

Supporting Information for:

## **Indazole enhances Ru-catalyzed hydrogenation of unsaturated bonds**

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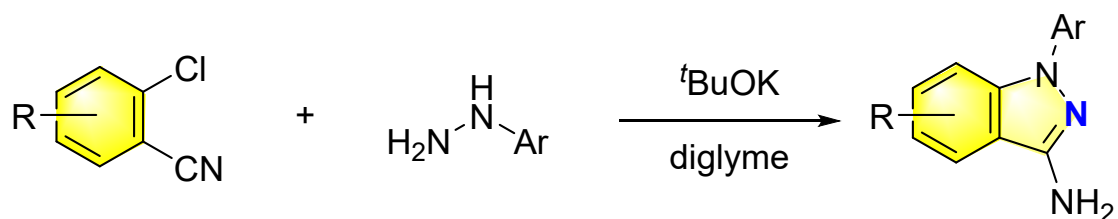
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## 1. General

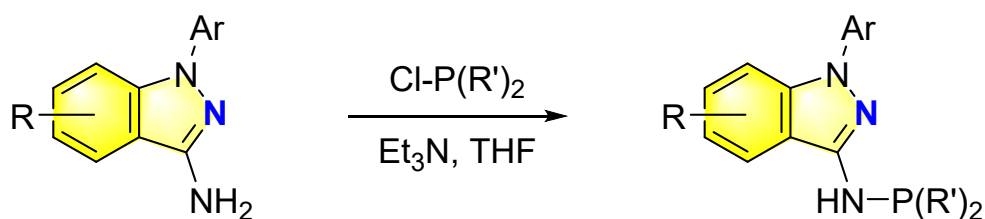
**Experimental:** All reactions and manipulations with air sensitive compounds being present were performed under dry argon (Ar 5.0) or nitrogen (N<sub>2</sub> 5.0), using Schlenk and glove box techniques. Non-halogenated solvents were dried over sodium benzophenone, 2-methyltetrahydrofuran (2-Me-THF) was dried over calcium hydride, and halogenated solvents were dried over P<sub>2</sub>O<sub>5</sub>. Deuterated solvents were bought from Cambridge Isotope Laboratories, distilled accordingly, and stored over molecular sieves (3 Å). Other chemicals were purchased from commercial vendors and used without further purification. NMR spectra were collected on a Varian INOVA 300 and 400 MHz spectrometer. Chemical shifts ( $\delta$ ) are reported in ppm relative to residual solvent signal. Coupling constants ( $J$ ) are given in Hz (coupling patterns: s: singlet, s\_br: broad singlet, d: doublet, t: triplet, q: quartet, m: multiplet). GC analyses were carried out using an Agilent Technologies 6890N system equipped with a Machinery-Nagel (MN) Optima 5 HT column (30 m, 320  $\mu$ m, 0.25  $\mu$ m) or an Agilent Technologies 6850 system equipped with a MN Optima 17 column (30 m, 320  $\mu$ m, 0.25  $\mu$ m). GC/MS analyses were carried out on an Agilent 7890A/MSD 5975C system equipped with a HP-5MS column (30 m, 320  $\mu$ m, 0.25  $\mu$ m). High resolution mass spectra (HRMS) were recorded on Bruker MicroTOF-QII mass (ESI). MN silica gel 60 (0.040 – 0.063 mm particle size) was used for flash column chromatography.

## 2. Synthesis of ligand

### 2.1 Synthesis of L1 – L8



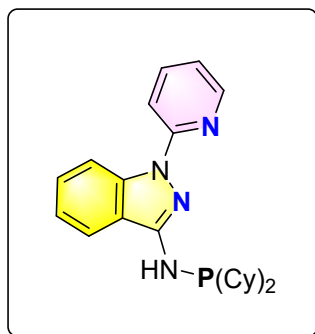
In a nitrogen-filled glove box, add a magnetic stirring bar, <sup>t</sup>BuOK (1.3 mmol), hydrazines (3.0 mmol), and diglyme (2.0 mL) to a dried pressure tube (38 mL volume). After stirring of 5 minutes, nitriles (1.0 mmol) were added the mixture reaction. Then the seal tube was closed tightly with a teflon cap, removed from the glove box and immersed into a pre-heated metal bath (130°C). After design time the reaction was cooled, quenched with saturated brine and extracted with DCM. The organic phase was dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated. A small aliquot of the organic phase was analyzed by GC or GC-MS to monitor product formation. Purification of the remainder by column chromatography on silica gel to give the corresponding product (pentane/ethyl ether = 15:1 - 5:1).



In a nitrogen-filled glove box, add a magnetic stirring bar, amines (1.0 mmol), Et<sub>3</sub>N (1.0 mmol), THF (2.0 mL) and phosphine chloride (1.0 mmol) to a dried schlenk tube. Then the seal tube was closed tightly with a teflon cap, removed from the glove box and immersed into a pre-heated metal bath (60°C) for 12 hours. After the reaction was finished, the reaction was analyzed by TLC to monitor product formation. A small aliquot of the organic phase was analyzed by GC or GC-MS to monitor product formation. The solvent was evaporated under reduced pressure and the residue was



purified by flash column chromatography on Al<sub>2</sub>O<sub>3</sub> to give the product **L1 - L8** in the reported yields.



***N*-(dicyclohexylphosphaneyl)-1-(pyridin-2-yl)-1*H*-indazol-3-amine (L1)<sup>1</sup>**

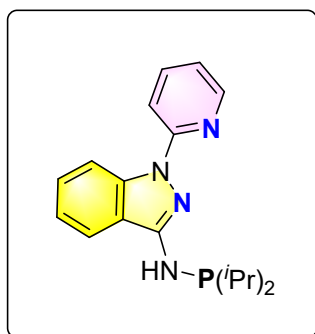
The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid, 80% yield.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.79 (d, *J* = 8.4 Hz, 1H), 8.44 – 8.42 (m, 1H), 8.16 (d, *J* = 7.6 Hz, 1H), 7.83 (d, *J* = 8.4 Hz, 1H), 7.73 – 7.70 (m, 1H), 7.47 (t, *J* = 8.0 Hz, 1H), 7.18 (t, *J* = 7.6 Hz, 1H), 6.70 – 6.97 (m, 1H), 4.61 (d, *J* = 4.8 Hz, 1H), 1.88 – 1.68 (m, 12H), 1.33 – 1.20 (m, 10H) ppm.

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 154.3, 147.5, 140.4, 137.8, 128.3, 121.9, 121.8, 120.7, 118.0, 115.2, 112.4, 36.7, 36.6, 29.1, 28.9, 27.2, 27.1, 26.8, 26.8 ppm.

<sup>31</sup>P NMR (162 MHz, CDCl<sub>3</sub>) δ 48.7 ppm.

HRMS (ESI) calcd for C<sub>24</sub>H<sub>32</sub>N<sub>4</sub>P: [M+H]: 407.2364, found:407.2360.



***N*-(diisopropylphosphaneyl)-1-(pyridin-2-yl)-1*H*-indazol-3-amine (L2)<sup>1</sup>**

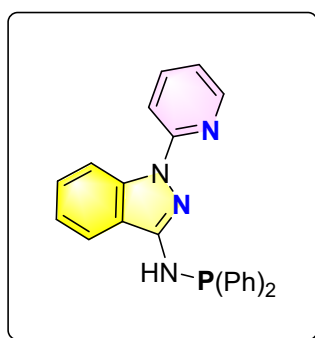
The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid, 77% yield.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.82 (d, *J* = 8.8 Hz, 1H), 8.45 – 8.44 (m, 1H), 8.14 (d, *J* = 8.0 Hz, 1H), 7.84 (d, *J* = 8.4 Hz, 1H), 7.73 – 7.70 (m, 1H), 7.49 – 7.43 (m, 1H), 7.22 – 7.17 (m, 1H), 6.98 – 6.93 (m, 1H), 4.61 (d, *J* = 5.6 Hz, 1H), 2.00 – 1.91 (m, 2H), 1.20 – 1.14 (m, 12H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 154.2, 151.3, 151.2, 147.4, 140.3, 137.7, 128.2, 121.5, 121.4, 120.7, 117.9, 117.9, 115.1, 112.3, 26.8, 26.7, 18.8, 18.6, 17.3, 17.2 ppm.

**<sup>31</sup>P NMR** (162 MHz, CDCl<sub>3</sub>) δ 55.6 ppm.

**HRMS** (ESI) calcd for C<sub>18</sub>H<sub>24</sub>N<sub>4</sub>P: [M+H]: 327.1738, found:327.1741.



***N*-(diphenylphosphaneyl)-1-(pyridin-2-yl)-1*H*-indazol-3-amine (L3)<sup>1</sup>**

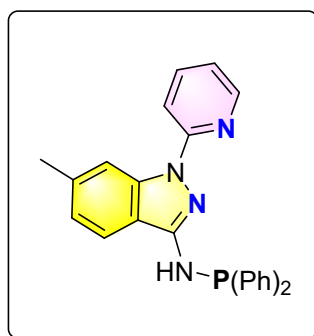
The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid, 85% yield.

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.84 (d, *J* = 8.8 Hz, 1H), 8.44 (d, *J* = 3.2 Hz, 1H), 7.99 (d, *J* = 8.0 Hz, 1H), 7.82 (d, *J* = 8.4 Hz, 1H), 7.69 (t, *J* = 8.8 Hz, 1H), 7.62 – 7.54 (m, 4H), 7.51 (t, *J* = 8.0 Hz, 1H), 7.46 – 7.35 (m, 6H), 7.20 (t, *J* = 7.6 Hz, 1H), 7.03 – 6.95 (m, 1H), 5.18 (d, *J* = 6.4 Hz, 1H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 154.3, 149.7, 149.6, 147.5, 140.3, 140.1, 140.0, 137.9, 131.5, 131.3, 129.4, 128.7, 128.6, 128.6, 121.2, 120.6, 120.5, 118.3, 118.0, 118.0, 115.5, 112.6 ppm.

**<sup>31</sup>P NMR** (162 MHz, CDCl<sub>3</sub>) δ 32.1 ppm.

**HRMS** (ESI) calcd for C<sub>24</sub>H<sub>20</sub>N<sub>4</sub>P: [M+H]: 395.1425, found: 395.1423.



***N*-(diphenylphosphaneyl)-6-methyl-1-(pyridin-2-yl)-1*H*-indazol-3-amine (L4)<sup>1</sup>**

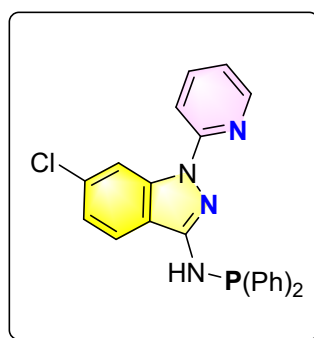
The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid, 86% yield.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.63 (s, 1H), 8.35 (d, *J* = 4.0 Hz, 1H), 7.79 (dd, *J* = 27.6, 8.4 Hz, 2H), 7.50 – 7.49 (m, 5H), 7.30 – 7.29 (m, 6H), 6.93 (d, *J* = 8.0 Hz, 1H), 6.86 (dd, *J* = 6.8, 5.2 Hz, 1H), 5.26 (d, *J* = 6.4 Hz, 1H), 2.47 (s, 3H) ppm.

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 153.2, 148.6, 148.5, 146.2, 139.8, 139.0, 138.9, 137.9, 136.7, 130.3, 130.1, 128.1, 127.5, 127.4, 122.1, 119.2, 119.0, 116.9, 115.1, 115.0, 113.9, 111.5, 21.1 ppm.

<sup>31</sup>P NMR (162 MHz, CDCl<sub>3</sub>) δ 32.2 ppm.

HRMS (ESI) calcd for C<sub>25</sub>H<sub>22</sub>N<sub>4</sub>P: [M+H]<sup>+</sup>: 409.1582, found: 409.1580.



**6-chloro-*N*-(diphenylphosphaneyl)-1-(pyridin-2-yl)-1*H*-indazol-3-amine (L5)<sup>1</sup>**

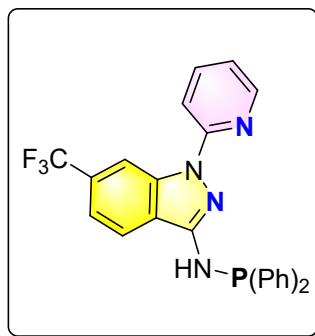
The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid, 81% yield.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.88 (s, 1H), 8.45 – 8.39 (m, 1H), 7.91 (d, *J* = 8.4 Hz, 1H), 7.77 (d, *J* = 8.4 Hz, 1H), 7.67 (d, *J* = 7.6 Hz, 1H), 7.51 – 7.36 (m, 10H), 7.11 (d, *J* = 8.4 Hz, 1H), 7.00 – 6.96 (m, 1H), 5.21 (d, *J* = 6.0 Hz, 1H) ppm.

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  153.8, 149.5, 149.3, 147.4, 140.4, 139.8, 139.7, 138.0, 134.7, 131.4, 131.2, 129.4, 128.7, 128.6, 122.0, 121.6, 121.5, 118.6, 115.3, 112.4 ppm.

$^{31}\text{P}$  NMR (162 MHz,  $\text{CDCl}_3$ )  $\delta$  32.6 ppm.

HRMS (ESI) calcd for  $\text{C}_{24}\text{H}_{19}\text{ClN}_4\text{P}$ :  $[\text{M}+\text{H}]$ : 429.1036, found: 429.1034.



***N*-(diphenylphosphaneyl)-1-(pyridin-2-yl)-6-(trifluoromethyl)-1*H*-indazol-3-amine (L6)<sup>1</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid, 72% yield.

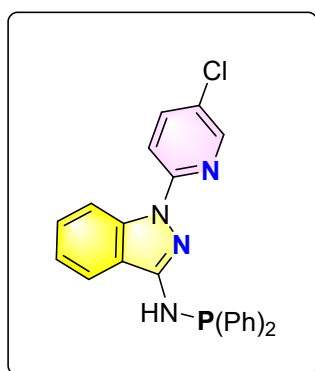
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  9.22 (s, 1H), 8.50 – 8.45 (m, 1H), 8.13 (d,  $J = 8.4$  Hz, 1H), 7.81 (d,  $J = 8.4$  Hz, 1H), 7.74 – 7.70 (m, 1H), 7.58 – 7.54 (m, 4H), 7.40 – 7.37 (m, 7H), 7.03 – 7.01 (m, 1H), 5.24 (d,  $J = 6.4$  Hz, 1H) ppm.

$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  153.8, 149.4, 149.2, 147.5, 139.6, 139.5, 139.2, 138.0, 131.4, 131.2, 130.4, 130.1, 129.4, 128.7, 128.6, 125.8, 123.1, 121.5, 121.4, 119.6, 118.9, 117.5, 113.3, 113.3, 112.5 ppm.

$^{31}\text{P}$  NMR (162 MHz,  $\text{CDCl}_3$ )  $\delta$  32.9 ppm.

$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -61.5 ppm.

HRMS (ESI) calcd for  $\text{C}_{25}\text{H}_{19}\text{F}_3\text{N}_4\text{P}$ :  $[\text{M}+\text{H}]$ : 463.1299, found: 463.1304.



**1-(5-chloropyridin-2-yl)-*N*-(diphenylphosphaneyl)-1*H*-indazol-3-amine (L7)<sup>1</sup>**

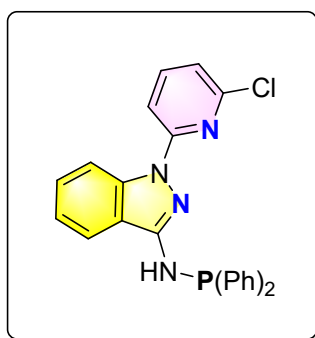
The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid, 80% yield.

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.71 (d, *J* = 8.8 Hz, 1H), 8.34 (d, *J* = 2.4 Hz, 1H), 7.94 (d, *J* = 8.0 Hz, 1H), 7.74 (d, *J* = 8.8 Hz, 1H), 7.58 (dd, *J* = 8.8, 2.4 Hz, 1H), 7.54 – 7.50 (m, 4H), 7.47 – 7.43 (m, 1H), 7.39 – 7.36 (m, 6H), 7.18 (t, *J* = 7.6 Hz, 1H), 5.17 (d, *J* = 6.4 Hz, 1H) ppm.

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 152.5, 150.1, 149.9, 145.9, 140.2, 140.0, 139.8, 137.7, 131.5, 131.3, 129.4, 128.8, 128.7, 128.7, 125.4, 121.5, 120.6, 120.5, 118.3, 118.3, 115.5, 113.5 ppm.

<sup>31</sup>P NMR (162 MHz, CDCl<sub>3</sub>) δ 32.3 ppm.

HRMS (ESI) calcd for C<sub>24</sub>H<sub>19</sub>ClN<sub>4</sub>P: [M+H]: 429.1036, found:429.1032.



**1-(6-chloropyridin-2-yl)-*N*-(diphenylphosphaneyl)-1*H*-indazol-3-amine (L8)<sup>1</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid, 72% yield.

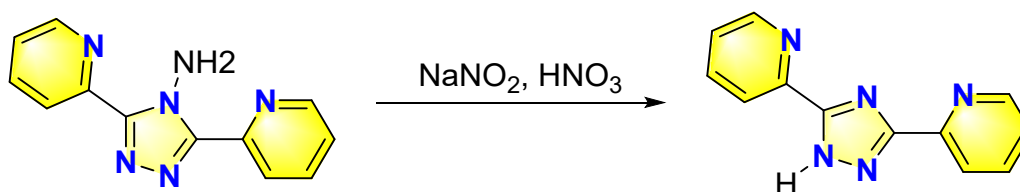
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.74 (d, *J* = 8.8 Hz, 1H), 7.92 (d, *J* = 8.0 Hz, 1H), 7.68 (d, *J* = 8.0 Hz, 1H), 7.59 (t, *J* = 8.0 Hz, 1H), 7.55 – 7.48 (m, 5H), 7.40 – 7.38 (m, 6H), 7.20 (t, *J* = 7.6 Hz, 1H), 6.96 (d, *J* = 7.6 Hz, 1H), 5.11 (d, *J* = 6.4 Hz, 1H) ppm.

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 153.6, 150.3, 150.1, 148.8, 140.2, 140.1, 139.8, 139.7, 131.4, 131.2, 129.4, 128.9, 128.6, 128.6, 121.7, 120.4, 120.3, 118.3, 117.6, 115.7, 110.3 ppm.

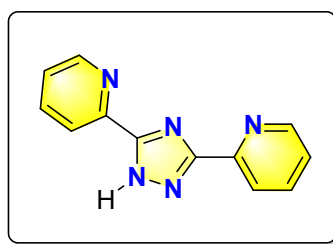
<sup>31</sup>P NMR (162 MHz, CDCl<sub>3</sub>) δ 32.1 ppm.

HRMS (ESI) calcd for C<sub>24</sub>H<sub>19</sub>ClN<sub>4</sub>P: [M+H]: 429.1036, found:429.1038.

## 2.2 Synthesis of L9



In a nitrogen-filled glove box, add a magnetic stirring bar, 3,5-di(2-pyridyl)-4-amino-1,2,4-triazole (1.0 mmol) was dissolved in aqueous boiling nitric acid (5 N) to a dried pressure tube (100 mL volume). The solution was cooled down to 0 °C, and saturated aqueous sodium nitrite solution was added. After that, it was kept under reflux for 2 hours and, upon cooling, poured into ammonium hydroxide solution to obtain a white precipitate of 2,2'-(1H-1,2,4-triazole-3,5-diyl)dipyridine, which was carefully washed several times with ice-cold water to get the corresponding product **L9** in the reported yield. A small aliquot of the organic solid was analyzed by GC-MS to monitor product formation.

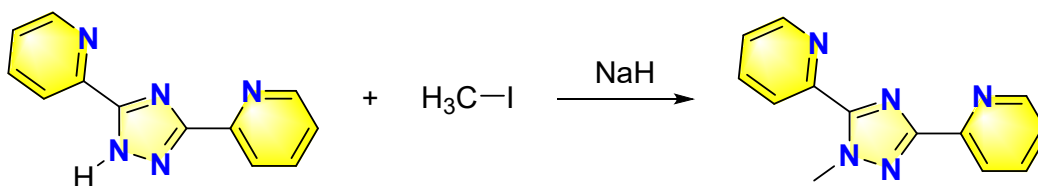


### 2,2'-(1H-1,2,4-triazole-3,5-diyl)dipyridine (L9)<sup>2</sup>

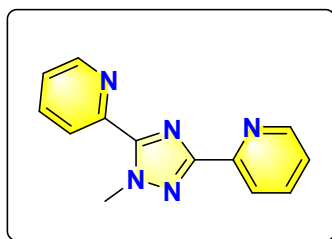
The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid, 71% yield. Purification by column chromatography on silica gel (pentane/ethyl ether = 20:1 - 2:1, DCM 5%).

<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>): δ 14.99 (s, 1H), 8.72 (s, 2H), 8.18 (s, 2H), 7.97 (s, 2H), 7.51 (s, 2H) ppm.

### 2.3 Synthesis of L10



In a nitrogen-filled glove box, add a magnetic stirring bar, 2,2'-(1H-1,2,4-triazole-3,5-diyl)dipyridine (2.0 mmol), NaH (2.5 mmol) into a dried pressure tube (38 mL volume) containing DMF (5.0 mL) at 0 °C for 10 min. Iodomethane (3.0 mmol) in 2.0 mL DMF was added dropwise to the reaction mixture, and then the solution was stirred at room temperature for an additional 12 hours. After that, the mixture was poured into the ice water, and extracted with DCM. Then, the organic layer was dried over Na<sub>2</sub>SO<sub>4</sub>. A small aliquot of the organic phase was analyzed by GC-MS to monitor product formation. Purification of the remainder by column chromatography on silica gel gave the corresponding products **L10** in the reported yields.

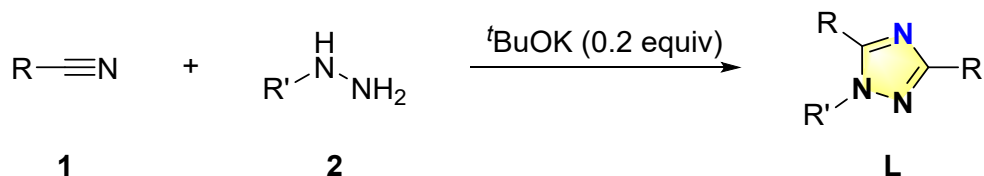


#### 2,2'-(1-methyl-1H-1,2,4-triazole-3,5-diyl)dipyridine (L10)<sup>3</sup>

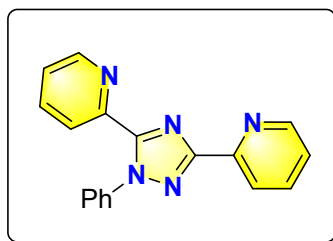
The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid, 66% yield. Purification by column chromatography on silica gel (pentane/ethyl ether = 20:1 - 2:1, DCM 5%).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 8.73 (d, *J* = 4.0, 2.0 Hz, 1H), 8.66 (d, *J* = 4.4, 2.0 Hz, 1H), 8.37 (d, *J* = 4.4, 2.0 Hz, 1H), 8.17 (d, *J* = 4.0, 2.0 Hz, 1H), 7.87 – 7.84 (m, 1H), 7.82 – 7.79 (m, 1H), 7.37 – 7.35 (m, 1H), 7.31 – 7.28 (m, 1H), 4.43 (s, 3H) ppm.

## 2.4 Synthesis of L11 – L13



In a nitrogen-filled glove box, add a magnetic stirring bar, <sup>t</sup>BuOK (112.0 mg, 1.0 mmol), nitriles **1** (5.0 mmol), hydrazines **2** (5.0 mmol), and 1,4-dioxane (5.0 mL) to a dried pressure tube (38 mL volume). Then the seal tube was closed tightly with a teflon cap, remove the pressure tube from the glove box and immersed into a pre-heated metal bath (120 °C). After 24 h the reaction was cooled, then quenched with saturated brine (200 μL) and EA, and dried with Na<sub>2</sub>SO<sub>4</sub>. A small aliquot of the organic phase was analyzed by GC or GC-MS to monitor product formation. Then the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 100:1 - 1:4, DCM 2%) on silica gel to give the corresponding product **L11 - L14**.

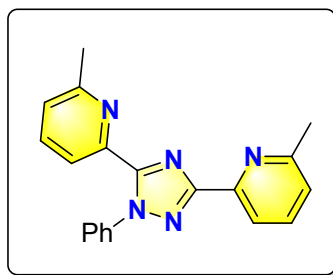


### 2,2'-(1-phenyl-1H-1,2,4-triazole-3,5-diyl)dipyridine (**L11**)<sup>4</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid, 83% yield. Purification by column chromatography on silica gel (pentane/ethyl ether = 20:1 - 2:1, DCM 5%).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.79 (d, *J* = 4.4 Hz, 1H), 8.46 (d, *J* = 4.4 Hz, 1H), 8.28 (d, *J* = 8.0 Hz, 1H), 8.06 (d, *J* = 8.0 Hz, 1H), 7.83 – 7.80 (m, 2H), 7.47 – 7.40 (m, 2H), 7.43 – 7.39 (m, 3H), 7.37 – 7.32 (m, 1H), 7.28 (dd, *J* = 4.4, 3.2 Hz, 1H) ppm.

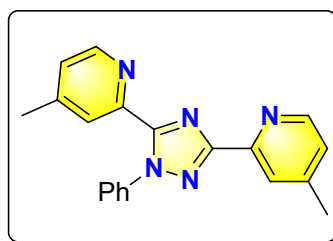




**6,6'-(1-phenyl-1H-1,2,4-triazole-3,5-diyl)bis(2-methylpyridine) (L12)<sup>5</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid, 65% yield. Purification by column chromatography on silica gel (pentane/ethyl ether = 20:1 - 2:1, DCM 5%).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.10 (d, *J* = 7.6 Hz, 1H), 7.81 (d, *J* = 7.6 Hz, 1H), 7.73 – 7.61 (m, 2H), 7.49 – 7.30 (m, 5H), 7.21 (d, *J* = 7.6 Hz, 1H), 7.11 (d, *J* = 7.2 Hz, 1H), 2.69 (s, 3H), 2.33 (s, 3H) ppm.



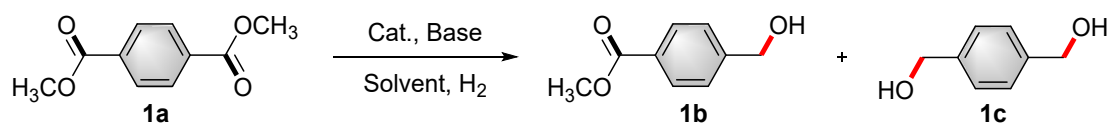
**2,2'-(1-phenyl-1H-1,2,4-triazole-3,5-diyl)bis(4-methylpyridine) (L13)<sup>5</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid, 73% yield. Purification by column chromatography on silica gel (pentane/ethyl ether = 20:1 - 2:1, DCM 5%).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.63 (d, *J* = 5.2 Hz, 1H), 8.30 (d, *J* = 5.2 Hz, 1H), 8.15 (d, *J* = 0.8 Hz, 1H), 8.01 (d, *J* = 0.8 Hz, 1H), 7.51 – 7.43 (m, 2H), 7.41 – 7.38 (m, 3H), 7.16 (dd, *J* = 5.2, 0.8 Hz, 1H), 7.13 (dd, *J* = 4.8, 0.8 Hz, 1H), 2.42 (s, 3H), 2.40 (s, 3H) ppm.

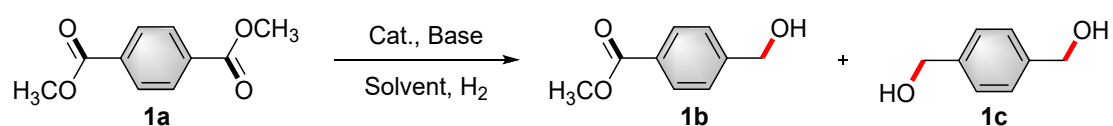
### 3. Screening of hydrogenation reaction conditions

Closed system:



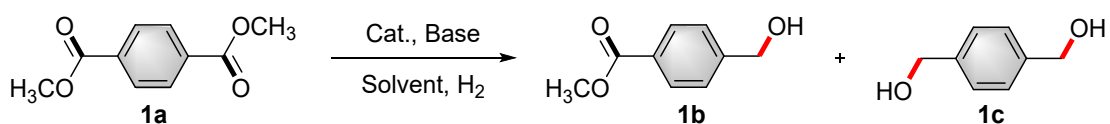
In a nitrogen-filled glove box, add a magnetic stirring bar, dimethyl terephthalate (1a), base, catalyst, and solvent to a dried high-pressure reactor. After sealing, remove the high-pressure reactor from the glove box. Then fill the reactor with hydrogen (design pressure) and immerse it in a pre-heated metal bath (design temperature). After design time the reaction was cooled, quenched with saturated brine (100  $\mu$ L) and dried over Na<sub>2</sub>SO<sub>4</sub>. A small aliquot of the organic phase was analyzed by GC or GC-MS to monitor product formation and gave the corresponding product in the reported yield.

Entry	Conditions
Table S1	Screening of different solvents
Table S2	Screening of different base
Table S3	Screening the loading of 'BuOK
Table S4	Screening the press of H <sub>2</sub> in hydrogenation reaction
Table S5	Screening the temperature of hydrogenation reaction
Table S6	Screening the loading of catalyst
Table S7	Screening of different catalyst
Table S8	Screening the time of hydrogenation reaction

**Table S1.** Screening of different solvents. [a]

Entry	Solvent (2 mL)	Yield <b>1b</b> (%)	<b>1b/1c</b>
1	toluene	16	5:1
2	anisole	32	19:1
<b>3</b>	<b>1,4-dioxane</b>	<b>45</b>	<b>5:1</b>
4	THF	44	5:1
5	diglyme	17	17:1
6	<i>t</i> AmOH	17	15:1
7	<i>t</i> BuOH	14	10:1
8	EtOH	<5	-
9	MeOH	<5	-
10	EA	<5	-
11	MeCN	<5	-
12	DCM	<5	-
13	NMP	<5	-
14	DMF	<5	-
15	DMAc	<5	-
16	DMSO	<5	-

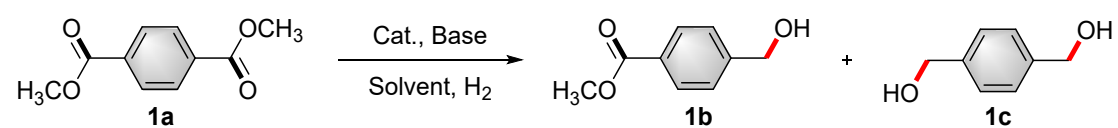
[a] Reaction conditions: **1a** (0.5 mmol), H<sub>2</sub> (4.0 MPa), RuCl<sub>2</sub>(PPh<sub>3</sub>)<sub>3</sub> (1 mol%), **L3** (1.2 mol%), *t*BuOK (20 mol%), solvent (2.0 mL), 130 °C for 24 h. Yield of **1b** and the ratio of **1b/1c** were detected by GC analysis with *n*-cetane as the internal standard.

**Table S2.** Screening of different base. [a]

Entry	Base	Yield <b>1b</b> (%)	<b>1b/1c</b>
1	<sup>t</sup> BuOLi	6	15:1
2	<sup>t</sup> BuONa	43	5:1
<b>3</b>	<b><sup>t</sup>BuOK</b>	<b>45</b>	<b>5:1</b>
4	( <sup>t</sup> BuO) <sub>2</sub> Mg <sup>[b]</sup>	<5	-
5	LiOH	5	10:1
6	NaOH	10	12:1
7	KOH	8	10:1
8	CsOH	<5	-
9	Li <sub>2</sub> CO <sub>3</sub>	<5	-
10	Na <sub>2</sub> CO <sub>3</sub>	<5	-
11	K <sub>2</sub> CO <sub>3</sub>	<5	-
12	Cs <sub>2</sub> CO <sub>3</sub>	<5	-
13	Py	<5	-
14	DBU	<5	-
15	TBD	<5	-
16	Et <sub>3</sub> N	<5	-

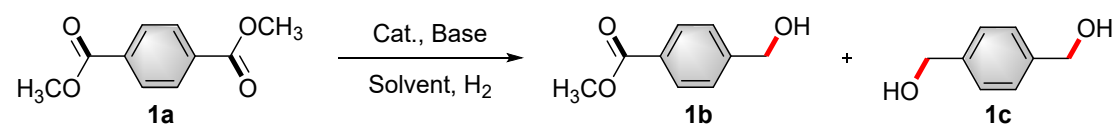
[a] Reaction conditions: **1a** (0.5 mmol), H<sub>2</sub> (4.0 MPa), RuCl<sub>2</sub>(PPh<sub>3</sub>)<sub>3</sub> (1 mol%), **L3** (1.2 mol%), base (20 mol%), 1,4-dioxane (2.0 mL), 130 °C for 24 h. Yield of **1b** and the ratio of **1b/1c** were detected by GC analysis with *n*-cetane as the internal standard.

[b] (<sup>t</sup>BuO)<sub>2</sub>Mg (10 mol%).

**Table S3.** Screening the loading of <sup>t</sup>BuOK. [a]

Entry	<sup>t</sup> BuOK	Yield <b>1b</b> (%)	<b>1b/1c</b>
1	0	0	-
2	1	<5	-
3	2	<5	-
4	4	13	-
5	6	38	19:1
6	8	62	10:1
7	<b>10</b>	<b>77</b>	<b>6:1</b>
8	12	74	5:1
9	15	65	5:1
10	20	45	5:1
11	30	25	3:1
12	40	16	2:1
13	50	9	2:1
14	100	<5	-
15	200	<5	-
16	300	<5	-

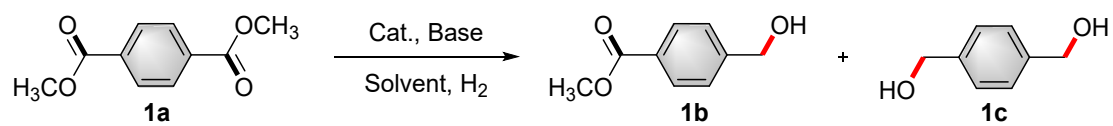
[a] Reaction conditions: **1a** (0.5 mmol), H<sub>2</sub> (4.0 MPa), RuCl<sub>2</sub>(PPh<sub>3</sub>)<sub>3</sub> (1 mol%), **L3** (1.2 mol%), <sup>t</sup>BuOK (x mol%), 1,4-dioxane (2.0 mL), 130 °C for 24 h. Yield of **1b** and the ratio of **1b/1c** were detected by GC analysis with *n*-cetane as the internal standard.

**Table S4.** Screening the press of H<sub>2</sub> in hydrogenation reaction. [a]

Entry	H <sub>2</sub> (MPa)	Yield <b>1b</b> (%)	<b>1b/1c</b>
1	4.0	77	6:1
2	3.5	79	7:1
3	3.0	78	7:1
4	2.5	80	8:1
<b>5</b>	<b>2.0</b>	<b>79</b>	<b>8:1</b>
6	1.5	68	10:1
7	1.0	50	15:1
8	0.5	27	19:1
9	0.1	<5	-
10	Ar or N <sub>2</sub>	0	-

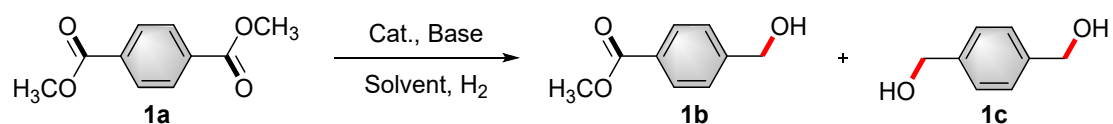
[a] Reaction conditions: **1a** (0.5 mmol), H<sub>2</sub> (x MPa), RuCl<sub>2</sub>(PPh<sub>3</sub>)<sub>3</sub> (1 mol%), **L3** (1.2 mol%), <sup>t</sup>BuOK (10 mol%), 1,4-dioxane (2.0 mL), 130 °C for 24 h. Yield of **1b** and the ratio of **1b/1c** were detected by GC analysis with *n*-cetane as the internal standard.

**Table S5.** Screening the temperature of hydrogenation reaction. <sup>[a]</sup>



Entry	T (°C)	Yield <b>1b</b> (%)	<b>1b/1c</b>
1	130	79	8:1
<b>2</b>	<b>110</b>	<b>83</b>	<b>12:1</b>
3	90	72	15:1
4	70	28	18:1
5	50	<5	-
6	rt	<5	-

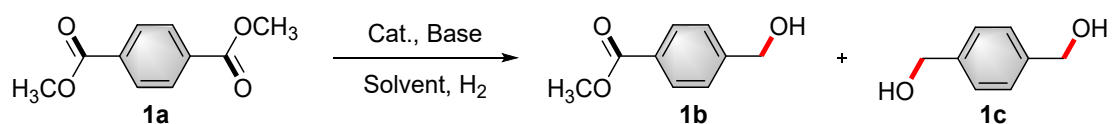
[a] Reaction conditions: **1a** (0.5 mmol), H<sub>2</sub> (2.0 MPa), RuCl<sub>2</sub>(PPh<sub>3</sub>)<sub>3</sub> (1 mol%), **L3** (1.2 mol%), *t*BuOK (10 mol%), 1,4-dioxane (2.0 mL), T °C for 24 h. Yield of **1b** and the ratio of **1b/1c** were detected by GC analysis with *n*-cetane as the internal standard.

**Table S6.** Screening the loading of catalyst. <sup>[a]</sup>

Entry	RuCl <sub>2</sub> (PPh <sub>3</sub> ) <sub>3</sub> (mol%)	<b>L3</b> (mol%)	Yield <b>1b</b> (%)	<b>1b/1c</b>
1	0	0	0	-
2	0.1	0.12	24	19:1
3	0.25	0.3	55	17:1
4	0.5	0.6	74	14:1
<b>5</b>	<b>1.0</b>	<b>1.2</b>	<b>83</b>	<b>12:1</b>
6	2.0	2.4	77	5:1

[a] Reaction conditions: **1a** (0.5 mmol), H<sub>2</sub> (2.0 MPa), RuCl<sub>2</sub>(PPh<sub>3</sub>)<sub>3</sub> (x mol%), **L3** (1.2x mol%), <sup>t</sup>BuOK (10 mol%), 1,4-dioxane (2.0 mL), 110 °C for 24 h. Yield of **1b** and the ratio of **1b/1c** were detected by GC analysis with *n*-cetane as the internal standard.

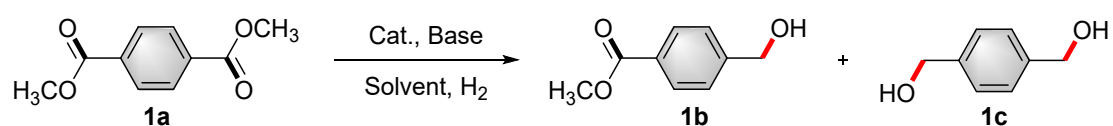


**Table S7.** Screening of different catalyst. <sup>[a]</sup>

Entry	[Cat.]	<b>1b</b>	<b>1b/1c</b>
1	FeCl <sub>2</sub>	0	-
2	FeBr <sub>2</sub>	0	-
3	Fe(OAc) <sub>2</sub>	0	-
4	CoCl <sub>2</sub>	0	-
5	Co(OAc) <sub>2</sub>	0	-
6	MnCl <sub>2</sub>	0	-
7	NiCl <sub>2</sub>	0	-
8	NiBr <sub>2</sub>	0	-
9	Ni(OAc) <sub>2</sub>	0	-
10	CuCl <sub>2</sub>	0	-
11	CuBr <sub>2</sub>	0	-
12	Cu(OAc) <sub>2</sub>	0	-
<b>13</b>	<b>RuCl<sub>2</sub>(PPh<sub>3</sub>)<sub>3</sub></b>	<b>83</b>	<b>11:1</b>
14	RuCl <sub>3</sub>	<5	-
15	-	0	-

[a] Reaction Conditions: **1a** (0.5 mmol), H<sub>2</sub> (2.0 MPa), [Cat.] (1.0 mol%), **L3** (1.2 mol%), <sup>t</sup>BuOK (10 mol%), 1,4-dioxane (2.0 mL), 110°C for 24 h. Yield of **1b** and the ratio of **1b/1c** were determined by GC analysis using *n*-cetane as the internal standard.

**Table S8.** Screening the time of hydrogenation reaction. [a]



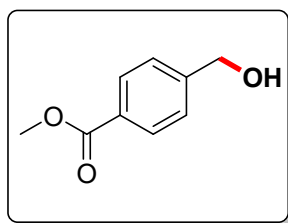
Entry	t (h)	Yield <b>1b</b> (%)	<b>1b/1c</b>
1	24	84	12:1
2	12	85	13:1
3	6	87	16:1
4	4	90	20:1
<b>5</b>	<b>2</b>	<b>96</b>	<b>&gt;20:1</b>
6	1.5	70	>20:1
7	1	41	>20:1

[a] Reaction conditions: **1a** (0.5 mmol), H<sub>2</sub> (2.0 MPa), RuCl<sub>2</sub>(PPh<sub>3</sub>)<sub>3</sub> (1 mol%), **L3** (1.2 mol%), <sup>t</sup>BuOK (10 mol%), 1,4-dioxane (2.0 mL), 110 °C. Yield of **1b** and the ratio of **1b/1c** were detected by GC analysis with *n*-cetane as the internal standard.

#### 4. General procedure for the hydrogenation reaction

In a nitrogen-filled glove box, add a magnetic stirring bar, substrates **a** (0.5 mmol), <sup>t</sup>BuOK (0.05 mmol, 10 mol%), RuCl<sub>2</sub>(PPh<sub>3</sub>)<sub>3</sub> (1 mol%), **L3** (1.2 mol%), and 1,4-dioxane (2.0 mL) to a dried high-pressure reactor. After sealing, remove the high-pressure reactor from the glove box. Then the reactor was purged and charged with H<sub>2</sub> (2.0 MPa) and immersed into a pre-heated metal bath (110 °C) and stirred at 600 rpm. After design time the reaction was cooled, release H<sub>2</sub> from the reactor. Then quenched with saturated brine (100 μL) and DCM, and dried with Na<sub>2</sub>SO<sub>4</sub>. A small aliquot of the organic phase was analyzed by GC or GC-MS to monitor product formation. Then the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 20:1 – 2:1) on silica gel to give the corresponding product.

## 5. Characterization data



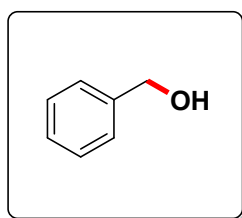
### methyl 4-(hydroxymethyl)benzoate (1b, 65b)<sup>6</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 80 mg, 96% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.98 (d, *J* = 8.0 Hz, 2H), 7.39 (d, *J* = 8.0 Hz, 2H), 4.72 (s, 2H), 3.89 (s, 3H), 2.56 (s, 1H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 167.1, 146.1, 129.8, 129.2, 126.5, 64.6, 52.1 ppm.

**HRMS** (ESI) calcd for C<sub>9</sub>H<sub>11</sub>O<sub>3</sub> [M+H]: 167.0708, found: 167.0711.



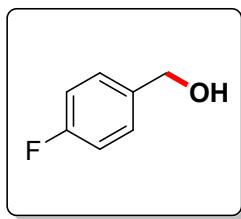
### benzyl alcohol (2b, 12b)<sup>7</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 50 mg, 92% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.43 – 7.25 (m, 5H), 4.68 (s, 2H), 2.19 – 1.91 (b, 1H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 140.9, 128.6, 127.6, 127.0, 65.2 ppm.

**HRMS** (ESI) calcd for C<sub>7</sub>H<sub>9</sub>O [M+H]: 109.0653, found: 109.0655.



**(4-fluorophenyl)methanol (3b)<sup>6</sup>**

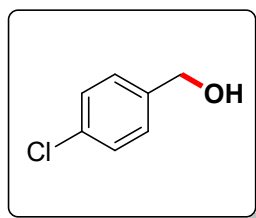
The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 59 mg, 94% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.36 – 7.25 (m, 2H), 7.04 (t, *J* = 8.8 Hz, 2H), 4.60 (s, 2H), 2.78 – 2.59 (b, 1H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 163.5, 161.1, 136.6, 136.5, 128.8, 128.7, 115.5, 115.2, 64.4, 64.4 ppm.

**<sup>19</sup>F NMR** (376 MHz, CDCl<sub>3</sub>) δ -114.9 ppm.

**HRMS** (ESI) calcd for C<sub>7</sub>H<sub>8</sub>FO [M+H]: 127.0558, found: 127.0560.



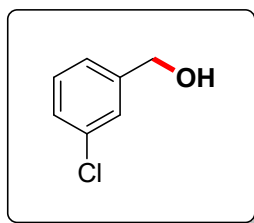
**(4-chlorophenyl)methanol (4b, 18b)<sup>7</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 65 mg, 91% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.30 (d, *J* = 8.4 Hz, 2H), 7.24 (d, *J* = 8.4 Hz, 2H), 4.59 (s, 2H), 2.54 (s, 1H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 139.2, 133.3, 128.7, 128.3, 64.4 ppm.

**HRMS** (ESI) calcd for C<sub>7</sub>H<sub>8</sub>ClO [M+H]: 143.0264, found: 143.0268.



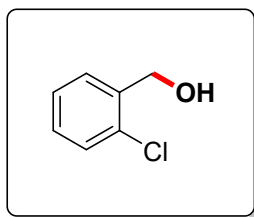
**(3-chlorophenyl)methanol (5b)<sup>6</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 62 mg, 87% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.38 (s, 1H), 7.33 – 7.21 (m, 3H), 4.68 (s, 2H), 2.05 (s, 1H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 142.8, 134.4, 129.8, 127.7, 127.0, 124.9, 64.5 ppm.

**HRMS** (ESI) calcd for C<sub>7</sub>H<sub>8</sub>ClO [M+H]: 143.0264, found: 143.0266.



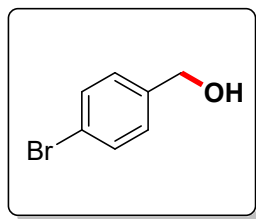
**(2-chlorophenyl)methanol (6b)<sup>6</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 58 mg, 82% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.45 (d, *J* = 7.2 Hz, 1H), 7.34 (d, *J* = 7.6 Hz, 1H), 7.29 – 7.16 (m, 2H), 4.74 (s, 2H), 2.44 (s, 1H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 138.2, 132.7, 129.4, 128.8, 128.7, 127.0, 62.8 ppm.

**HRMS** (ESI) calcd for C<sub>7</sub>H<sub>8</sub>ClO [M+H]: 143.0264, found: 143.0261.



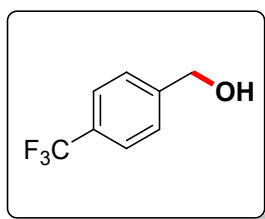
**(4-bromophenyl)methanol (7b)**<sup>7</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 76 mg, 81% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.46 (d, *J* = 8.0 Hz, 2H), 7.20 (d, *J* = 8.0 Hz, 2H), 4.61 (d, *J* = 5.2 Hz, 2H), 2.16 (s, 1H) ppm.

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 139.9, 131.7, 128.7, 121.5, 64.6 ppm.

**HRMS** (ESI) calcd for C<sub>7</sub>H<sub>8</sub>BrO [M+H]: 186.9760, found: 186.9759.



**(4-(trifluoromethyl)phenyl)methanol (8b, 21b)**<sup>7</sup>

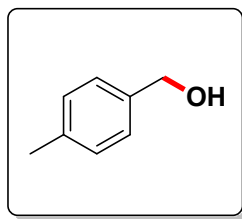
The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 86 mg, 98% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.59 (d, *J* = 8.0 Hz, 2H), 7.42 (d, *J* = 8.0 Hz, 2H), 4.70 (s, 2H), 2.67 – 2.54 (b, 1H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 144.7, 130.2, 129.9, 129.6, 129.3, 128.2, 126.8, 125.5, 125.5, 125.4, 125.4, 125.4, 122.8, 120.1, 64.3 ppm.

**<sup>19</sup>F NMR** (376 MHz, CDCl<sub>3</sub>) δ -62.5 ppm.

**HRMS** (ESI) calcd for C<sub>8</sub>H<sub>8</sub>F<sub>3</sub>O [M+H]: 177.0528, found: 177.0524.



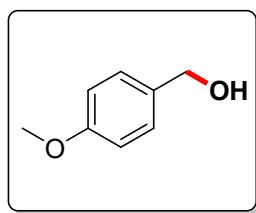
#### 4-methylbenzyl alcohol (9b, 13b)<sup>6</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 51 mg, 83% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.28 – 7.24 (m, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 4.65 (s, 2H), 2.35 (s, 3H), 1.61 (s, 1H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 137.9, 137.3, 129.2, 127.1, 65.0, 21.1.

**HRMS** (ESI) calcd for C<sub>8</sub>H<sub>11</sub>O [M+H]: 123.0810, found: 123.0808.



#### (4-methoxyphenyl)methanol (10b)<sup>7</sup>

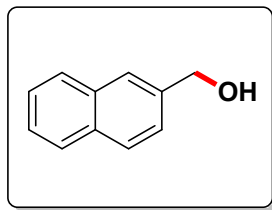
The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 53 mg, 77% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.28 (d, *J* = 8.8 Hz, 2H), 6.90 (d, *J* = 8.8 Hz, 2H), 4.59 (s, 2H), 3.81 (s, 3H), 2.22 (s, 1H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 159.1, 133.1, 128.6, 113.9, 64.9, 55.3 ppm.

**HRMS** (ESI) calcd for C<sub>8</sub>H<sub>11</sub>O<sub>2</sub> [M+H]: 139.0757, found: 139.0760.





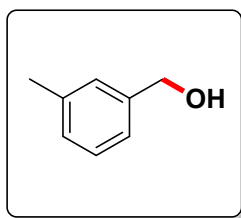
### naphthalen-2-ylmethanol (11b, 23b)<sup>6</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 66 mg, 84% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.90 – 7.73 (m, 4H), 7.55 – 7.41 (m, 3H), 4.79 (s, 2H), 2.56 (s, 1H) ppm.

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 138.4, 133.4, 133.0, 128.3, 128.0, 127.8, 126.2, 125.9, 125.5, 125.2, 65.3 ppm.

HRMS (ESI) calcd for C<sub>11</sub>H<sub>11</sub>O [M+H]: 159.0810, found: 159.0811.



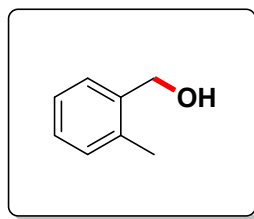
### 3-methylbenzyl alcohol (14b)<sup>8</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 56 mg, 92% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.26 (t, *J* = 7.6 Hz, 1H), 7.20 – 7.10 (m, 3H), 4.63 (s, 2H), 2.37 (s, 3H), 2.13 (s, 1H) ppm.

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 140.8, 138.2, 128.5, 128.4, 127.8, 124.1, 65.3, 21.4 ppm.

HRMS (ESI) calcd for C<sub>8</sub>H<sub>11</sub>O [M+H]: 123.0810, found: 123.0813.



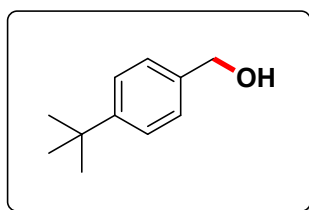
### 2-methylbenzyl alcohol (15b)<sup>7</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 60 mg, 98% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.33 – 7.28 (m, 1H), 7.22 – 7.11 (m, 3H), 4.61 (s, 2H), 2.31 (s, 3H), 2.13 (s, 1H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 138.7, 136.1, 130.3, 127.7, 127.5, 126.0, 63.3, 18.6 ppm.

**HRMS** (ESI) calcd for C<sub>8</sub>H<sub>11</sub>O [M+H]: 123.0810, found: 123.0811.



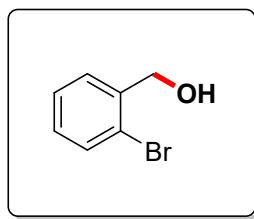
### (4-(tert-butyl)phenyl)methanol (16b)<sup>6</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 77 mg, 94% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.40 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 4.66 (s, 2H), 1.75 (s, 1H), 1.34 (s, 9H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 150.8, 138.1, 127.0, 125.6, 65.3, 34.7, 31.5 ppm.

**HRMS** (ESI) calcd for C<sub>11</sub>H<sub>17</sub>O [M+H]: 165.1281, found: 165.1285.



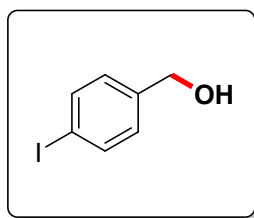
**(2-bromophenyl)methanol (19b)<sup>8</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 80 mg, 86% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.54 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.47 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.32 (td, *J* = 7.6, 1.2 Hz, 1H), 7.16 (td, *J* = 7.6, 2.0 Hz, 1H), 4.73 (s, 2H), 2.22 (s, 1H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 139.7, 132.5, 129.0, 128.8, 127.6, 122.5, 64.9 ppm.

**HRMS** (ESI) calcd for C<sub>7</sub>H<sub>8</sub>BrO [M+H]: 186.9760, found: 186.9758.



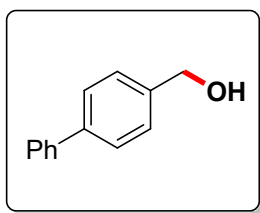
**(4-iodophenyl)methanol (20b)<sup>7</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 95 mg, 81% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.66 (d, *J* = 8.4 Hz, 2H), 7.07 (d, *J* = 8.4 Hz, 2H), 4.59 (s, 2H), 2.19 (s, 1H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 140.4, 137.5, 128.8, 93.0, 64.5 ppm.

**HRMS** (ESI) calcd for C<sub>7</sub>H<sub>8</sub>IO [M+H]: 234.9618, found: 234.9620.



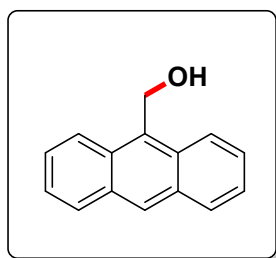
#### 4-biphenylmethanol (22b)<sup>6</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 79 mg, 86% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.61 (d, *J* = 8.0 Hz, 4H), 7.48 – 7.42 (m, 4H), 7.37 (t, *J* = 7.2 Hz, 1H), 4.74 (s, 2H), 1.97 (s, 1H) ppm.

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 140.9, 140.7, 140.0, 128.9, 127.6, 127.4, 127.2, 65.1 ppm.

HRMS (ESI) calcd for C<sub>13</sub>H<sub>13</sub>O [M+H]: 185.0968, found: 185.0971.



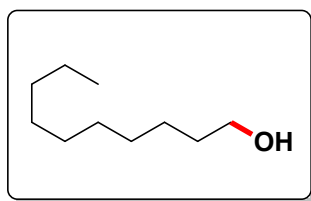
#### anthracen-9-ylmethanol (24b)<sup>9</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 91 mg, 87% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.46 (s, 1H), 8.40 (d, *J* = 8.8 Hz, 2H), 8.03 (d, *J* = 8.0 Hz, 2H), 7.63 – 7.54 (m, 2H), 7.52 – 7.45 (m, 2H), 5.65 (s, 2H), 1.75 (s, 1H) ppm.

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 131.6, 131.0, 130.3, 129.2, 128.4, 126.5, 125.1, 123.9, 57.4 ppm.

HRMS (ESI) calcd for C<sub>15</sub>H<sub>13</sub>O [M+H]: 209.0967, found: 209.0965.



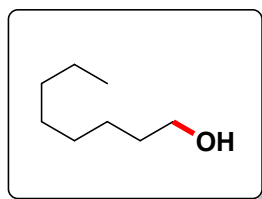
**decan-1-ol (25b)<sup>8</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 76 mg, 96% yield. Purification by column chromatography on silica gel (petroleum ether).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 3.63 (t, *J* = 6.8 Hz, 2H), 1.71 (m, 1H), 1.56 (m, 2H), 1.38 – 1.23 (m, 14H), 0.87 (t, *J* = 6.8 Hz, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 63.1, 32.8, 31.9, 29.6, 29.6, 29.4, 29.3, 25.7, 22.7, 14.1 ppm.

**HRMS** (ESI) calcd for C<sub>10</sub>H<sub>23</sub>O [M+H]: 159.1748, found: 159.1745.



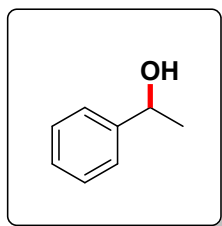
**octan-1-ol (26b)<sup>10</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 61 mg, 94% yield. Purification by column chromatography on silica gel (petroleum ether).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 3.64 (t, *J* = 6.8 Hz, 2H), 1.62 – 1.50 (m, 3H), 1.38 – 1.21 (m, 10H), 0.92 – 0.84 (m, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 63.1, 32.8, 31.8, 29.4, 29.3, 25.7, 22.7, 14.1 ppm.

**HRMS** (ESI) calcd for C<sub>8</sub>H<sub>19</sub>O [M+H]: 131.1439, found: 131.1441.



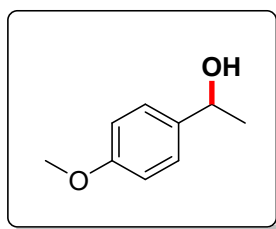
### 1-phenylethanol (27b)<sup>11</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 60 mg, 99% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.43 – 7.27 (m, 5H), 4.91 (q, *J* = 6.4 Hz, 1H), 2.29 (s, 1H), 1.52 (d, *J* = 6.4 Hz, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 145.8, 128.5, 127.4, 125.4, 70.3, 25.1 ppm.

**HRMS** (ESI) calcd for C<sub>8</sub>H<sub>11</sub>O [M+H]: 123.0810, found: 123.0808.



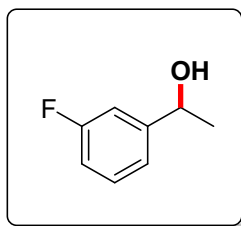
### 1-(4-methoxyphenyl)ethanol (28b)<sup>11</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 71 mg, 93% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.30 (d, *J* = 8.4 Hz, 2H), 6.88 (d, *J* = 8.8 Hz, 2H), 4.85 (q, *J* = 6.4 Hz, 1H), 3.80 (s, 3H), 1.90 (s, 1H), 1.47 (d, *J* = 6.4 Hz, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 159.0, 138.1, 126.7, 113.9, 70.0, 55.4, 25.1 ppm.

**HRMS** (ESI) calcd for C<sub>9</sub>H<sub>13</sub>O<sub>2</sub> [M+H]: 153.0915, found: 153.0917.



### 1-(3-fluorophenyl)ethanol (29b)<sup>12</sup>

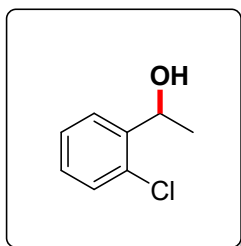
The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 69 mg, 99% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.33 – 7.24 (m, 1H), 7.13 – 7.04 (m, 2H), 6.94 (m, 1H), 4.86 (q, *J* = 6.4 Hz, 1H), 2.32 (s, 1H), 1.46 (d, *J* = 6.4 Hz, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 163.01 (d, *J* = 245.8 Hz), 148.56 (d, *J* = 6.6 Hz), 130.00 (d, *J* = 8.1 Hz), 120.97 (d, *J* = 2.9 Hz), 114.21 (d, *J* = 21.2 Hz), 112.33 (d, *J* = 21.8 Hz), 69.78 (d, *J* = 1.8 Hz), 25.21 ppm.

**<sup>19</sup>F NMR** (376 MHz, CDCl<sub>3</sub>) δ -112.9 ppm.

**HRMS** (ESI) calcd for C<sub>8</sub>H<sub>10</sub>FO [M+H]: 141.0716, found: 141.0718.



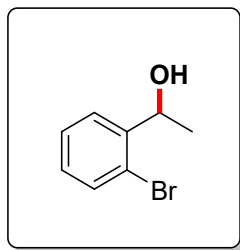
### 1-(2-chlorophenyl)ethanol (30b)<sup>11</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 76 mg, 97% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.59 (d, *J* = 9.6 Hz, 1H), 7.34 – 7.27 (m, 2H), 7.20 (t, *J* = 7.6 Hz, 1H), 5.29 (q, *J* = 6.4 Hz, 1H), 2.11 (s, 1H), 1.49 (d, *J* = 6.4 Hz, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 143.2, 131.7, 129.5, 128.5, 127.3, 126.5, 67.1, 23.6.

**HRMS** (ESI) calcd for C<sub>8</sub>H<sub>10</sub>ClO [M+H]: 157.0420, found: 157.0423.



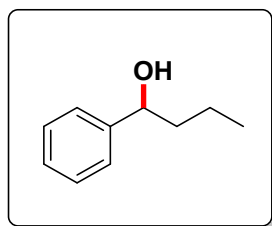
### 1-(2-bromophenyl)ethanol (31b)<sup>11</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 93 mg, 93% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.51 (m, 2H), 7.30 (t, *J* = 7.6 Hz, 1H), 7.12 – 7.05 (m, 1H), 5.18 (q, *J* = 6.4 Hz, 1H), 3.11 (s, 1H), 1.42 (d, *J* = 6.4 Hz, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 144.6, 132.5, 128.6, 127.8, 126.6, 121.5, 69.0, 23.5 ppm.

**HRMS** (ESI) calcd for C<sub>8</sub>H<sub>10</sub>BrO [M+H]: 200.9915, found: 200.9917.



### 1-phenyl-1-butanol (32b)<sup>13</sup>

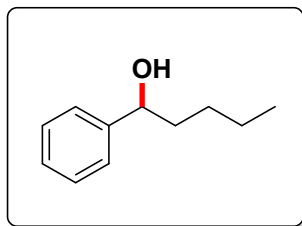
The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 74 mg, 99% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.36 (d, *J* = 4.4 Hz, 4H), 7.31 – 7.25 (m, 1H), 4.68 (dd, *J* = 7.6, 6.0 Hz, 1H), 1.90 (s, 1H), 1.87 – 1.63 (m, 2H), 1.52 – 1.25 (m, 2H), 0.94 (t, *J* = 7.6 Hz, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 145.1, 128.6, 127.6, 126.0, 74.6, 41.4, 19.2, 14.1 ppm.

**HRMS** (ESI) calcd for C<sub>10</sub>H<sub>15</sub>O [M+H]: 151.1123, found: 151.1121.





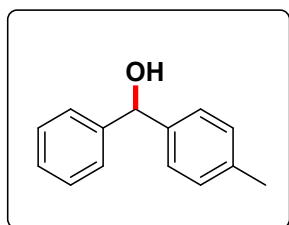
### 1-Phenylpentan-1-ol (33b)<sup>13</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 81 mg, 99% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.34 (d, *J* = 4.0 Hz, 4H), 7.30 – 7.24 (m, 1H), 4.65 (t, *J* = 5.6 Hz, 1H), 1.91 (s, 1H), 1.87 – 1.63 (m, 2H), 1.45 – 1.19 (m, 4H), 0.88 (t, *J* = 7.2 Hz, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 145.0, 128.5, 127.5, 126.0, 74.7, 38.9, 28.0, 22.7, 14.1 ppm.

**HRMS** (ESI) calcd for C<sub>11</sub>H<sub>17</sub>O [M+H]: 165.1279, found: 165.1283.



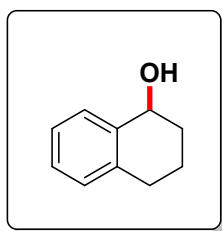
### 4-methylbenzhydrol (34b)<sup>14</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 96 mg, 97% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.42 – 7.32 (m, 4H), 7.28 (d, *J* = 7.2 Hz, 3H), 7.16 (d, *J* = 8.0 Hz, 2H), 5.82 (s, 1H), 2.35 (s, 3H), 2.25 (s, 1H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 144.1, 141.1, 137.4, 129.3, 128.6, 127.6, 126.6, 126.6, 76.2, 21.2 ppm.

**HRMS** (ESI) calcd for C<sub>14</sub>H<sub>15</sub>O [M+H]: 199.1123, found: 199.1126.



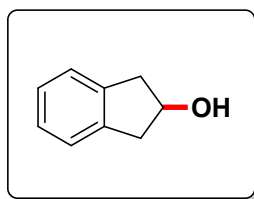
### 1,2,3,4-tetrahydronaphthalen-1-ol (35b)<sup>11</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the yellow oil 72 mg, 97% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.46 – 7.41 (m, 1H), 7.24 – 7.17 (m, 2H), 7.14 – 7.08 (m, 1H), 4.78 (s, 1H), 2.87 – 2.80 (m, 1H), 2.78 – 2.69 (m, 1H), 2.04 – 1.71 (m, 5H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 138.9, 137.2, 129.1, 128.8, 127.7, 126.3, 68.2, 32.4, 29.3, 18.9 ppm.

**HRMS** (ESI) calcd for C<sub>10</sub>H<sub>13</sub>O [M+H]: 149.0966, found: 149.0963.



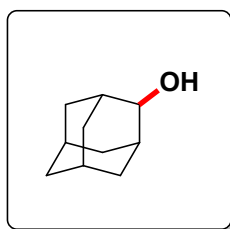
### indan-2-ol (36b)<sup>15</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 66 mg, 99% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.32 – 7.27 (m, 2H), 7.25 – 7.20 (m, 2H), 4.76 – 4.71 (m, 1H), 3.26 (dd, *J* = 16.4, 6.0 Hz, 2H), 2.96 (dd, *J* = 16.4, 3.2 Hz, 2H), 1.91 (s, 1H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 140.9, 126.8, 125.1, 73.3, 42.8 ppm.

**HRMS** (ESI) calcd for C<sub>9</sub>H<sub>11</sub>O [M+H]: 135.0810, found: 135.0807.



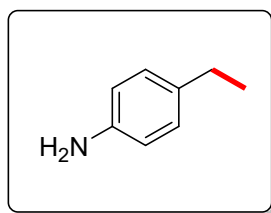
**1-adamantanol (37b)<sup>10</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 75 mg, 99% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 3.86 (s, 1H), 2.06 (d, *J* = 12.8 Hz, 2H), 1.90 – 1.77 (m, 6H), 1.69 (d, *J* = 12.8 Hz, 5H), 1.52 (d, *J* = 10.0 Hz, 2H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 74.7, 37.7, 36.7, 34.7, 31.2, 27.7, 27.2 ppm.

**HRMS** (ESI) calcd for C<sub>10</sub>H<sub>17</sub>O [M+H]: 153.1279, found: 153.1283.

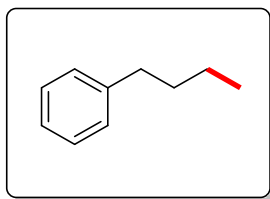


**4-ethylaniline (38b)<sup>16</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the yellow oil 58 mg, 96% yield. Purification by column chromatography on silica gel (petroleum ether).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.04 (d, *J* = 8.4 Hz, 2H), 6.67 (d, *J* = 8.4 Hz, 2H), 3.56 (s, 2H), 2.59 (q, *J* = 7.6 Hz, 2H), 1.23 (t, *J* = 7.6 Hz, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 144.0, 134.6, 128.7, 115.4, 28.0, 16.0 ppm.

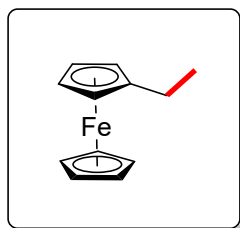


**butylbenzene (39b)<sup>17</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 54 mg, 80% yield. Purification by column chromatography on silica gel (petroleum ether).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.41 – 7.32 (m, 2H), 7.30 – 7.22 (m, 3H), 2.75 – 2.66 (m, 2H), 1.77 – 1.63 (m, 2H), 1.53 – 1.38 (m, 2H), 1.08 – 0.98 (m, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 143.0, 128.5, 128.3, 125.6, 35.8, 33.8, 22.5, 14.0 ppm.

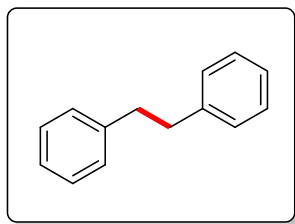


**ethylferrocene (40b)<sup>18</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the orange oil 97 mg, 91% yield. Purification by column chromatography on silica gel (petroleum ether).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 4.27 (d, *J* = 18.8 Hz, 9H), 2.24 (d, *J* = 7.6 Hz, 2H), 1.09 (t, *J* = 6.8 Hz, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 93.2, 70.1, 68.9, 68.4, 22.3, 14.6 ppm.

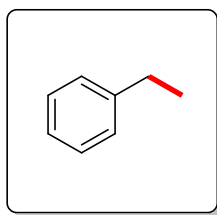


**1,2-diphenylethane (41b, 52b)<sup>16</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 86 mg, 94% yield. Purification by column chromatography on silica gel (petroleum ether).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.34 – 7.28 (m, 4H), 7.25 – 7.20 (m, 6H), 2.95 (s, 4H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 141.8, 128.5, 128.4, 126.0, 38.0 ppm.

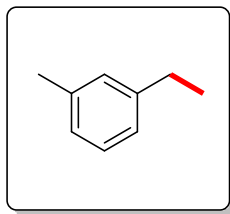


**ethylbenzene (42b)<sup>16</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 52 mg, 99% yield. Purification by column chromatography on silica gel (petroleum ether).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.44 – 7.26 (m, 5H), 2.82 – 2.73 (m, 2H), 1.40 – 1.33 (m, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 144.3, 128.4, 127.9, 125.7, 29.0, 15.7 ppm.

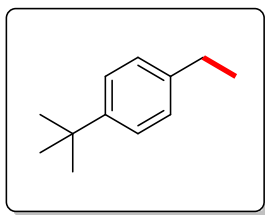


**1-ethyl-3-methylbenzene (43b)<sup>16</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 59 mg, 99% yield. Purification by column chromatography on silica gel (petroleum ether).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.27 (t, *J* = 7.2 Hz, 1H), 7.13 – 7.06 (m, 3H), 2.71 (q, *J* = 7.6 Hz, 2H), 2.42 (s, 3H), 1.32 (t, *J* = 7.6 Hz, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 144.3, 137.9, 128.8, 128.3, 126.4, 124.9, 28.9, 21.5, 15.7 ppm.

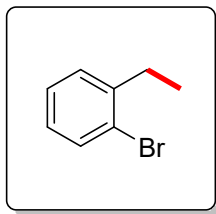


**1-(tert-butyl)-4-ethylbenzene (44b)<sup>16</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 79 mg, 97% yield. Purification by column chromatography on silica gel (petroleum ether).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.39 – 7.32 (m, 2H), 7.22 – 7.14 (m, 2H), 2.70 – 2.61 (m, 2H), 1.38 – 1.32 (m, 9H), 1.32 – 1.23 (m, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 148.4, 141.2, 127.5, 125.2, 34.4, 31.5, 28.3, 15.5 ppm.

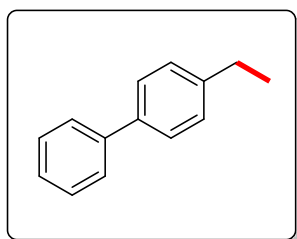


**1-bromo-2-ethylbenzene (45b)<sup>16</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 80 mg, 87% yield. Purification by column chromatography on silica gel (petroleum ether).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.57 (d, *J* = 7.6 Hz, 1H), 7.33 – 7.24 (m, 2H), 7.13 – 7.05 (m, 1H), 2.81 (q, *J* = 7.6 Hz, 2H), 1.28 (t, *J* = 7.6 Hz, 3H) ppm.

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 143.3, 132.7, 129.5, 127.5, 127.4, 124.4, 29.4, 14.3 ppm.

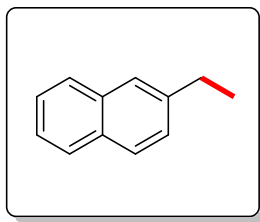


**4-ethyl-1,1'-biphenyl (46b)<sup>16</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 89 mg, 98% yield. Purification by column chromatography on silica gel (petroleum ether).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.60 (d, *J* = 6.8 Hz, 2H), 7.54 (d, *J* = 8.4 Hz, 2H), 7.44 (t, *J* = 7.6 Hz, 2H), 7.37 – 7.27 (m, 3H), 2.72 (q, *J* = 7.6 Hz, 2H), 1.30 (t, *J* = 7.6 Hz, 3H) ppm.

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 143.4, 141.2, 138.6, 128.7, 128.3, 127.1, 127.0, 127.0, 28.5, 15.6 ppm.

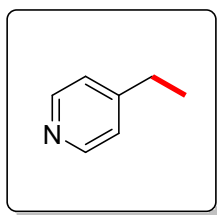


### 2-ethylnaphthalene (47b)<sup>19</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 76 mg, 97% yield. Purification by column chromatography on silica gel (petroleum ether).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.89 – 7.79 (m, 3H), 7.68 (d, *J* = 4.4 Hz, 1H), 7.54 – 7.36 (m, 3H), 2.92 – 2.81 (m, 2H), 1.43 – 1.35 (m, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 141.8, 133.8, 132.0, 127.9, 127.7, 127.5, 127.2, 125.9, 125.6, 125.1, 29.1, 15.6 ppm.



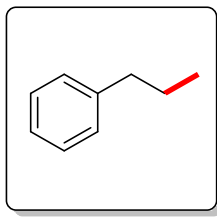
### 4-ethylpyridine (48b)<sup>20</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the yellow oil 50 mg, 94% yield. Purification by column chromatography on silica gel (petroleum ether).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.46 – 8.39 (m, 2H), 7.07 – 7.04 (m, 2H), 2.62 – 2.55 (m, 2H), 1.21 – 1.16 (m, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 153.0, 149.5, 123.4, 28.1, 14.3 ppm.



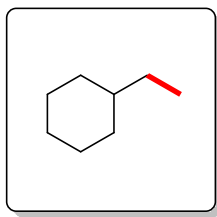


**propylbenzene (49b)<sup>21</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 58 mg, 97% yield. Purification by column chromatography on silica gel (petroleum ether).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.37 – 7.29 (m, 2H), 7.26 – 7.19 (m, 3H), 2.67 – 2.61 (m, 2H), 1.77 – 1.65 (m, 2H), 1.00 (t, *J* = 7.2 Hz, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 142.7, 128.5, 128.3, 125.7, 38.1, 24.7, 13.9 ppm.

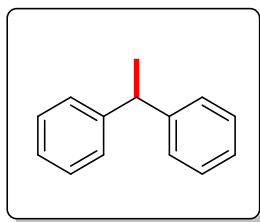


**ethylcyclohexane (50b)<sup>22</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 55 mg, 98% yield. Purification by column chromatography on silica gel (petroleum ether).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 1.74 – 1.60 (m, 5H), 1.29 – 1.03 (m, 6H), 0.86 (t, *J* = 7.4 Hz, 5H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 39.5, 33.0, 30.1, 26.8, 26.5, 11.4 ppm.

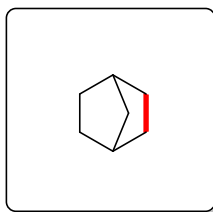


**1,1-diphenylethane (51b)<sup>23</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 87 mg, 95% yield. Purification by column chromatography on silica gel (petroleum ether).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.43 – 7.24 (m, 10H), 4.27 (q, *J* = 7.2 Hz, 1H), 1.76 (d, *J* = 7.2 Hz, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 146.5, 128.5, 127.7, 126.1, 44.9, 22.0 ppm.

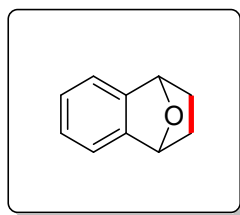


**norbornane (53b)<sup>24</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless solid 47 mg, 99% yield. Purification by column chromatography on silica gel (petroleum ether).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 2.22 – 2.16 (m, 2H), 1.52 – 1.37 (m, 4H), 1.24 – 1.07 (m, 6H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 38.4, 36.4, 29.7 ppm.

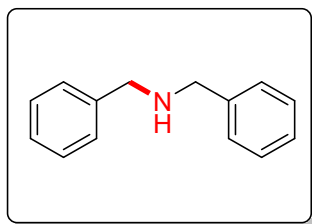


**1,2,3,4-tetrahydro-1,4-epoxynaphthalene (54b)<sup>25</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the yellow oil 72 mg, 98% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.17 – 7.10 (m, 2H), 7.06 (dd, *J* = 5.2, 3.2 Hz, 2H), 5.31 (dd, *J* = 3.2, 2.0 Hz, 2H), 2.03 – 1.91 (m, 2H), 1.36 – 1.22 (m, 2H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 145.7, 126.5, 118.7, 78.9, 26.6 ppm.

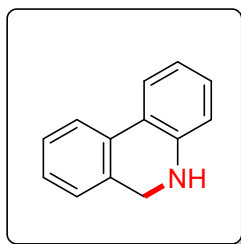


**dibenzylamine (55b)<sup>26</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 97 mg, 98% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.37 – 7.29 (m, 8H), 7.27 – 7.21 (m, 2H), 3.80 (s, 4H), 1.70 (s, 1H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 140.4, 128.5, 128.2, 127.0, 53.2 ppm.

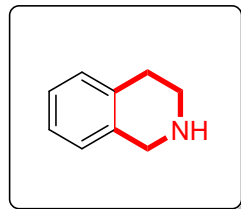


### 5,6-dihydrophenanthridine (56b)<sup>27</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 81 mg, 89% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.66 (dd, *J* = 8.0, 3.6 Hz, 2H), 7.28 (t, *J* = 7.6 Hz, 1H), 7.18 (t, *J* = 7.2 Hz, 1H), 7.07 (d, *J* = 8.0 Hz, 2H), 6.81 (t, *J* = 7.6 Hz, 1H), 6.61 (d, *J* = 8.0 Hz, 1H), 4.33 (s, 2H), 3.57 (s, 1H) ppm.

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 145.8, 132.8, 132.1, 128.9, 127.7, 127.2, 126.1, 123.6, 122.5, 122.1, 119.3, 115.2, 46.4 ppm.

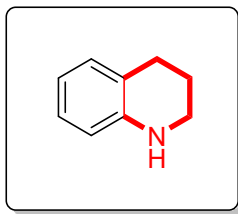


### 1,2,3,4-tetrahydroisoquinoline (57b)<sup>28</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the yellow oil 47 mg, 70% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.19 – 7.05 (m, 3H), 7.04 – 6.96 (m, 1H), 4.01 (s, 2H), 3.13 (t, *J* = 6.0 Hz, 2H), 2.79 (t, *J* = 6.0 Hz, 2H), 1.84 (s, 1H) ppm.

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 136.0, 134.8, 129.4, 126.2, 126.0, 125.7, 48.4, 44.0, 29.2 ppm.

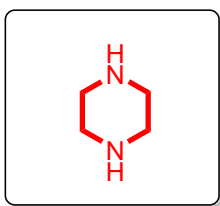


**1,2,3,4-tetrahydroquinoline (58b)<sup>28</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the yellow oil 62 mg, 93% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.04 – 6.96 (m, 2H), 6.65 (t, *J* = 7.6 Hz, 1H), 6.51 (d, *J* = 8.0 Hz, 1H), 3.73 (s, 1H), 3.37 – 3.26 (m, 2H), 2.80 (t, *J* = 6.4 Hz, 2H), 2.03 – 1.93 (m, 2H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 144.8, 129.6, 126.8, 121.5, 117.0, 114.3, 42.0, 27.0, 22.2 ppm.

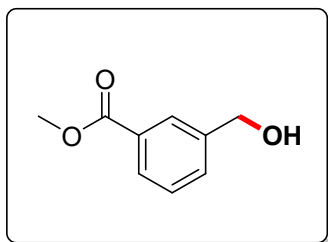


**piperazine (59b)<sup>28</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the yellow solid 40 mg, 93% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 2.83 (s, 8H), 1.91 (d, *J* = 4.4 Hz, 2H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 47.2 ppm.



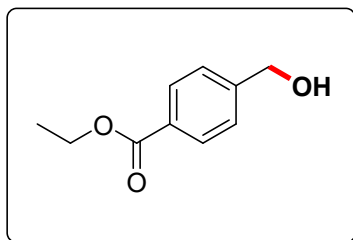
**methyl 3-(hydroxymethyl)benzoate (60b)<sup>29</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 67 mg, 81% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.02 (s, 1H), 7.94 (d, *J* = 7.6 Hz, 1H), 7.56 (d, *J* = 7.6 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 1H), 4.73 (s, 2H), 3.91 (s, 3H), 2.03 (s, 1H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 167.1, 141.3, 131.4, 130.3, 128.8, 128.6, 127.9, 64.7, 52.2 ppm.

**HRMS** (ESI) calcd for C<sub>9</sub>H<sub>11</sub>O<sub>3</sub> [M+H]: 167.0708, found: 167.0712.



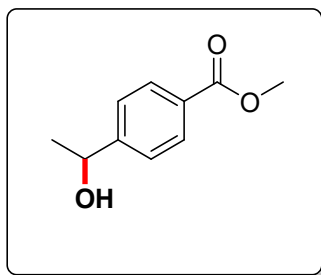
**ethyl 4-(hydroxymethyl)benzoate (62b)<sup>30</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 77 mg, 85% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.99 (d, *J* = 8.4 Hz, 2H), 7.39 (d, *J* = 8.0 Hz, 2H), 4.73 (s, 2H), 4.35 (q, *J* = 7.2 Hz, 2H), 2.48 (s, 1H), 1.38 (t, *J* = 7.2 Hz, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 166.6, 146.0, 129.8, 129.5, 126.4, 64.6, 61.0, 14.3 ppm.

**HRMS** (ESI) calcd for C<sub>10</sub>H<sub>13</sub>O<sub>3</sub> [M+H]: 181.0865, found: 181.0868.



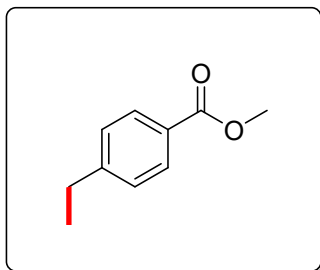
**methyl 4-(1-hydroxyethyl)benzoate (64b)<sup>31</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 80 mg, 89% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.98 (d, *J* = 8.4 Hz, 2H), 7.41 (d, *J* = 7.2 Hz, 2H), 4.93 (q, *J* = 6.4 Hz, 1H), 3.89 (s, 3H), 2.21 (s, 1H), 1.48 (d, *J* = 6.4 Hz, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 167.0, 151.0, 129.8, 129.1, 125.3, 69.9, 52.1, 25.3 ppm.

**HRMS** (ESI) calcd for C<sub>10</sub>H<sub>13</sub>O<sub>3</sub> [M+H]: 181.0865, found: 181.0866.



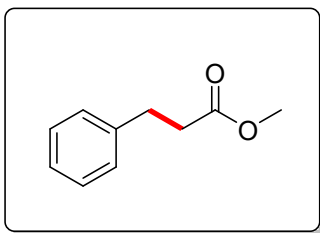
**methyl 4-ethylbenzoate (66b)<sup>32</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 50 mg, 61% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.98 (d, *J* = 8.4 Hz, 2H), 7.28 (d, *J* = 8.4 Hz, 2H), 3.92 (s, 3H), 2.72 (q, *J* = 7.6 Hz, 2H), 1.27 (t, *J* = 7.6 Hz, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 167.2, 149.8, 129.7, 127.9, 127.6, 51.9, 29.0, 15.2 ppm.

**HRMS** (ESI) calcd for C<sub>10</sub>H<sub>13</sub>O<sub>2</sub> [M+H]: 165.0916, found: 165.0921.



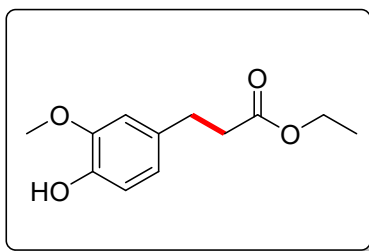
**methyl 3-phenylpropanoate (67b, 89b)<sup>33</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 80 mg, 98% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.35 – 7.27 (m, 2H), 7.26 – 7.18 (m, 3H), 3.68 (s, 3H), 2.97 (t, *J* = 8.0 Hz, 2H), 2.65 (t, *J* = 8.0 Hz, 2H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 173.4, 140.5, 128.5, 128.3, 126.3, 51.6, 35.7, 31.0 ppm.

**HRMS** (ESI) calcd for C<sub>10</sub>H<sub>13</sub>O<sub>2</sub> [M+H]: 165.0916, found: 165.0916.



**ethyl 3-(4-hydroxy-3-methoxyphenyl)propanoate (68b)<sup>34</sup>**

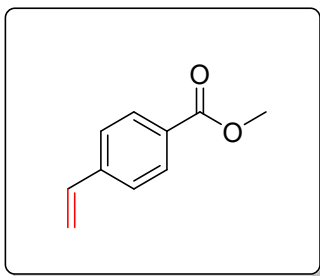
The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 110 mg, 98% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 6.82 (d, *J* = 8.0 Hz, 1H), 6.73 – 6.65 (m, 2H), 5.66 (s, 1H), 4.12 (q, *J* = 7.2 Hz, 2H), 3.85 (s, 3H), 2.87 (t, *J* = 8.0 Hz, 2H), 2.59 (dd, *J* = 8.4, 7.2 Hz, 2H), 1.23 (t, *J* = 7.2 Hz, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 173.1, 146.5, 144.0, 132.5, 120.8, 114.4, 111.0, 60.4, 55.8, 36.4, 30.7, 14.2 ppm.

**HRMS** (ESI) calcd for C<sub>12</sub>H<sub>17</sub>O<sub>4</sub> [M+H]: 225.1127, found: 225.1126.





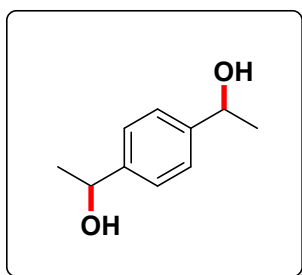
**ethyl 3-(4-hydroxy-3-methoxyphenyl)propanoate (69b)<sup>35</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 33 mg, 41% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.99 (d, *J* = 8.4 Hz, 2H), 7.46 (d, *J* = 8.4 Hz, 2H), 6.75 (dd, *J* = 17.6, 10.8 Hz, 1H), 5.86 (d, *J* = 16.8 Hz, 1H), 5.38 (d, *J* = 10.0 Hz, 1H), 3.91 (s, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 166.9, 141.9, 136.0, 129.9, 129.3, 126.1, 116.5, 52.1.

**HRMS** (ESI) calcd for C<sub>10</sub>H<sub>11</sub>O<sub>2</sub> [M+H]: 163.0759, found: 163.0763.



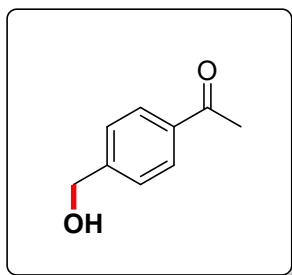
**1,1'-(1,4-phenylene)bis(ethan-1-ol) (73b)<sup>36</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 80 mg, 96% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.35 (s, 4H), 4.89 (q, *J* = 6.4 Hz, 2H), 1.87 (s, 2H), 1.49 (d, *J* = 6.4 Hz, 6H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 145.1, 125.6, 70.2, 25.2 ppm.

**HRMS** (ESI) calcd for C<sub>10</sub>H<sub>15</sub>O<sub>2</sub> [M+H]: 167.1073, found: 167.1070.



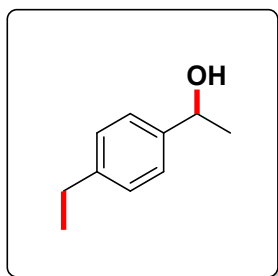
**1-(4-(hydroxymethyl)phenyl)ethan-1-one (74b)<sup>37</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 62 mg, 82% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.91 (d, *J* = 8.4 Hz, 2H), 7.43 (d, *J* = 8.0 Hz, 2H), 4.75 (s, 2H), 2.57 (s, 3H), 2.43 (s, 1H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 198.2, 146.4, 136.2, 128.6, 126.6, 64.5, 26.7 ppm.

**HRMS** (ESI) calcd for C<sub>9</sub>H<sub>11</sub>O<sub>2</sub> [M+H]: 151.0759, found: 151.0757.



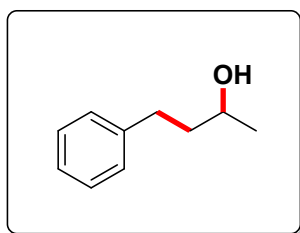
**1-(4-ethylphenyl)ethan-1-ol (75b)<sup>31</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 74 mg, 99% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.30 (d, *J* = 8.0 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 2H), 4.88 (q, *J* = 6.4 Hz, 1H), 2.65 (q, *J* = 7.6 Hz, 2H), 1.78 (s, 1H), 1.49 (d, *J* = 6.4 Hz, 3H), 1.24 (t, *J* = 7.6 Hz, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 143.6, 143.1, 128.0, 125.5, 70.3, 28.5, 25.0, 15.6 ppm.

**HRMS** (ESI) calcd for C<sub>10</sub>H<sub>15</sub>O [M+H]: 151.1123, found: 151.1128.



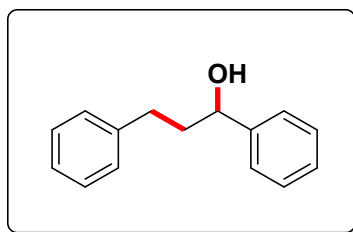
#### 4-phenylbutan-2-ol (76b)<sup>14</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 65 mg, 87% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.34 – 7.28 (m, 2H), 7.26 – 7.16 (m, 3H), 3.90 – 3.78 (m, 1H), 2.84 – 2.63 (m, 2H), 1.96 (s, 1H), 1.88 – 1.71 (m, 2H), 1.25 (d, *J* = 6.4 Hz, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 142.1, 128.4, 125.9, 67.5, 40.9, 32.2, 23.6 ppm.

**HRMS** (ESI) calcd for C<sub>10</sub>H<sub>15</sub>O [M+H]: 151.1123, found: 151.1122.



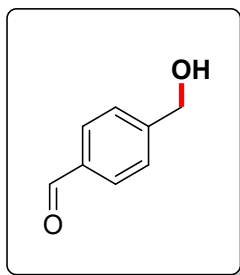
#### 1,3-diphenylpropan-1-ol (77b)<sup>14</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 93 mg, 88% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.38 (d, *J* = 5.2 Hz, 4H), 7.34 – 7.27 (m, 3H), 7.25 – 7.17 (m, 3H), 4.70 (dd, *J* = 7.6, 5.2 Hz, 1H), 2.85 – 2.58 (m, 2H), 2.22 – 1.98 (m, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 144.58, 141.82, 128.56, 128.49, 128.44, 127.69, 125.99, 125.91, 73.93, 40.48, 32.09 ppm.

**HRMS** (ESI) calcd for C<sub>15</sub>H<sub>17</sub>O [M+H]: 213.1279, found: 213.1282.



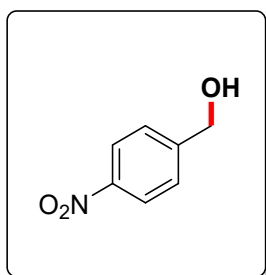
#### 4-(hydroxymethyl)benzaldehyde (82b)<sup>6</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the yellow oil 60 mg, 88% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 9.97 (s, 1H), 7.85 (d, *J* = 8.4 Hz, 2H), 7.51 (d, *J* = 8.0 Hz, 2H), 4.78 (s, 2H), 2.27 (s, 1H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 192.2, 147.9, 135.6, 130.0, 127.0, 64.5 ppm.

**HRMS** (ESI) calcd for C<sub>8</sub>H<sub>9</sub>O<sub>2</sub> [M+H]: 137.0603, found: 137.0605.



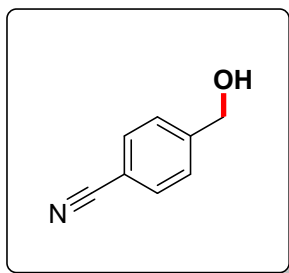
#### (4-nitrophenyl)methanol (83b)<sup>7</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the yellow solid 61 mg, 80% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.22 (d, *J* = 8.8 Hz, 2H), 7.53 (d, *J* = 8.4 Hz, 2H), 4.84 (s, 2H), 1.99 (s, 1H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 148.1, 127.0, 123.8, 64.0 ppm.

**HRMS** (ESI) calcd for C<sub>7</sub>H<sub>8</sub>NO<sub>3</sub> [M+H]: 154.0504, found: 154.0506.



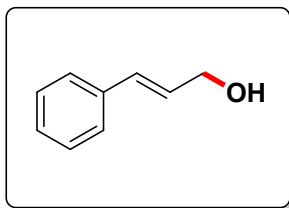
**4-(hydroxymethyl)benzonitrile (84b)<sup>40</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 37 mg, 56% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.64 (d, *J* = 8.4 Hz, 2H), 7.47 (d, *J* = 8.4 Hz, 2H), 4.78 (s, 2H), 2.03 (s, 1H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 146.2, 132.3, 127.0, 118.9, 111.2, 64.2 ppm.

**HRMS** (ESI) calcd for C<sub>8</sub>H<sub>8</sub>NO [M+H]: 134.0606, found: 134.0610.



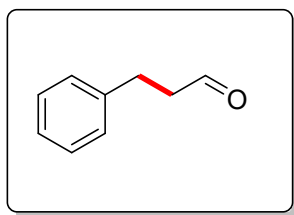
**Cinnamyl alcohol (85b)<sup>7</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 39 mg, 58% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.41 – 7.37 (m, 2H), 7.35 – 7.30 (m, 2H), 7.28 – 7.21 (m, 1H), 6.62 (d, *J* = 16.0 Hz, 1H), 6.46 – 6.25 (m, 1H), 4.33 (d, *J* = 5.6 Hz, 2H), 1.65 (s, 1H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 136.7, 131.2, 128.6, 128.5, 127.7, 126.5, 63.8 ppm.

**HRMS** (ESI) calcd for C<sub>9</sub>H<sub>11</sub>O [M+H]: 135.0810, found: 135.0807.



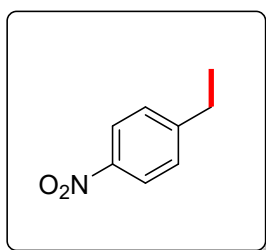
### 3-phenylpropanal (86b)<sup>38</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 27 mg, 41% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 9.83 (s, 1H), 7.31 (t, *J* = 7.3 Hz, 2H), 7.26 – 7.17 (m, 3H), 2.97 (t, *J* = 7.5 Hz, 2H), 2.79 (t, *J* = 7.3 Hz, 2H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 201.6, 140.4, 128.6, 128.3, 126.3, 45.3, 28.1 ppm.

**HRMS** (ESI) calcd for C<sub>9</sub>H<sub>10</sub>O [M+H]: 135.0810, found: 135.0812.

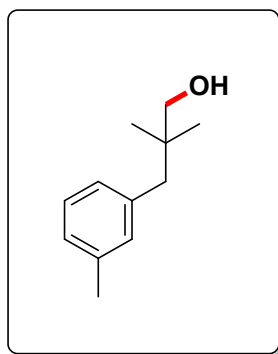


### 1-ethyl-4-nitrobenzene (87b)<sup>39</sup>

The title compound was prepared according to the general procedure and purified by column chromatography to give the yellow oil 32 mg, 42% yield. Purification by column chromatography on silica gel (petroleum ether).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 8.13 (d, *J* = 8.8 Hz, 2H), 7.34 (d, *J* = 8.8 Hz, 2H), 2.75 (q, *J* = 7.6 Hz, 2H), 1.28 (t, *J* = 7.6 Hz, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 152.0, 146.3, 128.7, 123.6, 28.9, 15.1 ppm.



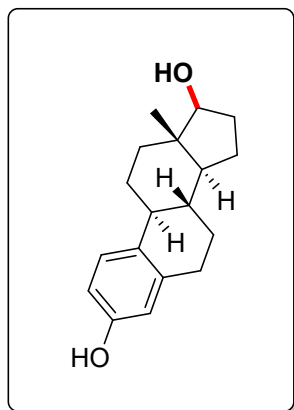
**Majantol (90b)<sup>5</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the colorless oil 80 mg, 90% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.18 (d, *J* = 7.6 Hz, 1H), 7.05 (d, *J* = 7.6 Hz, 1H), 6.99 (d, *J* = 8.0 Hz, 2H), 3.34 (s, 2H), 2.56 (s, 2H), 2.36 (s, 3H), 1.67 (s, 1H), 0.91 (s, 6H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 138.8, 137.4, 131.3, 127.8, 127.6, 126.7, 71.3, 44.7, 36.4, 24.1, 21.5 ppm.

**HRMS** (ESI) calcd for C<sub>12</sub>H<sub>19</sub>O [M+H]: 179.1436, found: 179.1440.



### Estradiol (91b)<sup>5</sup>

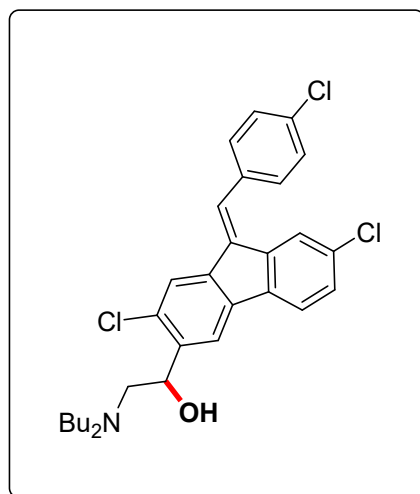
The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 114 mg, 84% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  8.96 (s, 1H), 7.05 (d, *J* = 8.8 Hz, 1H), 6.50 (dd, *J* = 8.4, 2.6 Hz, 1H), 6.43 (d, *J* = 2.8 Hz, 1H), 4.34 (d, *J* = 4.4 Hz, 1H), 3.61 – 3.54 (m, 1H), 2.80 – 2.63 (m, 2H), 2.31 – 2.22 (m, 1H), 2.11 – 1.98 (m, 2H), 1.85 – 1.64 (m, 3H), 1.61 – 1.49 (m, 1H), 1.46 – 1.06 (m, 6H), 0.61 (s, 3H) ppm.

**<sup>13</sup>C NMR** (101 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  156.6, 138.8, 132.2, 127.8, 116.6, 114.4, 79.7, 48.9, 46.7, 45.4, 40.6, 33.8, 33.2, 31.0, 29.6, 27.8, 25.6, 18.7 ppm.

**HRMS** (ESI) calcd for C<sub>18</sub>H<sub>25</sub>O<sub>2</sub> [M+H]: 273.1855, found: 273.1852.





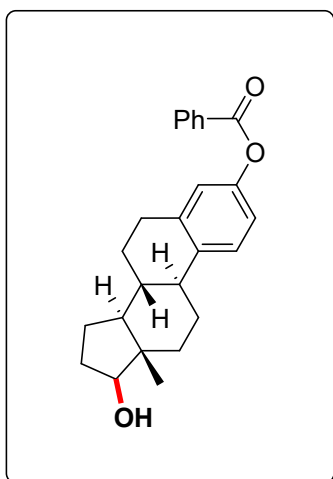
**Lumefantrine (92b)<sup>5</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the yellow solid 164 mg, 62% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

**<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>) δ 7.74 (d, *J* = 2.0 Hz, 1H), 7.68 (d, *J* = 2.0 Hz, 1H), 7.64 – 7.56 (m, 2H), 7.51 – 7.42 (m, 5H), 7.33 (dd, *J* = 8.3, 2.0 Hz, 1H), 5.36 (dd, *J* = 10.2, 3.5 Hz, 1H), 4.54 (s, 1H), 2.88 (dd, *J* = 13.0, 3.5 Hz, 1H), 2.75 – 2.63 (m, 2H), 2.58 – 2.40 (m, 3H), 1.56 – 1.45 (m, 4H), 1.43 – 1.28 (m, 4H), 0.97 (t, *J* = 7.3 Hz, 6H) ppm.

**<sup>13</sup>C NMR** (101 MHz, CDCl<sub>3</sub>) δ 141.6, 139.9, 138.3, 136.5, 135.0, 135.0, 134.7, 134.2, 133.2, 132.9, 130.6, 129.1, 128.4, 127.7, 126.4, 124.0, 123.1, 120.7, 65.5, 60.0, 53.4, 29.1, 20.7, 14.1 ppm.

**HRMS** (ESI) calcd for C<sub>30</sub>H<sub>33</sub>Cl<sub>3</sub>NO [M+H]: 529.1662, found: 529.1667.



**Estradiol benzoate (93b)<sup>5</sup>**

The title compound was prepared according to the general procedure and purified by column chromatography to give the white solid 134 mg, 71% yield. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1 – 2:1).

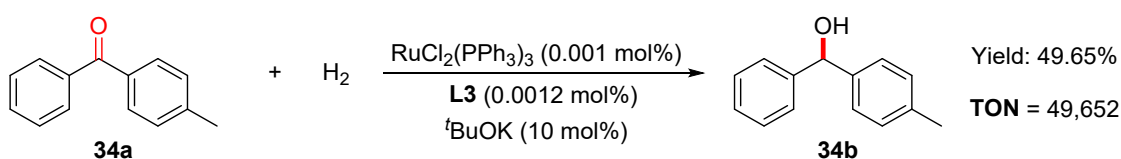
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.20 (dd, *J* = 8.4, 1.4 Hz, 2H), 7.63 (t, *J* = 7.6 Hz, 1H), 7.51 (t, *J* = 7.6 Hz, 2H), 7.34 (d, *J* = 8.8 Hz, 1H), 6.98 (dd, *J* = 8.4, 2.8 Hz, 1H), 6.93 (d, *J* = 2.4 Hz, 1H), 3.79 – 3.69 (m, 1H), 2.98 – 2.82 (m, 2H), 2.39 – 2.31 (m, 1H), 2.30 – 2.21 (m, 1H), 2.20 – 2.06 (m, 1H), 2.01 – 1.94 (m, 1H), 1.95 – 1.85 (m, 1H), 1.78 – 1.65 (m, 1H), 1.62 – 1.22 (m, 8H), 0.80 (s, 3H) ppm.

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 165.5, 148.7, 138.4, 138.1, 133.5, 130.2, 129.8, 128.6, 126.5, 121.7, 118.7, 81.9, 50.1, 44.2, 43.3, 38.5, 36.7, 30.6, 29.6, 27.1, 26.2, 23.2, 11.1 ppm.

HRMS (ESI) calcd for C<sub>25</sub>H<sub>29</sub>O<sub>3</sub> [M+H]: 377.2117, found: 377.2113.

## 6. Gram scale experiments

### 6.1



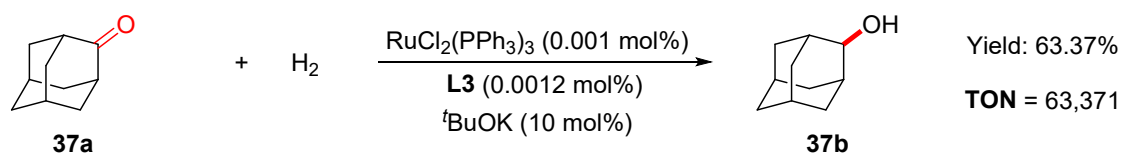
**10 mmol Scale:** In a nitrogen-filled glove box, add a magnetic stirring bar, 4-methylbenzophenone **34a** (10.0 mmol),  $t\text{BuOK}$  (1.0 mmol, 10 mol%),  $\text{RuCl}_2(\text{PPh}_3)_3$  (0.001 mol%), **L3** (0.0012 mol%), and 1,4-dioxane (20.0 mL) to a dried high-pressure reactor. After sealing, remove the high-pressure reactor from the glove box. Then the reactor was purged and charged with  $\text{H}_2$  (2.0 MPa) and immersed into a pre-heated metal bath (110°C). After 48 hours the reaction was cooled, release  $\text{H}_2$  from the reactor. Then quenched with saturated brine and DCM, and dried with  $\text{Na}_2\text{SO}_4$ . A small aliquot of the organic phase was analyzed by GC or GC-MS to monitor product formation. Then the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 100:1 – 2:1) on silica gel to give the corresponding product **34b** in the 49.65% yield.

$$TON = \frac{N_s}{N_c} = \frac{984.4 \text{ mg}}{198.26 \text{ mg/mmol}} = 49,652$$

$N_s$ : The number of molecules of the substrate transformed in the reaction

$N_c$ : The number of active catalyst centers involved in the reaction

## 6.2



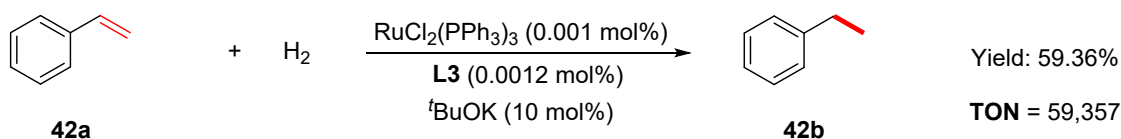
**10 mmol Scale:** In a nitrogen-filled glove box, add a magnetic stirring bar, 2-adamantanone **37a** (10.0 mmol), <sup>t</sup>BuOK (1.0 mmol, 10 mol%), RuCl<sub>2</sub>(PPh<sub>3</sub>)<sub>3</sub> (0.001 mol%), **L3** (0.0012 mol%), and 1,4-dioxane (20.0 mL) to a dried high-pressure reactor. After sealing, remove the high-pressure reactor from the glove box. Then the reactor was purged and charged with H<sub>2</sub> (2.0 MPa) and immersed into a pre-heated metal bath (110°C). After 48 hours the reaction was cooled, release H<sub>2</sub> from the reactor. Then quenched with saturated brine and DCM, and dried with Na<sub>2</sub>SO<sub>4</sub>. A small aliquot of the organic phase was analyzed by GC or GC-MS to monitor product formation. Then the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 100:1 – 2:1) on silica gel to give the corresponding product **37b** in the 63.37% yield.

$$TON = \frac{N_s}{N_c} = \frac{964.7 \text{ mg}}{\frac{152.23 \text{ mg/mmol}}{0.0001 \text{ mmol}}} = 63,371$$

N<sub>s</sub>: The number of molecules of the substrate transformed in the reaction

N<sub>c</sub>: The number of active catalyst centers involved in the reaction

## 6.3



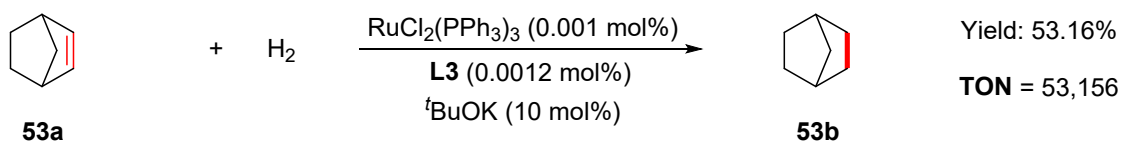
**10 mmol Scale:** In a nitrogen-filled glove box, add a magnetic stirring bar, styrene **42a** (10.0 mmol),  $t\text{BuOK}$  (1.0 mmol, 10 mol%),  $\text{RuCl}_2(\text{PPh}_3)_3$  (0.001 mol%), **L3** (0.0012 mol%), and 1,4-dioxane (20.0 mL) to a dried high-pressure reactor. After sealing, remove the high-pressure reactor from the glove box. Then the reactor was purged and charged with  $\text{H}_2$  (2.0 MPa) and immersed into a pre-heated metal bath (110°C). After 48 hours the reaction was cooled, release  $\text{H}_2$  from the reactor. Then quenched with saturated brine and DCM, and dried with  $\text{Na}_2\text{SO}_4$ . A small aliquot of the organic phase was analyzed by GC or GC-MS to monitor product formation. Then the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 100:1 – 2:1) on silica gel to give the corresponding product **42b** in the 59.36% yield.

$$TON = \frac{N_s}{N_c} = \frac{630.2 \text{ mg}}{\frac{106.17 \text{ mg/mmol}}{0.0001 \text{ mmol}}} = 59,357$$

$N_s$ : The number of molecules of the substrate transformed in the reaction

$N_c$ : The number of active catalyst centers involved in the reaction

## 6.4



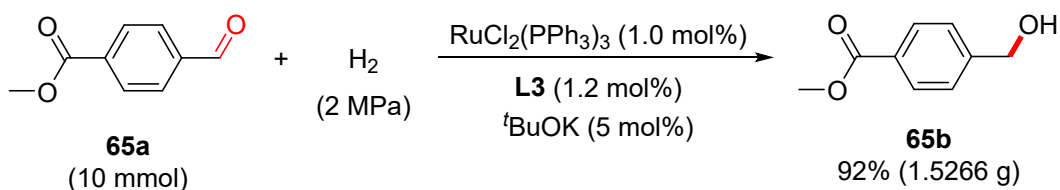
**10 mmol Scale:** In a nitrogen-filled glove box, add a magnetic stirring bar, norbornylene **53a** (10.0 mmol), <sup>t</sup>BuOK (1.0 mmol, 10 mol%), RuCl<sub>2</sub>(PPh<sub>3</sub>)<sub>3</sub> (0.001 mol%), **L3** (0.0012 mol%), and 1,4-dioxane (20.0 mL) to a dried high-pressure reactor. After sealing, remove the high-pressure reactor from the glove box. Then the reactor was purged and charged with H<sub>2</sub> (2.0 MPa) and immersed into a pre-heated metal bath (110°C). After 48 hours the reaction was cooled, release H<sub>2</sub> from the reactor. Then quenched with saturated brine and DCM, and dried with Na<sub>2</sub>SO<sub>4</sub>. A small aliquot of the organic phase was analyzed by GC or GC-MS to monitor product formation. Then the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 100:1 – 2:1) on silica gel to give the corresponding product **53b** in the 53.16% yield.

$$TON = \frac{N_s}{N_c} = \frac{511.2 \text{ mg}}{96.17 \text{ mg/mmol}} \div \frac{0.0001 \text{ mmol}}{1} = 53,156$$

N<sub>s</sub>: The number of molecules of the substrate transformed in the reaction

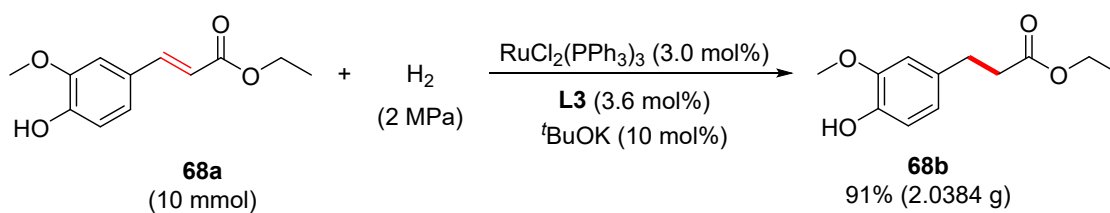
N<sub>c</sub>: The number of active catalyst centers involved in the reaction

## 6.5



**10 mmol Scale:** In a nitrogen-filled glove box, add a magnetic stirring bar, **65a** (10.0 mmol),  $t\text{BuOK}$  (5 mol%),  $\text{RuCl}_2(\text{PPh}_3)_3$  (1 mol%), **L3** (1.2 mol%), and 1,4-dioxane (20.0 mL) to a dried high-pressure reactor. After sealing, remove the high-pressure reactor from the glove box. Then the reactor was purged and charged with  $\text{H}_2$  (2.0 MPa) and immersed into a pre-heated metal bath (110°C). After 24 hours the reaction was cooled, release  $\text{H}_2$  from the reactor. Then quenched with saturated brine and DCM, and dried with  $\text{Na}_2\text{SO}_4$ . A small aliquot of the organic phase was analyzed by GC or GC-MS to monitor product formation. Then the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 20:1 – 2:1) on silica gel to give the corresponding product **65b** in the 92% yield (1.5266 g).

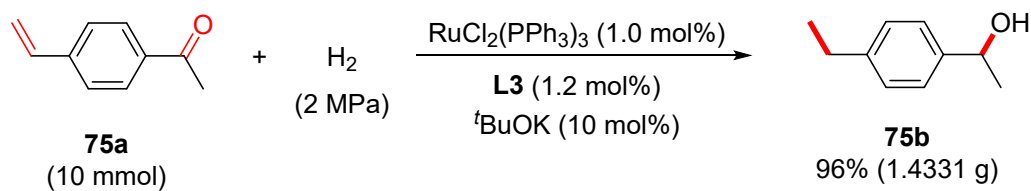
## 6.6



**10 mmol Scale:** In a nitrogen-filled glove box, add a magnetic stirring bar, **68a** (10.0 mmol), <sup>t</sup>BuOK (10 mol%), RuCl<sub>2</sub>(PPh<sub>3</sub>)<sub>3</sub> (3 mol%), **L3** (3.6 mol%), and 1,4-dioxane (20.0 mL) to a dried high-pressure reactor. After sealing, remove the high-pressure reactor from the glove box. Then the reactor was purged and charged with H<sub>2</sub> (2.0 MPa) and immersed into a pre-heated metal bath (110°C). After 24 hours the reaction was cooled, release H<sub>2</sub> from the reactor. Then quenched with saturated brine and DCM, and dried with Na<sub>2</sub>SO<sub>4</sub>. A small aliquot of the organic phase was analyzed by GC or GC-MS to monitor product formation. Then the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 20:1 – 2:1) on silica gel to give the corresponding product **68b** in the 91% yield (2.0384 g).

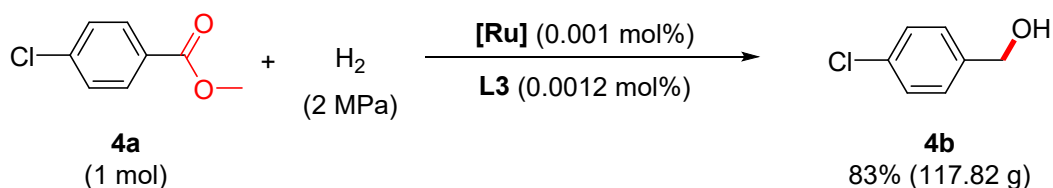


## 6.7



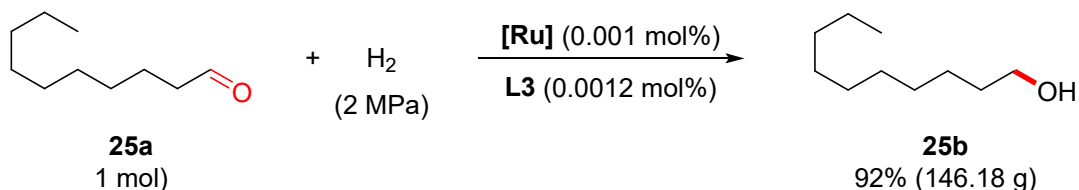
**10 mmol Scale:** In a nitrogen-filled glove box, add a magnetic stirring bar, **75a** (10.0 mmol), <sup>t</sup>BuOK (10 mol%), RuCl<sub>2</sub>(PPh<sub>3</sub>)<sub>3</sub> (1 mol%), **L3** (1.2 mol%), and 1,4-dioxane (20.0 mL) to a dried high-pressure reactor. After sealing, remove the high-pressure reactor from the glove box. Then the reactor was purged and charged with H<sub>2</sub> (2.0 MPa) and immersed into a pre-heated metal bath (110°C). After 24 hours the reaction was cooled, release H<sub>2</sub> from the reactor. Then quenched with saturated brine and DCM, and dried with Na<sub>2</sub>SO<sub>4</sub>. A small aliquot of the organic phase was analyzed by GC or GC-MS to monitor product formation. Then the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 20:1 – 2:1) on silica gel to give the corresponding product **75b** in the 96% yield (1.4331 g).

## 6.8



**1 mol Scale:** In a nitrogen-filled glove box, add a magnetic stirring bar, **4a** (1 mol), <sup>t</sup>BuOK (20 mol%), RuCl<sub>2</sub>(PPh<sub>3</sub>)<sub>3</sub> (0.001 mol%), **L3** (0.0012 mol%), and 1,4-dioxane (0.5 L) to a dried high-pressure reactor. After sealing, remove the high-pressure reactor from the glove box. Then the reactor was purged and charged with H<sub>2</sub> (2.0 MPa) and immersed into a pre-heated metal bath (110°C). After 48 hours the reaction was cooled, release H<sub>2</sub> from the reactor. Then quenched with saturated brine and DCM, and dried with Na<sub>2</sub>SO<sub>4</sub>. A small aliquot of the organic phase was analyzed by GC or GC-MS to monitor product formation. Then the residue was purified by recrystallization to obtain **4b** in the 83% yield (117.82 g).

## 6.9



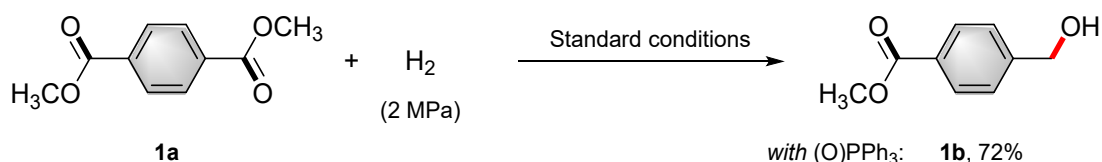
**1 mol Scale:** In a nitrogen-filled glove box, add a magnetic stirring bar, **25a** (1 mol), <sup>t</sup>BuOK (5 mol%), RuCl<sub>2</sub>(PPh<sub>3</sub>)<sub>3</sub> (0.001 mol%), **L3** (0.0012 mol%), and 1,4-dioxane (0.5 L) to a dried high-pressure reactor. After sealing, remove the high-pressure reactor from the glove box. Then the reactor was purged and charged with H<sub>2</sub> (2.0 MPa) and immersed into a pre-heated metal bath (110°C). After 48 hours the reaction was cooled, release H<sub>2</sub> from the reactor. Then quenched with saturated brine and DCM, and dried with Na<sub>2</sub>SO<sub>4</sub>. A small aliquot of the organic phase was analyzed by GC or GC-MS to monitor product formation. Then the residue was purified by distillation to obtain **25b** in the 92% yield (146.18 g).

## 7. Mechanism investigations

### 7.1



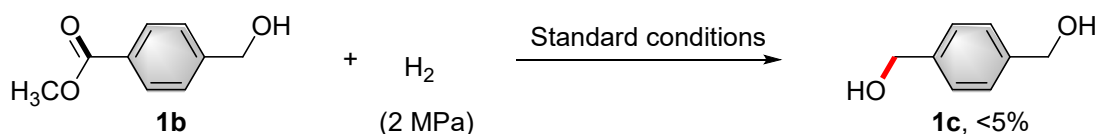
In a nitrogen-filled glove box, add a magnetic stirring bar, dimethyl terephthalate **1a** (0.5 mmol), *t*BuOK (0.05 mmol, 10 mol%), RuCl<sub>2</sub>(PPh<sub>3</sub>)<sub>3</sub> (1 mol%), **L3** (1.2 mol%), 1,4-dioxane (2.0 mL) and a drop of mercury to a dried high-pressure reactor. After sealing, remove the high-pressure reactor from the glove box. Then the reactor was purged and charged with H<sub>2</sub> (2.0 MPa) and immersed into a pre-heated metal bath (110°C) and stirred at 600 rpm. After design time the reaction was cooled, release H<sub>2</sub> from the reactor. Then quenched with saturated brine (100 μL) and DCM, and dried with Na<sub>2</sub>SO<sub>4</sub>. A small aliquot of the organic phase was analyzed by GC or GC-MS to monitor product formation. Then the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 100:1 – 2:1) on silica gel to give the corresponding product **1b** in the 78% yield.



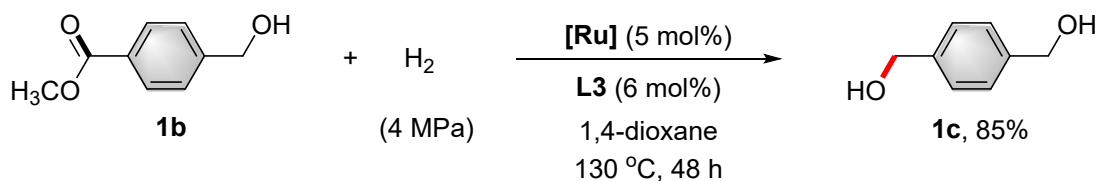
In a nitrogen-filled glove box, add a magnetic stirring bar, dimethyl terephthalate **1a** (0.5 mmol), *t*BuOK (0.05 mmol, 10 mol%), RuCl<sub>2</sub>(PPh<sub>3</sub>)<sub>3</sub> (1 mol%), **L3** (1.2 mol%), 1,4-dioxane (2.0 mL) and (O)PPh<sub>3</sub> (1.0 mmol) to a dried high-pressure reactor. After sealing, remove the high-pressure reactor from the glove box. Then the reactor was purged and charged with H<sub>2</sub> (2.0 MPa) and immersed into a pre-heated metal bath (110°C) and stirred at 600 rpm. After design time the reaction was cooled, release H<sub>2</sub> from the reactor. Then quenched with saturated brine (100 μL) and DCM, and dried with Na<sub>2</sub>SO<sub>4</sub>. A small aliquot of the organic phase was analyzed by GC or GC-MS to monitor

product formation. Then the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 100:1 – 2:1) on silica gel to give the corresponding product **1b** in the 72% yield.

## 7.2

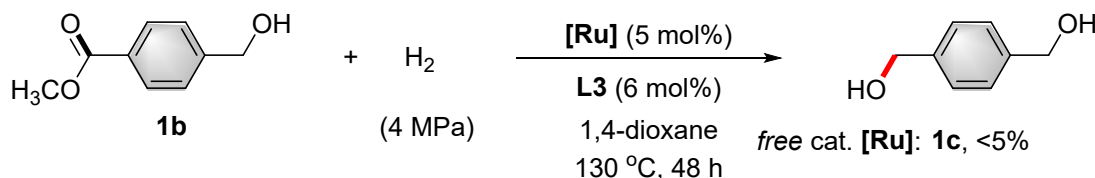


In a nitrogen-filled glove box, add a magnetic stirring bar, methyl 4-(hydroxymethyl)benzoate **1b** (0.5 mmol),  $t\text{BuOK}$  (0.05 mmol, 10 mol%),  $\text{RuCl}_2(\text{PPh}_3)_3$  (1 mol%), **L3** (1.2 mol%) and 1,4-dioxane (2.0 mL) to a dried high-pressure reactor. After sealing, remove the high-pressure reactor from the glove box. Then the reactor was purged and charged with  $\text{H}_2$  (2.0 MPa) and immersed into a pre-heated metal bath (110°C) and stirred at 600 rpm. After design time the reaction was cooled, release  $\text{H}_2$  from the reactor. Then quenched with saturated brine (100  $\mu\text{L}$ ) and DCM, and dried with  $\text{Na}_2\text{SO}_4$ . A small aliquot of the organic phase was analyzed by GC or GC-MS to monitor product formation. Then the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 100:1 – 2:1) on silica gel to reclaim the **1b** in the 97% yield.

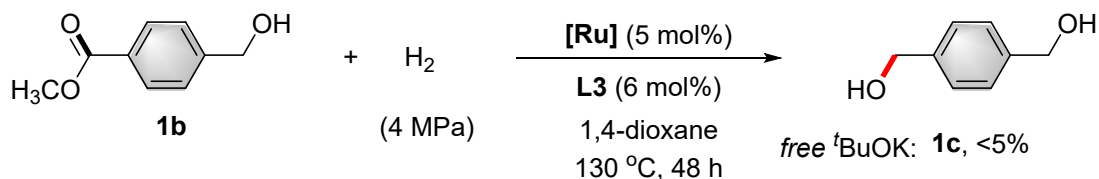


In a nitrogen-filled glove box, add a magnetic stirring bar, **1b** (0.5 mmol),  $t\text{BuOK}$  (20 mol%),  $\text{RuCl}_2(\text{PPh}_3)_3$  (5.0 mol%), **L3** (6.0 mol%), and 1,4-dioxane (2.0 mL) to a dried high-pressure reactor. After sealing, remove the high-pressure reactor from the glove box. Then the reactor was purged and charged with  $\text{H}_2$  (4.0 MPa) and immersed into a pre-heated metal bath (130°C). After 48 hours the reaction was cooled, release  $\text{H}_2$  from the reactor. Then quenched with saturated brine and DCM, and dried with  $\text{Na}_2\text{SO}_4$ . A small aliquot of the organic phase was analyzed by GC or GC-MS to monitor product

formation. Then the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 20:1 – 2:1) on silica gel to give the corresponding product **1c** in the 85% yield.



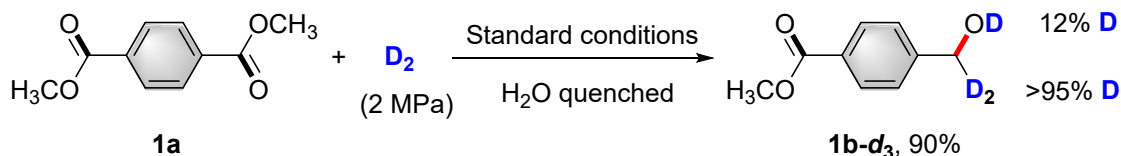
In a nitrogen-filled glove box, add a magnetic stirring bar, **1b** (0.5 mmol),  $t\text{BuOK}$  (20 mol%), **L3** (6.0 mol%), and 1,4-dioxane (2.0 mL) to a dried high-pressure reactor. After sealing, remove the high-pressure reactor from the glove box. Then the reactor was purged and charged with  $\text{H}_2$  (4.0 MPa) and immersed into a pre-heated metal bath (130°C). After 48 hours the reaction was cooled, release  $\text{H}_2$  from the reactor. Then quenched with saturated brine and DCM, and dried with  $\text{Na}_2\text{SO}_4$ . A small aliquot of the organic phase was analyzed by GC or GC-MS to monitor product formation. Then the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 100:1 – 2:1) on silica gel to reclaim the **1b** in the 96% yield.



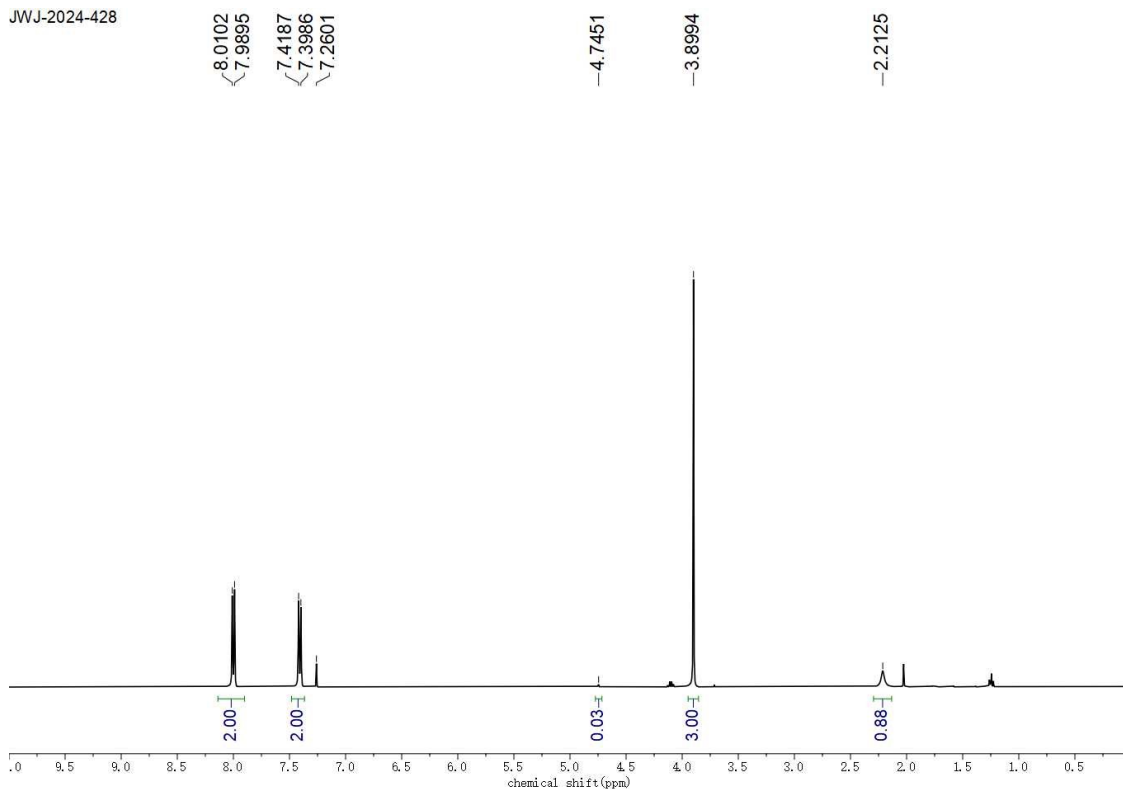
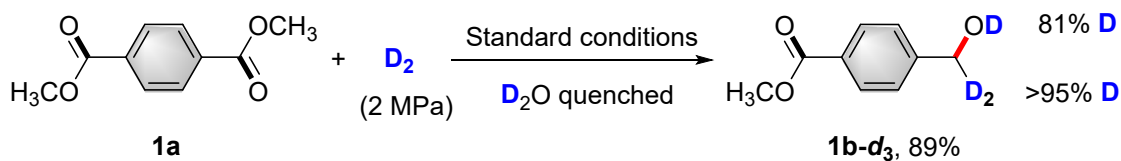
In a nitrogen-filled glove box, add a magnetic stirring bar, **1b** (0.5 mmol),  $\text{RuCl}_2(\text{PPh}_3)_3$  (5.0 mol%), **L3** (6.0 mol%), and 1,4-dioxane (2.0 mL) to a dried high-pressure reactor. After sealing, remove the high-pressure reactor from the glove box. Then the reactor was purged and charged with  $\text{H}_2$  (4.0 MPa) and immersed into a pre-heated metal bath (130°C). After 48 hours the reaction was cooled, release  $\text{H}_2$  from the reactor. Then quenched with saturated brine and DCM, and dried with  $\text{Na}_2\text{SO}_4$ . A small aliquot of the organic phase was analyzed by GC or GC-MS to monitor product

formation. Then the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 100:1 – 2:1) on silica gel to reclaim the **1b** in the 96% yield.

### 7.3 Deuterium-labeled investigations with D<sub>2</sub>



In a nitrogen-filled glove box, add a magnetic stirring bar, dimethyl terephthalate **1a** (0.5 mmol), <sup>t</sup>BuOK (0.05 mmol, 10 mol%), RuCl<sub>2</sub>(PPh<sub>3</sub>)<sub>3</sub> (1 mol%), **L3** (1.2 mol%) and 1,4-dioxane (2.0 mL) to a dried high-pressure reactor. After sealing, remove the high-pressure reactor from the glove box. Then the reactor was purged and charged with D<sub>2</sub> (2.0 MPa) and immersed into a pre-heated metal bath (110°C) and stirred at 600 rpm. After design time the reaction was cooled, release H<sub>2</sub> from the reactor. Then quenched with saturated brine (100 μL) and DCM, and dried with Na<sub>2</sub>SO<sub>4</sub>. A small aliquot of the organic phase was analyzed by GC or GC-MS to monitor product formation. Then the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 100:1 – 2:1) on silica gel to give the corresponding product **1b-d<sub>3</sub>** in the 90% yield.

Figure S1  $^1\text{H-NMR}$  of **1b-d<sub>3</sub>**

In a nitrogen-filled glove box, add a magnetic stirring bar, dimethyl terephthalate **1a** (0.5 mmol),  $t\text{BuOK}$  (0.05 mmol, 10 mol%),  $\text{RuCl}_2(\text{PPh}_3)_3$  (1 mol%), **L3** (1.2 mol%) and 1,4-dioxane (2.0 mL) to a dried high-pressure reactor. After sealing, remove the high-pressure reactor from the glove box. Then the reactor was purged and charged with  $\text{D}_2$  (2.0 MPa) and immersed into a pre-heated metal bath (110°C) and stirred at 600 rpm. After design time the reaction was cooled, release  $\text{H}_2$  from the reactor. Then quenched with saturated brine (100  $\mu\text{L}$ ) and DCM, and dried with  $\text{Na}_2\text{SO}_4$ . A small aliquot of the organic phase was analyzed by GC or GC-MS to monitor product formation. Then the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography (petroleum ether/ethyl acetate = 100:1 – 2:1) on silica gel to give the corresponding product **1b-d<sub>3</sub>** in the 89% yield.

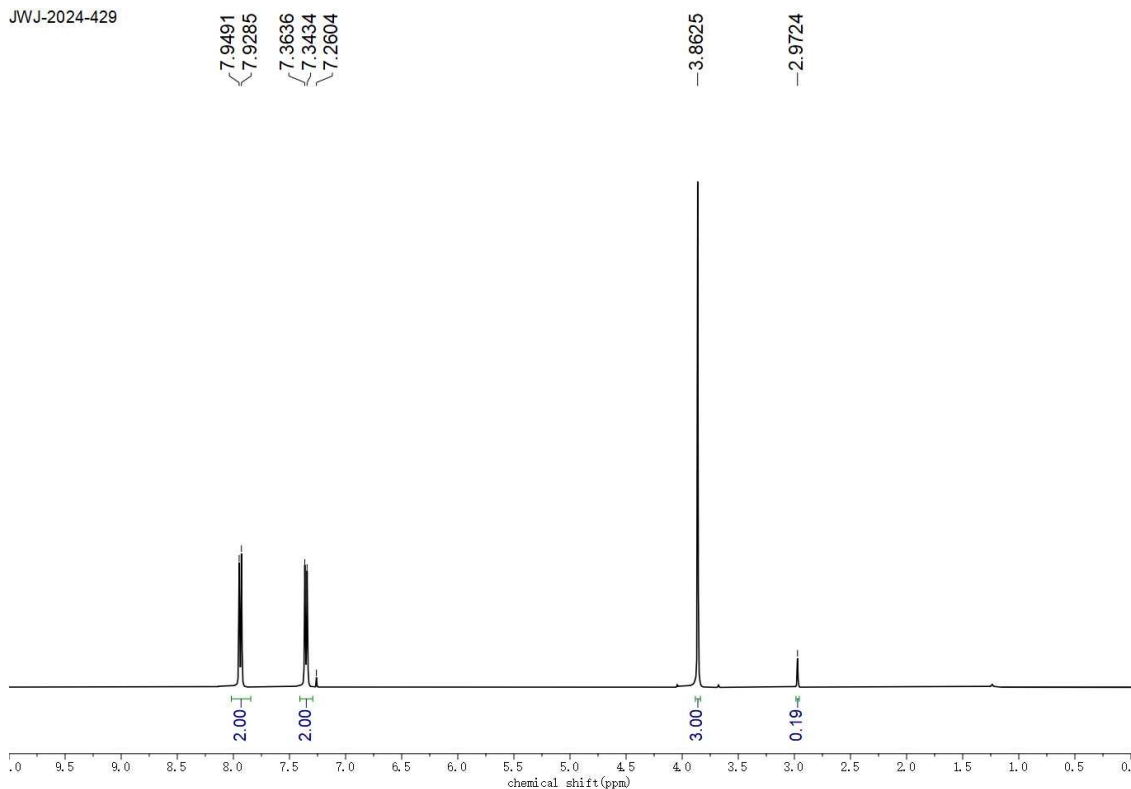


Figure S2  $^1\text{H-NMR}$  of  $1b-d_3$

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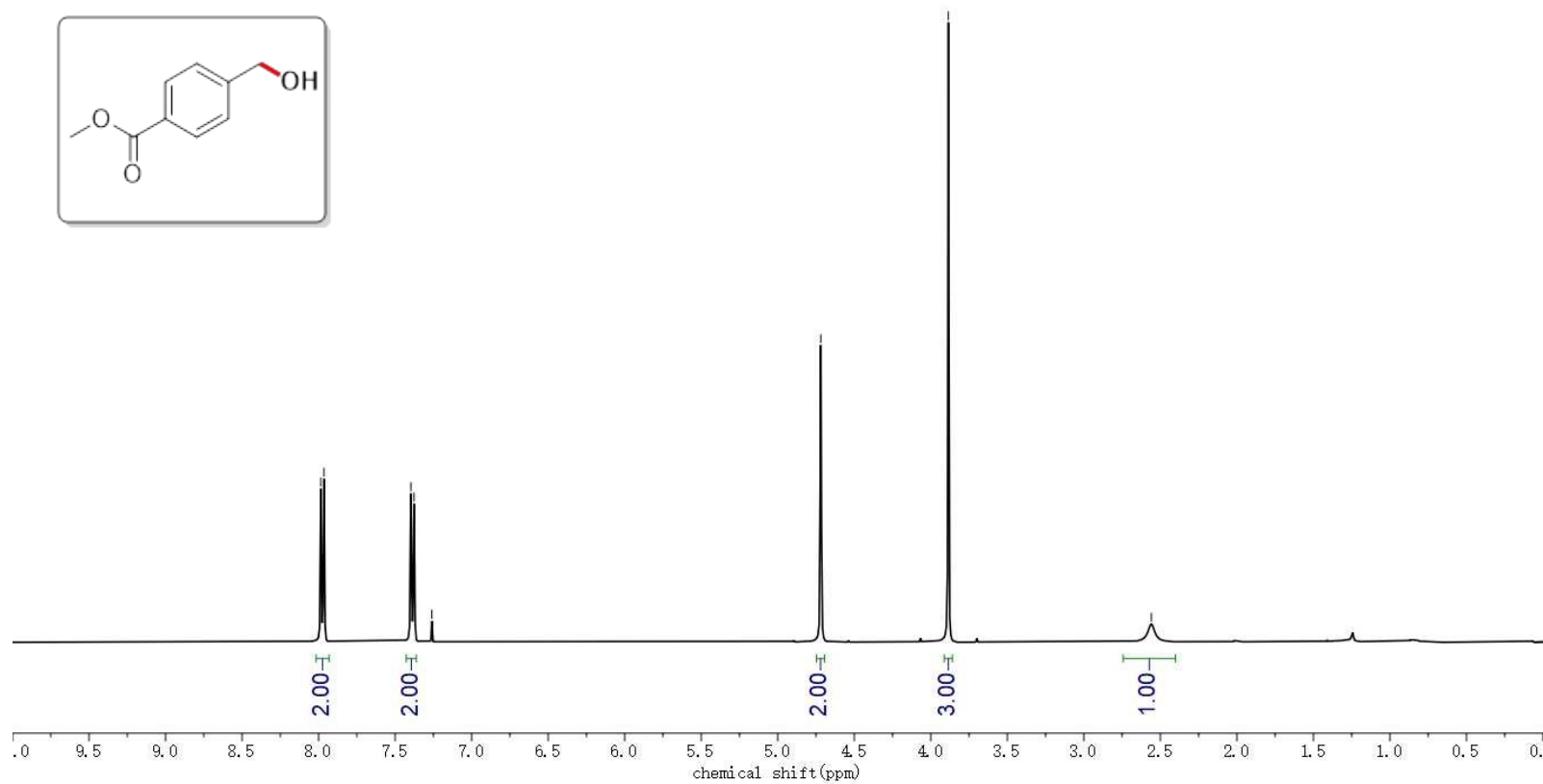
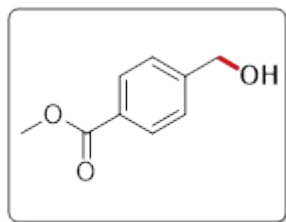
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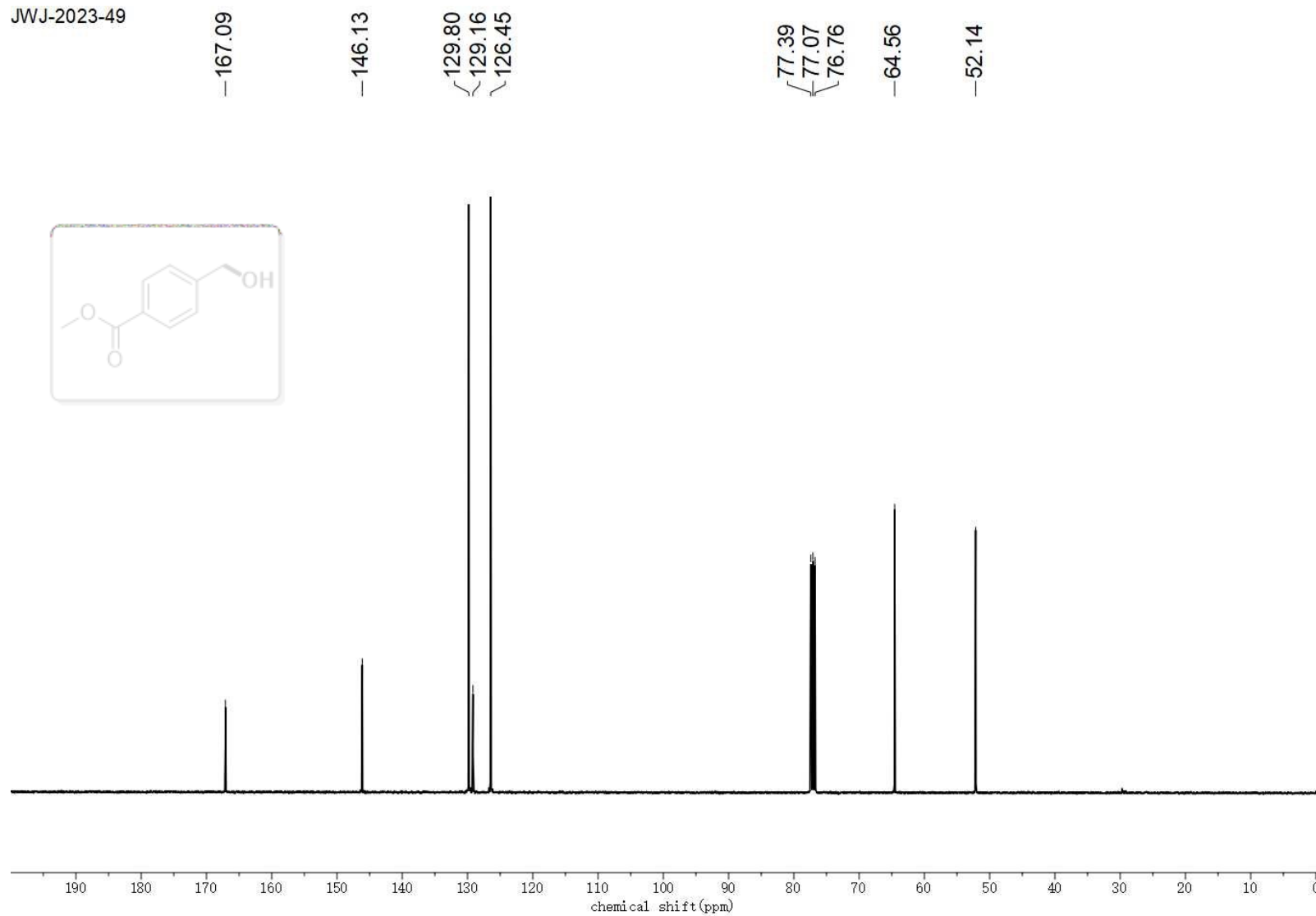
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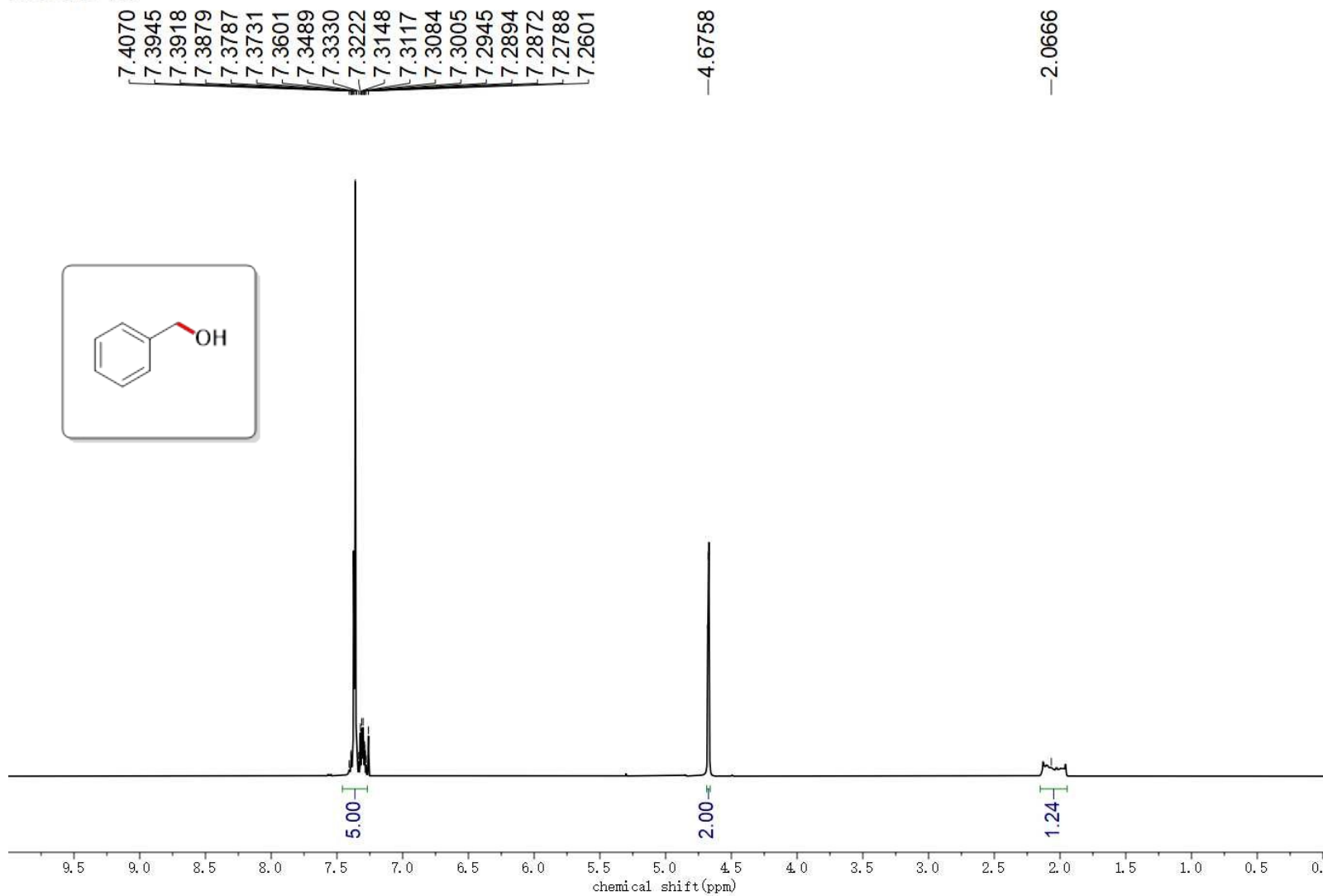
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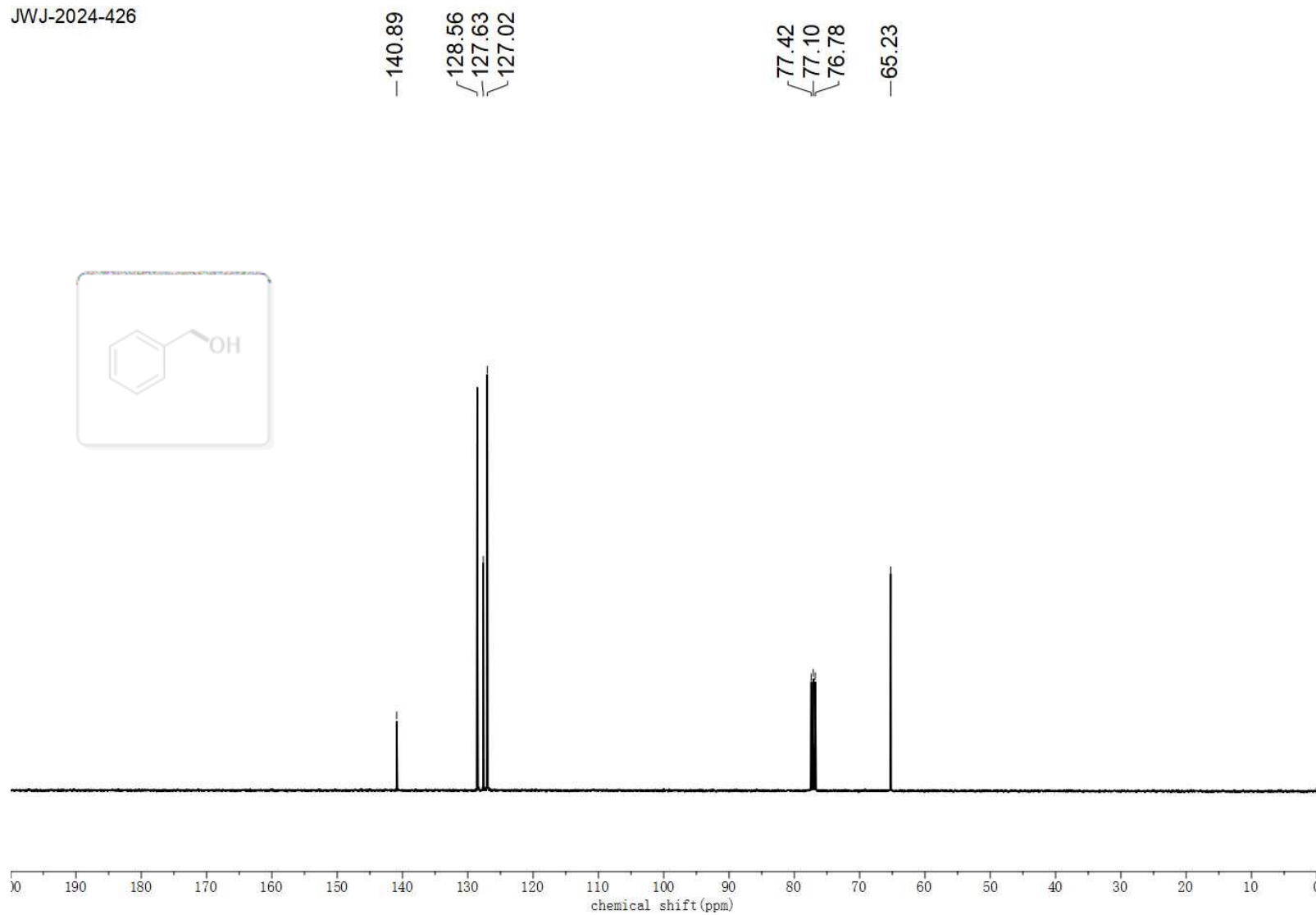
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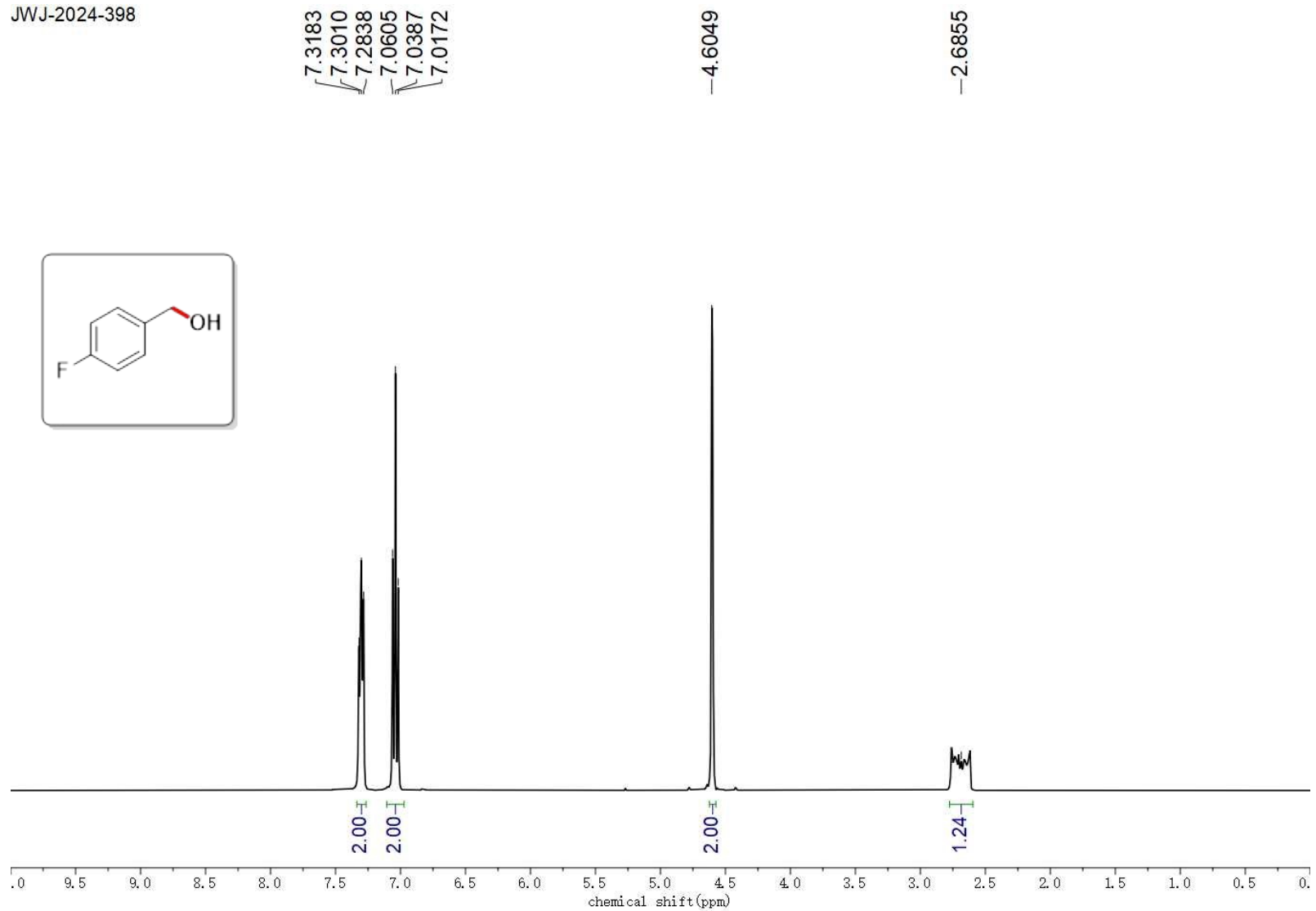
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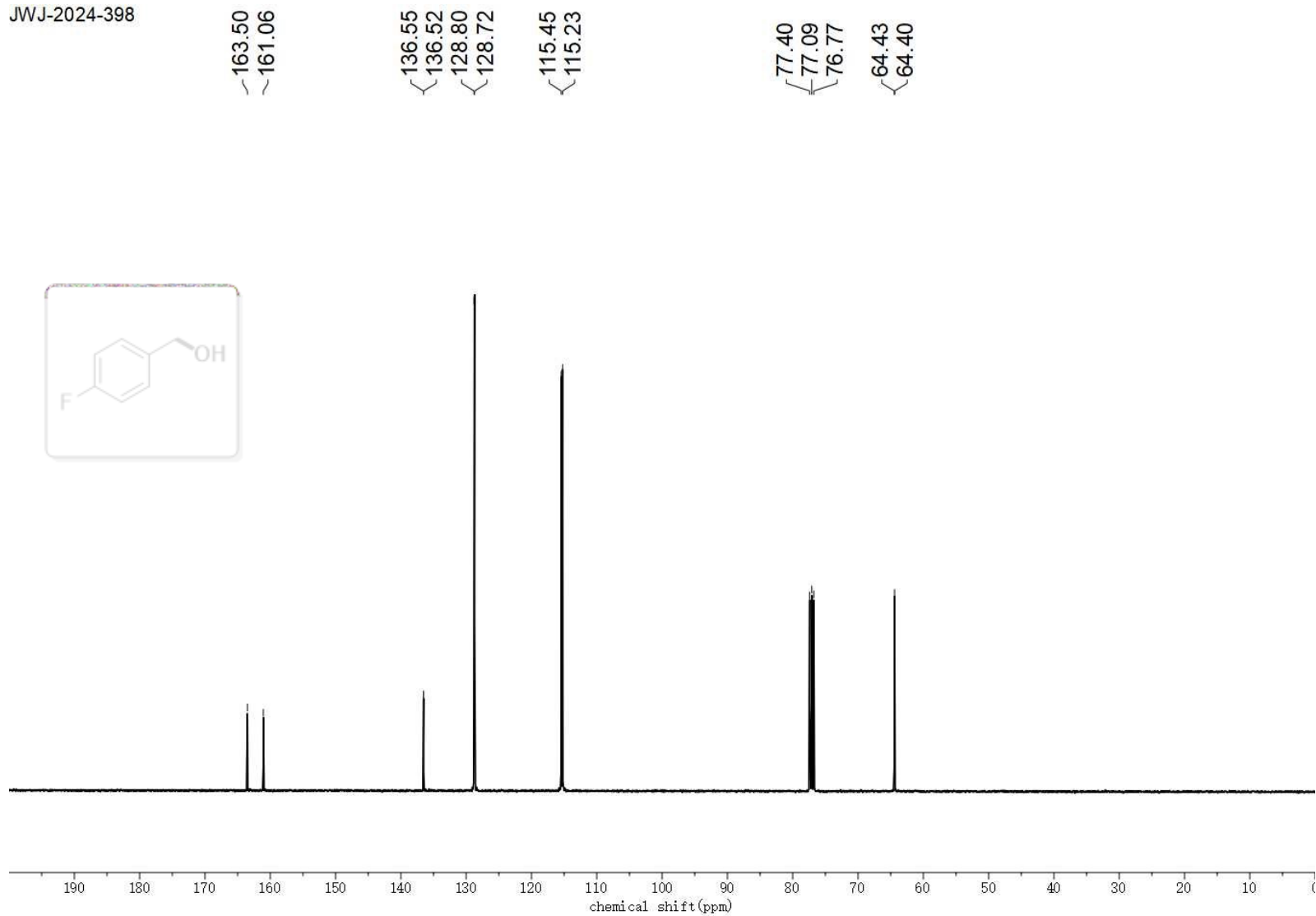


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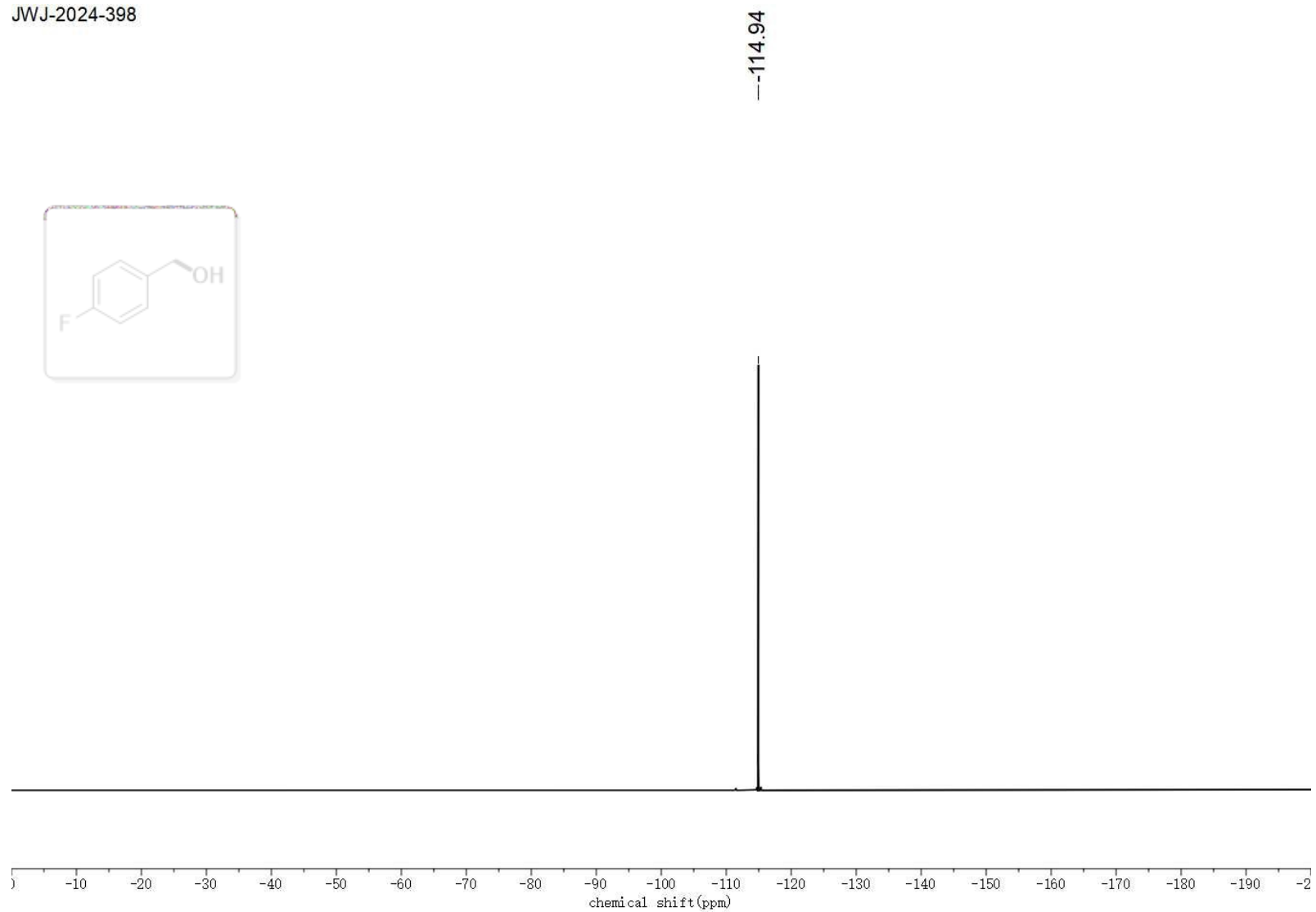
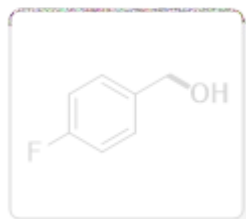




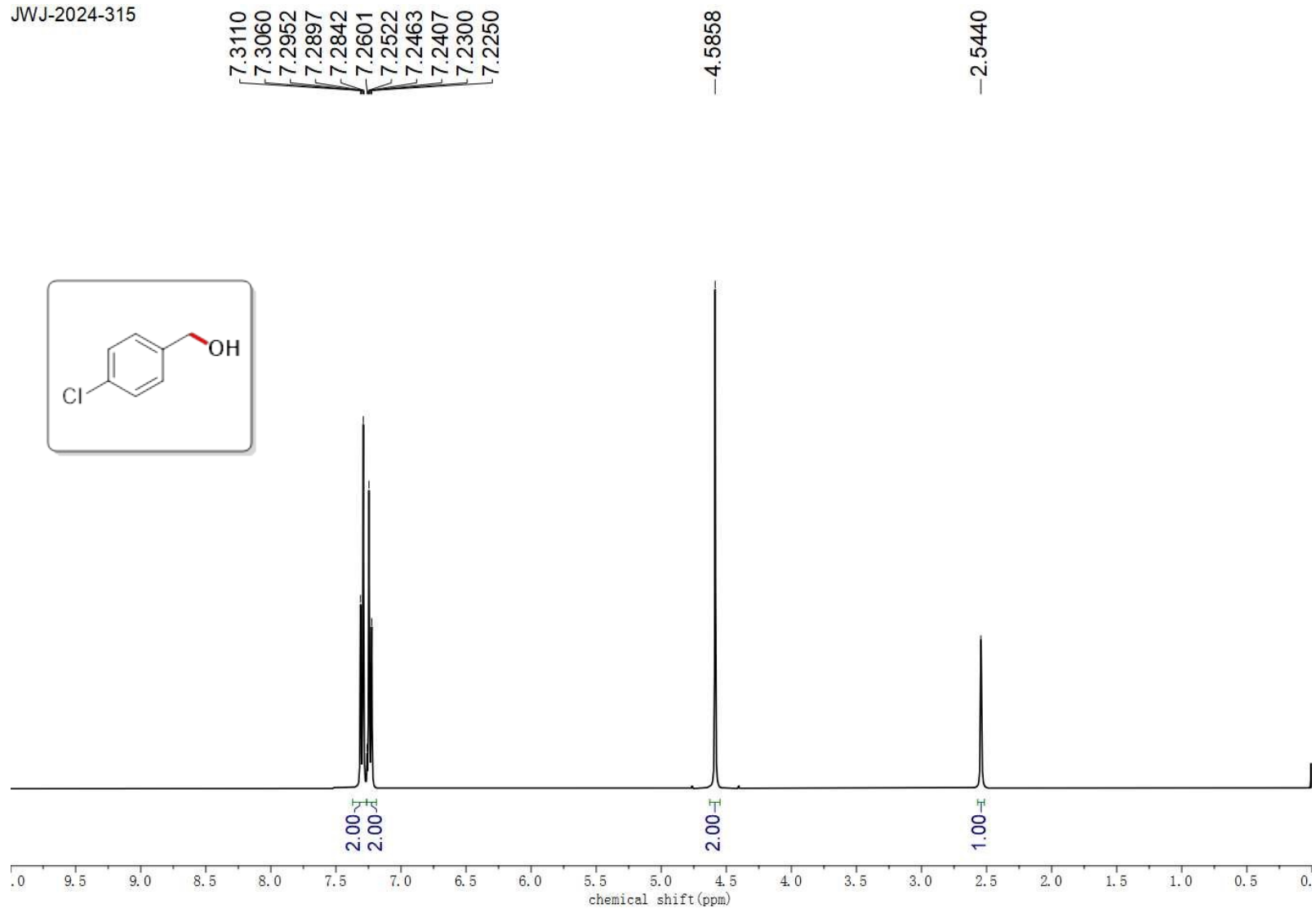
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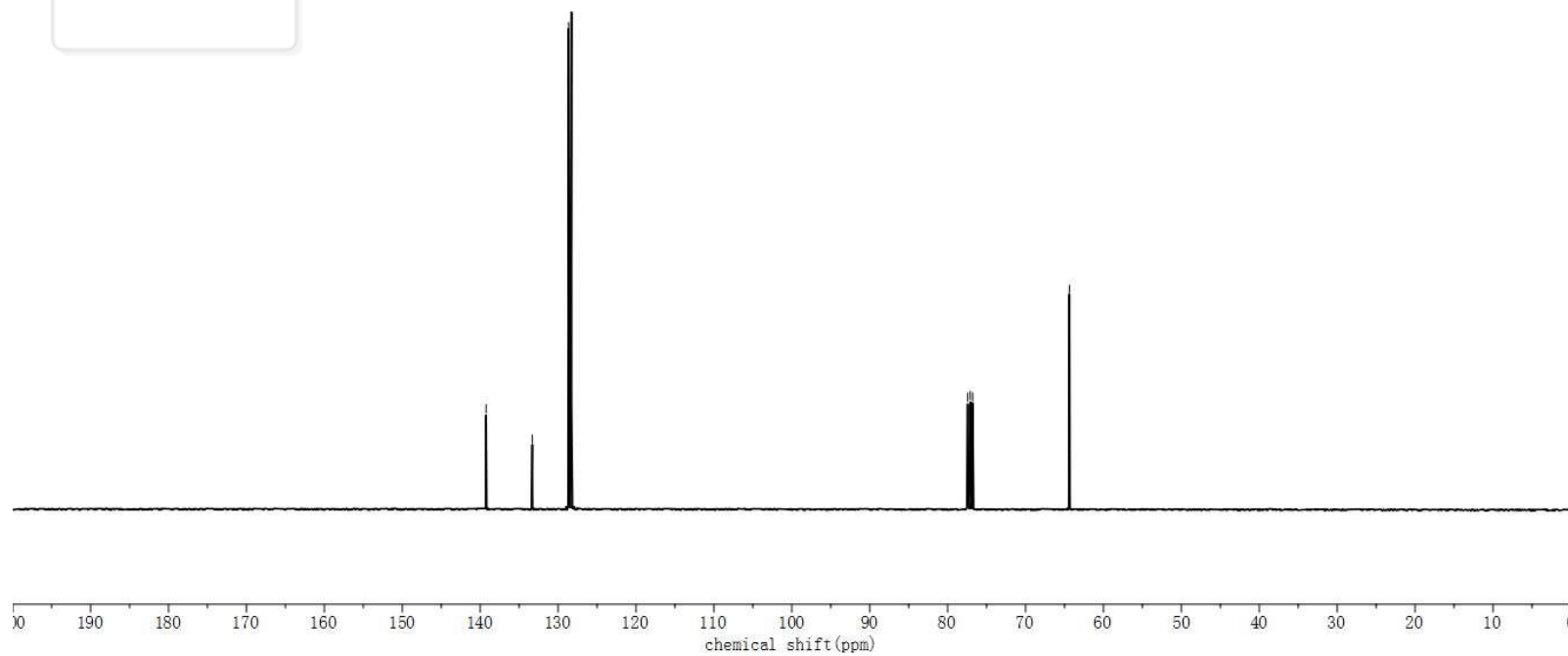
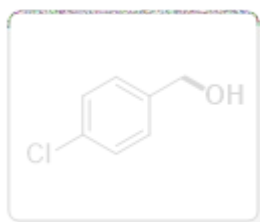
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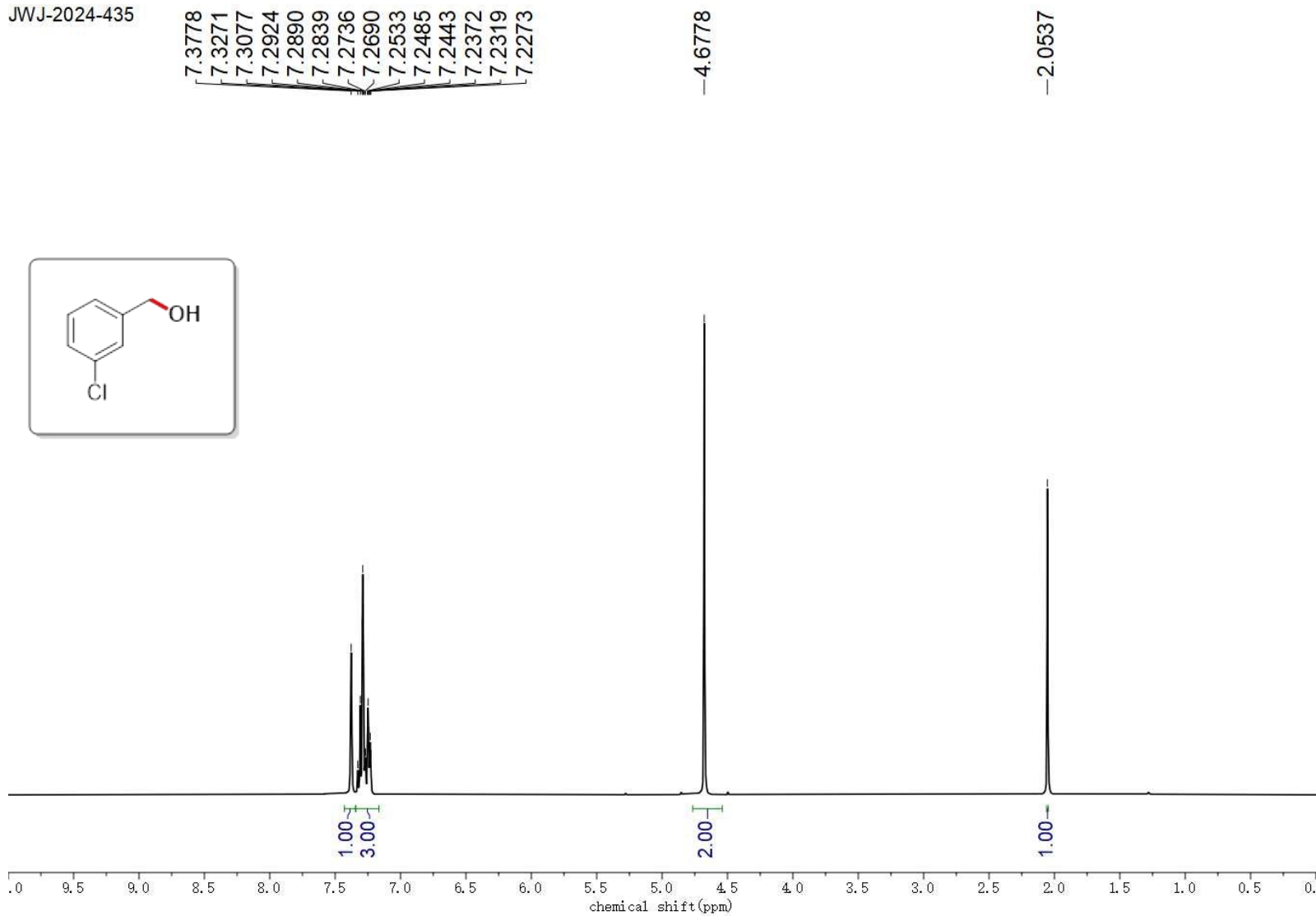
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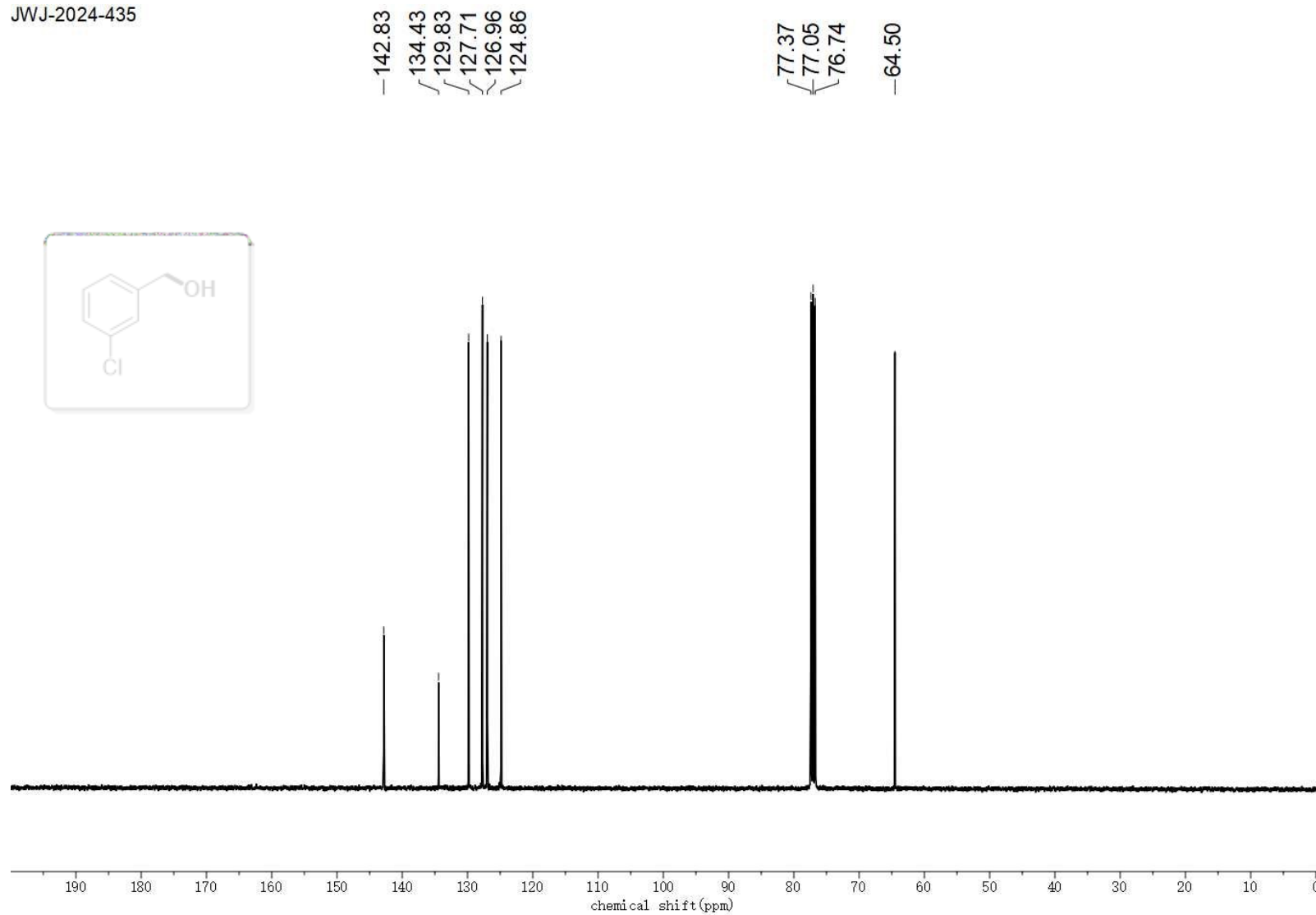
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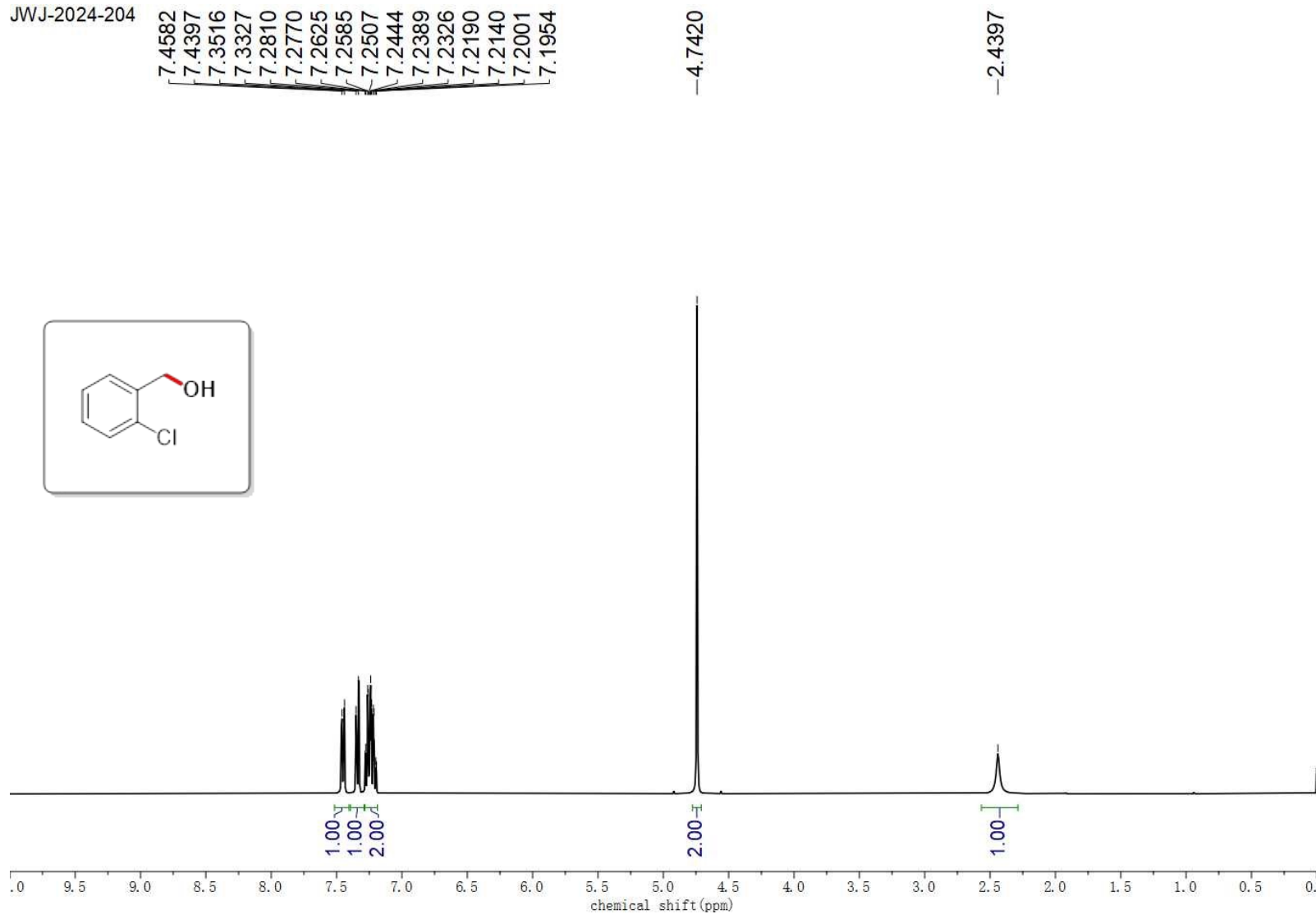
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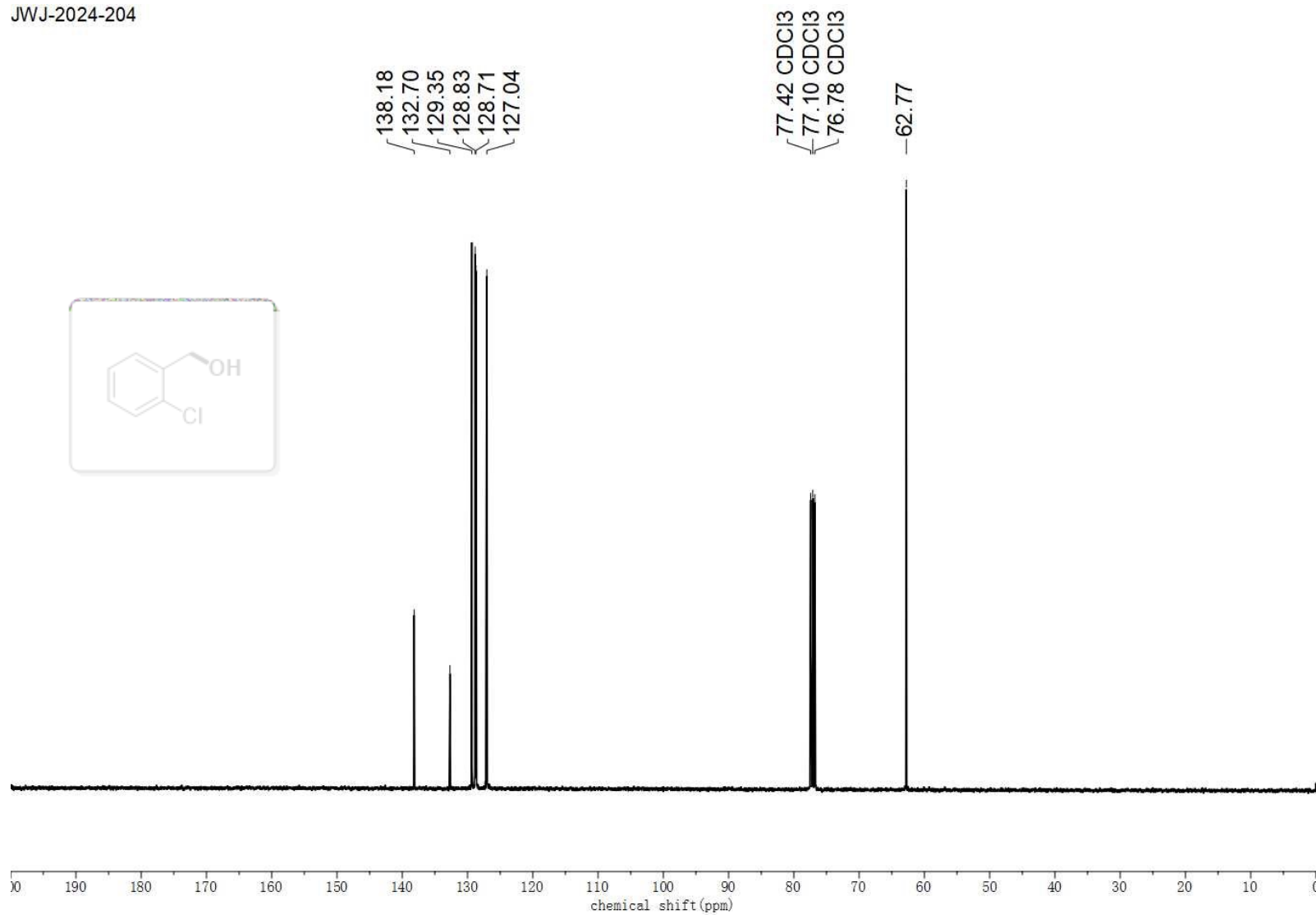
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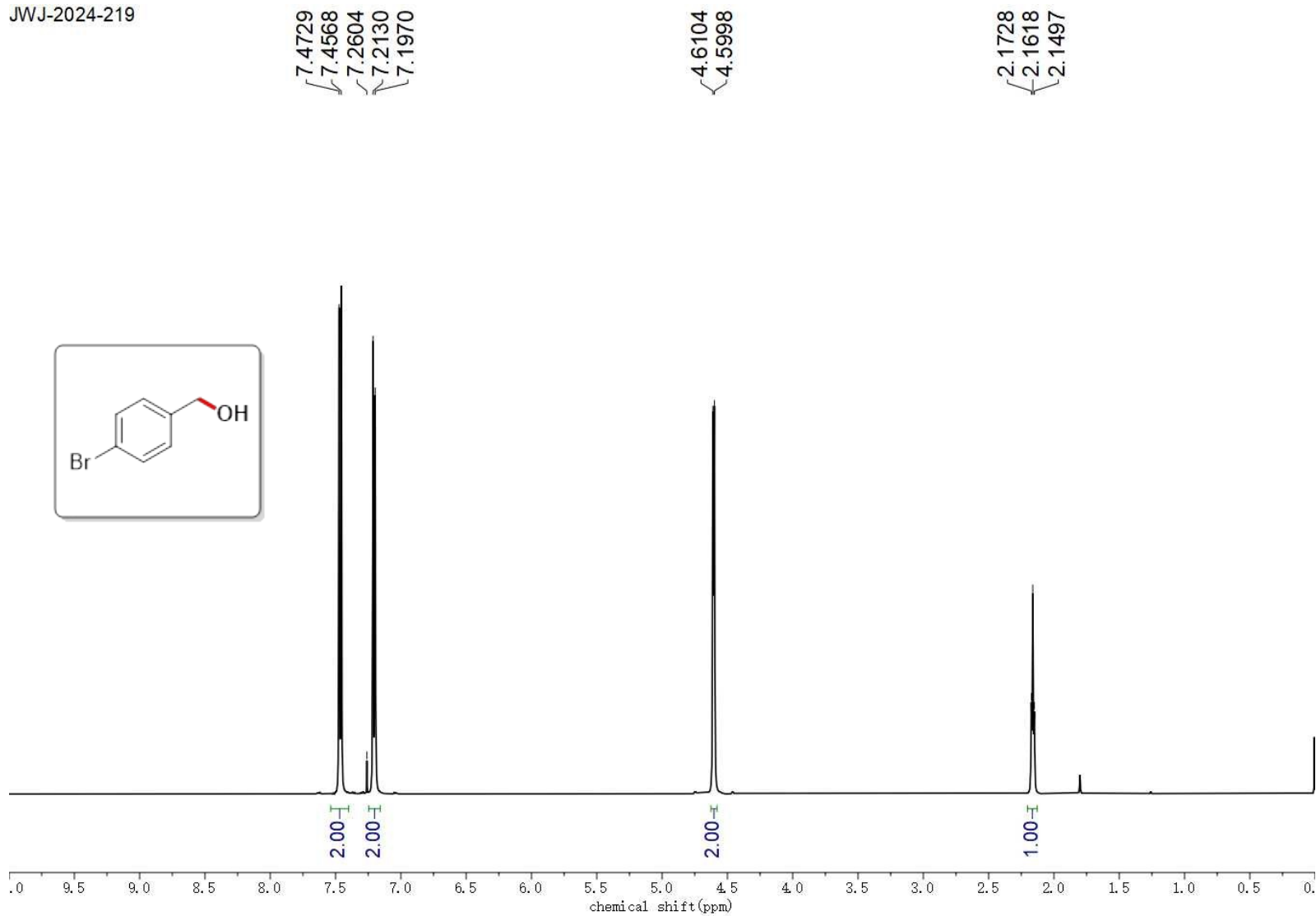


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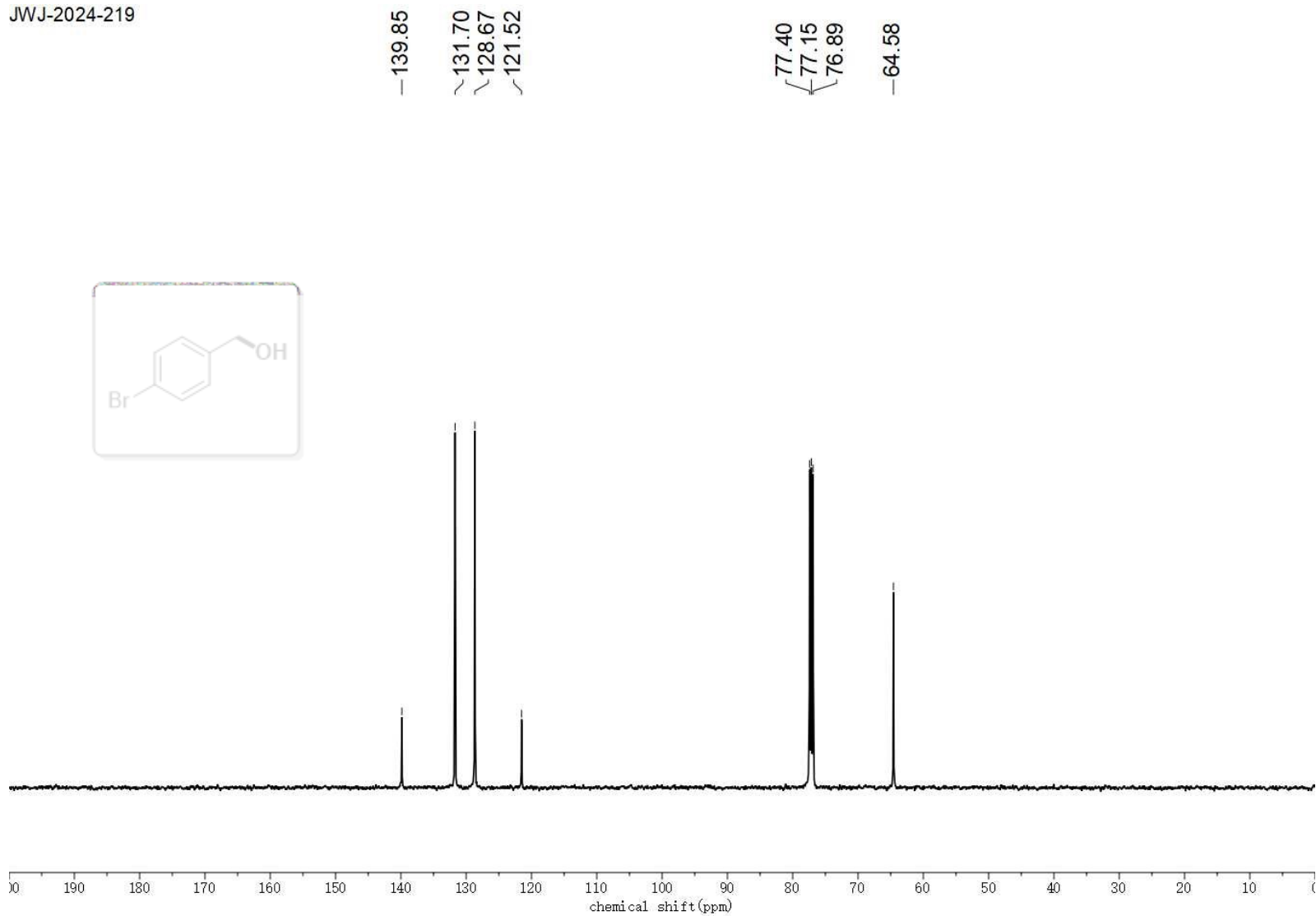




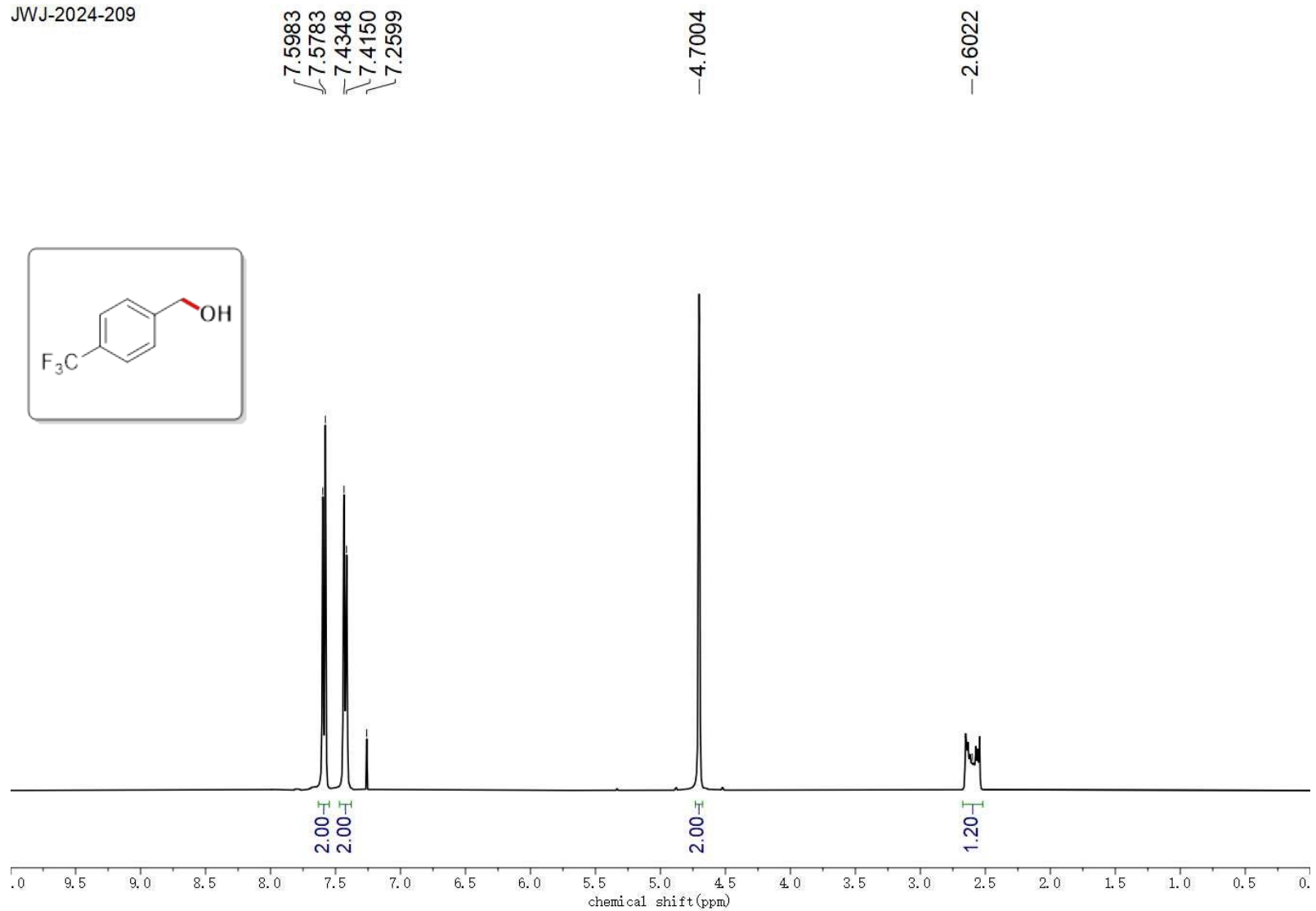
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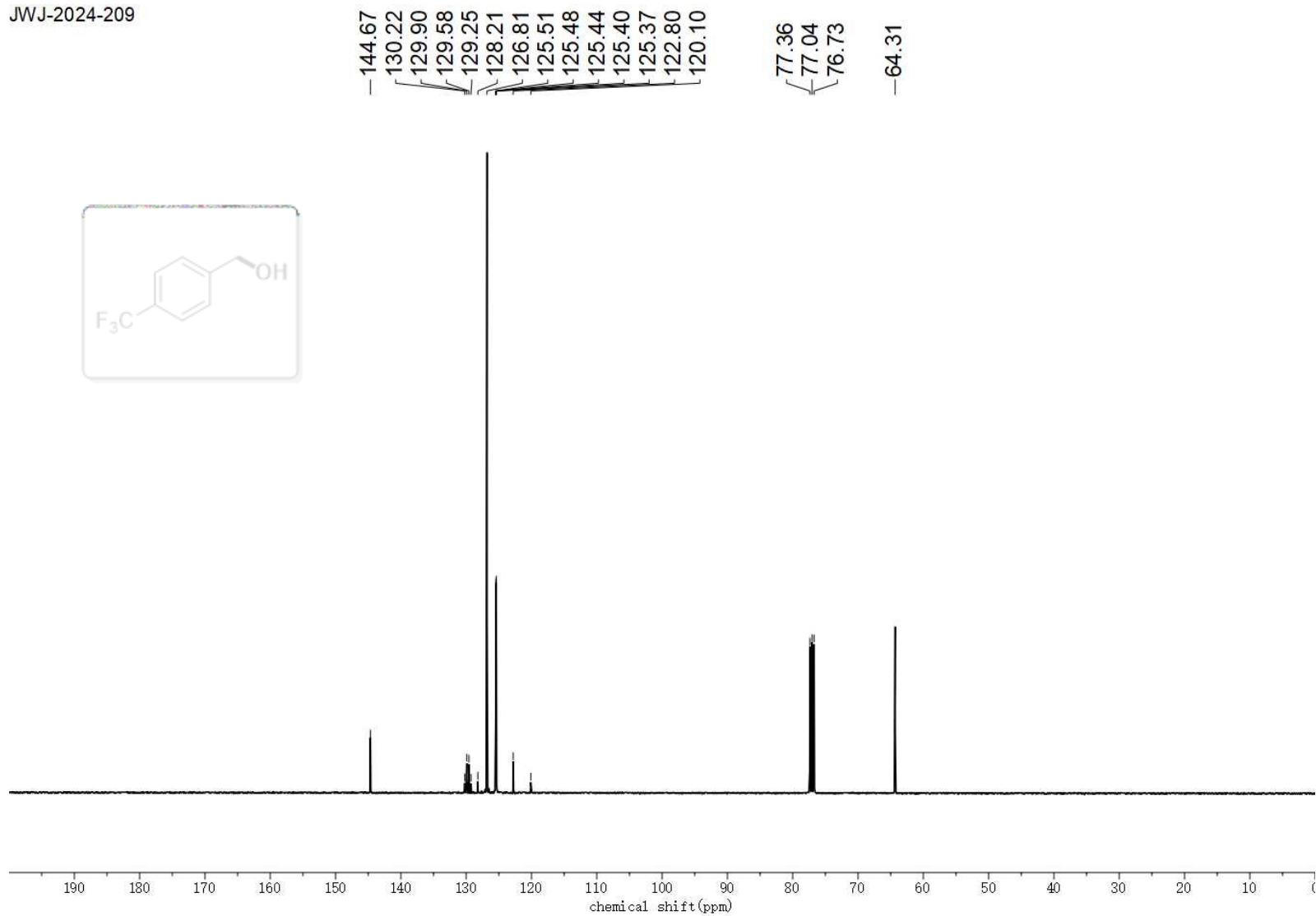
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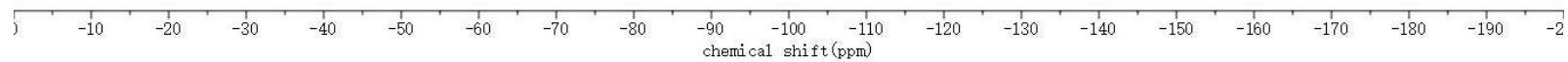
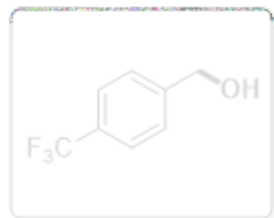


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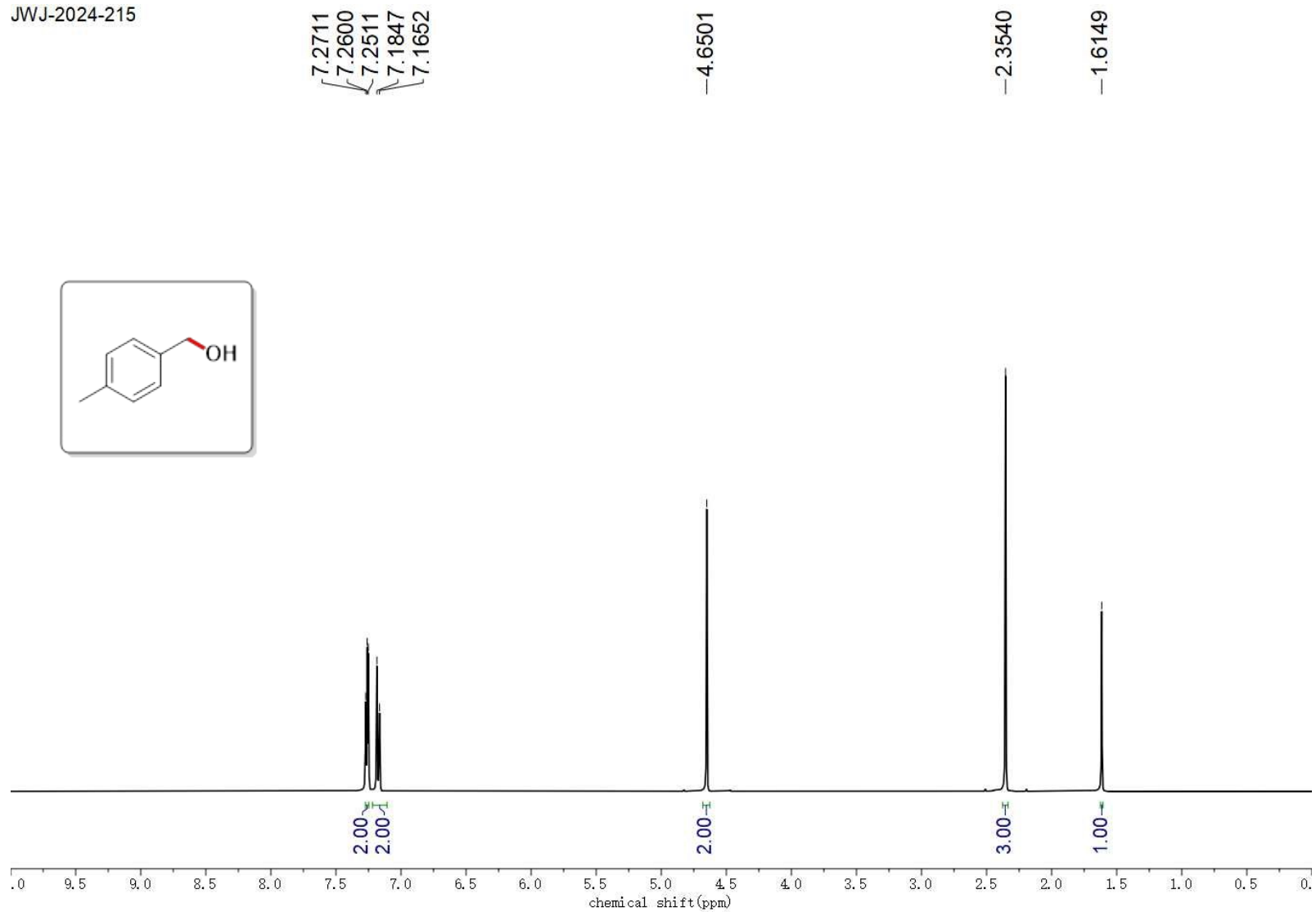


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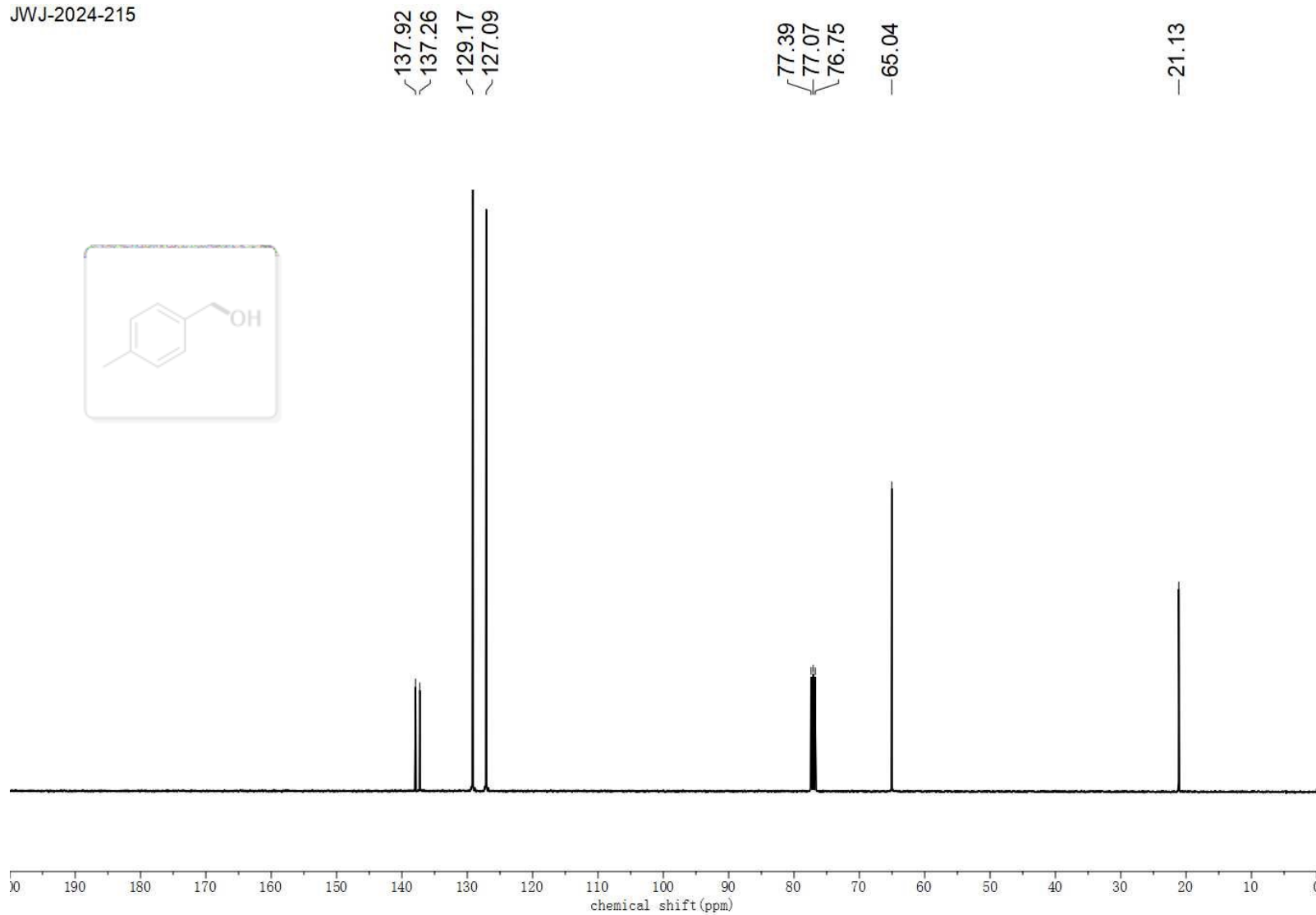
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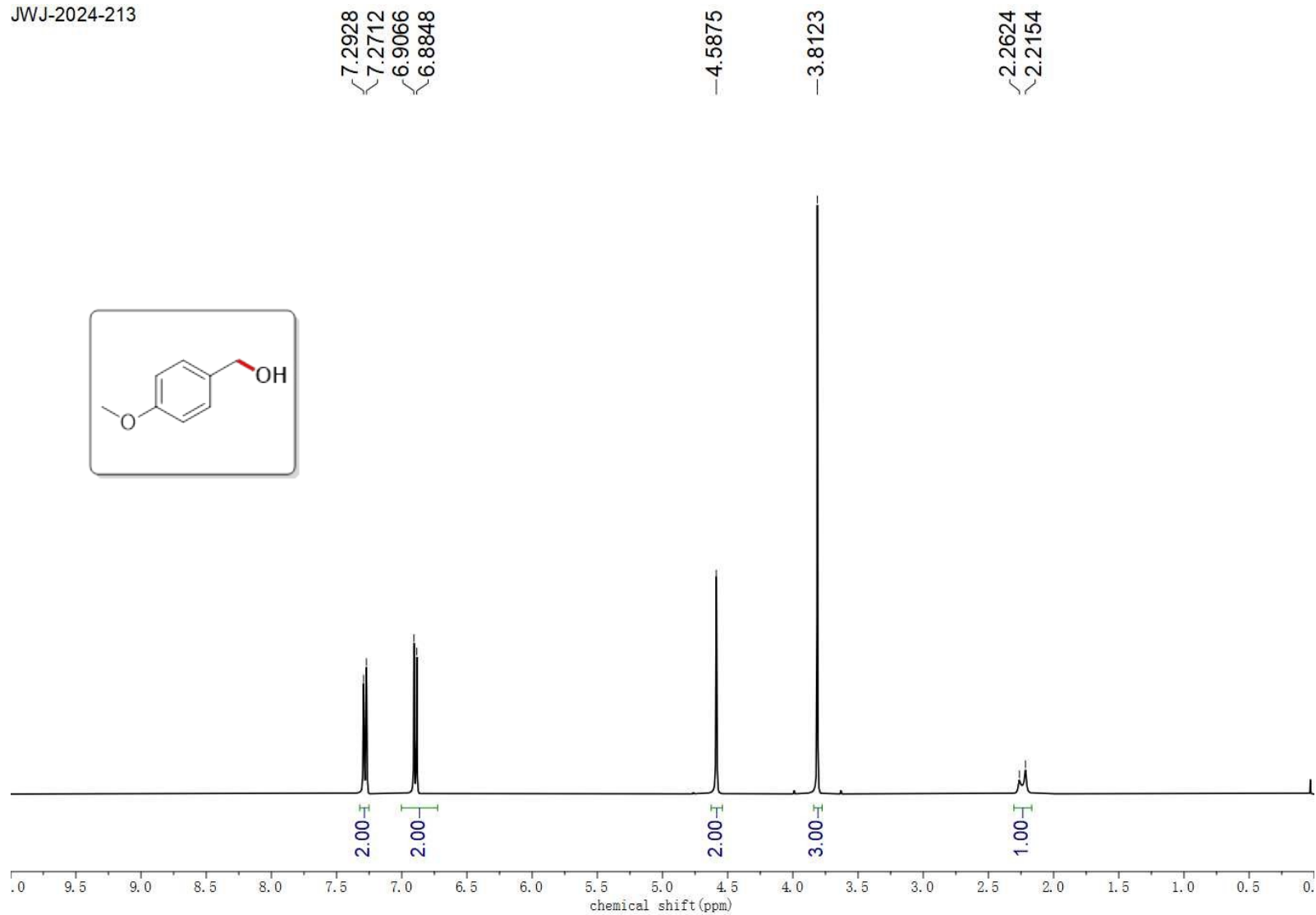
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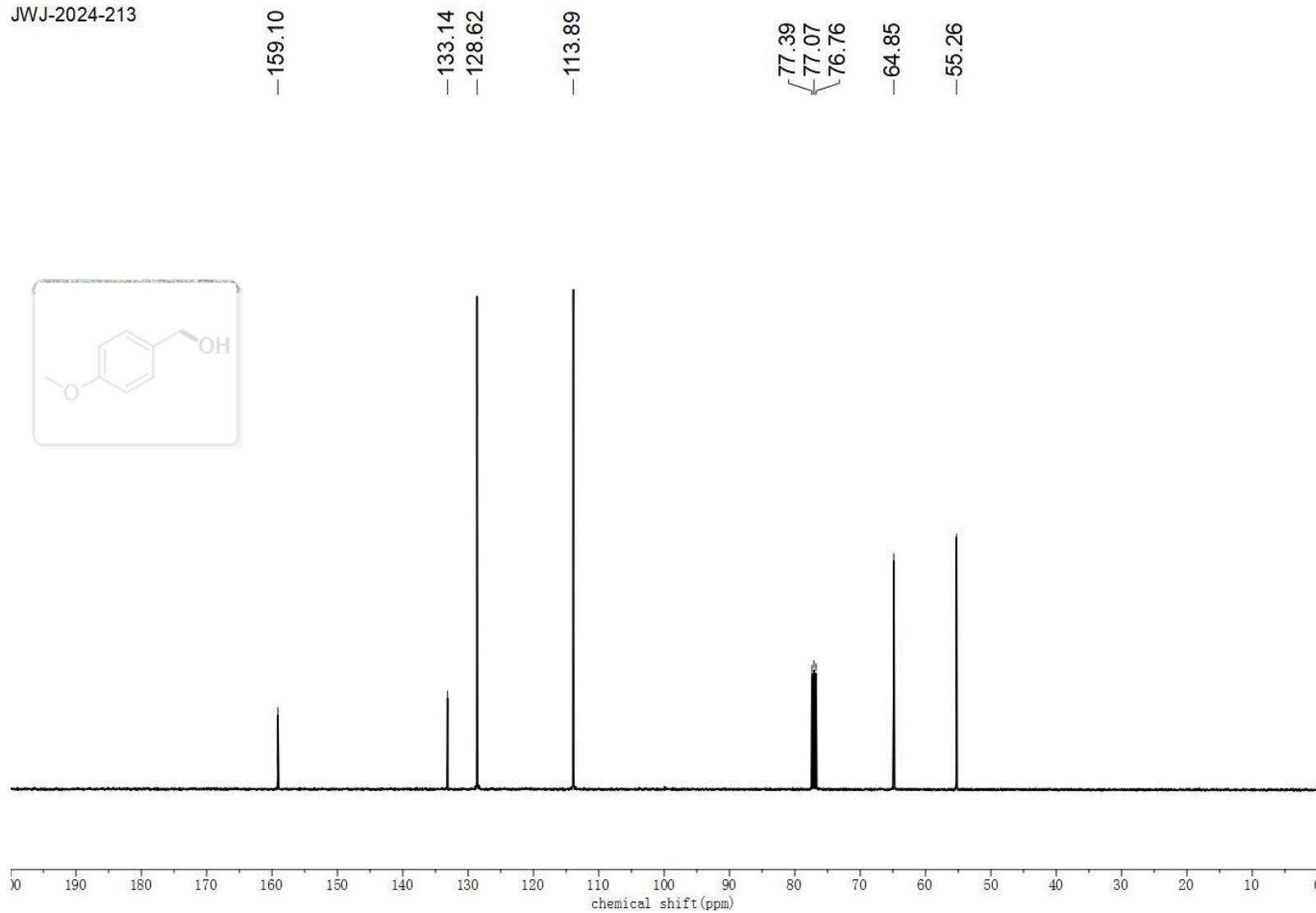


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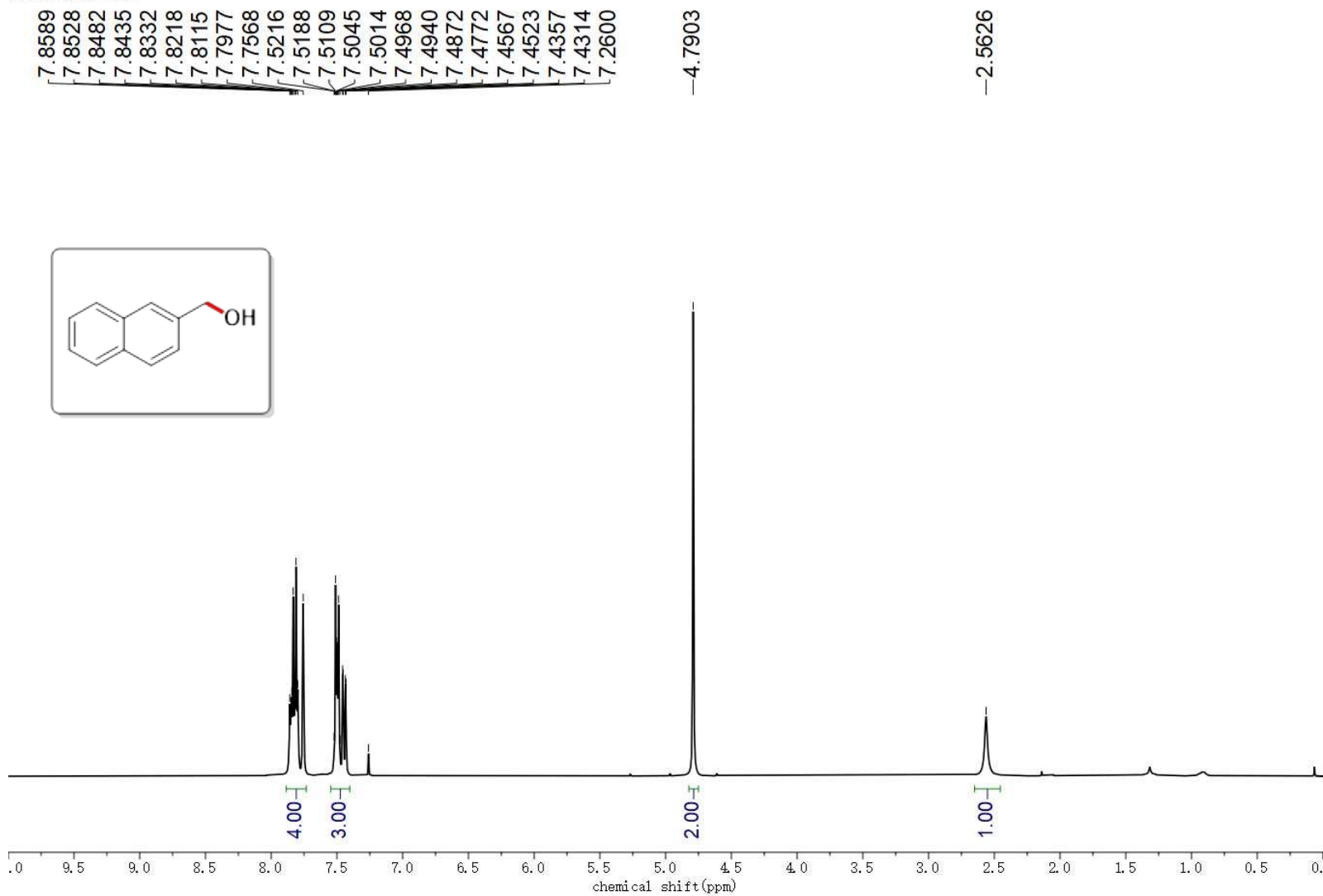




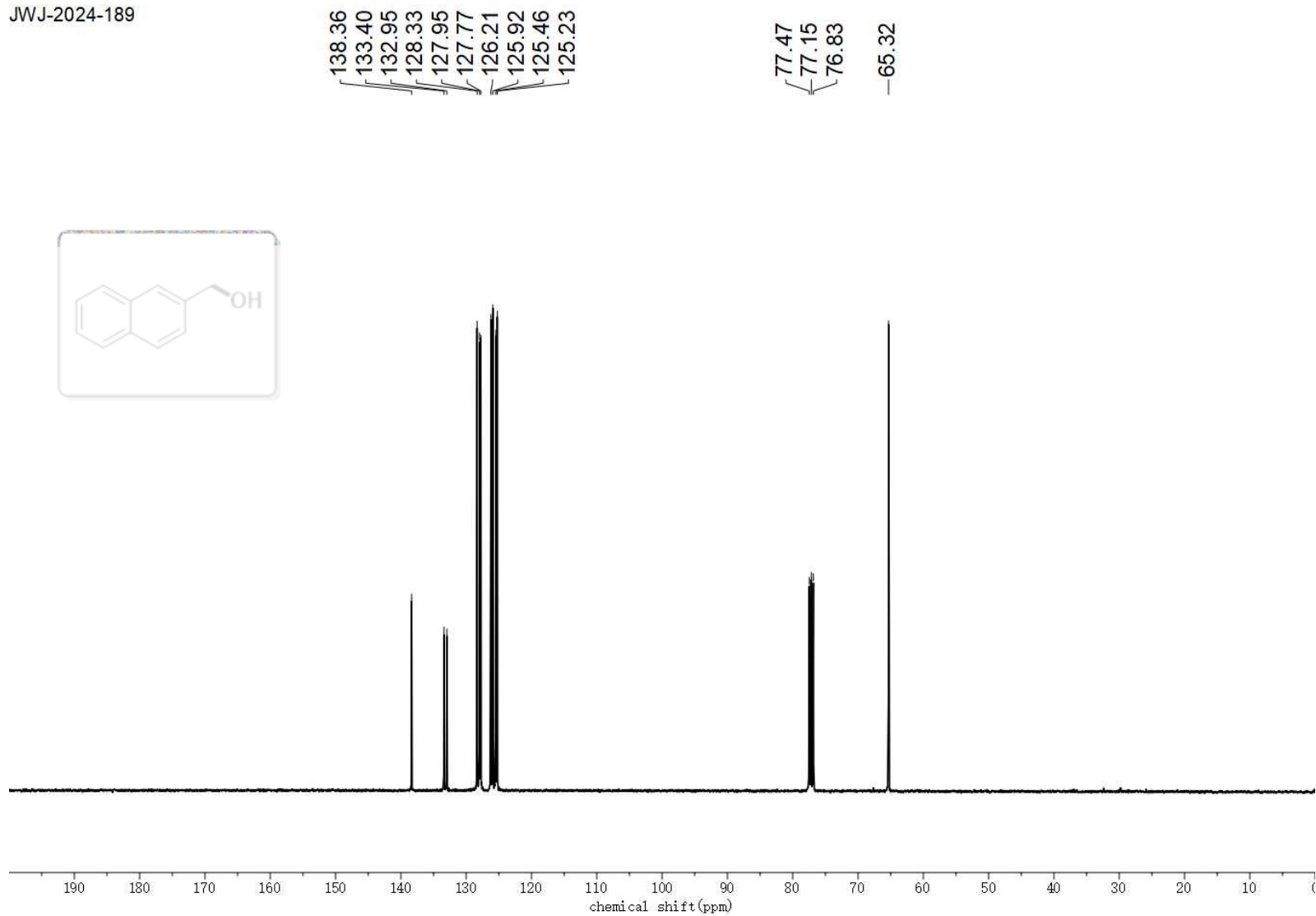
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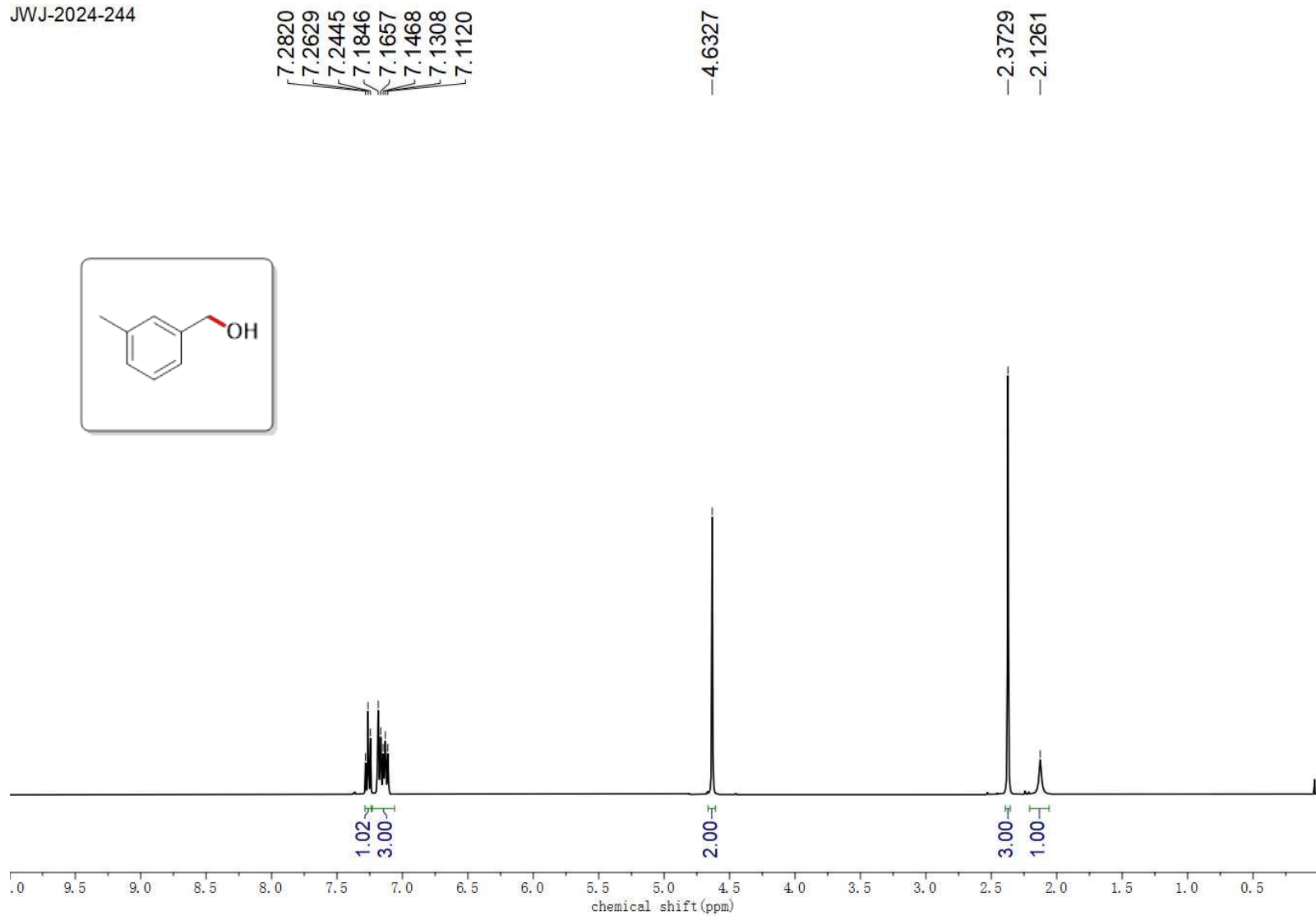
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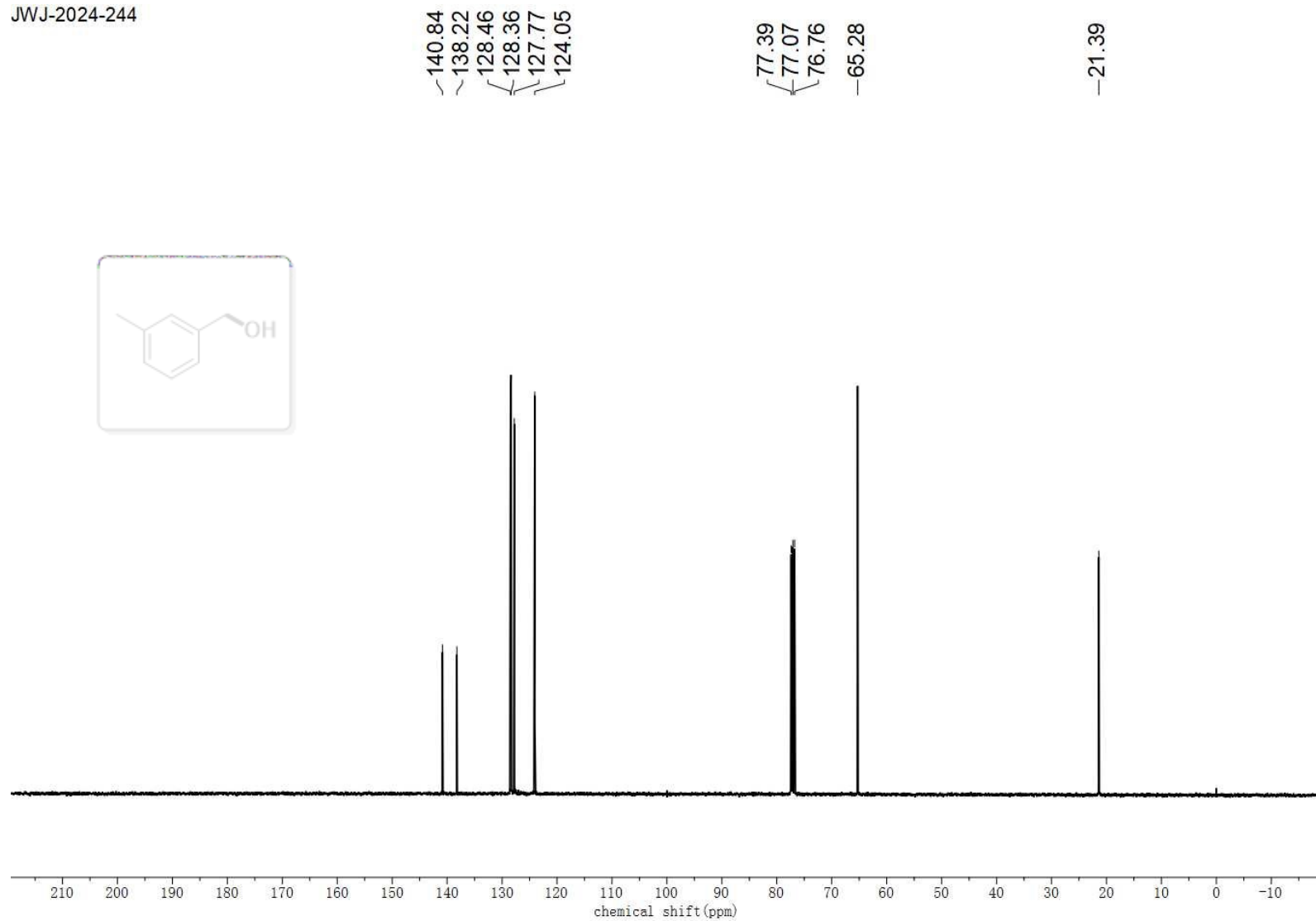
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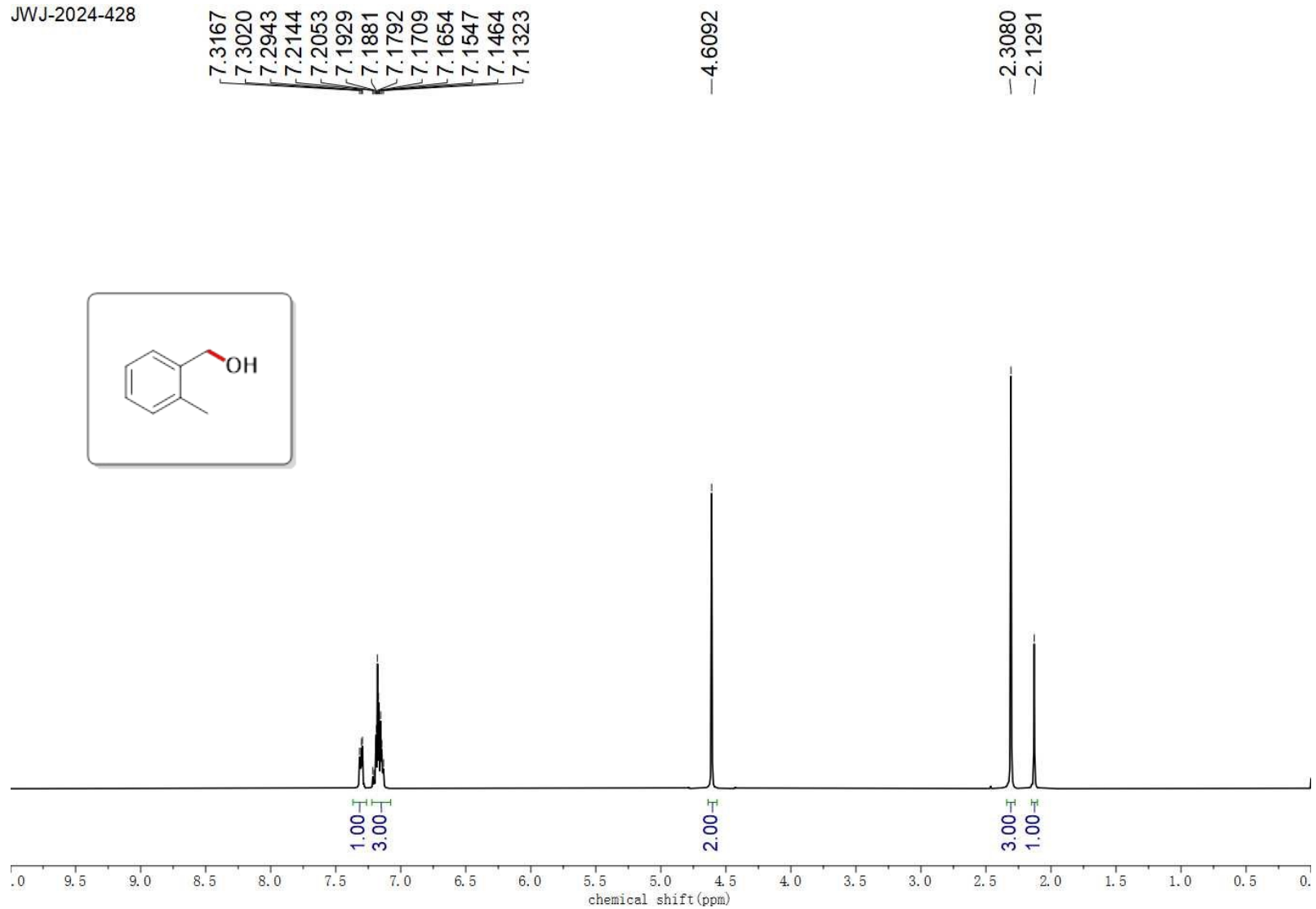
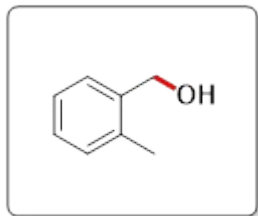
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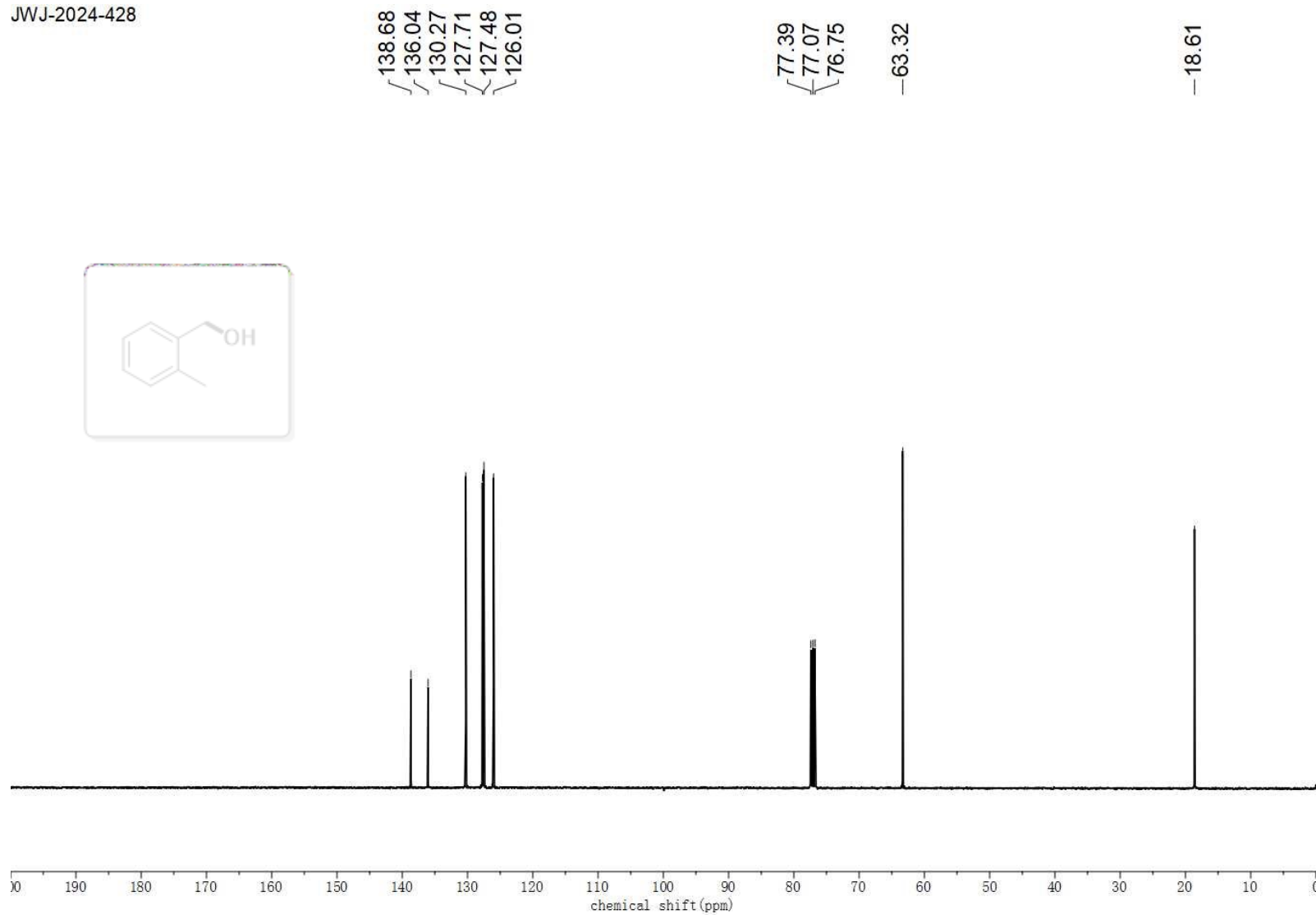
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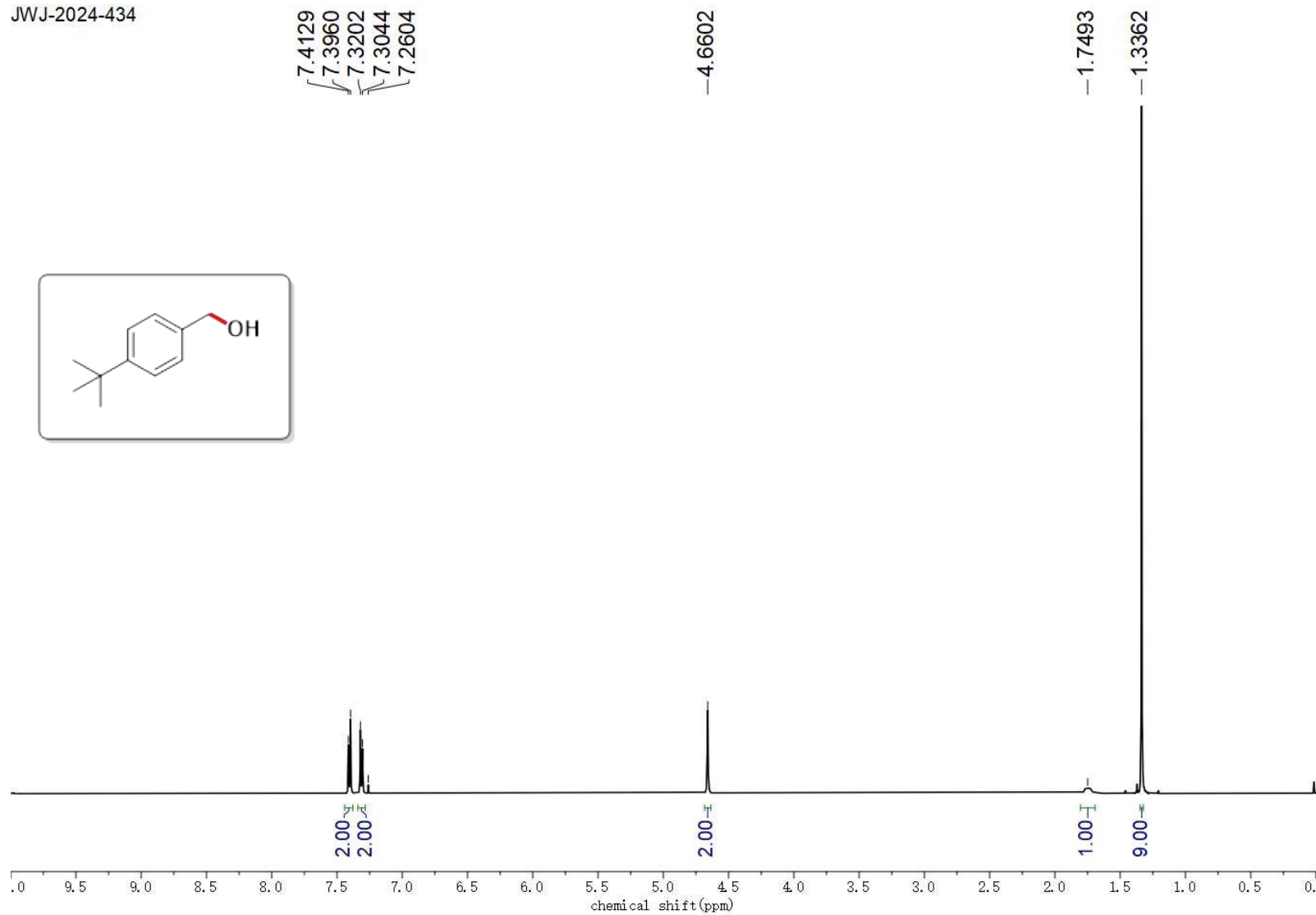
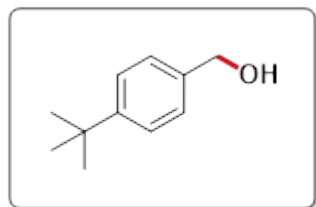
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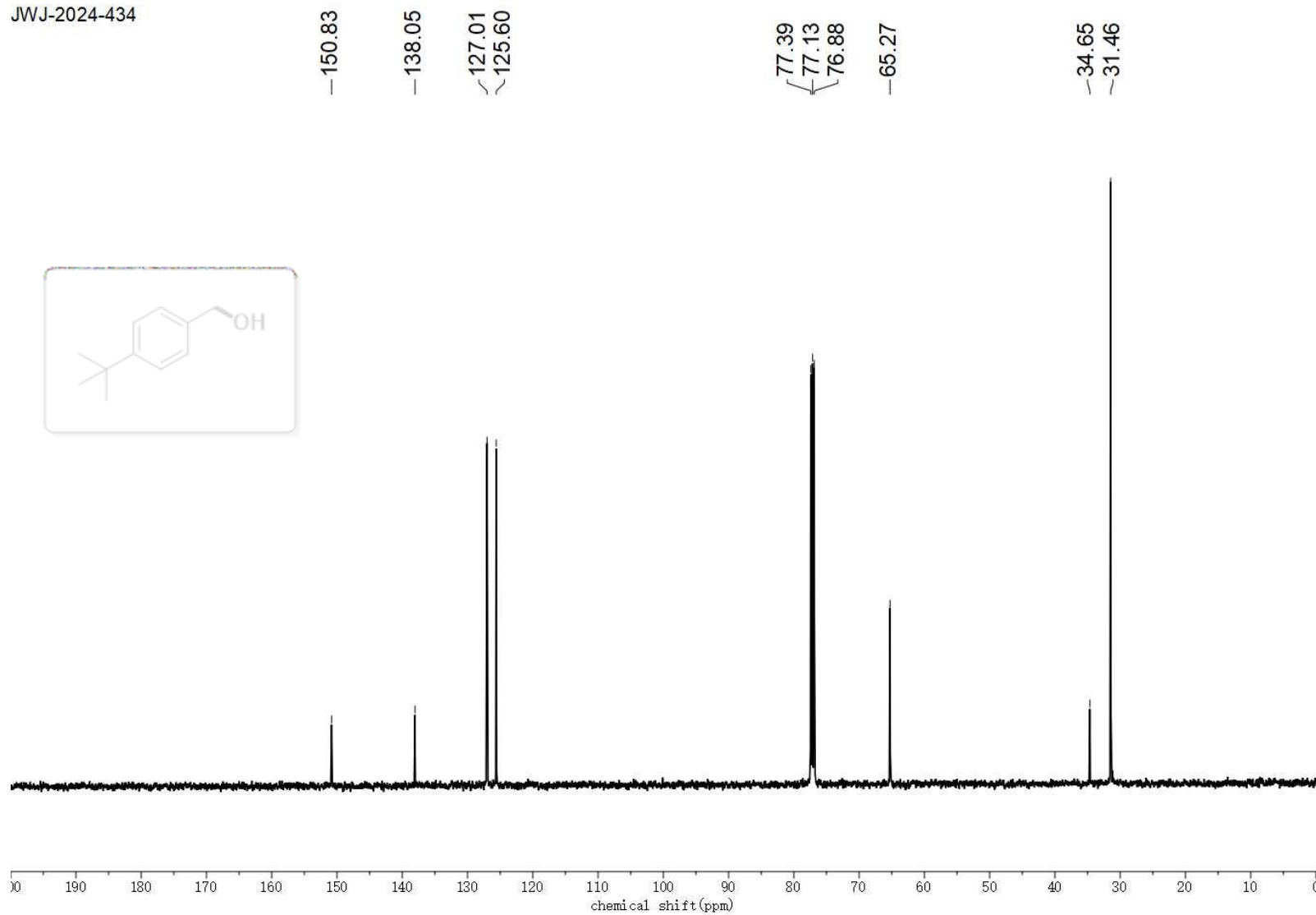


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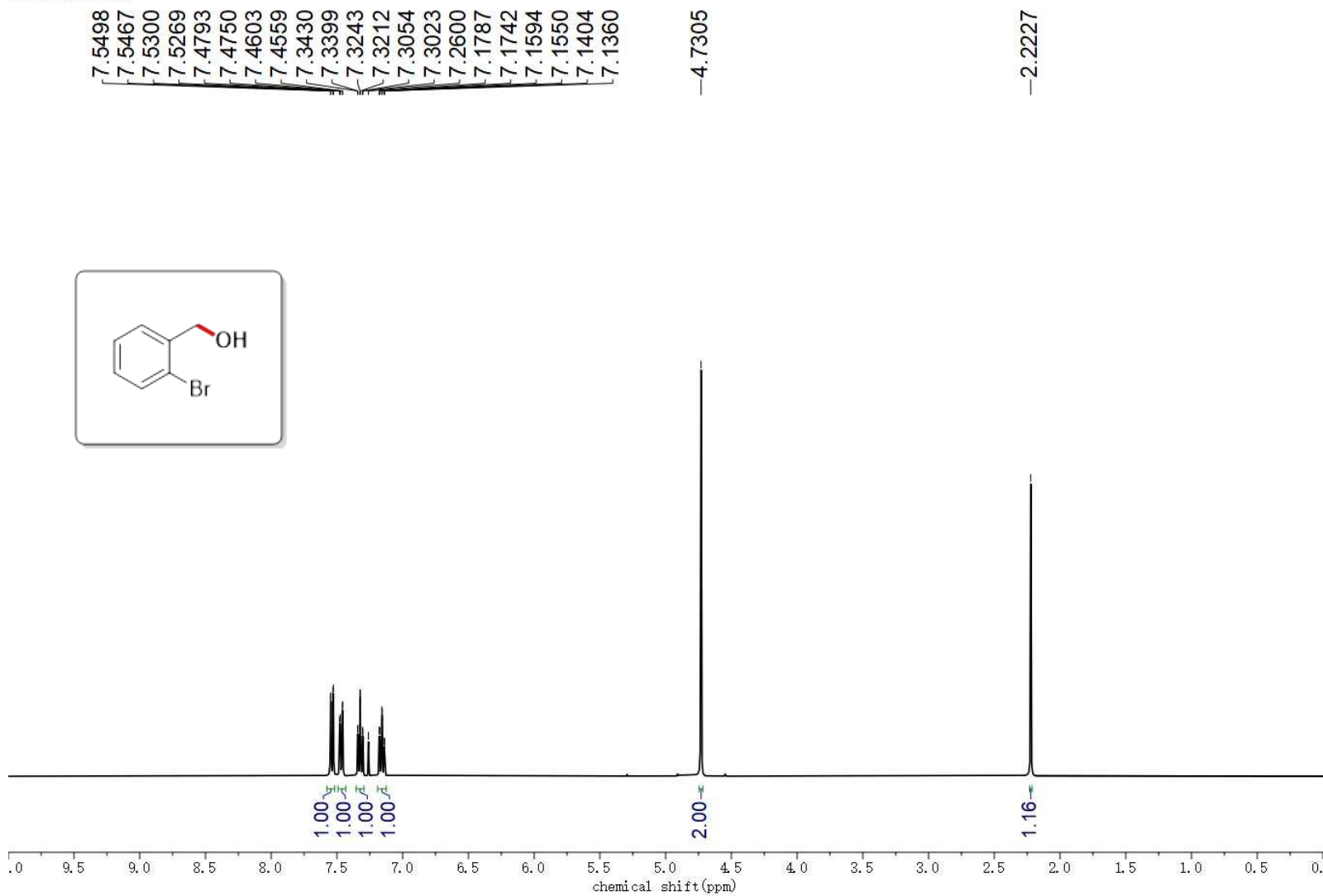




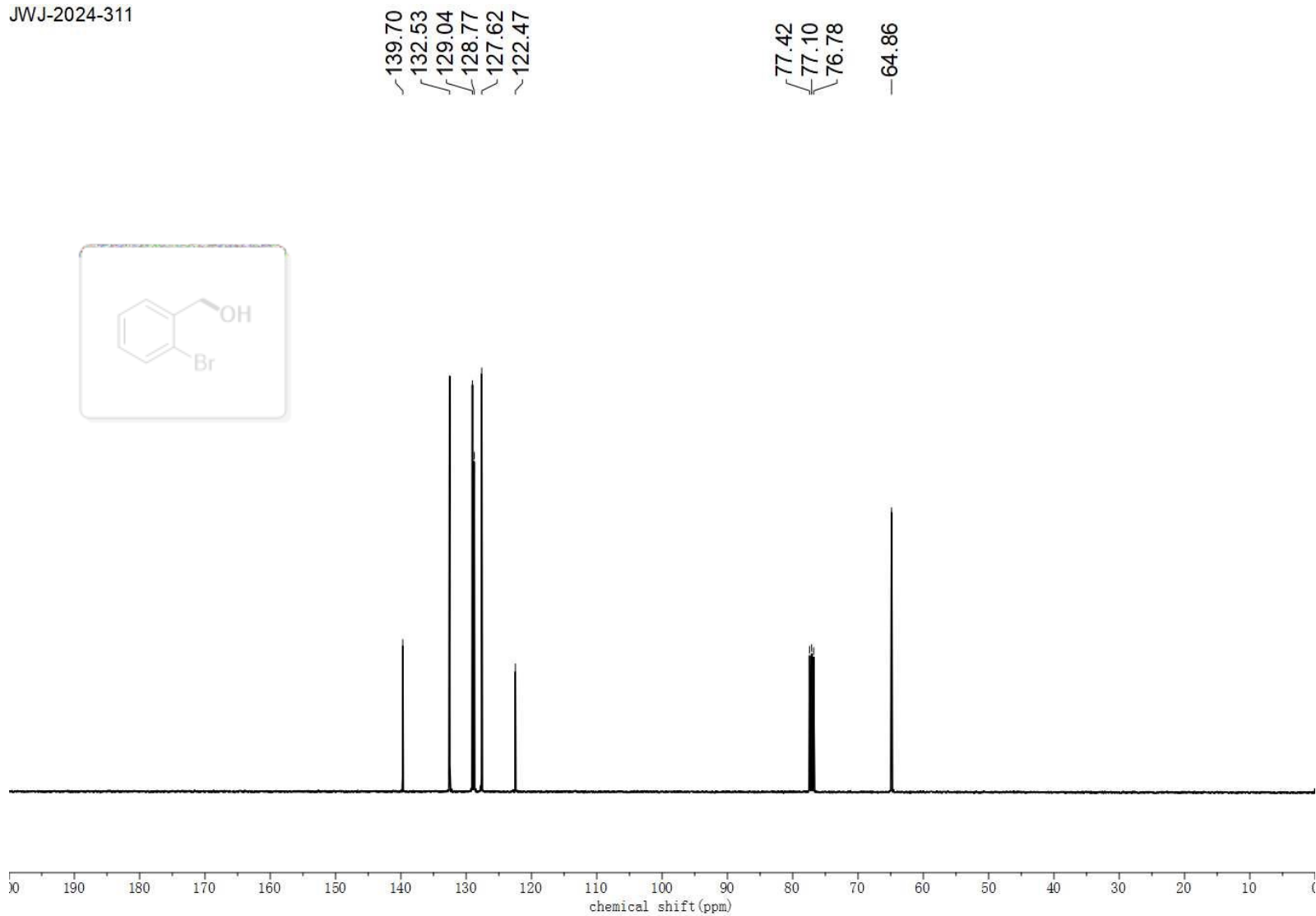
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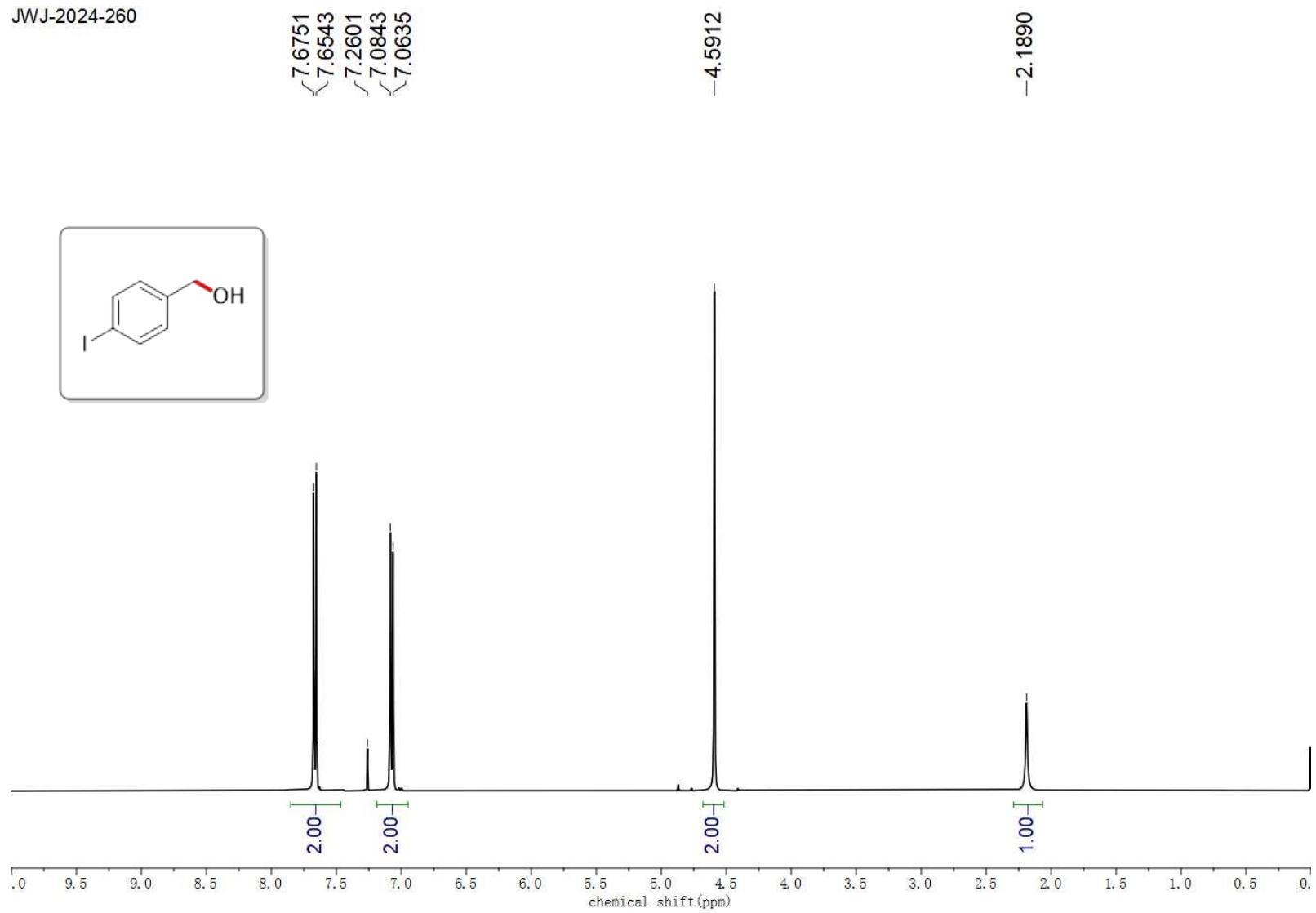
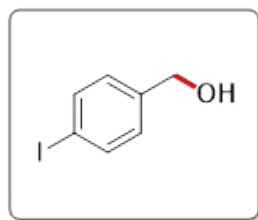
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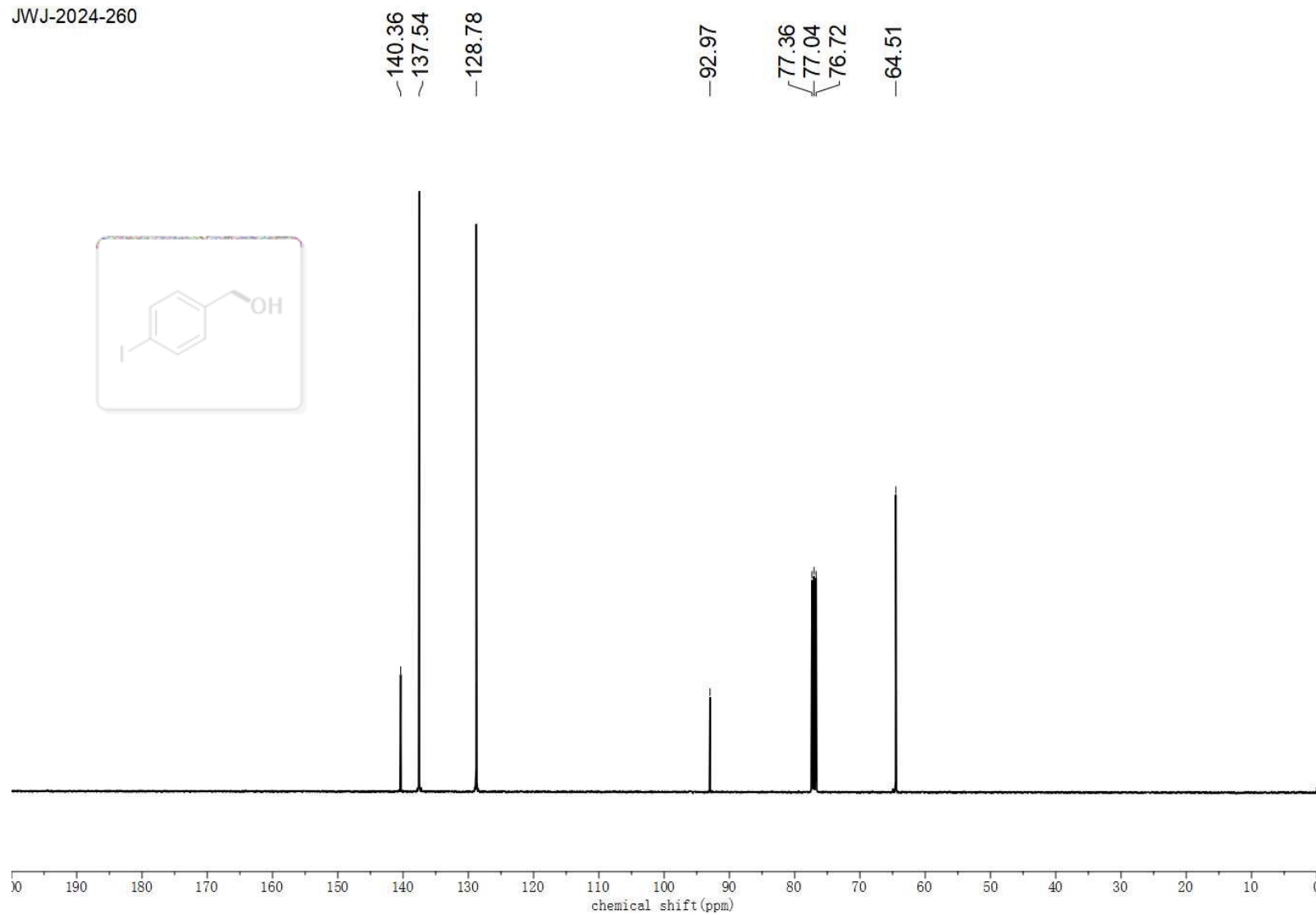
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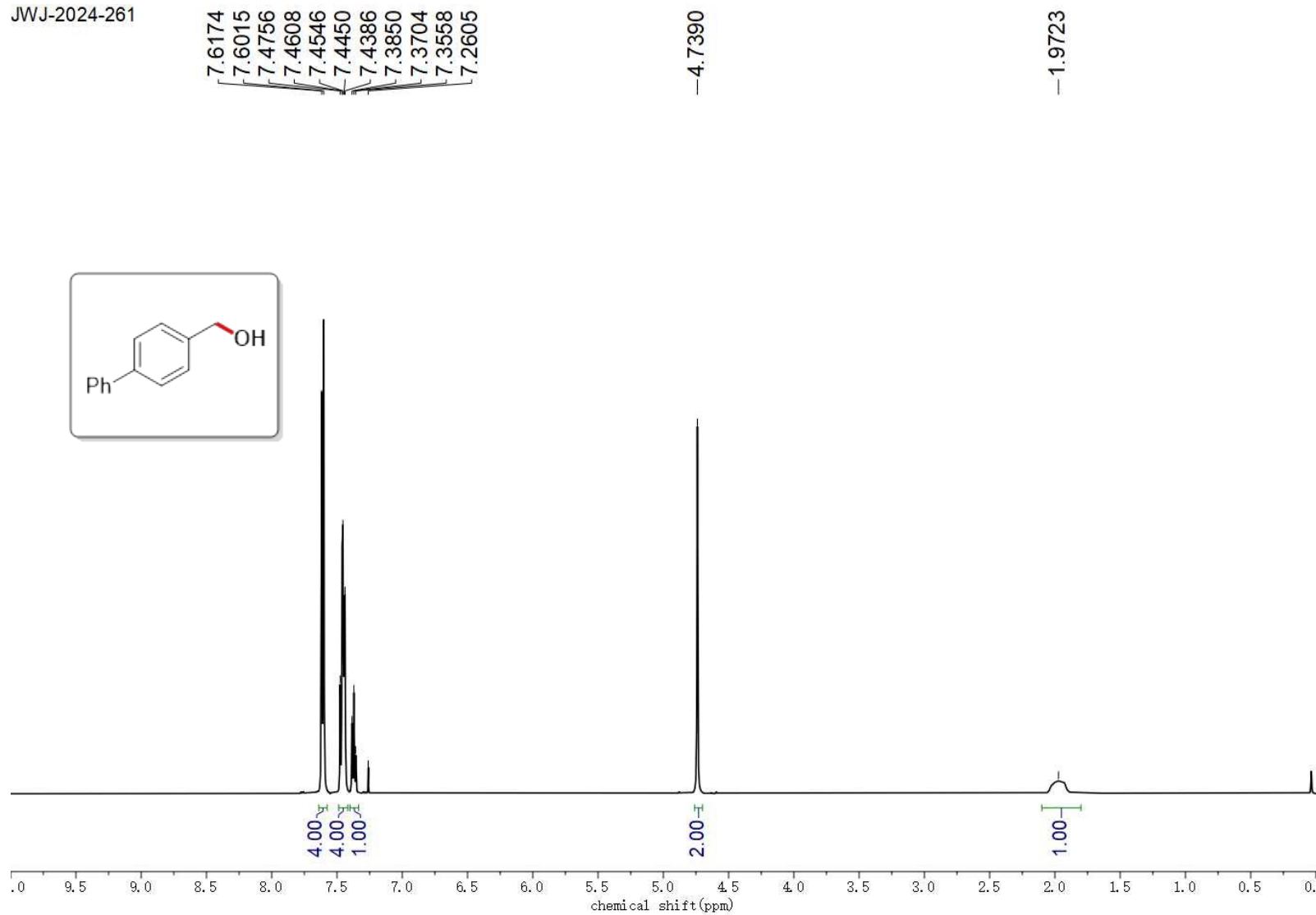
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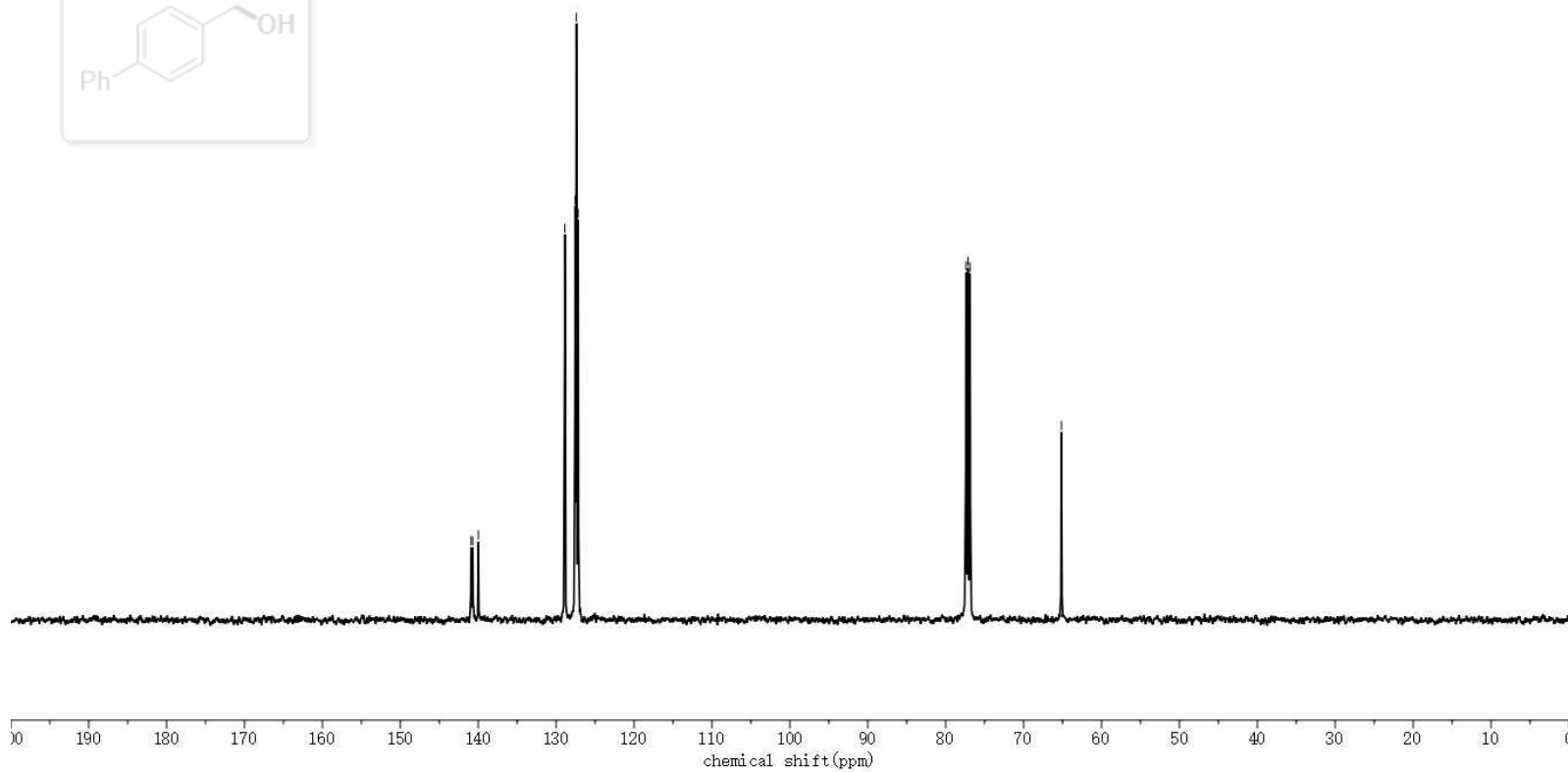
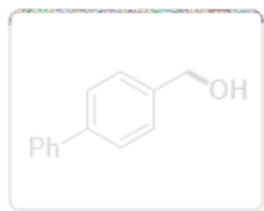
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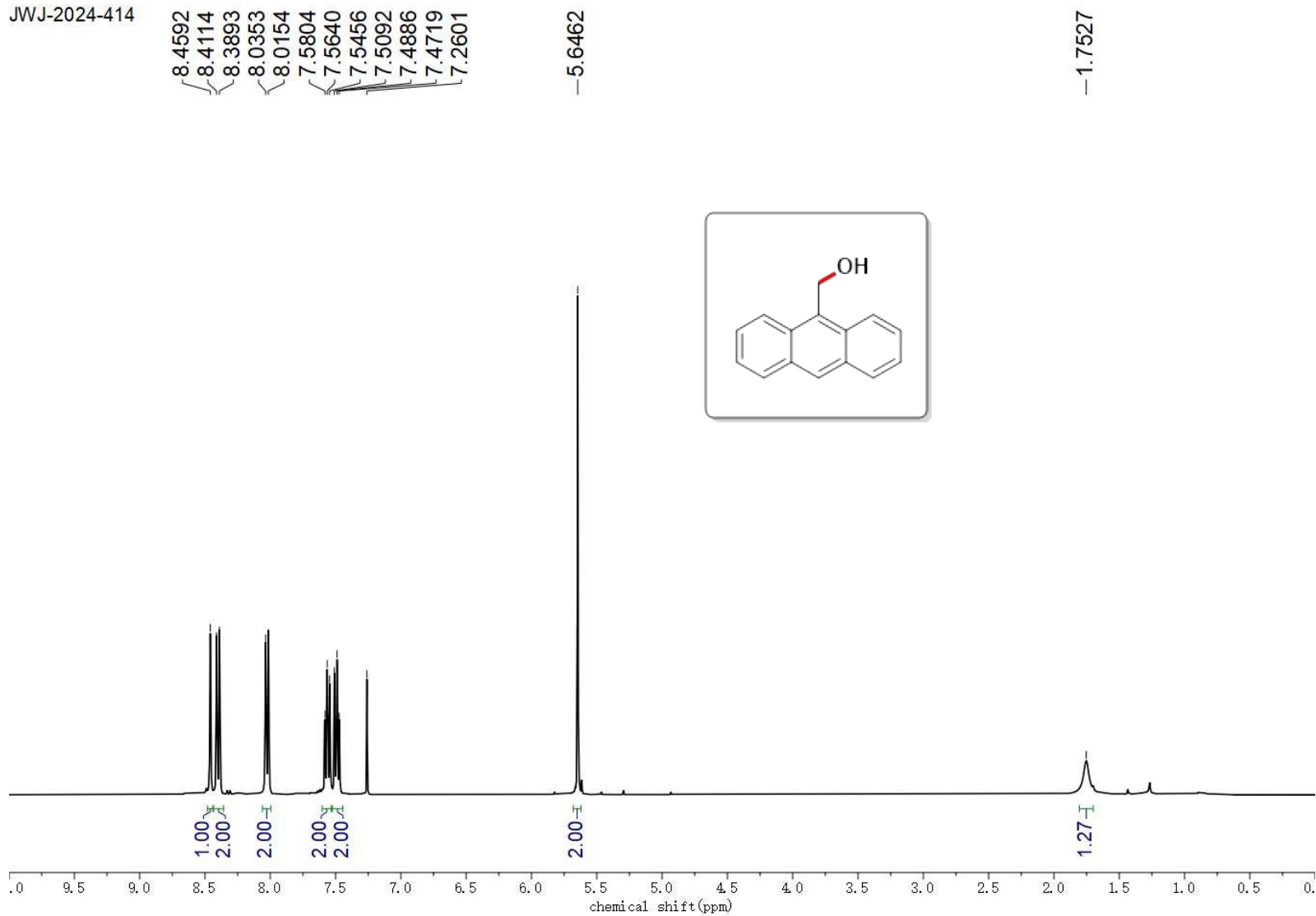
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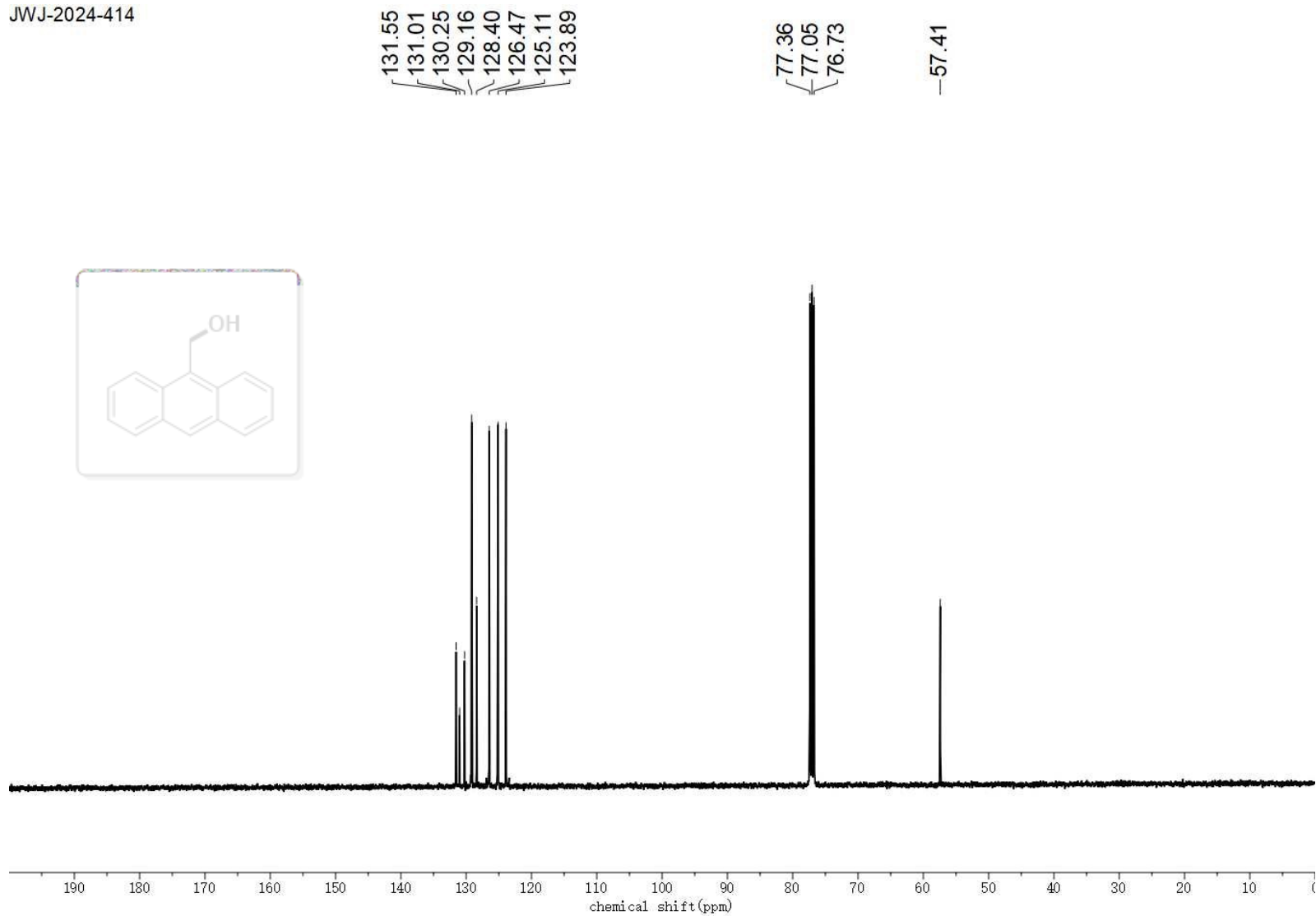


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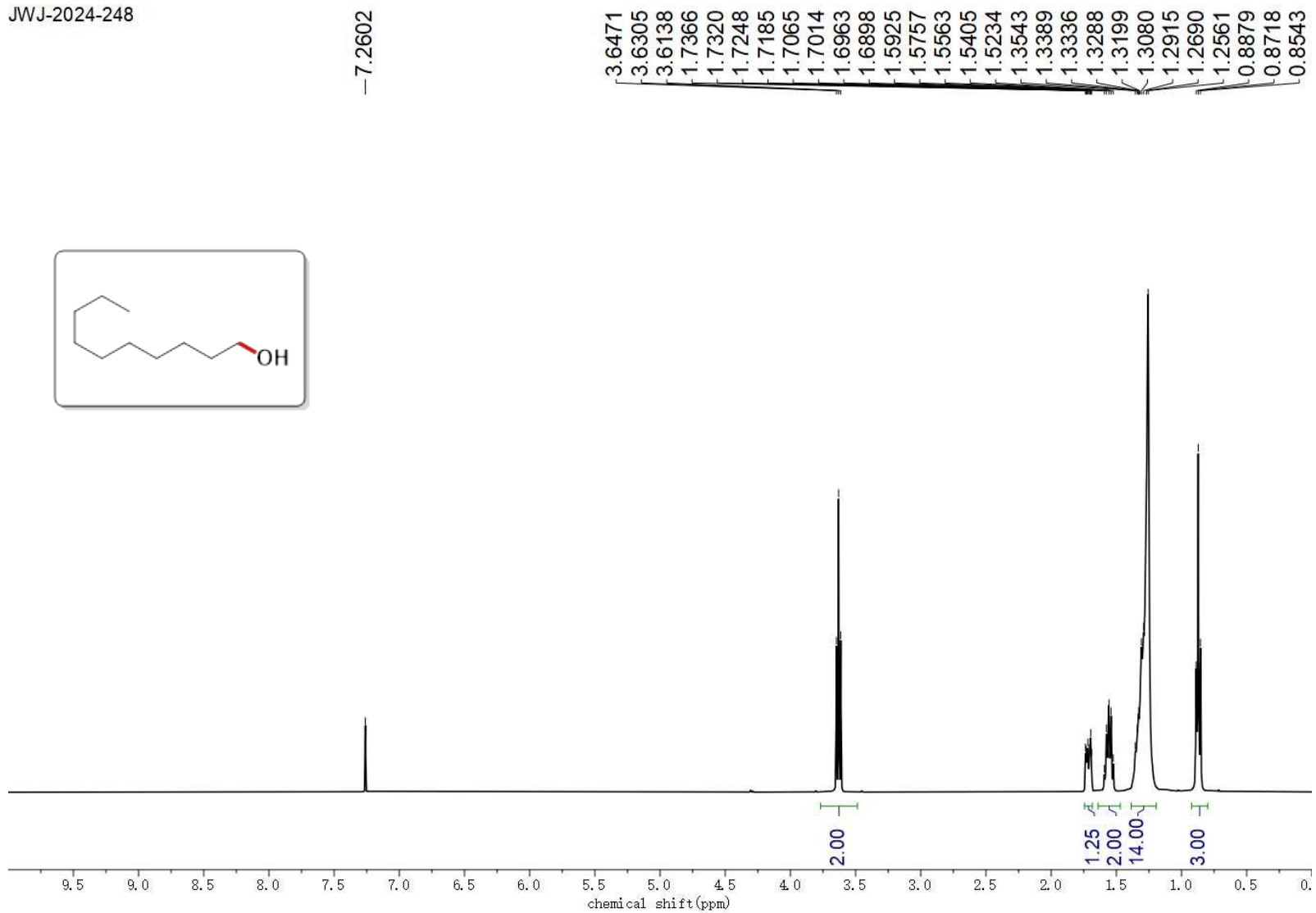
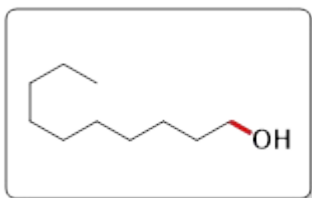




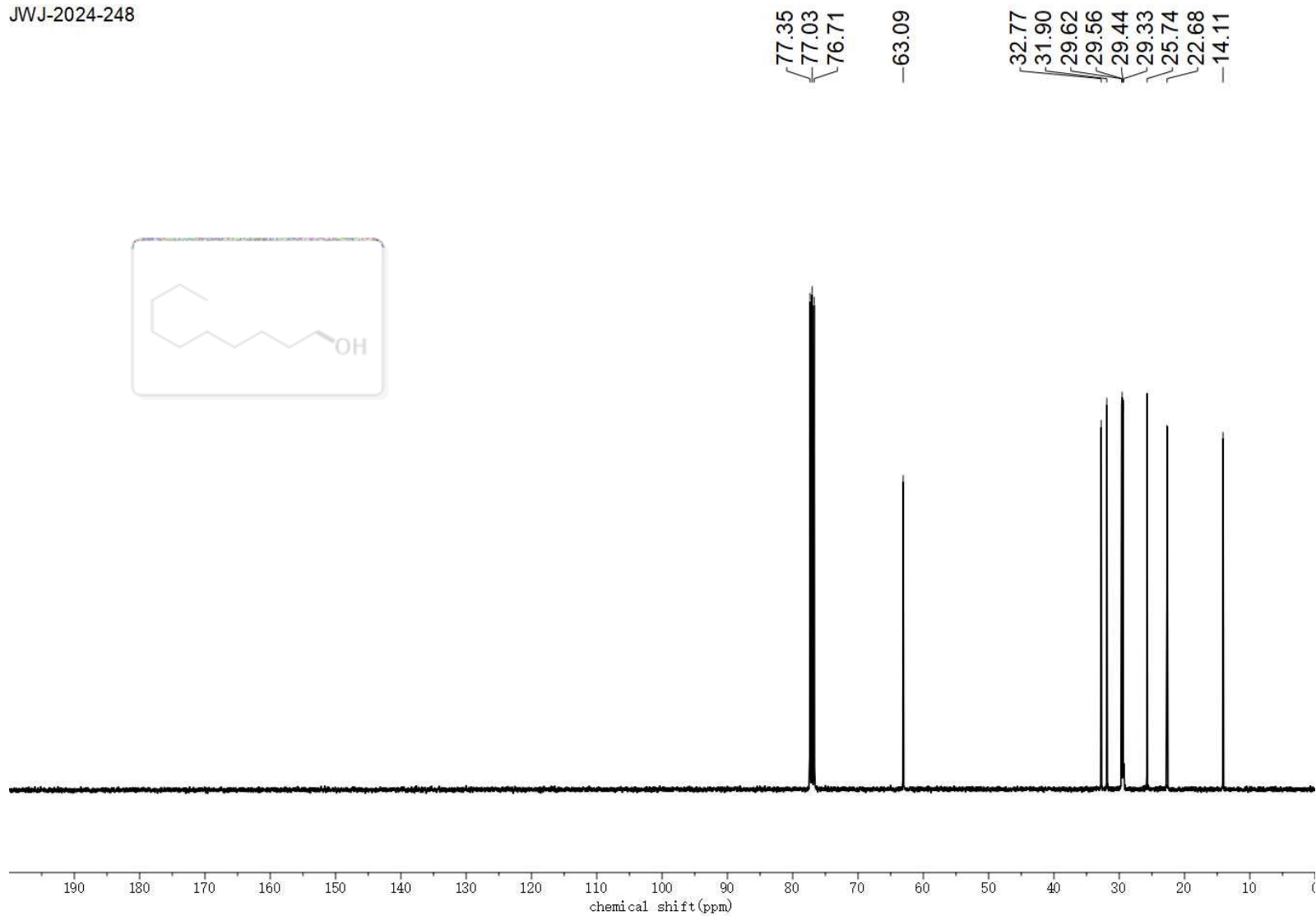
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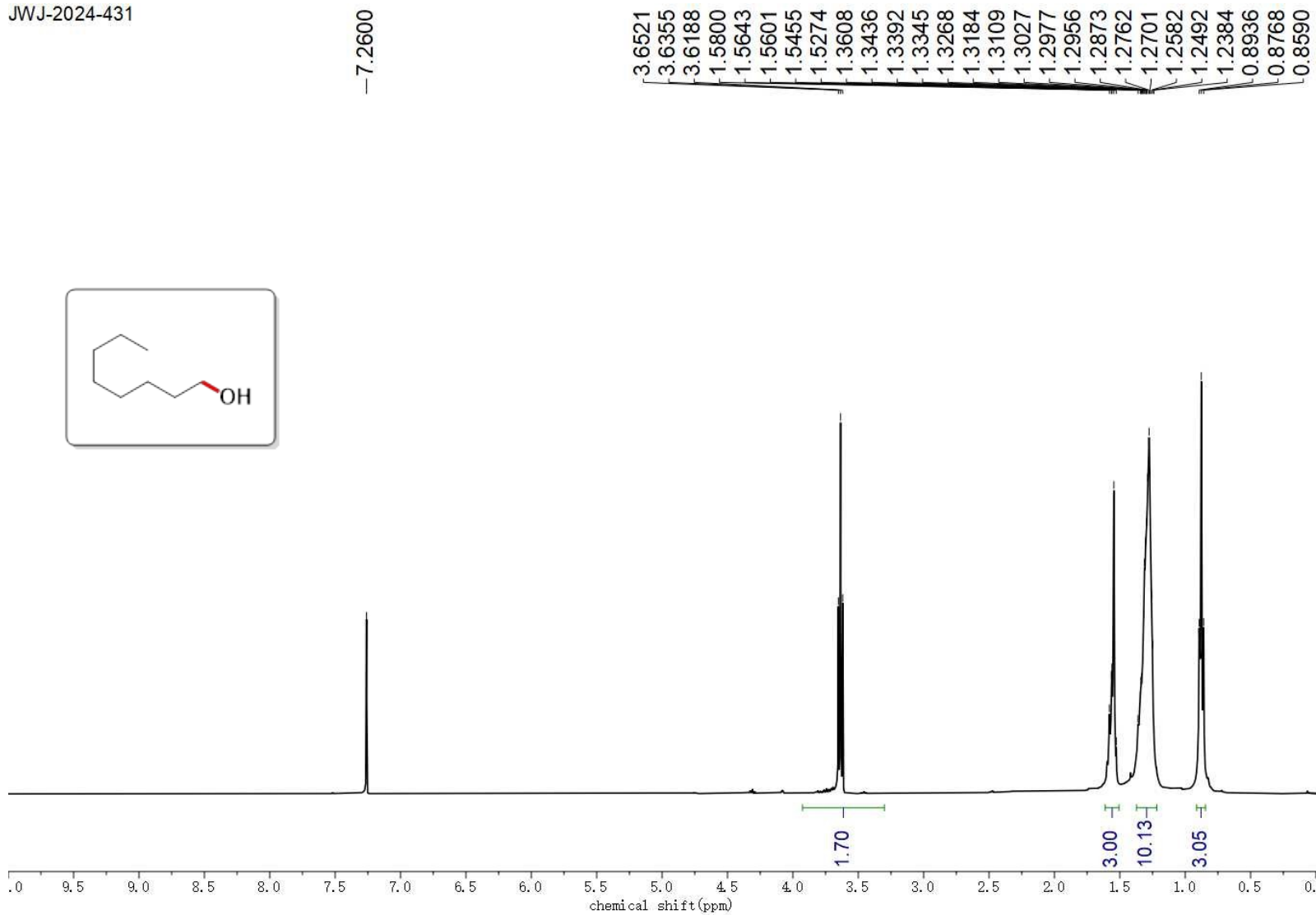
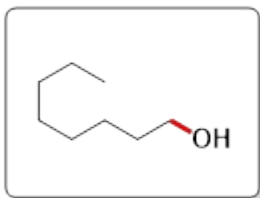
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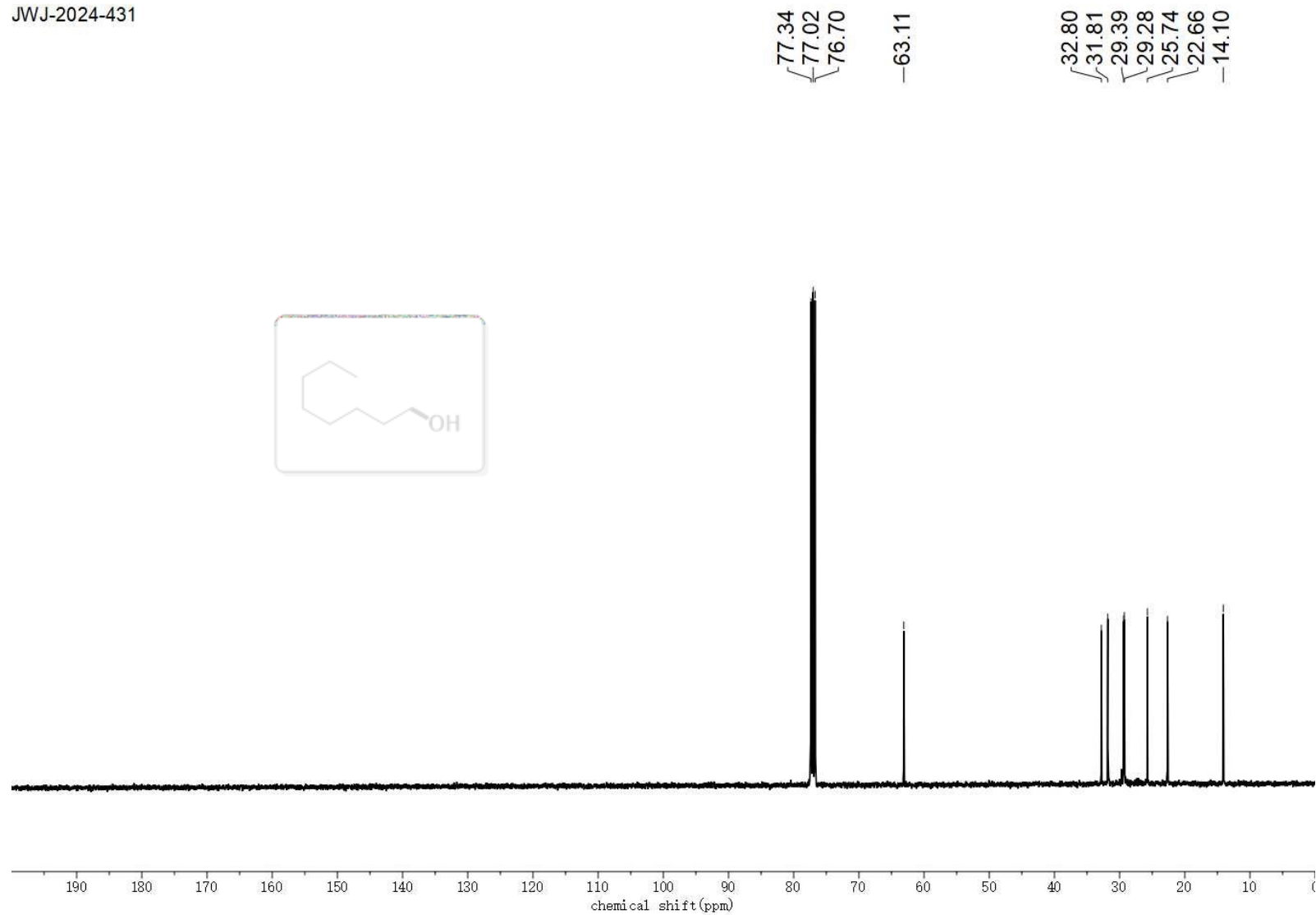
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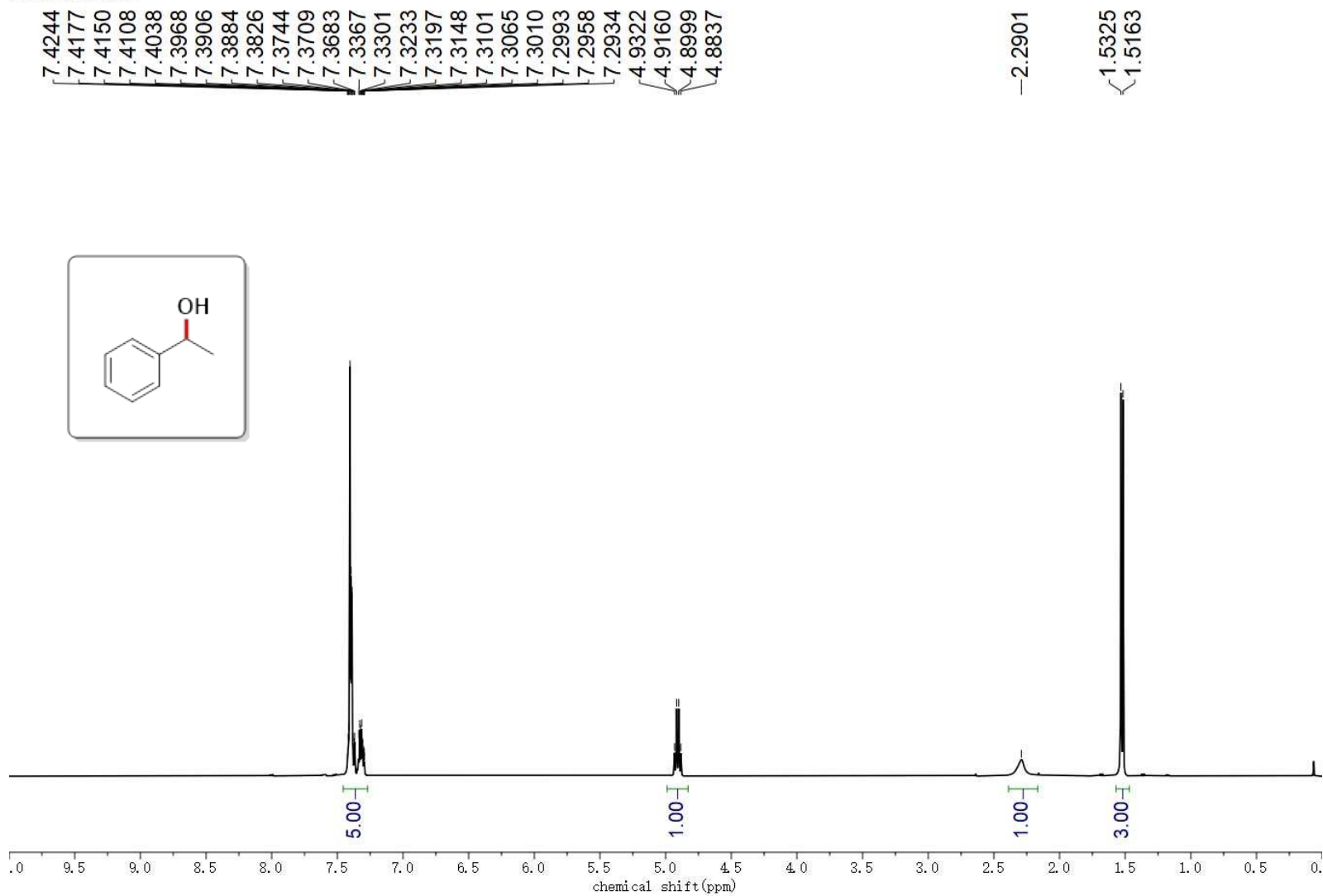
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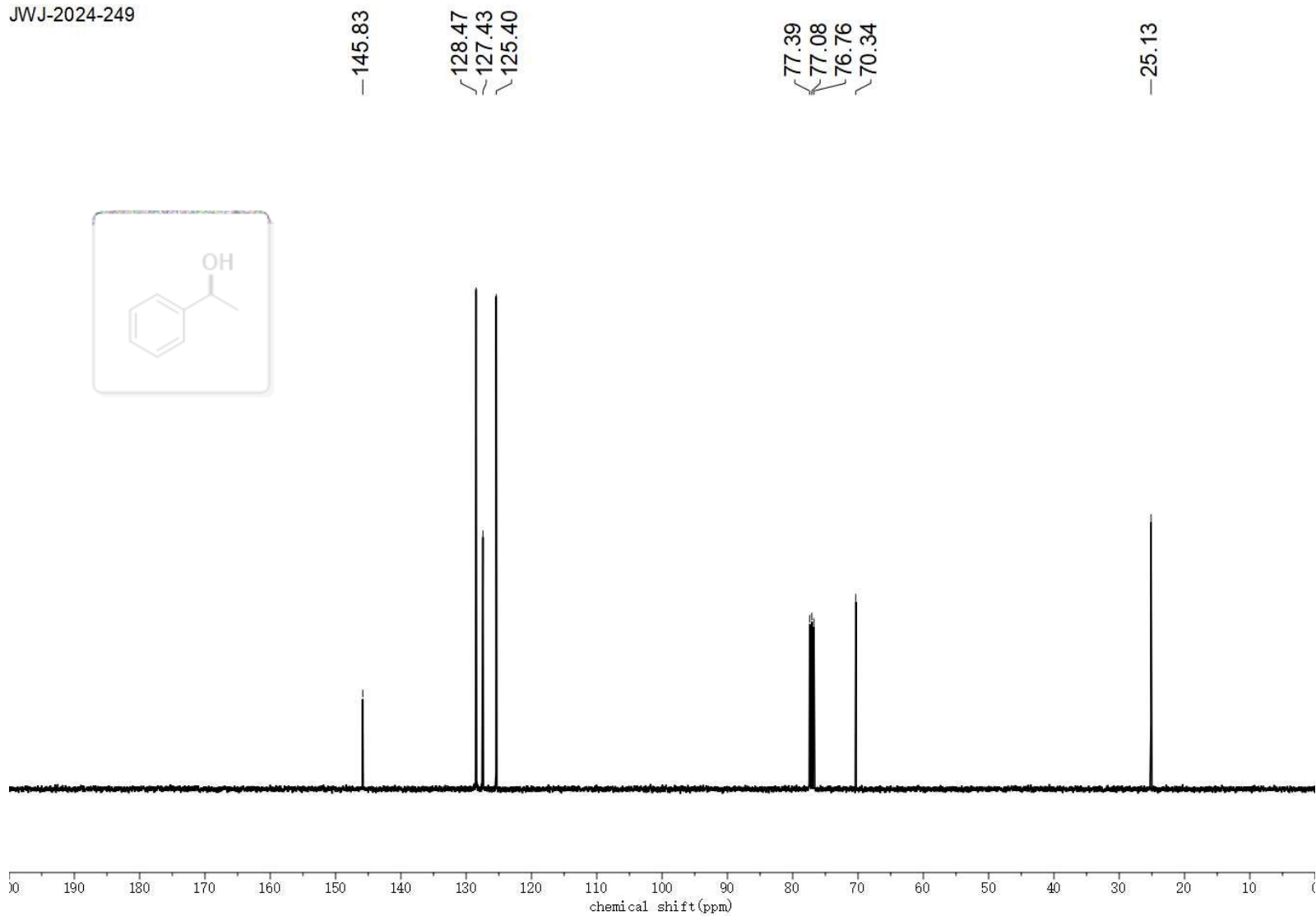
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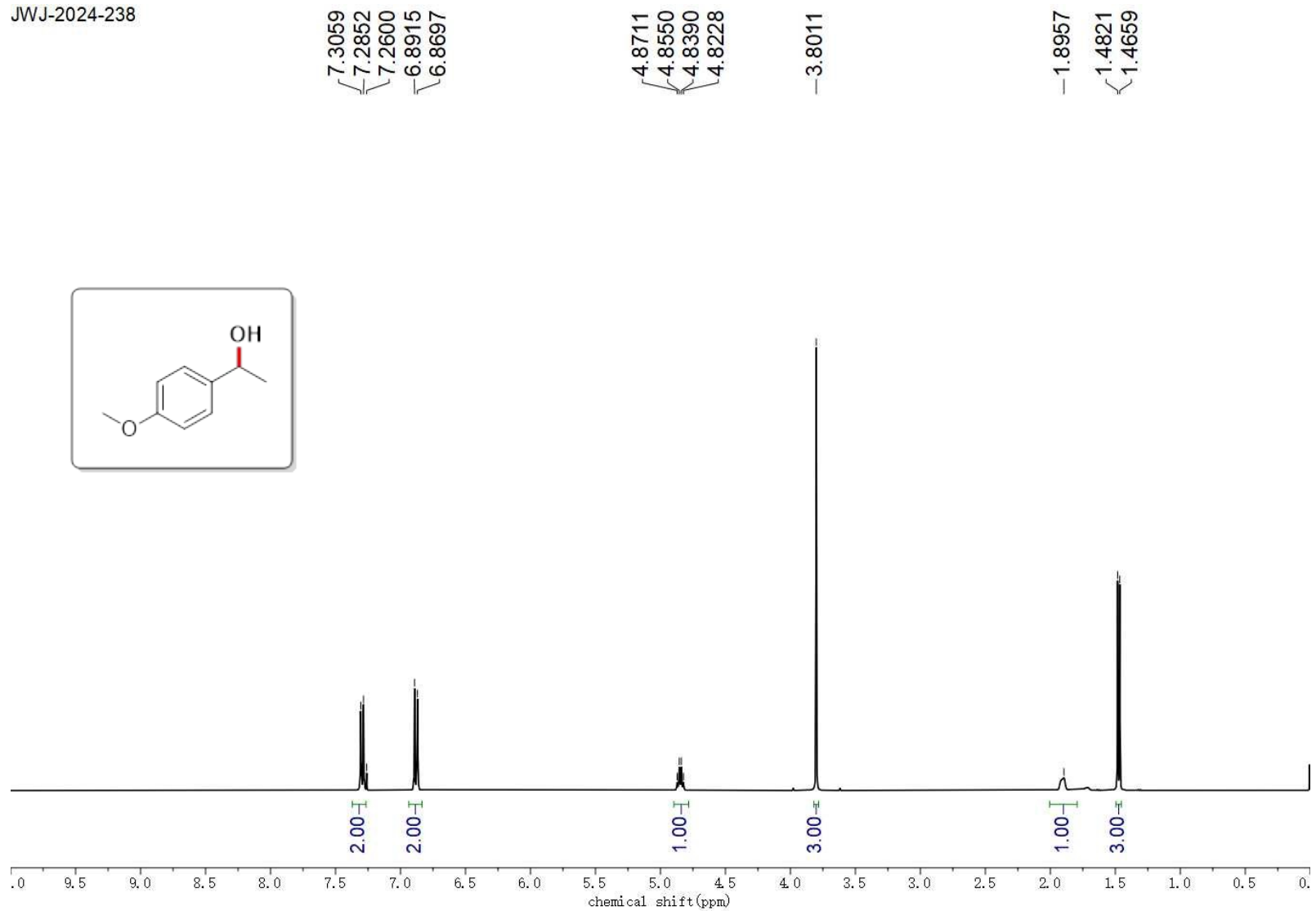
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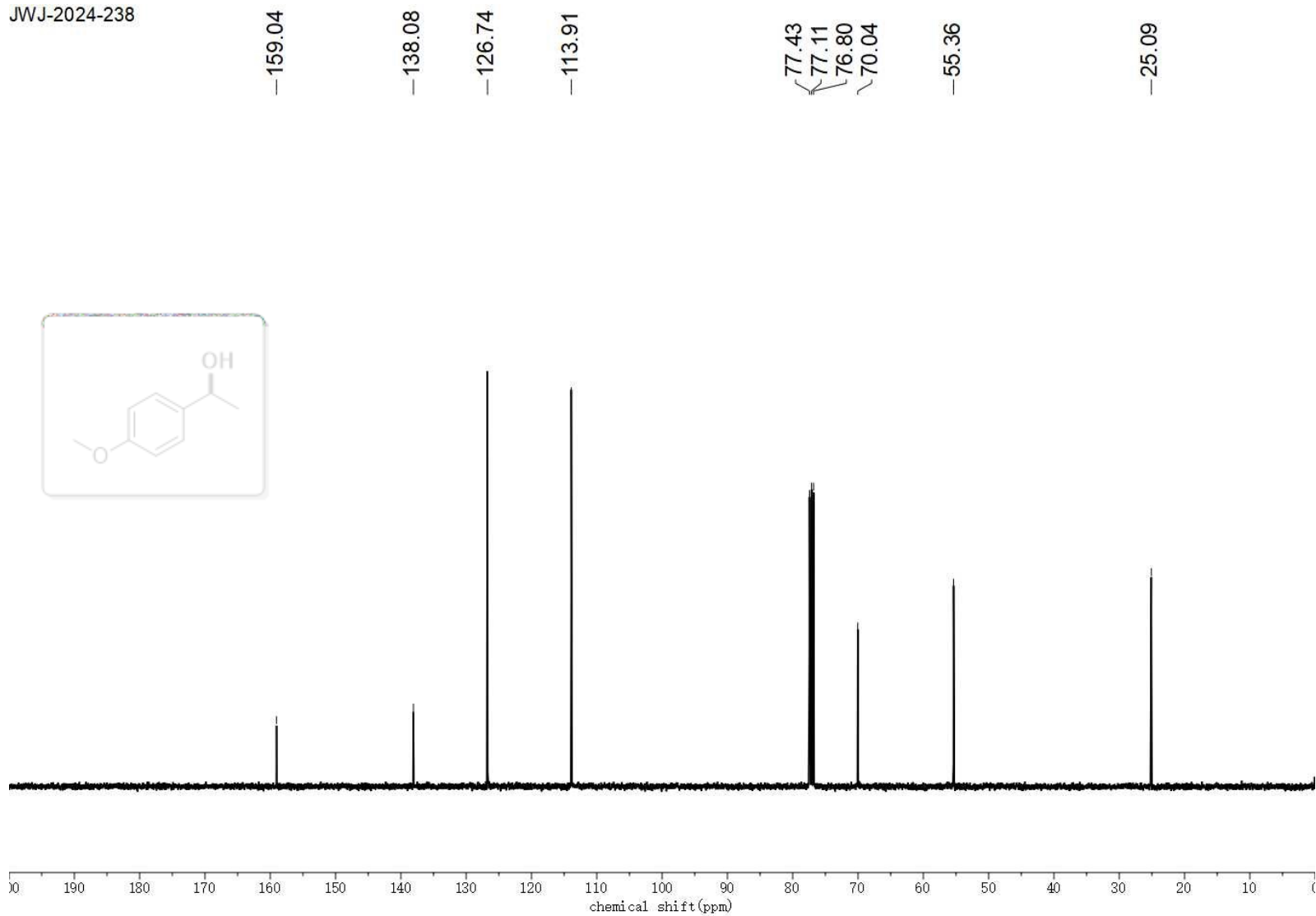


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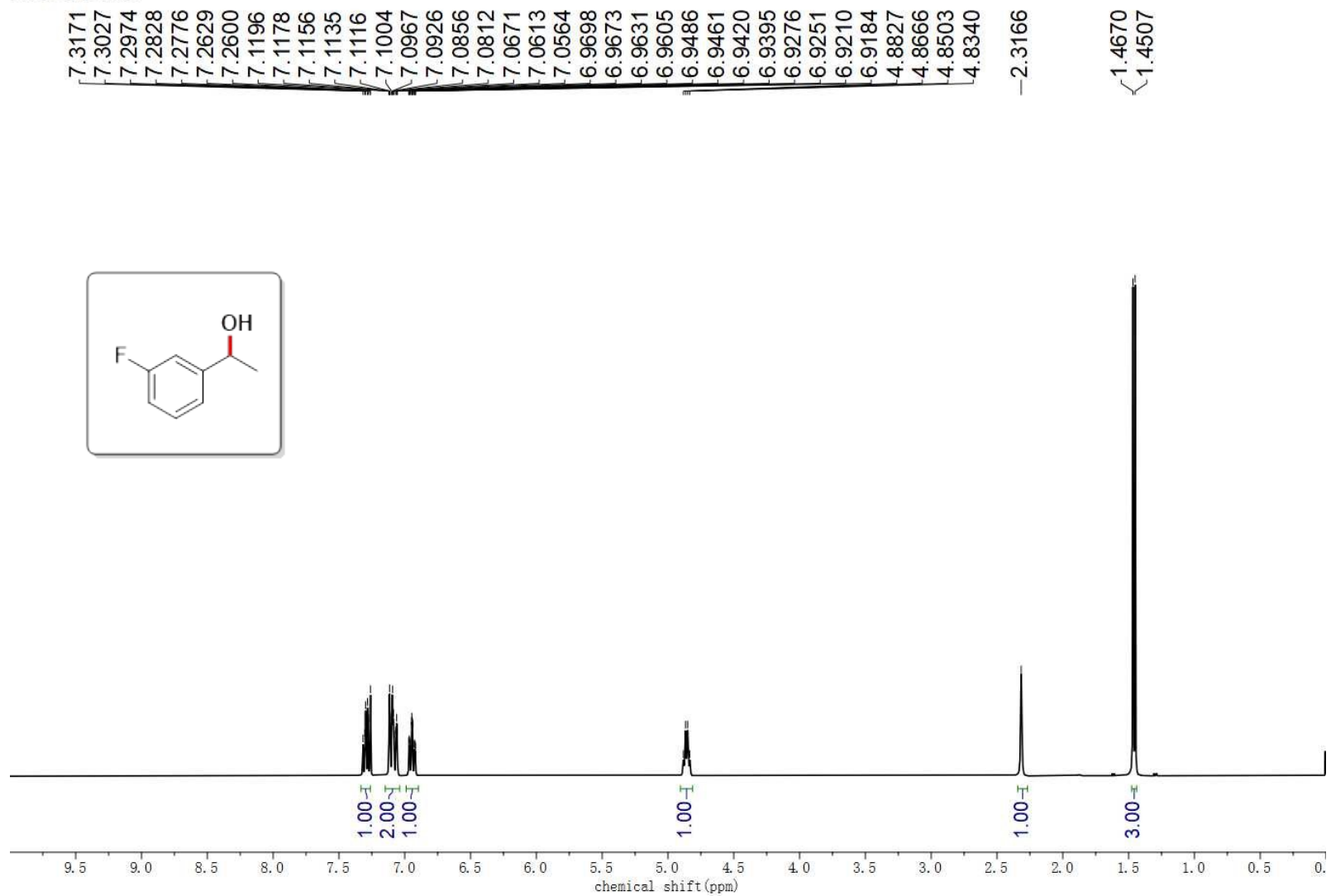




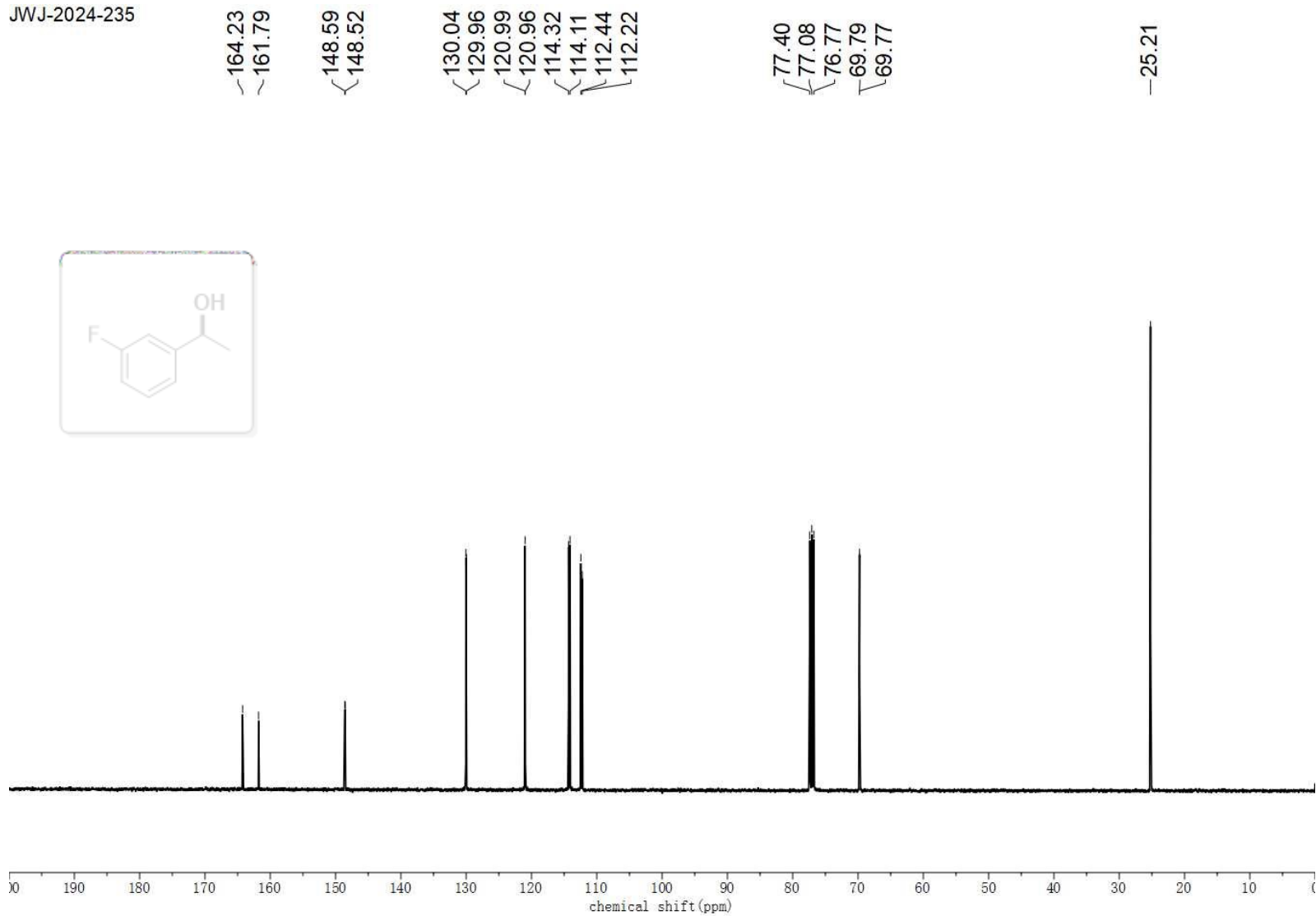
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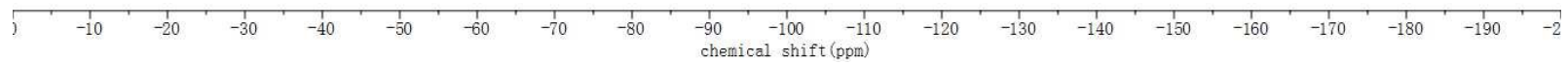
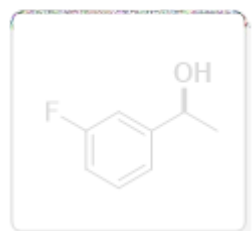


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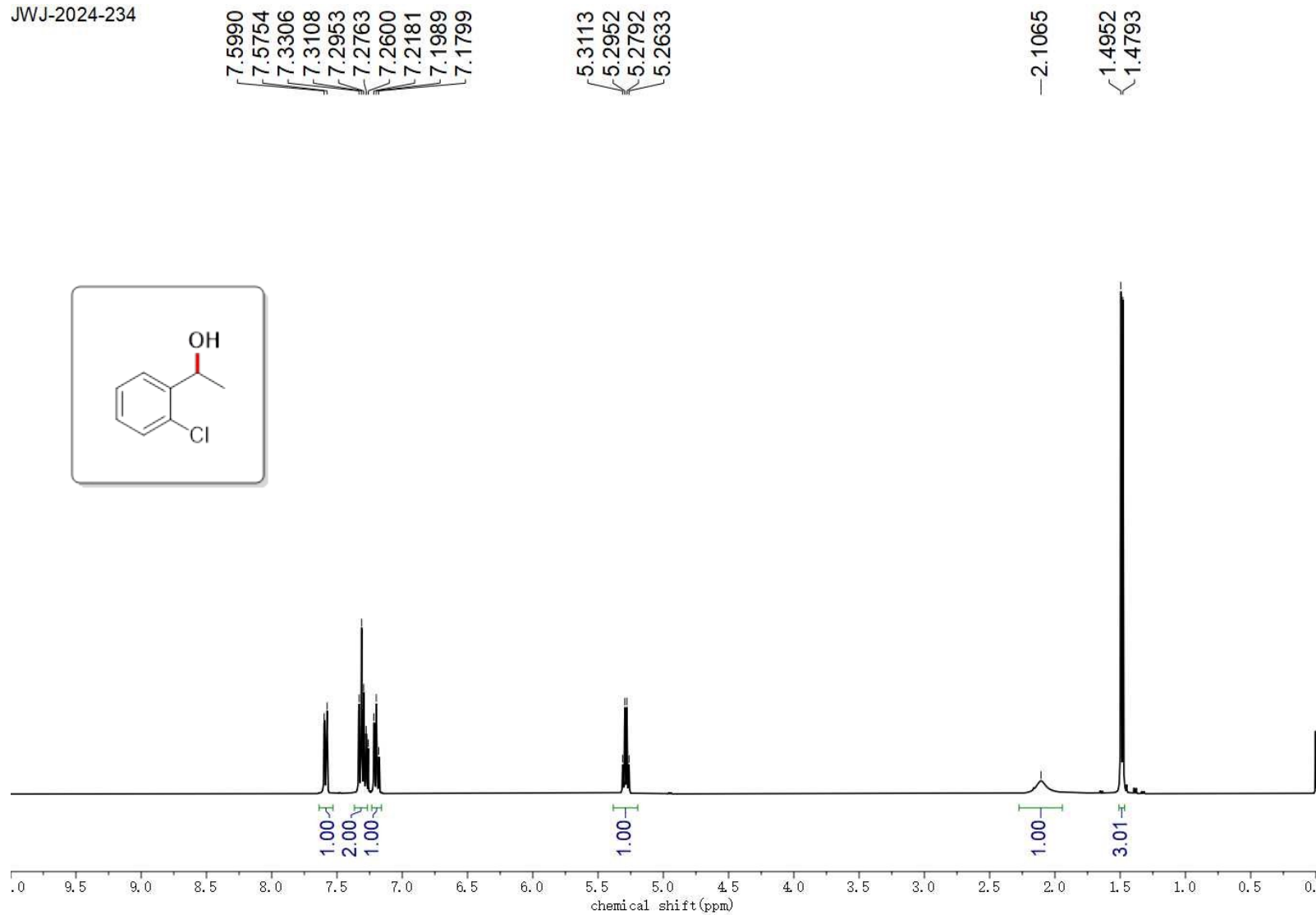


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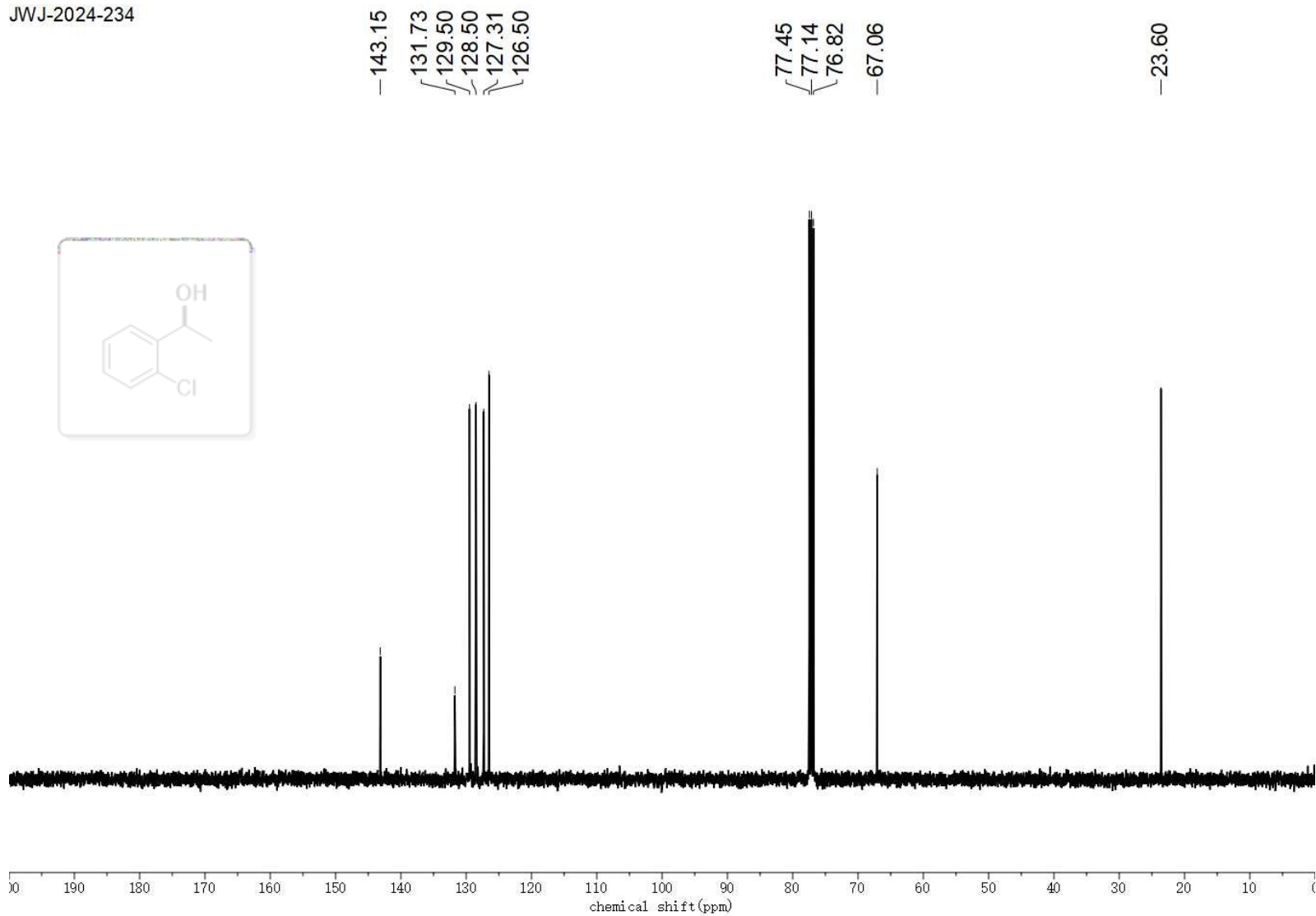
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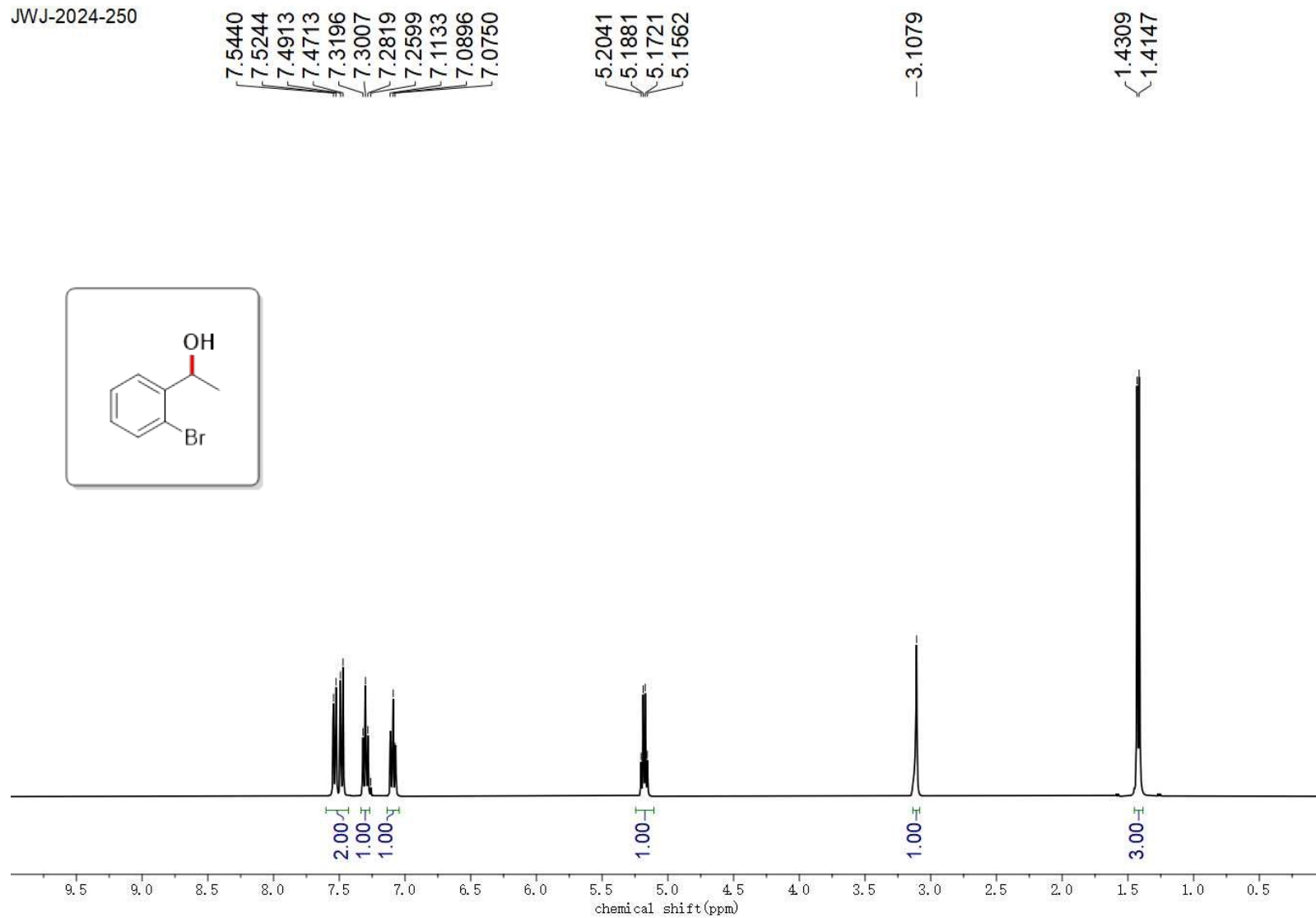
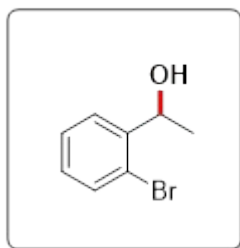
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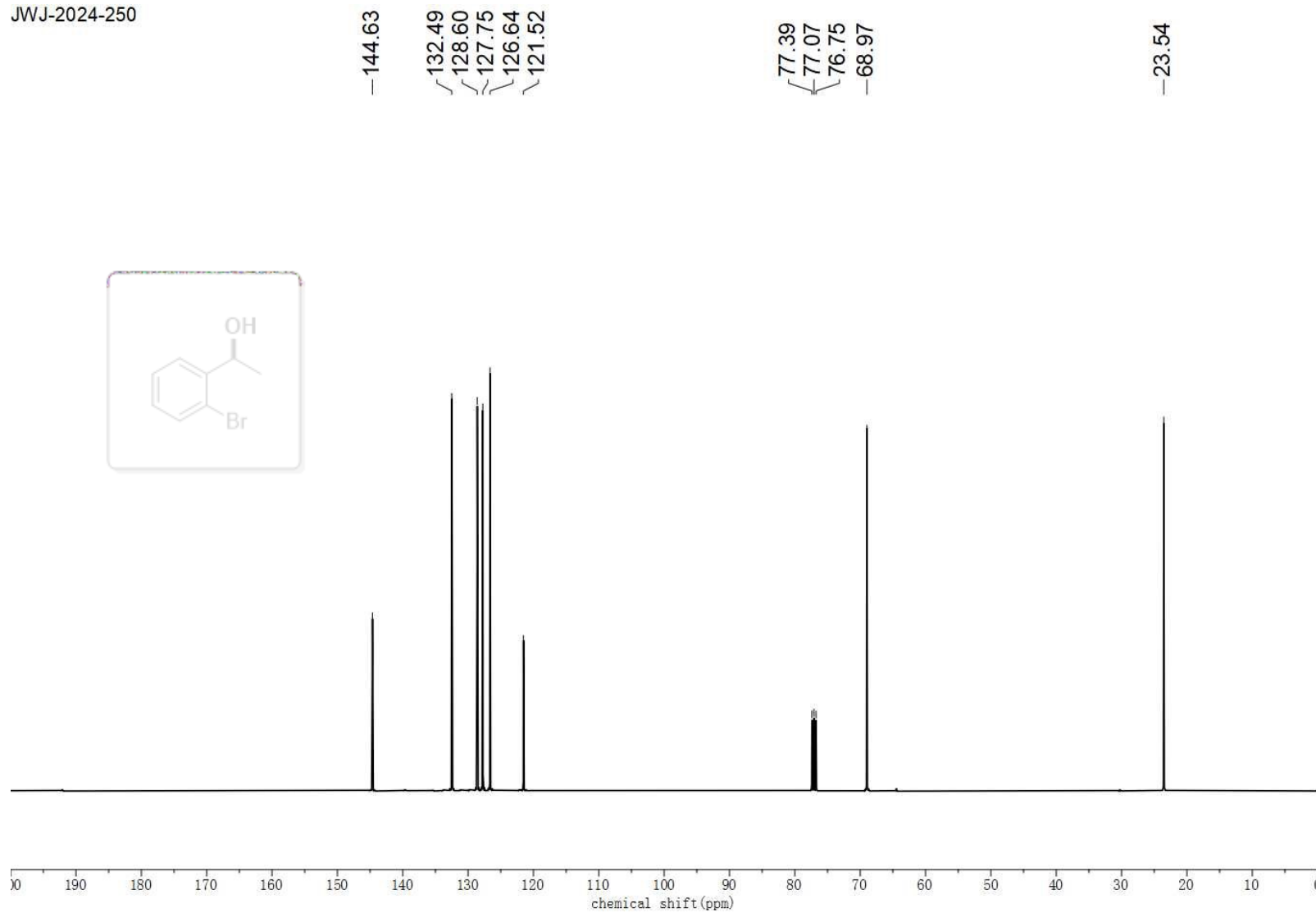
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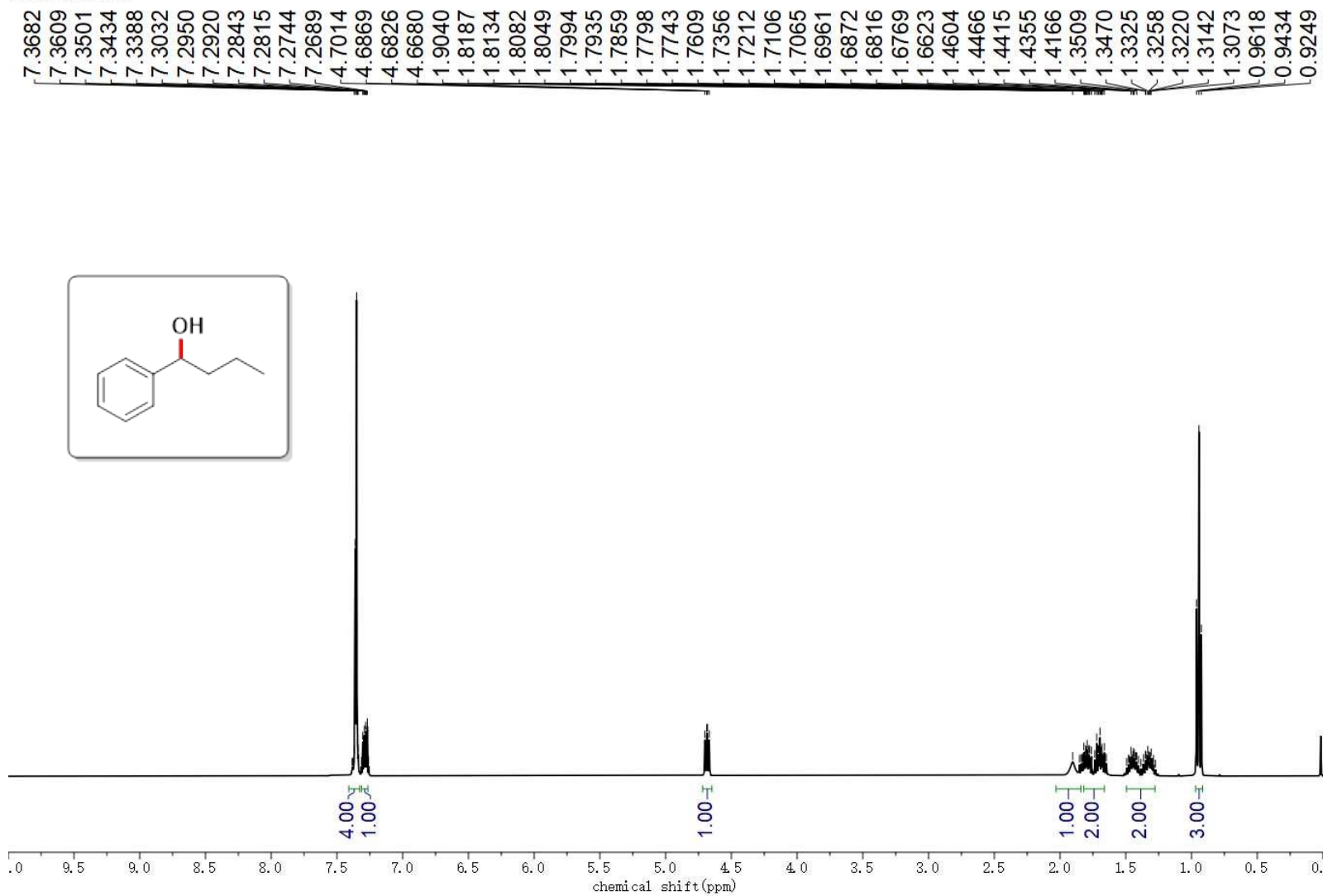


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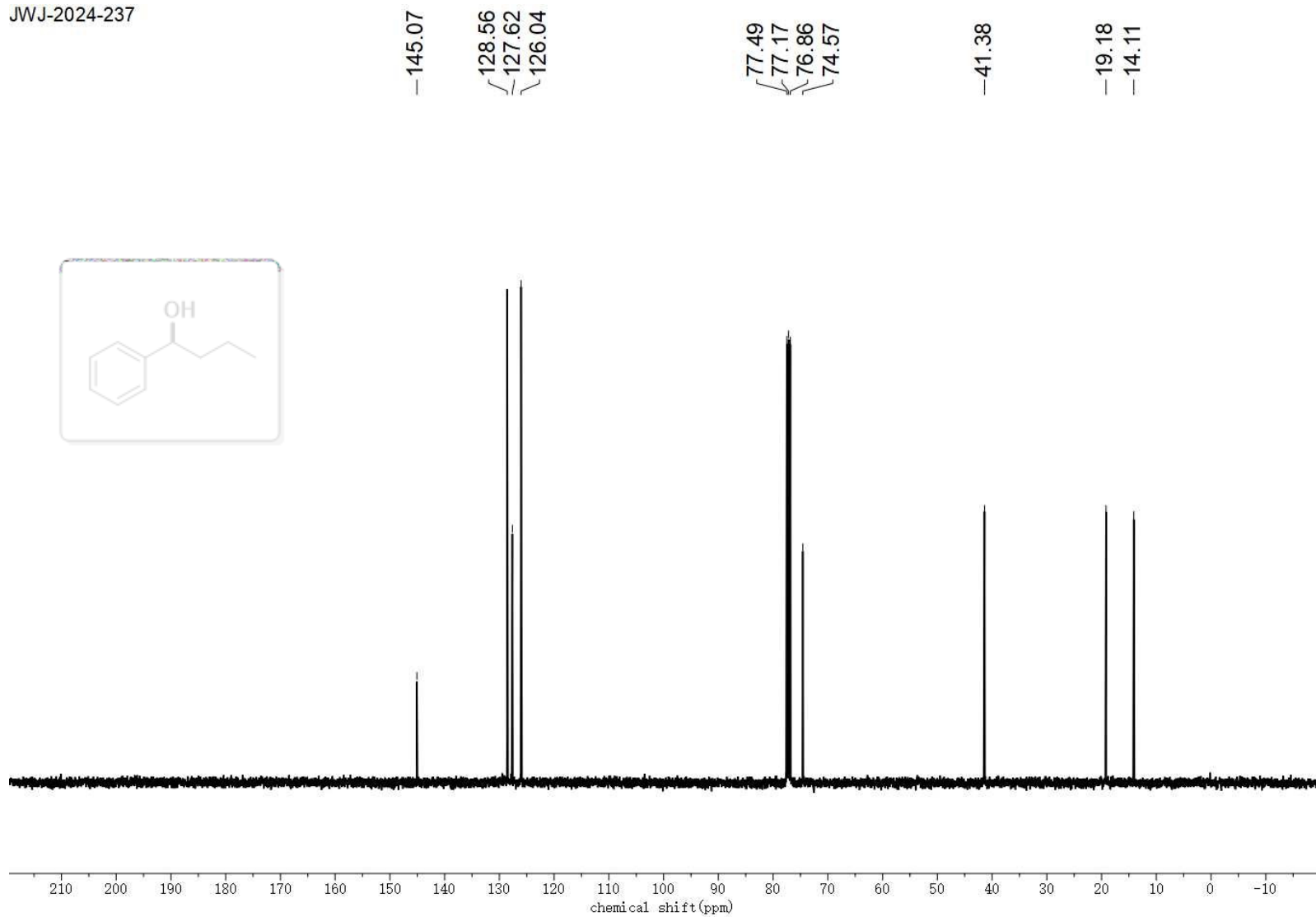




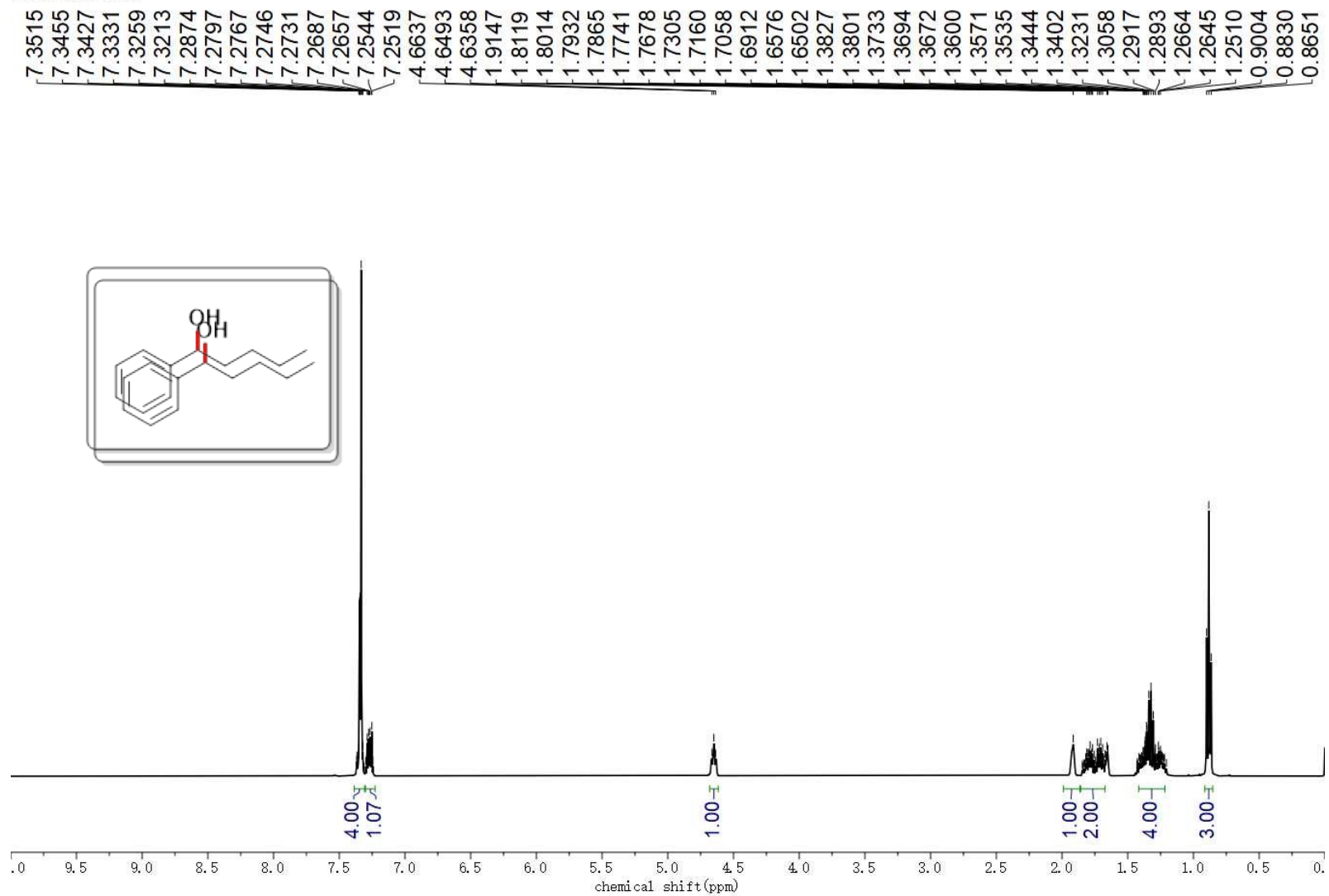
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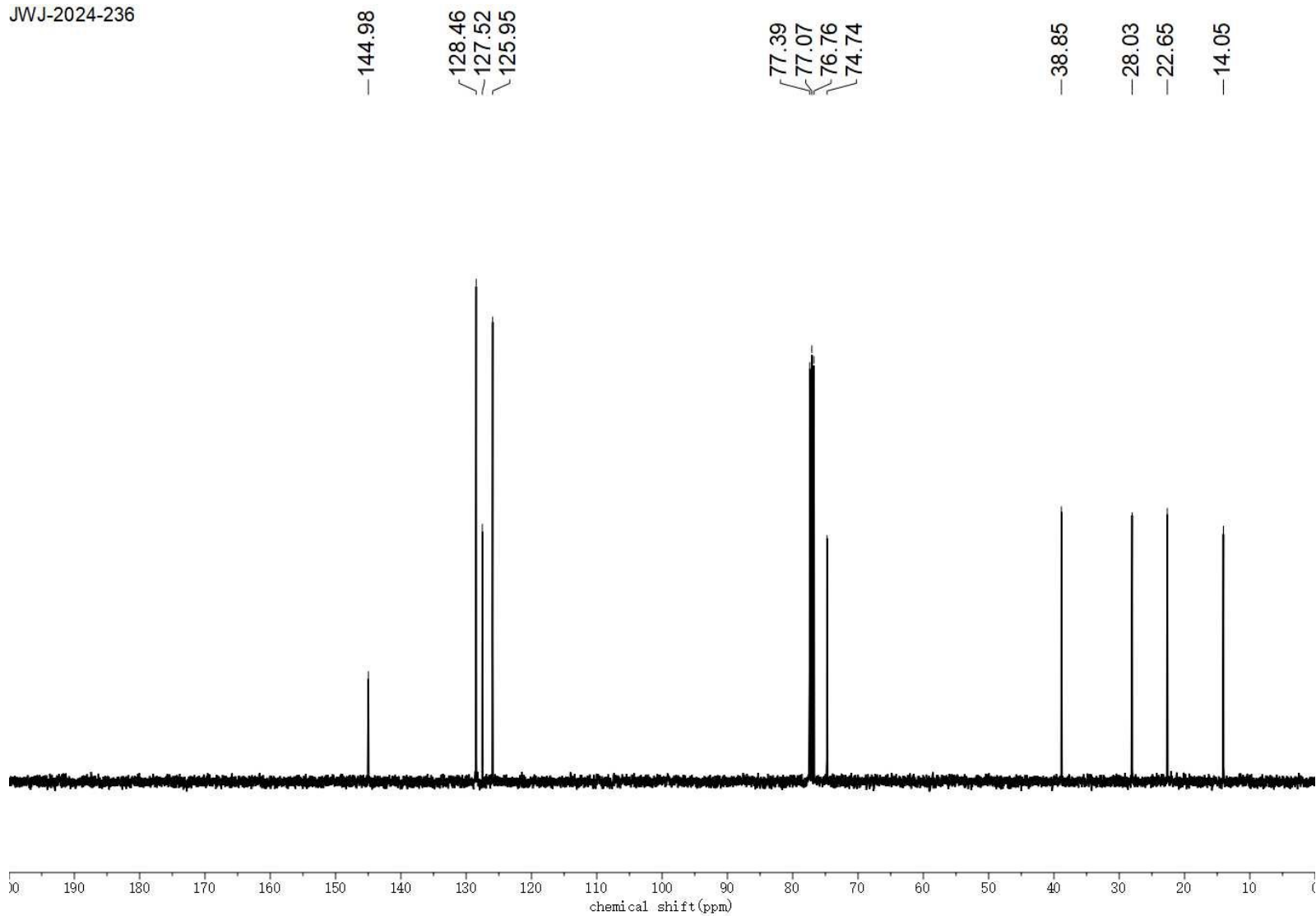
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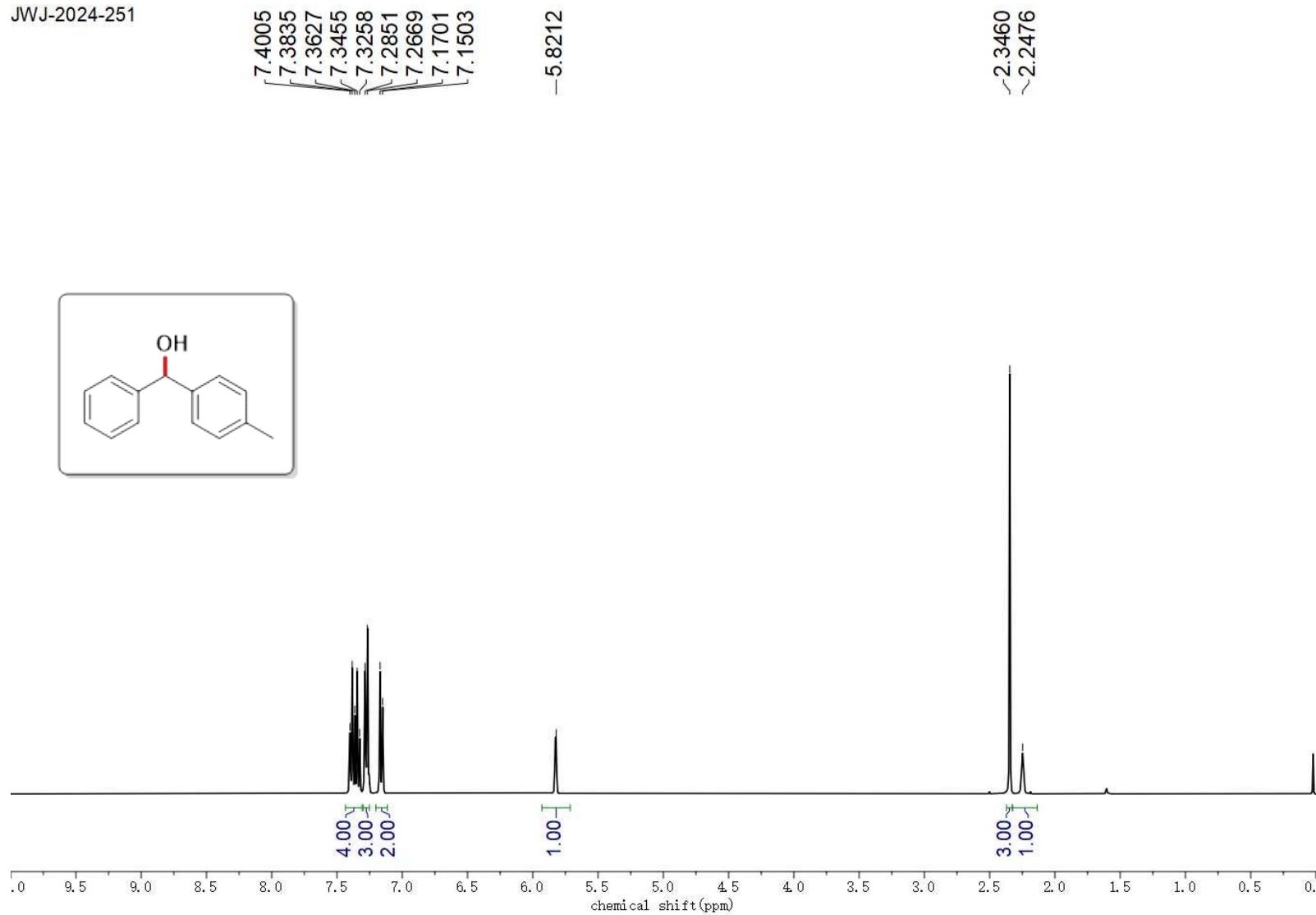
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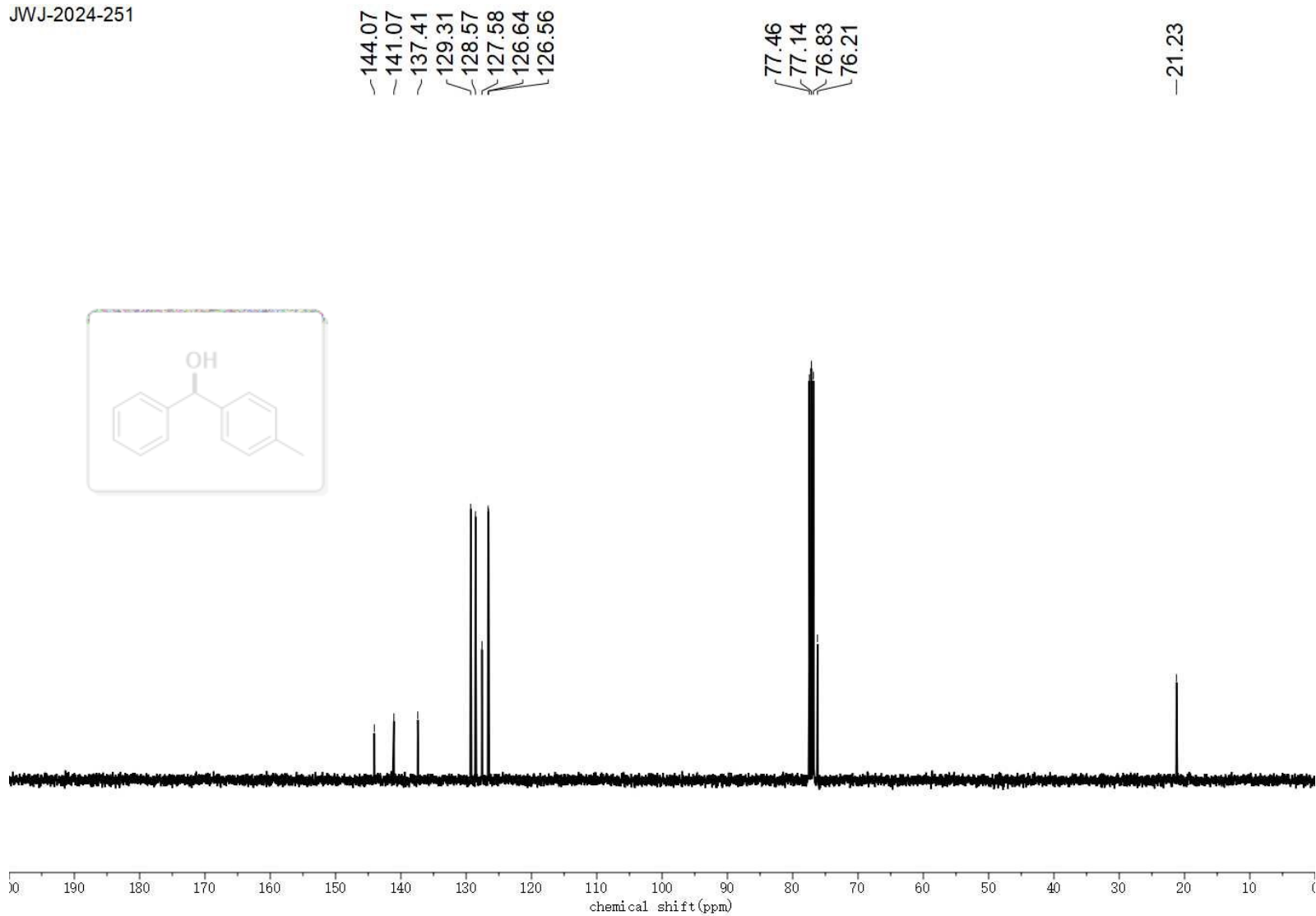
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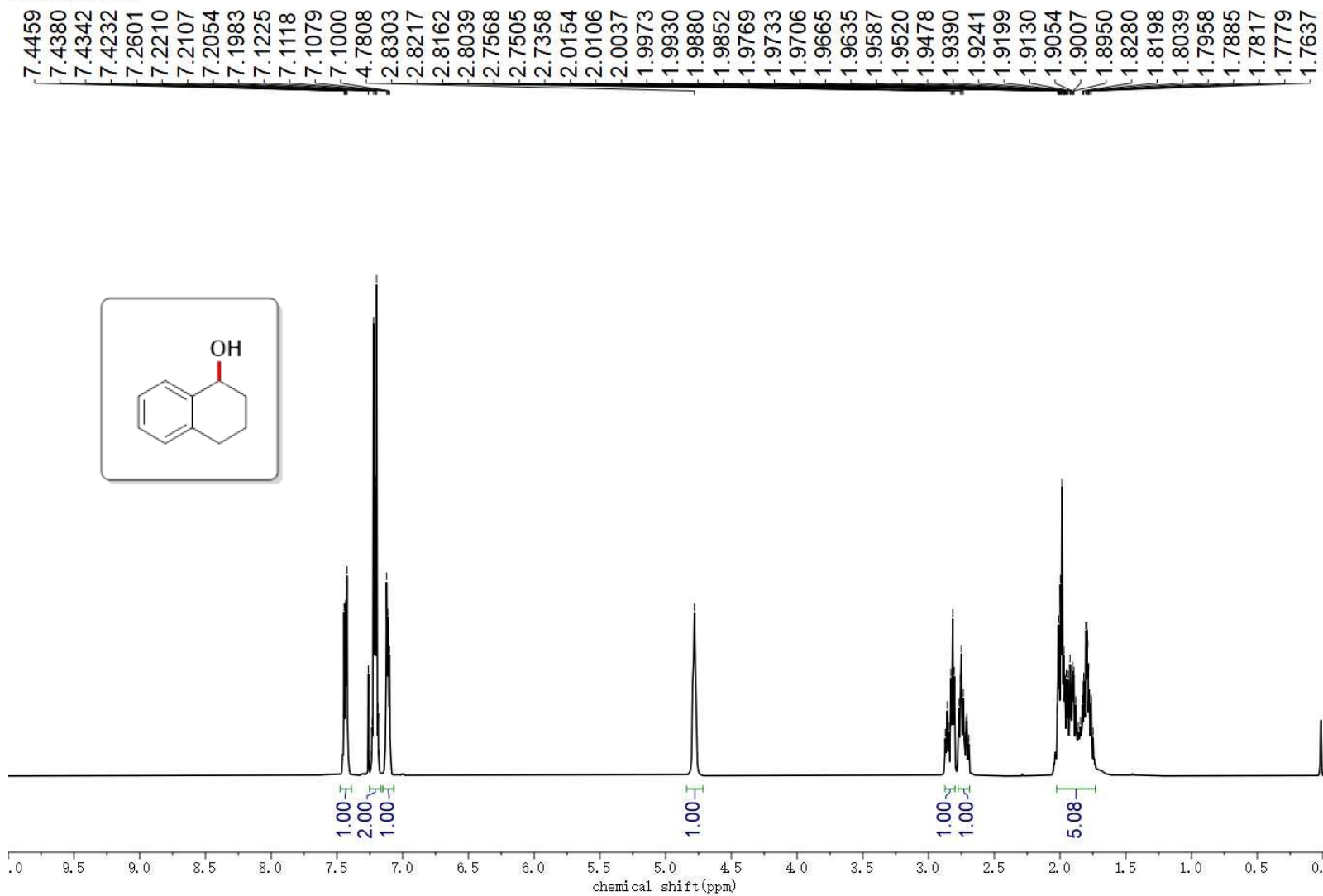
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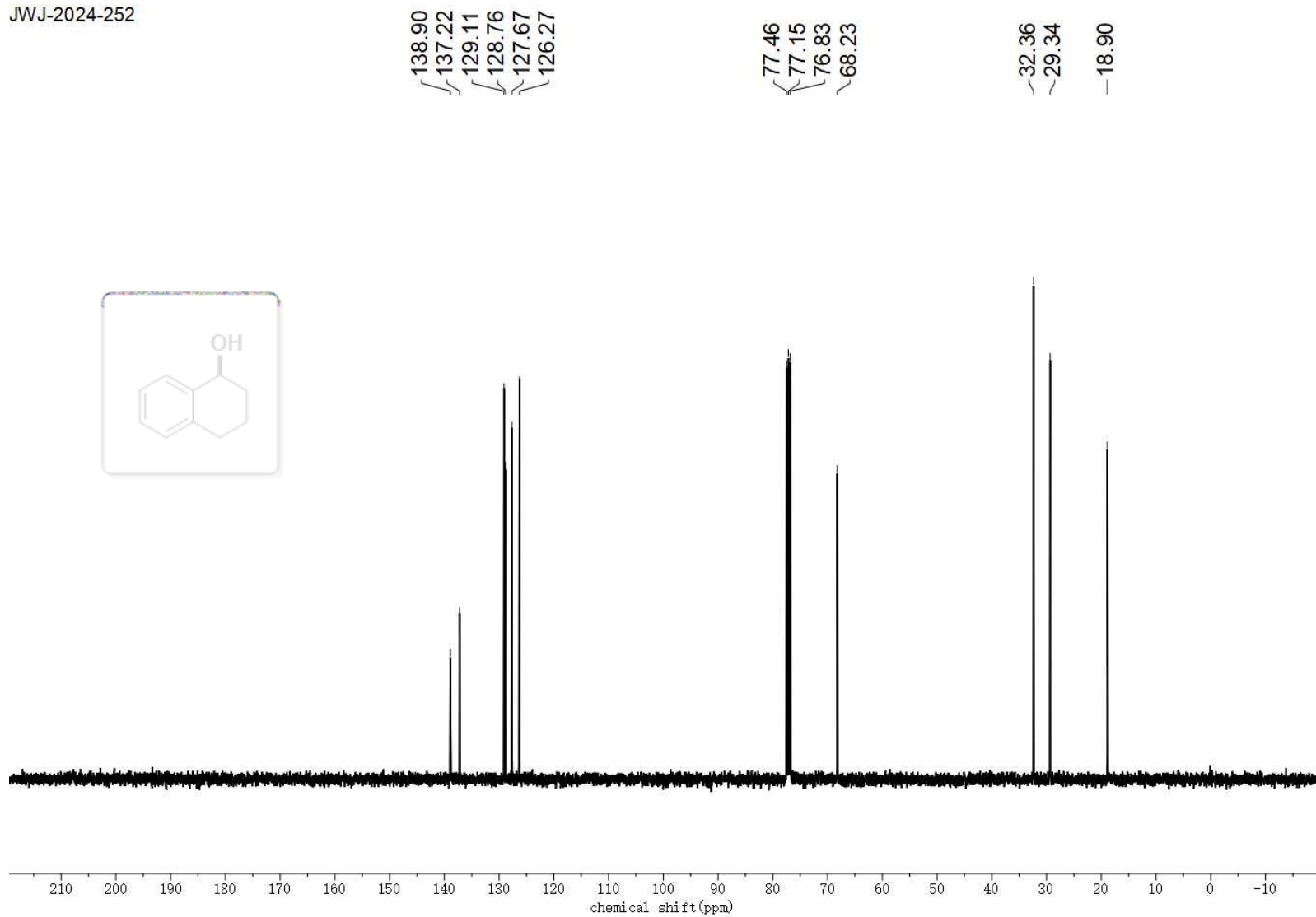
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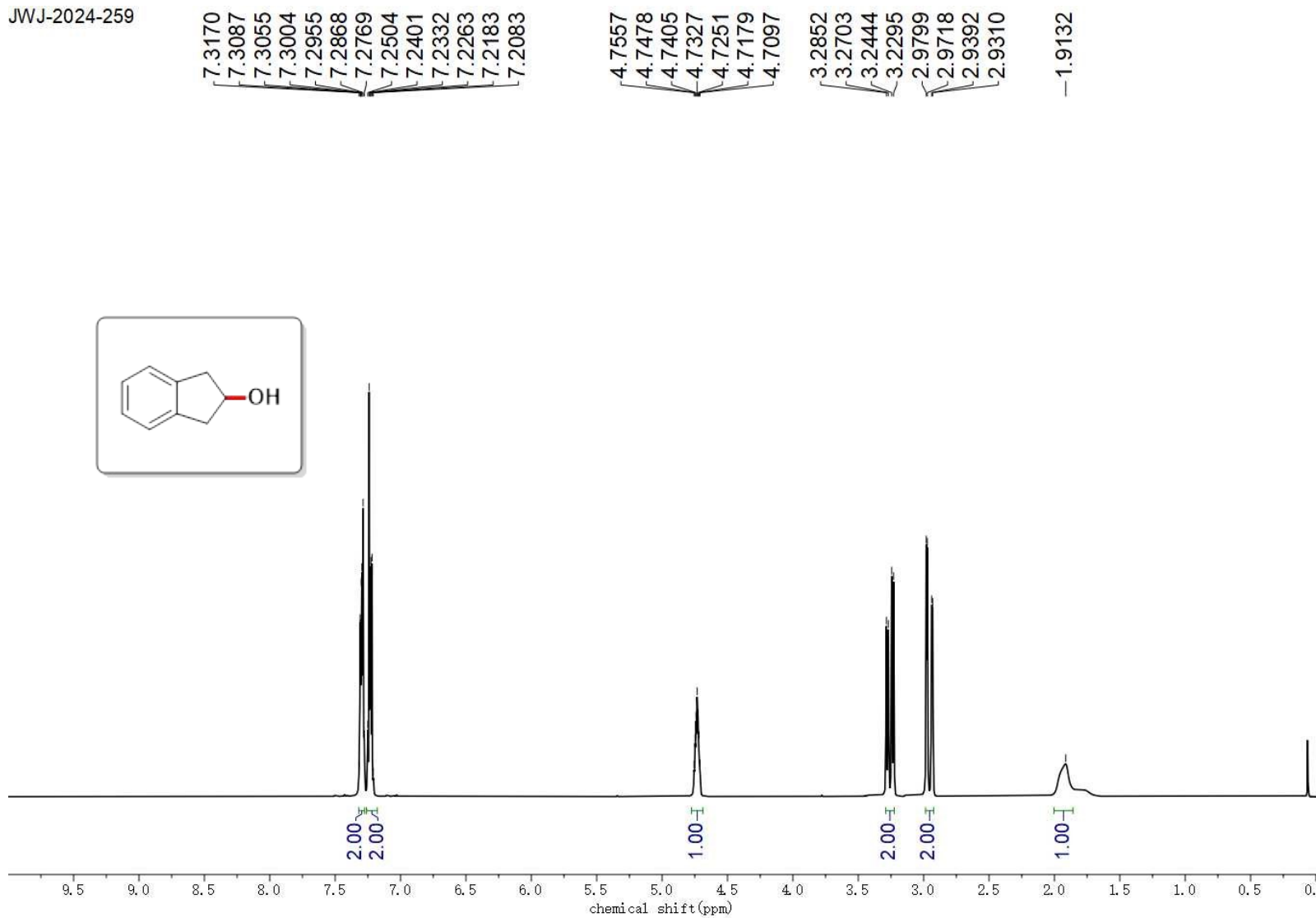


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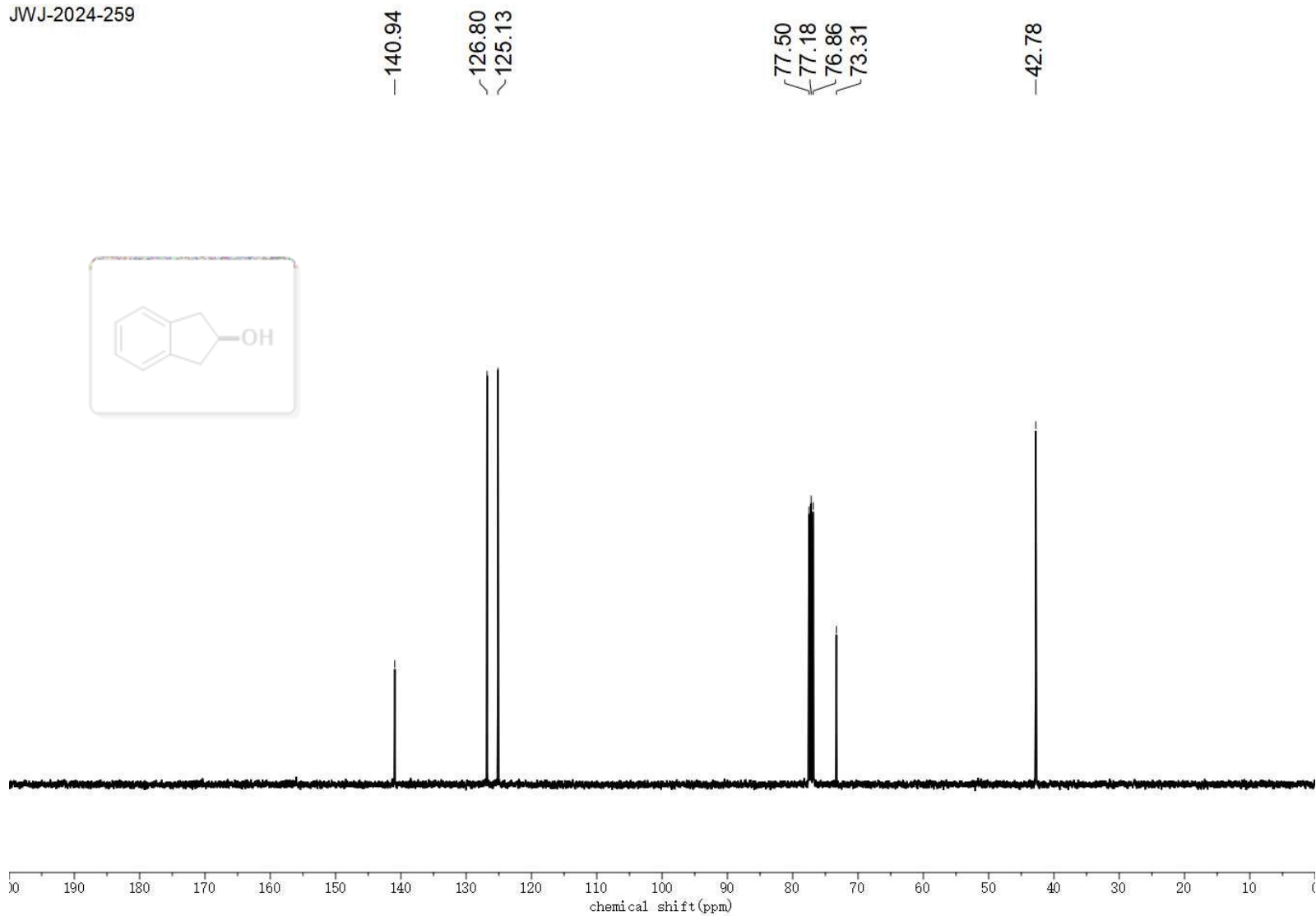




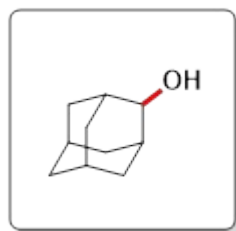
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JWJ-2024-259



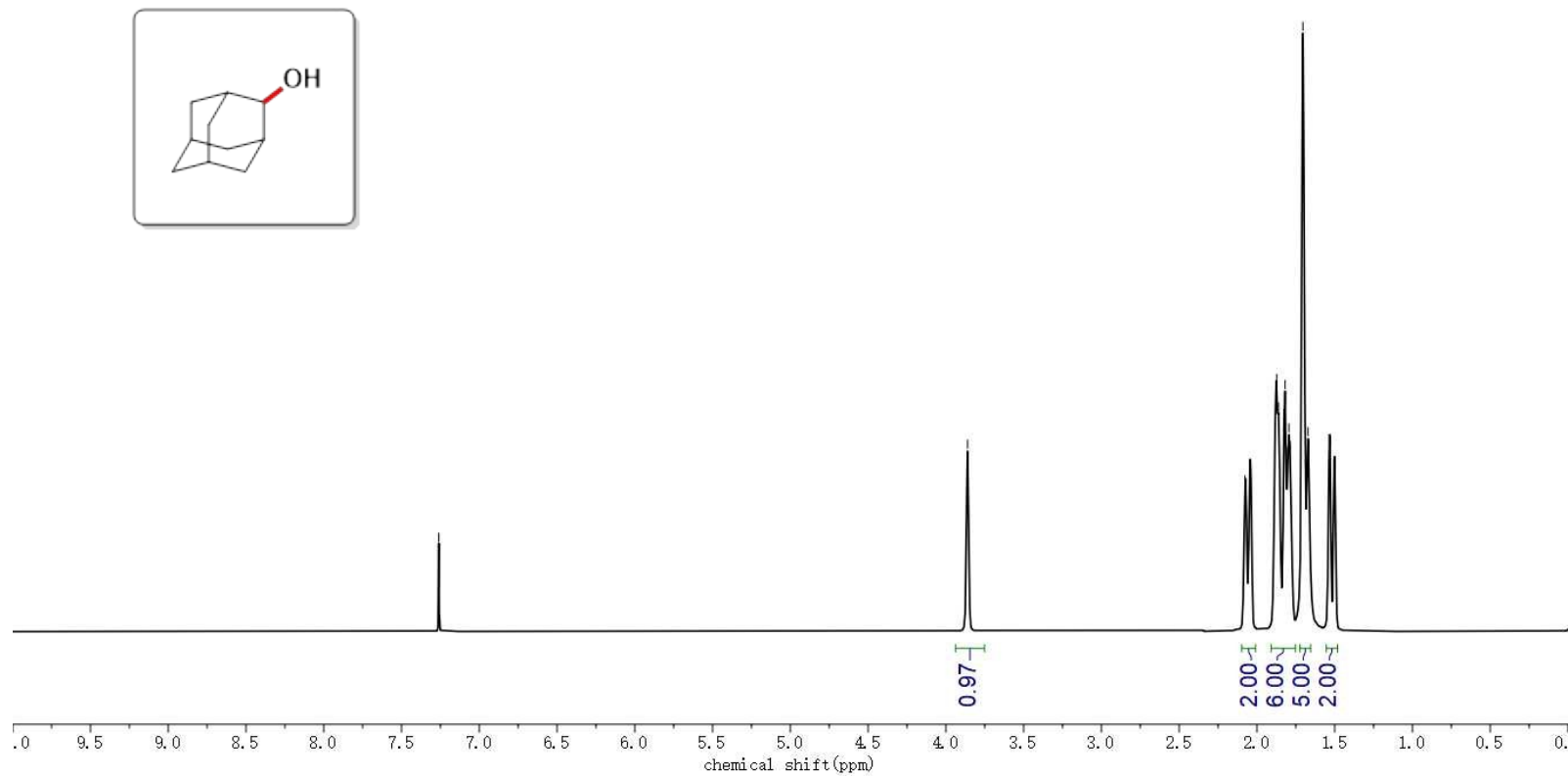
JWJ-2024-253



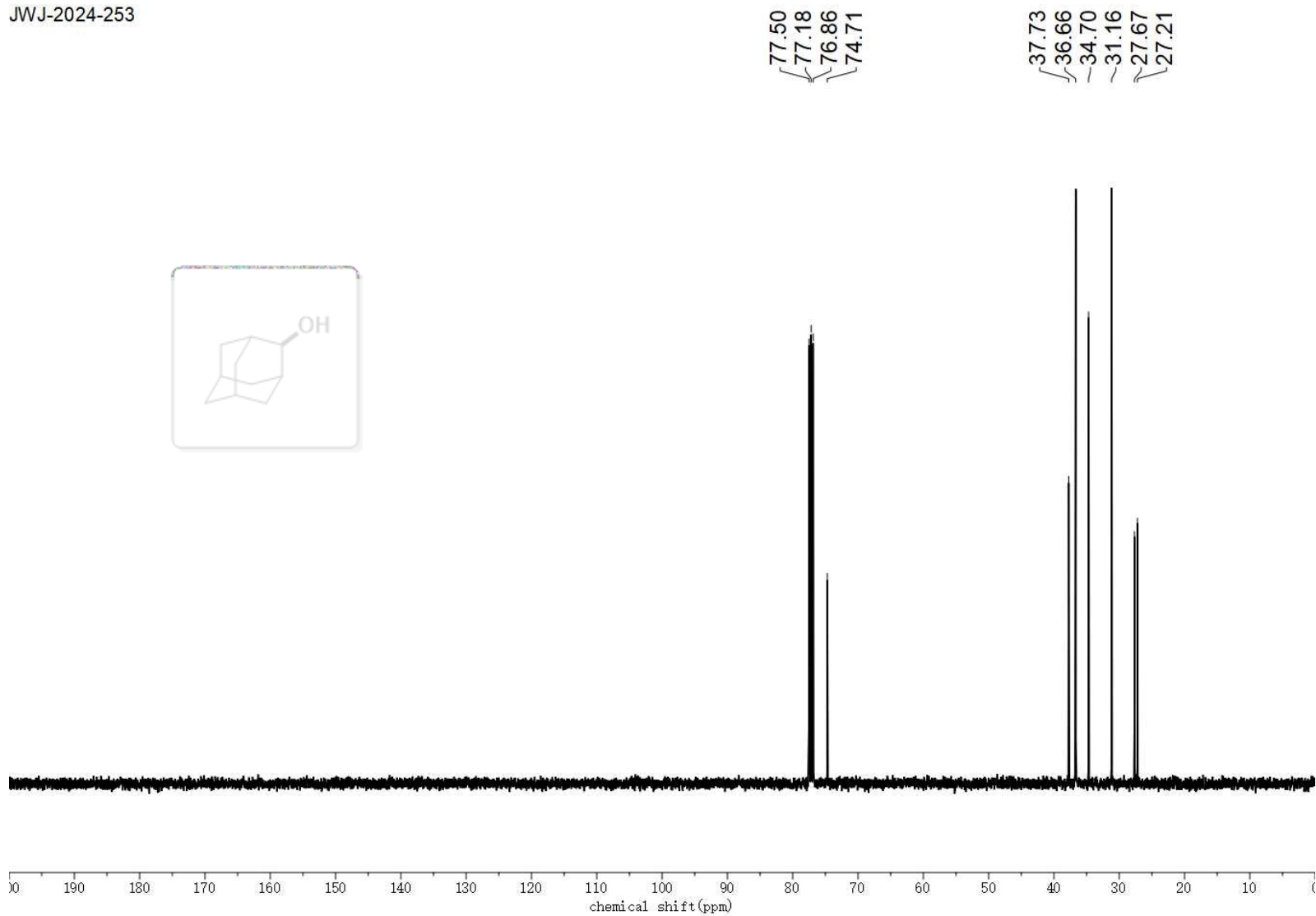
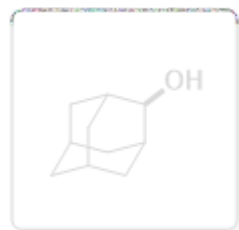
—7.2601

—3.8608

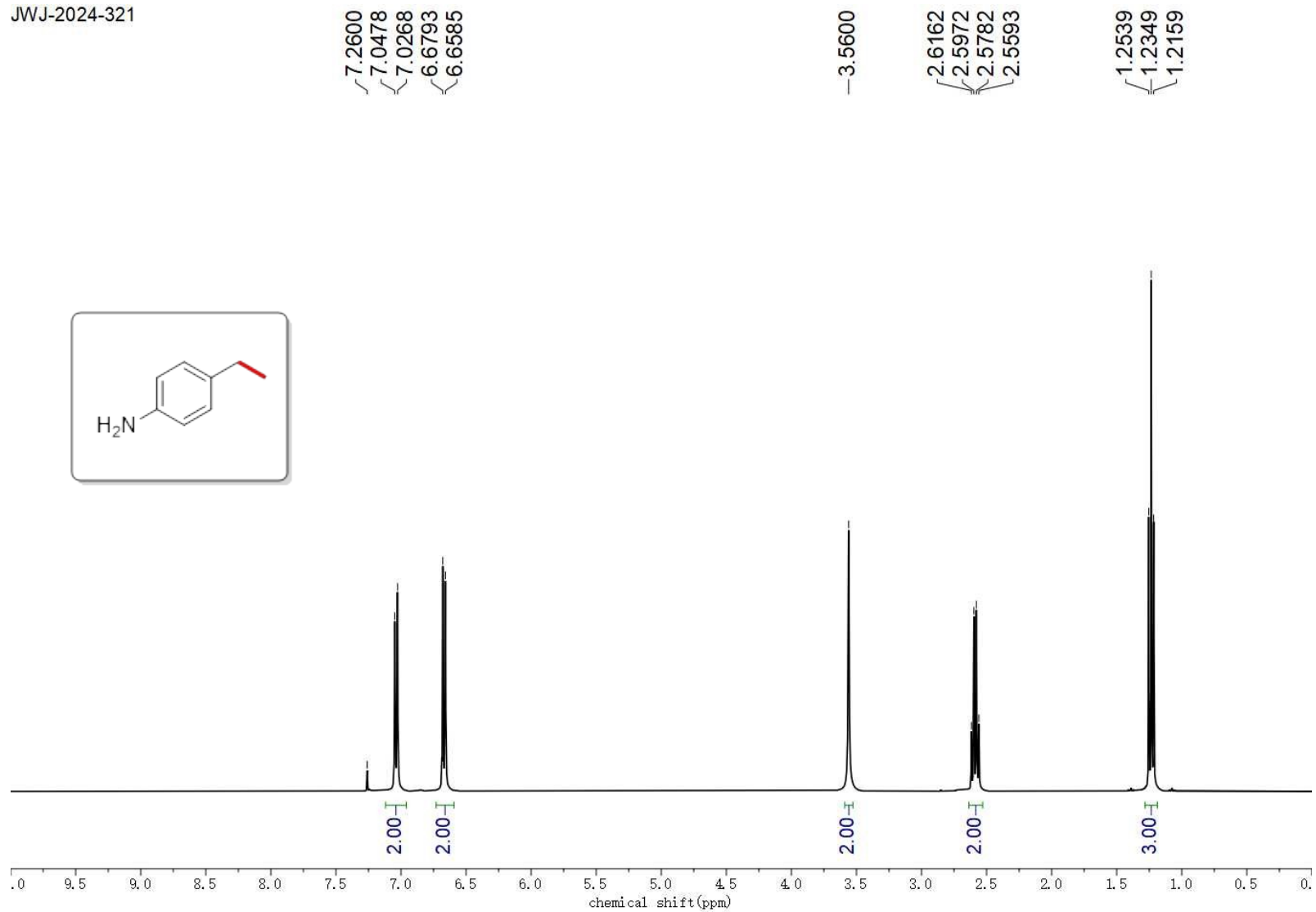
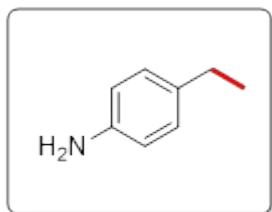
2.0773  
2.0459  
1.8731  
1.8610  
1.8262  
1.8190  
1.8012  
1.7938  
1.7868  
1.7044  
1.6719  
1.5298  
1.5045



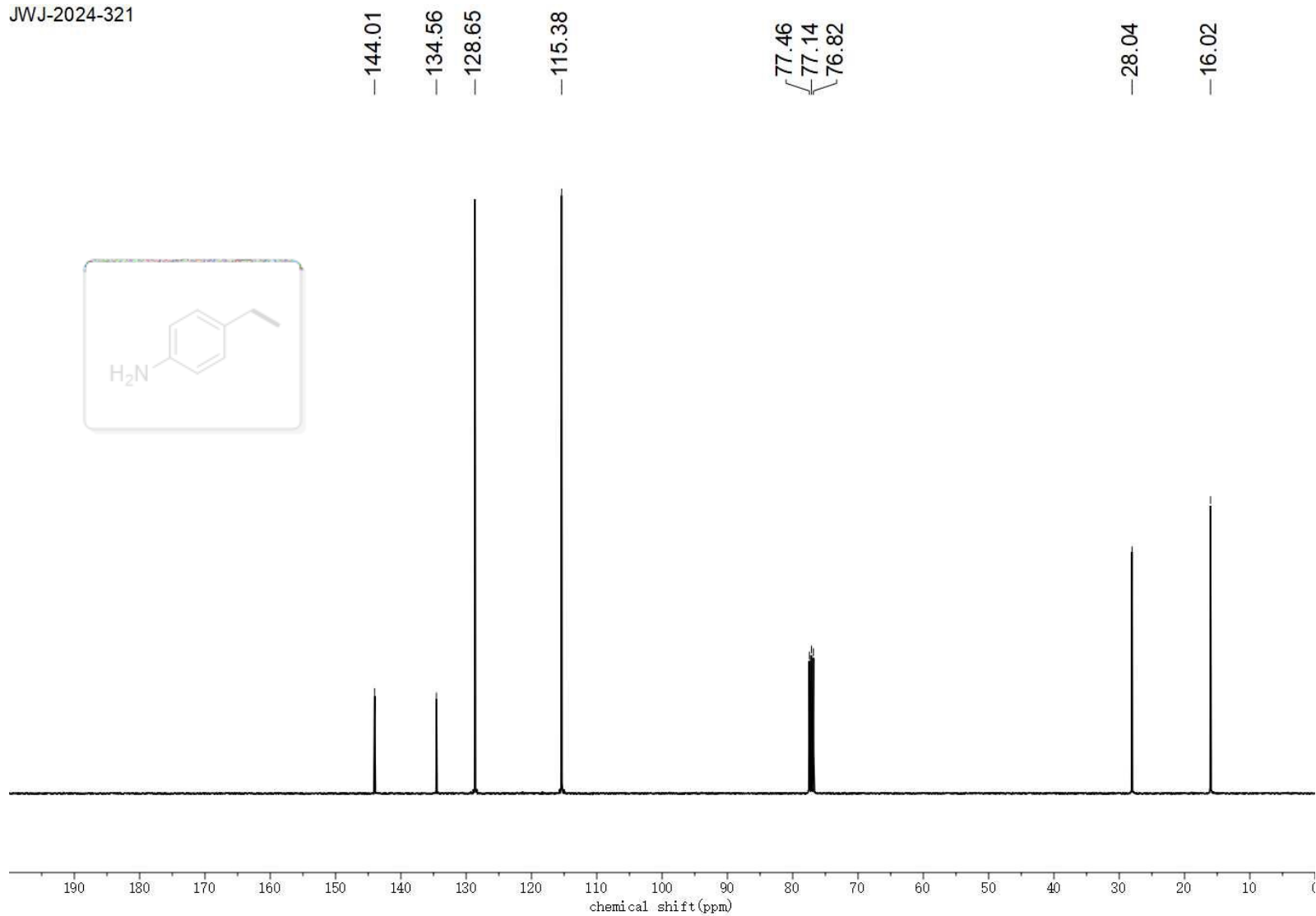
JWJ-2024-253



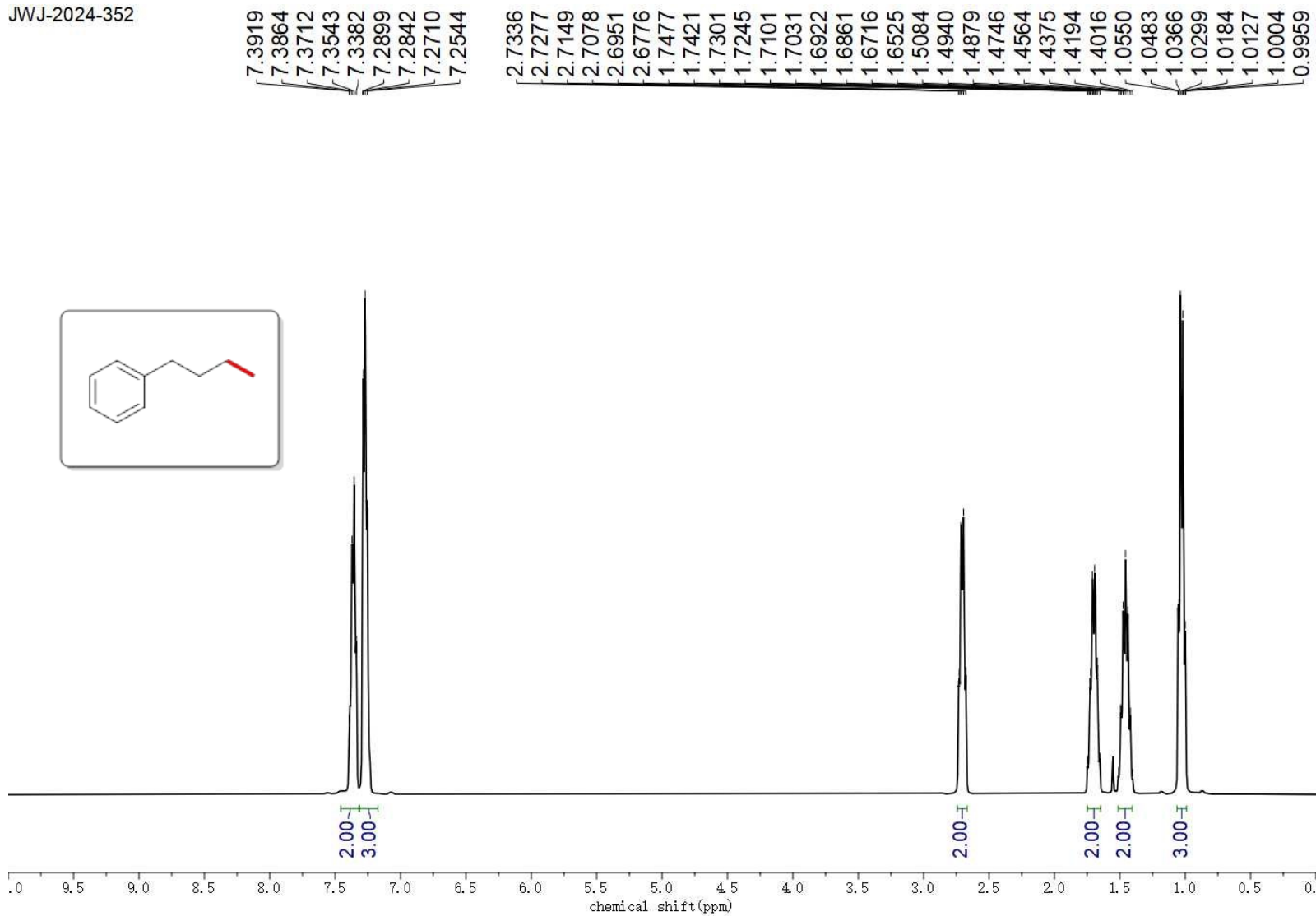
JWJ-2024-321



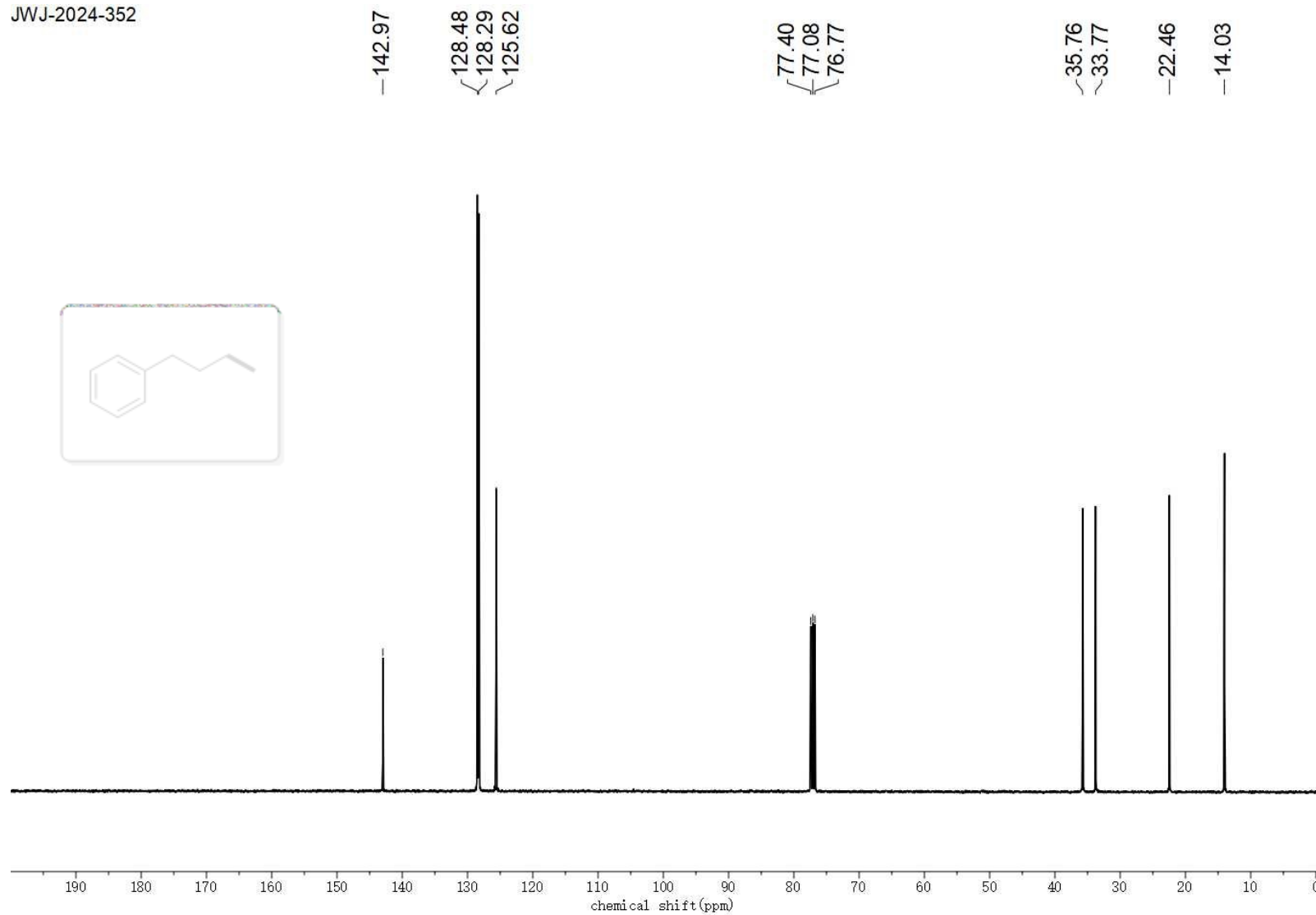
JWJ-2024-321



JWJ-2024-352

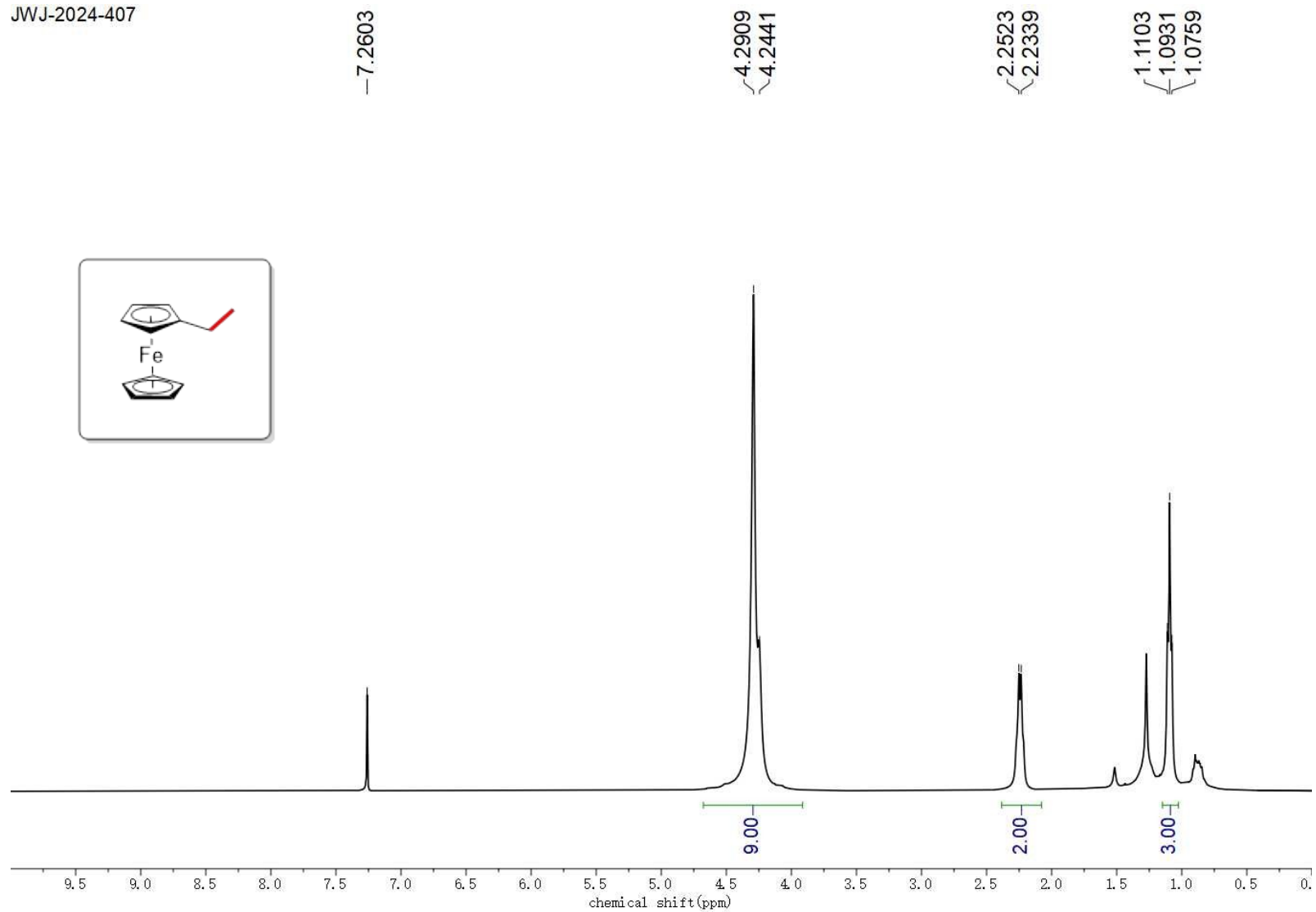


JWJ-2024-352

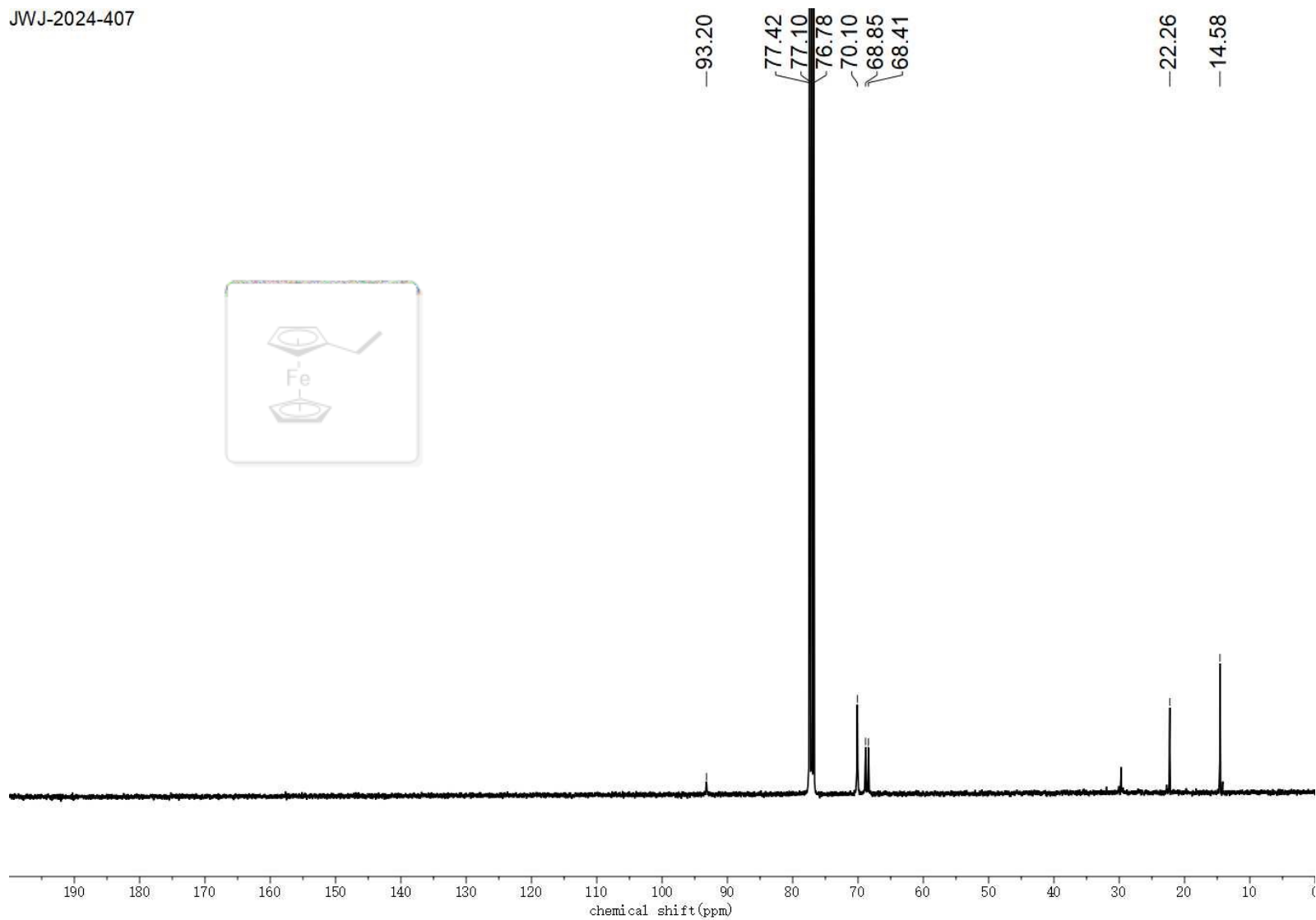




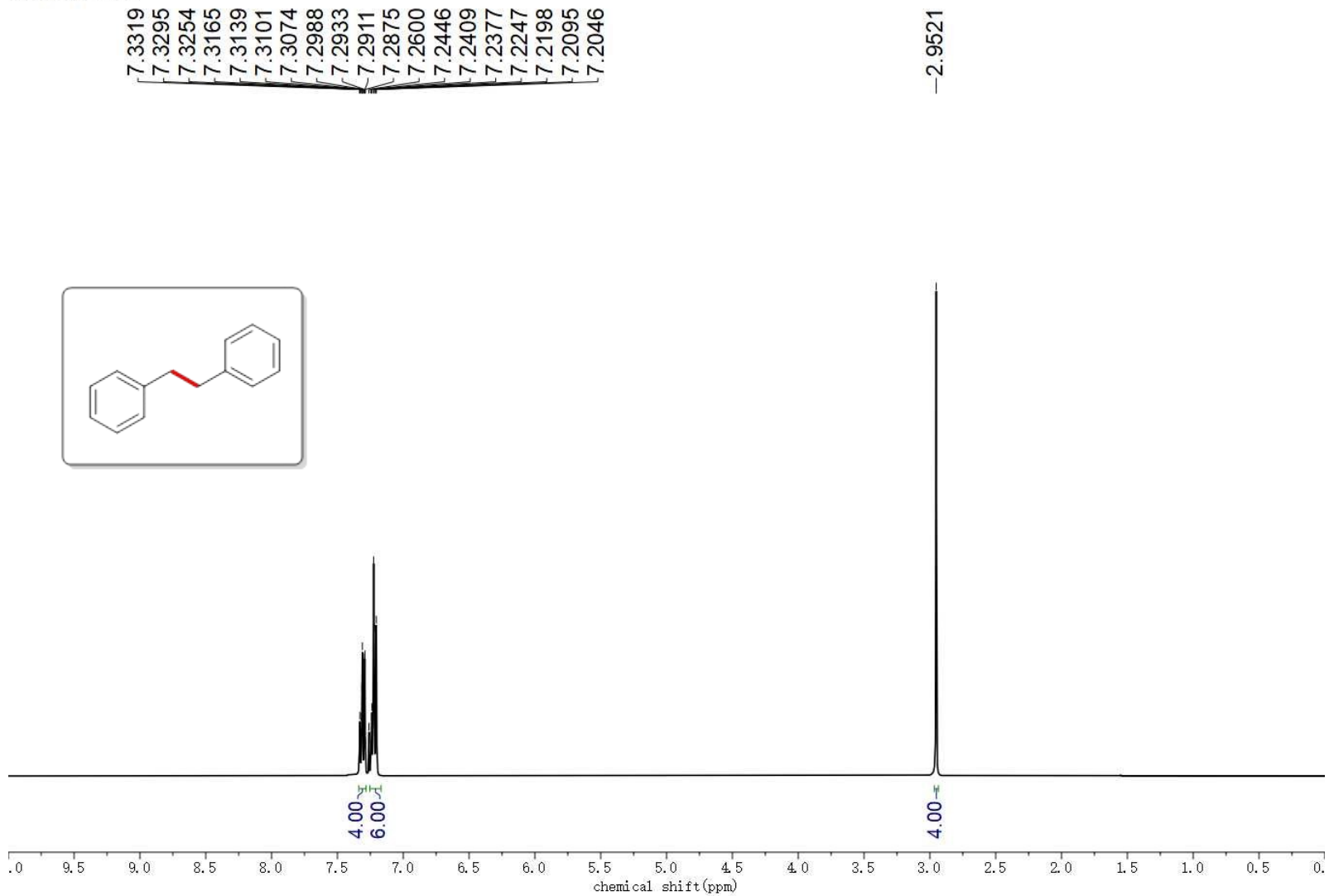
JWJ-2024-407



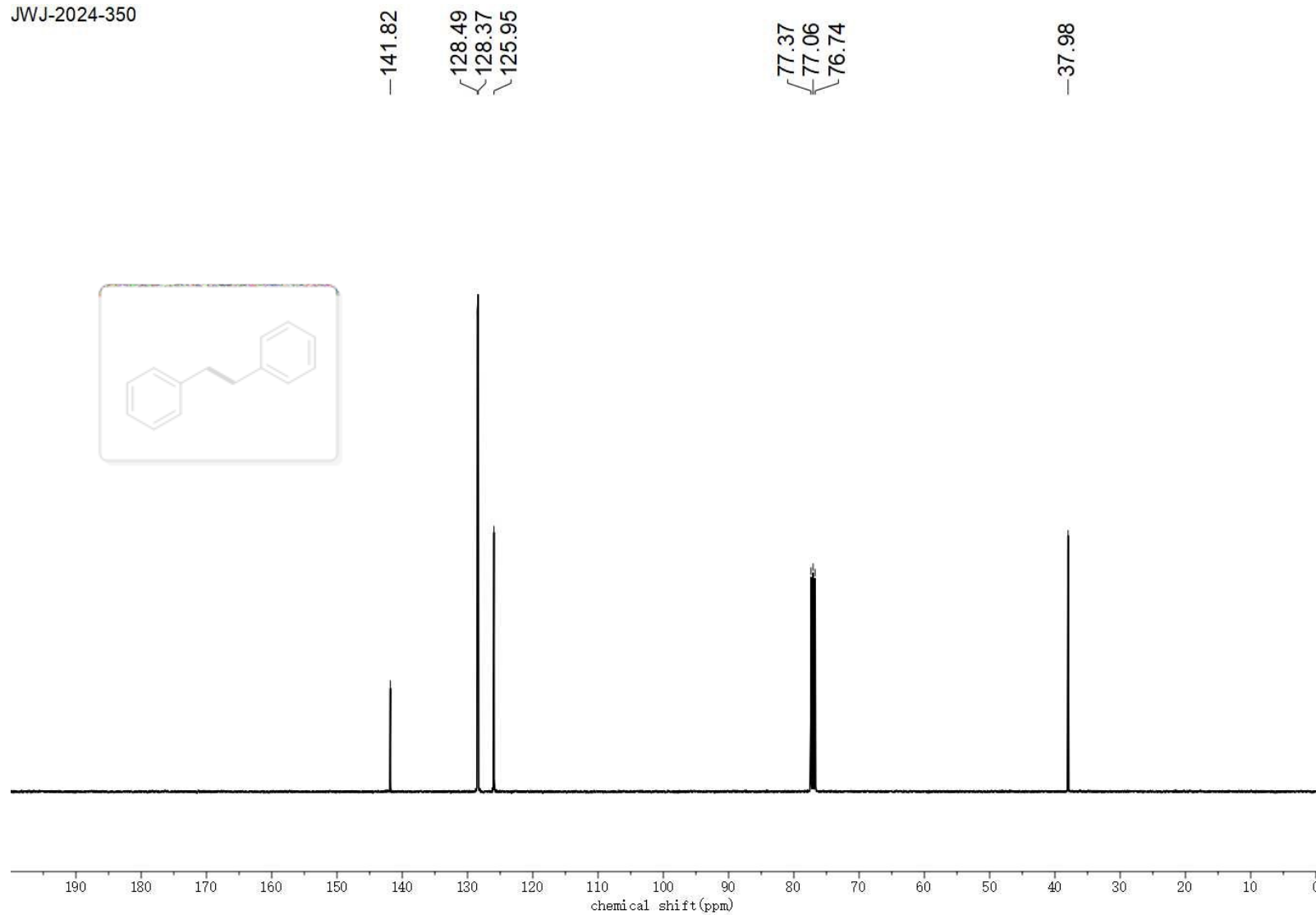
JWJ-2024-407



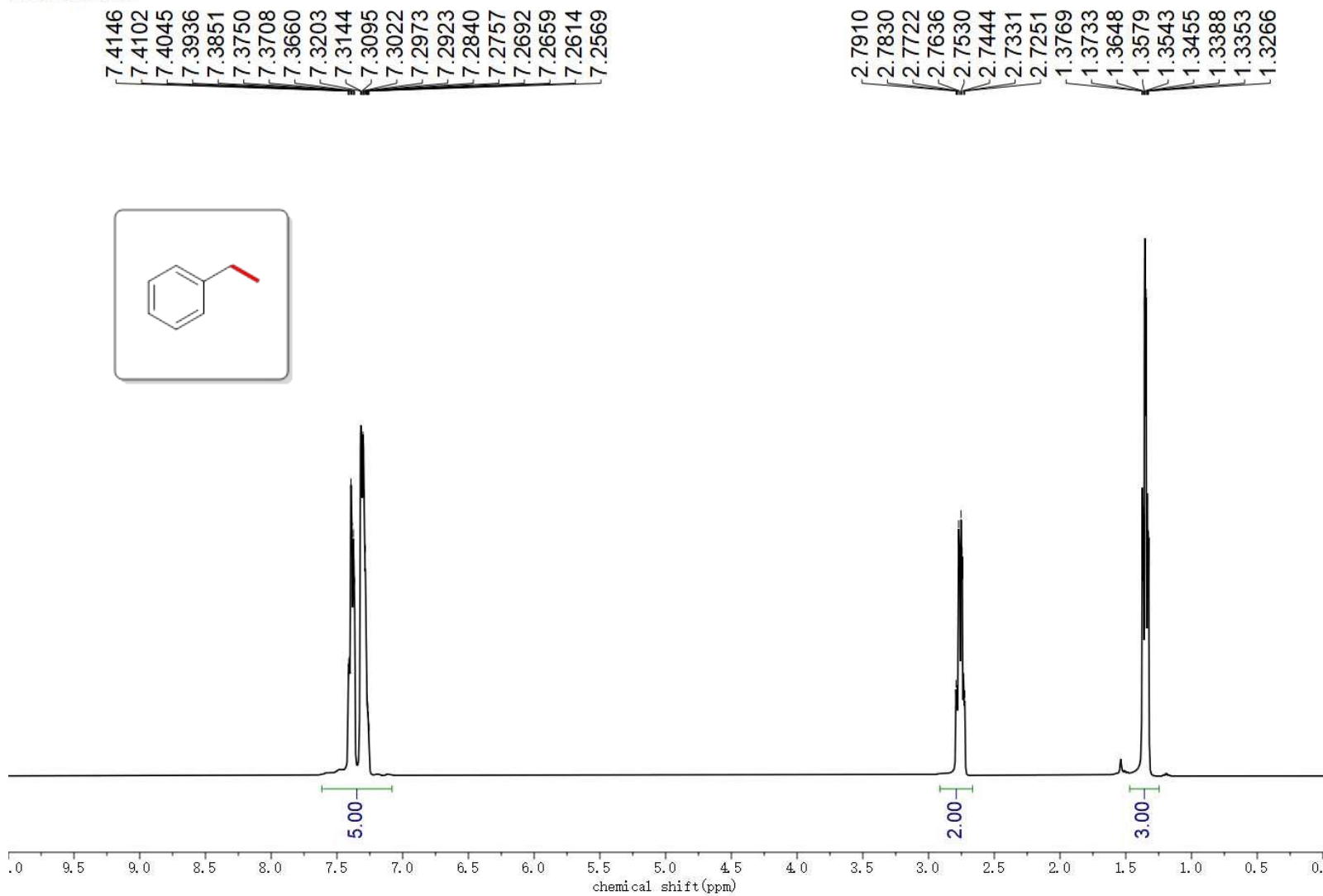
JWJ-2024-350



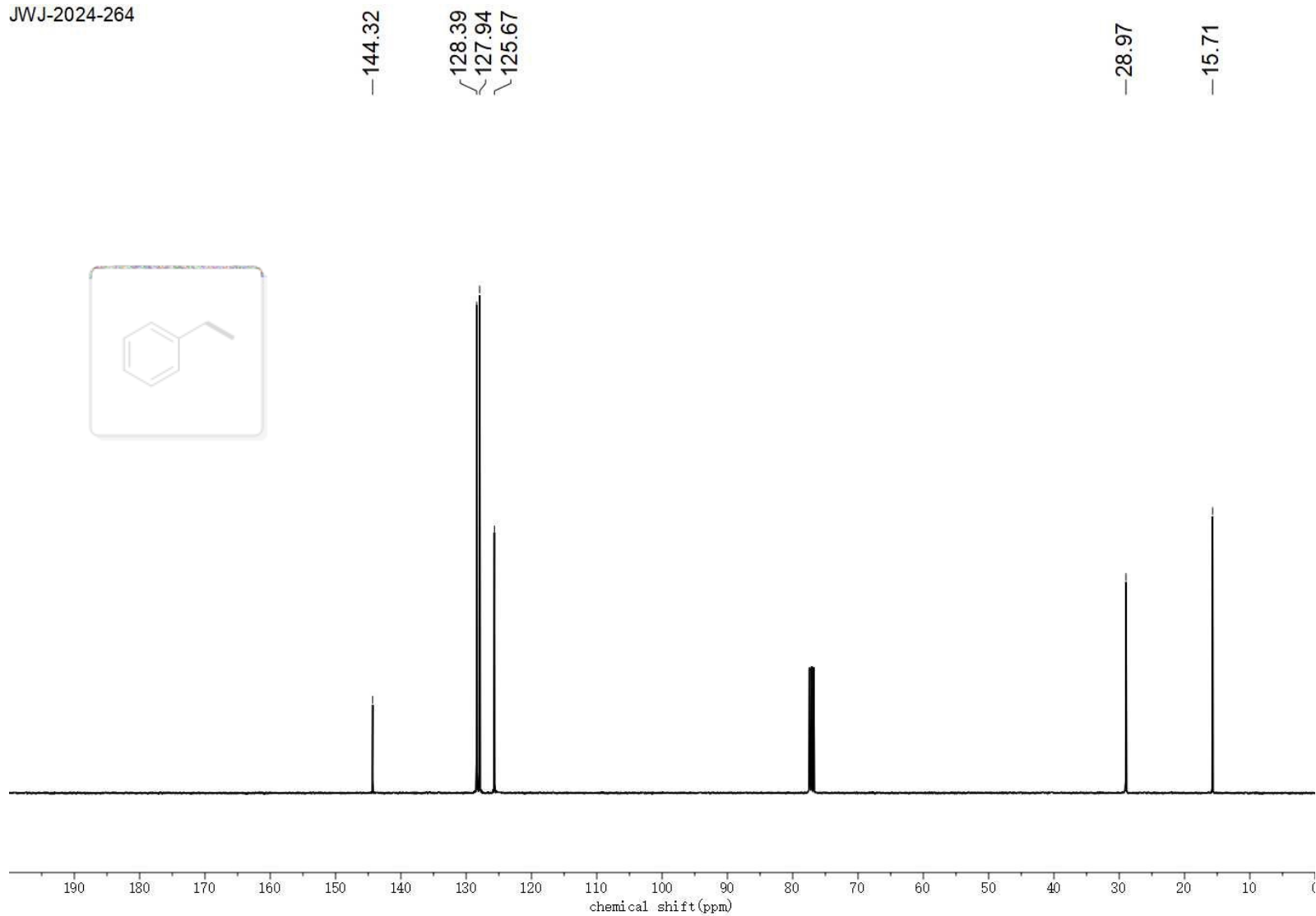
JWJ-2024-350



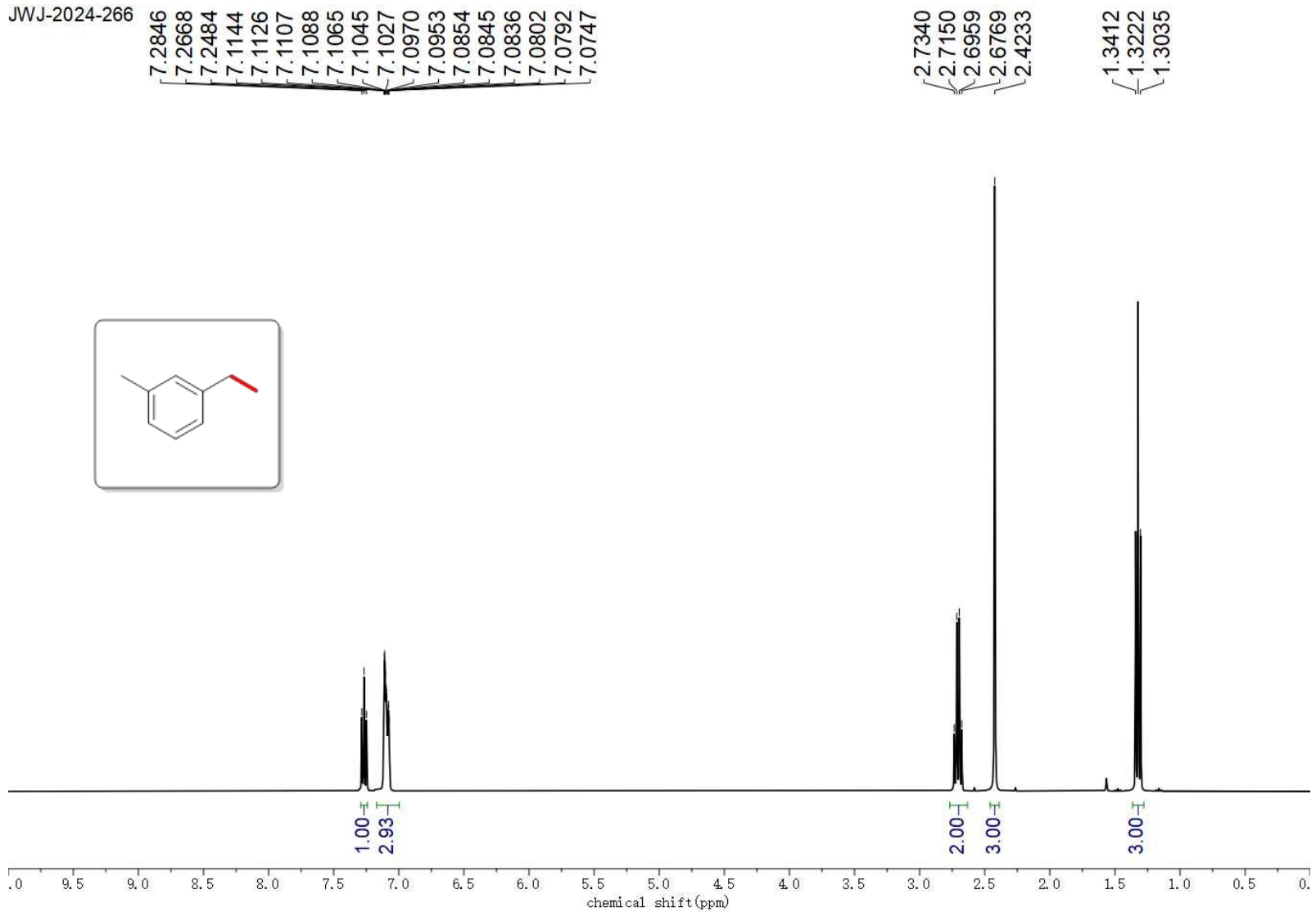
JWJ-2024-264



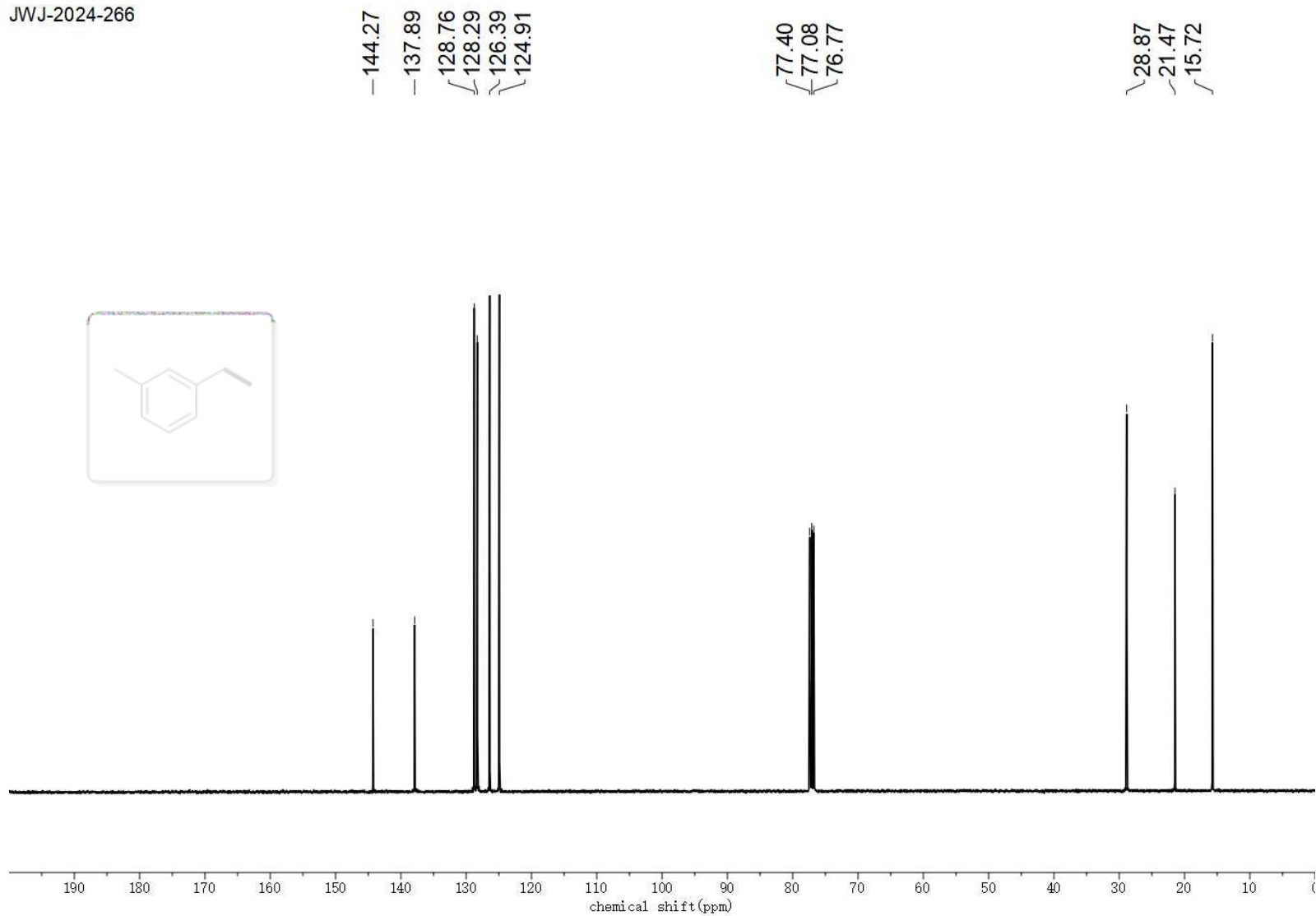
JWJ-2024-264



JWJ-2024-266

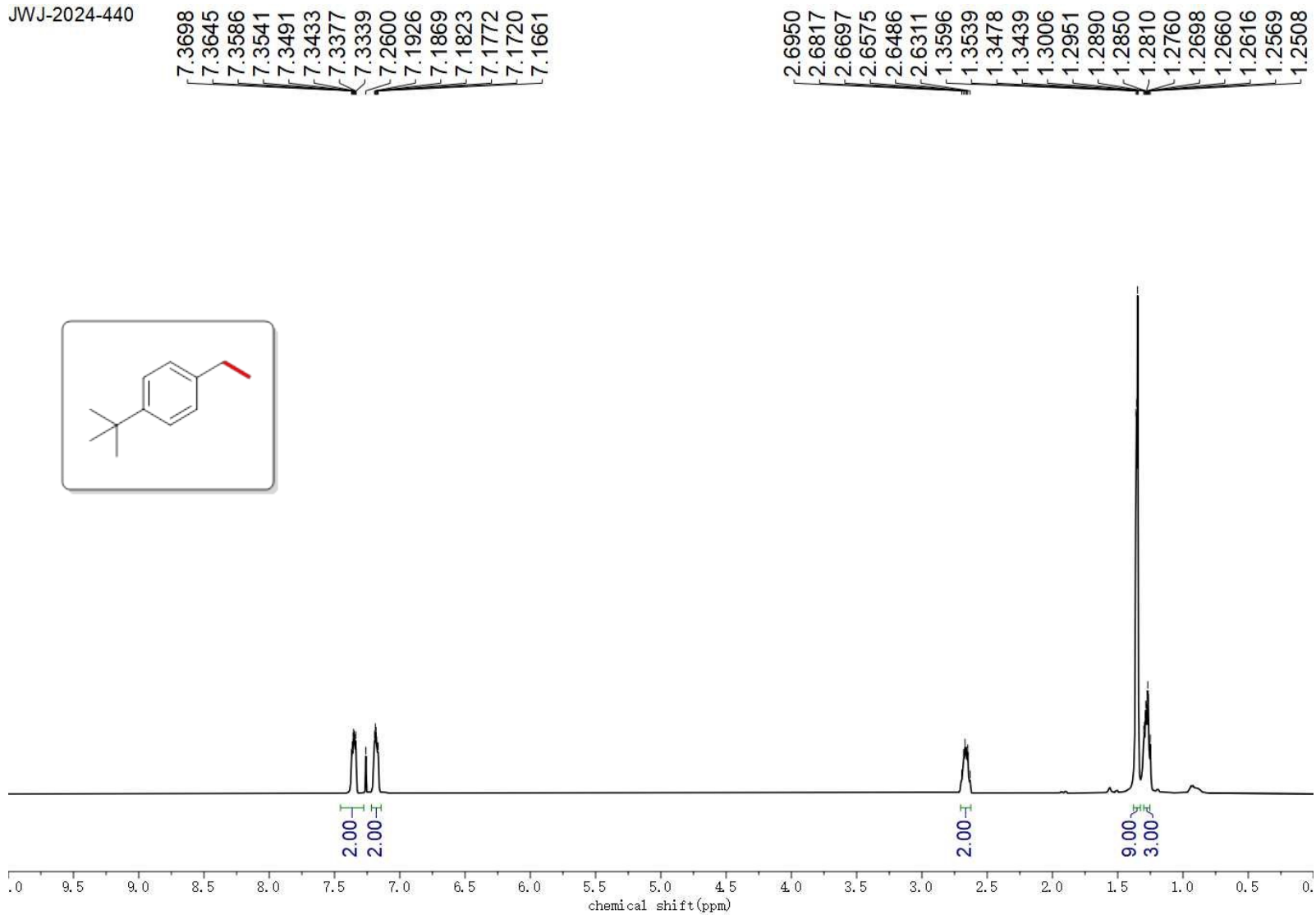
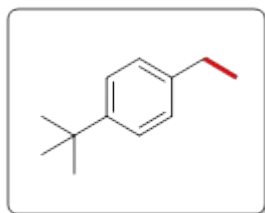


JWJ-2024-266

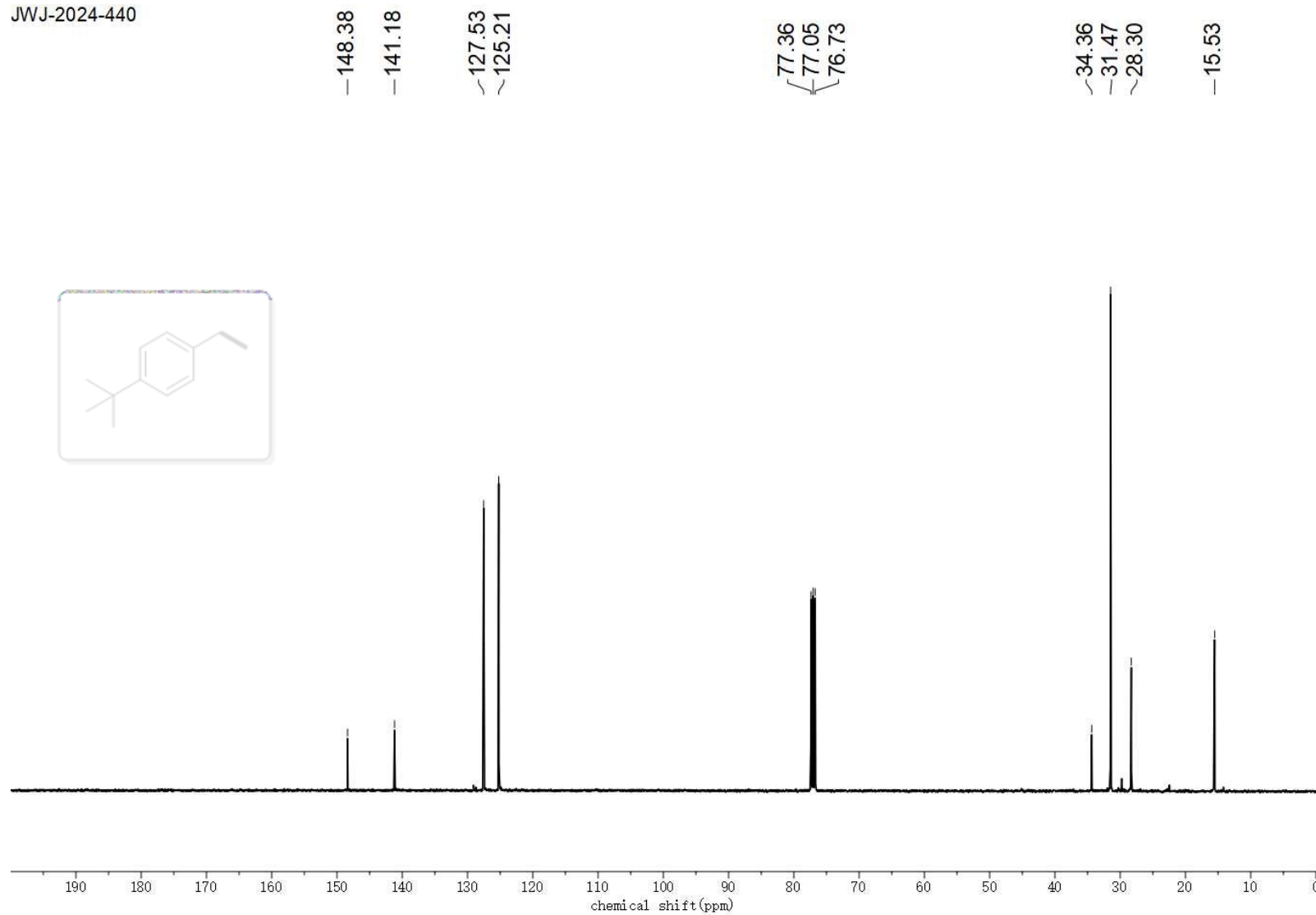




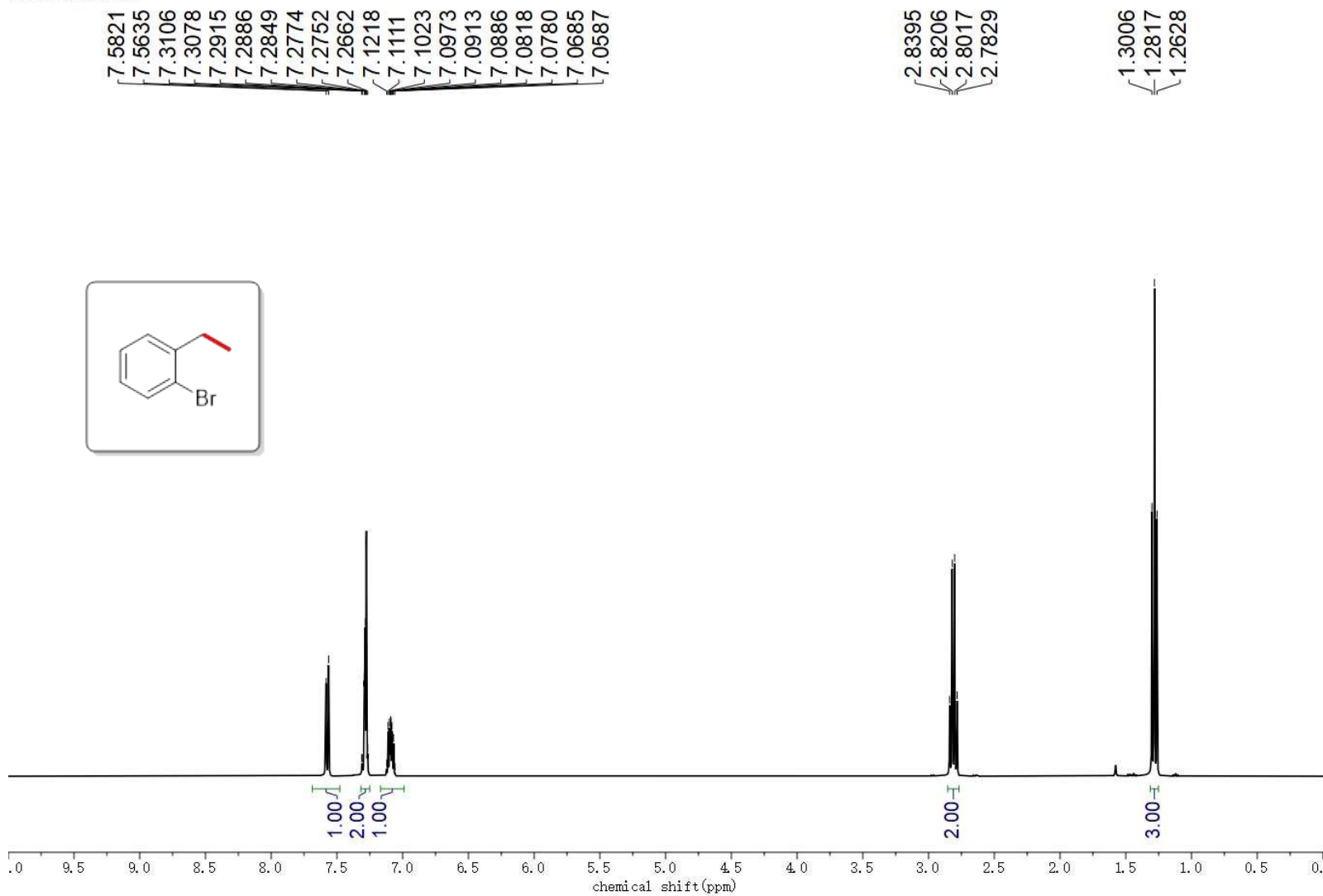
JWJ-2024-440



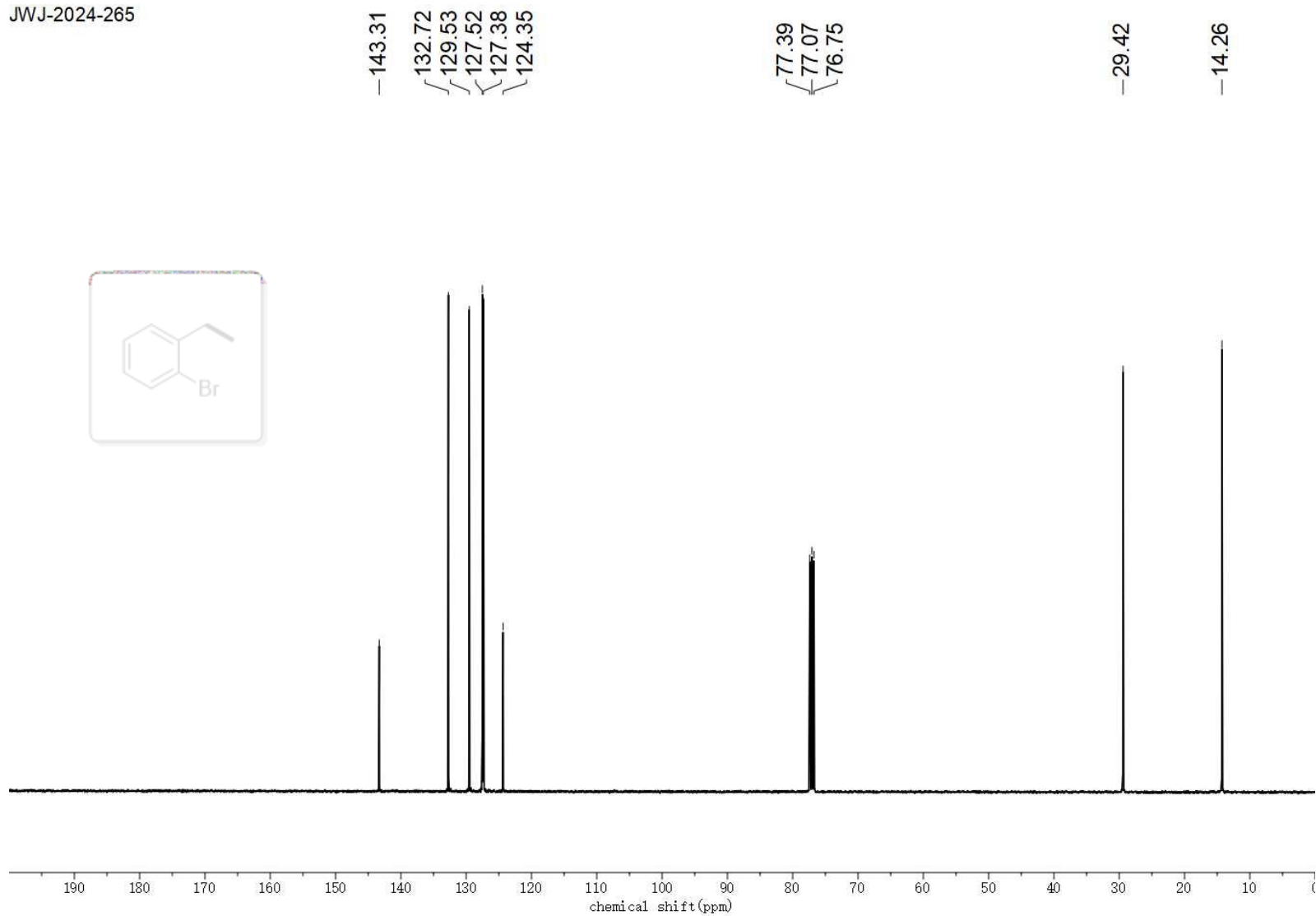
JWJ-2024-440



JWJ-2024-265



JWJ-2024-265

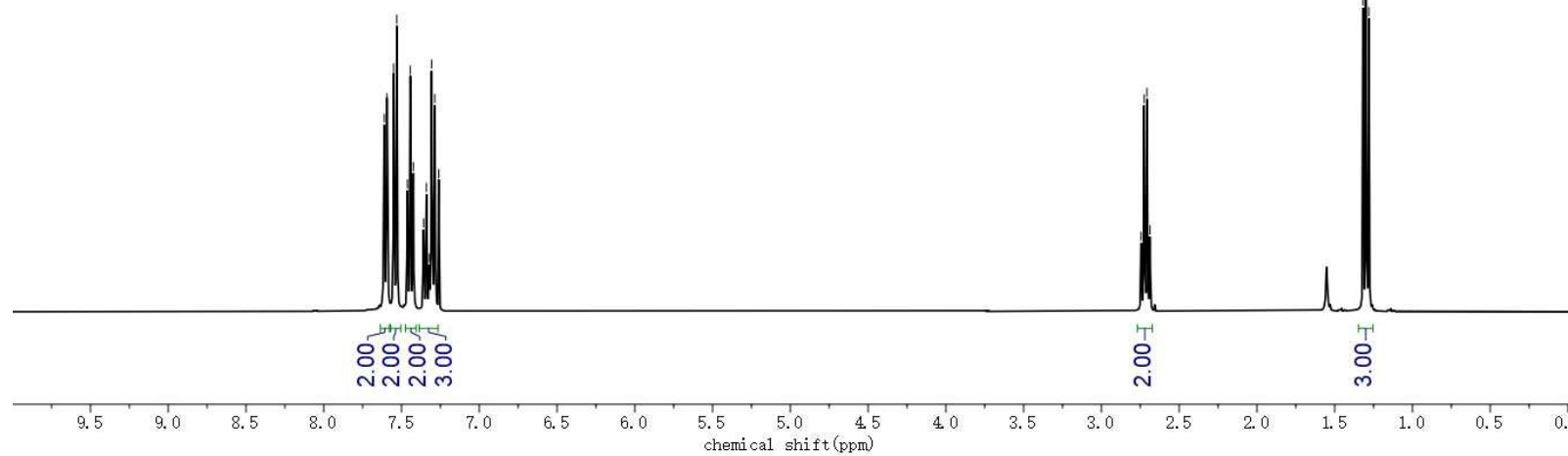
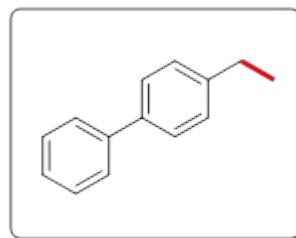


JWJ-2024-268

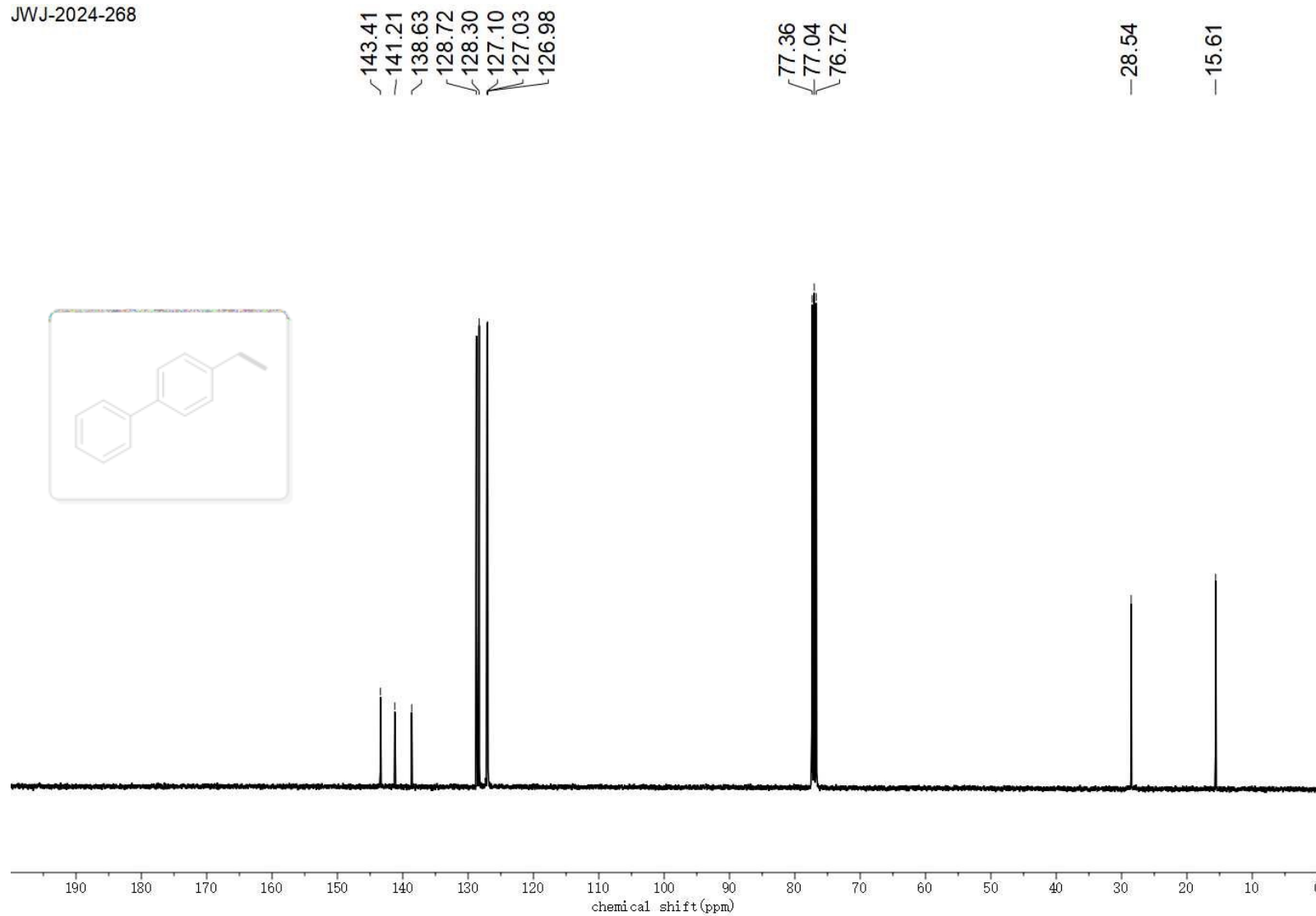
7.6109  
7.5936  
7.5508  
7.5303  
7.4623  
7.4438  
7.4240  
7.3582  
7.3398  
7.3215  
7.3065  
7.2862  
7.2599

2.7448  
2.7258  
2.7068  
2.6878

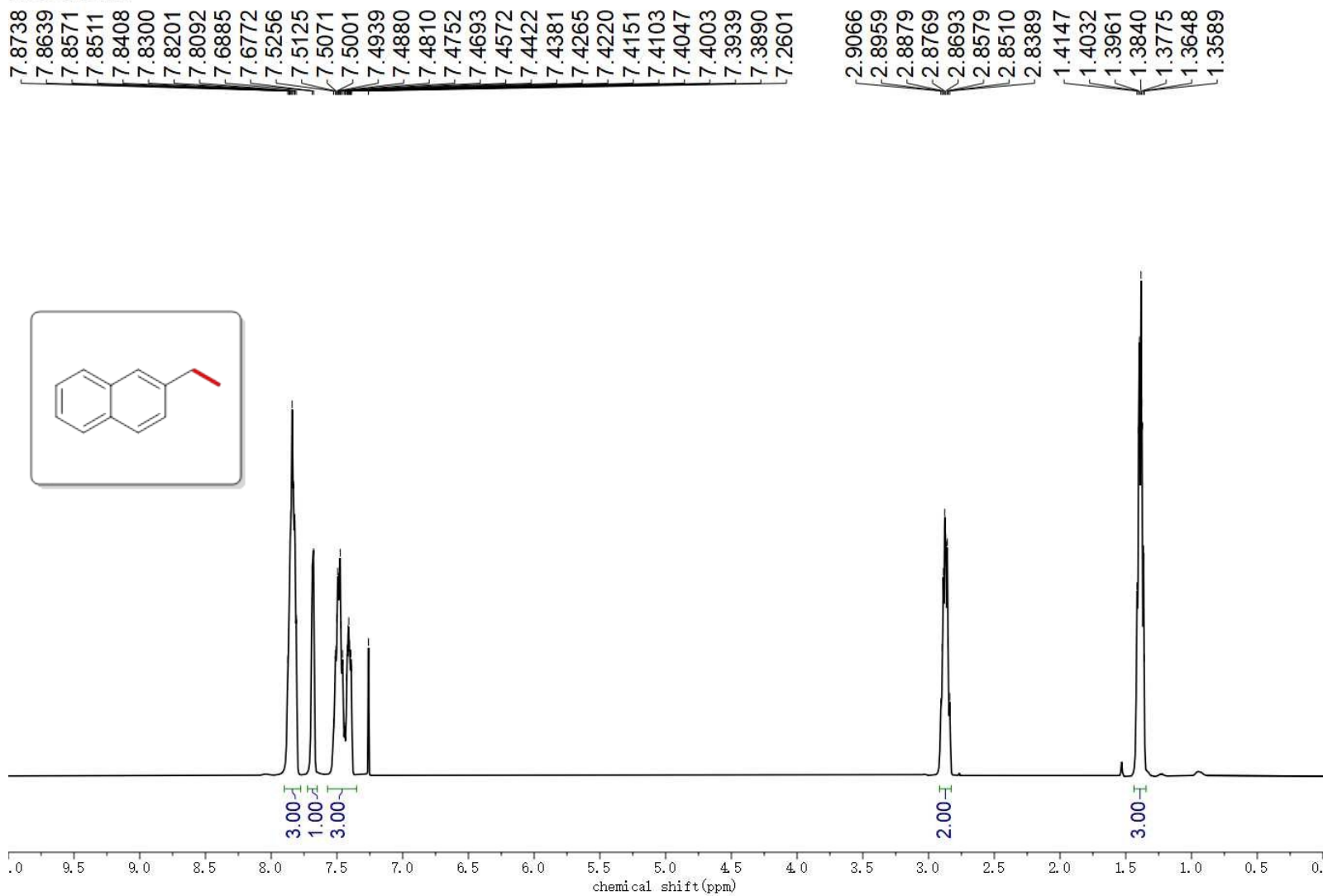
1.3183  
1.2992  
1.2801



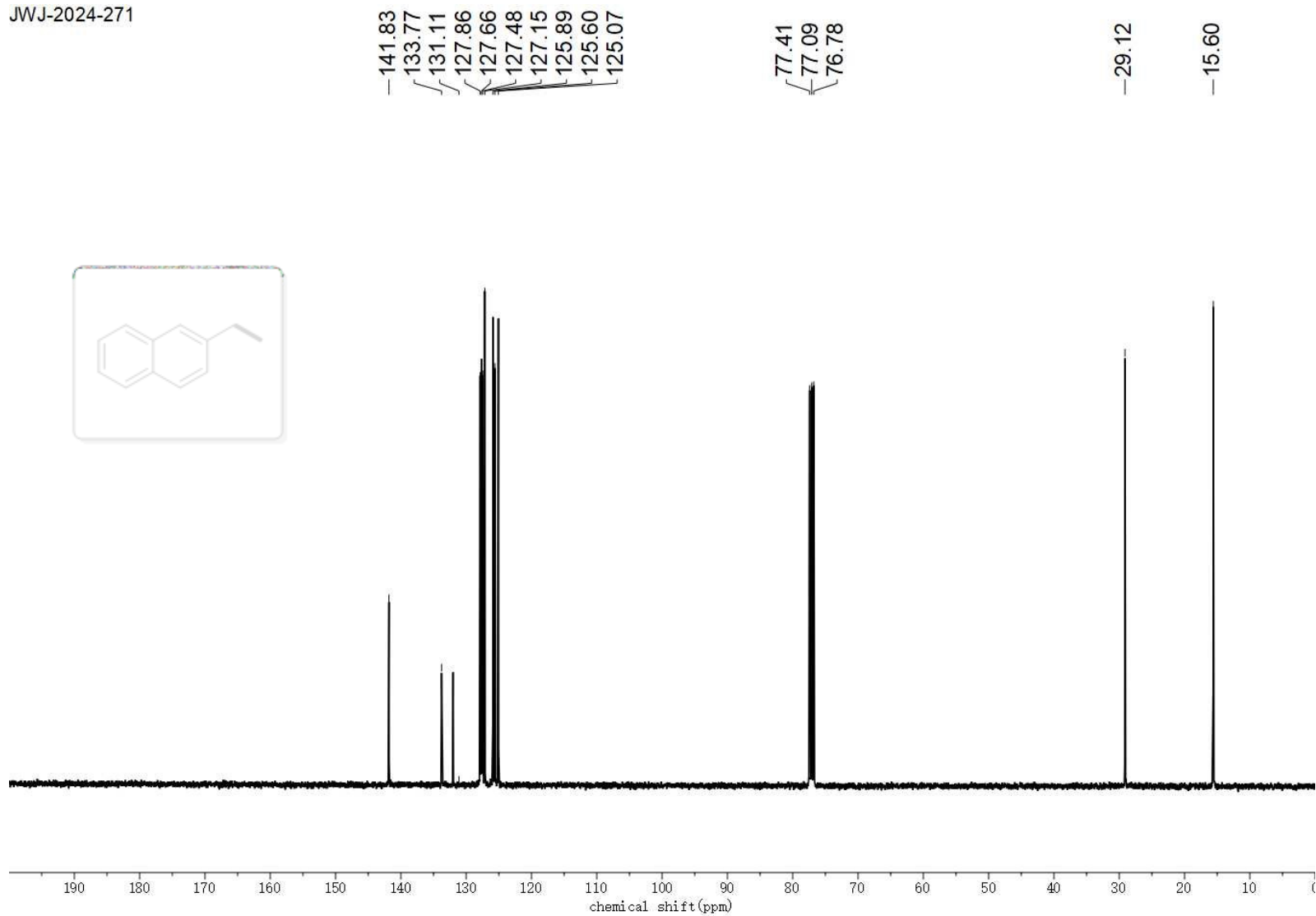
JWJ-2024-268



JWJ-2024-271

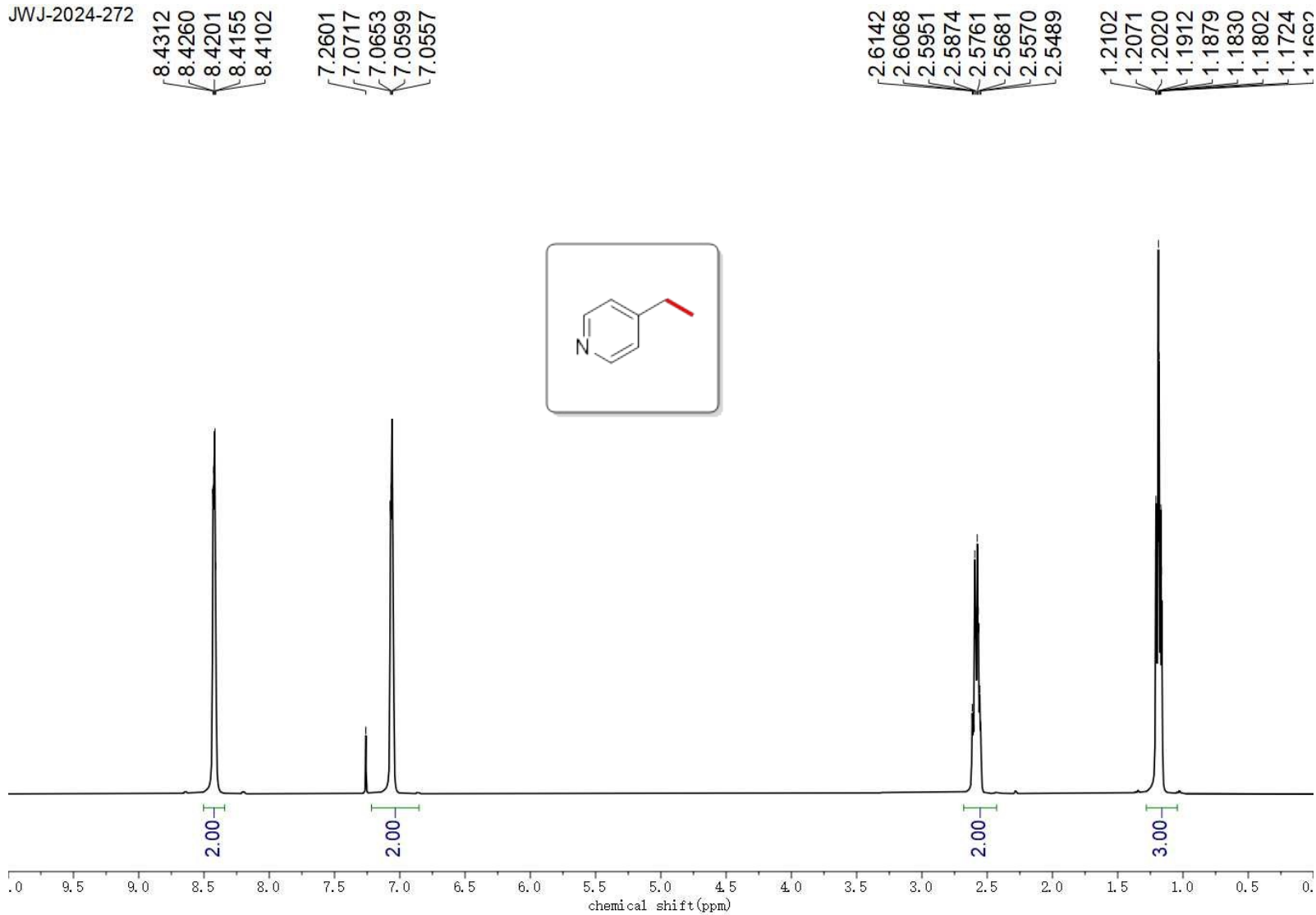


JWJ-2024-271

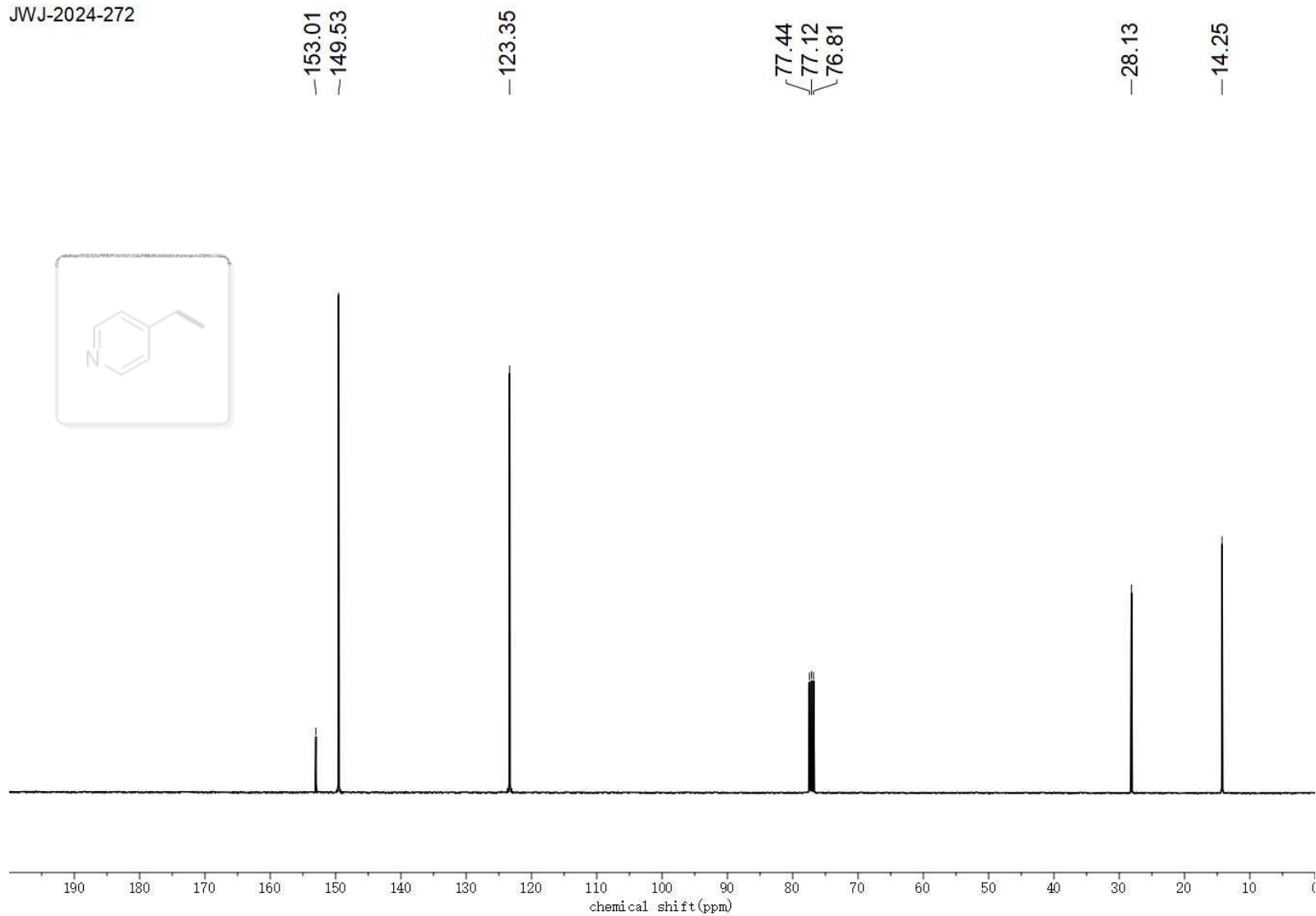




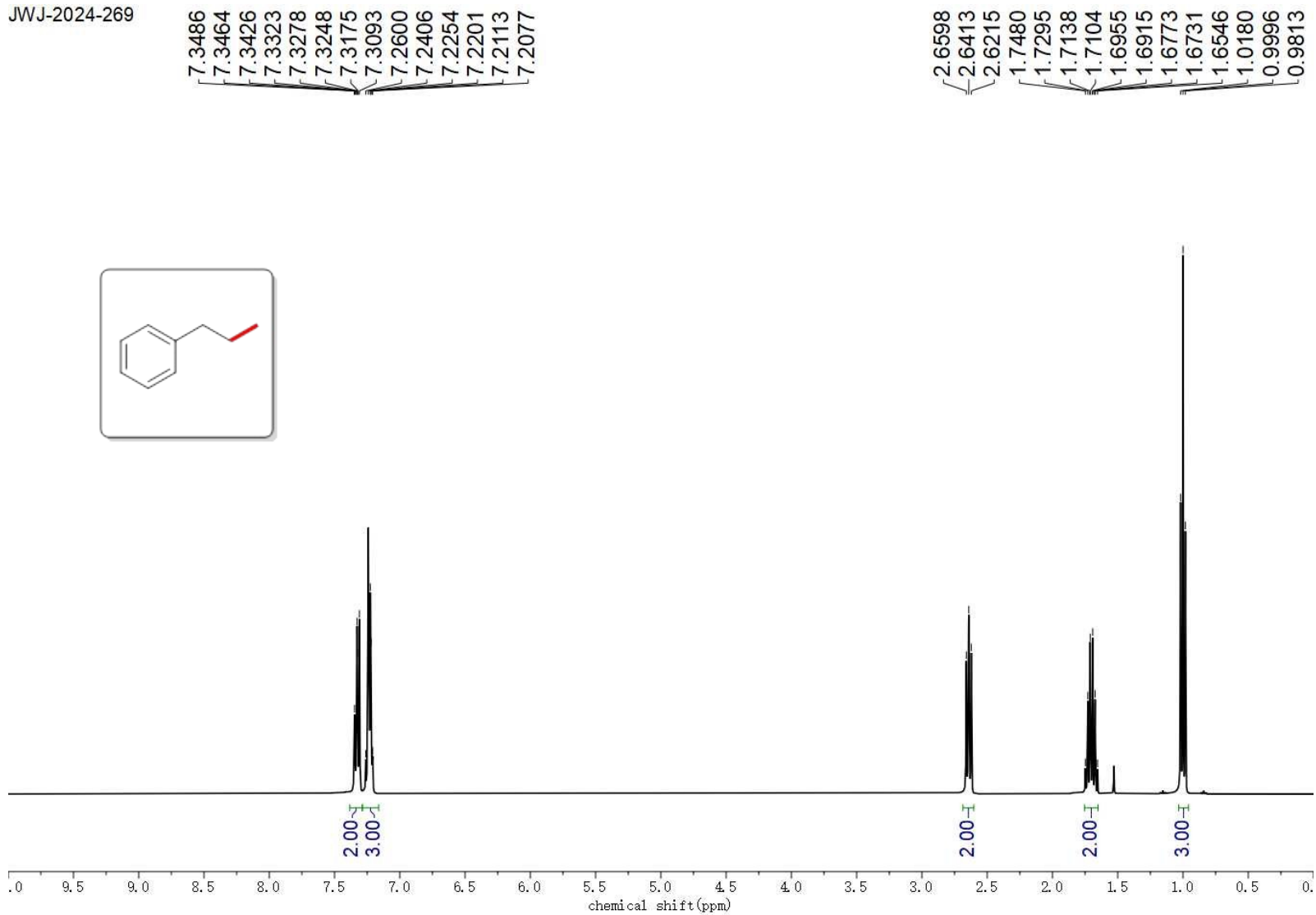
JWJ-2024-272



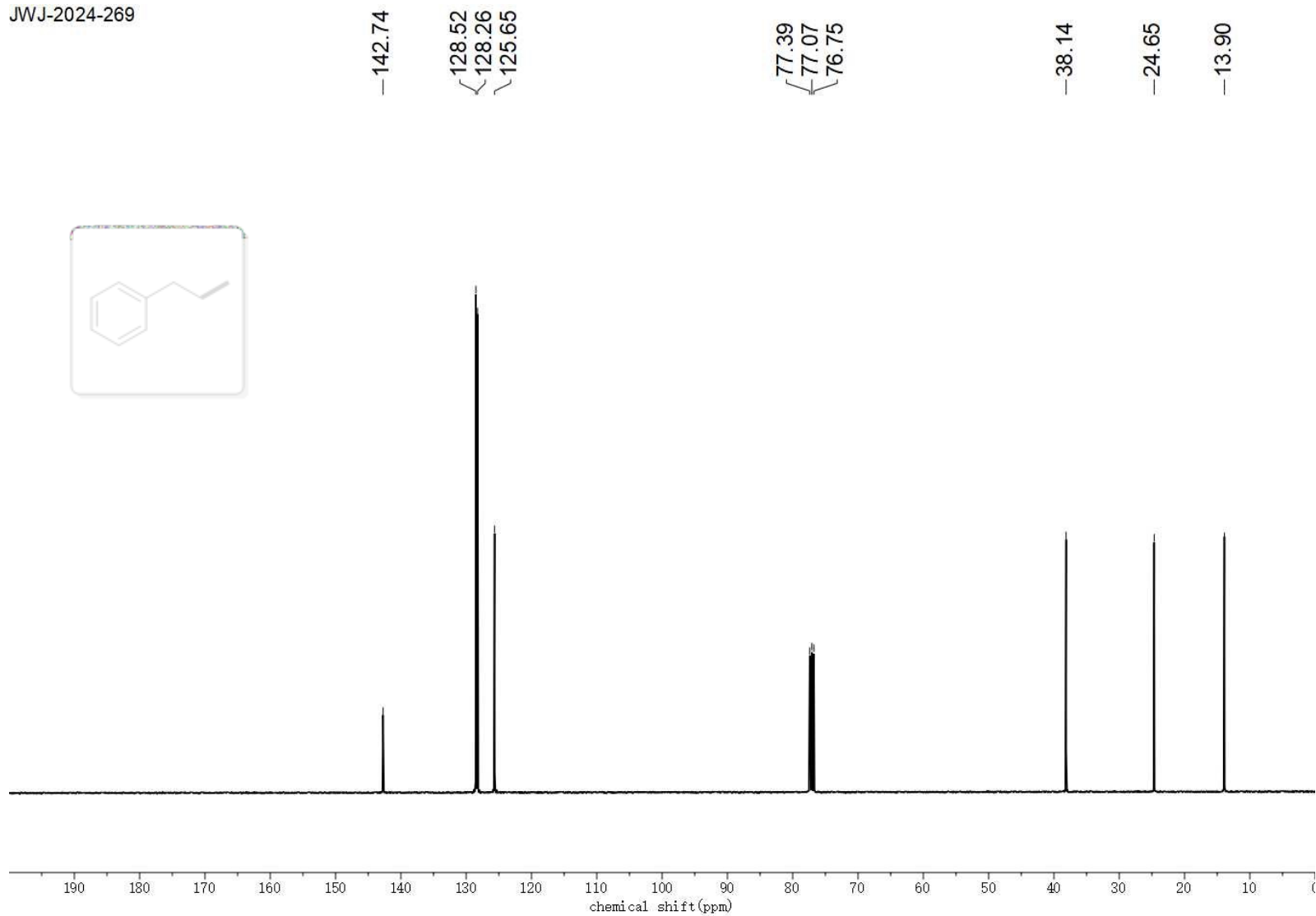
JWJ-2024-272



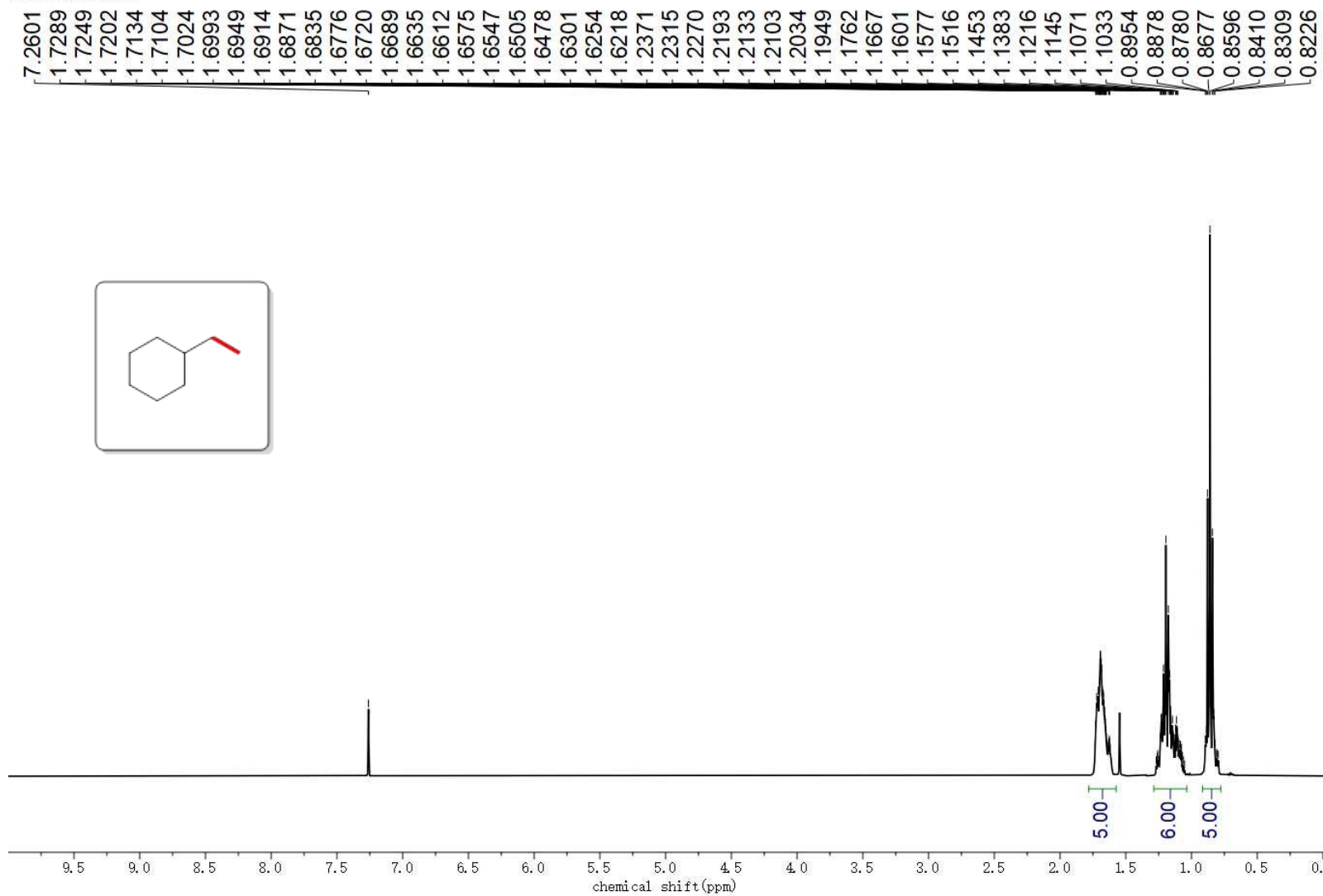
JWJ-2024-269



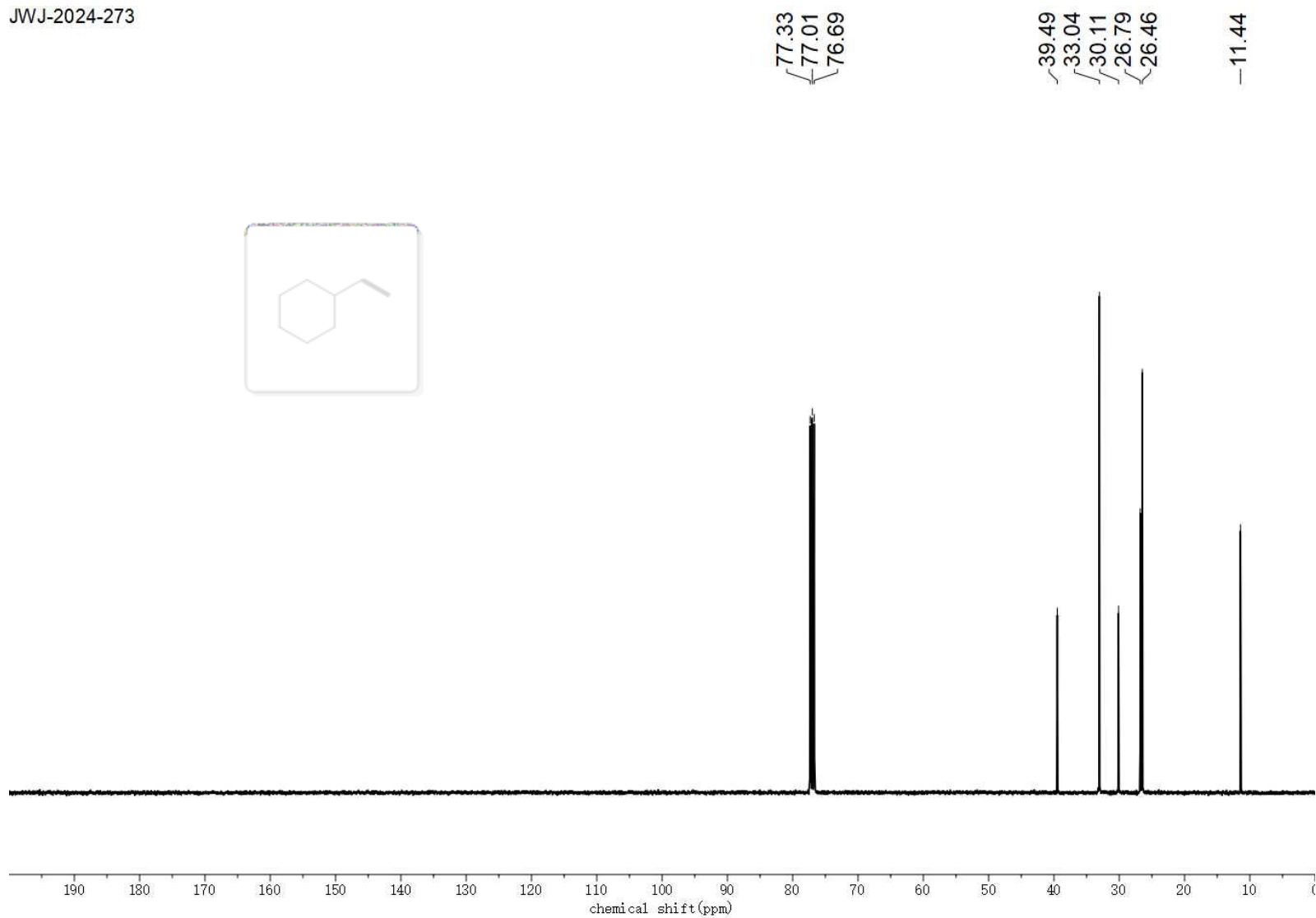
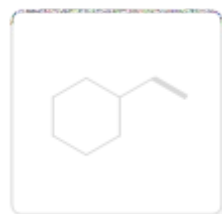
JWJ-2024-269



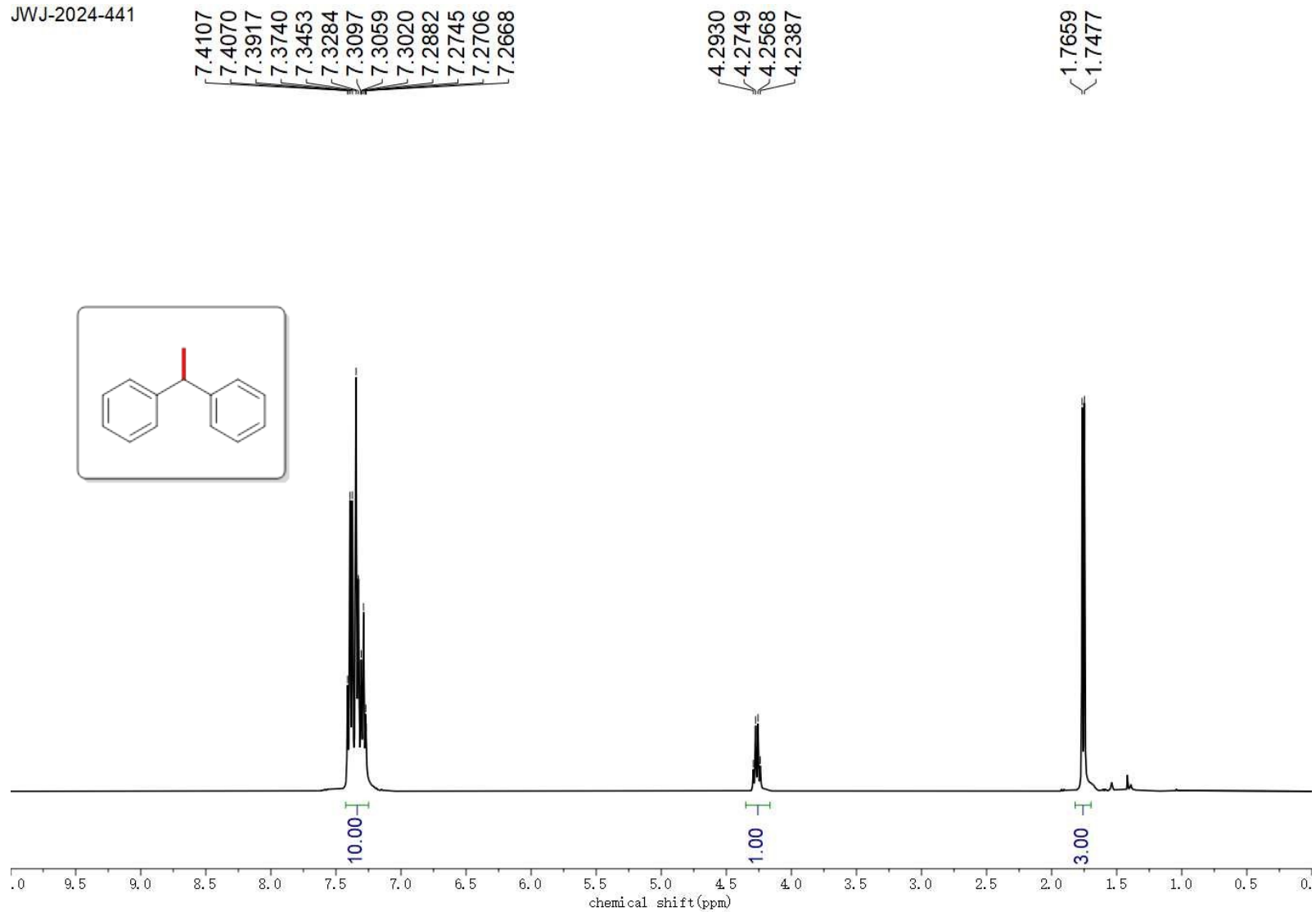
JWJ-2024-273



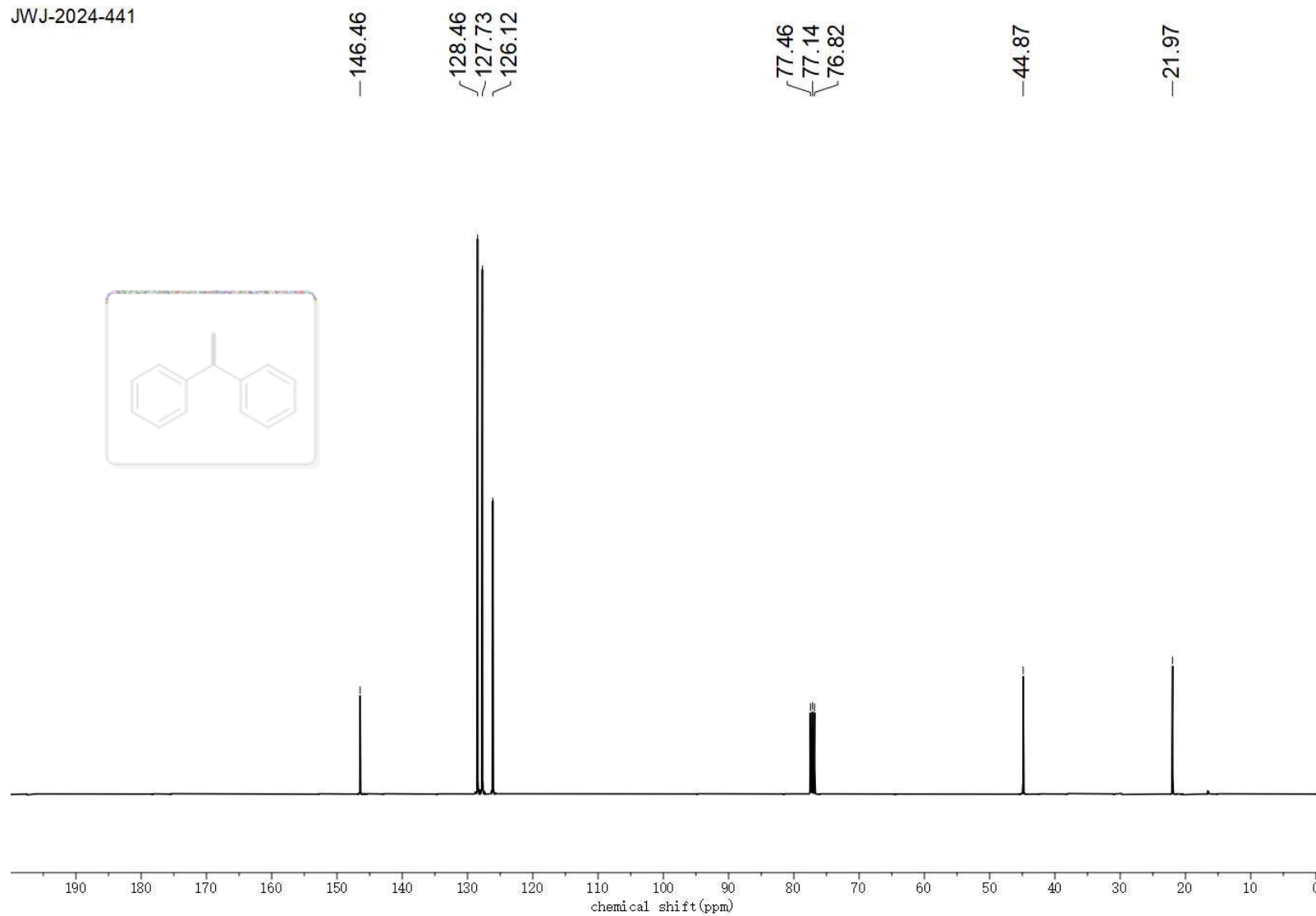
JWJ-2024-273



JWJ-2024-441

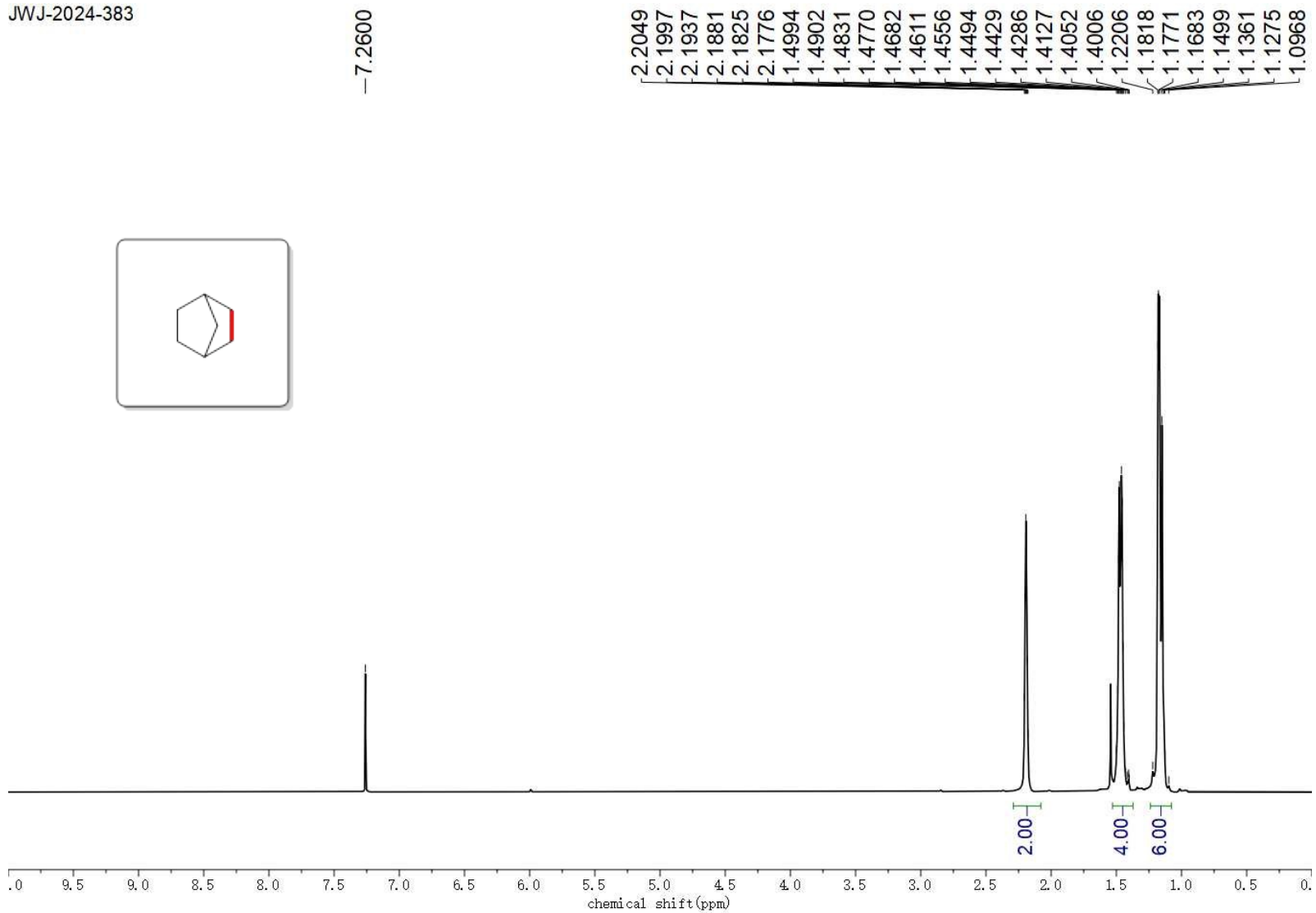
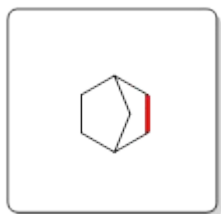


JWJ-2024-441

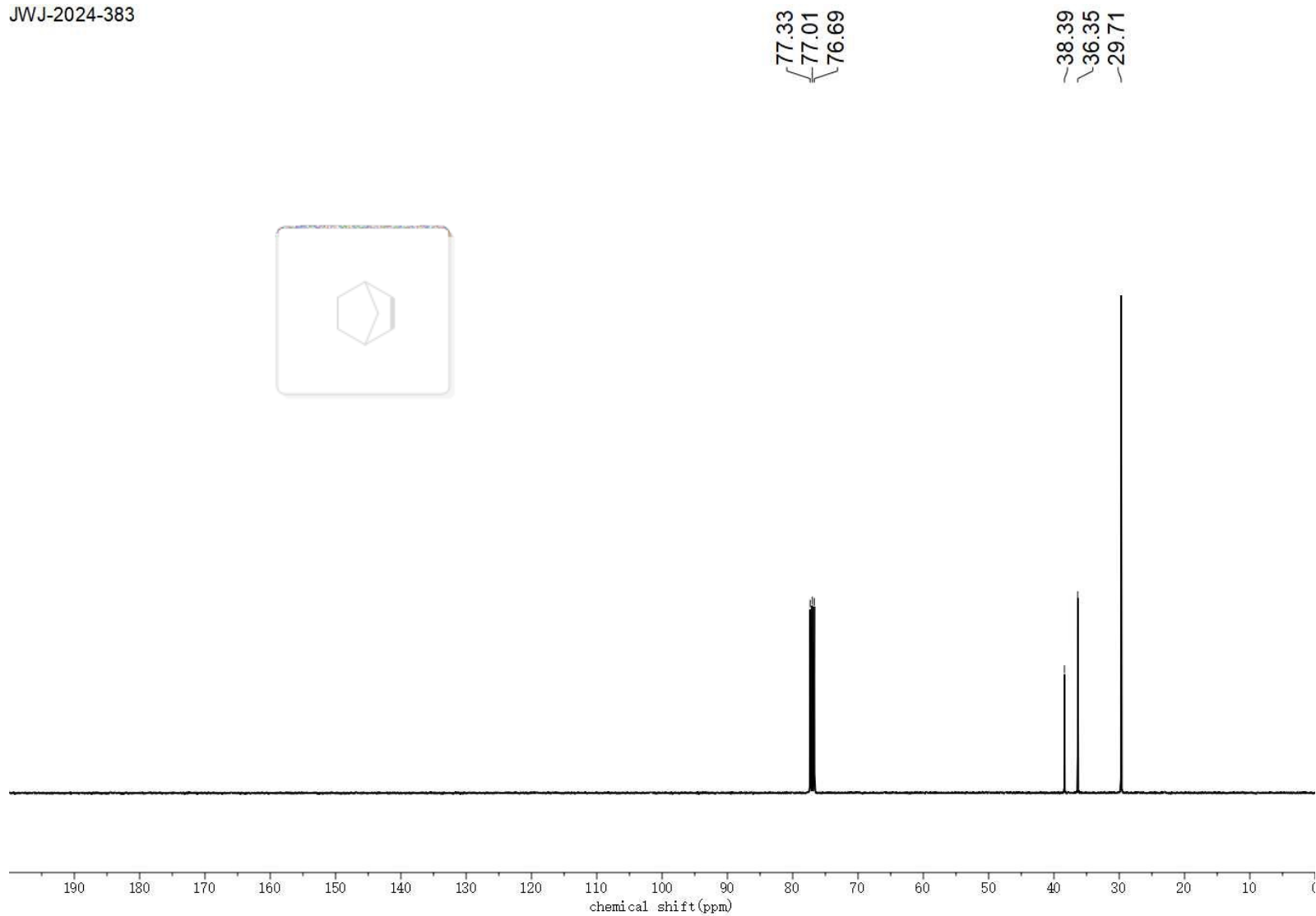




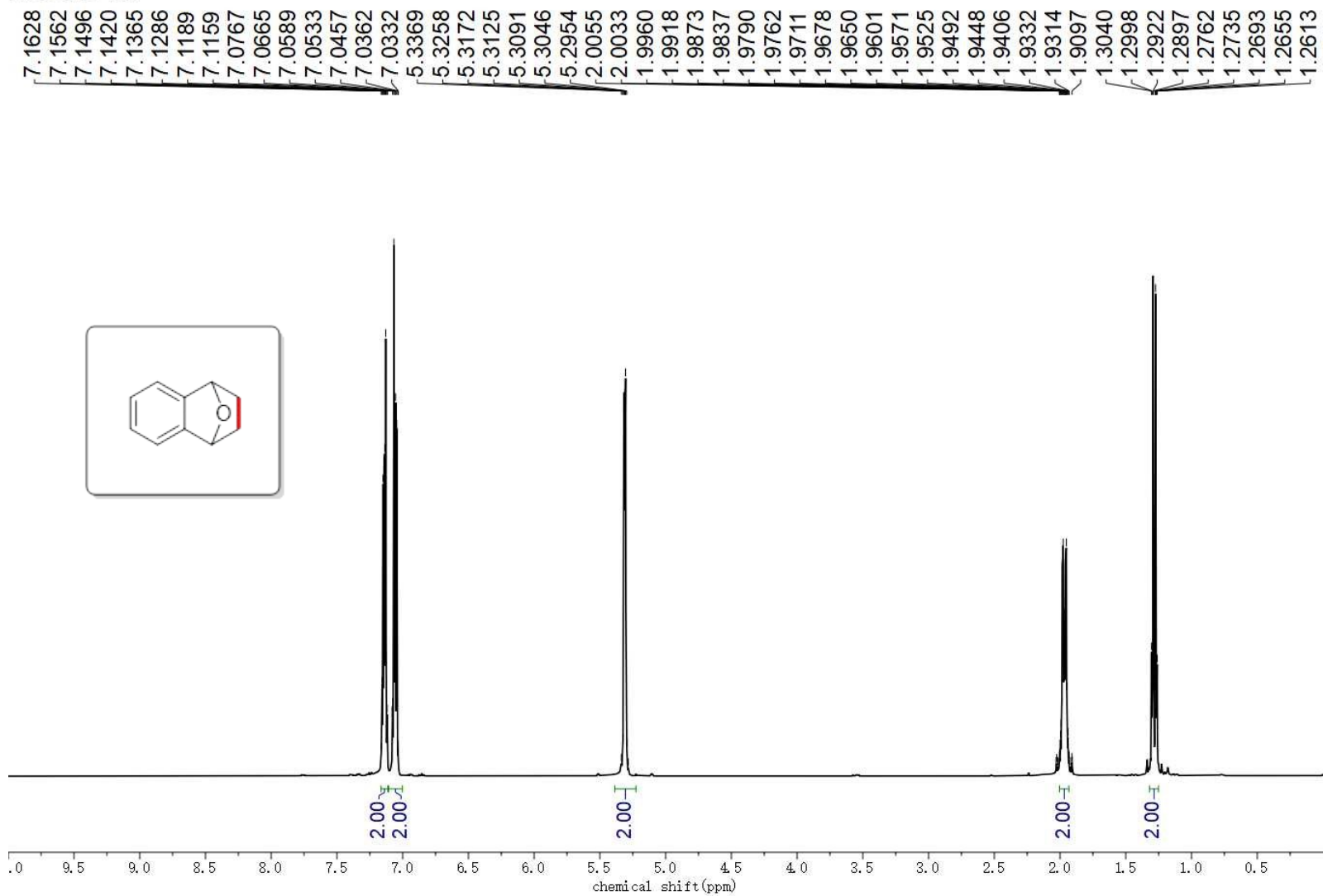
JWJ-2024-383



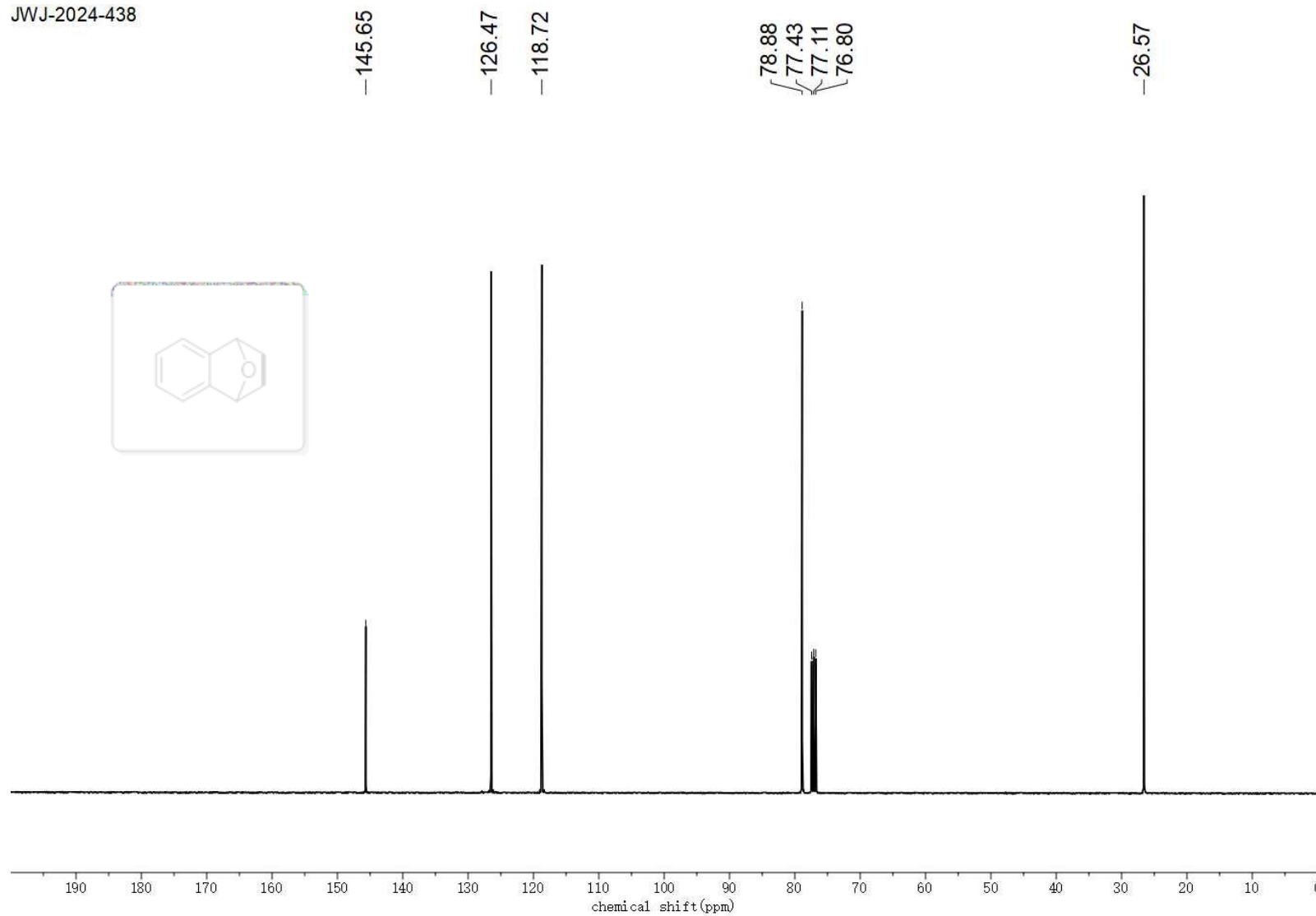
JWJ-2024-383



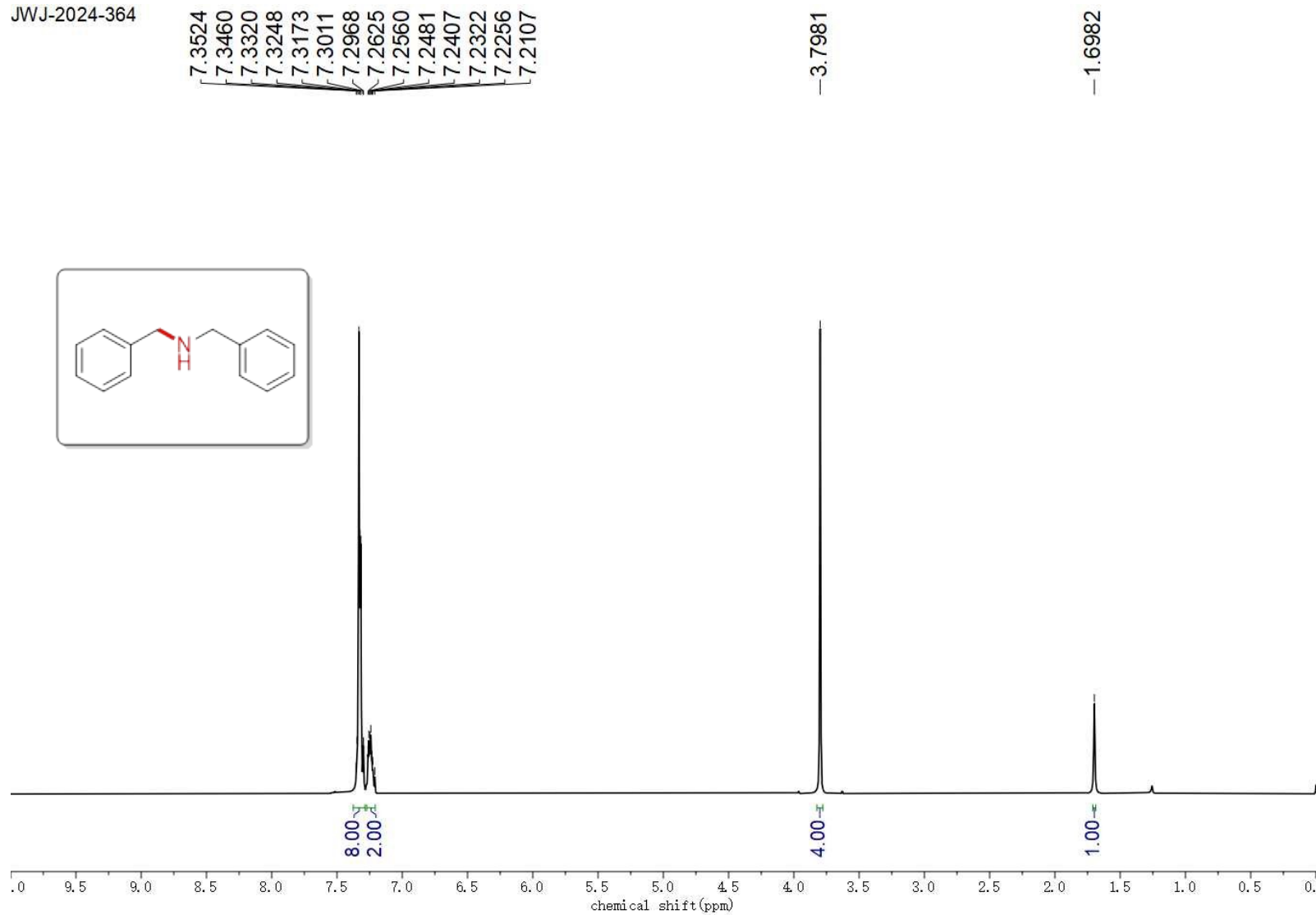
JWJ-2024-438



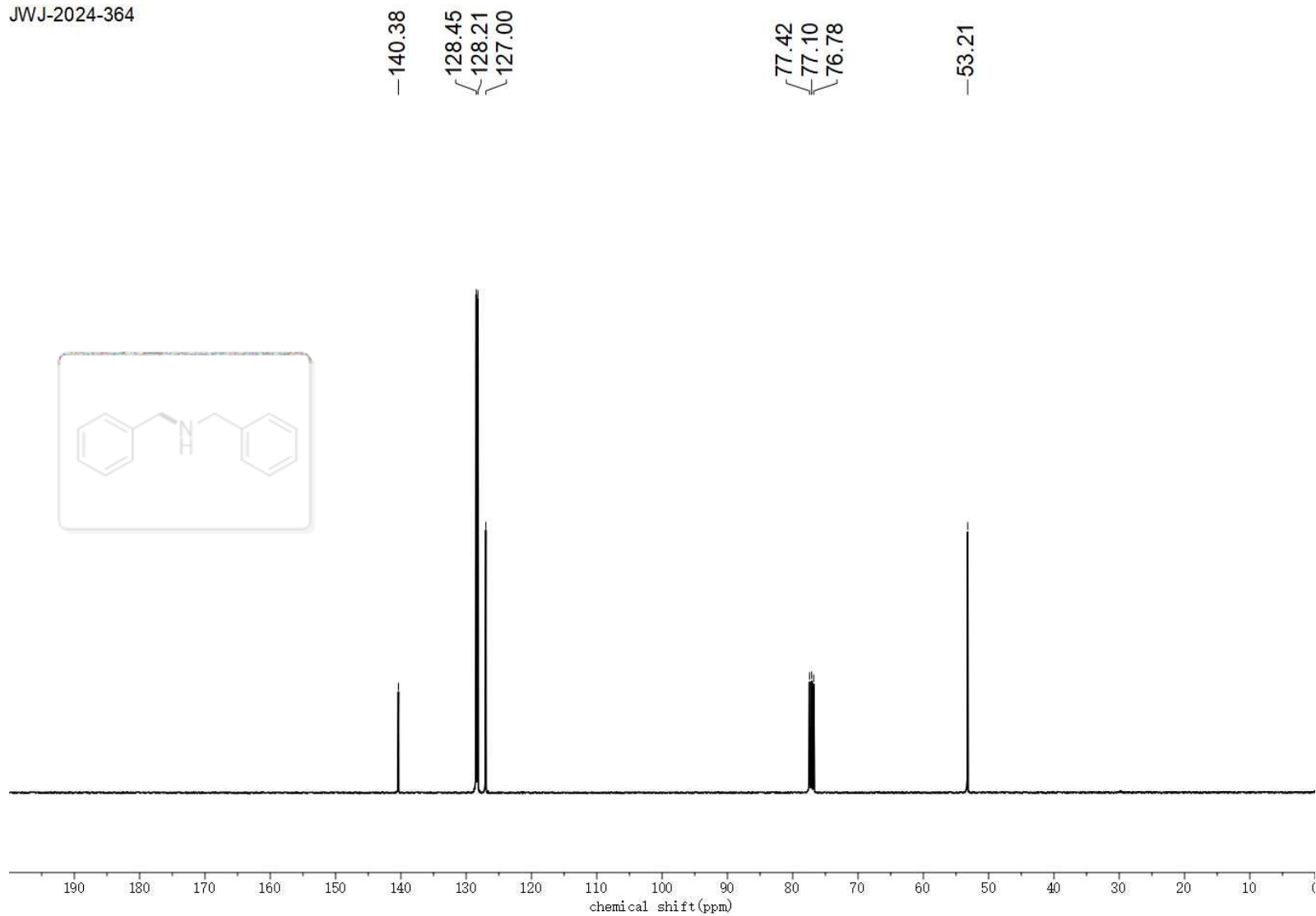
JWJ-2024-438



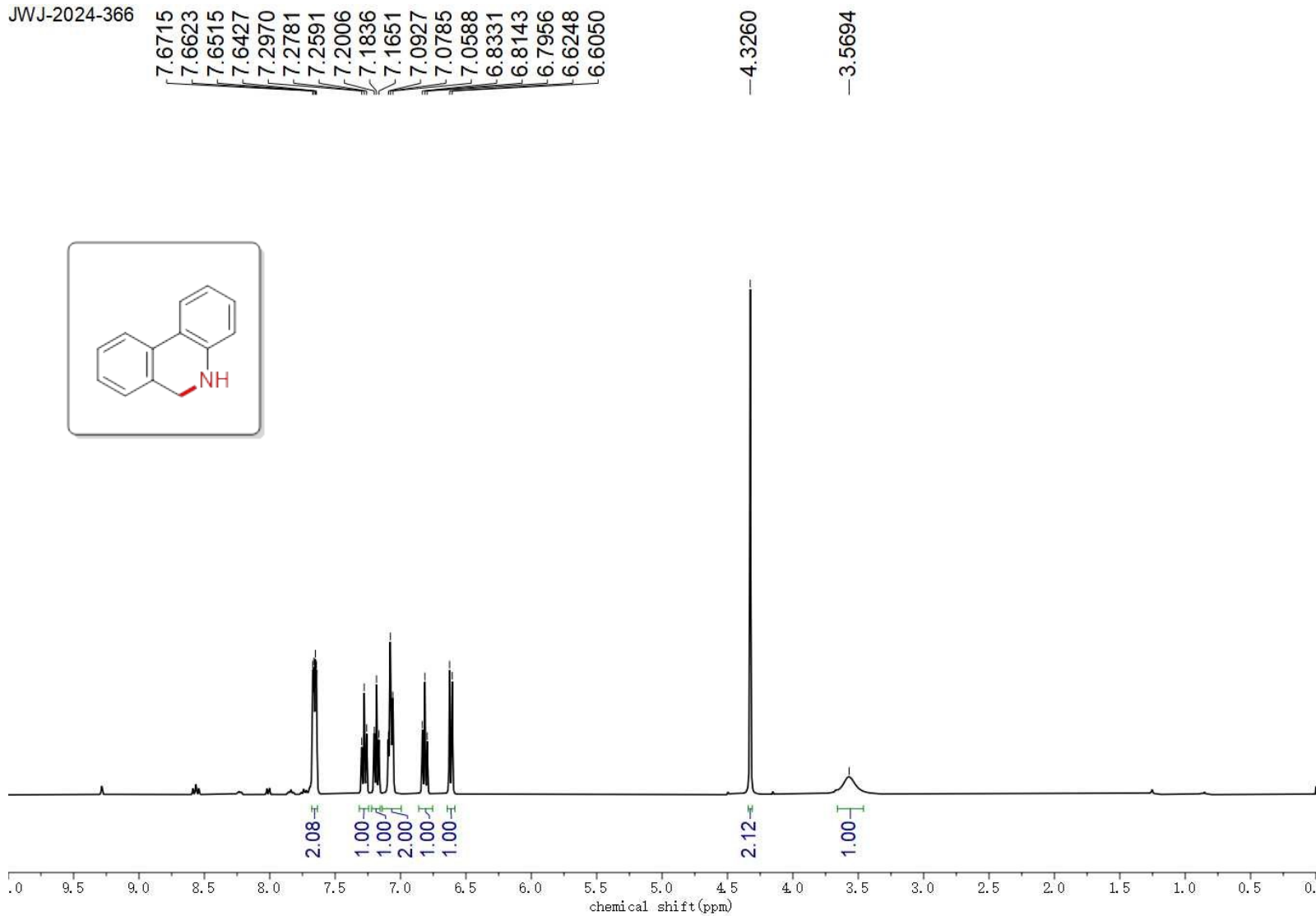
JWJ-2024-364



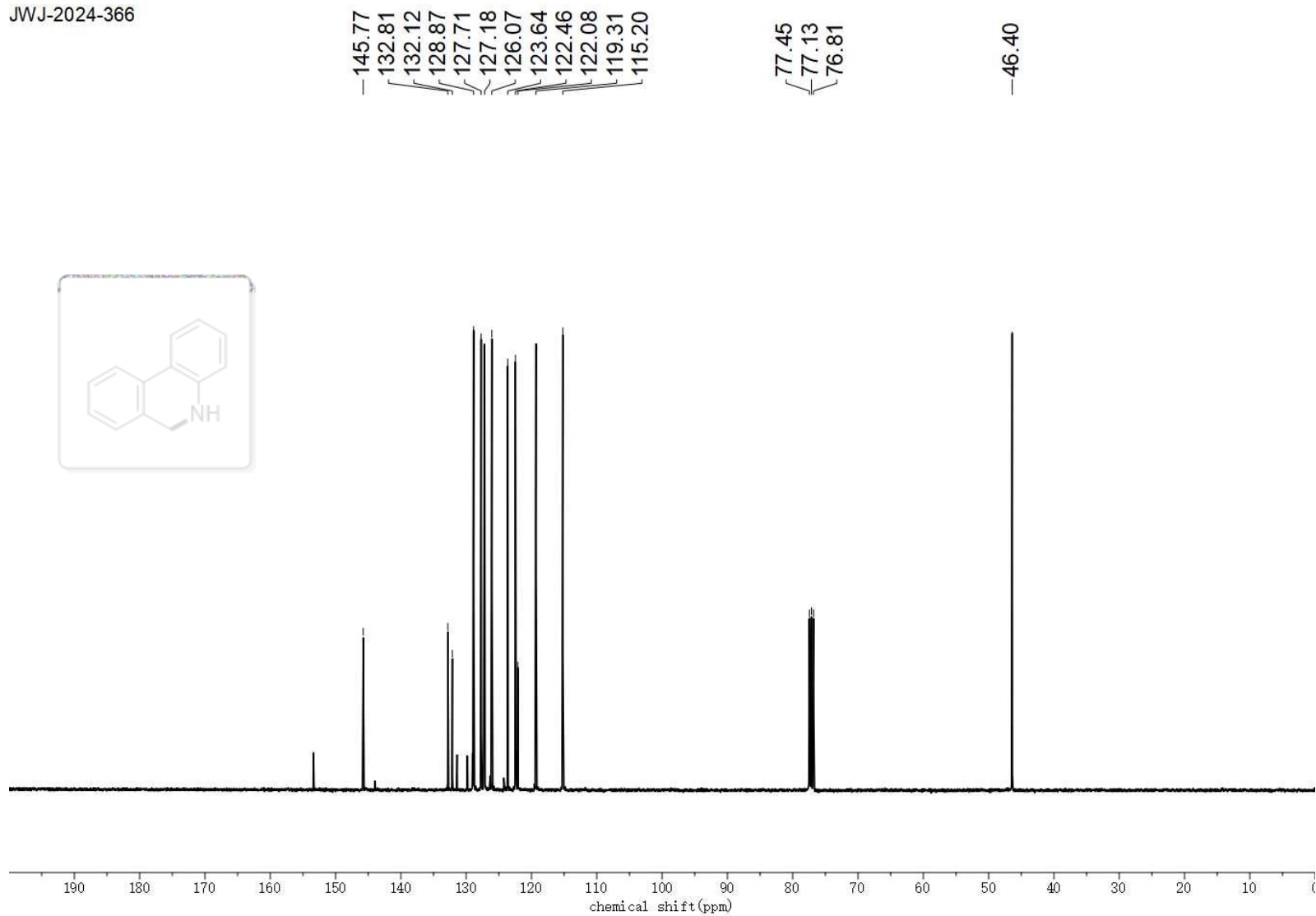
JWJ-2024-364



JWJ-2024-366

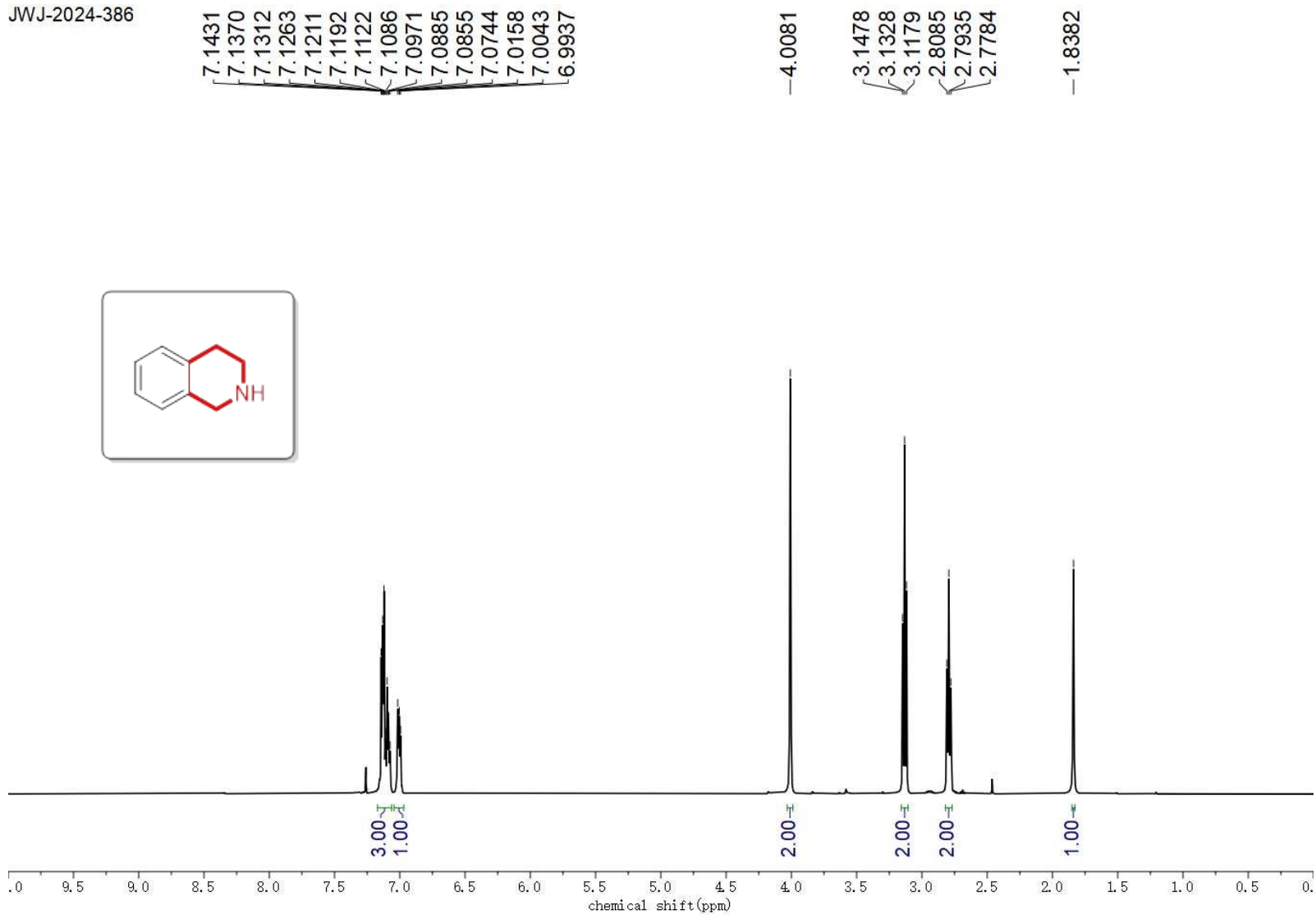


JWJ-2024-366

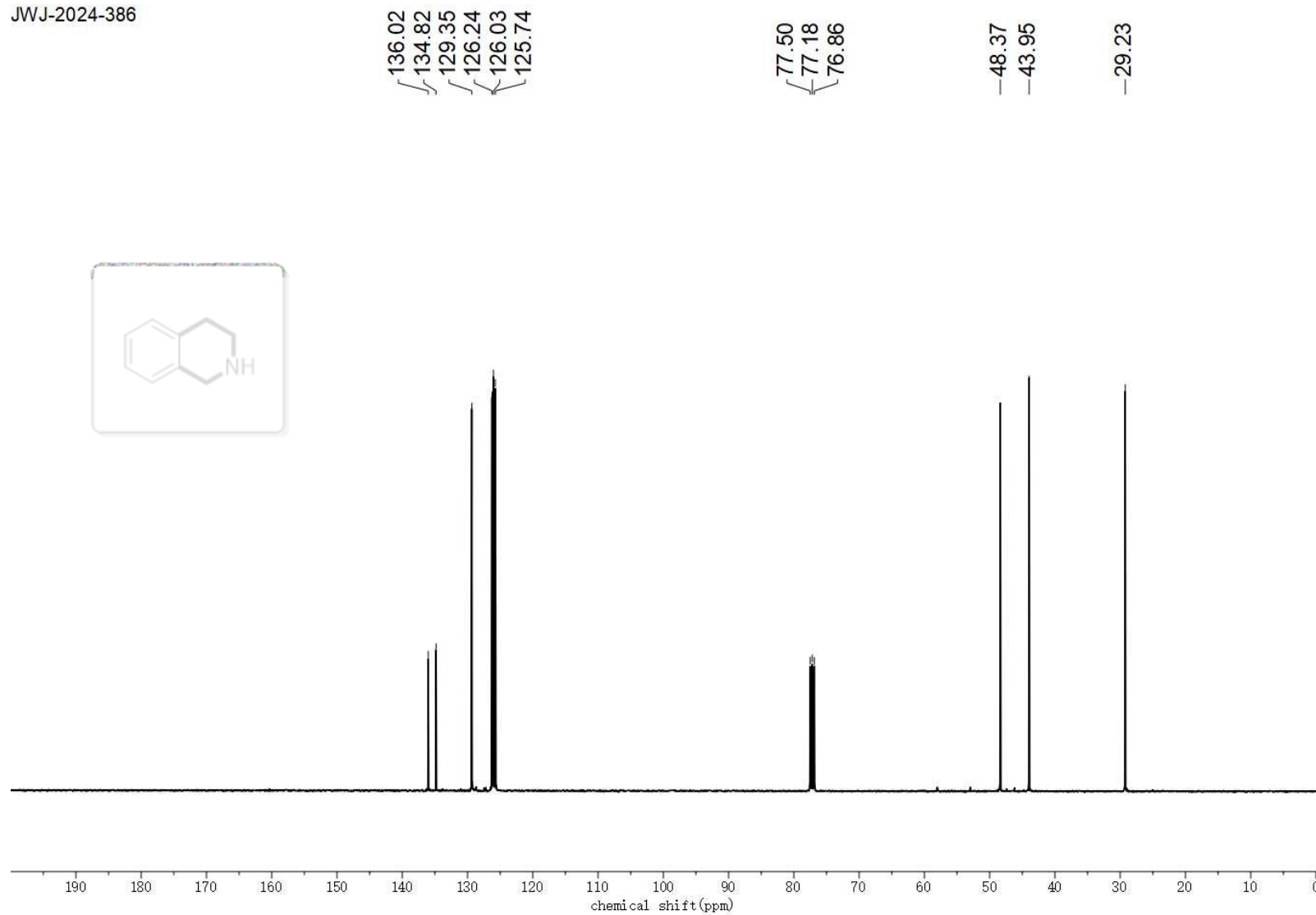




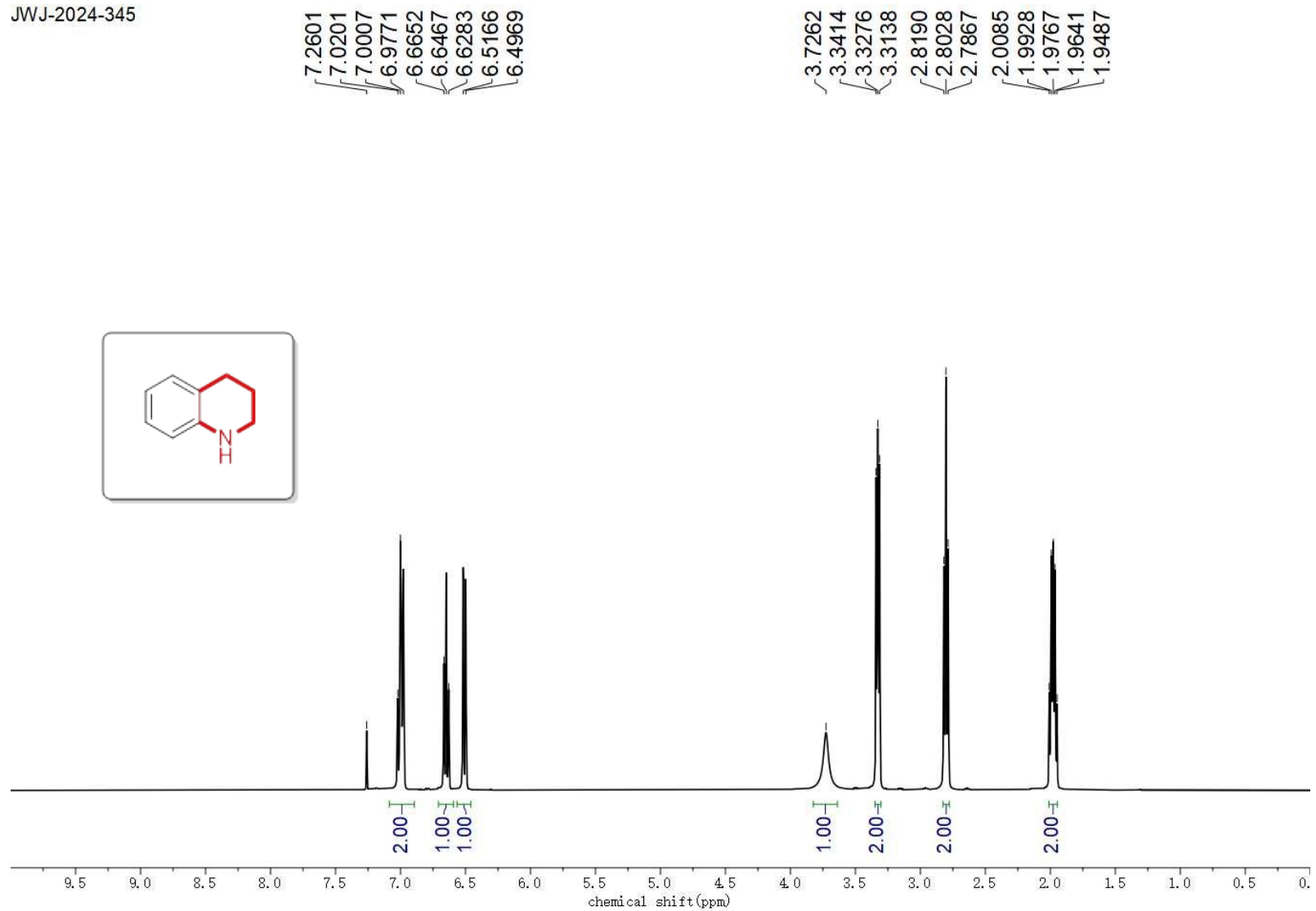
JWJ-2024-386



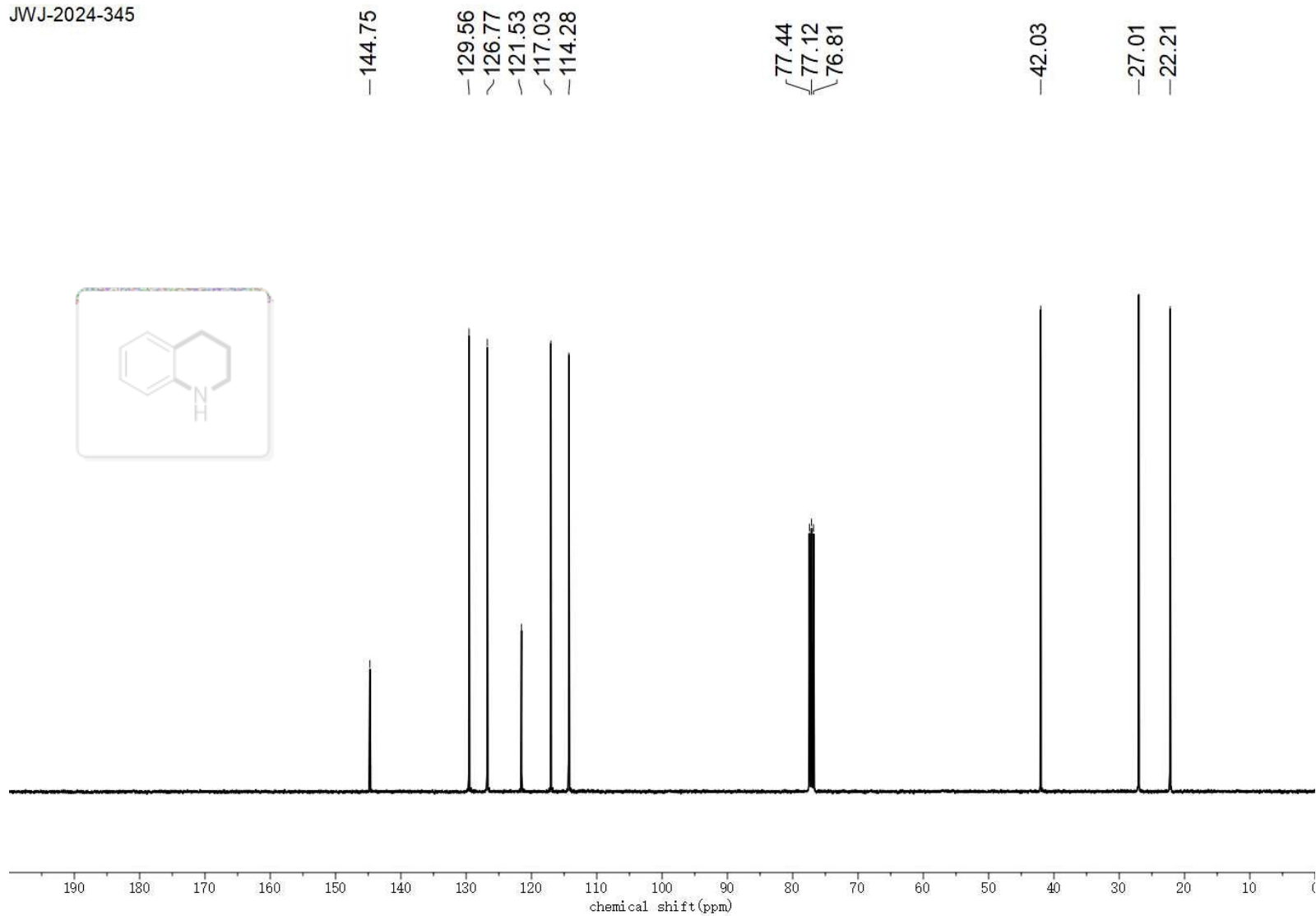
JWJ-2024-386



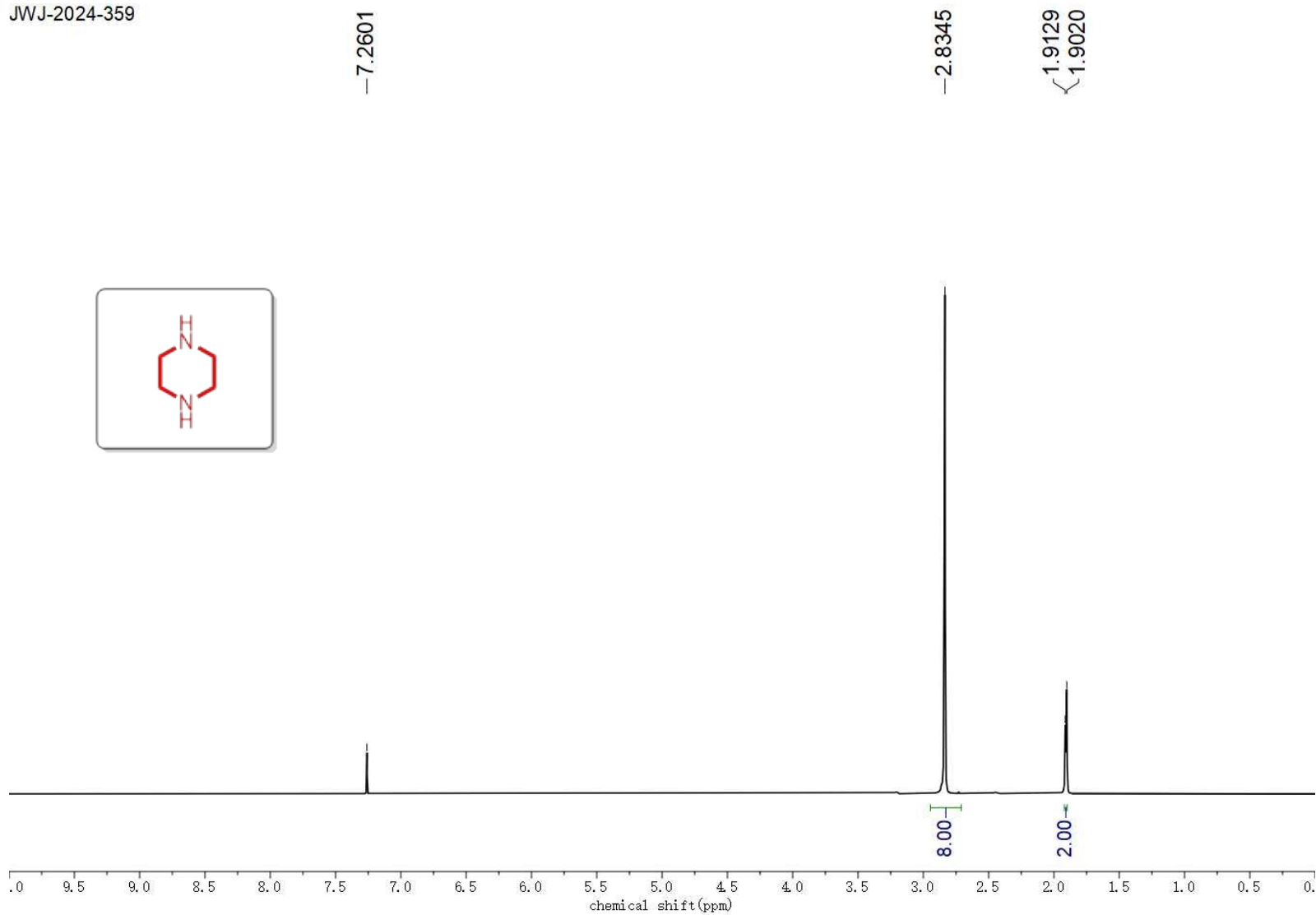
JWJ-2024-345



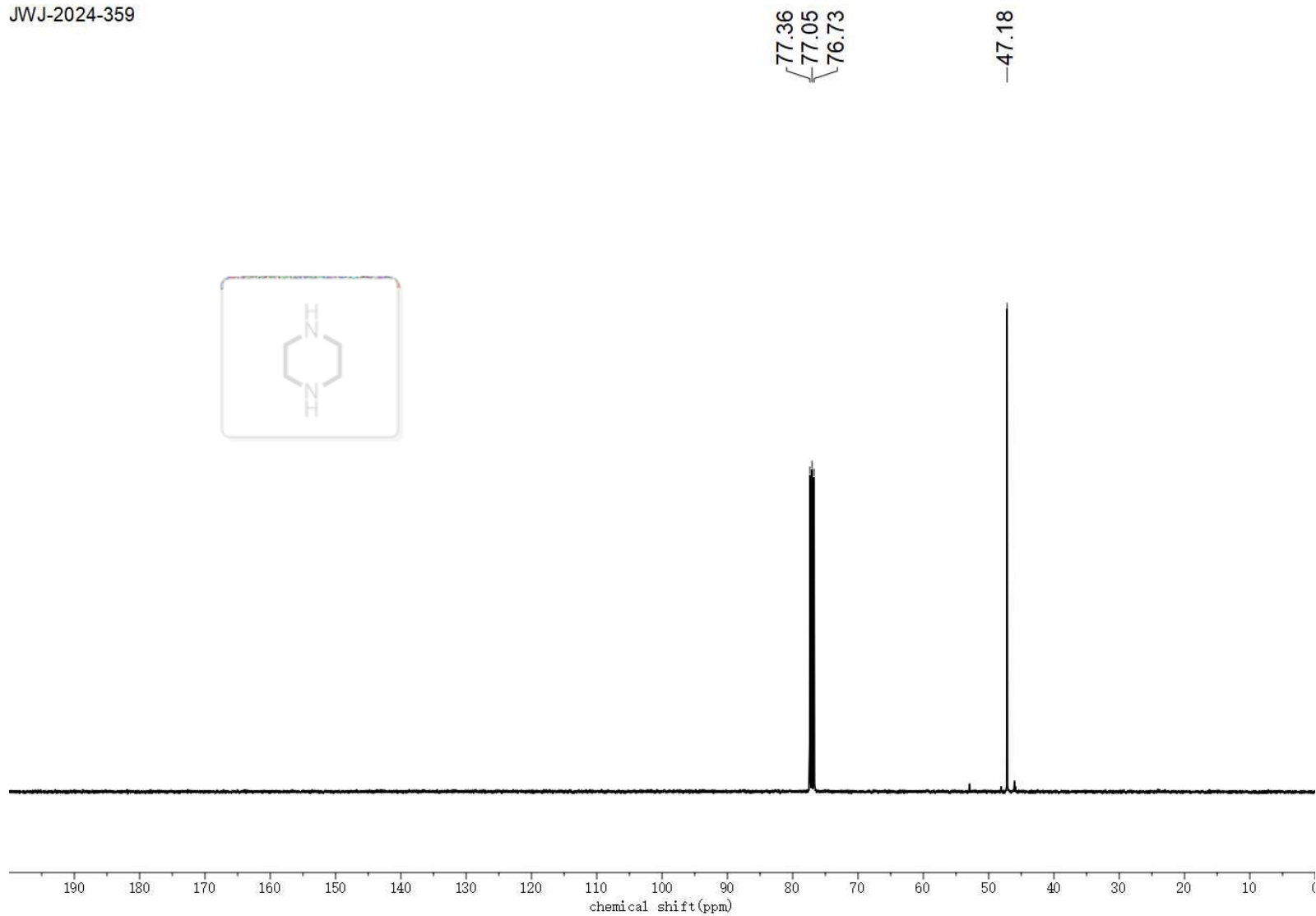
JWJ-2024-345



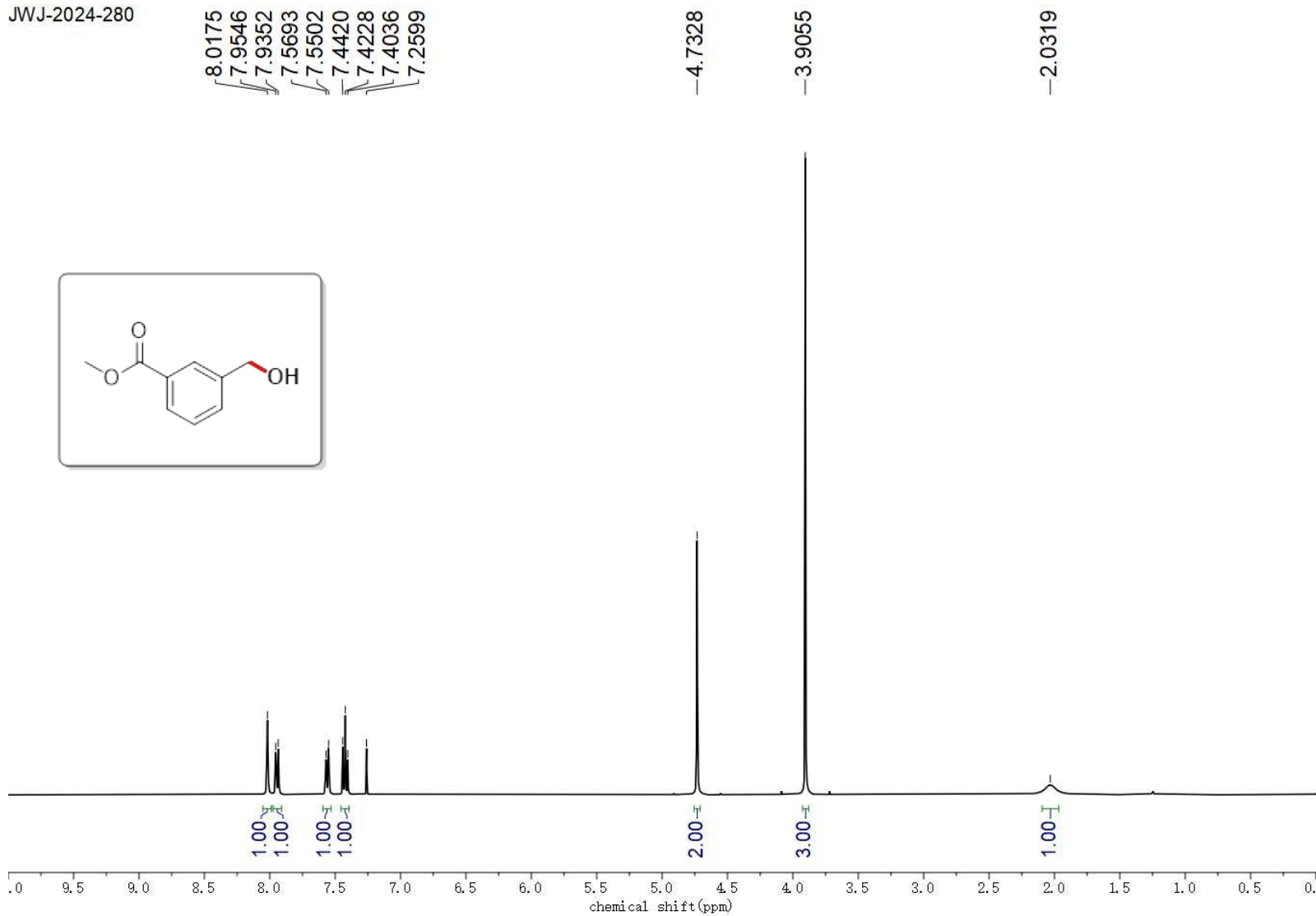
JWJ-2024-359



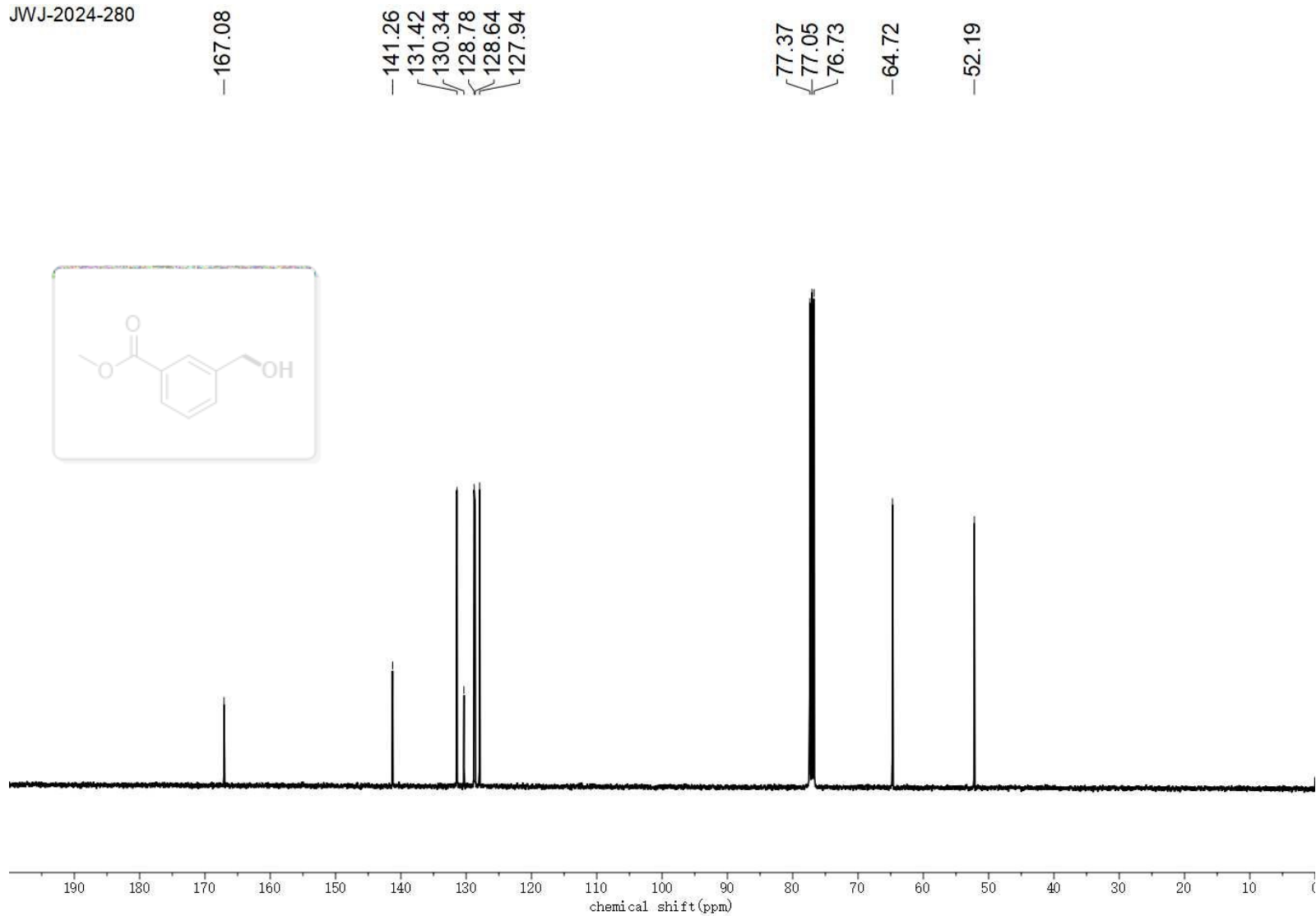
JWJ-2024-359



JWJ-2024-280



JWJ-2024-280





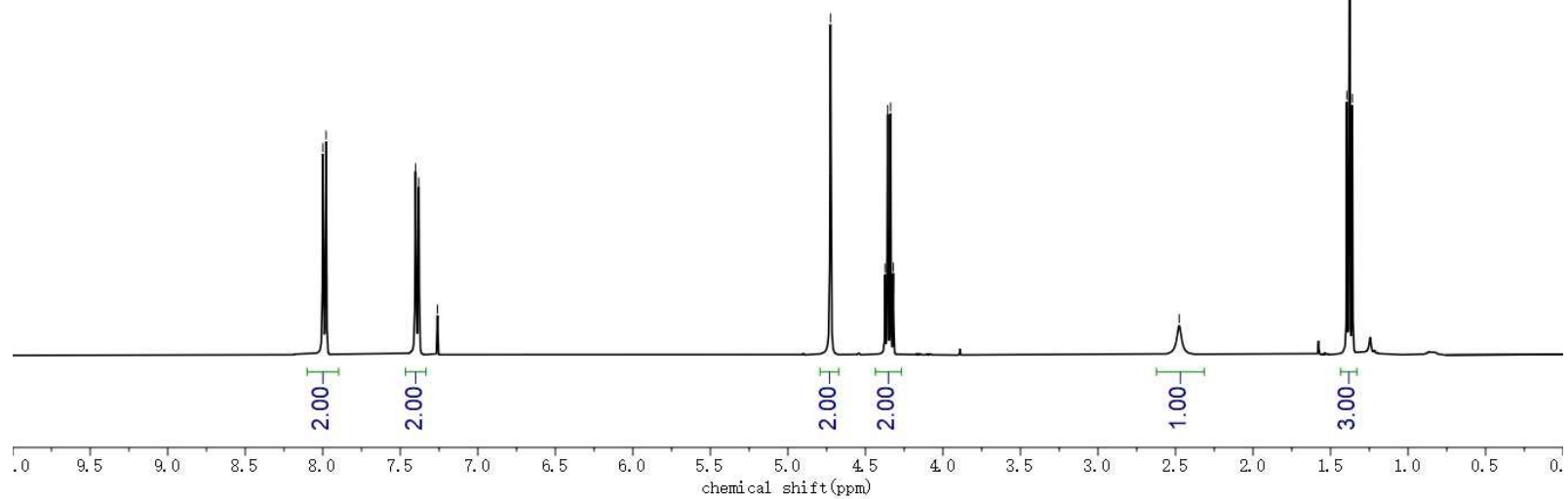
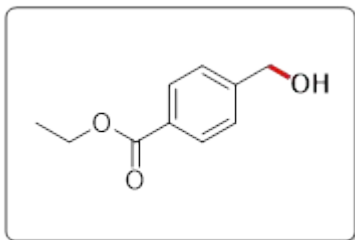
JWJ-2024-170

7.9995  
7.9787  
7.4011  
7.3810  
7.2601

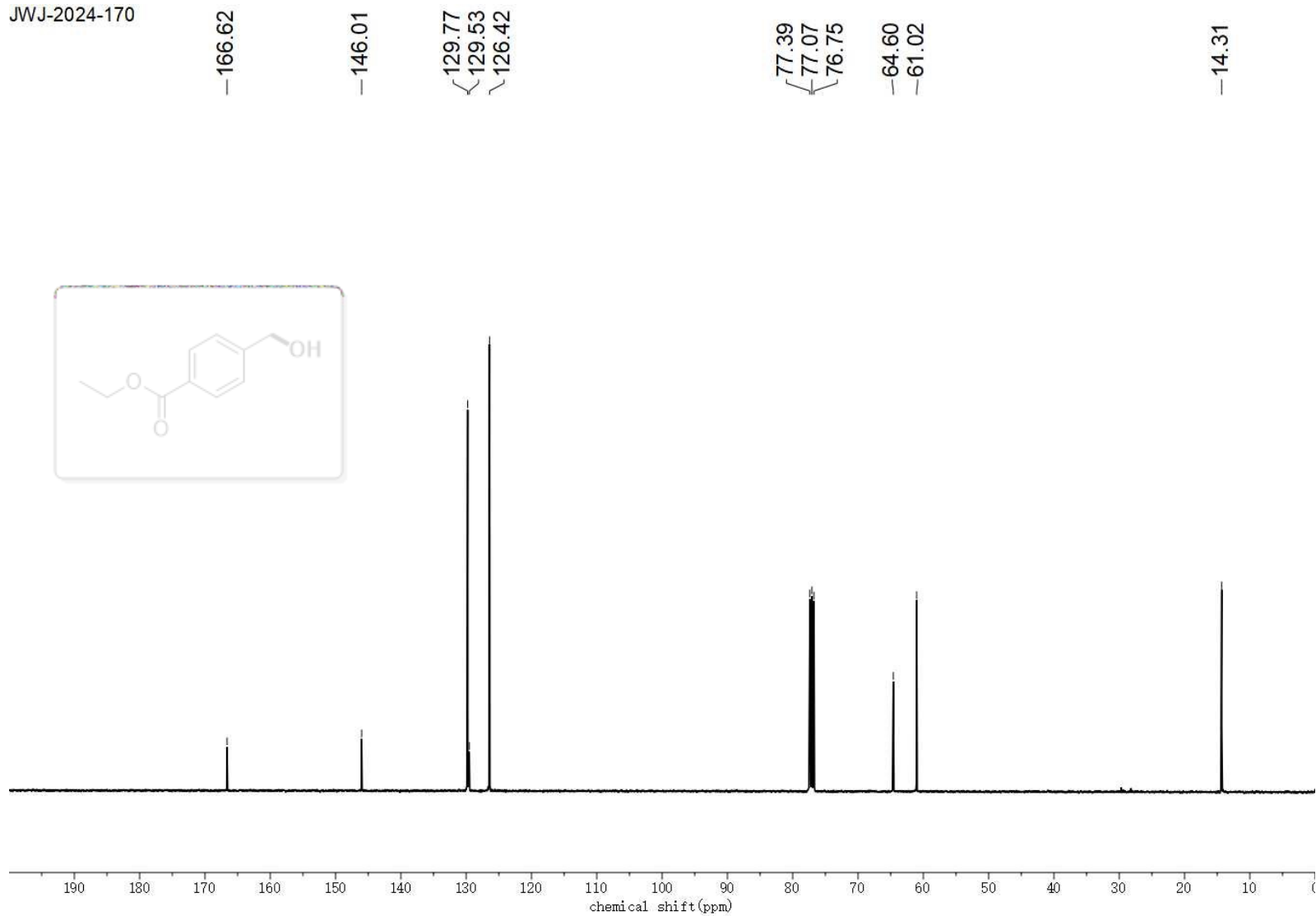
4.7253  
4.3735  
4.3557  
4.3378  
4.3200

-2.4762

1.3949  
1.3769  
1.3592



JWJ-2024-170



JWJ-2024-340

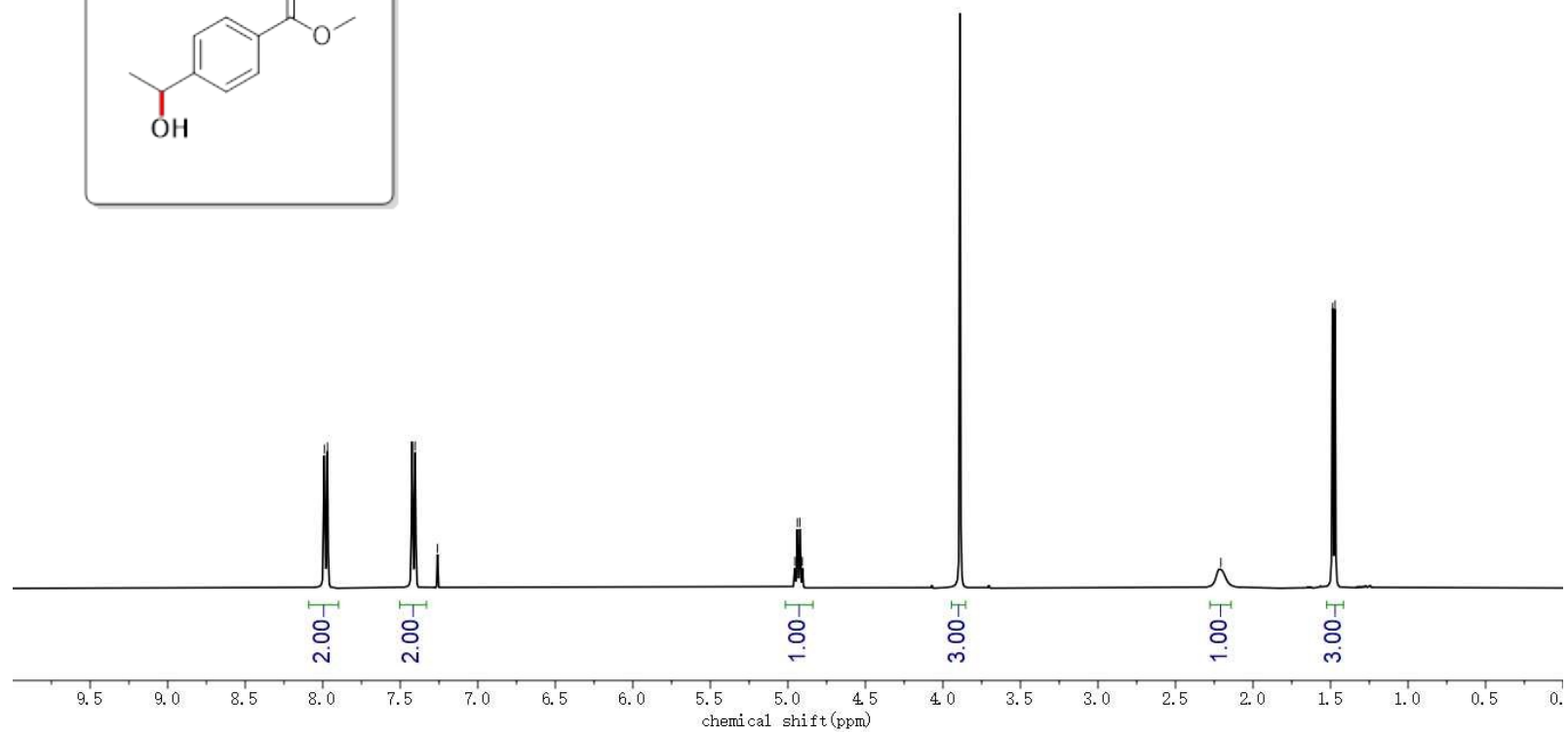
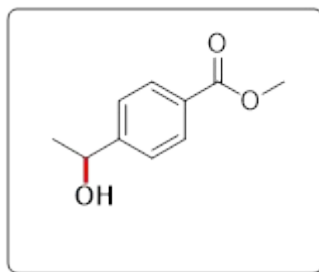
7.9899  
7.9694  
7.4221  
7.4037  
7.2598

4.9552  
4.9390  
4.9228  
4.9065

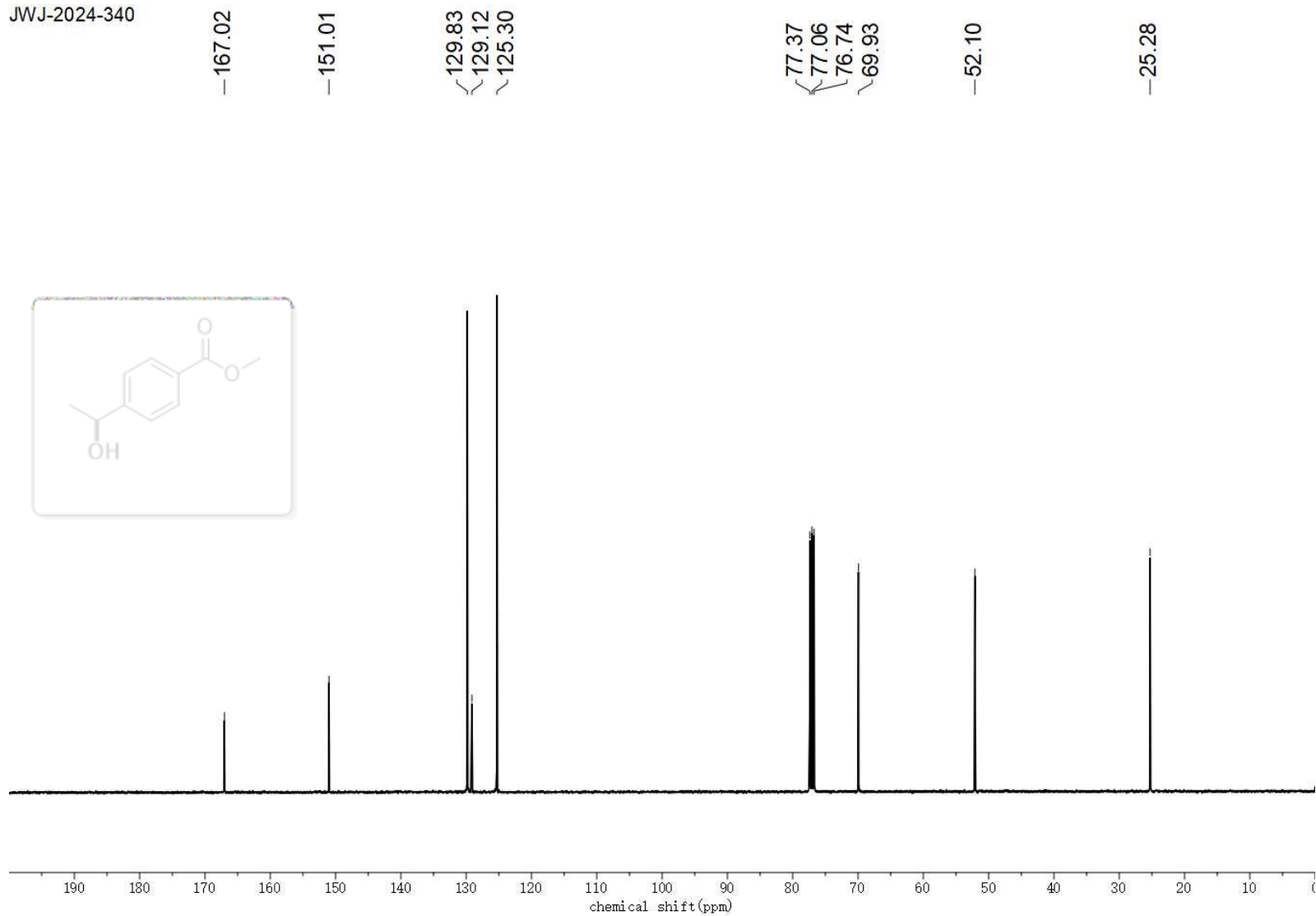
3.8889

2.2077

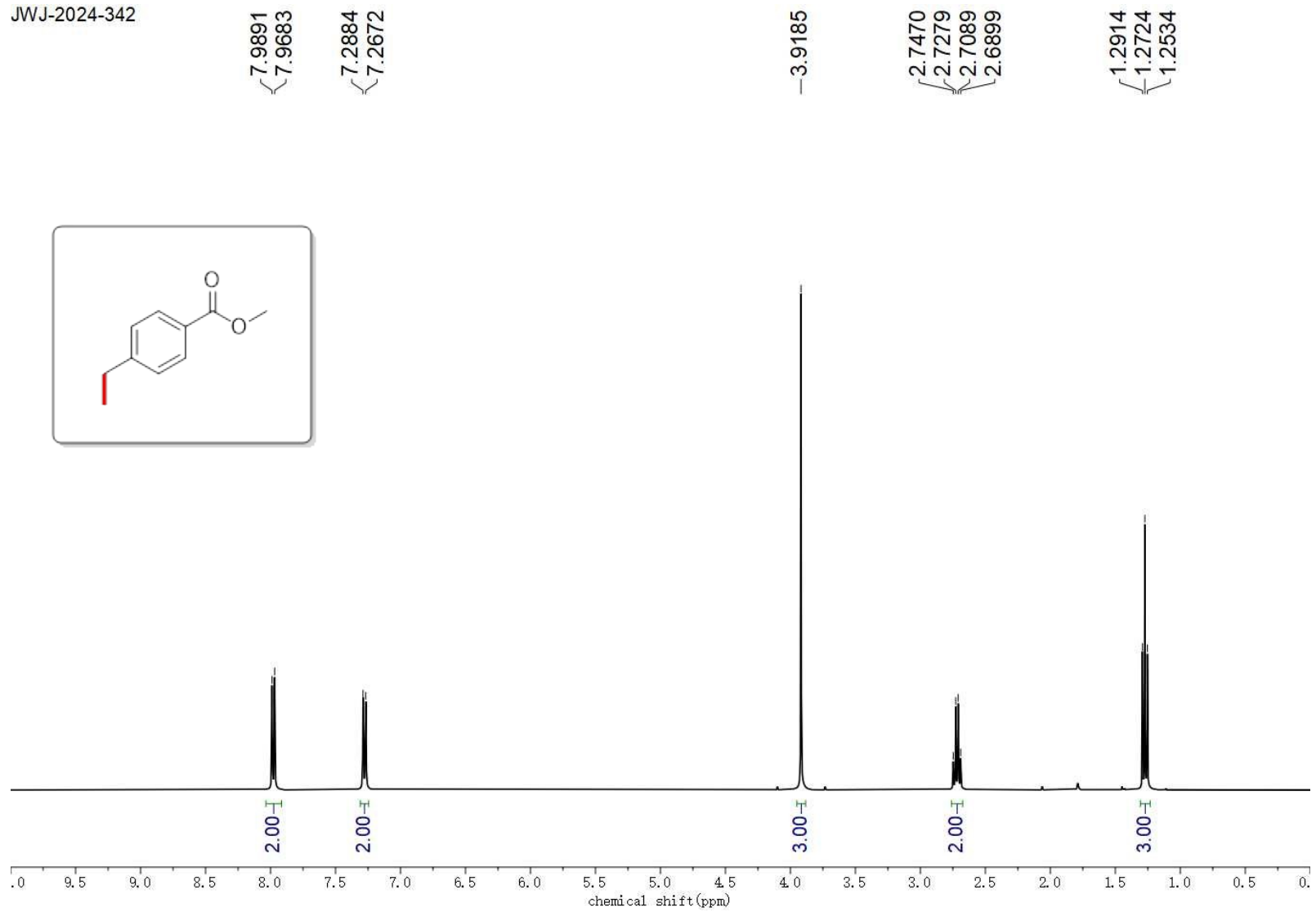
1.4869  
1.4708



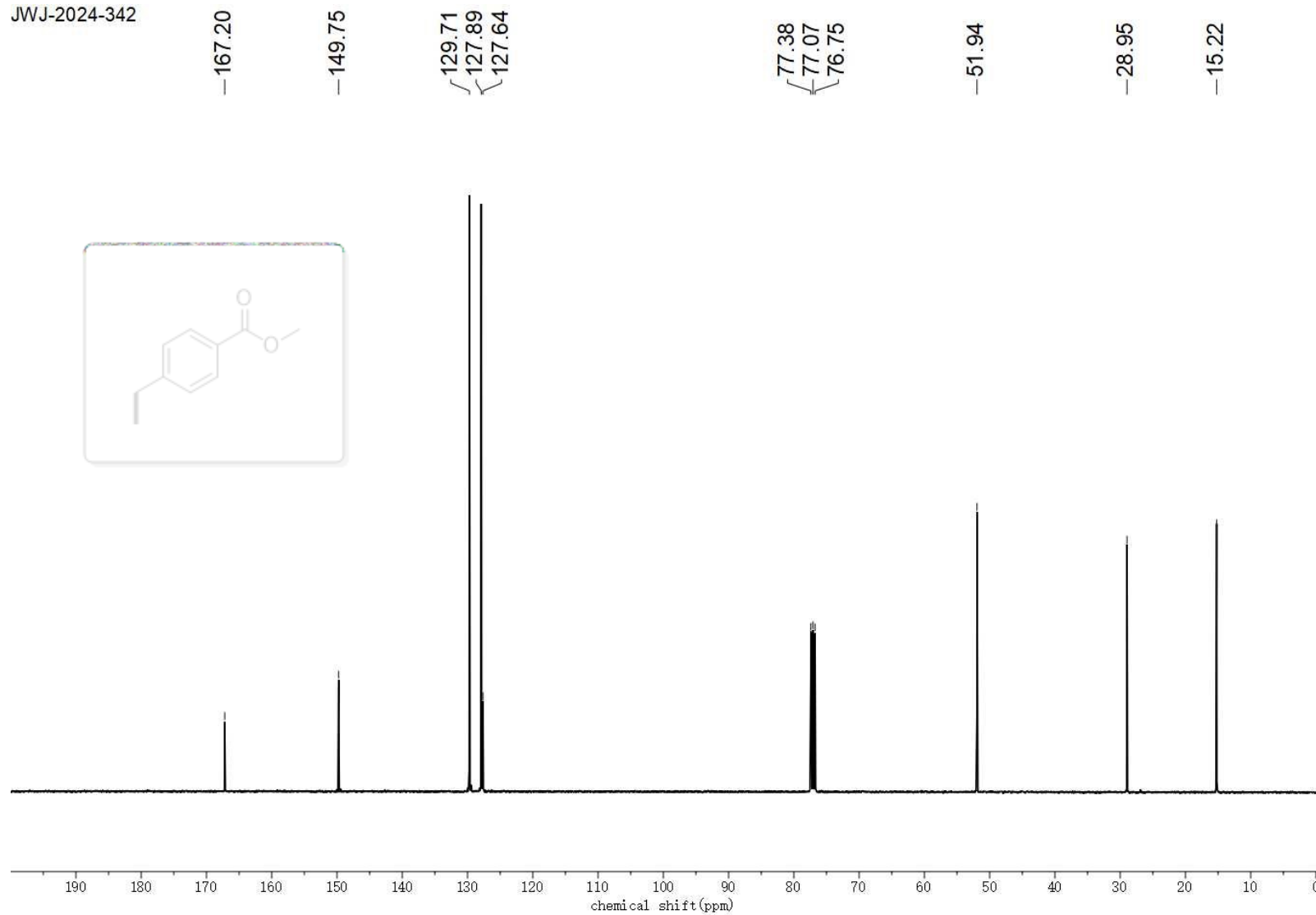
JWJ-2024-340



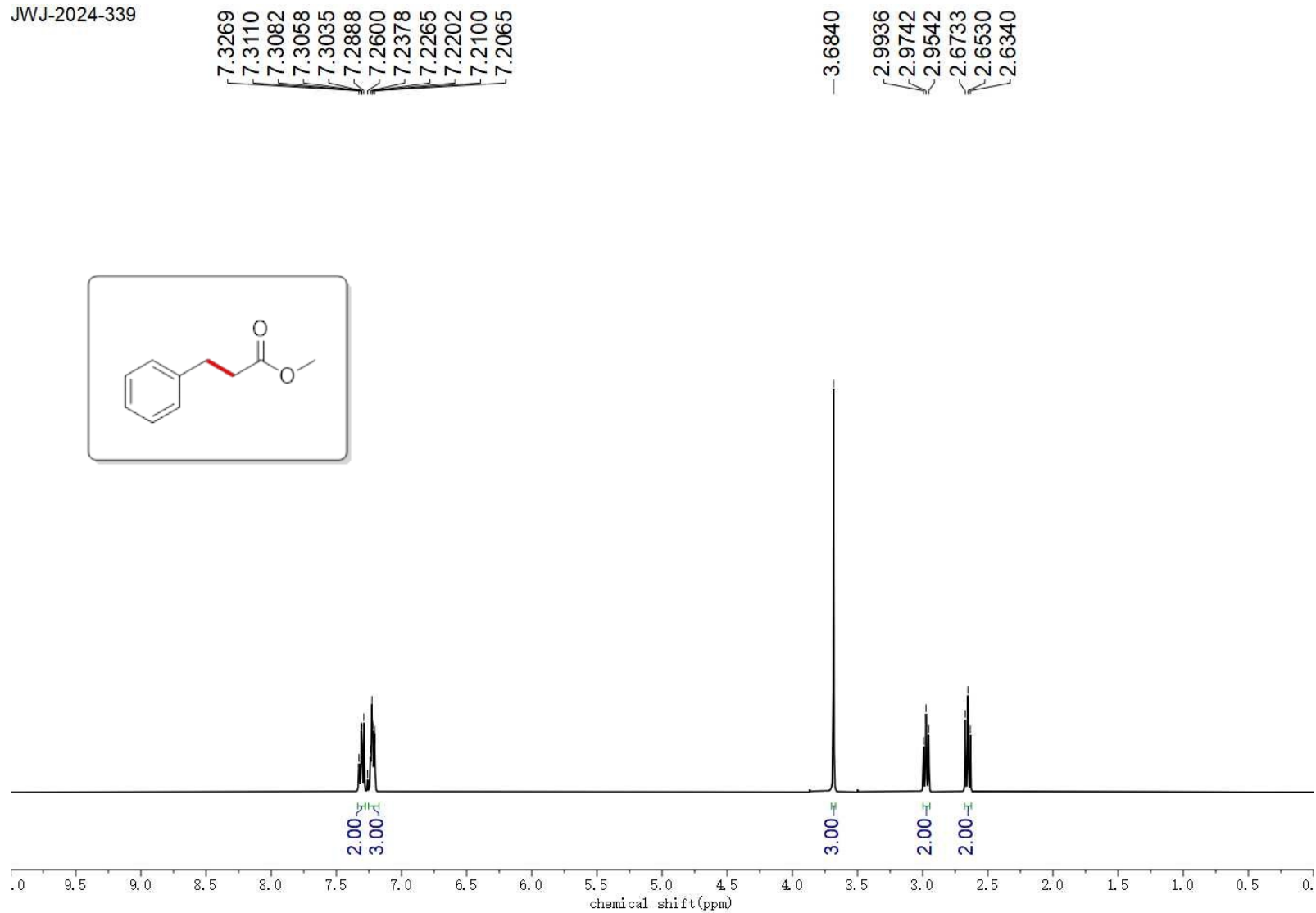
JWJ-2024-342



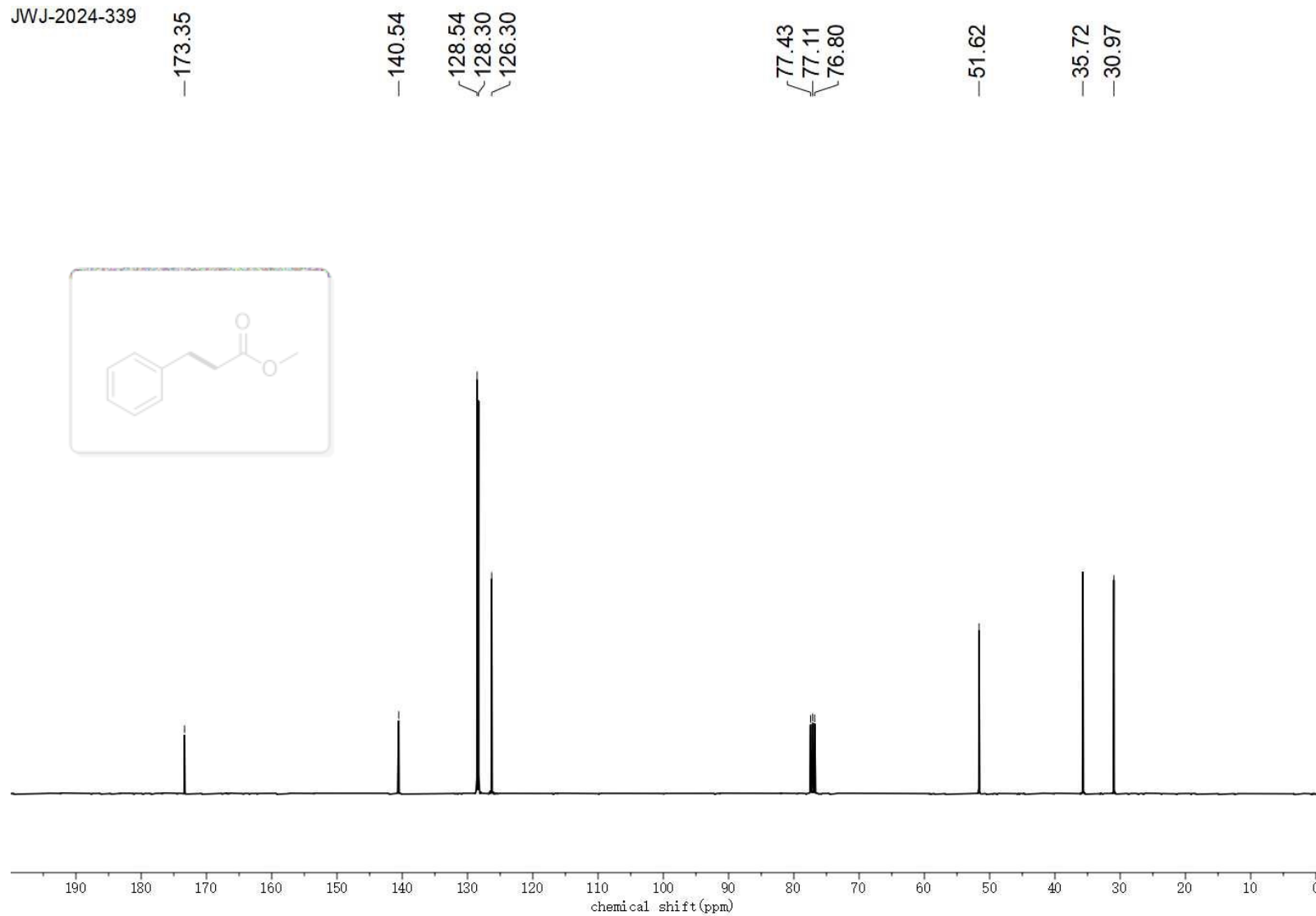
JWJ-2024-342



JWJ-2024-339

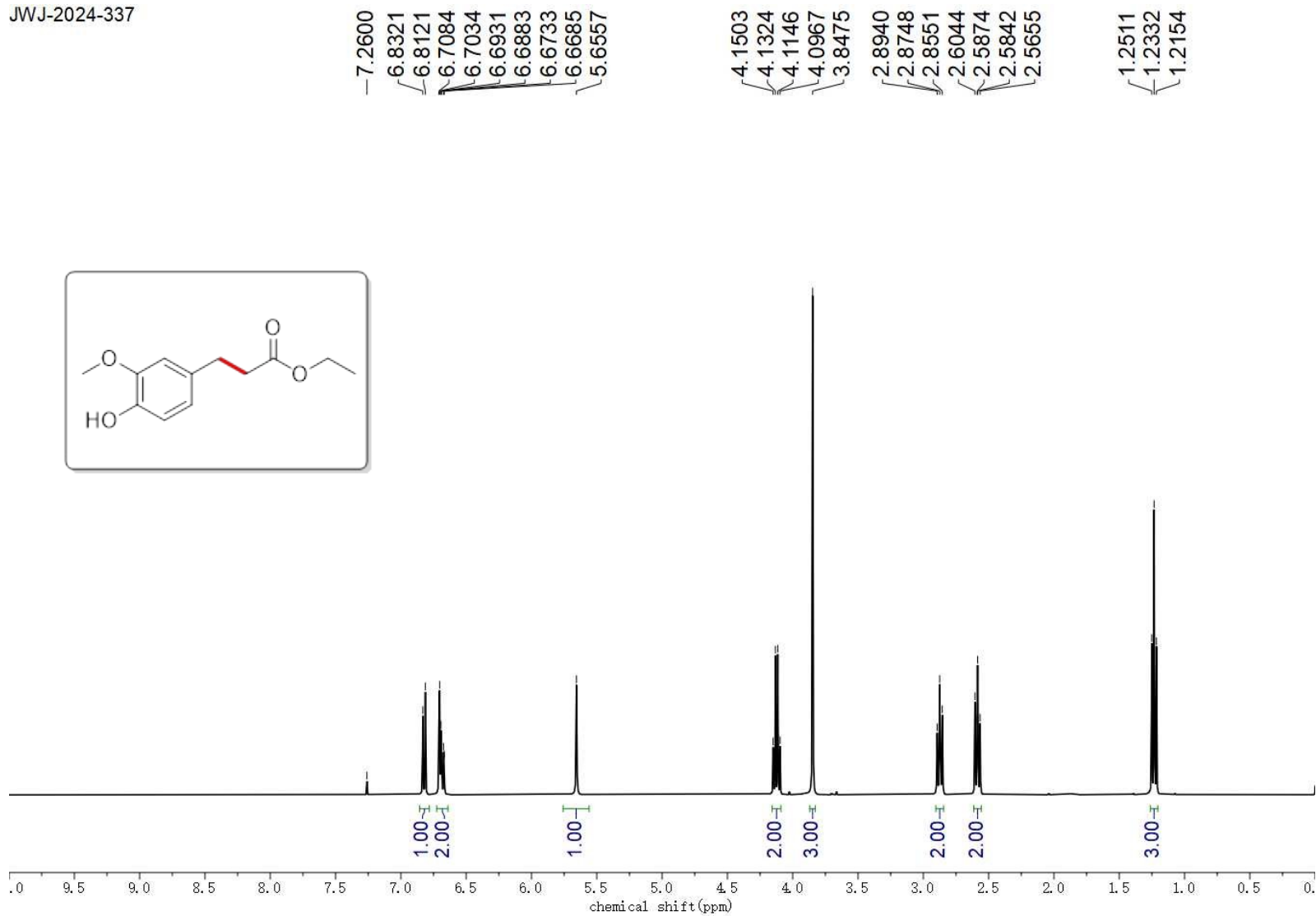


JWJ-2024-339

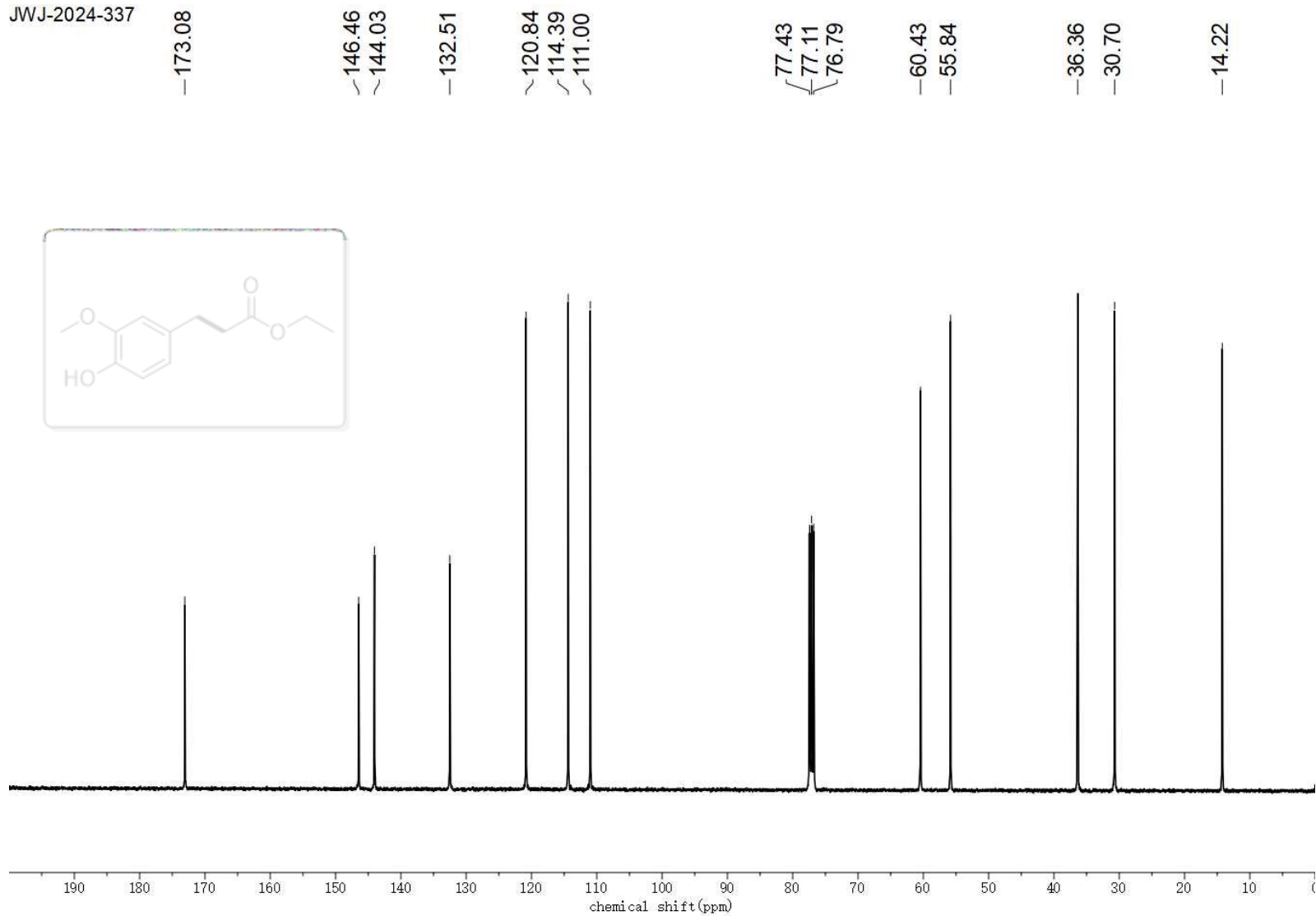




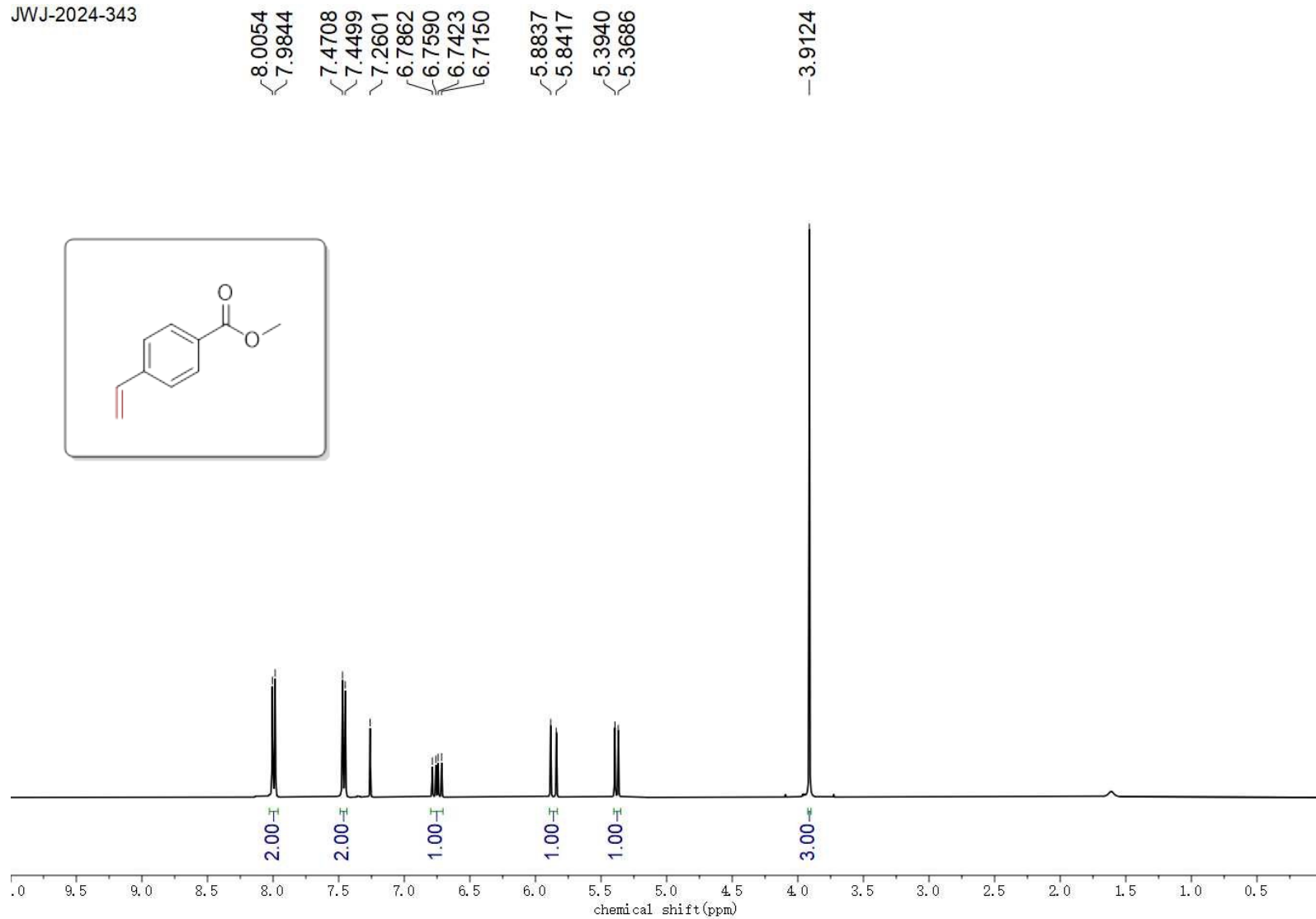
JWJ-2024-337



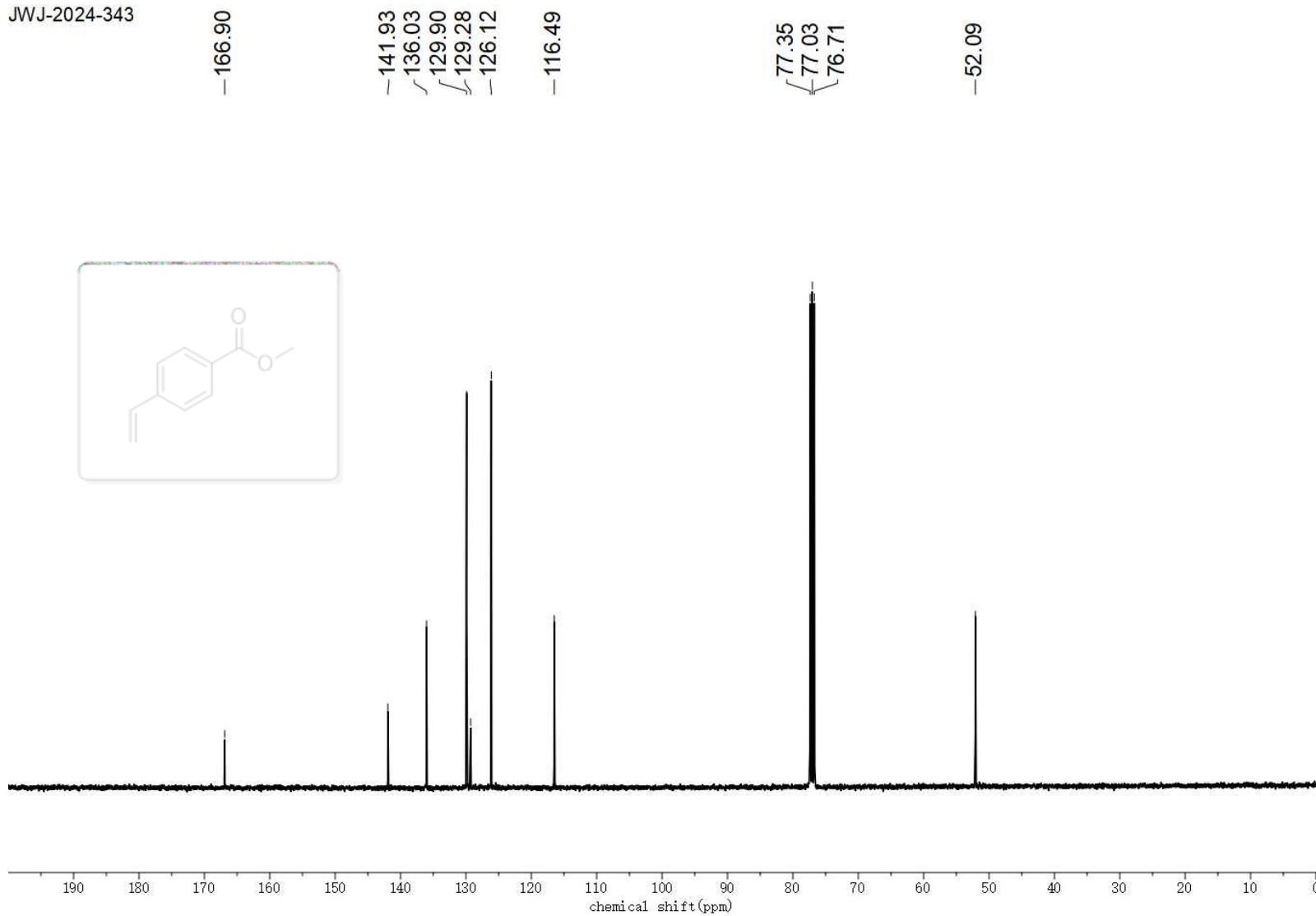
JWJ-2024-337



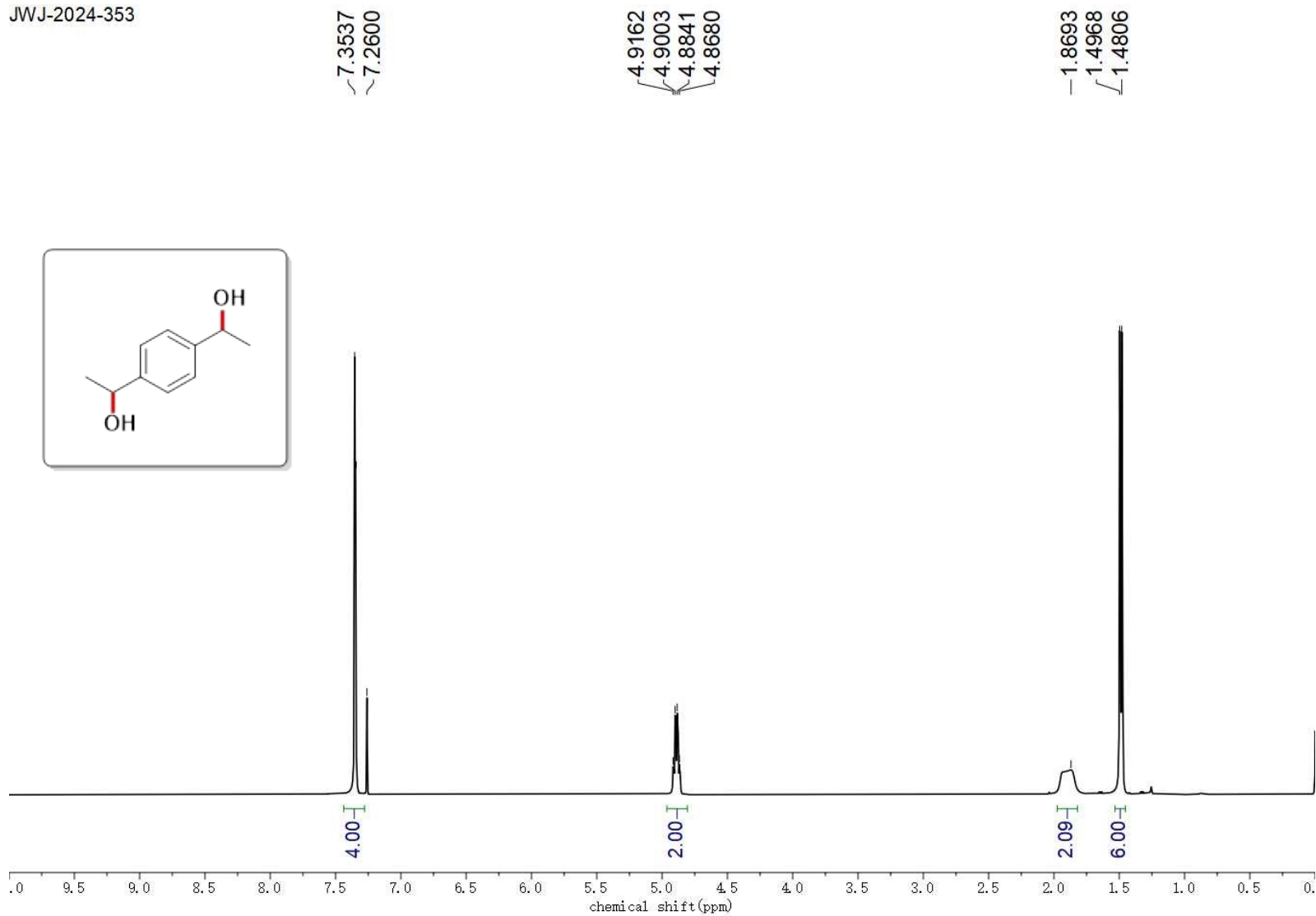
JWJ-2024-343



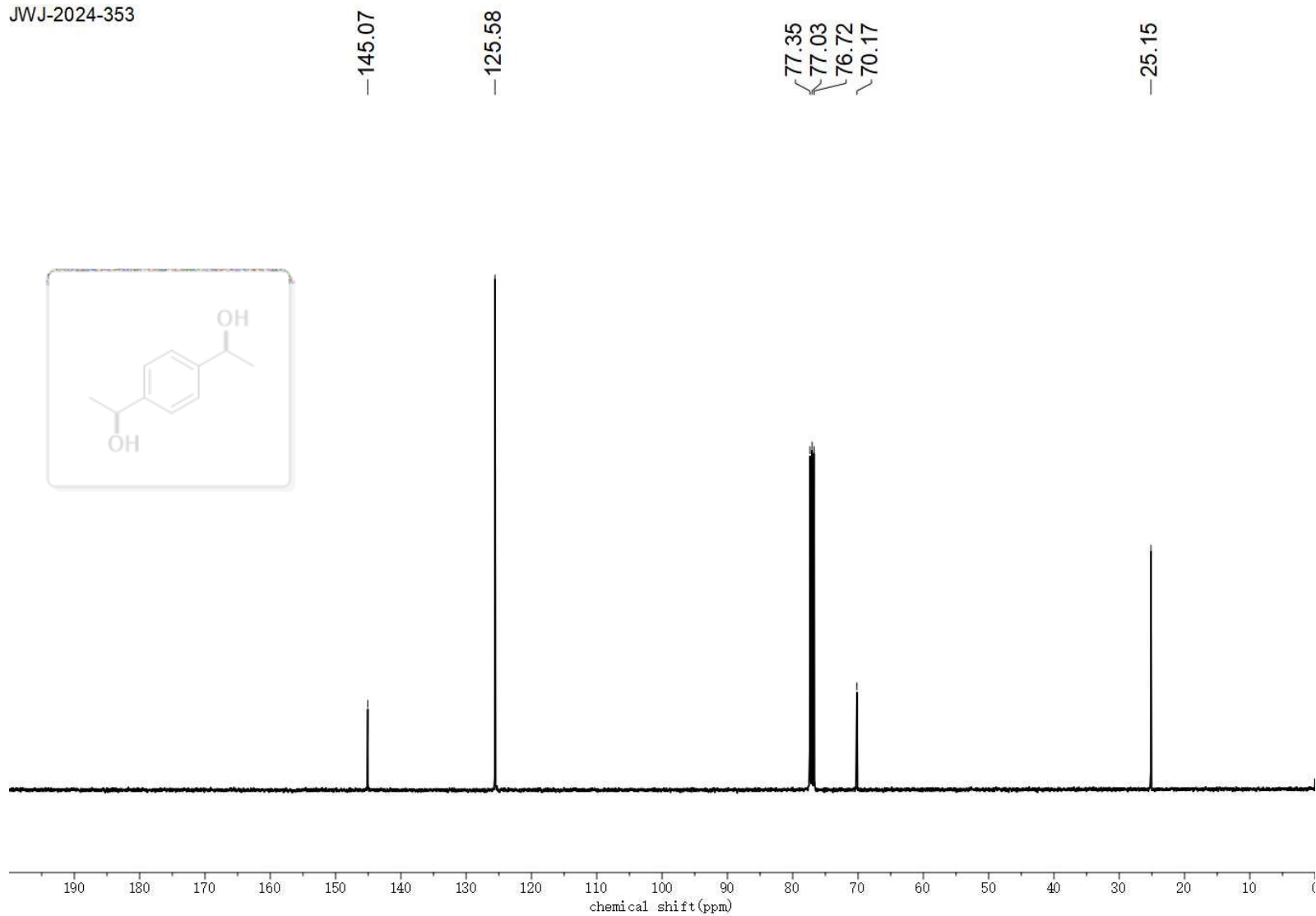
JWJ-2024-343



JWJ-2024-353



JWJ-2024-353

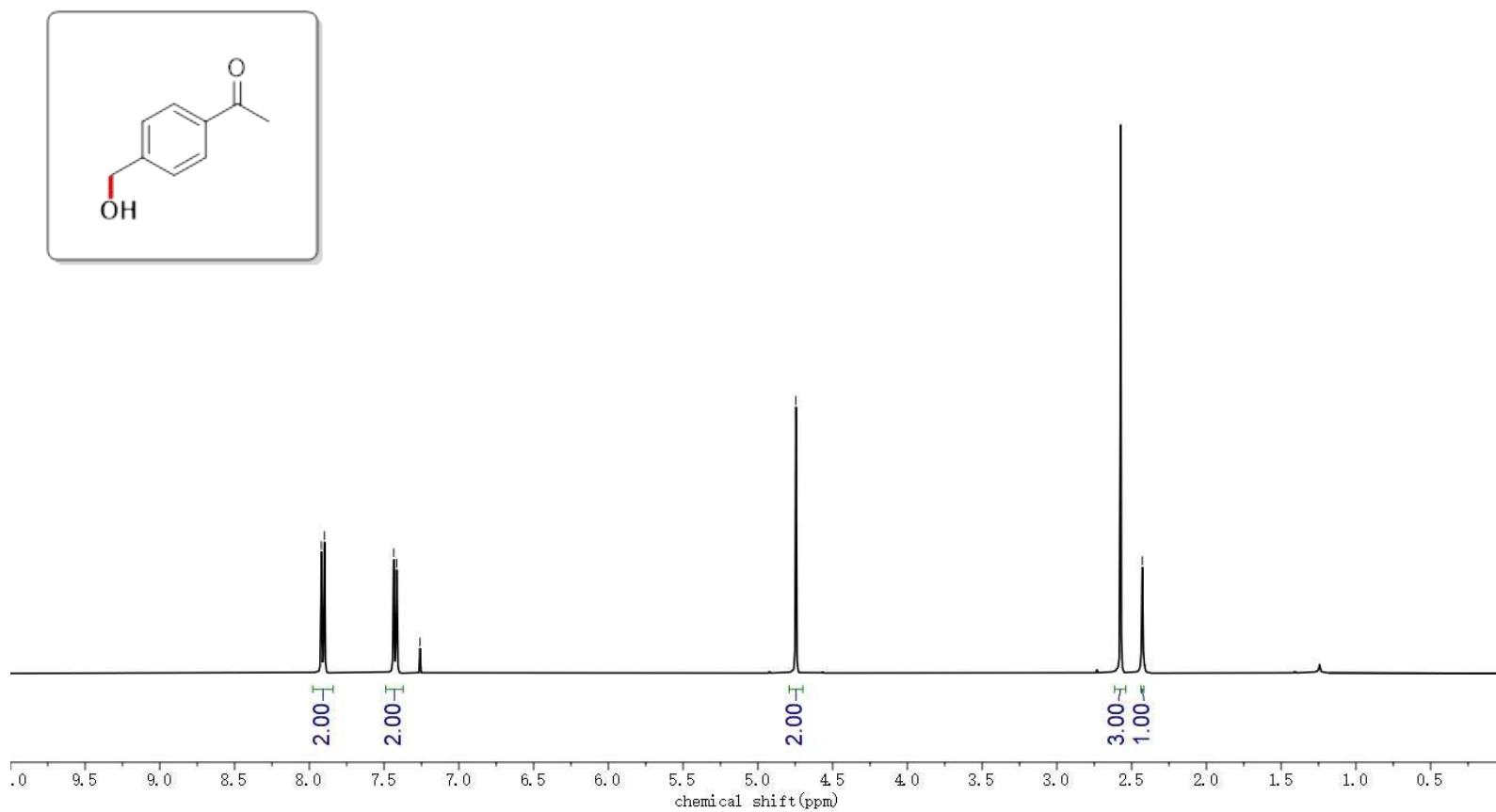
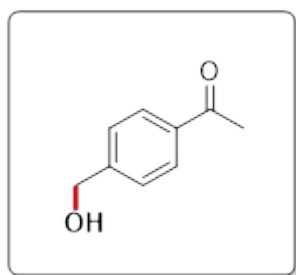


JWJ-2024-430

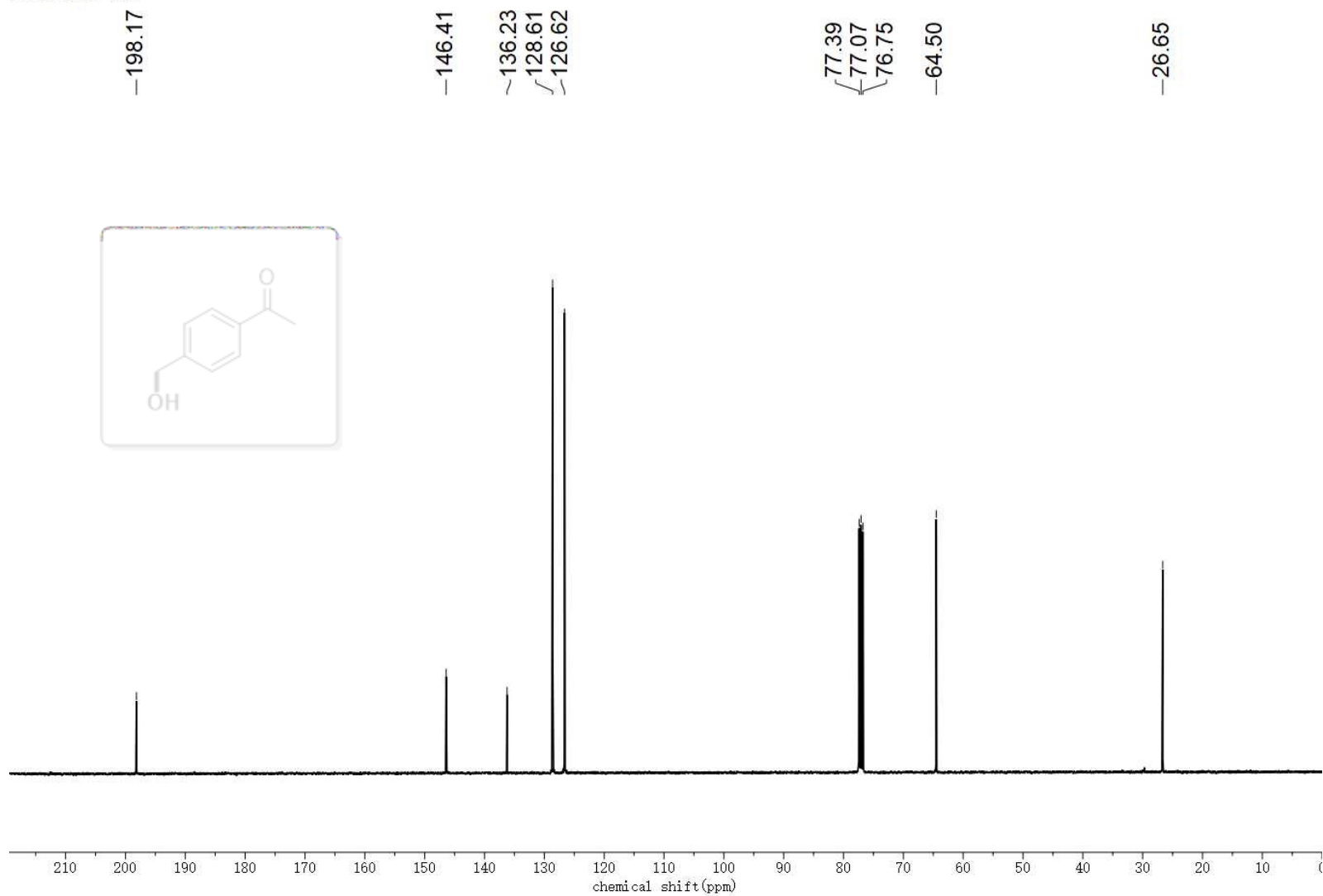
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7.8989  
7.4357  
7.4156  
7.2602

4.7457

2.5738  
2.4283

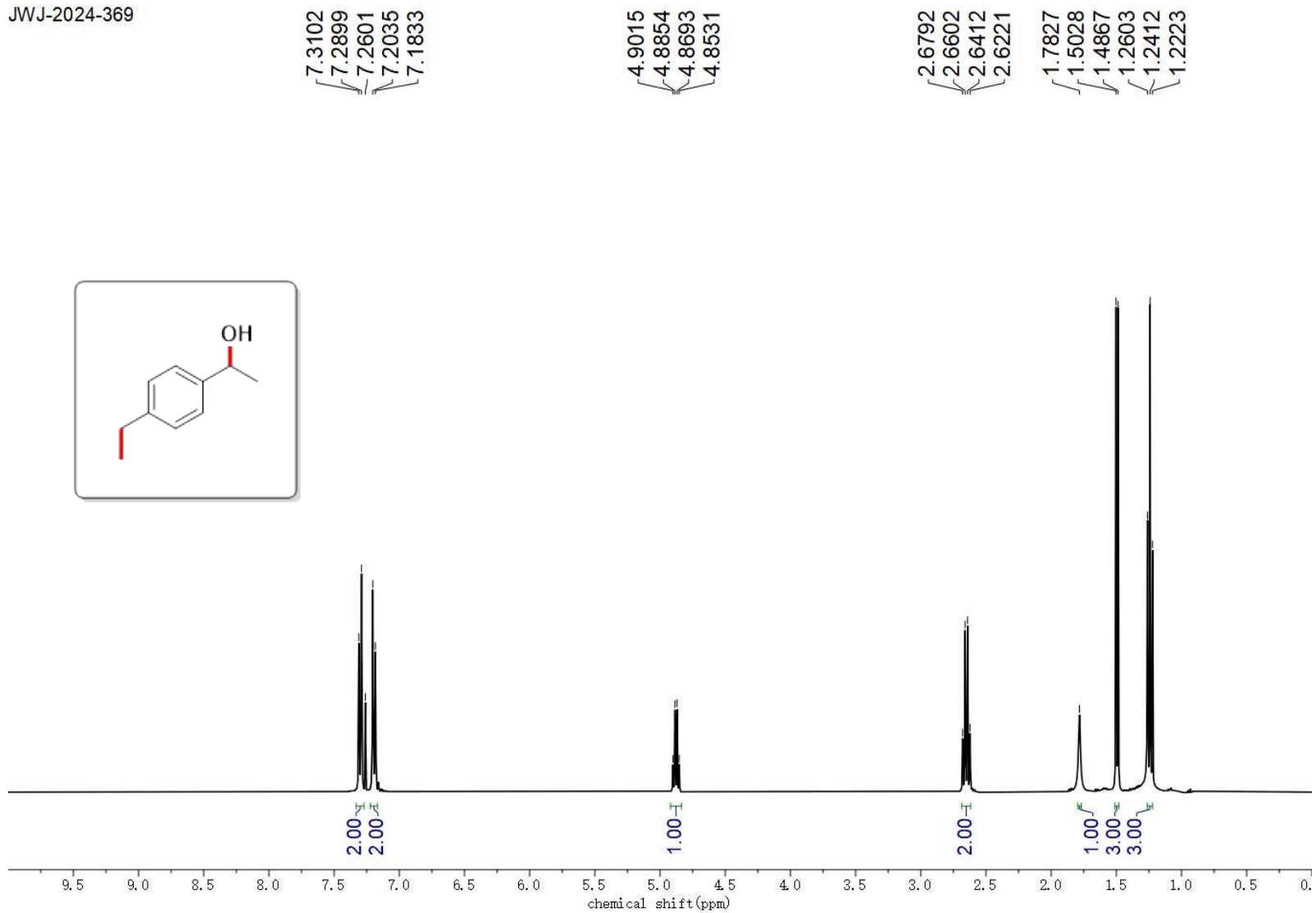


JWJ-2024-430

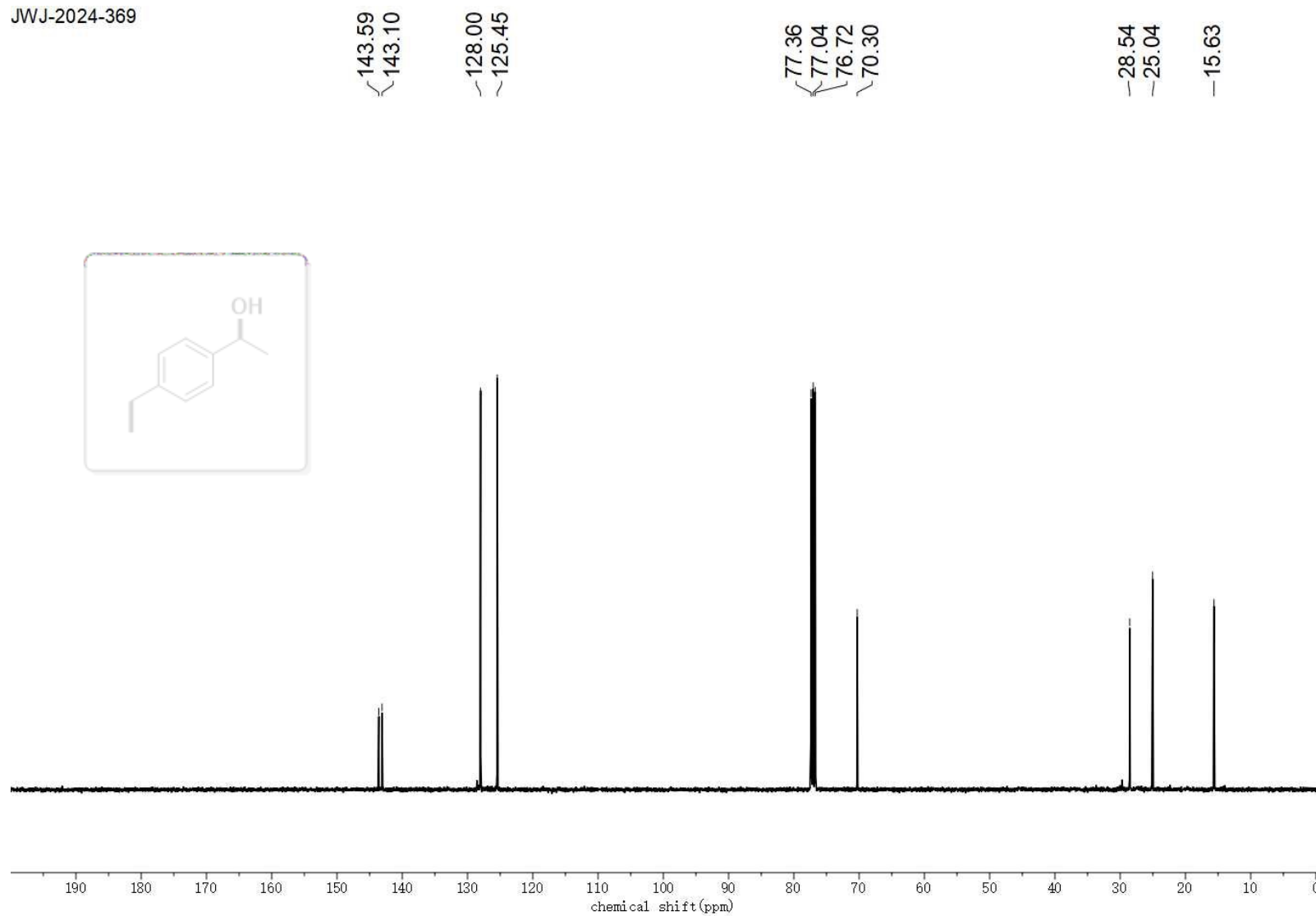




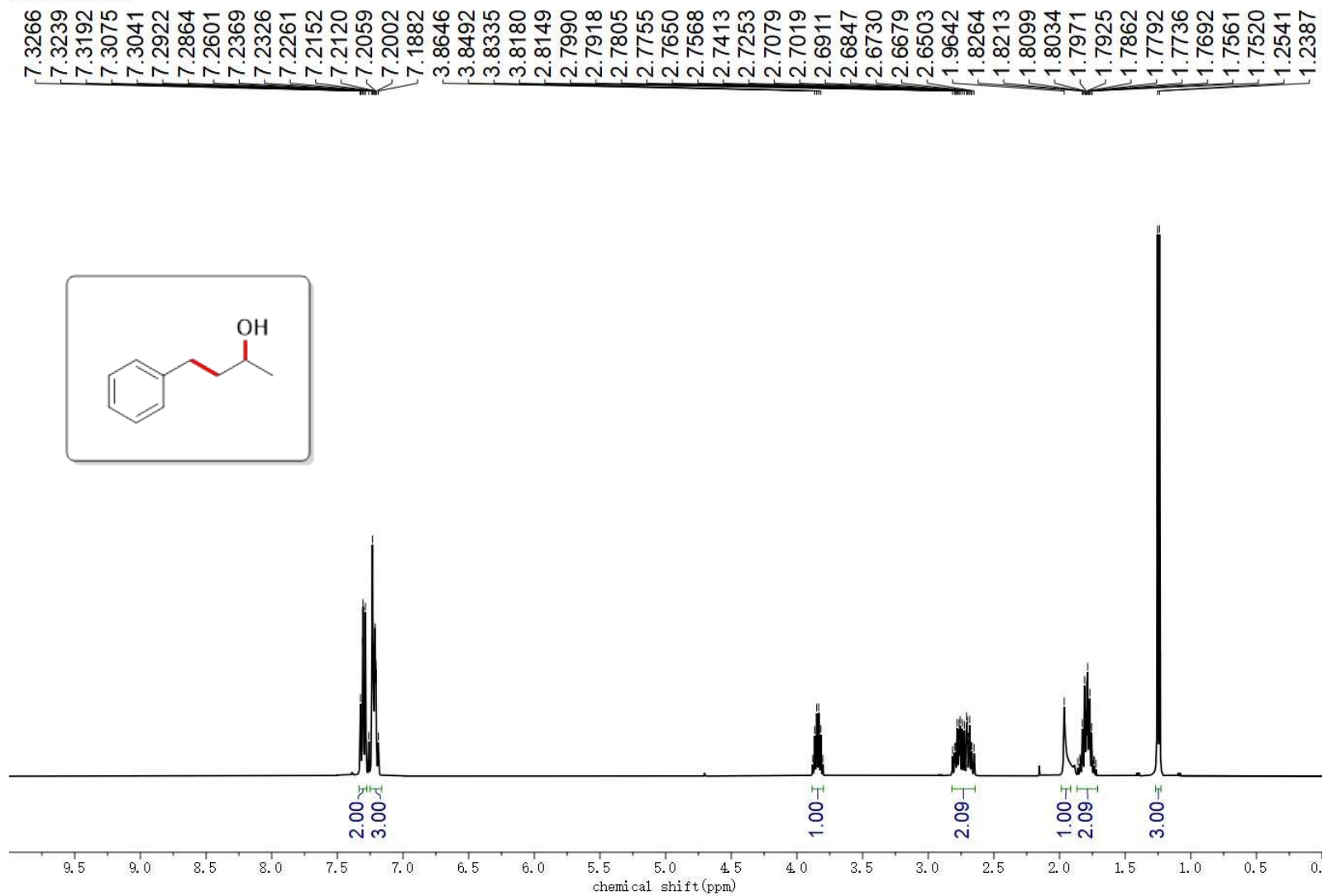
JWJ-2024-369



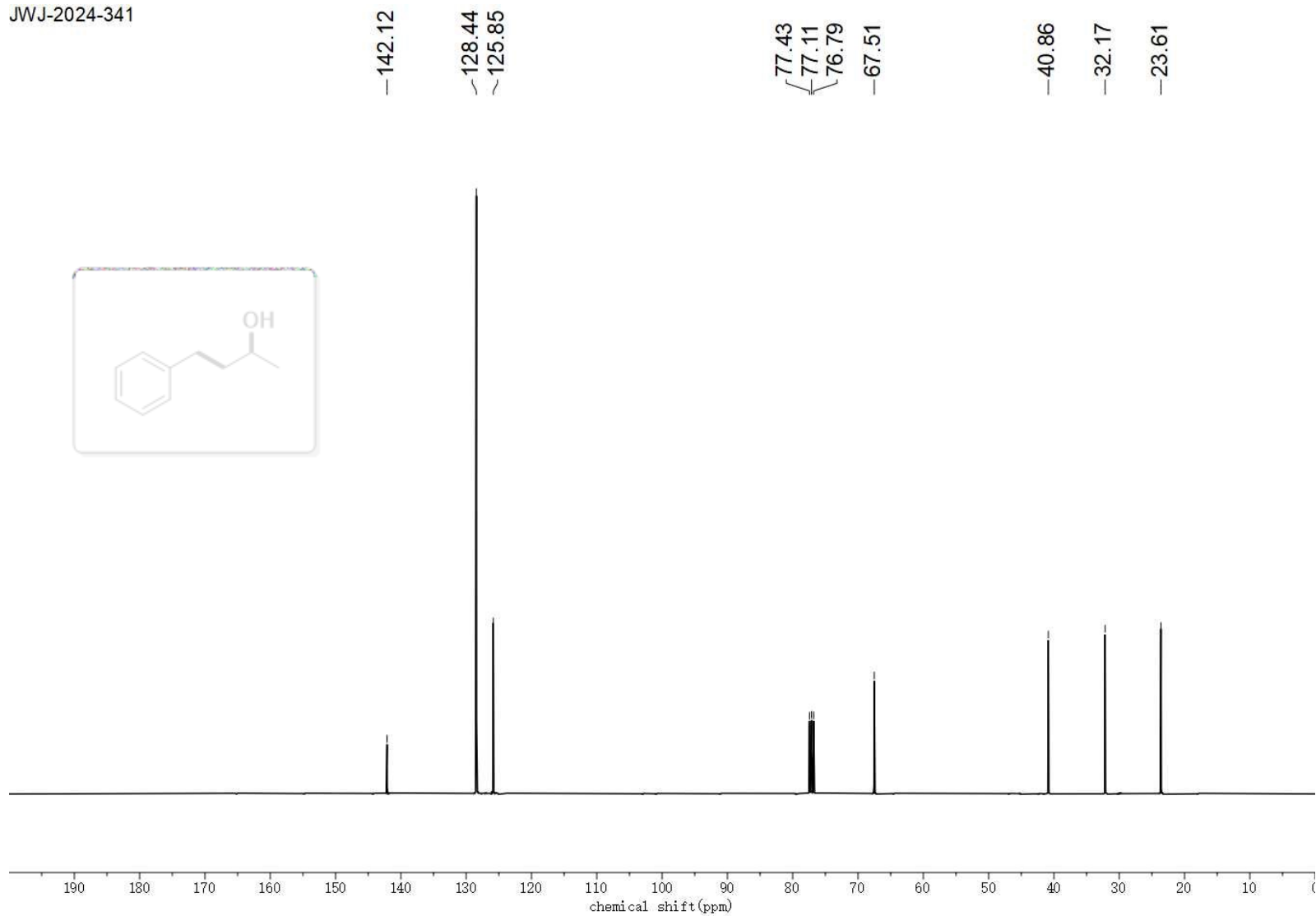
JWJ-2024-369



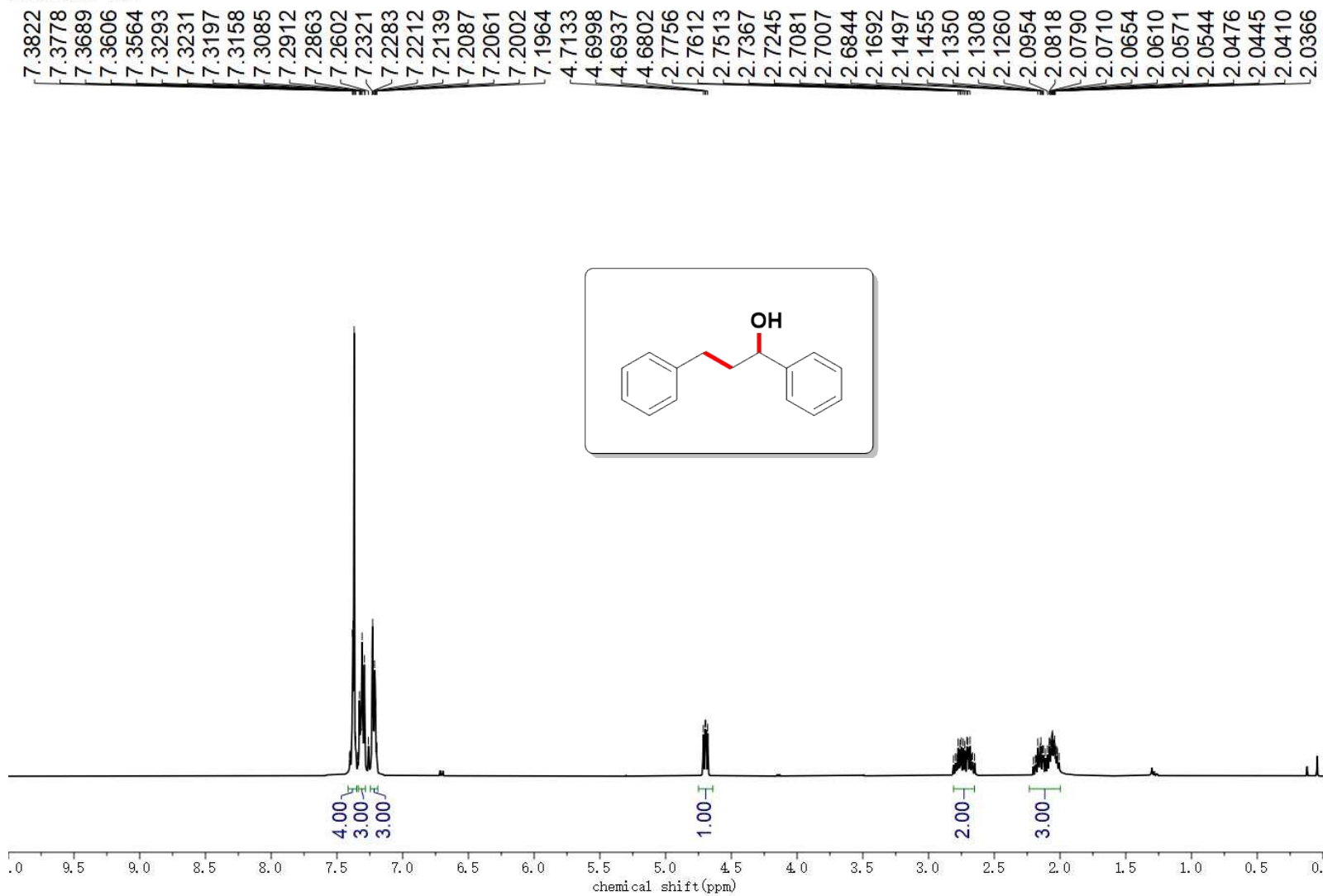
JWJ-2024-341



JWJ-2024-341



JWJ-2024-439



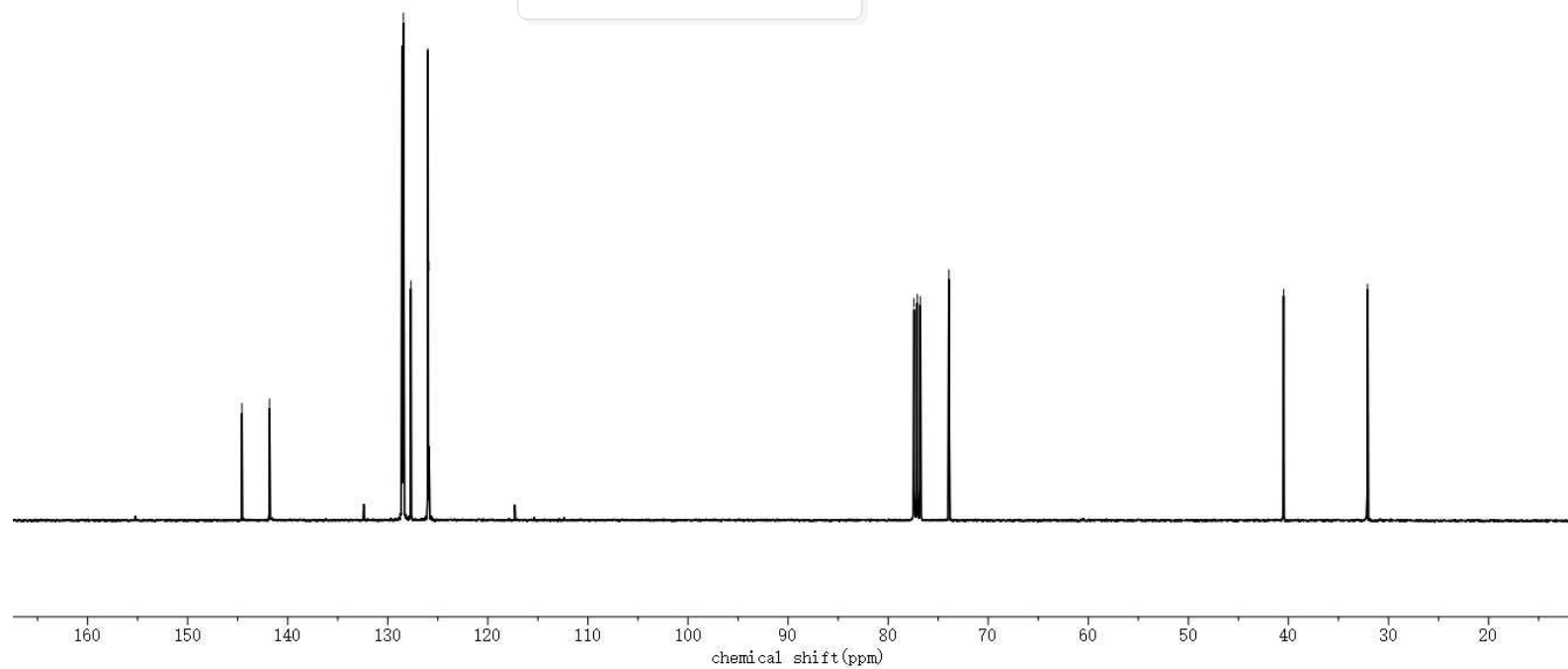
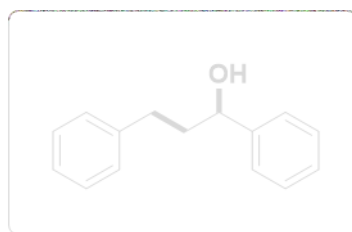
JWJ-2024-439

144.58  
141.82

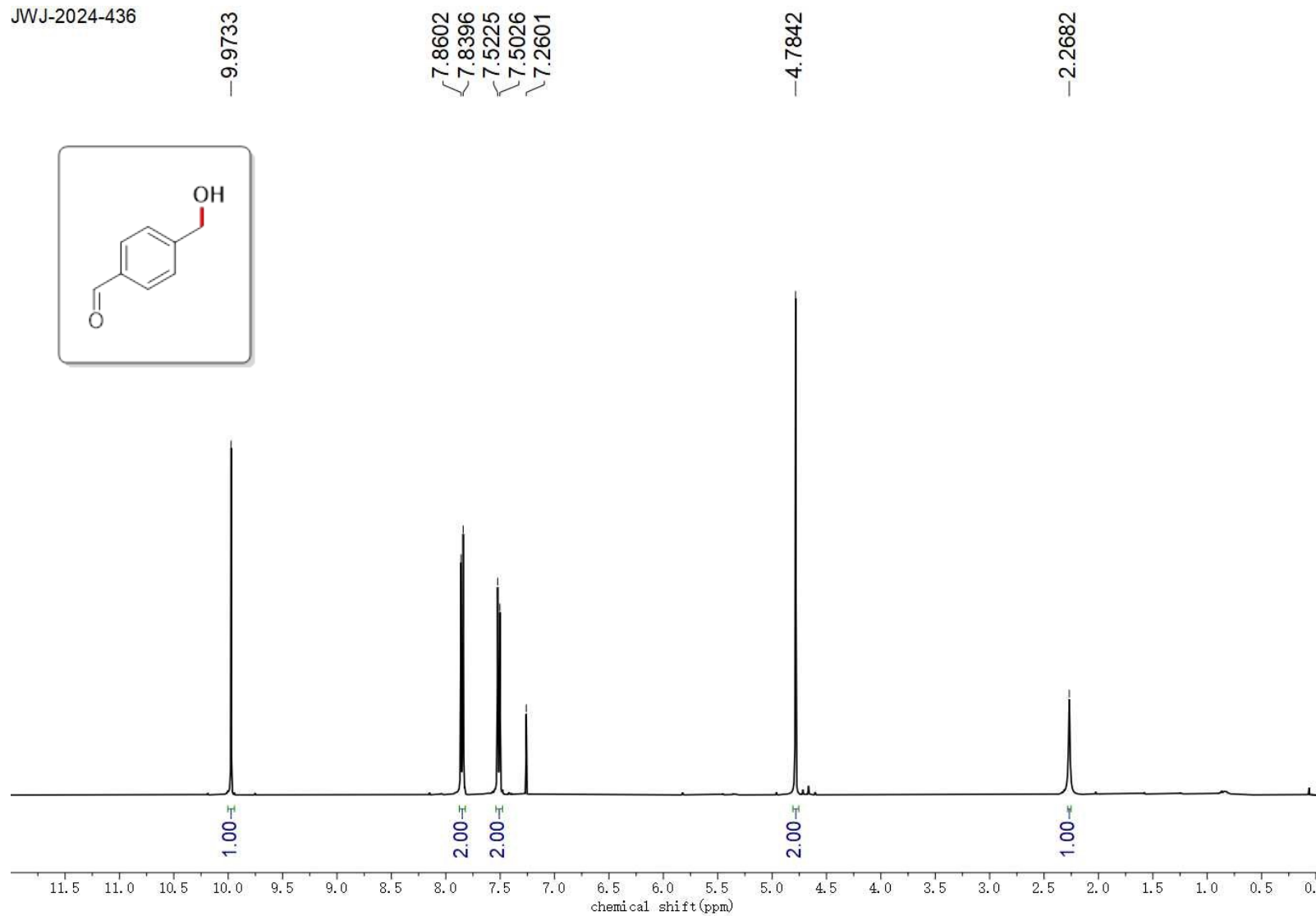
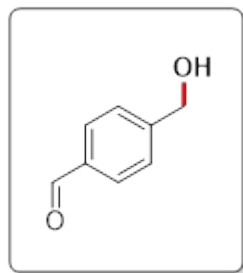
128.56  
128.49  
128.44  
127.69  
125.99  
125.91

77.42  
77.10  
76.78  
73.93

40.48  
32.09



JWJ-2024-436



JWJ-2024-436

-192.16

-147.89

-135.63

-130.04

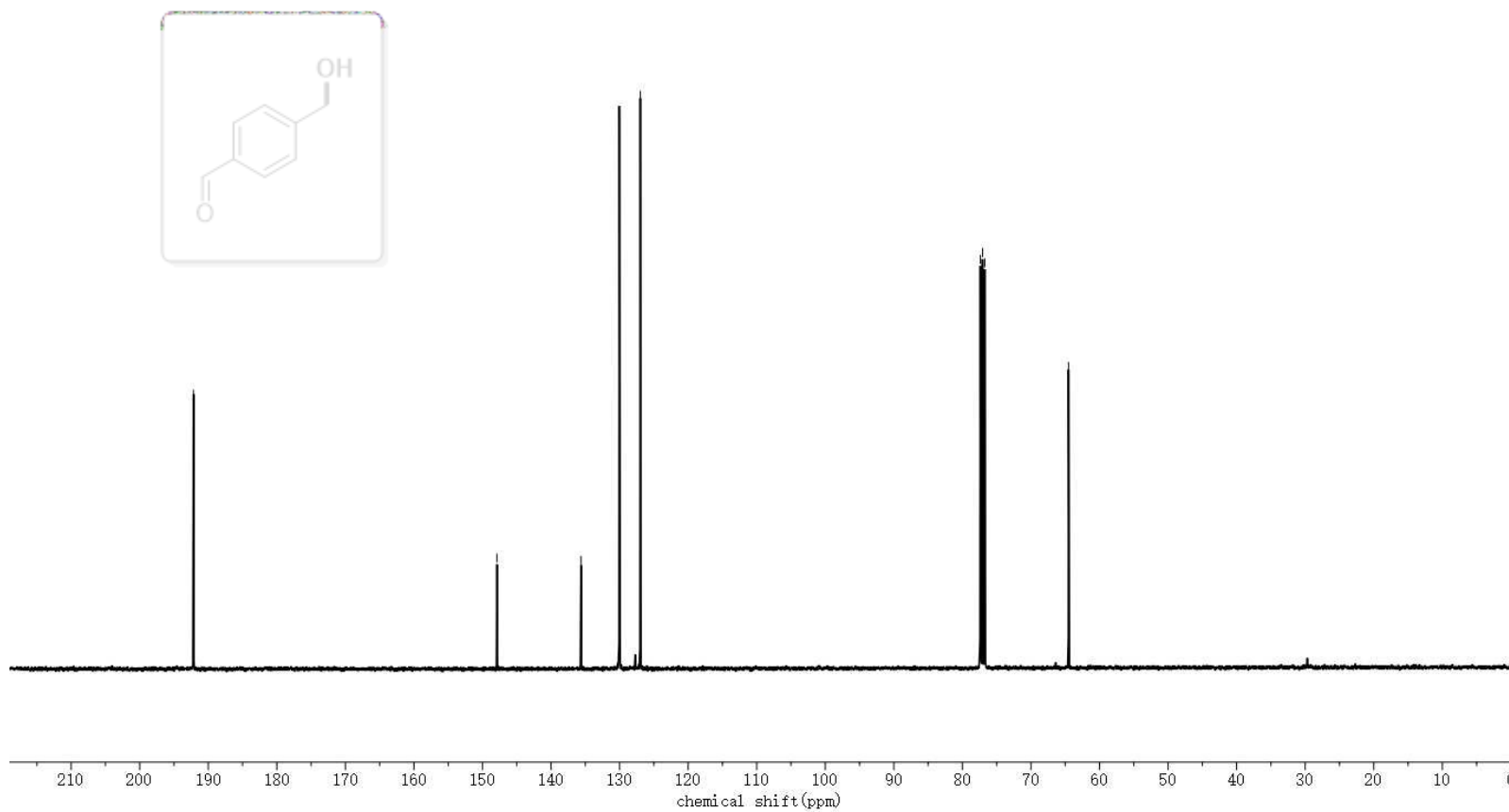
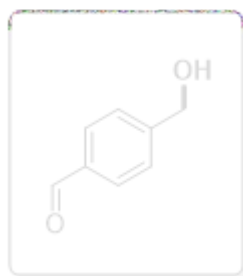
-126.97

77.37

77.06

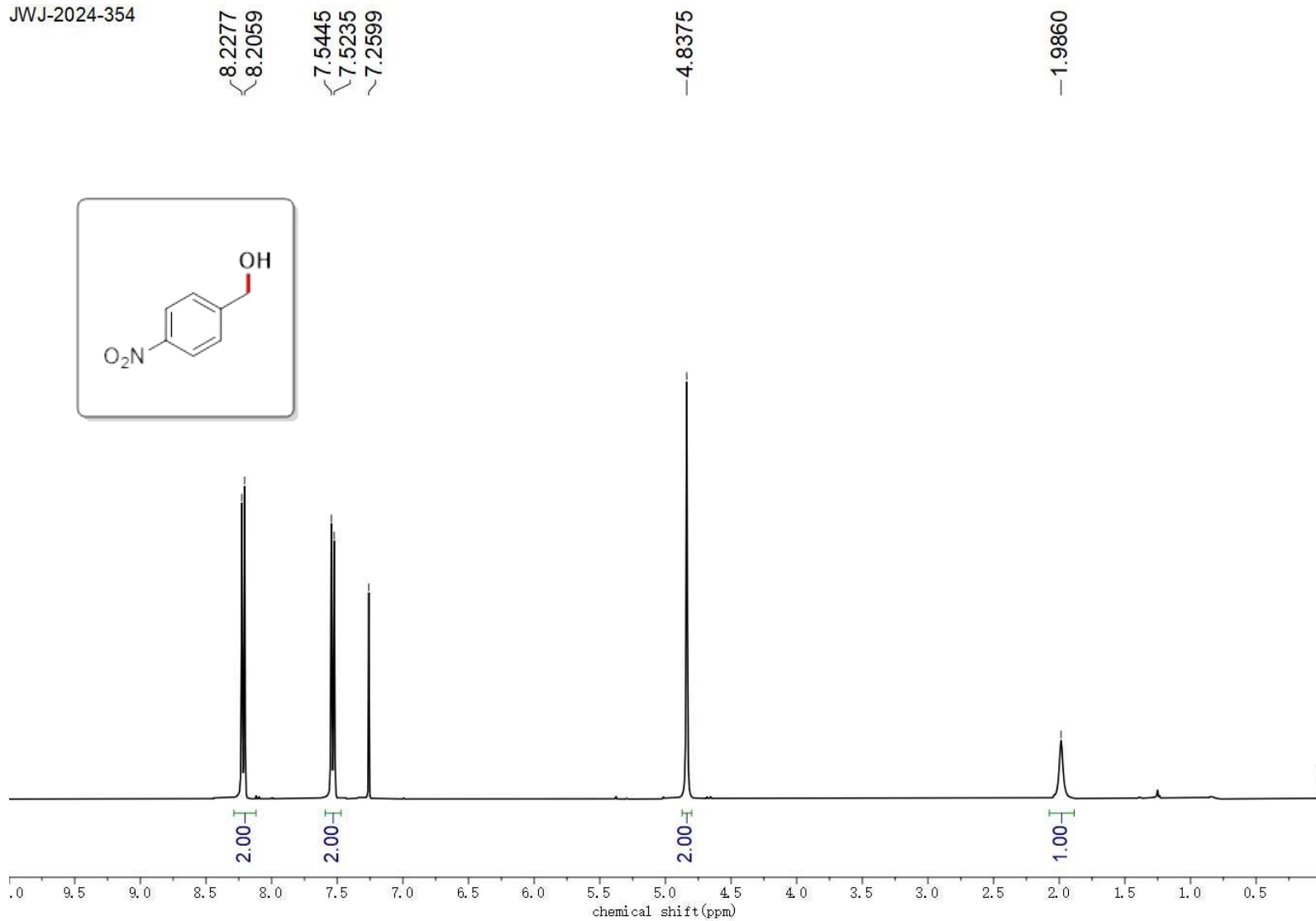
76.74

-64.52

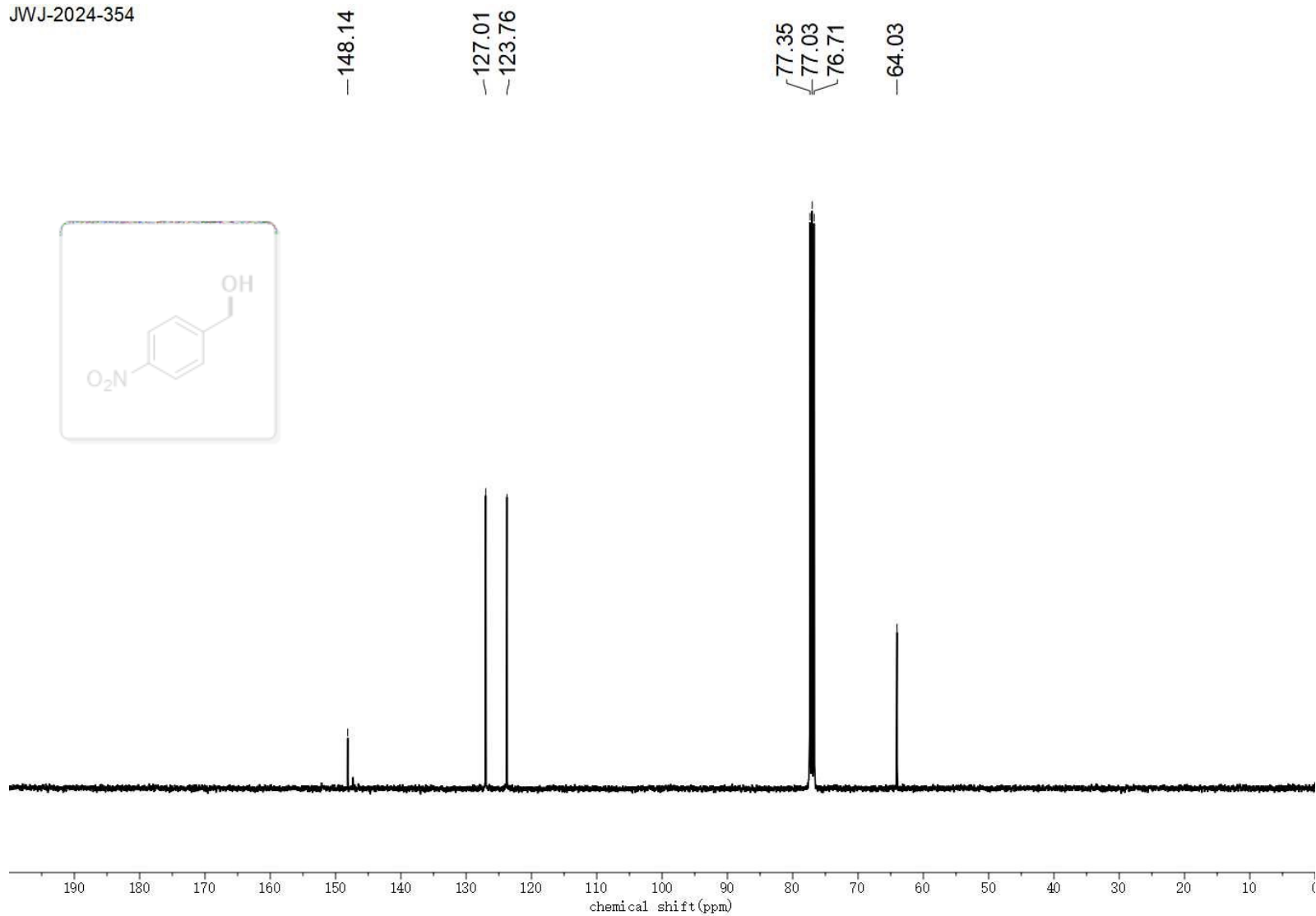




JWJ-2024-354



JWJ-2024-354

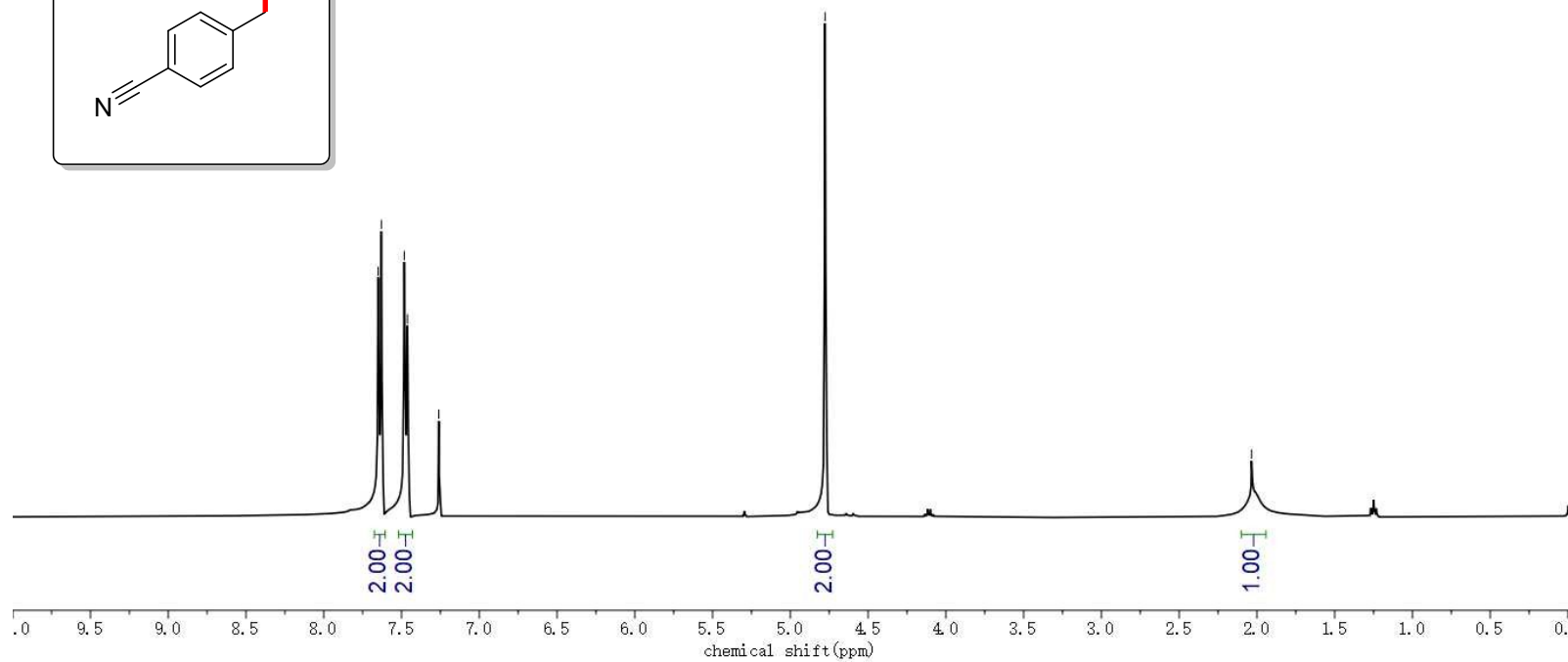
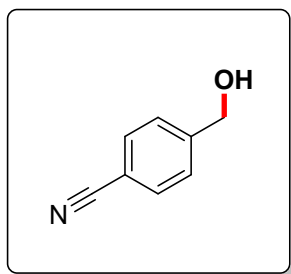


JWJ-2024-450

7.6501  
7.6295  
7.4825  
7.4620  
7.2601

4.7770

2.0347



JWJ-2024-450

-146.21

-132.34

-127.03

-118.87

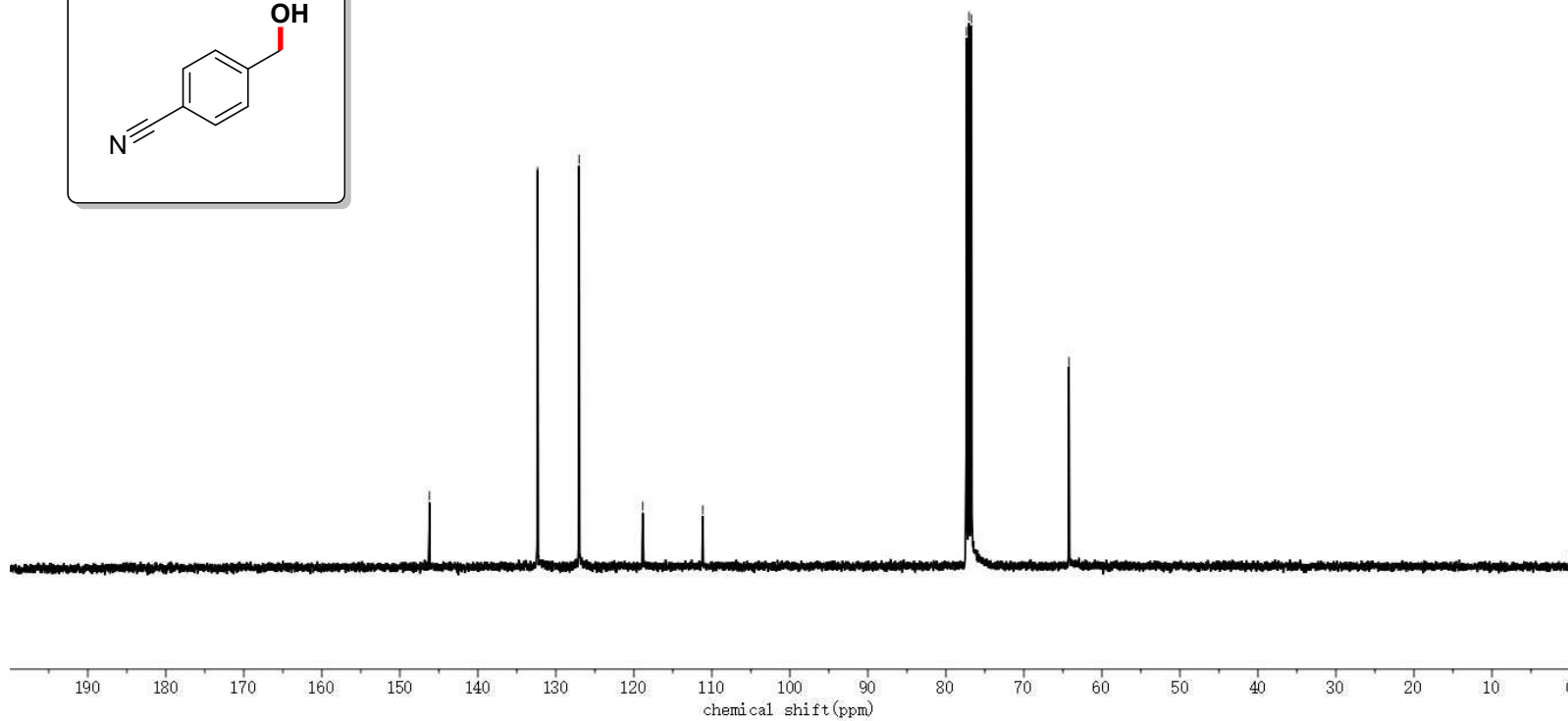
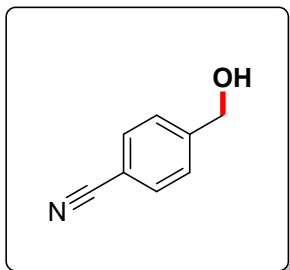
-111.17

77.36

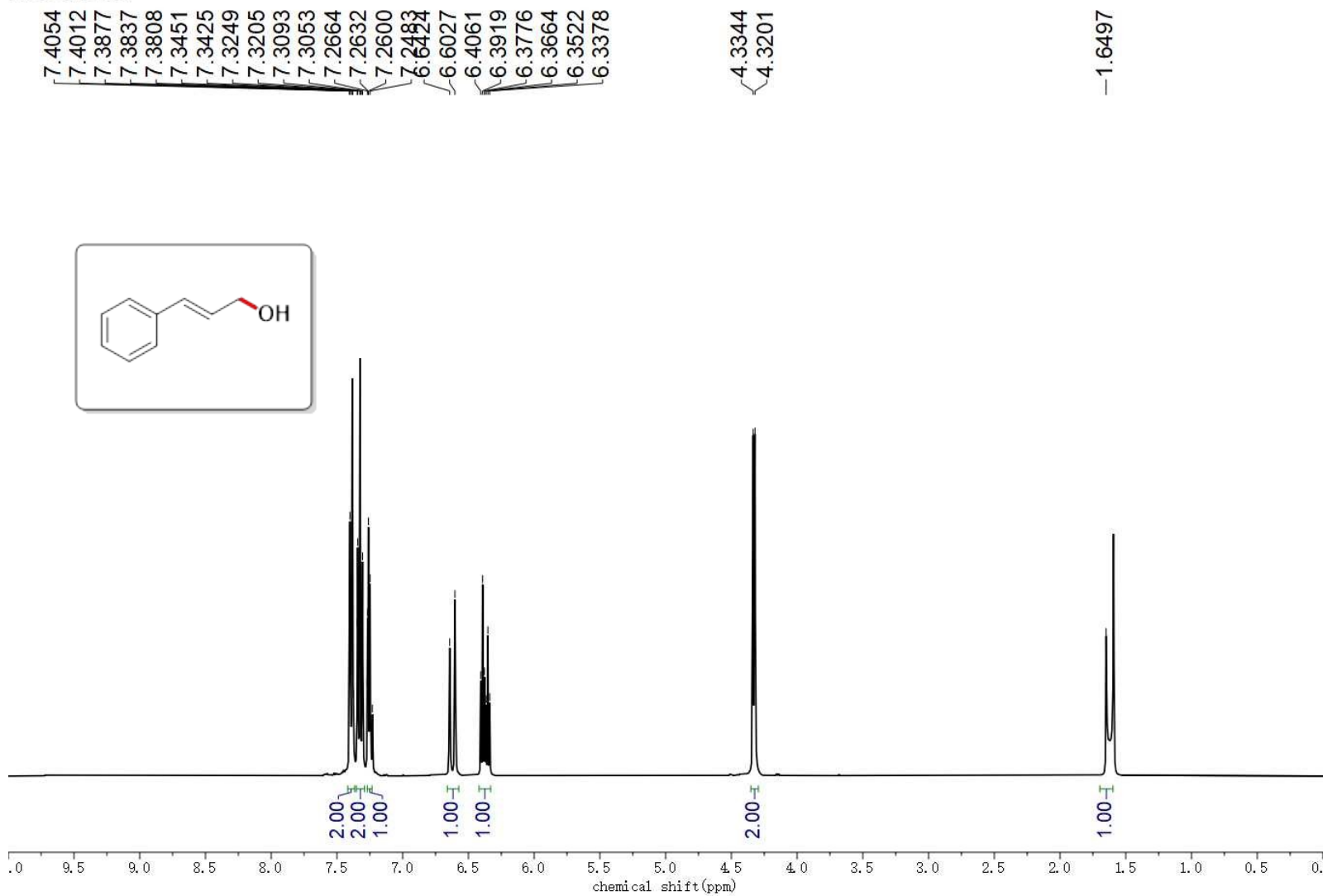
77.04

76.73

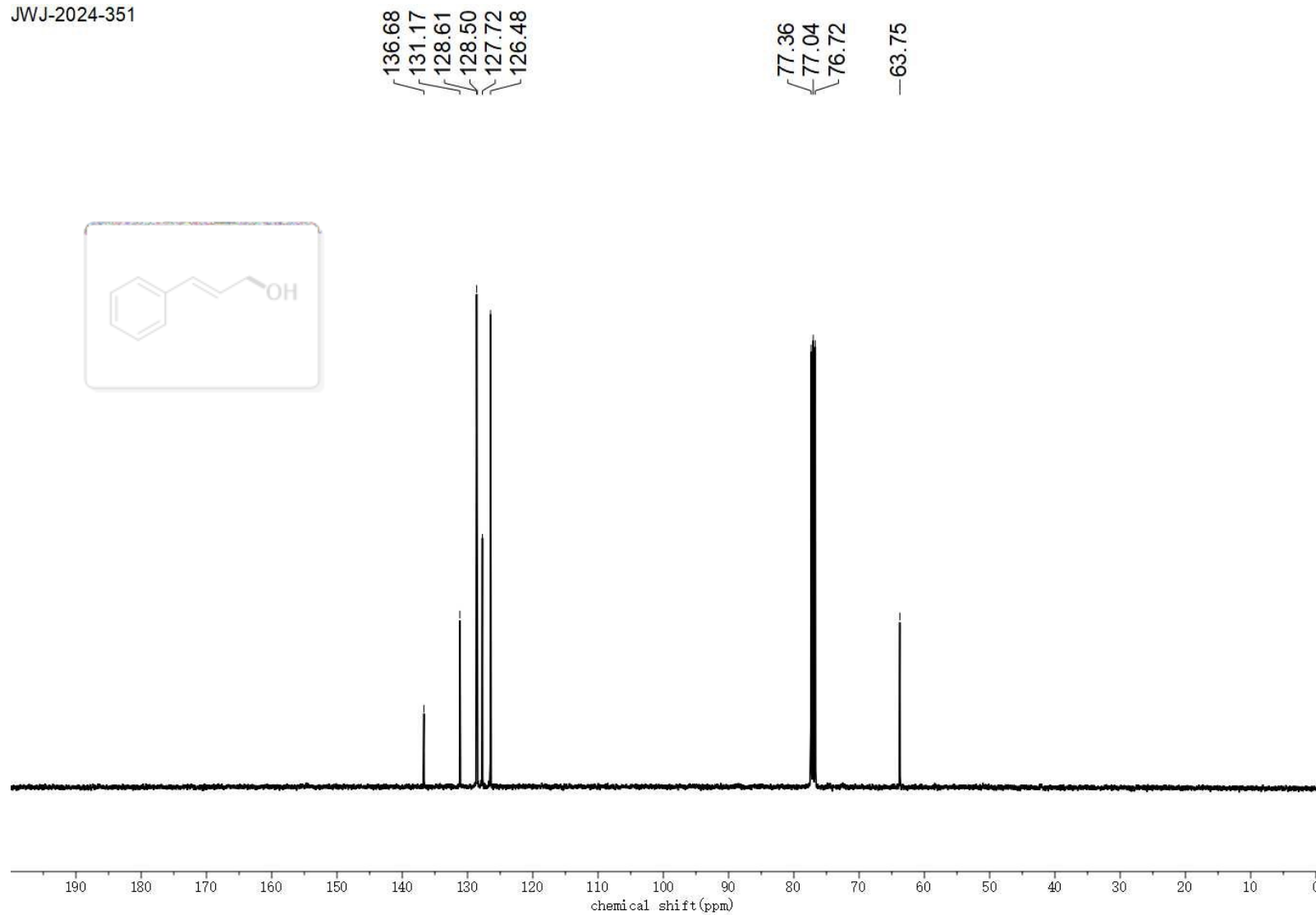
-64.23



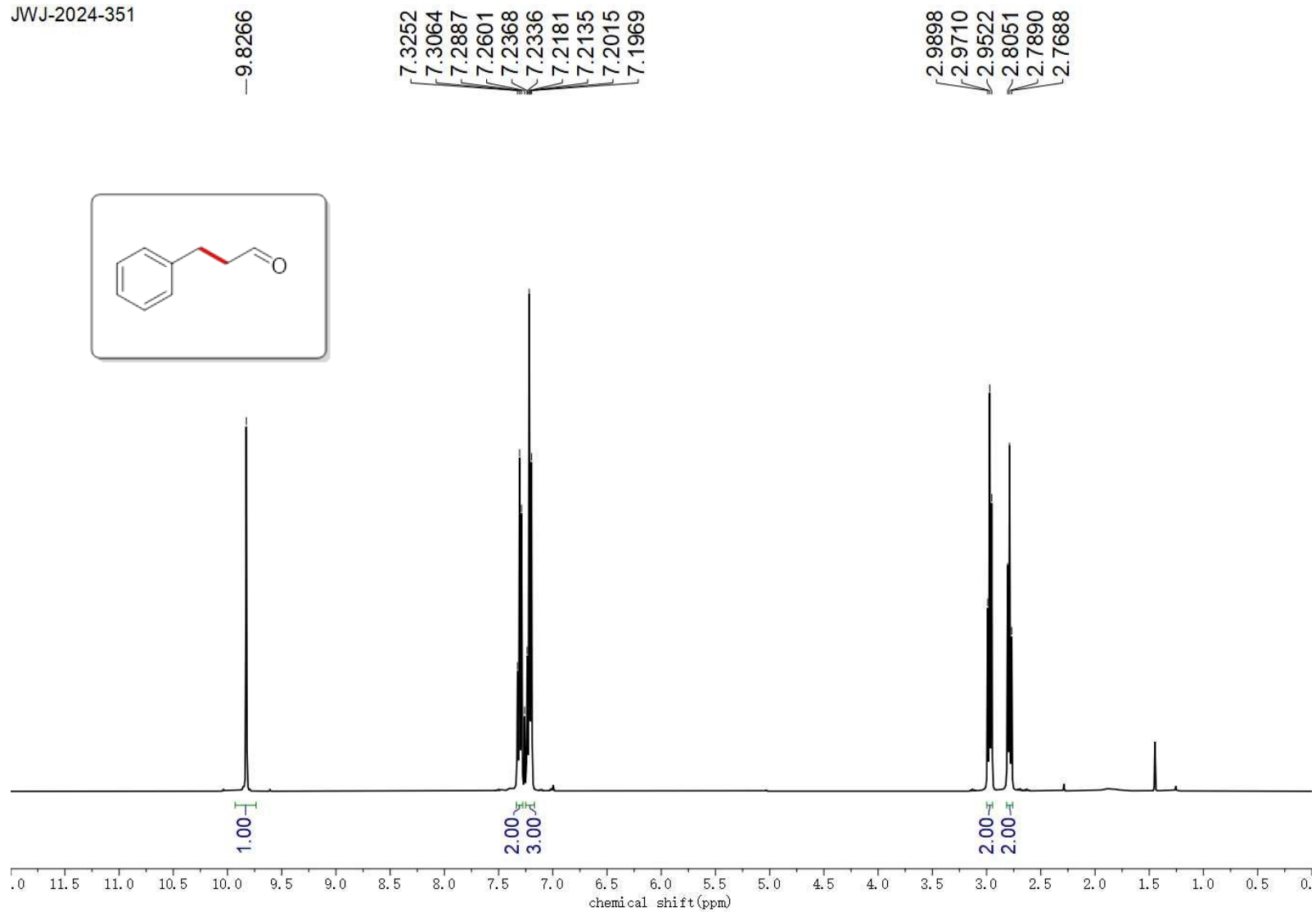
JWJ-2024-351



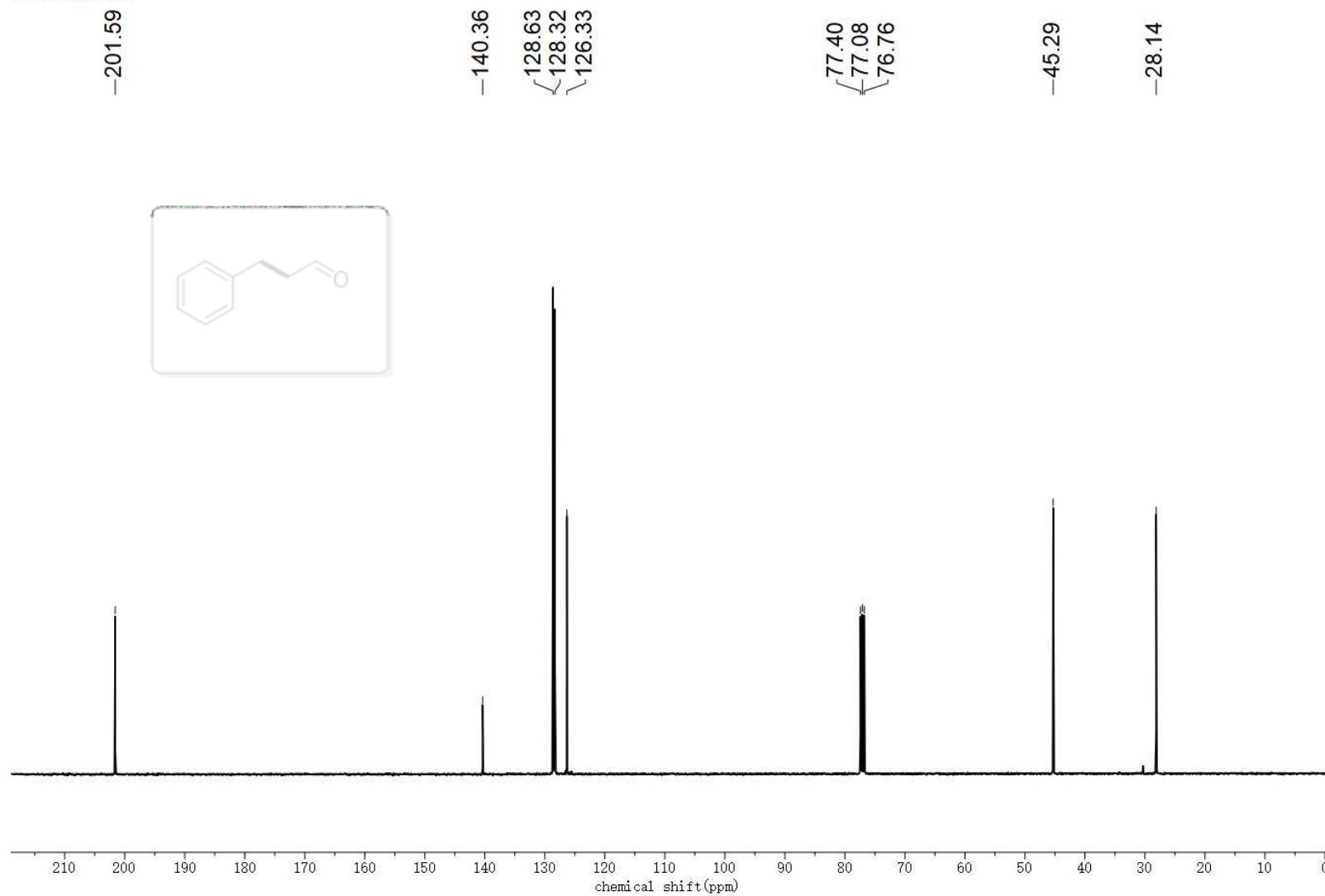
JWJ-2024-351



JWJ-2024-351



JWJ-2024-351





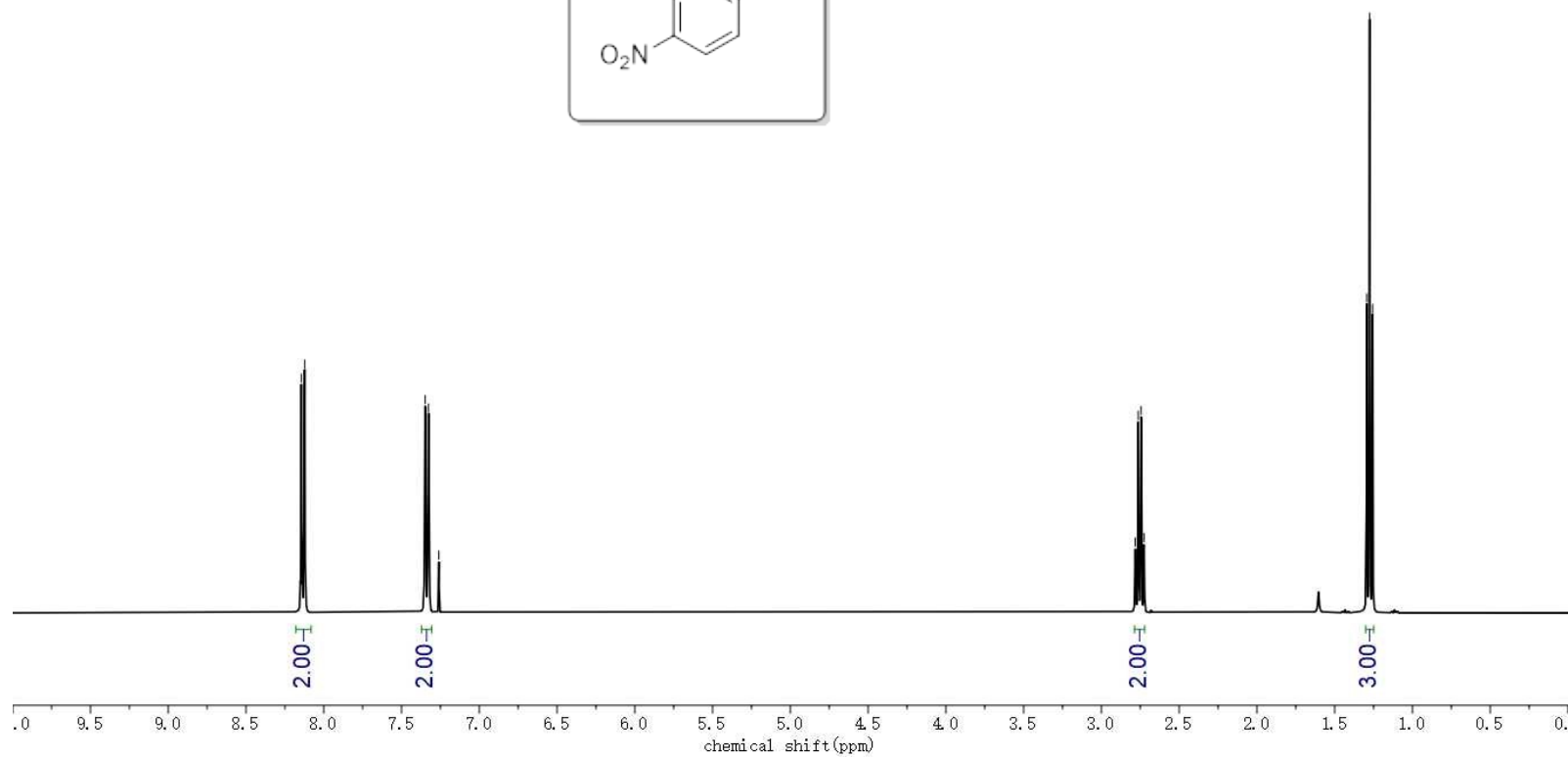
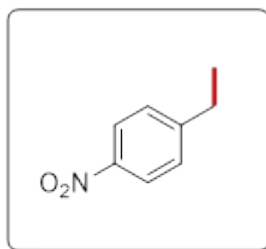
JWJ-2024-443

8.14  
8.12

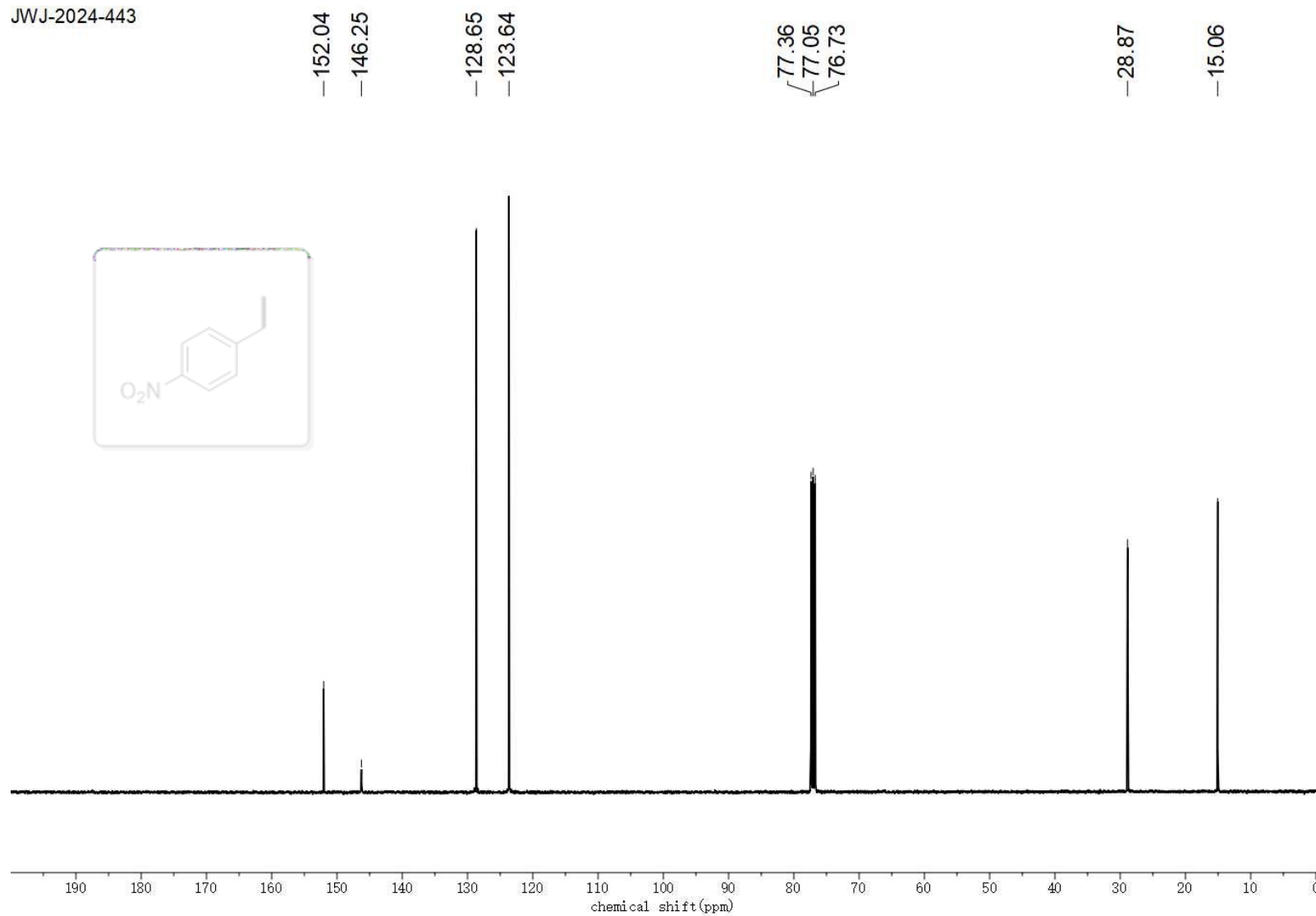
7.35  
7.33  
7.26

2.78  
2.76  
2.74  
2.73

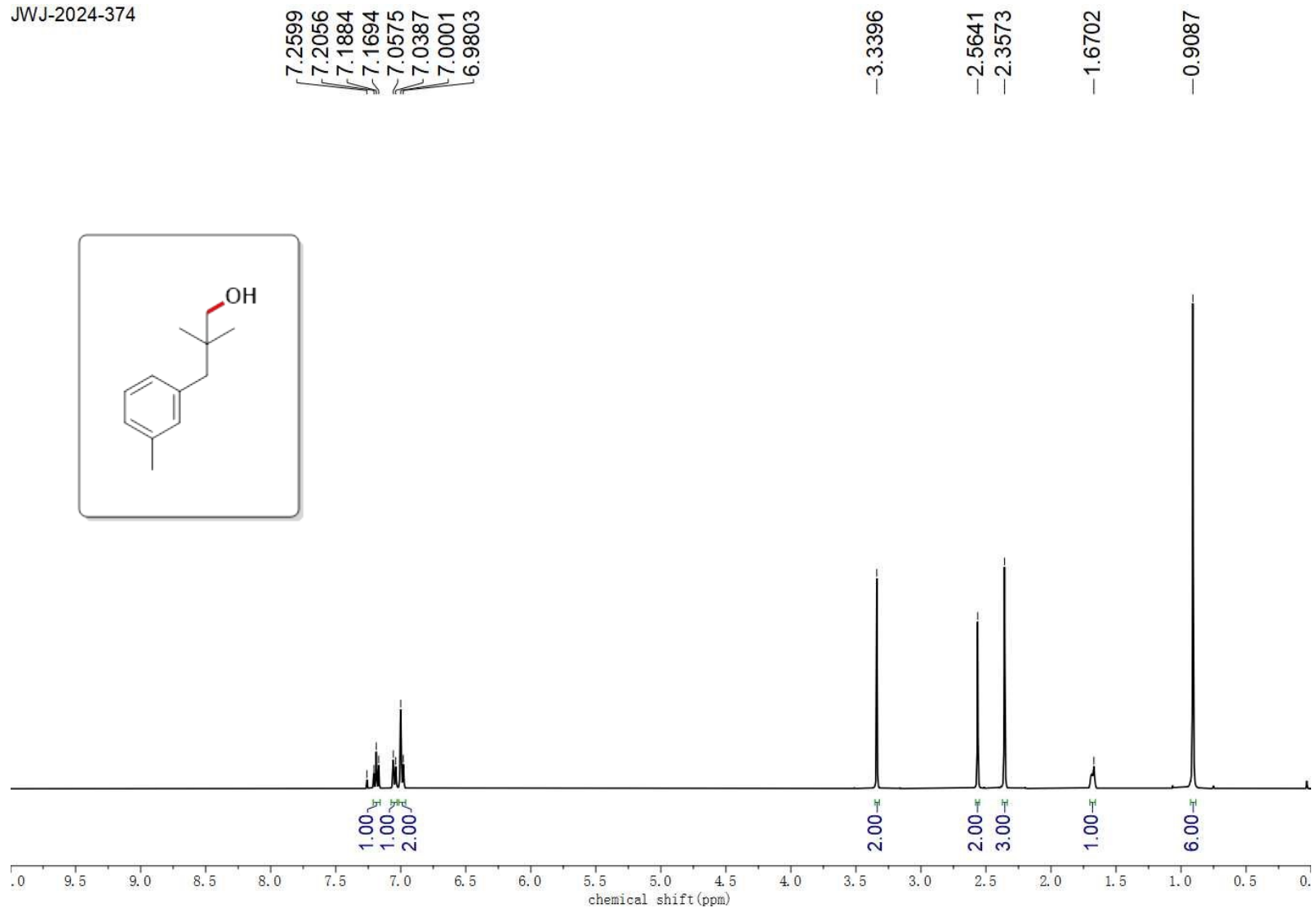
1.29  
1.28  
1.26



JWJ-2024-443



JWJ-2024-374



JWJ-2024-374

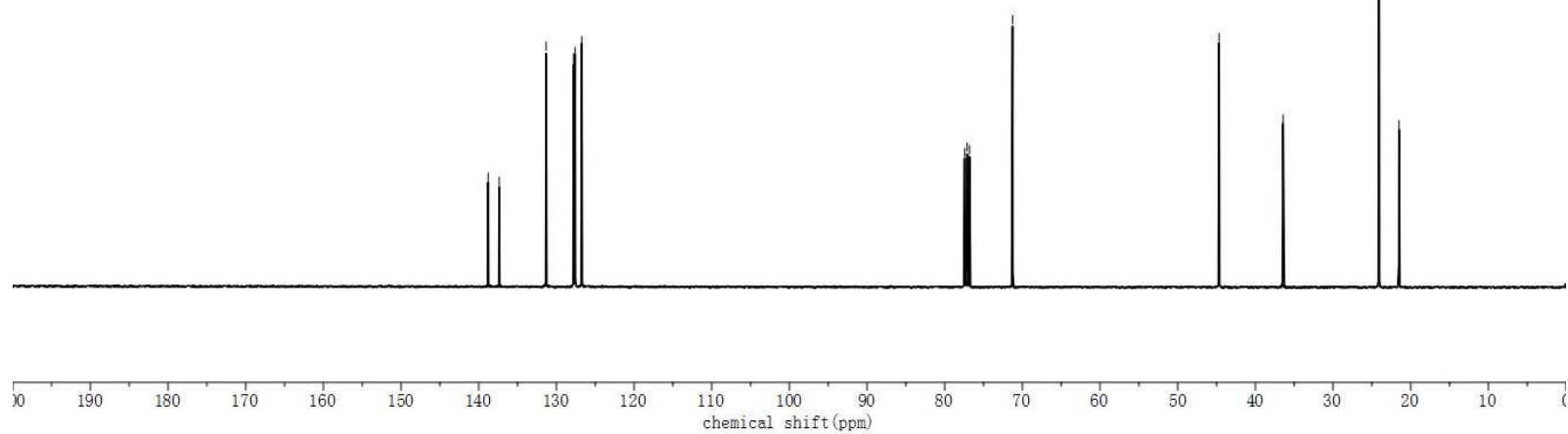
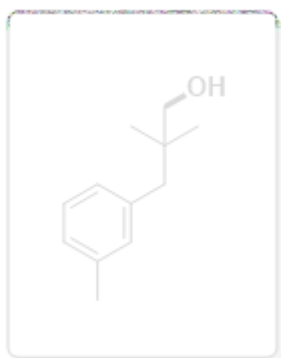
138.78  
137.37  
131.33  
127.78  
127.60  
126.73

77.43  
77.11  
76.79  
71.26

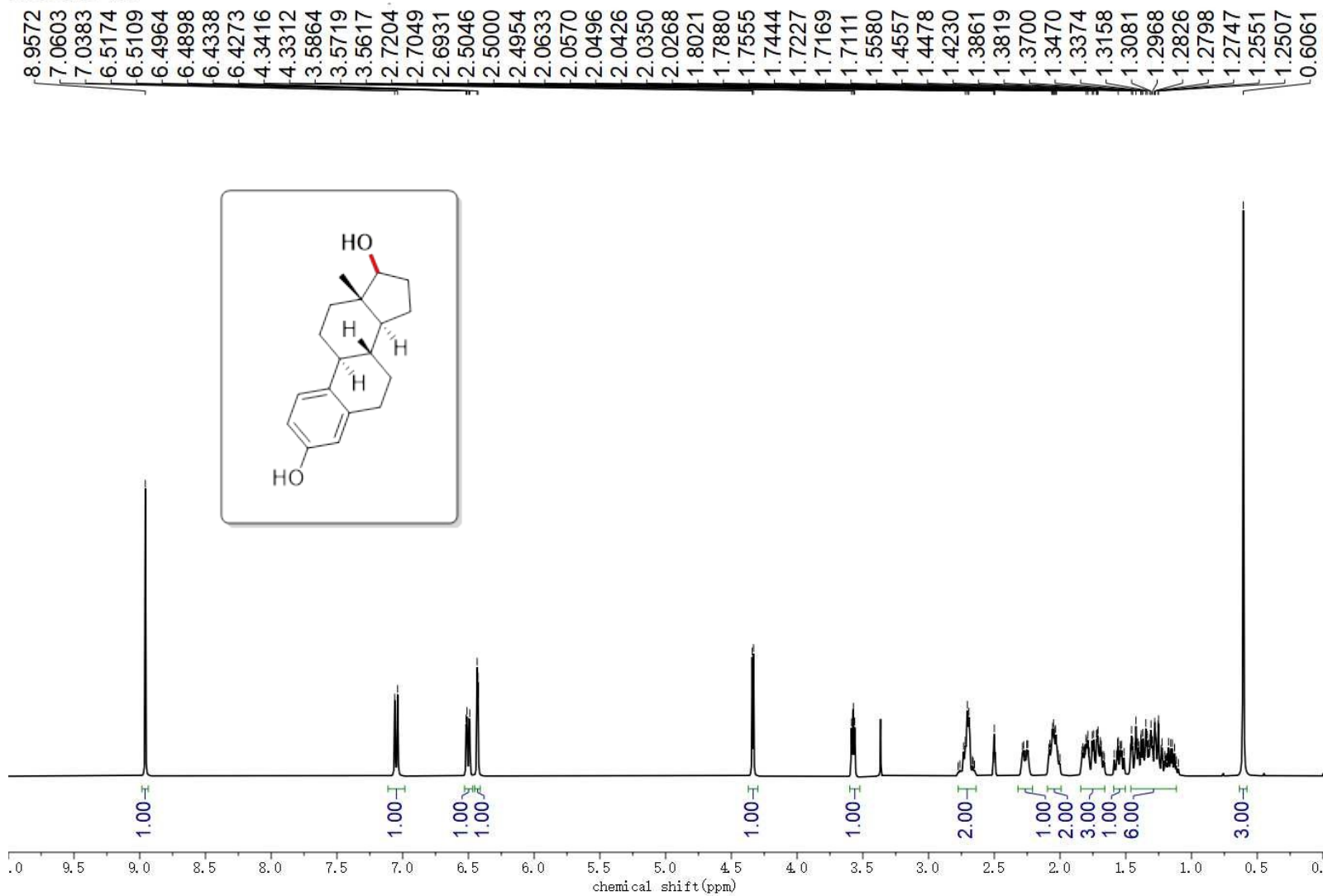
44.68

36.43

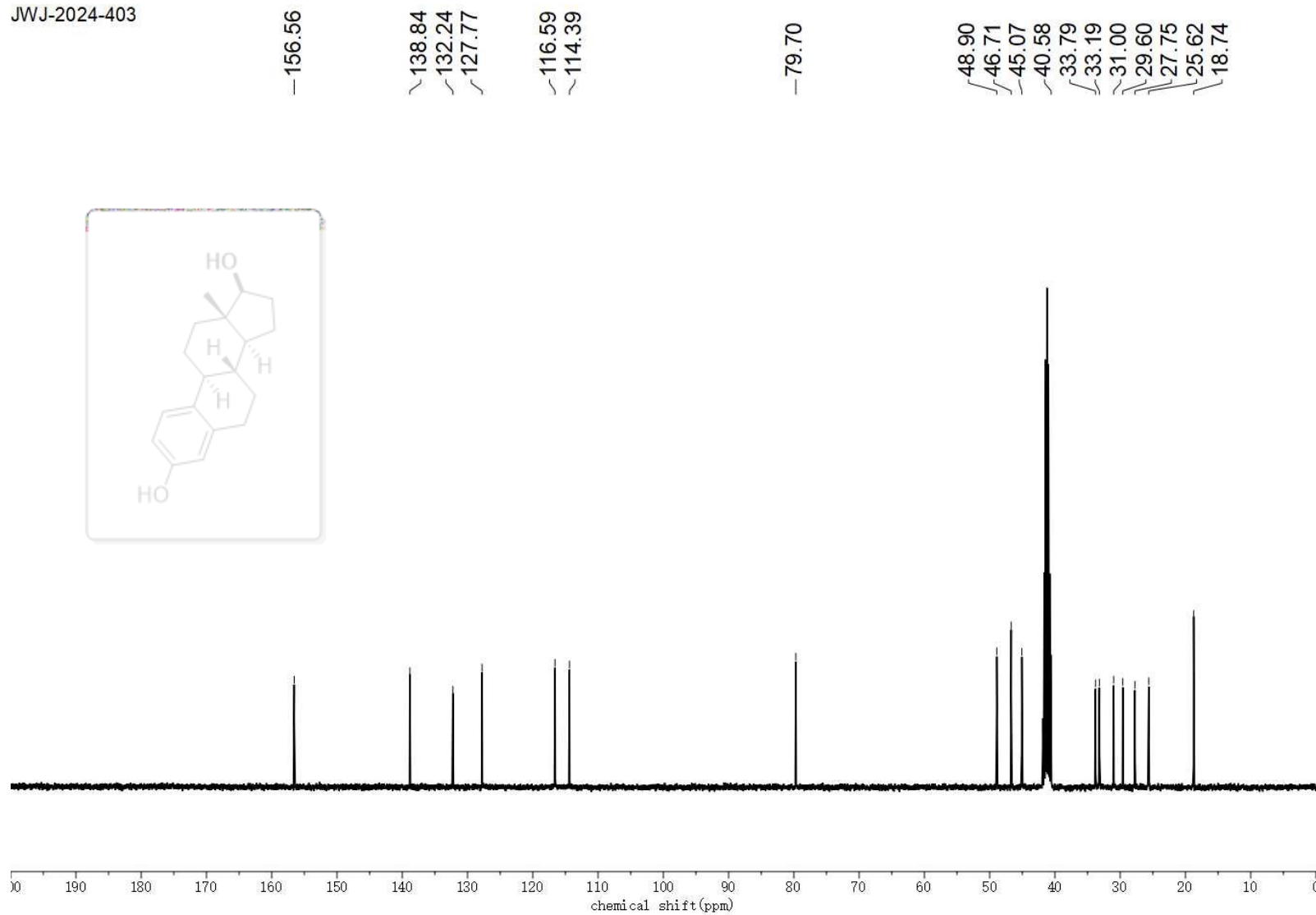
24.08  
21.51



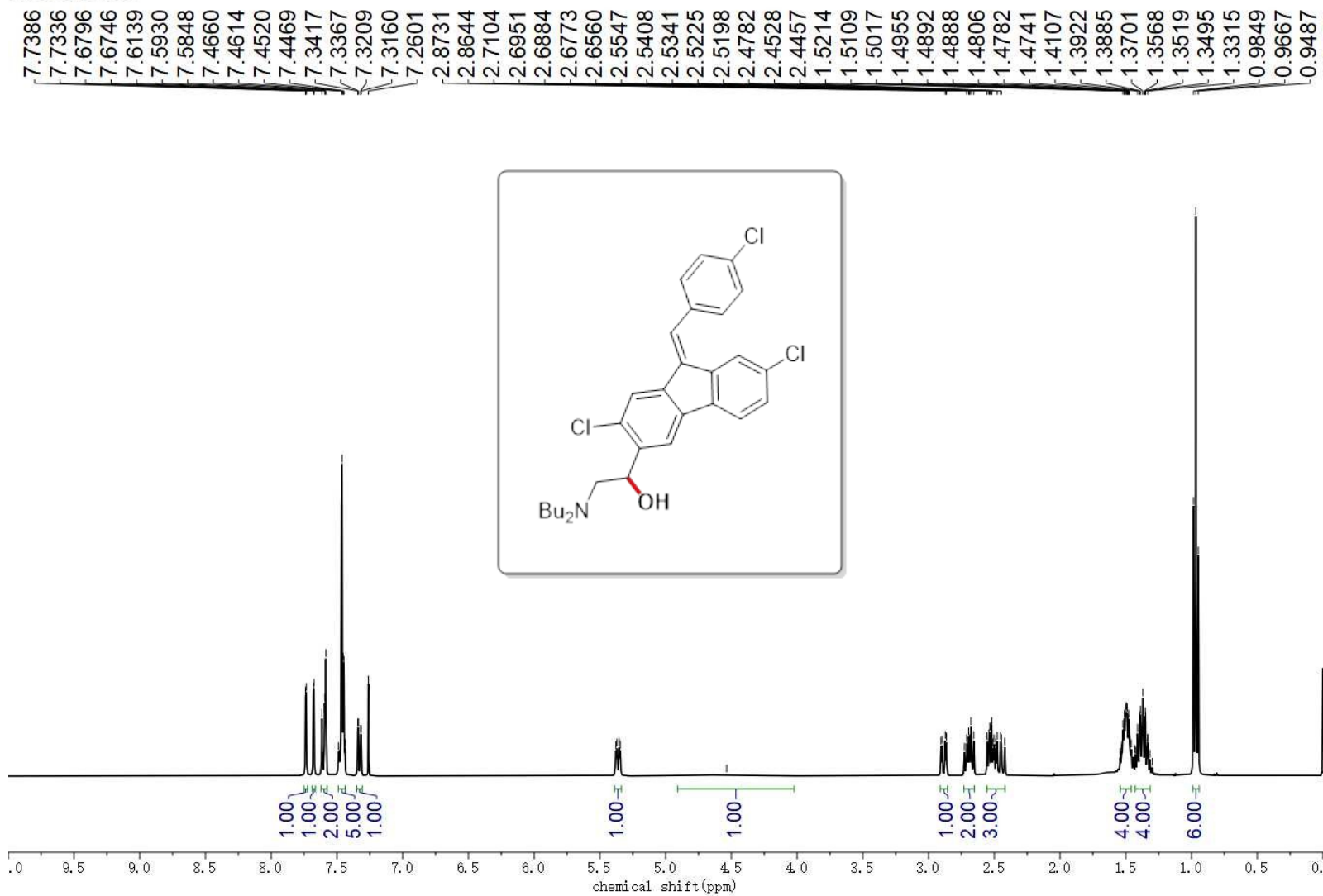
JWJ-2024-403



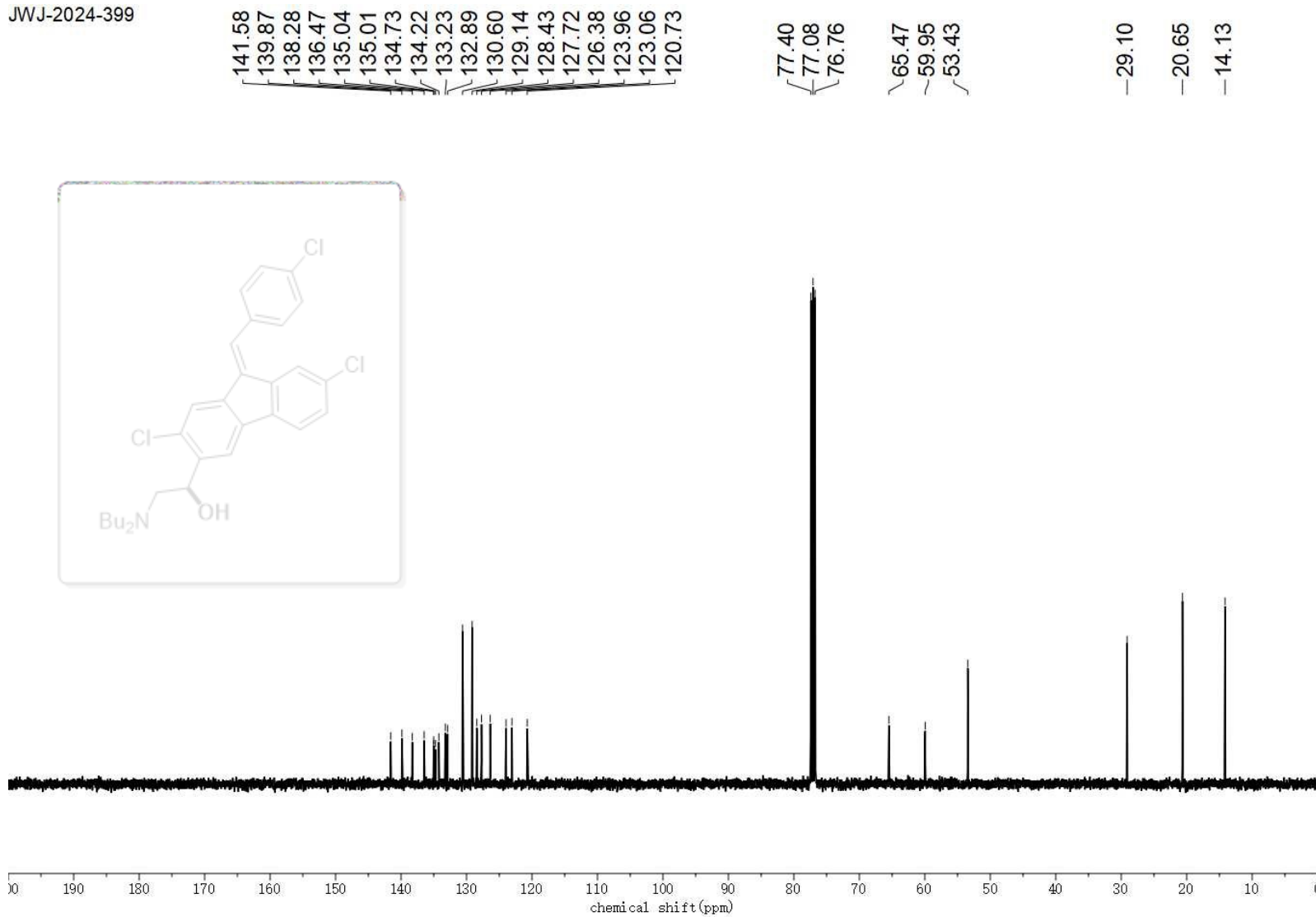
JWJ-2024-403



JWJ-2024-399

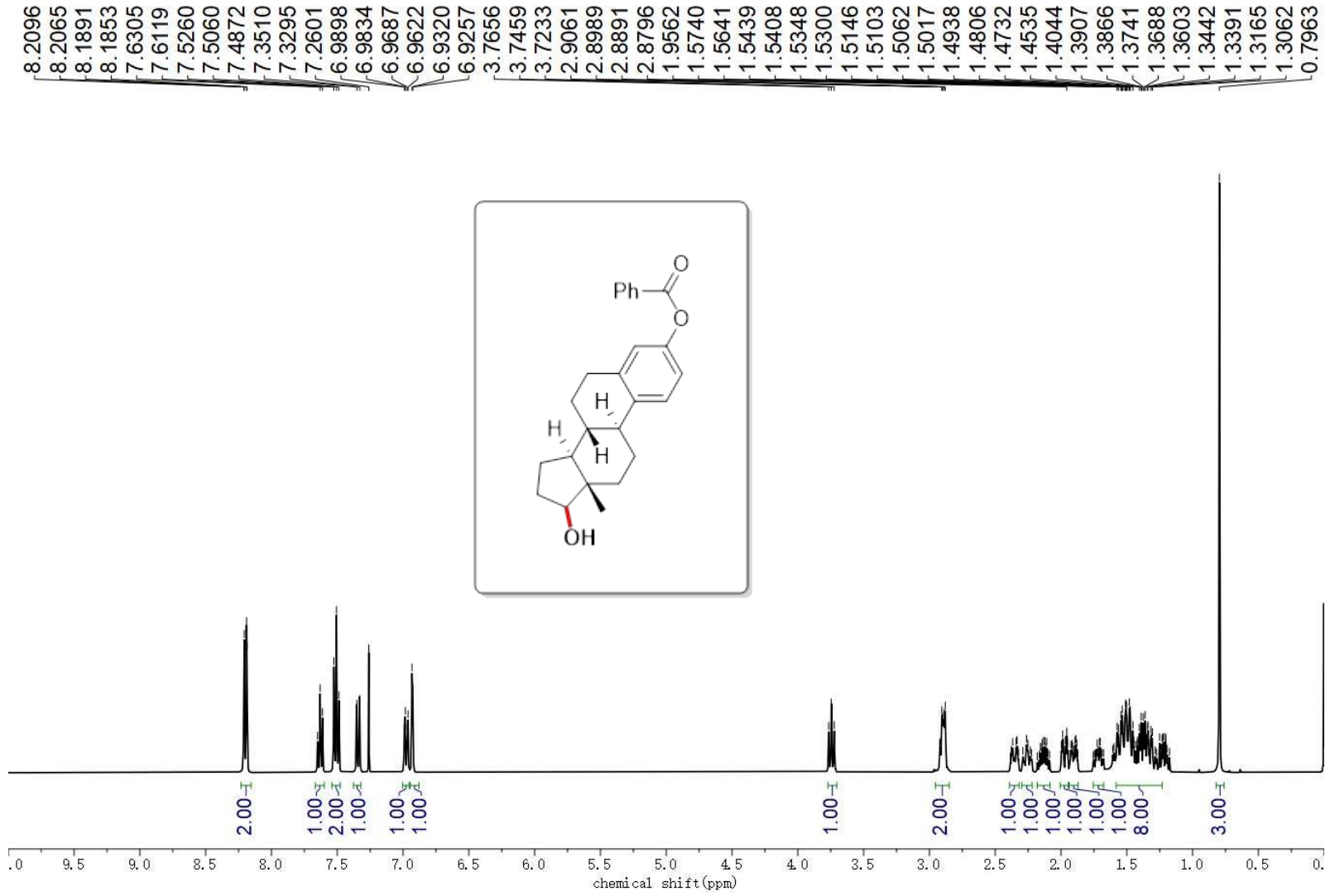


JWJ-2024-399





JWJ-2024-417



JWJ-2024-417

