

Aryl Iodine Catalysed Divergent Synthesis of Isobenzofuranone and Isocoumarins via Oxidative 1,2-Aryl Migration/Elimination

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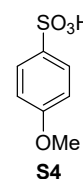
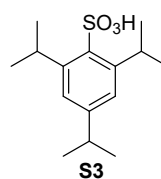
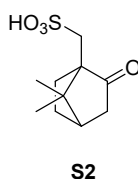
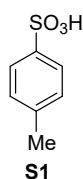
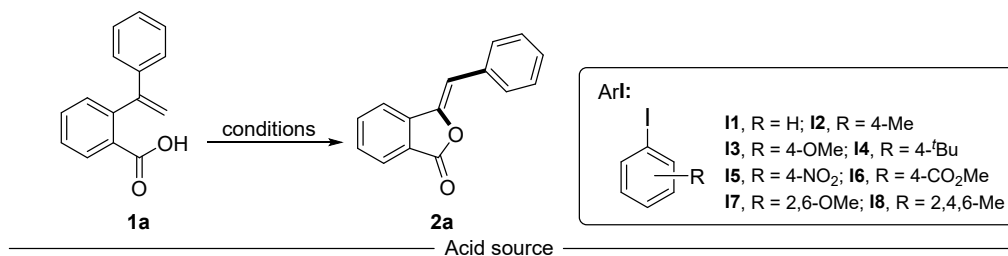
I. General Information

^1H and ^{13}C NMR spectra were recorded on a 400 MHz or 600 MHz spectrometer at 25 °C. Chemical shifts values are given in ppm and referred as the internal standard to TMS: 0.00 ppm. Chemical shifts were expressed in parts per million (δ) downfield from the internal standard tetramethylsilane, and were reported as s (singlet), d (doublet), t (triplet), q (quadruple), dd (doublet of doublet), m (multiplet), etc. The coupling constants J , are reported in Hertz (Hz). High resolution mass spectrometry (HRMS) data were recorded on Q Exactive HF (Q ExactiveTM HF/UtiMateTM 3000 RSLCnano) using electron spray ionization (ESI) in positive (or negative) mode. Melting points were determined with a Micromelting point apparatus. TLC plates were visualized by exposure to ultraviolet light.

Reagents and solvents were purchased as reagent grade and were used without further purification. All reactions were performed in standard glassware, heated at 70 °C for 3 h before used. The starting materials **1**^[1] were prepared according to literature methods. Flash column chromatography was performed over silica gel (200-300 m) using a mixture of ethyl acetate (EtOAc) and petroleum ether (PE).

II. Experimental Procedures and Spectroscopic Data

1. Table S1. Optimization study for the formation of 2a^a



Entry	Ar1	Oxidant	Solvent	Acid	Yield (%) ^b
1	I1	<i>m</i> CPBA	DCM	S1	35
2	I1	<i>m</i> CPBA	EA	S1	trace
3	I1	<i>m</i> CPBA	MeCN	S1	0
4	I1	<i>m</i> CPBA	HFIP	S1	23
5	I1	<i>m</i> CPBA	toluene	S1	0
6	I1	<i>m</i> CPBA	DCE	S1	43
7	I1	<i>m</i> CPBA	THF	S1	0
8	I1	<i>m</i> CPBA	TFE	S1	37
9	I1	<i>m</i> CPBA	DMF	S1	0
10	I1	<i>m</i> CPBA	MeOH	S1	0
11	I2	<i>m</i> CPBA	DCE	S1	65
12	I3	<i>m</i> CPBA	DCE	S1	43
13	I4	<i>m</i> CPBA	DCE	S1	70
14	I5	<i>m</i> CPBA	DCE	S1	trace
15	I6	<i>m</i> CPBA	DCE	S1	trace
16	I7	<i>m</i> CPBA	DCE	S1	59
17	I8	<i>m</i> CPBA	DCE	S1	62
18	I4	selectfluor	DCE	S1	trace

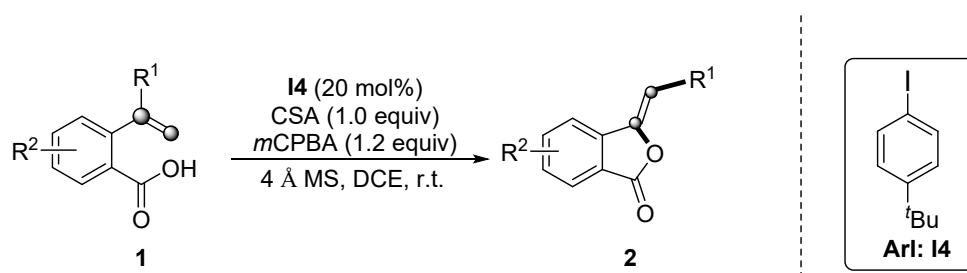
19	I4	H ₂ O ₂	MeCN	S1	NR
20	I4	TBHP	MeCN	S1	NR
21 ^c	I4	<i>m</i> CPBA	DCE	S1	77
22^c	I4	<i>m</i>CPBA	DCE	S2	82
23 ^c	I4	<i>m</i> CPBA	DCE	S3	72
24 ^c	I4	<i>m</i> CPBA	DCE	S4	80

^a Unless otherwise indicated, the reaction of **1a** (0.5 mmol) was carried out at rt for 2-12 h in solvent (5.0 mL) in the presence of ArI (20 mol%), *m*CPBA (1.2 equiv), and acid (1.0 equiv). ^b Isolated yield. NR = no reaction. ^c 4 Å molecular sieves (0.1 g) were added.

The initial investigation was carried out using iodobenzene (20 mol%) as catalyst and *meta*-chloroperoxybenzoic acid (*m*CPBA) as the oxidant in the presence of *para*-toluenesulfonic acid (*p*-TsOH) in DCM at room temperature. To our delight, the transformation indeed took place, and the desired product **2a** was obtained in 35% yield (Table S1, entry 1). With the acceptable result in hand, we came to further optimize the reaction conditions. First, a detailed screening of solvent was carried out, the result indicated dichloroethane (DCE) was the most appropriate solvent for this transformation and gave the product **2a** in moderate yield (Table S1, entries 6). Second, a series of aryl iodide catalysts were screened (Table S1, entries 11-17). It was found when 4-iodotoluene **I2** was applied, the yield of product **2a** could be raised to 65% (Table S1, entry 11). This result might indicate that the electron-donating group on the benzene ring was beneficial for this transformation. Gratifyingly, the presence of an electron-donating group with large steric hindrance (**I4**) further afforded the desired product with a satisfying 70% yield (Table S1, entry 13). On the contrary, when the aryl iodine with an electron-withdrawing substituting groups (-NO₂, -CO₂Me) were applied, the transformation was greatly inhibited. Next, the other oxidants including selectfluor, H₂O₂ and TBHP were investigated. Regrettably, none of them was capable of promoting the transformation (Table S1, entries 18-20). Whereas better yields were afforded with the addition of 4 Å molecular sieves (Table S1, entry 21), which prevented water

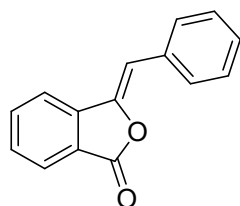
as the nucleophile and promoted yield to 77%. Furthermore, the sulfonic acid with different substituents and stereoscopic structures were tested (Table S1, entries 22-24). Notably, the optimum yield was obtained when (\pm)-10-camphorsulfonic acid (CSA) was applied to this reaction (Table S1, entry 22). Based on the above results, the optimal conditions for this transformation were finalized to be **I4** (20 mol%), *m*CPBA (1.2 equiv), CSA (1.0 equiv) and 4 Å molecular sieves in DCE at room temperature.

2. Typical Synthetic Procedure A and Spectroscopic Data of 2a-u



To a reaction flask filled with arylidene **I4** (20 mol %) in DCE (5.0 mL) was added *m*CPBA (1.2 equiv, 0.6 mmol), (\pm)-10-camphorsulfonic acid (1.0 equiv, 0.5 mmol) and 4 Å molecular sieves (0.1 g). The mixture was stirred at rt for 5 min and reactant **1** (0.5 mmol) was added. The resulting mixture was kept stirring until TLC indicated the total consumption of substrate **1**. Then the reaction mixture was quenched with aqueous saturated NaHCO₃ (5 mL), and extracted with dichloromethane (10 mL x 3). The combined organic layers were dried over anhydrous Na₂SO₄ and the solvent was removed *in vacuo*. The residue was purified by flash column chromatography on silica gel to afford target product **2**.

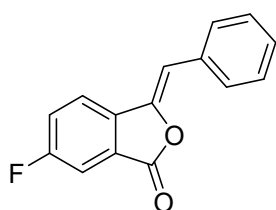
(*Z*)-3-Benzylideneisobenzofuran-1(3*H*)-one (**2a**)



According to the procedure A, **2a** was purified by silica gel chromatography (PE/EtOAc

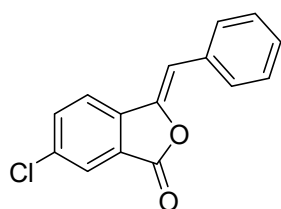
= 20/1). A white solid (91.1 mg, yield: 82%). mp: 82-84 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.94 (dd, *J* = 7.7, 0.9 Hz, 1H), 7.88 – 7.83 (m, 2H), 7.78 (dd, *J* = 7.9, 0.9 Hz, 1H), 7.75 – 7.70 (m, 1H), 7.57 – 7.52 (m, 1H), 7.45 – 7.39 (m, 2H), 7.35 – 7.29 (m, 1H), 6.43 (s, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 167.1, 144.6, 140.6, 134.5, 133.1, 130.1, 129.8, 128.8, 128.4, 125.6, 123.4, 119.8, 107.1. HRMS (ESI) calcd for C₁₅H₁₀NaO₂⁺ [*M* + Na⁺] 245.0573, found 245.0571.

(*Z*)-3-Benzylidene-6-fluoroisobenzofuran-1(3*H*)-one (2b)



According to the procedure A, **2b** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (91.3 mg, yield: 76%). mp: 154-156 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.83 (d, *J* = 7.3 Hz, 2H), 7.76 (dd, *J* = 8.5, 4.2 Hz, 1H), 7.58 (dd, *J* = 7.1, 2.3 Hz, 1H), 7.46 (dd, *J* = 8.6, 2.4 Hz, 1H), 7.44 – 7.39 (m, 2H), 7.34 (d, *J* = 7.4 Hz, 1H), 6.38 (s, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 165.9, (d, ⁴*J*_{C-F} = 4.1 Hz), 163.4, (d, ¹*J*_{C-F} = 253.1 Hz), 143.8, 136.6, (d, ⁴*J*_{C-F} = 2.3 Hz), 132.9, 130.1, 128.8, 128.6, 125.3, (d, ³*J*_{C-F} = 9.4 Hz), 122.9, (d, ²*J*_{C-F} = 24.9 Hz), 121.8, (d, ³*J*_{C-F} = 8.9 Hz), 111.8, (d, ²*J*_{C-F} = 24.2 Hz), 107.2, (d, ⁵*J*_{C-F} = 2.3 Hz). HRMS (ESI) calcd for C₁₅H₉FNao₂⁺ [*M* + Na⁺] 263.0479, found 263.0476.

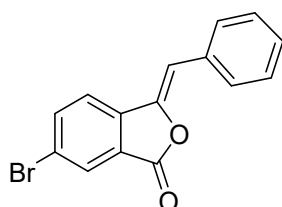
(*Z*)-3-Benzylidene-6-chloroisobenzofuran-1(3*H*)-one (2c)



According to the procedure A, **2c** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (92.4 mg, yield: 72%). mp: 163-165 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.91 (dd, *J* = 1.8, 0.8 Hz, 1H), 7.86 – 7.82 (m, 2H), 7.74 – 7.67 (m, 2H), 7.42

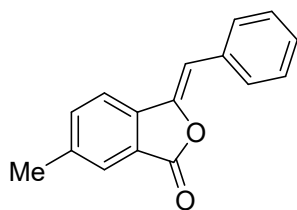
(td, $J = 6.8, 1.5$ Hz, 2H), 7.36 – 7.31 (m, 1H), 6.42 (s, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 165.6, 143.8, 138.8, 136.0, 134.9, 132.8, 130.2, 128.9, 128.8, 125.5, 124.9, 121.1, 107.9. HRMS (ESI) calcd for $\text{C}_{15}\text{H}_9^{35}\text{ClNaO}_2^+$ [$\text{M} + \text{Na}^+$] 279.0183, found 279.0185.

(Z)-3-Benzylidene-6-bromoisobenzofuran-1(3H)-one (2d)



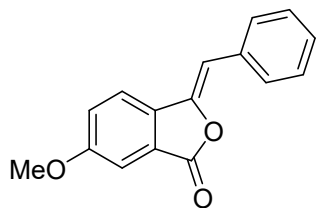
According to the procedure A, **2d** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (119.0 mg, yield: 79%). mp: 182-184 °C. ^1H NMR (400 MHz, CDCl_3) δ 8.06 (s, 1H), 7.83 (d, $J = 7.9$ Hz, 3H), 7.64 (d, $J = 8.3$ Hz, 1H), 7.42 (t, $J = 7.5$ Hz, 3H), 7.33 (t, $J = 7.4$ Hz, 1H), 6.42 (s, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 165.5, 143.8, 139.2, 137.6, 132.8, 130.2, 128.9, 128.8, 128.5, 125.1, 123.7, 121.2, 108.0. HRMS (ESI) calcd for $\text{C}_{15}\text{H}_9^{79}\text{BrNaO}_2^+$ [$\text{M} + \text{Na}^+$] 322.9678, found 322.9675.

(Z)-3-Benzylidene-6-methylisobenzofuran-1(3H)-one (2e)



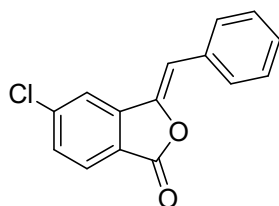
According to the procedure A, **2e** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (94.5 mg, yield: 80%). mp: 131-133 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.82 (d, $J = 7.6$ Hz, 2H), 7.68 (s, 1H), 7.62 (d, $J = 8.0$ Hz, 1H), 7.50 (d, $J = 7.9$ Hz, 1H), 7.39 (t, $J = 7.6$ Hz, 2H), 7.30 (t, $J = 7.3$ Hz, 1H), 6.32 (s, 1H), 2.47 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 167.2, 144.7, 140.5, 138.2, 135.8, 133.3, 130.0, 128.7, 128.2, 125.4, 123.6, 119.6, 106.2, 21.6. HRMS (ESI) calcd for $\text{C}_{16}\text{H}_{12}\text{NaO}_2^+$ [$\text{M} + \text{Na}^+$] 259.0730, found 259.0732.

(Z)-3-Benzylidene-6-methoxyisobenzofuran-1(3H)-one (2f)



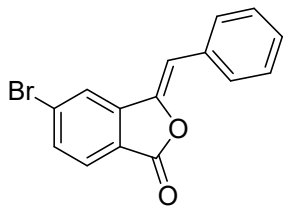
According to the procedure A, **2f** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (68.1 mg, yield: 54%). mp: 102-104 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.82 (d, *J* = 7.1 Hz, 2H), 7.66 (d, *J* = 8.6 Hz, 1H), 7.40 (t, *J* = 7.7 Hz, 2H), 7.34 (d, *J* = 2.4 Hz, 1H), 7.32 – 7.27 (m, 2H), 6.29 (s, 1H), 3.90 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 167.1, 161.4, 144.6, 133.6, 133.4, 129.9, 128.7, 128.1, 125.0, 124.0, 121.1, 106.8, 105.6, 55.9. HRMS (ESI) calcd for C₁₆H₁₂NaO₃⁺ [M + Na⁺] 275.0679, found 275.0676.

(Z)-3-Benzylidene-5-chloroisobenzofuran-1(3H)-one (2g)



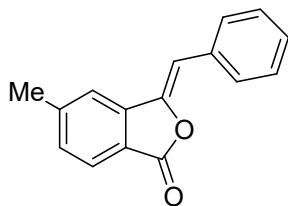
According to the procedure A, **2g** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (101.4 mg, yield: 79%). mp: 184-186 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.89 – 7.85 (m, 2H), 7.84 (s, 1H), 7.77 (d, *J* = 1.5 Hz, 1H), 7.51 (dd, *J* = 8.19, 1.7 Hz, 1H), 7.43 (t, *J* = 7.5 Hz, 2H), 7.37 – 7.32 (m, 1H), 6.43 (s, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 166.0, 143.3, 142.1, 141.4, 132.7, 130.4, 130.3, 128.9, 126.8, 121.8, 120.0, 108.3. HRMS (ESI) calcd for C₁₅H₉³⁵ClNaO₂⁺ [M + Na⁺] 279.0183, found 279.0181.

(Z)-3-Benzylidene-5-bromoisobenzofuran-1(3H)-one (2h)



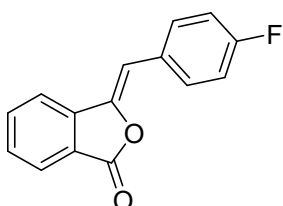
According to the procedure A, **2h** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (125.0 mg, yield: 83%). mp: 194-197 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.95 (d, *J* = 1.3 Hz, 1H), 7.86 – 7.79 (m, 3H), 7.67 (dd, *J* = 8.2, 1.5 Hz, 1H), 7.46 – 7.40 (m, 2H), 7.35 (t, *J* = 7.4 Hz, 1H), 6.43 (s, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 166.1, 143.2, 142.2, 133.2, 132.7, 130.3, 129.8, 128.9, 126.8, 123.1, 122.1, 108.4. HRMS (ESI) calcd for C₁₅H₉⁷⁹BrNaO₂⁺ [M + Na⁺] 322.9678, found 322.9677.

(Z)-3-Benzylidene-5-methylisobenzofuran-1(3H)-one (2i)



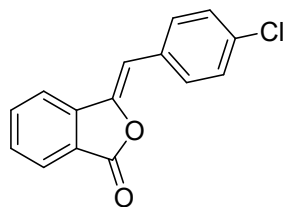
According to the procedure A, **2i** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (100.4 mg, yield: 85%). mp: 107-110 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.86 – 7.79 (m, 3H), 7.56 (s, 1H), 7.41 (t, *J* = 7.6 Hz, 2H), 7.35 (d, *J* = 7.9 Hz, 1H), 7.31 (tt, *J* = 6.6, 1.3 Hz, 1H), 6.38 (s, 1H), 2.53 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 167.1, 145.8, 144.7, 141.1, 133.2, 131.2, 130.1, 128.8, 128.3, 125.3, 121.0, 120.0, 106.6, 22.3. HRMS (ESI) calcd for C₁₆H₁₂NaO₂⁺ [M + Na⁺] 259.0730, found 259.0732.

(Z)-3-(4-Fluorobenzylidene)isobenzofuran-1(3H)-one (2j)



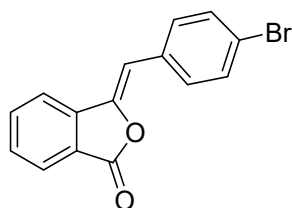
According to the procedure A, **2j** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (100.9 mg, yield: 84%). mp: 114-117 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.93 (d, *J* = 7.7 Hz, 1H), 7.83 (dd, *J* = 8.68, 5.5 Hz, 2H), 7.74 (q, *J* = 7.5 Hz, 2H), 7.58 – 7.52 (m, 1H), 7.09 (t, *J* = 8.7 Hz, 2H), 6.38 (s, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 167.0, 162.5, (d, ¹*J*_{C-F} = 250.9 Hz), 144.2, (d, ⁵*J*_{C-F} = 2.7 Hz) 140.5, 134.6, 131.9, (d, ³*J*_{C-F} = 8.2 Hz), 129.8, 129.3, (d, ⁴*J*_{C-F} = 3.5 Hz), 125.6, 123.3, 119.8, 115.9, (d, ²*J*_{C-F} = 21.8 Hz), 105.9. HRMS (ESI) calcd for C₁₅H₉FN₂O₂⁺ [M + Na⁺] 263.0479, found 263.0480.

(Z)-3-(4-Chlorobenzylidene)isobenzofuran-1(3H)-one (2k)



According to the procedure A, **2k** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (104.0 mg, yield: 81%). mp: 148-150 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.95 (d, *J* = 7.7 Hz, 1H), 7.80 (s, 1H), 7.77 (d, *J* = 5.0 Hz, 2H), 7.76 – 7.71 (m, 1H), 7.57 (ddd, *J* = 8.0, 6.8, 1.4 Hz, 1H), 7.38 (d, *J* = 8.6 Hz, 2H), 6.38 (s, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 166.8, 144.9, 140.4, 134.6, 134.2, 131.6, 131.3, 130.0, 129.0, 125.7, 123.4, 119.9, 105.7. HRMS (ESI) calcd for C₁₅H₉³⁵ClNaO₂⁺ [M + Na⁺] 279.0183, found 279.0184.

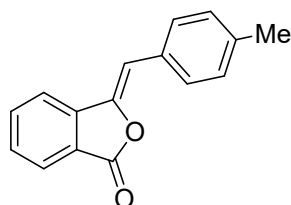
(Z)-3-(4-Bromobenzylidene)isobenzofuran-1(3H)-one (2l)



According to the procedure A, **2l** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (128.0 mg, yield: 85%). mp: 173-176 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.95 (d, *J* = 7.7 Hz, 1H), 7.79 – 7.69 (m, 4H), 7.60 – 7.51 (m, 3H), 6.36 (s, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 166.8, 145.1, 140.4, 134.6, 132.0, 132.0, 131.5,

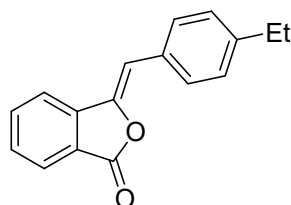
130.1, 125.7, 123.5, 122.6, 119.9, 105.8. HRMS (ESI) calcd for $C_{15}H_9^{79}BrNaO_2^+$ [$M + Na^+$] 322.9678, found 322.9675.

(Z)-3-(4-Methylbenzylidene)isobenzofuran-1(3H)-one (2m)



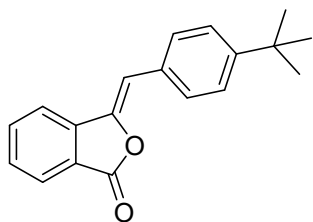
According to the procedure A, **2m** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (105.1 mg, yield: 89%). mp: 141-144 °C. 1H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, $J = 7.7$ Hz, 1H), 7.78 – 7.74 (m, 3H), 7.72 (t, $J = 7.5$ Hz, 1H), 7.56 – 7.51 (m, 1H), 7.23 (d, $J = 7.9$ Hz, 2H), 6.41 (s, 1H), 2.39 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.2, 144.0, 140.7, 138.7, 134.4, 130.3, 130.1, 129.6, 125.6, 123.3, 119.7, 107.2, 21.5. HRMS (ESI) calcd for $C_{16}H_{12}NaO_2^+$ [$M + Na^+$] 259.0730, found 259.0728.

(Z)-3-(4-Ethylbenzylidene)isobenzofuran-1(3H)-one (2n)



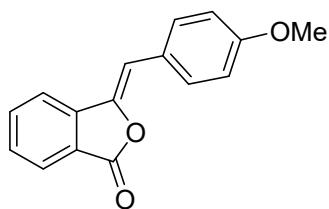
According to the procedure A, **2n** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (107.6 mg, yield: 86%). mp: 108-110°C. 1H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, $J = 7.7$ Hz, 1H), 7.80 – 7.75 (m, 3H), 7.74 – 7.69 (m, 1H), 7.56 – 7.51 (m, 1H), 7.25 (d, $J = 8.0$ Hz, 2H), 6.42 (s, 1H), 2.68 (q, $J = 7.6$ Hz, 2H), 1.26 (t, $J = 7.6$ Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.2, 145.0, 144.0, 140.7, 134.4, 130.5, 130.2, 129.5, 128.4, 125.6, 123.4, 119.7, 107.2, 28.8, 15.4. HRMS (ESI) calcd for $C_{17}H_{14}NaO_2^+$ [$M + Na^+$] 273.0886, found 273.0884.

(Z)-3-(4-(Tert-butyl)benzylidene)isobenzofuran-1(3H)-one (2o)



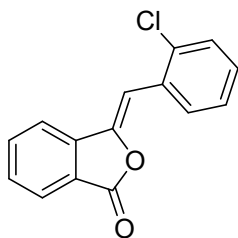
According to the procedure A, **2o** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (110.0 mg, yield: 79%). mp: 104-106°C. ¹H NMR (400 MHz, CDCl₃) δ 7.93 (d, *J* = 7.4 Hz, 1H), 7.82 – 7.74 (m, 3H), 7.71 (t, *J* = 7.4 Hz, 1H), 7.53 (t, *J* = 7.3 Hz, 1H), 7.44 (d, *J* = 8.4 Hz, 2H), 6.42 (s, 1H), 1.35 (s, 9H). ¹³C NMR (101 MHz, CDCl₃) δ 167.2, 151.8, 144.1, 140.7, 134.4, 130.3, 130.0, 129.6, 125.8, 125.5, 123.3, 119.7, 107.1, 34.8, 31.2. HRMS (ESI) calcd for C₁₉H₁₈NaO₂⁺ [M + Na⁺] 301.1199, found 301.1196.

(Z)-3-(4-Methoxybenzylidene)isobenzofuran-1(3H)-one (2p)



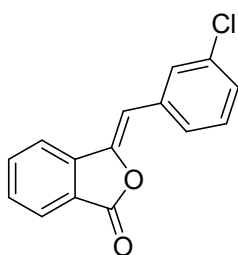
According to the procedure A, **2p** was purified by silica gel chromatography (PE/EtOAc = 20/1). A yellow solid (97.1 mg, yield: 77%). mp: 114-116°C. ¹H NMR (400 MHz, CDCl₃) δ 7.92 (d, *J* = 5.7 Hz, 1H), 7.80 (dd, *J* = 8.6, 4.3 Hz, 2H), 7.71 (q, *J* = 7.0 Hz, 2H), 7.54 – 7.47 (m, 1H), 6.93 (dd, *J* = 8.7, 3.9 Hz, 2H), 6.37 (s, 1H), 3.85 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 167.3, 159.8, 143.1, 140.8, 134.4, 131.7, 129.3, 125.9, 125.5, 123.1, 119.5, 114.3, 107.0, 55.3. HRMS (ESI) calcd for C₁₆H₁₂NaO₃⁺ [M + Na⁺] 275.0679, found 275.0677.

(Z)-3-(2-Chlorobenzylidene)isobenzofuran-1(3H)-one (2q)



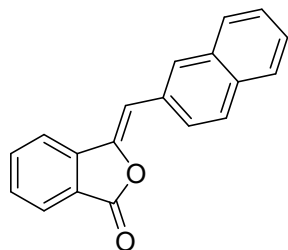
According to the procedure A, **2q** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (100.1 mg, yield: 78%). 5 mmol scale (921.6 mg, yield: 72%). mp: 161-163°C. ¹H NMR (400 MHz, CDCl₃) δ 8.32 (dd, *J* = 8.0, 1.7 Hz, 1H), 7.96 (d, *J* = 7.7 Hz, 1H), 7.87 (d, *J* = 7.9 Hz, 1H), 7.79 – 7.73 (m, 1H), 7.62 – 7.56 (m, 1H), 7.43 (dd, *J* = 8.0, 1.3 Hz, 1H), 7.35 (td, *J* = 7.50, 1.1 Hz, 1H), 7.28 – 7.21 (m, 1H), 6.91 (s, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 166.8, 145.8, 140.5, 134.7, 133.8, 131.8, 131.0, 130.2, 129.6, 129.3, 127.2, 125.6, 123.5, 120.3, 102.3. HRMS (ESI) calcd for C₁₅H₉³⁵ClNaO₂⁺ [M + Na⁺] 279.0183, found 279.0184.

(Z)-3-(3-Chlorobenzylidene)isobenzofuran-1(3H)-one (2r)



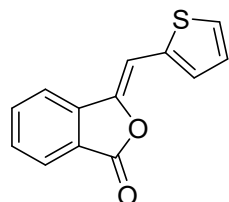
According to the procedure A, **2r** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (104.0 mg, yield: 81%). mp: 152-154°C. ¹H NMR (400 MHz, CDCl₃) δ 7.94 (d, *J* = 7.7 Hz, 1H), 7.82 (s, 1H), 7.75 (q, *J* = 10.8, 9.4 Hz, 3H), 7.57 (t, *J* = 7.1 Hz, 1H), 7.33 (t, *J* = 7.8 Hz, 1H), 7.28 (d, *J* = 7.2 Hz, 1H), 6.34 (s, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 166.7, 145.5, 140.3, 134.8, 134.7, 134.7, 130.2, 130.0, 129.7, 128.3, 128.1, 125.7, 123.5, 120.0, 105.5. HRMS (ESI) calcd for C₁₅H₉³⁵ClNaO₂⁺ [M + Na⁺] 279.0183, found 279.0182.

(Z)-3-(Naphthalen-2-ylmethylene)isobenzofuran-1(3H)-one (2s)



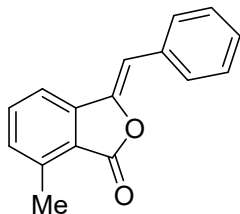
According to the procedure A, **2s** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (114.4 mg, yield: 84%). mp: 170-172°C. ¹H NMR (400 MHz, CDCl₃) δ 8.24 (s, 1H), 8.04 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.95 (d, *J* = 7.7 Hz, 1H), 7.91 – 7.87 (m, 1H), 7.85 (d, *J* = 8.8 Hz, 1H), 7.84 – 7.77 (m, 2H), 7.73 (t, *J* = 7.4 Hz, 1H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.52 – 7.47 (m, 2H), 6.57 (s, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 167.1, 144.8, 140.6, 134.5, 133.5, 133.0, 130.7, 129.9, 129.8, 128.5, 128.4, 127.7, 127.3, 126.7, 126.4, 125.6, 123.4, 119.8, 107.3. HRMS (ESI) calcd for C₁₉H₁₂NaO₂⁺ [M + Na⁺] 295.0730, found 295.0731.

(Z)-3-(Thiophen-2-ylmethylene)isobenzofuran-1(3H)-one (2t)



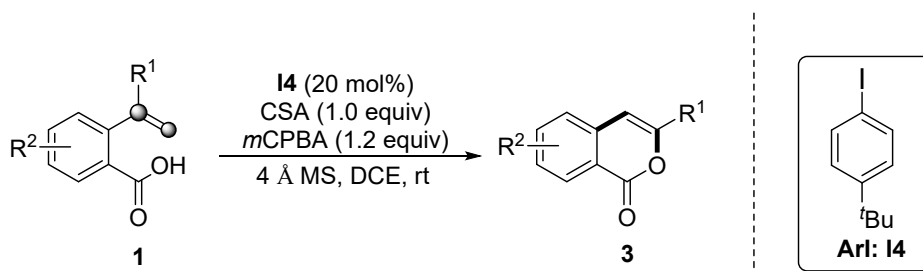
According to the procedure A, **2t** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (93.6 mg, yield: 82%). mp: 112-114°C. ¹H NMR (400 MHz, CDCl₃) δ 7.93 (dt, *J* = 7.71, 0.9 Hz, 1H), 7.74 – 7.71 (m, 1H), 7.71 – 7.68 (m, 1H), 7.53 (ddd, *J* = 8.0, 5.41, 2.7 Hz, 1H), 7.45 (d, *J* = 5.1 Hz, 1H), 7.40 (d, *J* = 3.6 Hz, 1H), 6.70 (s, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 166.5, 142.9, 139.8, 136.2, 134.5, 129.6, 129.6, 128.9, 127.5, 125.8, 123.8, 119.6, 101.1. HRMS (ESI) calcd for C₁₃H₈NaO₂⁺ [M + Na⁺] 251.0137, found 251.0135.

(Z)-3-benzylidene-7-methylisobenzofuran-1(3H)-one (2u)



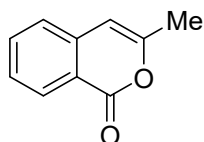
According to the procedure A, **2u** was purified by silica gel chromatography (petroleum ester/EtOAc = 20/1). A white solid (92.1 mg, yield: 78%). mp: 149-151 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.83 (d, *J* = 8.3 Hz, 2H), 7.54 (d, *J* = 4.3 Hz, 2H), 7.40 (t, *J* = 7.6 Hz, 2H), 7.33 – 7.28 (m, 1H), 7.26 (d, *J* = 3.8 Hz, 1H), 6.35 (s, 1H), 2.69 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 167.2, 144.5, 141.0, 139.6, 134.2, 133.3, 131.3, 130.0, 128.7, 128.2, 121.1, 117.2, 106.3, 17.5. HRMS (ESI) calcd for C₁₆H₁₂NaO₂⁺ [M + Na⁺] 259.0730, found 259.0732.

3. Typical Synthetic Procedure B and Spectroscopic Data of 3a-l



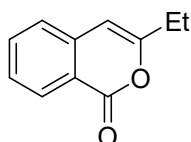
To a reaction flask filled with aryl iodide **I4** (20 mol %) in DCE (5.0 ml) was added *m*CPBA (1.2 equiv, 0.6 mmol), (±)-10-camphorsulfonic acid (1.0 equiv, 0.5 mmol) and 4 Å molecular sieves (0.1 g). The mixture was stirred at rt for 5 min and then reactant **1** (0.5 mmol) was added. The resulting mixture was kept stirring until TLC indicated the total consumption of substrate **1**. Then the reaction mixture was quenched with aqueous saturated NaHCO₃ (5 mL), and extracted with dichloromethane (10 mL x 3). The combined organic layers were dried over anhydrous Na₂SO₄ and the solvent was removed *in vacuo*. The residue was purified by flash column chromatography on silica gel to afford target product **3**.

3-Methyl-1*H*-isochromen-1-one (3a)



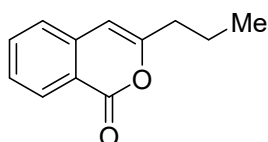
According to the procedure B, **3a** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (69.7 mg, yield: 87%). mp: 69-72°C. ¹H NMR (400 MHz, CDCl₃) δ 8.25 (d, *J* = 8.1 Hz, 1H), 7.67 (td, *J* = 7.6, 1.4 Hz, 1H), 7.47 – 7.42 (m, 1H), 7.34 (d, *J* = 7.9 Hz, 1H), 6.26 (s, 1H), 2.28 (d, *J* = 1.0 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 163.0, 154.6, 137.7, 134.7, 129.5, 127.6, 124.9, 119.9, 103.5, 19.7. HRMS (ESI) calcd for C₁₀H₈NaO₂⁺ [M + Na⁺] 183.0417, found 183.0416.

3-Ethyl-1H-isochromen-1-one (3b)



According to the procedure B, **3b** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (76.6 mg, yield: 88%). mp: 68-70°C. ¹H NMR (400 MHz, CDCl₃) δ 8.26 – 8.20 (m, 1H), 7.65 (td, *J* = 7.7, 1.4 Hz, 1H), 7.46 – 7.40 (m, 1H), 7.34 (d, *J* = 7.9 Hz, 1H), 6.23 (s, 1H), 2.55 (q, *J* = 7.5 Hz, 2H), 1.26 (t, *J* = 7.5 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 163.0, 159.5, 137.6, 134.7, 129.5, 127.5, 125.1, 120.1, 102.0, 26.7, 11.2. HRMS (ESI) calcd for C₁₁H₁₀NaO₂⁺ [M + Na⁺] 197.0573, found 197.0571.

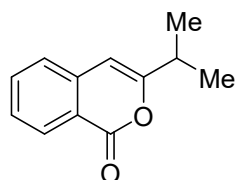
3-Propyl-1H-isochromen-1-one (3c)



According to the procedure B, **3c** was purified by silica gel chromatography (PE/EtOAc = 20/1). Colorless liquid (66.8 mg, yield: 71%). ¹H NMR (400 MHz, CDCl₃) δ 8.22 (d, *J* = 8.0 Hz, 1H), 7.64 (td, *J* = 7.8, 1.3 Hz, 1H), 7.44 – 7.39 (m, 1H), 7.33 (d, *J* = 7.9 Hz, 1H), 6.23 (s, 1H), 2.48 (t, *J* = 7.5 Hz, 2H), 1.77 – 1.66 (m, 2H), 0.97 (t, *J* = 7.4 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 163.1, 158.0, 137.6, 134.7,

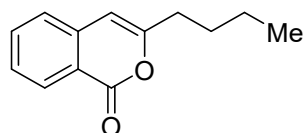
129.4, 127.5, 125.0, 120.1, 103.0, 35.4, 20.2, 13.5. HRMS (ESI) calcd for $C_{12}H_{12}NaO_2^+$ $[M + Na^+]$ 211.0730, found 211.0732.

3-Isopropyl-1*H*-isochromen-1-one (3d)



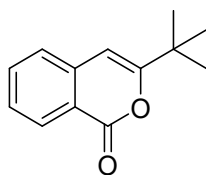
According to the procedure B, **3d** was purified by silica gel chromatography (PE/EtOAc = 20/1). Colorless liquid (81.9 mg, yield: 87%). 1H NMR (400 MHz, $CDCl_3$) δ 8.18 (d, $J = 8.0$ Hz, 1H), 7.64 – 7.58 (m, 1H), 7.38 (t, $J = 7.6$ Hz, 1H), 7.31 (d, $J = 7.9$ Hz, 1H), 6.20 (s, 1H), 2.72 (hept, $J = 6.6$ Hz, 1H), 1.24 (d, $J = 6.9$ Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.0, 162.9, 137.6, 134.7, 129.3, 127.5, 125.3, 120.2, 100.6, 32.3, 20.2. HRMS (ESI) calcd for $C_{12}H_{12}NaO_2^+$ $[M + Na^+]$ 211.0730, found 211.0729.

3-Butyl-1*H*-isochromen-1-one (3e)



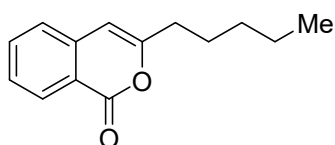
According to the procedure B, **3e** was purified by silica gel chromatography (PE/EtOAc = 20/1). Colorless liquid (91.0 mg, yield: 90%). 1H NMR (400 MHz, $CDCl_3$) δ 8.24 (d, $J = 8.0$ Hz, 1H), 7.66 (td, $J = 8.0, 1.3$ Hz, 1H), 7.47 – 7.41 (m, 1H), 7.34 (d, $J = 7.8$ Hz, 1H), 6.25 (s, 1H), 2.55 – 2.49 (m, 2H), 1.69 (p, $J = 7.5$ Hz, 2H), 1.40 (h, $J = 7.4$ Hz, 2H), 0.94 (t, $J = 7.4$ Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.1, 158.3, 137.6, 134.7, 129.5, 127.5, 125.0, 120.1, 102.9, 33.2, 29.0, 22.1, 13.8. HRMS (ESI) calcd for $C_{13}H_{14}NaO_2^+$ $[M + Na^+]$ 225.0886, found 225.0884.

3-(Tert-butyl)-1*H*-isochromen-1-one (3f)



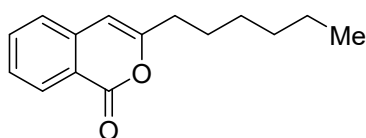
According to the procedure B, **3f** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (90.0 mg, yield: 89%). mp: 59-62°C. ¹H NMR (400 MHz, CDCl₃) δ 8.24 (d, *J* = 7.9 Hz, 1H), 7.70 – 7.62 (m, 1H), 7.43 (t, *J* = 7.3 Hz, 1H), 7.37 (d, *J* = 7.8 Hz, 1H), 6.30 (s, 1H), 1.32 (s, 9H). ¹³C NMR (101 MHz, CDCl₃) δ 165.1, 163.0, 137.7, 134.6, 129.4, 127.6, 125.5, 120.1, 99.7, 35.6, 28.0. HRMS (ESI) calcd for C₁₃H₁₄NaO₂⁺ [M + Na⁺] 225.0886, found 225.0885.

3-Pentyl-1H-isochromen-1-one (3g)



According to the procedure B, **3g** was purified by silica gel chromatography (PE/EtOAc = 20/1). Colorless liquid (96.2 mg, yield: 89%). ¹H NMR (400 MHz, CDCl₃) δ 8.24 (d, *J* = 8.5 Hz, 1H), 7.66 (td, *J* = 7.8, 1.3 Hz, 1H), 7.46 – 7.41 (m, 1H), 7.34 (d, *J* = 7.8 Hz, 1H), 6.24 (d, *J* = 0.9 Hz, 1H), 2.55 – 2.47 (m, 2H), 1.70 (p, *J* = 7.5 Hz, 2H), 1.35 (dq, *J* = 7.1, 3.5 Hz, 4H), 0.92 – 0.87 (m, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 163.1, 158.3, 137.6, 134.7, 129.5, 127.5, 125.0, 120.1, 102.9, 33.5, 31.2, 26.6, 22.4, 14.0. HRMS (ESI) calcd for C₁₄H₁₆NaO₂⁺ [M + Na⁺] 239.1043, found 239.1041.

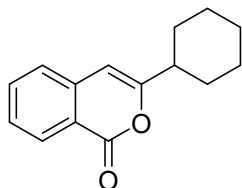
3-Hexyl-1H-isochromen-1-one (3h)



According to the procedure B, **3h** was purified by silica gel chromatography (PE/EtOAc = 20/1). Colorless liquid (99.0 mg, yield: 86%). ¹H NMR (400 MHz, CDCl₃) δ 8.22 (d, *J* = 7.9 Hz, 1H), 7.64 (td, *J* = 7.8, 1.2 Hz, 1H), 7.44 – 7.39 (m, 1H), 7.33 (d, *J* = 7.8 Hz, 1H), 6.23 (s, 1H), 2.49 (t, *J* = 7.6 Hz, 2H), 1.67 (p, *J* = 7.5 Hz, 2H),

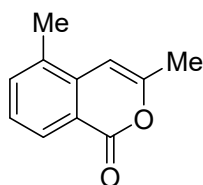
1.39 – 1.32 (m, 2H), 1.32 – 1.22 (m, 4H), 0.89 – 0.83 (m, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 163.1, 158.3, 137.6, 134.7, 129.4, 127.5, 125.0, 120.1, 102.9, 33.5, 31.5, 28.7, 26.9, 22.5, 14.1. HRMS (ESI) calcd for $\text{C}_{15}\text{H}_{18}\text{NaO}_2^+$ [$\text{M} + \text{Na}^+$] 253.1199, found 253.1196.

3-Cyclohexyl-1*H*-isochromen-1-one (3i)



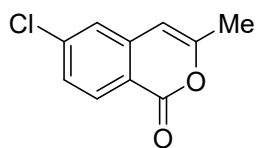
According to the procedure B, **3i** was purified by silica gel chromatography (PE/EtOAc = 20/1). Colorless liquid (97.0 mg, yield: 85%). ^1H NMR (400 MHz, CDCl_3) δ 8.25 (d, $J = 8.0$ Hz, 1H), 7.66 (td, $J = 7.7, 1.3$ Hz, 1H), 7.46 – 7.42 (m, 1H), 7.36 (d, $J = 7.9$ Hz, 1H), 6.23 (s, 1H), 2.49 – 2.40 (m, 1H), 2.04 (d, $J = 11.7$ Hz, 2H), 1.85 (dt, $J = 12.1, 2.8$ Hz, 2H), 1.78 – 1.70 (m, 1H), 1.46 (ddd, $J = 24.0, 12.1, 2.4$ Hz, 2H), 1.36 – 1.18 (m, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 163.2, 162.4, 137.8, 134.6, 129.5, 127.5, 125.2, 120.3, 100.9, 41.9, 30.6, 26.0, 25.9. HRMS (ESI) calcd for $\text{C}_{15}\text{H}_{16}\text{NaO}_2^+$ [$\text{M} + \text{Na}^+$] 251.1043, found 251.1044.

3,5-Dimethyl-1*H*-isochromen-1-one (3j)



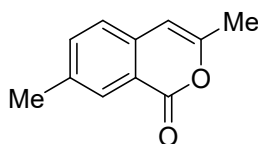
According to the procedure B, **3j** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (74.9 mg, yield: 86%). mp: 130-132°C. ^1H NMR (400 MHz, CDCl_3) δ 8.08 (d, $J = 7.9$ Hz, 1H), 7.48 (d, $J = 7.3$ Hz, 1H), 7.30 (t, $J = 7.7$ Hz, 1H), 6.34 (s, 1H), 2.42 (s, 3H), 2.28 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 163.3, 154.1, 136.3, 135.6, 132.5, 127.3, 127.0, 119.9, 100.4, 19.9, 18.7. HRMS (ESI) calcd for $\text{C}_{11}\text{H}_{10}\text{NaO}_2^+$ [$\text{M} + \text{Na}^+$] 197.0573, found 197.0575.

6-Chloro-3-methyl-1*H*-isochromen-1-one (3k)



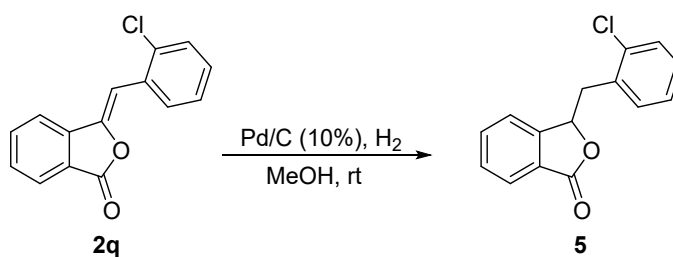
According to the procedure B, **3k** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (62.3 mg, yield: 64%). mp: 148-150°C. ¹H NMR (400 MHz, CDCl₃) δ 8.09 (d, *J* = 8.5 Hz, 1H), 7.32 (d, *J* = 8.4 Hz, 1H), 7.25 (s, 1H), 6.13 (s, 1H), 2.23 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 162.0, 156.1, 141.3, 138.9, 131.1, 128.0, 124.4, 118.1, 102.7, 19.7. HRMS (ESI) calcd for C₁₀H₇³⁵ClNaO₂⁺ [M + Na⁺] 217.0027, found 217.0026.

3,7-Dimethyl-1*H*-isochromen-1-one (3l)



According to the procedure B, **3l** was purified by silica gel chromatography (PE/EtOAc = 20/1). A white solid (67.1 mg, yield: 77%). mp: 119-121°C. ¹H NMR (400 MHz, CDCl₃) δ 8.00 (s, 1H), 7.47 – 7.42 (m, 1H), 7.20 (d, *J* = 8.0 Hz, 1H), 6.19 (s, 1H), 2.40 (s, 3H), 2.23 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 163.2, 153.6, 137.7, 136.0, 135.2, 129.1, 124.8, 119.7, 103.4, 21.3, 19.6. HRMS (ESI) calcd for C₁₁H₁₀NaO₂⁺ [M + Na⁺] 197.0573, found 197.0570.

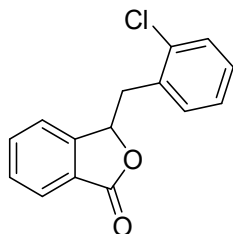
4. Synthetic Procedure^[2] and Spectroscopic Data of 3-(2-Chlorobenzyl)isobenzofuran-1(3*H*)-one (**5**)



A solution of (*Z*)-3-(2-Chlorobenzylidene)isobenzofuran-1(3*H*)-one **2q** (0.5mmol) in dry methanol (10 mL) was stirred with palladium/charcoal (10%, 20.0 mg) under a

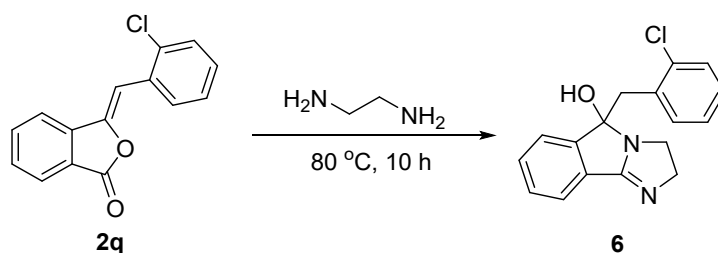
hydrogen atmosphere at rt for 16 h. The catalyst was removed by suction filtration and the solvent evaporated under reduced pressure. The mixture was purified by silica gel chromatography to obtain the hydrogenated product **5** as colorless liquid.

3-(2-Chlorobenzyl)isobenzofuran-1(3H)-one (**5**)



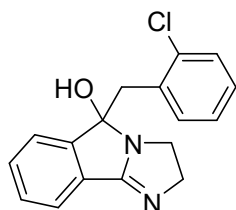
According to the procedure above, **5** was purified by silica gel chromatography (PE/EtOAc = 10/1). Colorless liquid (125.5 mg, yield: 97%). ¹H NMR (400 MHz, CDCl₃) δ 7.85 (d, *J* = 7.6 Hz, 1H), 7.65 – 7.59 (m, 1H), 7.50 (t, *J* = 7.5 Hz, 1H), 7.31 (dd, *J* = 6.6, 1.5 Hz, 1H), 7.28 (dd, *J* = 4.2, 2.0 Hz, 1H), 7.25 – 7.19 (m, 3H), 5.71 (t, *J* = 6.4 Hz, 1H), 3.29 (dd, *J* = 14.1, 6.7 Hz, 1H), 3.18 (dd, *J* = 14.1, 6.1 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 170.3, 149.1, 135.0, 133.8, 129.7, 129.2, 128.5, 127.2, 126.2, 125.7, 122.4, 81.2, 40.9. HRMS (ESI) calcd for C₁₅H₁₁³⁵ClNaO₂⁺ [*M* + Na⁺] 281.0340, found 281.0338.

5. Synthetic Procedure^[3] and Spectroscopic Data of 5-(2-Chlorobenzyl)-2,5-dihydro-3H-imidazo[2,1-*a*]isoindol-5-ol (**6**)



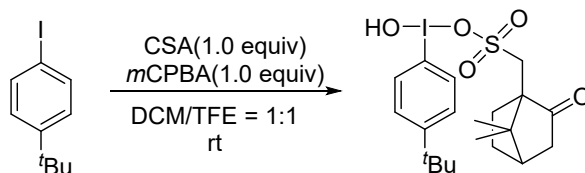
Compound **2q** (0.5 mmol) was dissolved into ethylenediamine (5 mL) and the mixture was heated at 80 °C with magnetic stirring for 10 h. After cooling to rt, the reaction mixture was extracted with EtOAc (10 mL x 3) and washed with water to pH = 7. The combined organic layers were dried over anhydrous Na₂SO₄ and the solvent was removed in vacuo. The residue was purified by recrystallization from EtOAc/petroleum ether to give the product **3a** as a white solid.

5-(2-Chlorobenzyl)-2,5-dihydro-3H-imidazo[2,1-a]isoindol-5-ol (6)



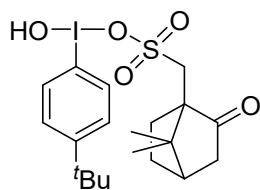
According to the procedure above, **6** was purified by crystallization. A white solid (143.4 mg, yield: 96%). mp: 209-212 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.62 (d, *J* = 6.39 Hz, 1H), 7.51 – 7.43 (m, 1H), 7.35 (dd, *J* = 21.89, 6.55 Hz, 2H), 7.22 (d, *J* = 6.90 Hz, 1H), 7.13 – 6.96 (m, 3H), 4.14 (d, *J* = 14.26 Hz, 1H), 3.61 (d, *J* = 13.95 Hz, 1H), 3.38 (d, *J* = 13.94 Hz, 1H), 3.19 (t, *J* = 12.10 Hz, 1H), 3.02 (d, *J* = 12.75 Hz, 1H), 2.90 (t, *J* = 11.42 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 167.5, 147.9, 134.9, 133.2, 131.9, 131.7, 130.6, 129.5, 128.9, 128.2, 126.3, 122.9, 122.6, 88.9, 41.8, 40.7, 39.2. HRMS (ESI) calcd for C₁₇H₁₅³⁵ClN₂NaO⁺ [*M* + Na⁺] 321.0675, found 321.0673.

5. Synthetic Procedure^[4] and Spectroscopic Data of modified Koser reagent HCIB



To a stirred solution of iodoarene (0.50 mmol) in dichloromethane/TFE (1:1 v/v, 5.0 mL) was added *m*CPBA (0.50 mmol), followed by (±)-10-camphorsulfonic acid (0.50 mmol). The resulting solution was stirred at rt for 30 min and concentrated under a stream of air, then diethyl ether (10.0 mL) was added to the remaining residue. The resulting precipitate was filtered off and dried *in vacuo* to give the corresponding Koser reagent as a white solid.

HCIB

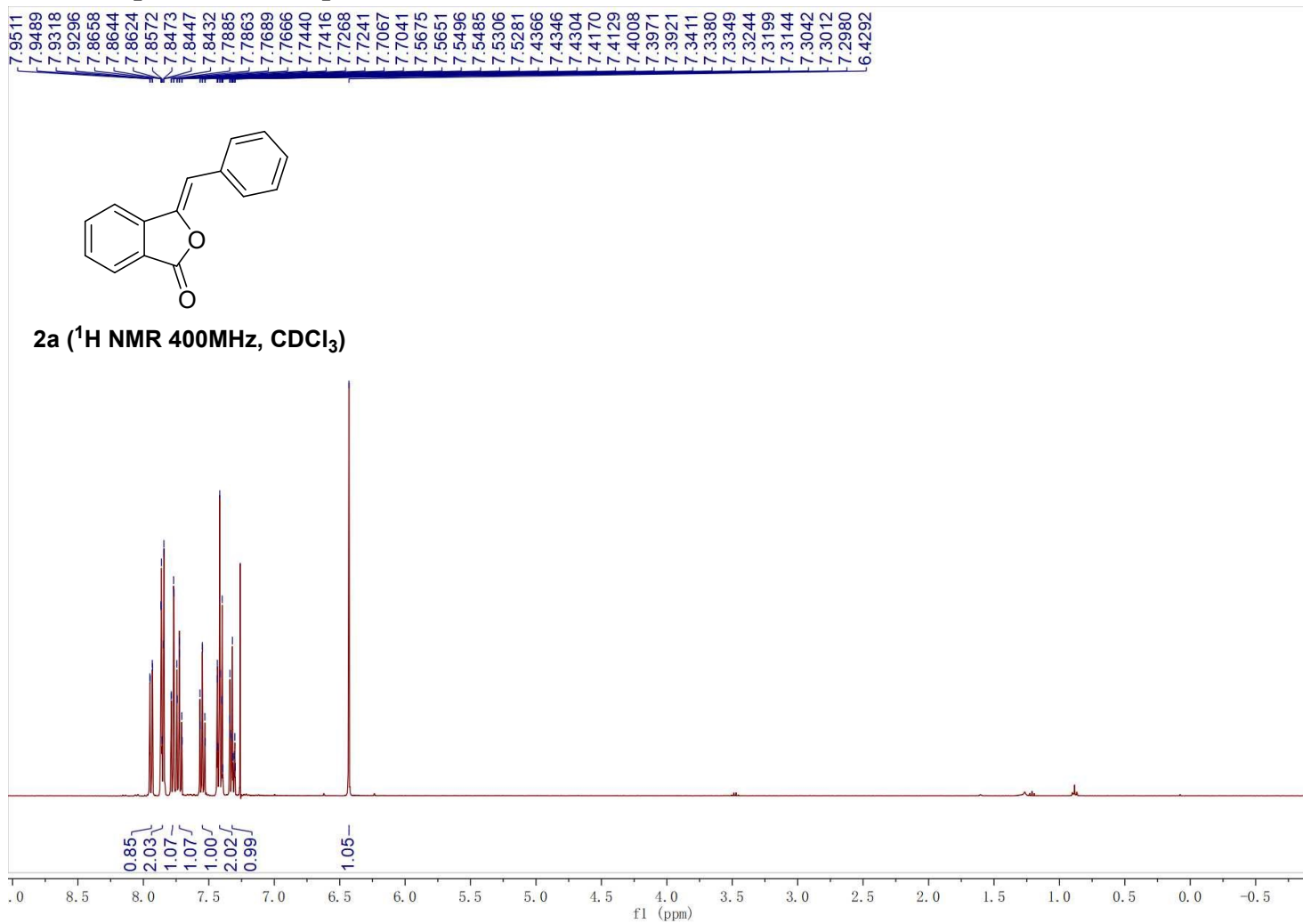


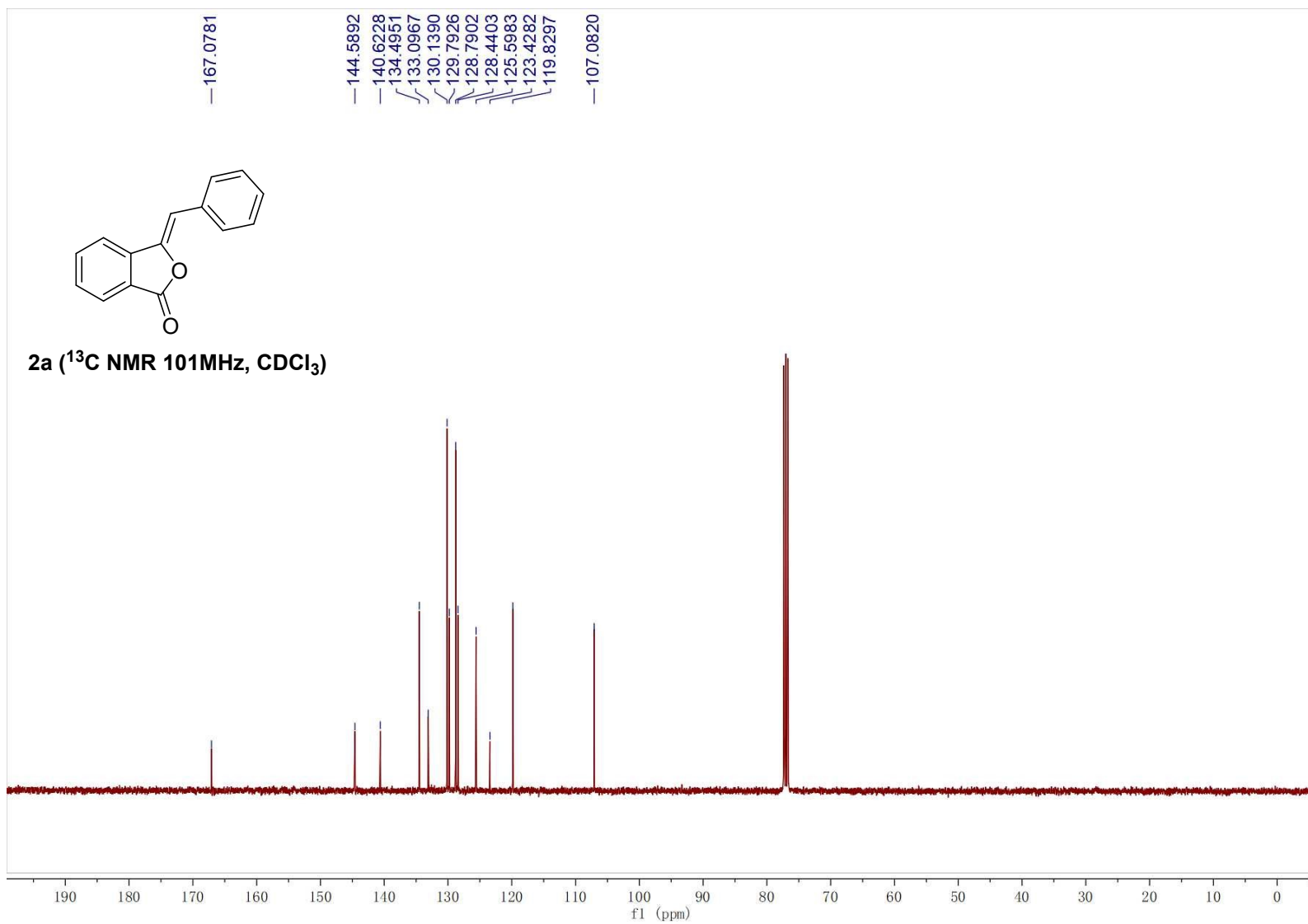
According to the procedure above, **HCIB** was purified by crystallization. A colorless solid (228.8 mg, yield: 90%). mp: 125-128 °C. ^1H NMR (400 MHz, CDCl_3) δ 8.08 (s, 2H), 7.54 – 7.44 (m, 2H), 3.27 (d, $J = 13.8$ Hz, 1H), 2.73 (d, $J = 14.1$ Hz, 1H), 2.31 (d, $J = 18.2$ Hz, 2H), 2.02 (s, 1H), 1.86 (d, $J = 18.4$ Hz, 2H), 1.54 (s, 1H), 1.32 (s, 10H), 0.96 (s, 3H), 0.76 (s, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 216.4, 156.4, 134.3, 128.5, 58.3, 48.0, 47.9, 42.8, 42.6, 35.3, 31.1, 26.9, 24.6, 19.8, 19.8. HRMS (ESI) calcd for $\text{C}_{20}\text{H}_{29}\text{INaO}_5\text{S}^+$ [$\text{M} + \text{Na}^+$] 531.0673, found 531.0671.

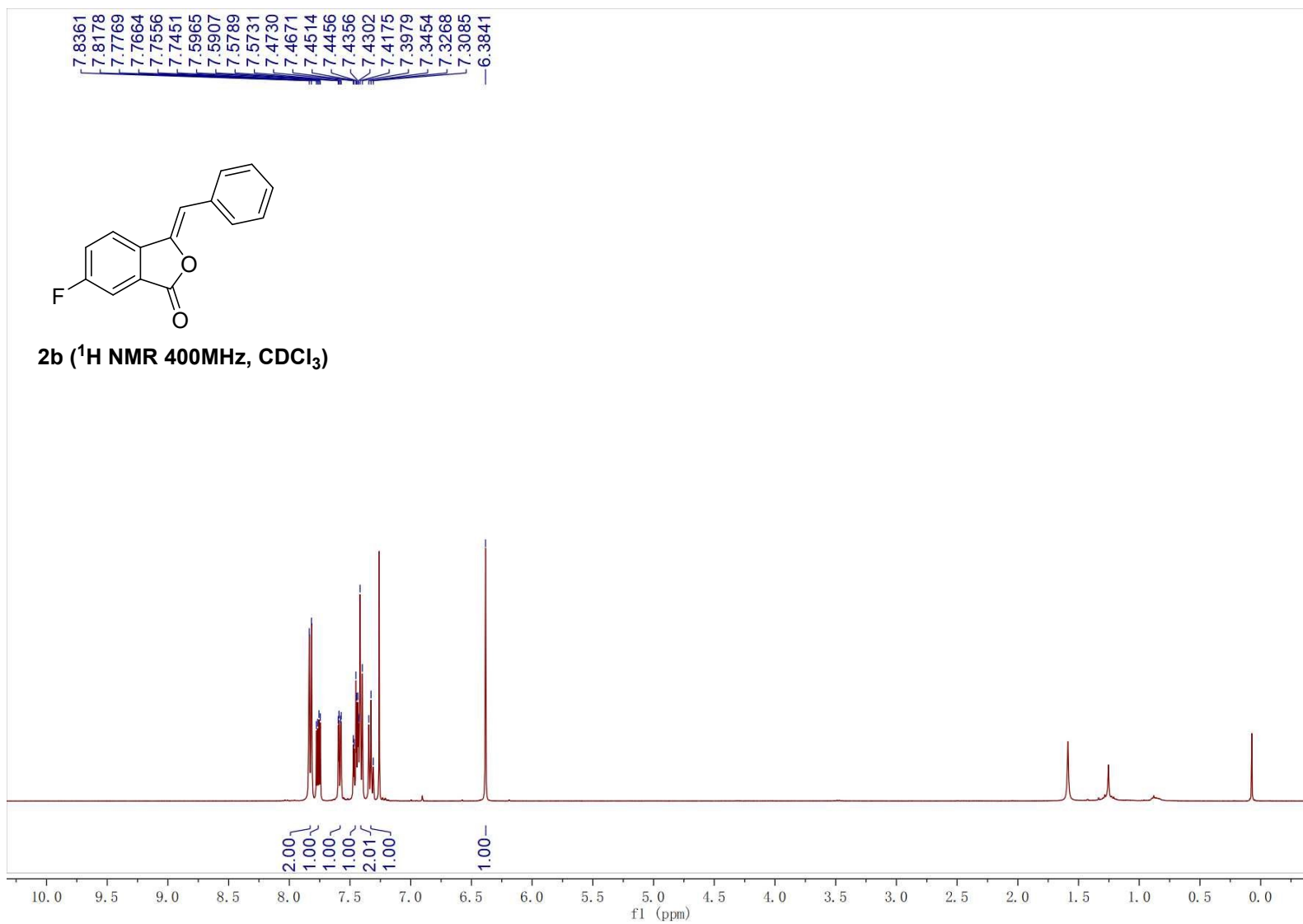
III. References:

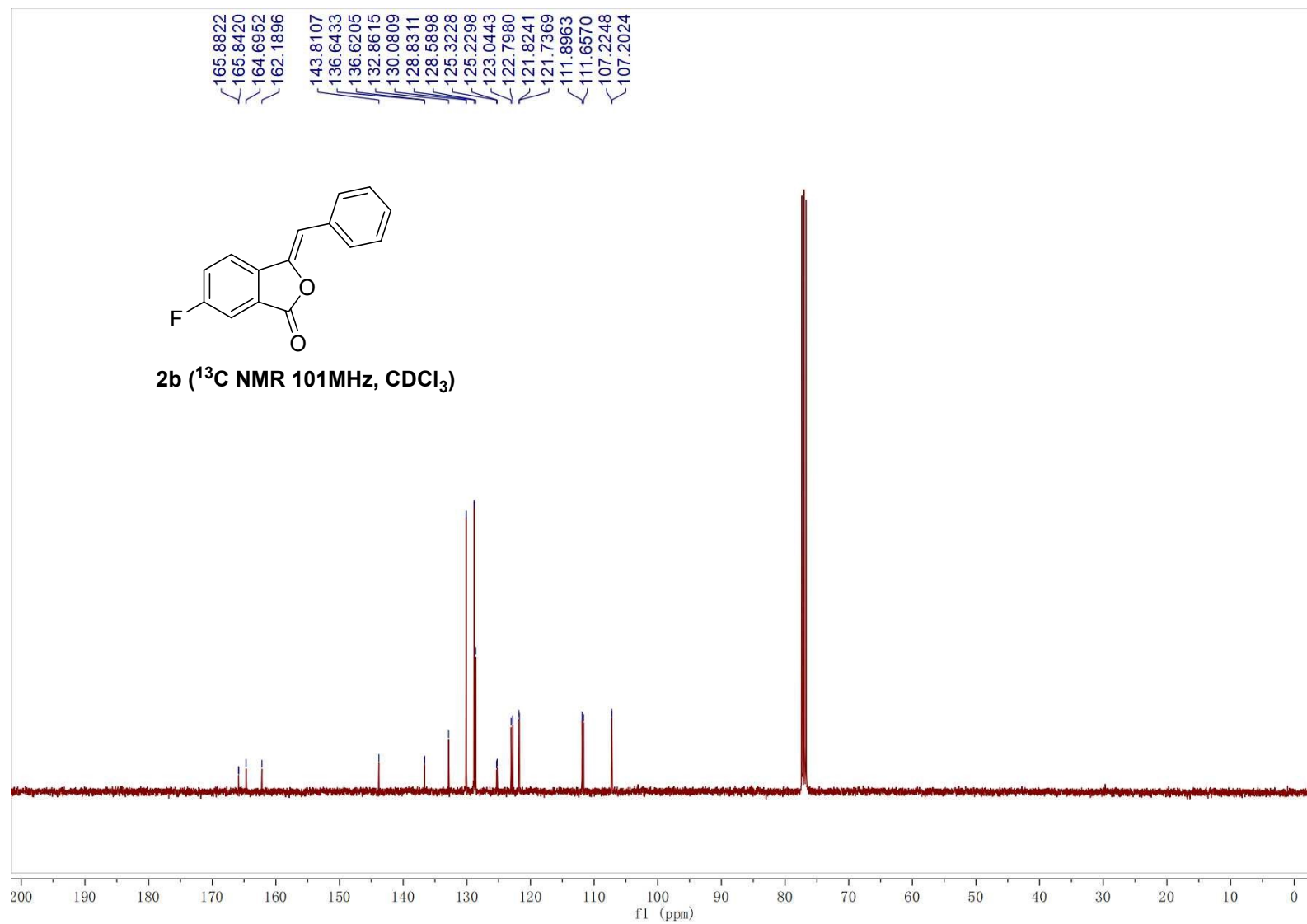
1. (a) S. Yang; S.-F. Zhu; N. Guo; S. Song; Q.-L. Zhou. *Org. Biomol. Chem.*, 2014, **12**, 2049-2052; (b) B. Suchand; G. Satyanarayana. *J. Org. Chem.*, 2016, **81**, 6409-6423; (c) B. N. Hemric; K. Shen; Q. Wang. *J. Am. Chem. Soc.*, 2016, **138**, 5813-5816; (d) J. Zhang; A. Jalil; J. He; Z. Yu; Y. Cheng; G. Li; Y. Du; K. Zhao. *Chem. Commun.*, 2021, **57**, 7426-7429.
2. N. G. Kundu; M. Pal; B. Nandi. *J. Chem. Soc., Perkin Trans. 1*, 1998, 561-568.
3. E. d. Olmo; B. Barboza; L. D. Chiaradia; A. Moreno; J. Carrero-Lérida; D. González-Pacanowska; V. Muñoz; J. L. López-Pérez; A. Giménez; A. Benito; A. R. Martínez; L. M. Ruiz-Pérez; A. San Feliciano. *Eur. J. Med. Chem.*, 2011, **46**, 5379-5386.
4. E. A. Merritt; V. M. T. Carneiro; L. F. Silva; B. Olofsson. *J. Org. Chem.*, 2010, **75**, 7416-7419.

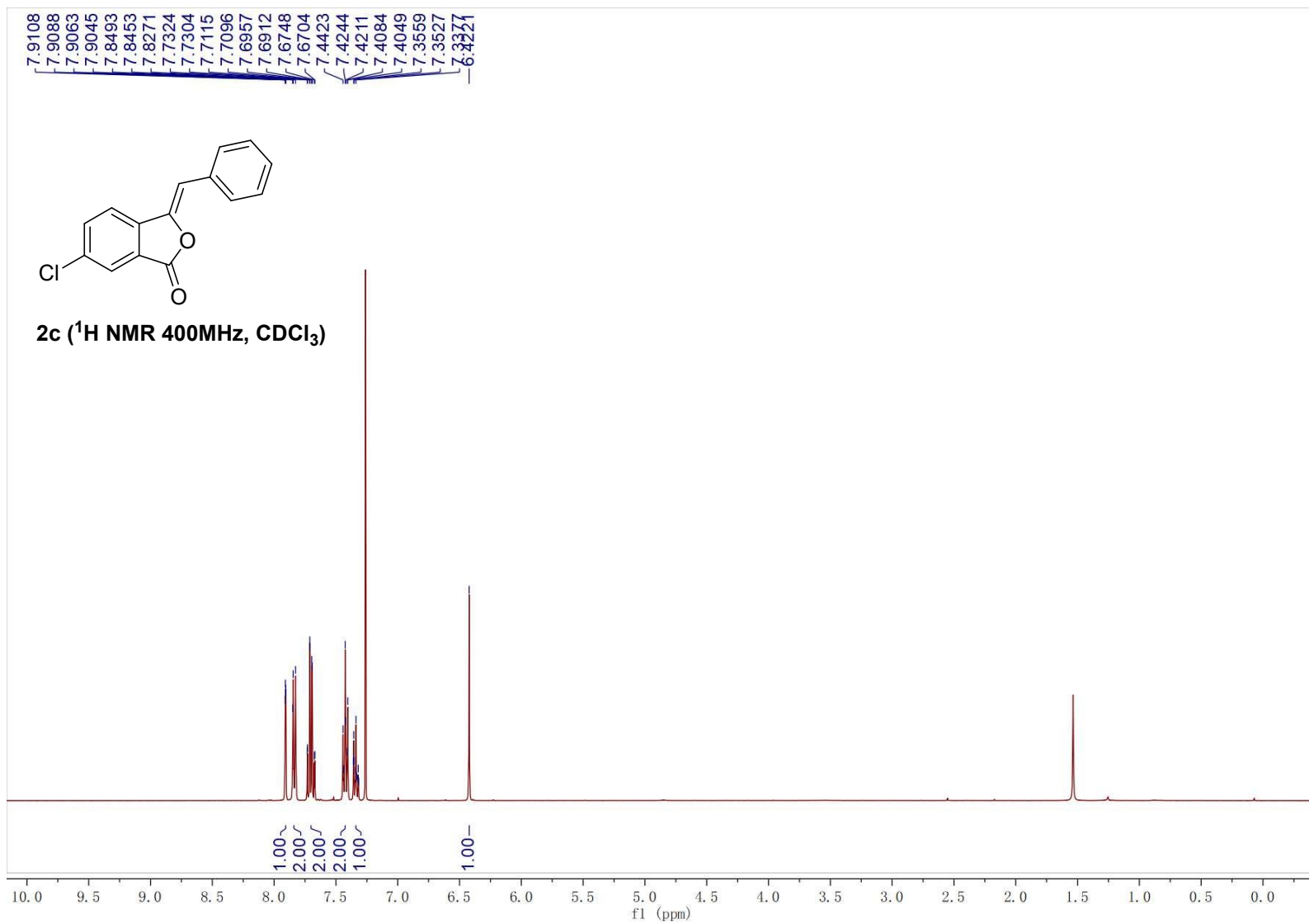
IV. ^1H and ^{13}C NMR Spectra of Compounds 2a-u, 3a-l, 5, 6:

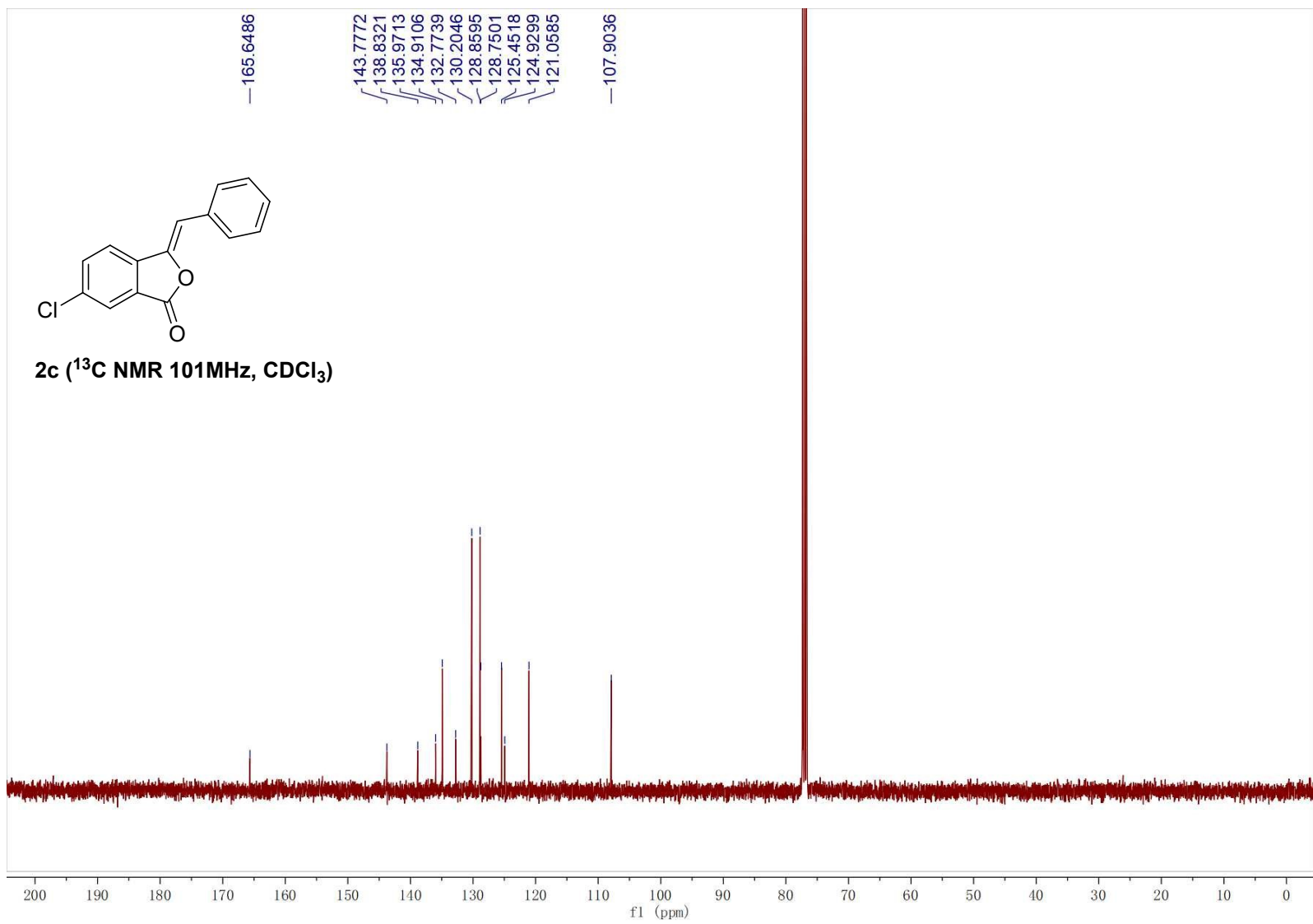


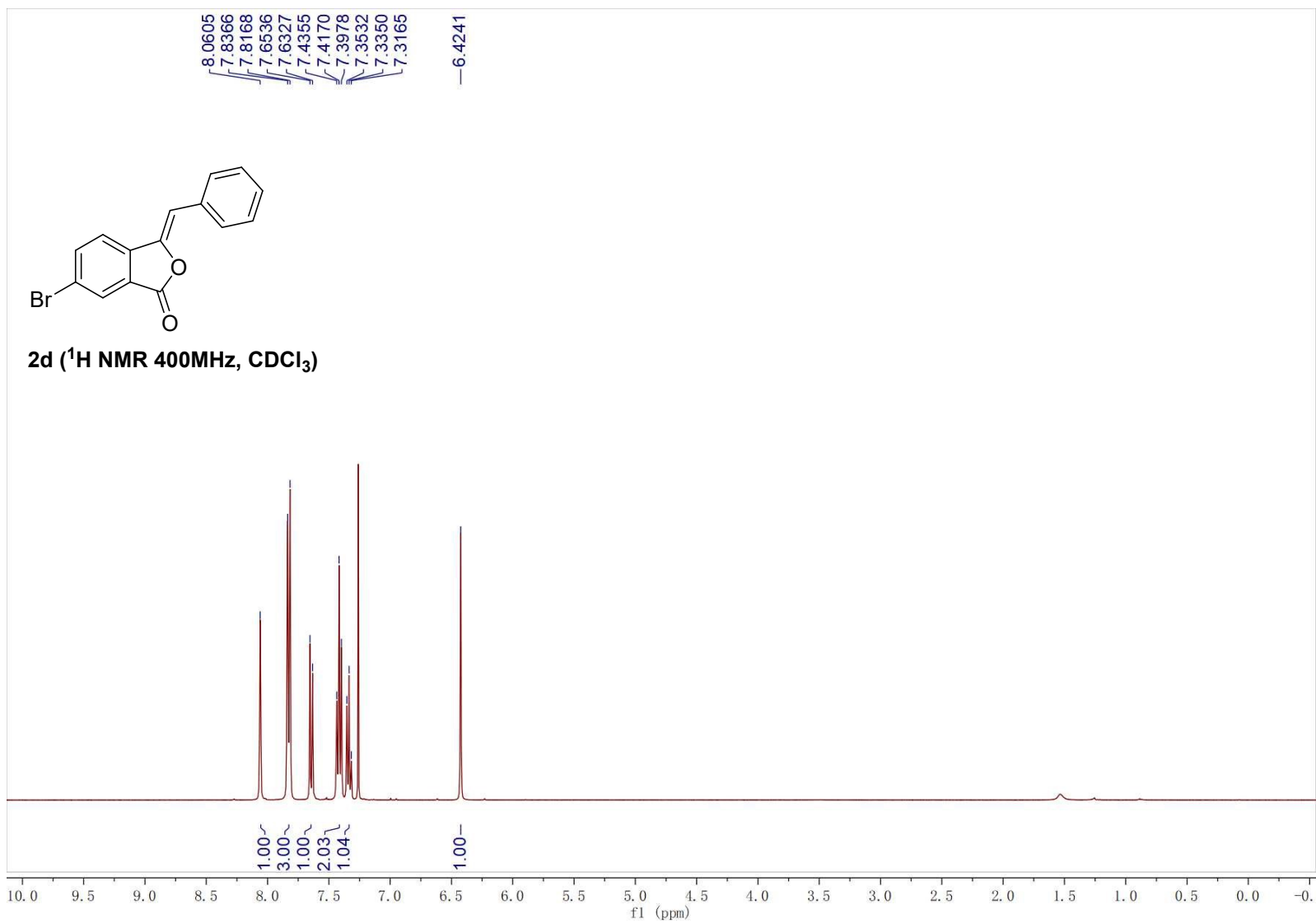


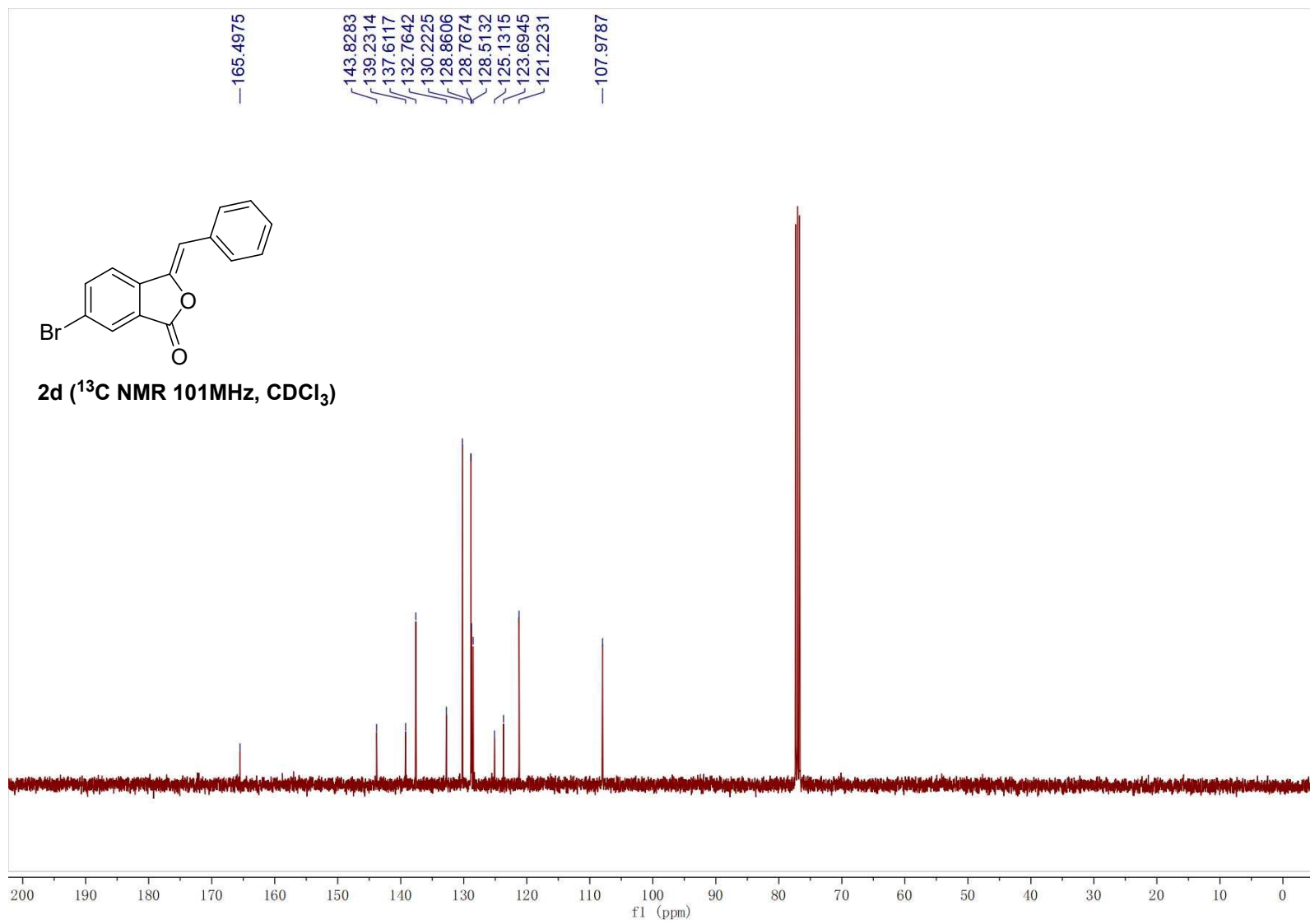


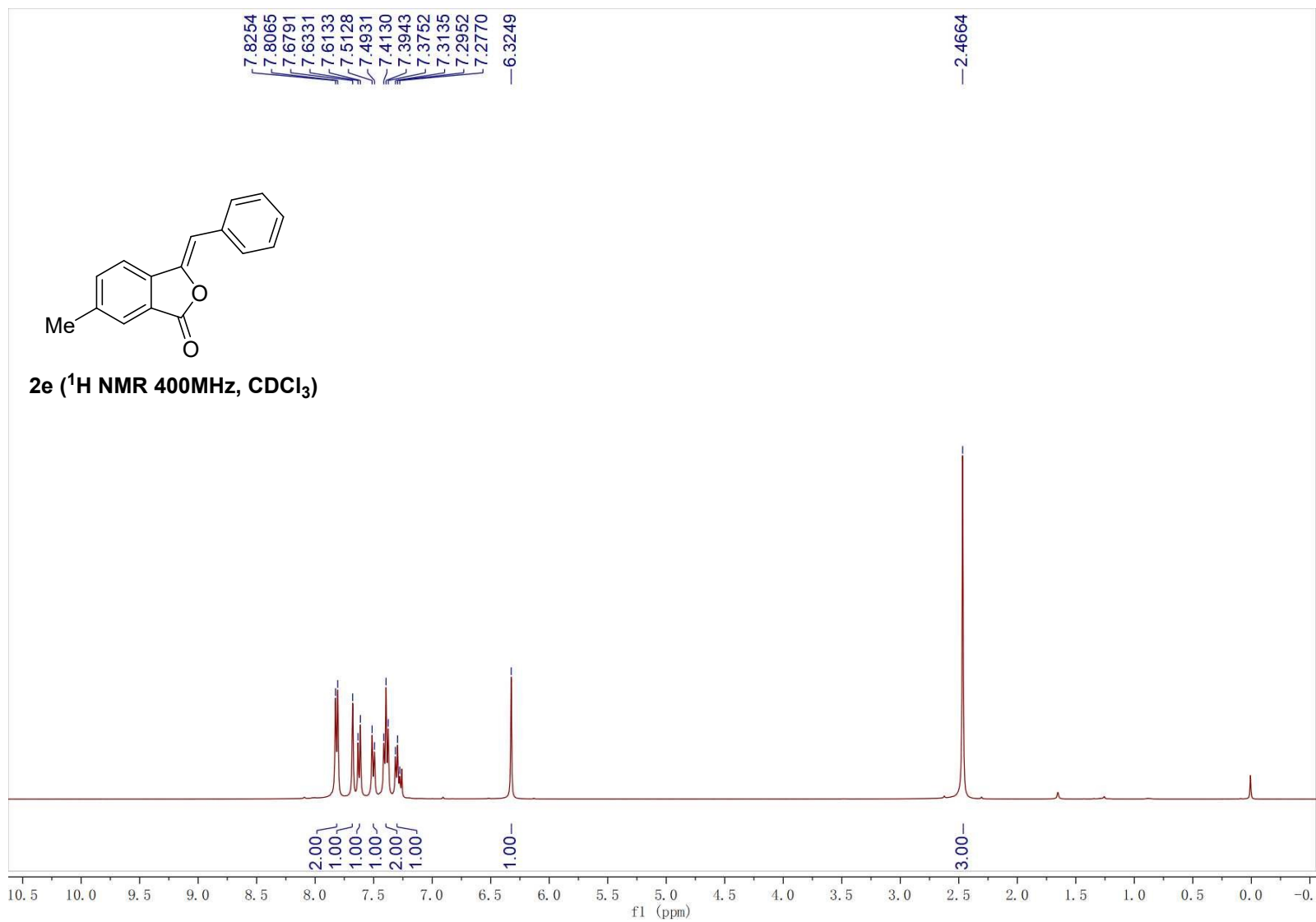


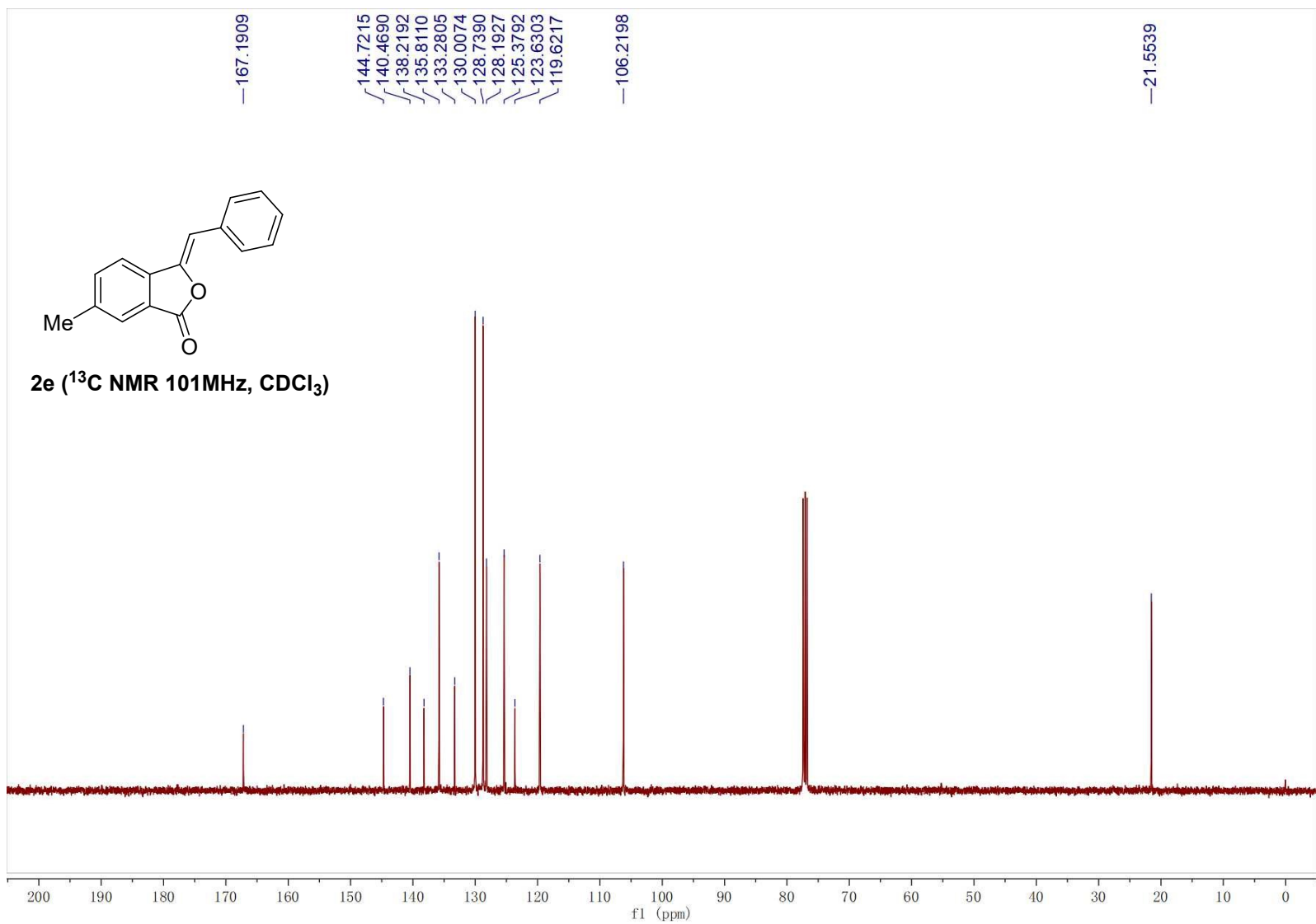


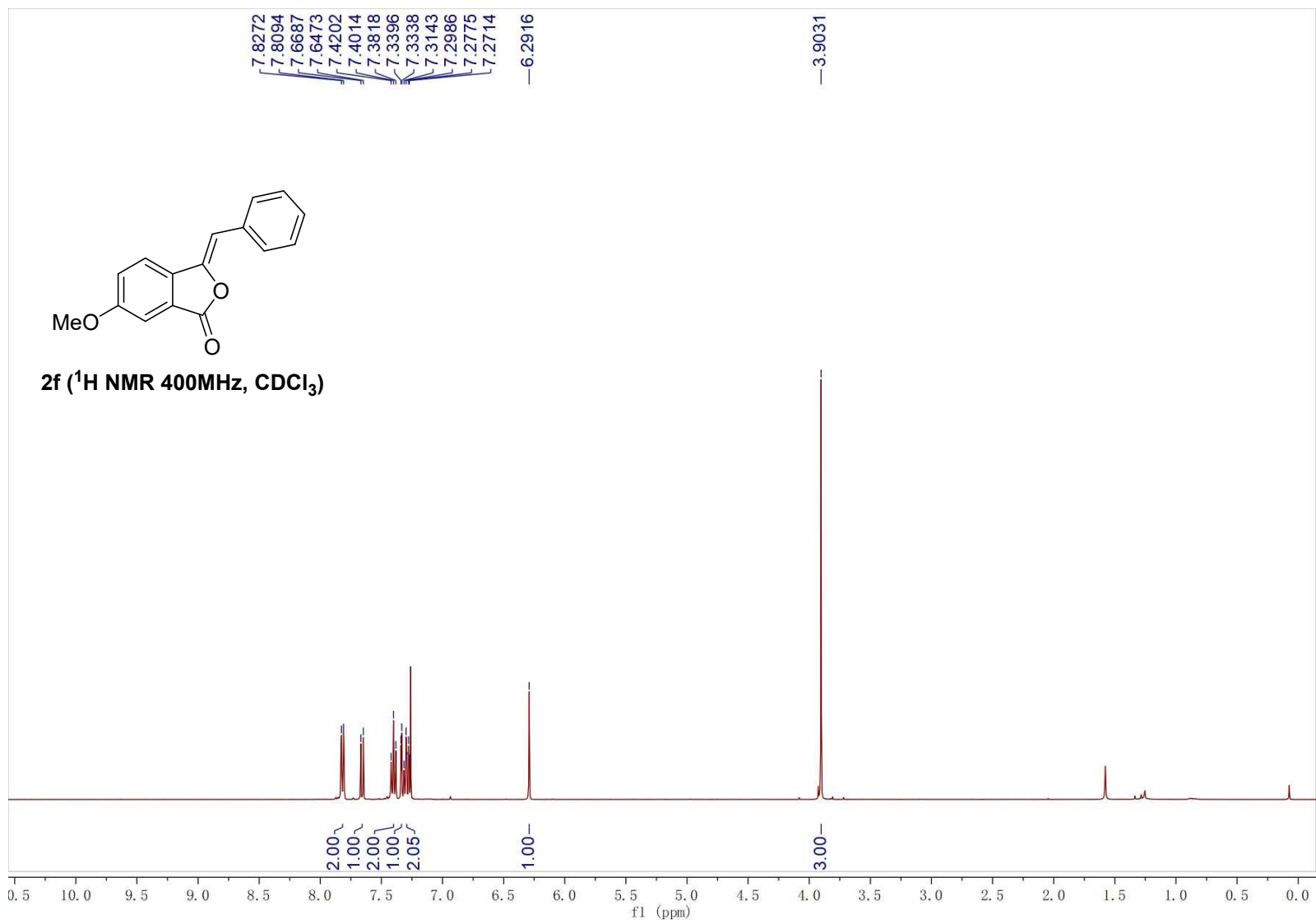


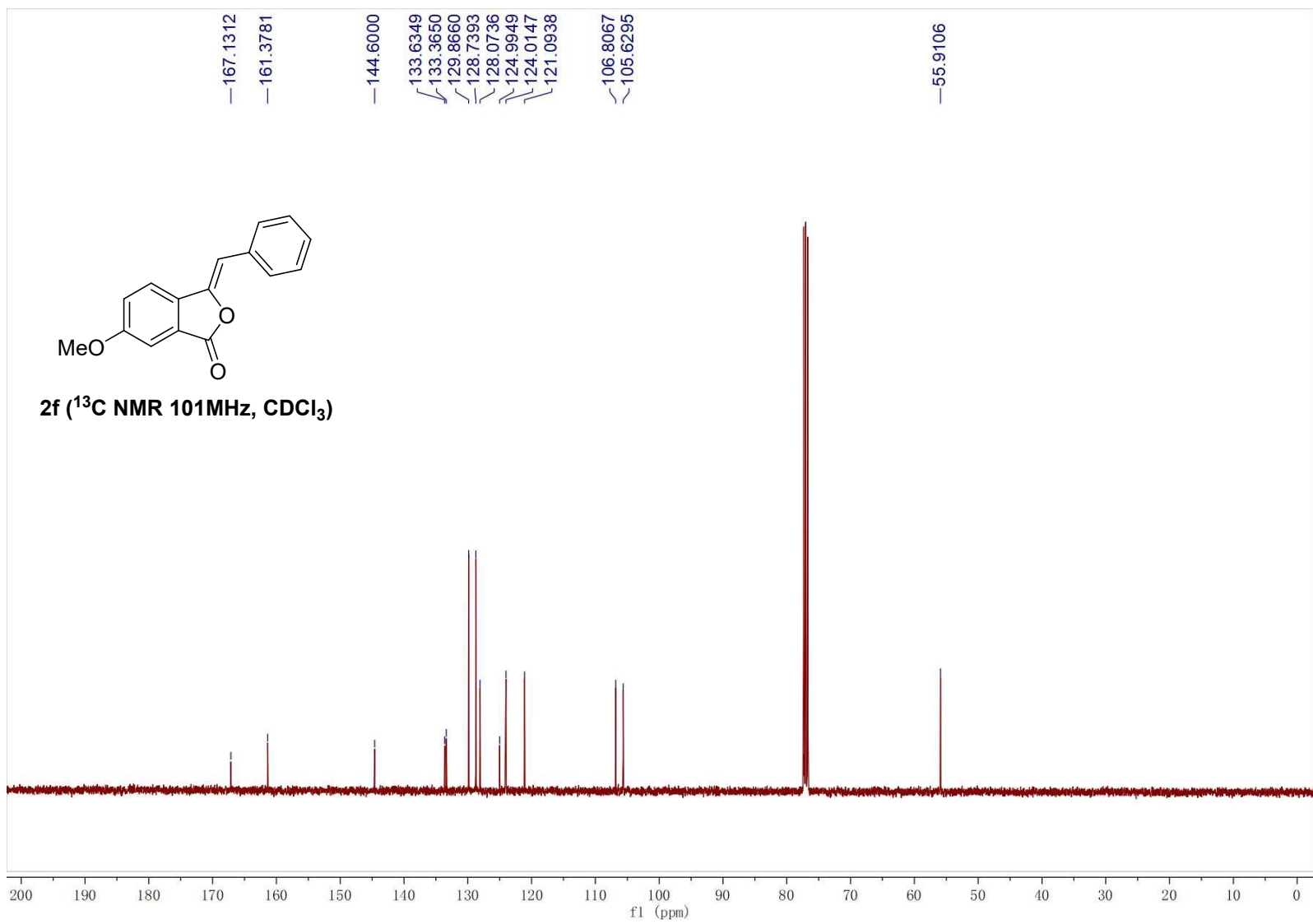


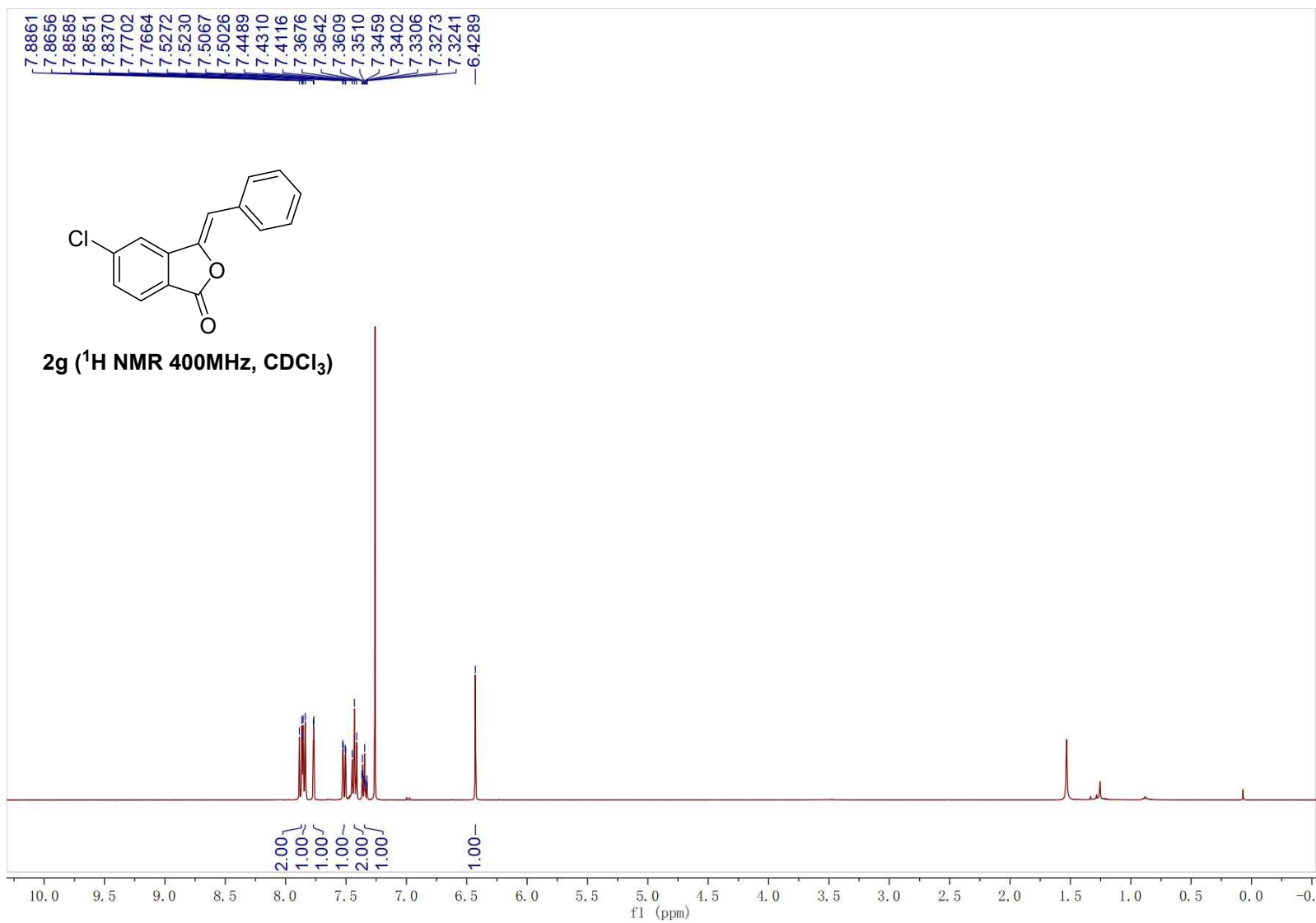


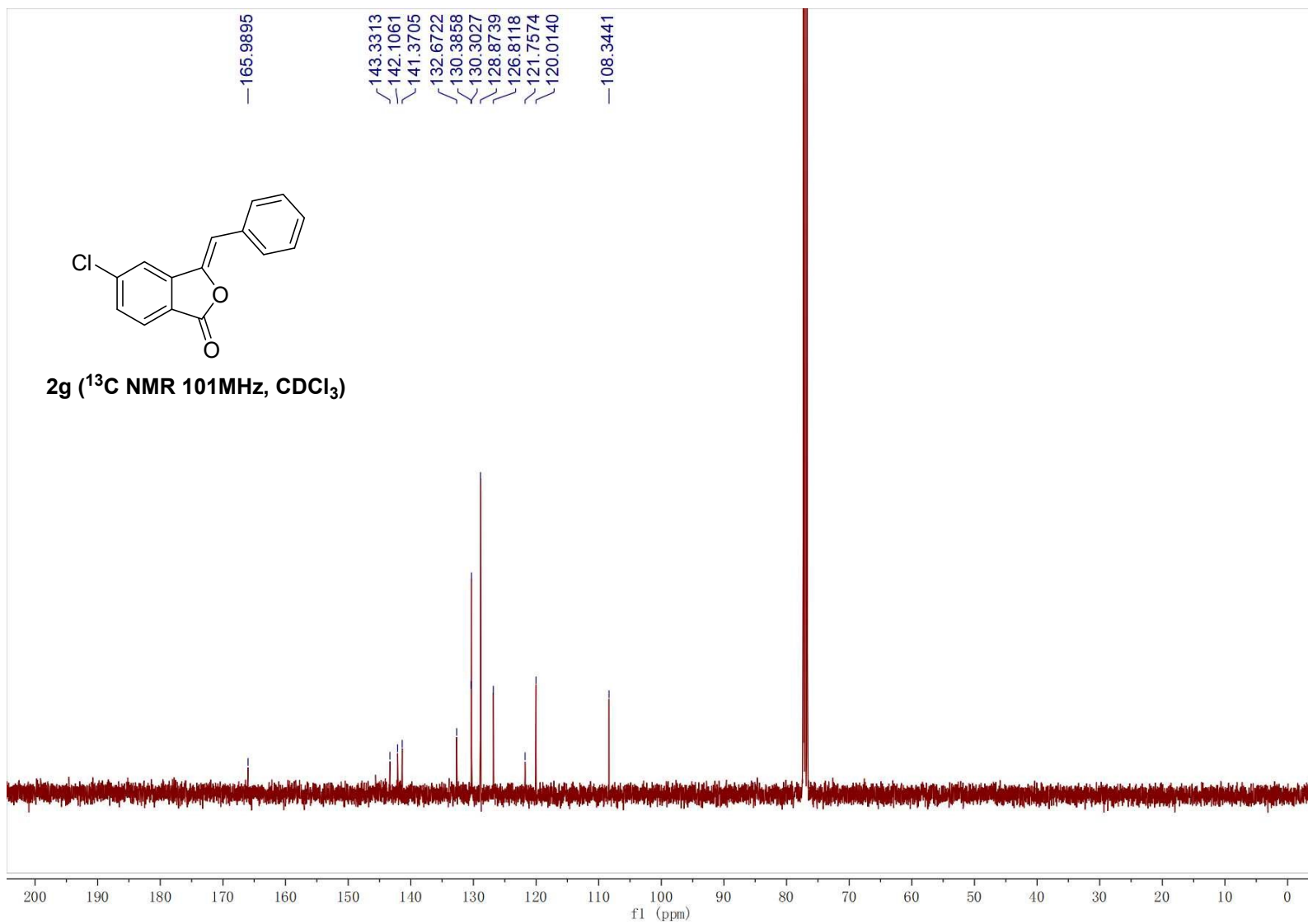


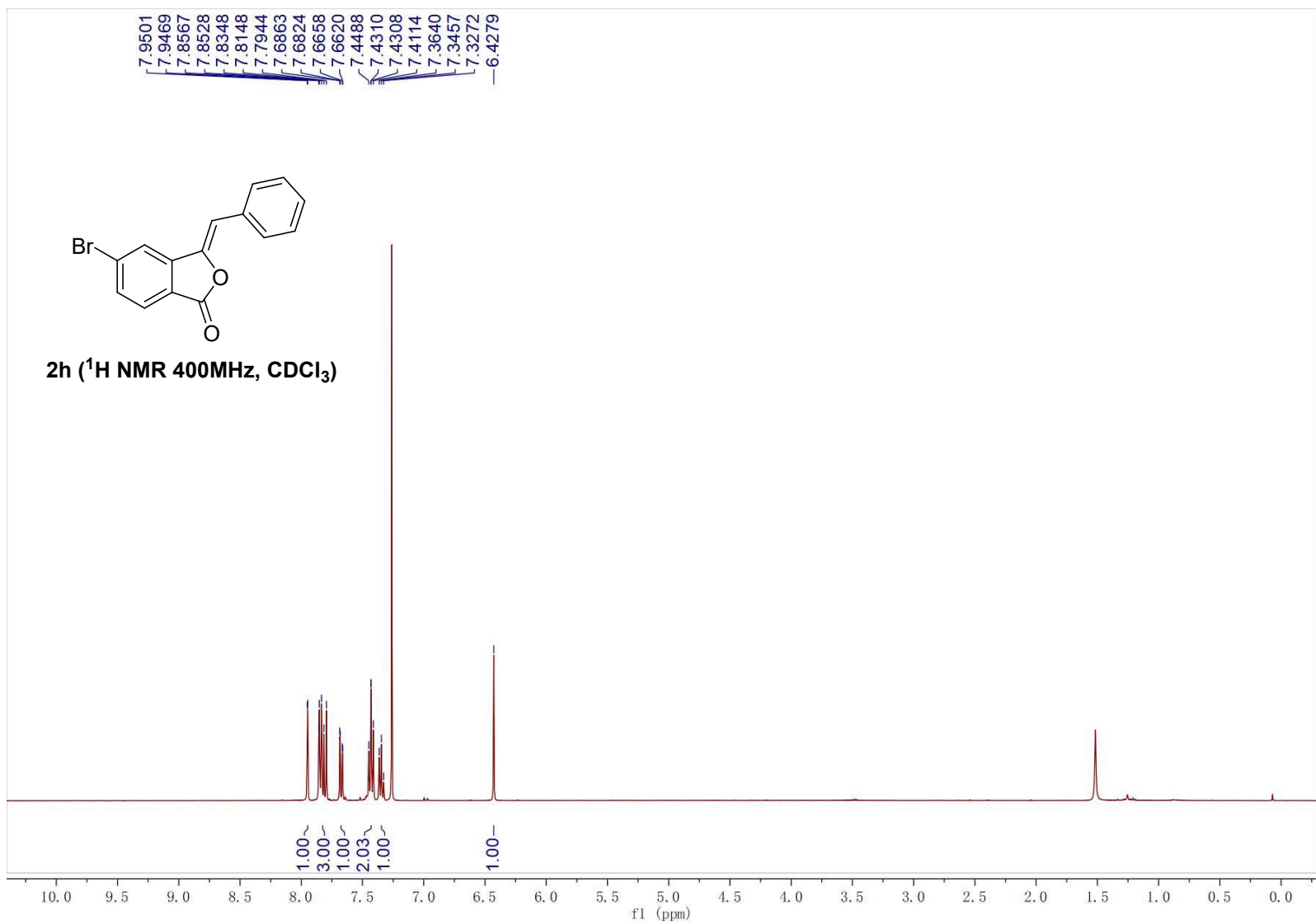


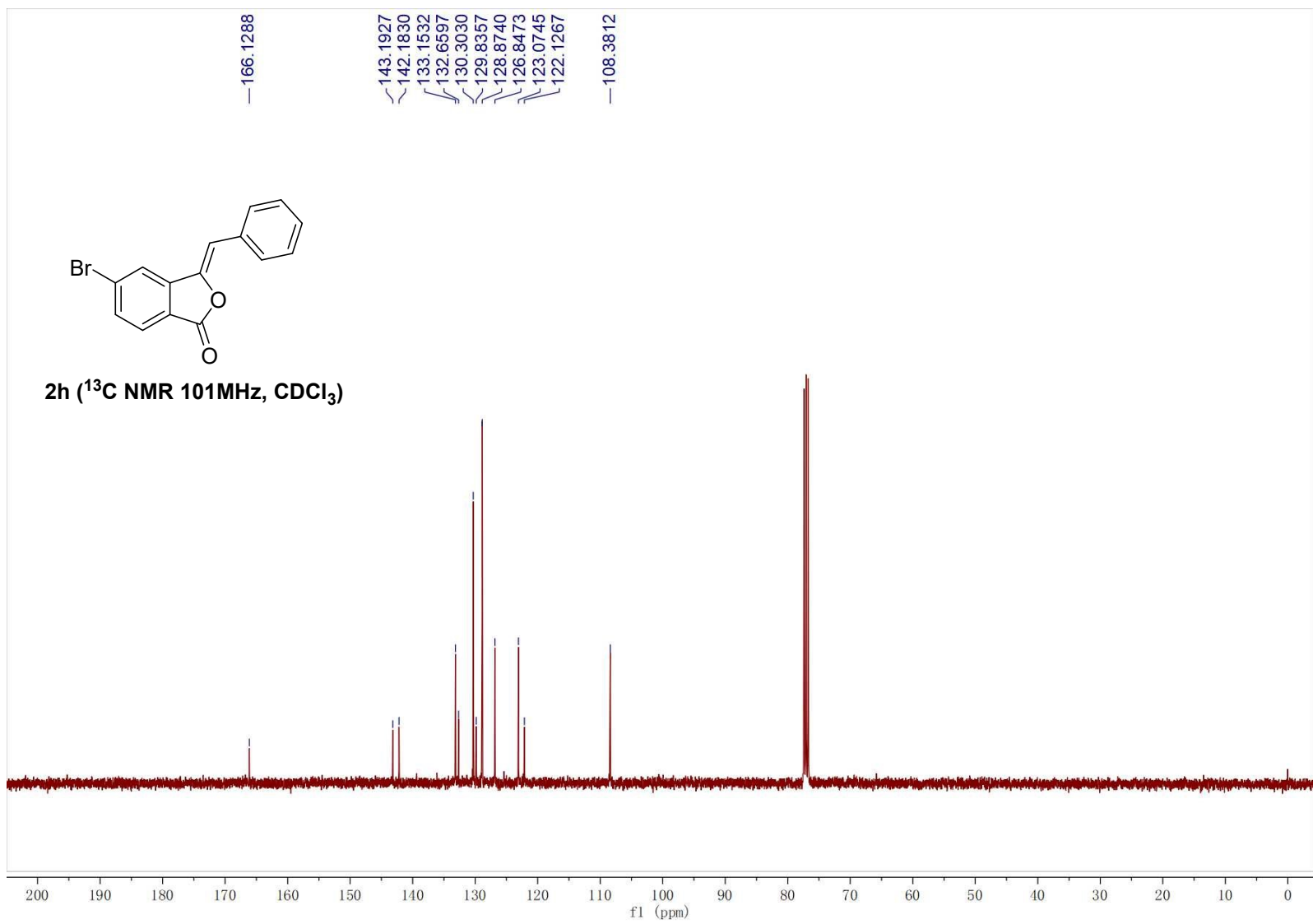


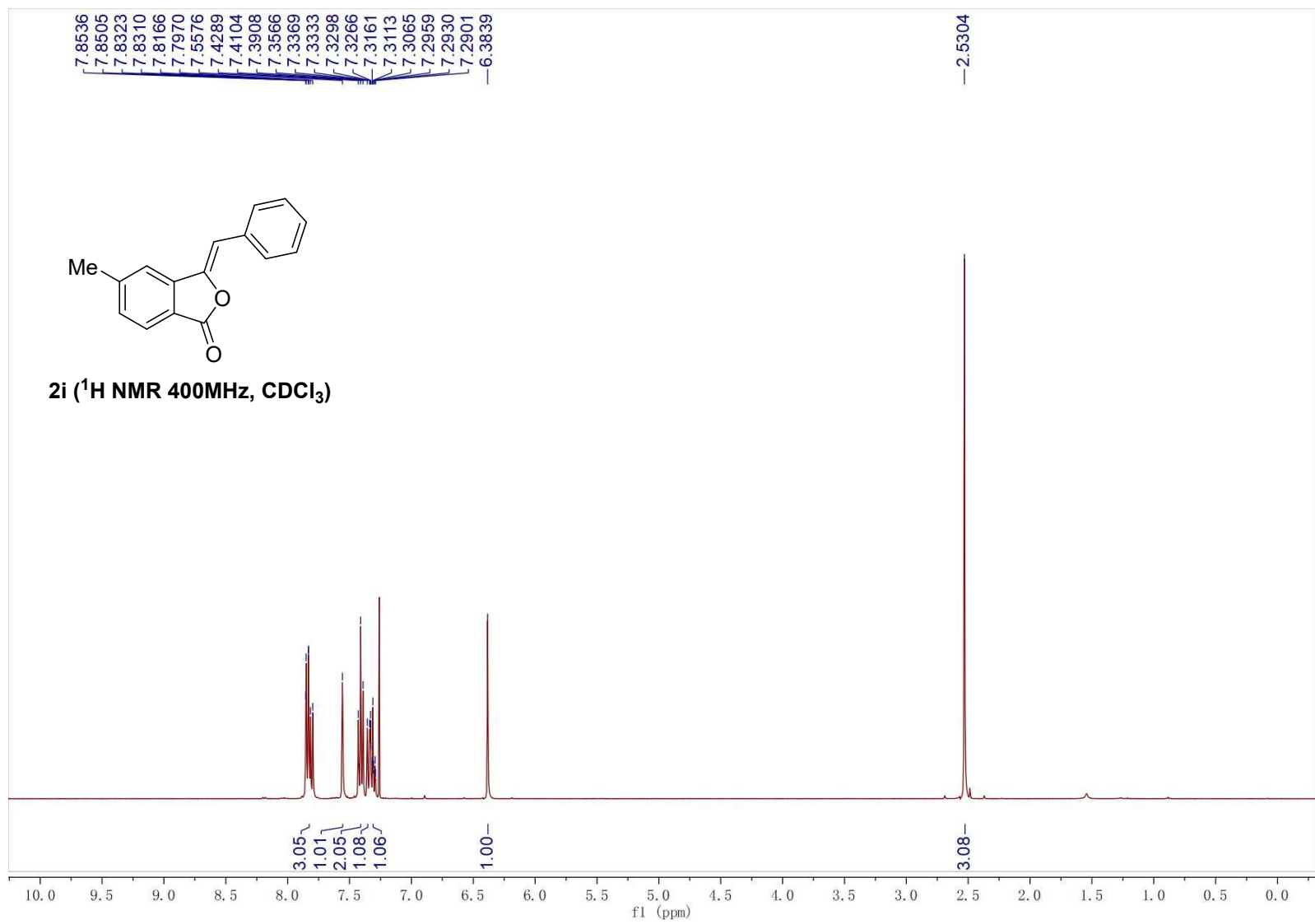


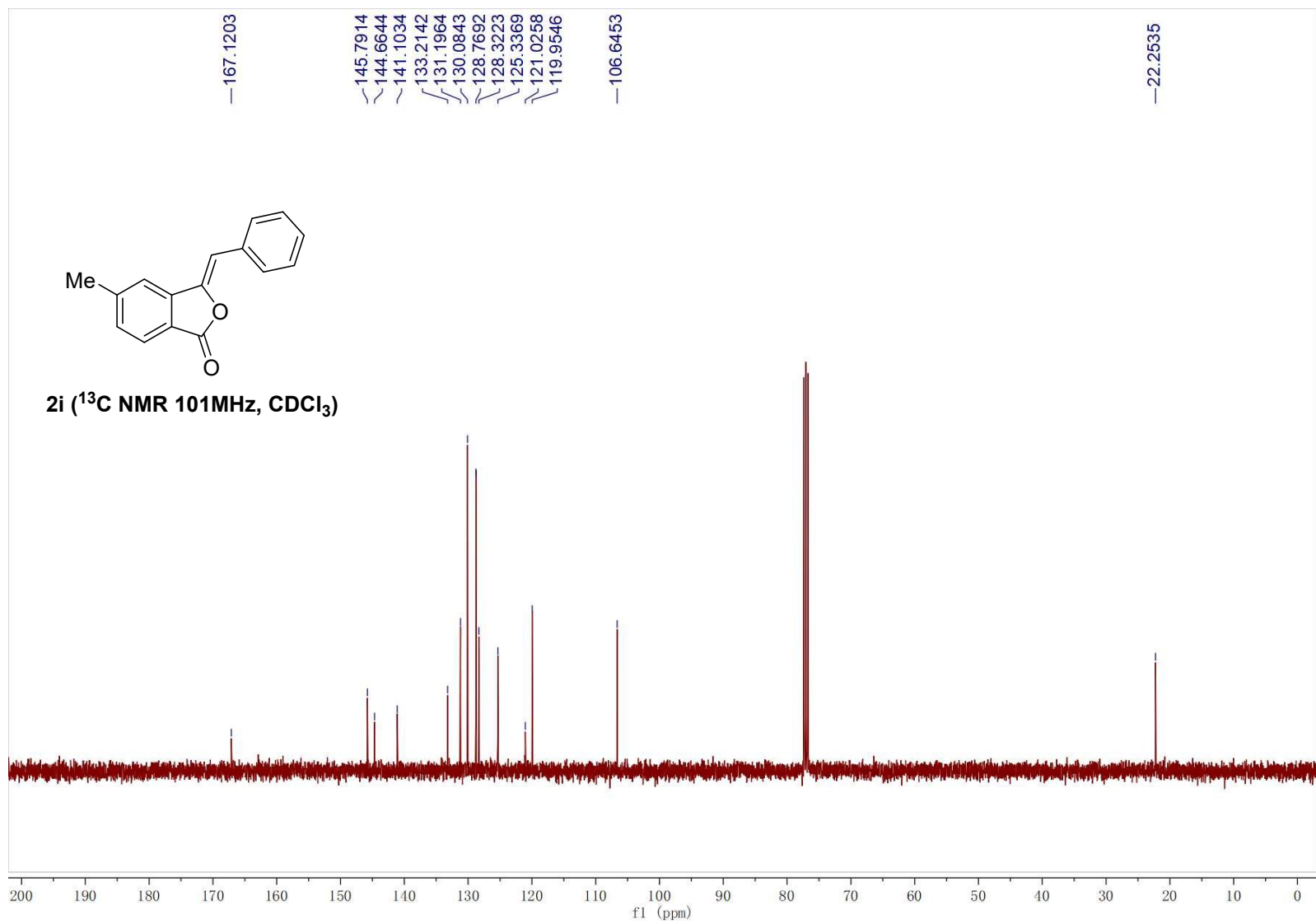


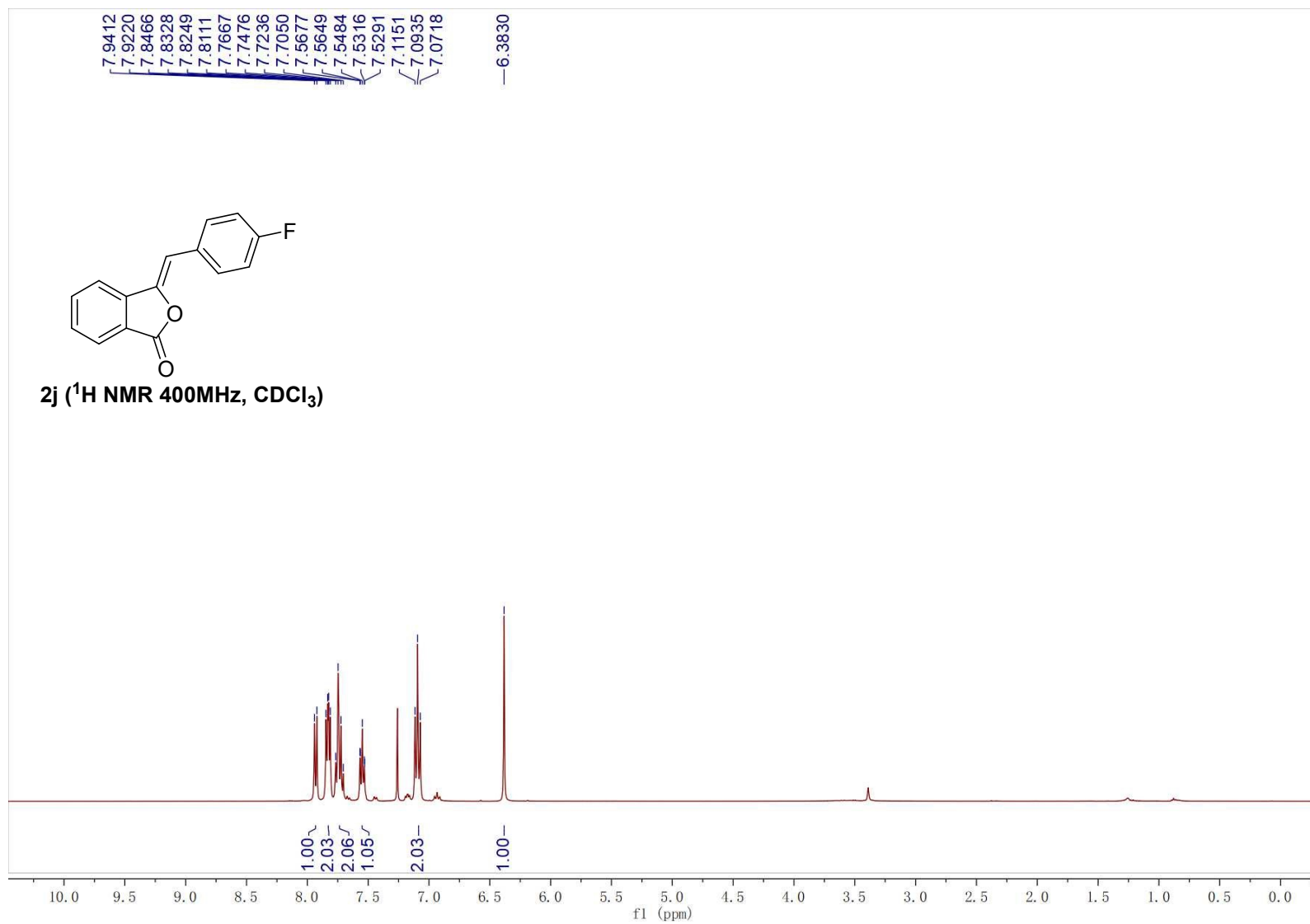


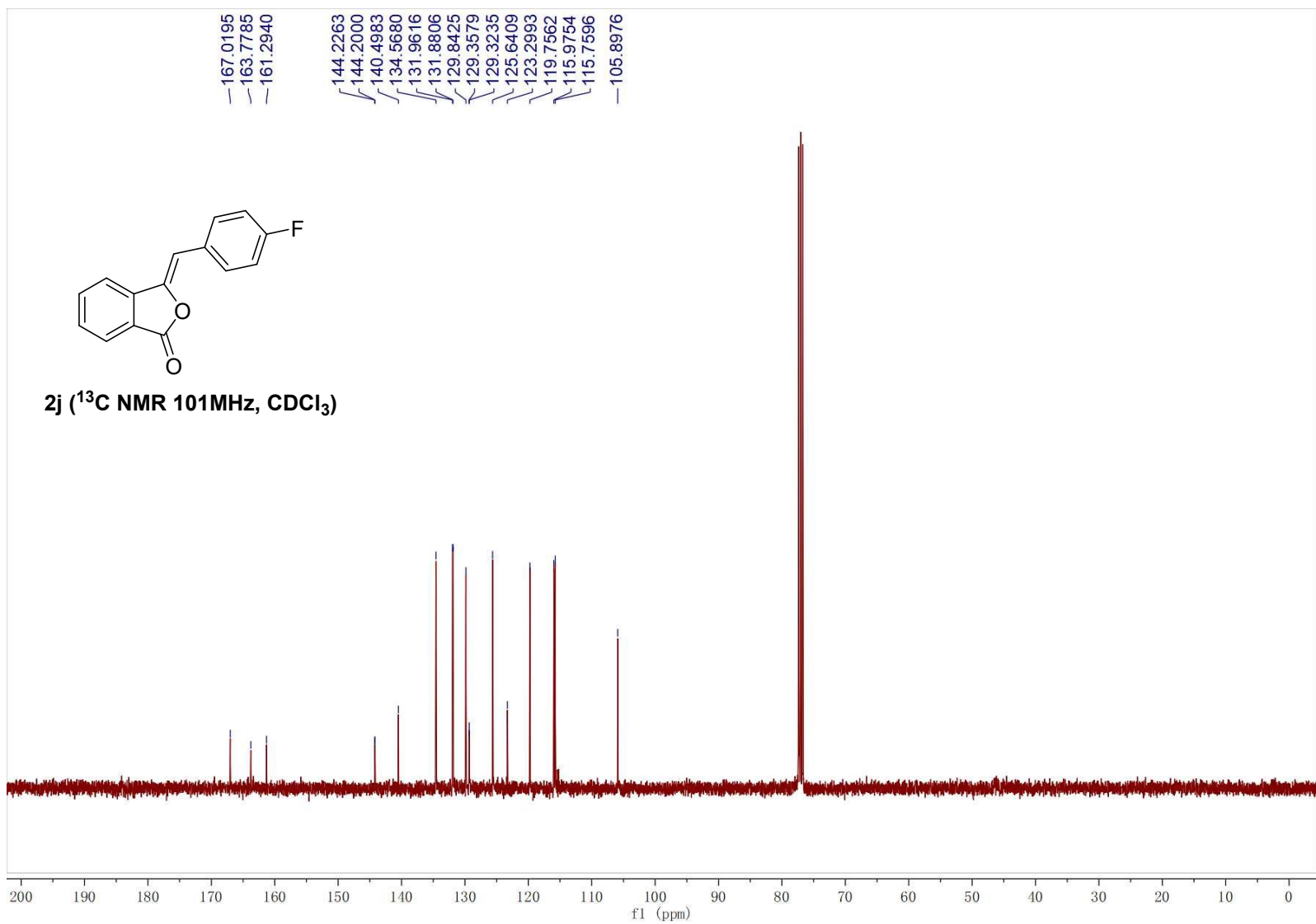


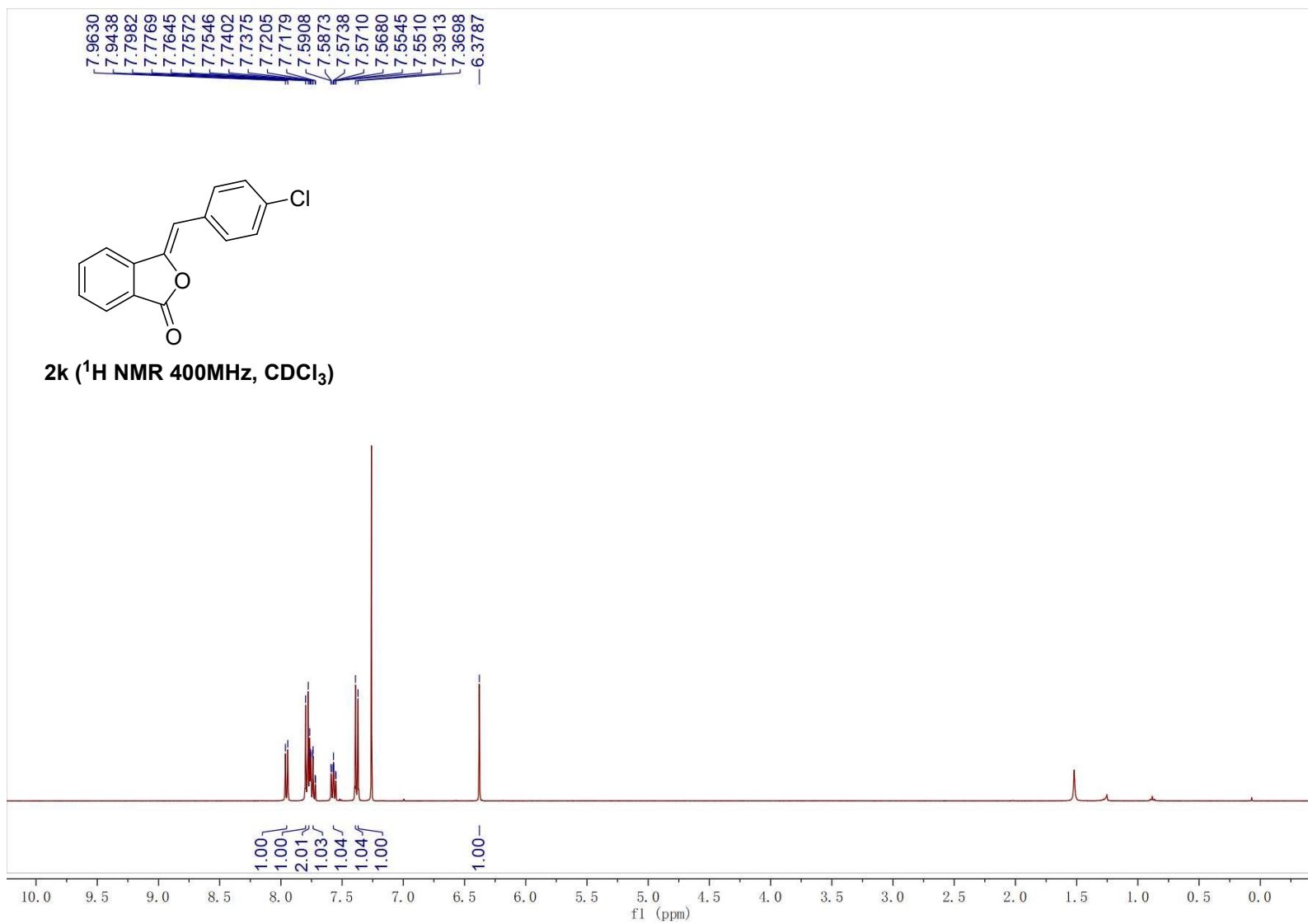


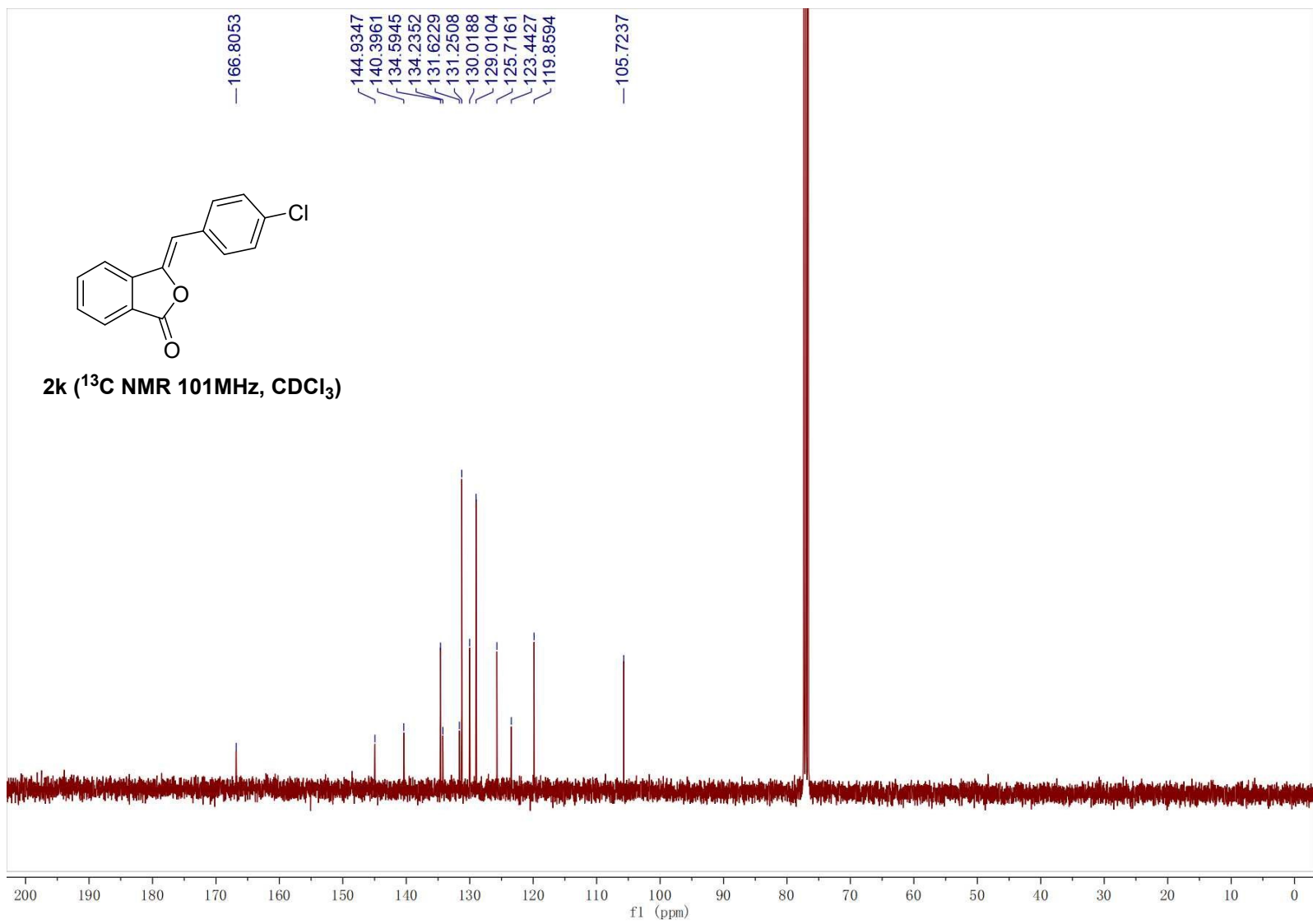


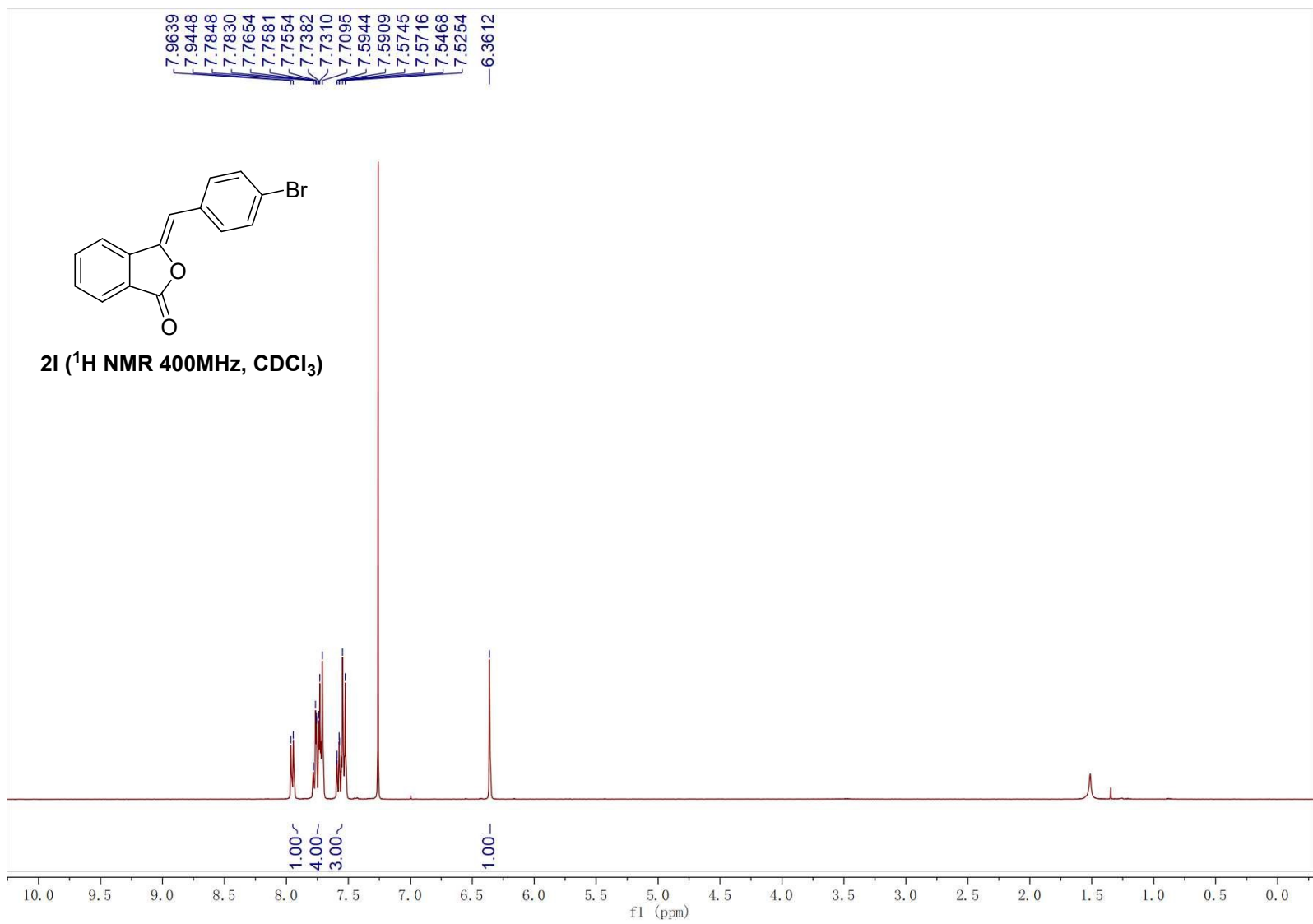


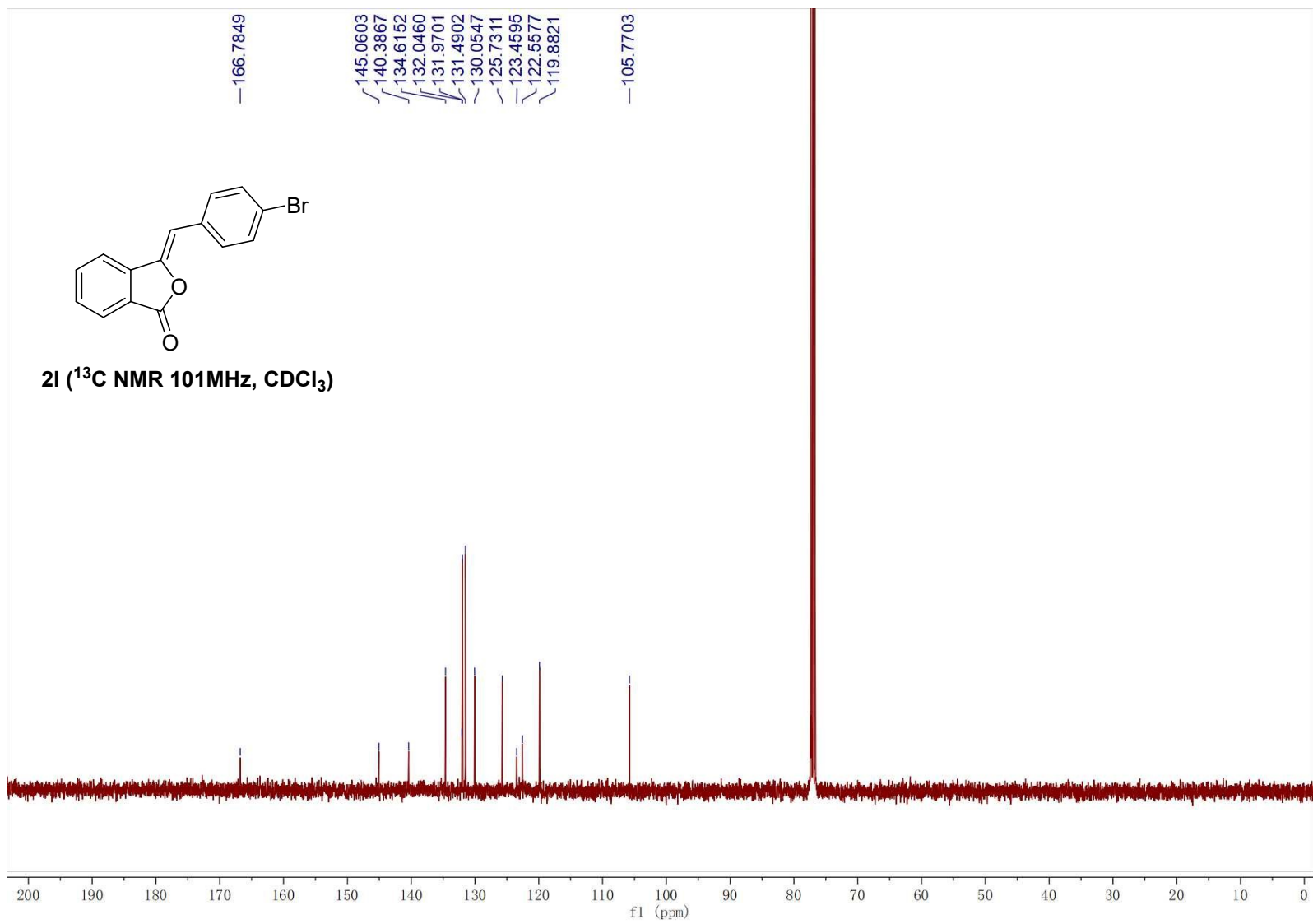


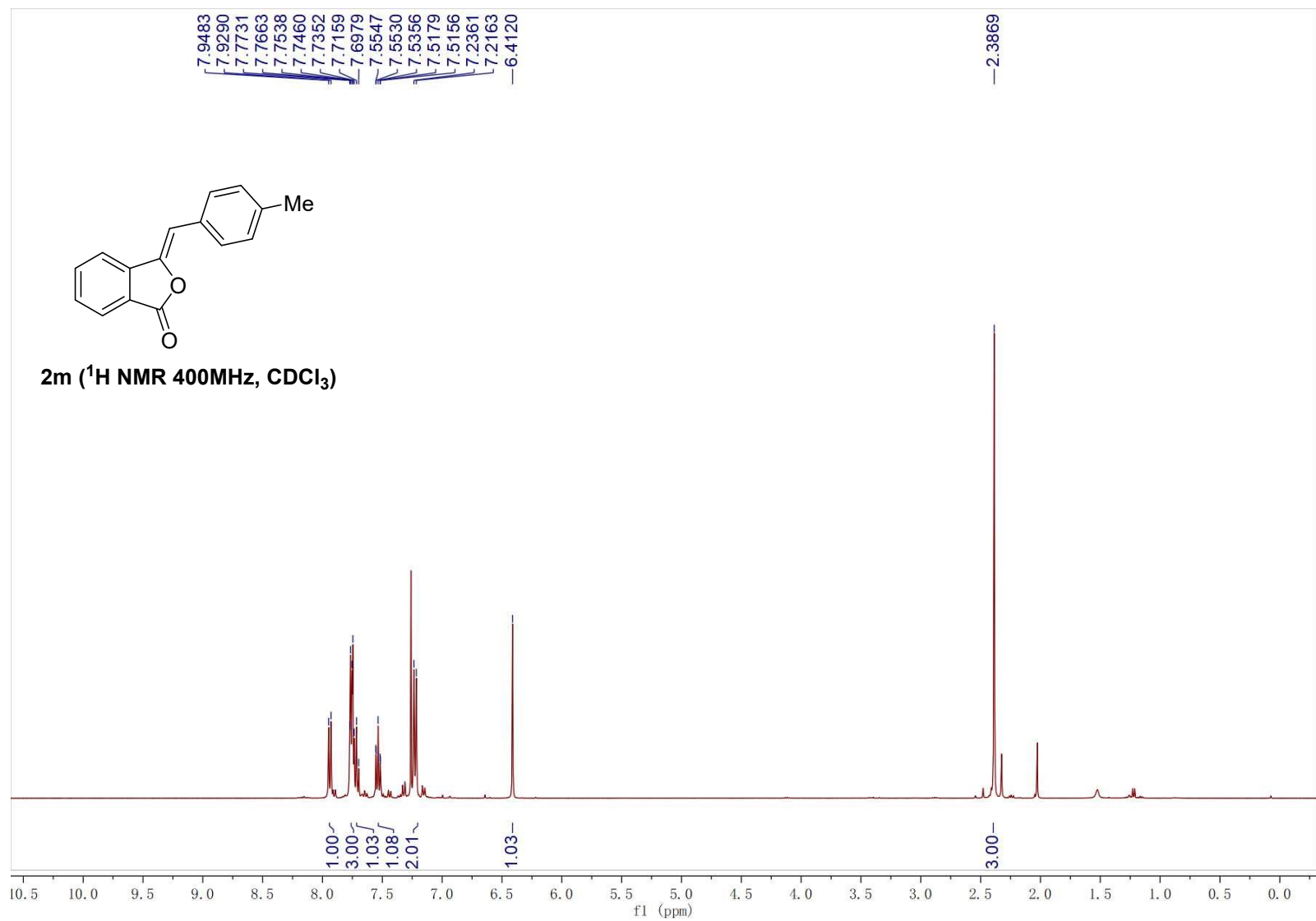


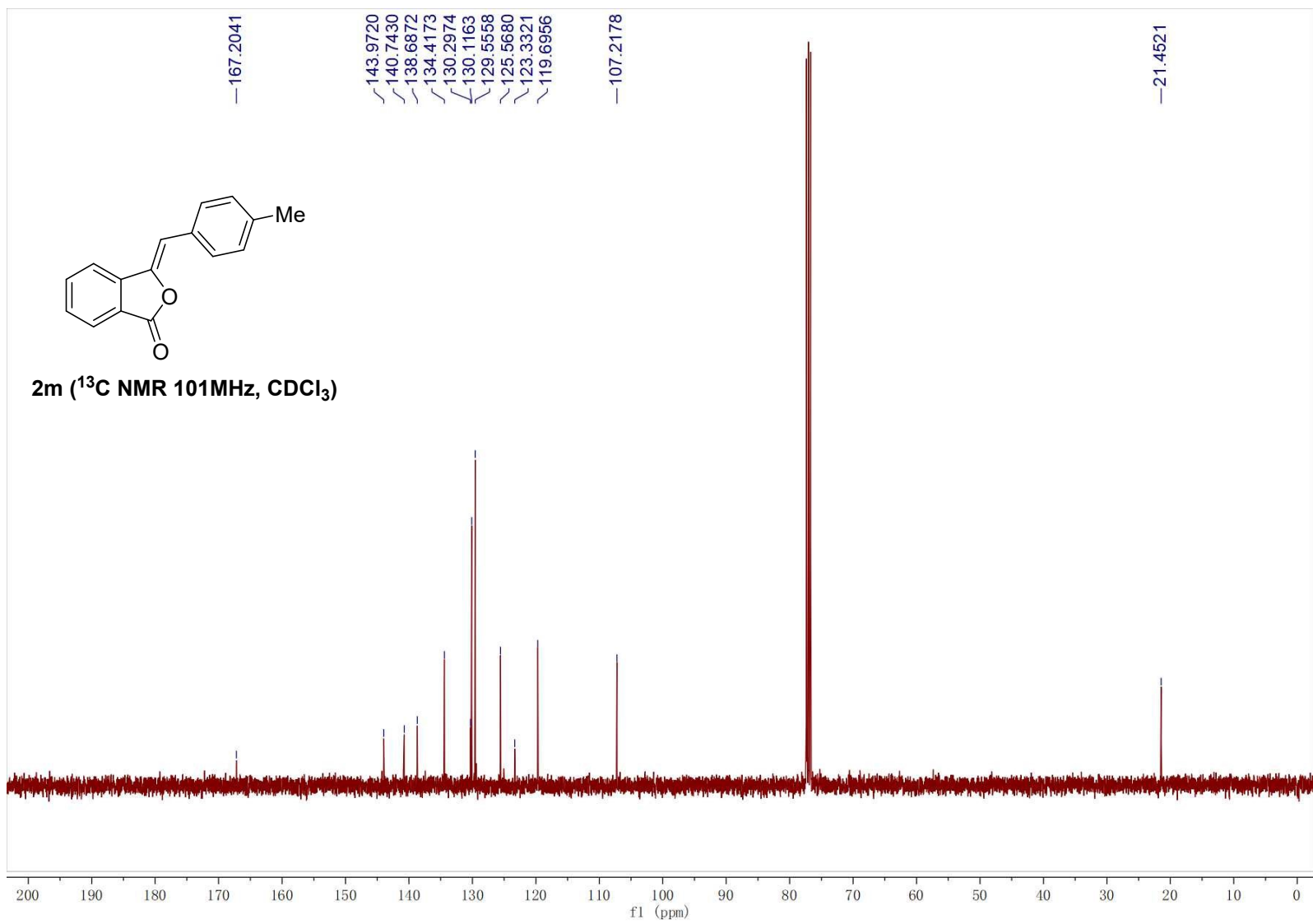


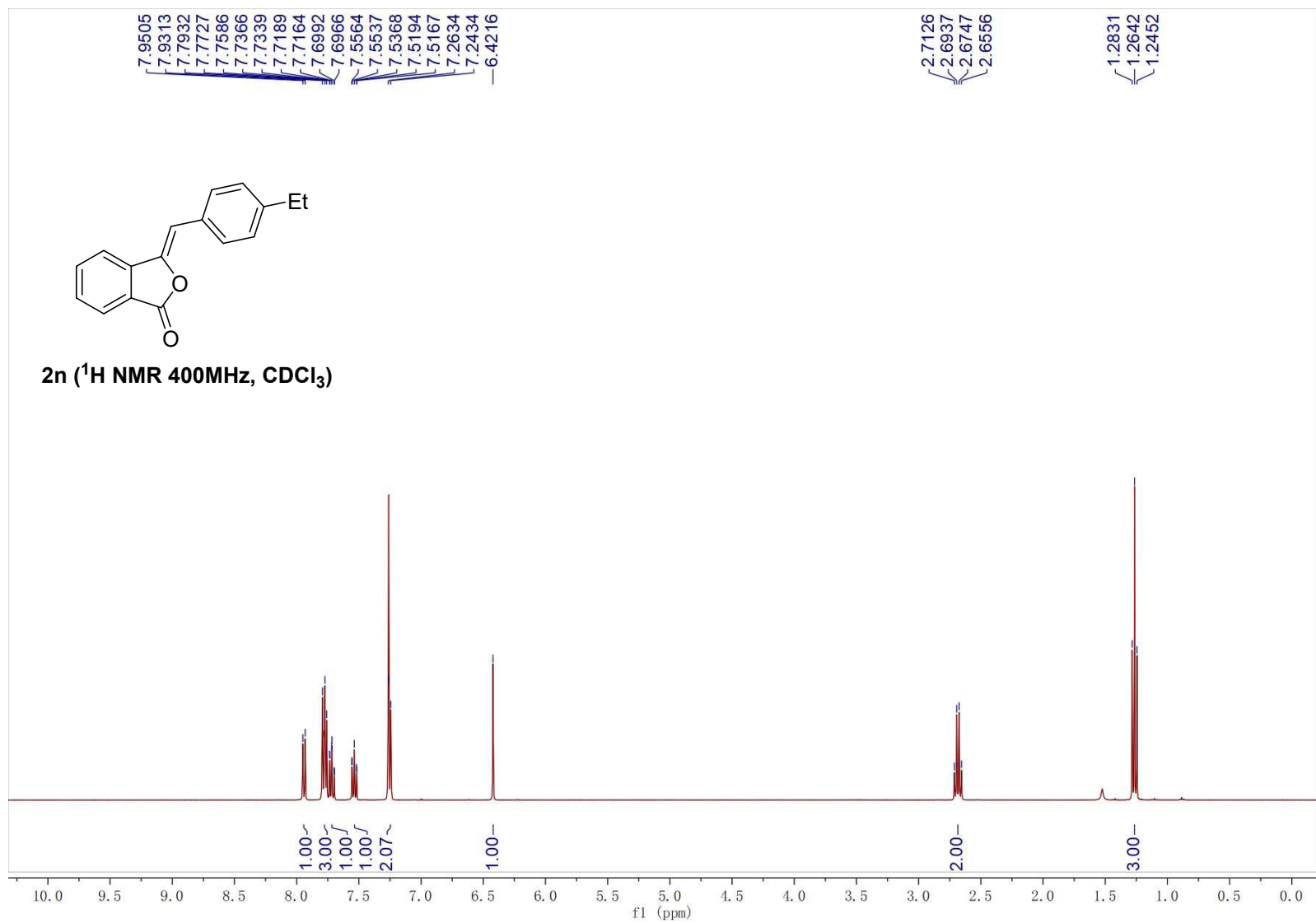


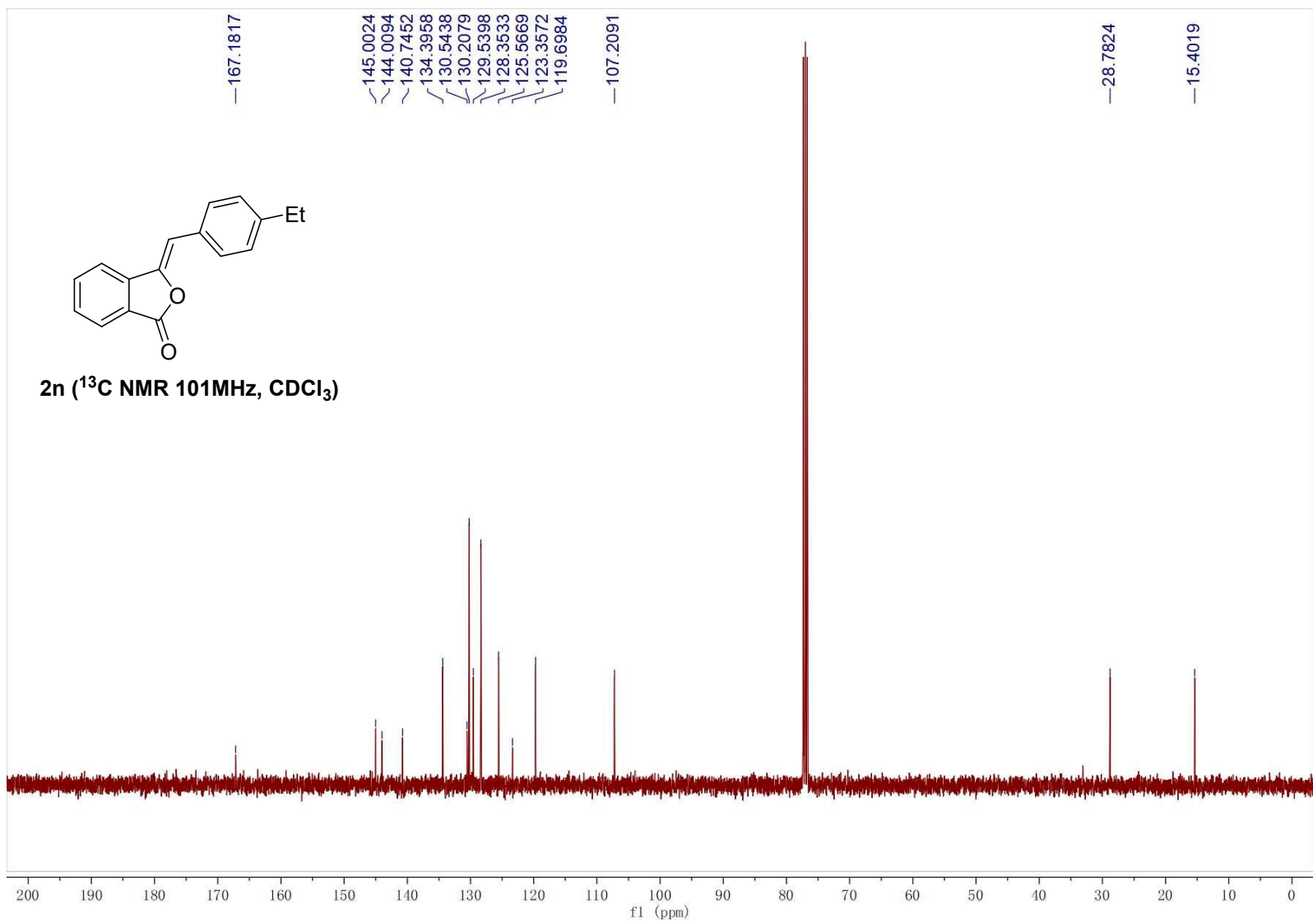


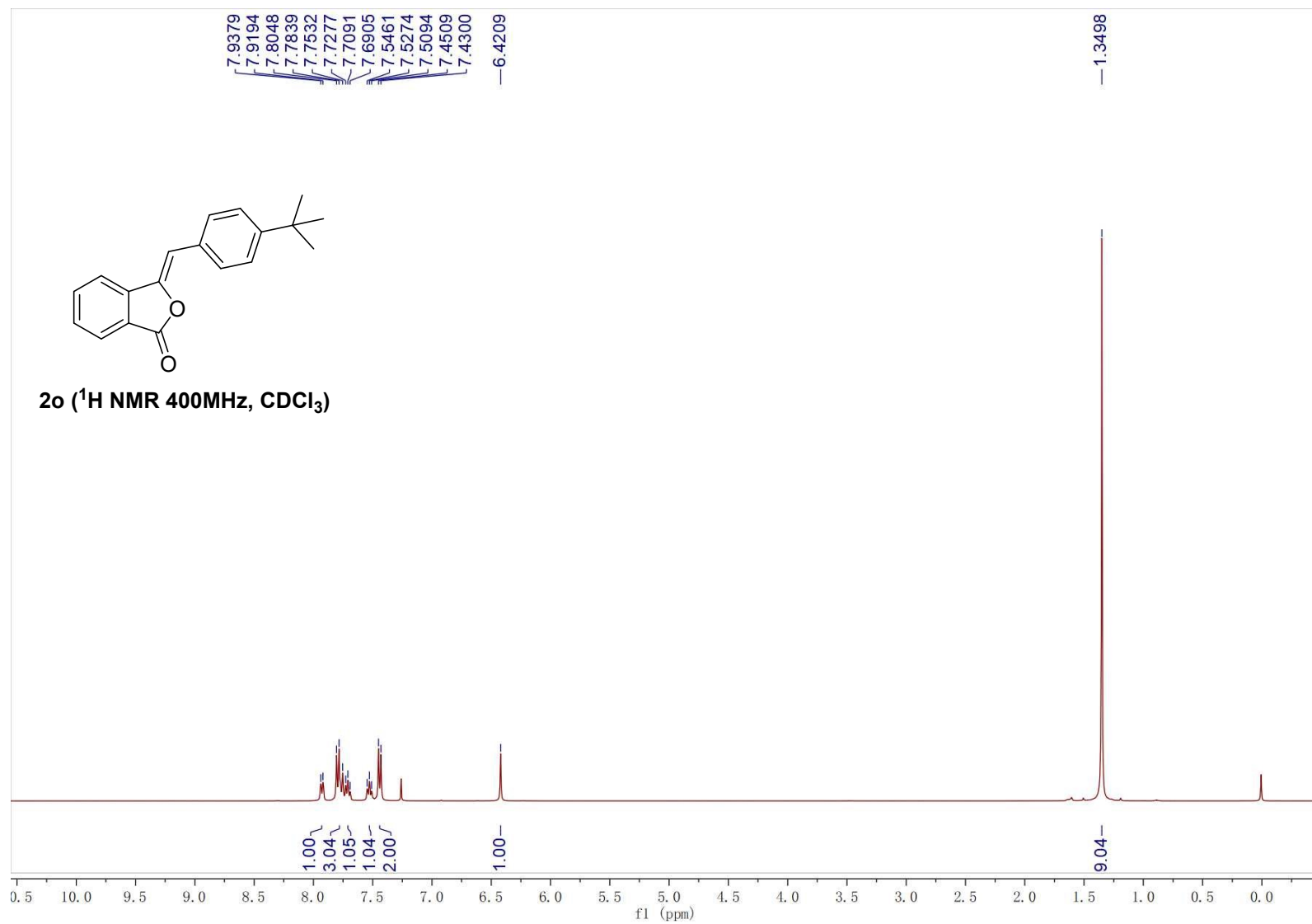


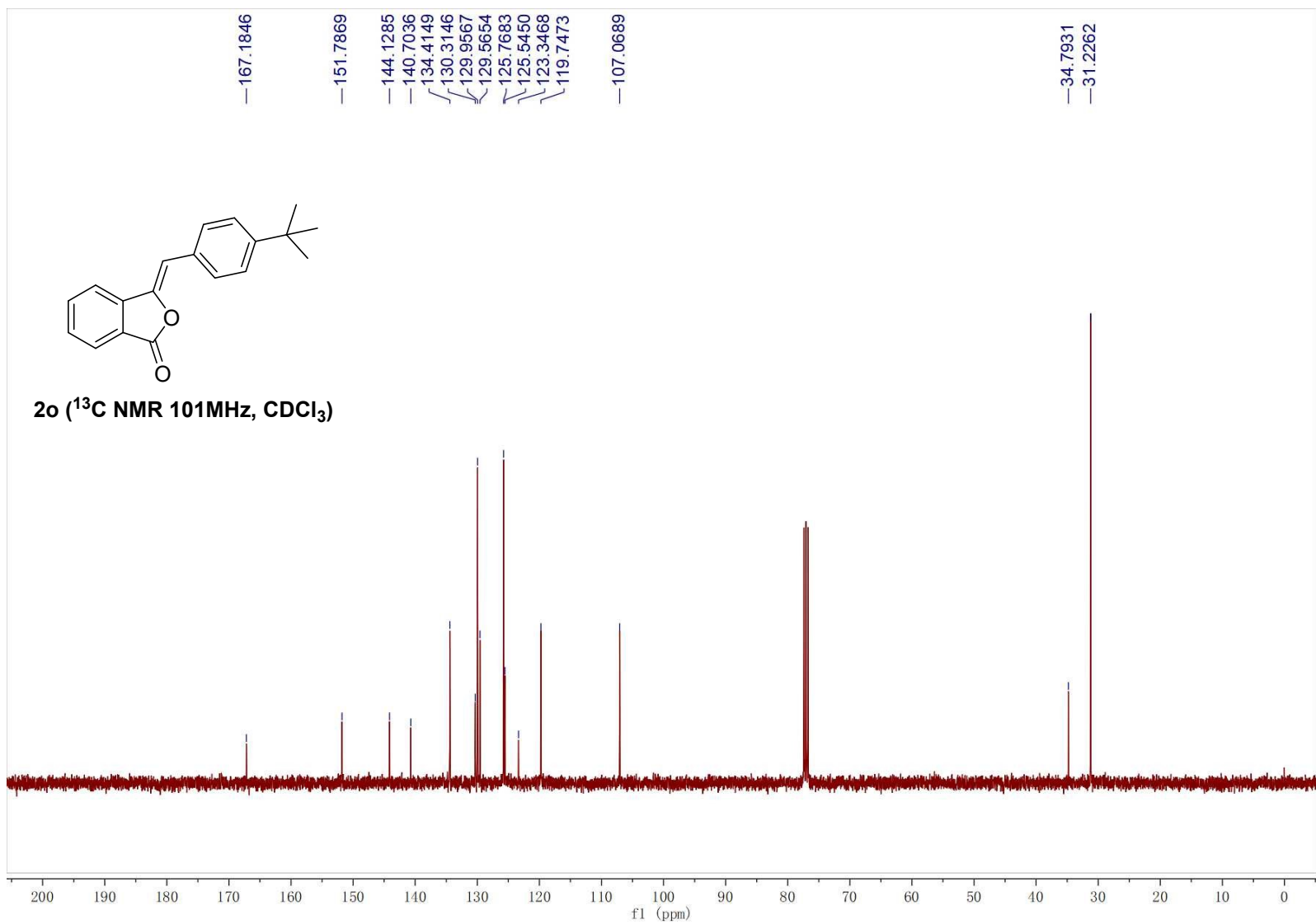


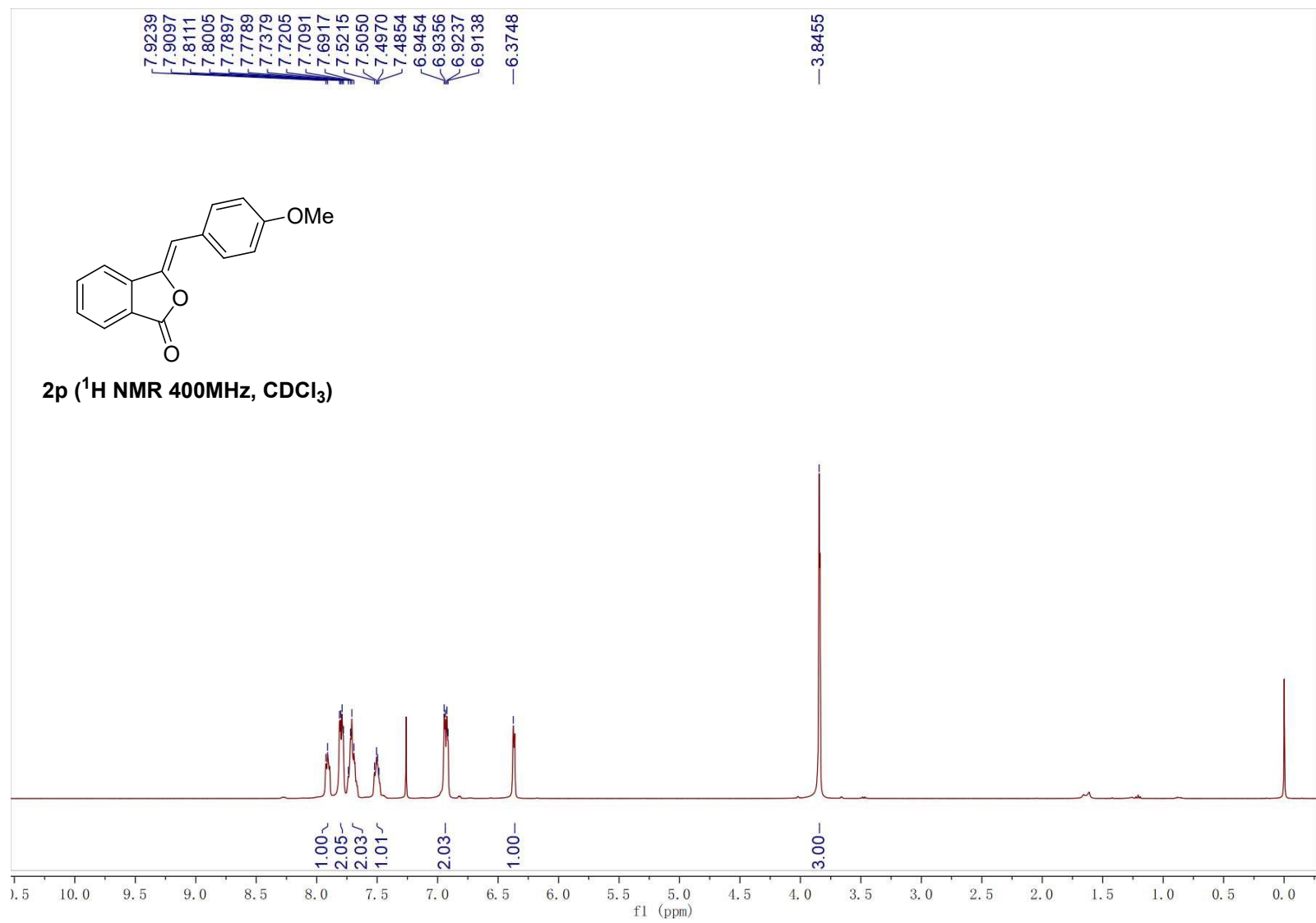


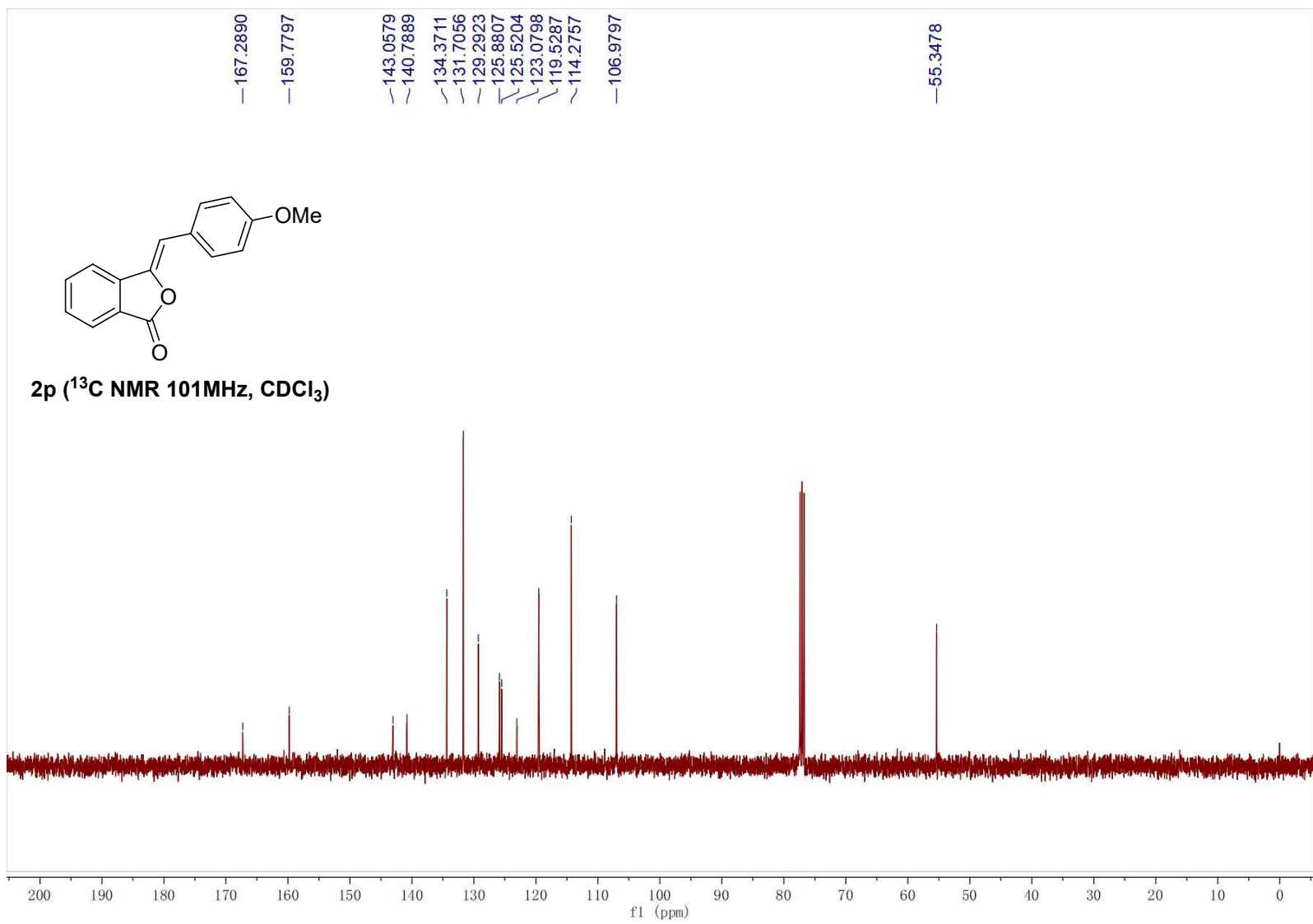


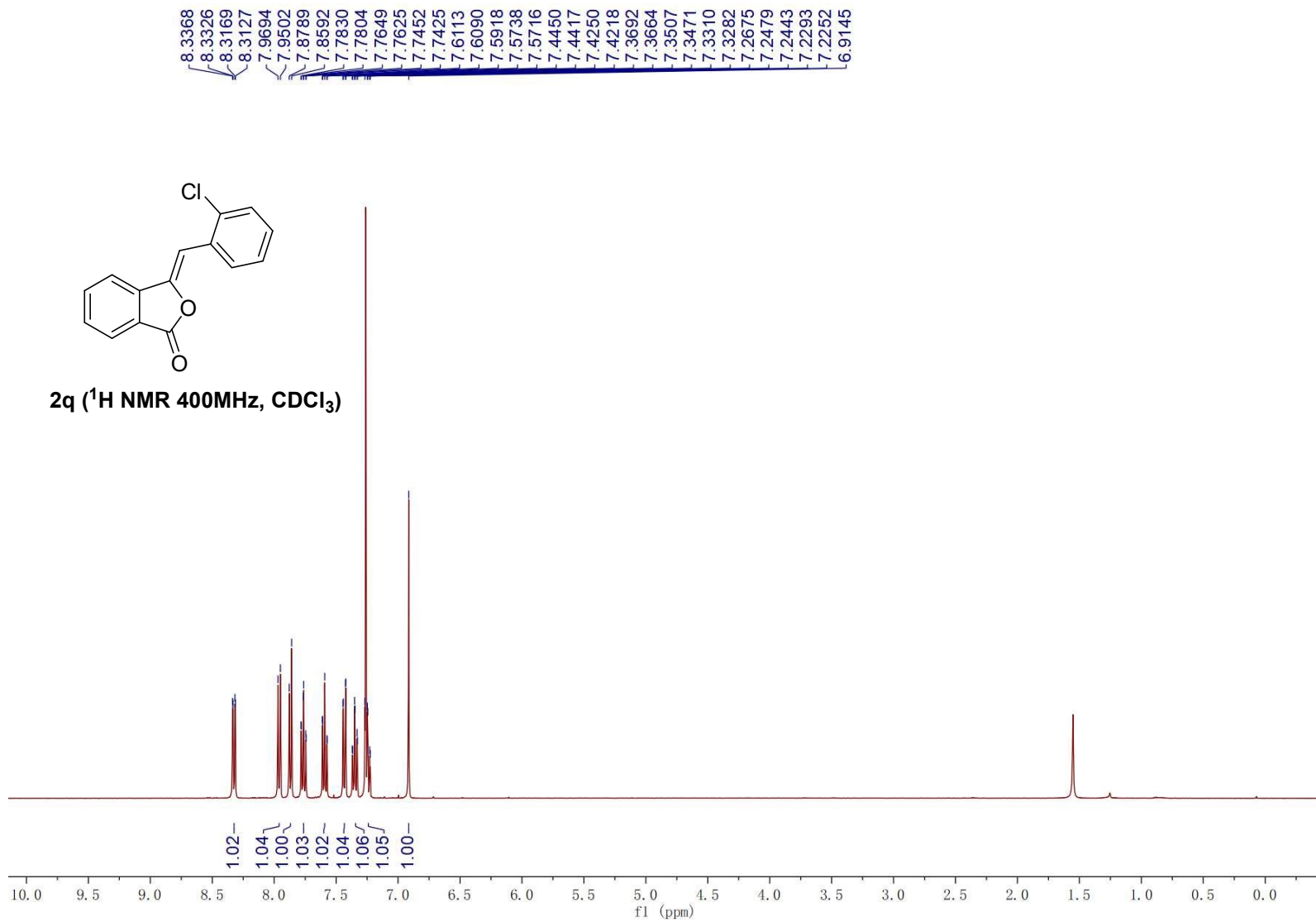


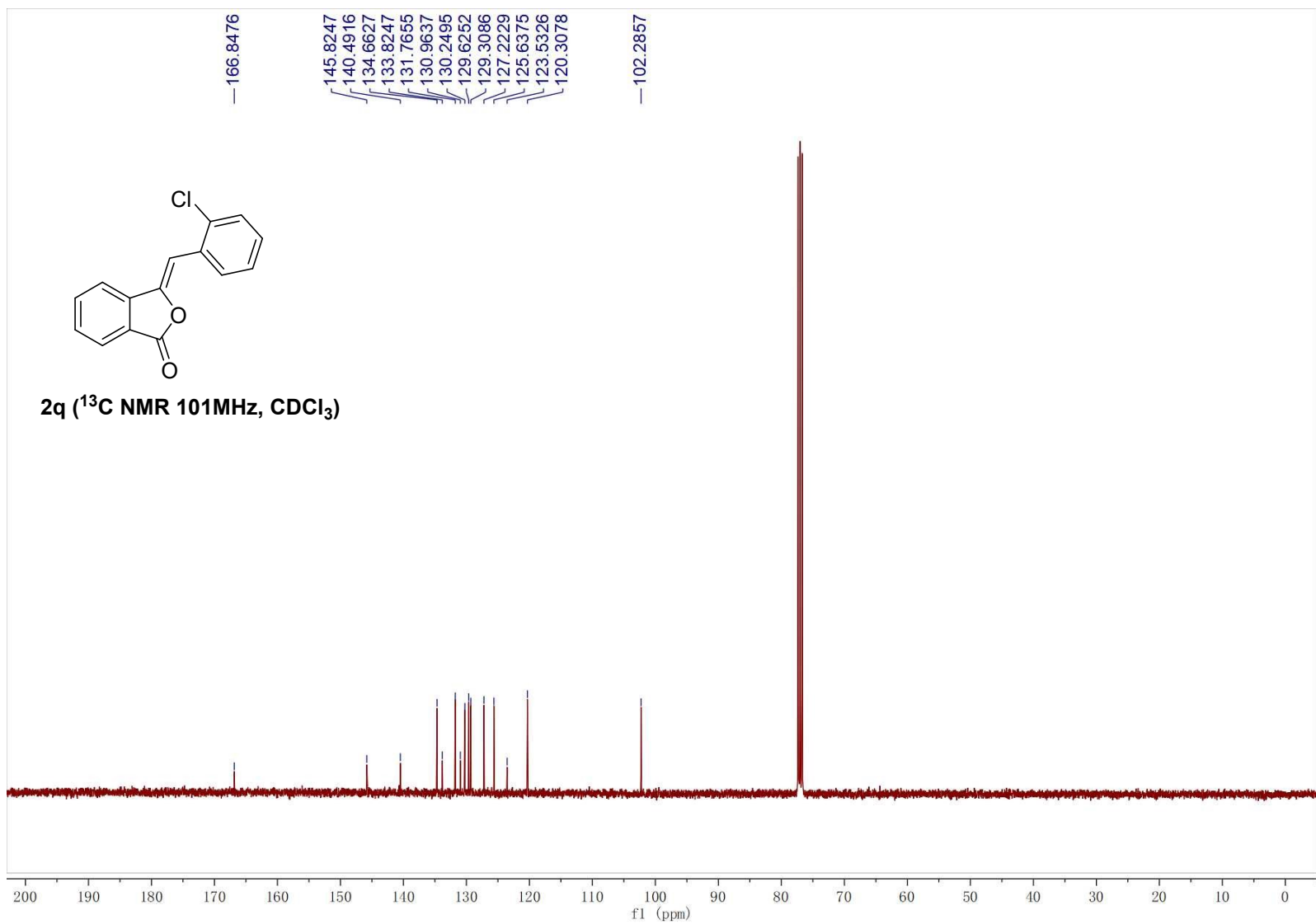


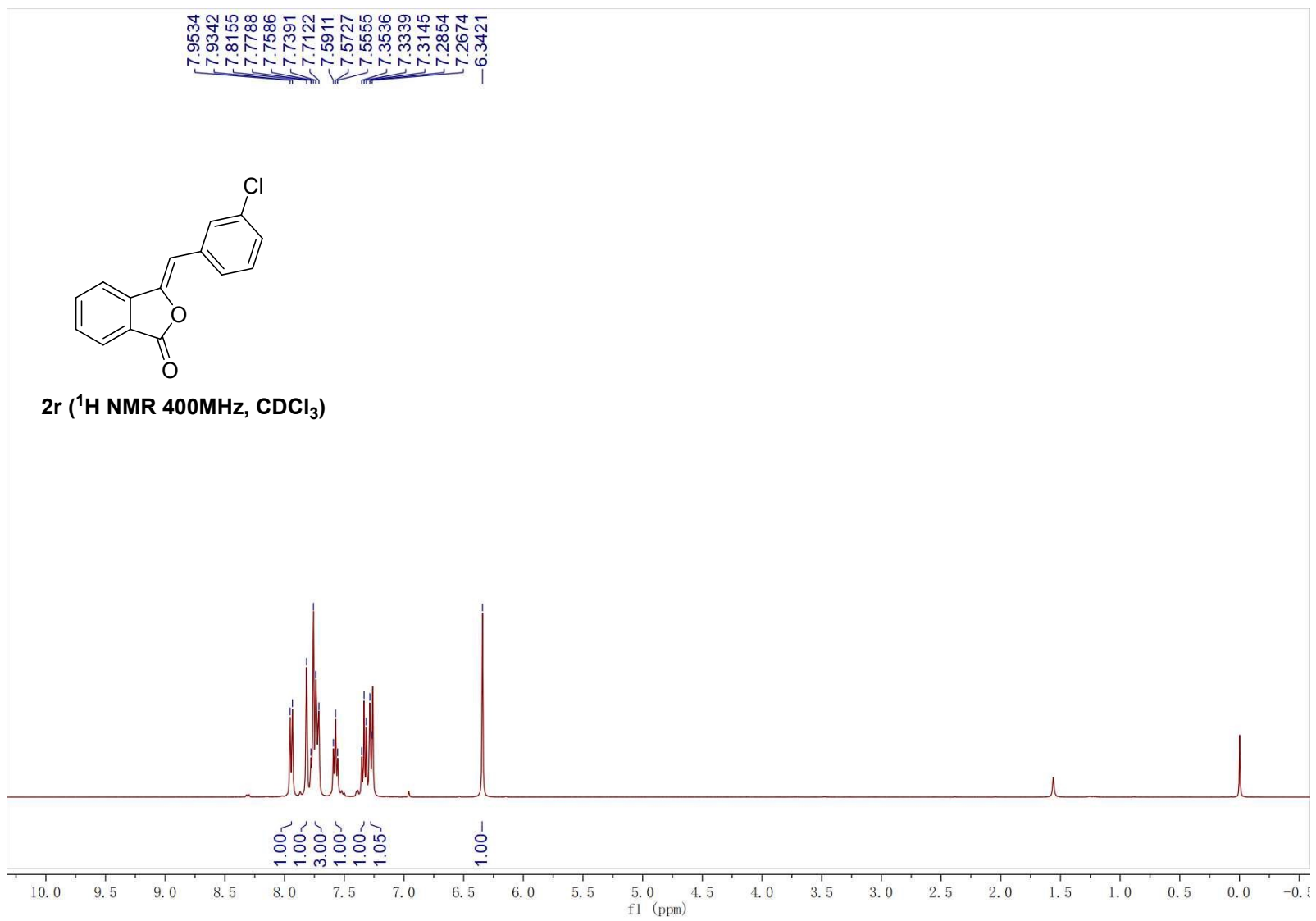


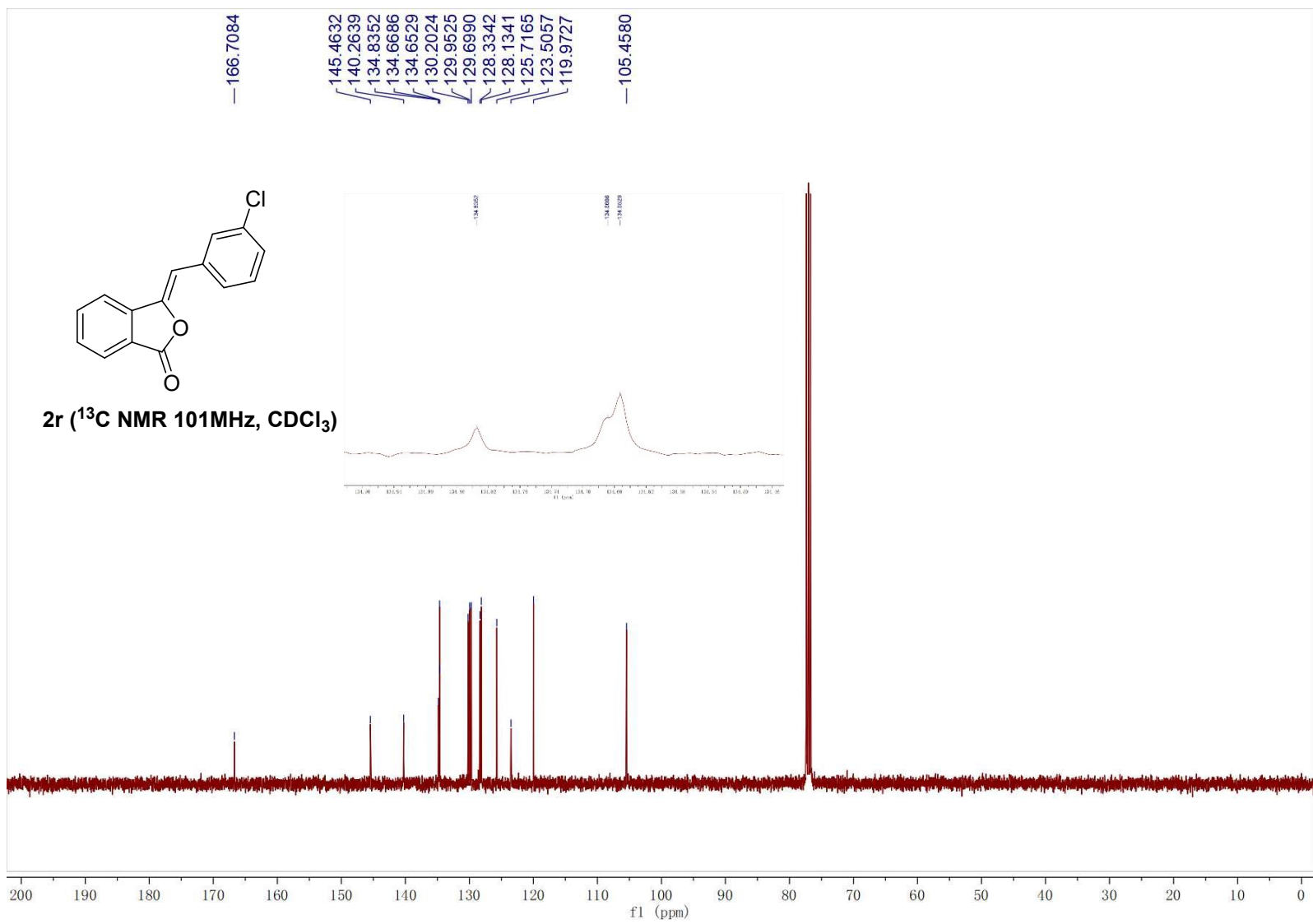


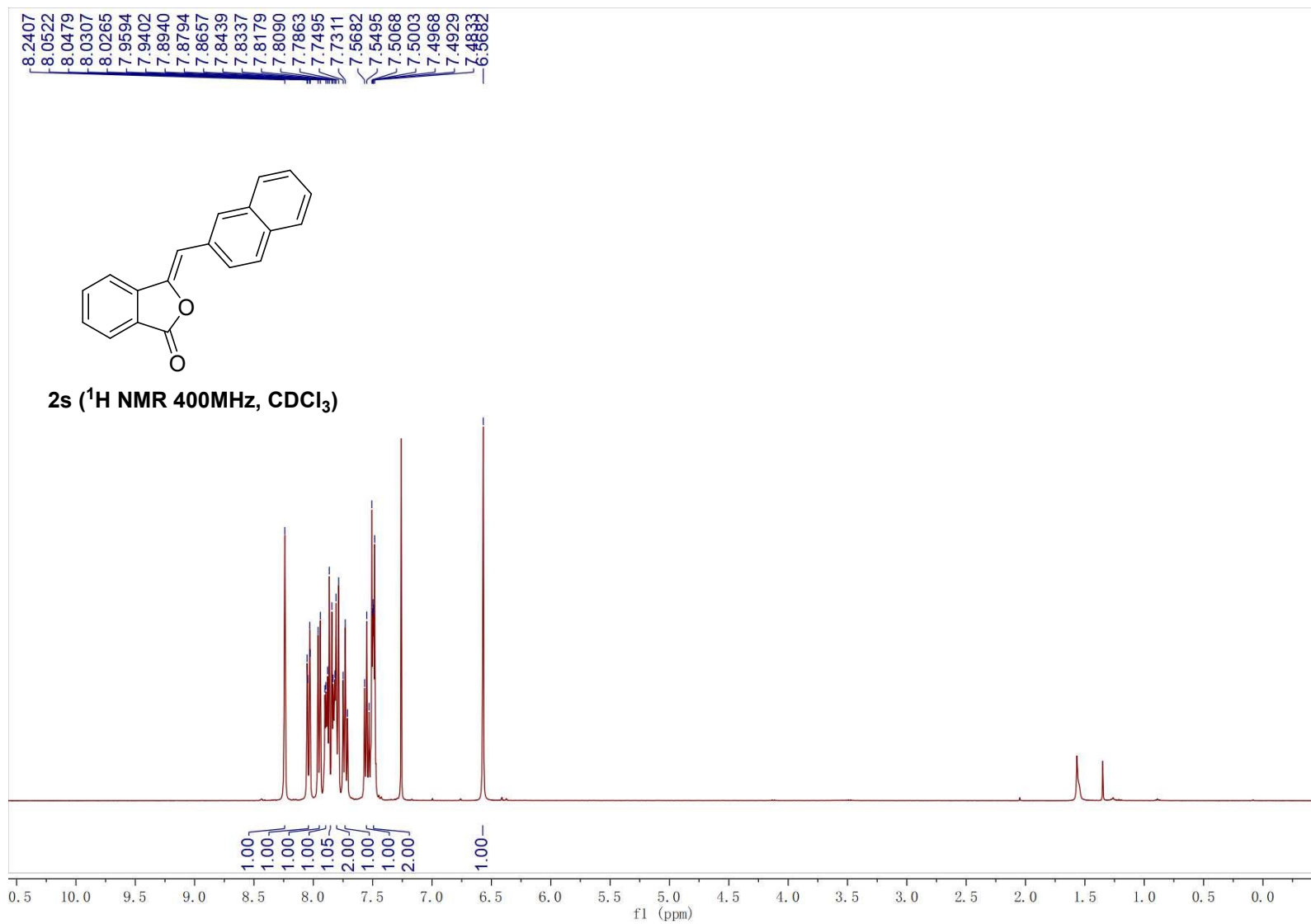


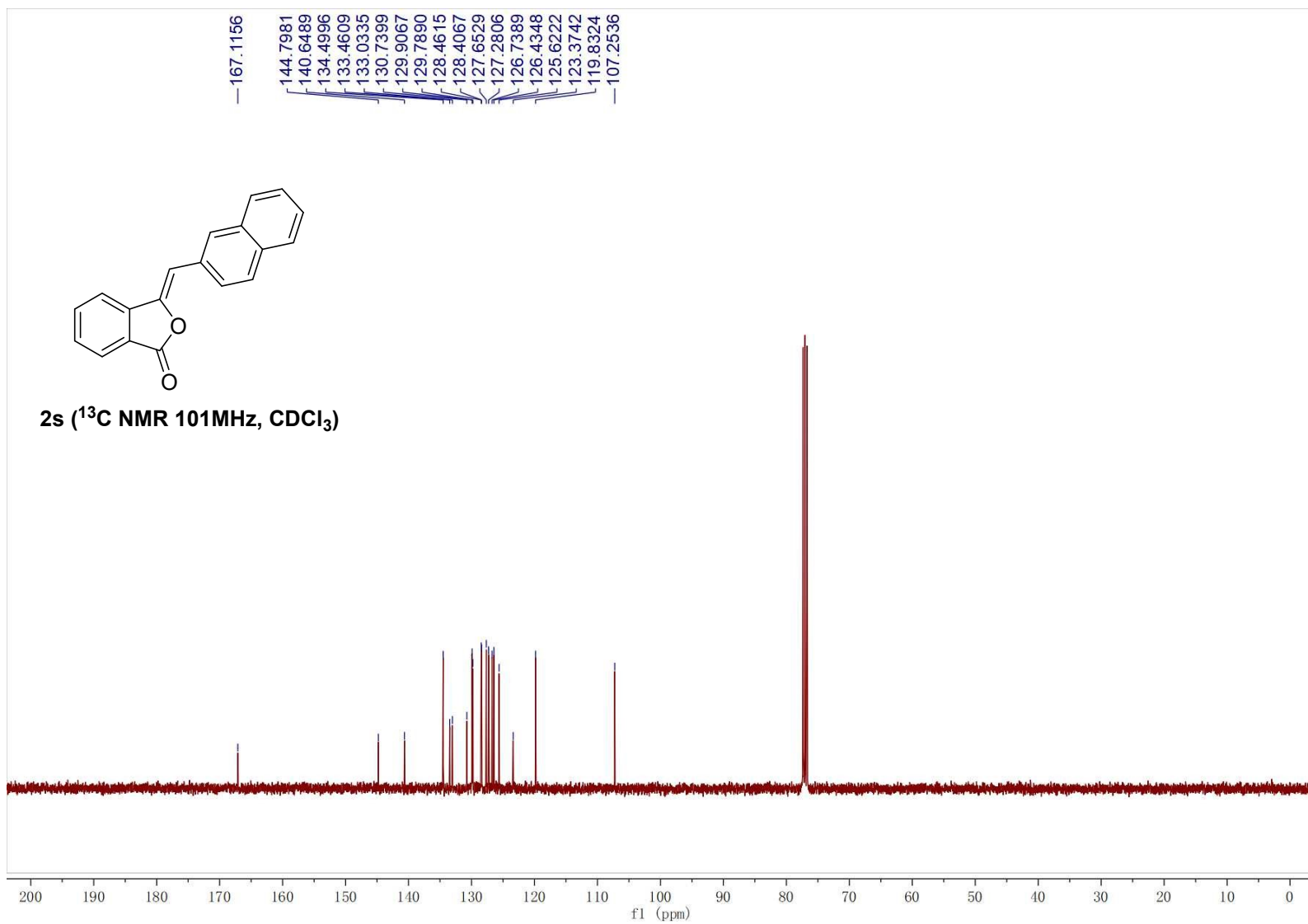


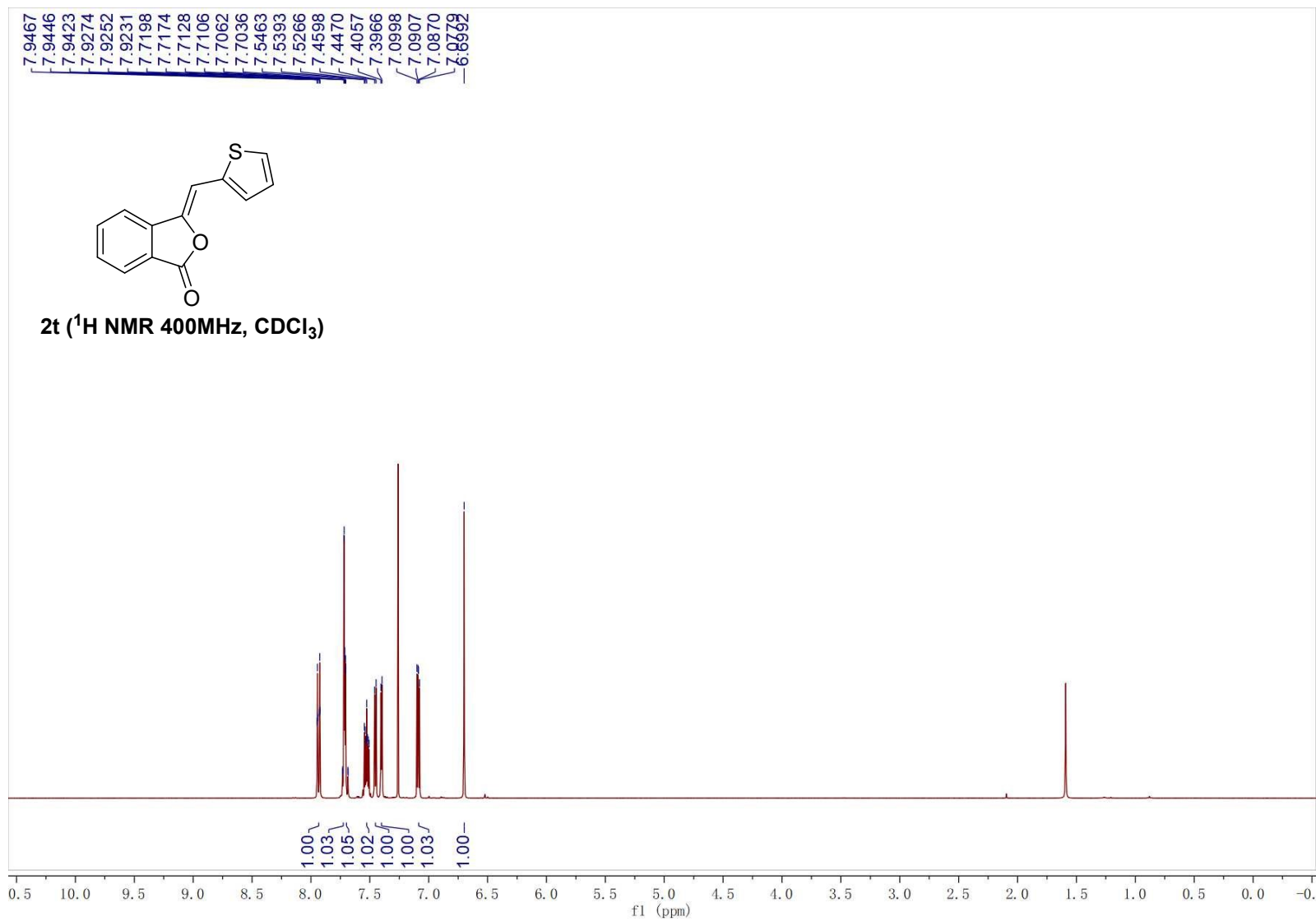


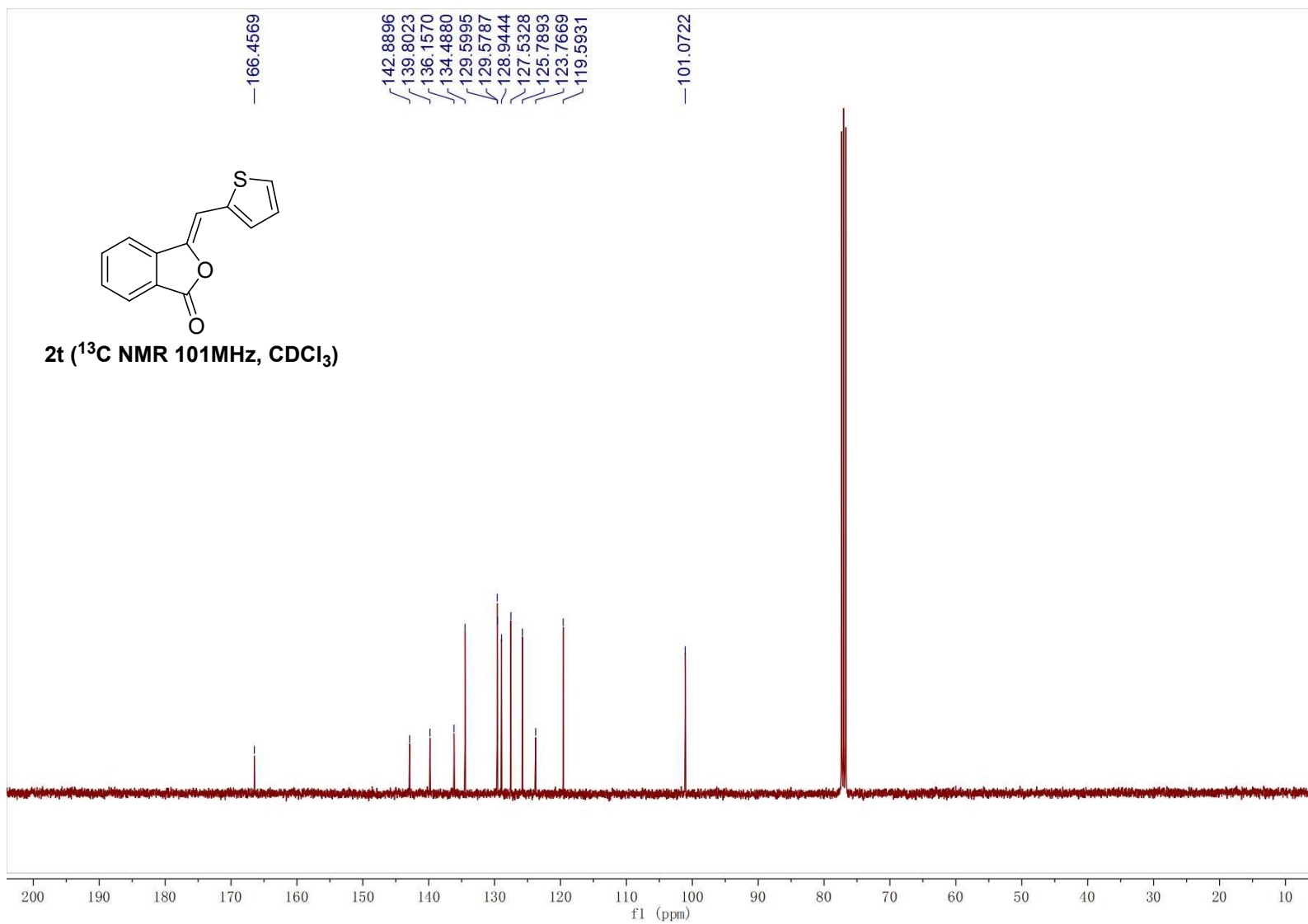


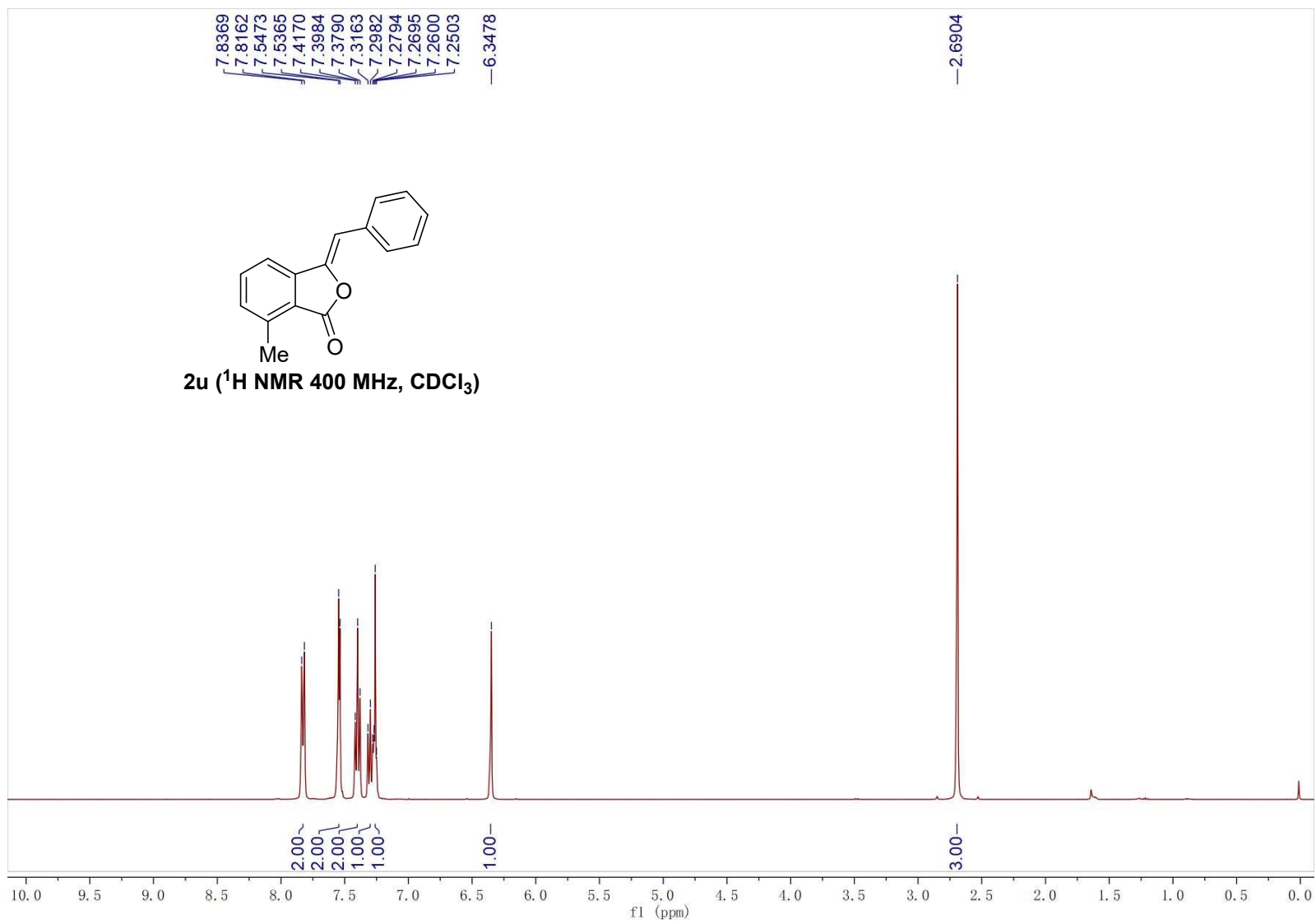


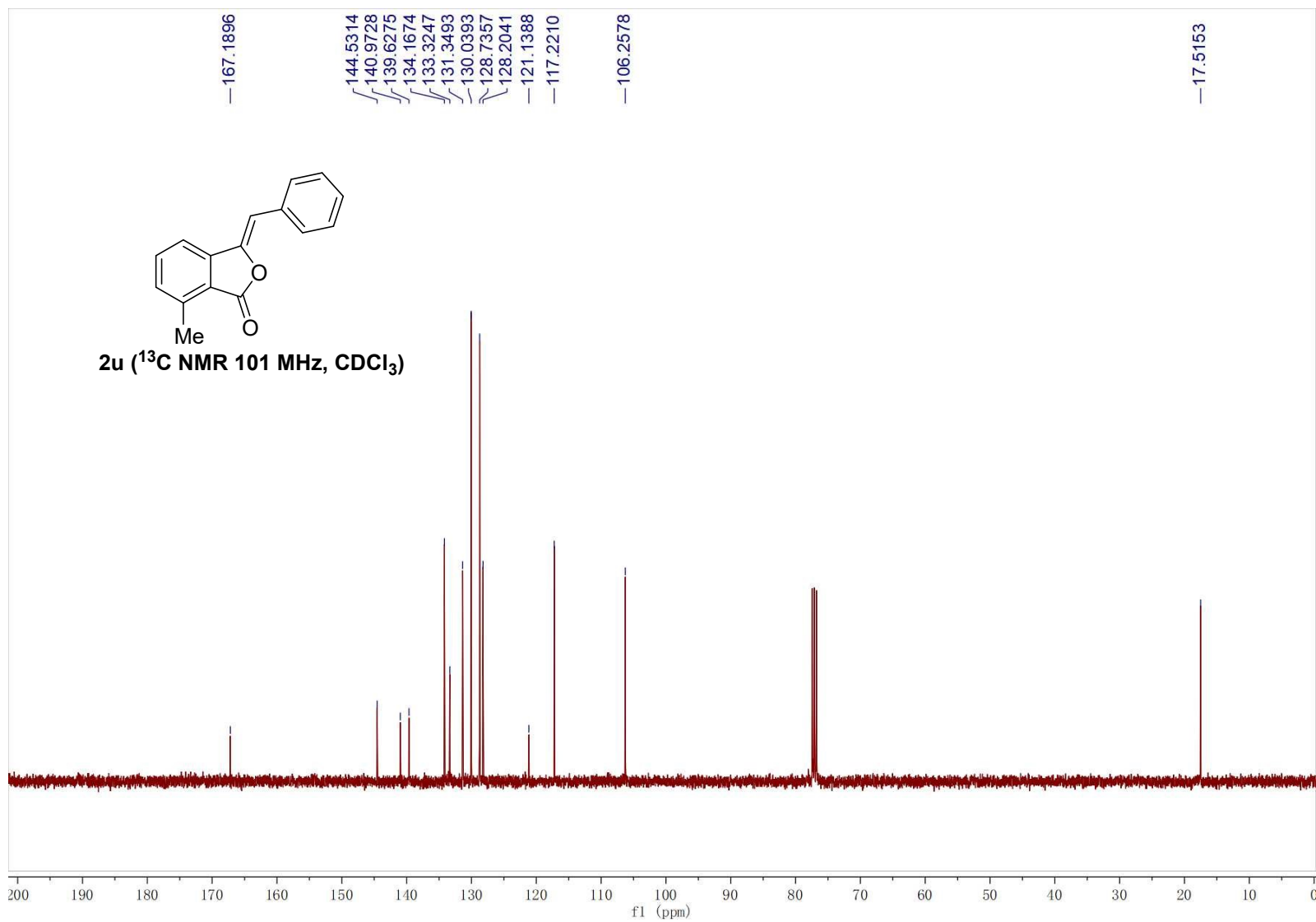


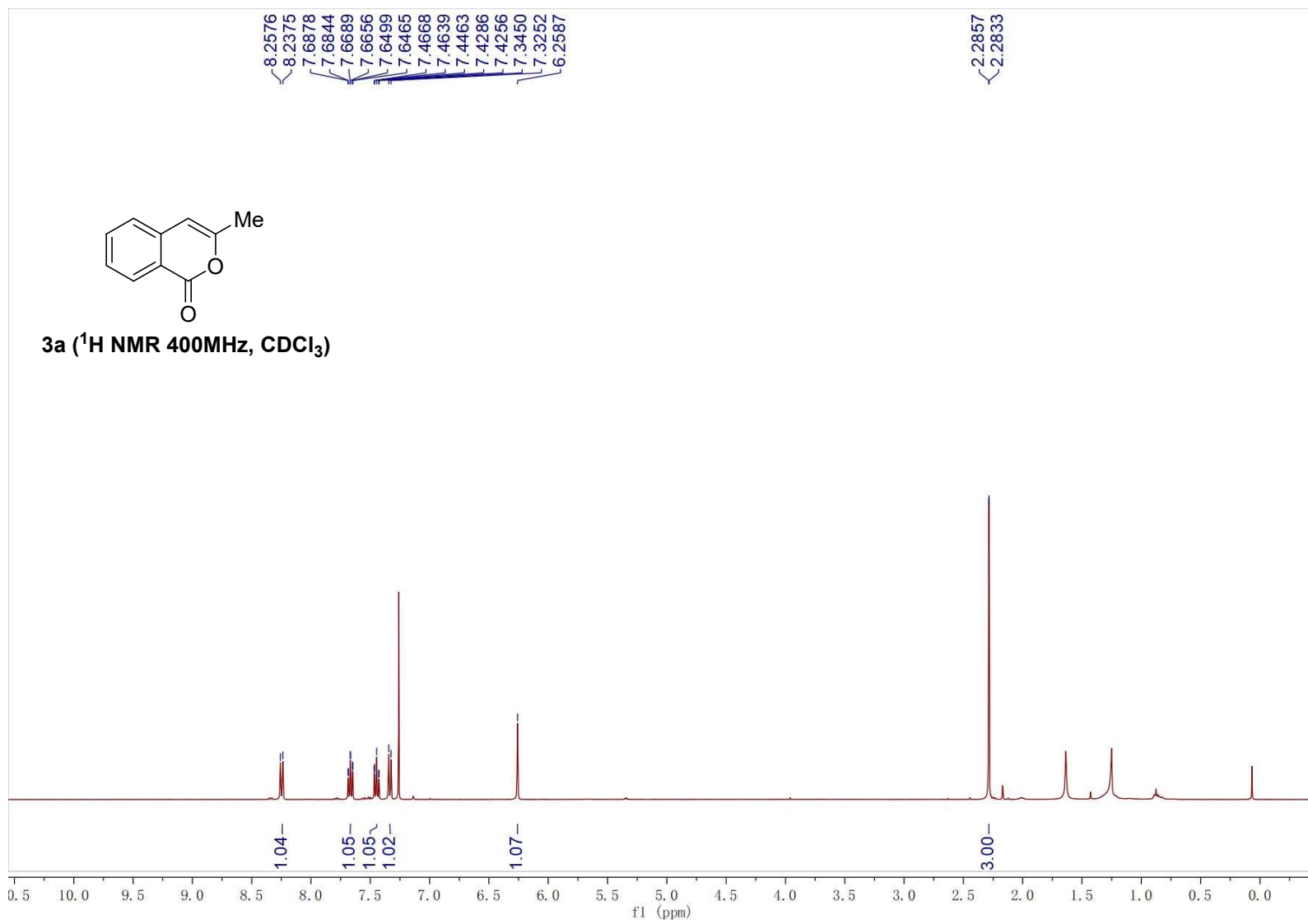


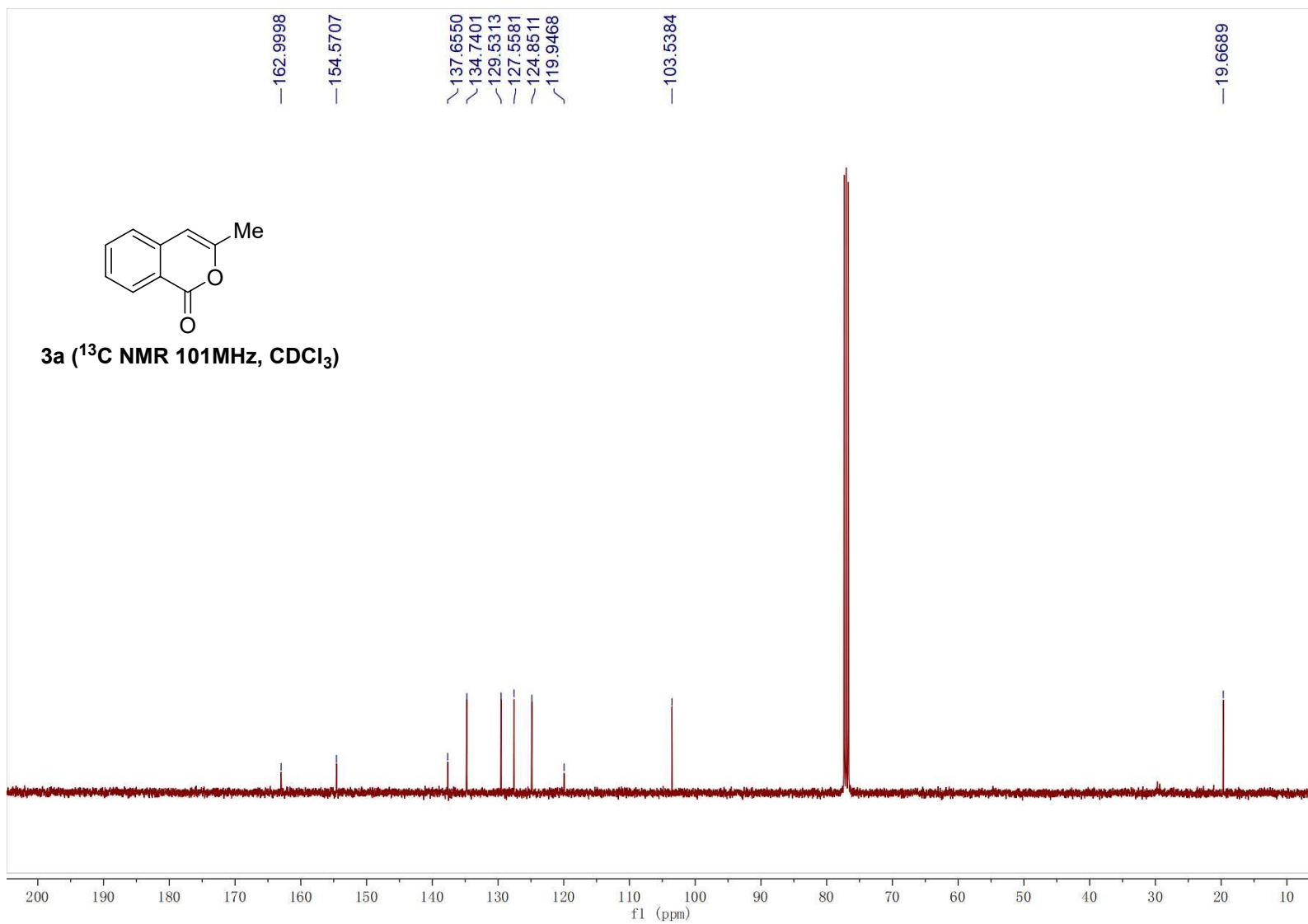


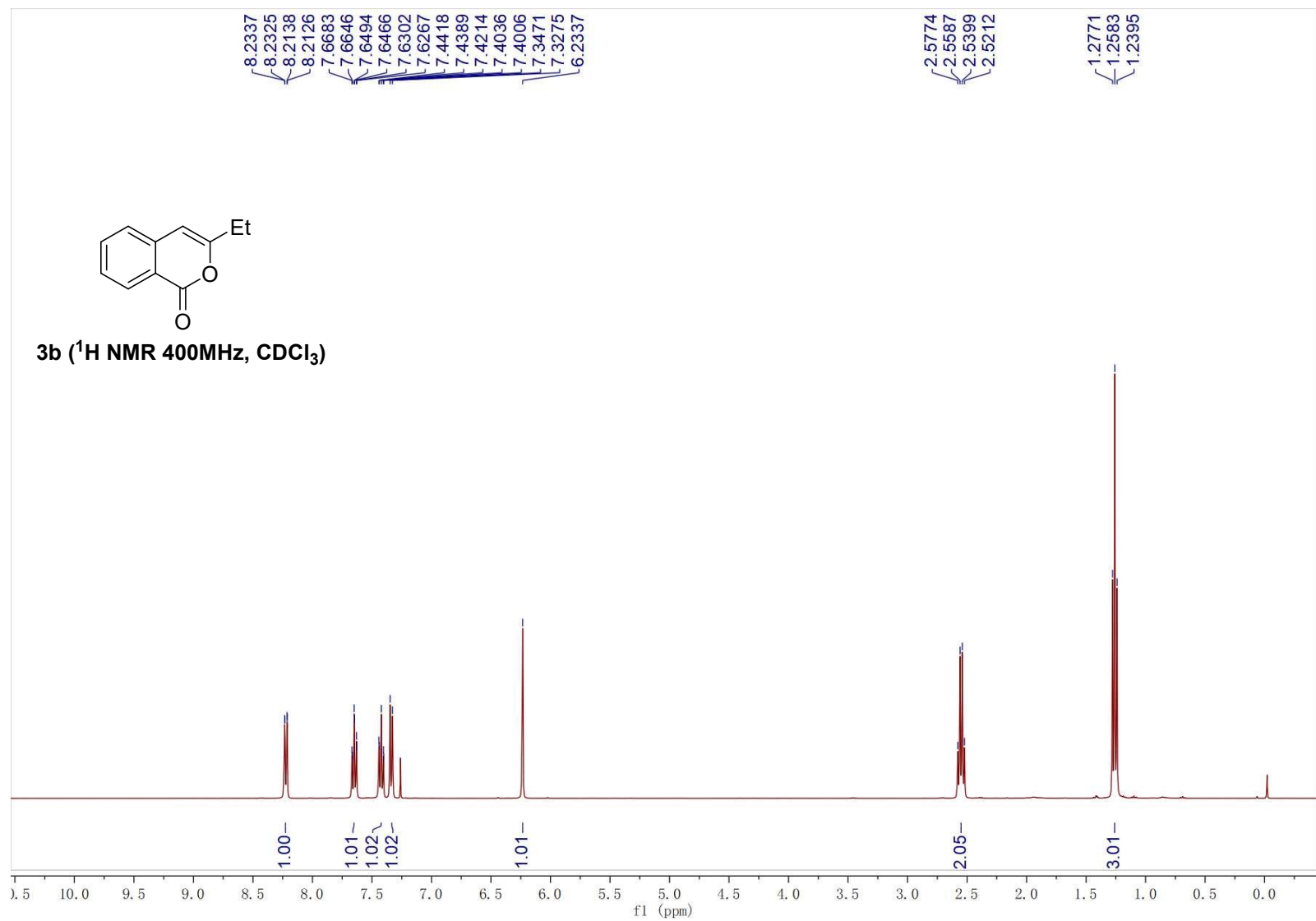


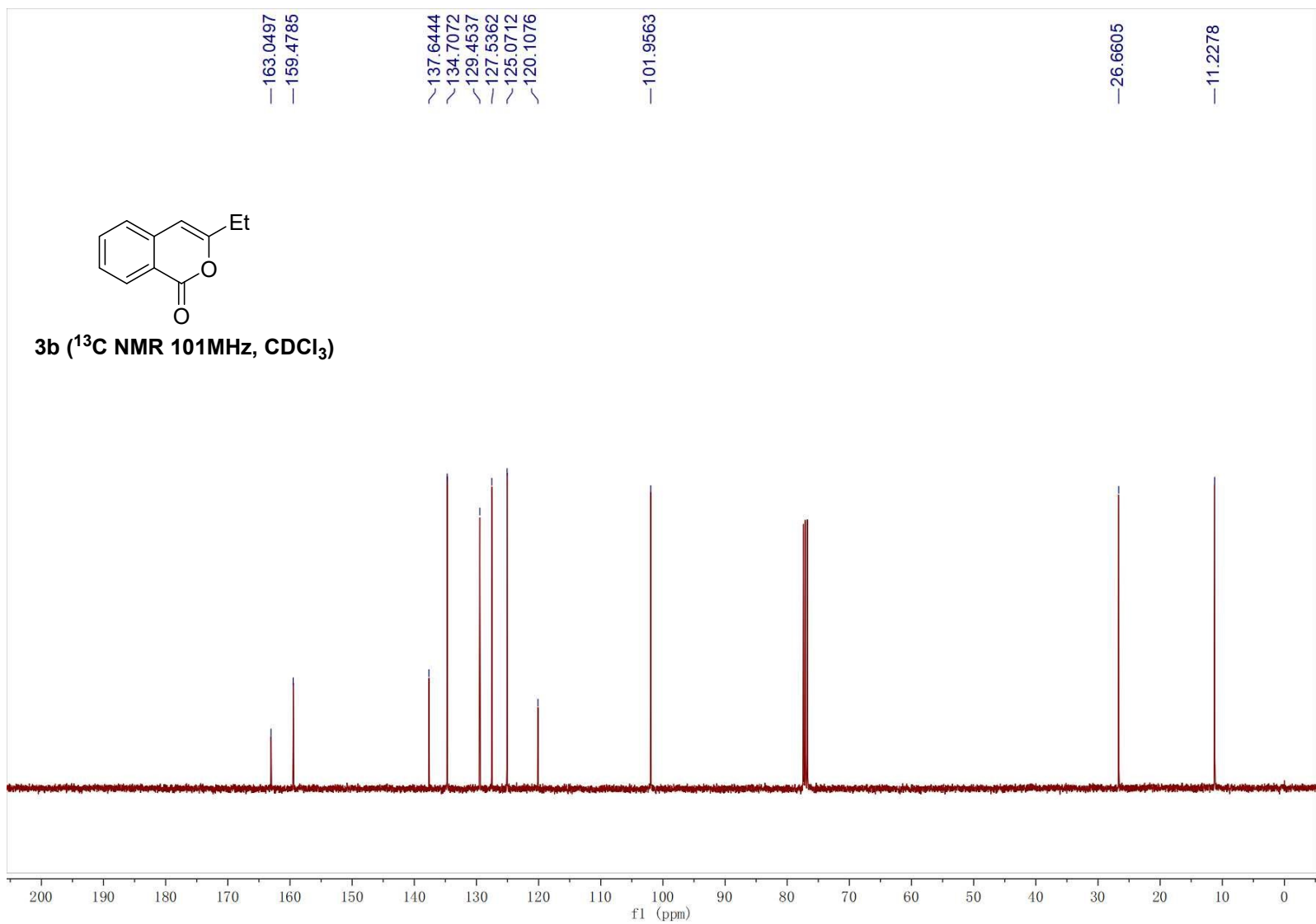


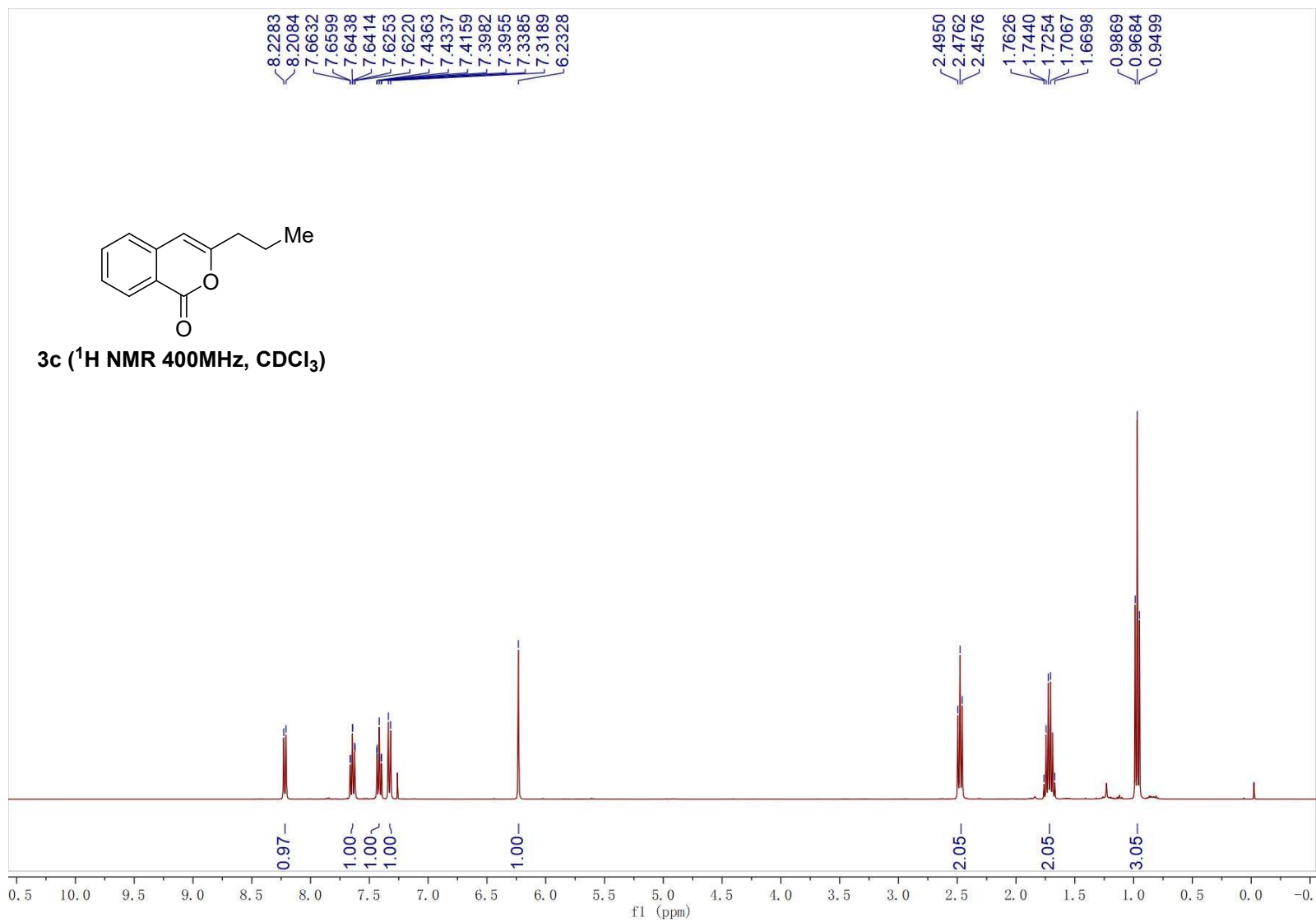


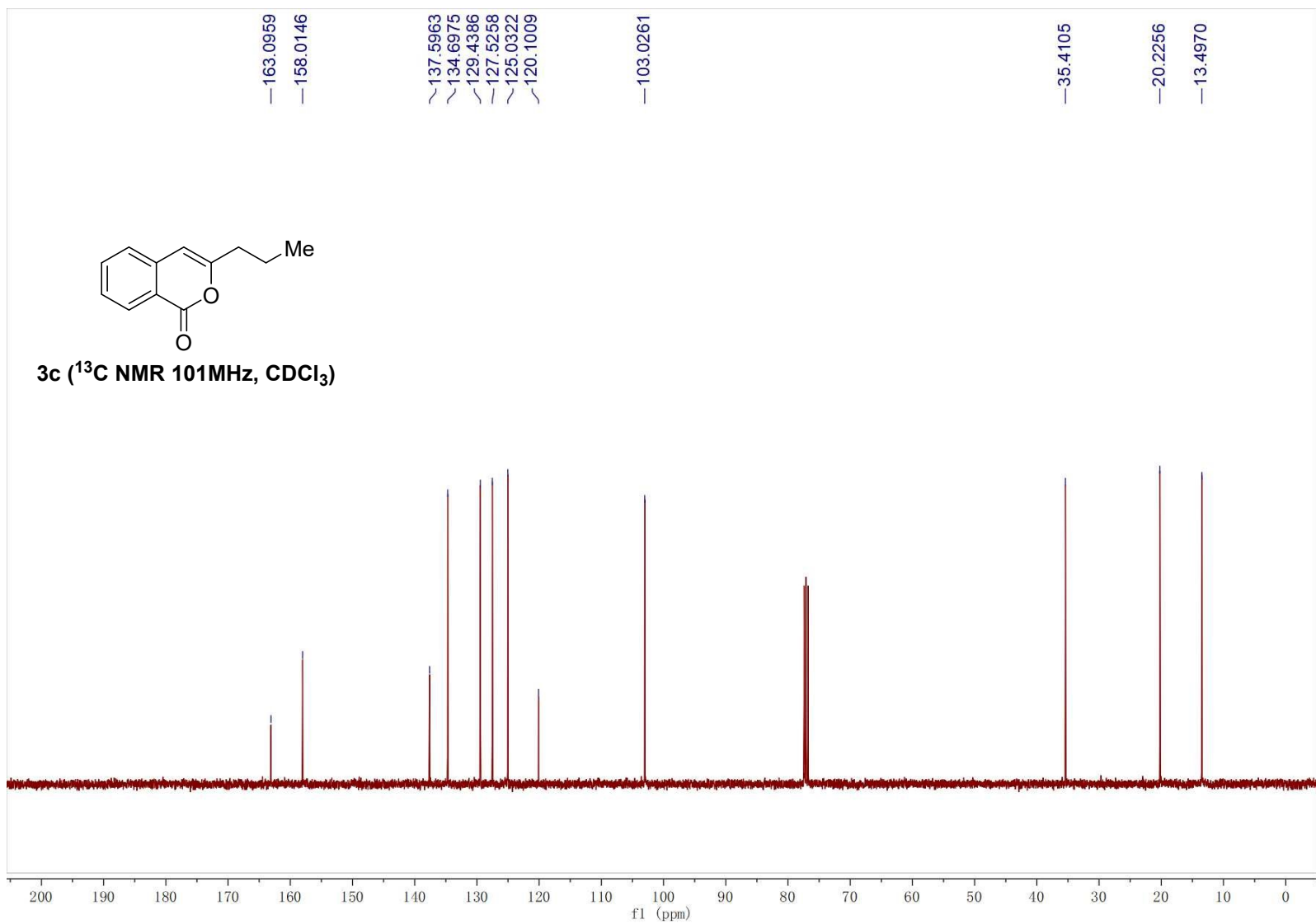


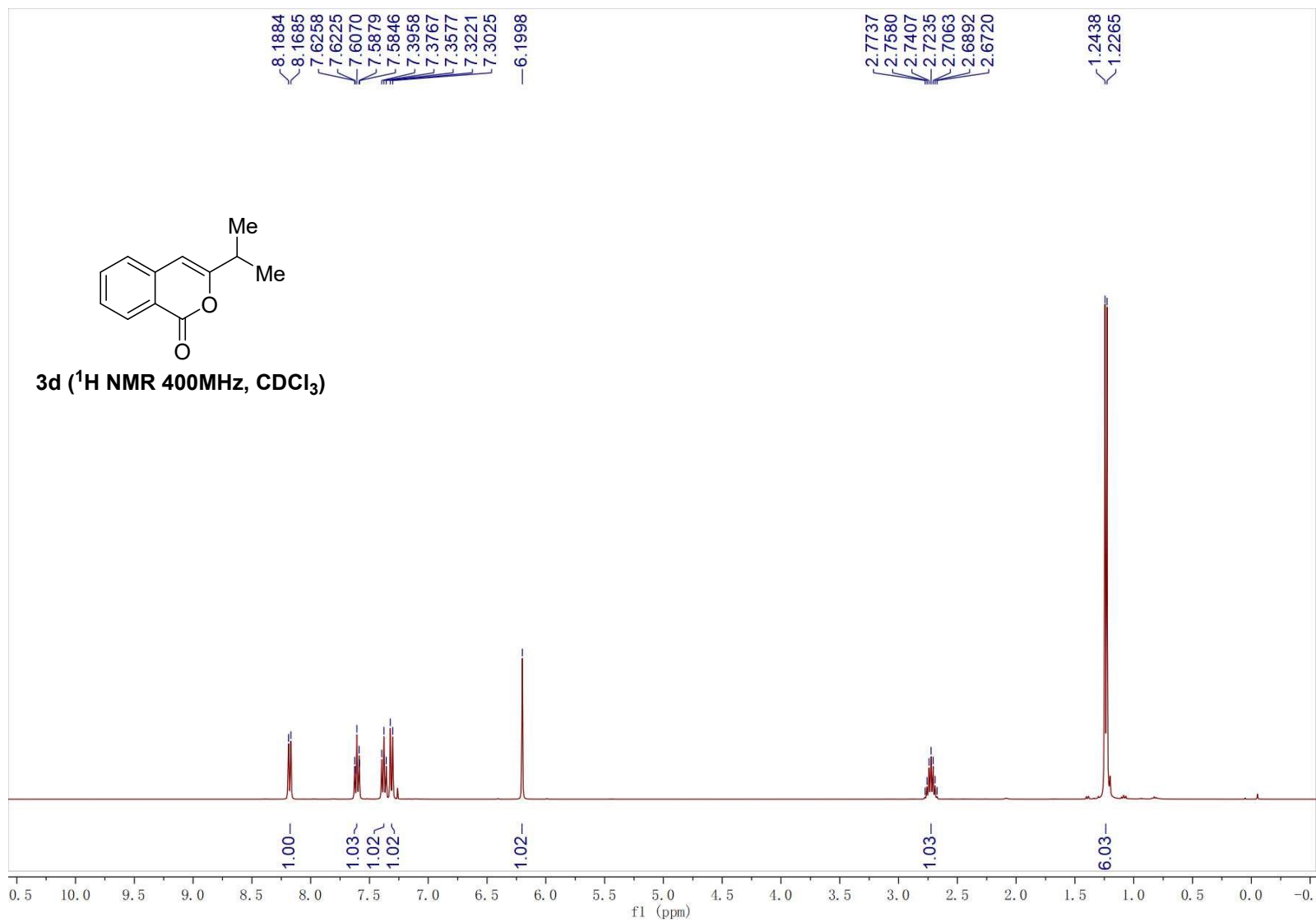


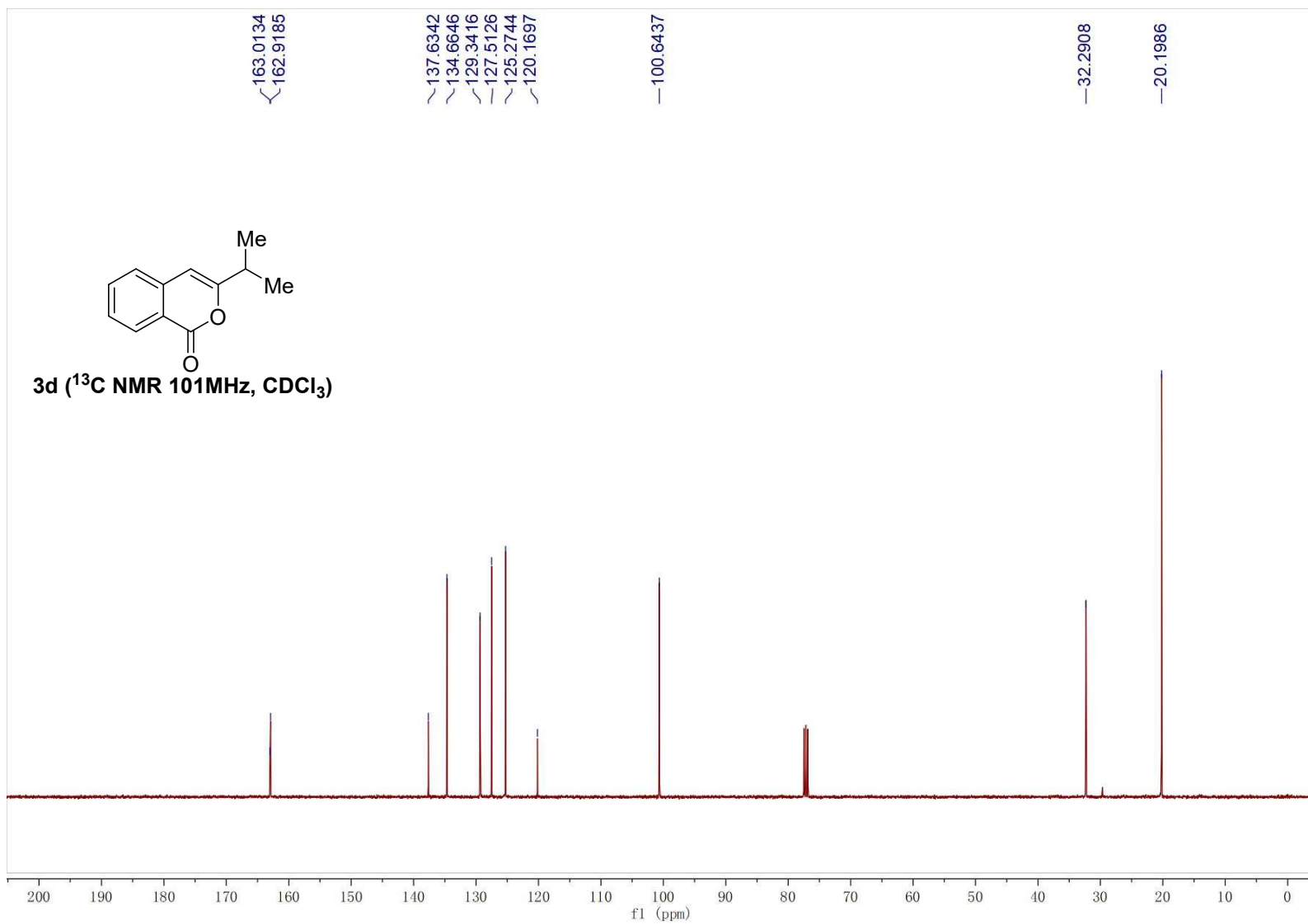


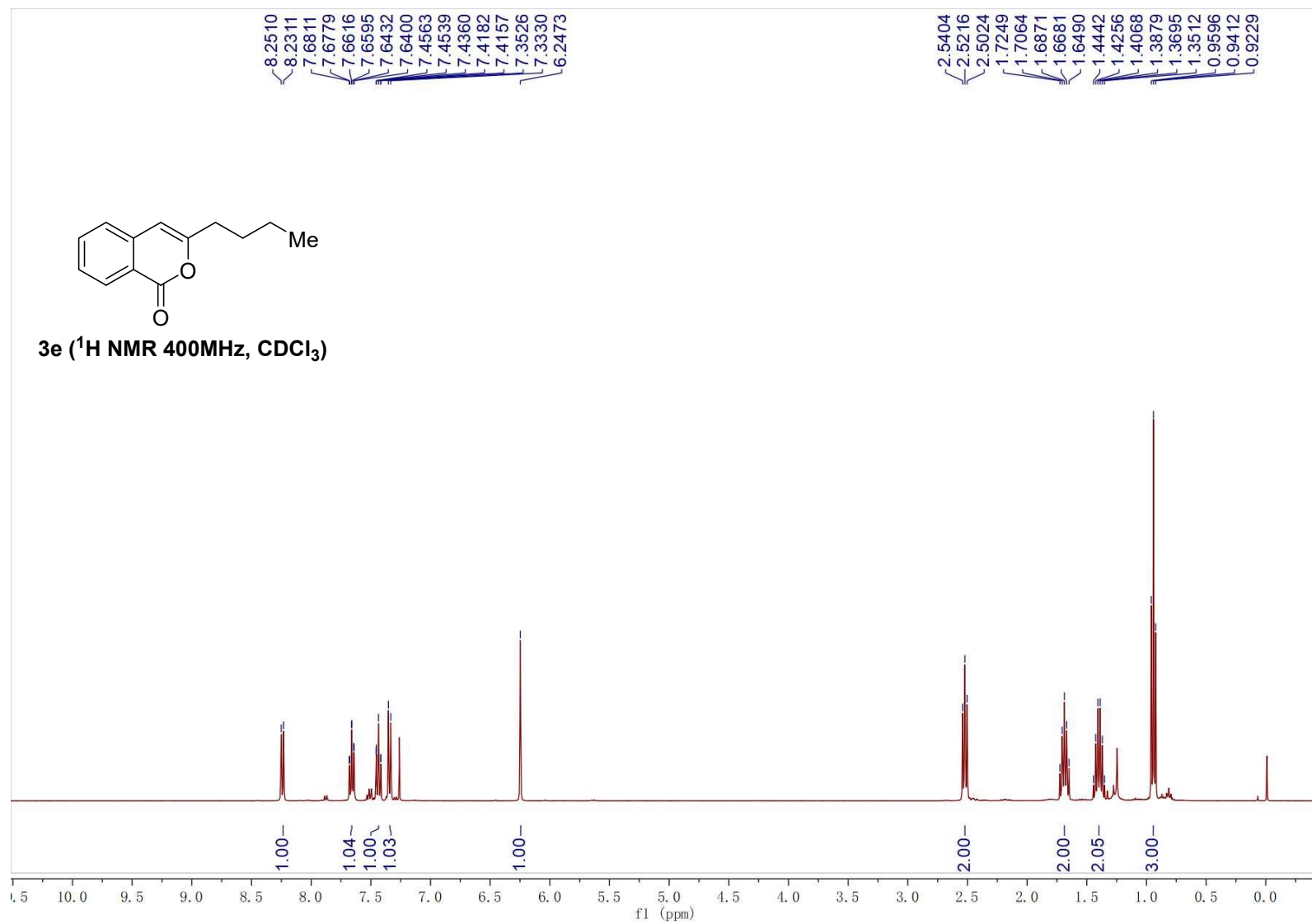


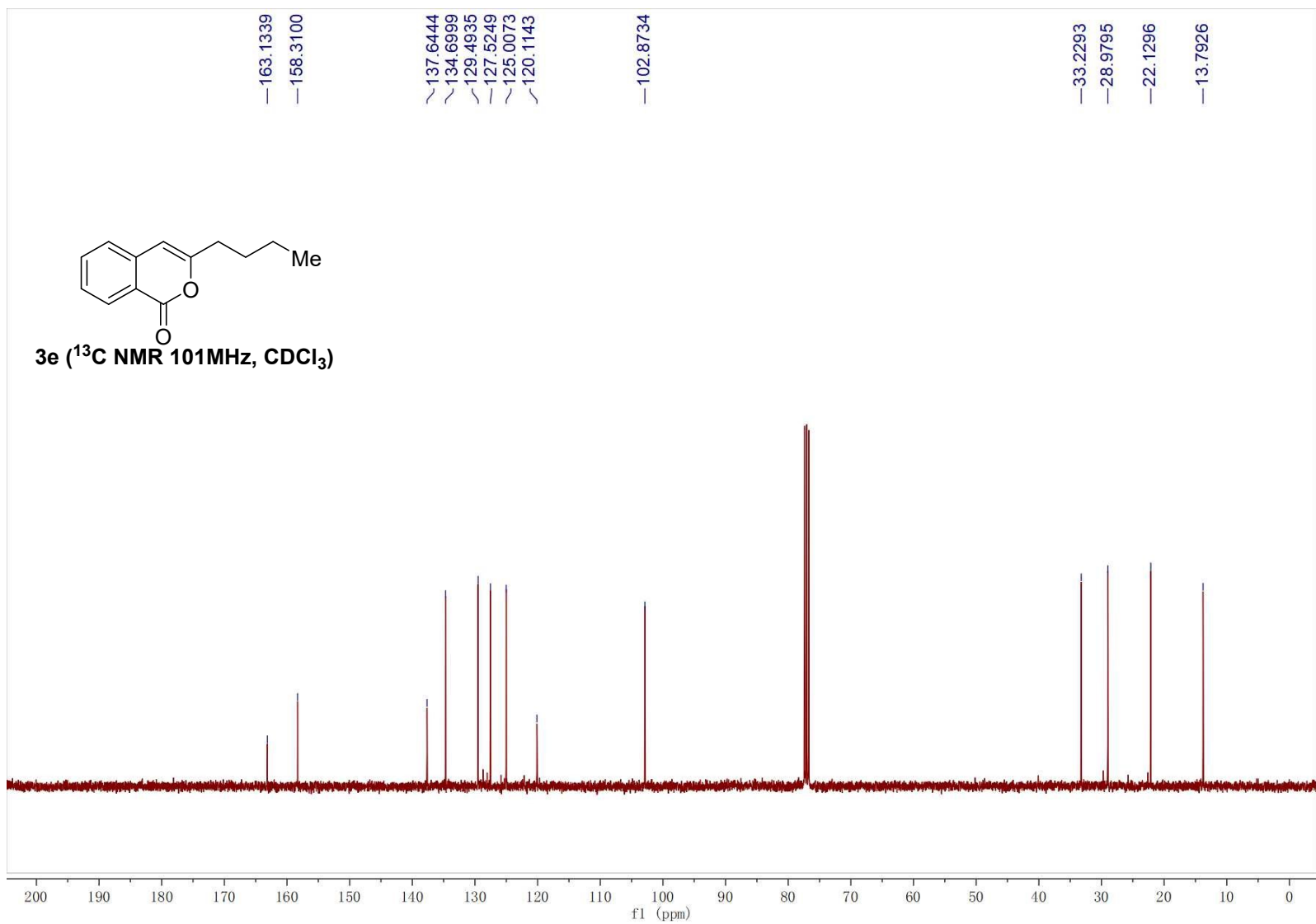


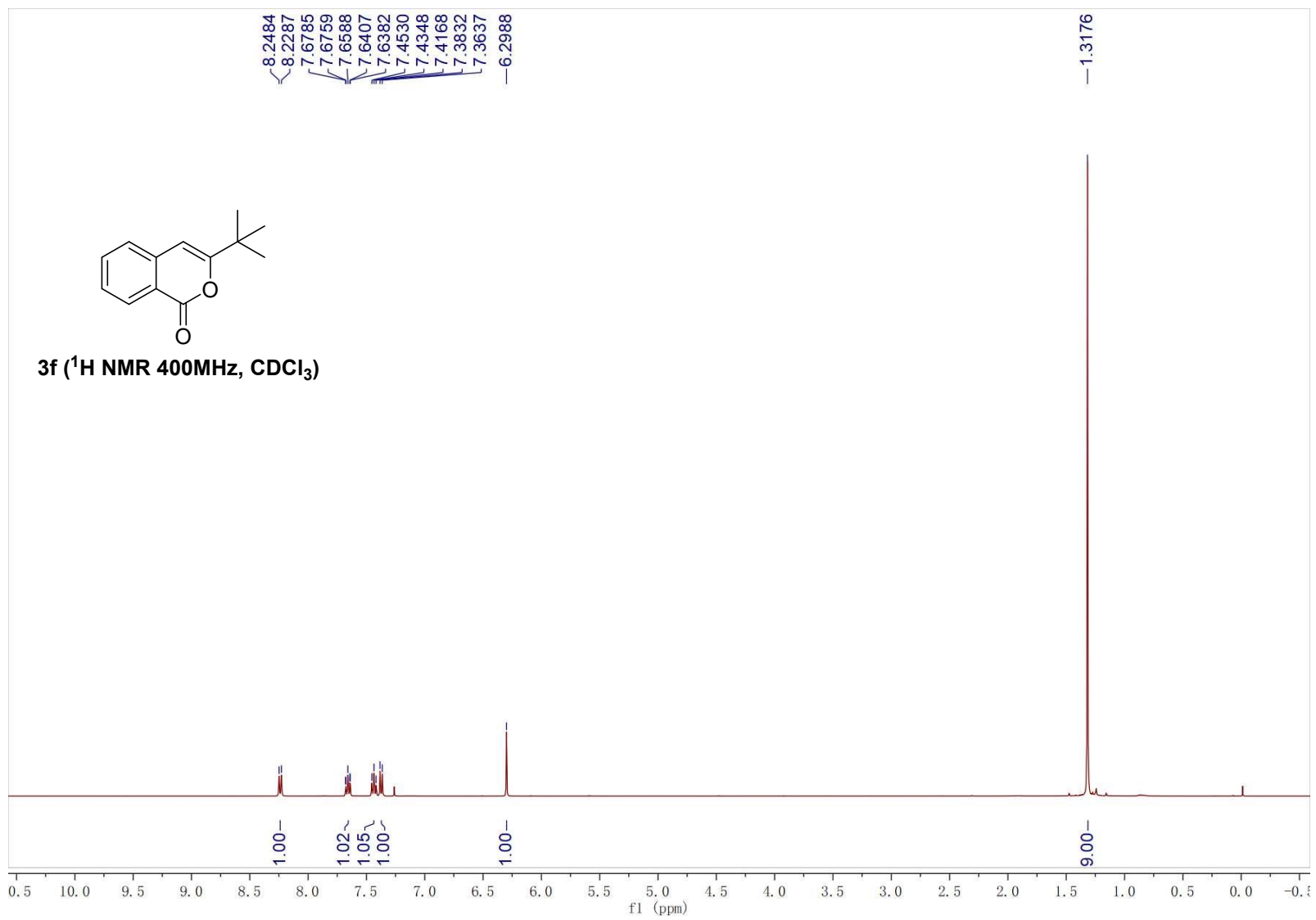


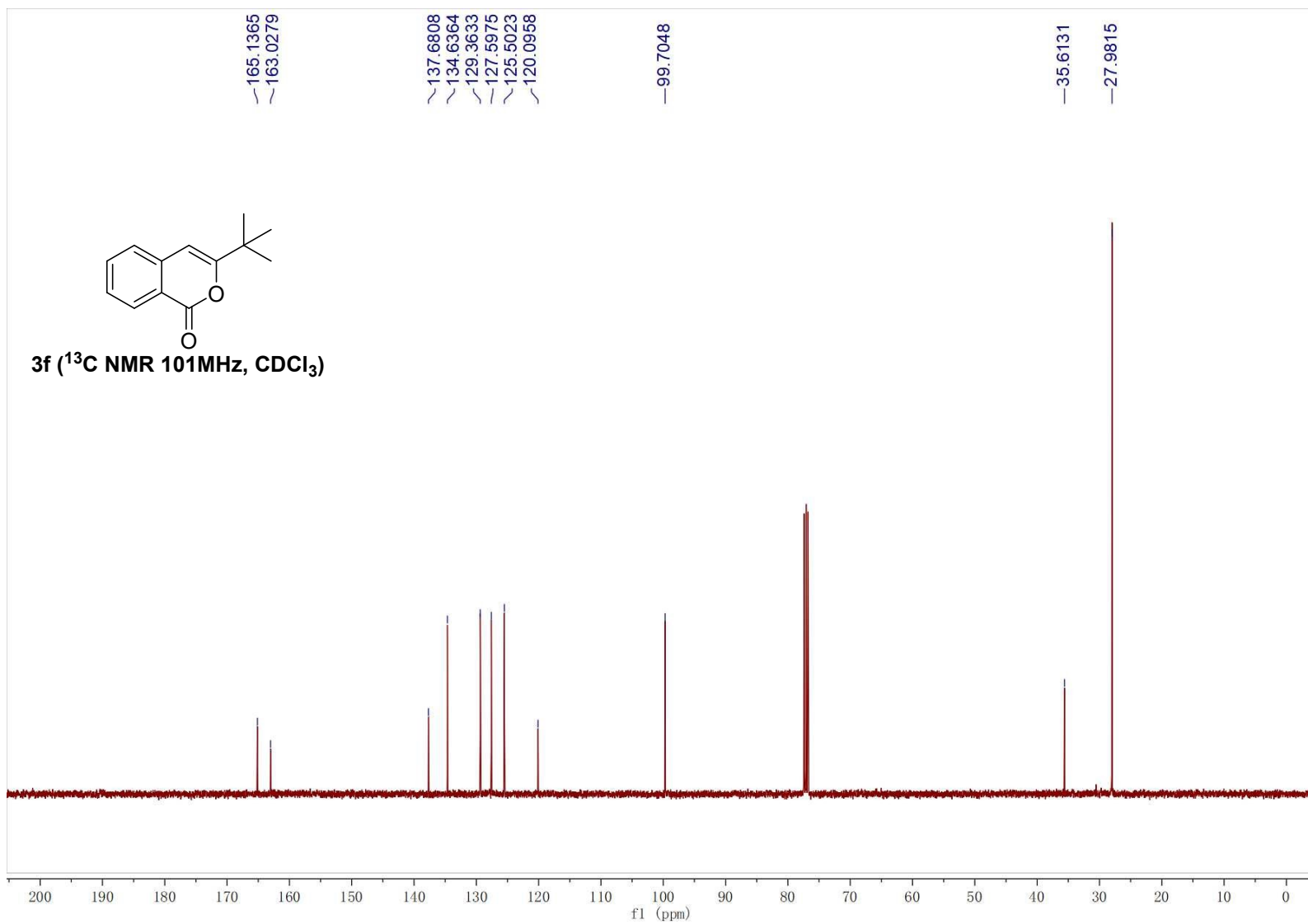


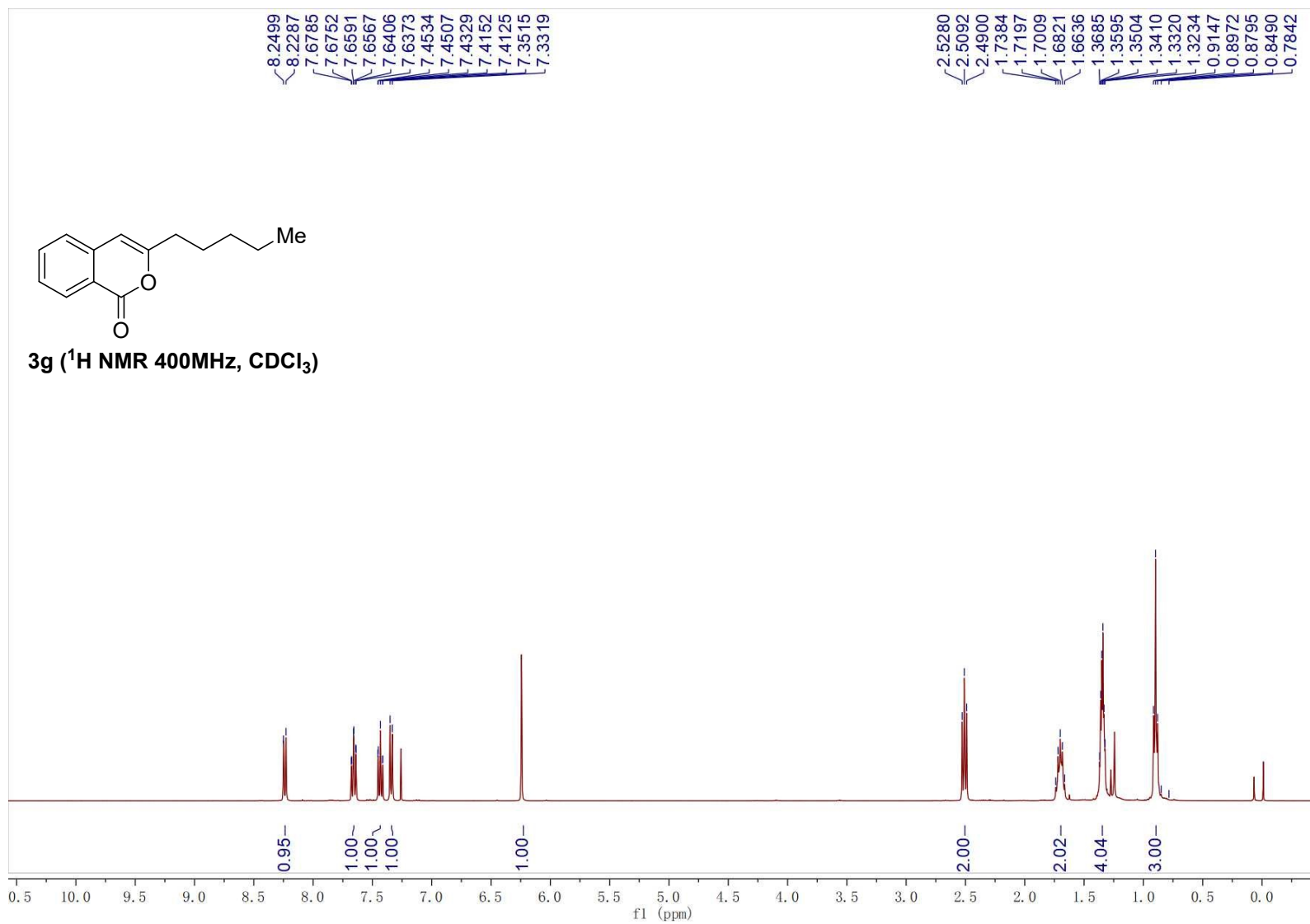


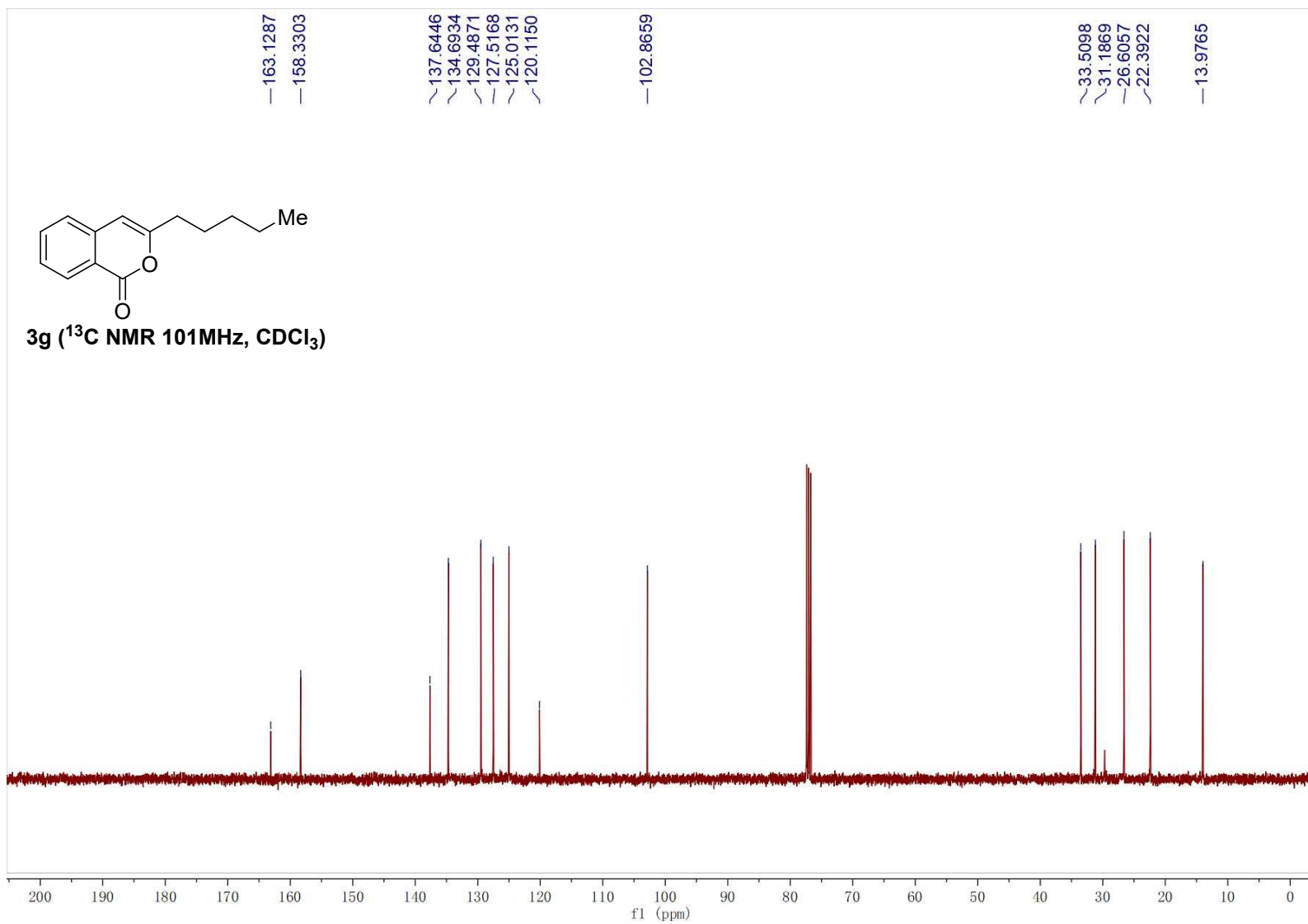


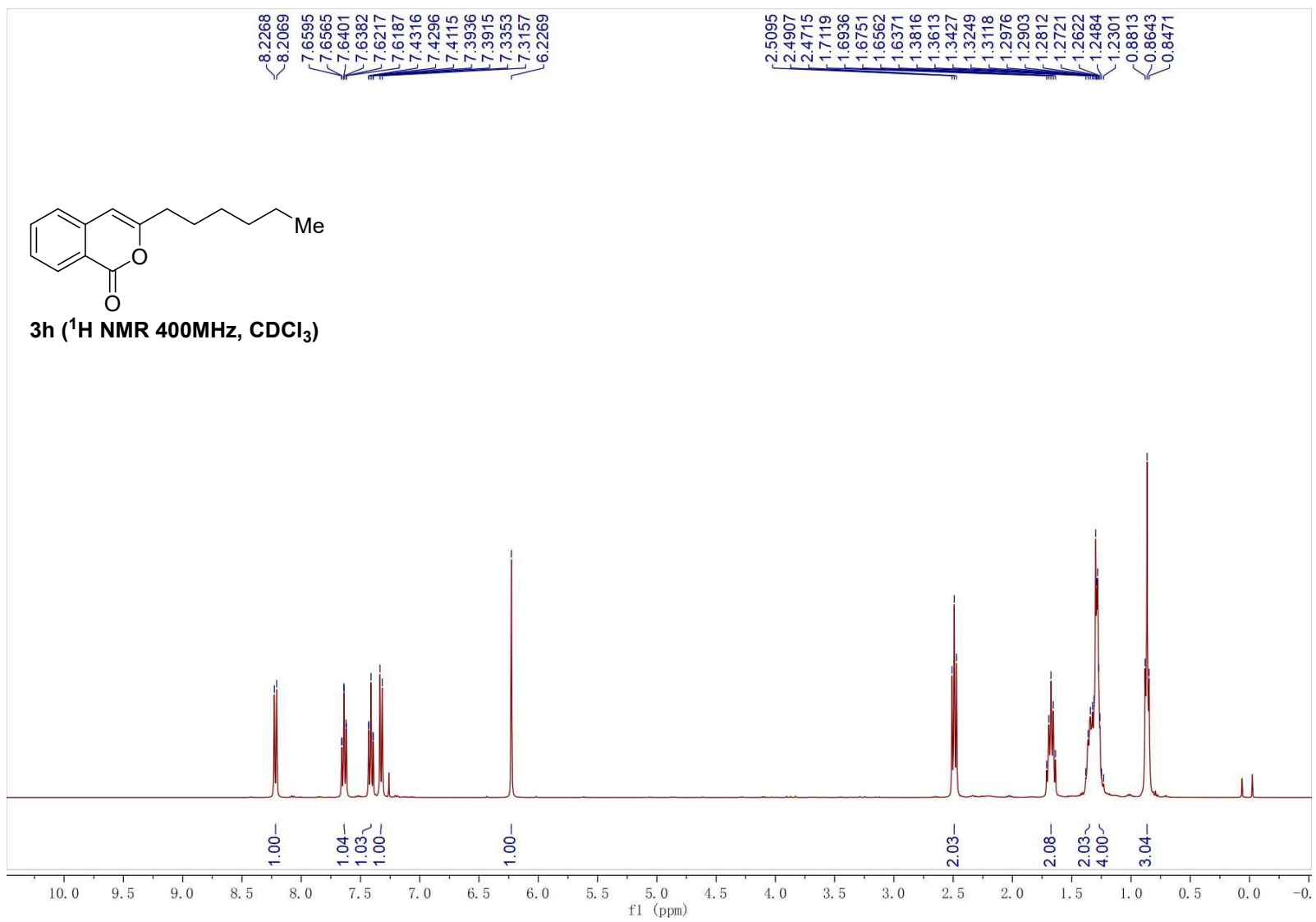


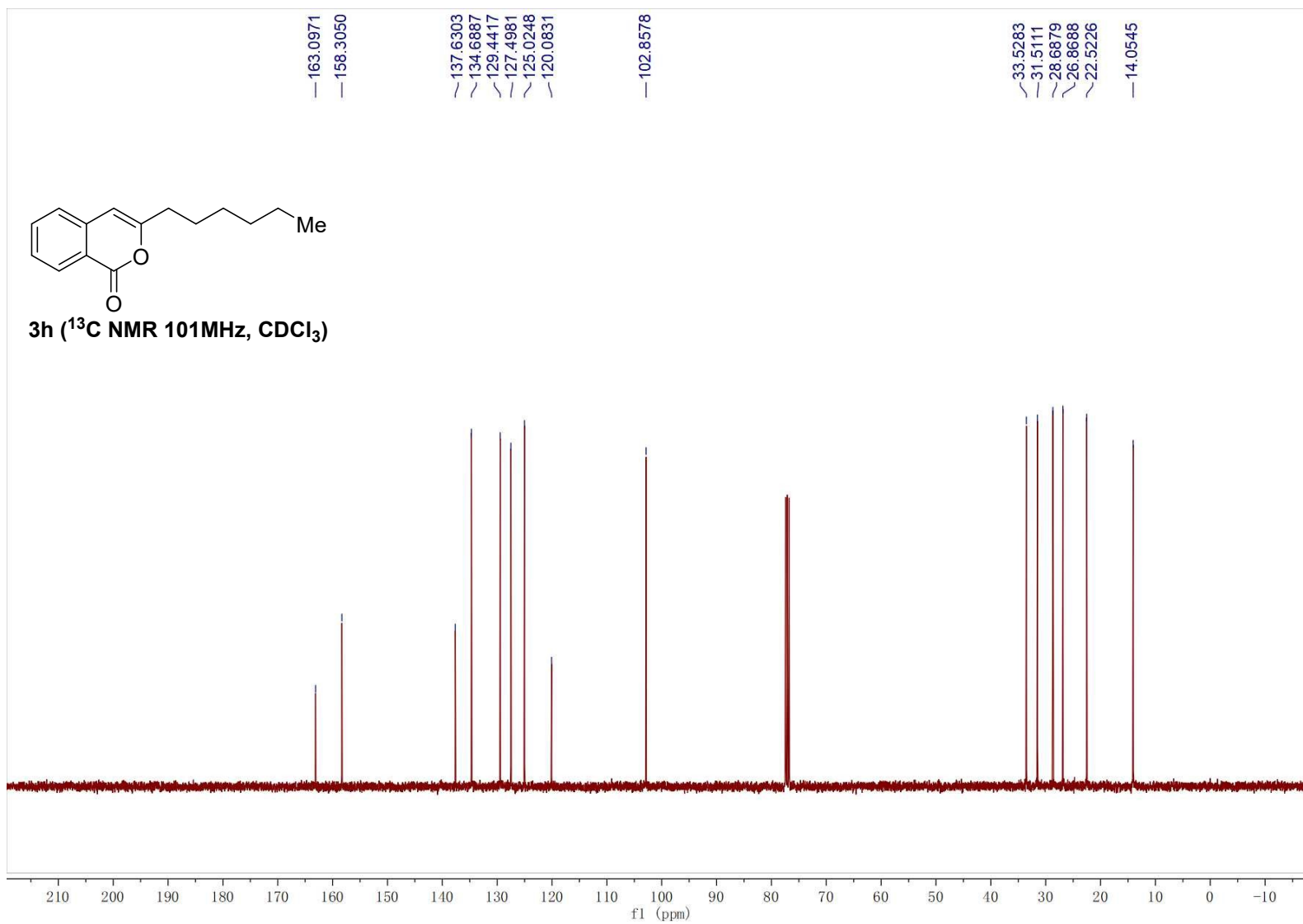


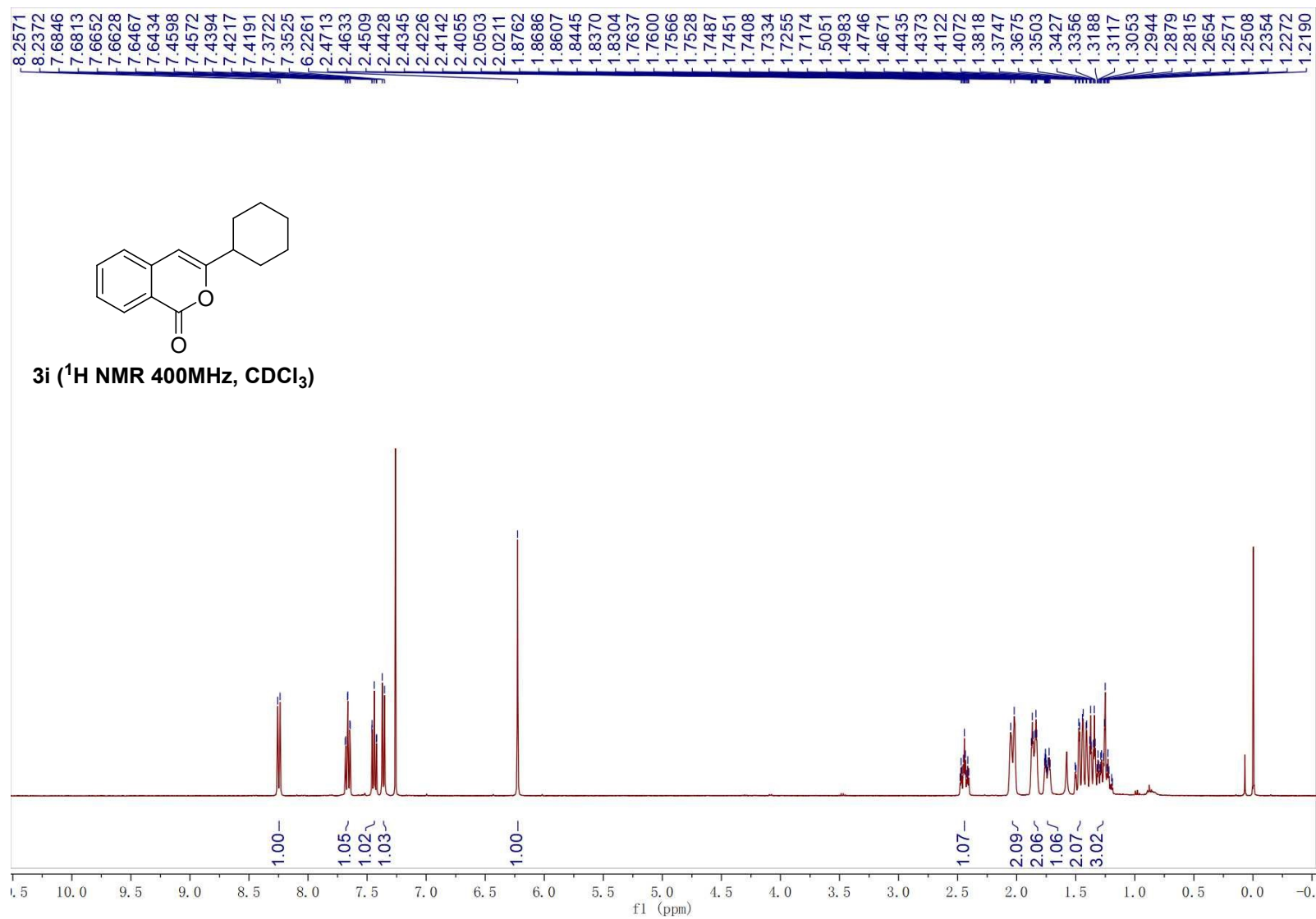


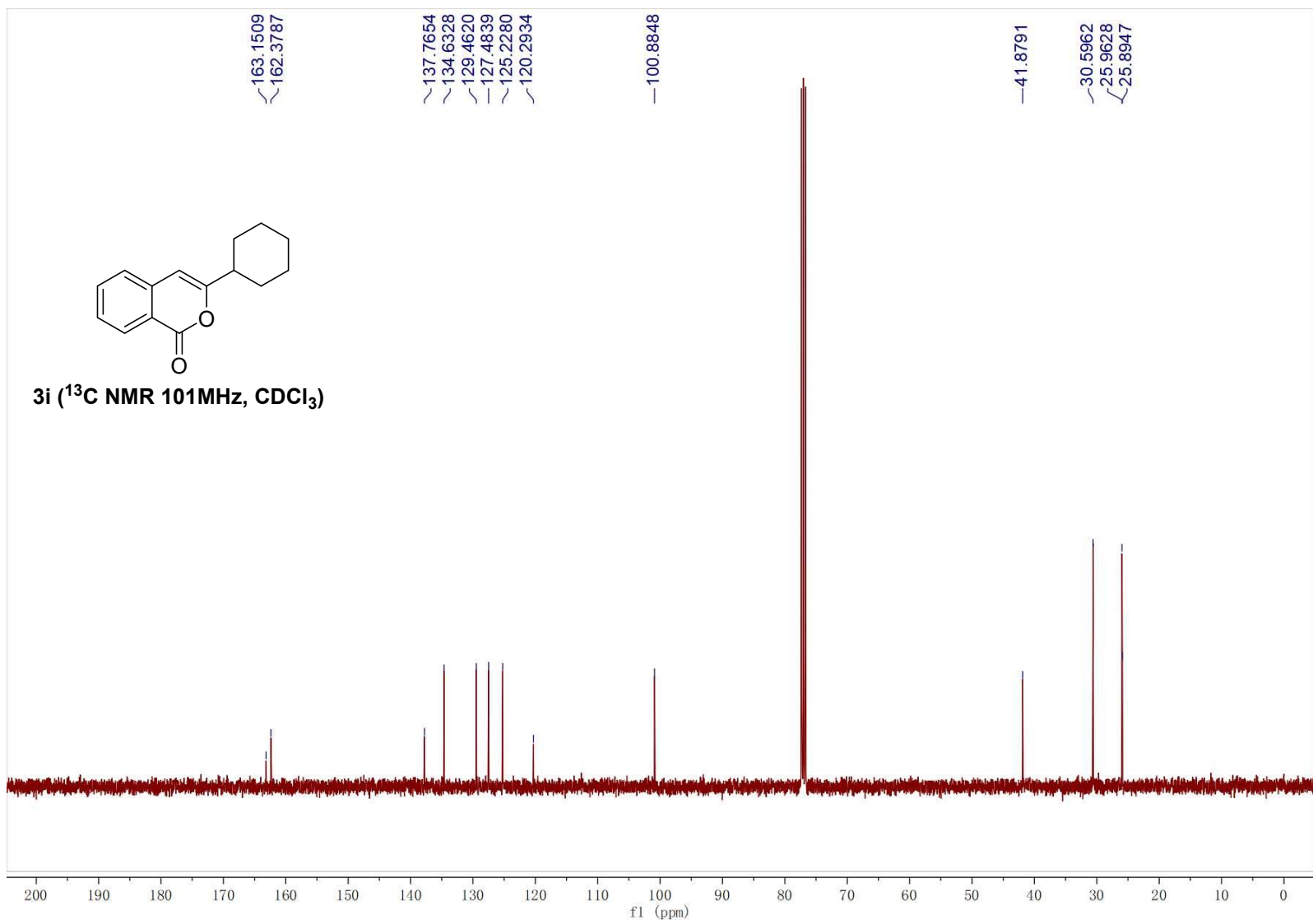


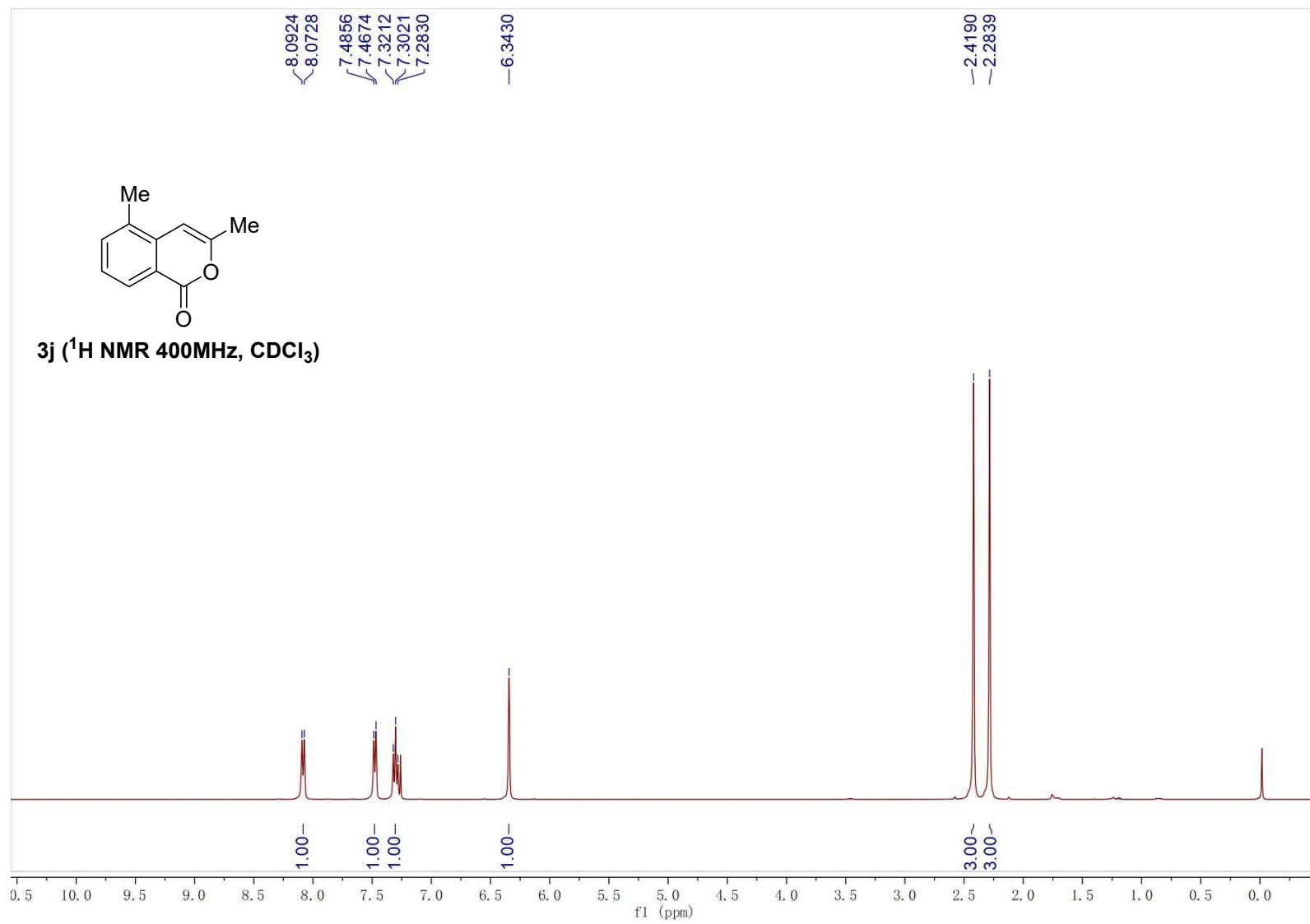


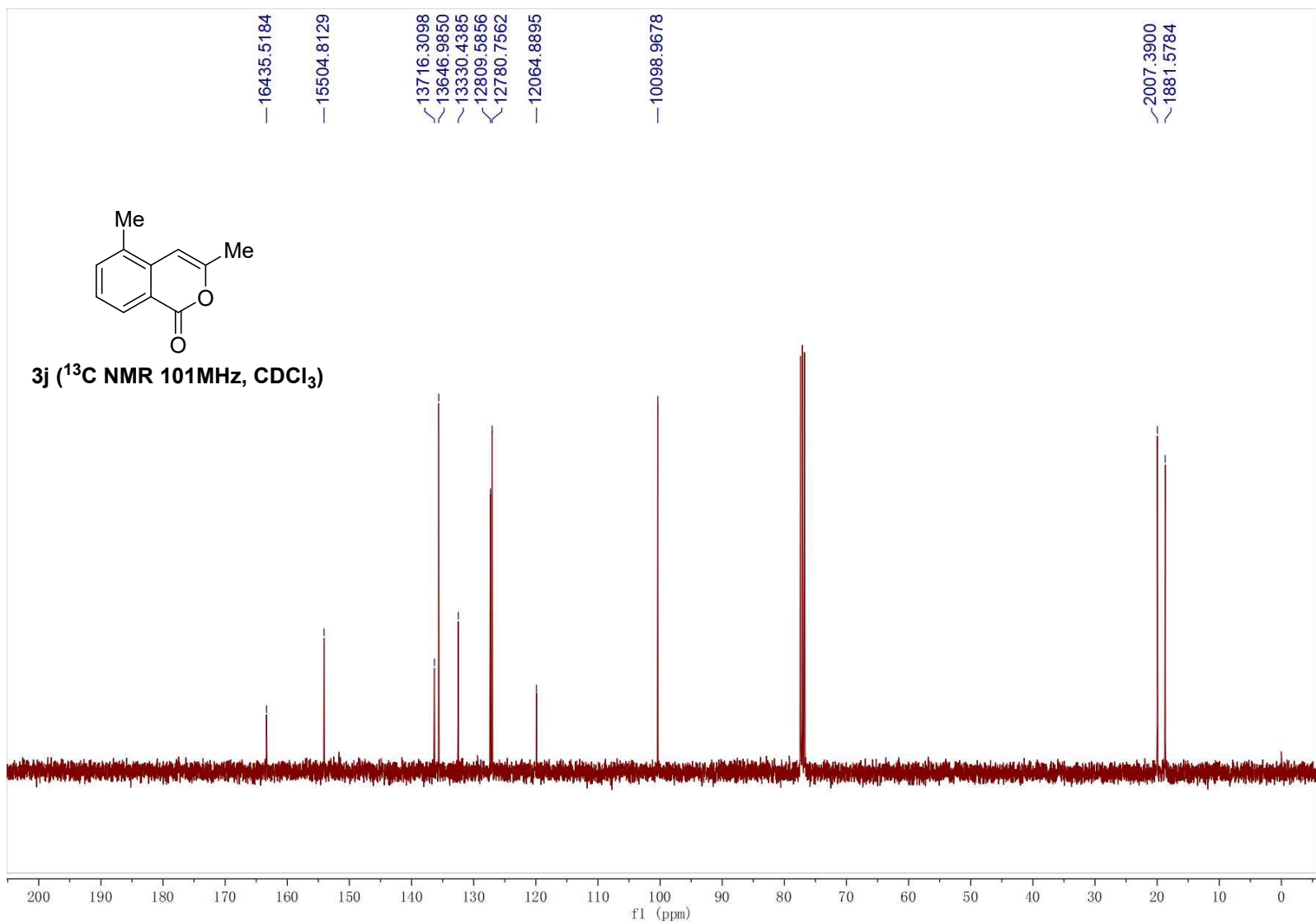


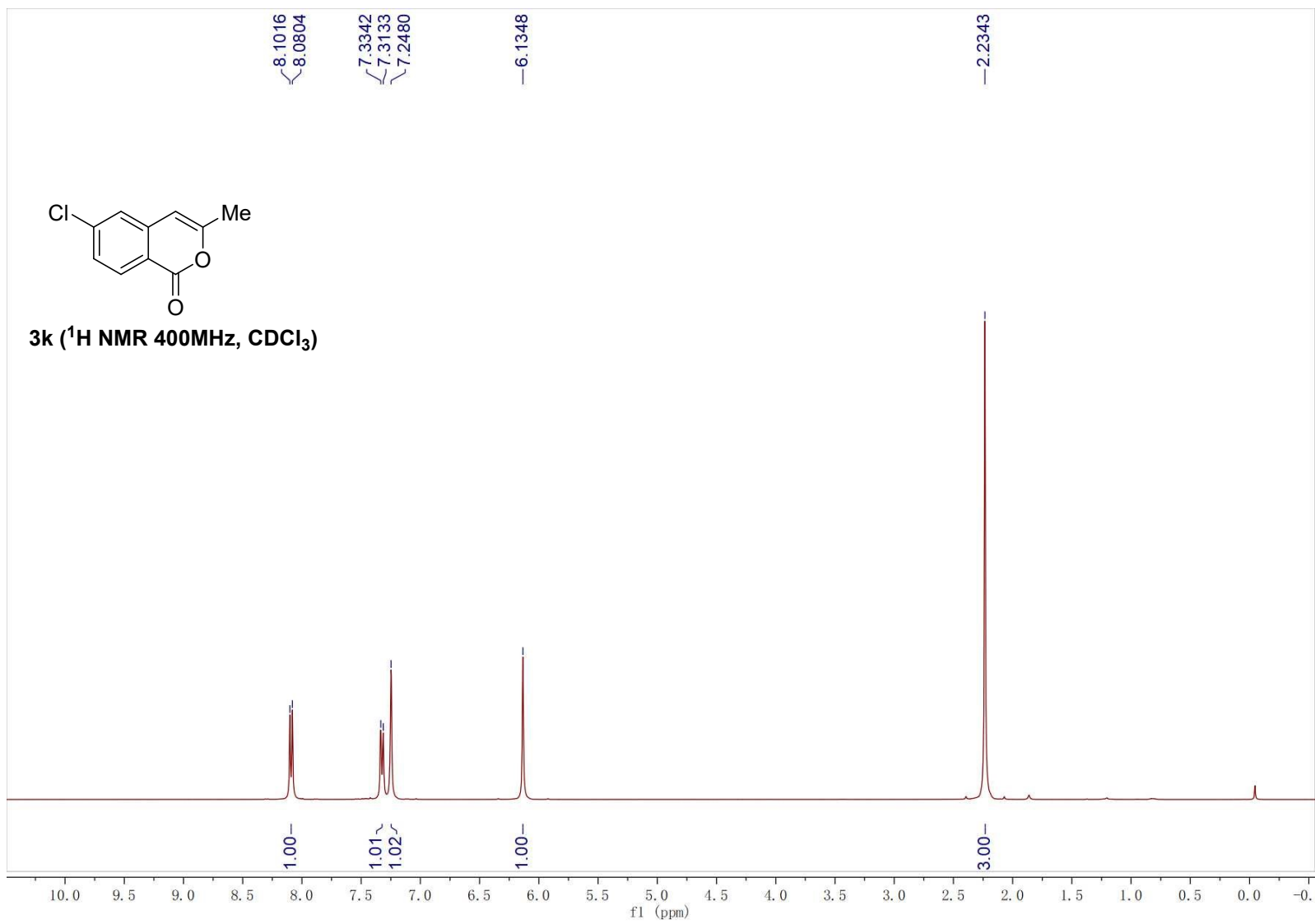


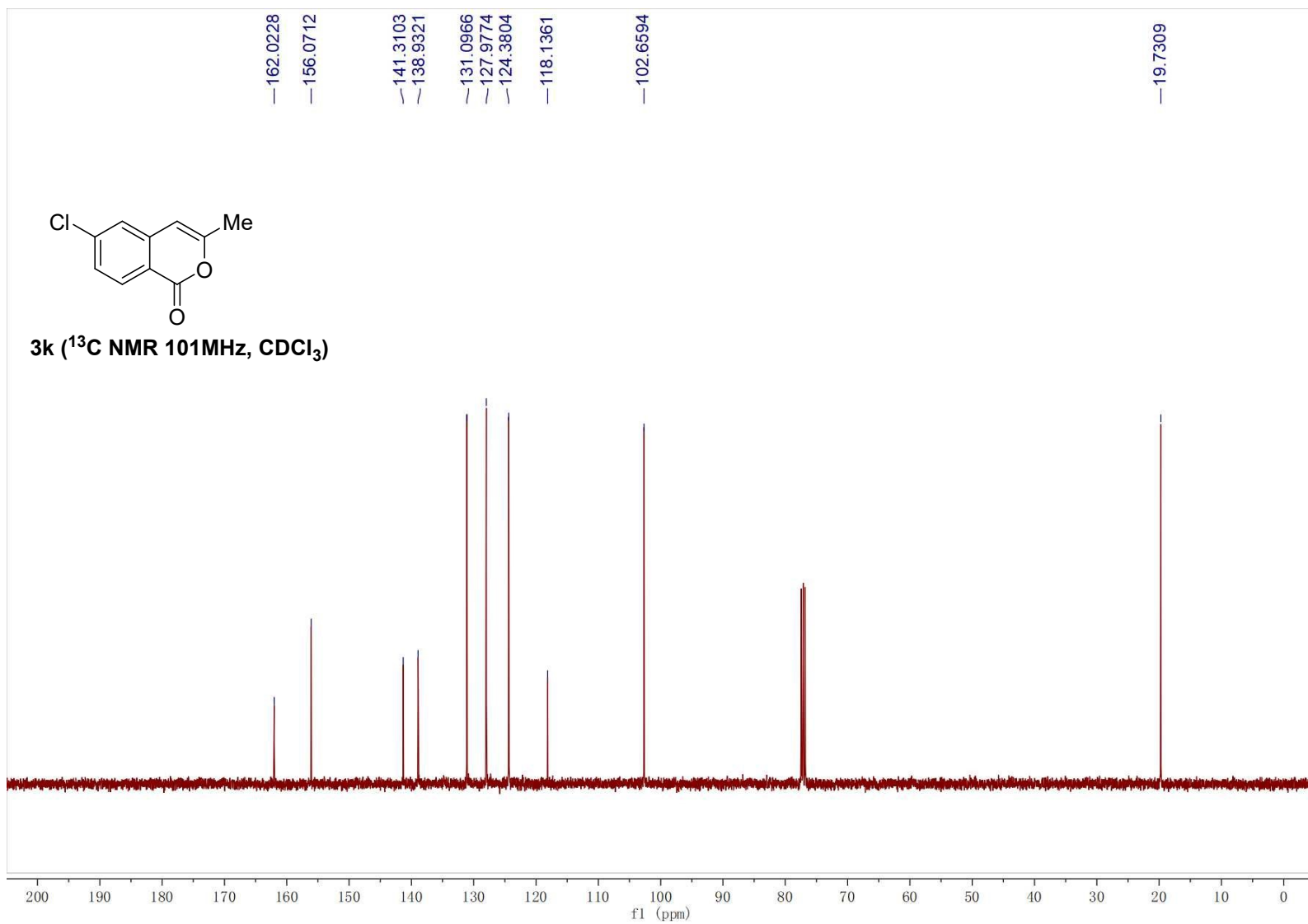


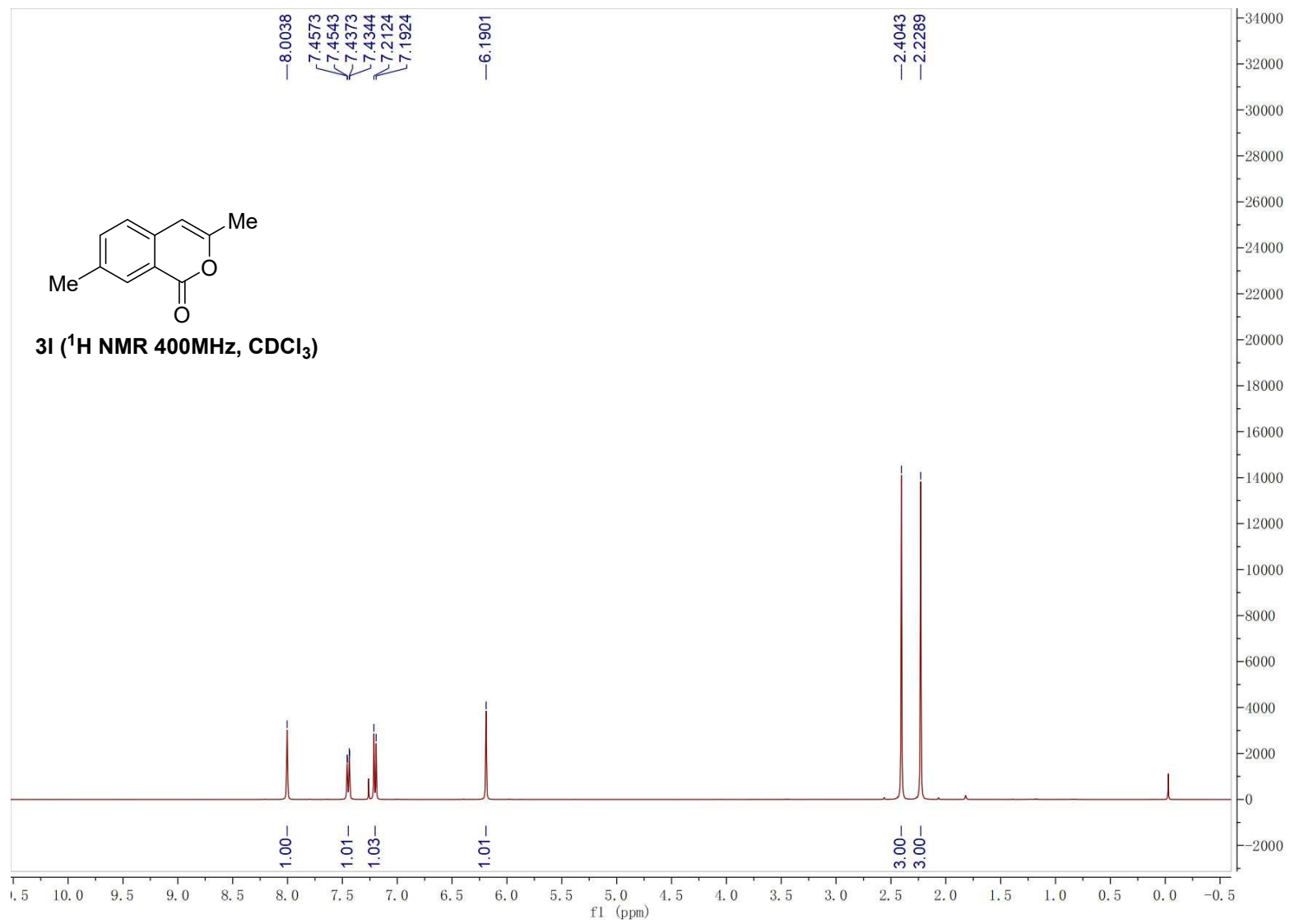


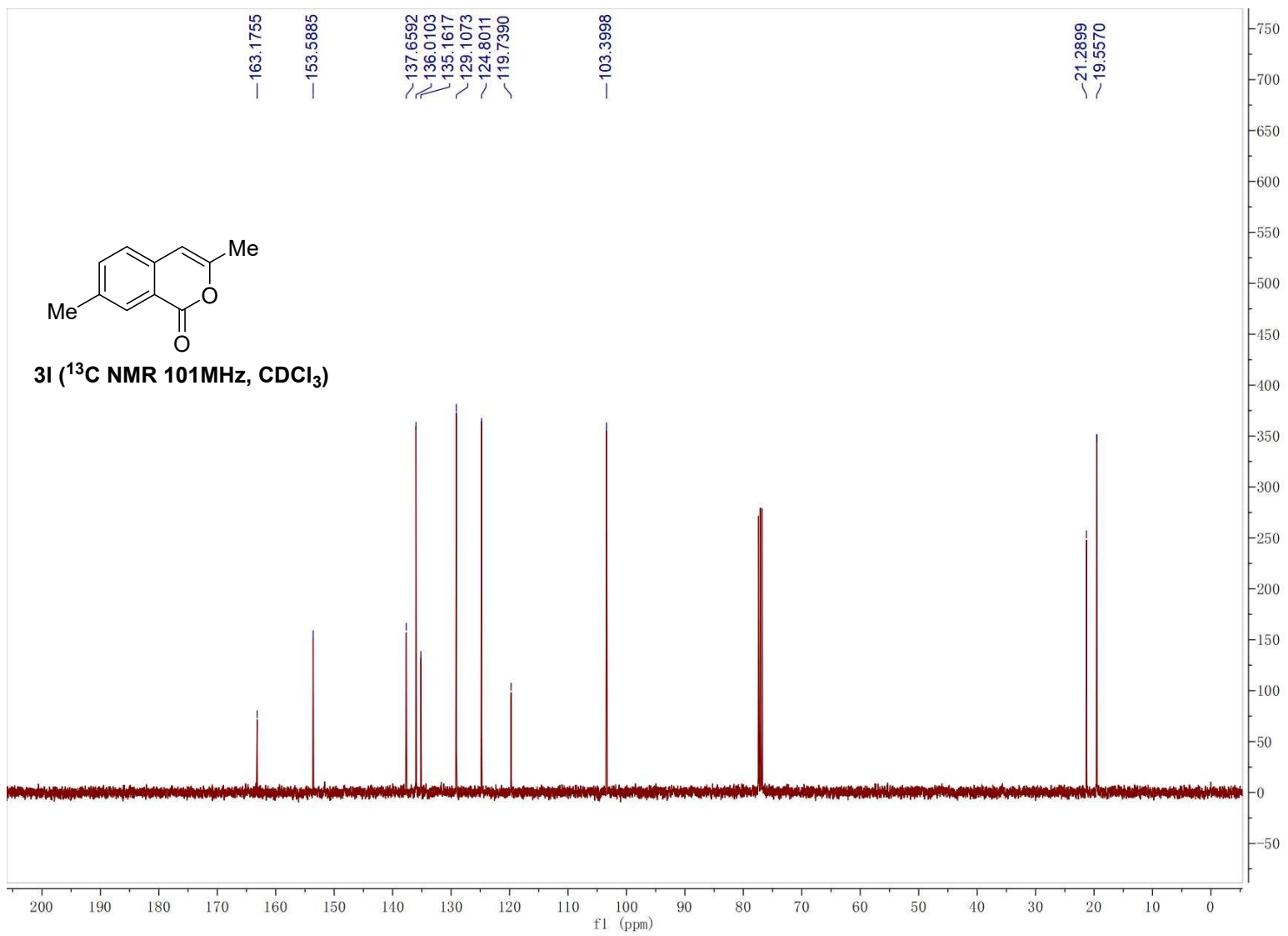


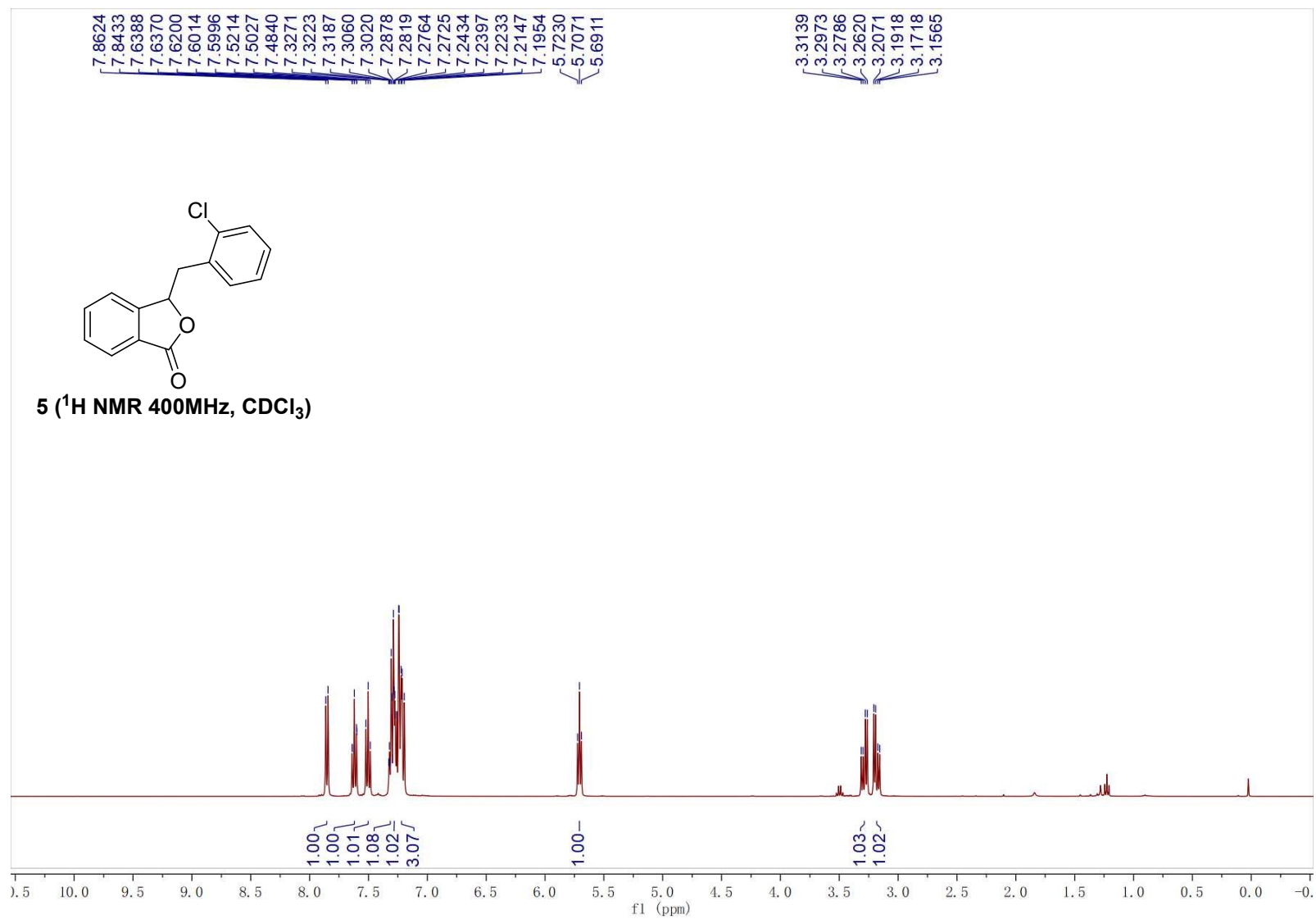


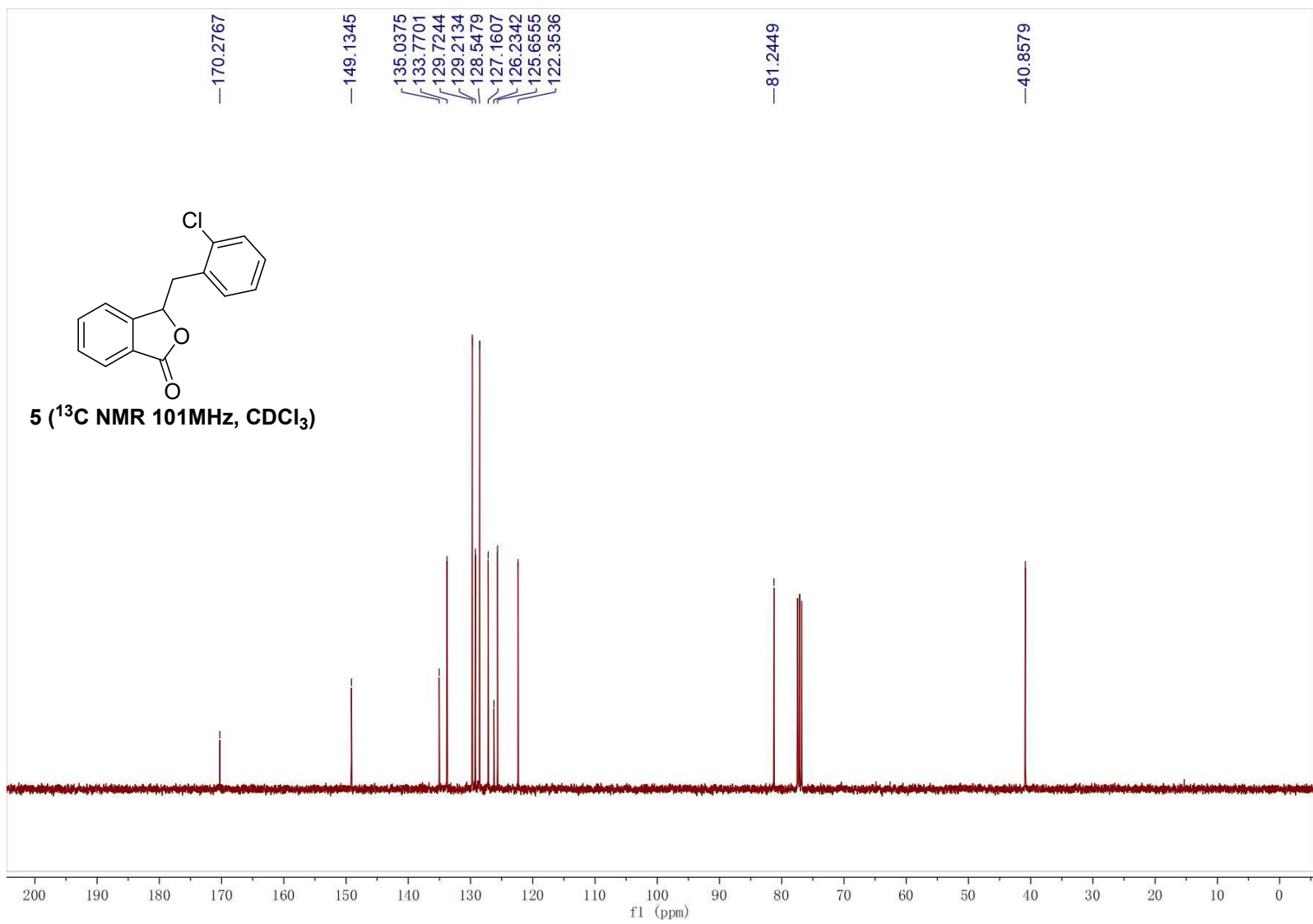


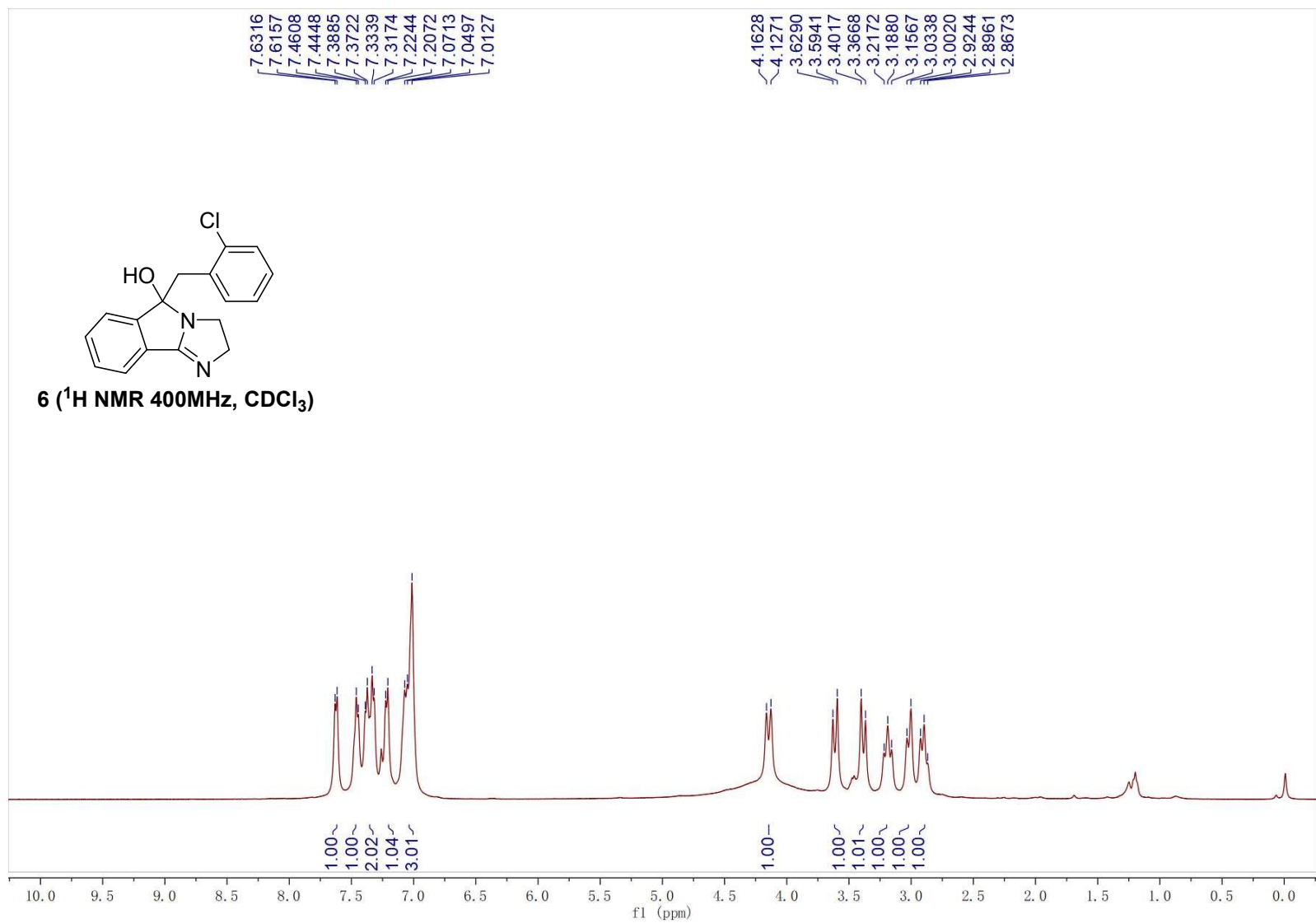


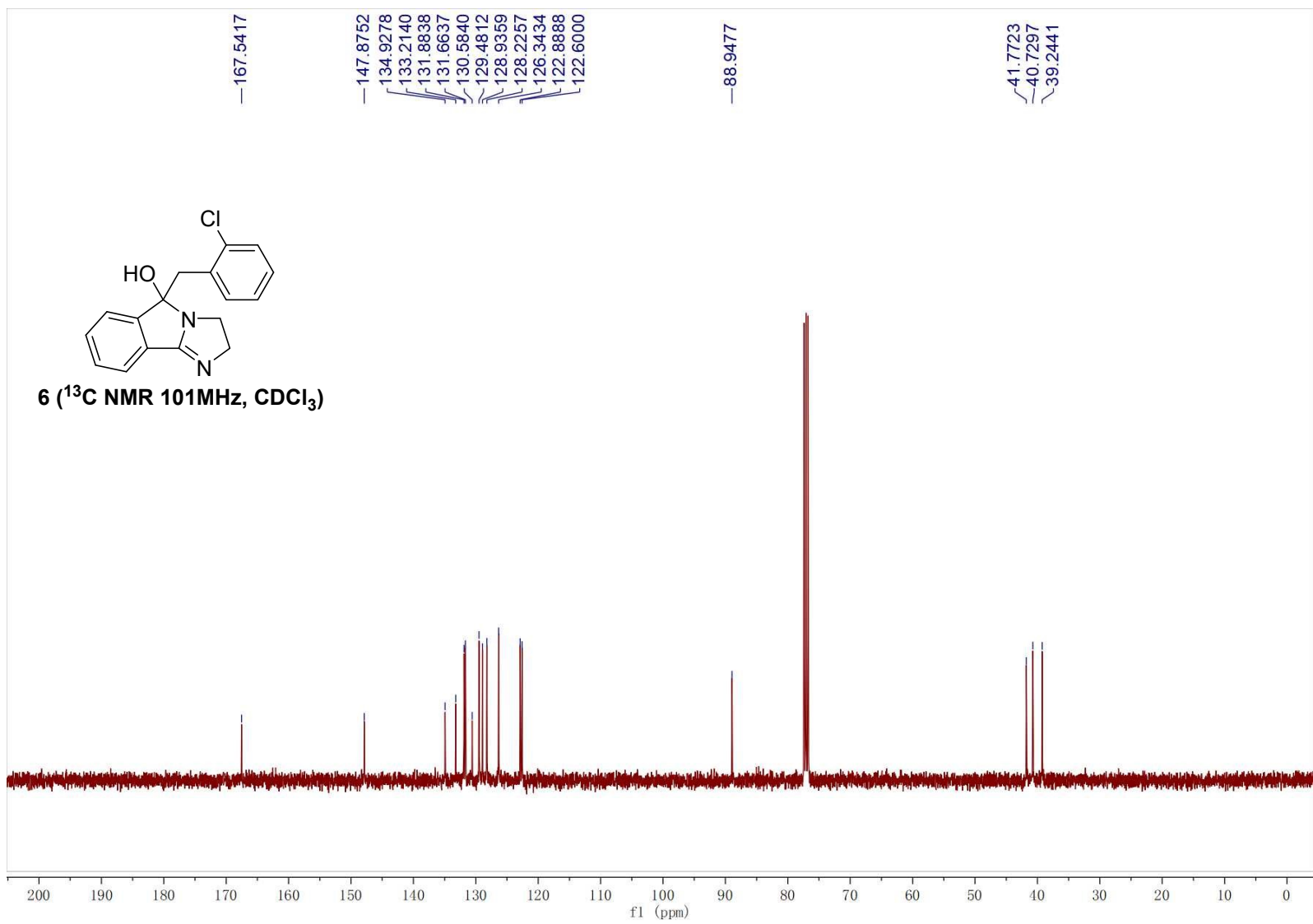


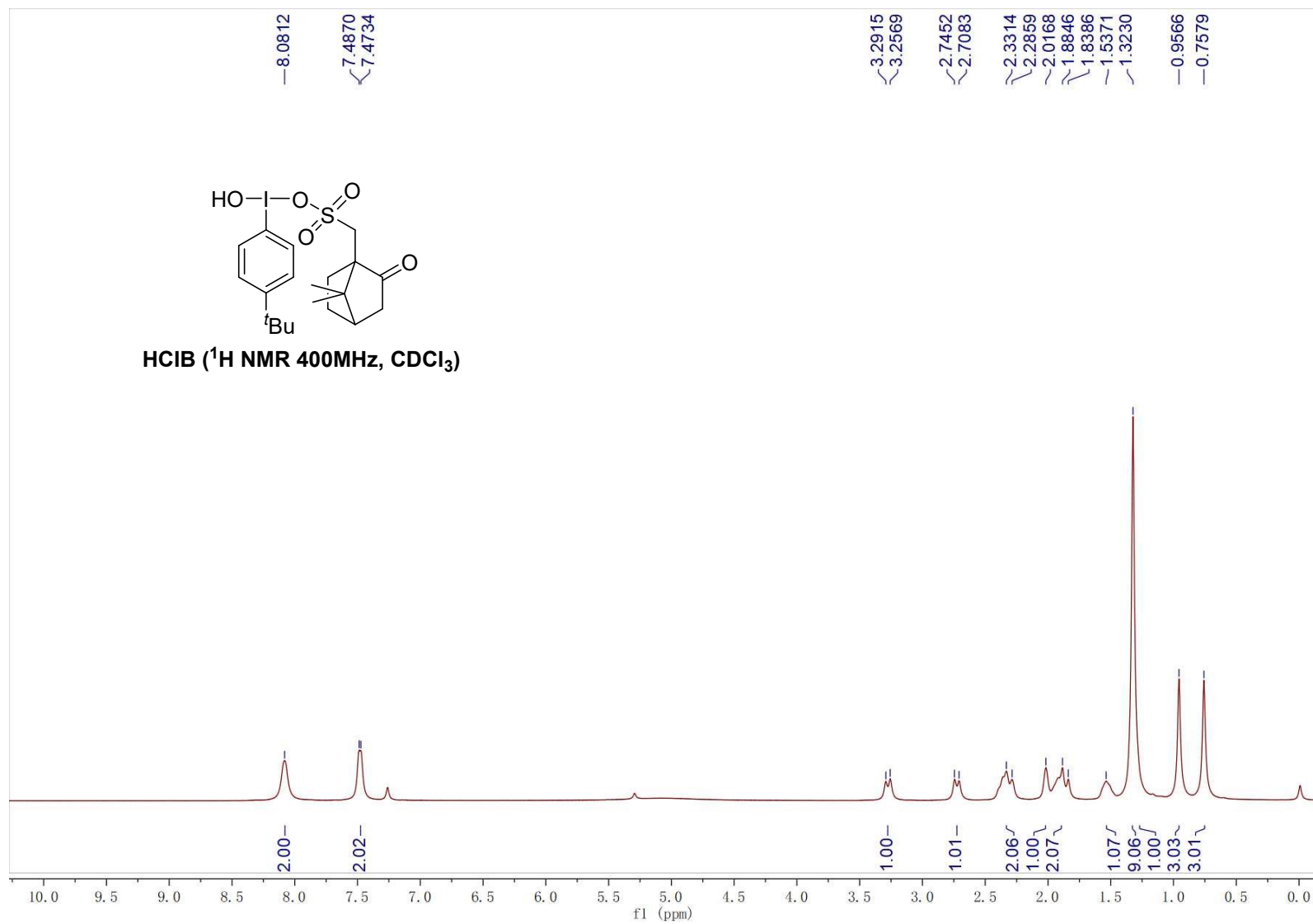


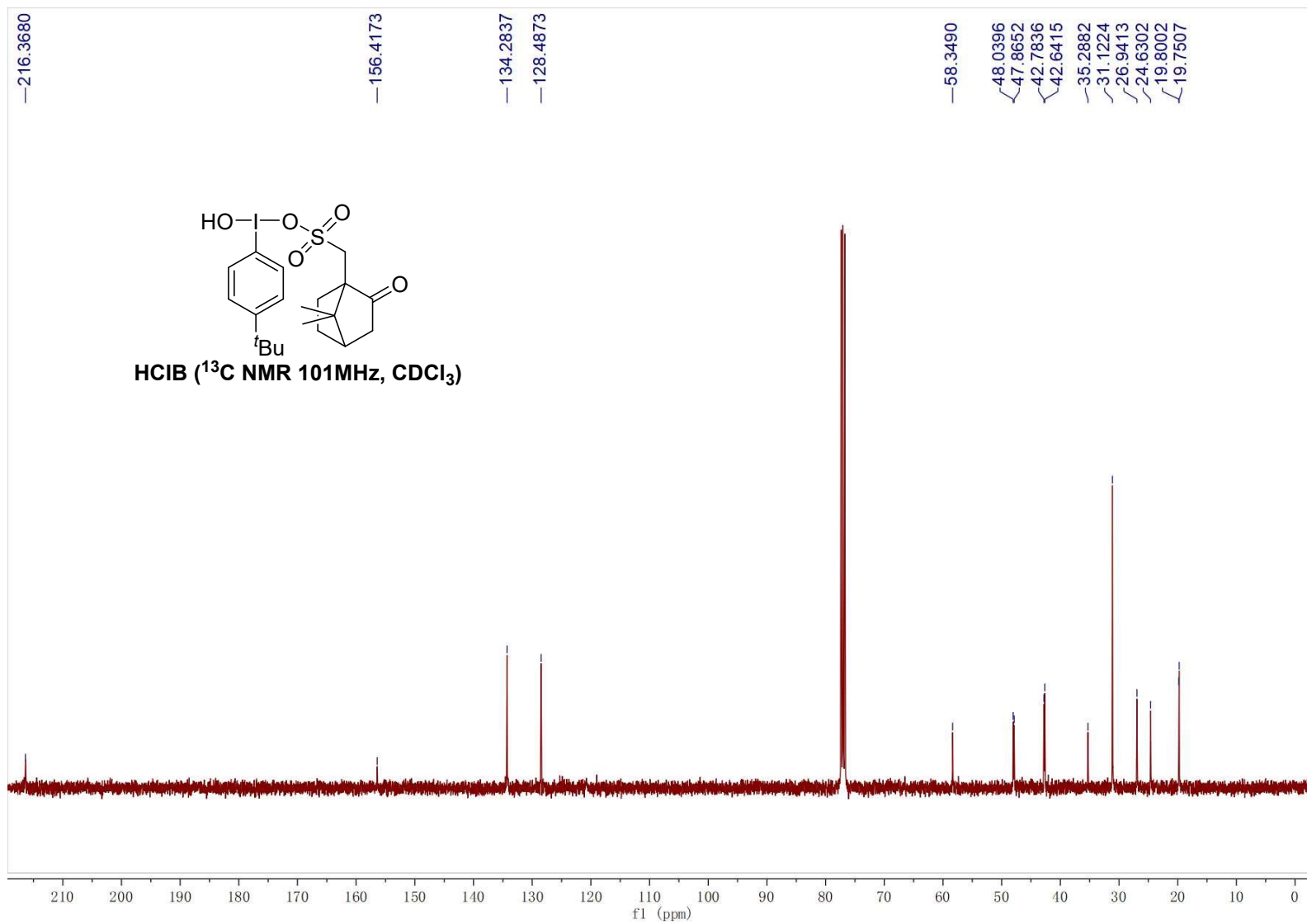












V..X-ray Crystal Structure and Data of HCIB

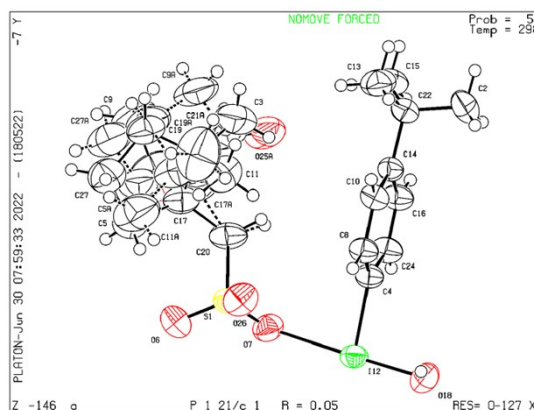
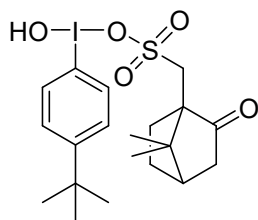


Table 1 Crystal data and structure refinement for HCIB

Identification code	HCIB
Empirical formula	C ₂₀ H ₂₉ IO ₅ S
Formula weight	508.39
Temperature/K	298
Crystal system	monoclinic
Space group	P2 ₁ /c
a/Å	14.715(3)
b/Å	7.0850(14)
c/Å	21.523(4)
α/°	90
β/°	107.03(3)
γ/°	90
Volume/Å ³	2145.5(8)
Z	4
ρ _{calc} /cm ³	1.574
μ/mm ⁻¹	1.617
F(000)	1032.0
Crystal size/mm ³	0.15 × 0.12 × 0.05
Radiation	MoKα (λ = 0.71073)
2θ range for data collection/°	6.082 to 52.742
Index ranges	-18 ≤ h ≤ 15, -8 ≤ k ≤ 8, -26 ≤ l ≤ 26
Reflections collected	17283
Independent reflections	4317 [R _{int} = 0.0720, R _{sigma} = 0.0661]
Data/restraints/parameters	4317/362/346
Goodness-of-fit on F ²	1.063
Final R indexes [I ≥ 2σ (I)]	R ₁ = 0.0512, wR ₂ = 0.1277

Final R indexes [all data] $R_1 = 0.0680$, $wR_2 = 0.1382$

Largest diff. peak/hole / $e \text{ \AA}^{-3}$ 1.54/-0.85

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for A. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{ij} tensor

Atom	x	y	z	U(eq)
I12	4562.0(3)	4004.7(5)	3228.8(2)	46.28(16)
S1	6739.1(11)	3900.9(19)	2859.6(7)	47.7(3)
O18	3372(3)	4808(7)	3366(2)	59.5(10)
O7	6083(3)	2851(6)	3142(2)	59.6(10)
O26	6325(3)	5612(6)	2552(2)	67.5(12)
C4	5391(4)	5917(7)	3894(3)	43.7(11)
O6	7113(4)	2695(7)	2454(2)	79.8(13)
C8	5551(4)	7716(7)	3710(2)	46.1(12)
C10	6106(4)	8926(7)	4172(3)	45.5(12)
C24	5757(4)	5340(8)	4529(3)	50.7(13)
C14	6510(4)	8372(8)	4811(2)	43.2(10)
C16	6303(4)	6555(8)	4984(3)	51.1(13)
C22	7097(4)	9731(9)	5334(3)	52.1(11)
C13	7530(6)	11327(10)	5043(4)	72.9(18)
C20	7700(5)	4473(10)	3548(3)	65.6(14)
C15	7908(6)	8684(11)	5820(4)	80(2)
C2	6420(6)	10589(10)	5686(4)	77.9(19)
C19	9735(8)	7407(18)	3422(7)	78(2)
C21	9564(11)	4280(20)	3802(9)	103(4)
C17	8601(7)	5304(14)	3379(6)	65.4(19)
C11	8054(10)	8471(17)	3263(8)	87(3)
C5	8739(12)	5210(20)	2662(6)	81(3)
C23	8853(8)	7252(15)	3601(6)	72(2)
C3	8983(13)	7650(30)	4281(7)	112(4)
O25	9596(8)	2779(16)	4048(8)	163(5)
C9	10329(11)	5720(20)	3895(10)	108(4)
C27	9494(12)	6630(20)	2697(7)	100(4)
C3A	8520(20)	9070(30)	3143(15)	143(7)
C11A	8640(20)	6370(40)	2558(10)	130(8)
C17A	8612(13)	5600(30)	3595(8)	93(3)

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for A. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor

Atom	x	y	z	$U(\text{eq})$
O25A	8771(12)	6000(20)	4787(7)	105(6)
C21A	9027(14)	6360(30)	4328(8)	84(5)
C9A	9838(16)	7590(40)	4302(9)	108(6)
C19A	9834(14)	7390(30)	3542(11)	111(5)
C5A	9496(15)	4170(30)	3515(12)	98(4)
C27A	10293(16)	5380(40)	3504(14)	117(6)
C23A	8800(14)	7130(30)	3244(10)	106(4)

Table 3 Anisotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for A. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[\text{h}^2\text{a}^*2U_{11}+2\text{hka}^*\text{b}^*U_{12}+\dots]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
I12	44.5(3)	51.2(2)	39.5(2)	-2.42(15)	6.45(16)	-8.02(15)
S1	44.8(9)	48.9(8)	44.5(8)	-1.7(6)	5.4(6)	-0.5(6)
O18	47(2)	75(3)	57(3)	9(2)	15.1(19)	-2(2)
O7	52(2)	51(2)	75(3)	-4.4(18)	15.9(19)	-5.6(18)
O26	58(3)	72(2)	63(3)	21(2)	3(2)	13(2)
C4	45(3)	45(2)	35.8(19)	2.1(16)	3.4(18)	-1.2(18)
O6	85(4)	79(3)	80(3)	-27(2)	31(3)	-3(2)
C8	53(3)	46(2)	34(2)	5.3(16)	5.5(19)	-1.8(19)
C10	54(3)	39(2)	39.2(19)	2.2(15)	8.0(18)	1.1(19)
C24	62(4)	44(2)	38(2)	7.2(16)	4(2)	-3(2)
C14	42(3)	49(2)	36.6(18)	-1.7(15)	6.9(17)	0.5(18)
C16	57(4)	54(2)	34(2)	6.1(17)	2(2)	-2(2)
C22	53(3)	60(3)	41(2)	-10.1(18)	9.9(17)	-6.0(19)
C13	69(5)	70(3)	76(4)	-9(3)	16(3)	-25(3)
C20	65(3)	69(4)	47(3)	9(2)	-8(2)	-11(2)
C15	68(4)	94(5)	56(4)	-7(3)	-13(3)	-2(3)
C2	90(5)	78(4)	74(4)	-27(3)	39(4)	-5(3)
C19	65(4)	61(5)	98(6)	-3(4)	9(4)	-10(3)
C21	69(4)	80(5)	135(8)	38(6)	-11(6)	4(4)
C17	61(3)	59(3)	63(4)	4(3)	-2(3)	-4(3)
C11	78(6)	64(5)	118(8)	9(6)	27(6)	7(5)
C5	77(7)	82(7)	80(4)	-13(4)	19(4)	-12(5)
C23	70(5)	63(4)	75(4)	-5(3)	7(4)	-7(3)
C3	108(10)	136(12)	83(5)	-36(6)	14(5)	-37(9)

Table 3 Anisotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for A. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^*^2U_{11}+2hka^*b^*U_{12}+\dots]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O25	91(7)	105(6)	251(12)	92(7)	-16(8)	0(5)
C9	66(4)	95(7)	141(9)	25(7)	-1(6)	-2(4)
C27	97(8)	109(8)	100(6)	-11(5)	36(5)	-29(7)
C3A	161(17)	165(10)	117(15)	65(11)	64(15)	14(12)
C11A	108(18)	199(16)	74(6)	17(9)	13(8)	-50(20)
C17A	80(5)	124(8)	63(6)	4(5)	4(5)	-39(4)
O25A	78(10)	161(15)	64(6)	1(7)	2(6)	-29(9)
C21A	52(8)	119(11)	65(5)	3(6)	-7(5)	-22(7)
C9A	70(9)	148(13)	91(7)	-3(8)	-1(6)	-45(9)
C19A	88(7)	143(10)	94(8)	8(8)	13(6)	-47(7)
C5A	78(6)	139(9)	66(12)	-7(8)	4(9)	-36(6)
C27A	85(6)	153(11)	104(15)	3(11)	14(10)	-39(7)
C23A	88(7)	142(9)	78(6)	23(7)	6(6)	-39(6)

Table 4 Bond Lengths for HCIB

Atom	Atom	Length/ \AA	Atom	Atom	Length/ \AA
I12	O18	1.945(4)	C19	C27	1.594(18)
I12	O7	2.441(4)	C21	C17	1.616(17)
I12	C4	2.087(5)	C21	O25	1.181(15)
S1	O7	1.482(4)	C21	C9	1.489(18)
S1	O26	1.430(4)	C17	C5	1.614(17)
S1	O6	1.441(5)	C17	C23	1.472(13)
S1	C20	1.770(6)	C11	C23	1.468(17)
C4	C8	1.375(7)	C5	C27	1.481(19)
C4	C24	1.376(7)	C23	C3	1.447(17)
C8	C10	1.384(7)	C3A	C23A	1.43(2)
C10	C14	1.386(7)	C11A	C23A	1.52(2)
C24	C16	1.372(8)	C17A	C21A	1.61(2)
C14	C16	1.398(8)	C17A	C5A	1.70(2)
C14	C22	1.540(8)	C17A	C23A	1.390(17)
C22	C13	1.521(9)	O25A	C21A	1.183(18)
C22	C15	1.529(9)	C21A	C9A	1.49(2)
C22	C2	1.541(9)	C9A	C19A	1.64(2)
C20	C17	1.588(12)	C19A	C27A	1.59(2)
C20	C17A	1.540(15)	C19A	C23A	1.481(19)
C19	C23	1.462(15)	C5A	C27A	1.46(2)

Table 4 Bond Lengths for HCIB

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C19	C9	1.645(17)			

Table 5 Bond Angles for HCIB

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O18	I12	O7	175.20(16)	C20	C17	C5	123.5(9)
O18	I12	C4	94.4(2)	C5	C17	C21	100.7(11)
C4	I12	O7	84.56(19)	C23	C17	C20	114.9(9)
O7	S1	C20	103.2(3)	C23	C17	C21	98.2(10)
O26	S1	O7	112.0(3)	C23	C17	C5	104.9(10)
O26	S1	O6	114.2(3)	C27	C5	C17	103.1(10)
O26	S1	C20	108.7(3)	C19	C23	C17	98.3(9)
O6	S1	O7	111.2(3)	C19	C23	C11	118.0(12)
O6	S1	C20	106.8(4)	C11	C23	C17	107.6(11)
S1	O7	I12	125.8(2)	C3	C23	C19	112.8(12)
C8	C4	I12	121.2(4)	C3	C23	C17	117.1(13)
C8	C4	C24	120.8(5)	C3	C23	C11	103.7(12)
C24	C4	I12	118.0(4)	C21	C9	C19	99.9(10)
C4	C8	C10	118.8(5)	C5	C27	C19	103.5(10)
C8	C10	C14	121.9(5)	C20	C17A	C21A	108.1(12)
C16	C24	C4	120.0(5)	C20	C17A	C5A	111.1(14)
C10	C14	C16	117.5(5)	C21A	C17A	C5A	102.4(15)
C10	C14	C22	122.3(5)	C23A	C17A	C20	132.6(15)
C16	C14	C22	120.0(5)	C23A	C17A	C21A	101.3(15)
C24	C16	C14	121.0(5)	C23A	C17A	C5A	97.5(15)
C14	C22	C2	107.5(5)	O25A	C21A	C17A	128.8(16)
C13	C22	C14	112.1(5)	O25A	C21A	C9A	127.5(17)
C13	C22	C15	107.8(6)	C9A	C21A	C17A	103.7(13)
C13	C22	C2	108.5(6)	C21A	C9A	C19A	102.6(13)
C15	C22	C14	110.8(5)	C27A	C19A	C9A	104.4(18)
C15	C22	C2	110.1(6)	C23A	C19A	C9A	98.3(15)
C17	C20	S1	114.1(6)	C23A	C19A	C27A	105.2(16)
C17A	C20	S1	129.8(7)	C27A	C5A	C17A	107.0(15)
C23	C19	C9	97.7(11)	C5A	C27A	C19A	99.9(15)
C23	C19	C27	105.7(10)	C3A	C23A	C11A	103.6(18)
C27	C19	C9	106.2(11)	C3A	C23A	C19A	99.0(19)
O25	C21	C17	124.7(14)	C17A	C23A	C3A	137(2)
O25	C21	C9	128.9(13)	C17A	C23A	C11A	104.6(17)

Table 5 Bond Angles for HCIB

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C9	C21	C17	105.7(10)	C17A	C23A	C19A	102.0(14)
C20	C17	C21	110.9(10)	C19A	C23A	C11A	108.4(18)

Table 6 Torsion Angles for HCIB

A	B	C	D	Angle/°	A	B	C	D	Angle/°
I12	C4	C8	C10	179.9(4)	C5	C17	C23	C19	-46.7(13)
I12	C4	C24	C16	-179.7(5)	C5	C17	C23	C11	76.1(13)
S1	C20	C17	C21	-132.6(10)	C5	C17	C23	C3	-167.7(13)
S1	C20	C17	C5	-13.2(12)	C23	C19	C9	C21	41.3(15)
S1	C20	C17	C23	117.2(10)	C23	C19	C27	C5	-29.9(16)
S1	C20	C17A	C21A	158.7(12)	C23	C17	C5	C27	29.1(15)
S1	C20	C17A	C5A	-89.6(15)	O25	C21	C17	C20	22(3)
S1	C20	C17A	C23A	33(3)	O25	C21	C17	C5	-111(2)
O7	S1	C20	C17	169.9(6)	O25	C21	C17	C23	142(2)
O7	S1	C20	C17A	-178.4(13)	O25	C21	C9	C19	-178(2)
O26	S1	O7	I12	-8.2(4)	C9	C19	C23	C17	-61.9(13)
O26	S1	C20	C17	-71.1(7)	C9	C19	C23	C11	-176.9(12)
O26	S1	C20	C17A	-59.3(14)	C9	C19	C23	C3	62.2(14)
C4	C8	C10	C14	0.6(9)	C9	C19	C27	C5	73.2(14)
C4	C24	C16	C14	-1.2(10)	C9	C21	C17	C20	-149.7(13)
O6	S1	O7	I12	-137.3(3)	C9	C21	C17	C5	78.0(15)
O6	S1	C20	C17	52.6(6)	C9	C21	C17	C23	-29.0(17)
O6	S1	C20	C17A	64.3(14)	C27	C19	C23	C17	47.4(13)
C8	C4	C24	C16	-1.2(9)	C27	C19	C23	C11	-67.6(14)
C8	C10	C14	C16	-2.8(9)	C27	C19	C23	C3	171.5(13)
C8	C10	C14	C22	-177.6(5)	C27	C19	C9	C21	-67.7(15)
C10	C14	C16	C24	3.1(9)	C17A	C21A	C9A	C19A	-1(3)
C10	C14	C22	C13	-24.7(8)	C17A	C5A	C27A	C19A	-3(2)
C10	C14	C22	C15	-145.2(6)	O25A	C21A	C9A	C19A	177(3)
C10	C14	C22	C2	94.5(7)	C21A	C17A	C5A	C27A	-70(2)
C24	C4	C8	C10	1.5(9)	C21A	C17A	C23A	C3A	-63(3)
C16	C14	C22	C13	160.6(6)	C21A	C17A	C23A	C11A	167.5(18)
C16	C14	C22	C15	40.1(8)	C21A	C17A	C23A	C19A	55(2)
C16	C14	C22	C2	-80.2(7)	C21A	C9A	C19A	C27A	-76(2)
C22	C14	C16	C24	178.0(6)	C21A	C9A	C19A	C23A	32(2)
C20	S1	O7	I12	108.5(4)	C9A	C19A	C27A	C5A	75.4(19)
C20	C17	C5	C27	163.4(11)	C9A	C19A	C23A	C3A	88(2)

Table 6 Torsion Angles for HCIB

A	B	C	D	Angle/°	A	B	C	D	Angle/°
C20	C17	C23	C19	174.4(9)	C9A	C19A	C23A	C11A	-164.5(18)
C20	C17	C23	C11	-62.7(14)	C9A	C19A	C23A	C17A	-55(2)
C20	C17	C23	C3	53.4(16)	C5A	C17A	C21A	O25A	-110(3)
C20	C17A	C21A	O25A	7(3)	C5A	C17A	C21A	C9A	68(2)
C20	C17A	C21A	C9A	-174.1(19)	C5A	C17A	C23A	C3A	-167(3)
C20	C17A	C5A	C27A	174.8(16)	C5A	C17A	C23A	C11A	63.1(19)
C20	C17A	C23A	C3A	65(4)	C5A	C17A	C23A	C19A	-49.7(18)
C20	C17A	C23A	C11A	-65(3)	C27A	C19A	C23A	C3A	-164.7(19)
C20	C17A	C23A	C19A	-178(2)	C27A	C19A	C23A	C11A	-57(2)
C21	C17	C5	C27	-72.5(13)	C27A	C19A	C23A	C17A	53(2)
C21	C17	C23	C19	56.7(13)	C23A	C17A	C21A	O25A	149(3)
C21	C17	C23	C11	179.6(12)	C23A	C17A	C21A	C9A	-32(2)
C21	C17	C23	C3	-64.2(15)	C23A	C17A	C5A	C27A	33(2)
C17	C21	C9	C19	-6.9(17)	C23A	C19A	C27A	C5A	-28(2)
C17	C5	C27	C19	0.3(16)					

Table 7 Hydrogen Atom Coordinates ($\text{\AA} \times 10^4$) and Isotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for HCIB

Atom	x	y	z	U(eq)
H8	5290.66	8111.8	3282.79	55
H10	6210.88	10147.12	4050.31	55
H24	5635.33	4128.25	4650.07	61
H16	6538.55	6163.72	5413.86	61
H13A	7947.97	10812.23	4817.68	109
H13B	7881.63	12149.01	5383.18	109
H13C	7032.33	12028.93	4743.27	109
H20A	7484.36	5389.55	3808.84	79
H20B	7889.67	3345.6	3810.01	79
H20C	7410.4	5110.86	3841.35	79
H20D	7921.85	3268.8	3750.78	79
H15A	7652.03	7680.36	6017.64	120
H15B	8250.72	9546.1	6150.33	120
H15C	8330.29	8169.32	5598.45	120
H2A	5904.32	11214.67	5375.43	117
H2B	6760.78	11483.47	6004.39	117
H2C	6173.17	9603.72	5896.61	117
H19	10036.92	8653.63	3492.5	94

Table 7 Hydrogen Atom Coordinates ($\text{\AA}\times 10^4$) and Isotropic Displacement Parameters ($\text{\AA}^2\times 10^3$) for HCIB

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U(eq)
H11A	8190.63	9749.7	3408.84	131
H11B	7487.83	8054.13	3357.2	131
H11C	7962.47	8407.53	2803.62	131
H5A	8937.54	3964.46	2570.59	97
H5B	8156.36	5547.22	2331.32	97
H3A	9427.82	6762.6	4543.07	168
H3B	8384.67	7531.66	4372.46	168
H3C	9222.81	8905.42	4379.52	168
H9A	10854.28	5265.52	3749.18	129
H9B	10564.66	6125.28	4343.91	129
H27A	9266.53	7629.51	2383.7	120
H27B	10047.22	6045.15	2619.38	120
H3AA	8709.89	9562.68	2785.62	214
H3AB	8818.81	9777.55	3528.84	214
H3AC	7841.38	9160.74	3048.37	214
H11D	7986.55	6580.65	2309.14	194
H11E	8766.54	5036.74	2576.08	194
H11F	9050	7002.99	2354.91	194
H9AA	10432.25	7144.3	4595.99	130
H9AB	9734.15	8886.86	4408.55	130
H19A	10118.06	8448.71	3370.35	134
H5AA	9683.33	3293.31	3877.1	118
H5AB	9283.29	3446.51	3115.35	118
H27C	10468.01	5226.5	3106.49	140
H27D	10843.27	5153.16	3875.33	140
H18	3320(40)	5870(50)	3140(20)	41(16)

Table 8 Atomic Occupancy for HCIB

Atom	Occupancy	Atom	Occupancy	Atom	Occupancy
H20A	0.617(8)	H20B	0.617(8)	H20C	0.383(8)
H20D	0.383(8)	C19	0.617(8)	H19	0.617(8)
C21	0.617(8)	C17	0.617(8)	C11	0.617(8)
H11A	0.617(8)	H11B	0.617(8)	H11C	0.617(8)
C5	0.617(8)	H5A	0.617(8)	H5B	0.617(8)
C23	0.617(8)	C3	0.617(8)	H3A	0.617(8)
H3B	0.617(8)	H3C	0.617(8)	O25	0.617(8)

Table 8 Atomic Occupancy for HCIB

Atom	<i>Occupancy</i>	Atom	<i>Occupancy</i>	Atom	<i>Occupancy</i>
C9	0.617(8)	H9A	0.617(8)	H9B	0.617(8)
C27	0.617(8)	H27A	0.617(8)	H27B	0.617(8)
C3A	0.383(8)	H3AA	0.383(8)	H3AB	0.383(8)
H3AC	0.383(8)	C11A	0.383(8)	H11D	0.383(8)
H11E	0.383(8)	H11F	0.383(8)	C17A	0.383(8)
O25A	0.383(8)	C21A	0.383(8)	C9A	0.383(8)
H9AA	0.383(8)	H9AB	0.383(8)	C19A	0.383(8)
H19A	0.383(8)	C5A	0.383(8)	H5AA	0.383(8)
H5AB	0.383(8)	C27A	0.383(8)	H27C	0.383(8)
H27D	0.383(8)	C23A	0.383(8)		