Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Iron-Catalyzed Intramolecular Acyl Nitrene/Alkyne Metalation for the Divergent Synthesis of Pyrrolo[2,1-*a*]isoindol-5-ones and Isoindol-1-ones

Xiaojing Lai,[a] Jin-Biao Liu,[b] Yu-Chao Wang,[a] and Guanyinsheng Qiu*,[a]

[a] College of Biological, Chemical Sciences and Engineering, Jiaxing University, Jiaxing 314001, Zhejiang, China; E-mail: qiuguanyinsheng@mail.zjxu.edu.cn.

[b] School of Metallurgical and Chemical Engineering, Jiangxi University of Science and Technology, Ganzhou 341000, China

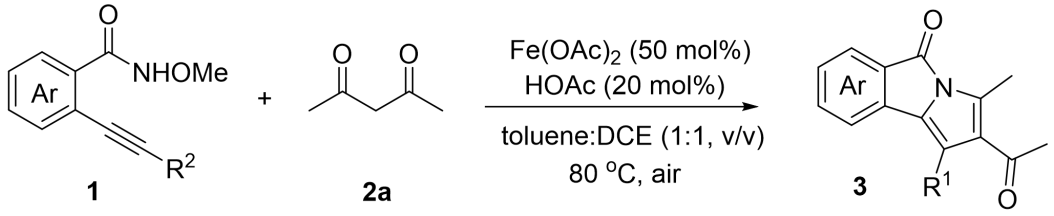
Table of contents

1. General information

The UV–Vis spectra were recorded on an Ultraviolet spectrophotometer (UV-2550). Emission spectra were performed on a Fluorescence spectrometer (F-4600). Error limits were estimated: λ (±1 nm); τ (±10%); φ (±10%). The solvents were distilled from standard drying agents. Unless otherwise stated, commercial reagents purchased from Alfa Aesar, Acros and Aldrich chemical companies were used without further purification. Purification of reaction products was carried out by flash chromatography using Qing Dao Sea Chemical Reagent silica gel (200–300 mesh). ^{1}H NMR spectra were recorded on a Bruker Avance III 400 (400 MHz) spectrometer and referenced internally to the residual proton resonance in $CDCl_3$ (δ = 7.26 ppm), or with tetramethylsilane (TMS, δ = 0.00 ppm) as the internal standard. Chemical shifts were reported as parts per million (ppm) in the δ scale downfield from TMS. Multiplicity is indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), quint (quintet), m (multiplet), dd (doublet of doublet), bs (broad singlet). ^{13}C NMR spectra were recorded on Bruker spectrometer with complete proton decoupling, and chemical shifts were reported in ppm from TMS with the solvent as the internal reference ($CDCl_3$, δ = 77.0 ppm).

2. General procedure

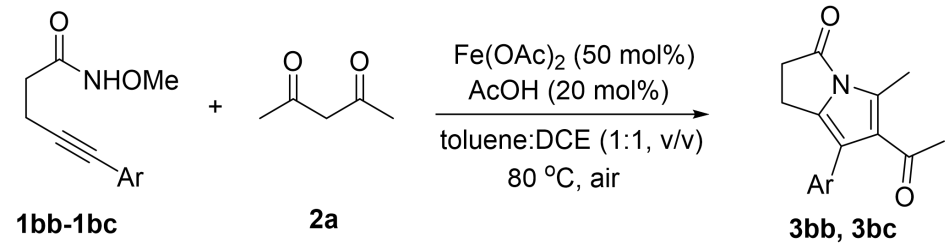
(a) Synthesis of compounds 3



N-methoxy-2-(phenylethynyl)benzamide **1** (0.2 mmol), Acetylacetone **2a** (1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (50 mol%) were added to a test tube, the solvent (toluene:DCE, 1:1, v/v, 2 mL) was added. The mixture was stirred at 80 °C for 6 h. After the disappearance of substrate as indicated by TLC, the mixture was filtrated, Evaporation of the solvent and purification by flash column chromatograph provided

the desired products **3**.

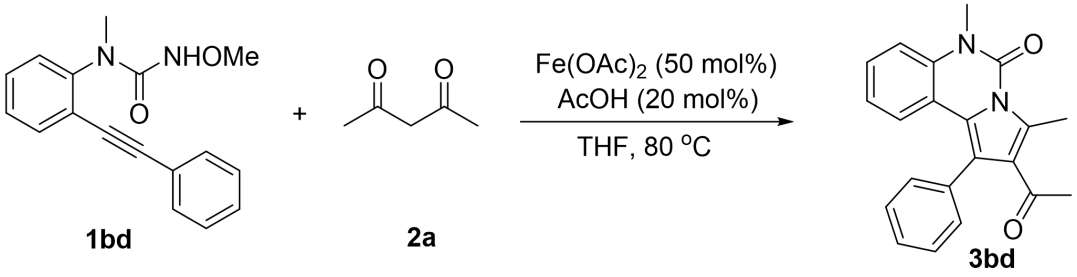
(b) Synthesis of compounds 3bc and 3bd



1bb-1bc **2a** **3bb, 3bc**

N-methoxy-2-(phenylethynyl)acetamide **1bb-1bc** (0.2 mmol), Acetylacetone **2a** (1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (50 mol%) were added to a test tube, the solvent (toluene:DCE, 1:1, v/v, 2 mL) was added. The mixture was stirred at 80 °C for 6 h. After the disappearance of substrate as indicated by TLC, the mixture was filtrated, Evaporation of the solvent and purification by flash column chromatograph provided the desired products **3bb** and **3bc**.

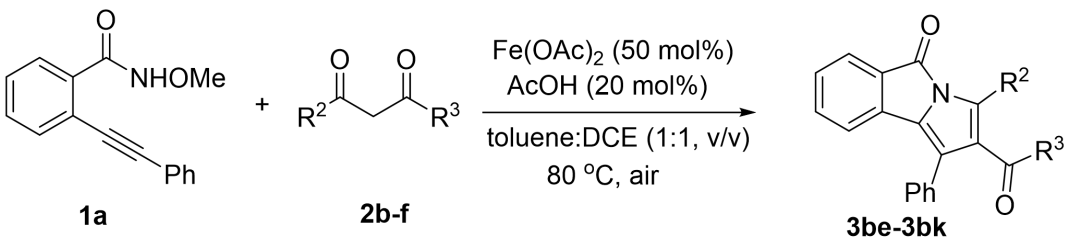
(c) Synthesis of compound 3be



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

3-methoxy-1-methyl-1-(2-(phenylethynyl)phenyl)urea 1bd (0.2 mmol), Acetylacetone **2a** (1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (50 mol%) were added to a test tube, the solvent THF (2.0 mL) was added. The mixture was stirred at 80 °C for 6 h. After the disappearance of substrate as indicated by TLC, the mixture was filtrated, Evaporation of the solvent and purification by flash column chromatograph provided the desired products **3bd**.

(d) Synthesis of compounds 3be-3bk



1a **2b-f** **3be-3bk**

N-methoxy-2-(phenylethynyl)acetamide **1a** (0.2 mmol), Acetylacetone **2b-2f** (1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (50 mol%) were added to a test tube, the solvent (toluene:DCE, 1:1, v/v, 2 mL) was added. The mixture was stirred at 80 °C for 6 h. After the disappearance of substrate as indicated by TLC, the mixture was filtrated, Evaporation of the solvent and purification by flash column chromatograph provided the desired products **3be-3bk**.

3. Fluorescence experiment

3.1 Excitation and emission wavelengths for products

	3aa	**3ab**	**3ac**	**3ad**	**3ae**	**3af**	**3ai**	**3aj**	**3ak**
Ex/nm	410	481	456	419	420	440	399	416	421
Em/nm	538	573	529	519	551	573	524	523	530

	3al	**3am**	**3an**	**3ao**	**3ap**	**3aq**	**3ar**	**3as**	**3at**
Ex/nm	505	480	432	438	404	486	439	487	423
Em/nm	586	568	561	562	538	559	566	552	531

	3au	**3av**	**3aw**	**3ax**	**3ay**	**3az**	**3ba**	**3be**	**3bf**
Ex/nm	423	402	420	429	410	461	460	402	402
Em/nm	543	530	540	549	522	547	550	518	517

3.2 The selectivity of the Hg^{2+} detection method using 3ak as the probe.

8 mM of various ions (Be^{2+}, Cr^{3+}, Fe^{2+}, Ni^{2+}, Ca^{2+}, Al^{3+}, Sn^{2+}, Co^{2+}, Zn^{2+}, Cu^{2+}, Pb^{2+}, Mg^{2+}, Mn^{2+}, Ba^{2+}, K^{+}, Na^{+}, Li^{+}, Fe^{3+}, Ag^{+}, Ce^{4+}, Hg^{2+}) were prepared in water as stock solutions. Various ions (120 μM) were added to DCE/H_2O solution (v = 19:1) containing 5-acetyl-6-methyl-4-phenyl-8*H*-thieno[3,2-*a*]pyrrolizin-8-one (**3ak**) (40 μM). Then the mixture was incubated at room temperature for 1h. Emission spectra were measured in the range of 461 nm to 721 nm with an excitation wavelength at 421 nm, and the slit width is 10 nm/20 nm.

The results showed that only Hg^{2+} triggered a significant fluorescence enhancement, and encouragingly even Ce^{4+} only slightly enhance the fluorescence of the system (Figure 1). To demonstrate the specificity of the sensor to Hg^{2+}, a

competition experiment was carried out by adding Hg^{2+} (120 μM) to a system containing the same concentration of another metal cations. No significant change in fluorescence was observed, indicating that this method possesses great potential for the detection of Hg^{2+}, even in the presence of other common amines. These results demonstrat the high selectivity of this method towards Hg^{2+}.

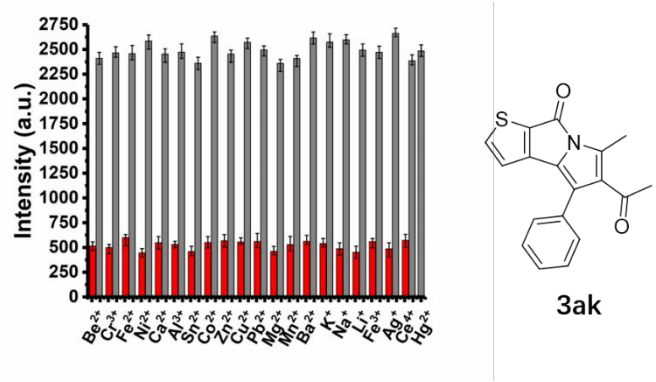


Figure 1. Fluorescence intensity upon addition of different ions (Be^{2+}, Cr^{3+}, Fe^{2+}, Ni^{2+}, Ca^{2+}, Al^{3+}, Sn^{2+}, Co^{2+}, Zn^{2+}, Cu^{2+}, Pb^{2+}, Mg^{2+}, Mn^{2+}, Ba^{2+}, K^{+}, Na^{+}, Li^{+}, Fe^{3+}, Ag^{+}, Ce^{4+}, Hg^{2+}). The red bars represent the addition of 120 μM different ions to the sensoring system. The black bars represent the subsequent addition of 120 μM Hg^{2+} to the system. λ_{ex} = 421 nm, λ_{em} = 530 nm.

3.3 Detection of Hg^{2+} using 3ak in DCE

The stock solution of **3ai** (8 mM) and Hg^{2+} (8 mM) were prepared in DCE solvent. Solutions of **3ak** (40 μM), Hg^{2+} (40 μM), and **3ak** (40 μM) with Hg^{2+} (40 μM) were incubated in DCE at room temperature for 1 h, respectively. Then, emission spectra were measured in the range of 461 nm to 721 nm with an excitation wavelength at 421 nm, and the slit width is 10 nm/20 nm.

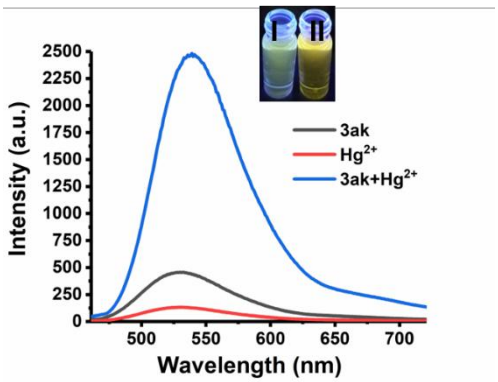


Figure 2. Fluorescence emission spectra (λ_{ex} = 421 nm) of the control experiments containing 40 μM **3ak** (λ_{em} = 530 nm), Hg^{2+} (40 μM), and **3ak** with Hg^{2+} (λ_{em} = 539 nm) in the DCE. Photographs of 1 mM of **3ak** (I), 1 mM

of **3ak** with 1 mM of Hg^{2+} (II) under 365 nm UV illumination.

The results show that fluorescence enhancement occurs when **3ak** and Hg^{2+} are presented (Figure 2). Bright yellow fluorescence could be observed with the naked eye via irradiation with an ultraviolet lamp at 365 nm (Figure 2) when **3ak** (1 mM) and Hg^{2+} (1 mM) were mixed at room temperature. From λ_{em} data, slight red-shift effect was observed.

3.4 Detection of Hg^{2+} in various ratios in mixed solvent of water:DCE

The stock solutions of **3ak** (8 mM) and Hg^{2+} (8 mM) were prepared in DCE. **3ak** (40 μM) and Hg^{2+} (0.4 μM) were added into various percentages of water in DCE (water content 0-100%), and then the mixture was incubated at room temperature for 1 h. Emission spectra were measured in the range of 461 nm to 721 nm with an excitation wavelength at 421 nm, and the slit width is 10 nm/20 nm.

To obtain the optimum performance of this sensing platform, we examined the luminescence response of the reaction between the probe molecule **3ak** and Hg^{2+} for 1 h in solvent systems consisting of various DCE/H_2O ratios. As shown in figure 3, the luminous efficiency is enhanced when the water content in DCE increases. The highest luminescence enhancement was obtained with a 4:6 DCE/H_2O ratio. The luminescence rate gradually decreases thereafter. We therefore chose a 4:6 DCE/H_2O solution as the best detection system for subsequent experiments. The results were presneted in Figure 3.

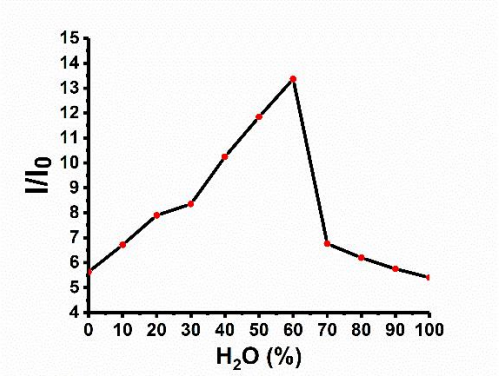


Figure 3. Fluorescence enhancement of 5-acetyl-6-methyl-4-phenyl-8*H*-thieno[3,2-*a*]pyrrolizin-8-one (3ak) (40 μM) with 120 μM of Hg^{2+} in various percentages of water in DCE.

3.5 Emission titration experiments

Different concentrations of Hg^{2+} were added to DCE containing **3ak** (40 μM) in a cuvette. Then emission spectra were recorded after 1-hour incubation in the range of 461 nm to 721 nm at λ_{ex} = 421 nm, and the slit width is 10 nm/20 nm.

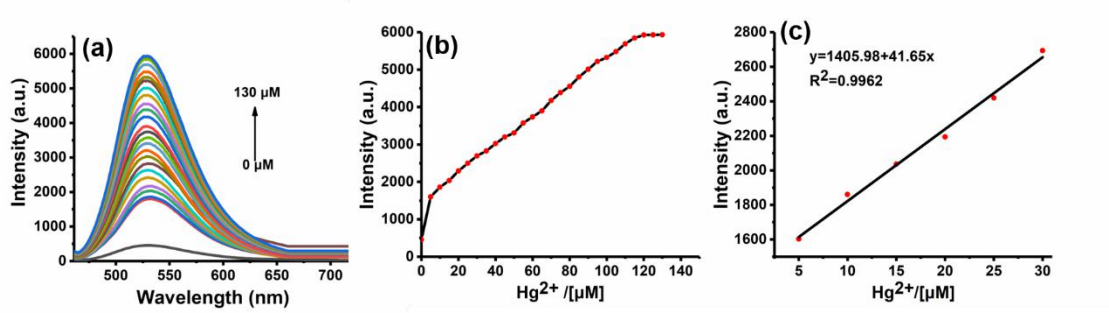


Figure 4. (a) Fluorescence responses of the system to various concentrations of Hg^{2+}. The concentration of 5-acetyl-6-methyl-4-phenyl-8*H*-thieno[3,2-*a*]pyrrolizin-8-one (3ak) is 40 μM and the concentration of Hg^{2+} is in the range of 0−130 μM; (b) Fluorescence responses at 530 nm of the system at various concentrations of Hg^{2+} (1−130 μM). (c) The linear correlation between fluorescence intensity and the concentration of Hg^{2+} (5−30 μM). λ_{ex} = 421 nm, λ_{em} = 530 nm.

We next examined the fluorescence responses of the method to various concentrations of Hg^{2+}. The system containing 5-acetyl-6-methyl-4-phenyl-8*H*-thieno[3,2-*a*]pyrrolizin-8-one (**3ak**) (40 μM) was incubated with the indicated concentrations of Hg^{2+} at rt for 1 h (Figure 4a). The fluorescence intensity of the system increased with increasing concentration of Hg^{2+} (0−130 μM). Upon the addition of Hg^{2+}, the sensor displayed more than a 13-fold enhancement in fluorescence, according to literature reported calculations. A good linear relationship was established at a range of 5−30 μM (Linear correlation index: R^2 = 0.9962). The limit of detection (LOD) was calculated as 2.90 μM, according to LOD = 3σ/s.

3.6 Study of the detection of Hg^{2+} in various water samples

River water was collected from the river, tap water was collected from chemistry laboratory, and rainwater was collected from Zhejiang. Hg^{2+} was spiked into arious water samples to obtain sample solutions with 19.2 μM of Hg^{2+} in DCE/H_2O (v =

19:1).

Lateritic soils were collected from Zhejiang. The samples were pre-treated according to a reported method[1] as noted below: Hg^{2+} (0.05 mmol) was spiked into 5 g of soil and stired well before use. Then the sample was extracted with water. After filtration, the sample was diluted to obtain a solution with 19.2 μM of Hg^{2+} in DCE/H_2O (v = 19:1).

The **3ak** was added into above real samples to a final solution with 40 μM. Then the mixture solutions were incubated at room temperature for 1 h. Emission spectra were measured in the range of 461 nm to 721 nm with an excitation wavelength at 421 nm, and the slit width is 10 nm/20 nm.

The results were presented in Table 1.

Table 1. Study of the detection of Hg^{2+} in water samples and lateritic soils samples[a]

	Samples	Added amount (μM)	Found amount (μM)	Std Dev (μM)	RSD (%, n=3)	Recovery (%, n=3)
Entry 1	River water	19.20	18.50	0.68	1.56	96.15
Entry 2	Tap water	19.20	18.23	1.12	2.37	95.58
Entry 3	Rainwater	19.20	18.56	0.55	1.52	97.26
Entry 4	Lateritic soils water	19.20	17.12	1.34	2.50	94.06

[a]Results of the determination of Hg^{2+} in water samples and lateritic soils sample. Experimental condition: DCE/H_2O (v/v = 4:6); Hg^{2+} (19.2 μM), 5-acetyl-6-methyl-4-phenyl-8*H*-thieno[3,2-*a*]pyrrolizin-8-one (**3ak**) (40 μM); reaction time: 1 h.

First, we collected Zhejiang river water, rainwater, tap water, lateritic soils, and pre-treated the samples according to a reported method.[2,3] Next, exogenous Hg^{2+} was added and the samples studied. The results show that Hg^{2+} (19.20 μM) can enhance the luminescence intensity of the sample significantly. The relative standard deviations and recoveries are shown in Table 1. The recovery rates for lateritic soils and rainwater samples were 94.06% and 97.26%, with relative standard deviations of 2.50% and 1.52%, respectively. The fluorescent probe can be used for the detection of Hg^{2+} in water samples and lateritic soils, and has good selectivity and sensitivity.

3.7 The selectivity of the Cys detection method using 3bf as the probe.

8 mM of various amino acids (Lysine, Tryptophan, Arginine, Histidine, Leucine, Isoleucine, Phenylalanine, Threonine, Valine, Methionine, Glutamate, Glycine, Serine, Aspartic acid, Tyrosine, L-Cysteine) were prepared in water as stock solutions. Various amino acid interferers (120 μM) were added to DCE/H_2O solution (v = 19:1) containing 2-acetyl-1-phenyl-3-(trifluoromethyl)-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (**3bf**) (40 μM). Then the mixture was incubated at room temperature for 1 h. Emission spectra were measured in the range of 450 nm to 710 nm with an excitation wavelength at 402 nm, and the slit width is 10 nm/20 nm. The results were showed in Figure 5.

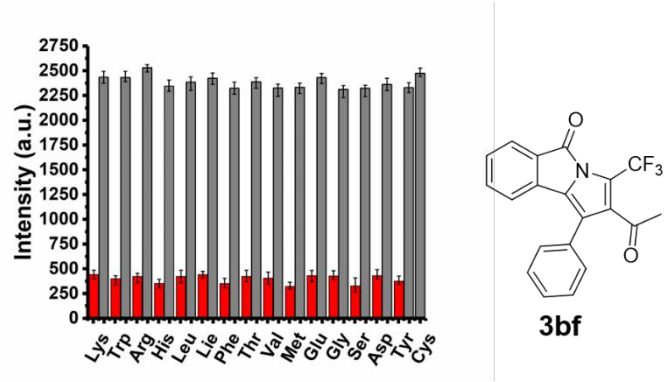


Figure 5. Fluorescence intensity upon addition of different amino acids (define lysine, tryptophan, arginine, histidine, leucine, Isoleucine, phenylalanine, threonine, valine, methionine, glutamate, glycine, serine, aspartic acid, tyrosine, L-cysteine, as Lys, Trp, Arg, His, Leu, Lle, Phe, Thr, Val, Met, Glu, Gly, Ser, Asp, Tyr, Cys). The red bars represent the addition of 120 μM different amino acids to the sensoring system. The black bars represent the subsequent addition of 120 μM Cys to the system. λ_{ex} = 402 nm, λ_{em} = 517 nm.

The results showed that only Cys triggered a significant fluorescence enhancement，and other amino acids exhibited low fluorescence enhancement. (Figure 6). To demonstrate the specificity of the probe to Cys, a competition experiment was carried out by adding Cys (120 μM) to a system containing the same concentration of another amino acids. No significant change in fluorescence was observed, indicating that this reaction-based probe possesses great potential for the detection of Cys, even in the presence of other amino acids. The results demonstrate that the compound possesses application as the highly specific Cys fluorescence probe. It is anticipated

that further optimization would lead to the development of a practical probe with great potential for the vivo detection of Cys.

3.8 The emission spectra of 3bf with Cys in DCE

The stock solution of **3bf** (8 mM) and Cys (8 mM) were prepared in DCE solvent. **3bf** (40 μM), Cys (40 μM), and **3bf** (40 μM) with Cys (40 μM) were mixed in DCE, and then the mixture was incubated at room temperature for 1 h. Emission spectra were measured in the range of 450 nm to 710 nm with an excitation wavelength at 402 nm, and the slit width is 10 nm/20 nm.

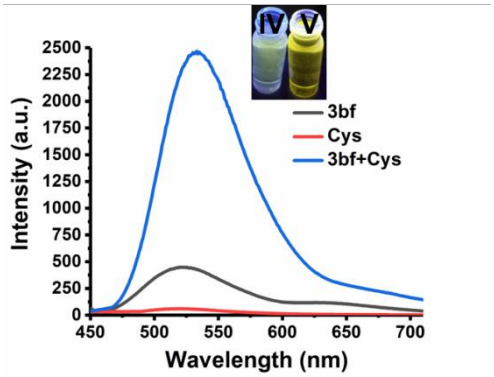


Figure 6. Fluorescence emission spectra (λ_{ex} = 402 nm) of the control experiments containing 40 μM **3bf** (λ_{em} = 517 nm), Cys (40 μM), **3bf** with Cys (λ_{em} = 533 nm) in the DCE. Photographs of 1 mM of **3bf** (IV), 1 mM of **3bf** with 1 mM of Hg^{2+} (V) under 365 nm UV illumination

The results show that fluorescence enhancement occurs only when **3bf** and Cys are present at the same time (Figure 6). Bright yellow fluorescence could be observed with the naked eye via irradiation with an ultraviolet lamp at 365 nm. From λ_{em} data, slight red-shift effect was observed.

3.9 The emission spectra of 3ak with N_2H_4 in DCE

The stock solution of **3ak** (8 mM) and N_2H_4 (40 μM) were prepared in DCE solvent. **3ak** (8 mM), N_2H_4 (40 μM), **3ak** (40 μM) with N_2H_4 (40 μM) were mixed in DCE, respectively. Then, the mixture was incubated at room temperature for 1 h. Emission spectra were measured in the range of 461 nm to 721 nm with an excitation

wavelength at 421 nm, and the slit width is 10 nm/20 nm.

The results show that fluorescence enhancement occurs only when **3ak** and N_2H_4 are present (Figure 7). Bright yellow fluorescence could be observed with the naked eye via irradiation with an ultraviolet lamp at 365 nm when **3ak** (1 mM) and N_2H_4 (1 mM) were mixed at room temperature. From λ_{em} data, slight red-shift effect was observed.

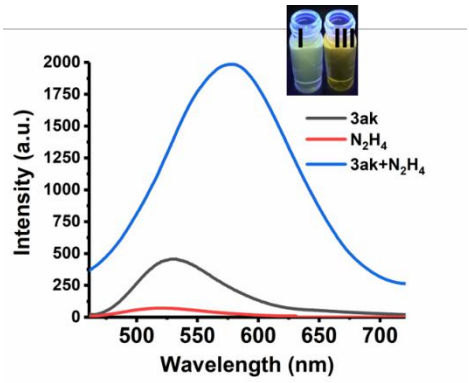


Figure 7. Fluorescence emission spectra (λ_{ex} = 421 nm) of the control experiments containing 40 μM **3ak** (λ_{em} = 530 nm), N_2H_4 (40 μM), **3ak** with N_2H_4 (λ_{em} = 576 nm) in the DCE. Photographs of 1 mM of **3ak** (I), 1 mM of **3ak** with 1 mM of N_2H_4 (III) under 365 nm UV illumination

3.10 The correlation between emission wavelength and the R substituents Hammett constant (σ_p) in phenyl ring of the products (3ad (OMe), 3ac (Me), 3aa (H), 3ae (Cl), 3af (Br))

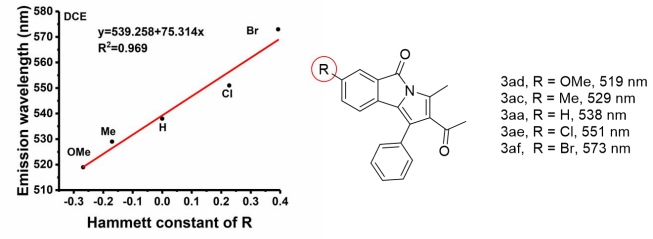


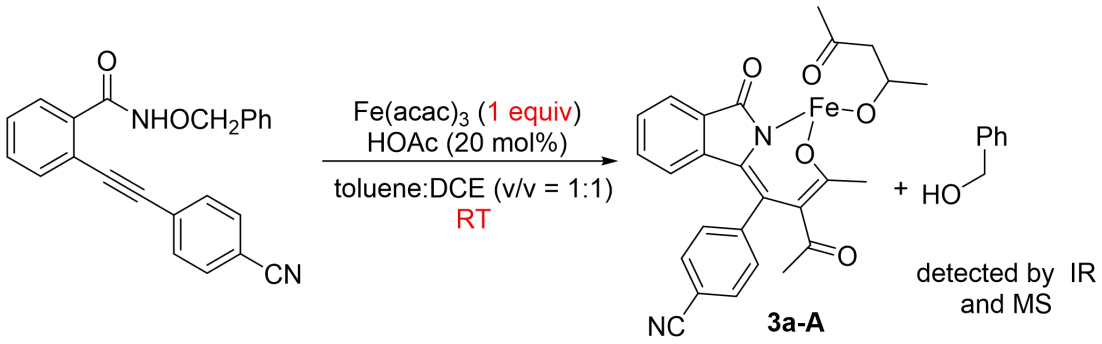
Figure 8. Structure-photophysical property relationship of fused-ring compounds. The compound order is **3ad** (OMe), **3ac** (Me), **3aa** (H), **3ae** (Cl), **3af** (Br), and stock solution of the compound was prepared in DCE (120 μM). Correlation between emission wavelength and Hammett constant (σp) of R substituents in fused-ring compounds.

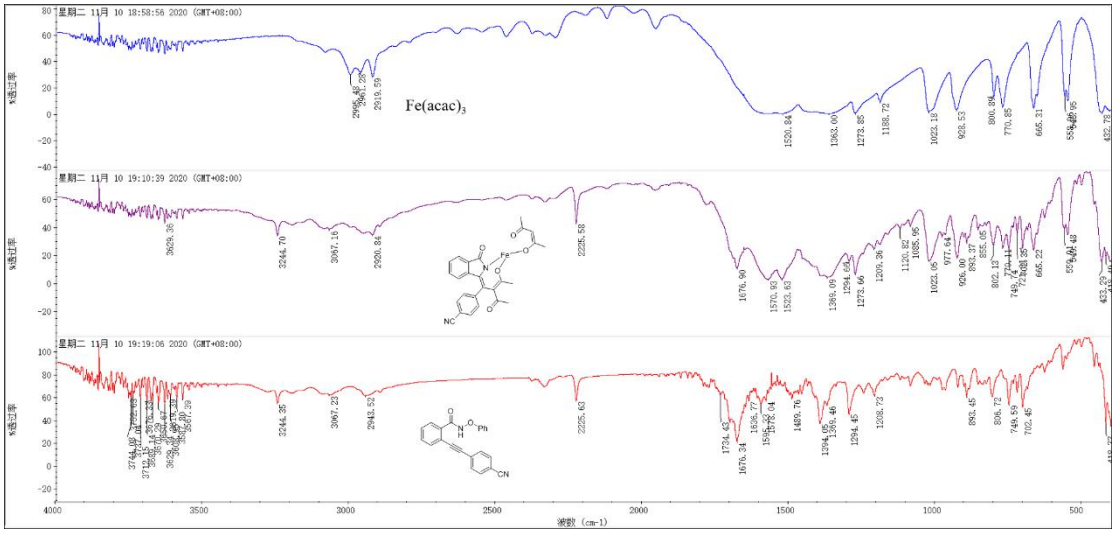
1. Y.-M. Lv, *Modern. Agrochem.*, **2004**, *3*, 9.

2. A. R. Jacobson, S. Dousset, F. Andreux and P. C. Baveye, *Environ. Sci. Tec*., **2007**, *41*, 6343.

3. Y. Wang, J. Li, Y. Gao, Y. Yang, Y. Gao and Z. Xu, *Hydrometallurgy*., **2020**, *191*, 105220.

4. Mechanism

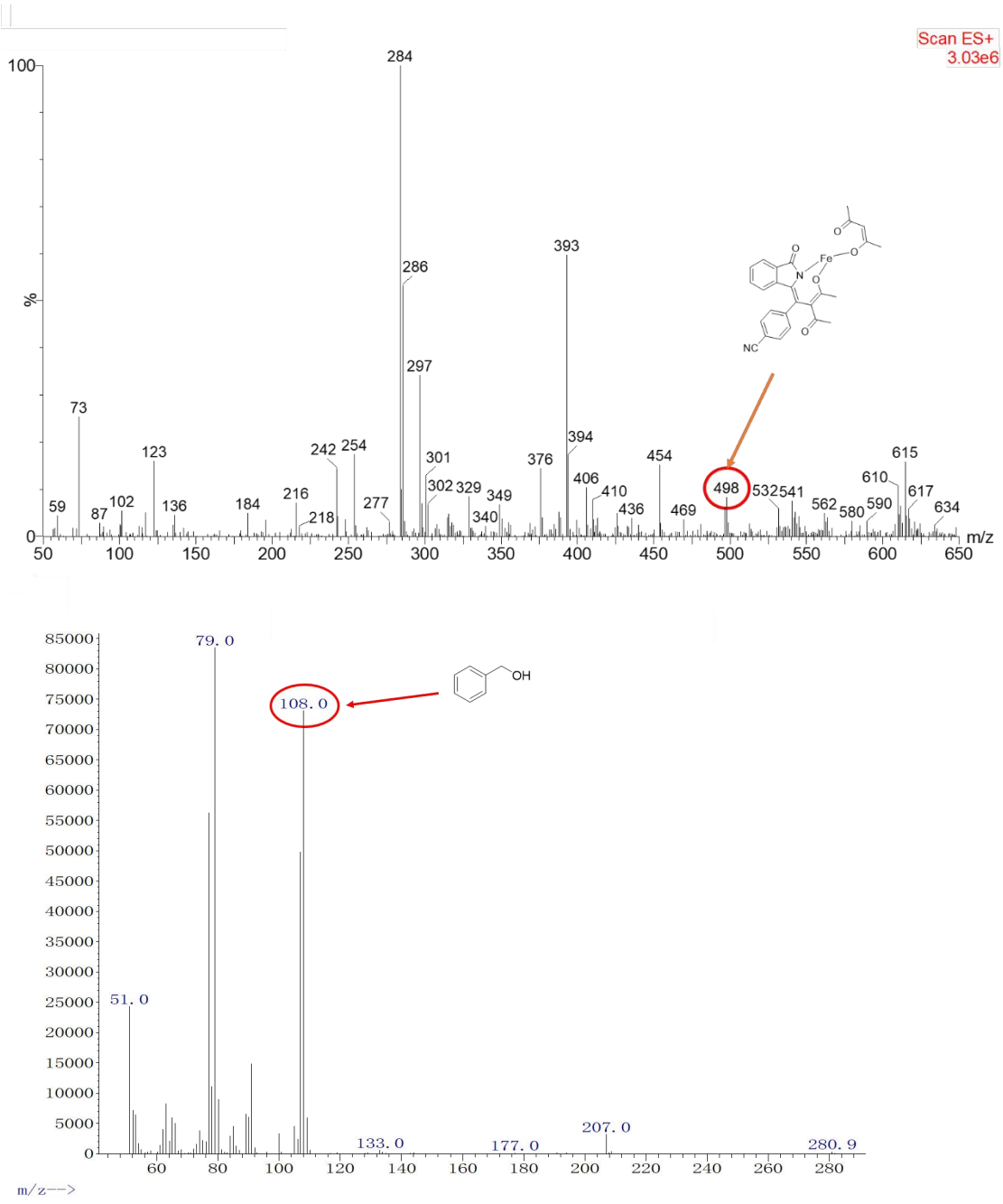


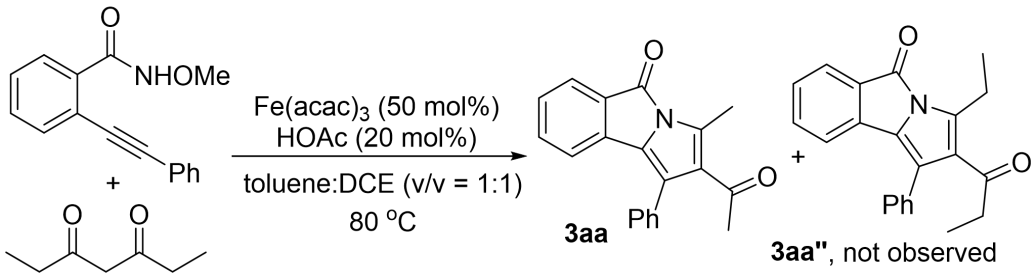


$Fe(acac)_3$: IR (KBr): 2995, 2961, 2919, 1520, 1363, 1274, 1189, 1023, 929, 801, 771, 665, 559, 433 cm^{-1};

3a-A: IR (KBr): 3245, 3067, 2920, 2226, 1677, 1571, 1524, 1369, 1295, 1274, 1209, 1120, 1085, 1023, 926, 893, 855, 802, 770, 750, 749, 665, 559, 433 cm^{-1};

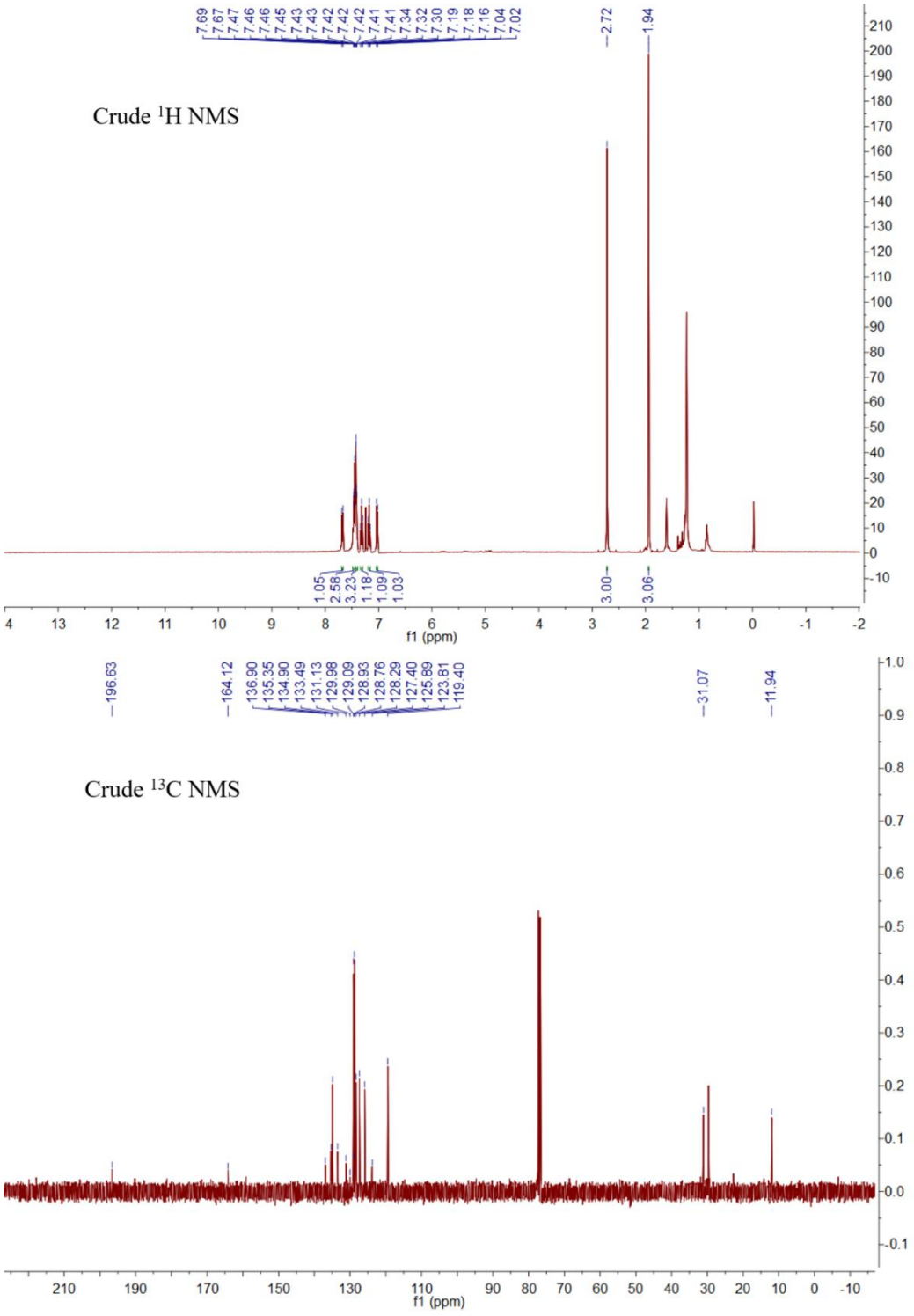
3a: IR (KBr): 3244, 3067, 2944, 2226, 1676, 1595, 1394, 1294, 1209, 893, 807, 750, 702 cm^{-1}



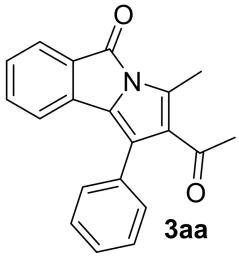


^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, J = 7.4 Hz, 1H), 7.46 (m, 3H), 7.43 – 7.39 (m, 3H), 7.32 (t, 1H), 7.18 (t, 1H), 7.03 (d, J = 7.6 Hz, 1H), 2.72 (s, 3H), 1.94 (s, 3H). ^{13}C

NMR (100 MHz, $CDCl_3$) δ 196.6, 164.1, 136.9, 135.4, 134.9, 133.5, 131.1, 130.0, 129.2 – 128.7, 128.3, 127.4, 125.9, 123.8, 119.4, 31.1, 11.9.

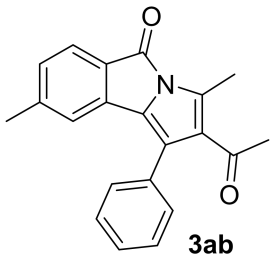


5. Characterization data



3aa

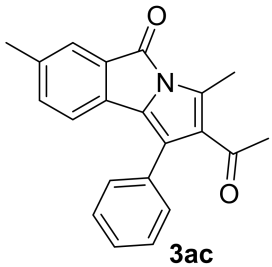
2-acetyl-3-methyl-1-phenyl-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3aa): Following the general procedure, the reaction of *N*-methoxy-2-(phenylethynyl)benzamide (50.2 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3aa** 51.2 mg (85% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (d, *J* = 7.5 Hz, 1H), 7.49 – 7.40 (m, 5H), 7.30 (t, 1H), 7.15 (t, 1H), 7.02 (d, *J* = 7.3 Hz, 1H), 2.70 (s, 3H), 1.94 (s, 3H). ^{13}C{^{1}H}NMR (100 MHz, $CDCl_3$) δ 196.5, 164.0, 136.9, 135.3, 134.9, 133.5, 131.1, 130.0, 129.2 – 128.7, 128.3, 127.4, 125.8, 123.8, 119.4, 31.1, 11.9; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{20}H_{16}NO_2$ 302.1204; Found: 302.1206



3ab

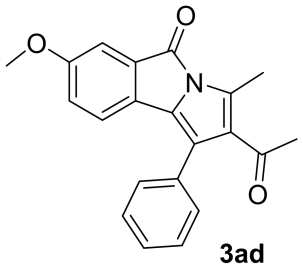
2-acetyl-3,8-dimethyl-1-phenyl-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3ab): Following the general procedure, the reaction of *N*-methoxy-4-methyl-2-(phenylethynyl)benzamide (53.0 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3ab** 51.7 mg (82% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 – 7.40 (m, 4H), 7.35 (d, *J* = 8.1 Hz, 2H), 7.13 (d, *J* = 7.8 Hz, 1H), 6.91 (d, *J* = 7.8 Hz, 1H), 2.69 (s, 3H), 2.32 (s, 3H), 1.99 (s, 3H). ^{13}C{^{1}H}NMR (100 MHz, $CDCl_3$) δ 196.0, 164.1, 138.0, 136.7, 135.6, 134.1,

132.4, 132.2, 131.3, 130.4, 129.0, 128.6, 126.5, 121.6, 119.2, 31.2, 21.2, 11.9; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{20}H_{18}NO_2$ 316.1328; Found: 316.1329



3ac

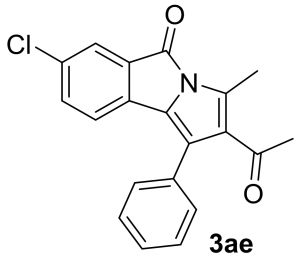
2-acetyl-3,7-dimethyl-1-phenyl-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3ac): Following the general procedure, the reaction of *N*-methoxy-5-methyl-2-(phenylethynyl)benzamide (53.0 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3ac** 51.7 mg (89% yield) as yellow solid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.43 (m, 7H), 7.11 (d, J = 7.7 Hz, 1H), 6.92 (d, J = 7.7 Hz, 1H), 2.70 (s, 3H), 2.31 (s, 3H), 1.94 (s, 3H). $^{13}C\{^1H\}$NMR (100 MHz, $CDCl_3$) δ 196.6, 164., 137.7 136.6, 135.5, 133.6, 132.7, 131.4, 130.1, 129.1, 128.7, 128.2, 126.3, 123.0, 119.2, 31.1, 21.2, 11.9; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{21}H_{18}NO_2$ 316.1316; Found: 316.1317



3ad

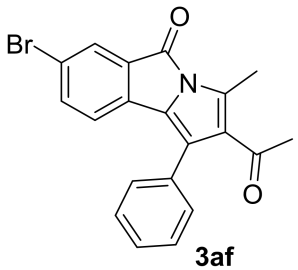
2-acetyl-7-methoxy-3-methyl-1-phenyl-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3ad): Following the general procedure, the reaction of *N*,5-dimethoxy-2-(phenylethynyl)benzamide (56.2 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3ad** 53.0 mg (80% yield) as yellow solid; ^1H

NMR (400 MHz, $CDCl_3$) δ 7.45 (d, *J* = 6.0 Hz, 2H), 7.42 – 7.38 (m, 4H), 7.19 (d, *J* = 2.4 Hz, 1H), 6.96 (d, *J* = 8.4 Hz, 1H), 6.85 (m, 1H), 3.80 (s, 3H), 2.69 (s, 3H), 1.95 (s, 3H).$^{13}C\{^1H\}$NMR (100 MHz, $CDCl_3$) δ 196.6, 164.0, 159.5, 136.6, 133.7, 132.7, 130.2, 129.1, 128.7, 128.1, 122.2, 121.3, 120.5, 110.2, 55.7, 31.0, 11.9 ; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{21}H_{18}NO_3$ 332.1310; Found: 332.1311



3ae

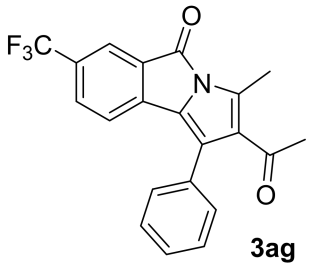
2-acetyl-7-chloro-3-methyl-1-phenyl-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3ae): Following the general procedure, the reaction of 5-chloro-*N*-methoxy-2-(phenylethynyl)benzamide (57.0 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3ae** 52.3 mg (78% yield) as yellow solid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, *J* = 1.7 Hz, 1H), 7.49 – 7.45 (m, 3H), 7.42 – 7.39 (m, 2H), 7.30 – 7.27 (m, 1H), 6.96 (d, *J* = 8.1 Hz, 1H), 2.71 (s, 3H), 1.95 (s, 3H). $^{13}C\{^1H\}$NMR (100 MHz, $CDCl_3$) δ 196.4, 162.8, 137.2, 134.7, 133.5, 133.1, 132.7, 129.1, 128.5, 126.0, 124.4, 120.3, 31.1, 11.9; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{20}H_{15}ClNO_2$ 336.0833; Found: 336.0832



3af

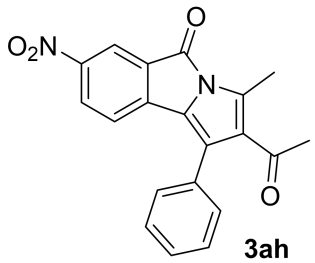
2-acetyl-7-bromo-3-methyl-1-phenyl-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3af): Following the general procedure, the reaction of

5-bromo-*N*-methoxy-2-(phenylethynyl)benzamide (65.8 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3af** 60.6 mg (80% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (s, 1H), 7.48 – 7.43 (m, 3H), 7.40 (d, *J* = 7.1 Hz, 3H), 6.90 (d, *J* = 8.1 Hz, 1H), 2.71 (s, 3H), 1.95 (s, 3H). ^{13}C{^{1}H}NMR (100 MHz, $CDCl_3$) δ 196.5, 162.6, 137.6, 137.1, 134.0, 133.1, 132.8, 130.2, 129.2, 128.9, 128.5, 124.5, 120.9, 120.6, 31.1, 12.0; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{20}H_{15}BrNO_2$ 380.0316; Found: 380.0317



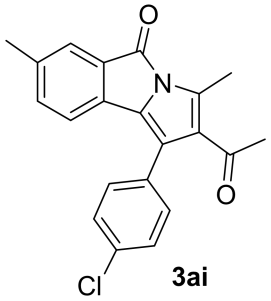
3ag

2-acetyl-3-methyl-1-phenyl-7-(trifluoromethyl)-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3ag): Following the general procedure, the reaction of N-methoxy-2-(phenylethynyl)-5-(trifluoromethyl)benzamide (63.8 mg, 0.2 mmol), Acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 25/1) to give **3ag** 51.7 mg (70% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (s, 1H), 7.58 (d, *J* = 7.9 Hz, 1H), 7.52 – 7.47 (m, 3H), 7.44 – 7.39 (m, 2H), 7.12 (d, *J* = 7.9 Hz, 1H), 2.75 (s, 3H), 1.97 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.4, 162.7, 138.2, 137.9, 132.9, 131.9, 131.5, 129.4, 128.9, 125.9, 122.9, 119.5, 31.1, 12.0.^{19}F NMR (376 MHz, $CDCl_3$) δ -62.8; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{21}H_{15}F_3NO_2$ 370.0871; Found: 370.0871



3ah

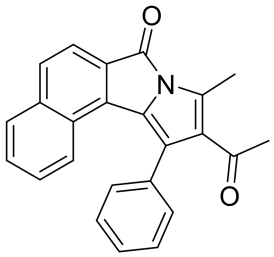
2-acetyl-3-methyl-7-nitro-1-phenyl-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3ah): Following the general procedure, the reaction of *N*-methoxy-5-nitro-2-(phenylethynyl)benzamide (59.2 mg, 0.2 mmol), Acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3ah** 51.2 mg (74% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 8.2 Hz, 1H), 7.85 (d, *J* = 8.2 Hz, 1H), 7.80 (d, *J* = 1.8 Hz, 1H), 7.52 (m, 3H), 7.45 – 7.41 (m, 2H), 2.74 (s, 3H), 1.97 (s, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 196.4, 152.3, 137.8, 136.4, 135.7, 132.3, 129.8, 129.2, 128.8, 126.6, 122.4, 114.1, 105.0, 31.1, 12.0. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{20}H_{15}N_2O_4$ 347.0854; Found: 347.0854



3ai

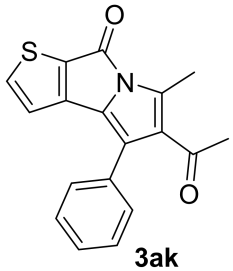
2-acetyl-1-(4-chlorophenyl)-3,7-dimethyl-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3ai): Following the general procedure, the reaction of 2-((4-chlorophenyl)ethynyl)-*N*-methoxy-5-methylbenzamide (59.8 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3ai** 55.2 mg (79% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 7.7 Hz, 1H), 7.47 (d, *J* = 6.7 Hz, 2H),

7.42 (d, J = 7.6 Hz, 3H), 6.98 (d, J = 7.7 Hz, 1H), 2.72 (s, 3H), 2.26 (s, 3H), 1.94 (s, 3H). $^{13}C\{^{1}H\}$NMR (100 MHz, $CDCl_3$) δ 196.6, 164.1, 146.2, 136.9, 135.7, 133.6, 129.8, 129.1, 128.8, 128.6, 128.2, 125.8, 123.6, 120.0, 31.1, 22.2, 11.9; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{21}H_{17}ClNO_2$ 350.0965; Found: 350.0966



3aj

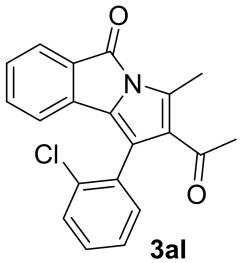
2-acetyl-3-methyl-1-phenyl-5*H*-benzo[*f*]pyrrolo[2,1-*a*]isoindol-5-one (3aj): Following the general procedure, the reaction of *N*-methoxy-3-(phenylethynyl)-2-naphthamide (60.2 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3aj** 43.5 mg (62% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (m, 3H), 7.61 (d, J = 8.3 Hz, 1H), 7.53 (d, J = 6.8 Hz, 4H), 7.45 (d, J = 6.4 Hz, 2H), 7.37 (t, 1H), 6.93 (t, 1H), 6.62 (d, J = 8.6 Hz, 1H), 2.79 (s, 3H), 1.83 (s, 3H).$^{13}C\{^{1}H\}$NMR (100 MHz, $CDCl_3$) δ 196.1, 164.7, 138.3, 137.6, 136.0, 135.5, 131.1, 130.5, 129.4, 129.0 – 128.5, 128.3, 128.1, 127.8, 127.1, 126.9, 125.8, 125.6, 120.57, 31.2, 12.3; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{24}H_{18}NO_2$ 352.1355; Found: 352.1356



3ak

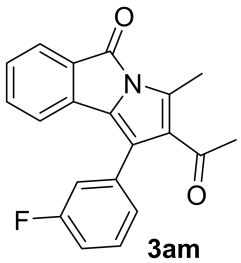
5-acetyl-6-methyl-4-phenyl-8*H*-thieno[3,2-*a*]pyrrolizin-8-one (3ak): Following the general procedure, the reaction of

N-methoxy-3-(phenylethynyl)thiophene-2-carboxamide (51.4 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3ak** 38.1 mg (62% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 (d, *J* = 4.7 Hz, 1H), 7.41 (m, 5H), 6.77 (d, *J* = 4.7 Hz, 1H), 2.67 (s, 3H), 2.01 (s, 3H).^{13}C{^{1}H}NMR (100 MHz, $CDCl_3$) δ 196.4, 158.8, 147.8, 140.1, 139.6, 133.3, 130.3, 128.7, 128.3, 127.4, 126.8, 123.3, 119.0, 31.0, 12.0; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{18}H_{14}NO_2S$ 308.0743; Found: 308.0744



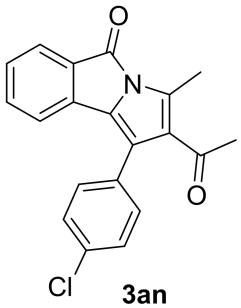
3al

2-acetyl-1-(2-chlorophenyl)-3-methyl-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3al): Following the general procedure, the reaction of 2-((2-chlorophenyl)ethynyl)-*N*-methoxybenzamide (57.0 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3al** 57.0 mg (85% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 7.5 Hz, 1H), 7.55 – 7.51 (m, 1H), 7.44 (d, *J* = 3.0 Hz, 1H), 7.40 – 7.37 (m, 2H), 7.32 (t, 1H), 7.19 (t, 1H), 6.77 (d, *J* = 7.6 Hz, 1H), 2.78 (s, 3H), 1.96 (s, 3H). ^{13}C{^{1}H}NMR (100 MHz, $CDCl_3$) δ 195.6, 164.1, 137.2, 135.0, 134.1, 132.8, 131.2, 130.8, 130.0, 128.6, 127.6, 127.2, 125.9, 120.1, 119.7, 30.1, 12.2; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{20}H_{15}ClNO_2$ 336.0831; Found: 336.0830



3am

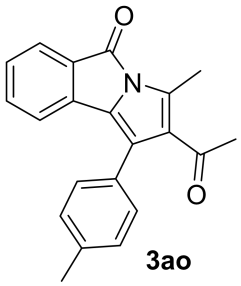
2-acetyl-1-(3-fluorophenyl)-3-methyl-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3am): Following the general procedure, the reaction of 2-((3-fluorophenyl)ethynyl)-N-methoxybenzamide (52.3 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3am** 31.9 mg (82% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 7.3 Hz, 1H), 7.45 (m, 1H), 7.35 (t, 1H), 7.16 (m, 5H), 7.03 (d, *J* = 7.4 Hz, 1H), 2.72 (s, 3H), 1.99 (s, 3H). ^{13}C{^{1}H}NMR (100 MHz, $CDCl_3$) δ 196.0, 164.0, 161.6, 137.0, 135.8, 135.0, 131.1, 130.4, 128.7, 127.7, 126.0, 125.0, 122.3, 119.4, 116.3, 116.1, 115.4, 115.2, 31.0, 11.9; ^{19}F NMR (376 MHz, $CDCl_3$) δ -150.8; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{20}H_{15}FNO_2$ 320.1123; Found: 320.1122



3an

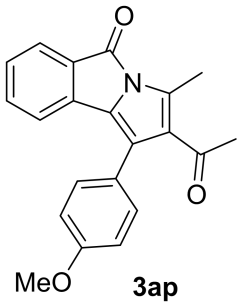
2-acetyl-1-(4-chlorophenyl)-3-methyl-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3an): Following the general procedure, the reaction of 2-((4-chlorophenyl)ethynyl)-*N*-methoxybenzamide (57.0 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3an** 50.3 mg (75% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.5 Hz, 1H), 7.46 (d, *J* = 8.1 Hz, 2H),

7.36 (m, 3H), 7.20 (t, 1H), 7.02 (d, J = 7.6 Hz, 1H), 2.73 (s, 3H), 2.00 (s, 3H). $^{13}C\{^{1}H\}$NMR (100 MHz, $CDCl_3$) δ 196.0, 137.0, 135.0, 134.3, 132.0, 131.1, 130.5, 130.2, 129.0, 128.7, 127.6, 126.0, 122.4, 119.4, 31.2, 12.0; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{20}H_{15}ClNO_2$ 336.0832; Found: 336.0830



3ao

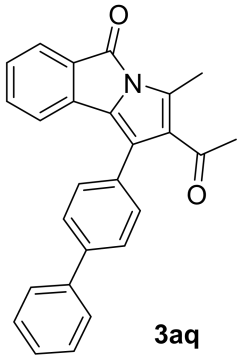
2-acetyl-3-methyl-1-(*p*-tolyl)-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3ao): Following the general procedure, the reaction of *N*-methoxy-2-(*p*-tolylethynyl)benzamide (53.0 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3ao** 501.7 mg (82% yield) as yellow solid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, J = 7.5 Hz, 1H), 7.35 – 7.27 (m, 6H), 7.19 – 7.15 (m, 1H), 7.06 (d, J = 7.6 Hz, 1H), 2.71 (s, 3H), 2.43 (s, 3H), 1.96 (s, 3H). $^{13}C\{^{1}H\}$NMR (100 MHz, $CDCl_3$) δ 196.8, 164.1, 138.2, 136.8, 135.4, 134.8, 131.1, 130.4, 129.8, 129.5, 129.0, 127.3, 125.7, 124.0, 119.4, 31.1, 21.4, 11.9; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{21}H_{18}NO_2$ 316.1362; Found: 316.1360



3ap

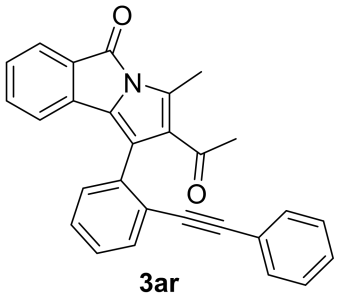
2-acetyl-1-(4-methoxyphenyl)-3-methyl-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3ap): Following the general procedure, the reaction of *N*-methoxy-2-((4-methoxyphenyl)ethynyl)benzamide (56.2 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%),

and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3ap** 51.7 mg (78% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 7.5 Hz, 1H), 7.35 – 7.30 (m, 4H), 7.16 (t, 1H), 7.05 (d, *J* = 7.6 Hz, 1H), 7.00 (d, *J* = 8.7 Hz, 2H), 3.87 (s, 3H), 2.71 (s, 3H), 1.97 (s, 3H). ^{13}C{^{1}H}NMR (100 MHz, $CDCl_3$) δ 196.7, 164.10, 159.6, 136.8, 135.4, 134.9, 131.1, 130.3, 129.8, 129.0, 127.2, 125.8, 125.5, 123.7, 119.3, 114.2, 55.3, 31.1, 11.9; HRMS (ESI-TOF) m/z: [M + H]$^+$ Calcd for $C_{21}H_{18}NO_3$ 332.1312; Found: 332.1311



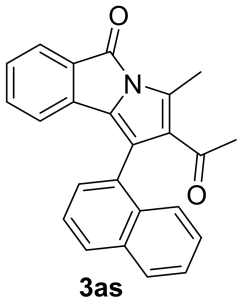
3aq

1-([1,1'-biphenyl]-4-yl)-2-acetyl-3-methyl-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3aq): Following the general procedure, the reaction of 2-([1,1'-biphenyl]-4-ylethynyl)-*N*-methoxybenzamide (65.4 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3aq** 58.8 mg (78% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.3 Hz, 2H), 7.68 (d, *J* = 7.8 Hz, 2H), 7.51 (d, *J* = 7.6 Hz, 2H), 7.47 (d, *J* = 7.2 Hz, 2H), 7.40 – 7.33 (m, 3H), 7.20 (t, 1H), 7.15 (d, *J* = 7.5 Hz, 1H), 2.75 (s, 3H), 2.04 (s, 3H). ^{13}C{^{1}H}NMR (100 MHz, $CDCl_3$) δ 196.6, 164.1, 141.0, 140.0, 136.9, 135.3, 134.9, 132.4, 131.2, 130.1, 129.6, 129.0, 127.7, 127.4, 127.0, 125.9, 123.5, 119.5, 31.2, 12.0; HRMS (ESI-TOF) m/z: [M + H]$^+$ Calcd for $C_{26}H_{20}NO_2$ 378.1520; Found: 378.1520



3ar

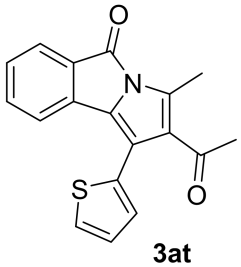
2-acetyl-3-methyl-1-(2-(phenylethynyl)phenyl)-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3ar): Following the general procedure, the reaction of *N*-methoxy-2-((2-(phenylethynyl)phenyl)ethynyl)benzamide (70.2 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3ar** 72.2 mg (90% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 6.7 Hz, 2H), 7.49 (s, 1H), 7.47 – 7.43 (m, 2H), 7.33 (t, 1H), 7.24 (s, 5H), 7.18 (t, 1H), 6.98 (d, *J* = 7.5 Hz, 1H), 2.80 (s, 3H), 2.01 (s, 3H). ^{13}C{^{1}H}NMR (100 MHz, $CDCl_3$) δ 196.1, 164.3, 136.7, 135.9, 135.3, 134.9, 132.7, 131.4, 131.2, 130.7, 129.7, 129.2, 128.4, 127.4, 125.8, 123.6, 122.7, 122.1, 120.0, 93.9, 87.9, 30.4, 12.1; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{28}H_{20}NO_2$ 402.1518; Found: 402.1519



3as

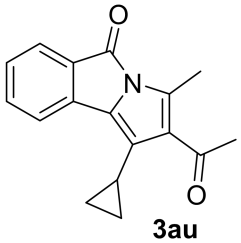
2-acetyl-3-methyl-1-(naphthalen-2-yl)-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3as): Following the general procedure, the reaction of *N*-methoxy-2-(naphthalen-2-ylethynyl)benzamide (60.2 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3as** 58.3 mg (83% yield) as yellow solid; ^{1}H

NMR (400 MHz, $CDCl_3$) δ 7.94 (d, *J* = 5.3 Hz, 2H), 7.88 (d, *J* = 7.8 Hz, 1H), 7.69 (d, *J* = 6.8 Hz, 1H), 7.54 (m, 3H), 7.47 (d, *J* = 6.7 Hz, 1H), 7.19 – 7.12 (m, 2H), 6.47 (d, *J* = 6.4 Hz, 1H), 2.87 – 2.85 (s, 3H), 1.66 – 1.64 (s, 3H). $^{13}C\{^1H\}$NMR (100 MHz, $CDCl_3$) δ 196.3, 164.2, 137.7, 135.2, 134.9, 133.7, 132.2, 131.1, 128.9, 128.5, 127.4, 127.0, 126.4, 125.8, 125.5, 121.2, 119.8, 105.1 – 104.9, 30.2, 12.3; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{24}H_{18}NO_2$ 352.1362; Found: 352.1361



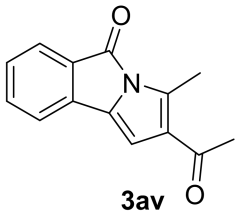
3at

2-acetyl-3-methyl-1-(thiophen-2-yl)-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3at): Following the general procedure, the reaction of *N*-methoxy-2-(thiophen-2-ylethynyl)benzamide (51.4 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3at** 47.9 mg (78% yield) as yellow solid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 7.5 Hz, 1H), 7.46 – 7.43 (m, 1H), 7.37 (t, 1H), 7.19 (m, 2H), 7.16 – 7.11 (m, 2H), 2.72 (s, 3H), 2.06 (s, 3H). $^{13}C\{^1H\}$NMR (100 MHz, $CDCl_3$) δ 196.3, 163.9, 136.9, 135.0, 133.6, 131.5, 131.0, 129.3, 128.3, 127.7, 126.8, 125.9, 119.9, 115.5, 77.3, 77.0, 76.7, 30.6, 12.0; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{18}H_{14}NO_2S$ 308.0767; Found: 308.0767



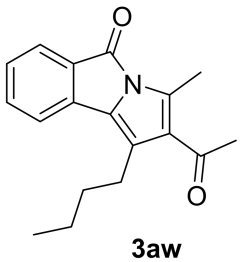
3au

2-acetyl-1-cyclopropyl-3-methyl-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3au): Following the general procedure, the reaction of 2-(cyclopropylethynyl)-*N*-methoxybenzamide (43.0 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3au** 36.6 mg (69% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, *J* = 7.4 Hz, 1H), 7.67 (d, *J* = 7.4 Hz, 1H), 7.52 (t, 1H), 7.21 (t, 1H), 2.69 (s, 3H), 2.58 (s, 3H), 1.90 – 1.84 (m, 1H), 1.08 (m, 2H), 0.66 (m, 2H). ^{13}C{^{1}H}NMR (100 MHz, $CDCl_3$) δ 196.3, 164.2, 137.1, 135.2, 134.8, 131.4, 131.2, 129.8, 126.9, 125.6, 121.3, 31.3, 12.2, 8.4, 7.8; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{17}H_{18}NO_2$ 266.1208; Found: 266.1207



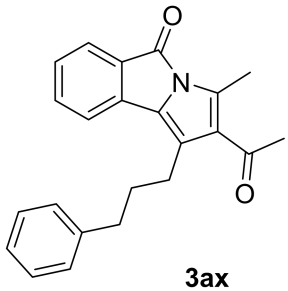
3av

2-acetyl-3-methyl-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3av): Following the general procedure, the reaction of *N*-methoxy-2-((trimethylsilyl)ethynyl)benzamide (49.4 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3av** 31.5 mg (70% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 7.5 Hz, 1H), 7.49 (t, 1H), 7.32 (d, *J* = 7.5 Hz, 1H), 7.26 – 7.21 (m, 1H), 6.51 (s, 1H), 2.77 (s, 3H), 2.40 (s, 3H). ^{13}C{^{1}H}NMR (100 MHz, $CDCl_3$) δ 194.6, 163.9 – 163.7, 137.4, 135.4, 135.1, 132.8, 131.1, 127.9, 127.5, 125.9, 119.7, 106.9, 29.2, 12.0; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{14}H_{12}NO_2$ 226.0890; Found: 226.0891



3aw

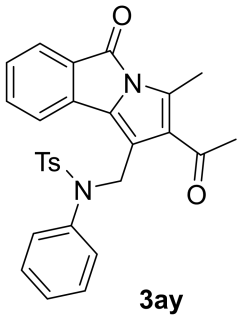
2-acetyl-1-butyl-3-methyl-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3aw): Following the general procedure, the reaction of 2-(hex-1-yn-1-yl)-*N*-methoxybenzamide (46.2 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3aw** 40.5 mg (72% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (d, *J* = 7.6 Hz, 1H), 7.47 (t, 1H), 7.35 (d, *J* = 7.5 Hz, 1H), 7.18 (t, 1H), 2.80 (t, 2H), 2.73 (s, 3H), 2.43 (s, 3H), 1.57 – 1.52 (m, 2H), 1.41 (m, 2H), 0.93 (t, 3H).^{13}C{^{1}H}NMR (100 MHz, $CDCl_3$) δ 195.2, 164.3, 137.1, 135.7, 134.9, 131.2, 130.0, 128.8, 126.7, 125.8, 119.6, 32.7, 31.3, 25.9, 22.8, 14.0, 12.7; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{18}H_{20}NO_2$ 282.1512; Found: 282.1513



3ax

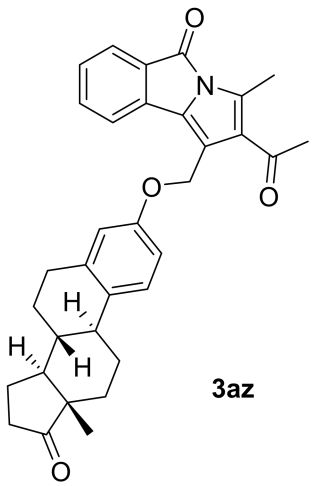
2-acetyl-3-methyl-1-(3-phenylpropyl)-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3ax): Following the general procedure, the reaction of *N*-methoxy-2-(5-phenylpent-1-yn-1-yl)benzamide (58.6 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3ax** 49.4 mg (72% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (d, *J* = 7.1 Hz, 1H), 7.37 (t, 1H), 7.29 (d, *J* = 7.4 Hz, 1H), 7.25 (s, 1H), 7.21 – 7.15 (m, 4H), 6.92 (d, *J* = 7.2 Hz, 1H), 2.84 – 2.79 (m, 2H),

2.72 (m, 5H), 2.42 (s, 3H), 1.95 – 1.90 (m, 2H). $^{13}C\{^{1}H\}$NMR (100 MHz, $CDCl_3$) δ 195.2, 164.4, 142.0, 137.1, 135.5, 135.0, 131.1, 128.7, 128.3, 126.7, 125.8, 125.3, 119.7, 35.7, 31.9, 31.3, 25.4, 12.7; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{23}H_{22}NO_2$ 344.1672; Found: 344.1673



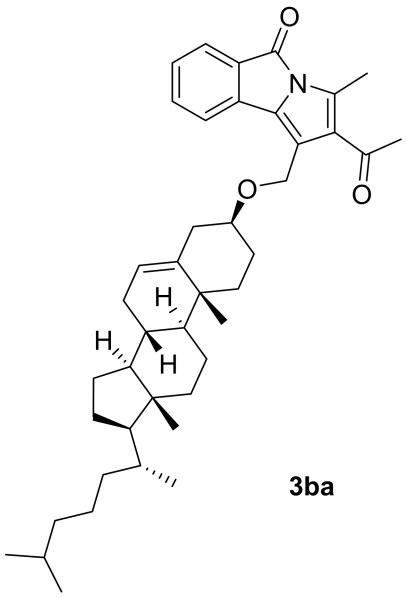
3ay

***N*-((2-acetyl-3-methyl-5-oxo-5*H*-pyrrolo[2,1-*a*]isoindol-1-yl)methyl)-4-methyl-*N*-phenylbenzenesulfonamide (3ay):** Following the general procedure, the reaction of *N*-methoxy-2-(3-((4-methyl-*N*-phenylphenyl)sulfonamido)prop-1-yn-1-yl)benzamide (86.8 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3ay** 79.4 mg (82% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 – 8.05 (d, 1H), 7.68 – 7.63 (m, 2H), 7.45 (d, *J* = 4.8 Hz, 2H), 7.27 (d, *J* = 5.9 Hz, 3H), 7.11 (d, *J* = 7.7 Hz, 3H), 6.88 (d, *J* = 5.8 Hz, 2H), 5.09 (s, 2H), 2.58 (s, 3H), 2.44 (s, 3H), 2.22 (s, 3H). $^{13}C\{^{1}H\}$NMR (100 MHz, $CDCl_3$) δ 195.8, 143.8, 138.6, 135.8, 135.5, 134.6, 133.7, 130.7, 129.5, 128.4, 128.0, 127.7, 125.6, 122.7, 118.2, 43.8, 31.2, 21.6, 12.5; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{28}H_{25}N_2O_4S$ 485.1552; Found: 485.1551



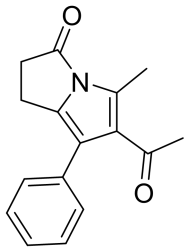
3az

2-acetyl-3-methyl-1-((((8*R*,9*S*,13*S*,14*S*)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta[*a*]phenanthren-3-yl)oxy)methyl)-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3az): Following the general procedure, the reaction of *N*-methoxy-2-(3-(((8*R*,9*S*,13*S*,14*S*)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta[*a*]phenanthren-3-yl)oxy)prop-1-yn-1-yl)benzamide (91.4 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3az**62.9 mg (62% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, *J* = 7.5 Hz, 1H), 7.35 (m, 2H), 7.19 (m, 2H), 7.14 (t, 2H), 2.97 – 2.93 (m, 2H), 2.70 (s, 3H), 2.58 – 2.41 (m, 3H), 2.36 (d, *J* = 10.5 Hz, 1H), 2.21 – 2.05 (m, 4H), 2.00 (s, 3H), 1.66 (m, 3H), 1.60 – 1.53 (m, 3H), 0.95 (s, 3H). ^{13}C{^{1}H}NMR (100 MHz, $CDCl_3$) δ 220.67, 196.88, 164.09, 139.90, 136.76, 135.46, 134.82, 131.14, 130.63, 129.75, 129.47, 129.06, 127.28, 126.37, 125.84, 125.64, 124.01, 119.45, 50.53, 47.95, 44.42, 37.99, 35.82, 31.68 – 31.10, 29.30, 26.44, 25.63, 21.58, 13.88, 11.88; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{33}H_{34}NO_4$ 508.2509; Found: 508.2508



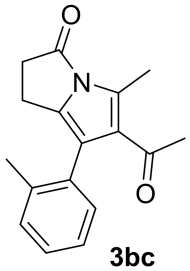
3ba

2-acetyl-1-((((3*S*,8*S*,9*S*,10*R*,13*R*,14*S*,17*R*)-10,13-dimethyl-17-((*R*)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl)oxy)methyl)-3-methyl-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3ba): Following the general procedure, the reaction of 2-(3-(((3*S*,8*S*,9*S*,10*R*,13*R*,14*S*,17*R*)-10,13-dimethyl-17-((*R*)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[a]phenanthren-3-yl)oxy)prop-1-yn-1-yl)-*N*-methoxybenzamide (114.7 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3ba** 106.0 mg (85% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.6 Hz, 1H), 7.66 (d, *J* = 7.5 Hz, 1H), 7.49 (t, 1H), 7.21 (t, 1H), 5.36 (s, 1H), 4.78 (s, 1H), 3.37 (s, 1H), 2.76 (s, 3H), 2.46 (s, 3H), 2.31 (t, 1H), 2.00 (m, 3H), 1.92 – 1.76 (m, 3H), 1.66 (s, 1H), 1.50 (m, 8H), 1.32 (s, 3H), 1.26 (d, *J* = 12.2 Hz, 3H), 1.10 (m, 7H), 1.01 (s, 5H), 0.90 (d, *J* = 6.0 Hz, 3H), 0.86 (d, *J* = 6.4 Hz, 5H), 0.67 (s, 2H). ^{13}C{^{1}H}NMR (100 MHz, $CDCl_3$) δ 195.2, 164.6, 140.6, 137.0, 135.7, 134.9, 131.0, 128.1, 127.1, 125.5, 122.7, 121.9, 79.5, 63.4, 56.7, 56.1, 50.1, 42.3, 39.7, 39.5, 39.0, 37.2, 36.9, 36.2, 35.8, 31.9, 31.1, 30.2, 29.7, 28.3, 28.0, 24.3, 23.8, 22.8, 22.6, 21.1, 19.4, 18.7, 12.6, 11.9; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{42}H_{58}NO_3$ 624.4432; Found: 624.4431



3bb

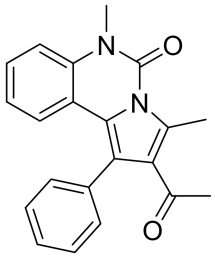
6-acetyl-5-methyl-7-phenyl-1,2-dihydro-3*H*-pyrrolizin-3-one (3bb): Following the general procedure, the reaction of *N*-methoxy-5-phenylpent-4-ynamide (40.6 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3bb** 35.4 mg (70% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (t, 2H), 7.32 (d, *J* = 7.1 Hz, 1H), 7.26 (d, *J* = 7.1 Hz, 2H), 3.02 (t, 2H), 2.99 (t, 2H), 2.68 (s, 3H), 2.03 (s, 3H). ^{13}C{^{1}H}NMR (100 MHz, $CDCl_3$) δ 198.3, 173.6, 134.9, 134.1, 129.3, 128.7, 127.1, 119.0, 34.8, 31.3, 18.2, 11.1; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{16}H_{16}NO_2$ 254.1209; Found: 254.1208



3bc

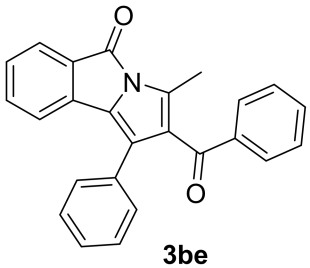
6-acetyl-5-methyl-7-(*o*-tolyl)-1,2-dihydro-3*H*-pyrrolizin-3-one (3bc): Following the general procedure, the reaction of *N*-methoxy-5-(o-tolyl)pent-4-ynamide (43.4 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3bc** 37.9 mg (71% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 – 7.25 (m, 2H), 7.20 (dd, *J* = 8.0, 3.4 Hz, 1H), 7.15 (d, *J* = 7.2 Hz, 1H), 3.03 (t, 2H), 2.83 (t, 2H), 2.75 (s, 3H), 2.17 (s, 3H), 1.83 (s, 3H). ^{13}C{^{1}H}NMR (100 MHz, $CDCl_3$) δ 197.6, 173.8, 137.0,

134.7, 133.9, 130.2, 129.8, 127.9, 126.0, 118.2, 105.0, 34.9, 30.5, 20.1, 18.0, 11.5; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{17}H_{18}NO_2$ 278.1360; Found: 278.1361



3bd

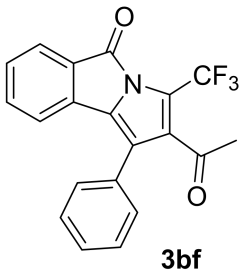
2-acetyl-3,6-dimethyl-1-phenylpyrrolo[1,2-*c*]quinazolin-5(6*H*)-one (3bd): Following the general procedure, the reaction of 3-methoxy-1-methyl-1-(2-(phenylethynyl)phenyl)urea (56.0 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and THF (2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 3/1) to give **3bd** 42.9 mg (65% yield) as yellow solid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.49 (d, *J* = 7.2 Hz, 3H), 7.40 – 7.37 (m, 2H), 7.22 (d, *J* = 7.2 Hz, 1H), 7.14 (d, *J* = 8.3 Hz, 1H), 7.10 (d, *J* = 8.1 Hz, 1H), 6.84 (t, 1H), 3.66 (s, 3H), 3.01 (s, 3H), 1.86 (s, 3H). $^{13}C\{^1H\}$NMR (100 MHz, $CDCl_3$) δ 198.8, 148.2, 135.8, 134.7, 133.8, 130.4, 129.3, 128.4, 128.2, 127.4, 123.5, 122.9, 120.2, 116.4, 114.07, 31.8, 30.8, 14.3; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{21}H_{19}N_2O_2$ 331.1468; Found: 331.1467



3be

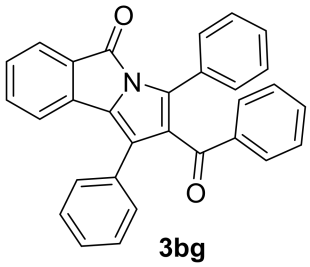
2-benzoyl-3-methyl-1-phenyl-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3be): Following the general procedure, the reaction of *N*-methoxy-2-(phenylethynyl)benzamide (50.2 mg, 0.2 mmol), 1-phenyl-1,3-butanedione (123.9 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3be**

42.1 mg (58% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 7.2 Hz, 3H), 7.37 (s, 3H), 7.29 (d, *J* = 6.7 Hz, 2H), 7.25 – 7.19 (m, 6H), 2.52 (s, 3H). ^{13}C{^{1}H}NMR (100 MHz, $CDCl_3$) δ 192.5, 164.0, 138.4, 135.6, 134.9, 132.6, 131.3, 129.5, 128.7, 128.3, 128.1, 127.6, 127.0, 125.9, 125.0, 119.6, 11.9; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{25}H_{18}NO_2$ 364.1360; Found: 364.1361



3bf

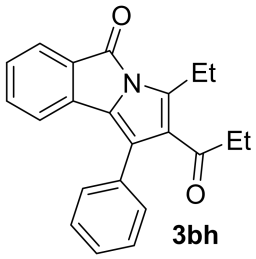
2-acetyl-1-phenyl-3-(trifluoromethyl)-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3bf): Following the general procedure, the reaction of *N*-methoxy-2-(phenylethynyl)benzamide (50.2 mg, 0.2 mmol), 1,1,1-trifluoro-2,4-pentanedione (46.2 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3bf** 44.0 mg (62% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 7.8 Hz, 1H), 7.45 (m, 6H), 7.33 – 7.29 (m, 2H), 2.21 (s, 3H). ^{13}C{^{1}H}NMR (100 MHz, $CDCl_3$) δ 197.6, 161.0, 135.2, 134.4, 133.8, 130.6, 129.4 – 128.8, 128.6, 126.7, 121.4, 120.6, 32.2. ^{19}F NMR (376 MHz, cdcl$_3$) δ -59.4; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{20}H_{13}F_3NO_2$ 356.0919; Found: 356.0919



3bg

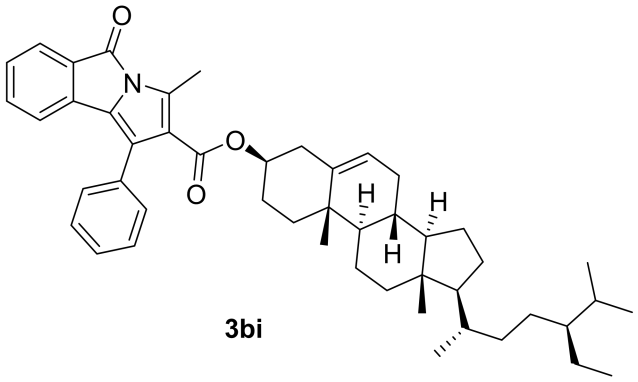
2-benzoyl-1,3-diphenyl-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3bg): Following the general procedure, the reaction of *N*-methoxy-2-(phenylethynyl)benzamide (50.2 mg, 0.2 mmol), dibenzoylmethane (67.3 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$

(24.6 mg, 50 mol%), and (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3bg** 38.2 mg (45% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 7.1 Hz, 2H), 7.55 (s, 2H), 7.43 (d, *J* = 6.7 Hz, 2H), 7.38 (s, 2H), 7.30 (d, *J* = 6.3 Hz, 3H), 7.28 (s, 1H), 7.24 (m, 4H), 7.17 (m, 3H). ^{13}C{^{1}H}NMR (100 MHz, $CDCl_3$) δ 192.8, 163.3, 137.6, 135.3, 134.9, 132.8, 132.1, 131.6, 130.0 – 129.7, 129.7 – 129.2, 128.9, 128.8 – 128.0, 127.8, 126.0, 125.3, 119.9; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{30}H_{20}NO_2$ 426.1512; Found: 426.1511



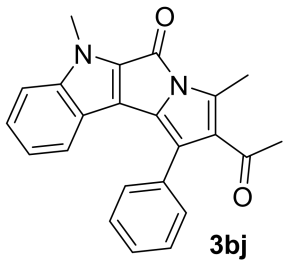
3bh

3-ethyl-1-phenyl-2-propionyl-5*H*-pyrrolo[2,1-*a*]isoindol-5-one (3bh): Following the general procedure, the reaction of *N*-methoxy-2-(phenylethynyl)benzamide (50.2 mg, 0.2 mmol), heptane-3,5-dione (38.4 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3bh** 54.6 mg (83% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 7.3 Hz, 1H), 7.51 – 7.45 (m, 2H), 7.44 – 7.39 (m, 3H), 7.32 (t, 1H), 7.18 (t, 1H), 7.06 (d, *J* = 7.6 Hz, 1H), 3.10 (m, 2H), 2.19 (m, 2H), 1.32 (t, 3H), 0.92 (t, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.4, 163.8, 142.1, 135.5, 134.8, 133.6 131.3, 130.0, 128.9, 128.3, 127.4, 125.9, 123.5, 119.4, 36.2, 19.0, 13.3, 8.4; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{22}H_{20}NO_2$ 330.1226; Found: 330.1224



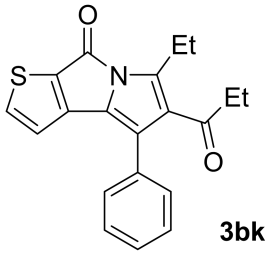
3bi

(3*R*,8*R*,9*R*,10*S*,13*S*,14*R*,17*S*)-17-((2*S*,5*S*)-5-ethyl-6-methylheptan-2-yl)-10,13-dimethyl-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl 3-methyl-5-oxo-1-phenyl-5*H*-pyrrolo[2,1-*a*]isoindole-2-carboxylate (3bi): Following the general procedure, the reaction of *N*-methoxy-2-(phenylethynyl)benzamide (50.2 mg, 0.2 mmol), (3*R*,8*R*,9*R*,10*S*,13*S*,14*R*,17*S*)-17-((2*S*,5*S*)-5-ethyl-6-methylheptan-2-yl)-10,13-dimethyl-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl 3-oxobutanoate (149.5 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 25/1) to give **3bi** 69.9 mg (50% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.6 Hz, 1H), 7.40 (m, 5H), 7.32 (t, 1H), 7.18 (t, 1H), 7.09 (d, *J* = 7.6 Hz, 1H), 5.32 (d, *J* = 3.4 Hz, 1H), 4.62 (t, 1H), 2.79 (d, *J* = 1.3 Hz, 3H), 2.09 – 1.89 (m, 3H), 1.84 – 1.75 (m, 2H), 1.60 – 1.29 (m, 9H), 1.28 – 1.18 (m, 3H), 1.07 (m, 9H), 0.91 (d, *J* = 9.3 Hz, 8H), 0.81 (m, 10H), 0.66 (d, *J* = 6.8 Hz, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 164.1, 163.7, 139.6, 138.5, 135.5, 134.8, 133.3, 131.1, 129.9, 129.5, 127.8, 127.3, 125.9, 122.5, 119.6, 73.8, 56.6, 56.0, 50.0, 45.8, 42.3, 39.7, 37.9, 36.9, 36.5, 36.2, 33.9, 31.8, 29.1, 28.2, 27.5, 26.0, 24.3, 23.0, 21.0, 20.2, 19.8, 19.2, 19.0, 18.8, 12.1 – 11.5; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{48}H_{62}NO_3$ 700.3651; Found: 700.3651



3bj

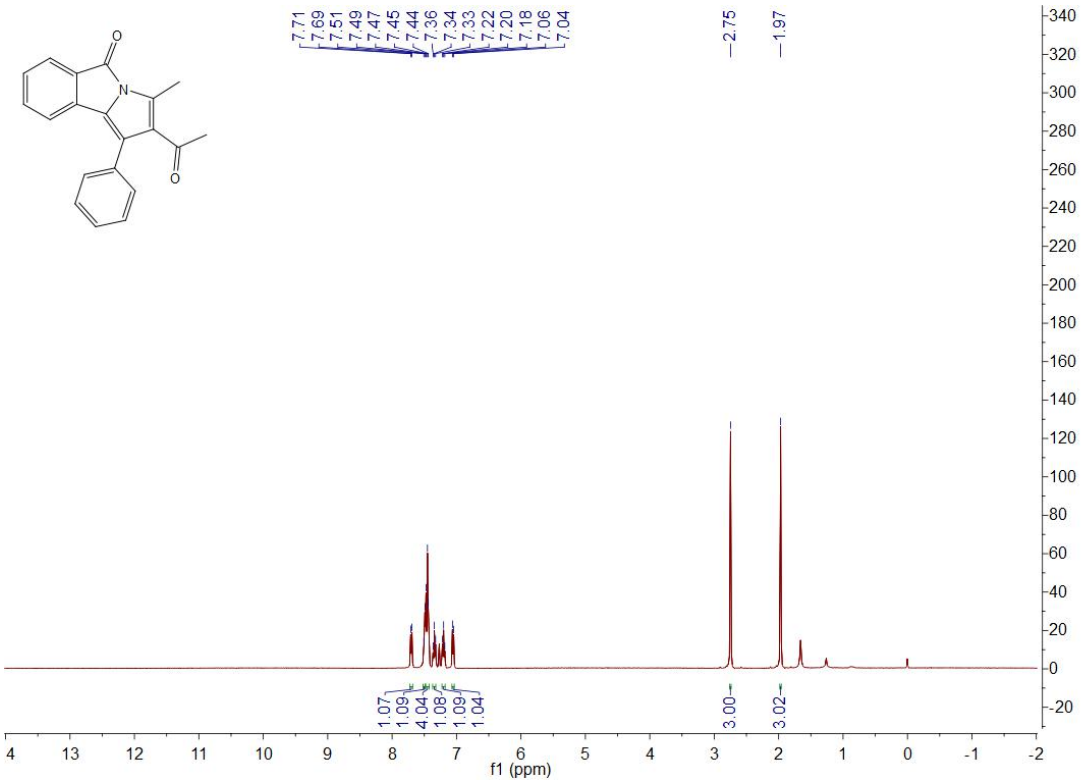
2-**acetyl-3,6-dimethyl-1-phenylpyrrolizino[2,1-*b*]indol-5(6*H*)-one (3bj):** Following the general procedure, the reaction of *N*-methoxy-1-methyl-3-(phenylethynyl)-1*H*-indole-2-carboxamide (60.8 mg, 0.2 mmol), acetylacetone (30.0 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3bj** 31.9 mg (45% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 – 7.45 (m, 6H), 7.24 (s, 1H), 6.96 (m, 1H), 6.85 (d, *J* = 8.2 Hz, 1H), 3.86 (s, 3H), 2.66 (s, 3H), 1.94 (s, 3H). ^{13}C{^{1}H}NMR (100 MHz, $CDCl_3$) δ 196.1, 157.6, 143.6, 139.0, 134.4, 132.0, 131.6, 129.4, 128.6, 128.3, 127.7, 126.2, 122.9, 122.6, 122.3, 121.7, 119.0, 111.0, 31.0, 30.5, 12.1; HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{23}H_{19}N_2O_2$ 355.1473; Found: 308.1472

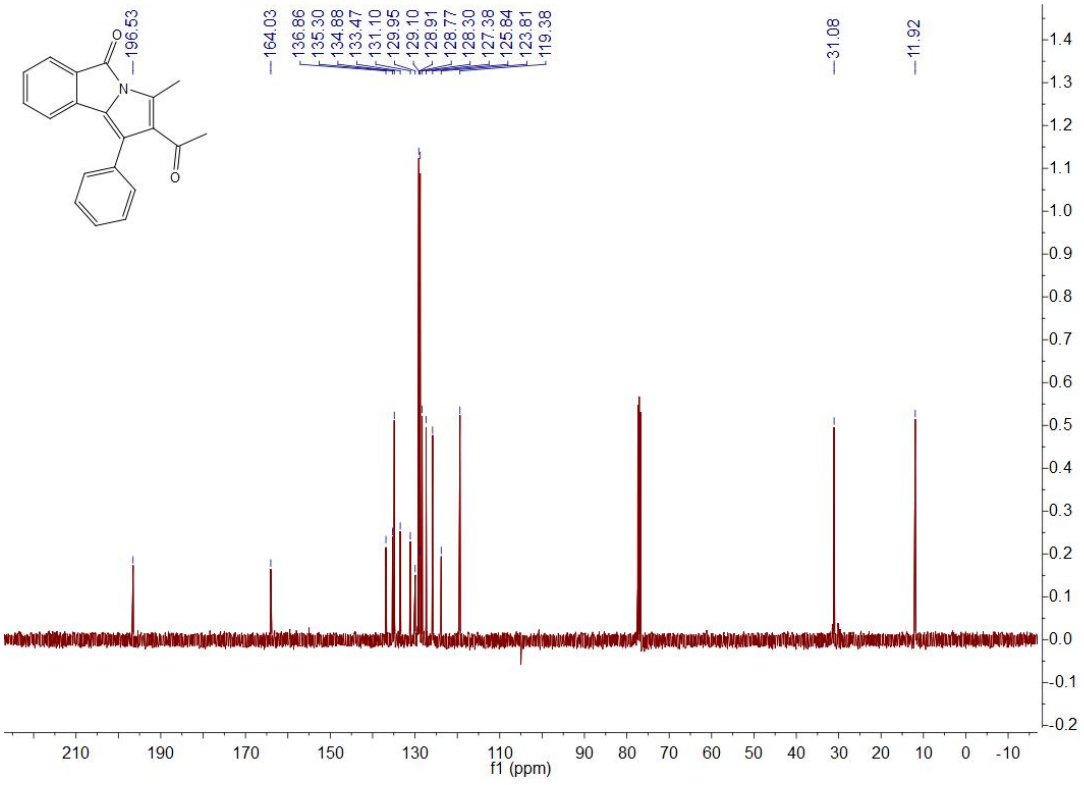


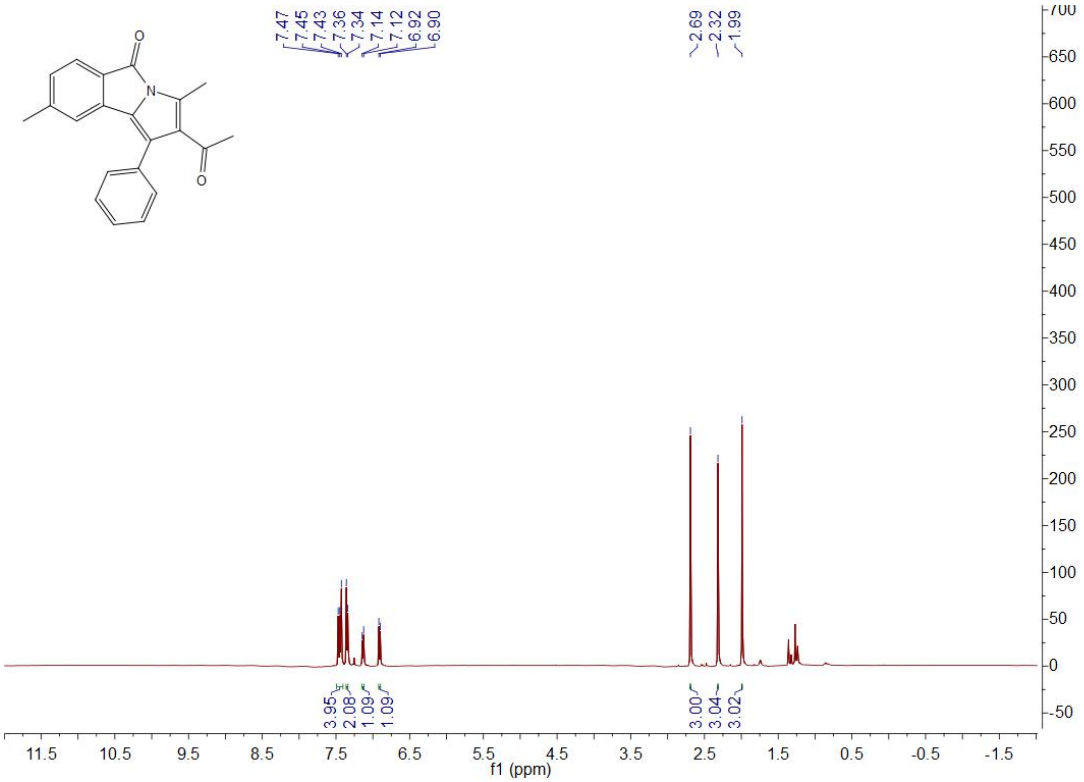
3bk

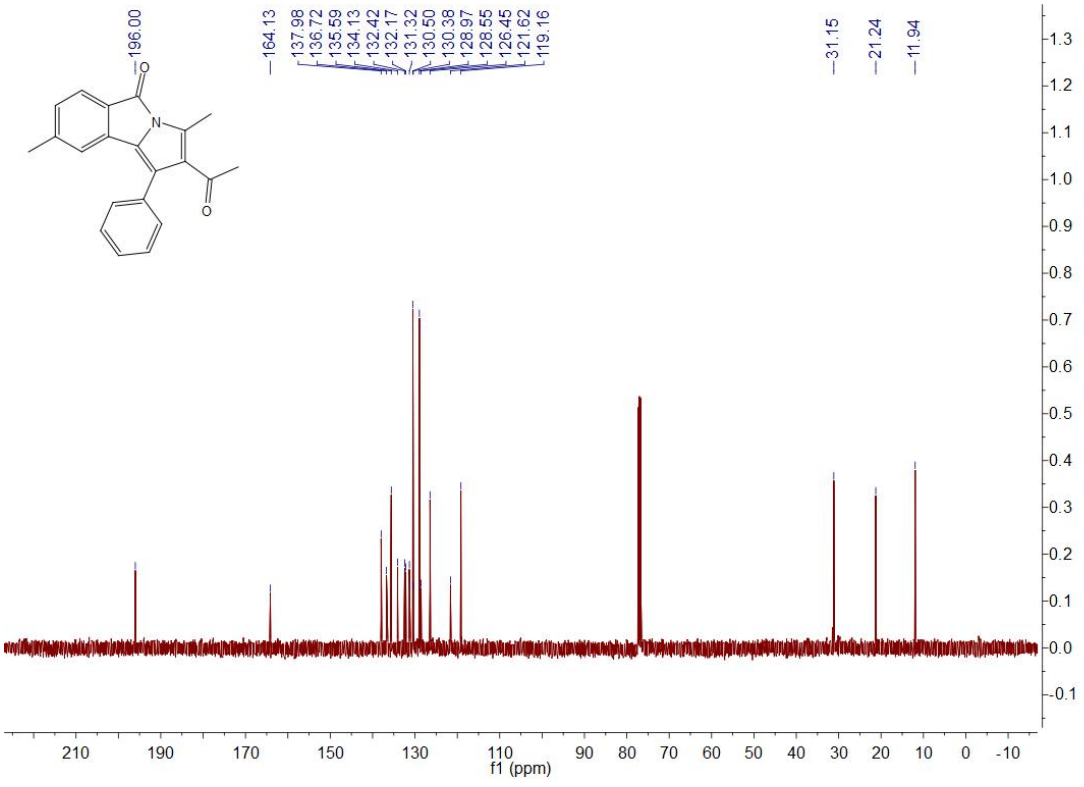
6-ethyl-4-phenyl-5-propionyl-8H-thieno[3,2-*a*]pyrrolizin-8-one (3bk): Following the general procedure, the reaction of *N*-methoxy-3-(phenylethynyl)thiophene-2-carboxamide (51.4 mg, 0.2 mmol), heptane-3,5-dione (38.4 mg, 1.5 equiv), HOAc (20 mol%), $Fe(OAc)_2$ (24.6 mg, 50 mol%), and solvent (toluene:DCE, 1:1, v/v, 2 mL) at 80 °C for 6 h. The product was purified by flash chromatography (PE/EA = 15/1) to give **3bk** 41.6 mg (62% yield) as yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (d, *J* = 4.8 Hz, 1H), 7.43 – 7.36 (m,

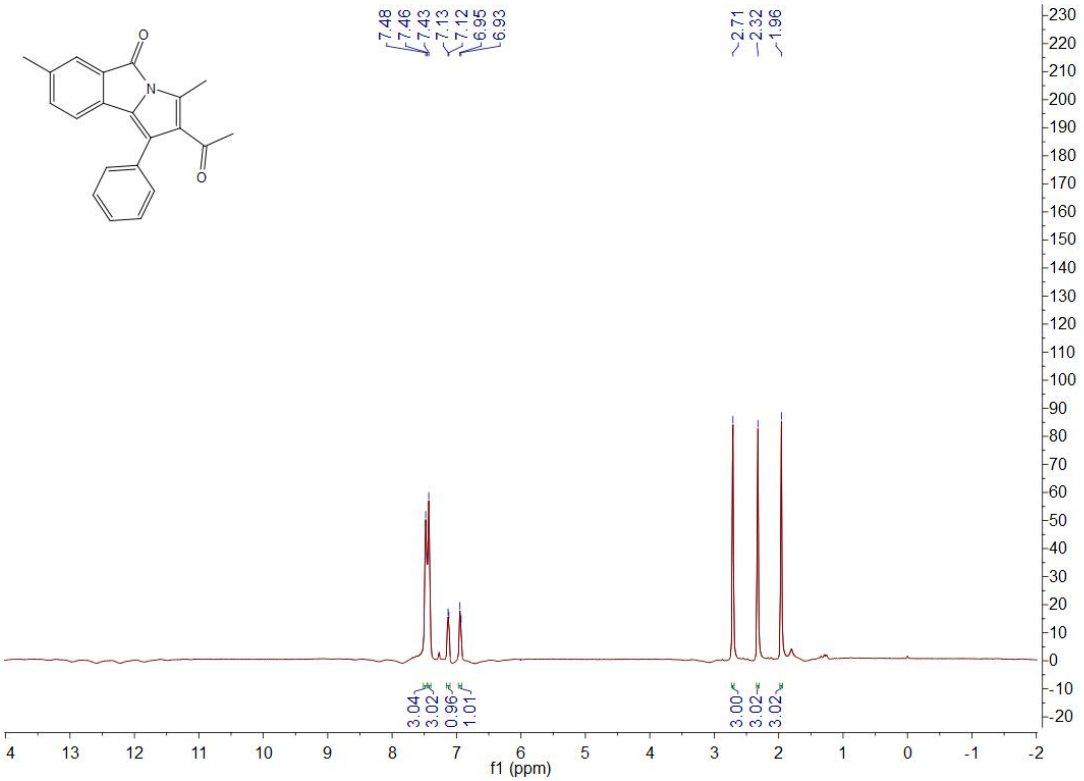
2H), 7.33 (t, 3H), 6.72 (d, J = 4.8 Hz, 1H), 2.97 (m, 2H), 2.19 (m, 2H), 1.25 (t, 3H), 0.90 (t, 3H). $^{13}C\{^{1}H\}$NMR (100 MHz, $CDCl_3$) δ 200.1, 158.4, 147.9, 145.1, 139.3, 133.3, 130.4, 128.7, 128.4, 128.2, 127.4, 125.9, 122.85 (s), 119.0, 36.1, 18.9, 13.0, 8.5.HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{20}H_{18}NO_2S$ 336.0871; Found: 336.0871

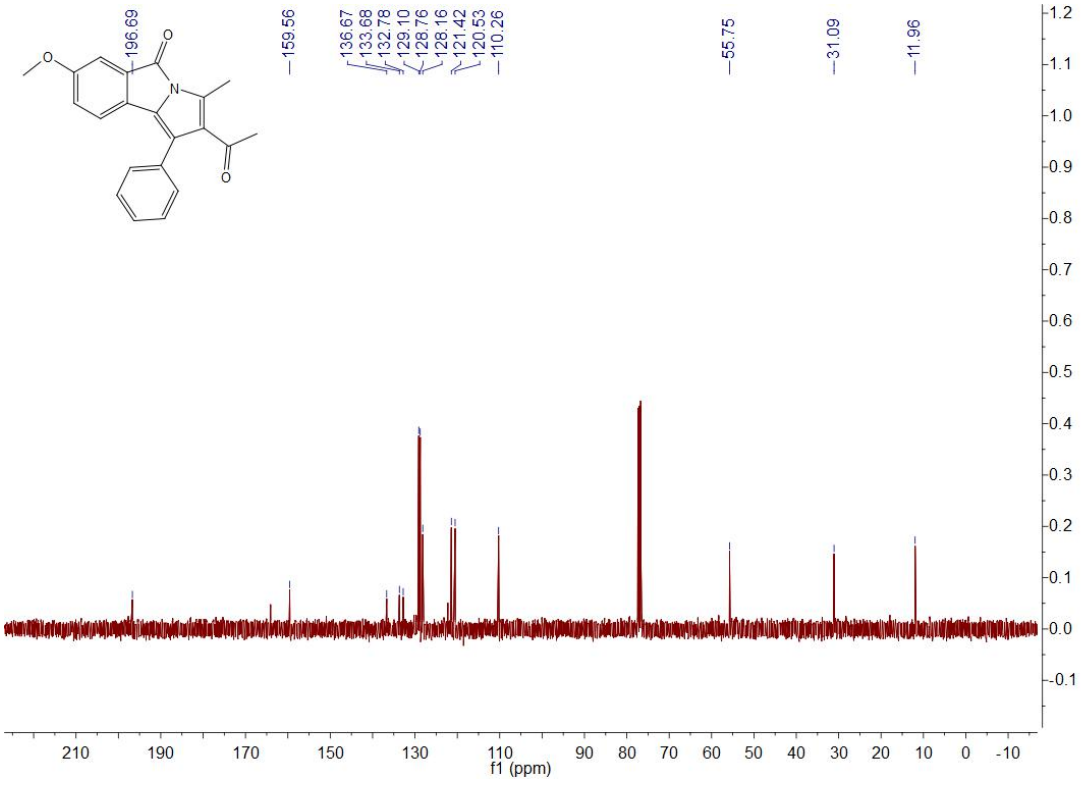

7.71
7.69
7.51
7.49
7.47
7.45
7.44
7.36
7.34
7.33
7.22
7.20
7.18
7.06
7.04
2.75
1.97
1.07
1.09
4.04
1.08
1.09
1.04
3.00
3.02
f1 (ppm)

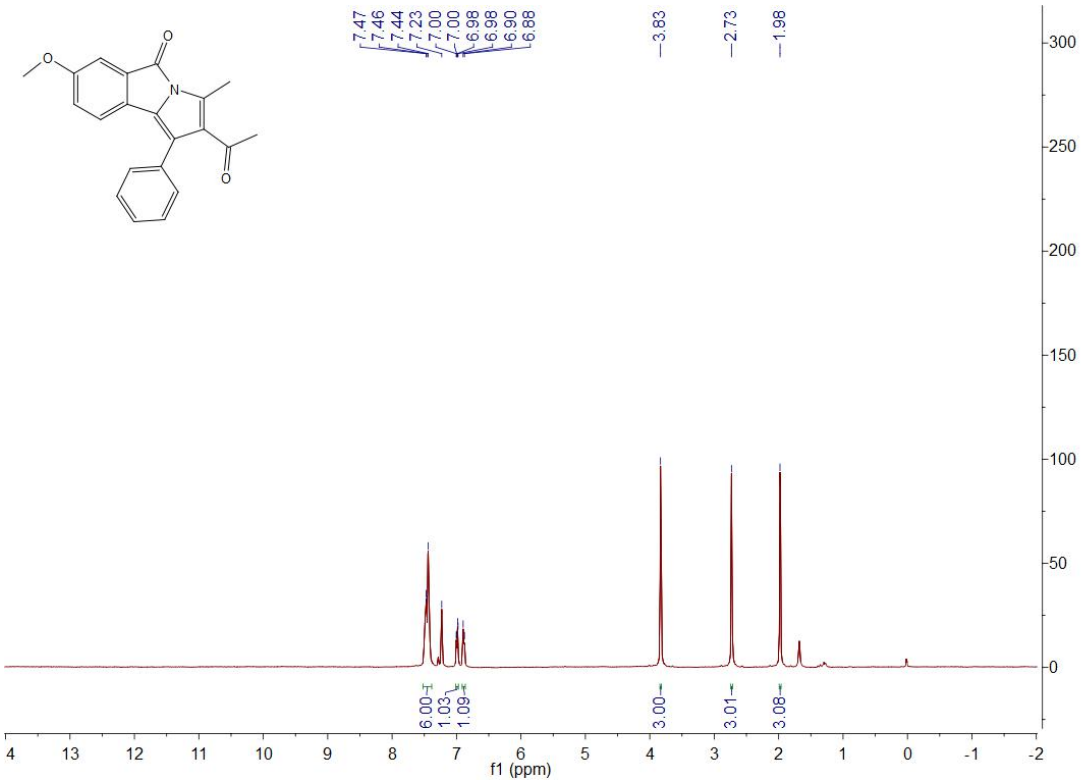

196.53
164.03
136.86
135.30
134.88
133.47
131.10
129.95
129.10
128.91
128.77
128.30
127.38
125.84
123.81
119.38
31.08
11.92
f1 (ppm)

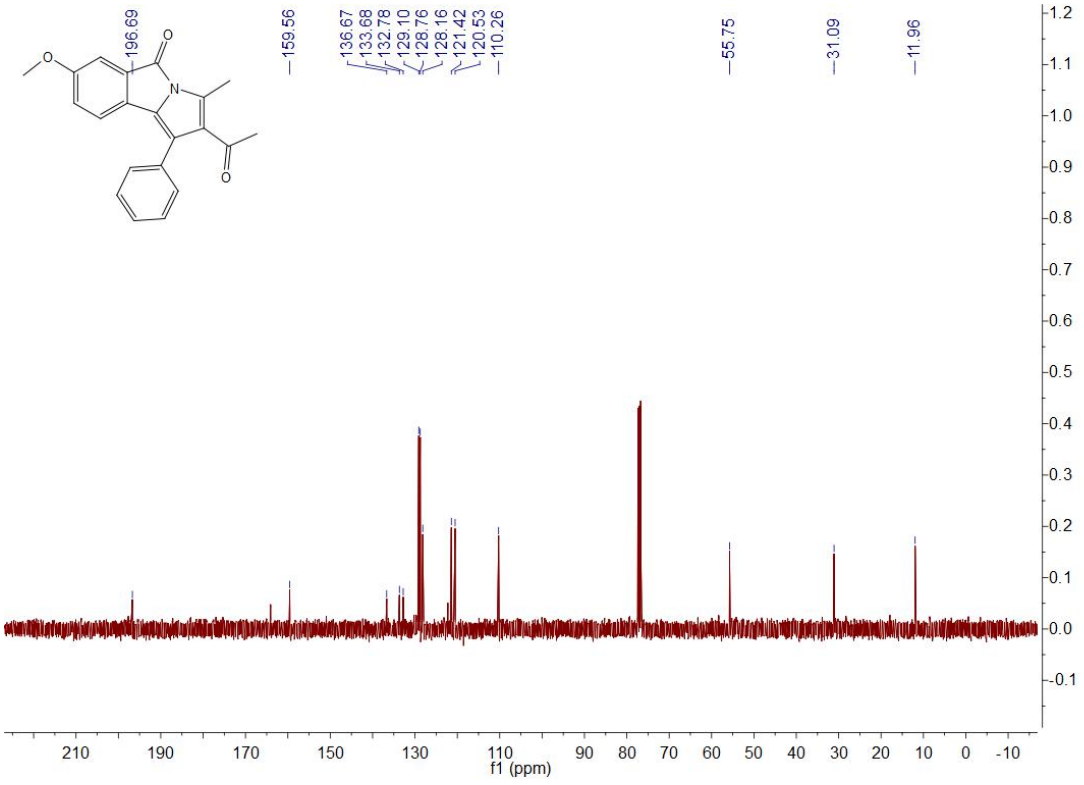

7.47
7.45
7.43
7.36
7.34
7.14
7.12
6.92
6.90
2.69
2.32
1.99
3.95
2.08
1.09
1.09
3.00
3.04
3.02
f1 (ppm)

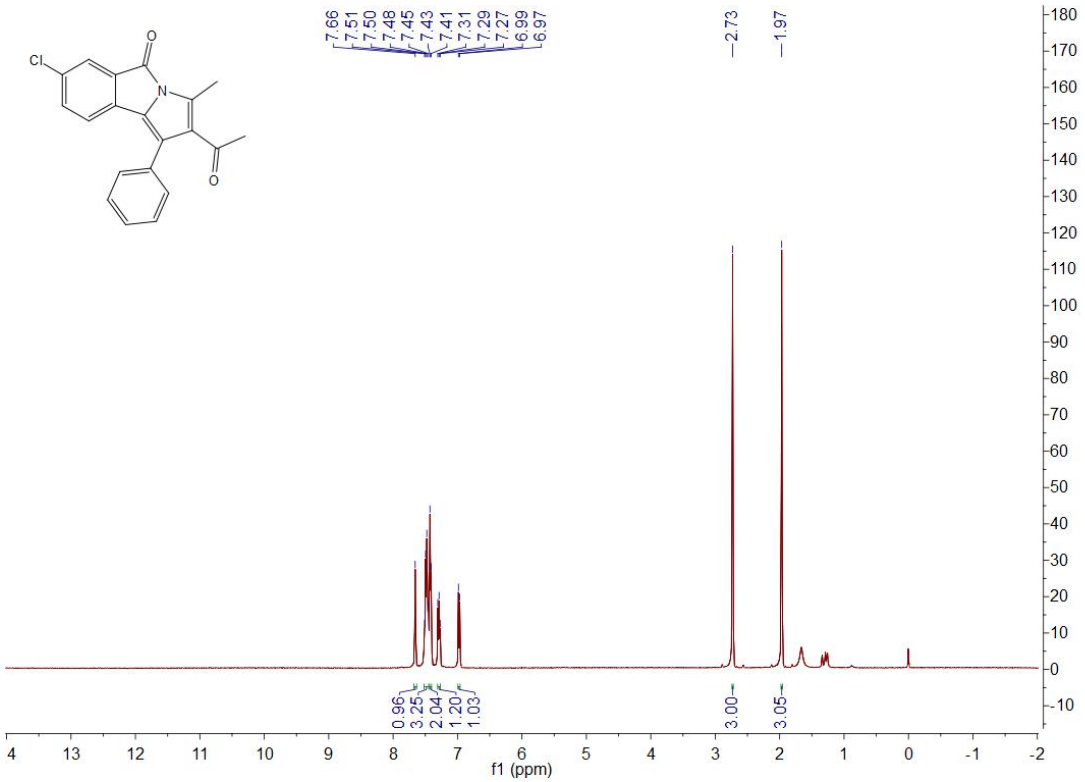

196.00
164.13
137.98
136.72
135.59
134.13
132.42
132.17
131.32
130.50
130.38
128.97
128.55
126.45
121.62
119.16
31.15
21.24
11.94
f1 (ppm)

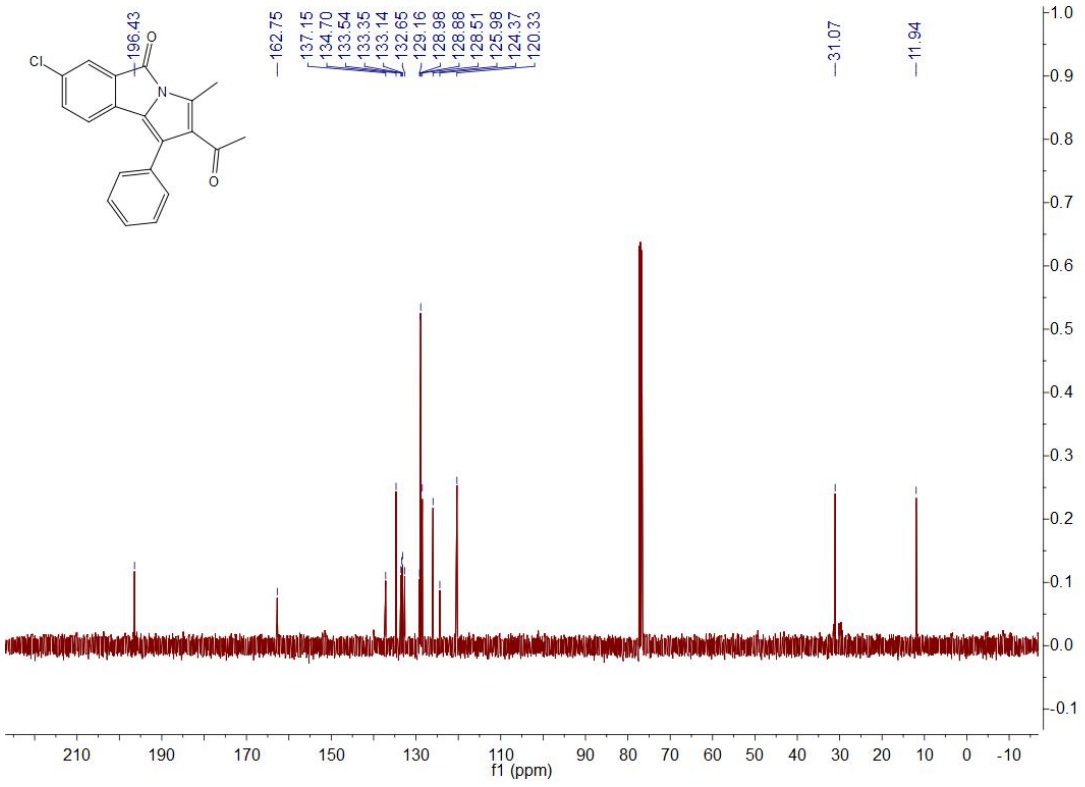

7.48
7.46
7.43
7.13
7.12
6.95
6.93
2.71
2.32
1.96
3.04
3.02
0.96
1.01
3.00
3.02
3.02
f1 (ppm)

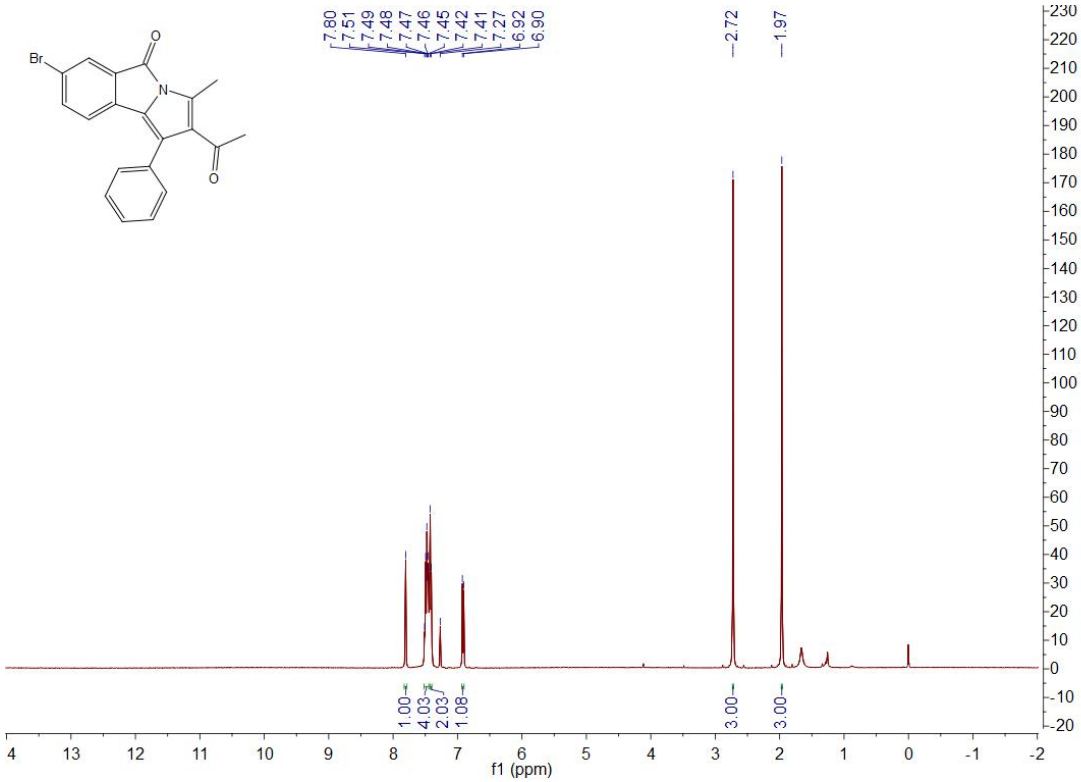

196.69
159.56
136.67
133.68
132.78
129.10
128.76
128.16
121.42
120.53
110.26
55.75
31.09
11.96
f1 (ppm)

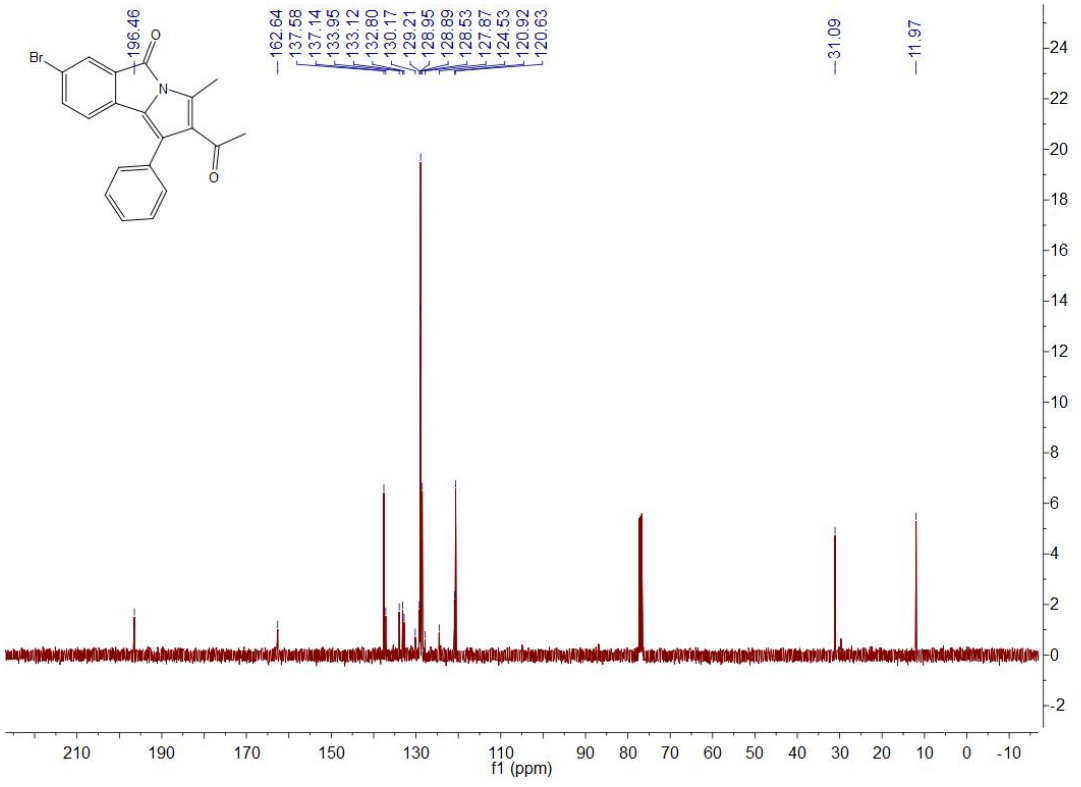

7.47
7.46
7.44
7.23
7.00
7.00
6.98
6.98
6.90
6.88
3.83
2.73
1.98
6.00
1.03
1.09
3.00
3.01
3.08
f1 (ppm)

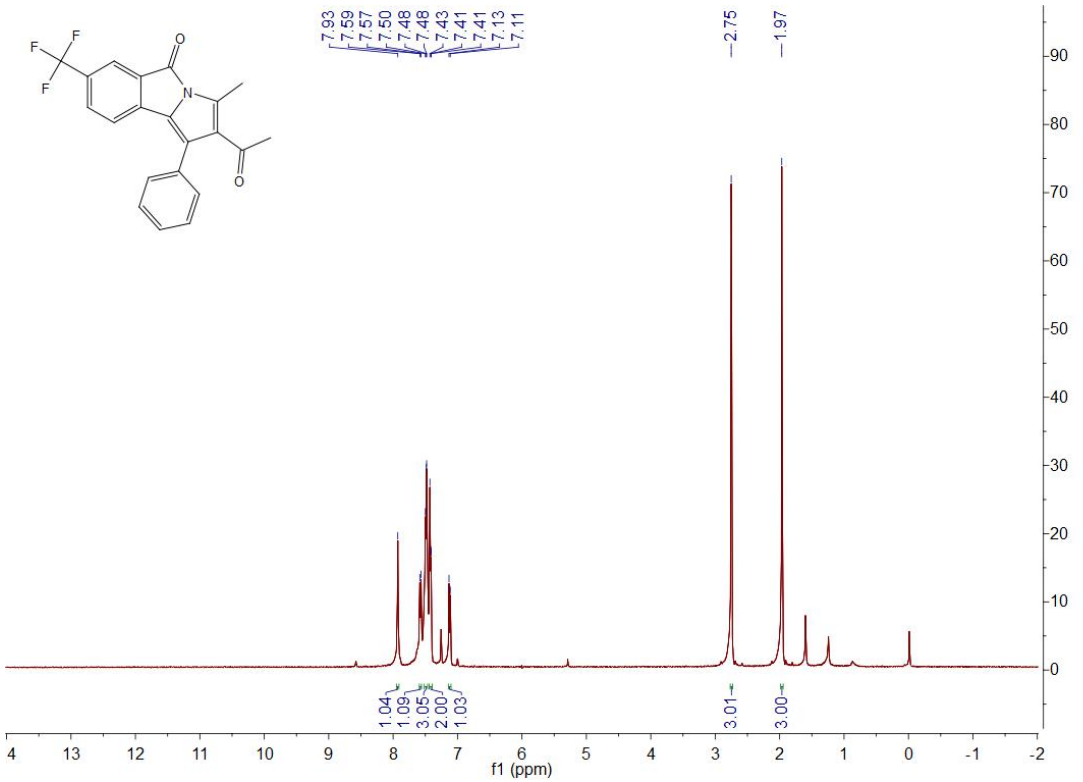

196.69
159.56
136.67
133.68
132.78
129.10
128.76
128.16
121.42
120.53
110.26
55.75
31.09
11.96
f1 (ppm)

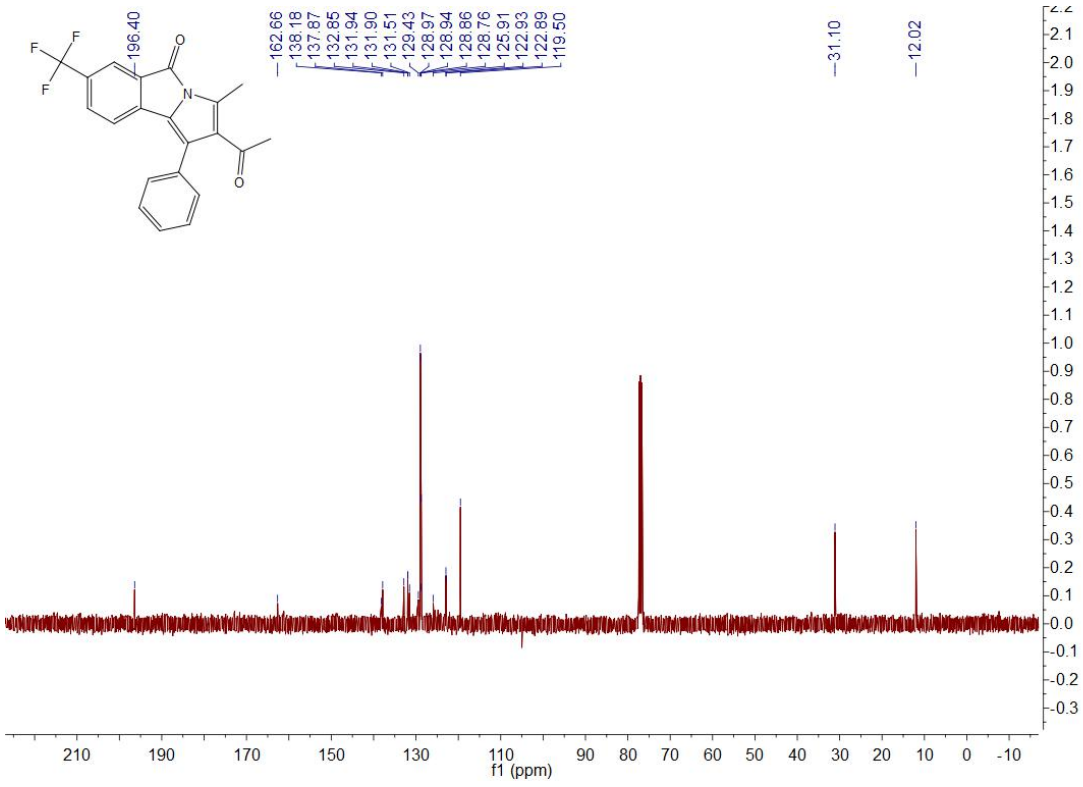

7.66
7.51
7.50
7.48
7.45
7.43
7.41
7.31
7.29
7.27
6.99
6.97
2.73
1.97
0.96
3.25
2.04
1.20
1.03
3.00
3.05
f1 (ppm)

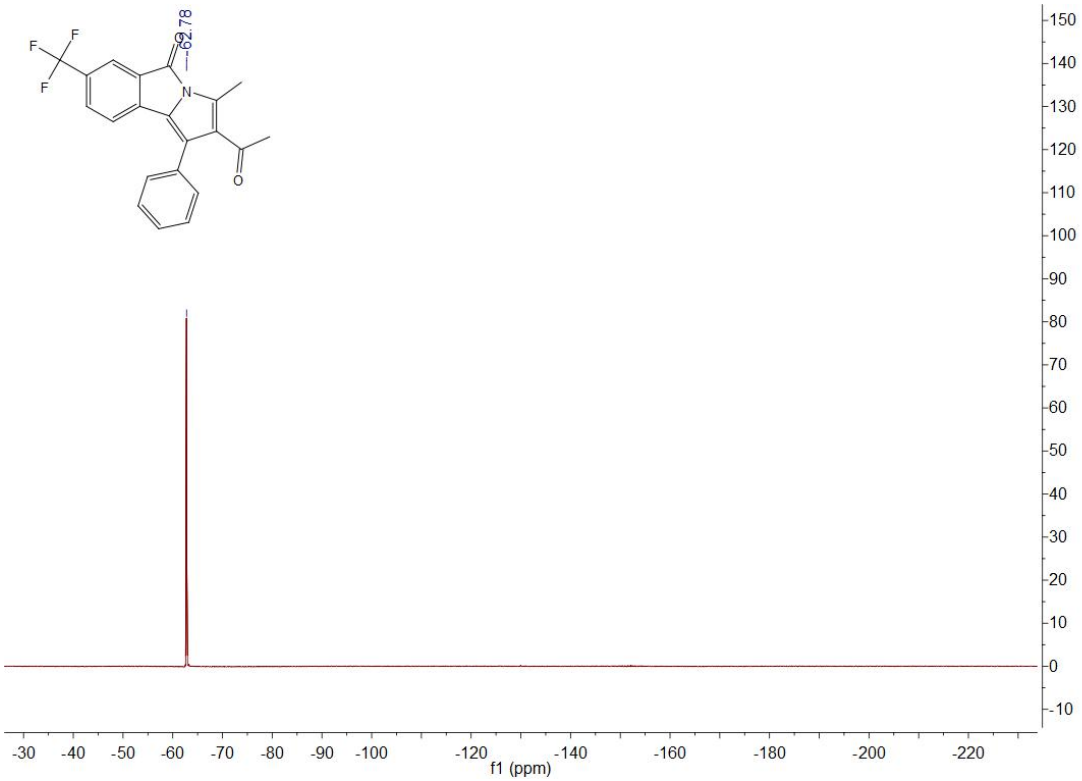

196.43
162.75
137.15
134.70
133.54
133.35
133.14
132.65
129.16
128.98
128.88
128.51
125.98
124.37
120.33
31.07
11.94
f1 (ppm)

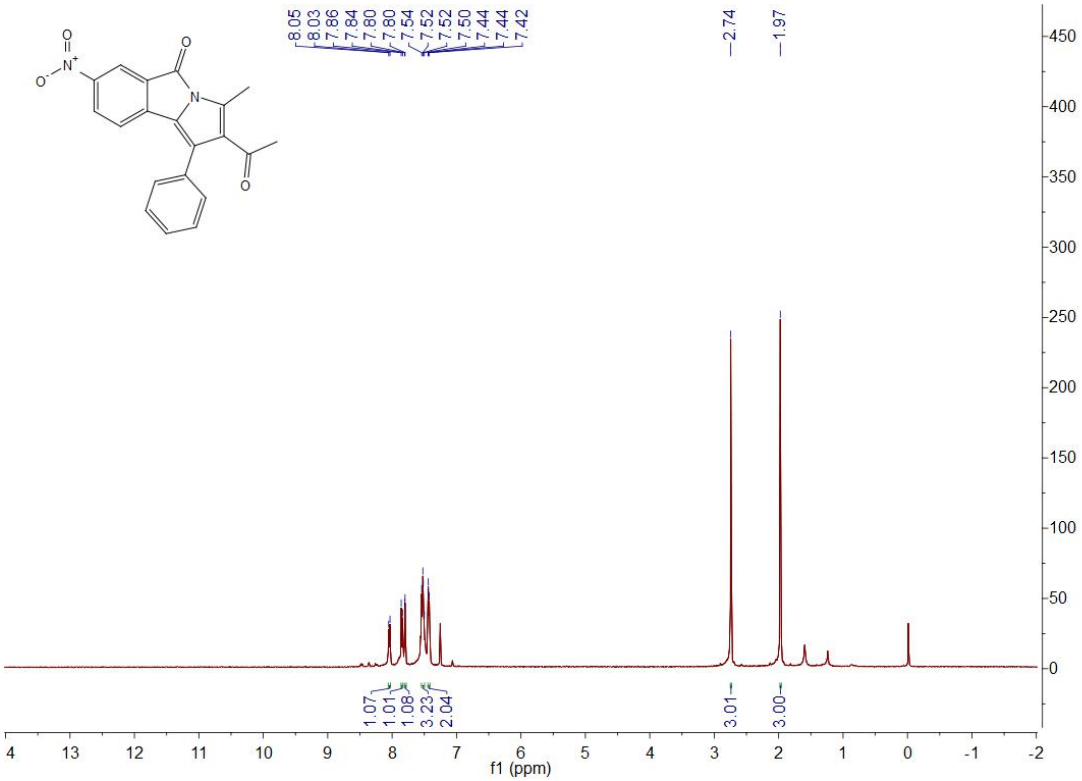

Br
O
N
O
7.80
7.51
7.49
7.48
7.47
7.46
7.45
7.42
7.41
7.27
6.92
6.90
2.72
1.97
1.00
4.03
2.03
1.08
3.00
3.00
f1 (ppm)

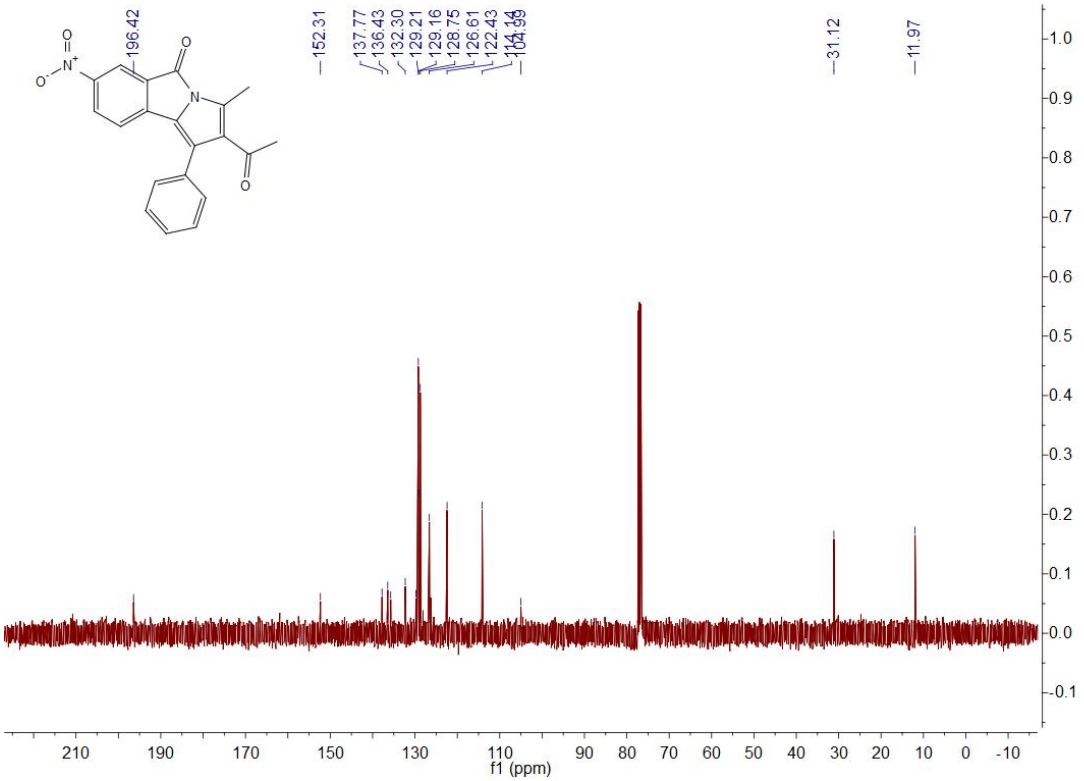

Br
O
N
O
196.46
162.64
137.58
137.14
133.95
133.12
132.80
130.17
129.21
128.95
128.89
128.53
127.87
124.53
120.92
120.63
31.09
11.97
f1 (ppm)

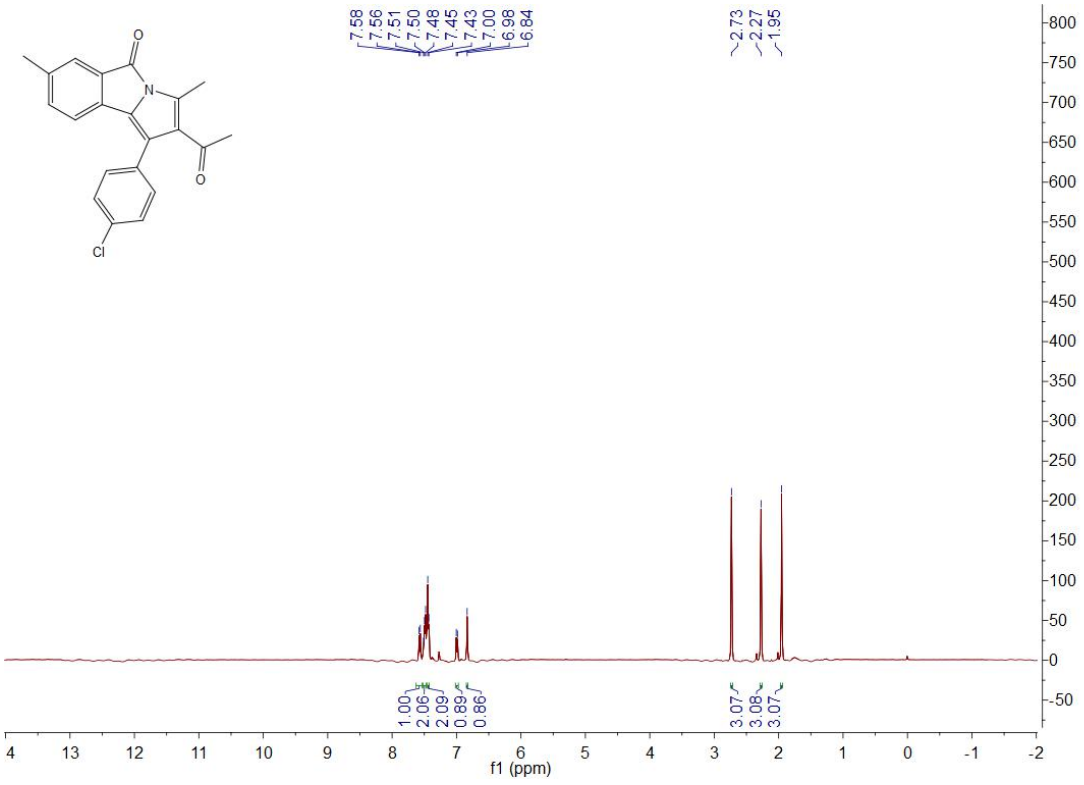

7.93
7.59
7.57
7.50
7.48
7.48
7.43
7.41
7.41
7.13
7.11
2.75
1.97
1.04
1.09
3.05
2.00
1.03
3.01
3.00
f1 (ppm)

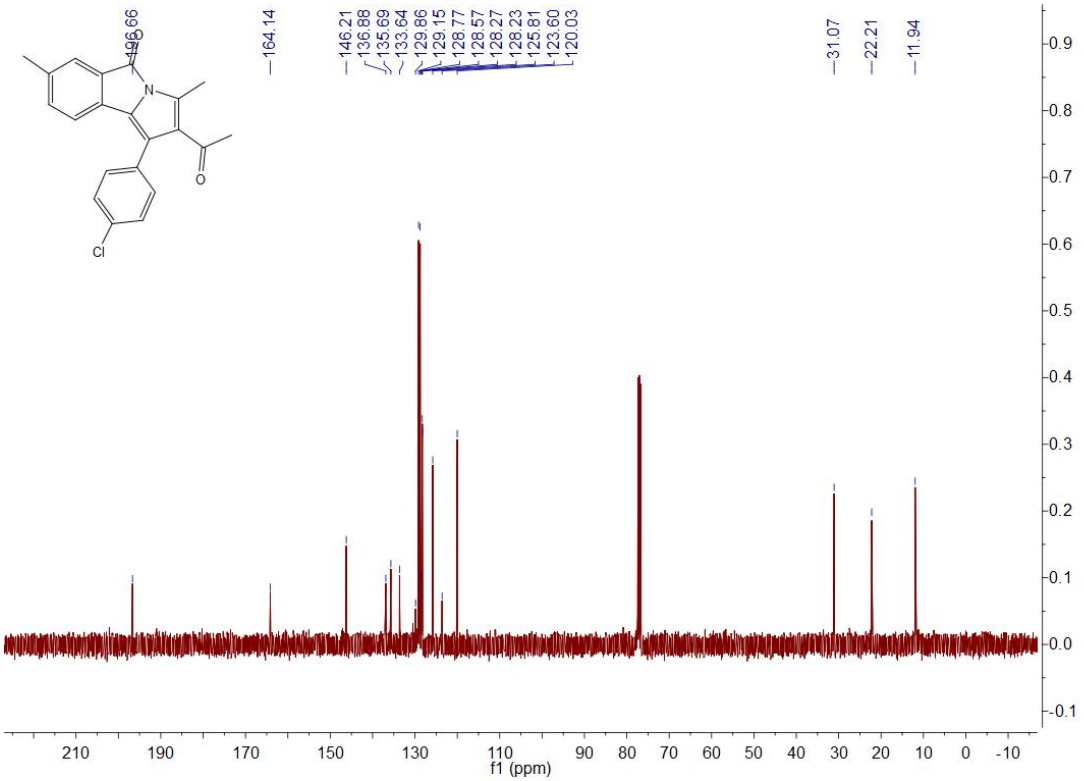

196.40
162.66
138.18
137.87
132.85
131.94
131.90
131.51
129.43
128.97
128.94
128.86
128.76
125.91
122.93
122.89
119.50
31.10
12.02
f1 (ppm)

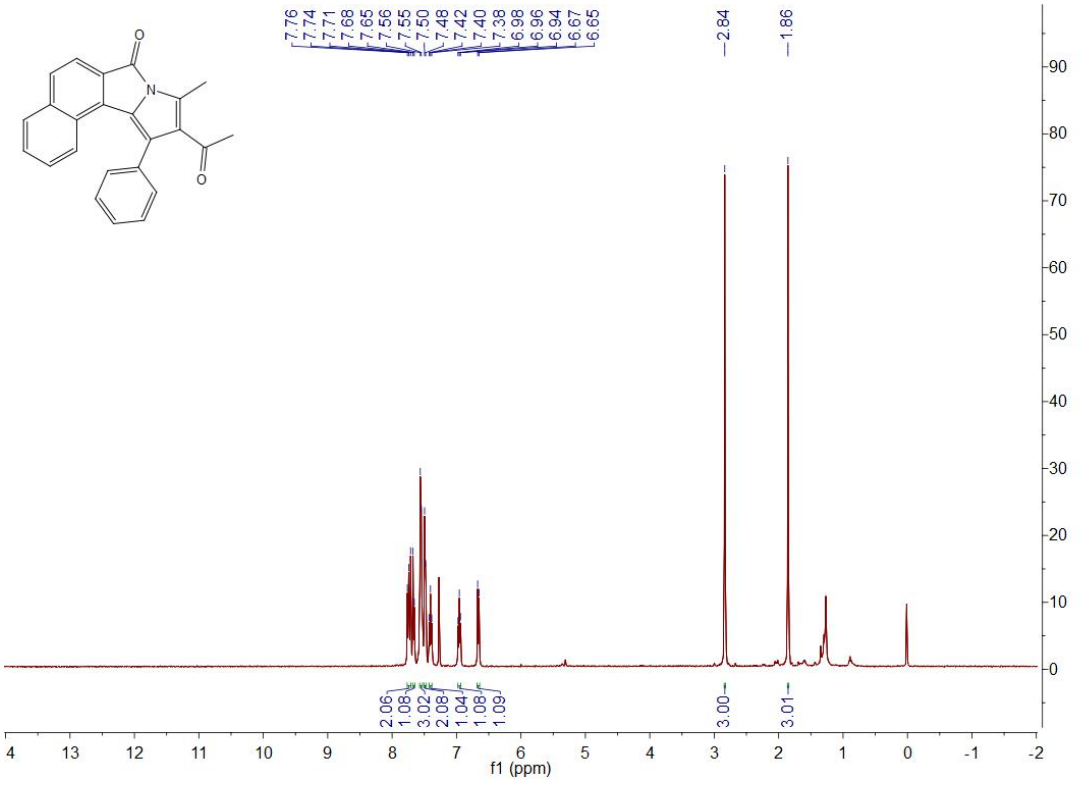

F
F
F
O
N
O
-62.78
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-30
-40
-50
-60
-70
-80
-90
-100
-120
-140
-160
-180
-200
-220
f1 (ppm)

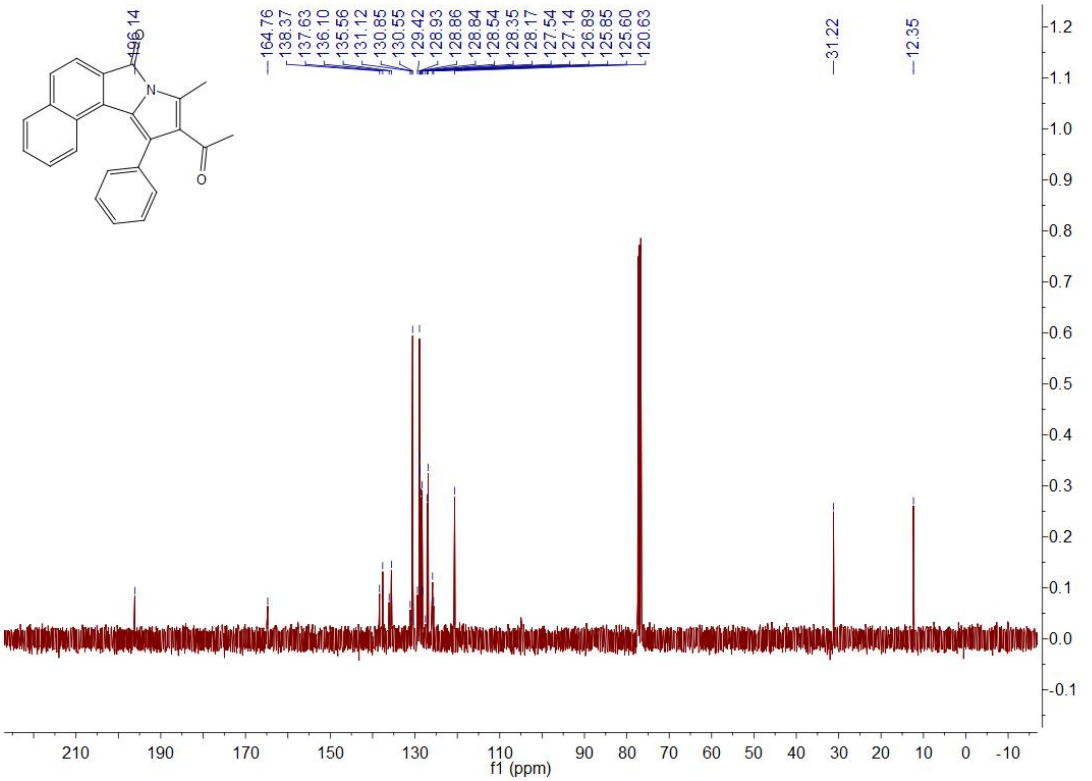

O
O
N
O
O
8.05
8.03
7.86
7.84
7.80
7.80
7.54
7.52
7.52
7.50
7.44
7.44
7.42
2.74
1.97
1.07
1.01
1.08
3.23
2.04
3.01
3.00
450
400
350
300
250
200
150
100
50
0
4
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)

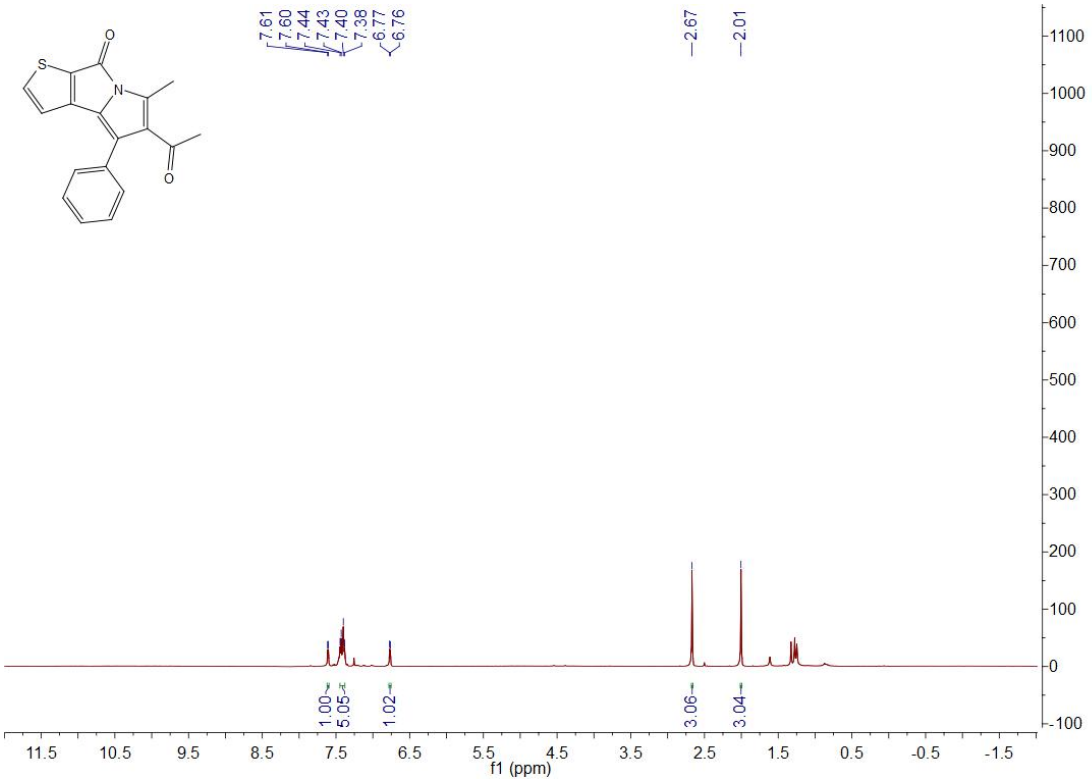

196.42
152.31
137.77
136.43
132.30
129.21
129.16
128.75
126.61
122.43
31.12
11.97
f1 (ppm)

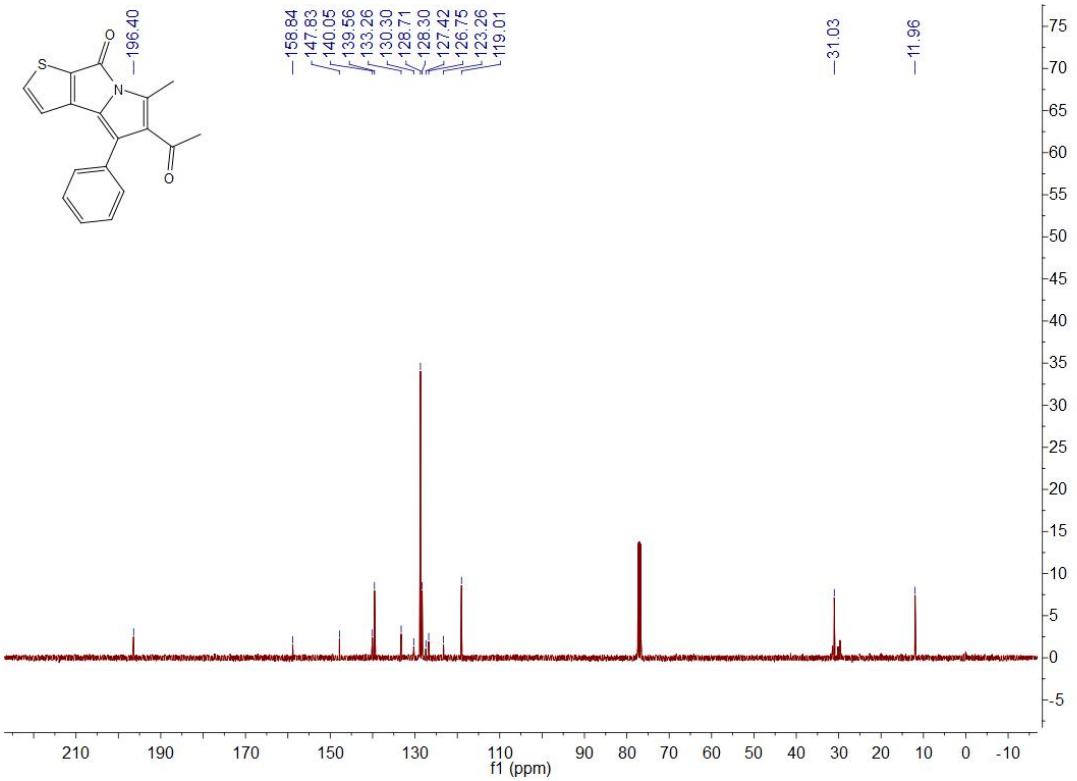

7.58
7.56
7.51
7.50
7.48
7.45
7.43
7.00
6.98
6.84
2.73
2.27
1.95
1.00
2.06
2.09
0.89
0.86
3.07
3.08
3.07
f1 (ppm)

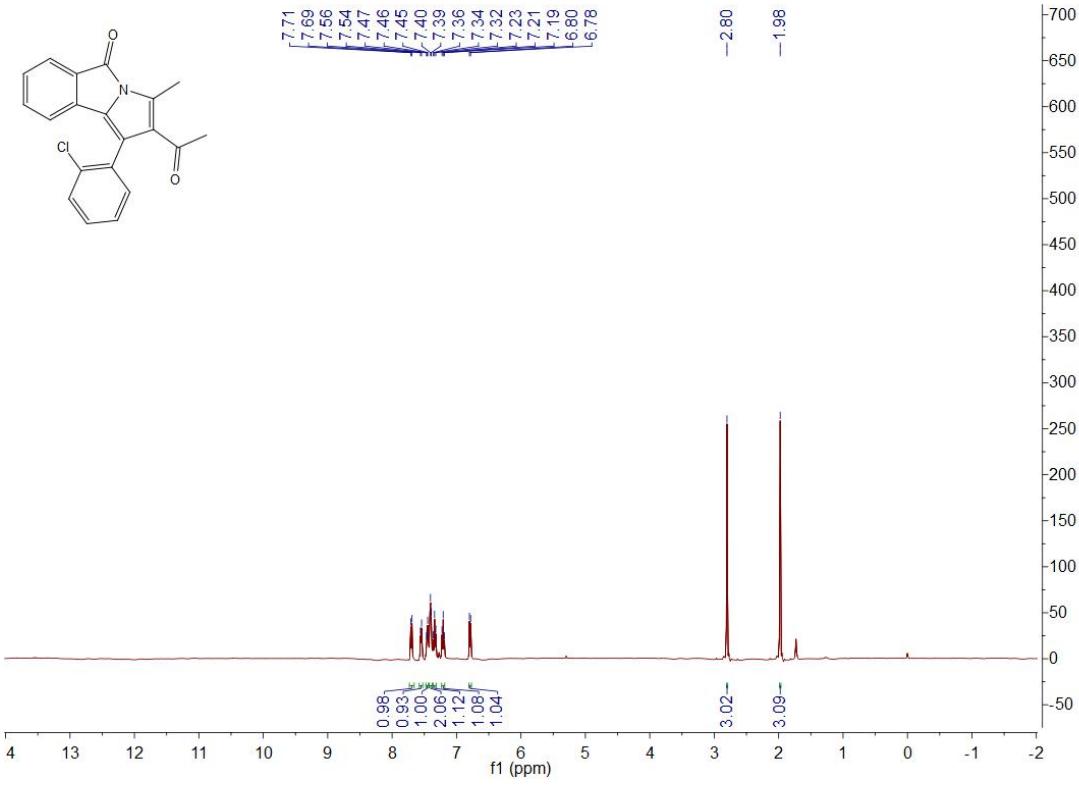

196.66
164.14
146.21
136.88
135.69
133.64
129.86
129.15
128.77
128.57
128.27
128.23
125.81
123.60
120.03
31.07
22.21
11.94
O
N
O
Cl
f1 (ppm)

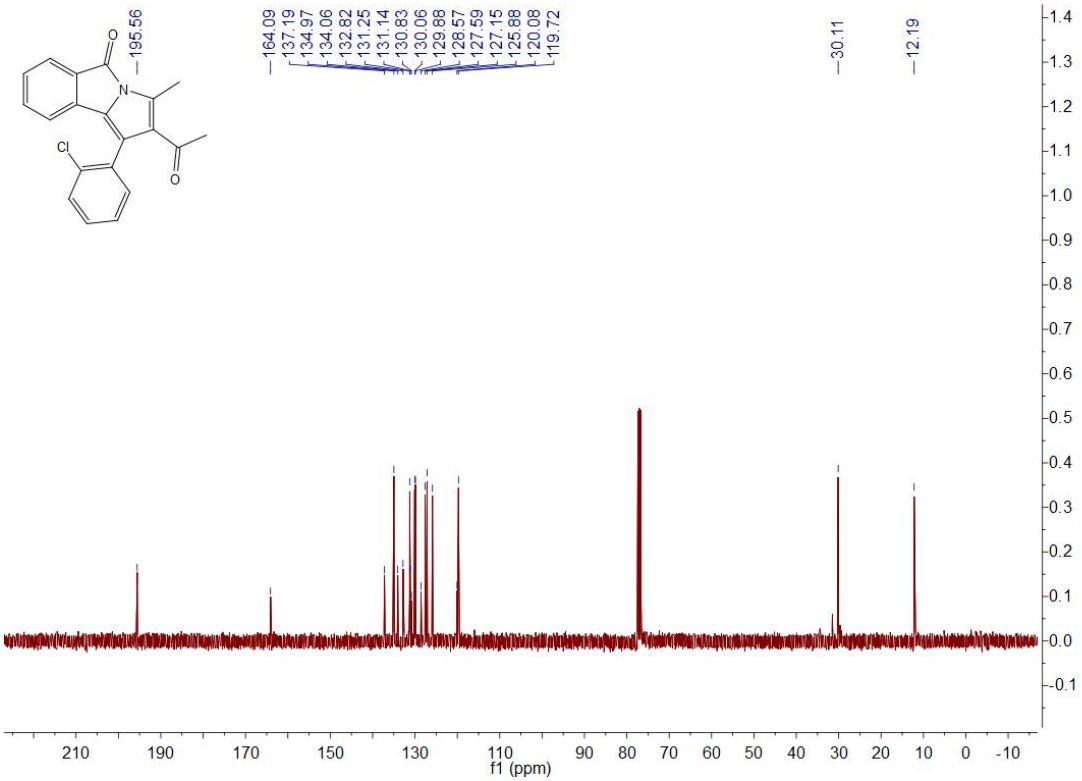

7.76
7.74
7.71
7.68
7.65
7.56
7.55
7.50
7.48
7.42
7.40
7.38
6.98
6.96
6.94
6.67
6.65
2.84
1.86
O
N
O
2.06
1.08
3.02
2.08
1.04
1.08
1.09
3.00
3.01
f1 (ppm)

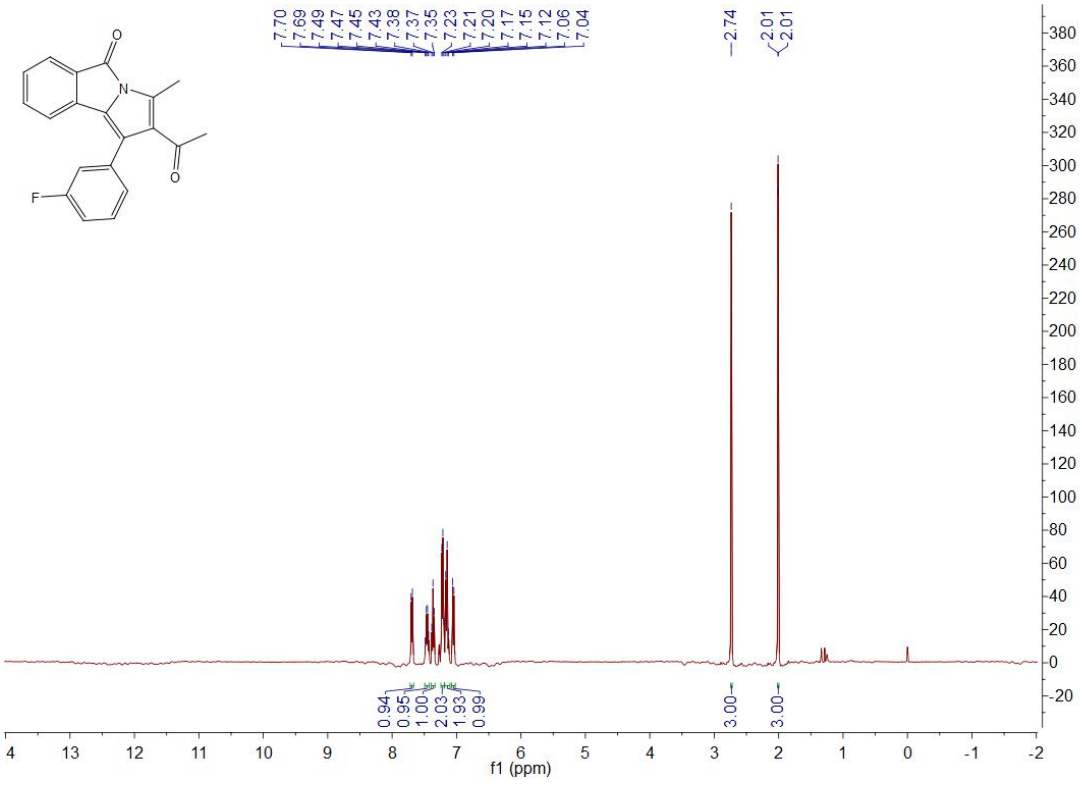

196.14
164.76
138.37
137.63
136.10
135.56
131.12
130.85
130.55
129.42
128.93
128.86
128.84
128.54
128.35
128.17
127.54
127.14
126.89
125.85
125.60
120.63
31.22
12.35
O
N
f1 (ppm)

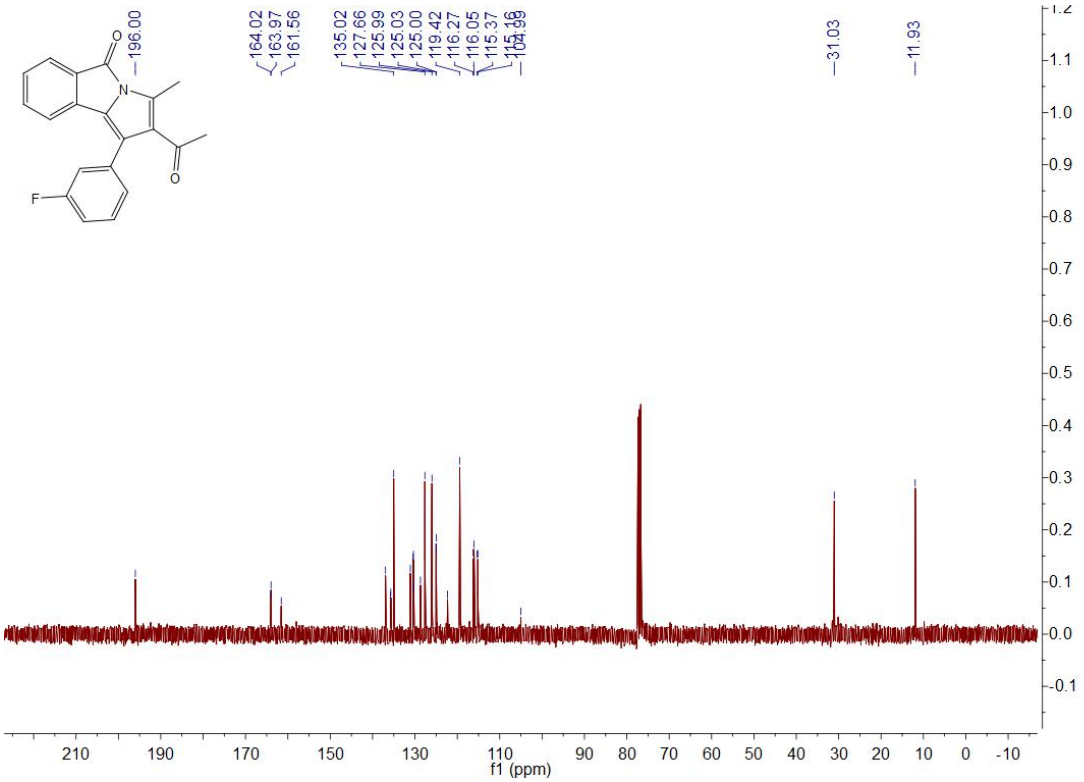

7.61
7.60
7.44
7.43
7.40
7.38
6.77
6.76
2.67
2.01
1.00
5.05
1.02
3.06
3.04
O
S
N
f1 (ppm)

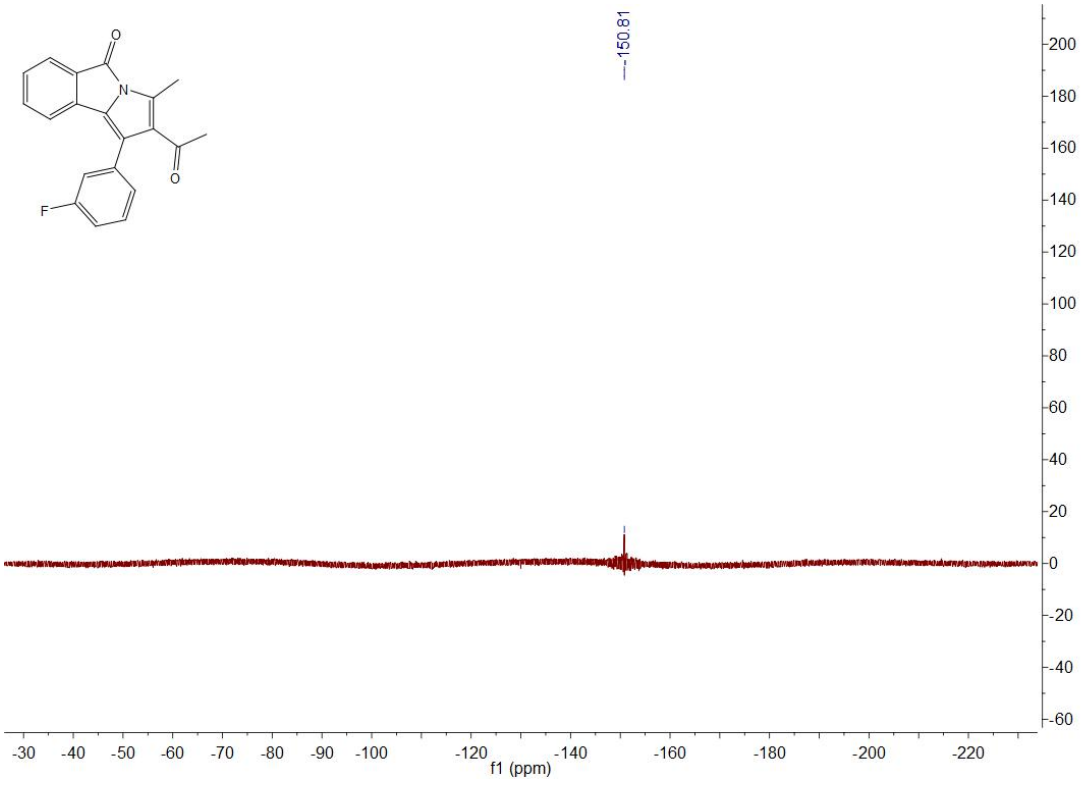

196.40
158.84
147.83
140.05
139.56
133.26
130.30
128.71
128.30
127.42
126.75
123.26
119.01
31.03
11.96
f1 (ppm)

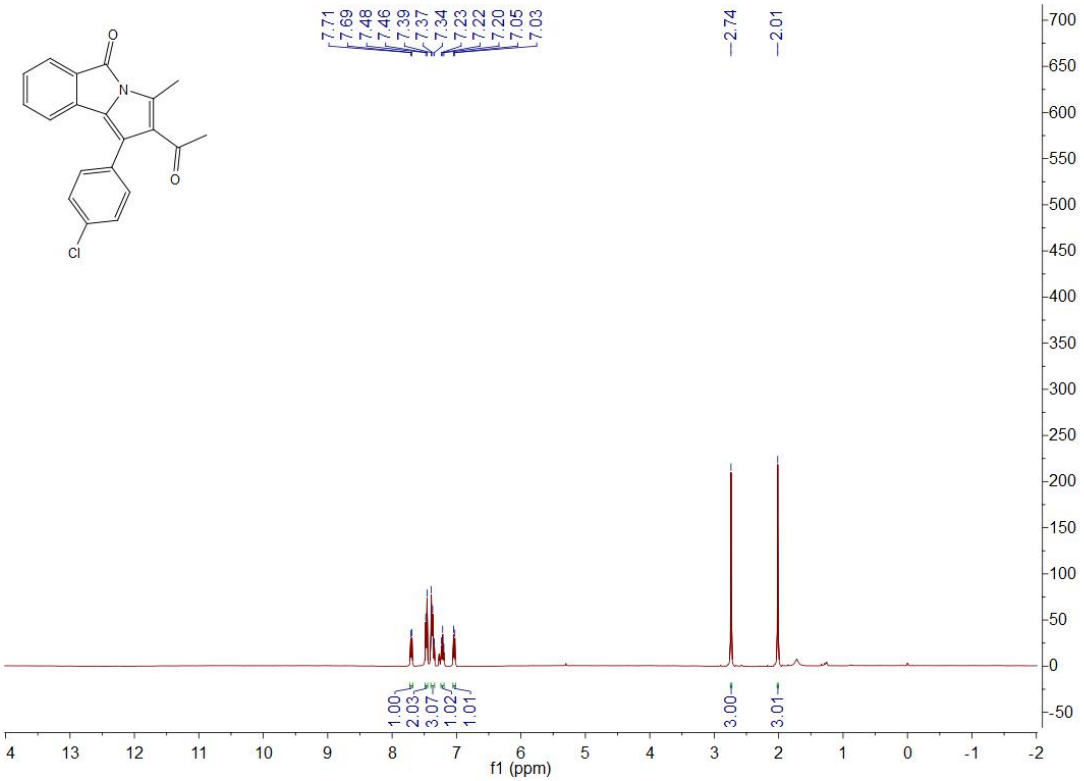

7.71
7.69
7.56
7.54
7.47
7.46
7.45
7.40
7.39
7.36
7.34
7.32
7.23
7.21
7.19
6.80
6.78
2.80
1.98
0.98
0.93
1.00
2.06
1.12
1.08
1.04
3.02
3.09
f1 (ppm)

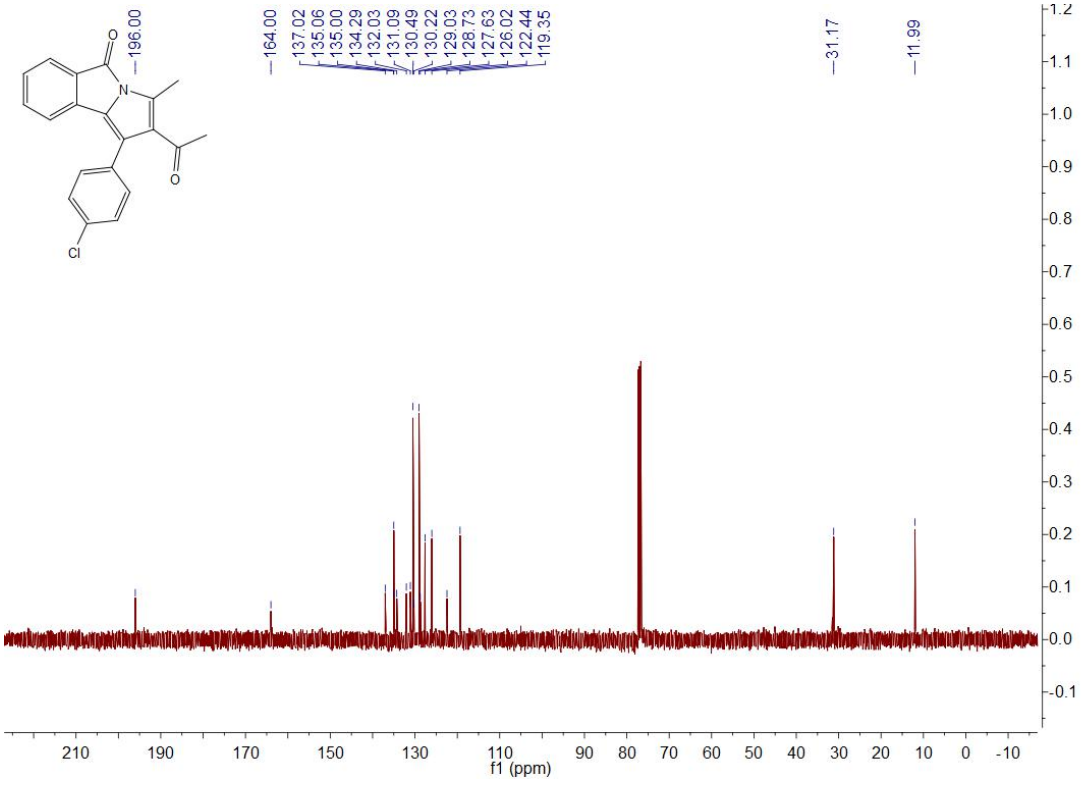

195.56
164.09
137.19
134.97
134.06
132.82
131.25
131.14
130.83
130.06
129.88
128.57
127.59
127.15
125.88
120.08
119.72
30.11
12.19
O
N
Cl
O
f1 (ppm)

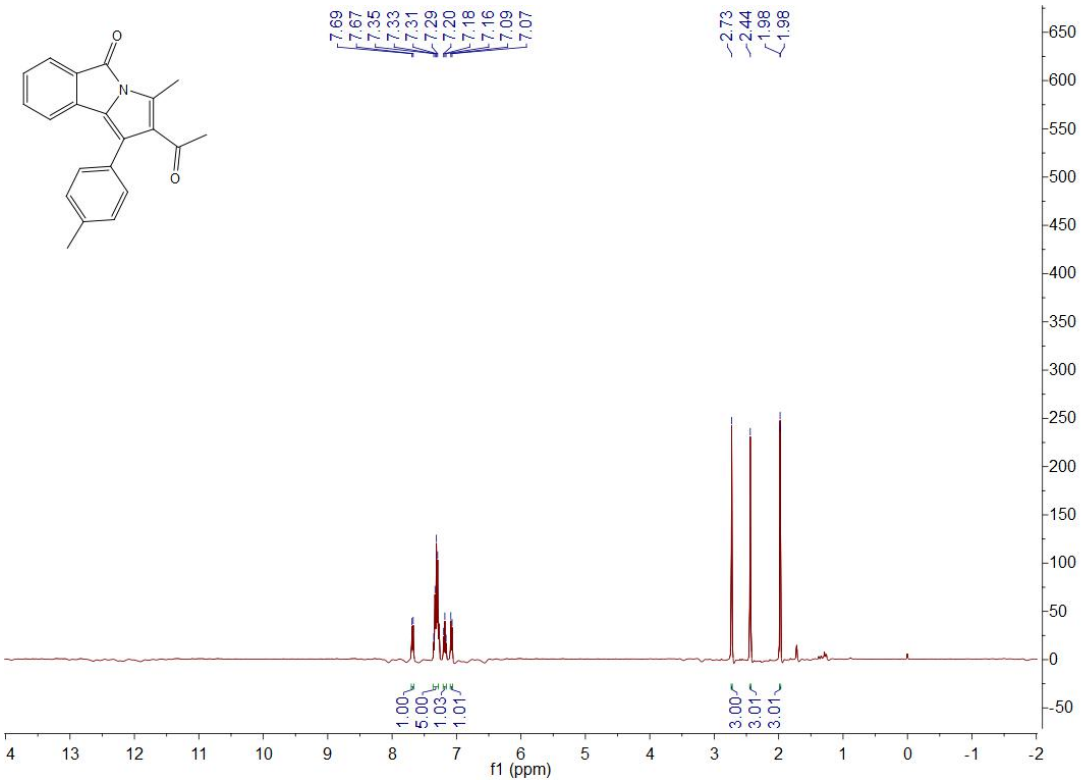

7.70
7.69
7.49
7.47
7.45
7.43
7.38
7.37
7.35
7.23
7.21
7.20
7.17
7.15
7.12
7.06
7.04
2.74
2.01
2.01
O
N
F
O
0.94
0.95
1.00
2.03
1.93
0.99
3.00
3.00
f1 (ppm)

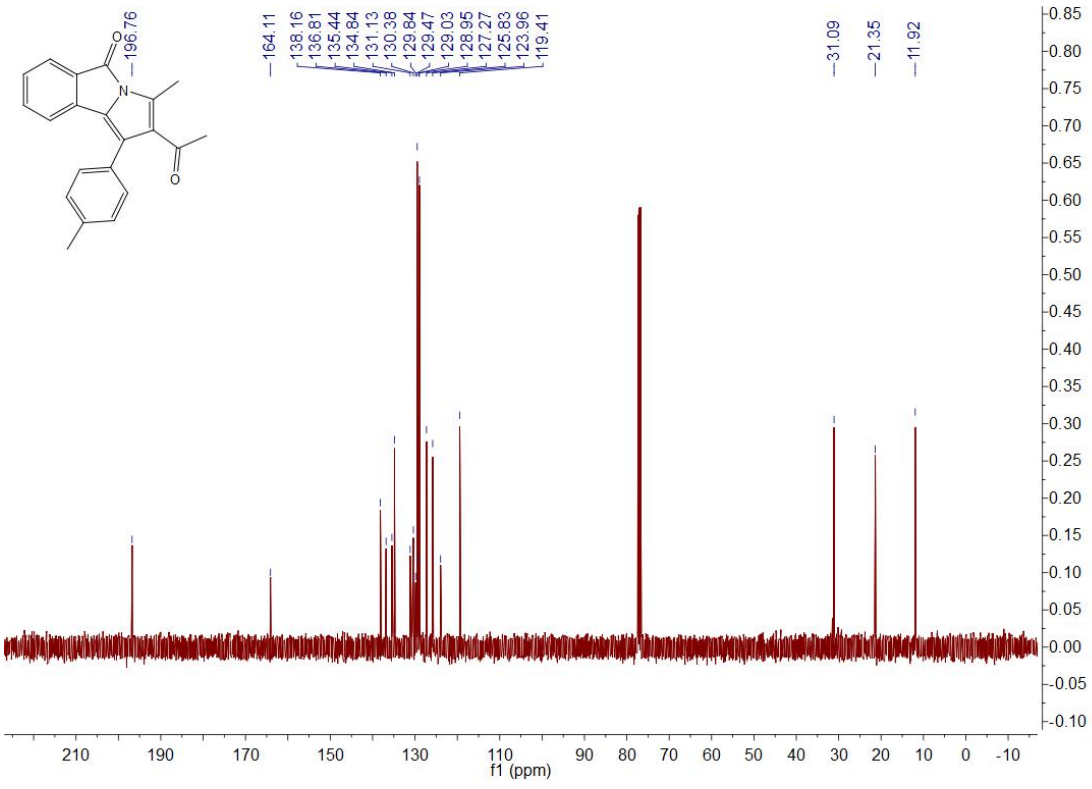

196.00
164.02
163.97
161.56
135.02
127.66
125.99
125.03
125.00
119.42
116.27
116.05
115.37
31.03
11.93
f1 (ppm)

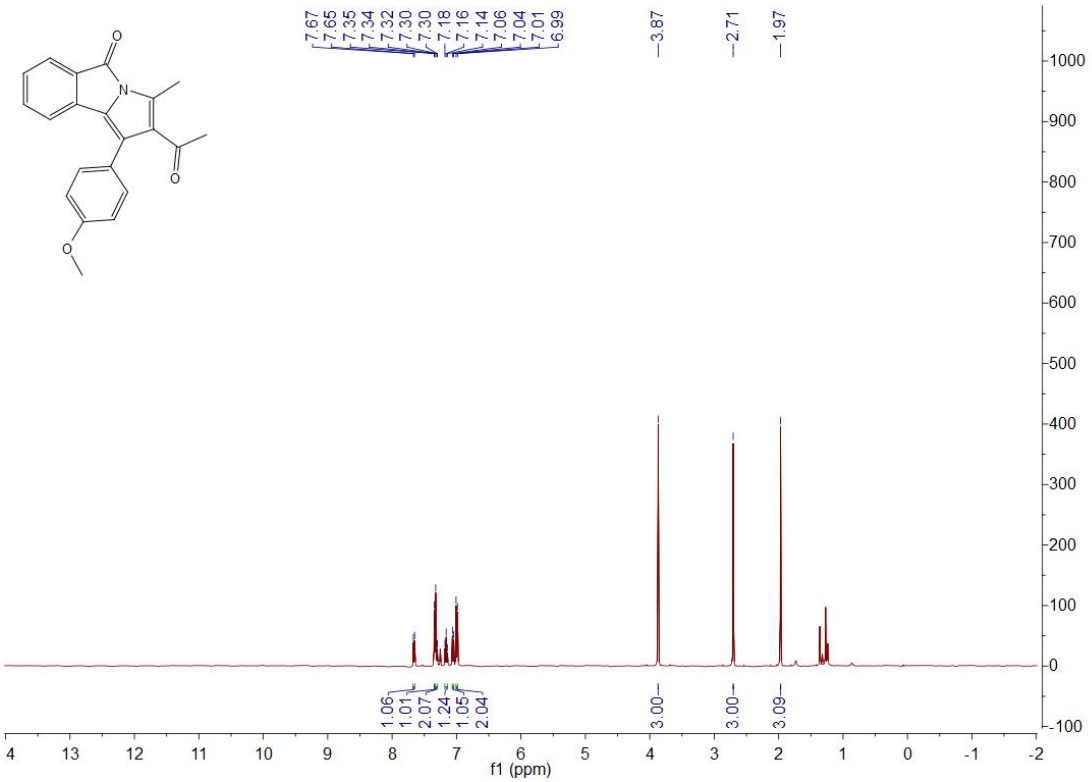

150.81
f1 (ppm)

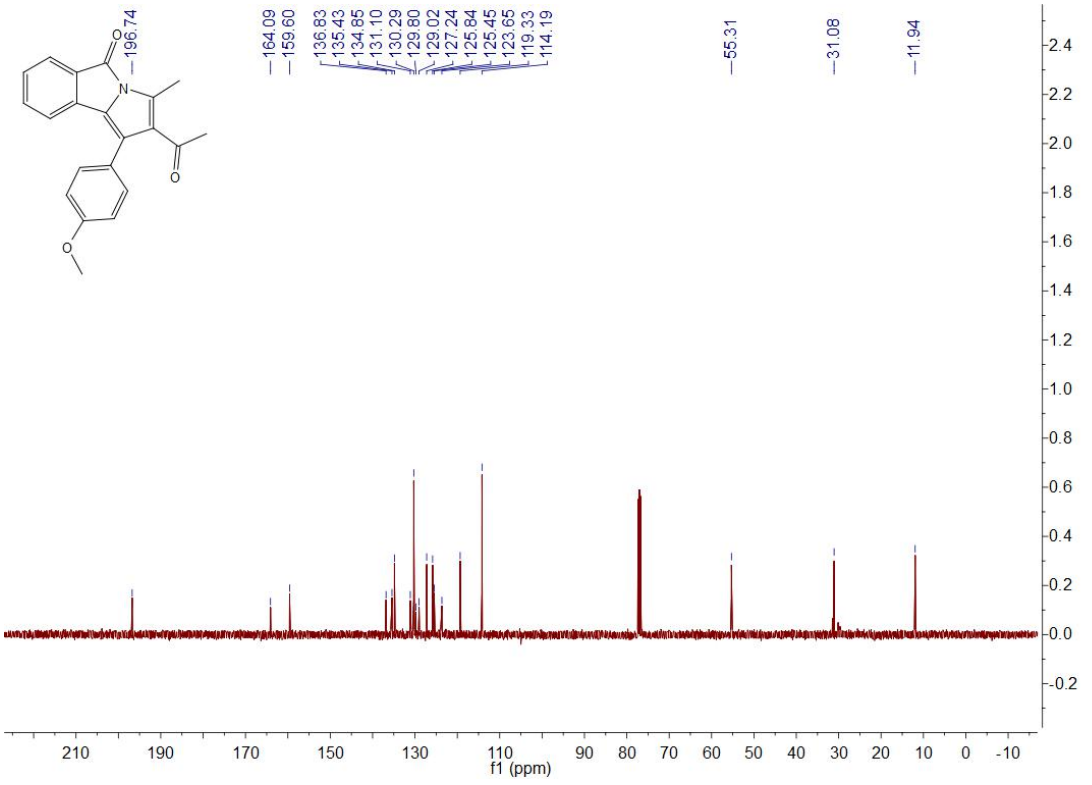

7.71
7.69
7.48
7.46
7.39
7.37
7.34
7.23
7.22
7.20
7.05
7.03
2.74
2.01
1.00
2.03
3.07
1.02
1.01
3.00
3.01
f1 (ppm)

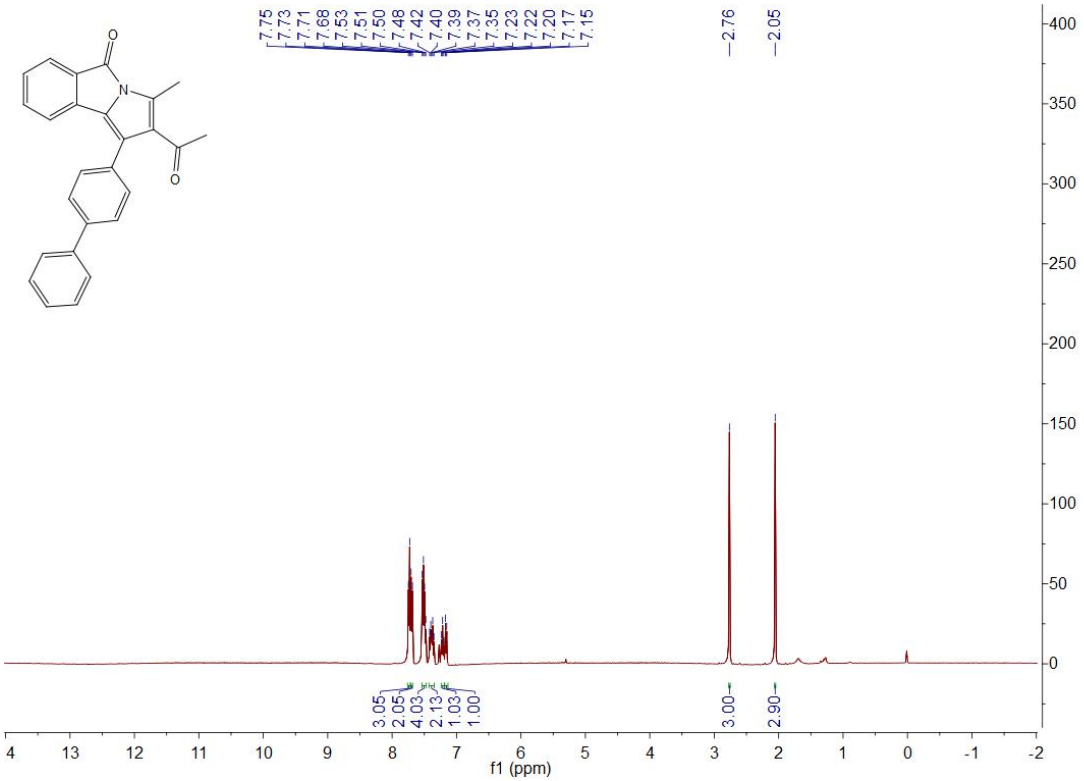

196.00
164.00
137.02
135.06
135.00
134.29
132.03
131.09
130.49
130.22
129.03
128.73
127.63
126.02
122.44
119.35
31.17
11.99
f1 (ppm)

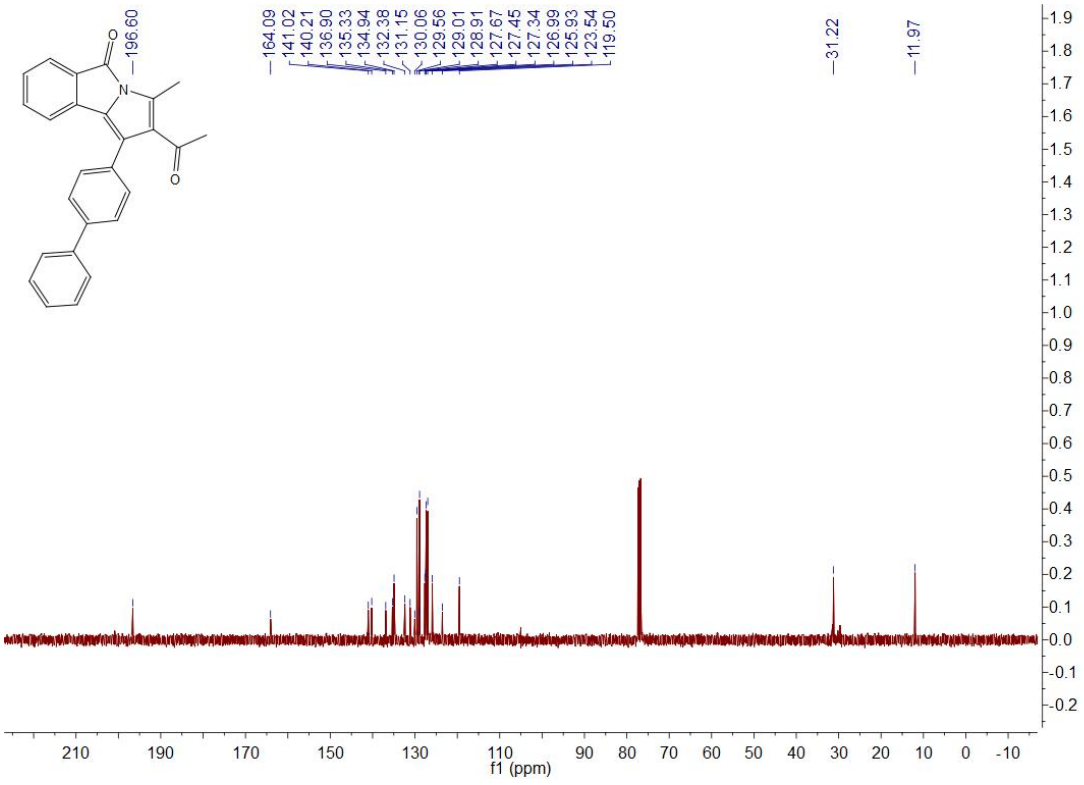

7.69
7.67
7.35
7.33
7.31
7.29
7.20
7.18
7.16
7.09
7.07
2.73
2.44
1.98
1.98
1.00
5.00
1.03
1.01
3.00
3.01
3.01
f1 (ppm)

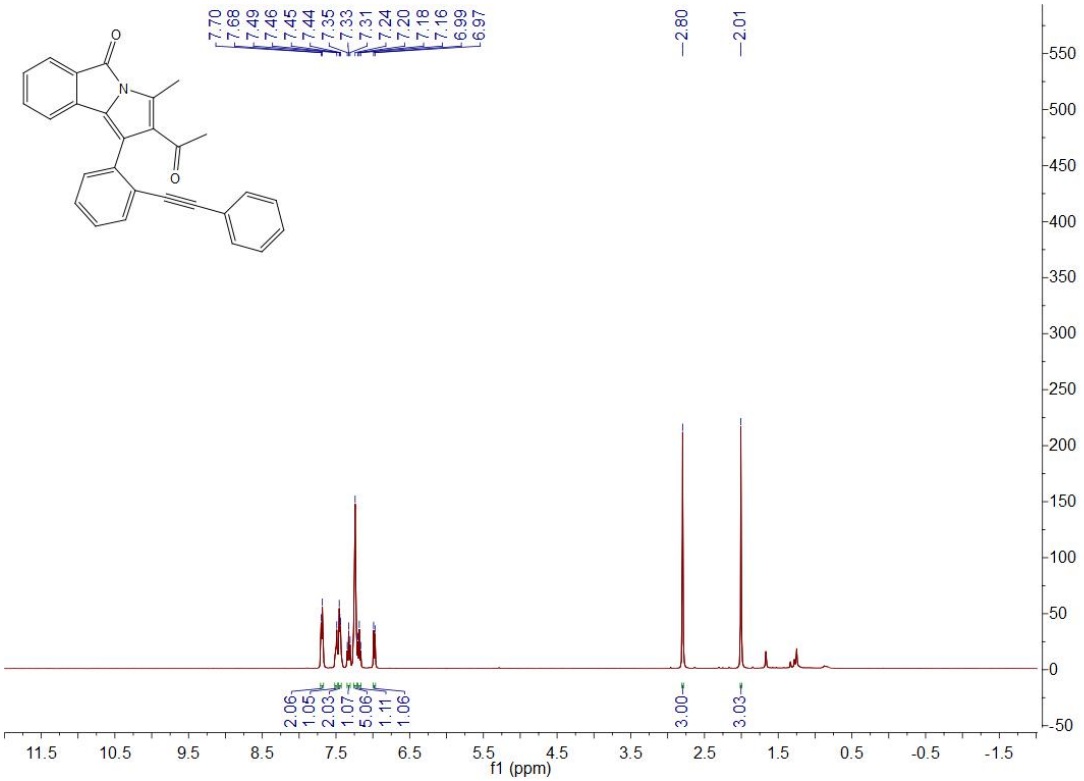

196.76
164.11
138.16
136.81
135.44
134.84
131.13
130.38
129.84
129.47
129.03
128.95
127.27
125.83
123.96
119.41
31.09
21.35
11.92
f1 (ppm)

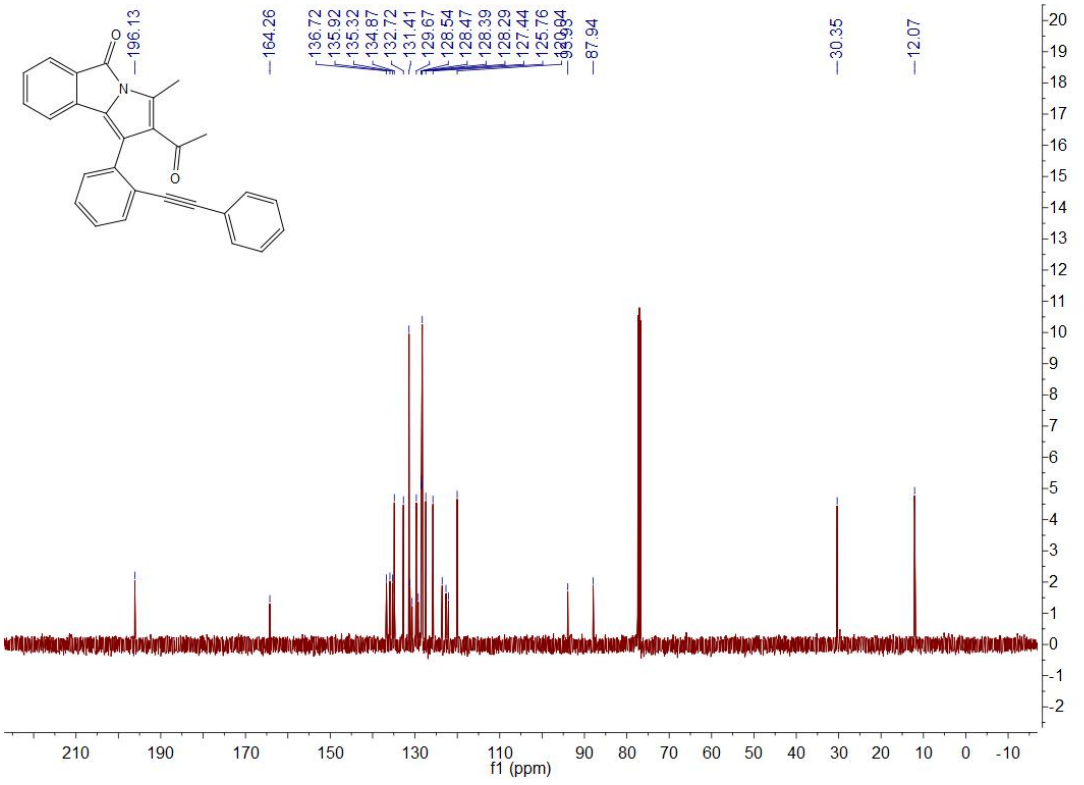

7.67
7.65
7.35
7.34
7.32
7.30
7.30
7.18
7.16
7.14
7.06
7.04
7.01
6.99
3.87
2.71
1.97
1.06
1.01
2.07
1.24
1.05
2.04
3.00
3.00
3.09
f1 (ppm)

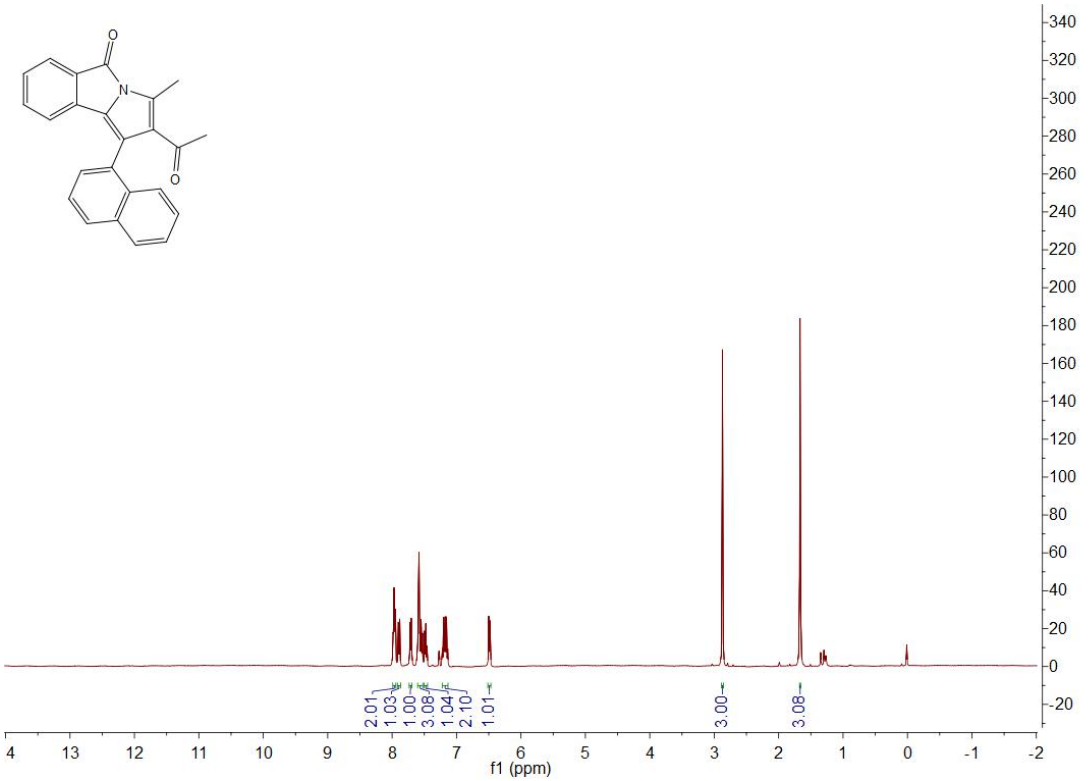

196.74
164.09
159.60
136.83
135.43
134.85
131.10
130.29
129.80
129.02
127.24
125.84
125.45
123.65
119.33
114.19
55.31
31.08
11.94
f1 (ppm)

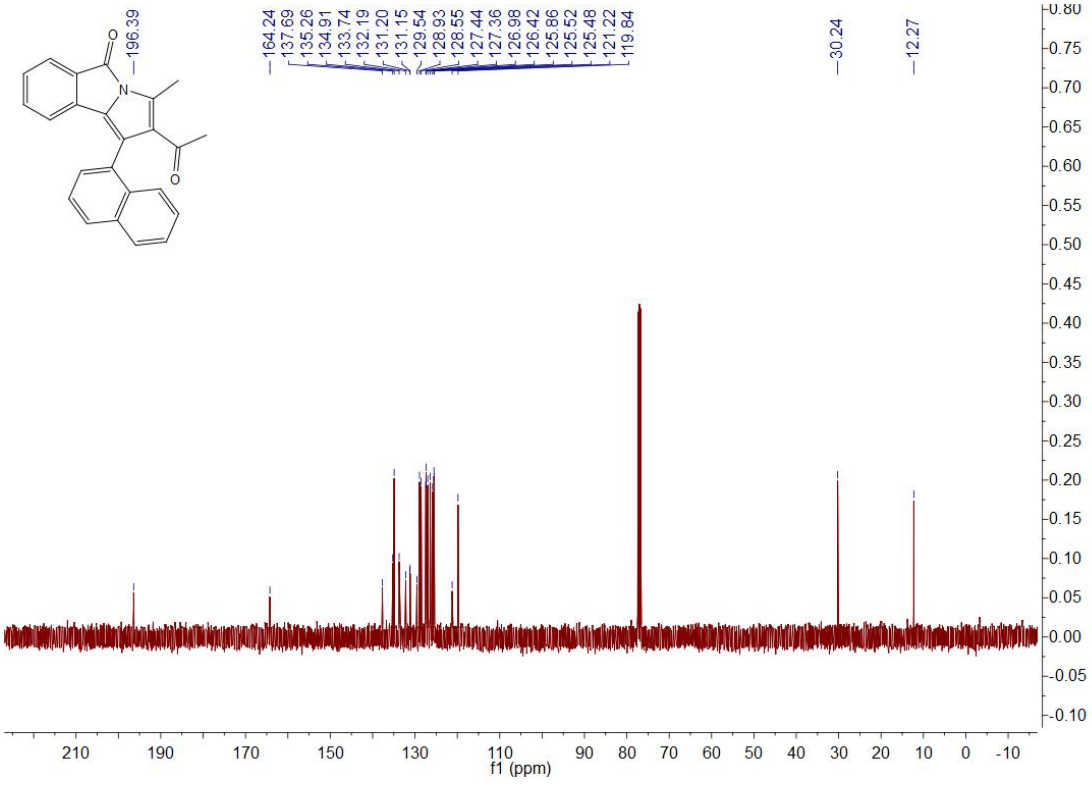

7.75
7.73
7.71
7.68
7.53
7.51
7.50
7.48
7.42
7.40
7.39
7.37
7.35
7.23
7.22
7.20
7.17
7.15
2.76
2.05
3.05
2.05
4.03
2.13
1.03
1.00
3.00
2.90
f1 (ppm)

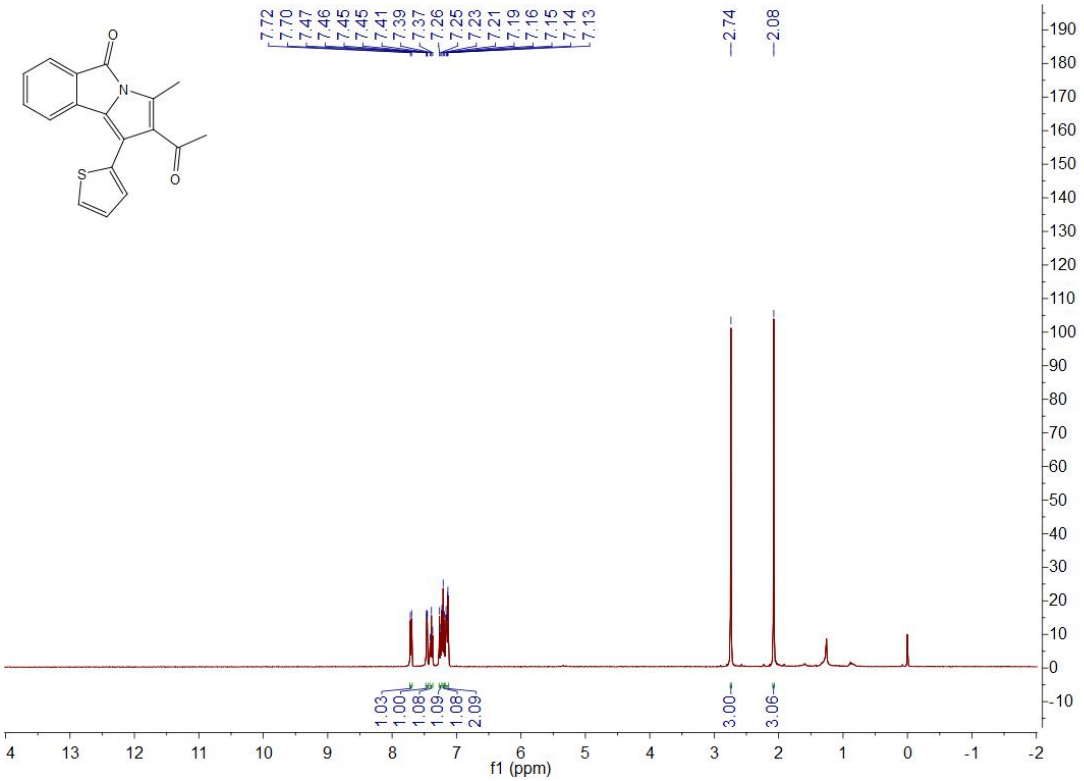

196.60
164.09
141.02
140.21
136.90
135.33
134.94
132.38
131.15
130.06
129.56
129.01
128.91
127.67
127.45
127.34
126.99
125.93
123.54
119.50
31.22
11.97
f1 (ppm)

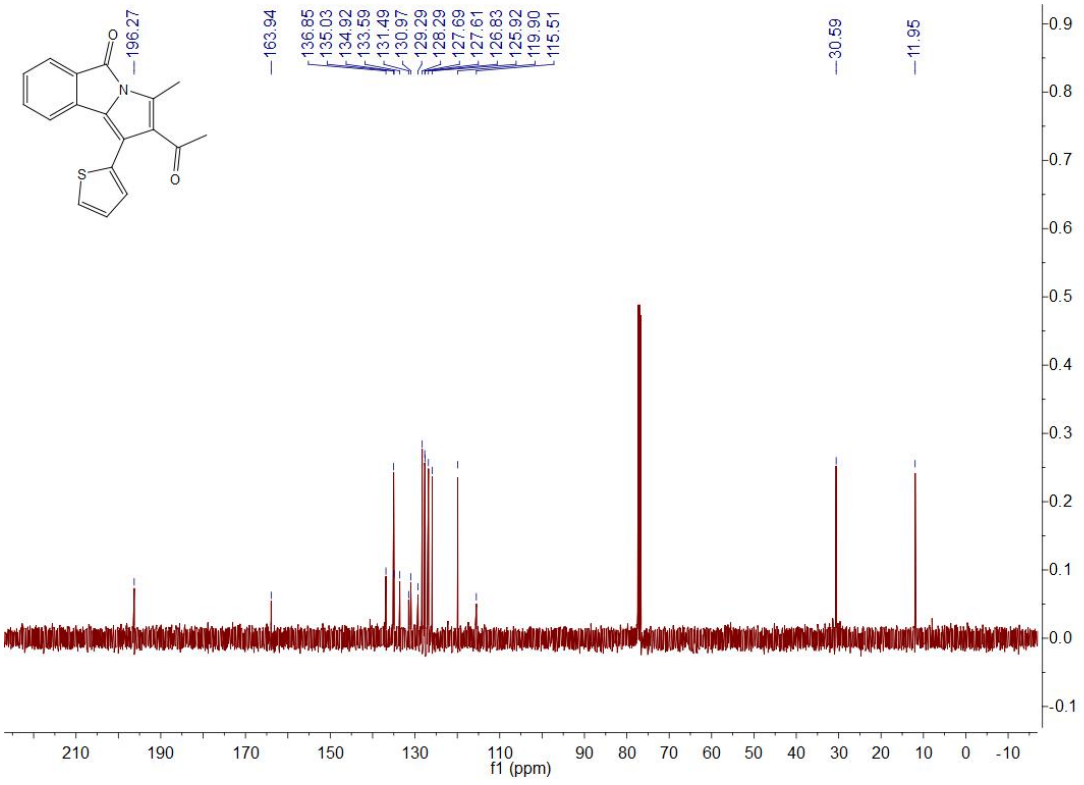

7.70
7.68
7.49
7.46
7.45
7.44
7.35
7.33
7.31
7.24
7.20
7.18
7.16
6.99
6.97
2.80
2.01
2.06
1.05
2.03
1.07
5.06
1.11
1.06
3.00
3.03
f1 (ppm)

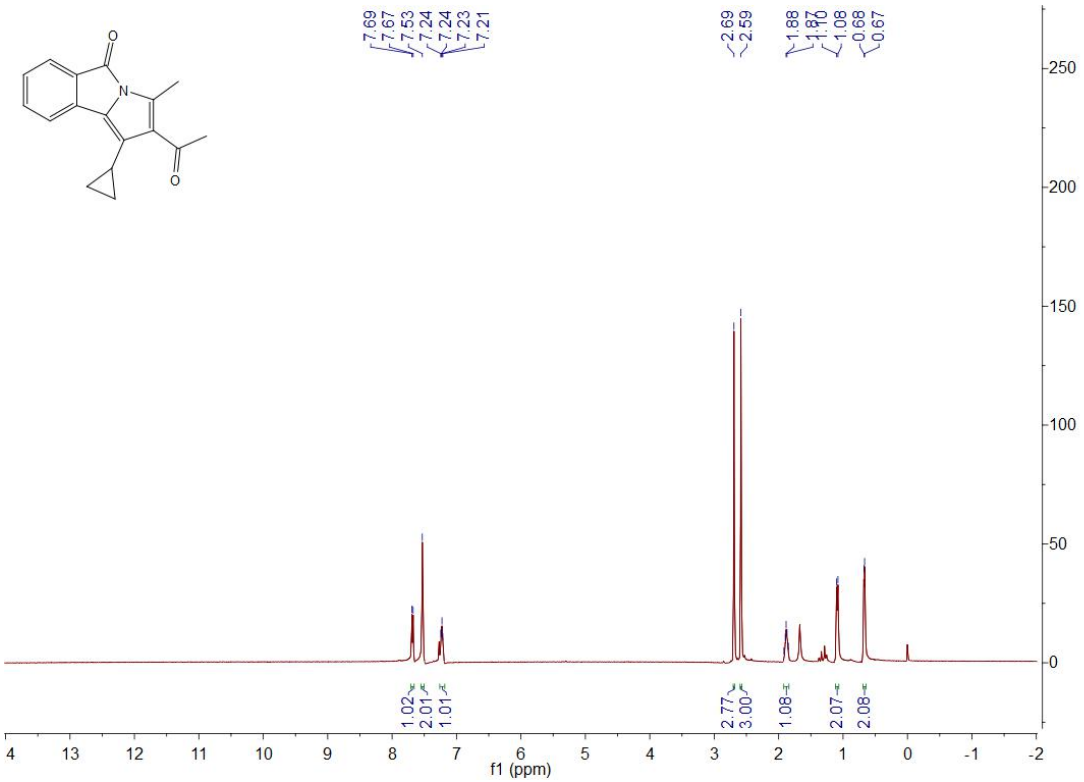

196.13
164.26
136.72
135.92
135.32
134.87
132.72
131.41
129.67
128.54
128.47
128.39
128.29
127.44
125.76
87.94
30.35
12.07
f1 (ppm)

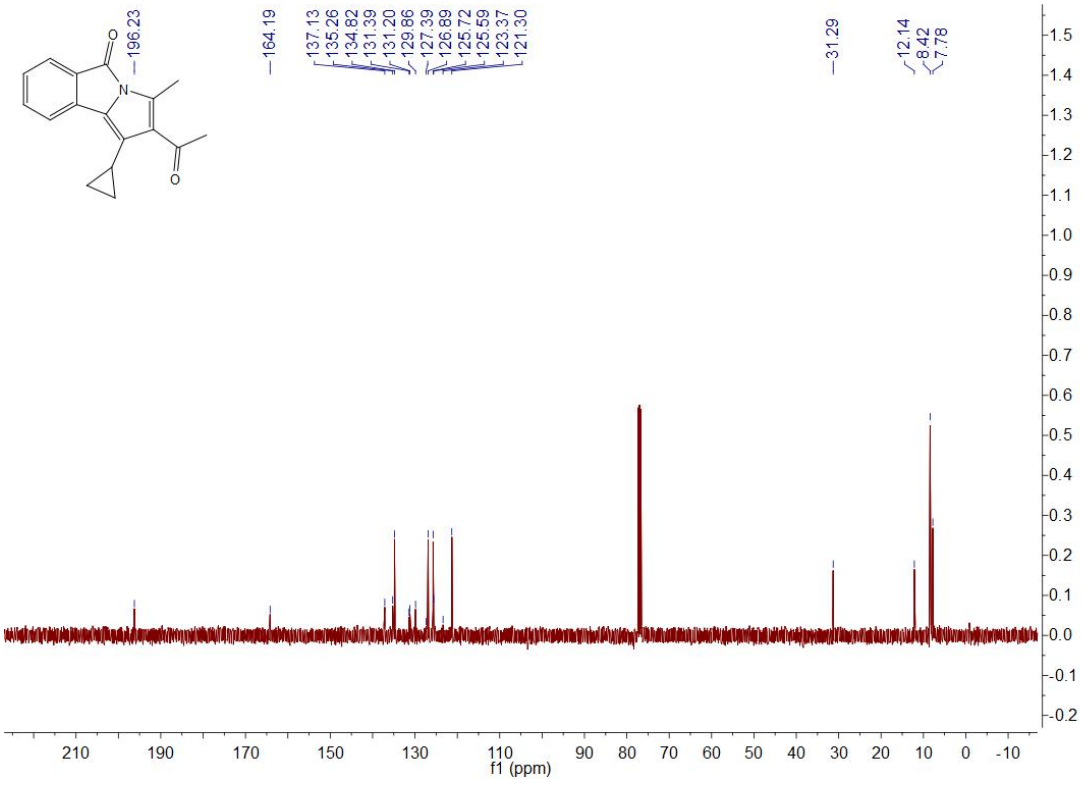

2.01
1.03
1.00
3.08
1.04
2.10
1.01
3.00
3.08
f1 (ppm)

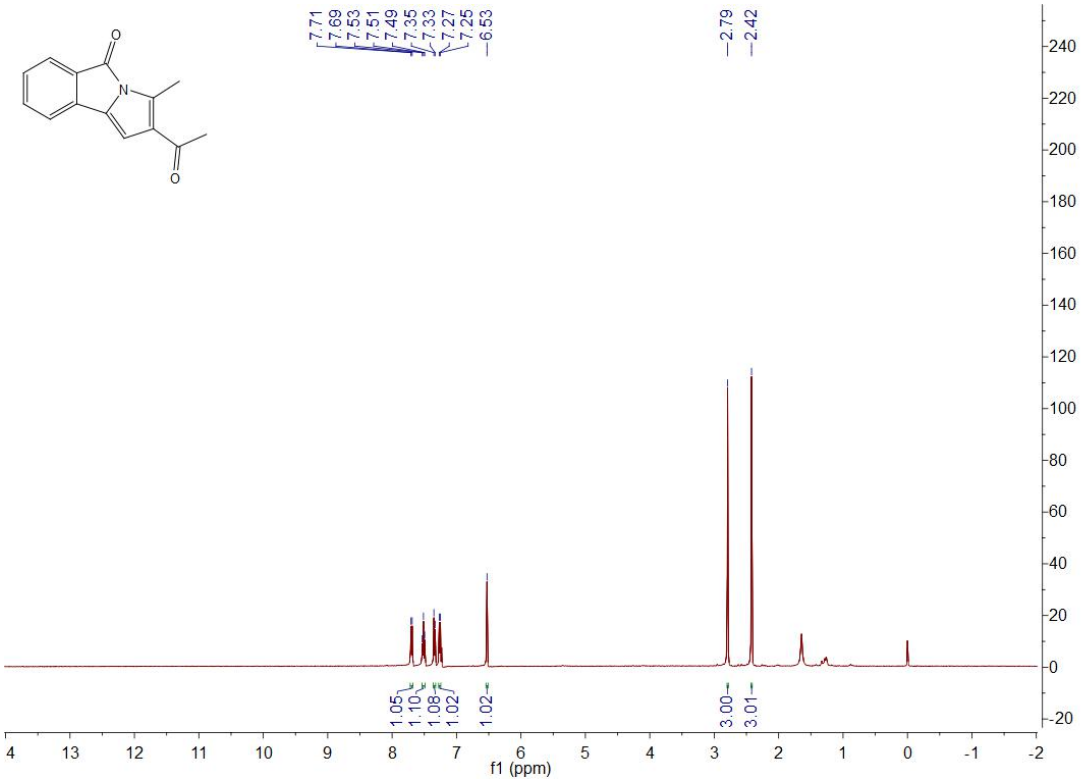

196.39
164.24
137.69
135.26
134.91
133.74
132.19
131.20
131.15
129.54
128.93
128.55
127.44
127.36
126.98
126.42
125.86
125.52
125.48
121.22
119.84
30.24
12.27
f1 (ppm)

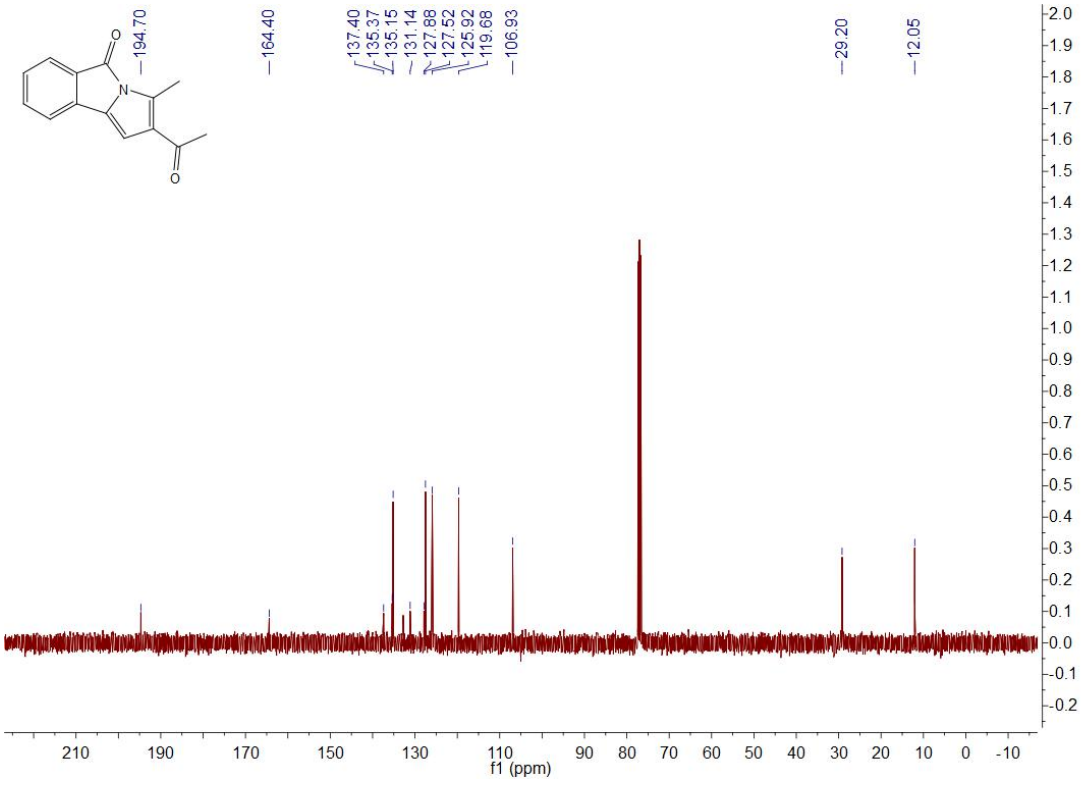

7.72
7.70
7.47
7.46
7.45
7.45
7.41
7.39
7.37
7.26
7.25
7.23
7.21
7.19
7.16
7.15
7.14
7.13
2.74
2.08
1.03
1.00
1.08
1.09
1.08
2.09
3.00
3.06
f1 (ppm)

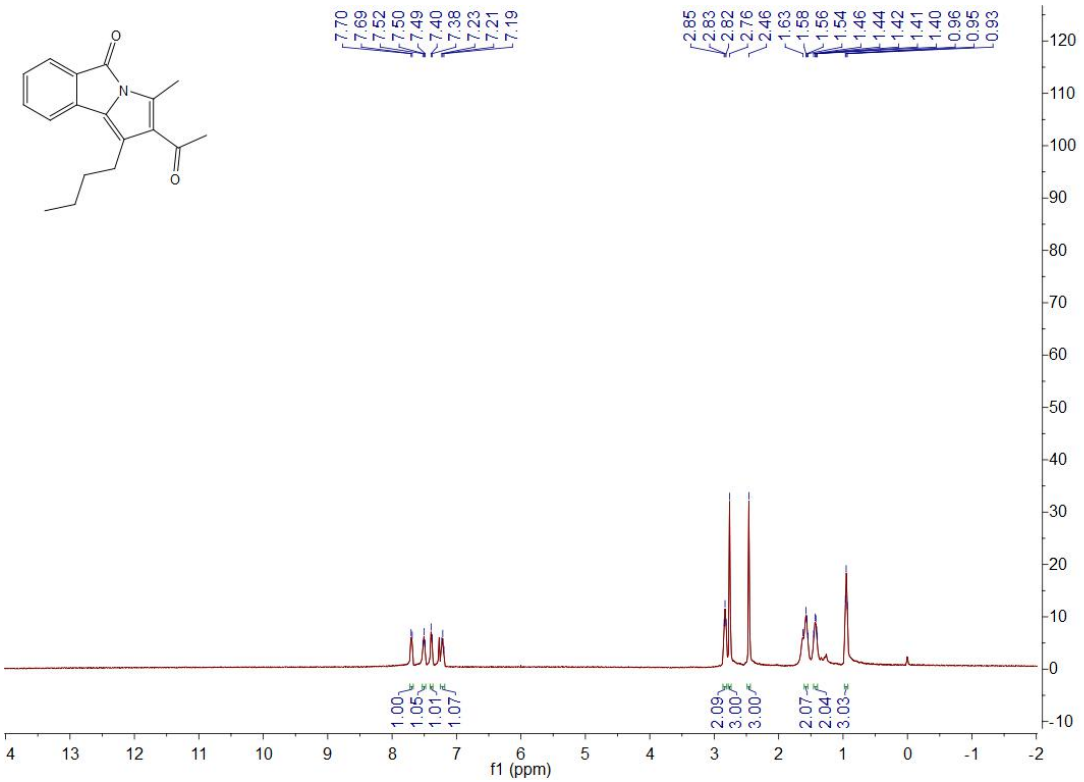

196.27
163.94
136.85
135.03
134.92
133.59
131.49
130.97
129.29
128.29
127.69
127.61
126.83
125.92
119.90
115.51
30.59
11.95
f1 (ppm)

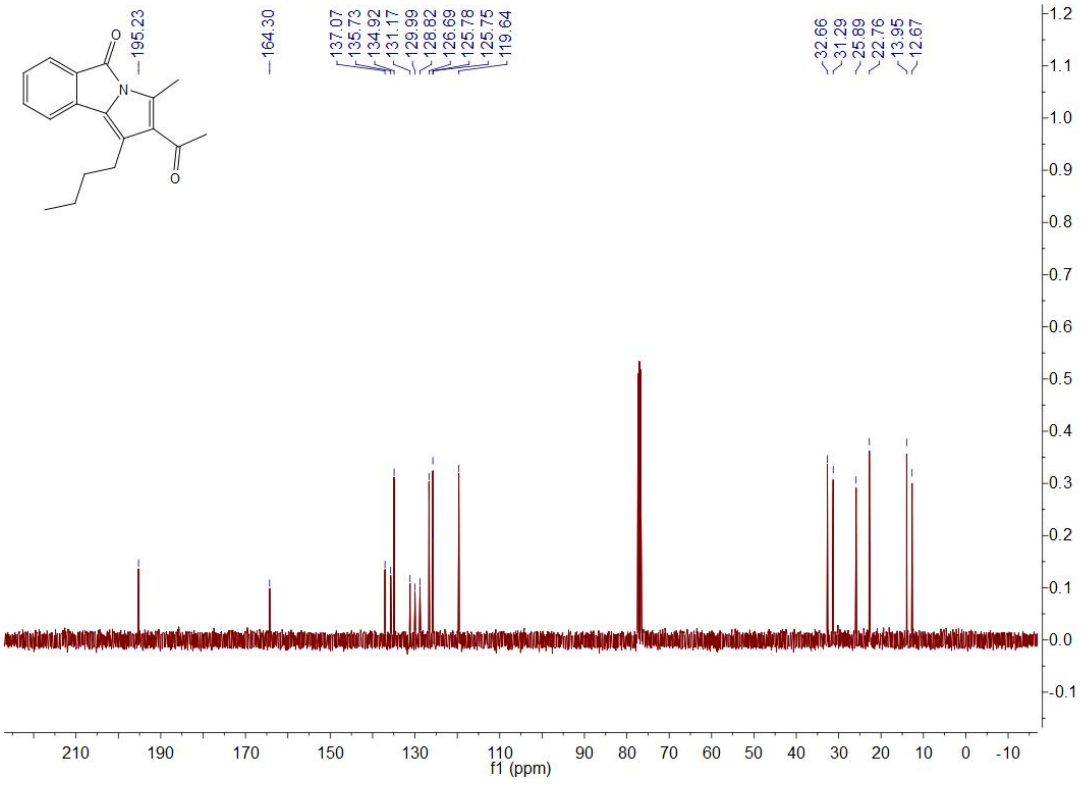

7.69
7.67
7.53
7.24
7.24
7.23
7.21
2.69
2.59
1.88
1.87
1.10
1.08
0.68
0.67
1.02
2.01
1.01
2.77
3.00
1.08
2.07
2.08
f1 (ppm)

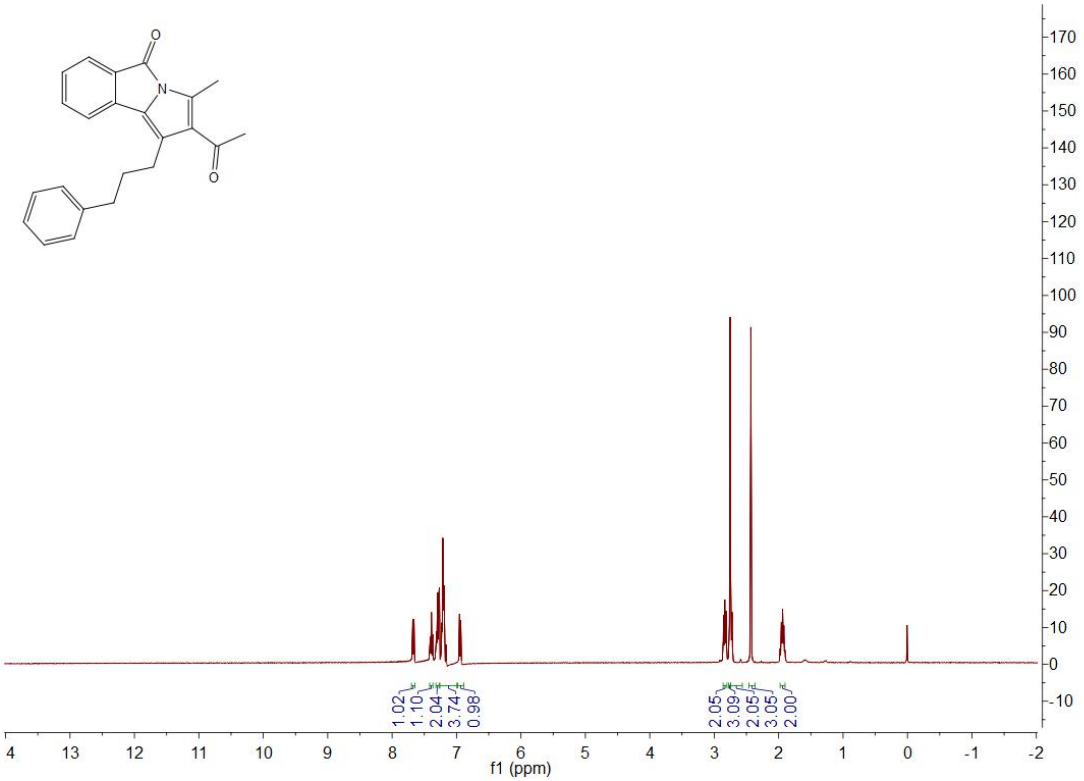

196.23
164.19
137.13
135.26
134.82
131.39
131.20
129.86
127.39
126.89
125.72
125.59
123.37
121.30
31.29
12.14
8.42
7.78
f1 (ppm)

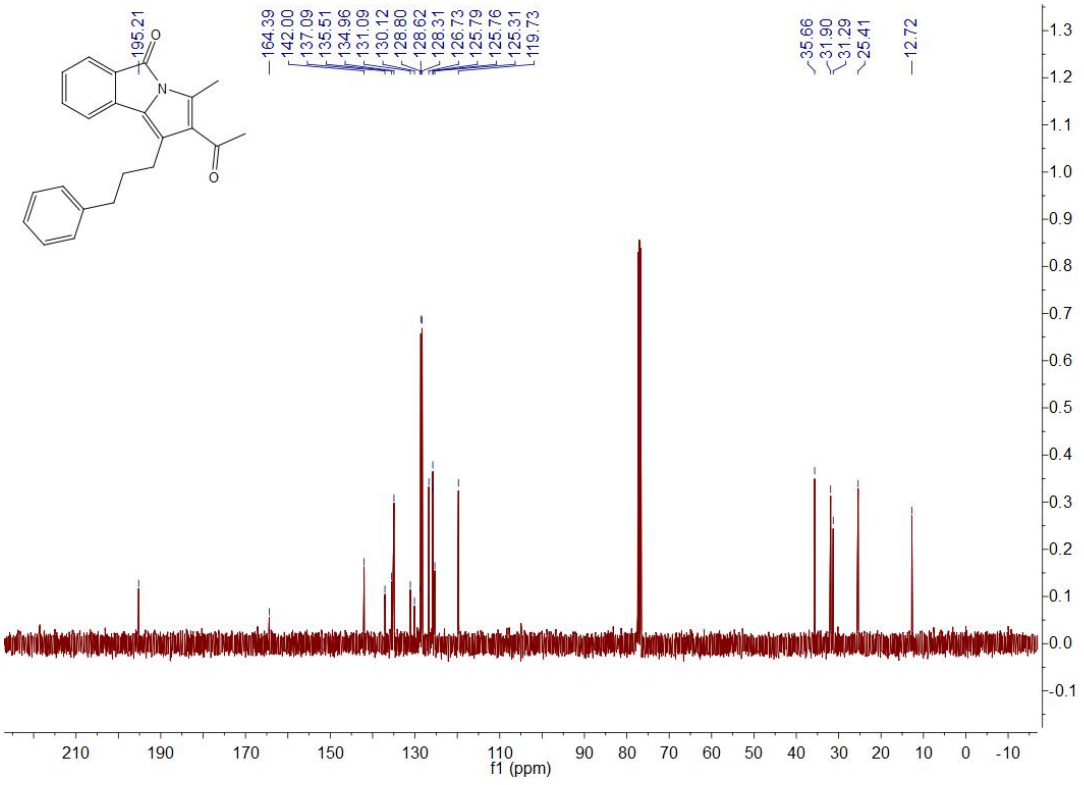

7.71
7.69
7.53
7.51
7.49
7.35
7.33
7.27
7.25
6.53
2.79
2.42
1.05
1.10
1.08
1.02
1.02
3.00
3.01
f1 (ppm)

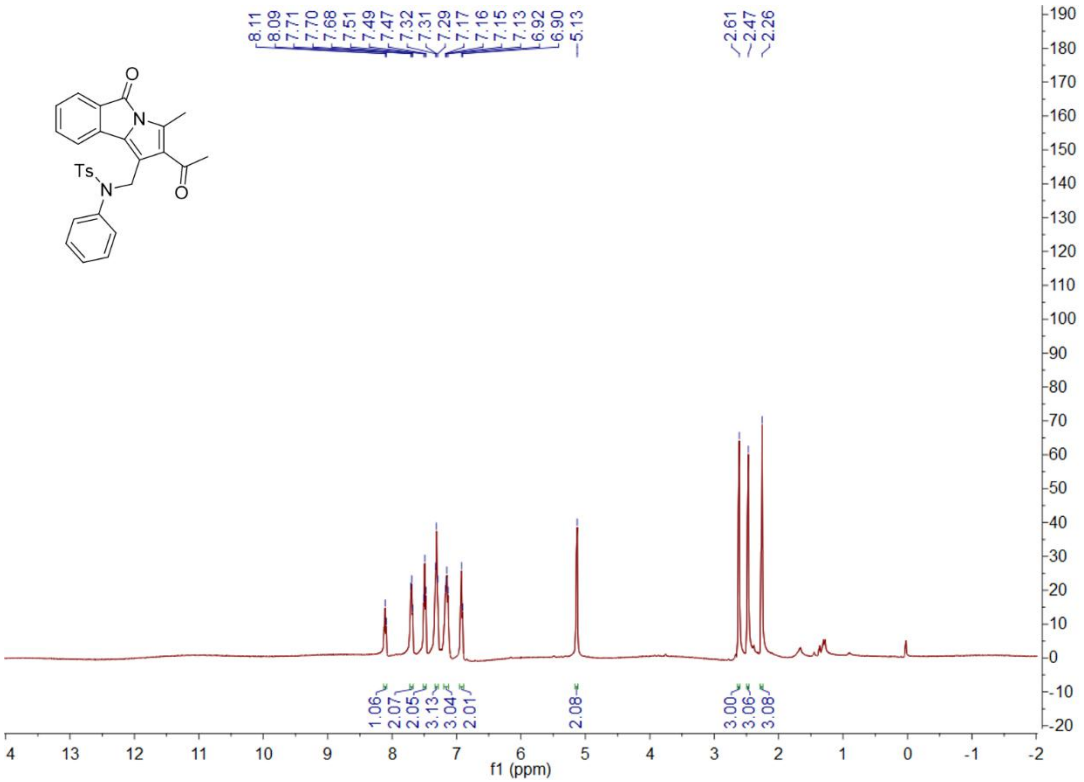

194.70
164.40
137.40
135.37
135.15
131.14
127.88
127.52
125.92
119.68
106.93
29.20
12.05
f1 (ppm)

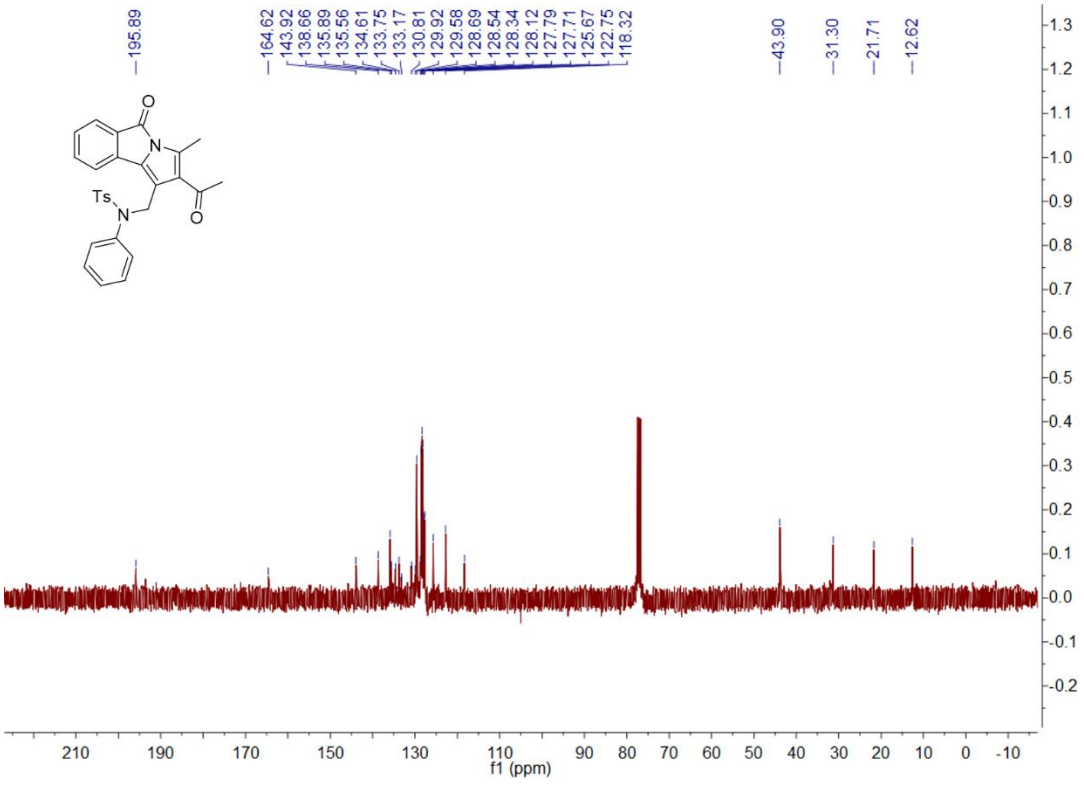

7.70
7.69
7.52
7.50
7.49
7.40
7.38
7.23
7.21
7.19
2.85
2.83
2.82
2.76
2.46
1.63
1.58
1.56
1.54
1.46
1.44
1.42
1.41
1.40
0.96
0.95
0.93
1.00
1.05
1.01
1.07
2.09
3.00
3.00
2.07
2.04
3.03
f1 (ppm)

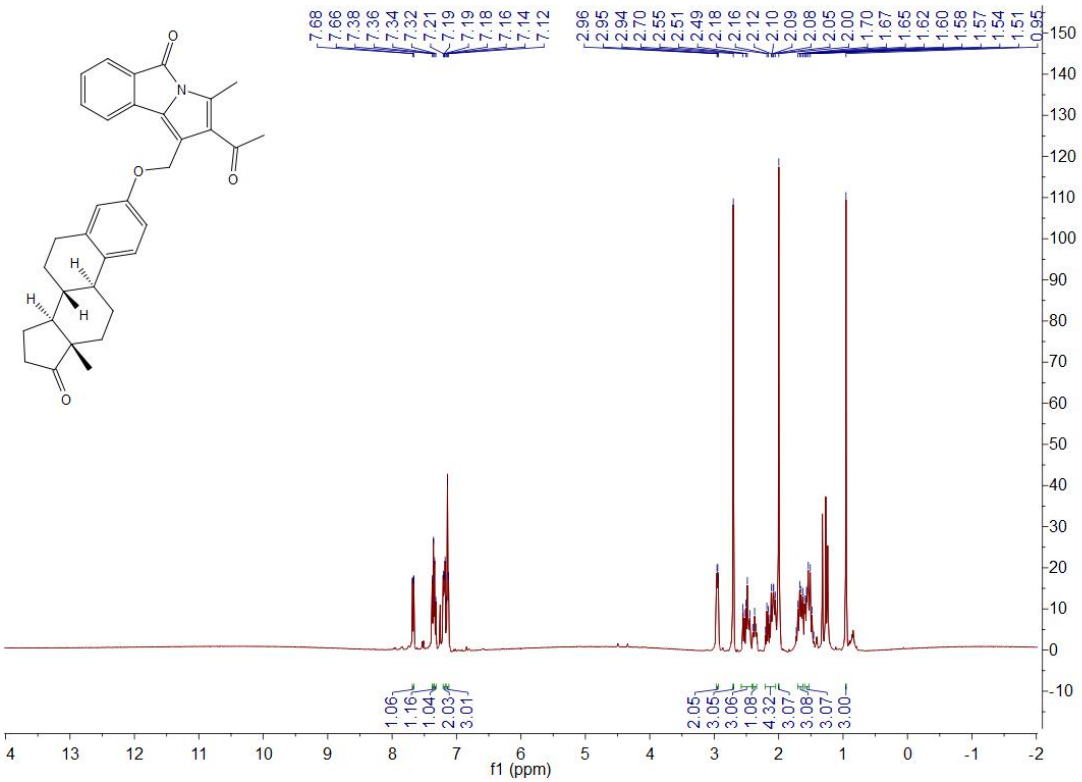

195.23
164.30
137.07
135.73
134.92
131.17
129.99
128.82
126.69
125.78
125.75
119.64
32.66
31.29
25.89
22.76
13.95
12.67
f1 (ppm)

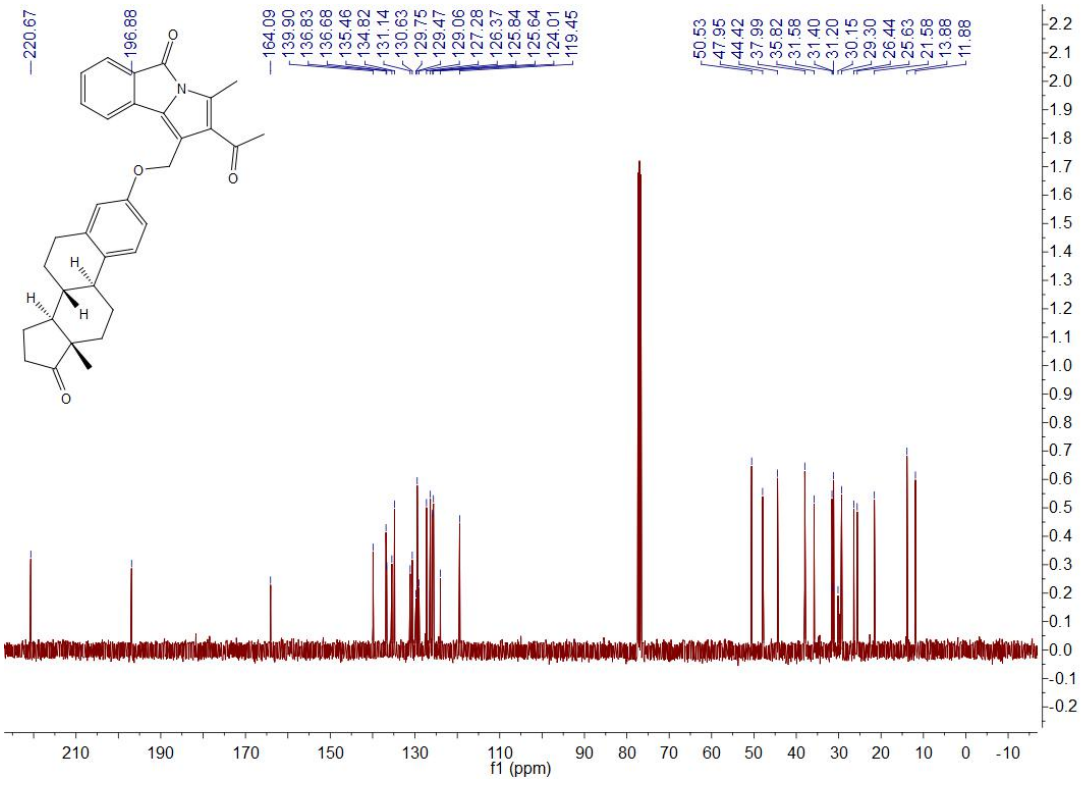

1.02
1.10
2.04
3.74
0.98
2.05
3.09
2.05
3.05
2.00
f1 (ppm)

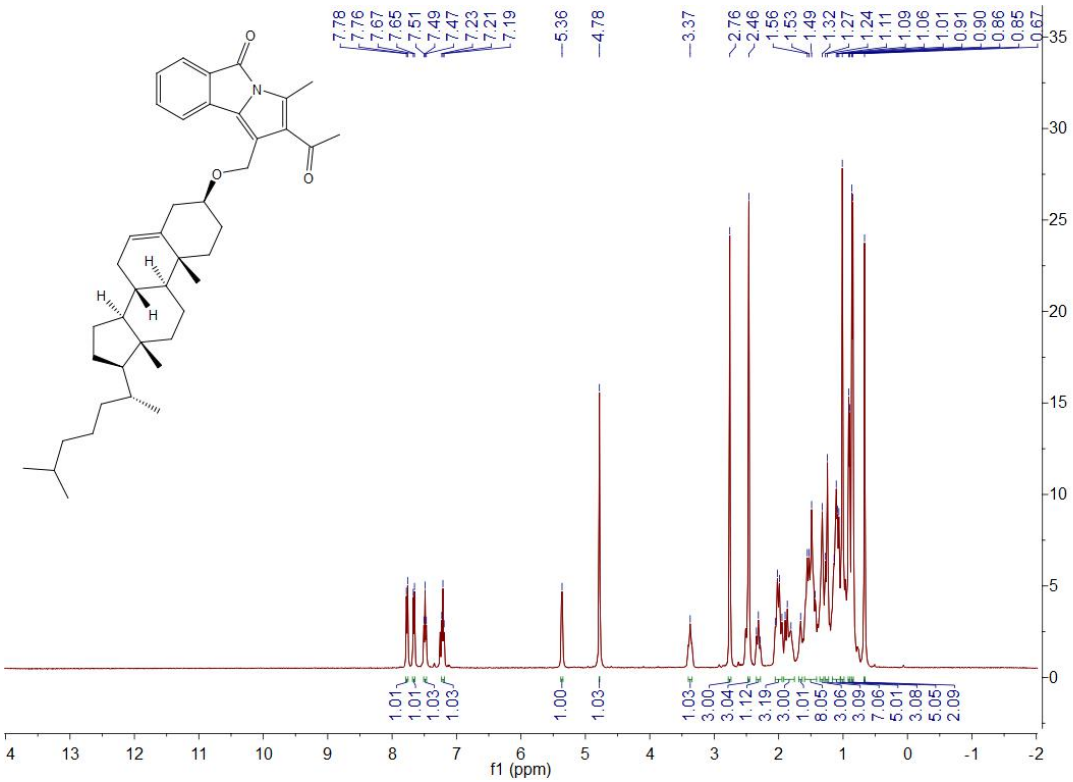

195.21
164.39
142.00
137.09
135.51
134.96
131.09
130.12
128.80
128.62
128.31
126.73
125.79
125.76
125.31
119.73
35.66
31.90
31.29
25.41
12.72
f1 (ppm)

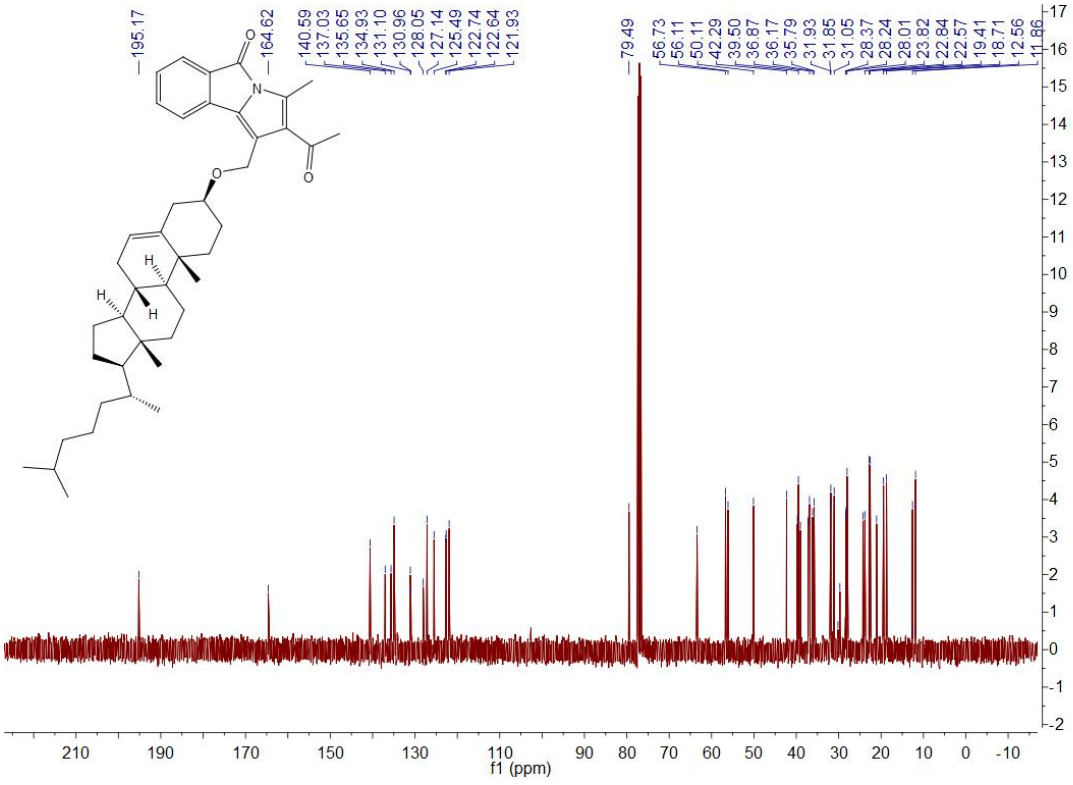

8.11
8.09
7.71
7.70
7.68
7.51
7.49
7.47
7.32
7.31
7.29
7.17
7.16
7.15
7.13
6.92
6.90
5.13
2.61
2.47
2.26
1.06
2.07
2.05
3.13
3.04
2.01
2.08
3.00
3.06
3.08
f1 (ppm)

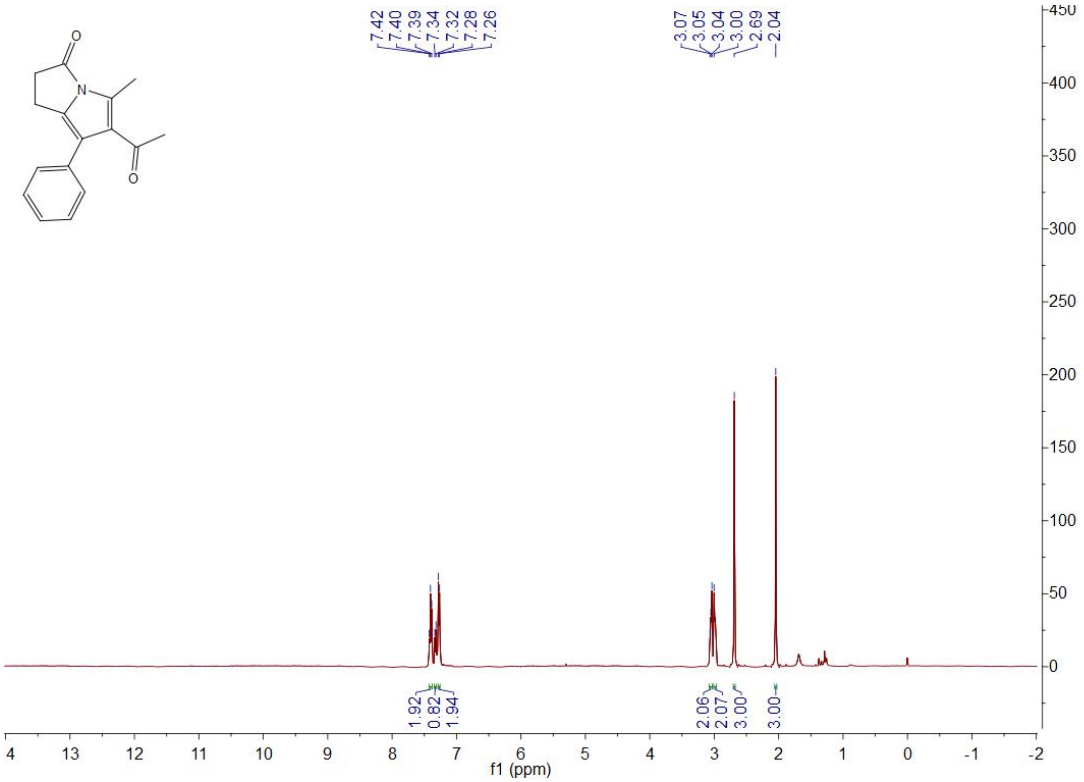

195.89
164.62
143.92
138.66
135.89
135.56
134.61
133.75
133.17
130.81
129.92
129.58
128.69
128.54
128.34
128.12
127.79
127.71
125.67
122.75
118.32
43.90
31.30
21.71
12.62
f1 (ppm)

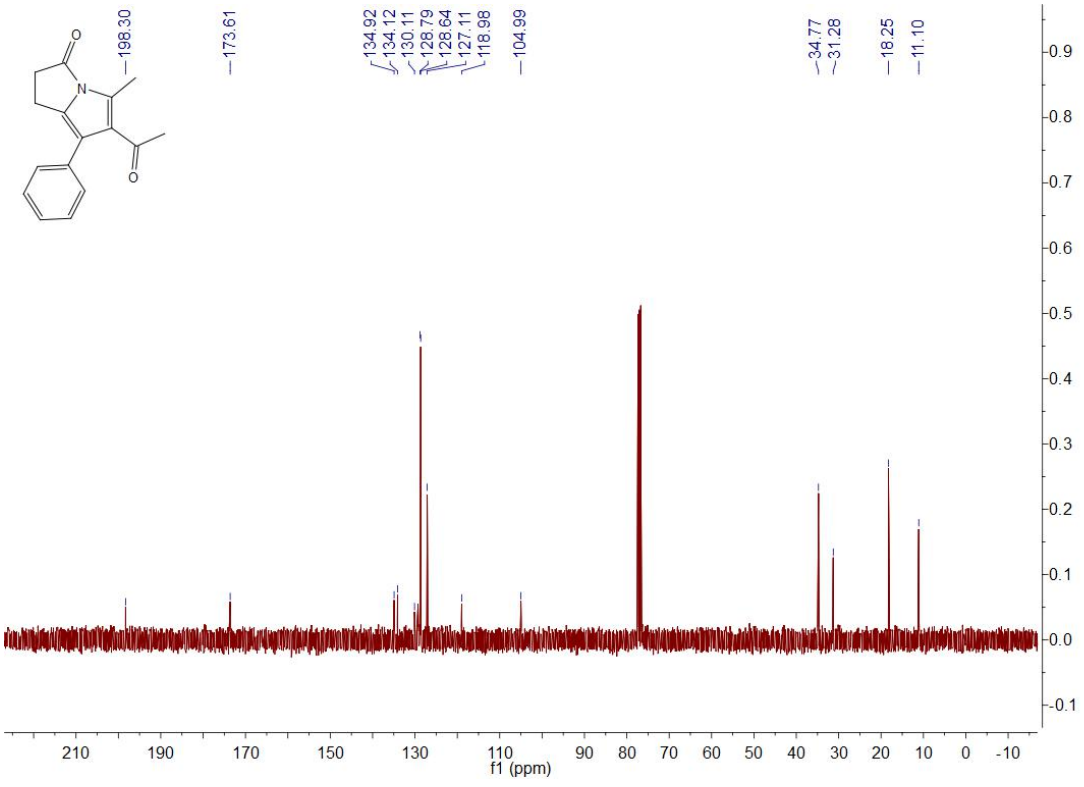

7.68
7.66
7.38
7.36
7.34
7.32
7.21
7.19
7.19
7.18
7.16
7.14
7.12
2.96
2.95
2.94
2.70
2.55
2.51
2.49
2.18
2.16
2.12
2.10
2.09
2.08
2.05
2.00
1.70
1.67
1.65
1.62
1.60
1.58
1.57
1.54
1.51
0.95
1.06
1.16
1.04
2.03
3.01
2.05
3.05
3.06
1.08
4.32
3.07
3.08
3.07
3.00
f1 (ppm)

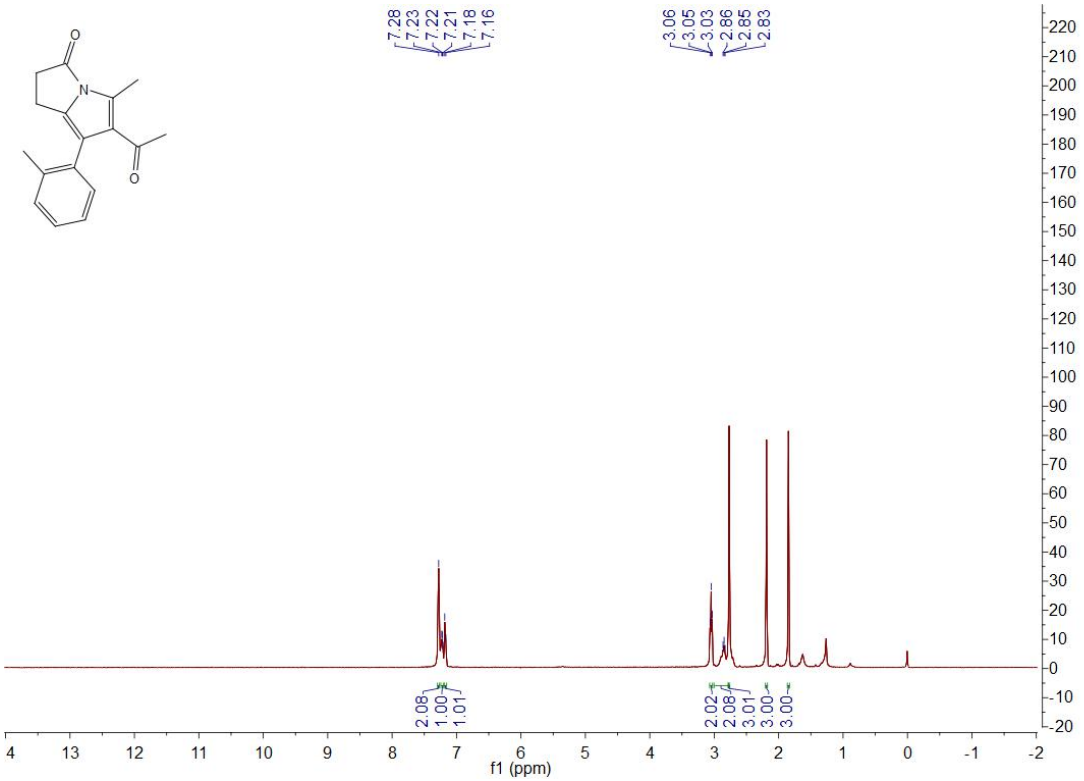

220.67
196.88
164.09
139.90
136.83
136.68
135.46
134.82
131.14
130.63
129.75
129.47
129.06
127.28
126.37
125.84
125.64
124.01
119.45
50.53
47.95
44.42
37.99
35.82
31.58
31.40
31.20
30.15
29.30
26.44
25.63
21.58
13.88
11.88
f1 (ppm)

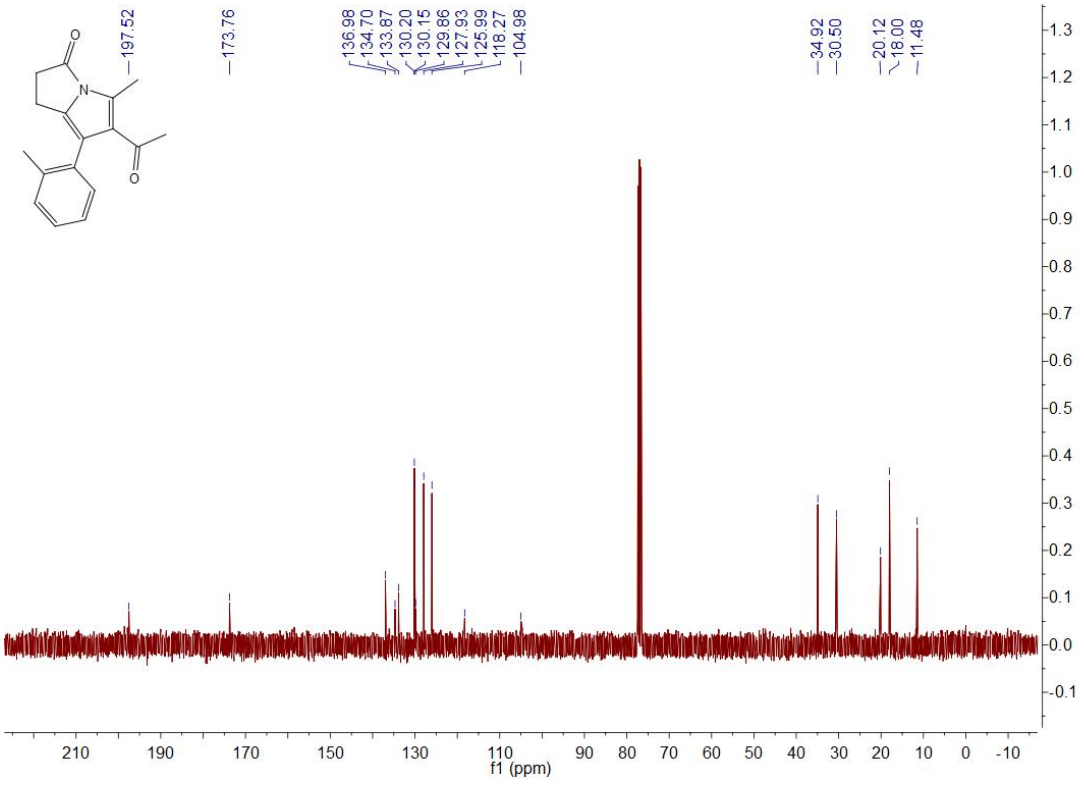

7.78
7.76
7.67
7.65
7.51
7.49
7.47
7.23
7.21
7.19
5.36
4.78
3.37
2.76
2.46
1.56
1.53
1.49
1.32
1.27
1.24
1.11
1.09
1.06
1.01
0.91
0.90
0.86
0.85
0.67
1.01
1.01
1.03
1.03
1.00
1.03
1.03
3.00
3.04
1.12
3.19
3.00
1.01
8.05
3.06
3.09
7.06
5.01
3.08
5.05
2.09
f1 (ppm)

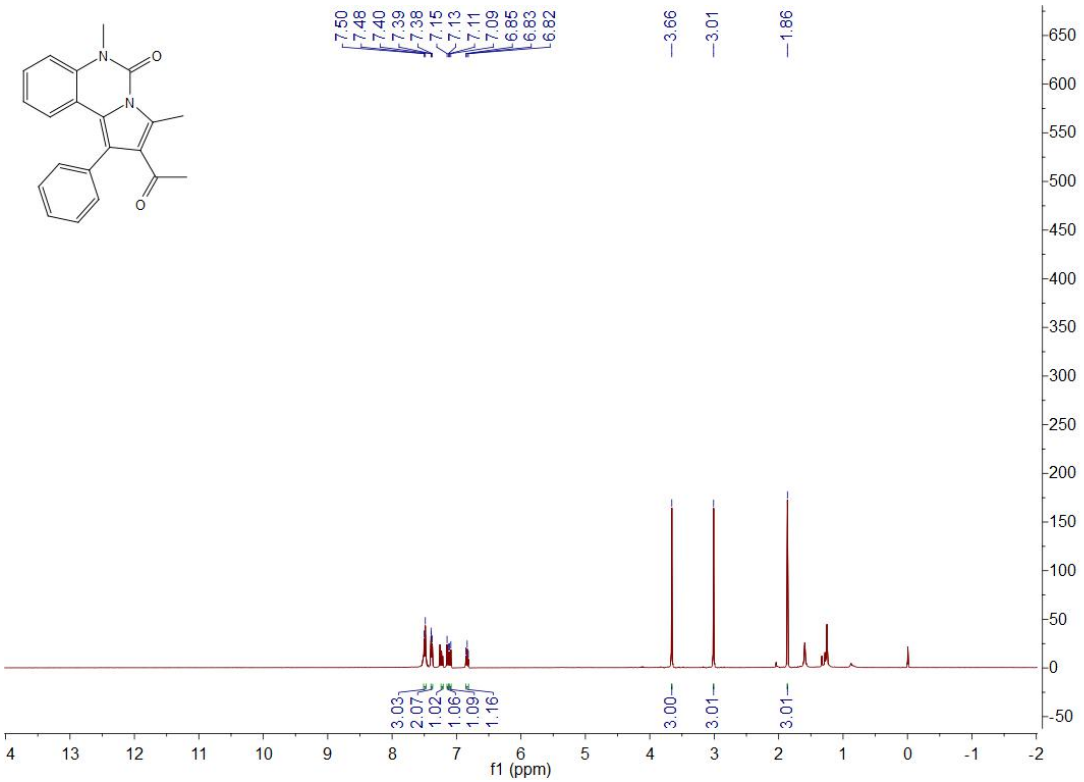

195.17
164.62
140.59
137.03
135.65
134.93
131.10
130.96
128.05
127.14
125.49
122.74
122.64
121.93
79.49
56.73
56.11
50.11
42.29
39.50
36.87
36.17
35.79
31.93
31.85
31.05
28.37
28.24
28.01
23.82
22.84
22.57
19.41
18.71
12.56
11.86
f1 (ppm)

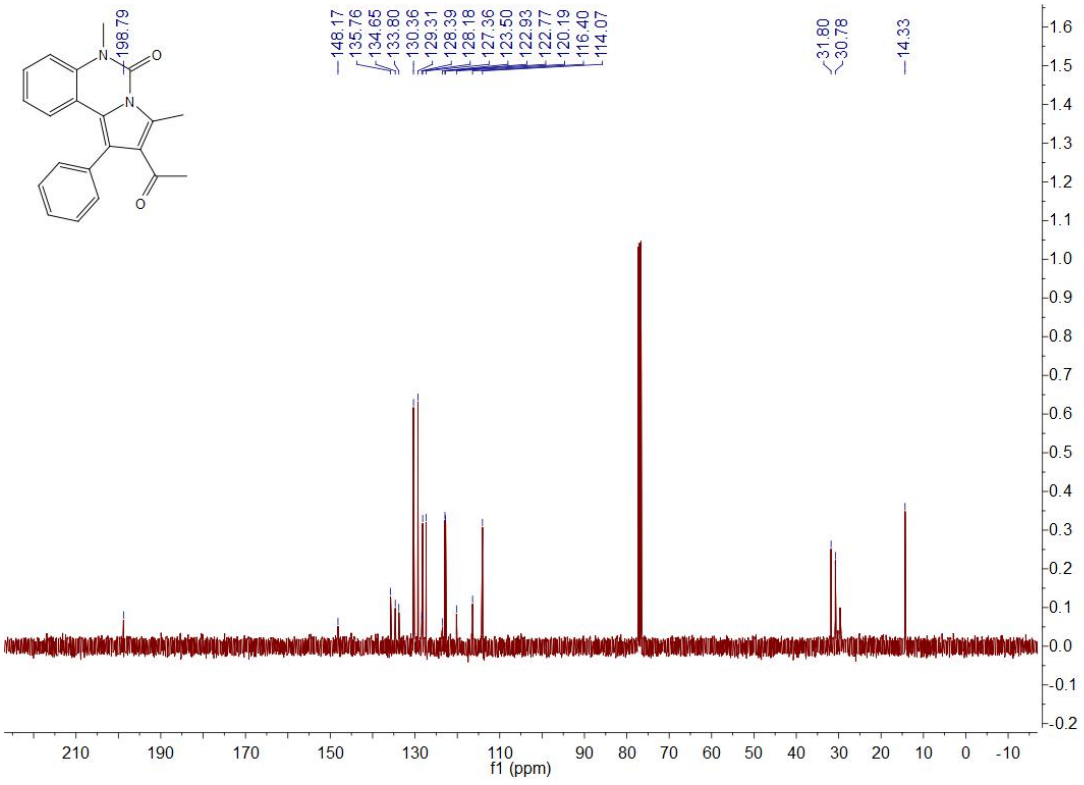

7.42
7.40
7.39
7.34
7.32
7.28
7.26
3.07
3.05
3.04
3.00
2.69
2.04
1.92
0.82
1.94
2.06
2.07
3.00
3.00
f1 (ppm)

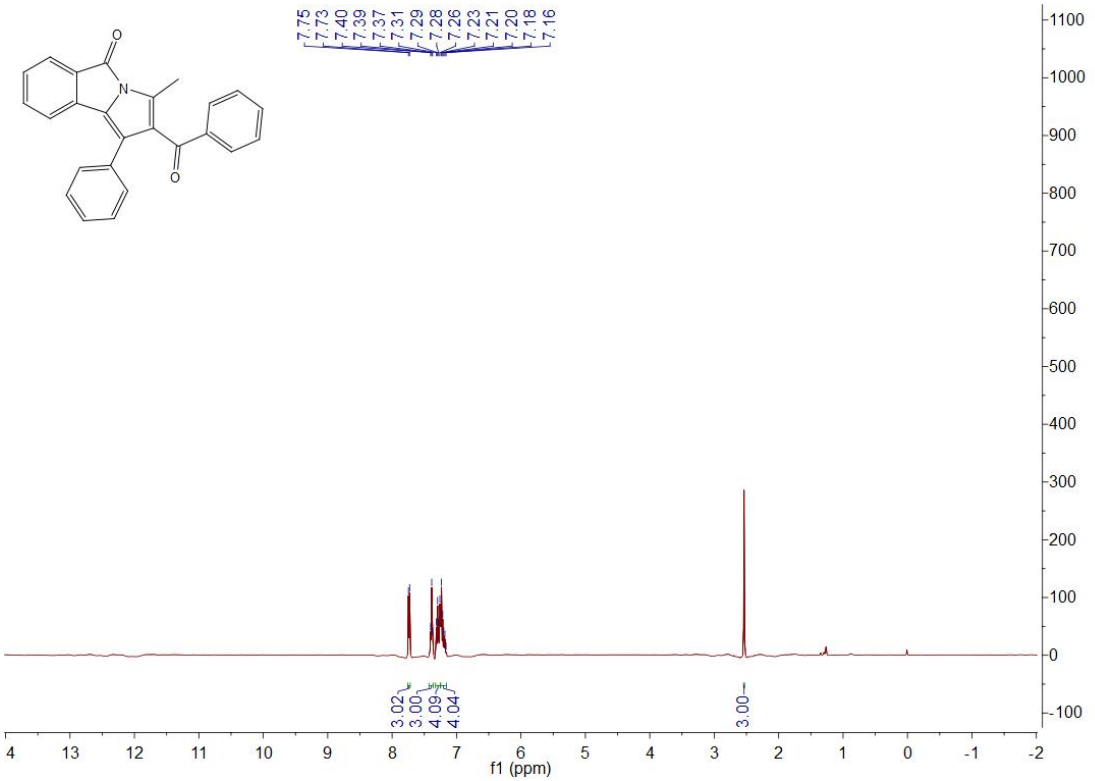

198.30
173.61
134.92
134.12
130.11
128.79
128.64
127.11
118.98
104.99
34.77
31.28
18.25
11.10
f1 (ppm)

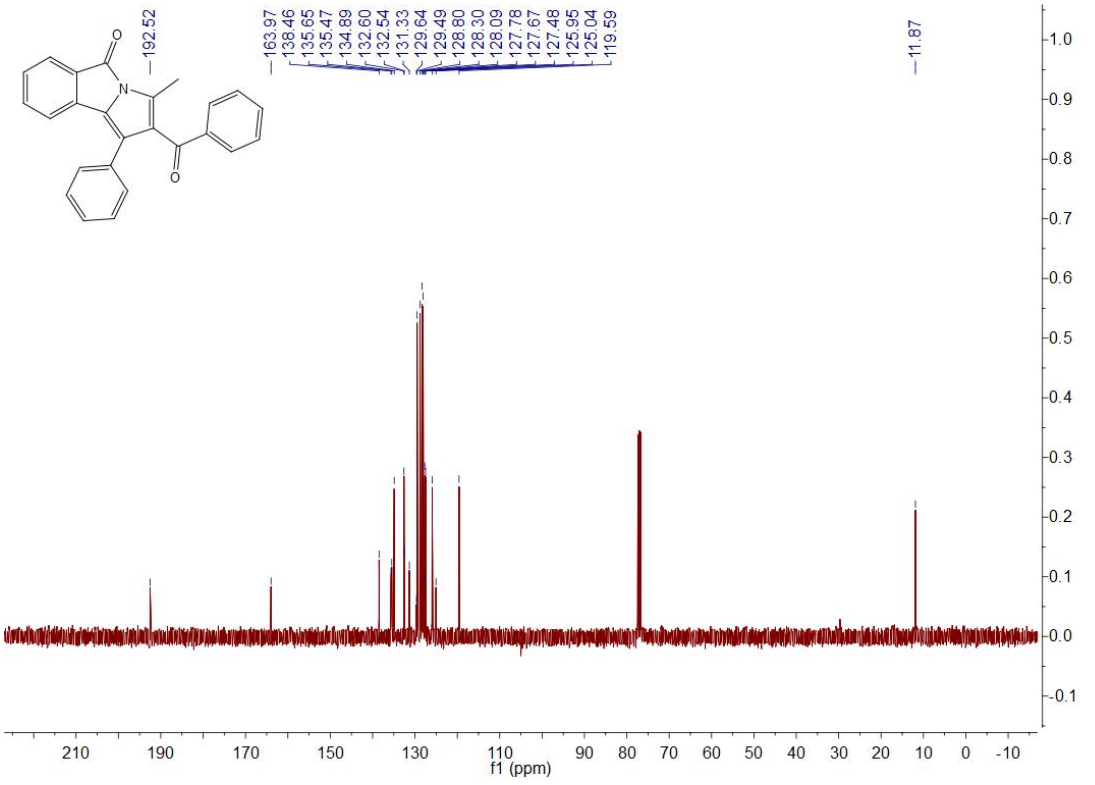

7.28
7.23
7.22
7.21
7.18
7.16
3.06
3.05
3.03
2.86
2.85
2.83
2.08
1.00
1.01
2.02
2.08
3.01
3.00
3.00
f1 (ppm)

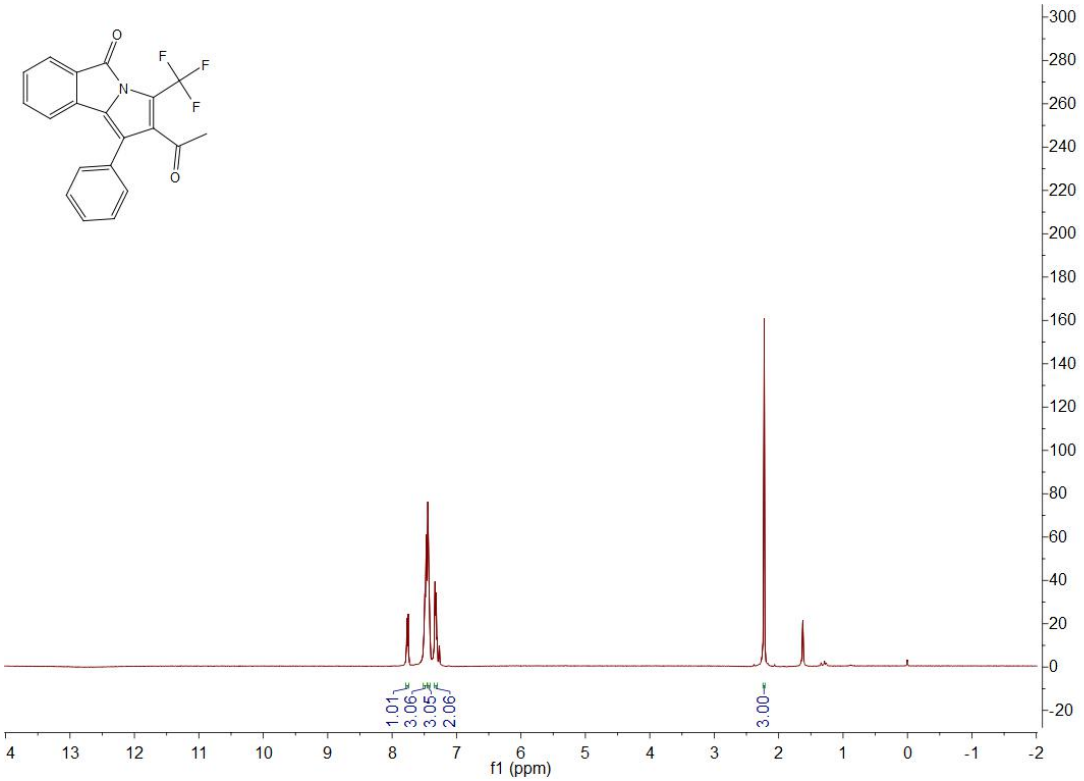

197.52
173.76
136.98
134.70
133.87
130.20
130.15
129.86
127.93
125.99
118.27
104.98
34.92
30.50
20.12
18.00
11.48
f1 (ppm)

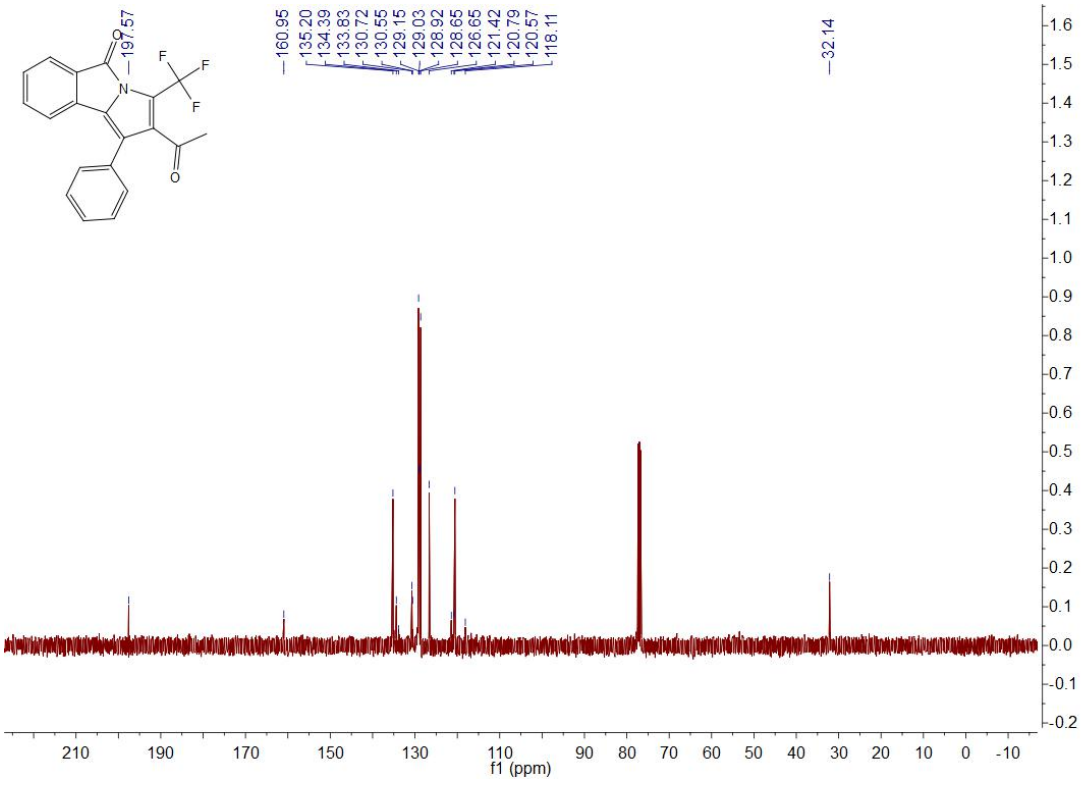

7.50
7.48
7.40
7.39
7.38
7.15
7.13
7.11
7.09
6.85
6.83
6.82
3.66
3.01
1.86
3.03
2.07
1.02
1.06
1.09
1.16
3.00
3.01
3.01
f1 (ppm)

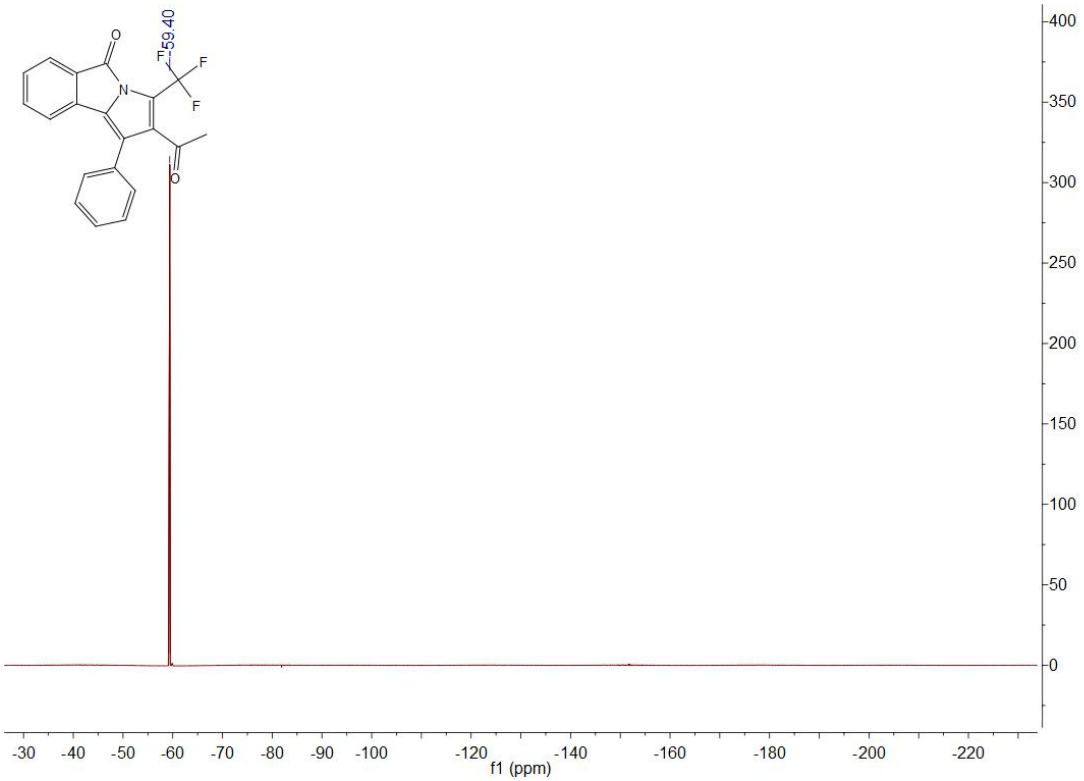

198.79
148.17
135.76
134.65
133.80
130.36
129.31
128.39
128.18
127.36
123.50
122.93
122.77
120.19
116.40
114.07
31.80
30.78
14.33
f1 (ppm)

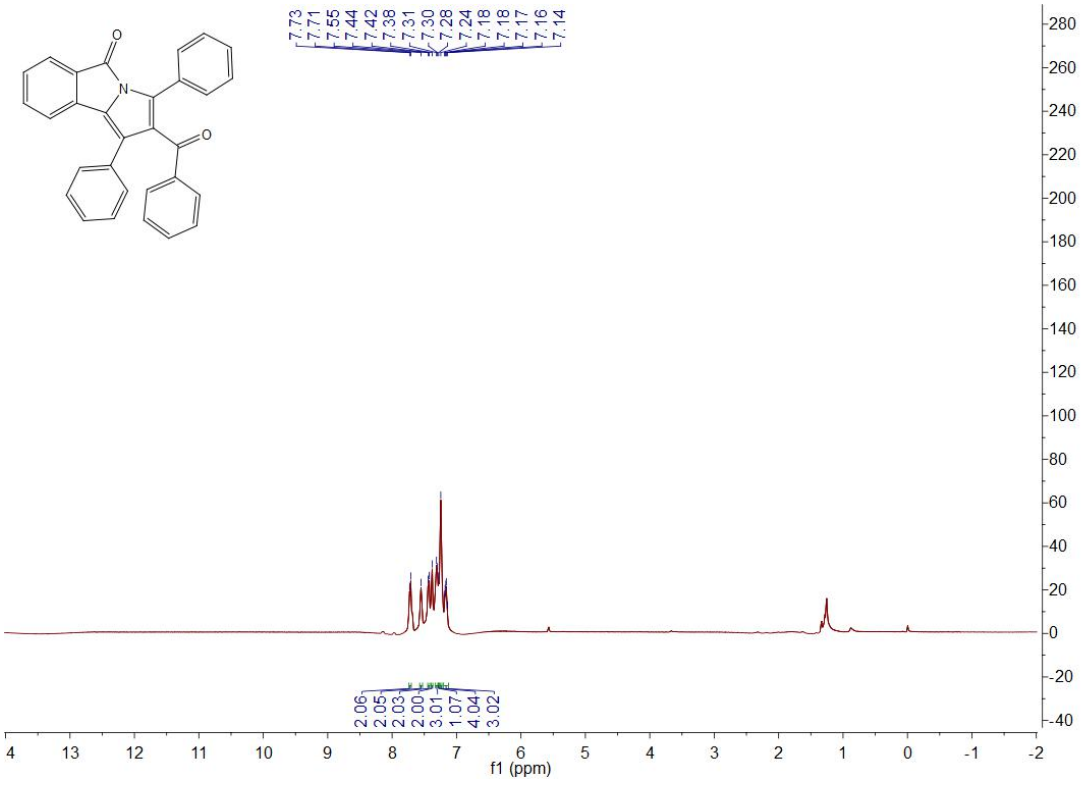

7.75
7.73
7.40
7.39
7.37
7.31
7.29
7.28
7.26
7.23
7.21
7.20
7.18
7.16
3.02
3.00
4.09
4.04
3.00
f1 (ppm)

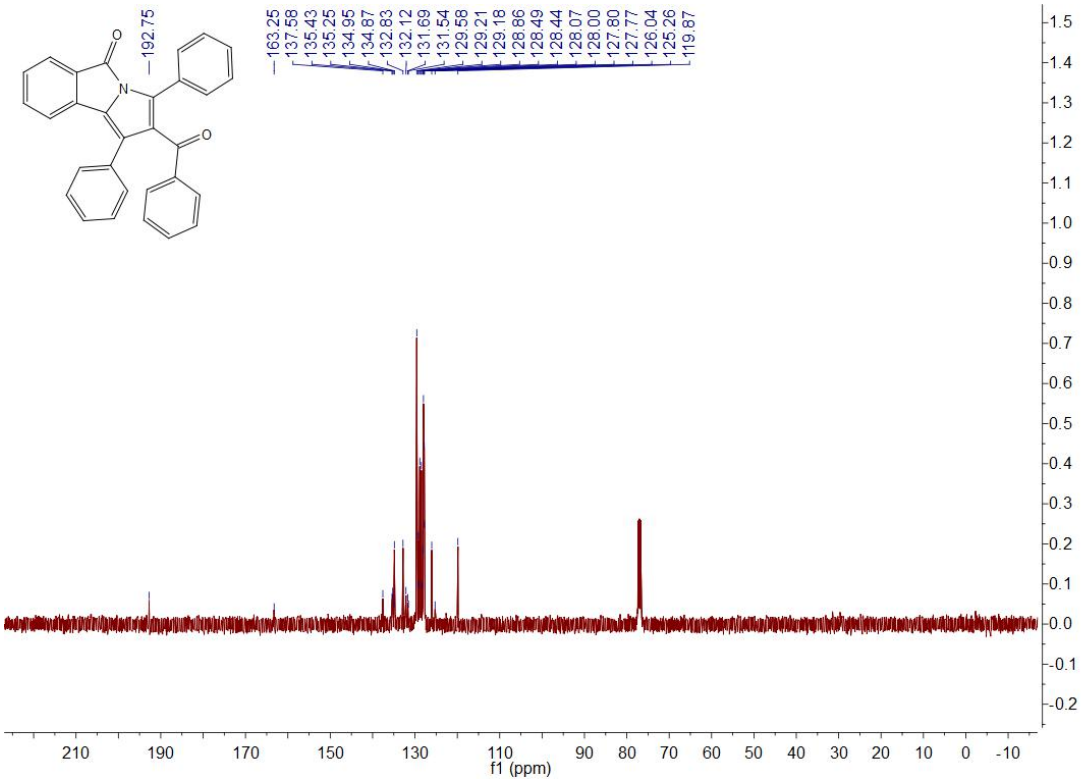

192.52
163.97
138.46
135.65
135.47
134.89
132.60
132.54
131.33
129.64
129.49
128.80
128.30
128.09
127.78
127.67
127.48
125.95
125.04
119.59
11.87
f1 (ppm)

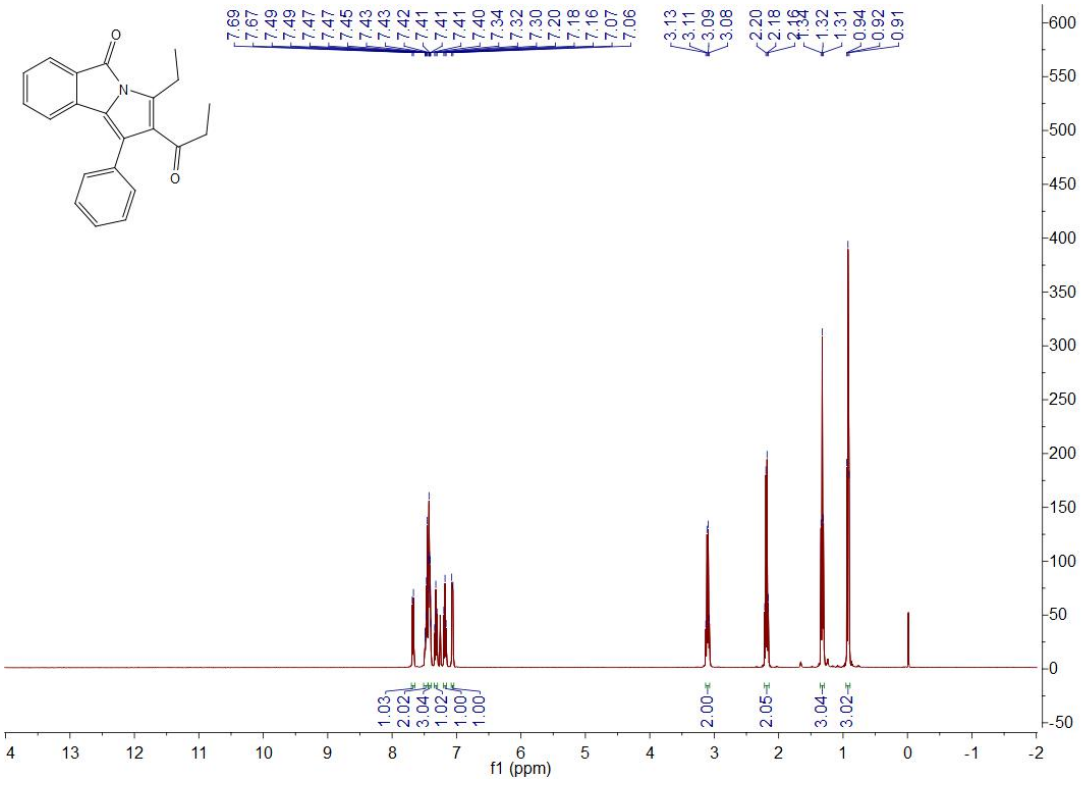

1.01
3.06
3.05
2.06
3.00
f1 (ppm)

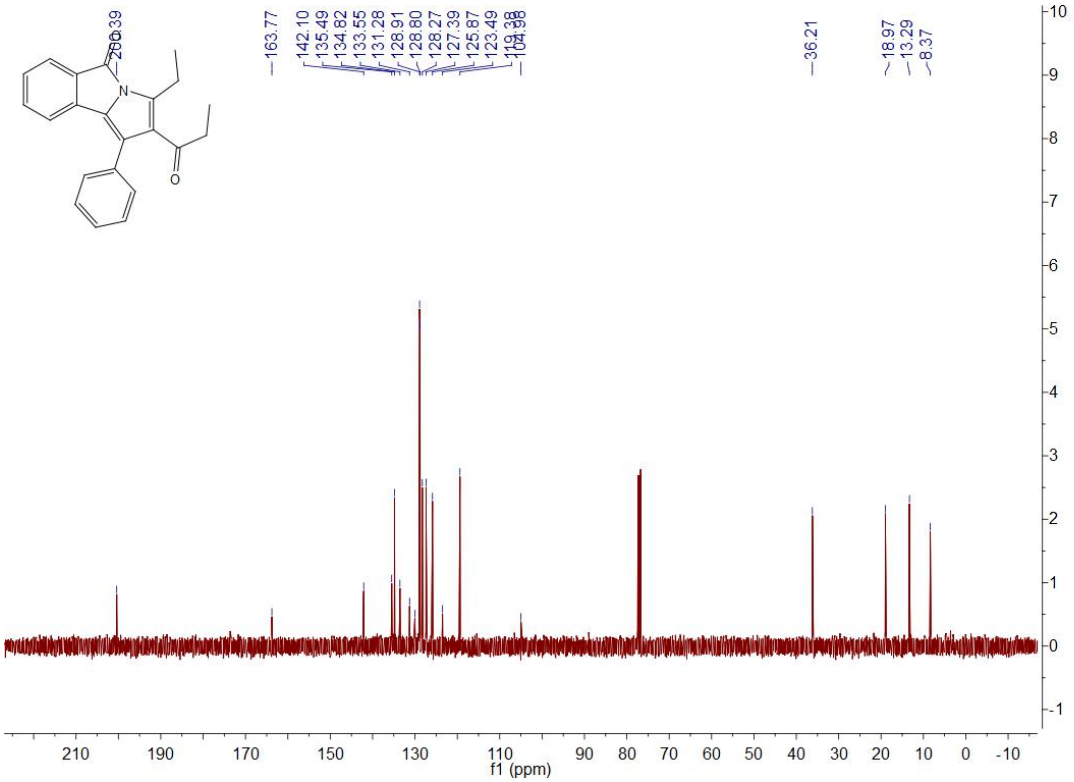

197.57
160.95
135.20
134.39
133.83
130.72
130.55
129.15
129.03
128.92
128.65
126.65
121.42
120.79
120.57
118.11
32.14
f1 (ppm)

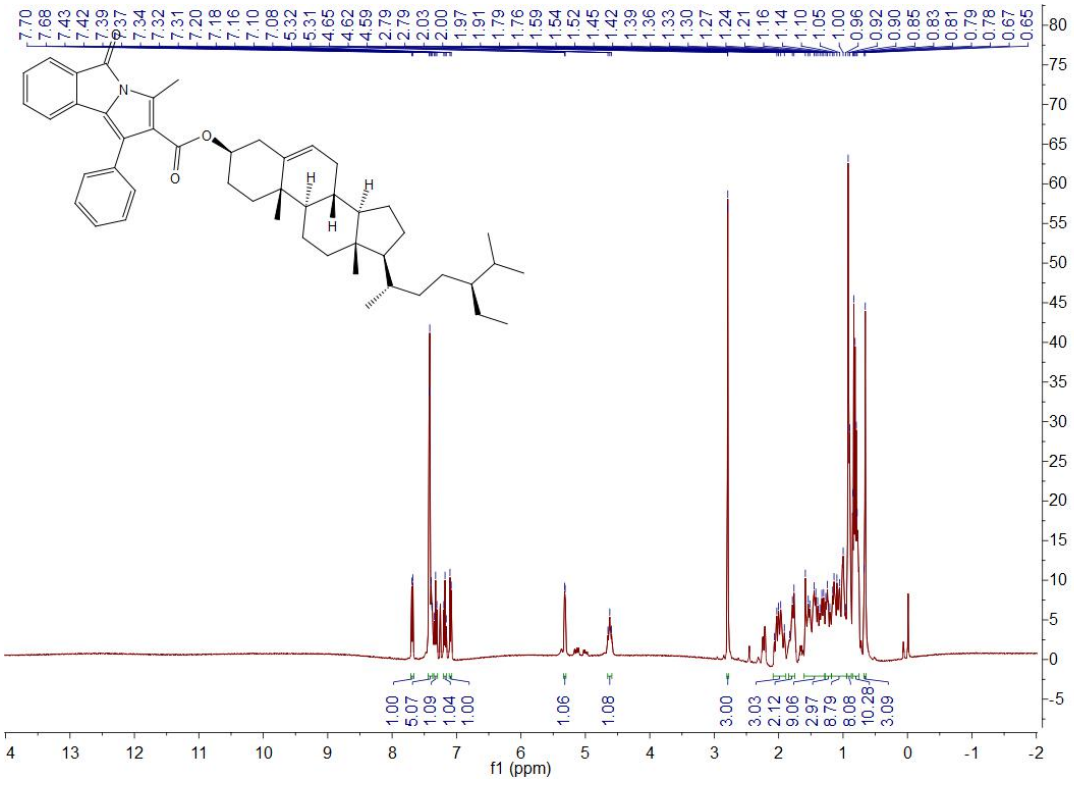

-59.40
F
F
O
O
N
f1 (ppm)

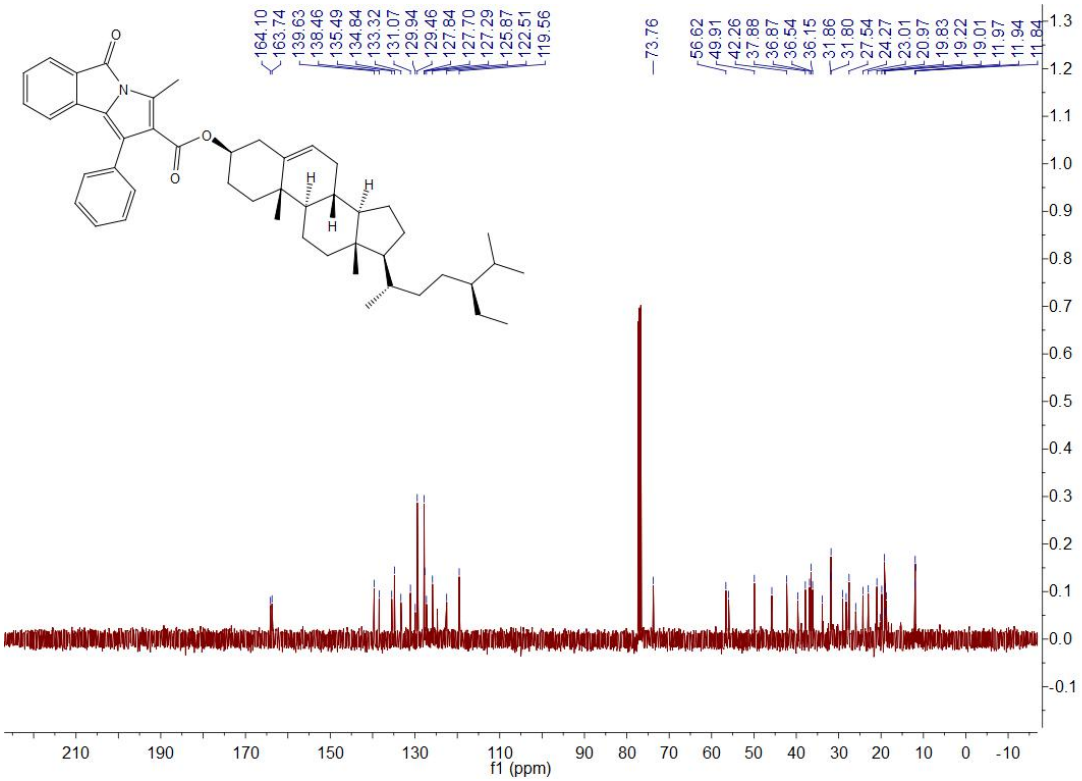

7.73
7.71
7.55
7.44
7.42
7.38
7.31
7.30
7.28
7.24
7.18
7.17
7.16
7.14
2.06
2.05
2.03
2.00
3.01
1.07
4.04
3.02
O
O
N
f1 (ppm)

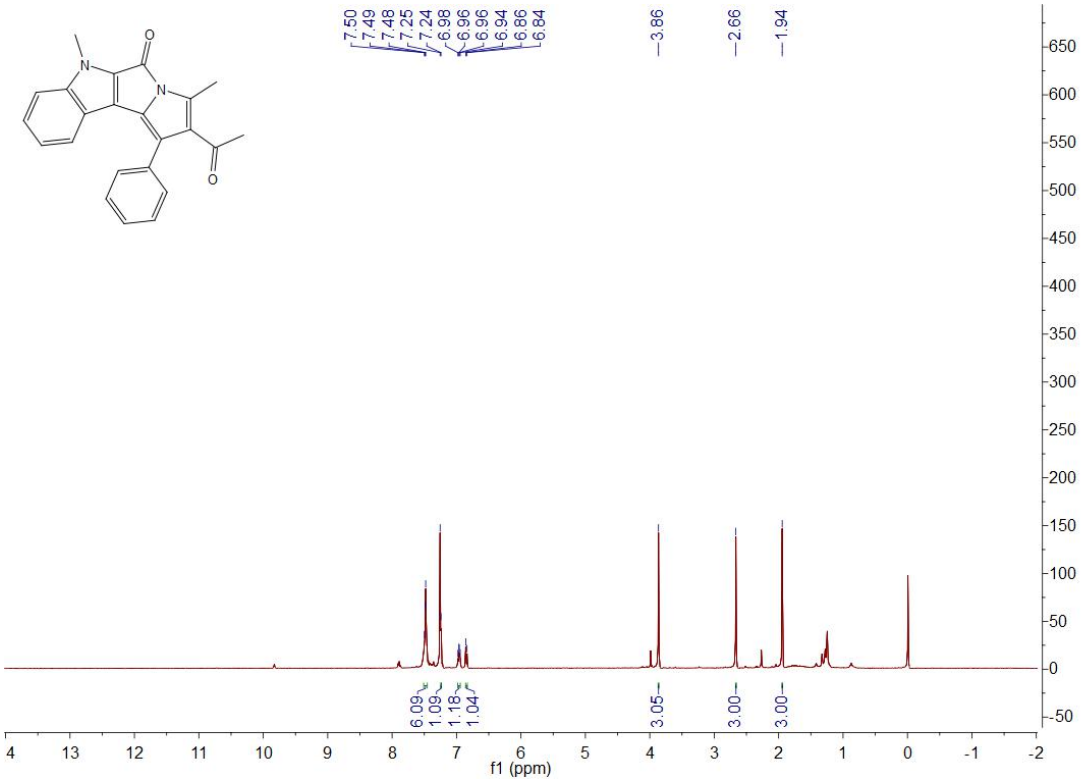

192.75
163.25
137.58
135.43
135.25
134.95
134.87
132.83
132.12
131.69
131.54
129.58
129.21
129.18
128.86
128.49
128.44
128.07
128.00
127.80
127.77
126.04
125.26
119.87
f1 (ppm)

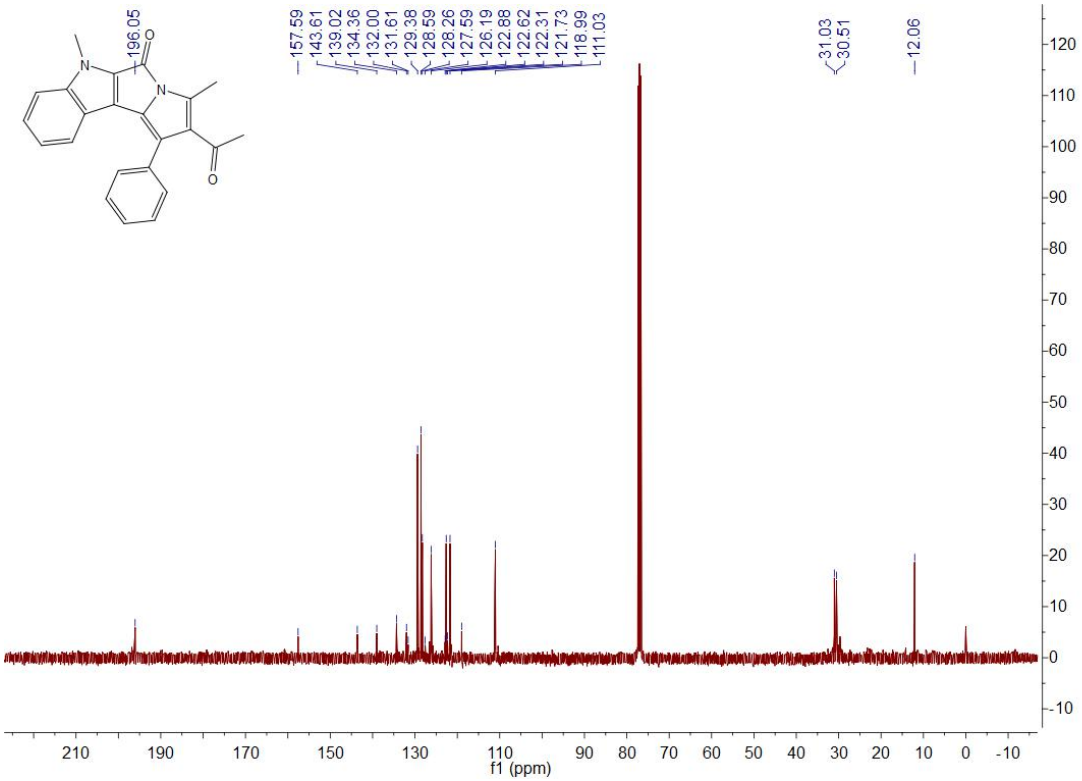

7.69
7.67
7.49
7.49
7.47
7.47
7.45
7.43
7.42
7.41
7.41
7.40
7.34
7.32
7.30
7.20
7.18
7.16
7.07
7.06
3.13
3.11
3.09
3.08
2.20
2.18
1.34
1.32
1.31
0.94
0.92
0.91
1.03
2.02
3.04
1.02
1.00
1.00
2.00
2.05
3.04
3.02
f1 (ppm)

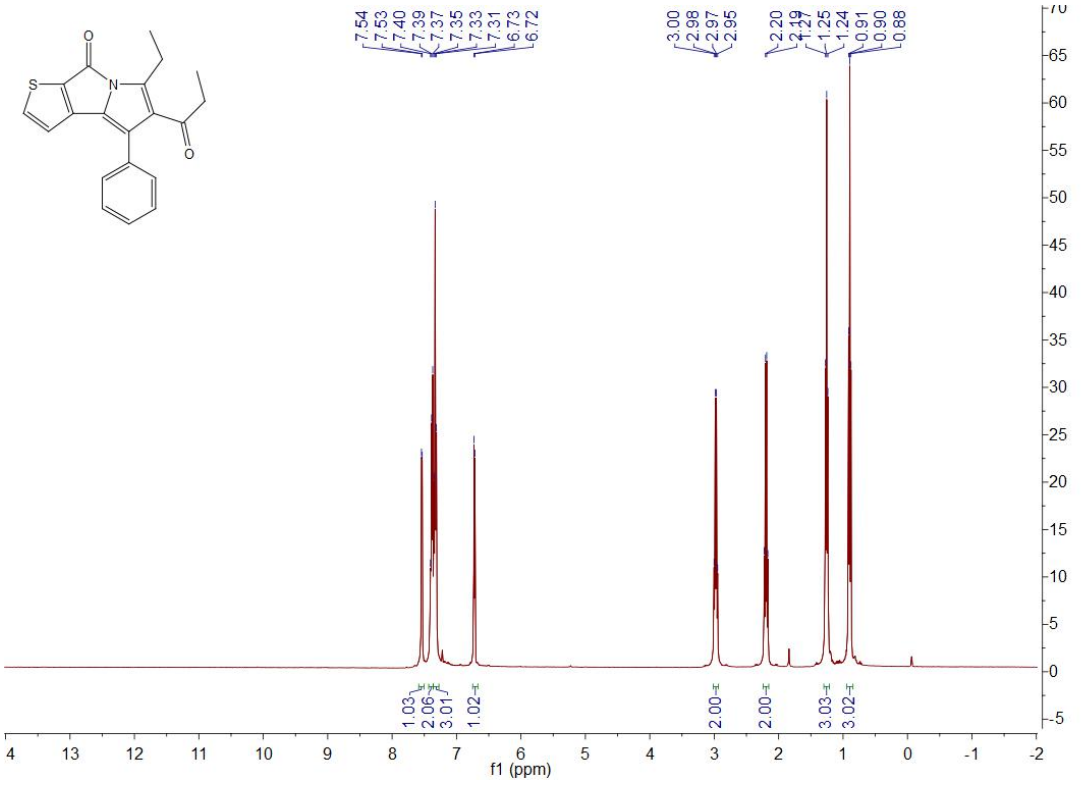

200.39
163.77
142.10
135.49
134.82
133.55
131.28
128.91
128.80
128.27
127.39
125.87
123.49
36.21
18.97
13.29
8.37
f1 (ppm)

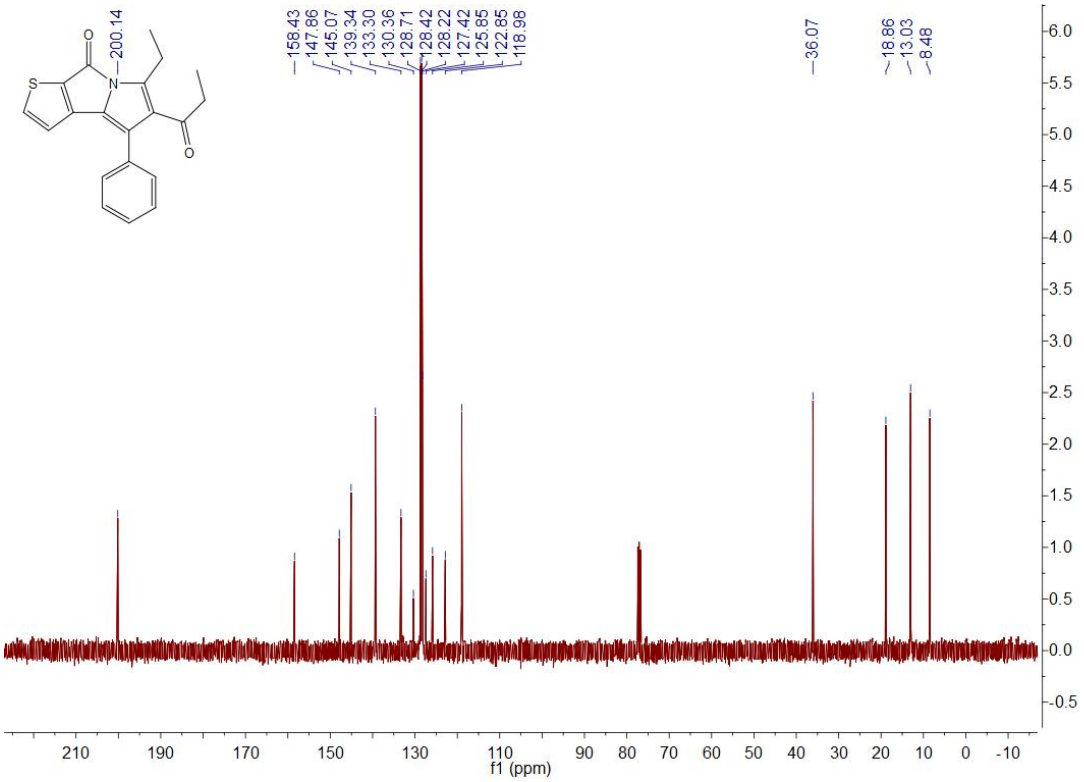

7.70
7.68
7.43
7.42
7.39
7.37
7.34
7.32
7.31
7.20
7.18
7.16
7.10
7.08
5.32
5.31
4.65
4.62
4.59
2.79
2.79
2.03
2.00
1.97
1.91
1.79
1.76
1.59
1.54
1.52
1.45
1.42
1.39
1.36
1.33
1.30
1.27
1.24
1.21
1.16
1.14
1.10
1.05
1.00
0.96
0.92
0.90
0.85
0.83
0.81
0.79
0.78
0.67
0.65
1.00
5.07
1.09
1.04
1.00
1.06
1.08
3.00
3.03
2.12
9.06
2.97
8.79
8.08
10.28
3.09
f1 (ppm)


164.10
163.74
139.63
138.46
135.49
134.84
133.32
131.07
129.94
129.46
127.84
127.70
127.29
125.87
122.51
119.56
73.76
56.62
49.91
42.26
37.88
36.87
36.54
36.15
31.86
31.80
27.54
24.27
23.01
20.97
19.83
19.22
19.01
11.97
11.94
11.84
f1 (ppm)


7.50
7.49
7.48
7.25
7.24
6.98
6.96
6.96
6.94
6.86
6.84
3.86
2.66
1.94
6.09
1.09
1.18
1.04
3.05
3.00
3.00
f1 (ppm)


196.05
157.59
143.61
139.02
134.36
132.00
131.61
129.38
128.59
128.26
127.59
126.19
122.88
122.62
122.31
121.73
118.99
111.03
31.03
30.51
12.06
f1 (ppm)


7.54
7.53
7.40
7.39
7.37
7.35
7.33
7.31
6.73
6.72
3.00
2.98
2.97
2.95
2.20
2.19
1.27
1.25
1.24
0.91
0.90
0.88
1.03
2.06
3.01
1.02
2.00
2.00
3.03
3.02
f1 (ppm)


200.14
158.43
147.86
145.07
139.34
133.30
130.36
128.71
128.42
128.22
127.42
125.85
122.85
118.98
36.07
18.86
13.03
8.48
f1 (ppm)