

**Facile Synthesis of 5*H*-Benzo[*b*]carbazol-6-yl Ketones via
Sequential Reaction of Cu-Catalyzed Friedel-Crafts
Alkylation/Iodine-Promoted Cyclization/Nucleophilic
Substitution/Aromatization**

Supplementary Information

Jing Wu, Dongping Wang, Haolong Wang, Fan Wu, Xincheng Li, and

Boshun Wan*

*Dalian Institute of Chemical Physics, Chinese Academy of Sciences,
457 Zhongshan Road, Dalian 116023, China.
E-mail: bswan@dicp.ac.cn*

Table of Contents

1. Experimental section.....	S1-S8
2. NMR Spectra (¹ H, ¹³ C)	S9-S38
3. X-ray structure of 5	S39-S41
4. TIC spectrum and MS spectrum of five substances with molecule weight of 591.1.....	S42-S47

General Considerations. ^1H NMR and ^{13}C NMR spectra were recorded on Bruker DRX-400 spectrometers (100 MHz for ^{13}C NMR) using CDCl_3 as solvent. Tetramethylsilane ($\delta = 0$) or CDCl_3 ($\delta = 7.27$) serves as the internal standard for ^1H NMR and CDCl_3 (77.16 ppm) for ^{13}C NMR. Coupling constants (J) are reported in Hz and refer to apparent peak multiplications. The abbreviations *s*, *d*, *t*, *q*, and *m* stand for singlet, doublet, triplet, quartet, and multiplet in that order. HRMS data were obtained with micromass HPLC-Q-TOF mass spectrometer. Flash column chromatography was performed on silica gel (300–400 mesh). TLC analysis was performed using silica gel GF254 and visualized by fluorescence quenching under UV light.

Commercially available reagents were used throughout without further purification. All solvents were purified according to the standard procedures.

General Procedure for the Asymmetric Friedel-Crafts Alkylation Reaction:

In a 25 mL Schlenk tube bis(sulfonamide)-diamine ligand **6** (13 mg, 0.0165 mmol, 5.5 mol%) and $\text{Cu}(\text{OTf})_2$ (5.4 mg, 0.015 mmol, 5 mol%) were dissolved in *i*-BuOH (1 mL), and the mixture was stirred at room temperature for 2 h to give the catalyst. After putting it into a freezer with the temperature of 0 °C, this mixture was added 2-(2-(alkynyl)benzylidene) malonate (0.3 mmol, 1.0 equiv.). After stirred for 15 min, indole was added (0.3 mmol, 1.0 equiv.). The reaction was stirred at 0 °C and monitored by TLC. When the reaction was finished (about 6–24 h), the solvent was evaporated under reduced pressure. Then CH_3CN (1 mL) with H_2O (1.0 equiv.) and I_2 (152mg, 2.0 equiv.) were added, the reaction was conducted at 50 °C and monitored

by TLC until finished (about 2h). Then the solvent was evaporated under reduced pressure and the crude residue was purified by column chromatography to afford the desired product.

(5H-benzo[b]carbazol-6-yl)(phenyl)methanone (4a).

Following the general procedure, the compound was obtained as yellow solid: 85 mg, 88% yield; mp 179–180 °C; $R_f = 0.43$ (petroleum ether/EtOAc = 8/1); ^1H NMR (400 MHz, CDCl_3) δ 9.41 (s, 1H), 8.69 (s, 1H), 8.19 (d, $J = 7.7$ Hz, 1H), 8.05 (d, $J = 8.2$ Hz, 1H), 7.74 (d, $J = 7.4$ Hz, 2H), 7.68 (d, $J = 8.6$ Hz, 1H), 7.55 (t, $J = 7.4$ Hz, 1H), 7.49 (t, $J = 7.6$ Hz, 1H), 7.39 (m, 4H), 7.32–7.20 (m, 2H); ^{13}C NMR (101 MHz, CDCl_3) δ 198.1, 142.1, 141.4, 140.5, 132.8, 131.0, 129.9, 129.4, 128.8, 128.3, 128.1, 126.2, 126.1, 126.0, 124.3, 123.1, 122.7, 121.2, 120.4, 113.4, 111.0; HRMS (ESI, m/z) Calculated for $\text{C}_{23}\text{H}_{15}\text{NO}$ $[\text{M} + \text{H}]^+$ 322.1226, found 322.1236.

(9-methoxy-5H-benzo[b]carbazol-6-yl)(phenyl)methanone (4b).

Following the general procedure, the compound was obtained as brown solid: 68 mg, 65% yield; mp 206 – 207 °C; $R_f = 0.3$ (petroleum ether/EtOAc = 8/1); ^1H NMR (400 MHz, CDCl_3) δ 9.31 (s, 1H), 8.55 (s, 1H), 8.13 (d, $J = 7.7$ Hz, 1H), 7.71 (d, $J = 7.3$ Hz, 2H), 7.60–7.48 (m, 2H), 7.44 (t, $J = 7.5$ Hz, 1H), 7.41–7.30 (m, 4H), 7.24 (t, $J = 7.4$ Hz, 1H), 6.96 (dd, $J = 9.3, 2.6$ Hz, 1H), 3.90 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 198.0, 155.4, 142.2, 140.4, 140.0, 132.8, 129.9, 129.4, 128.7, 127.9, 127.4, 126.5, 126.4, 122.8, 122.5, 121.1, 120.1, 118.8, 113.8, 110.9, 107.0, 55.4; HRMS HRMS (ESI, m/z) Calculated for $\text{C}_{24}\text{H}_{17}\text{NO}_2$ $[\text{M} + \text{H}]^+$ 352.1332, found 352.1348.

(9-chloro-5H-benzo[b]carbazol-6-yl)(phenyl)methanone (4c).

Following the general procedure, the compound was obtained as yellow solid: 83 mg, 78% yield; mp 208–209 °C; $R_f = 0.35$ (petroleum ether/EtOAc = 8/1); ^1H NMR (400 MHz, CDCl_3) δ 9.37 (s, 1H), 8.52 (s, 1H), 8.14 (d, $J = 7.6$ Hz, 1H), 7.98 (s, 1H), 7.69 (d, $J = 7.5$ Hz, 2H), 7.56 (t, $J = 8.9$ Hz, 2H), 7.48 (t, $J = 7.5$ Hz, 1H), 7.44–7.33 (m, 3H), 7.27 (t, $J = 7.4$ Hz, 1H), 7.18 (d, $J = 9.0$ Hz, 1H); ^{13}C NMR (101 MHz, CDCl_3) δ 197.6, 142.2, 141.2, 140.2, 133.1, 129.9, 129.0, 128.9, 128.6, 128.5, 127.6, 127.5, 127.1, 126.6, 123.0, 122.3, 121.3, 120.6, 113.5, 111.1; HRMS (ESI, m/z) Calculated for $\text{C}_{23}\text{H}_{14}\text{ClNO}$ $[\text{M} + \text{H}]^+$ 356.0837, found 356.0840.

(9-fluoro-5H-benzo[b]carbazol-6-yl)(phenyl)methanone (4d).

Following the general procedure, the compound was obtained as yellow solid: 72 mg, 71% yield; mp 209–210 °C; $R_f = 0.35$ (petroleum ether/EtOAc = 8/1); ^1H NMR (400 MHz, CDCl_3) δ 9.28 (s, 1H), 8.64 (s, 1H), 8.20 (d, $J = 7.7$ Hz, 1H), 7.74 (d, $J = 7.5$ Hz, 2H), 7.71–7.65 (m, 2H), 7.60 (t, $J = 7.4$ Hz, 1H), 7.52 (t, $J = 7.6$ Hz, 1H), 7.48–7.39 (m, 3H), 7.31 (t, $J = 7.4$ Hz, 1H), 7.14–7.06 (m, 1H); ^{13}C NMR (101 MHz, CDCl_3) δ 197.7, 158.7 (d, $J_{\text{C-F}} = 243.8$ Hz), 142.3, 140.7 (d, $J_{\text{C-F}} = 1.6$ Hz), 140.2, 133.1, 129.9, 128.86 (d, $J_{\text{C-F}} = 8.5$ Hz), 128.85, 128.4, 128.1 (d, $J_{\text{C-F}} = 8.4$ Hz), 127.8, 127.2, 123.0 (d, $J_{\text{C-F}} = 5.2$ Hz), 122.2, 121.3, 120.4, 116.2 (d, $J_{\text{C-F}} = 25.3$ Hz), 113.8, 111.8 (d, $J_{\text{C-F}} = 20.3$ Hz), 111.0; HRMS (ESI, m/z) Calculated for $\text{C}_{23}\text{H}_{14}\text{FNO}$ $[\text{M} + \text{H}]^+$ 340.1132, found 340.1136.

(8-methyl-5H-benzo[b]carbazol-6-yl)(phenyl)methanone (4e).

Following the general procedure, the compound was obtained as yellow solid: 60 mg, 60% yield; mp 199–200 °C; $R_f = 0.37$ (petroleum ether/EtOAc = 8/1); ^1H NMR (400

MHz, CDCl₃) δ 9.34 (s, 1H), 8.63 (s, 1H), 8.16 (d, *J* = 7.4 Hz, 1H), 7.94 (d, *J* = 8.2 Hz, 1H), 7.75 (d, *J* = 7.2 Hz, 2H), 7.55 (m, 1H), 7.41 (m, 5H), 7.32–7.11 (m, 2H), 2.28 (s, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 198.2, 142.0, 141.5, 140.5, 135.9, 132.7, 131.3, 129.9, 129.2, 128.7, 127.8, 126.7, 125.5, 125.4, 125.3, 124.1, 122.8, 121.0, 120.3, 112.9, 110.9, 22.2; HRMS (ESI, *m/z*) Calculated for C₂₄H₁₇NO [M + H]⁺ 336.1383, found 336.1396.

(5H-benzo[b]carbazol-6-yl)(p-tolyl)methanone (4f).

Following the general procedure, the compound was obtained as yellow solid: 91 mg, 90% yield; mp 175–176 °C; R_f = 0.4 (petroleum ether/EtOAc = 8/1); ¹H NMR (400 MHz, CDCl₃) δ 9.26 (s, 1H), 8.68 (s, 1H), 8.18 (d, *J* = 7.7 Hz, 1H), 8.05 (d, *J* = 8.1 Hz, 1H), 7.73 (d, *J* = 8.5 Hz, 1H), 7.66 (d, *J* = 7.9 Hz, 2H), 7.47 (t, *J* = 7.6 Hz, 1H), 7.41–7.24 (m, 4H), 7.20 (d, *J* = 7.8 Hz, 2H), 2.41 (s, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 197.7, 143.8, 142.1, 141.0, 137.6, 131.0, 130.2, 129.5, 129.3, 128.3, 128.0, 126.1, 126.1, 125.9, 123.8, 123.1, 122.7, 121.2, 120.3, 113.9, 111.0, 21.9; HRMS (ESI, *m/z*) Calculated for C₂₄H₁₇NO [M + H]⁺ 336.1383, found 336.1392.

(5H-benzo[b]carbazol-6-yl)(4-methoxyphenyl)methanone (4g).

Following the general procedure, the compound was obtained as yellow solid: 64 mg, 61% yield; mp 160–161 °C; R_f = 0.38 (petroleum ether/EtOAc = 8/1); ¹H NMR (400 MHz, CDCl₃) δ 9.09 (s, 1H), 8.68 (s, 1H), 8.20 (d, *J* = 7.7 Hz, 1H), 8.07 (d, *J* = 7.9 Hz, 1H), 7.77 (d, *J* = 8.3 Hz, 3H), 7.48 (t, *J* = 7.4 Hz, 1H), 7.39–7.37 (m, 2H), 7.35–7.23 (m, 2H), 6.89 (d, *J* = 8.4 Hz, 2H), 3.86 (s, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 196.6, 163.8, 142.2, 140.6, 132.7, 132.6, 130.9, 129.3, 128.3, 128.0, 126.1,

126.0, 125.9, 123.3, 123.1, 122.8, 121.2, 120.3, 114.2, 114.1, 110.9, 55.6; HRMS (ESI, m/z) Calculated for $C_{24}H_{17}NO_2$ $[M + H]^+$ 352.1332, found 352.1335.

(5H-benzo[b]carbazol-6-yl)(4-Chlorophenyl)methanone (4h).

Following the general procedure, the compound was obtained as yellow solid: 99 mg, 93% yield; mp 204–205 °C; R_f = 0.53 (petroleum ether/EtOAc = 8/1); 1H NMR (400 MHz, $CDCl_3$) δ 9.49 (s, 1H), 8.66 (s, 1H), 8.16 (d, J = 7.5 Hz, 1H), 8.03 (d, J = 8.0 Hz, 1H), 7.64 (m, 3H), 7.48 (t, J = 7.4 Hz, 1H), 7.37 (m, 4H), 7.28 (t, J = 7.1 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.7, 142.1, 141.6, 139.2, 138.8, 131.3, 130.8, 129.5, 129.1, 128.3, 128.2, 126.3, 126.1, 126.0, 124.6, 123.2, 122.6, 121.2, 120.5, 112.8, 111.1; HRMS (ESI, m/z) Calculated for $C_{23}H_{14}ClNO$ $[M + H]^+$ 356.0837, found 356.0837.

(4-chlorophenyl)(1-methoxy-5H-benzo[b]carbazol-6-yl)methanone (4i).

Following the general procedure, the compound was obtained as yellow solid: 64 mg, 55% yield; mp 233–235 °C; R_f = 0.28 (petroleum ether/EtOAc = 8/1); 1H NMR (400 MHz, $CDCl_3$) δ 9.56 (s, 1H), 8.95 (s, 1H), 8.10 (d, J = 8.1 Hz, 1H), 7.72–7.60 (m, 3H), 7.48–7.34 (m, 4H), 7.30 (t, J = 7.5 Hz, 1H), 7.05 (d, J = 8.0 Hz, 1H), 6.78 (d, J = 8.1 Hz, 1H), 4.17 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.8, 157.0, 143.5, 141.3, 139.03, 139.00, 131.3, 130.3, 129.6, 129.2, 129.0, 128.8, 127.6, 125.9, 125.8, 125.4, 123.0, 112.3, 111.5, 103.9, 102.0, 55.7; HRMS (ESI, m/z) Calculated for $C_{24}H_{16}ClNO_2$ $[M + H]^+$ 386.0942, found 386.0948.

(4-chlorophenyl)(2-methyl-5H-benzo[b]carbazol-6-yl)methanone (4j).

Following the general procedure, the compound was obtained as yellow solid: 107 mg,

96% yield; mp 205–206 °C; $R_f = 0.3$ (petroleum ether/EtOAc = 8/1); ^1H NMR (400 MHz, CDCl_3) δ 9.40 (s, 1H), 8.64 (s, 1H), 8.03 (d, $J = 7.8$ Hz, 1H), 7.97 (s, 1H), 7.64–7.61 (m, 3H), 7.38–7.36 (d, $J = 7.6$ Hz, 3H), 7.29 (m, 3H), 2.54 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 196.7, 142.0, 140.2, 139.1, 138.9, 131.3, 130.8, 123.0, 129.5, 129.4, 129.1, 128.21, 126.3, 126.04, 125.97, 124.5, 123.1, 122.8, 121.3, 112.7, 110.7, 21.5; HRMS (ESI, m/z) Calculated for $[\text{C}_{24}\text{H}_{16}\text{ClNO} + \text{H}]^+$ 370.0993, found 370.0993.

(4-chlorophenyl)(2-methoxy-5H-benzo[b]carbazol-6-yl)methanone (4k).

Following the general procedure, the compound was obtained as yellow solid: 87 mg, 75% yield; mp 190–191 °C; $R_f = 0.15$ (petroleum ether/EtOAc = 8/1); ^1H NMR (400 MHz, CDCl_3) δ 9.39 (s, 1H), 8.63 (s, 1H), 8.02 (d, $J = 8.0$ Hz, 1H), 7.71–7.56 (m, 4H), 7.41–7.32 (m, 3H), 7.31–7.21 (m, 2H), 7.10 (dd, $J = 8.5, 1.7$ Hz, 1H); ^{13}C NMR (101 MHz, CDCl_3) δ 196.6, 154.7, 142.2, 139.1, 138.9, 136.7, 131.3, 130.9, 129.5, 129.0, 128.0, 126.4, 126.1, 125.9, 124.7, 123.2, 123.0, 116.6, 112.7, 111.7, 104.8, 56.3; HRMS (ESI, m/z) Calculated for $\text{C}_{24}\text{H}_{16}\text{ClNO}_2$ $[\text{M} + \text{H}]^+$ 386.0942, found 386.0942.

(2-bromo-5H-benzo[b]carbazol-6-yl)(4-chlorophenyl)methanone (4l).

Following the general procedure, the compound was obtained as yellow solid: 128 mg, 98% yield; mp 261 – 262 °C; $R_f = 0.4$ (petroleum ether/EtOAc = 8/1); ^1H NMR (400 MHz, DMSO) δ 11.29 (s, 1H), 9.02 (s, 1H), 8.57 (s, 1H), 8.15 (dd, $J = 6.6, 2.7$ Hz, 1H), 7.76 (d, $J = 8.4$ Hz, 2H), 7.66–7.53 (m, 4H), 7.51–7.37 (m, 3H); ^{13}C NMR (101 MHz, DMSO) δ 195.4, 141.5, 138.7, 138.0, 136.6, 131.5, 130.1, 129.5, 129.2, 127.3, 126.4, 124.3, 124.0, 123.7, 122.9, 122.8, 114.2, 113.1, 111.2; HRMS (ESI, m/z)

Calculated for $C_{23}H_{13}BrClNO$ $[M + H]^+$ 433.9942, found 433.9944.

(3-chloro-5H-benzo[b]carbazol-6-yl)(4-chlorophenyl)methanone (4m).

Following the general procedure, the compound was obtained as yellow solid: 89 mg, 76% yield; mp 199–201 °C; $R_f = 0.5$ (petroleum ether/EtOAc = 8/1); 1H NMR (400 MHz, $CDCl_3$) δ 9.48 (s, 1H), 8.57 (s, 1H), 8.00 (d, $J = 8.0$ Hz, 2H), 7.71–7.54 (m, 3H), 7.41–7.25 (m, 5H), 7.22 (d, $J = 8.1$ Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.6, 142.7, 141.6, 139.4, 138.6, 133.8, 131.3, 130.76, 129.4, 129.1, 128.5, 126.3, 126.0, 125.4, 124.5, 123.5, 121.9, 121.2, 121.0, 113.2, 111.2; HRMS (ESI, m/z)

Calculated for $C_{23}H_{13}Cl_2NO$ $[M + H]^+$ 390.0447, found 390.0439.

(4-chlorophenyl)(4-methyl-5H-benzo[b]carbazol-6-yl) methanone (4n).

Following the general procedure, the compound was obtained as yellow solid: 89 mg, 80% yield; mp 164–166 °C; $R_f = 0.55$ (petroleum ether/EtOAc = 8/1); 1H NMR (400 MHz, $CDCl_3$) δ 9.41 (s, 1H), 8.66 (s, 1H), 8.03 (t, $J = 7.6$ Hz, 2H), 7.66 (d, $J = 8.4$ Hz, 2H), 7.62 (d, $J = 8.6$ Hz, 1H), 7.42–7.34 (m, 3H), 7.33–7.25 (m, 2H), 7.21 (t, $J = 7.7$ Hz, 1H), 2.53 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.9, 141.7, 141.3, 139.1, 139.0, 131.3, 130.8, 129.5, 129.1, 128.9, 128.4, 126.8, 126.1, 124.8, 123.2, 122.2, 120.7, 120.4, 118.7, 112.8, 16.9; HRMS (ESI, m/z) Calculated for $C_{24}H_{16}ClNO$ $[M + H]^+$ 370.0993, found 370.0995.

10-Chloro-6-cyclopropyl-7-iodo-5,12-dihydrobenzo[4,5]cyclohepta[1,2-b]indoleyl-1-2-diethyl malonate (5)

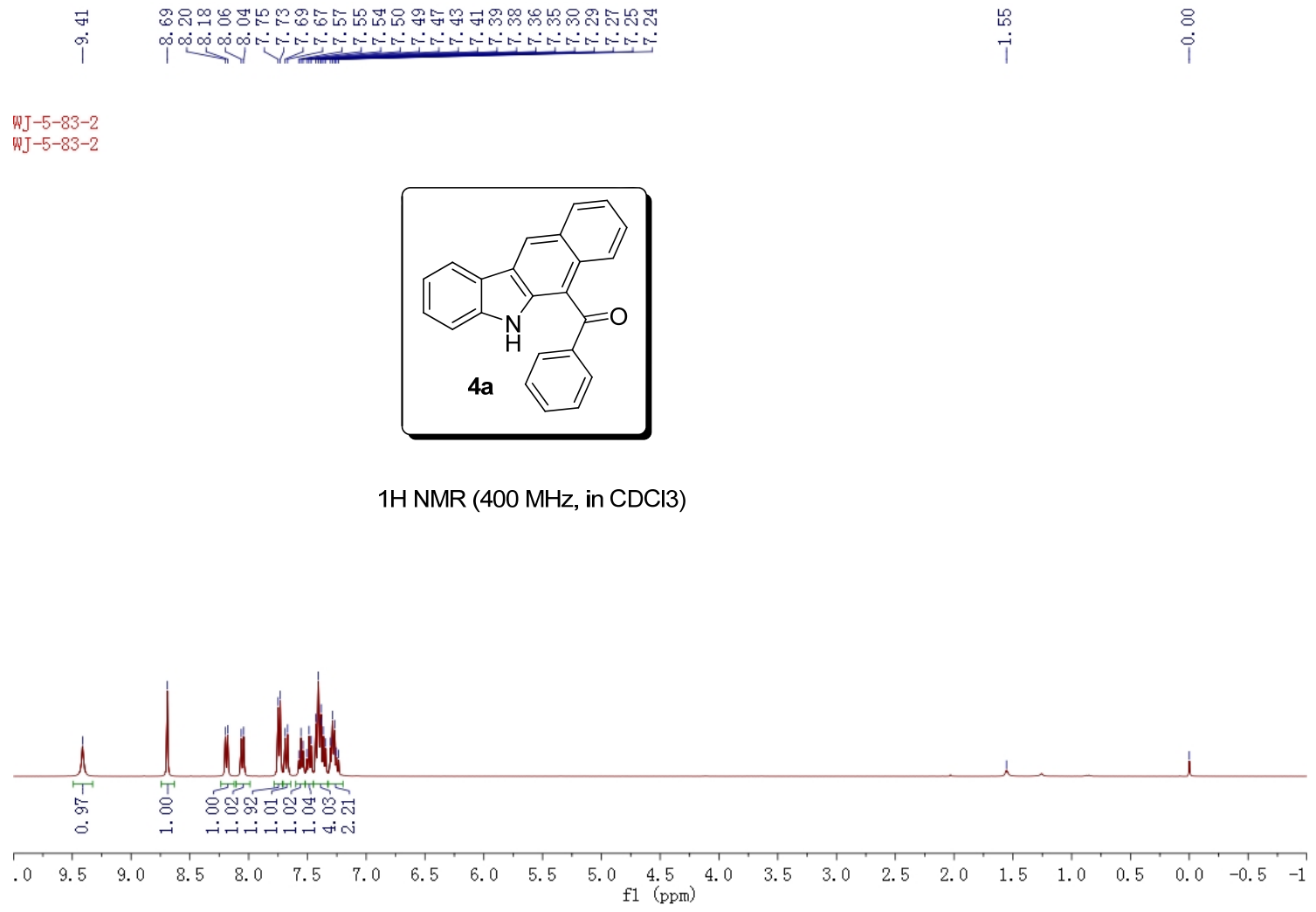
The compound was obtained as brown oil: 177 mg, 41% yield; $R_f = 0.2$ (petroleum ether/EtOAc = 8/1); 1H NMR (400 MHz, $CDCl_3$) δ 8.18 (s, 1H), 7.87–7.71 (m, 2H),

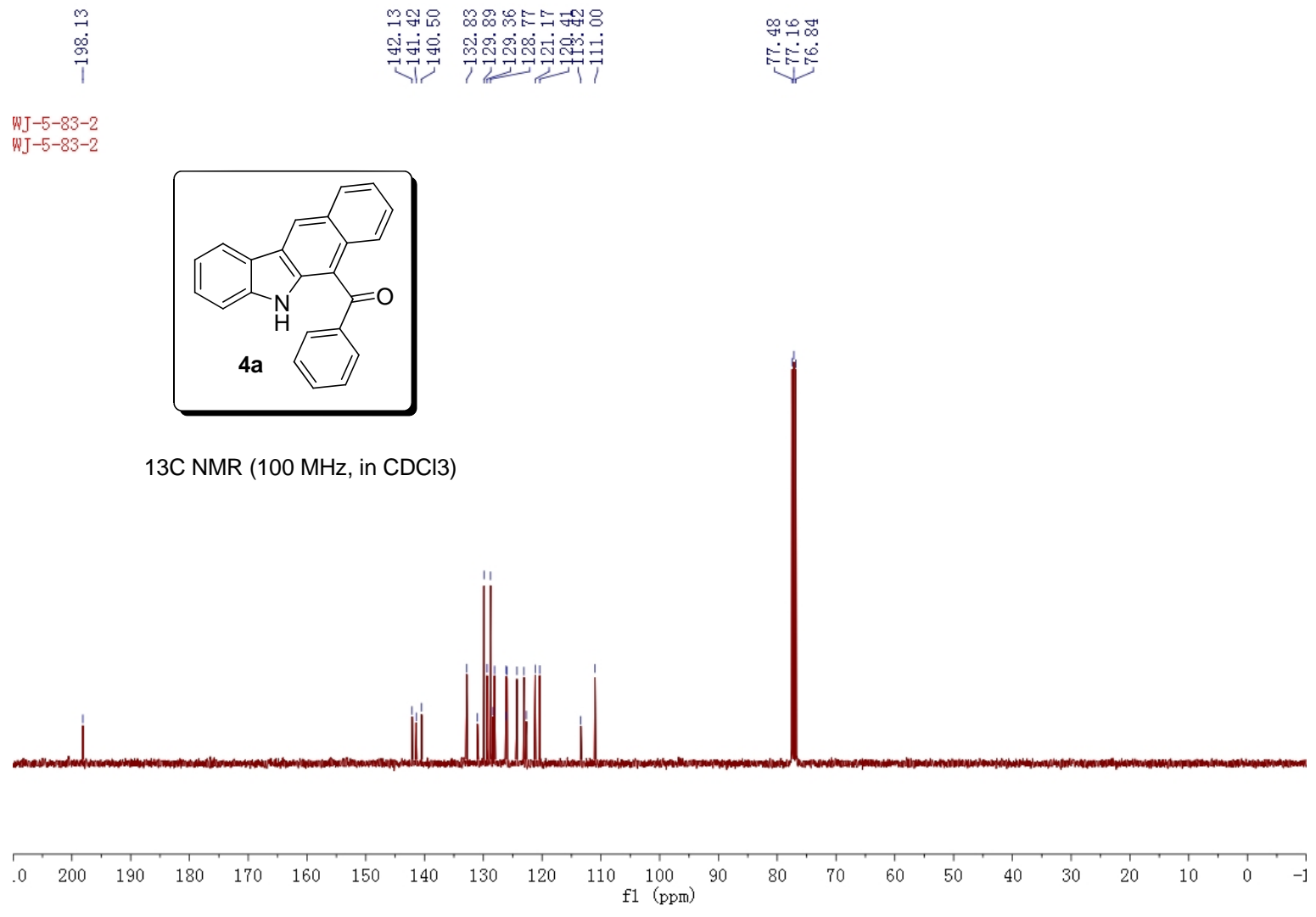
7.32–7.23 (m, 2H), 7.21–7.16 (m, 1H), 7.16–7.10 (m, 2H), 5.04 (d, $J = 11.6$ Hz, 1H), 4.12–3.95 (m, 2H), 3.76 (m, 3H), 2.08–1.94 (m, 1H), 1.35–1.24 (m, 1H), 1.19–1.11 (m, 1H), 1.11–1.03 (m, 3H), 0.87–0.79 (m, 4H), 0.73–0.60 (m, 1H); ^{13}C NMR (101 MHz, CDCl_3) δ 167.9, 167.2, 141.3, 139.2, 137.2, 136.5, 135.0, 130.7, 128.4, 126.1, 125.8, 123.6, 120.3, 118.8, 117.5, 110.9, 107.9, 61.6, 61.4, 50.9, 42.0, 24.3, 14.0, 13.7, 11.1, 10.7. HRMS (ESI, m/z) Calculated for $\text{C}_{27}\text{H}_{26}\text{ClINO}_4$ $[\text{M} + \text{H}]^+$ 590.0590, found 590.0580.

(5H-benzo[b]carbazol-6-yl)(phenyl)methanone with O^{18} label (4a').

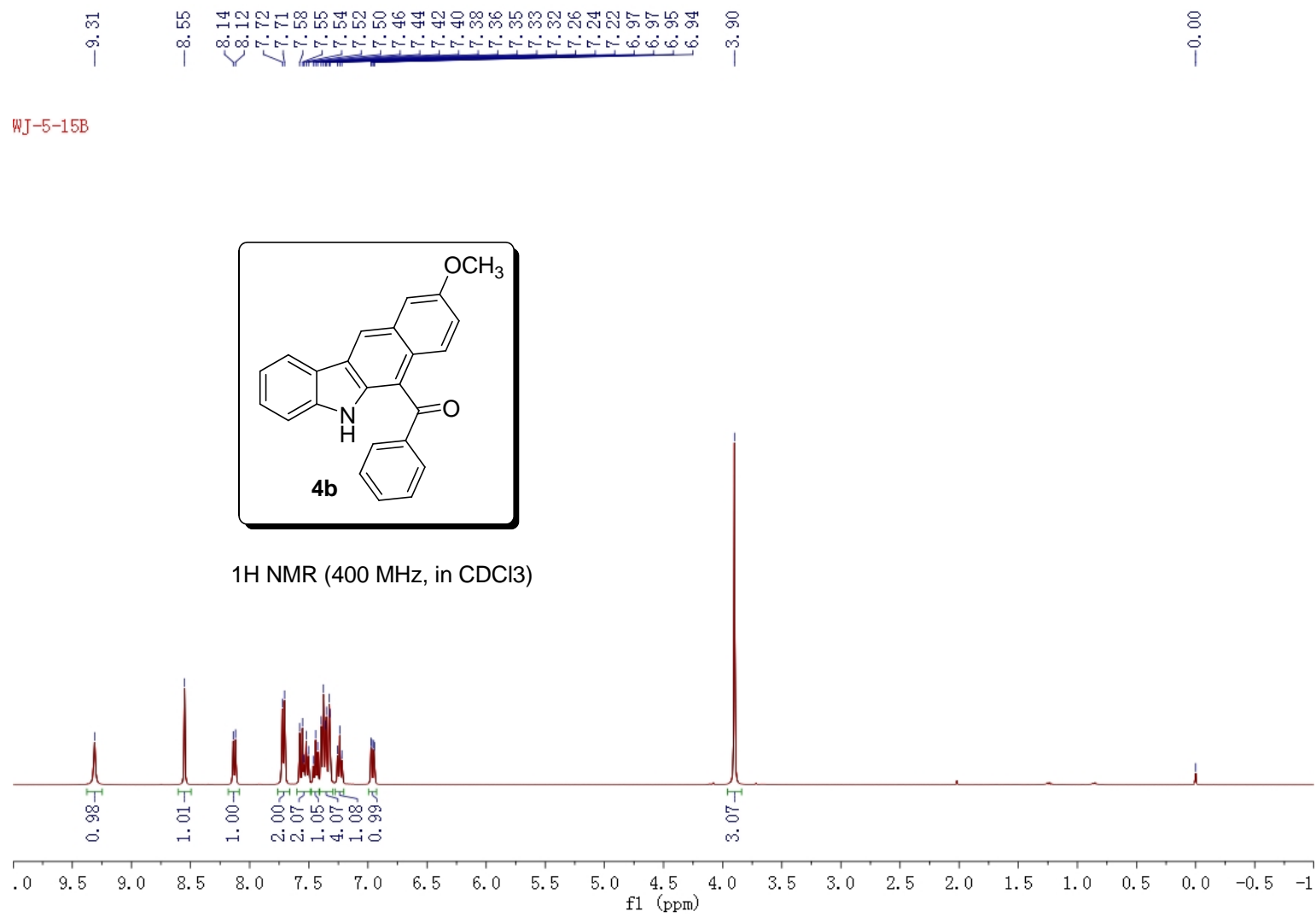
HRMS (ESI, m/z) Calculated for $\text{C}_{23}\text{H}_{15}\text{NO}^{18}$ $[\text{M} + \text{H}]^+$ 324.1269, found 324.1278

1. NMR Spectra (^1H , ^{13}C)

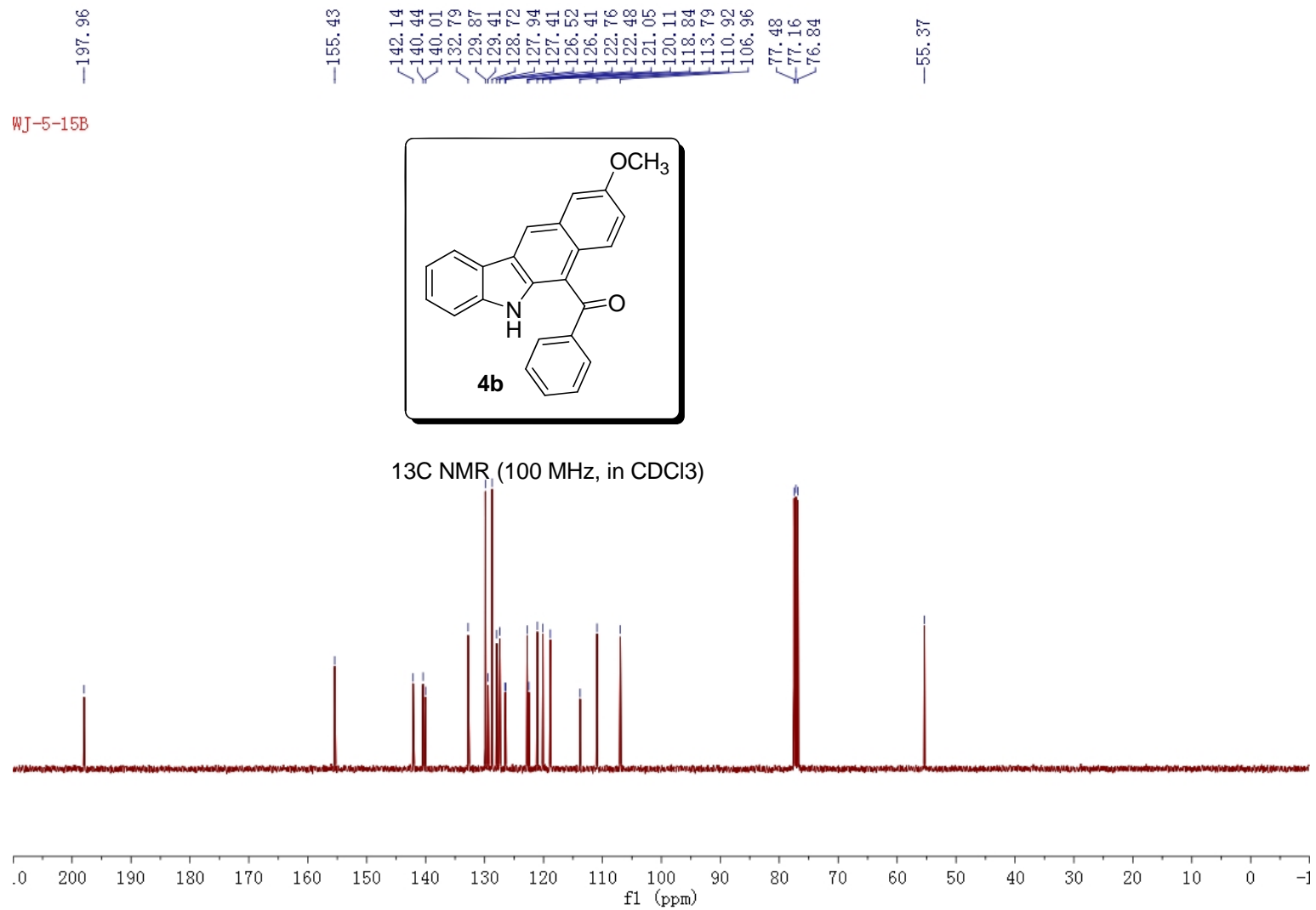




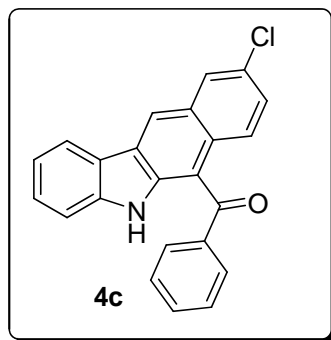
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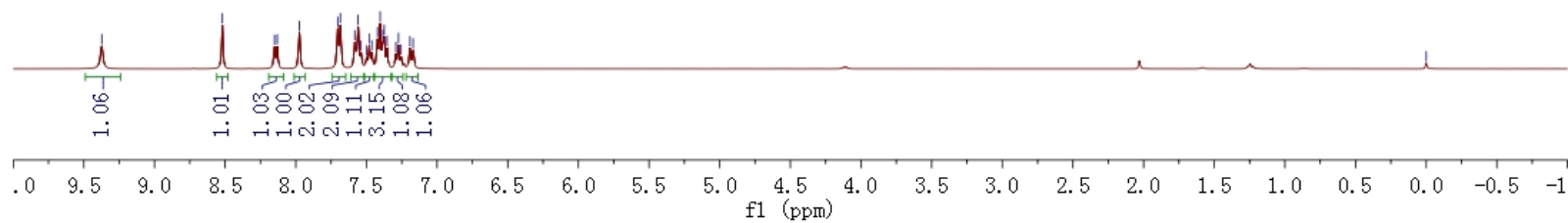
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WJ-6-15C



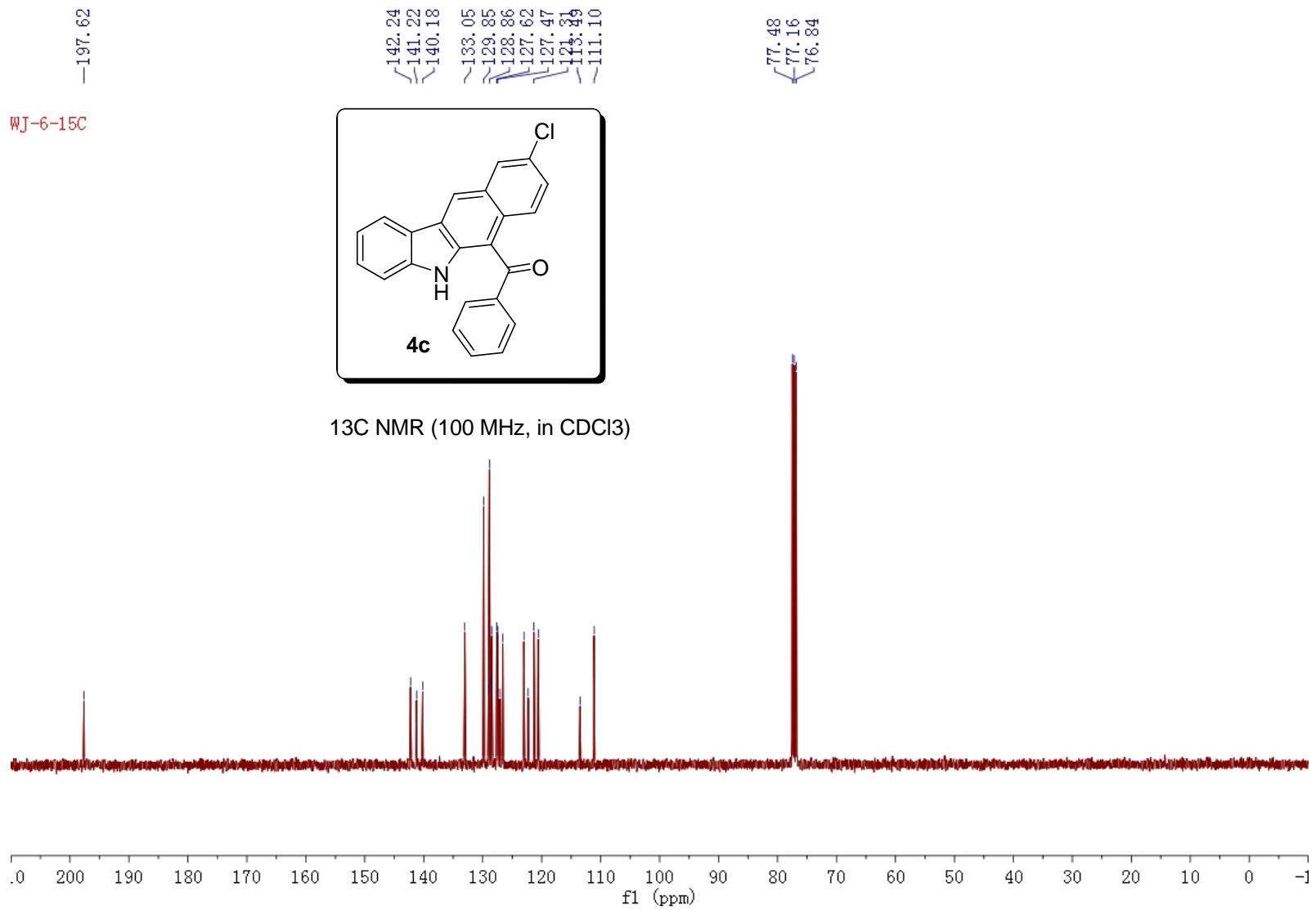
¹H NMR (400 MHz, in CDCl₃)



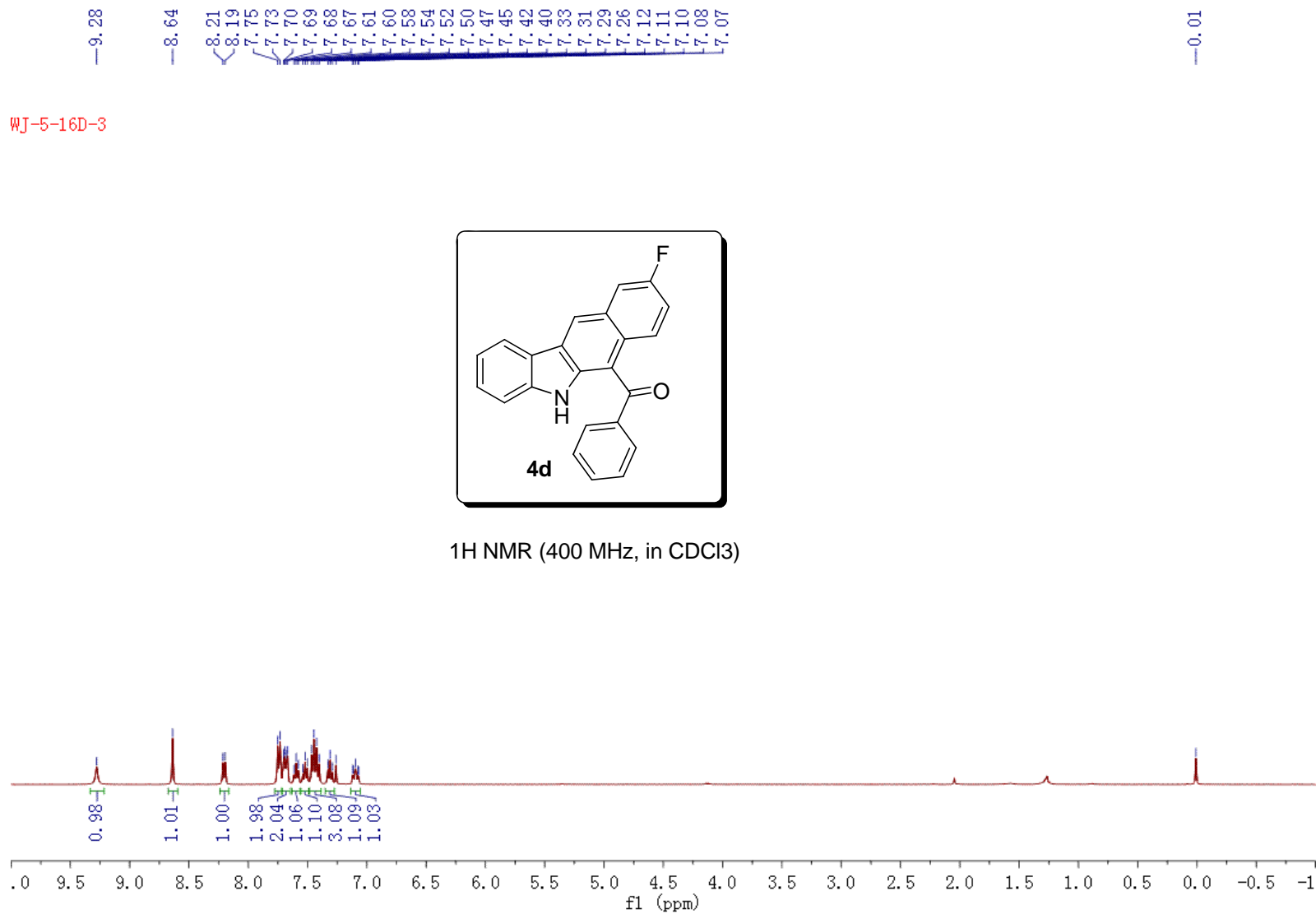
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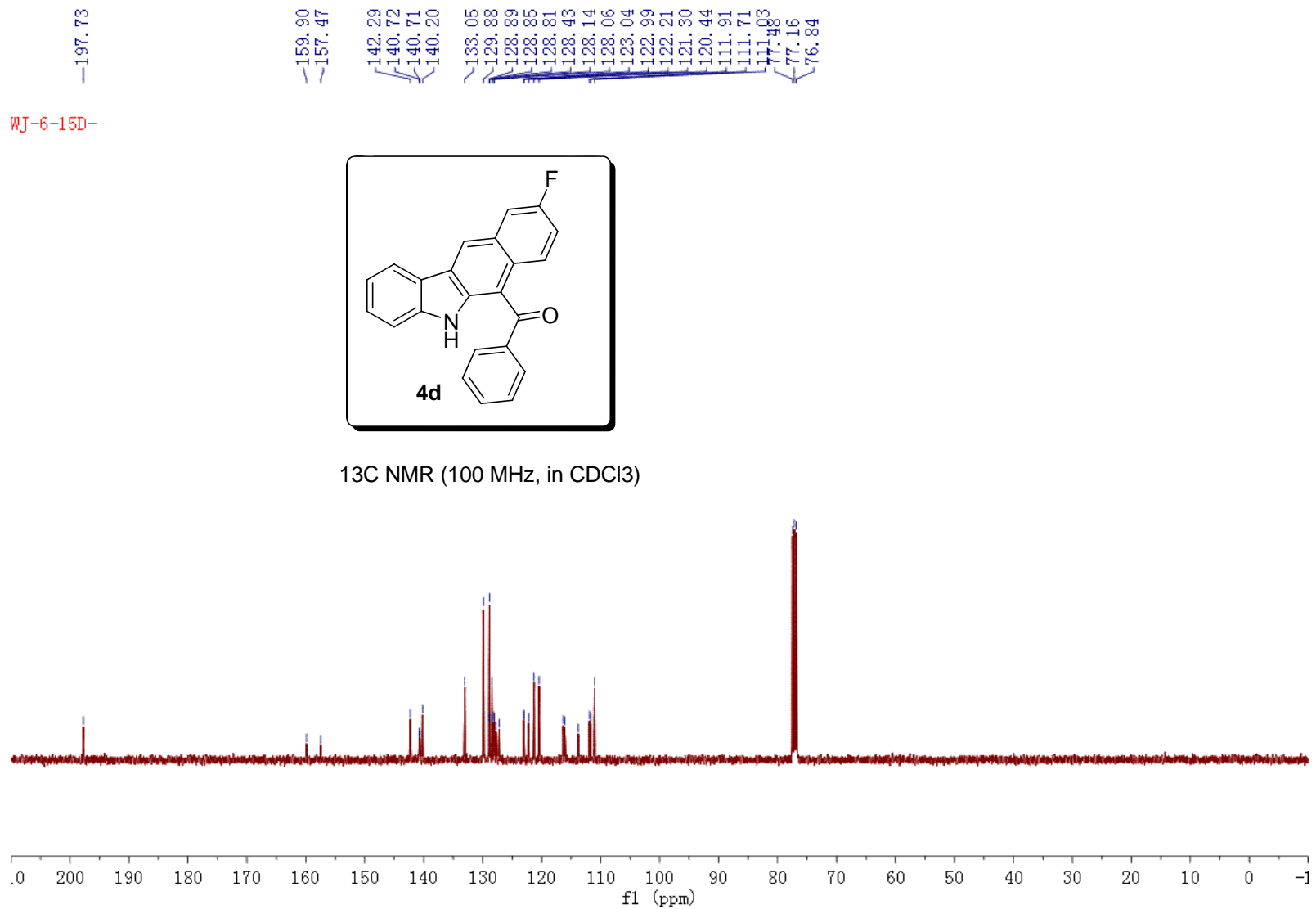
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7.68
7.58
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7.35
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7.25
7.19
7.17

0.00

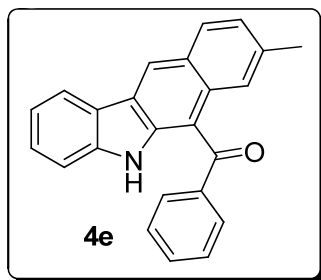


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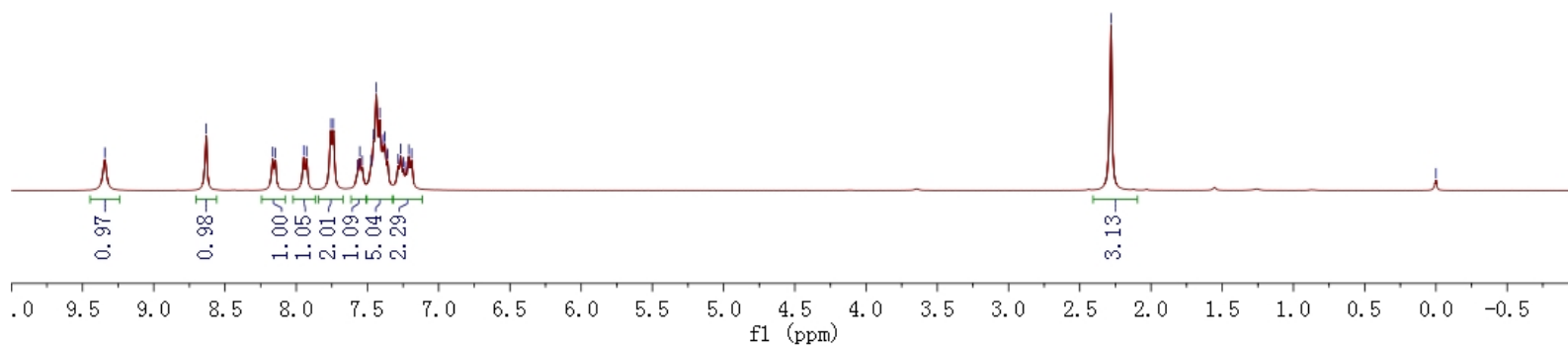




WJ-6-15A



¹H NMR (400 MHz, in CDCl₃)

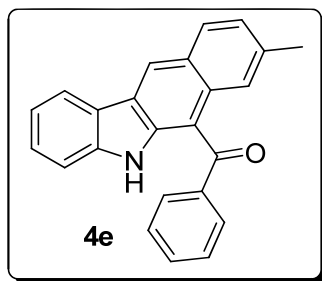


9.34
8.63
8.17
8.15
7.95
7.93
7.76
7.74
7.57
7.55
7.54
7.47
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7.19

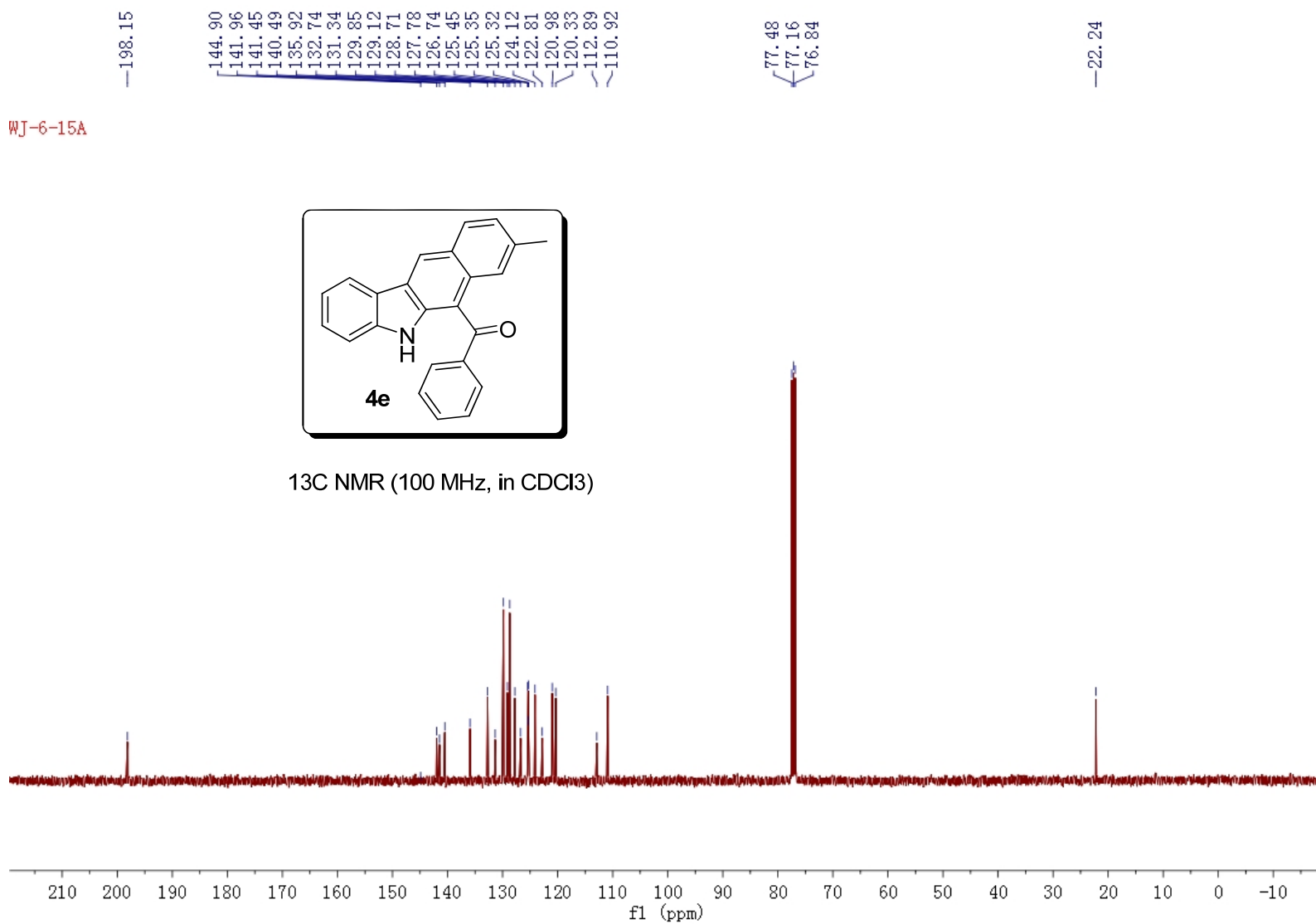
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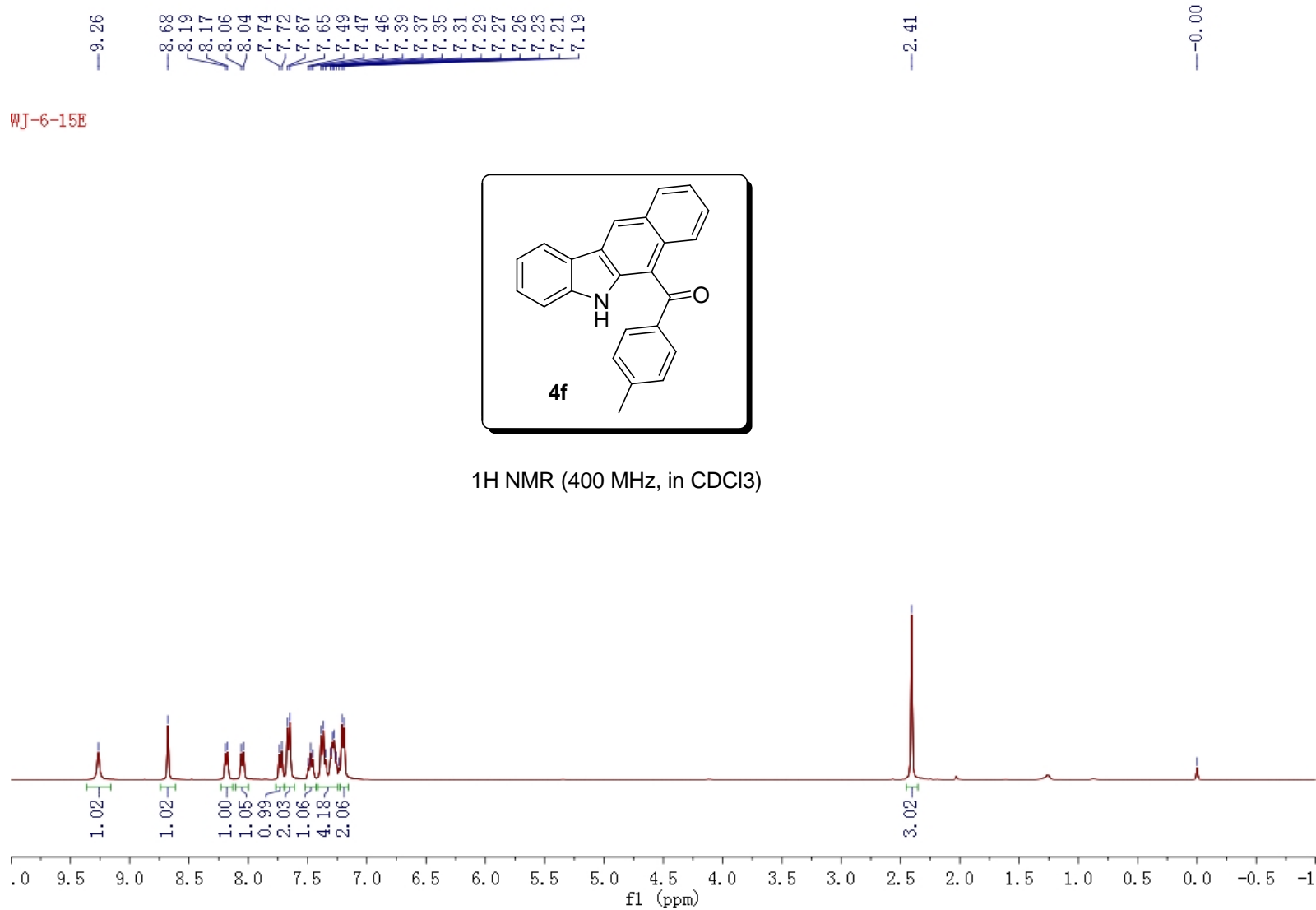
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¹³C NMR (100 MHz, in CDCl₃)



WJ-6-15E



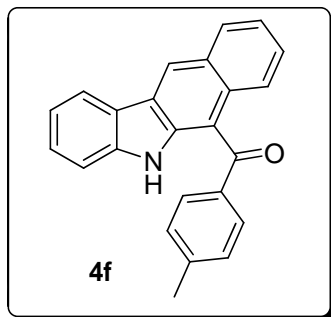
WJ-6-15E

—197.74

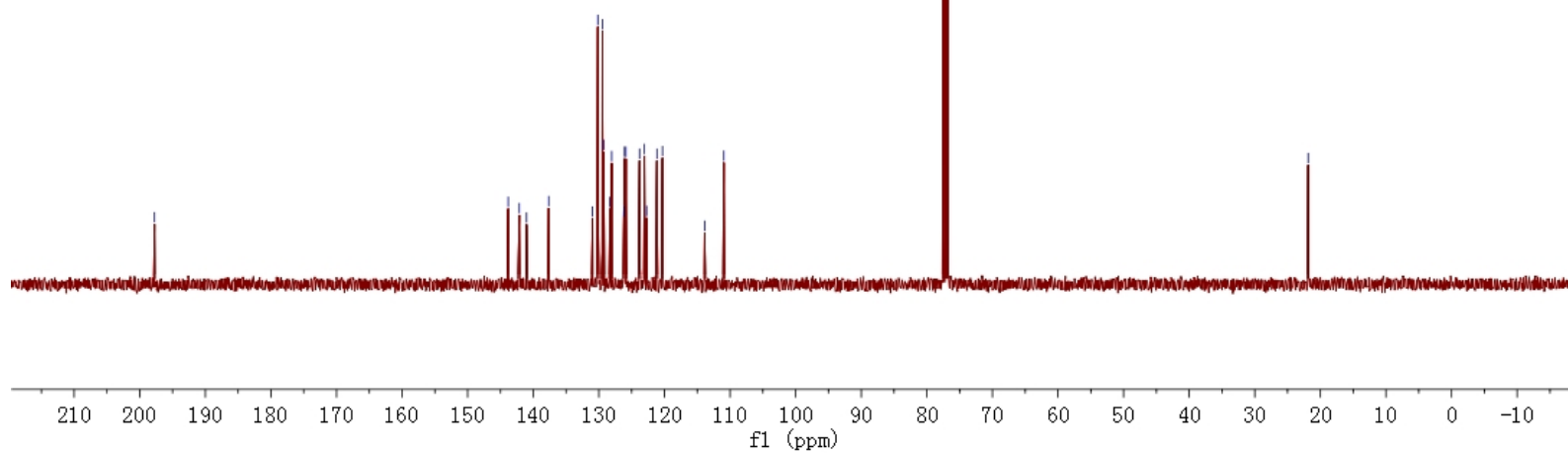
143.82
142.14
141.03
137.64
130.97
130.16
129.47
129.28
128.33
128.04
126.14
126.09
125.88
123.80
123.05
122.72
121.15
120.30
113.87
110.95

77.48
77.16
76.84

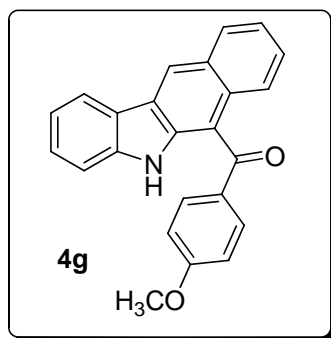
—21.86



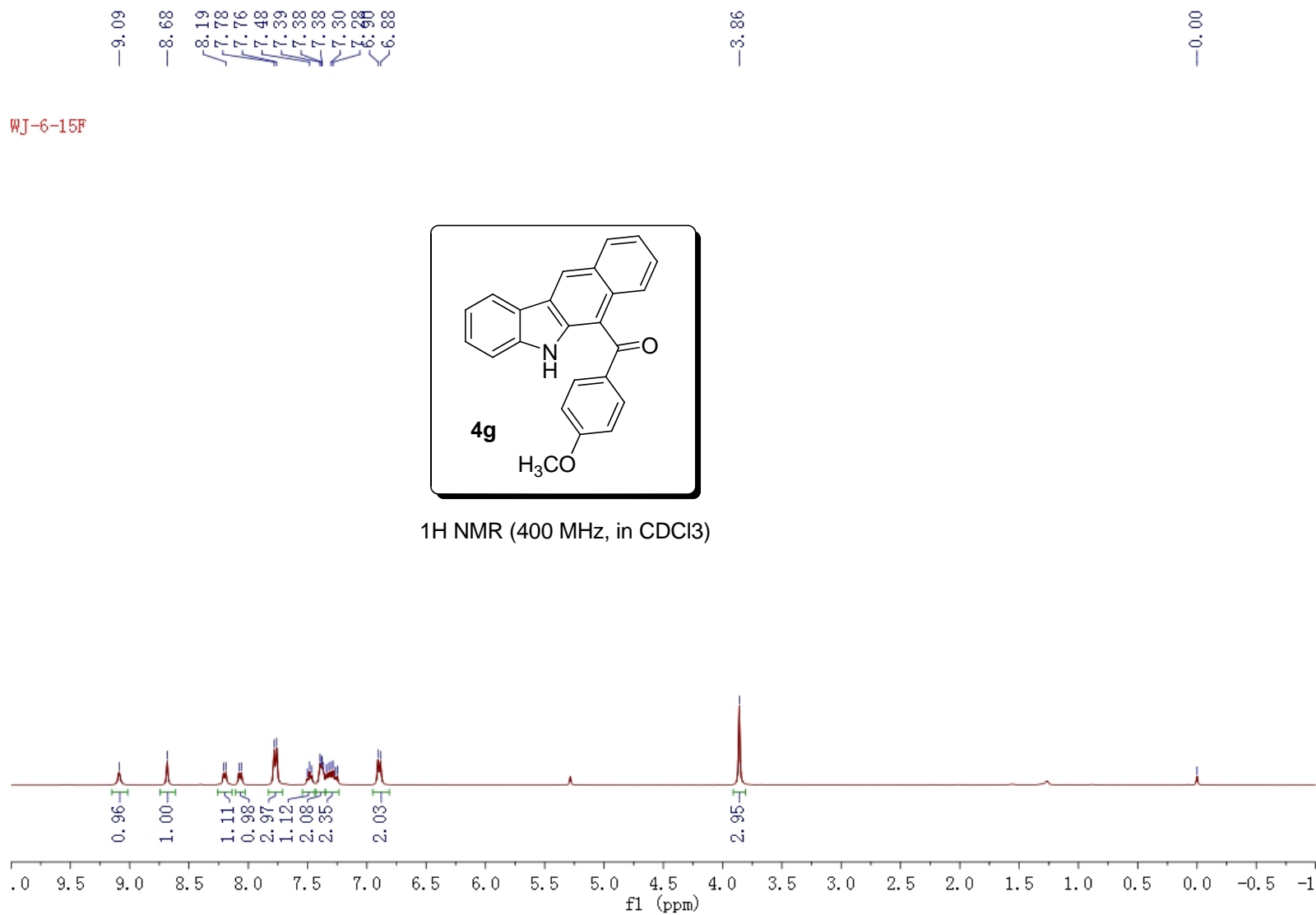
¹³C NMR (100 MHz, in CDCl₃)

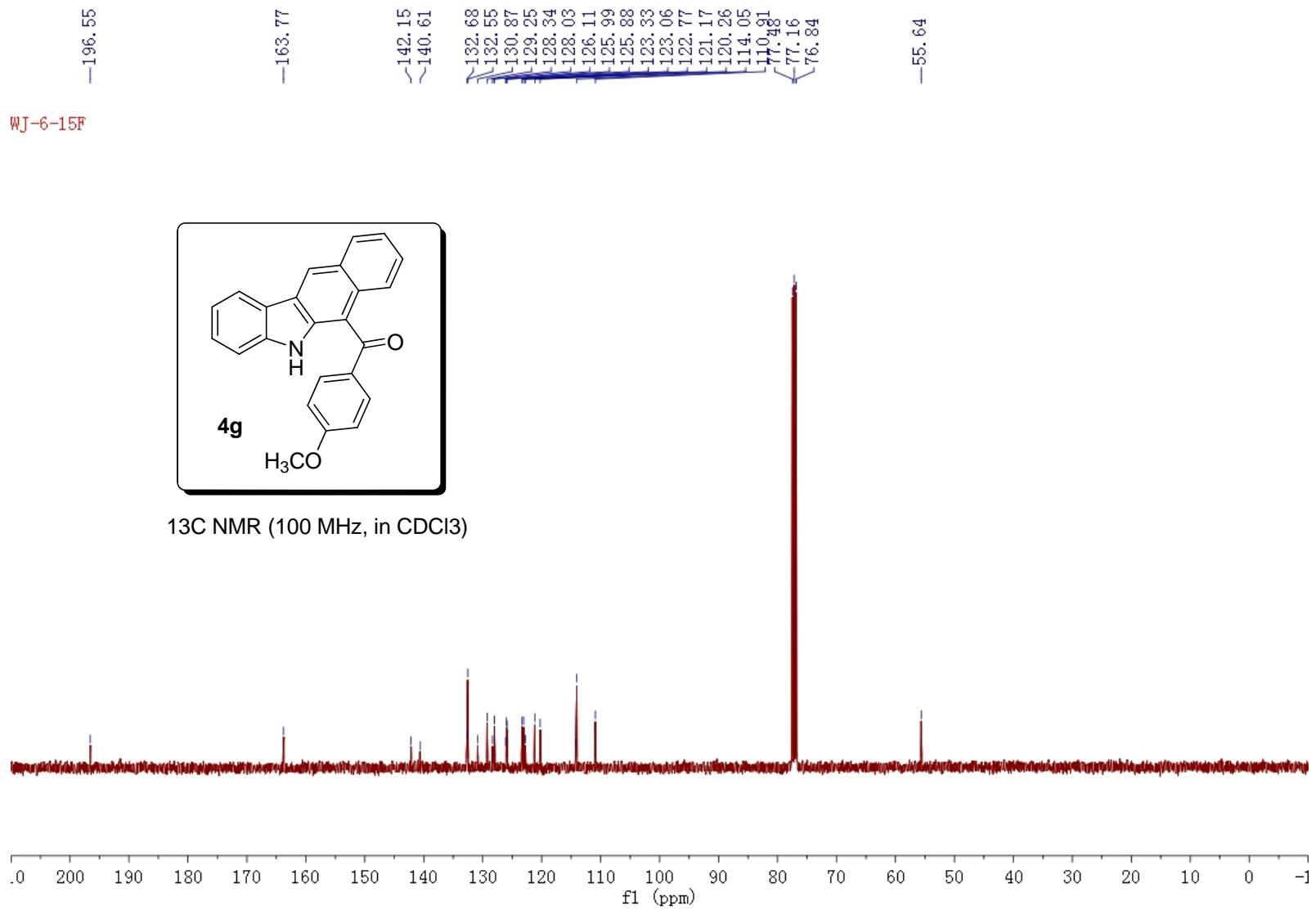


WJ-6-15F

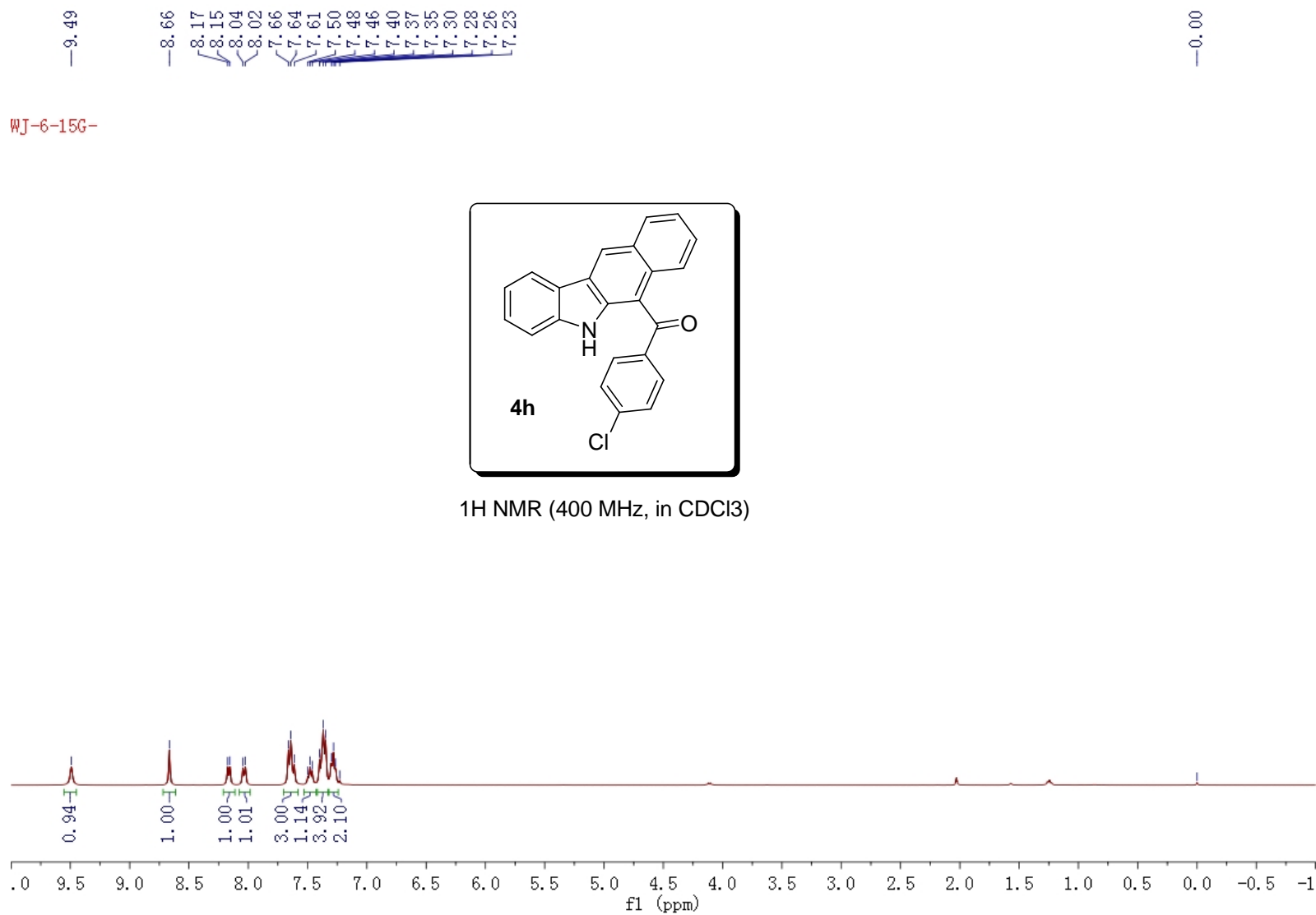


¹H NMR (400 MHz, in CDCl₃)





WJ-6-15G-

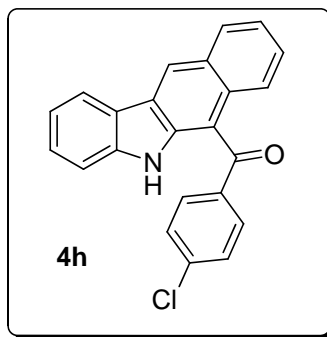


WJ-6-15G-

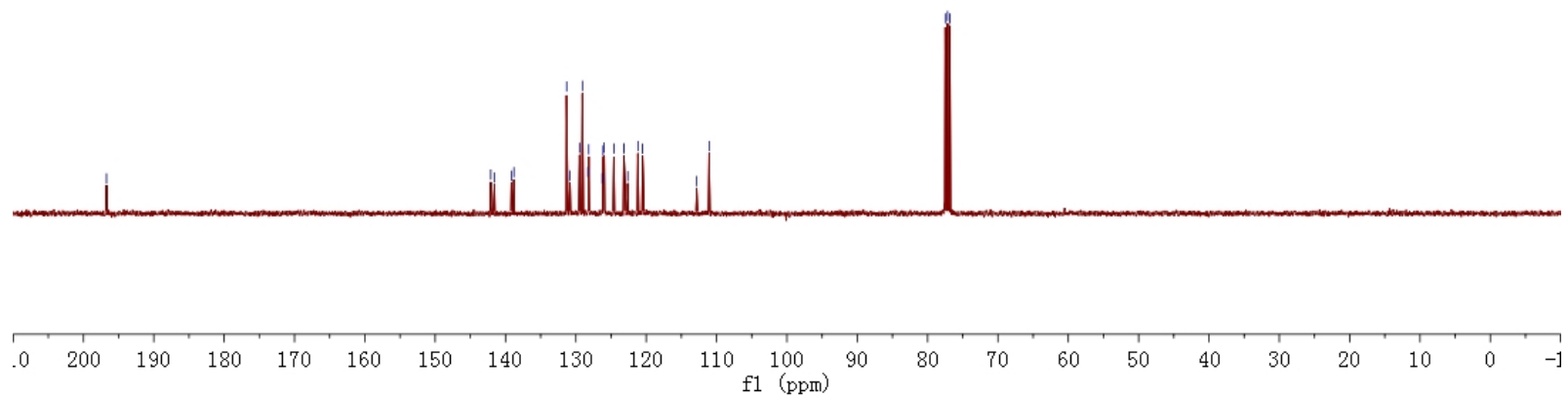
196.71

142.10
141.57
139.17
138.80
131.31
129.47
129.07
125.97
123.17
121.83
111.05

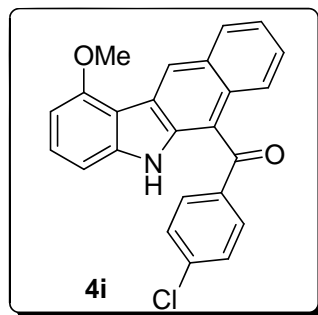
77.48
77.16
76.84



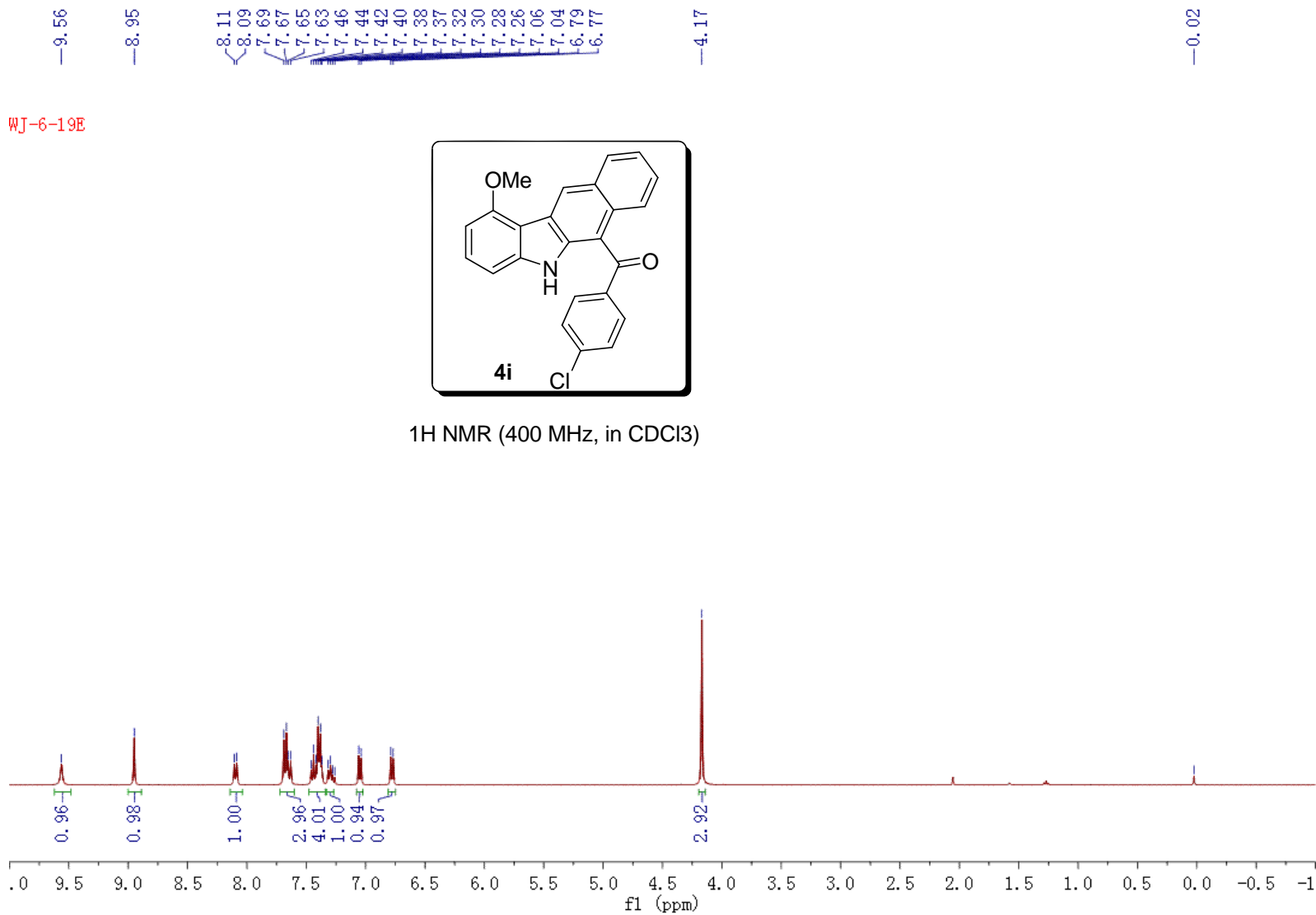
¹³C NMR (100 MHz, in CDCl₃)



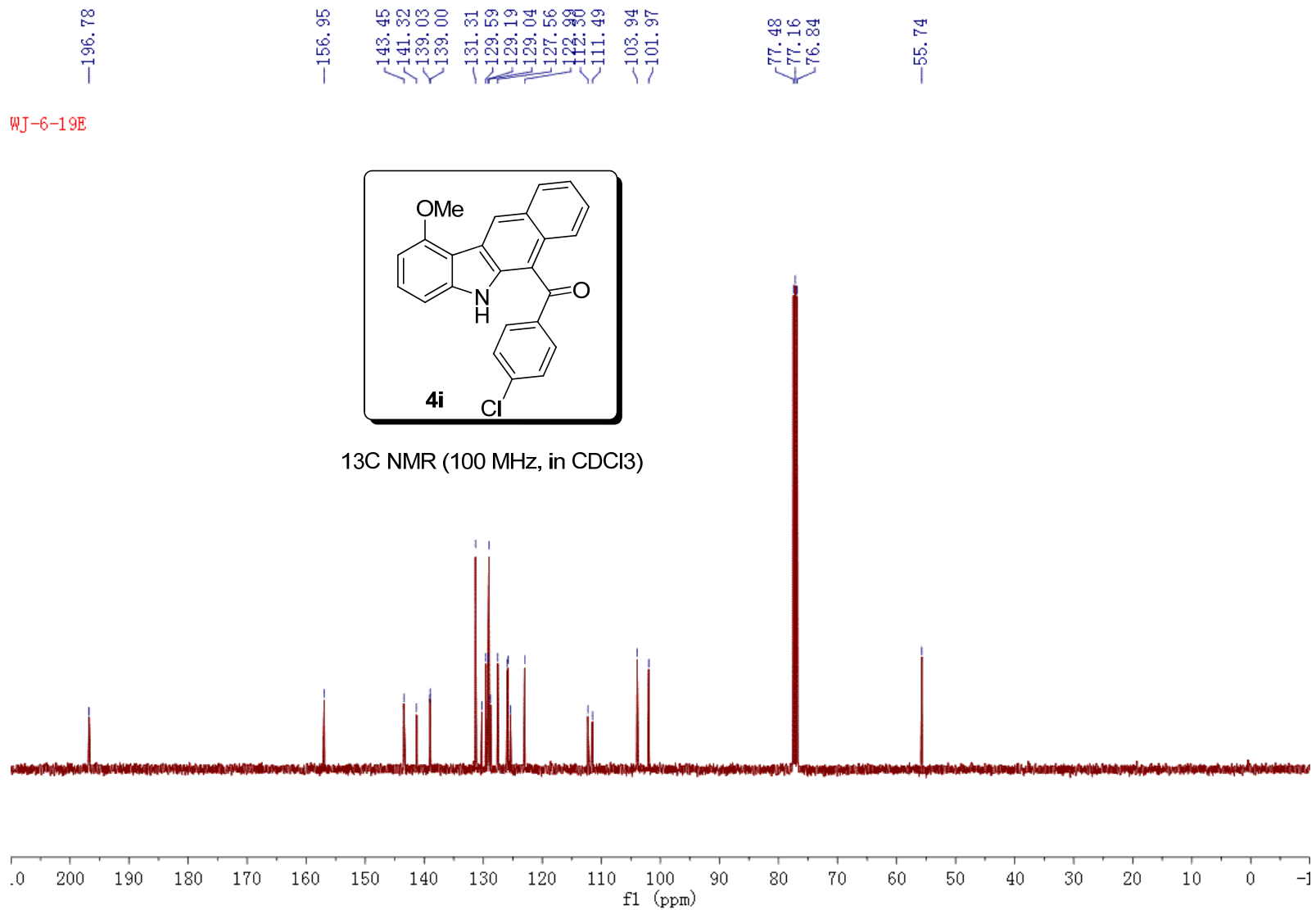
WJ-6-19E



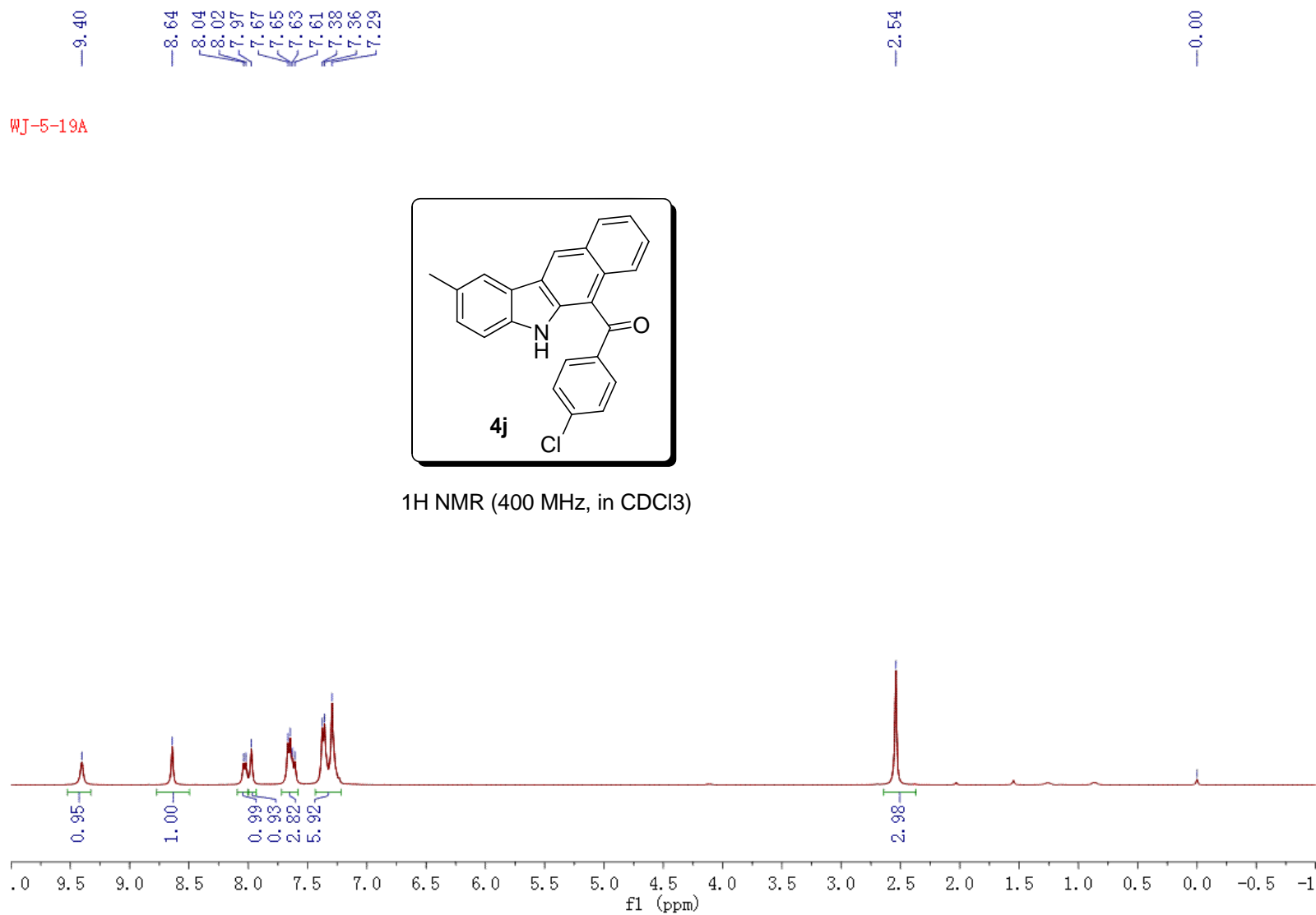
¹H NMR (400 MHz, in CDCl₃)

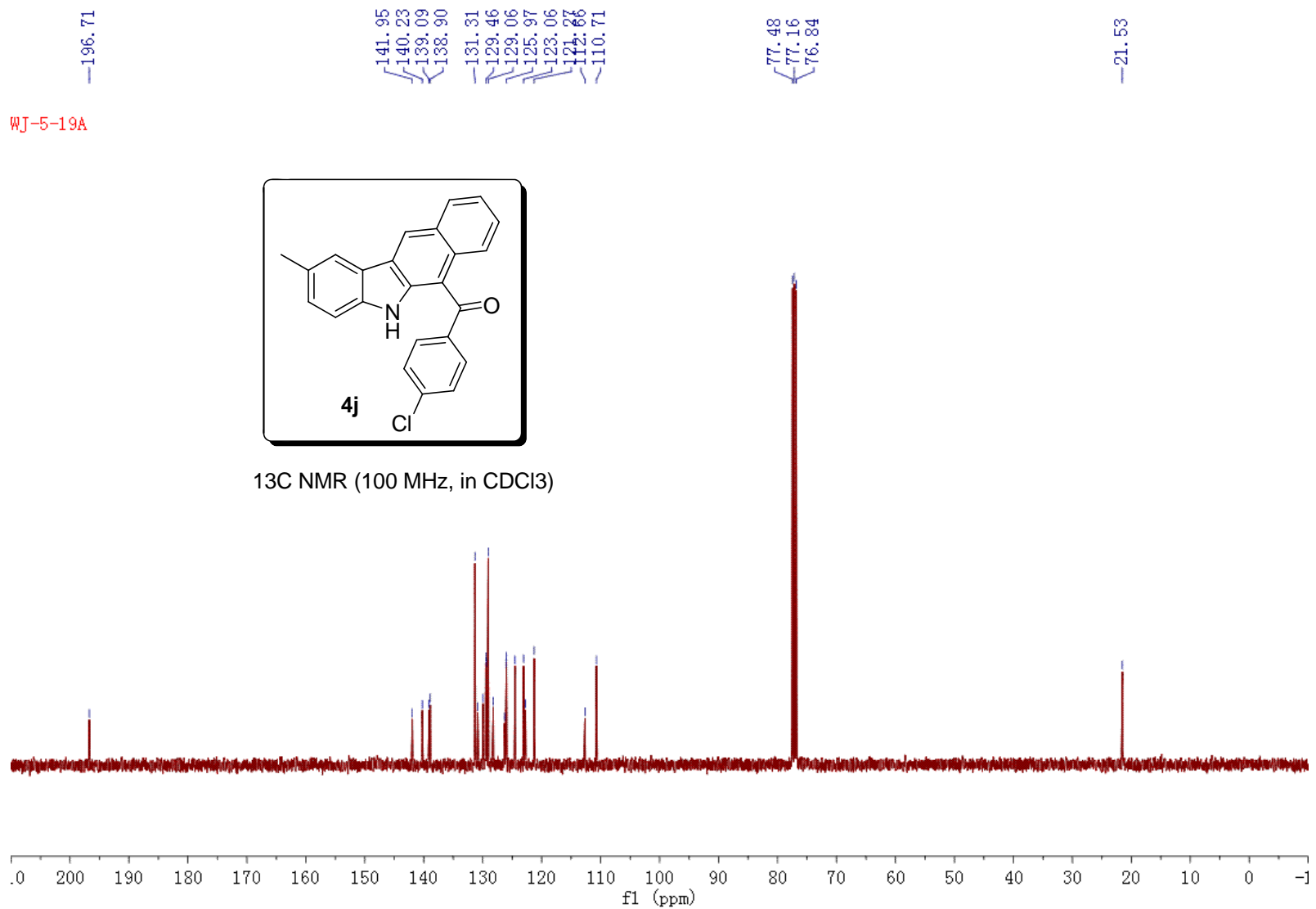


WJ-6-19E

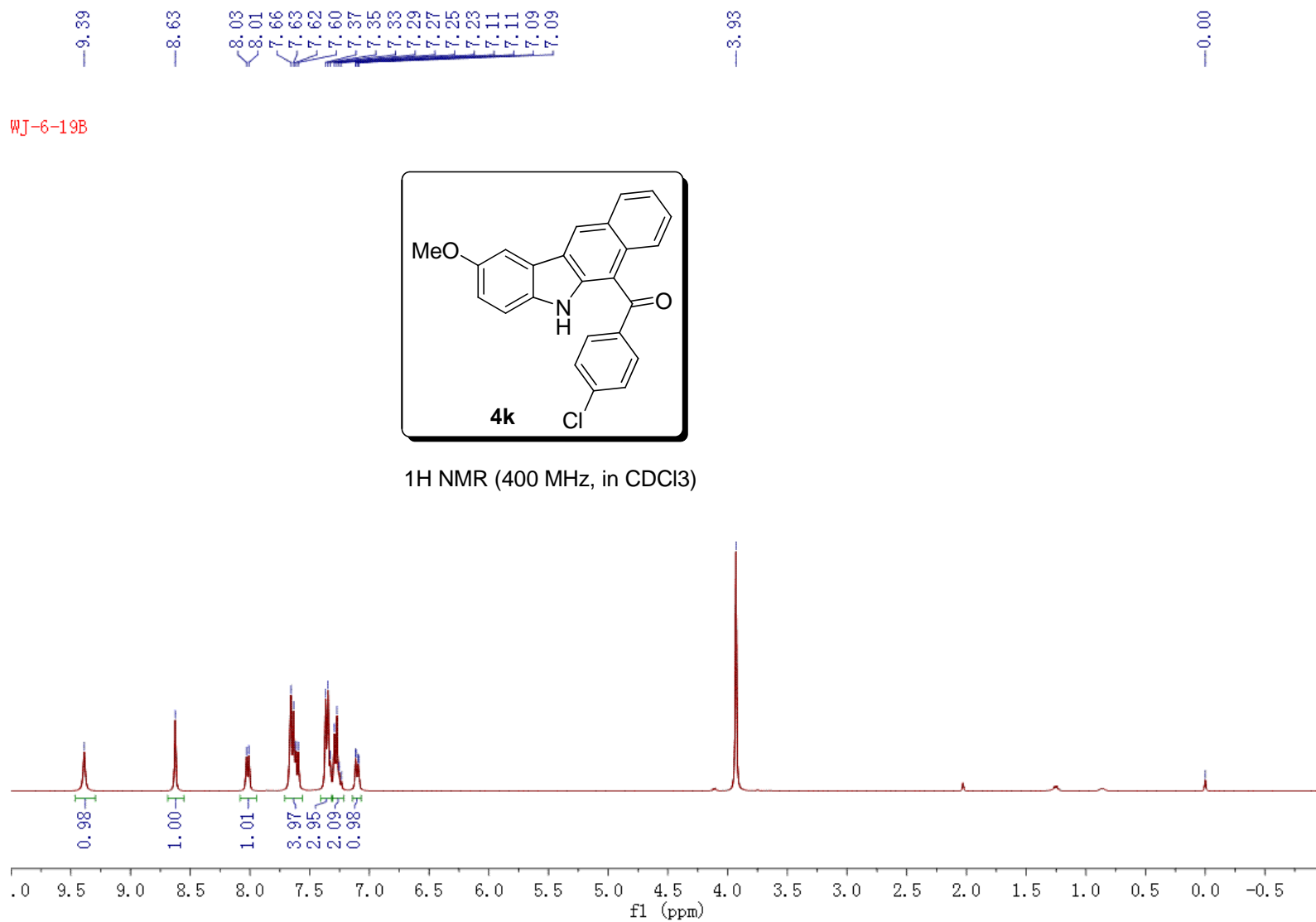


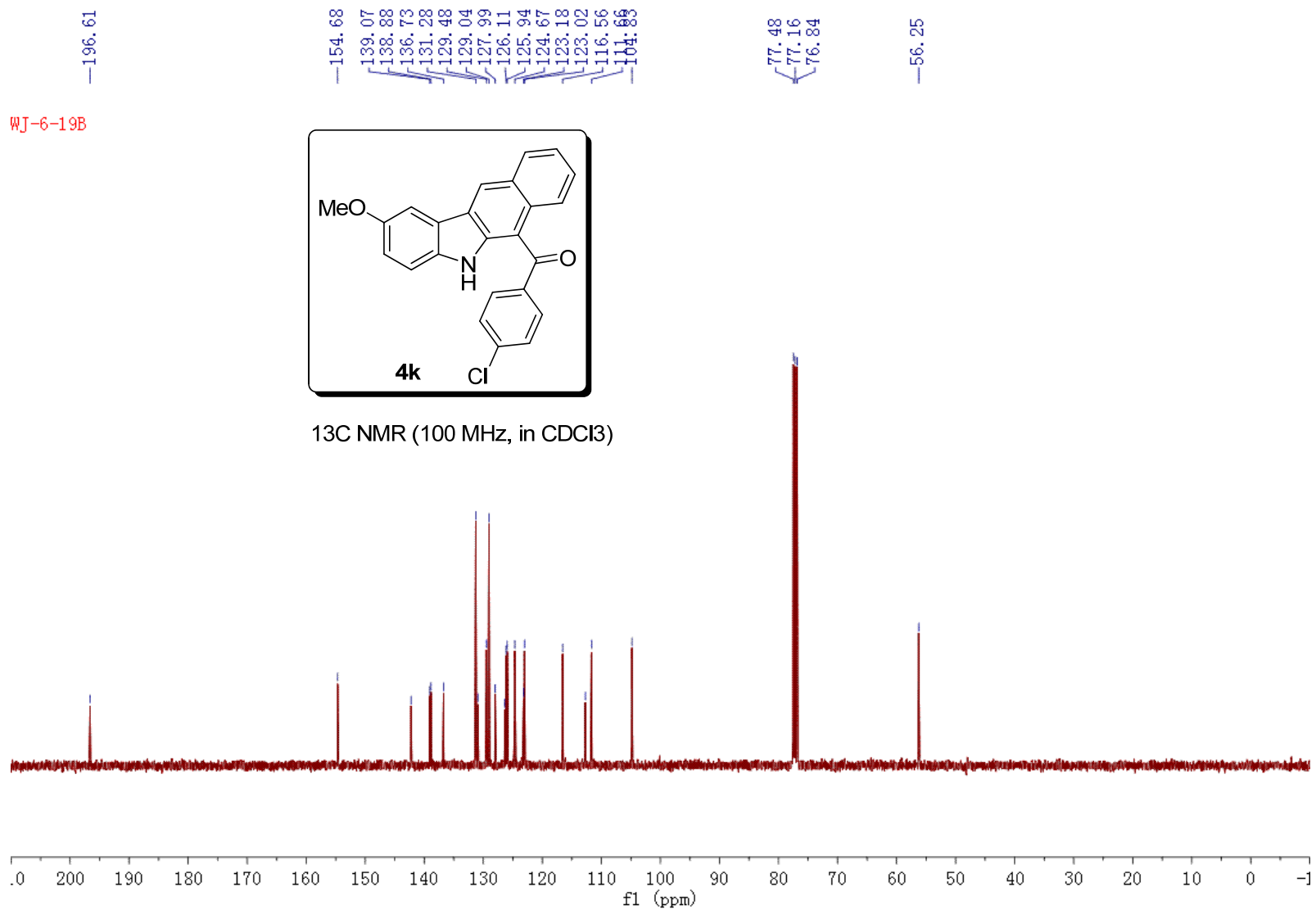
WJ-5-19A



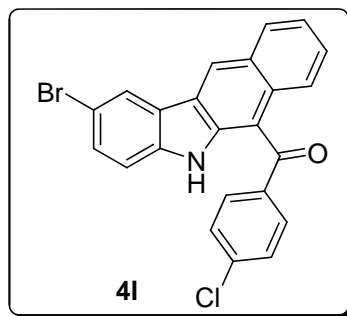


WJ-6-19B

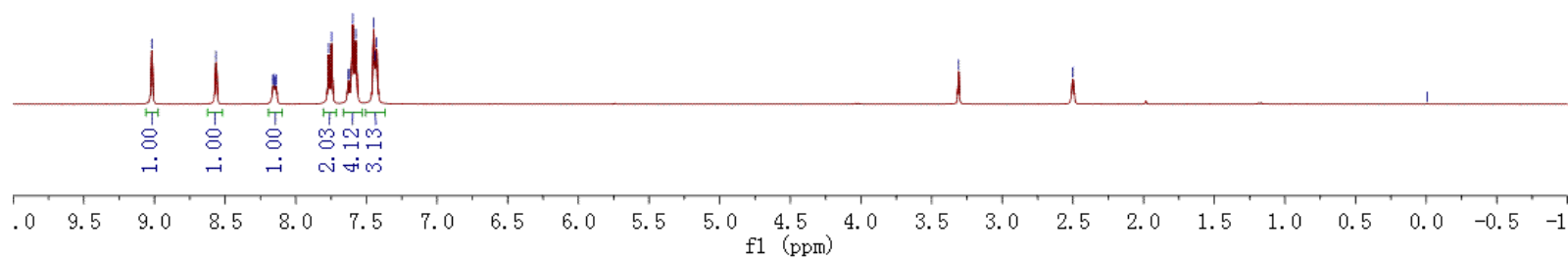


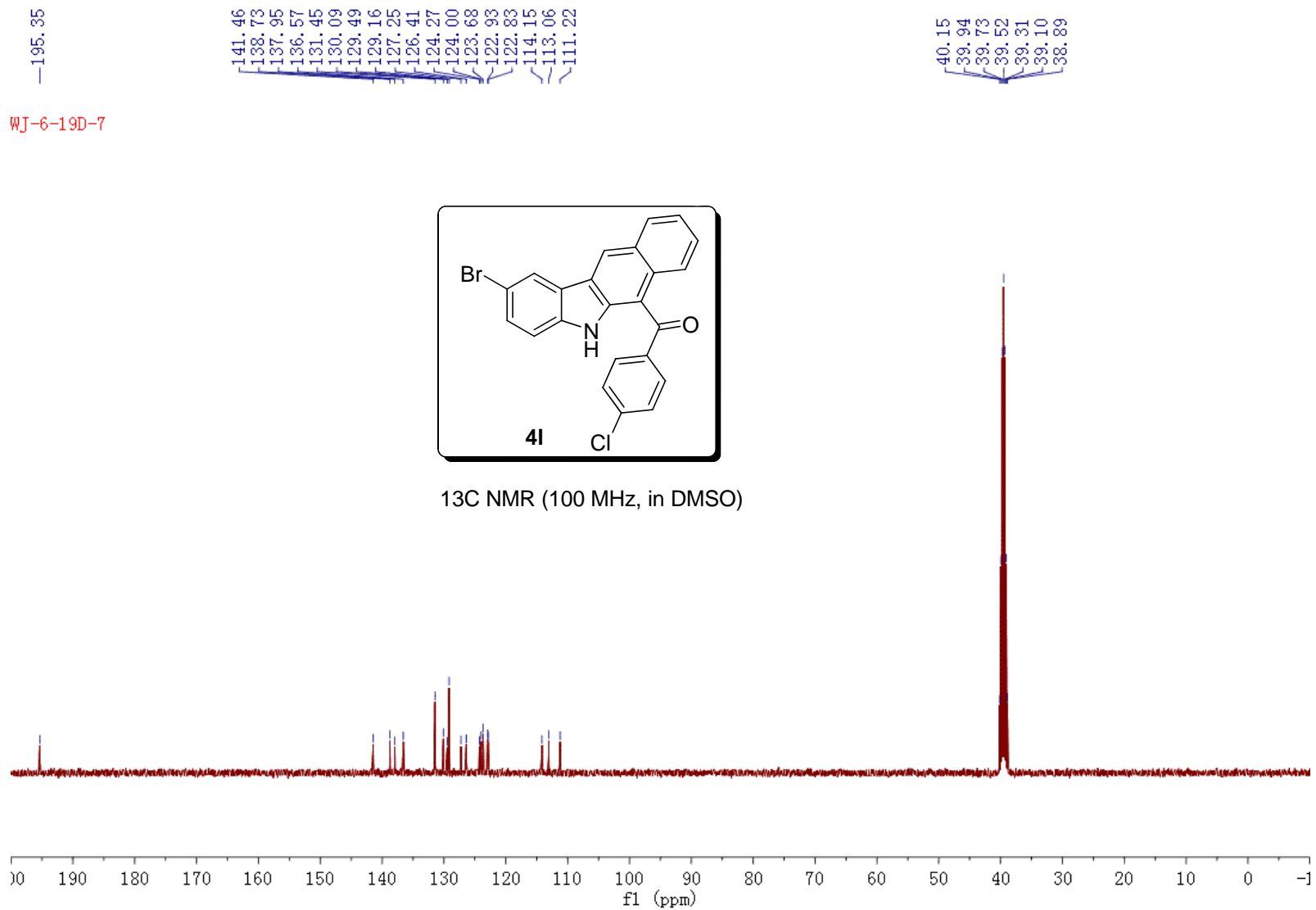


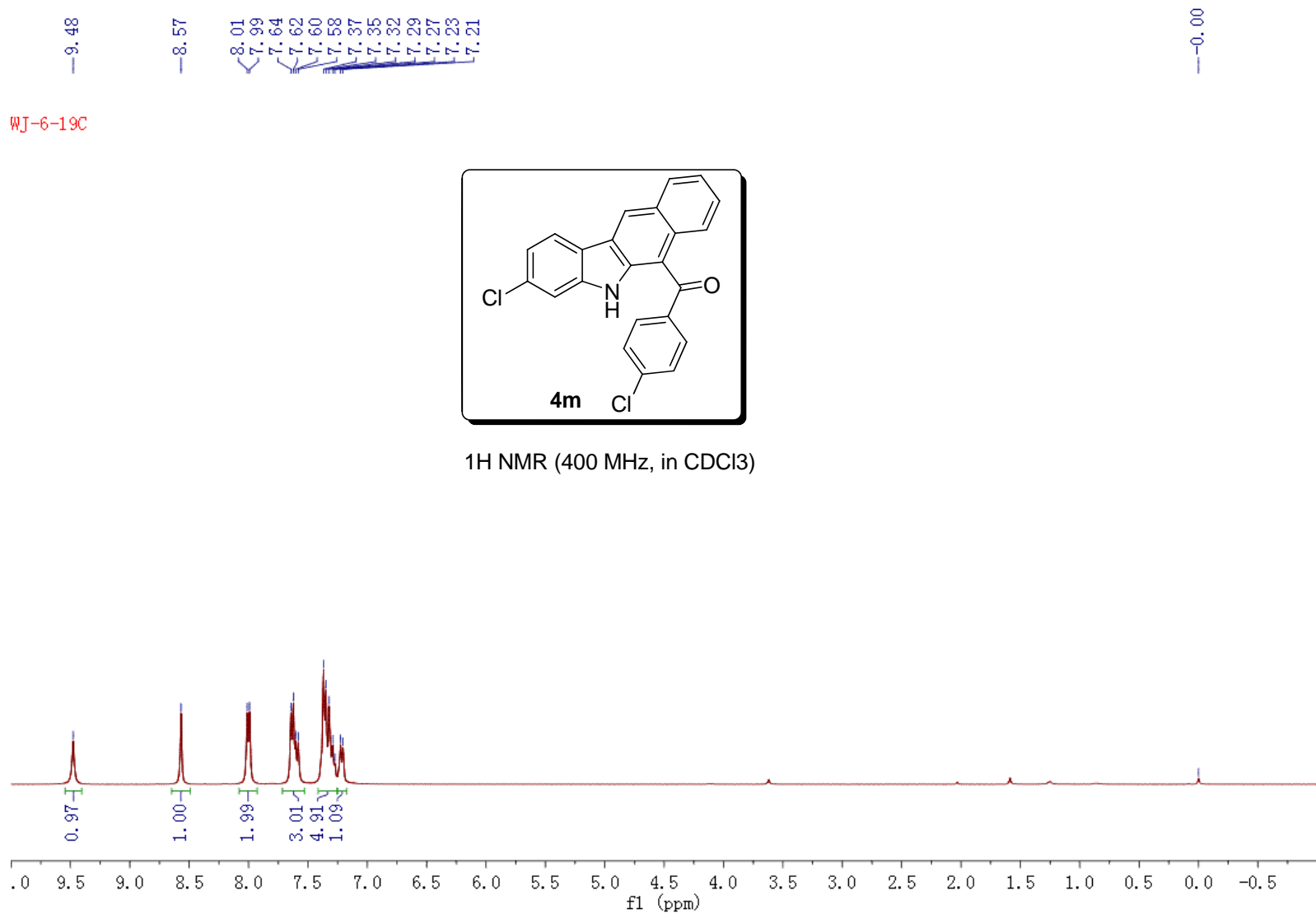
WJ-6-19D-7



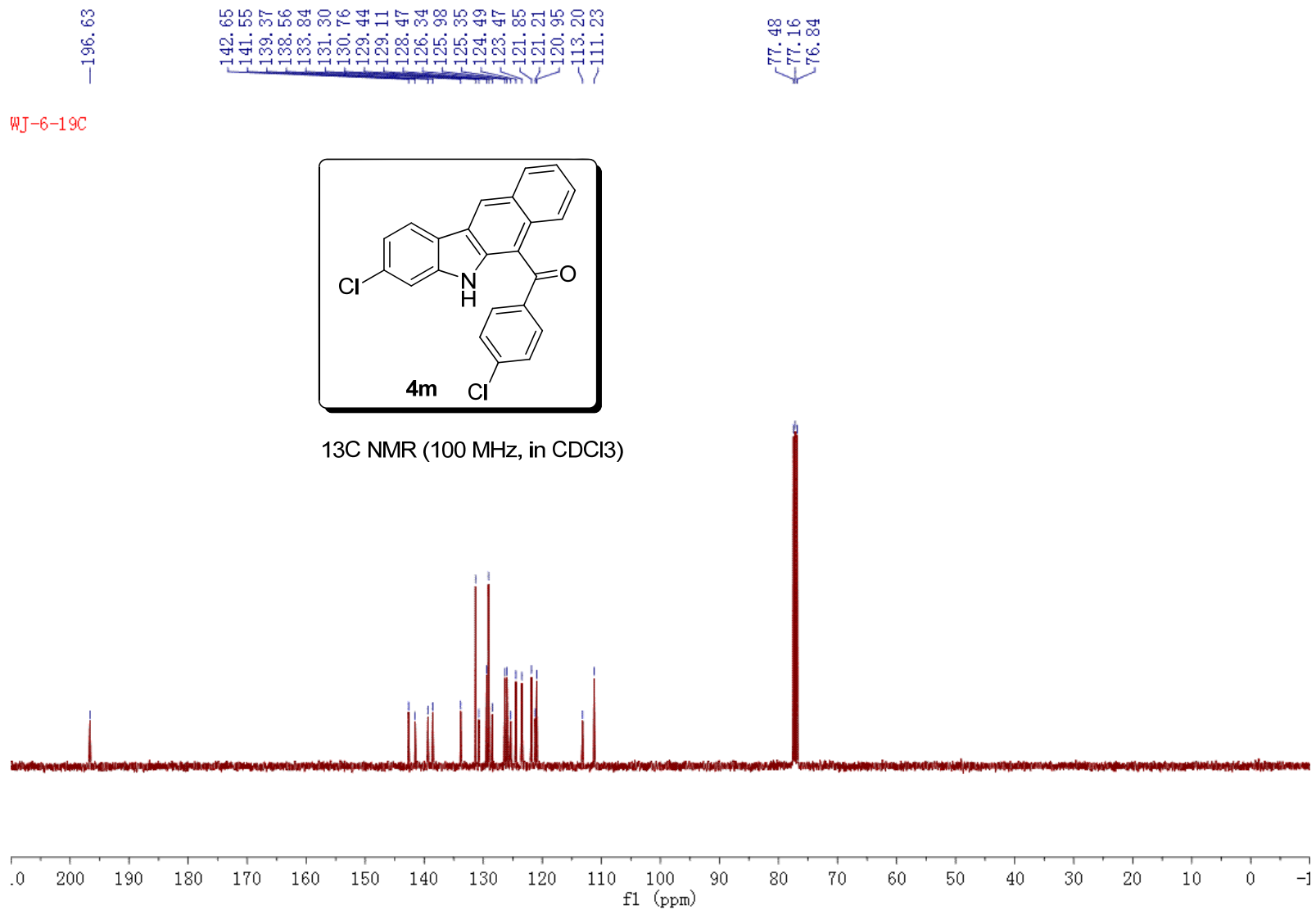
¹H NMR (400 MHz, in DMSO)



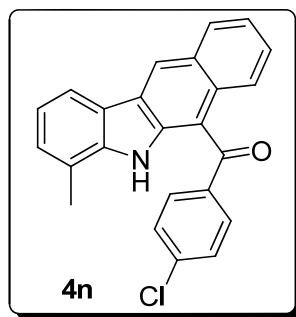




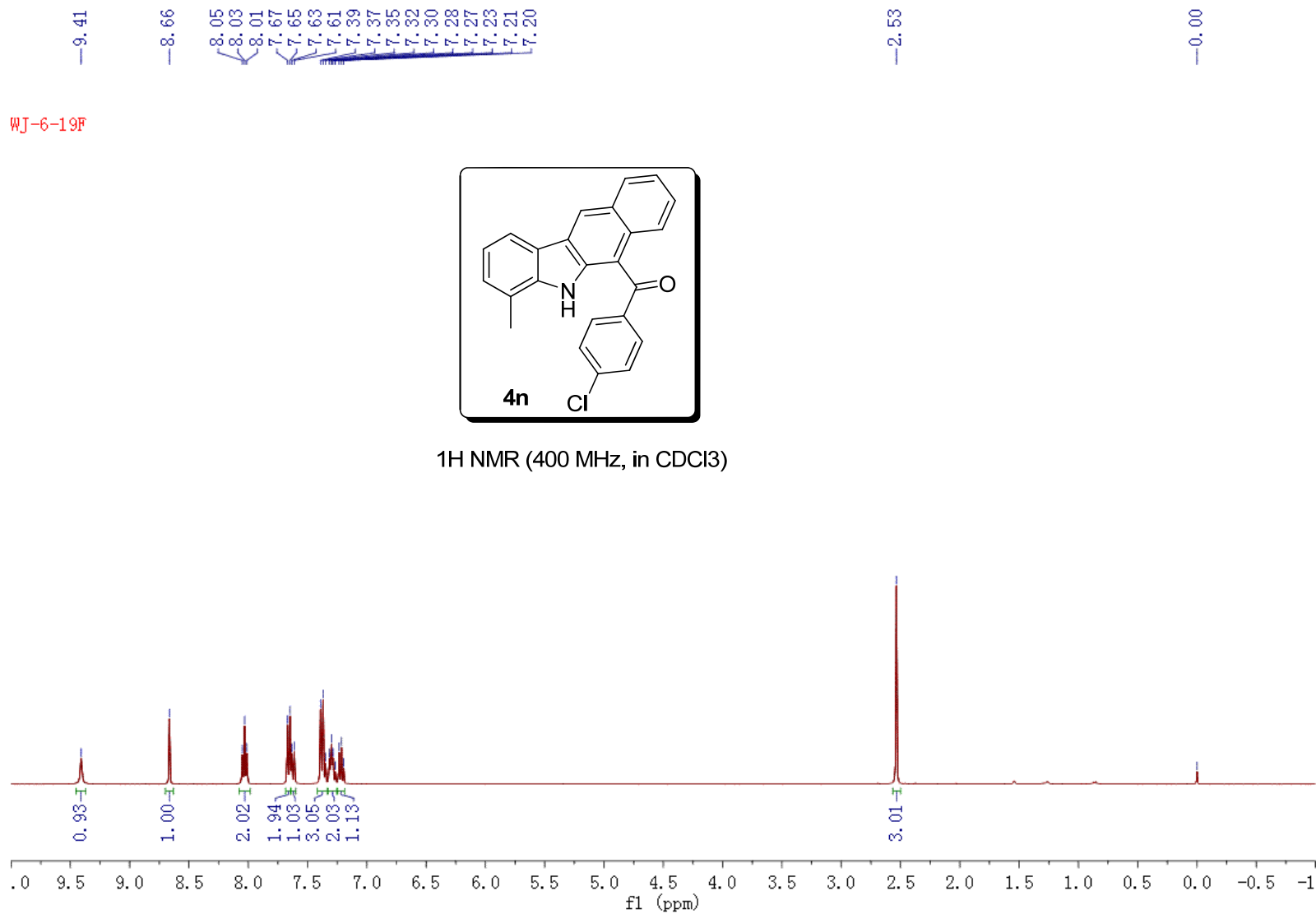
WJ-6-19C

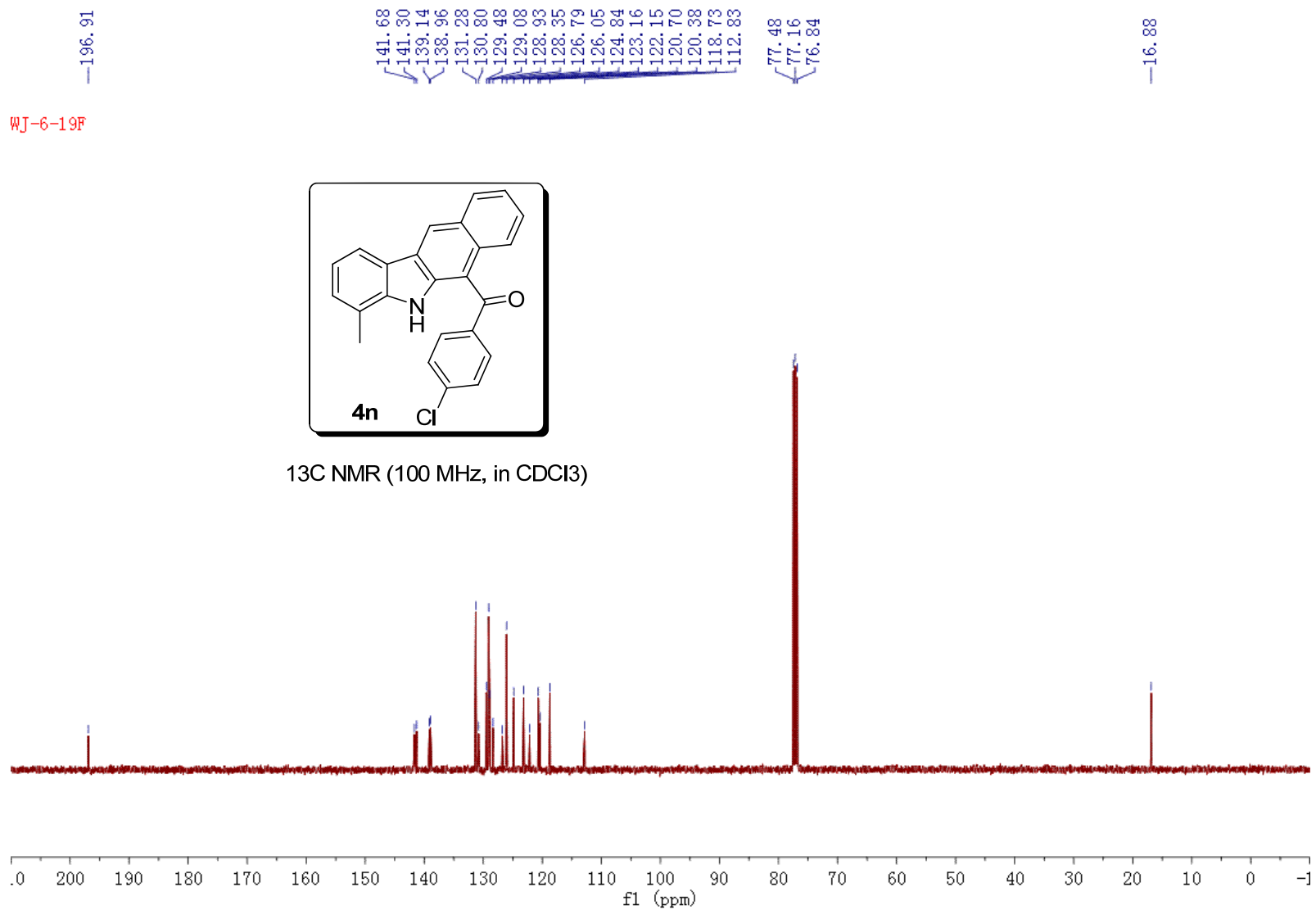


WJ-6-19F

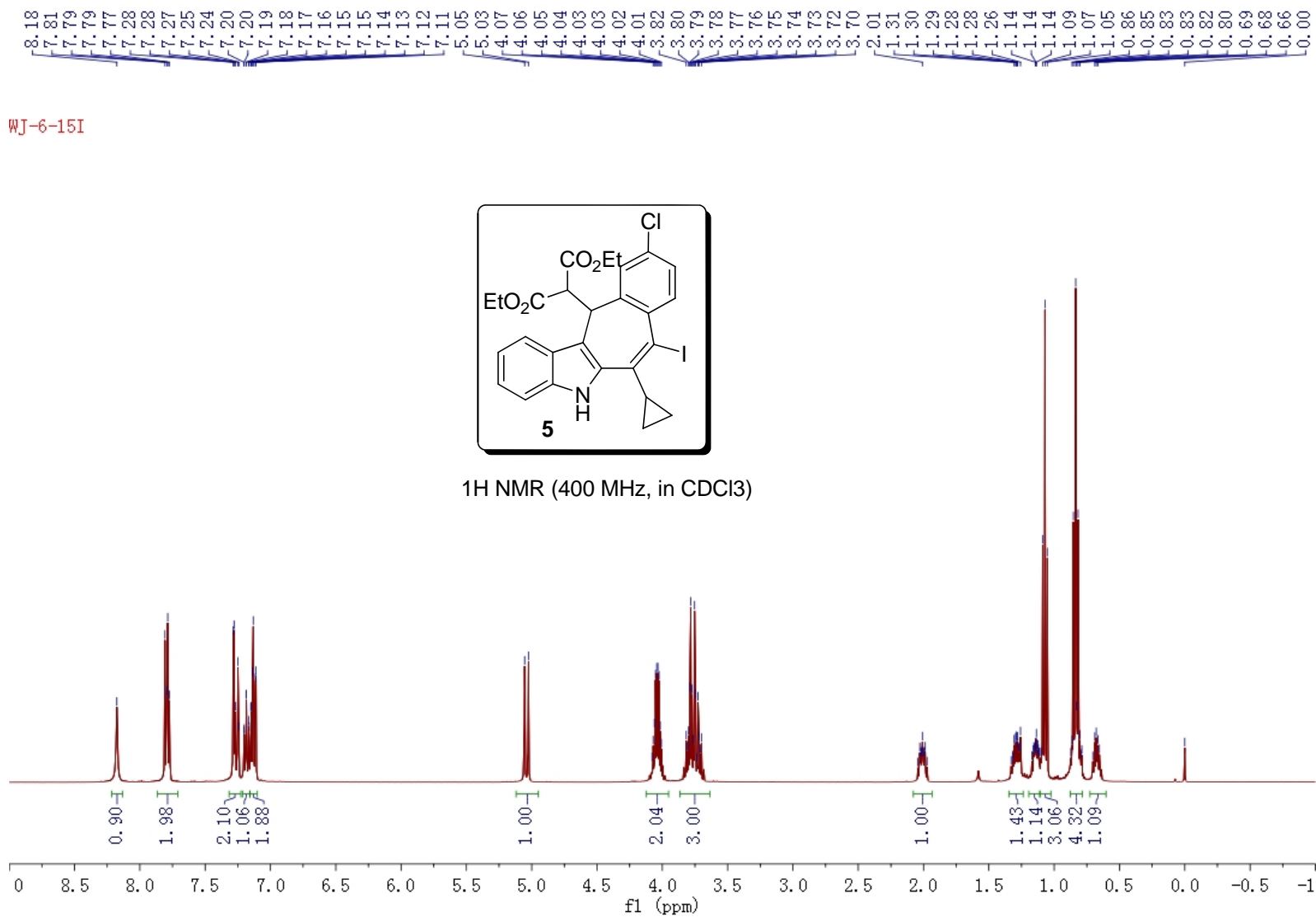


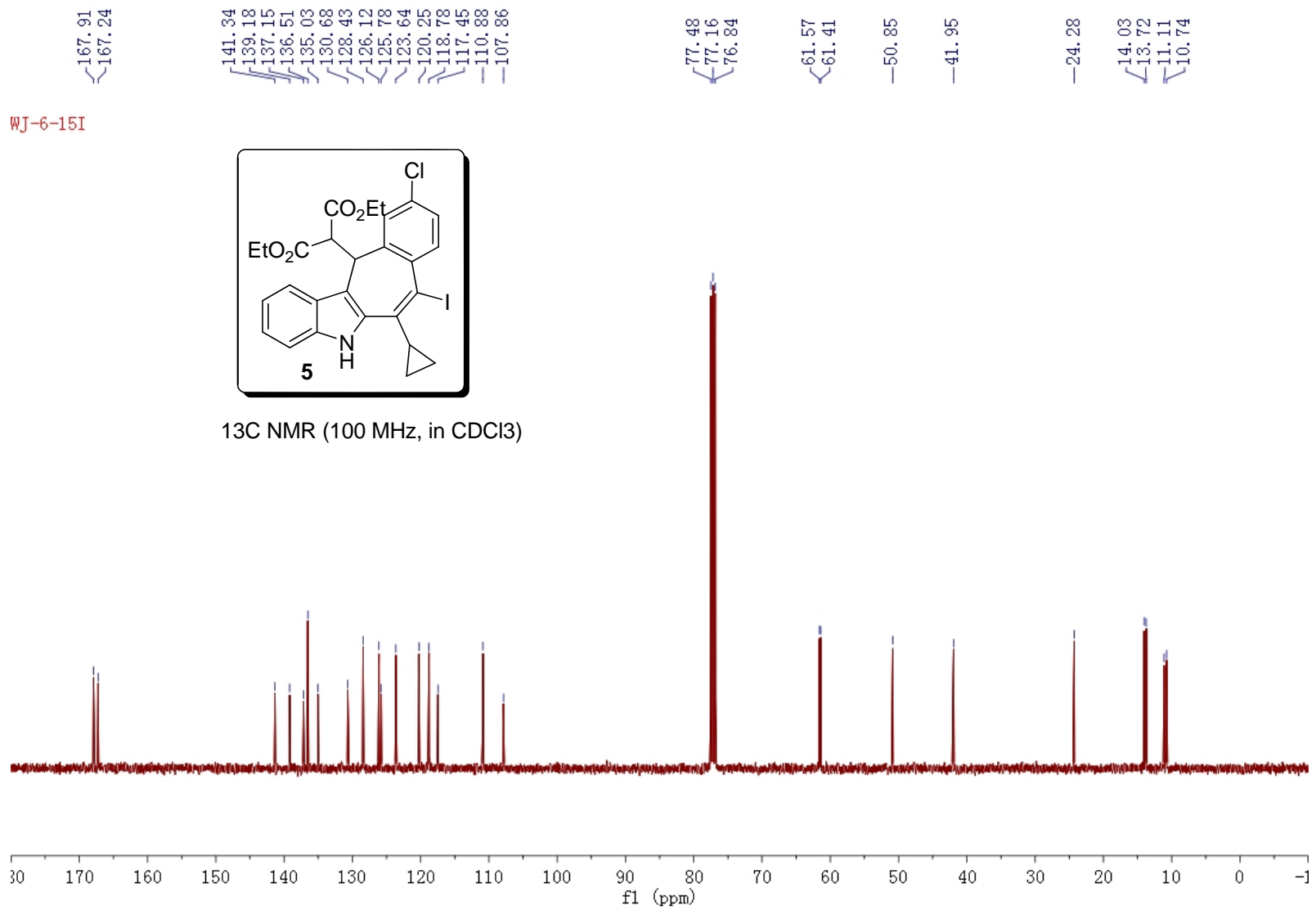
¹H NMR (400 MHz, in CDCl₃)





WJ-6-15I





2. X-ray structure of **5**

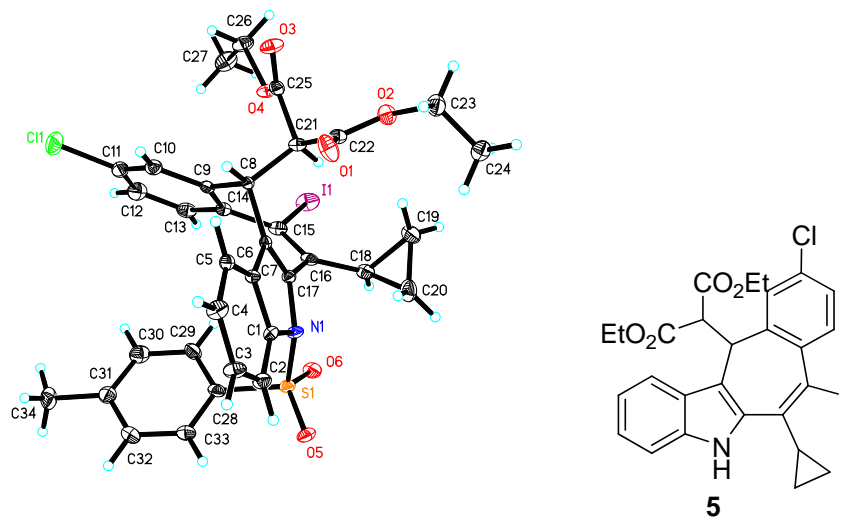


Table 1. Crystal data and structure refinement for mo_dm14148_0m.

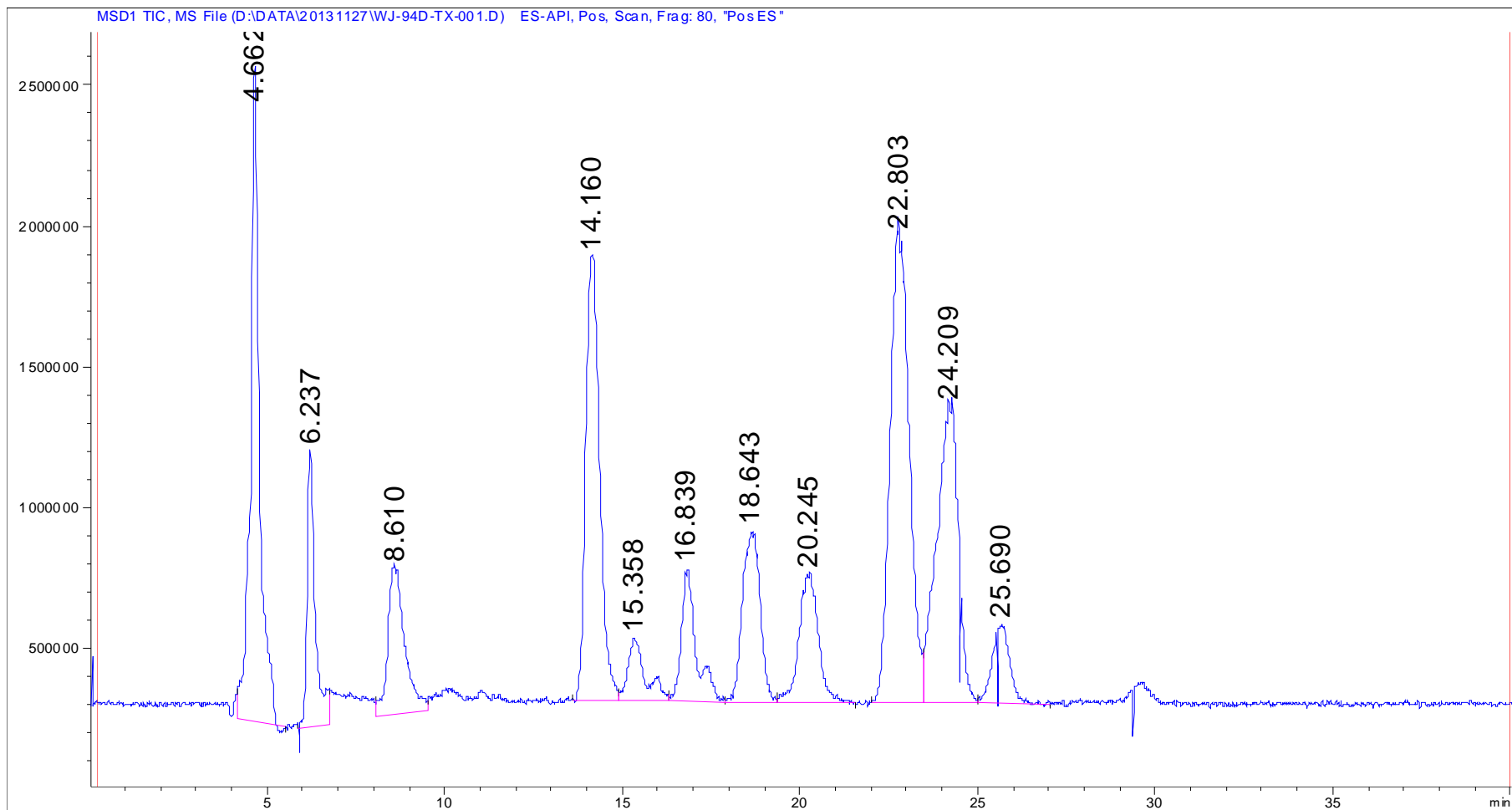
Identification code	mo_dm14148_0m	
Empirical formula	C ₃₄ H ₃₁ Cl I N O ₆ S	
Formula weight	744.01	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 8.380(3) Å	= 87.992(7)°.
b = 9.942(4) Å	= 88.509(7)°	

c = 19.911(7) Å	= 72.843(7)°.
Volume	1583.7(10) Å ³
Z	2
Density (calculated)	1.560 Mg/m ³
Absorption coefficient	1.208 mm ⁻¹
F(000)	752
Crystal size	0.250 x 0.120 x 0.080 mm ³
Theta range for data collection	1.023 to 30.278°.
Index ranges	-11<=h<=11, -13<=k<=13, -28<=l<=27
Reflections collected	15285
Independent reflections	9191 [R(int) = 0.0369]
Completeness to theta = 25.242°	99.3 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7461 and 0.6639
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	9191 / 0 / 397
Goodness-of-fit on F ²	1.002
Final R indices [I>2sigma(I)]	R1 = 0.0499, wR2 = 0.1329

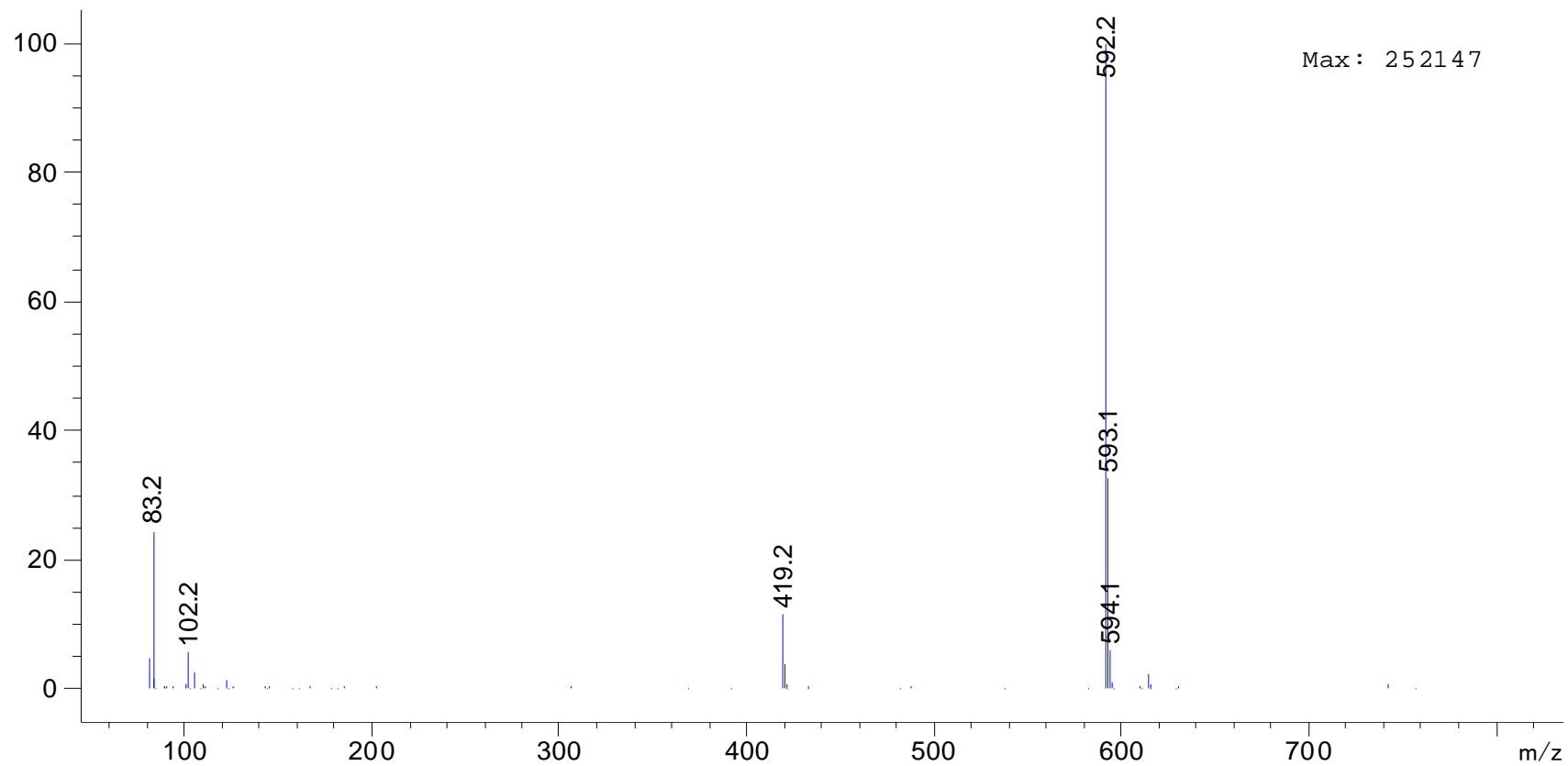
R indices (all data)	R1 = 0.0893, wR2 = 0.1837
Extinction coefficient	n/a
Largest diff. peak and hole	0.694 and -0.799 e.Å ⁻³

CCDC 992632 (5) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

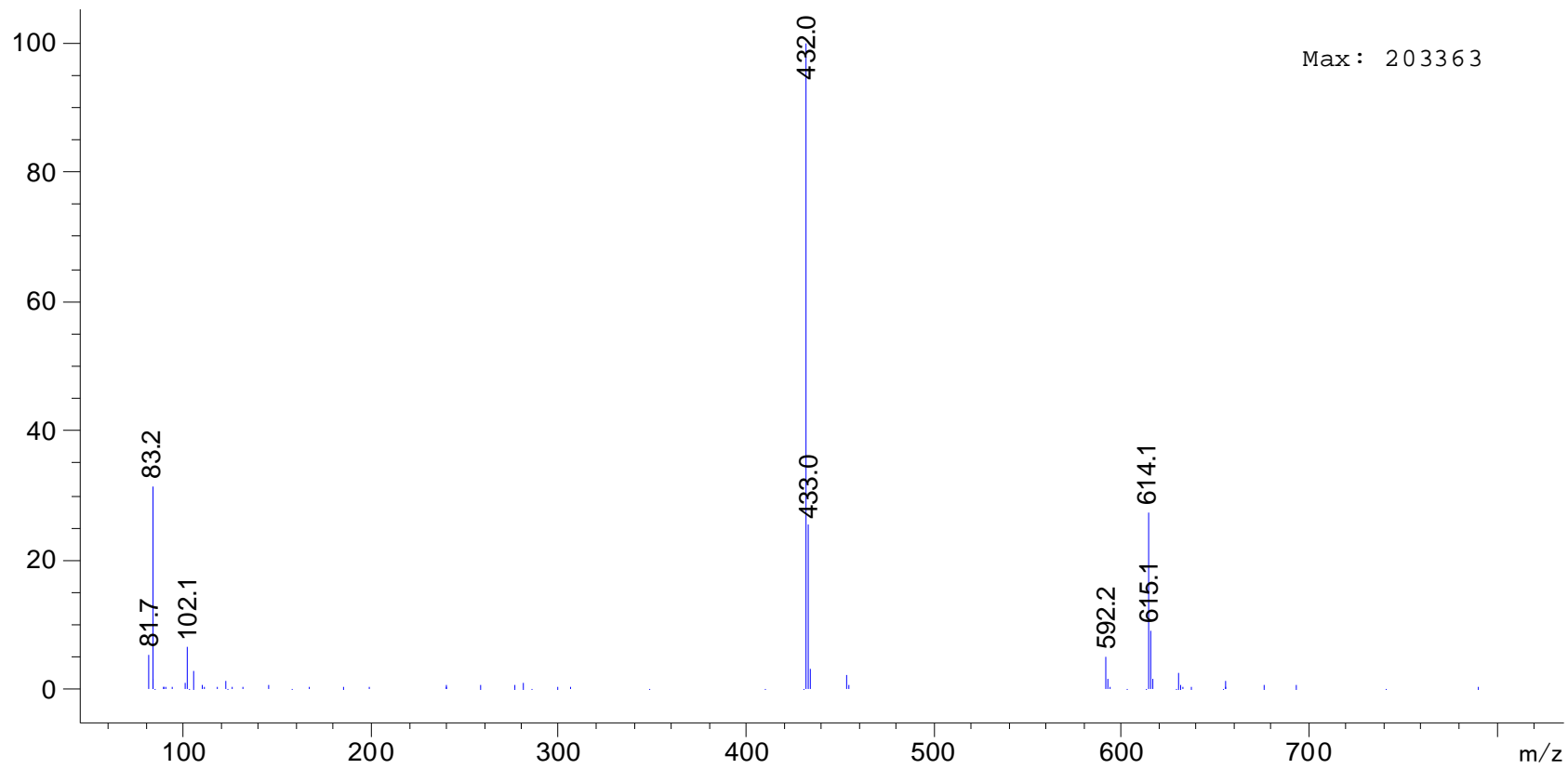
3. TIC spectrum and MS spectrum of five substances with molecule weight of 591.1



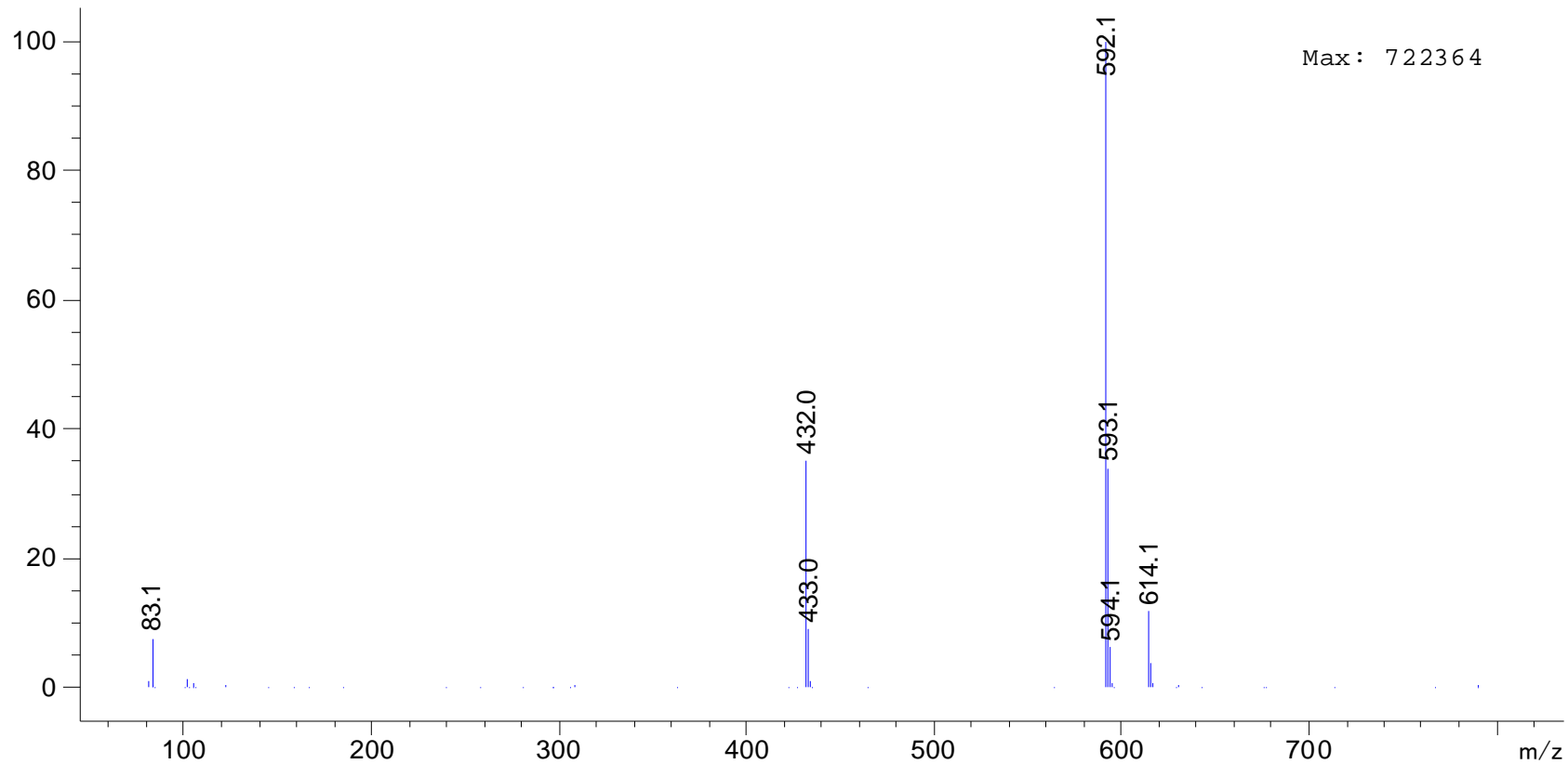
*MSD1 SPC, time=16.806:17.024 of D:\DATA\20131127WJ-94D-TX-001.D ES-API, Pos, Scan, Frag: 80, "Pos ES"



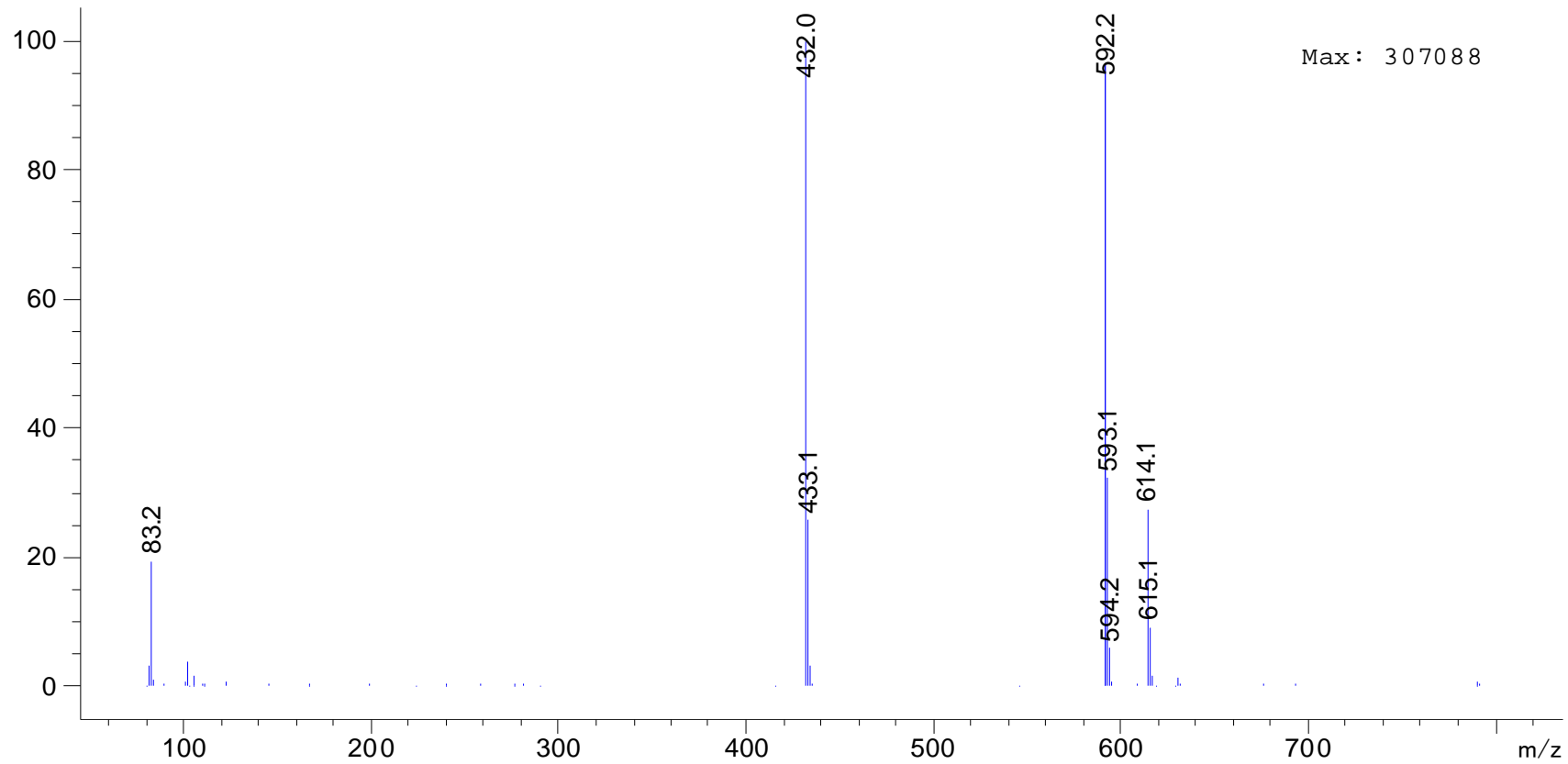
*MSD1 SPC, time=19.987:20.767 of D:\DATA\20131127WJ-94D-TX-001.D ES-API, Pos, Scan, Frag: 80, "Pos ES"



*MSD1 SPC, time=22.483:22.998 of D:\DATA\20131127WJ-94D-TX-001.D ES-API, Pos, Scan, Frag: 80, "Pos ES"



*MSD1 SPC, time=23.809:24.370 of D:\DATA\20131127WJ-94D-TX-001.D ES-API, Pos, Scan, Frag: 80, "Pos ES"



*MSD1 SPC, time=25.322:25.883 of D:\DATA\20131127WJ-94D-TX-001.D ES-API, Pos, Scan, Frag: 80, "Pos ES"

