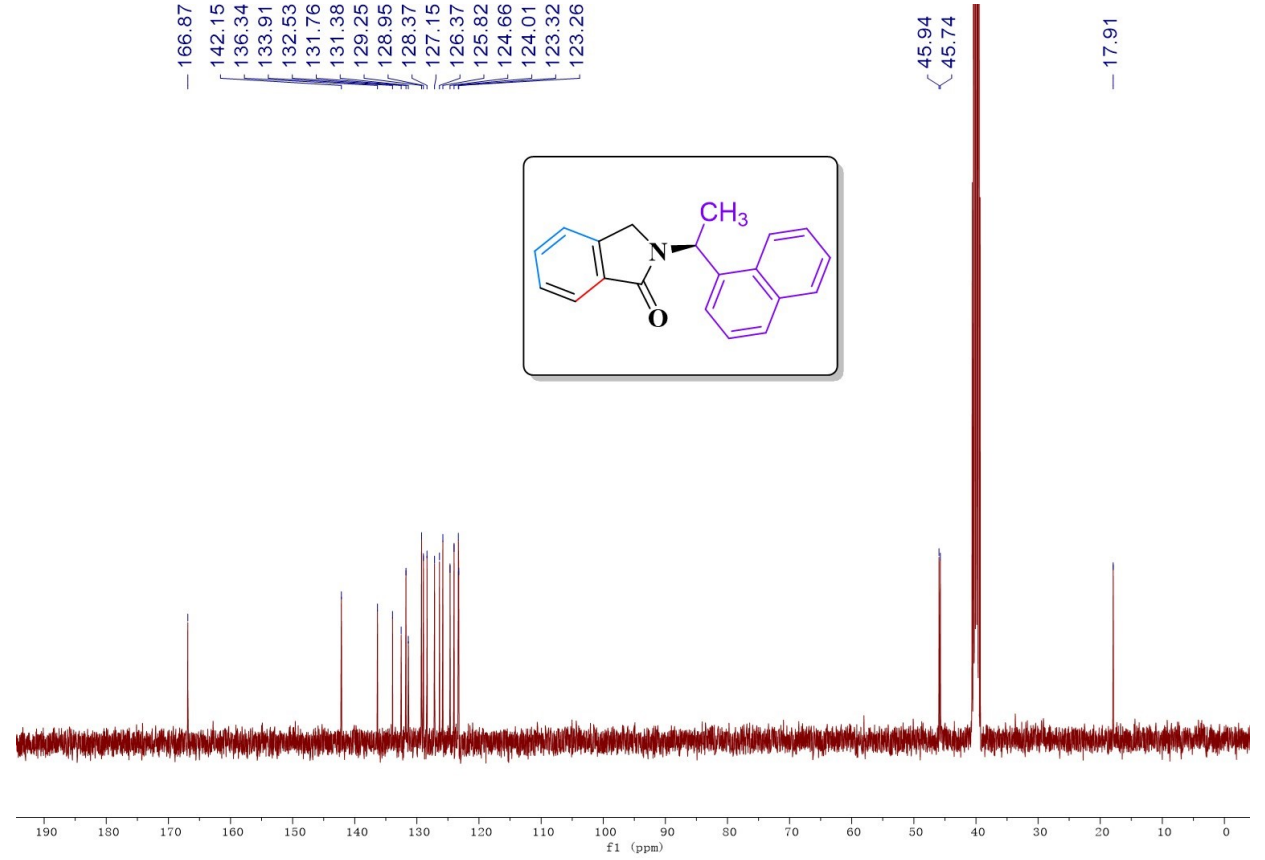

7.73
7.72
7.58
7.58
7.57
7.56
7.56
7.55
7.54
7.53
7.52
7.50
7.50
7.49
7.47
7.15
4.67
4.32
3.42
2.50
CH3
N
O
1.00
3.01
4.00
2.00
2.00
3.00
f1 (ppm)

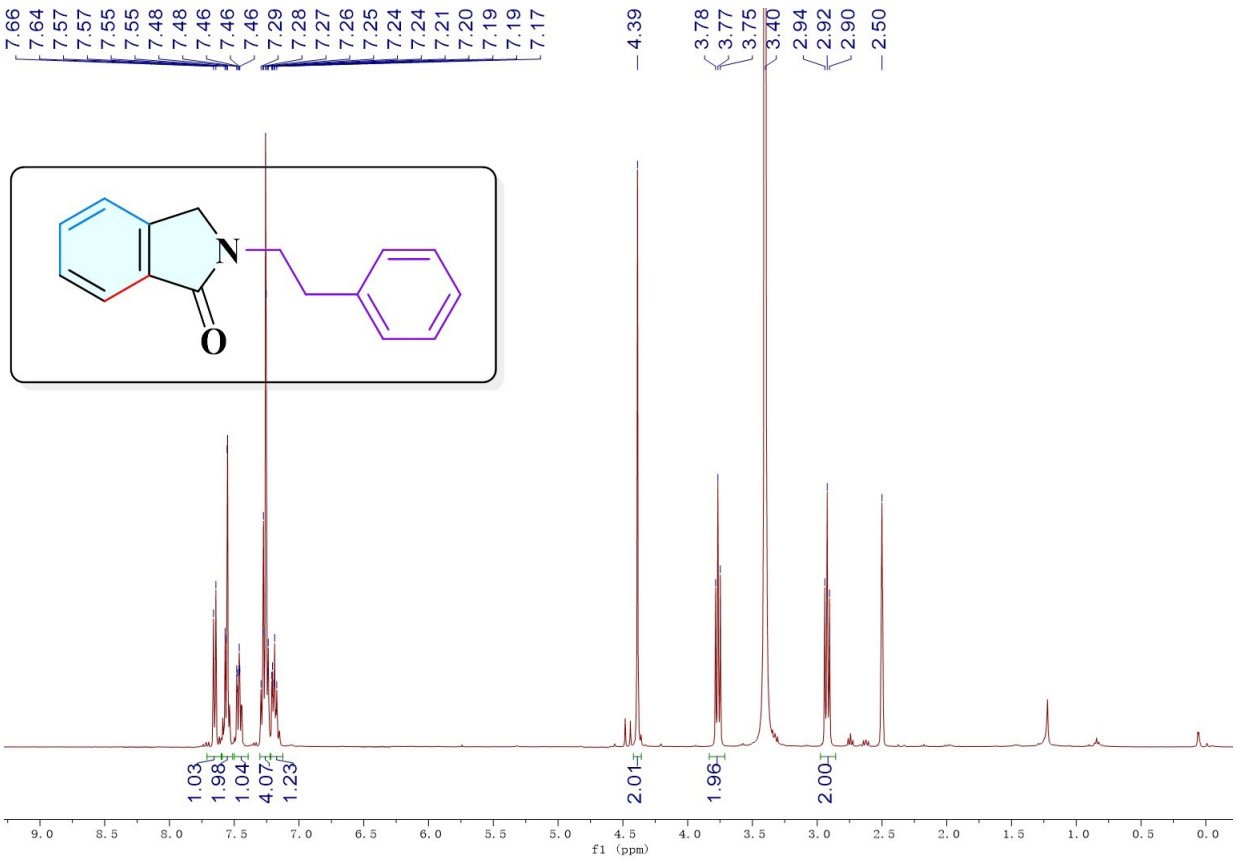

167.75
142.18
136.98
134.89
132.57
131.83
129.70
128.37
128.18
123.97
123.34
49.56
45.51
21.13
CH3
N
O
f1 (ppm)

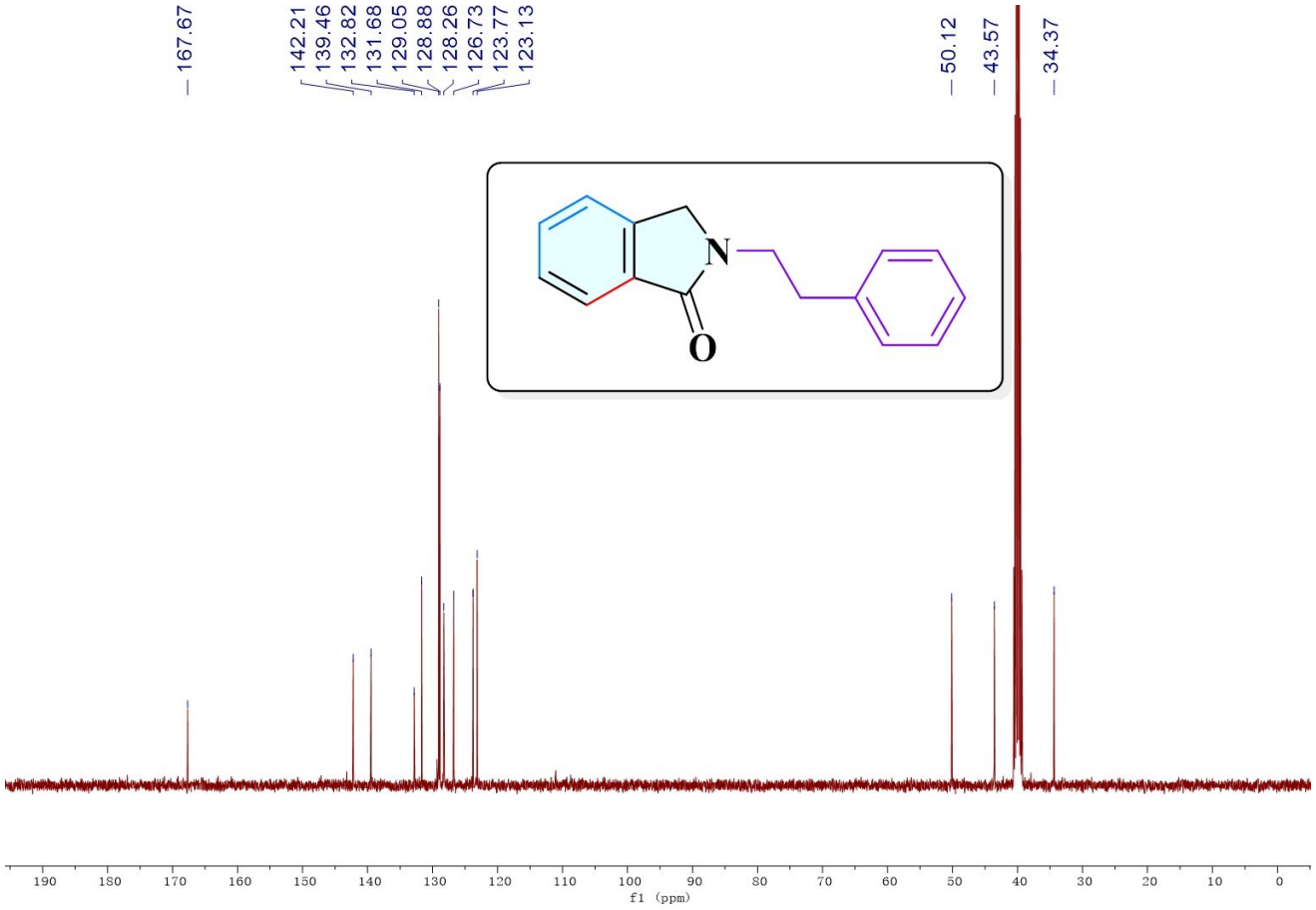

7.94
7.93
7.92
7.92
7.89
7.89
7.87
7.87
7.74
7.73
7.73
7.72
7.71
7.71
7.58
7.57
7.56
7.56
7.55
7.54
7.53
7.52
7.52
7.51
7.50
7.49
7.49
7.47
7.47
7.46
7.44
7.44
5.71
5.69
4.58
4.54
4.13
4.08
3.40
2.50
1.75
1.73
N
CH3
O
1.10
2.94
0.97
5.93
0.99
0.99
0.97
3.00
f1 (ppm)

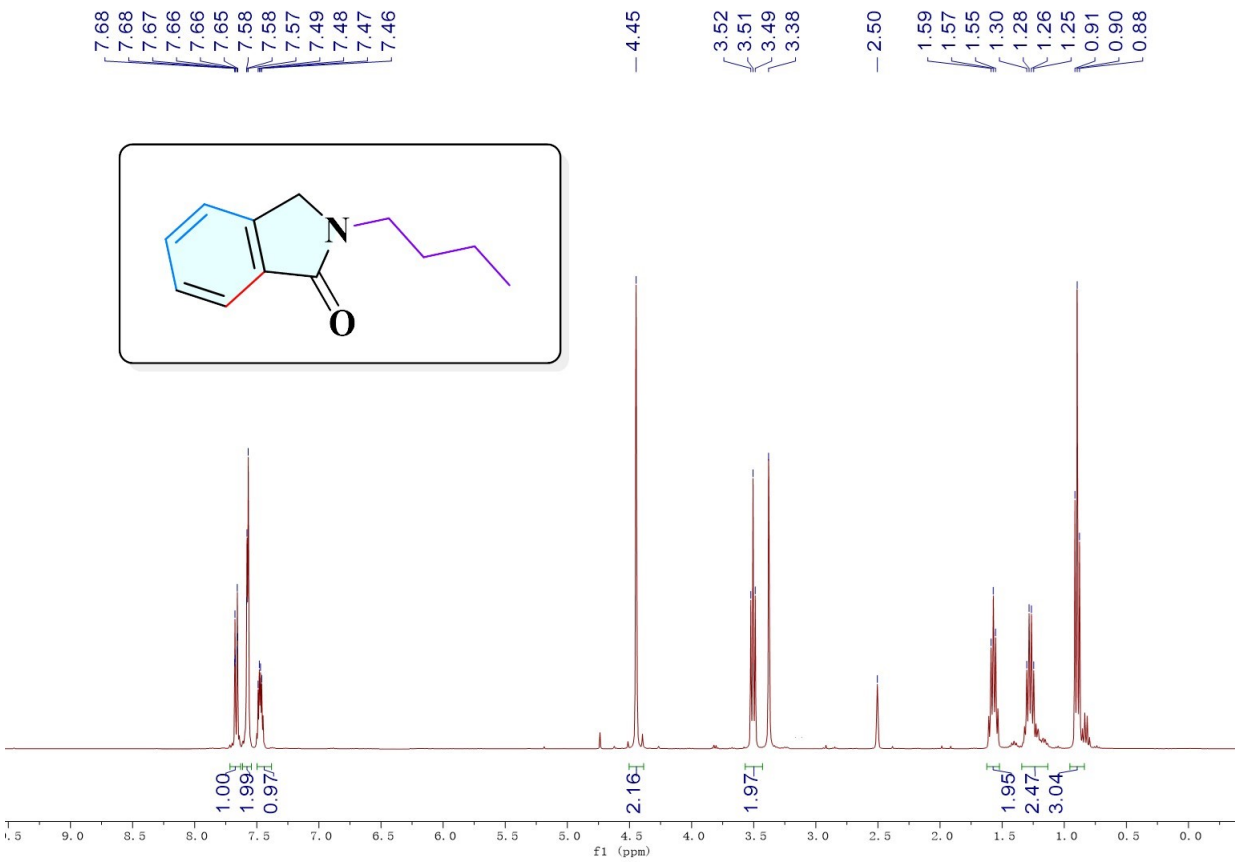

167.54
142.30
139.32
133.35
132.72
132.71
131.83
128.72
128.39
127.90
126.75
126.51
125.99
125.99
125.41
124.03
123.31
49.72
46.28
18.16
N
CH3
O
f1 (ppm)

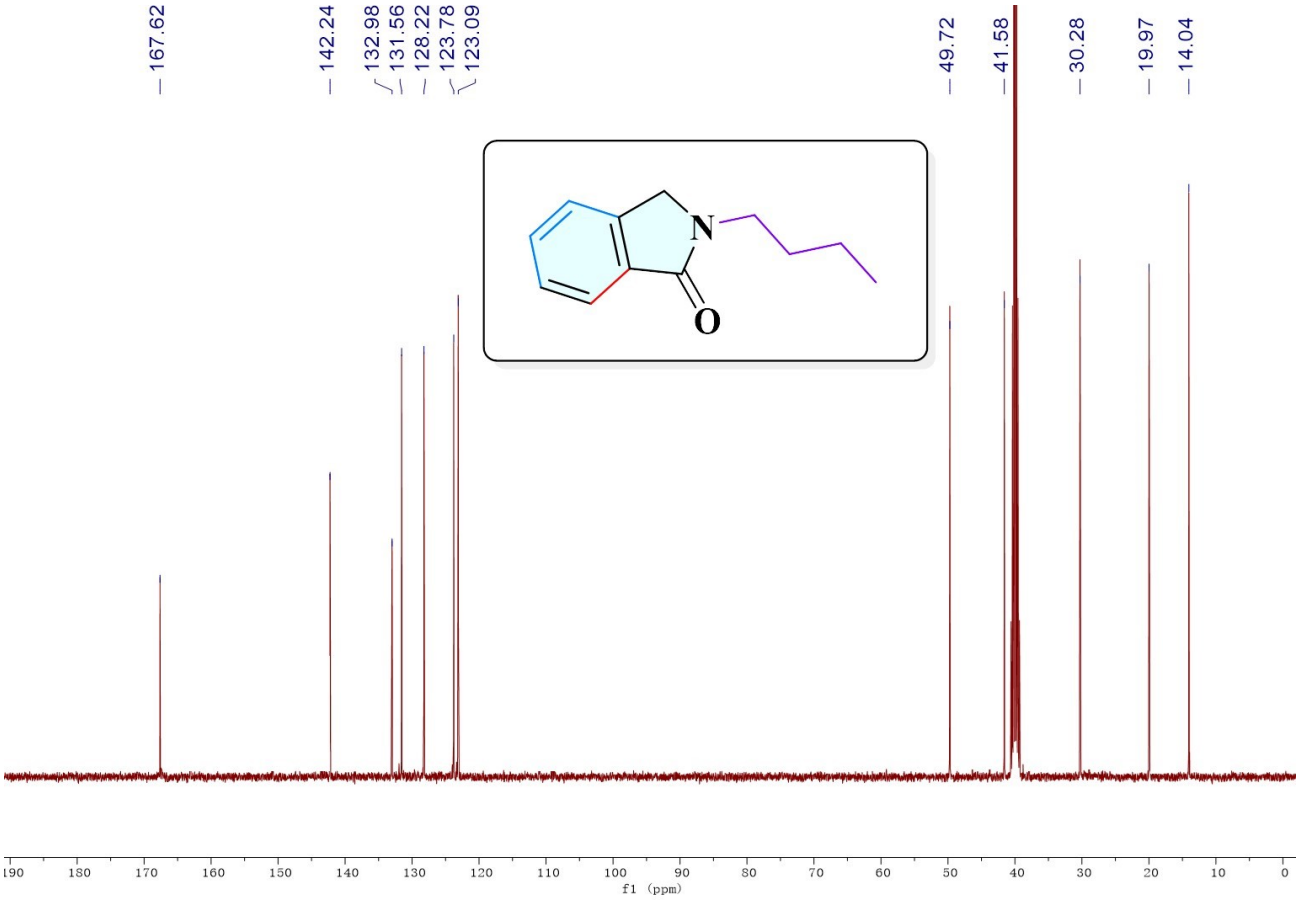

8.11 8.10 8.10 8.09 8.09 7.96 7.96 7.95 7.94 7.94 7.92 7.74 7.74 7.72 7.72 7.60 7.58 7.58 7.56 7.53 7.53 7.52 7.51 7.51 7.50 7.49 7.49 7.48 7.48 7.47 7.46 7.45 7.44 7.43 7.41 6.27 6.25 6.23 6.22 4.50 4.45 3.53 3.48 3.39 2.50 1.76 1.74
CH3
N
O
0.99 2.03 2.00 1.09 5.21 1.02 1.02 1.11 3.00
f1 (ppm)

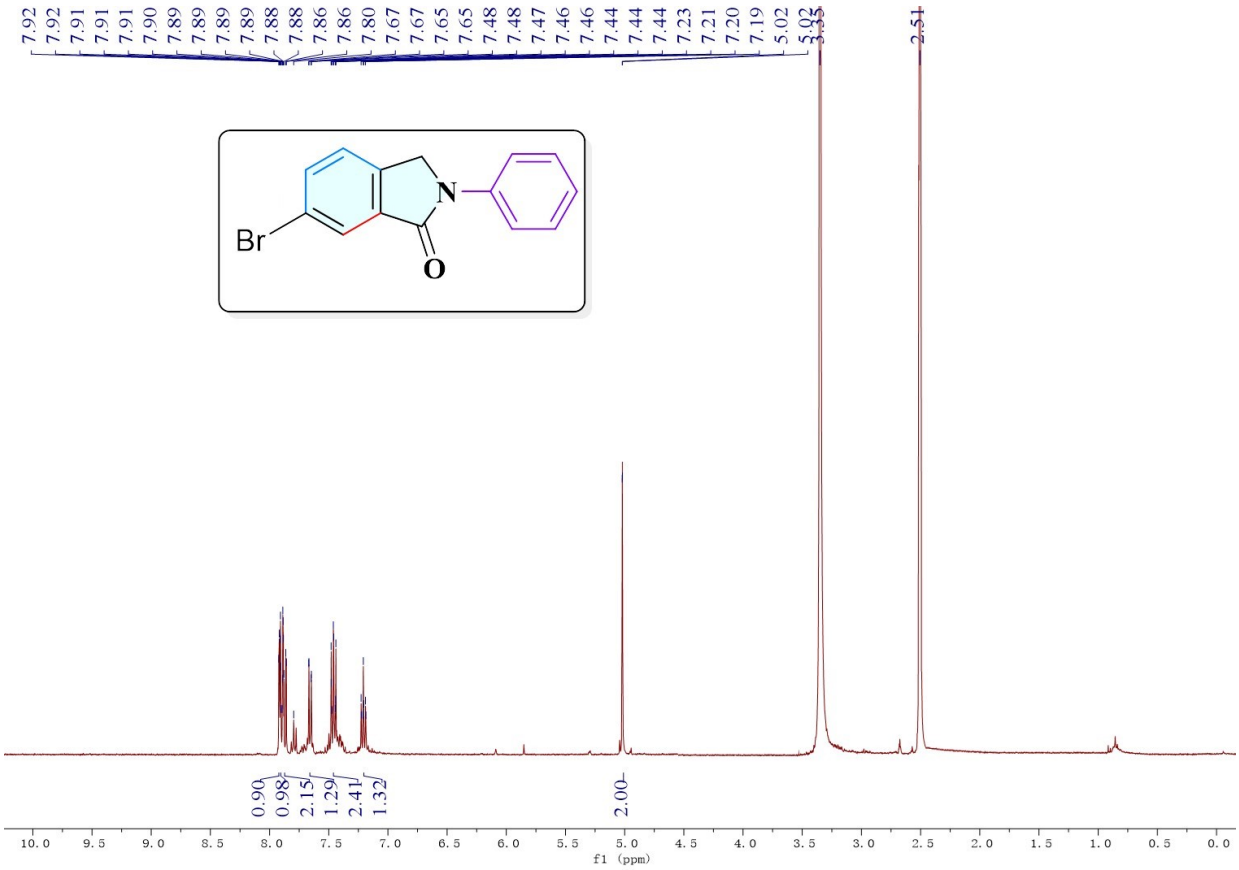

166.87 142.15 136.34 133.91 132.53 131.76 131.38 129.25 128.95 128.37 127.15 126.37 125.82 124.66 124.01 123.32 123.26
45.94 45.74
17.91
CH3
N
O
f1 (ppm)

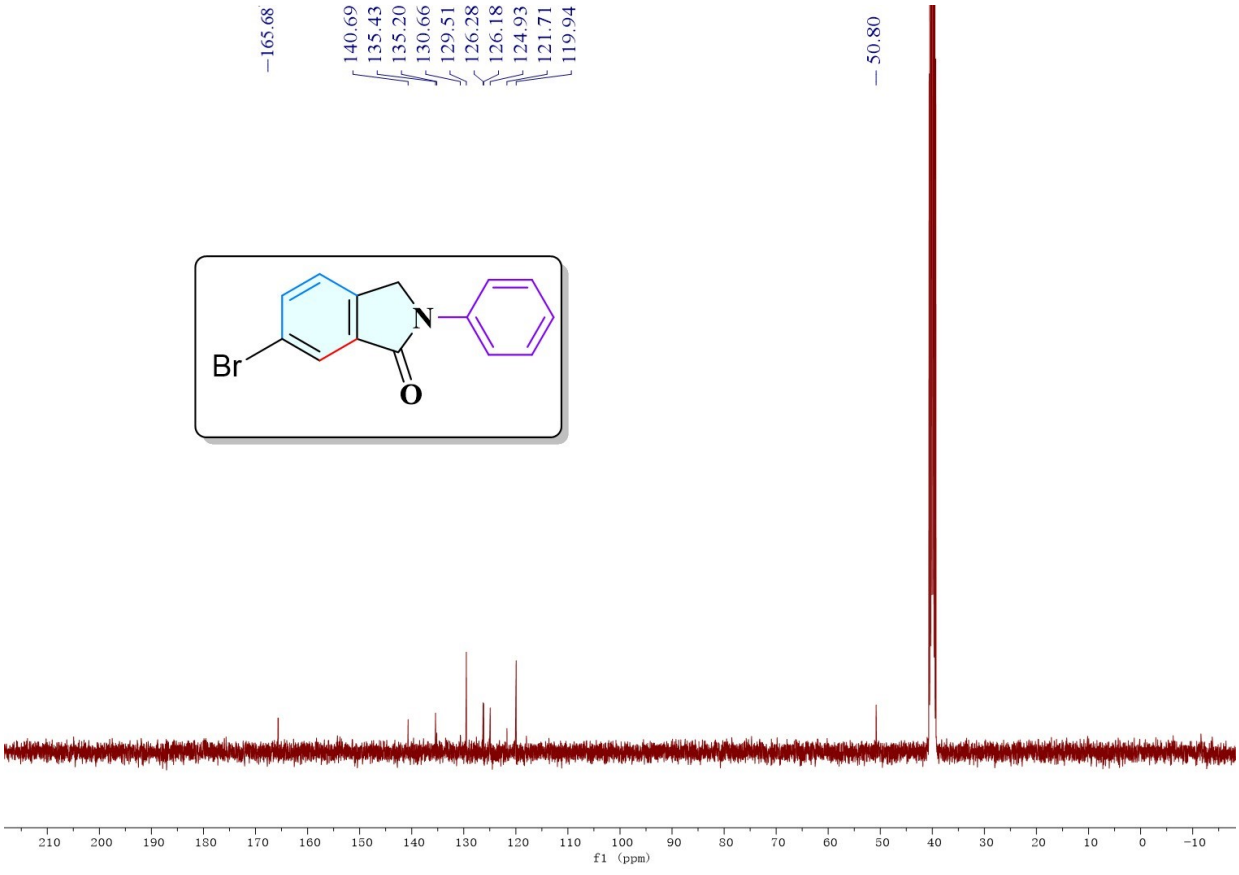

7.66
7.64
7.57
7.57
7.55
7.55
7.48
7.48
7.46
7.46
7.46
7.29
7.28
7.27
7.26
7.25
7.24
7.24
7.21
7.20
7.19
7.19
7.17
4.39
3.78
3.77
3.75
3.40
2.94
2.92
2.90
2.50
N
O
1.03
1.98
1.04
4.07
1.23
2.01
1.96
2.00
f1 (ppm)

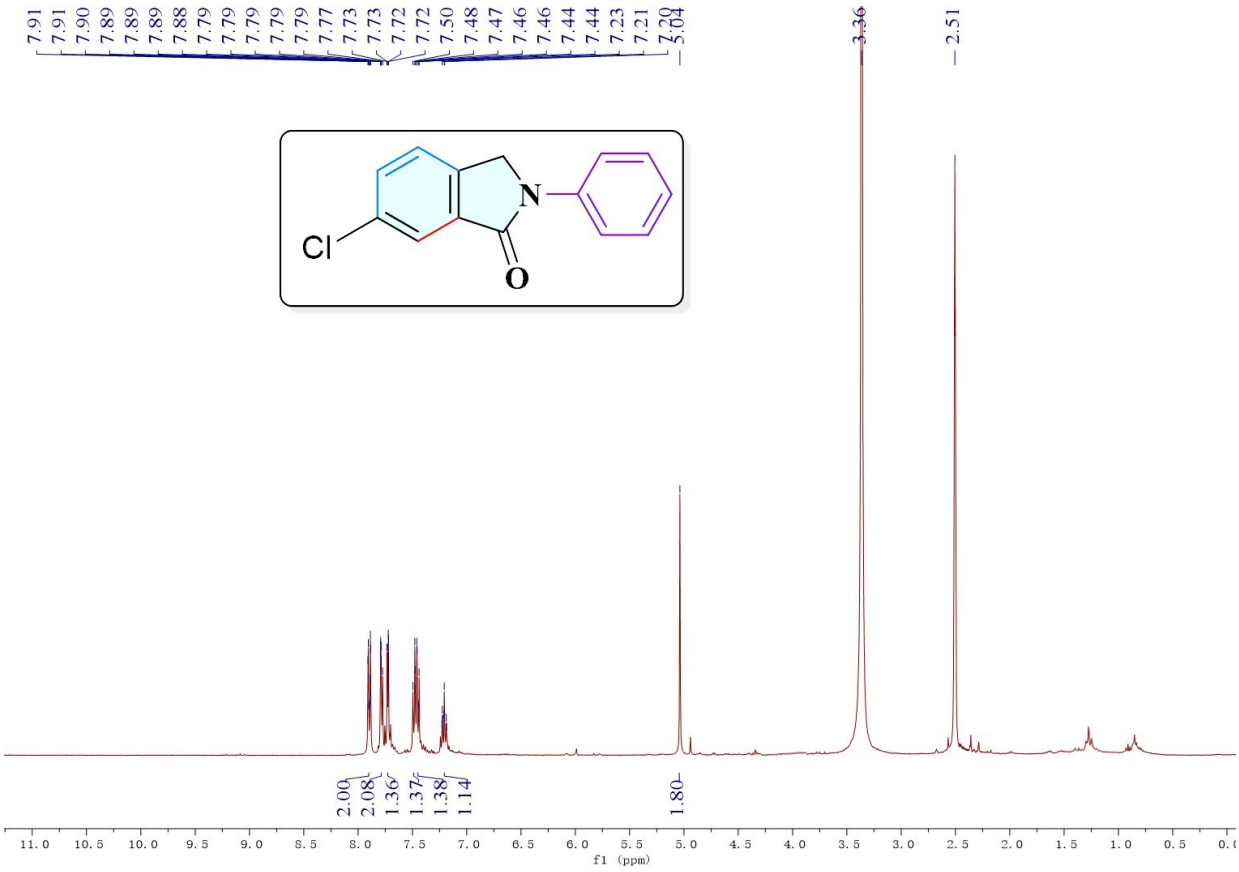

167.67
142.21
139.46
132.82
131.68
129.05
128.88
128.26
126.73
123.77
123.13
50.12
43.57
34.37
N
O
f1 (ppm)

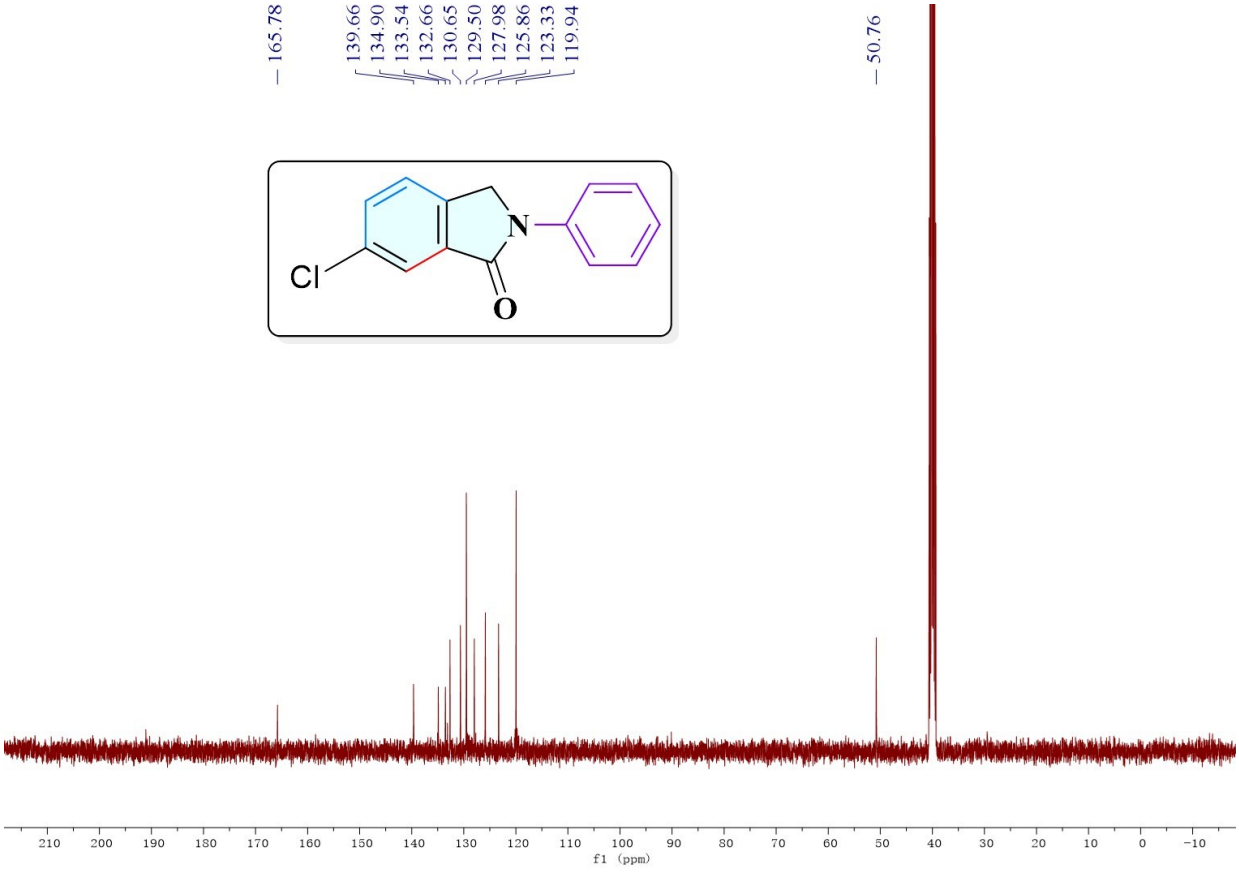

7.68
7.68
7.67
7.66
7.66
7.65
7.58
7.58
7.57
7.49
7.48
7.47
7.46
4.45
3.52
3.51
3.49
3.38
2.50
1.59
1.57
1.55
1.30
1.28
1.26
1.25
0.91
0.90
0.88
N
O
1.00
1.99
0.97
2.16
1.97
1.95
2.47
3.04
f1 (ppm)

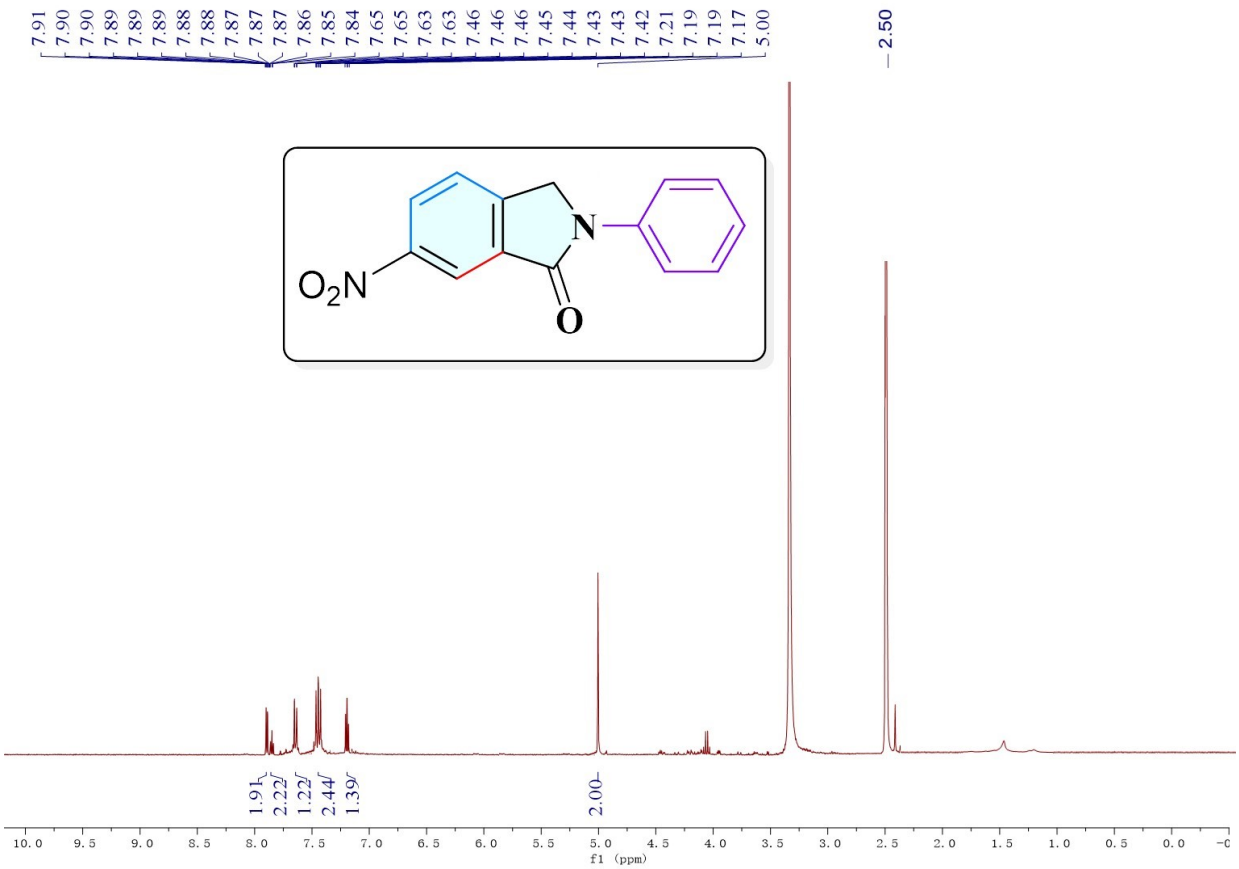

167.62
142.24
132.98
131.56
128.22
123.78
123.09
49.72
41.58
30.28
19.97
14.04
N
O
f1 (ppm)

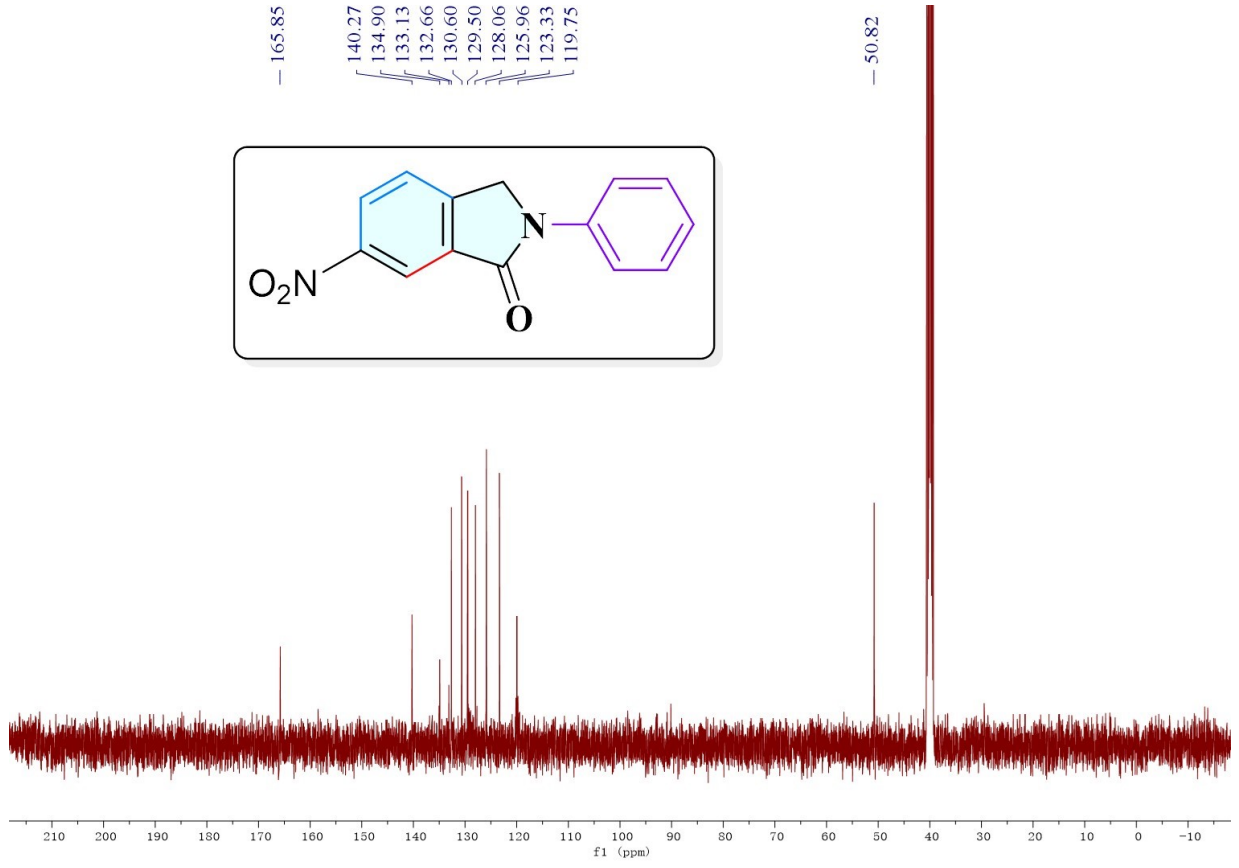

7.92
7.92
7.91
7.91
7.91
7.90
7.89
7.89
7.89
7.89
7.88
7.88
7.86
7.86
7.80
7.67
7.67
7.65
7.65
7.48
7.48
7.47
7.46
7.46
7.44
7.44
7.44
7.23
7.21
7.20
7.19
5.02
5.02
3.33
2.51
Br
N
O
0.90
0.98
2.15
1.29
2.41
1.32
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

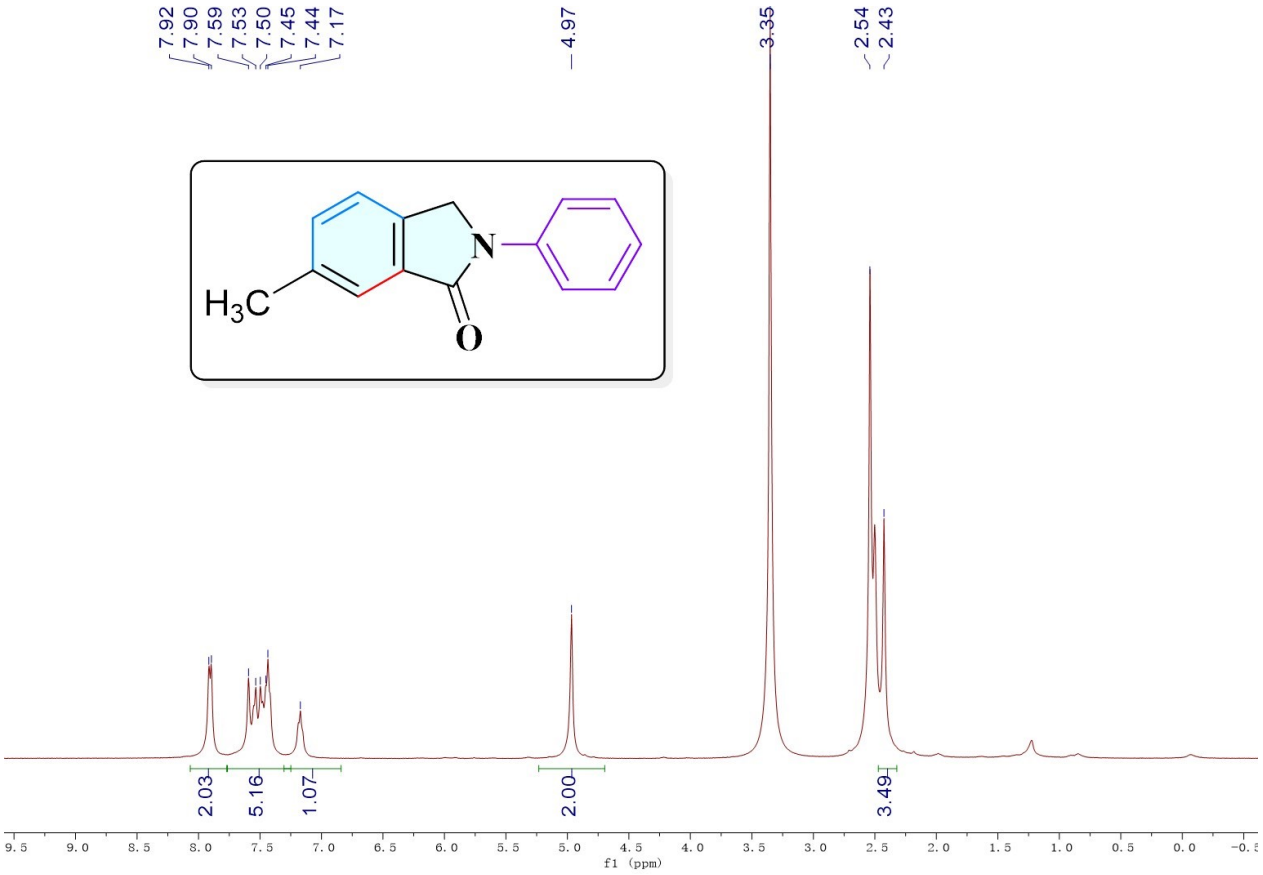

165.68
140.69
135.43
135.20
130.66
129.51
126.28
126.18
124.93
121.71
119.94
50.80
Br
N
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

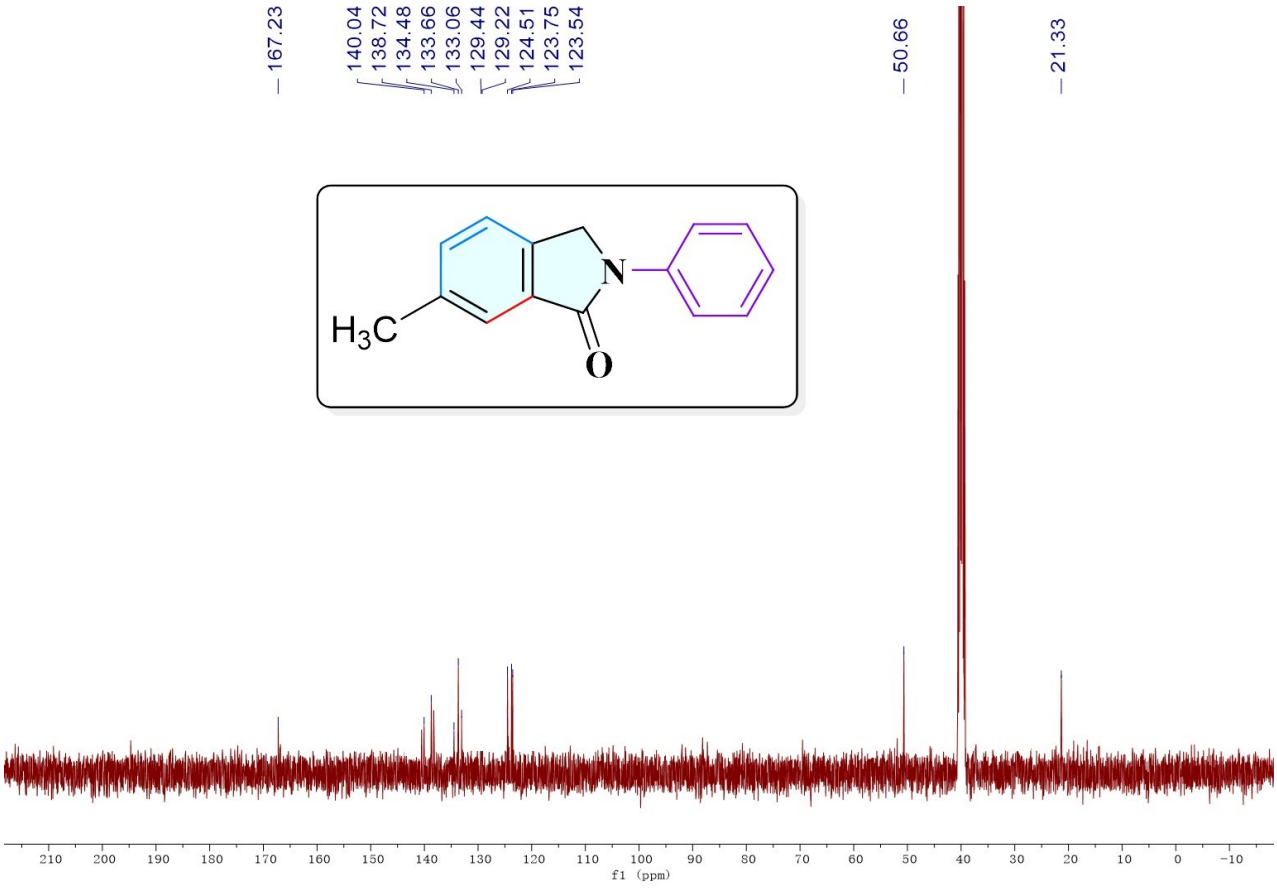

7.91
7.91
7.90
7.89
7.89
7.89
7.88
7.79
7.79
7.79
7.79
7.77
7.73
7.73
7.72
7.72
7.50
7.48
7.47
7.46
7.46
7.44
7.44
7.23
7.21
7.20
7.04
3.36
2.51
Cl
N
O
2.00
2.08
1.36
1.37
1.38
1.14
1.80
f1 (ppm)


165.78
139.66
134.90
133.54
132.66
130.65
129.50
127.98
125.86
123.33
119.94
50.76
Cl
N
O
f1 (ppm)


7.91
7.90
7.90
7.89
7.89
7.89
7.88
7.88
7.87
7.87
7.87
7.86
7.85
7.84
7.65
7.65
7.63
7.63
7.46
7.46
7.46
7.45
7.44
7.43
7.43
7.42
7.21
7.19
7.19
7.17
5.00
2.50
O2N
N
O
1.91
2.22
1.22
2.44
1.39
2.00
f1 (ppm)


165.85
140.27
134.90
133.13
132.66
130.60
129.50
128.06
125.96
123.33
119.75
50.82
O2N
N
O
f1 (ppm)


7.92
7.90
7.59
7.53
7.50
7.45
7.44
7.17
4.97
3.35
2.54
2.43
H3C
N
O
2.03
5.16
1.07
2.00
3.49
f1 (ppm)


167.23
140.04
138.72
134.48
133.66
133.06
129.44
129.22
124.51
123.75
123.54
50.66
21.33
H3C
N
O
f1 (ppm)

6 References

[S1] M. Frisch, G. Trucks, H. Schlegel, G. Scuseria, M. Robb, J. Cheeseman, G. Scalmani, V. Barone, G. Petersson and H. J. I. W. C. Nakatsuji, *Gaussian 16 A.03*. (Wallingford, CT, 2016).

[S2] S. Ketrat, T. Maihom, S. Wannakao, M. Probst, S. Nokbin and J. Limtrakul, *Inorg. Chem*., 2017, **56**, 14005-14012.

[S3] W. R. Wadt and o. c. p. Hay, *J.Chem. Phys*., 1985, **82**, 284-298.

[S4] C. J. C. Aleksandr V. Marenich and Donald G. Truhlar, *J. Phys. Chem. B*., 2009, **113**, 6378–6396.

[S5] A. S. A. Elyazed, Y. Sun, A. M. E. Nahas and A. M. Yousif, *RSC Adv*., 2020, **10**, 41283-41295.

[S6] V. Nozari, C. Calahoo, J. M. Tuffnell, D. A. Keen, T. D. Bennett and L. Wondraczek, *Nat. Commun*., 2021, **12**, 5703.

[S7] L. Xie, S. Liu, B. Gao, C. Zhang, C. Sun, D. Li and Z. Su, *Chem Commun*., 2005, **18**, 2402-2404.

[S8] H. Li, X. Liu, T. Yang, W. Zhao, S. Saravanamurugan and S. Yang, *ChemSusChem*., 2017, **10**, 1761-1770.

[S9] X. Wang, X. Ma, H. Wang, P. Huang, X. Du, X. Lu, *Microchim. Acta.,* 2017, **184**, 3681-3687.

[S10] a) F. A. Miller, Course Notes on the Interpretation of Infrared and Raman Spectra. *John Wiley & Sons, New York, USA,* 2003, 73-84; b) T. J. Jia, P. W. Li, Z. G. Shang, L. Zhang, T. C. He, Y. J. Mo, *J. Mol. Struct*., 2008, **873**, 1-4; c) T. Kim, R. S. Assary, C. L. Marshall, D. J. Gosztola, L. A. Curtiss, P. C. Stair, *Chem. Phys. Lett*., 2012, **531**, 210-215.

[S11] R. L. Rae, J. M. Zurek, M. J. Paterson, M. W. Bebbington, *Org. Biomol. Chem*., 2013, **11**, 7946-7952.