# Supporting Information

# Is Fe-catalyzed *ortho* C-H arylation of benzamides sensitive to steric hindrance and directing group?

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## 1. General experimental details

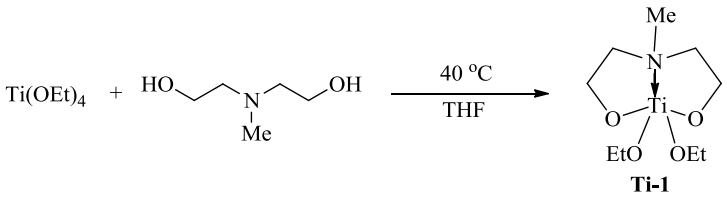
$^{1}$H NMR (400, 500 or 600 MHz) and $^{13}$C NMR (100, 125 or 150 MHz) spectra were recorded on Bruke 400 Hz or 500 Hz and JNM-ECE 400R or 600R spectrometers unless otherwise noted. The chemical shifts (δ) were quoted in parts per million from tetramethylsilane for $^{1}$H and $CDCl_3$ for $^{13}$C spectroscopy. ESI mass spectra were recorded on TRACE MS spectrometer. High resolution mass spectra (HRMS) were obtained with a Bruker microTOF (ESI). Infrared data were acquired using an AVATAR 360 FT-IR spectrophotometer. Elemental analysis was carried out with an Elementear Vario instrument. Melting points were recorded on a TECH X-4 microscopic instrument and uncorrected.

## 2. General remarks for the preparation of aryl Grignard reagents

All reagents and solvents used for aryl magnesium reagents or lithium reagents and reactions were freshly dehydrated before use. The corresponding glassware was oven dried (120 $^{o}$C) and cooled under a stream of argon gas.

Common aryl Grignard reagents such as phenyl magnesium or 4-methoxyphenyl magnesium bromides were prepared according to standard procedure. Functionalized aryl Grignard reagents such as 2-cyanophenyl magnesium chloride or 4-(ethoxycarbonyl)phenyl magnesium chloride were prepared *via* iodine-magnesium exchange using *i*-PrMgCl·LiCl according to Knochel's method.[1] All the Grignard reagents were titrated before use.[2]

## 3. Preparation of Ti-1[3]




**Ti-1**

Under Ar atmosphere, $Ti(OEt)_4$ (34.2 g, 150 mmol) was dissolved in 100 mL THF. Under stirring, *N*-methyldiethanolamine (17.9 g, 150 mmol) was added dropwise. The mixture was stirred at 40 $^{o}$C for 4—5 h and the solvent was removed by vacuum distillation. The residue was dissolved in hexane (20 mL) and frozen in the refrigerator to form a solid. After filtration and vacuum drying, the desired Ti-1 was obtained as a pale yellow solid (38.3g, 98%).

## 4. General Procedure for the Fe-catalyzed *ortho* C-H arylation of benzamides

### 4.1 Typical procedure for *ortho* arylation using common Grignard reagents (2a)

Under Ar atmosphere, **Ti-1** (306 mg; 1.2 mmol) was dissolved in 2 mL THF and stirred at room temperature for 20 to 30 min. A solution of 2-$MeC_6H_4MgBr$ (2.4 mmol, 1.0 M in THF) was added dropwise through a syringe to the resulting mixture during 10－15 min, and then stirred at room temperature for 30 to 40 min. After that, a solution of TMPMgCl·LiCl (1.0 M in THF, 2.4 mmol) was added dropwise through a syringe, and then stirred at room temperature for 30 min.

To another three-necked round-bottom flask were added *N*-methylbenzamide (270 mg, 2 mmol) and 2 mL THF. Under stirring, a solution of 2-$MeC_6H_4MgBr$ or *i*-PrMgCl (2.0 mmol, 1.0 M in THF) was added dropwise through a syringe to the resulting mixture during 10－15 min, and then stirred at room temperature for 30 to 40 min. $FeCl_3$ (32.5 mg; 0.2 mmol) and TMEDA (47 mg, 0.4 mmol) were added to the resulting solution and stirred for 10 min at room temperature. The mixture was heated to 45 $^oC$ and the above-prepared mixed titanate was added. The stirring was continued at 45 $^oC$ for 1 h before 1,2-dichloroethane (238 mg，2.4 mmol) was added. The progress of the reaction was monitored by TLC, and after the reaction finished (about 6 hr), the reaction was quenched by adding 100 mL distilled water. After being filtered, the solid and filtrate were extracted with $CH_2Cl_2$. The organic layer was dried over $Na_2SO_4$ and concentrated to yield the crude compound, which was purified by column chromatography to afford the desired product **2a** (388 mg, 86% yield).

### 4.2 Typical procedure for *ortho* arylation using functionalized Grignard reagents (2u)

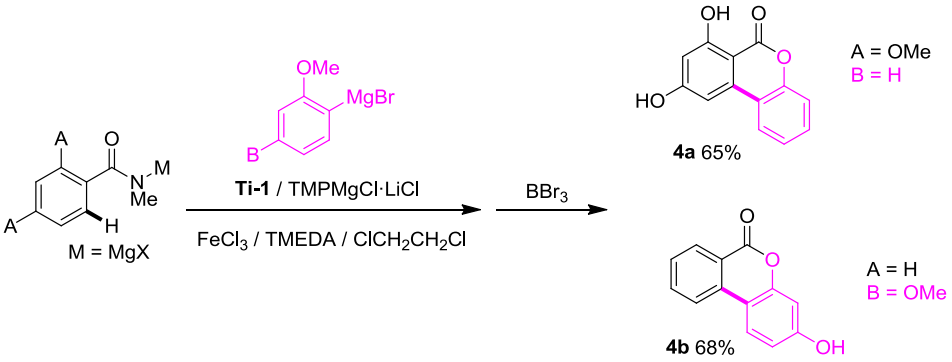
Under Ar atmosphere, the solution of 2-$IC_6H_4CN$ (550 mg, 2.4 mmol) in 5 mL THF was cooled to -40 $^oC$ under stirring. To this solution was added *i*-PrMgCl·LiCl (2.4 mmol, 1.0 M in THF) dropwise. The stirring was continued at that temperature untill the exchange reaction was completed (monitored by TLC).

Under Ar atmosphere, THF (2 mL); **Ti-1** (306 mg; 1.2 mmol) were added to another round-bottom flask, and the mixture was stirred and cooled to -45～-50 $^oC$. To

this solution, the above-prepared Knochel-type functionalized Grignard reagent was added dropwise through a syringe during 10-15 min with the temperature being kept below -40 $^{o}$C. After the addition, the mixture was allowed to come to room temperature in 2 hr, and stirred at that temperature for 30 min. The solvent as well as *i*-PrI were removed under vacuum (about 10 mmHg) during which the temperature was below 20 $^{o}$C until the mixture became a paste. THF (10 mL) was added to the paste and the stirring was continued until a solution was formed. After that, a solution of TMPMgCl·LiCl (1.0 M in THF, 2.4 mmol) was added dropwise through a syringe, and then stirred at room temperature for 30 min.

To another three-necked round-bottom flask were added *N*-methylbenzamide (270 mg, 2 mmol) and 2 mL THF. Under stirring, a solution of *i*-PrMgCl (2.0 mmol, 1.0 M in THF) was added dropwise through a syringe to the resulting mixture during 10—15 min, and then stirred at room temperature for 30 to 40 min. $FeCl_3$ (32.5 mg; 0.2 mmol) and TMEDA (47 mg, 0.4 mmol) were added to the resulting solution and stirred for 10 min at room temperature. The mixture was heated to 45 $^{o}$C and the above-prepared mixed titanate was added. The stirring was continued at 45 $^{o}$C for 1 h before 1,2-dichloroethane (238 mg, 2.4 mmol) was added. The progress of the reaction was monitored by TLC, and after the reaction finished (about 6 hr), the reaction was quenched by adding 100 mL distilled water. After being filtered, the solid and filtrate were extracted with $CH_2Cl_2$. The organic layer was dried over $Na_2SO_4$ and concentrated to yield the crude compound, which was purified by column chromatography to yield the desired product **2u** (321 mg, 68% yield).

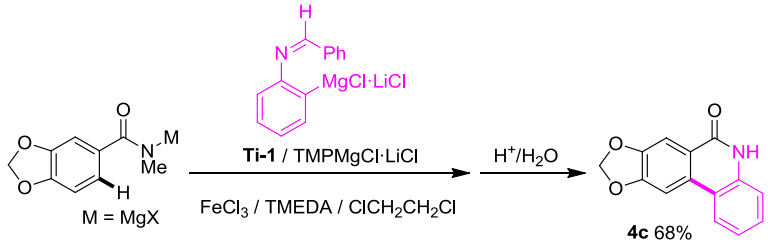
## 5. Preparations of 4a and 4b




According to typical procedure 4.1, the crude products of arylation reaction were obtained and dissolved in 20 mL $CH_2Cl_2$. The mixture was stirred and cooled to -78 $^{o}$C, and $BBr_3$ (for **4a**: 3.0 g, 12 mmol; for **4b**: 2.0 g, 8 mmol) was added dropwise.

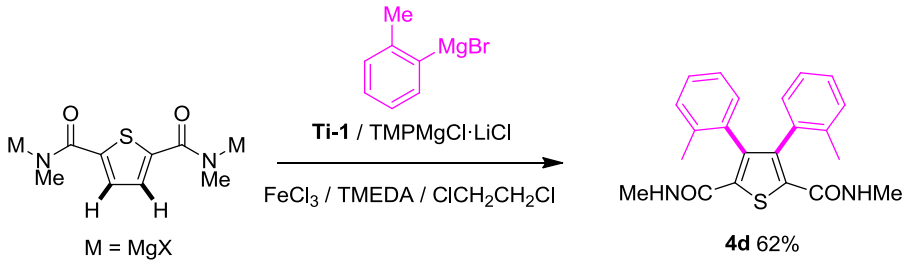
After addition, the mixture was allowed to come to room temperature and stirred for 24 h. The reaction was quenched with methanol (20 mL). The product was taken up with $CH_2Cl_2$ (4 × 50 mL) and washed with saturated NaCl aqueous solution (50 mL). After dried over $Na_2SO_4$ and concentrated by rotary evaporator, crude product was obtained, which was purified by column chromatography to afford the desired product (**4a**: 297 mg; **4b**: 289 mg).

## 6. Preparations of 4c



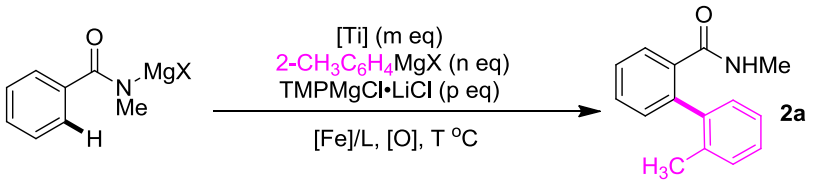

According to typical procedure 4.2, the crude product of arylation reaction was dissolved in 20 mL 2N HCl aqueous solution, and the mixture was stirred at 40 °C for 4 h. The product was taken up with $CH_2Cl_2$ (4 × 50 mL) and washed with saturated NaCl aqueous solution (50 mL). After dried over $Na_2SO_4$ and concentrated by rotary evaporator, crude product was obtained, which was purified by column chromatography to afford the desired product **4c** (325mg; 68%).

## 7. Preparations of 4d



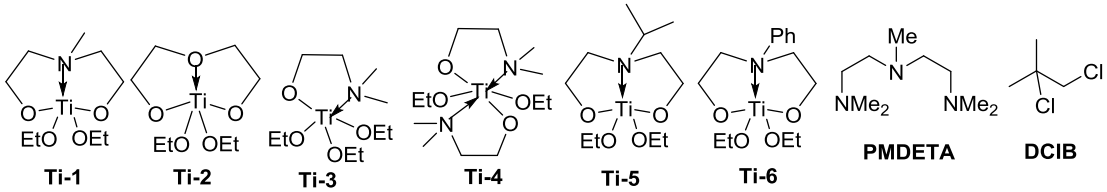

The reaction was conducted on a 2 mmol scale according to typical procedure 4.1. After the first part of mixed titanate [**Ti-1** (1.2 mmol) /2-$MeC_6H_4MgBr$ (2.4 mmol) /TMPMgCl·LiCl (2.4 mmol)] and 1,2-dichloroethane (2.4 mmol) was added, the reaction mixture was stirred at 45 °C for 6h; then the second part of mixed titanate and 1,2-dichloroethane (with equal amount to the first part) was added and the reaction was continued for 6h. The reaction was post-treated and purified by the typical procedure to afford **4d** (469 mg; 62%).

## 8. Table S1 condition optimization[a]

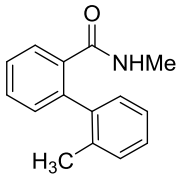



| entry | [Ti] | n | m | p | [Fe]/L | [O] | T (°C) | yield[b] (**2a**, %) |
|---|---|---|---|---|---|---|---|---|
| 1 | -- | 1.2 | 0 | 1.2 | FL1 | DCE | 45 | trace |
| 2 | $Ti(OEt)_4$ | 1.2 | 0.6 | 1.2 | FL1[d] | DCE[e] | 45 | 42 |
| **3** | **Ti-1**[c] | **1.2** | **0.6** | **1.2** | **FL1** | **DCE** | **45** | **86** |
| 4 | Ti-2[c] | 1.2 | 0.6 | 1.2 | FL1 | DCE | 45 | 46 |
| 5 | Ti-3[c] | 1.2 | 0.6 | 1.2 | FL1 | DCE | 45 | 47 |
| 6 | Ti-4[c] | 1.2 | 0.6 | 1.2 | FL1 | DCE | 45 | 52 |
| 7 | Ti-5[c] | 1.2 | 0.6 | 1.2 | FL1 | DCE | 45 | 59 |
| 8 | Ti-6[c] | 1.2 | 0.6 | 1.2 | FL1 | DCE | 45 | 30 |
| 9 | $Ti(OCH_2CH_2NMe_2)_4$ | 1.2 | 0.6 | 1.2 | FL1 | DCE | 45 | trace |
| 10 | Ti-1 | 1.2 | 0.3 | 1.2 | FL1 | DCE | 45 | 39 |
| 11 | Ti-1 | 1.2 | 1.0 | 1.2 | FL1 | DCE | 45 | 52 |
| 12 | Ti-1 | 1.2 | 1.5 | 1.2 | FL1 | DCE | 45 | 20 |
| **13** | **Ti-1** | **2.4** | **0.6** | **0** | **FL1** | **DCE** | **45** | **trace** |
| 14 | Ti-1 | 1.6 | 0.6 | 0.8 | FL1 | DCE | 45 | 43 |
| 15 | Ti-1 | 1.2 | 0.6 | 1.6 | FL1 | DCE | 45 | 86 |
| 16 | Ti-1 | 1.6 | 0.6 | 1.2 | FL1 | DCE | 45 | 85 |
| 17 | Ti-1 | 2.0 | 0.6 | 1.2 | FL1 | DCE | 45 | 83 |
| 18 | Ti-1 | 1.2 | 0.6 | 1.2 | FL1 | DCE | 25 | 33 |
| 19 | Ti-1 | 1.2 | 0.6 | 1.2 | FL1 | DCE | 65 | 75 |
| 20 | Ti-1 | 1.2 | 0.6 | 1.2 | FL1 | DCE[f] | 78 | 60 |
| 21 | Ti-1 | 1.2 | 0.6 | 1.2 | $FeCl_3$ | DCE | 45 | trace |
| 22 | Ti-1 | 1.2 | 0.6 | 1.2 | FL2[d] | DCE | 45 | 48 |
| 23 | Ti-1 | 1.2 | 0.6 | 1.2 | FL3[d] | DCE | 45 | 44 |
| 24 | Ti-1 | 1.2 | 0.6 | 1.2 | FL4[d] | DCE | 45 | trace |
| 25 | Ti-1 | 1.2 | 0.6 | 1.2 | FL1 | DCE[g] | 45 | 40 |
| 26 | Ti-1 | 1.2 | 0.6 | 1.2 | FL1 | DBE[e] | 45 | 47 |
| 27 | Ti-1 | 1.2 | 0.6 | 1.2 | FL1 | DCIB[e] | 45 | 54 |
| 28 | Ti-1 | 1.2 | 0.6 | 1.2 | FL1 | $O_2$ | 45 | 52 |

[a]The reaction was carried out on a 1 mmol scale and 10 mol % Fe salts and 20 mol % $Me_2NCH_2CH_2NMe_2$ (TMEDA) were charged in unless indicated otherwise; and all the reactions were conducted in THF unless indicated otherwise. [b]Isolated yields. [c]The structures of Ti-1, Ti-2, Ti-3, Ti-4, Ti-5, Ti-6 were outlined below. [d]FL1: 10 mol % $FeCl_3$/ 20 mol % TMEDA; FL2: 10 mol % $FeCl_3$/ 20 mol % PMDETA (outlined below); FL3: 10 mol % $FeCl_3$/ 20 mol % $PBu_3$; FL4: 10 mol % $Fe(acac)_3$/ 20 mol % TMEDA. [e]DCE: $ClCH_2CH_2Cl$; DBE: $BrCH_2CH_2Br$; DCIB: outlined below. These oxidants were charged in 1.2 equivalents unless indicated otherwise. [f]Solvent: THF/PhMe (3:1). [g]2.0 equivalents of DCE were used.



## 9. Characterization data

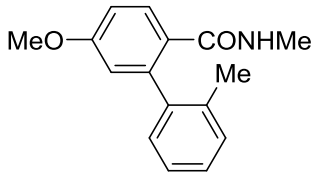



**2a**

***N*, 2'-Dimethyl-[1,1'-biphenyl]-2-carboxamide**[4]

Yellow solid; 86% yield (388 mg); m.p. = 98 $^{o}$C (lit. 95-96 $^{o}$C); $R_f$ = 0.30 (petroleum ether/ethyl acetate = 3:1, v/v).

IR ($cm^{-1}$, KBr): 3296, 1648, 1531, 756, 740; $^{1}$HNMR ($CDCl_3$, 400 MHz) δ (ppm) 7.91-7.89 (m, 1H), 7.47-7.42 (m, 2H), 7.28-7.25 (m, 3H), 7.20-7.18(m, 2H), 5.26 (br, 1H), 2.61 (d, *J* = 4.88 Hz, 3H), 2.10 (s, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 169.1, 140.3, 139.2, 136.2, 134.9, 130.51, 130.46, 130.39, 129.4, 129.2, 128.3, 127.8, 126.2, 26.8, 20.1.

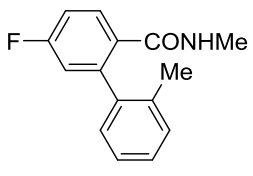



**2b**

**5-Methoxy-*N*, 2'-dimethyl-[1,1'-biphenyl]-2-carboxamide**

Yellow solid; 72% yield (368 mg); m.p. = 128-129 $^{o}$C; $R_f$ = 0.30 (petroleum ether/ethyl acetate = 2:1, v/v).

IR ($cm^{-1}$, KBr): 3300, 1651, 1601, 1223, 826, 764, 608; $^{1}$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.91 (d, *J* = 8.72 Hz, 1H), 7.26-7.22 (m, 3H), 7.16-7.14 (m, 1H), 6.91 (dd, *J* = 8.72, 2.64 Hz, 1H), 6.63 (d, *J* = 2.6 Hz, 1H), 5.16 (br, 1H), 3.78 (s, 3H), 2.54 (d, *J* = 4.80 Hz, 3H), 2.05 (s, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 168.3, 160.9, 140.9, 140.3, 136.2, 131.7, 130.4, 128.9, 128.3, 126.9, 126.3, 115.5, 113.0, 55.4, 26.7, 19.9; HRMS calcd for $C_{16}H_{18}NO_2^+$ $[M+H]^+$ 256.1338, Found 256.1331.

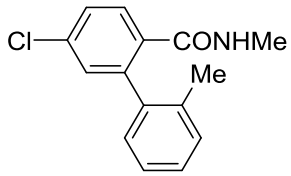



**2c**

**5-Fluoro-*N*, 2'-dimethyl-[1,1'-biphenyl]-2-carboxamide**

Yellow oil; 85% yield (414 mg); $R_f$ = 0.32 (petroleum ether/ethyl acetate = 3:1, v/v).

IR ($cm^{-1}$, KBr): 3297, 1658, 668, 568, 552; $^1H$ NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.97-7.94 (m, 1H), 7.34-7.25 (m, 3H), 7.18 (d, *J* = 7.20 Hz, 1H), 7.13-7.11 (m, 1H), 6.89 (dd, *J* = 9.20, 2.64 Hz, 1H), 5.21 (br, 1H), 2.60 (d, *J* = 4.88 Hz, 3H), 2.11 (s, 3H); $^{13}C$ NMR ($CDCl_3$, 100 MHz) δ (ppm) 167.9, 163.4 (d, *J* = 251.7 Hz, 1C), 141.7 (d, *J* = 8.4 Hz, 1C), 139.1, 136.0, 132.0 (d, *J* = 9.1 Hz, 1C), 131.0 (d, *J* = 3.3 Hz, 1C), 130.6, 128.9, 128.7, 126.4, 117.1 (d, *J* = 21.8 Hz, 1C), 114.7 (d, *J* = 21.2 Hz, 1C), 26.8, 20.0; HRMS calcd for $C_{15}H_{15}FNO^+$ $[M+H]^+$ 244.1138, Found 244.1128.

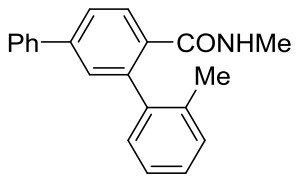



**2d**

**5-Chloro-*N*, 2'-dimethyl-[1,1'-biphenyl]-2-carboxamide**

Yellow oil; 73% yield (379 mg); $R_f$ = 0.32 (petroleum ether/ethyl acetate = 3:1, v/v).

IR ($cm^{-1}$, KBr): 3285, 2926, 1655, 1589, 758, 673; $^1H$ NMR (CDCl3, 400 MHz) δ (ppm) 7.89 (dd, *J* = 8.32, 1.20 Hz, 1H), 7.40 (dd, *J* = 8.52, 2.12 Hz, 1H), 7.33-7.26 (m, 3H), 7.20-7.17 (m, 2H), 5.25 (br, 1H), 2.60 (d, *J* = 4.84 Hz, 3H), 2.10 (s, 3H); $^{13}C$ NMR ($CDCl_3$, 150 MHz) δ (ppm) 167.9, 140.9, 138.9, 136.4, 136.1, 133.2, 131.1, 130.7, 130.3, 129.0, 128.8, 128.0, 126.5, 26.8, 20.0; HRMS calcd for $C_{15}H_{15}ClNO^+$ $[M+H]^+$ 260.0842, Found 260.0833.



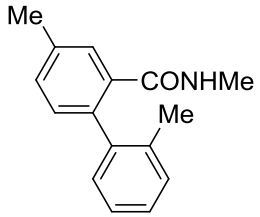

**2e**

***N*, 2''-Dimethyl-[1,1':3',1''-terphenyl]-4'-carboxamide**

Whitish solid; 75% yield (452 mg); m.p. = 152 $^oC$; $R_f$ = 0.35 (petroleum ether/ethyl acetate = 3:1, v/v).

IR ($cm^{-1}$, KBr): 3275, 1653, 1559, 758, 744, 696; $^1H$ NMR ($CDCl_3$, 400 MHz) δ (ppm) 8.03 (d, *J* = 8.12 Hz, 1H), 7.68 (d, *J* = 8.12, 1.76 Hz, 1H), 7.63 (d, *J* = 7.28 Hz,

2H), 7.46-7.42 (m, 3H), 7.38-7.26 (m, 5H), 5.34 (br, 1H), 2.65 (d, *J* = 4.88 Hz, 3H), 2.16 (s, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 168.6, 143.0, 140.2, 139.8, 139.6, 136.2, 133.4, 130.5, 130.1, 129.2, 129.0, 128.9, 128.3, 128.0, 127.2, 126.3, 126.2, 26.8, 20.1; HRMS calcd for $C_{21}H_{20}NO^+$ $[M+H]^+$ 302.1545, Found 302.1543.

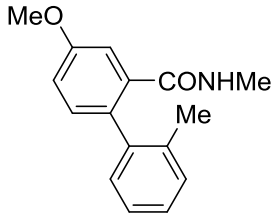



**2f**

***N*,2',4-Trimethyl-[1,1'-biphenyl]-2-carboxamide**

Yellow oil; 72% yield (345 mg); $R_f$ = 0.34 (petroleum ether/ethyl acetate = 3:1, v/v).

IR ($cm^{-1}$, KBr): 3292, 1656, 1539, 850, 750; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.71 (d, *J* = 0.32 Hz, 1H), 7.28-7.21 (m, 4H), 7.16 (d, *J* = 7.04 Hz, 2H), 7.07 (d, *J* = 7.72 Hz, 1H), 5.27 (br, 1H), 2.59 (d, *J* = 4.88 Hz), 2.41 (s, 3H), 2.10 (s, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 169.2, 140.2, 137.5, 136.23, 136.21, 134.6, 131.0, 130.4, 130.3, 129.7, 129.3, 128.0, 126.1, 26.7, 21.0, 20.0; HRMS calcd for $C_{16}H_{18}NO^+$ $[M+H]^+$ 240.1388, Found 240.1382.



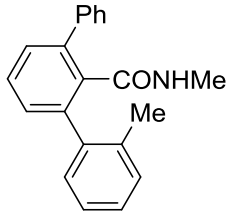

**2g**

**4-Methoxy-*N*,2'-dimethyl-[1,1'-biphenyl]-2-carboxamide**

Yellow oil; 80% yield (408 mg); $R_f$ = 0.30 (petroleum ether/ethyl acetate = 2:1, v/v).

IR ($cm^{-1}$, KBr): 3296, 2926, 1647, 1256, 786, 742; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.51 (d, *J* = 2.72 Hz, 1H), 7.32-7.26 (m, 3H), 7.21 (d, *J* = 7.0 Hz, 1H), 7.14 (d, *J* = 8.40 Hz, 1H ), 7.05 (dd, *J* = 8.40, 2.72 Hz, 1H), 5.33 (br, 1H), 3.91 (s, 3H), 2.64 (d, *J* = 4.88 Hz), 2.14 (s, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 168.7, 159.0,

139.9, 136.5, 135.8, 131.6, 131.4, 130.4, 129.5, 128.0, 126.2, 117.1, 113.5, 55.5, 26.7, 20.0; HRMS calcd for $C_{16}H_{18}NO_2^+$ $[M+H]^+$ 256.1338, Found 256.1331.

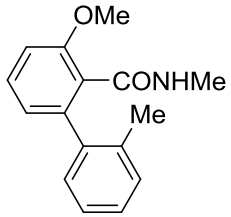



**2h**

***N*,2-Dimethyl-[1,1':3',1''-terphenyl]-2'-carboxamide**

Whitish solid; 76% yield (458 mg); m.p. = 161 $^oC$; $R_f$ = 0.38 (petroleum ether/ethyl acetate = 3:1, v/v).

IR ($cm^{-1}$, KBr): 3280, 1646, 1540, 801, 744, 693; $^1H$ NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.52 (t, *J* = 8.01 Hz, 1H), 7.48-7.35 (m, 11H), 5.31 (br, 1H), 2.69 (d, *J* = 4.94 Hz, 3H), 2.06 (s, 3H); $^{13}C$ NMR ($CDCl_3$, 100 MHz) δ (ppm) 168.9, 142.3, 140.4, 140.2, 139.4, 138.3, 137.5, 129.7, 129.3, 128.6, 128.3, 128.1, 127.3, 126.2, 125.2, 124.6, 124.2, 26.7, 21.0; HRMS calcd for $C_{21}H_{20}NO^+$ $[M+H]^+$ 302.1545, Found 302.1533.

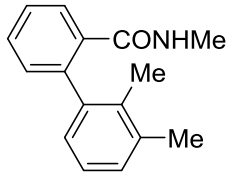



**2i**

**3-Methoxy-*N*,2'-dimethyl-[1,1'-biphenyl]-2-carboxamide**

Whitish solid; 70% yield (357 mg); m.p. = 151 $^oC$; $R_f$ = 0.35 (petroleum ether/ethyl acetate = 2:1, v/v)

IR ($cm^{-1}$, KBr): 3306, 1649, 1466, 1256, 1020, 802, 760; $^1H$ NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.31 (t, *J* = 8.04 Hz, 1H), 7.22-7.17 (m, 2H), 7.14-7.09 (m, 2H), 6.88 (d, *J* = 7.70, 1H), 6.77 (d, *J* = 7.64, 1H), 5.30 (br, 1H), 3.83 (s, 3H), 2.59 (d, *J* = 4.92 Hz, 3H), 2.12 (s, 3H); $^{13}C$ NMR ($CDCl_3$, 100 MHz) δ (ppm) 167.7, 156.4, 141.1, 139.6, 135.8, 130.0, 129.7, 129.0, 127.6, 126.6, 125.2, 122.3, 109.8, 55.9, 26.3, 20.2; HRMS calcd for $C_{16}H_{18}NO_2^+$ $[M+H]^+$ 256.1332, Found 256.1328.

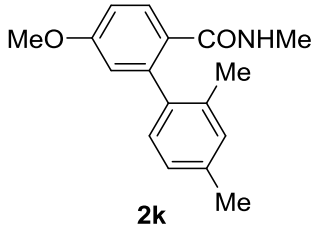



**2j**

***N*,2',3'-Trimethyl-[1,1'-biphenyl]-2-carboxamide**

Whitish solid; 73% yield (349 mg); m.p. = 105 $^{o}$C; $R_f$ = 0.34 (petroleum ether/ethyl acetate = 3:1, v/v).

IR (cm$^{-1}$, KBr): 3291, 2926, 1696, 1535, 1306, 787, 760; $^{1}$H NMR ($CDCl_3$, 400 MHz) δ (ppm) δ 7.92 (dd, *J* = 7.48, 1.56 Hz, 1H), 7.48-7.40 (m, 2H), 7.19-7.14 (m, 3H), 7.05-7.03 (m, 1H), 5.24 (br, 1H), 2.60 (d, *J* = 4.92 Hz, 3H), 2.32 (s, 3H), 1.99 (s, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 169.0, 140.4, 139.8, 137.6, 135.0, 134.8, 130.5, 130.3, 129.8, 129.4, 127.6, 127.0, 125.8, 26.8, 20.6, 16.7; HRMS calcd for $C_{16}H_{18}NO^{+}$ $[M+H]^{+}$ 240.1388, Found 240.1388.

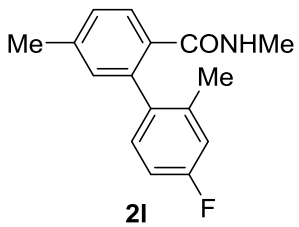



**2k**

**5-Methoxy-*N*,2',4'-trimethyl-[1,1'-biphenyl]-2-carboxamide**

Whitish solid; 71% yield (382 mg); m.p. = 145 $^{o}$C; $R_f$ = 0.34 (petroleum ether/ethyl acetate = 3:1, v/v).

IR (cm$^{-1}$, KBr): 3304, 1658, 1221, 826, 610; $^{1}$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.95 (dd, *J* = 8.68, 2.68 Hz, 1H), 7.09-7.07 (m, 3H), 6.92 (dt, *J* = 8.68, 2.36 Hz, 1H), 6.64 (d, *J* = 2.44 Hz, 1H), 5.30 (br, 1H), 3.81 (d, *J* = 1.48 Hz, 3H), 2.60 (dd, *J* = 4.84, 0.80 Hz, 3H), 2.36 (s, 3H), 2.05 (s, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 168.4, 160.9, 141.0, 138.0, 137.3, 135.9, 131.6, 131.2, 128.9, 127.0, 115.7, 112.9, 55.4, 26.7, 21.2, 19.8; HRMS calcd for $C_{17}H_{20}NO_2^{+}$ $[M+H]^{+}$ 270.1494, Found 270.1485.

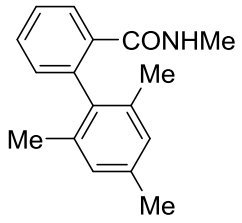



**2l**

**4'-Fluoro-*N*,2',5-trimethyl-[1,1'-biphenyl]-2-carboxamide**

Whitish solid; 78% yield (401 mg); m.p. = 96-97 $^{o}$C; $R_f$ = 0.34 (petroleum ether/ethyl acetate = 3:1, v/v).

IR (cm$^{-1}$, KBr): 3289, 1653, 1474, 857, 802; $^{1}$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.56 (d, *J* = 7.80 Hz, 1H), 7.21 (d, *J* = 7.76 Hz, 1H), 7.14 (s, 1H), 6.98 (s, 1H), 6.91-6.86 (m, 2H), 5.28 (br, 1H), 2.71 (d, *J* = 4.88 Hz, 3H), 2.40-2.38 (m, 6H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 170.0, 162.7 (d, *J* = 246.3 Hz, 1C), 142.3, 140.5, 140.3, 138.3, 132.9, 130.7, 128.9, 128.6, 125.1, 115.2 (d, *J* = 20.8 Hz, 1C), 112.5 (d, *J* = 22.3 Hz, 1C), 26.7, 21.4, 21.3; HRMS calcd for $C_{16}H_{17}FNO^{+}$ $[M+H]^{+}$ 258.1294, Found 258.1293.

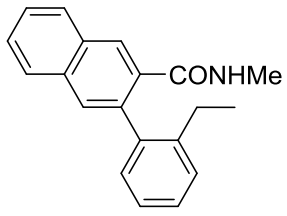



**2m**

***N*,2',4',6'-Tetramethyl-[1,1'-biphenyl]-2-carboxamide**

Whitish solid; 72% yield (365 mg); m.p. = 128 $^{o}$C; $R_f$ = 0.36 (petroleum ether/ethyl acetate = 3:1, v/v).

IR (cm$^{-1}$, KBr): 3288, 2910, 1689, 1548, 850, 770; $^{1}$H NMR ($CDCl_3$, 400 MHz) δ (ppm) δ 8.02 (d, *J* = 7.49, 1H), 7.52-7.47 (m, 2H), 7.12-7.09 (m, 1H), 6.89 (s, 2H), 5.13 (br, 1H), 2.65 (d, *J* = 4.89 Hz, 3H), 2.32 (s, 3H), 1.95 (s, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 168.9, 141.3, 140.4, 139.3, 137.5, 130.9, 130.3, 129.4, 127.5, 126.3, 26.7, 21.1, 20.4; HRMS calcd for $C_{17}H_{18}NO^{+}$ $[M+H]^{+}$ 254.1545, Found 254.1537.

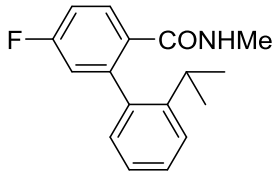



**2n**

**3-(2-Ethylphenyl)-*N*-methyl-2-naphthamide**

Yellow oil; 75% yield (434 mg); $R_f$ = 0.35 (petroleum ether/ethyl acetate = 3:1, v/v).

IR ($cm^{-1}$, KBr): 3308, 1654, 1537, 768, 665; $^1H$ NMR ($CDCl_3$, 400 MHz) δ (ppm) 8.51 (s, 1H), 7.97-7.95 (m, 1H), 7.83 (d, *J* = 7.20 Hz, 1H), 7.68 (s, 1H), 7.58-7.52 (m, 2H), 7.41-7.35 (m, 2H), 7.32-7.25 (m, 2H), 5.38 (br, 1H), 2.65 (d, *J* = 4.80 Hz, 3H), 2.54-2.39 (m, 2H), 1.02 (t, *J* = 7.52 Hz, 3H); $^{13}C$ NMR ($CDCl_3$, 100 MHz) δ (ppm) 168.6, 142.6, 139.9, 136.2, 133.9, 132.9, 132.2, 130.1, 129.7, 129.6, 128.84, 128.79, 128.6, 127.8, 127.6, 126.7, 126.2, 26.9, 26.2, 14.9; HRMS calcd for $C_{20}H_{20}NO^+$ $[M+H]^+$ 290.1545, Found 290.1542.

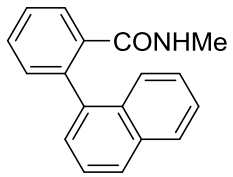



**2o**

**5-Fluoro-2'-isopropyl-*N*-methyl-[1,1'-biphenyl]-2-carboxamide**

Yellow oil; 79% yield (429 mg); $R_f$ = 0.34 (petroleum ether/ethyl acetate = 3:1, v/v).

IR ($cm^{-1}$, KBr): 3302, 1646, 1180, 831, 752, 606; $^1H$ NMR ($CDCl_3$, 400 MHz) δ (ppm) 8.01 (dd, *J* = 8.58, 5.98 Hz, 1H), 7.42-41 (m, 2H), 7.28-7.25 (m, 1H), 7.15-7.11 (m, 2H), 6.90 (d, *J* = 8.96, 2.36 Hz, 1H), 5.25 (br, 1H), 2.76-2.72 (m, 1H), 2.59 (d, *J* = 4.68 Hz, 3H), 1.14 (d, *J* = 6.72 Hz, 3H), 1.04 (d, *J* = 6.72 Hz, 3H); $^{13}C$ NMR ($CDCl_3$, 100 MHz) δ (ppm) 167.5, 163.4 (d, *J* = 252.1 Hz, 1C), 146.8, 141.7, 138.0, 132.1 (d, *J* = 8.9 Hz, 1C), 130.7, 129.2, 128.9, 126.3, 126.2, 117.4 (d, *J* = 21.8 Hz, 1C), 114.8 (d, *J* = 20.9 Hz, 1C), 29.9, 26.7, 24.7, 23.1; HRMS calcd for $C_{17}H_{19}FNO^+$ $[M+H]^+$ 272.1451, Found 272.1452.



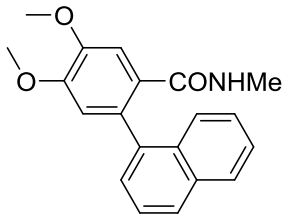

**2p**

***N*-Methyl-2-(naphthalen-1-yl)benzamide**

Whitish solid; 85% yield (444 mg); m.p. = 132 $^oC$; $R_f$ = 0.33 (petroleum ether/ethyl acetate = 3:1, v/v).

IR ($cm^{-1}$, KBr): 3296, 2957, 1645, 1539, 779, 669; $^1H$ NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.94-7.88 (m, 3H), 7.61 (d, *J* = 8.32 Hz , 1H), 7.56-7.48 (m, 4H), 7.45-7.41

(m, 2H), 7.38-7.36 (m, 1H), 5.14 (br, 1H), 2.34 (d, *J* = 4.92 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 169.1, 138.2, 137.6, 136.3, 133.6, 131.7, 131.3, 130.1, 129.2, 128.40, 128.36, 128.1, 126.9, 126.8, 126.2, 125.43, 125.38, 26.4; HRMS calcd for $C_{18}H_{16}NO^+$ $[M+H]^+$ 262.1232 , Found 262.1221.

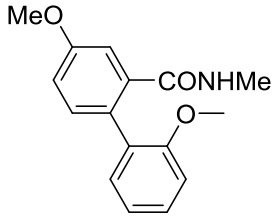



**2q**

**4,5-Dimethoxy-*N*-methyl-2-(naphthalen-1-yl)benzamide**

Yellow solid; 79% yield (508 mg); m.p. = 155 $^o$C; $R_f$ = 0.33 (petroleum ether/ethyl acetate = 2:1, v/v).

IR ($cm^{-1}$, KBr): 3293, 2934, 1647, 1506, 1211, 781, 623; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.91-7.88 (m, 2H), 7.62 (d, *J* = 8.32 Hz, 1H), 7.56 (s, 1H), 7.54-7.48 (m, 2H), 7.45-7.42 (m, 2H), 6.78 (s, 1H), 5.12 (br, 1H), 3.99 (s, 3H), 3.85 (s, 3H), 2.29 (d, *J* = 4.88 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 168.4, 150.2, 148.5, 138.2, 133.6, 131.9, 130.7, 128.5, 128.4, 128.1, 127.0, 126.9, 126.4, 125.5, 113.7, 112.2, 56.08, 56.06, 26.5; HRMS calcd for $C_{20}H_{20}NO_3^+$ $[M+H]^+$ 322.1443, Found 322.1440.



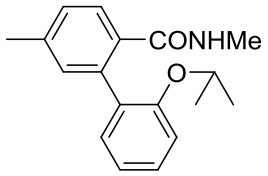

**2r**

**2',4-Dimethoxy-*N*-methyl-[1,1'-biphenyl]-2-carboxamide**

Yellow solid; 80% yield (434 mg); m.p. = 133 $^o$C; $R_f$ = 0.28 (petroleum ether/ethyl acetate = 2:1, v/v).

IR ($cm^{-1}$, KBr): 3318, 2957, 1647, 1238, 1043, 752; $^1$H NMR ($CDCl_3$, 600 MHz) δ (ppm) 7.33-7.31 (m, 2H), 7.22-7.17 (m, 2H), 7.01-6.91 (m, 3H), 5.49 (br, 1H), 3.84-3.74 (m, 6H), 2.64 (d, *J* = 4.80 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 150 MHz) δ (ppm)

169.6, 159.1, 156.6, 137.3, 132.3, 130.9, 129.4, 129.2, 128.1, 121.1, 116.8, 113.1, 110.8, 55.5, 26.5; HRMS calcd for $C_{16}H_{18}NO_3^+$ $[M+H]^+$ 272.1287, Found 272.1284.

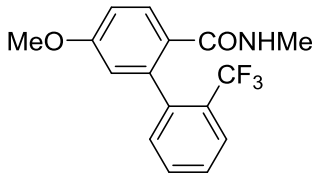



**2s**

**2'-Isopropoxy-*N*,5-dimethyl-[1,1'-biphenyl]-2-carboxamide**

Yellow oil; 68% yield (385 mg); $R_f$ = 0.35 (petroleum ether/ethyl acetate = 3:1, v/v).

IR ($cm^{-1}$, KBr): 3293, 2920, 1648, 1535, 826, 752; $^1H$ NMR ($CDCl_3$, 600 MHz) δ (ppm) 7.70 (d, *J* = 7.80 Hz, 1H), 7.31 (td, *J* = 7.86, 1.92 Hz, 1H), 7.22-7.20 (m, 2H), 7.03 (s, 1H), 7.01-6.98 (m, 1H), 6.93 (d, *J* = 8.22 Hz, 1H), 5.69 (br, 1H), 4.46-4.42 (m, 1H), 2.63 (d, *J* = 4.92 Hz, 3H), 2.38 (s, 3H), 1.20-1.19 (m, 6H); $^{13}C$ NMR ($CDCl_3$, 150 MHz) 169.7, 154.7, 140.0, 136.2, 133.4, 131.7, 131.1, 131.0, 129.3, 128.8, 128.2, 121.0, 113.8, 70.7, 26.5, 22.0, 21.4; HRMS calcd for $C_{18}H_{22}NO_2^+$ $[M+H]^+$ 284.1651, Found 284.1648.

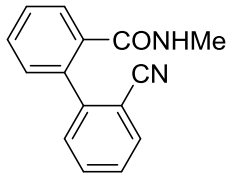



**2t**

**5-Methoxy-*N*-methyl-2'-(trifluoromethyl)-[1,1'-biphenyl]-2-carboxamide**

Yellow oil; 78% yield (483 mg); $R_f$ = 0.33 (petroleum ether/ethyl acetate = 2:1, v/v).

IR ($cm^{-1}$, KBr): 3298, 1653, 1559, 669; $^1H$ NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.74 (d, *J* = 7.80 Hz, 1H), 7.63 (d, *J* = 8.60 Hz, 1H), 7.56-7.52 (m, 1H), 7.49-7.46 (m, 1H ), 7.30 (d, *J* = 7.48 Hz, 1H), 6.92 (dd, *J* = 8.60, 1.48 Hz, 1H), 6.74 (d, *J* = 1.88 Hz, 1H), 5.44 (br, 1H), 3.80 (s, 3H), 2.60 (d, *J* = 4.84 Hz, 3H); $^{13}C$ NMR ($CDCl_3$, 100 MHz) δ (ppm) 168.8, 159.9, 139.4, 138.8, 131.6, 131.5, 129.8, 128.1, 127.9, 127.6 (d, *J* = 3.5 Hz, 1C), 126.1 (d, *J* = 5.2 Hz, 1C), 125.5, 116.1, 113.5, 55.4, 26.5; HRMS calcd for $C_{16}H_{15}F_3NO_2^+$ $[M+H]^+$ 310.1055, Found 310.1052.

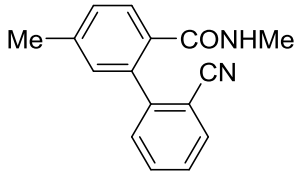



**2u**

**2'-Cyano-*N*-methyl-[1,1'-biphenyl]-2-carboxamide**

Yellow solid; 68% yield (321 mg); m.p. = 124 $^{o}$C; $R_f$ = 0.25 (petroleum ether/ethyl acetate = 3:1, v/v).

IR (cm$^{-1}$, KBr): 3308, 1653, 1541, 762; $^{1}$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.72 (d, *J* = 7.76 Hz, 1H), 7.65-7.60 (m, 2H), 7.56-7.44 (m, 4H), 7.38 (d, *J* = 7.33, 0.96 Hz, 1H), 5.72 (br, 1H), 2.81 (d, *J* = 4.88 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 169.4, 144.7, 136.7, 136.5, 132.8, 132.6, 130.8, 130.5, 130.2, 128.9, 127.9, 127.6, 126.4, 118.2, 111.8, 26.8; HRMS calcd for $C_{15}H_{12}N_2NaO^+$ $[M+Na]^+$ 259.0847, Found 259.0846.

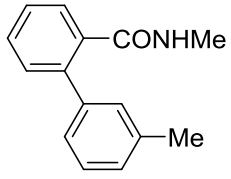



**2v**

**2'-Cyano-*N*,5-dimethyl-[1,1'-biphenyl]-2-carboxamide**

Yellow oil; 66% yield (330 mg); $R_f$ = 0.24 (petroleum ether/ethyl acetate = 3:1, v/v).

IR (cm$^{-1}$, KBr): 3300, 1648, 1420, 801, 735; $^{1}$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.56 (d, *J* = 7.85 Hz, 1H), 7.49-7.45 (m, 3H), 7.31-7.29 (m, 2H), 7.05 (d, *J* = 7.86 Hz, 1H), 6.81 (br, 1H), 2.73 (d, *J* = 4.7 Hz, 3H), 2.18 (s, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 169.6, 146.1, 145.0, 137.2, 132.9, 132.5, 132.2, 131.8, 130.9, 129.1, 128.3, 128.1, 127.5, 114.3, 26.8, 21.2; HRMS calcd for $C_{16}H_{15}N_2O^+$ $[M+H]^+$ 251.1184, Found 251.1182.

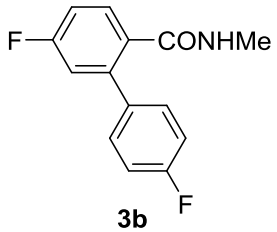



**3a**

***N*,3'-Dimethyl-[1,1'-biphenyl]-2-carboxamide**[4]

Yellow solid; 79% yield (356 mg); m.p. = 146-147 $^{o}$C (lit. 144-146 $^{o}$C); $R_f$ = 0.30 (petroleum ether/ethyl acetate = 3:1, v/v).

IR (cm$^{-1}$, KBr): 3279, 1634, 1314, 756, 700, 448; $^{1}$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.70 (d, *J* = 7.48 Hz, 1H), 7.46 (td, *J* = 7.48, 1.36 Hz, 1H), 7.42-7.35 (m, 2H), 7.31 (t, *J* = 7.48 Hz, 1H), 7.23-7.18 (m, 3H), 5.20 (br, 1H), 2.68 (d, *J* = 4.92 Hz, 3H), 2.40 (s, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 170.3, 140.1, 139.5, 138.3, 135.6, 130.1, 129.3, 128.9, 128.52, 128.46, 127.5, 125.7, 26.7, 21.4.

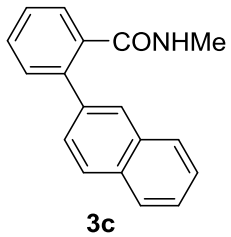



**3b**

**4',5-Difluoro-*N*-methyl-[1,1'-biphenyl]-2-carboxamide**

Whitish solid; 78% yield (386 mg); m.p. = 200 $^{o}$C; $R_f$ = 0.32 (petroleum ether/ethyl acetate = 3:1, v/v).

IR (cm$^{-1}$, KBr): 3242, 1626, 1223, 843, 610, 419; $^{1}$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.66 (dd, *J* = 8.44, 8.44 Hz, 3H), 7.39-7.36 (m, 2H), 7.15-7.03 (m, 4H), 5.19 (s, 1H), 2.71 (d, *J* = 4.88 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 169.2, 164.2(d, *J* = 45.7 Hz, 1C), 161.8(d, *J* = 43.6 Hz, 1C), 140.8(d, *J* = 8.3 Hz, 1C), 132.0(d, *J* = 3.3 Hz, 1C), 131.0(d, *J* = 8.9 Hz, 1C), 130.2(d, *J* = 8.13 Hz, 2C), 116.9(d, *J* = 22.1 Hz, 1C), 115.8(d, *J* = 21.8 Hz, 2C), 114.7(d, *J* = 21.5 Hz, 1C), 26.7; HRMS calcd for $C_{14}H_{12}F_2NO^+$ $[M+H]^+$ 248.0887, Found 248.0879.



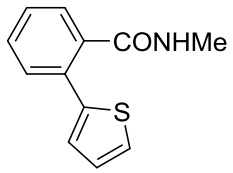

**3c**

***N*-Methyl-2-(naphthalen-2-yl)benzamide**

Yellow solid; 73% yield (381 mg); m. p. = 106 $^{o}$C; $R_f$ = 0.31 (petroleum ether/ethyl acetate = 3:1, v/v).

IR (cm$^{-1}$, KBr): 3298, 2922, 1631, 1410, 760, 662, 478; $^{1}$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.91-7.87 (m, 4H), 7.74 (d, *J* = 7.56 Hz, 1H), 7.55-7.47 (m, 5H),

7.46-7.42 (m, 1H), 5.27 (br, 1H), 2.62 (d, *J* = 4.88, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 170.2, 139.2, 137.7, 135.9, 133.4, 132.7, 130.5, 130.2, 129.0, 128.1, 127.7, 127.4, 126.9, 126.5, 126.4, 26.7; HRMS calcd for $C_{18}H_{15}NNaO^+$ $[M+Na]^+$ 284.1051, Found 284.1043.

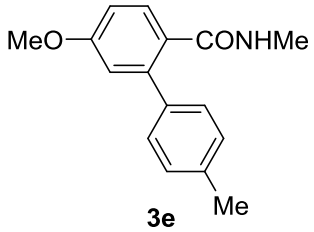



**3d**

***N*-Methyl-2-(thiophen-2-yl)benzamide**[5]

Yellow solid; 69% yield (300 mg); m.p. = 141 ℃; $R_f$ = 0.32 (petroleum ether/ethyl acetate = 3:1).

IR ($cm^{-1}$, KBr): 3318, 1628, 837, 818, 787, 770, 745; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.58 (d, *J* = 7.5 Hz, 1H), 7.45 (dtd, *J* = 9.1, 7.7, 1.4 Hz, 2H), 7.39-7.32 (m, 2H), 7.16 (dd, *J* = 3.6, 1.1 Hz, 1H), 7.07 (dd, *J* = 5.1, 3.6 Hz, 1H), 5.52 (br, 1H), 2.80 (d, *J* = 4.9 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 170.3, 141.3, 136.1, 131.7, 130.4, 129.9, 128.5, 128.0, 127.7, 126.7, 126.3, 26.8.

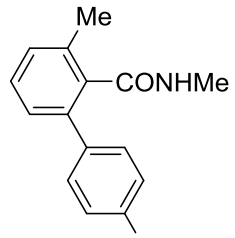



**3e**

**5-Methoxy-*N*,4'-dimethyl-[1,1'-biphenyl]-2-carboxamide**[4]

Whitish solid; 73% yield (373 mg); m.p. = 130-131 $^o$C (lit. 128-129 $^o$C); $R_f$ = 0.27 (petroleum ether/ethyl acetate = 2:1, v/v).

IR ($cm^{-1}$, KBr): 3293, 1647, 1559, 1221, 828; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.66 (d, *J* = 8.46, 2.78 Hz, 1H), 7.27-7.19 (m, 4H), 6.88 (d, *J* = 8.52 Hz, 1H), 6.80 (d, *J* = 2.24 Hz, 1H), 5.25 (br, 1H), 3.82 (s, 3H), 2.64 (d, *J* = 4.80 Hz, 3H), 2.38 (s, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 169.9, 160.6, 141.3, 137.7, 137.3, 130.9, 129.3, 128.5, 127.9, 115.4, 112.8, 55.4, 26.7, 21.2.

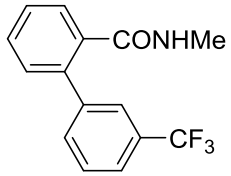



**3f**

***N*,3,4'-Trimethyl-[1,1'-biphenyl]-2-carboxamide**[4]

Whitish solid; 75% yield (359 mg); m.p. = 187 $^{o}$C (lit. 185-186 $^{o}$C); $R_f$ = 0.39 (petroleum ether/ethyl acetate = 3:1, v/v).

IR (cm$^{-1}$, KBr): 3231, 1624, 1458, 793, 714; $^{1}$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.34-7.28 (m, 3H), 7.20-7.18 (m, 4H), 5.23 (br, 1H), 2.66 (d, *J* = 4.92 Hz, 3H), 2.41 (s, 3H), 2.37 (s, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 170.8, 139.1, 137.6, 137.1, 136.4, 135.8, 129.11, 129.07, 128.9, 128.3, 127.2, 26.5, 21.1, 19.6.

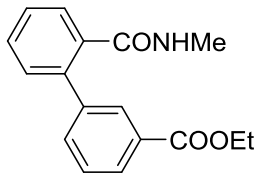



**3g**

***N*-Methyl-3'-(trifluoromethyl)-[1,1'-biphenyl]-2-carboxamide**

Whitish solid; 69% yield (385 mg); m.p. = 79 $^{o}$C; $R_f$ = 0.33 (petroleum ether/ethyl acetate = 3:1, v/v).

IR (cm$^{-1}$, KBr): 3264, 1645, 1335, 758, 702; $^{1}$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.67 (s, 1H), 7.62-7.60 (m, 3H), 7.54-7.37 (m, 4H), 5.32 (br, 1H), 2.72 (d, *J* = 4.88 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 170.0, 141.0, 138.0, 136.2, 132.1, 131.1 (d, *J* = 32.3 Hz, 1C), 130.4, 130.2, 129.0, 128.6, 128.3, 125.4 (d, *J* = 3.6 Hz, 1C), 124.5 (d, *J* = 3.6 Hz, 1C), 122.7, 26.7; HRMS calcd for $C_{15}H_{13}F_3NO^+$ $[M+H]^+$ 280.0949, Found 280.0947.

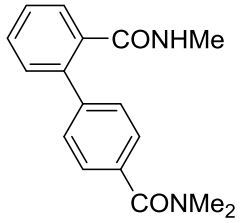



**3h**

**Ethyl 2'-(methylcarbamoyl)-[1,1'-biphenyl]-3-carboxylate**

Whitish oil; 69% yield (391 mg); $R_f$ = 0.30 (petroleum ether/ethyl acetate = 3:1, v/v).

IR ($cm^{-1}$, KBr): 3240, 1736, 1640, 1332, 1108, 881, 750, 701; $^1H$ NMR ($CDCl_3$, 400 MHz) δ (ppm) 8.08 (d, *J* = 1.7 Hz, 1H), 8.02 (d, *J* = 7.8 Hz, 1H), 7.73 (d, *J* = 7.1 Hz, 1H), 7.63-7.56 (m, 2H), 7.46 (d, *J* = 7.5 Hz, 1H), 7.39 (ddd, *J* = 12.3, 7.5, 4.3 Hz, 2H), 5.38 (br, 1H), 4.37 (q, *J* = 7.1 Hz, 2H), 2.68 (d, *J* = 4.9 Hz, 3H), 1.38 (t, *J* = 7.1 Hz, 3H); $^{13}C$ NMR ($CDCl_3$, 100 MHz) δ (ppm) 170.2, 166.4, 140.5, 138.5, 136.0, 134.7, 133.2, 131.4, 131.0, 130.3, 129.5, 128.9, 128.6, 126.9, 61.2, 26.7, 14.4; HRMS calcd for $C_{17}H_{18}NO_3^+$ $[M+H]^+$ 284.1287, Found 284.1288.

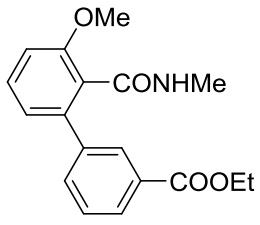



**3i**

***N*²,*N*⁴',*N*⁴'-Trimethyl-[1,1'-biphenyl]-2,4'-dicarboxamide**

Whitish oil; 67% yield (378 mg); $R_f$ = 0.12 (ethyl acetate).

IR ($cm^{-1}$, KBr): 3286, 1682, 1650, 850, 755; $^1H$ NMR (DMSO-$d_6$, 400 MHz) δ (ppm) 8.06 (br, 1H), 7.50 (ddd, *J* = 4.9, 4.5, 3.0 Hz, 1H), 7.45-7.38 (m, 7H), 2.96 (d, *J* = 18.4 Hz, 6H), 2.57 (d, *J* = 3.1 Hz, 3H); $^{13}C$ NMR (DMSO-$d_6$, 100 MHz) δ (ppm) 170.5, 170.0, 141.7, 138.8, 137.8, 135.6, 130.3, 130.0, 128.6, 128.3, 128.0, 127.4, 35.3, 26.5; HRMS calcd for $C_{17}H_{19}N_2O_2^+$ $[M+H]^+$ 283.1447, Found 283.1438.



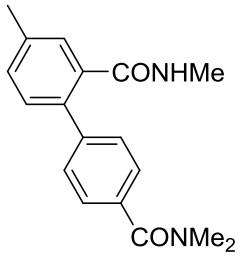

**3j**

**Ethyl 3'-methoxy-2'-(methylcarbamoyl)-[1,1'-biphenyl]-3-carboxylate**

Whitish oil; 68% yield (426 mg); $R_f$ = 0.25 (petroleum ether/ethyl acetate = 3:1, v/v).

IR ($cm^{-1}$, KBr): 3228, 1740, 1645, 1328, 1108, 891, 680; $^1H$ NMR ($CDCl_3$, 400 MHz) δ (ppm) 8.11 (td, *J* = 1.9, 0.4 Hz, 1H), 8.01 (ddd, *J* = 7.8, 1.6, 1.2 Hz, 1H), 7.70 (ddd, *J* = 7.7, 1.9, 1.2 Hz, 1H), 7.45 (td, *J* = 7.8, 0.4 Hz, 1H), 7.40 (dd, *J* = 8.3, 7.8 Hz,

1H), 7.01 (dd, *J* = 7.7, 0.9 Hz, 1H), 6.95 (dd, *J* = 8.4, 0.8 Hz, 1H), 5.47 (br, 1H), 4.41-4.36 (m, 2H), 3.88 (s, 3H), 2.73 (d, *J* = 4.9 Hz, 3H), 1.40 (t, *J* = 7.1 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 167.2, 165.6, 155.7, 139.4, 139.2, 132.0, 129.8, 129.4, 128.5, 127.8, 127.5, 125.2, 121.3, 109.5, 60.2, 55.1, 25.7, 13.5; HRMS calcd for $C_{18}H_{20}NO_4^+$ $[M+H]^+$ 314.1392, Found 314.1391.

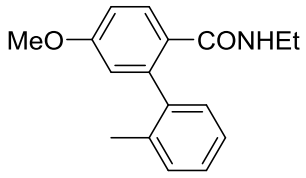



**3k**

***N*$^2$,*N*$^{4'}$,*N*$^{4'}$,4-Tetramethyl-[1,1'-biphenyl]-2,4'-dicarboxamide**

Brown oil; 66% yield (391 mg); $R_f$ = 0.11 (ethyl acetate).

IR ($cm^{-1}$, KBr): 3290, 1680, 1645, 883, 822; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.39-7.34 (m, 5H), 7.21 (dt, *J* = 12.0, 2.9 Hz, 2H), 5.56 (br, 1H), 3.00 (d, *J* = 34.9 Hz, 6H), 2.61 (d, *J* = 3.3 Hz, 3H), 2.33 (s, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 171.3, 170.5, 141.5, 138.0, 135.9, 135.6, 135.3, 130.9, 130.1, 129.3, 128.6, 127.4, 39.7, 35.5, 26.7, 21.0; HRMS calcd for $C_{18}H_{21}N_2O_2^+$ $[M+H]^+$ 297.1603, Found 297.1601.

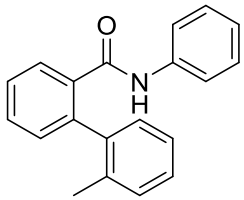



**3l**

***N*-Ethyl-5-methoxy-2'-methyl-[1,1'-biphenyl]-2-carboxamide**

Whitish solid; 73% yield (393 mg); m.p. = 141 $^o$C; $R_f$ = 0.34 (petroleum ether/ethyl acetate = 2:1, v/v).

IR ($cm^{-1}$, KBr): 3291, 1645, 1599, 1045, 480; $^1$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 7.91 (d, *J* = 8.72 Hz, 1H), 7.27-7.16 (m, 4H), 6.90 (dd, *J* = 8.68, 2.30 Hz, 1H), 6.63 (d, *J* = 2.16 Hz, 1H), 5.06 (br, 1H), 3.78 (s, 3H), 3.13-2.95 (m, 2H), 2.05 (s, 3H), 0.64 (t, J = 7.12 Hz, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 167.4, 160.9, 140.8, 140.4, 136.3, 131.7, 130.5, 128.9, 128.3, 127.1, 126.3, 115.3, 113.0, 55.4, 34.4, 19.9, 13.9; HRMS calcd for $C_{17}H_{19}NNaO_2^+$ $[M+Na]^+$ 292.1313, Found 292.1306.

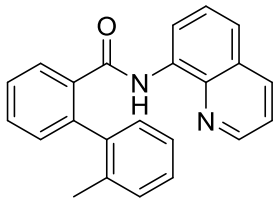


**3m**

**2'-Methyl-*N*-phenyl-[1,1'-biphenyl]-2-carboxamide**[4]

Whitish solid; 76% yield (437 mg); m. p. = 138 $^{o}$C (lit., 134-136 $^{o}$C); $R_f$ = 0.50 (petroleum ether/ethyl acetate = 10:1, v/v).

IR (cm$^{-1}$, KBr): 3063, 1651, 1441, 751, 690, 511; $^{1}$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 8.10 (d, *J* = 6.40 Hz, 1H), 7.53-7.46 (m, 2H), 7.36-7.30 (m, 4H), 7.23 (d, *J* = 6.12 Hz, 1H), 7.18-7.11 (m, 3H), 7.01-6.99 (m, 3H), 2.09 (s, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 165.7, 140.0, 139.1, 137.7, 136.6, 134.3, 131.0, 130.9, 130.5, 130.2, 129.3, 128.8, 128.7, 128.1, 126.7, 124.2, 119.7, 20.0.

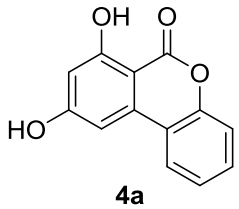


**3n**

**2'-Methyl-*N*-(quinolin-8-yl)-[1,1'-biphenyl]-2-carboxamide**

Yellow oil; 88% yield (596 mg); $R_f$ = 0.32 (petroleum ether/ethyl acetate = 20:1, v/v)

IR (cm$^{-1}$, KBr): 3047, 1682, 1520, 1068, 868, 667; $^{1}$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 9.82 (br, 1H), 8.79 (d, *J* = 7.44 Hz, 1H), 8.57 (d, *J* = 4.00 Hz, 1H), 8.07 (d, *J* = 8.20 Hz, 1H), 8.03 (dd, J = 7.28, 1.24 Hz, 1H), 7.57-7.43 (m, 4H), 7.38-7.32 (m, 3H), 7.21-7.11 (m, 3H), 2.26 (s, 3H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 167.2, 147.6, 140.0, 139.8, 138.2, 136.1, 134.7, 131.2, 130.3, 130.0, 129.9, 129.4, 127.8, 127.7, 127.6, 127.4, 126.7, 125.9, 121.9, 121.5, 121.3, 116.4, 20.3; HRMS calcd for $C_{23}H_{19}N_2O^+$ $[M+H]^+$ 339.1497, Found 339.1490.

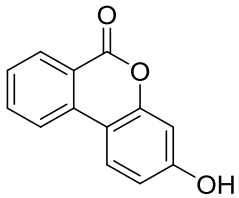



**4a**

**7,9-Dihydroxy-6*H*-benzo[c]chromen-6-one**[6]

Whitish solid; 65% yield (297 mg); m.p. = 267-269 $^{o}$C; $R_f$ = 0.11 (petroleum ether/ethyl acetate = 5:1, v/v).

IR (cm$^{-1}$, KBr): 3231, 1685, 1610, 1460, 1278; $^{1}$H NMR (DMSO-$d_6$, 600 MHz) δ (ppm) 11.32 (s, 1H), 11.05 (s, 1H), 8.16 (d, *J* = 8.0 Hz, 1H), 7.57 (t, *J* = 7.7 Hz, 1H), 7.41 (dd, *J* = 16.5, 8.1 Hz, 2H), 7.19 (d, *J* = 1.7 Hz, 1H), 6.48 (d, *J* = 2.0 Hz, 1H); $^{13}$C NMR (DMSO-$d_6$, 100 MHz) δ (ppm) 166.8, 165.2, 164.5, 151.1, 137.6, 132.0, 126.2, 124.9, 118.7, 118.3, 103.6, 101.7, 99.3.

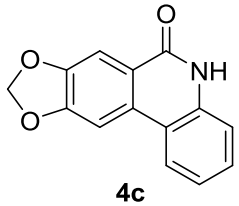



**4b**

**3-Hydroxy-6*H*-benzo[c]chromen-6-one**[7]

Brown solid; 68% yield (289 mg); m.p. = 205-206 $^{o}$C (lit. 207 $^{o}$C); $R_f$ = 0.15 (petroleum ether/ethyl acetate = 5:1, v/v).

IR (cm$^{-1}$, KBr): 3260, 1690, 1620, 1604, 1322, 1278; $^{1}$H NMR (DMSO-$d_6$, 400 MHz) δ (ppm) 10.31 (s, 1H), 8.23 (d, *J* = 5.4 Hz, 1H), 8.15 (dd, *J* = 8.5, 3.3 Hz, 2H), 7.85 (ddd, *J* = 5.5, 4.9, 0.9 Hz, 1H), 7.56-7.50 (m, 1H), 6.81 (dd, *J* = 5.8, 1.6 Hz, 1H), 6.72 (d, *J* = 1.6 Hz, 1H); $^{13}$C NMR (DMSO-$d_6$, 150 MHz) δ (ppm) 161.2, 160.4, 152.7, 135.8, 135.7, 130.2, 128.2, 125.4, 122.2, 119.5, 113.7, 109.9, 103.5.

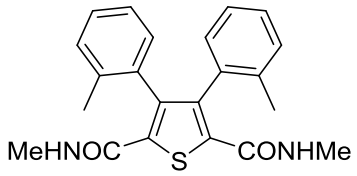



**4c**

**[1,3]Dioxolo[4,5-*j*]phenanthridin-6(5*H*)-one**[8]

Whitish solid; 68% yield (325 mg); m.p. >300 $^{o}$C; $R_f$ = 0.10 (petroleum ether/ethyl acetate = 1:1, v/v).

IR (cm$^{-1}$, KBr): 1660, 1580, 1471, 1422, 1248, 1009; $^{1}$H NMR ($CDCl_3$, 400 MHz) δ (ppm) 11.61 (br, 1H), 8.28 (d, *J* = 5.0 Hz, 1H), 8.02 (s, 1H), 7.62 (s, 1H), 7.41 (t, *J* = 5.0 Hz, 1H), 7.32 (d, *J* = 5.4 Hz, 1H), 7.21 (t, *J* = 5.0 Hz, 1H), 6.21 (s, 2H); $^{13}$C NMR ($CDCl_3$, 100 MHz) δ (ppm) 160.2, 152.2, 148.1, 135.9, 131.3, 128.7, 123.2, 122.1, 121.1, 117.8, 115.9, 105.1, 102.3, 101.6.




**4d**

***N*$^{2}$,*N*$^{5}$-Dimethyl-3,4-di-*o*-tolylthiophene-2,5-dicarboxamide**

Yellow oil; 62% yield (469 mg); $R_f$ = 0.12 (petroleum ether/ethyl acetate = 1:2, v/v).

IR (cm$^{-1}$, KBr): 3300, 3120, 1650, 1537, 1414, 740, 695; $^{1}$H NMR (DMSO-$d_6$, 400 MHz) δ (ppm) 8.14 (br, 2H), 7.31 (m, 4H), 7.26-7.17 (m, 4H), 2.74 (d, *J* = 4.8 Hz, 6H), 2.32 (s, 6H); $^{13}$C NMR (DMSO-$d_6$, 100 MHz) δ (ppm) 165.3, 145.6, 138.3, 136.5, 132.3, 130.7, 130.1, 127.8, 127.0, 27.2, 21.6; HRMS calcd for $C_{22}H_{23}N_2O_2S^+$ $[M+H]^+$ 379.1480, Found 379.1483.

## 10. References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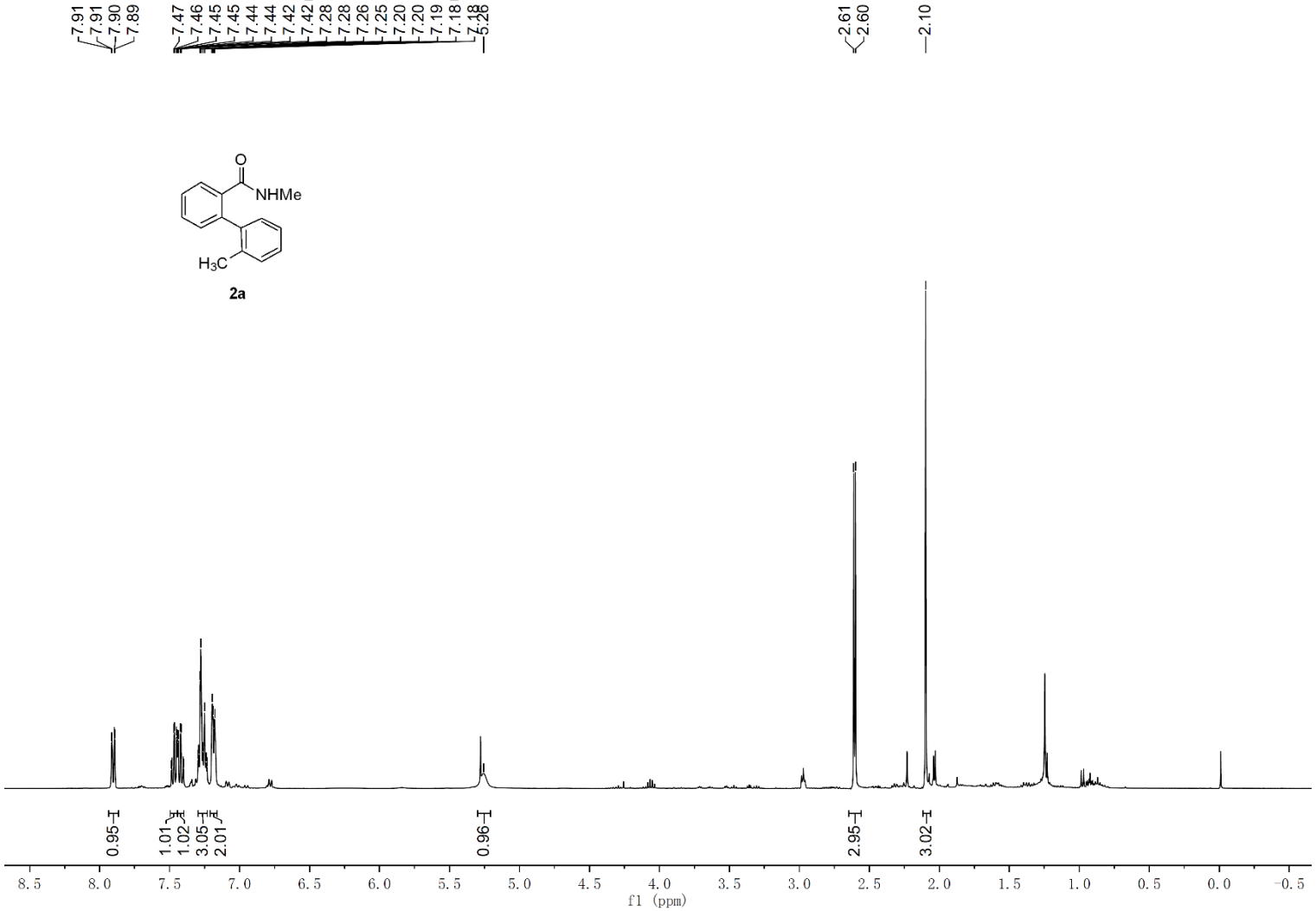
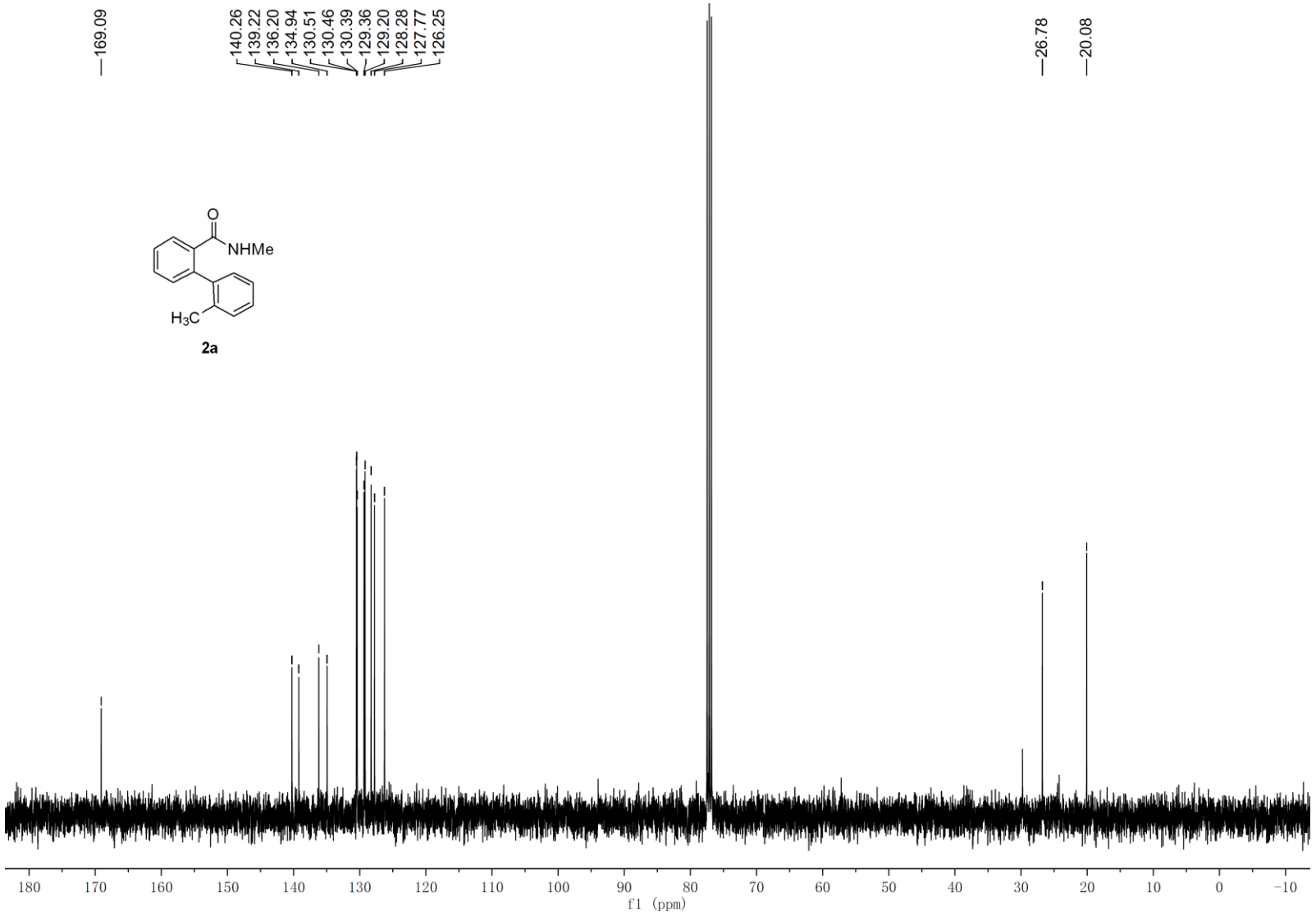
# 11. $^{1}$H and $^{13}$C NMR spectra for products

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20.08
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H3C
2a
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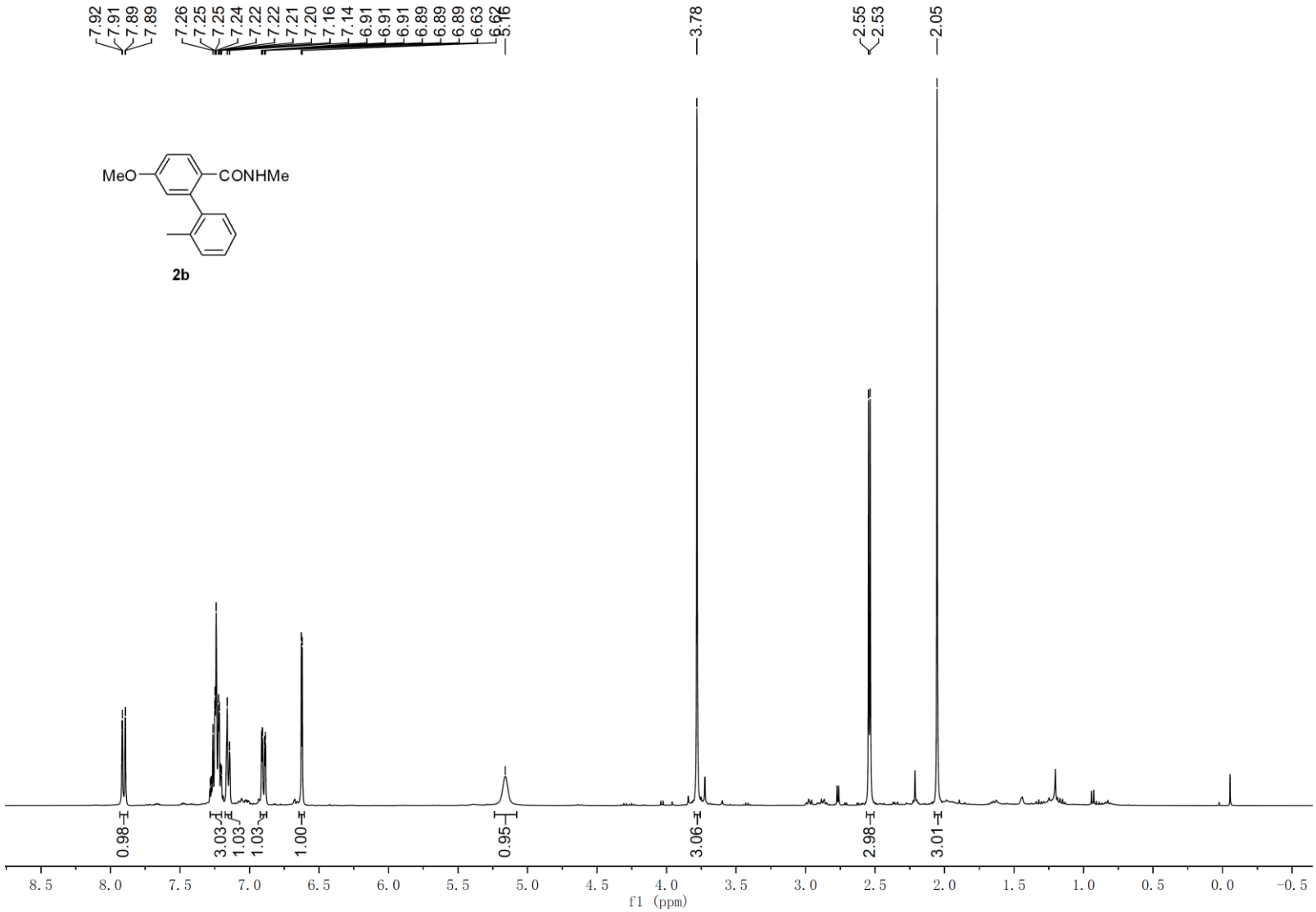
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6.89
6.89
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3.78
2.55
2.53
2.05
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CONHMe
2b
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3.03
1.03
1.03
1.00
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3.06
2.98
3.01
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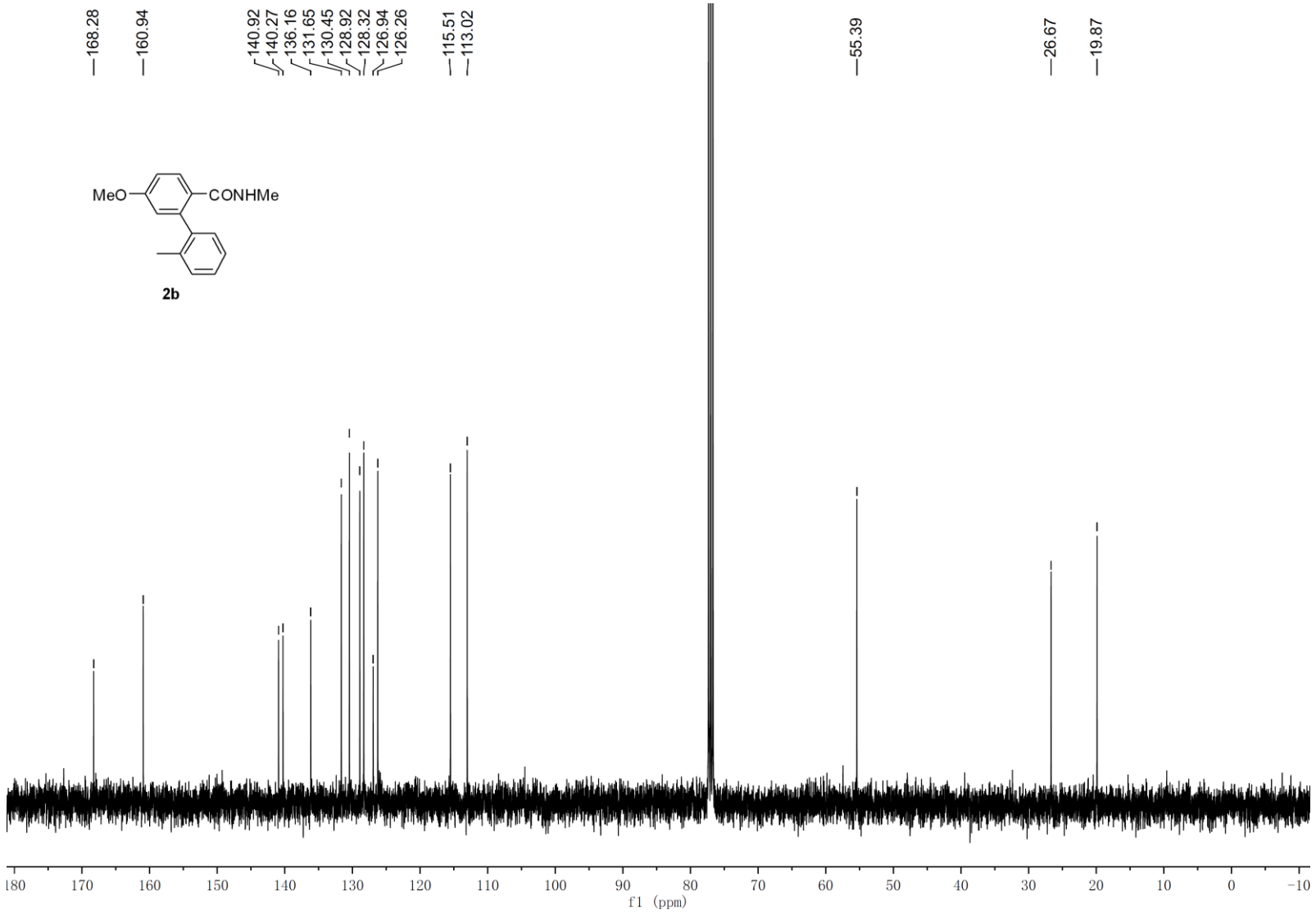
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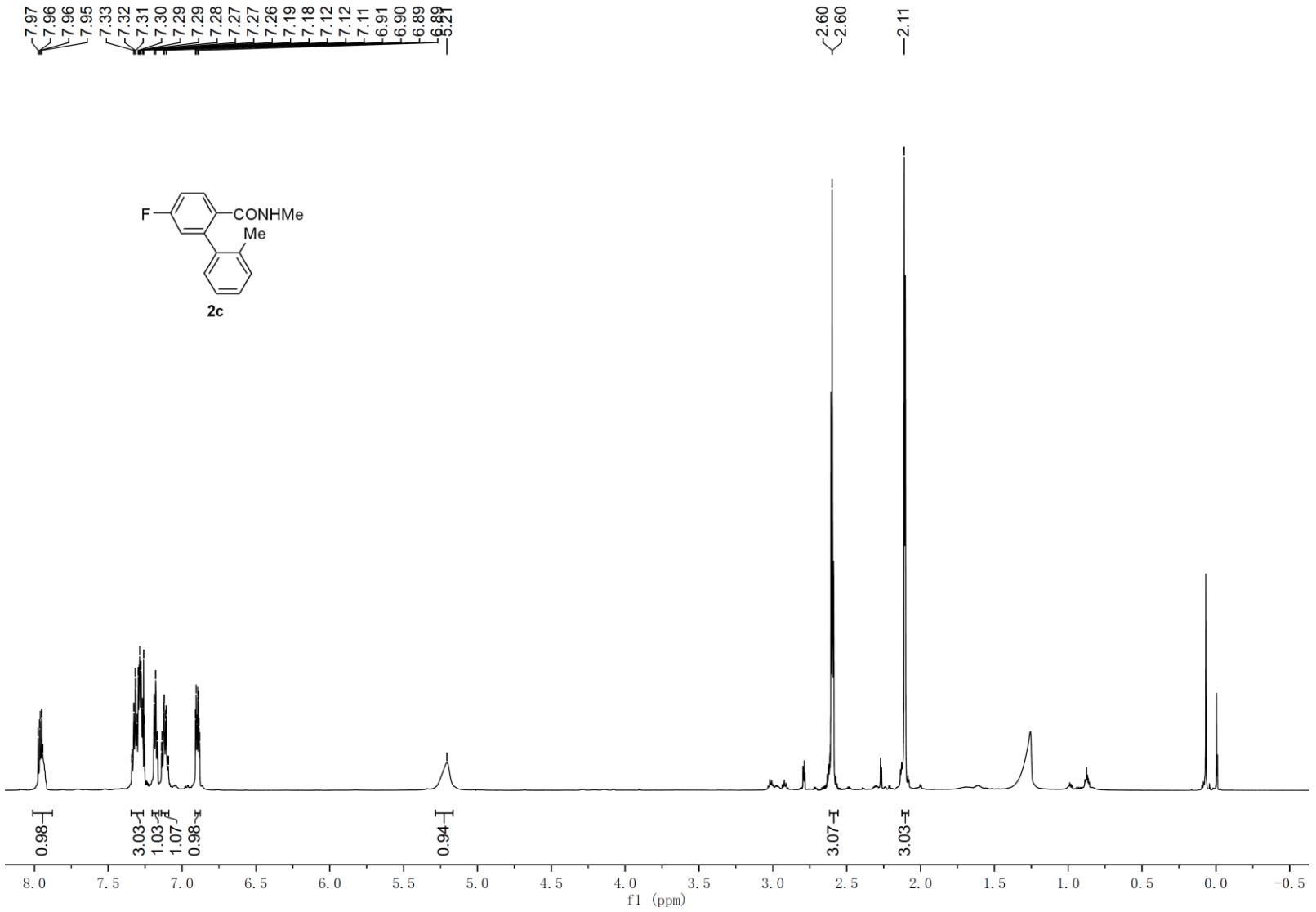
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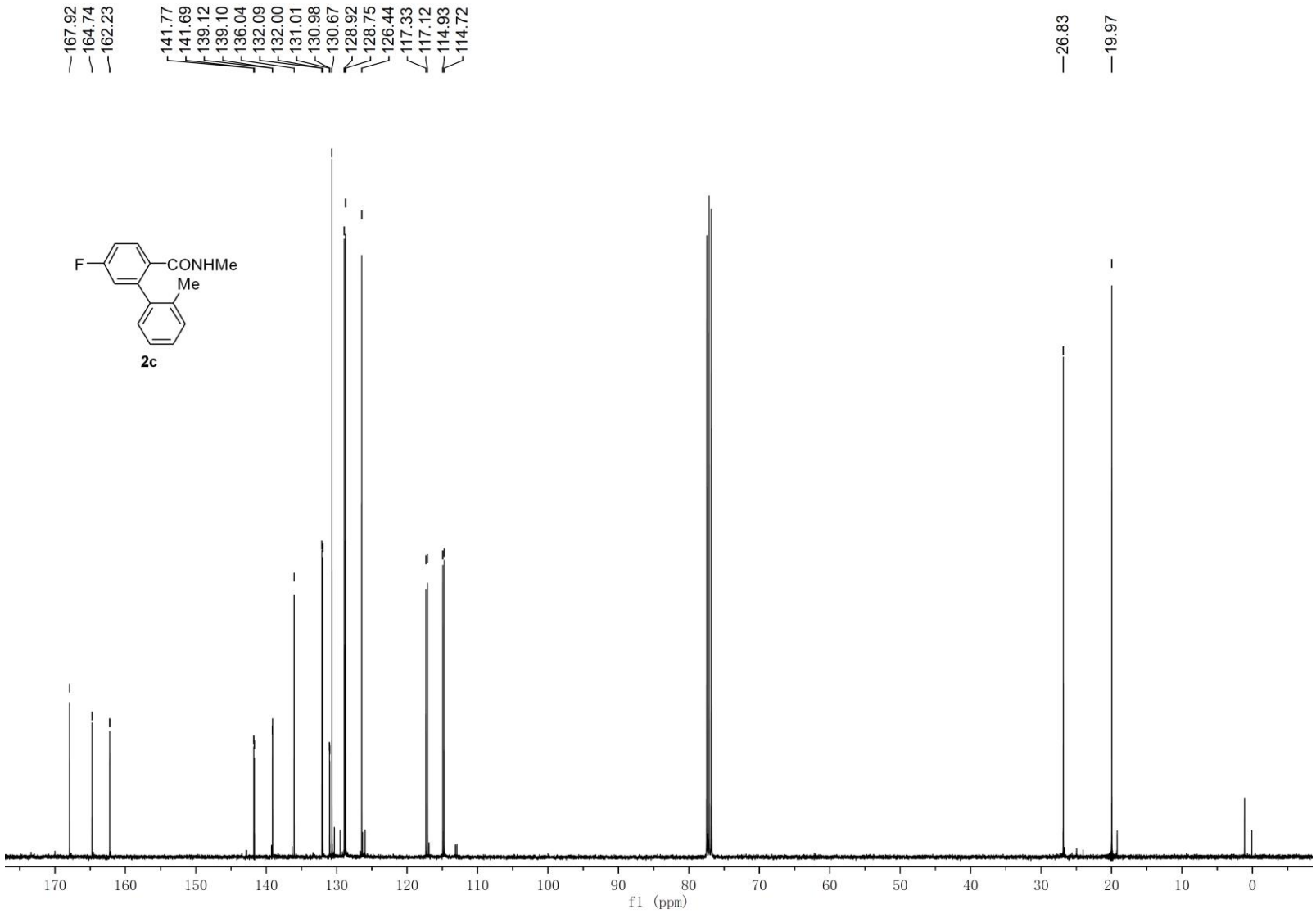
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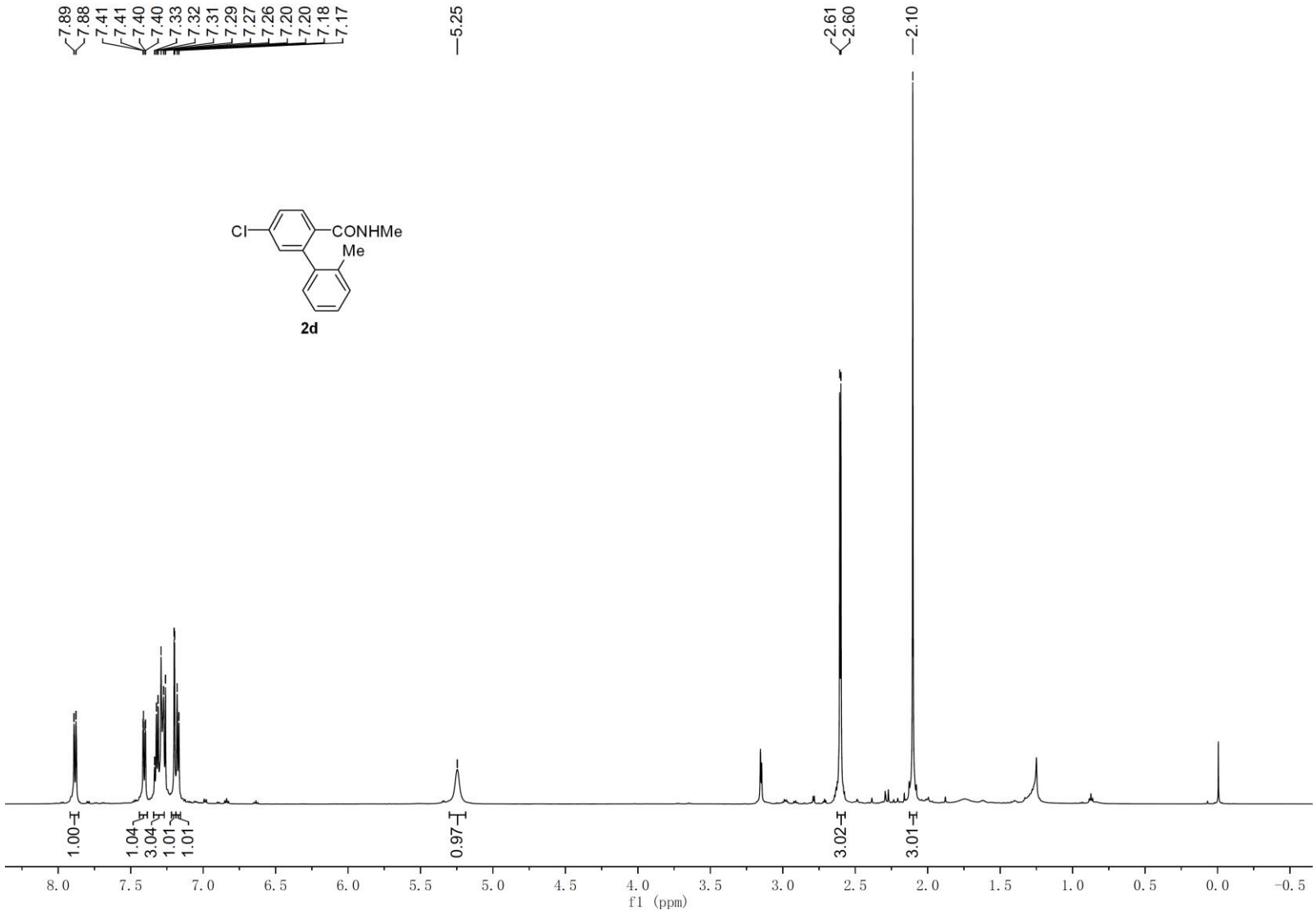
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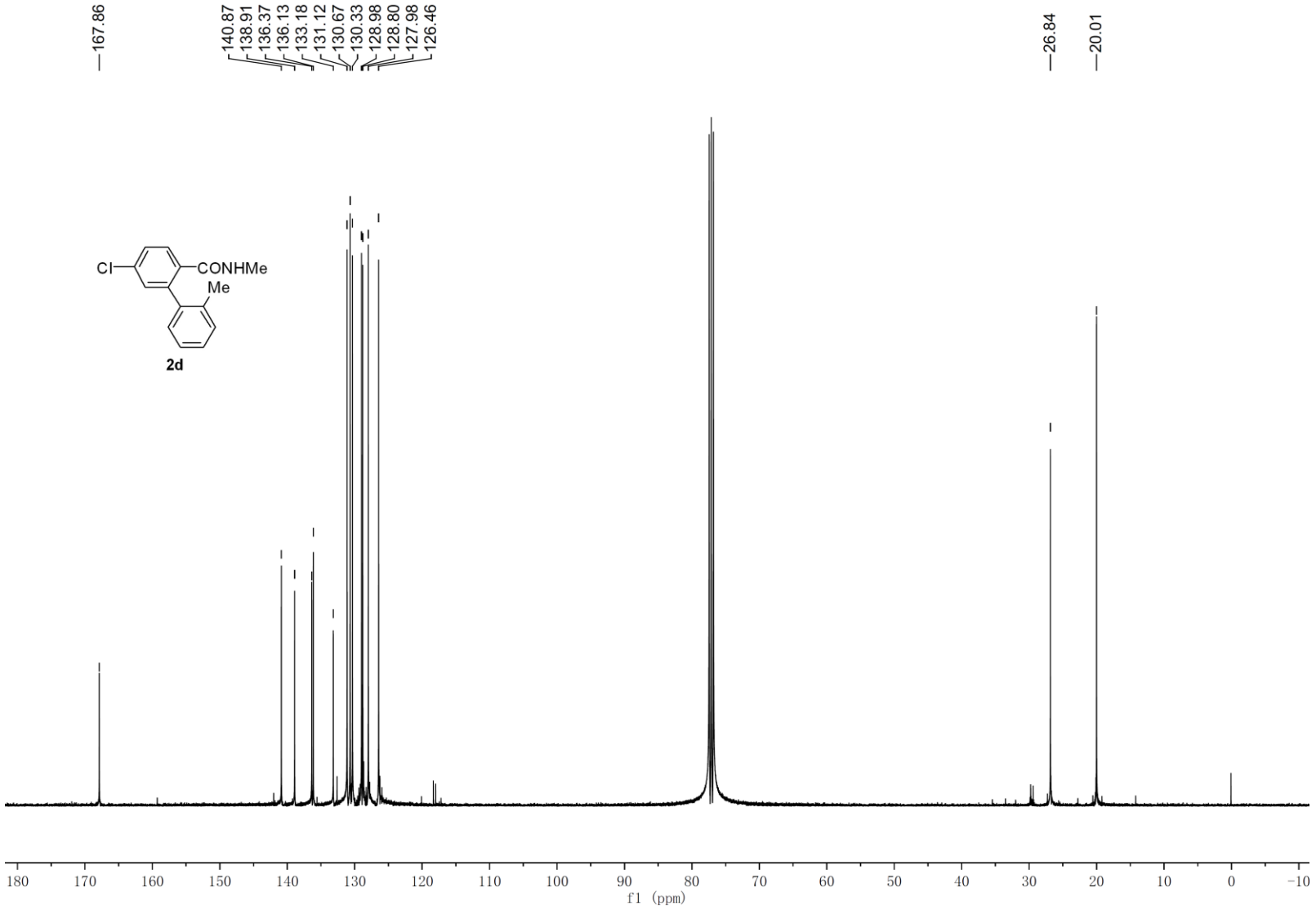
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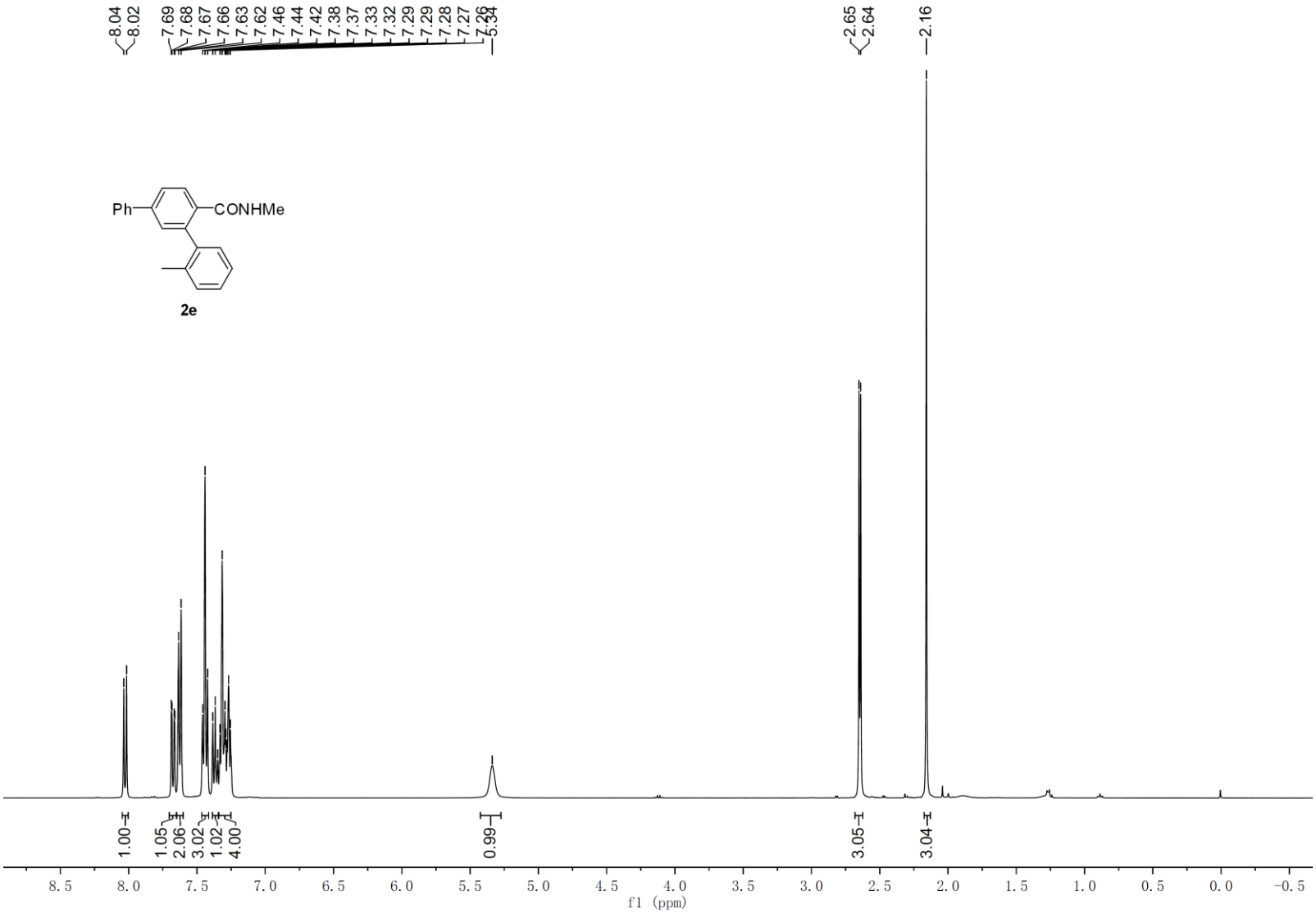
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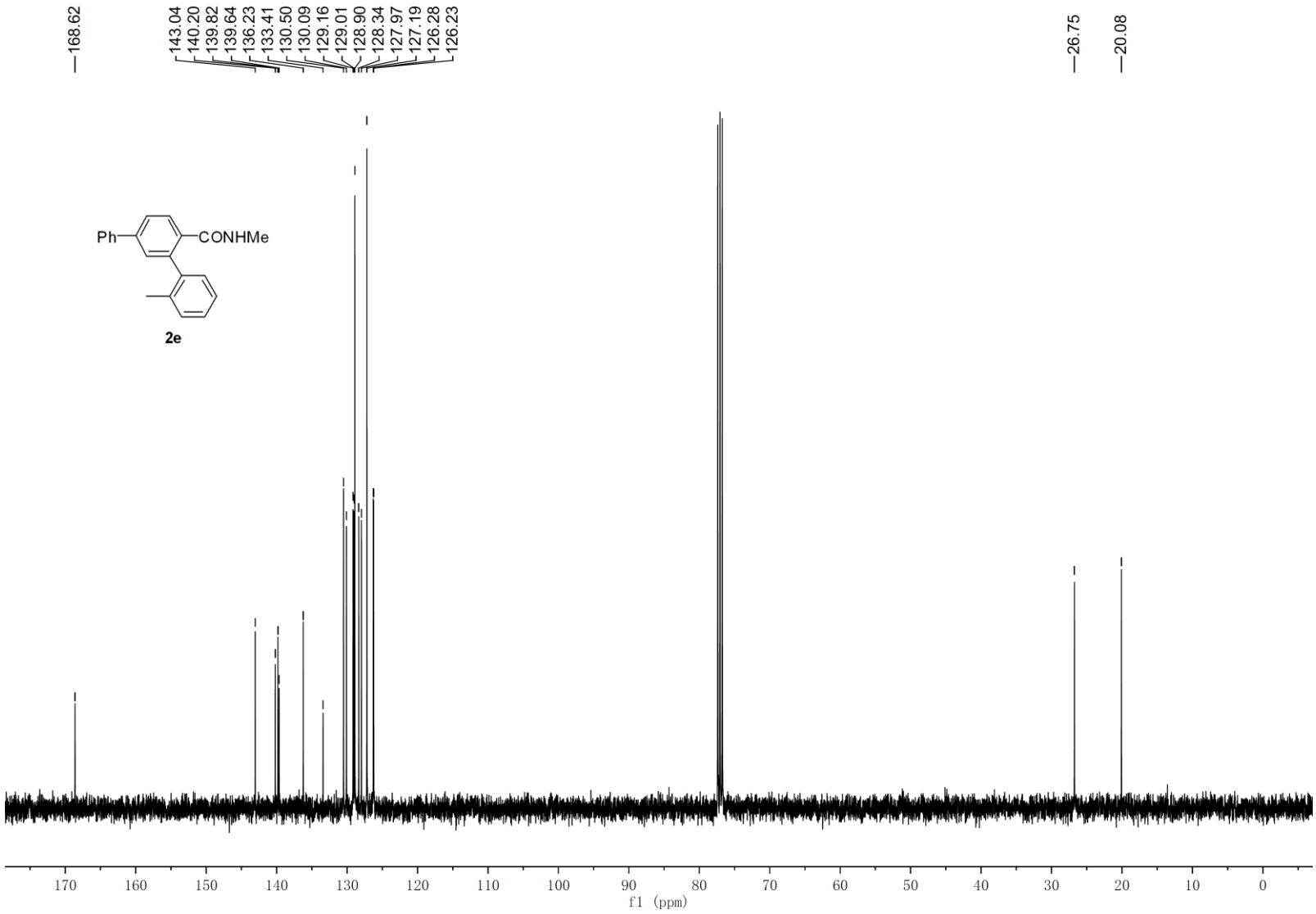
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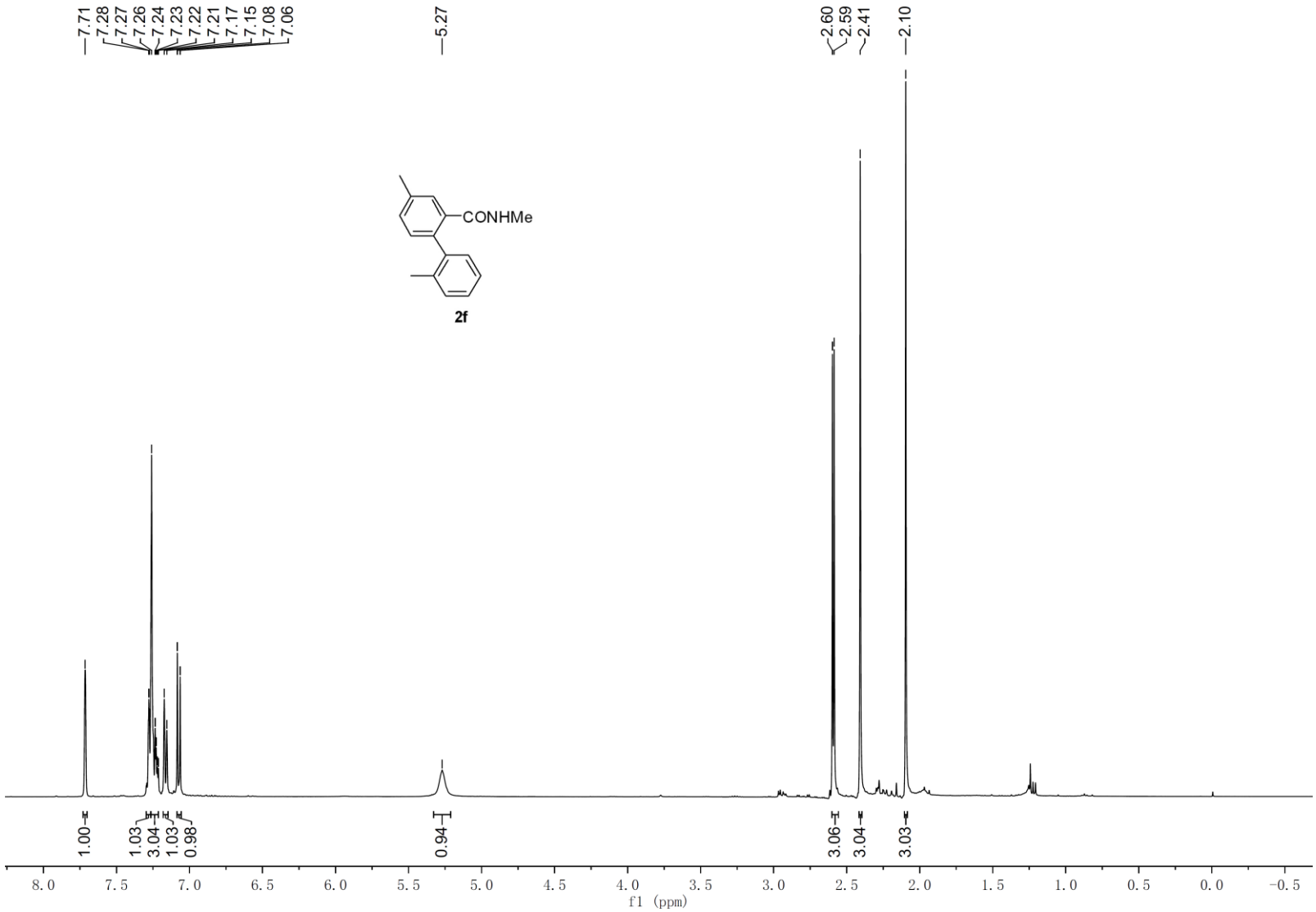
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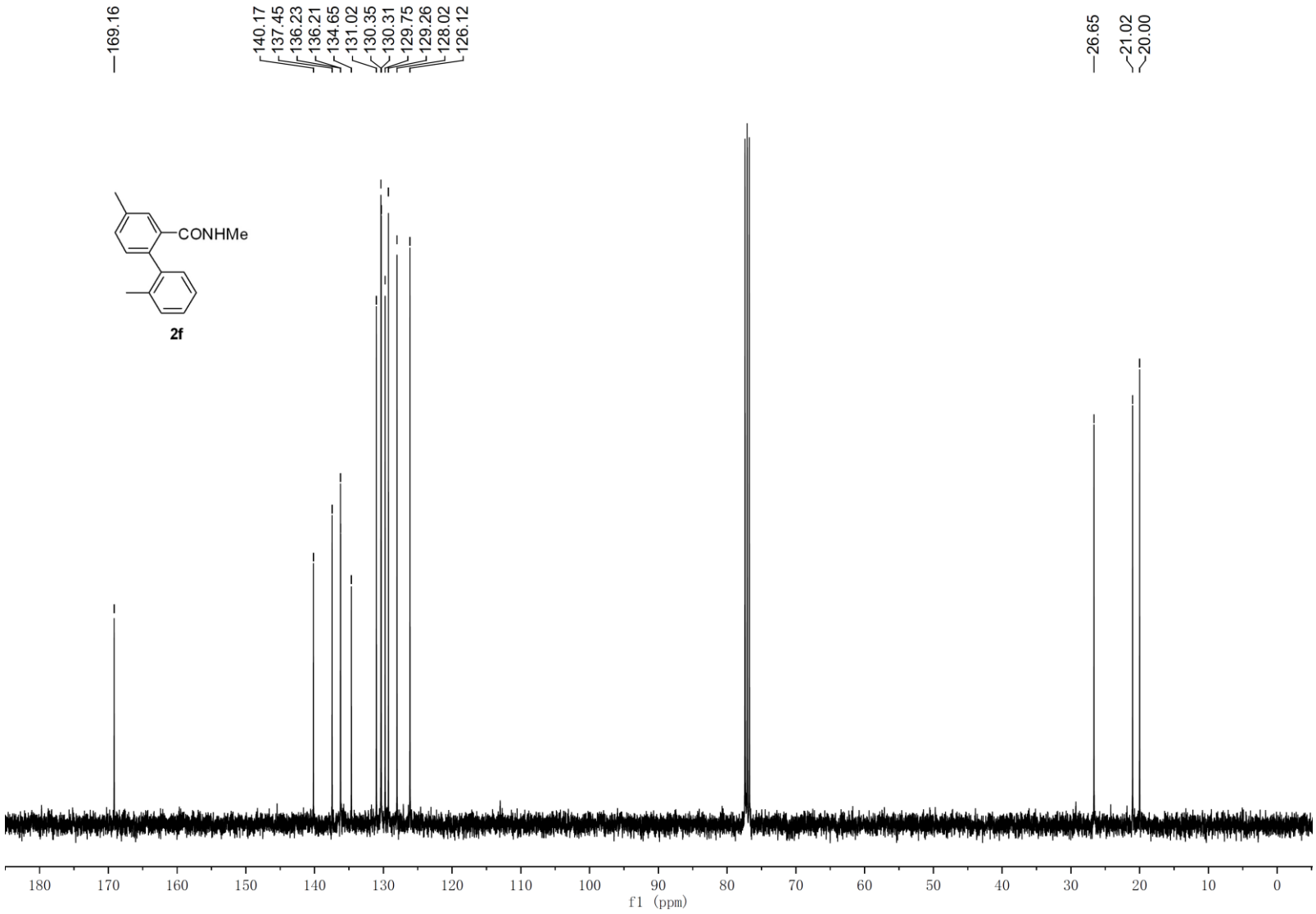
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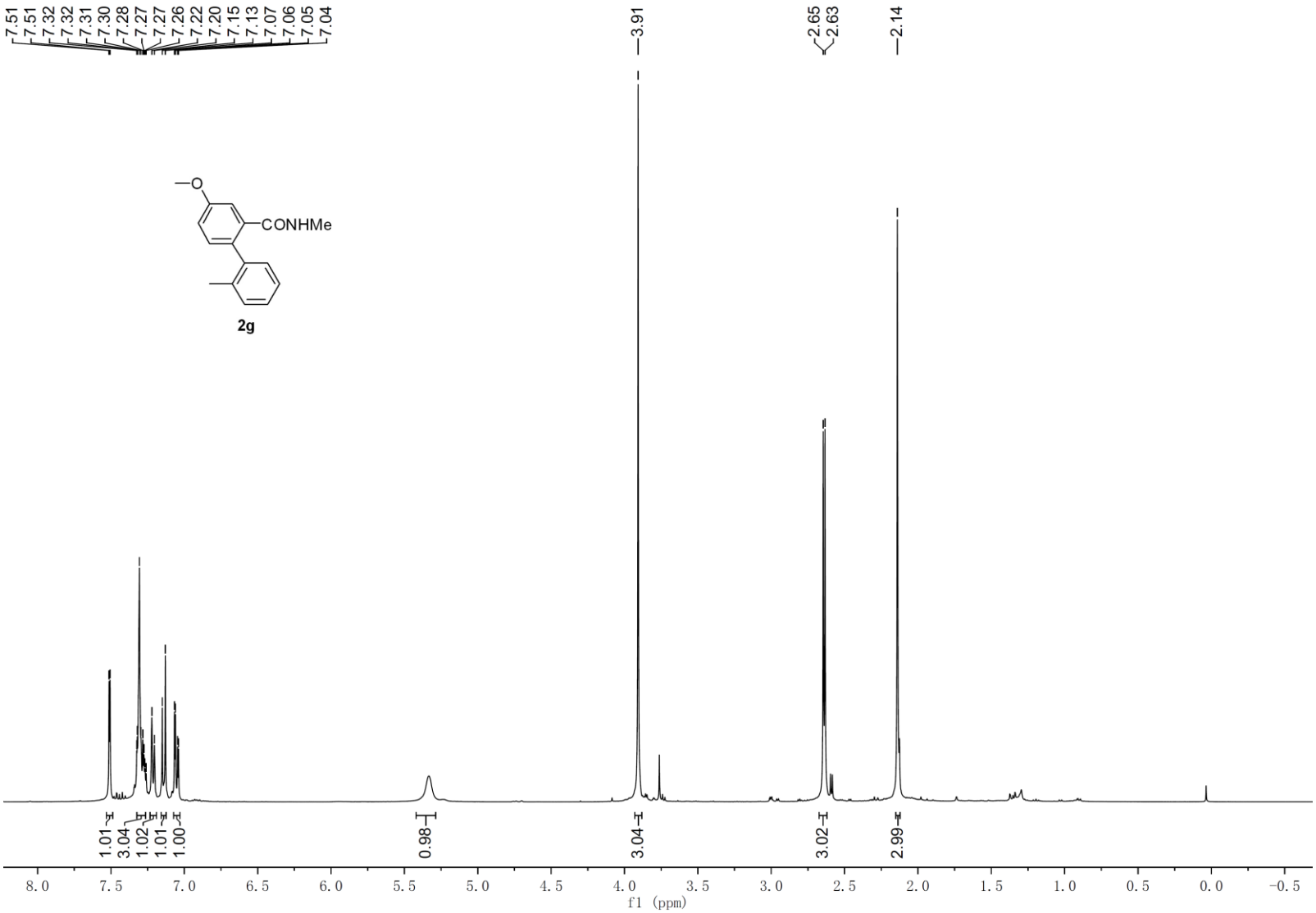
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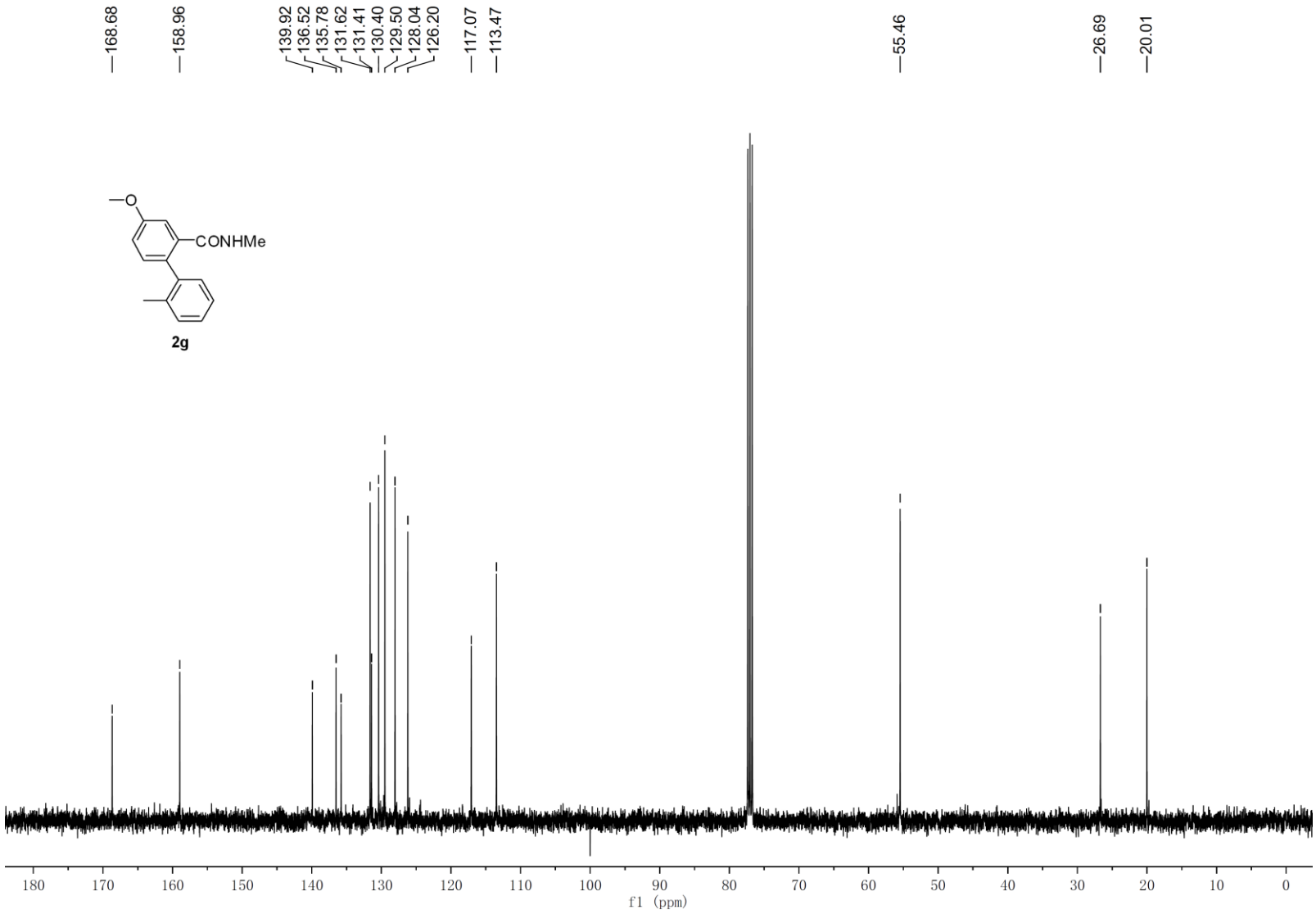
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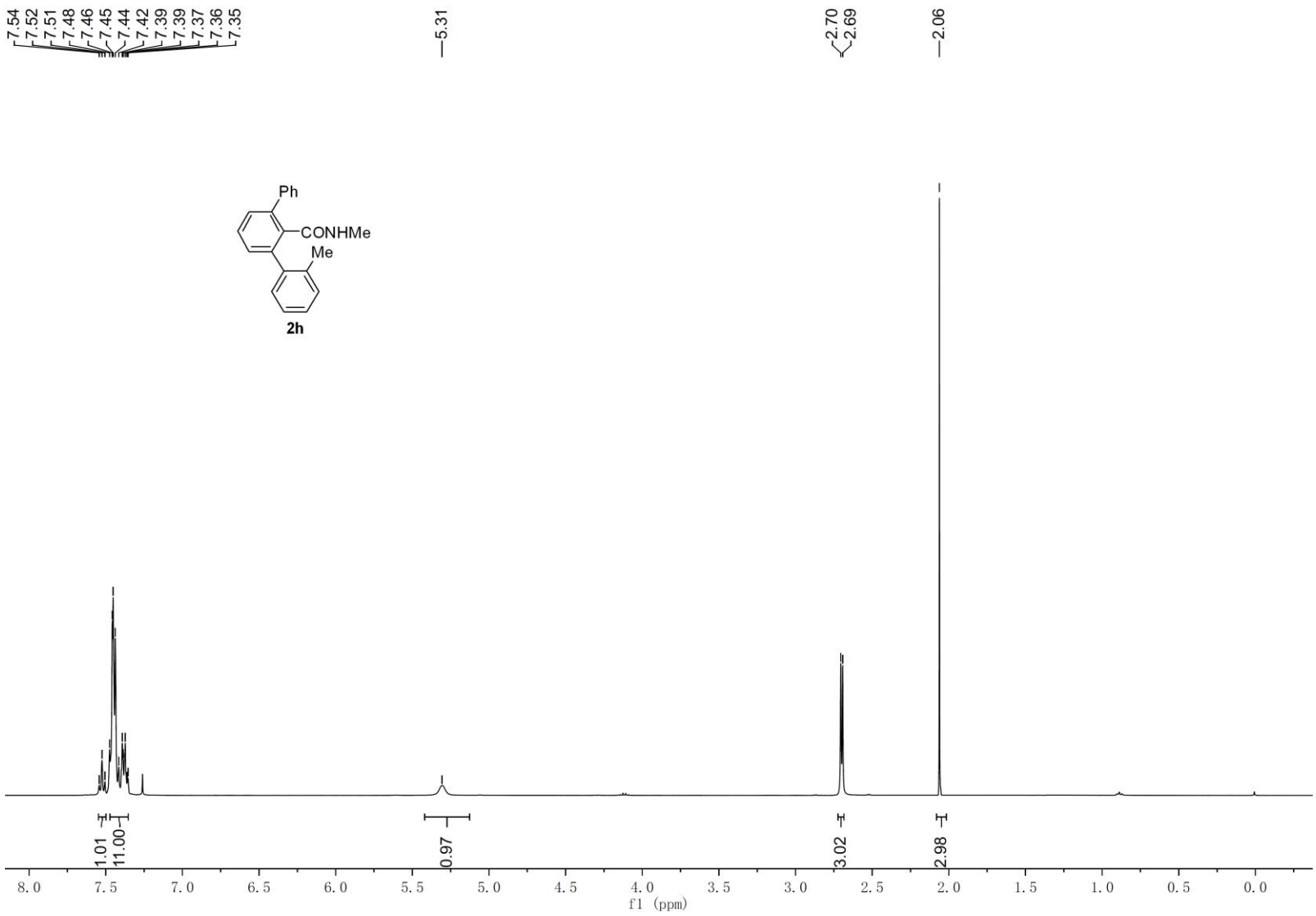
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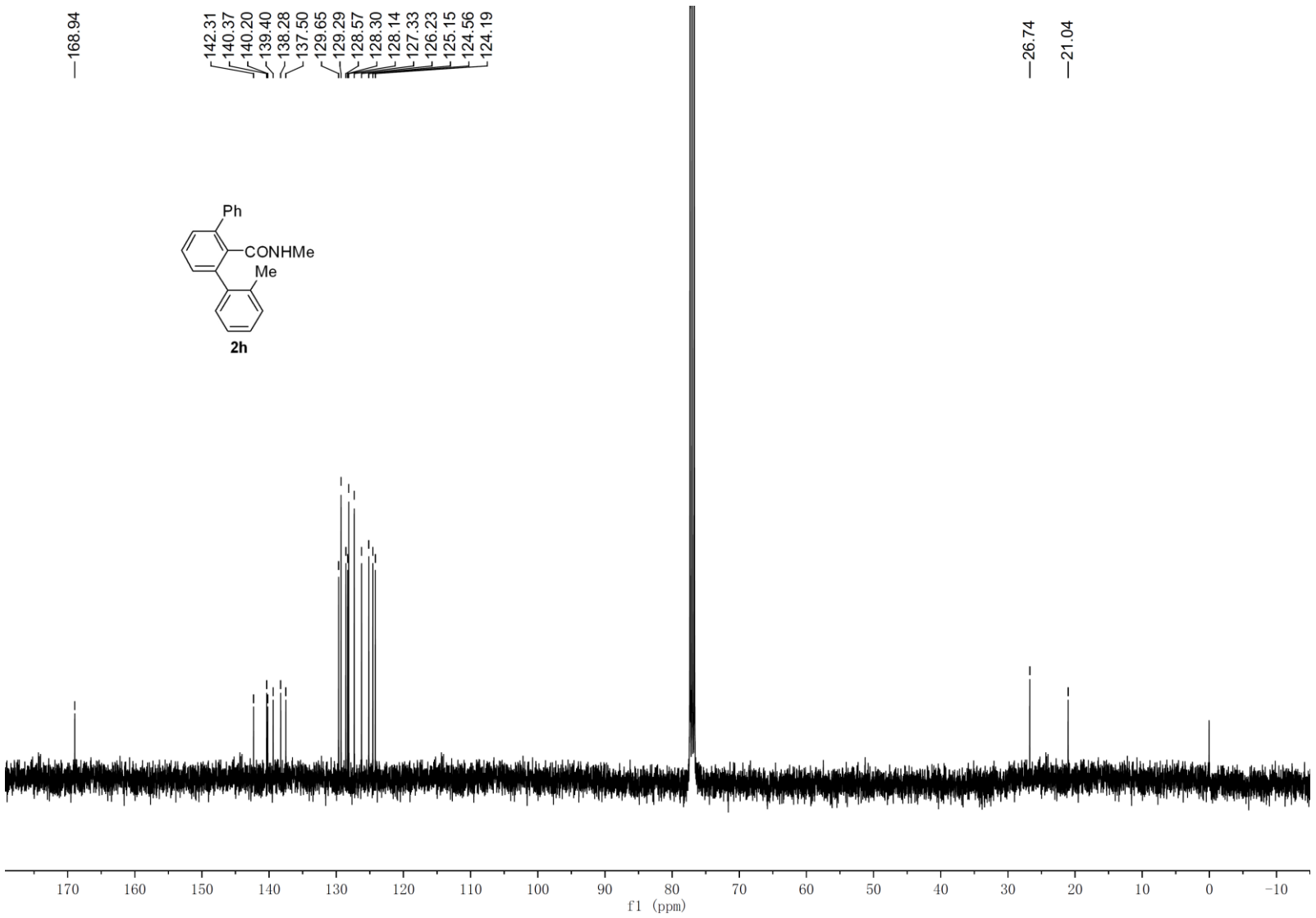
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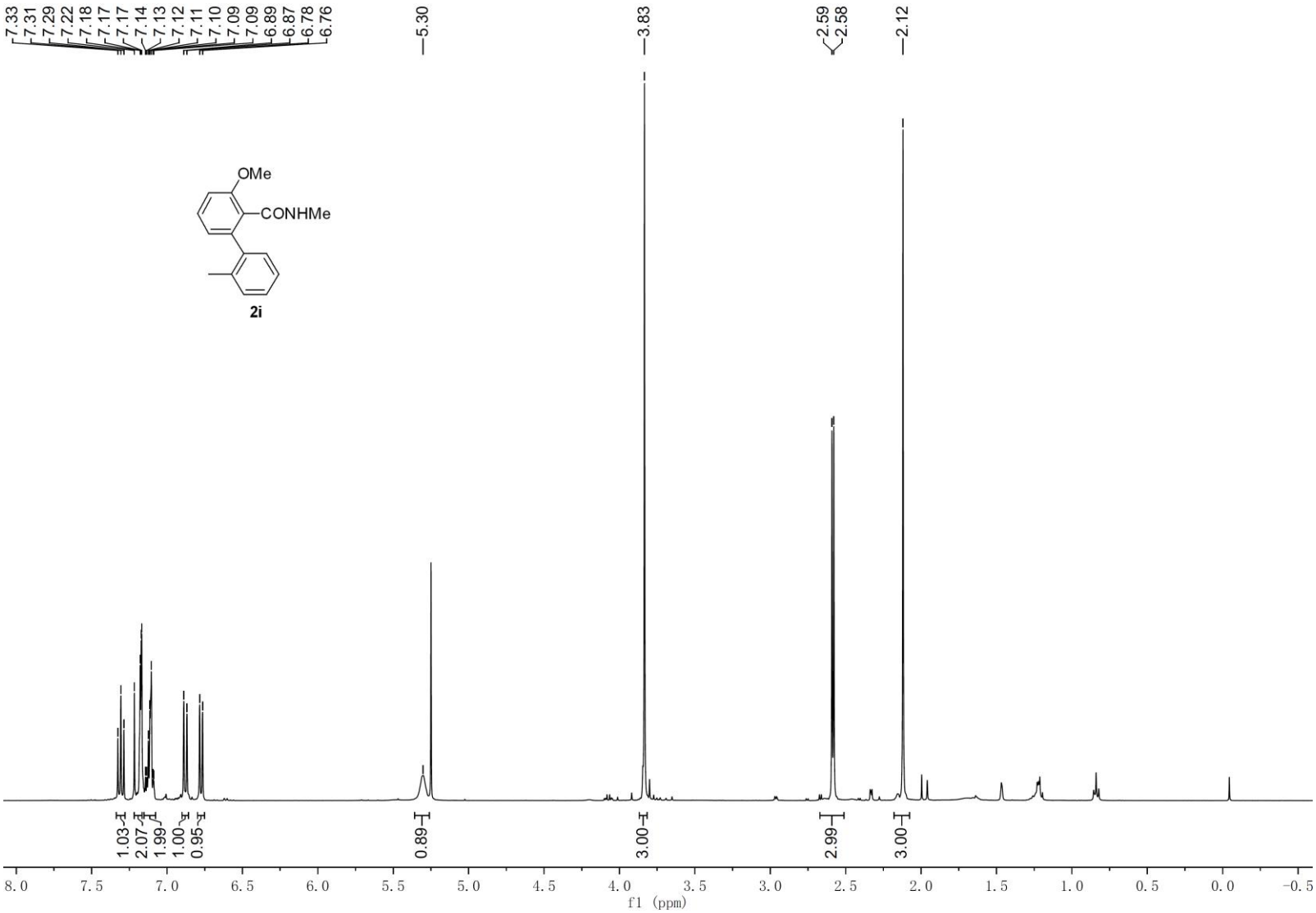
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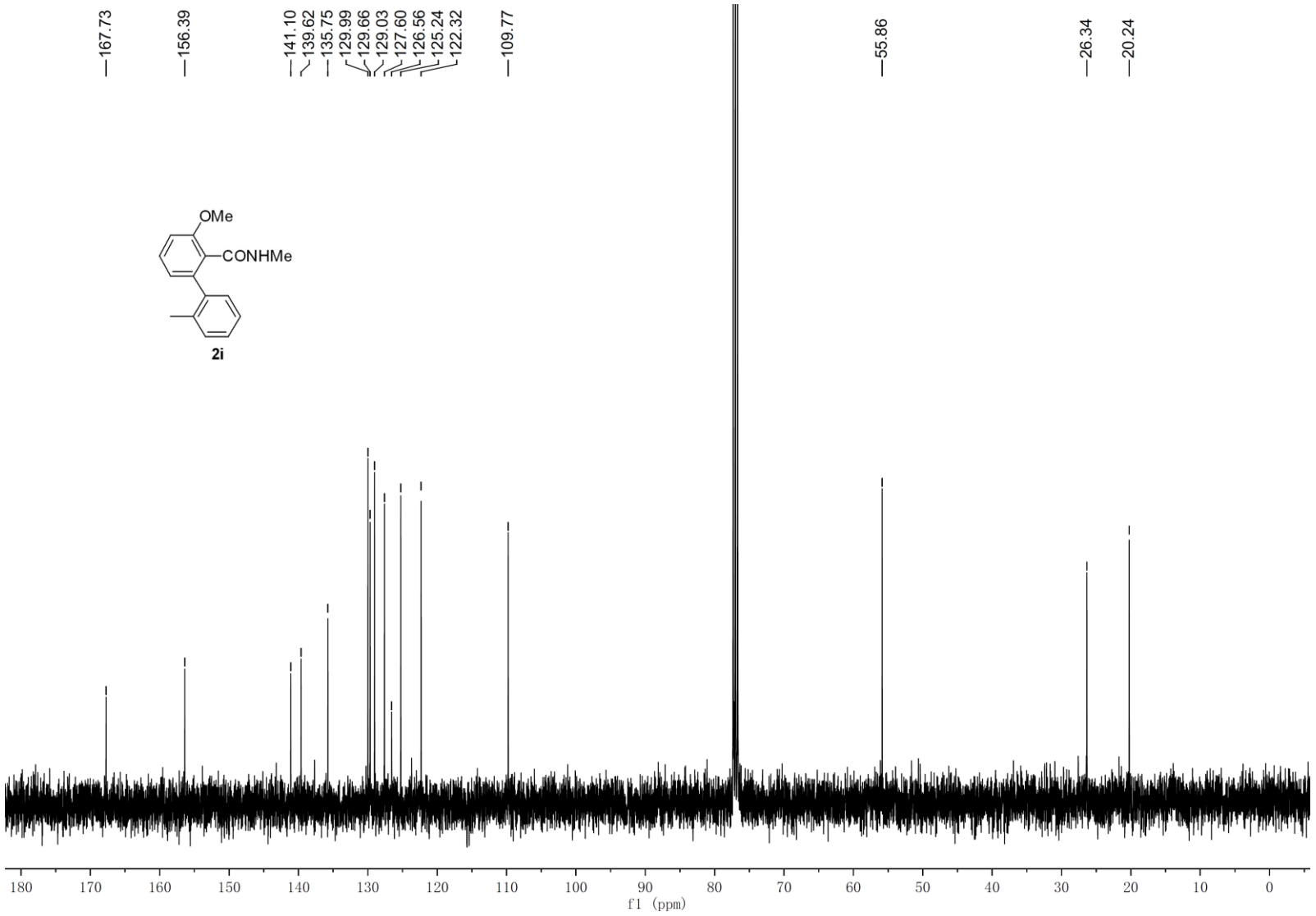
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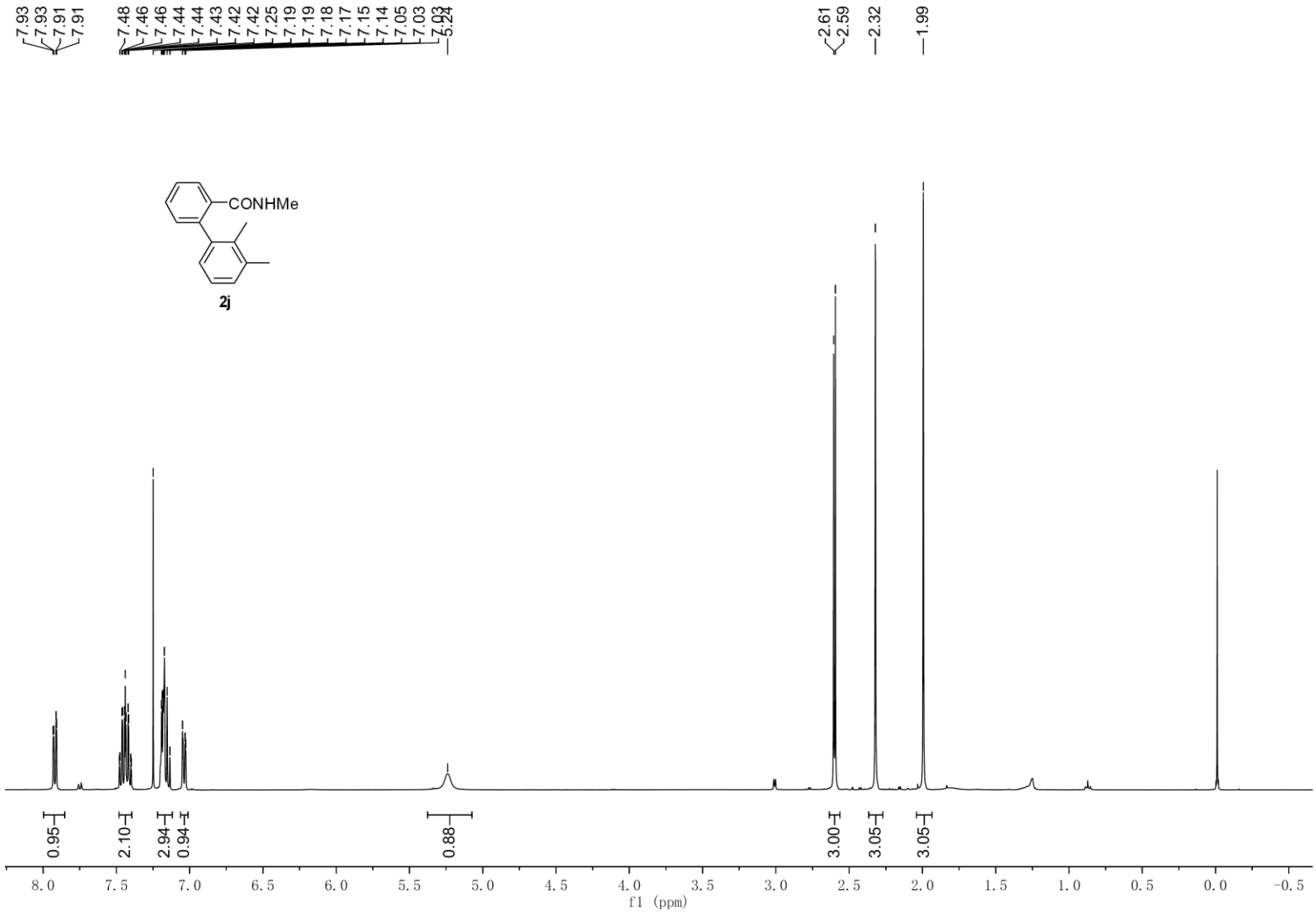
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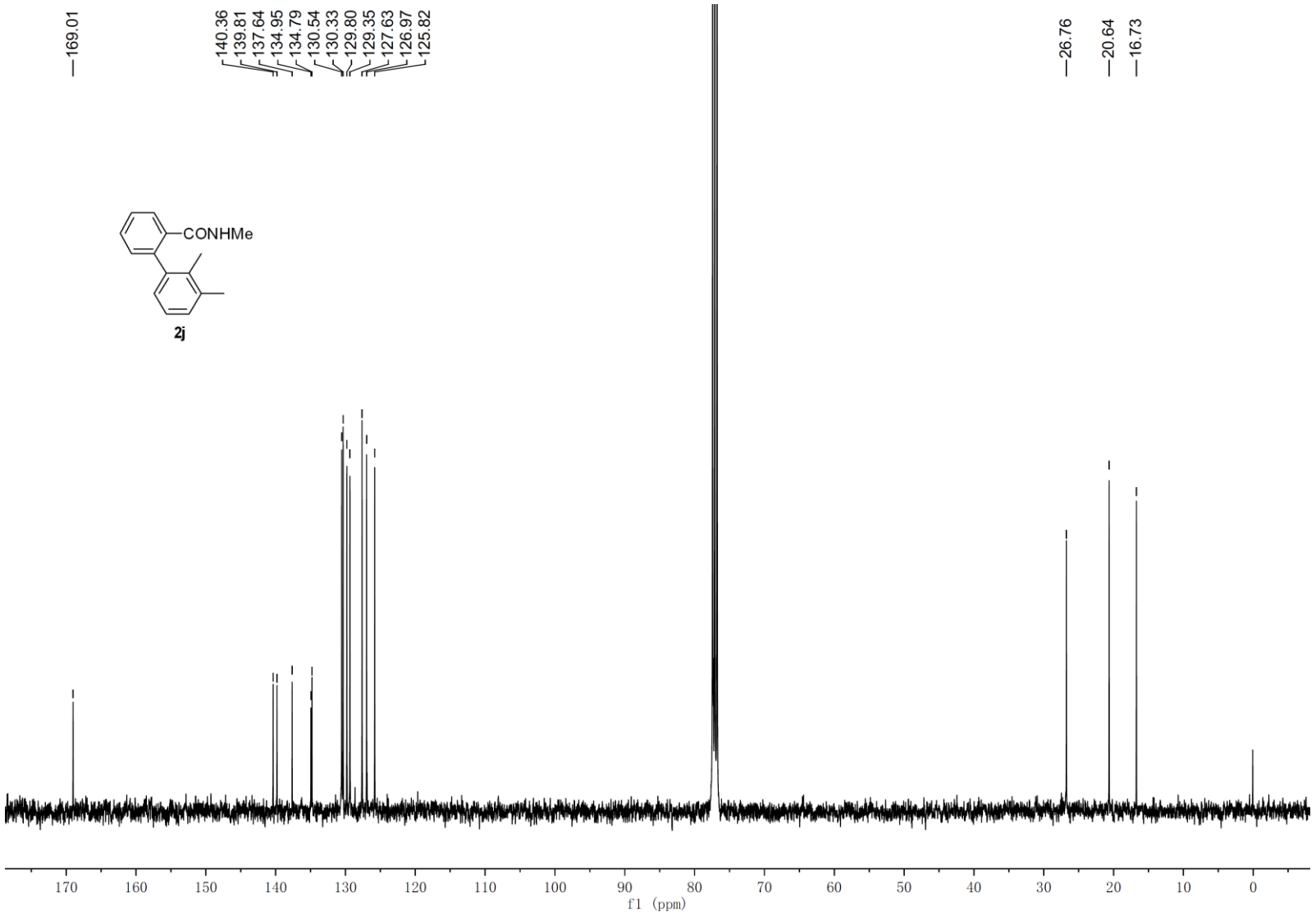
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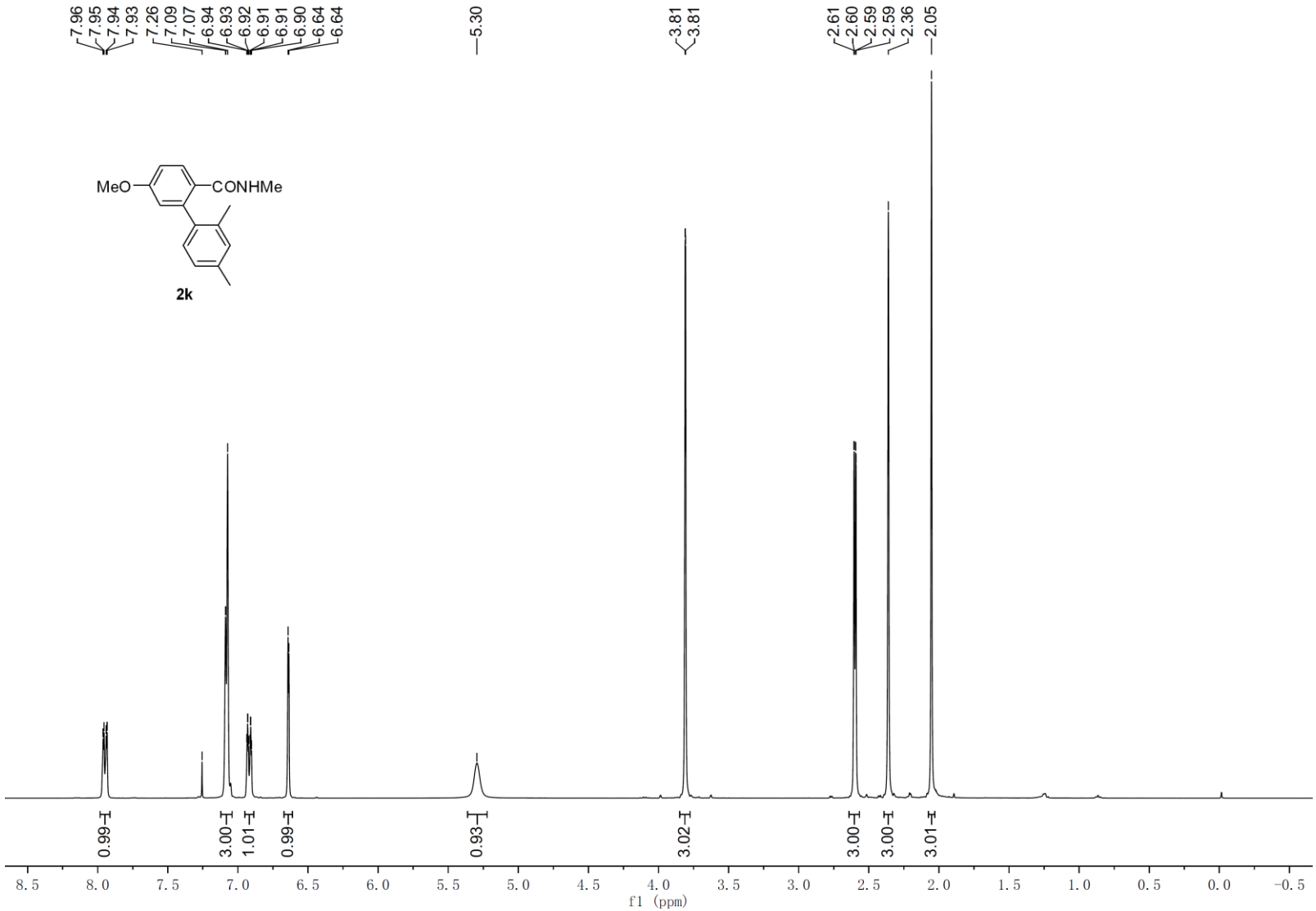
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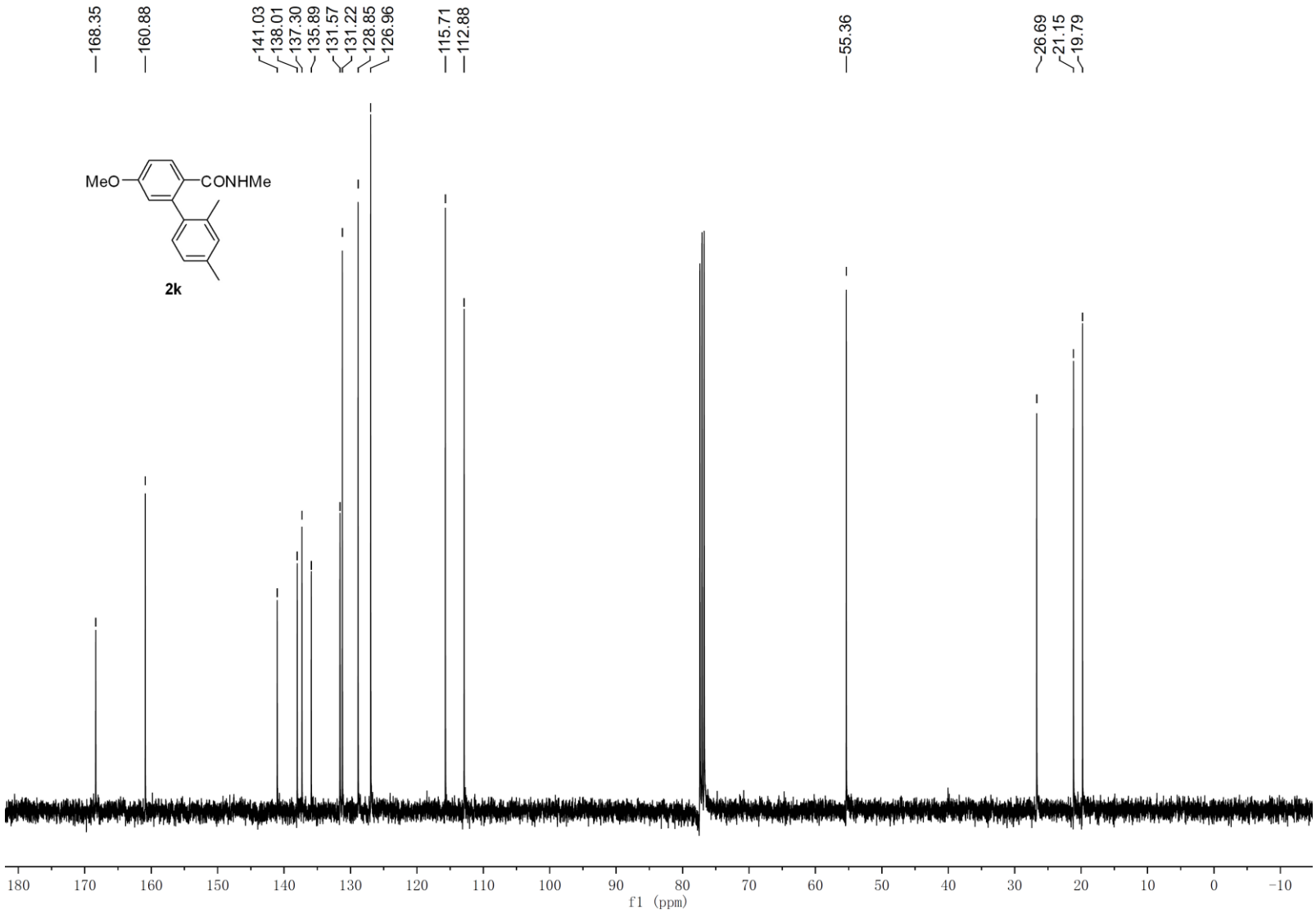
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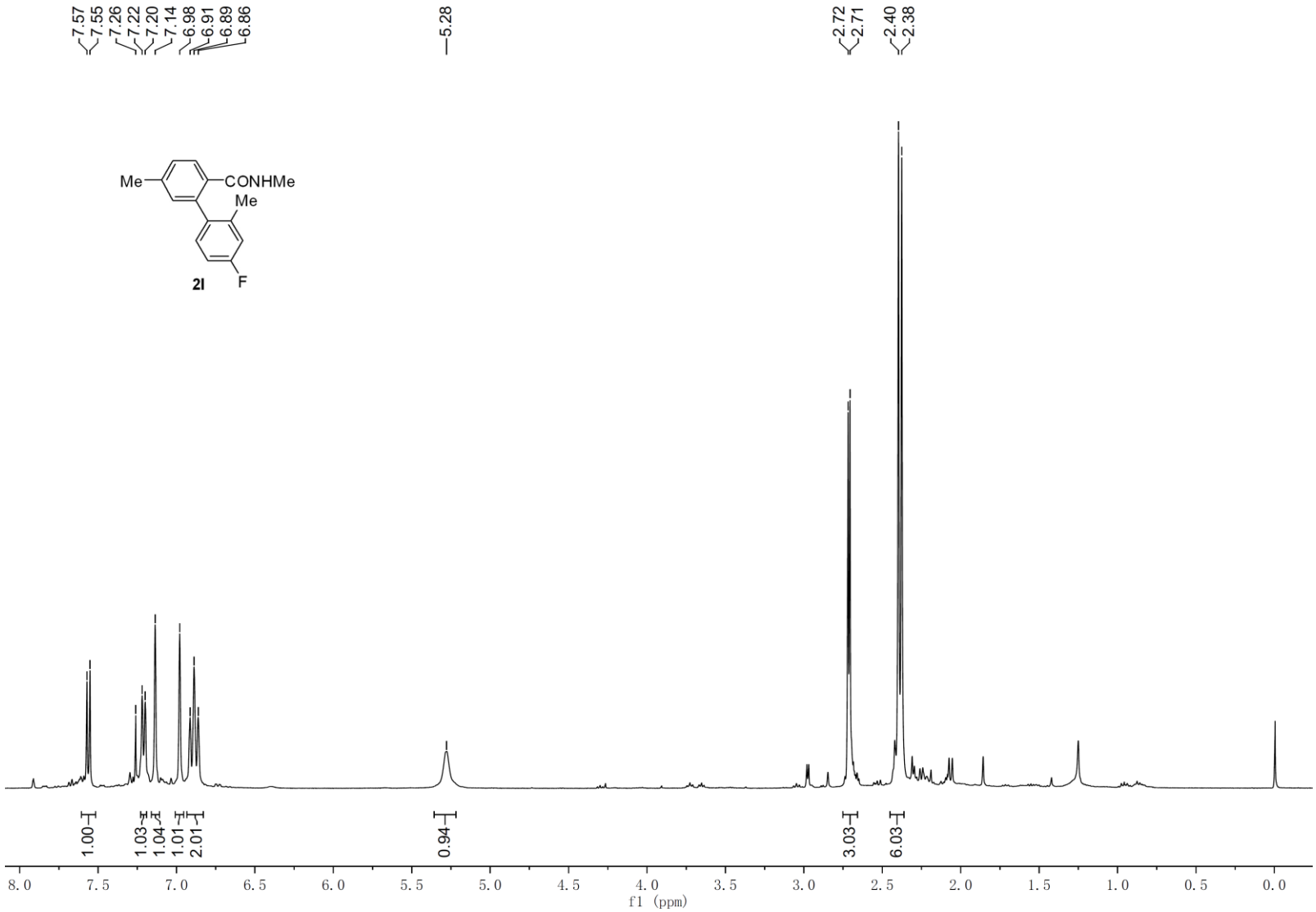
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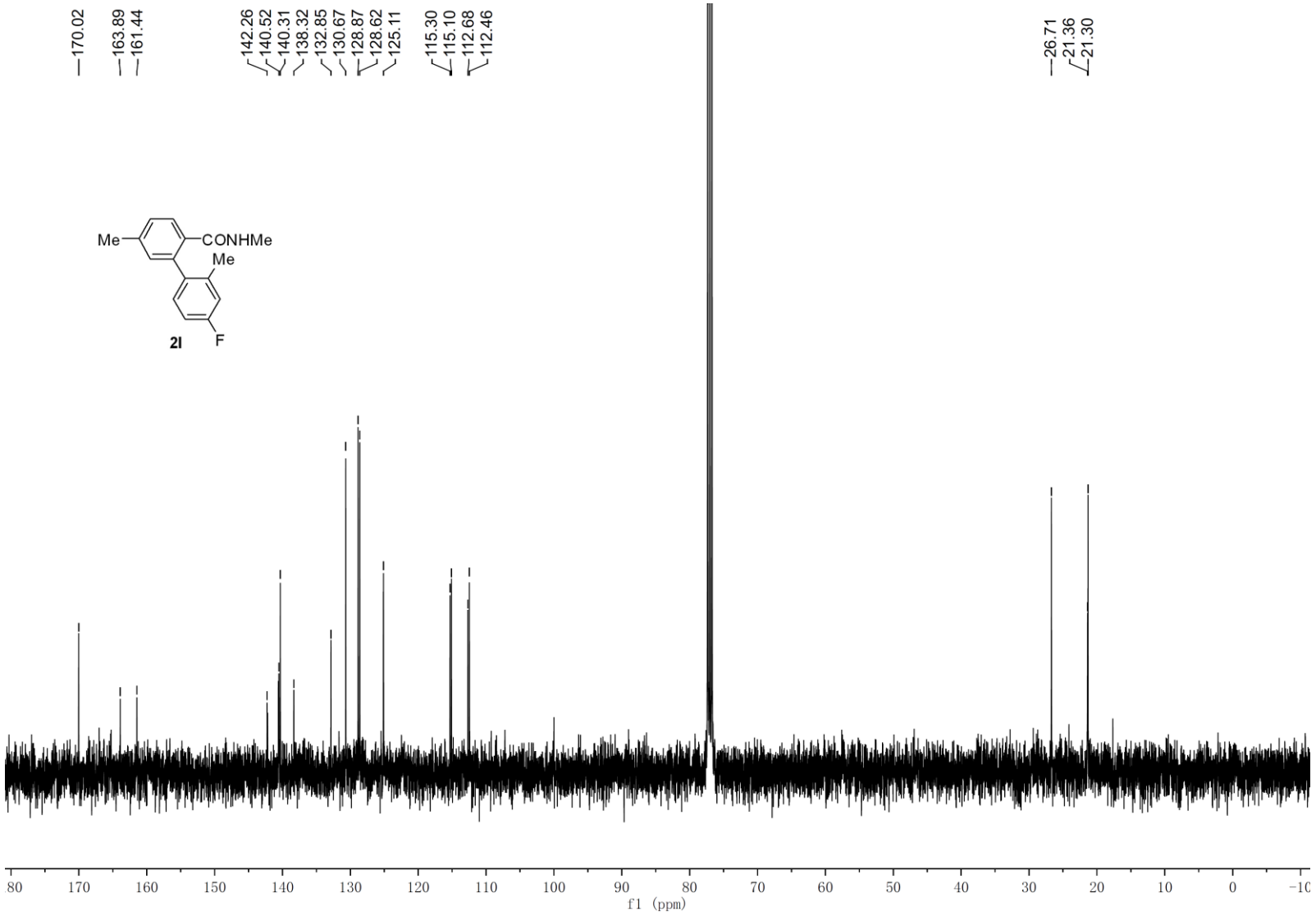
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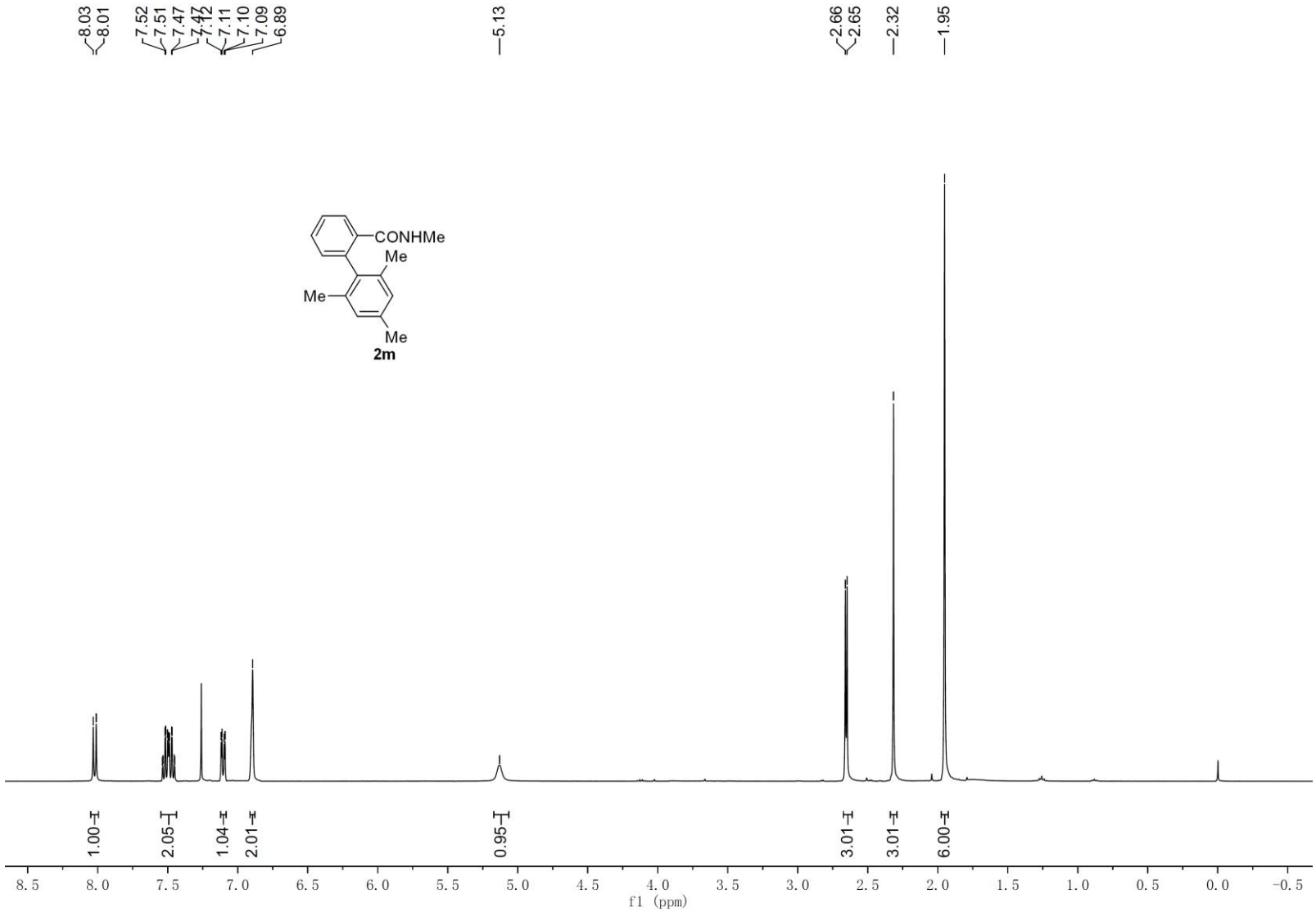
Me
F
2l
f1 (ppm)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CONHMe
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Me
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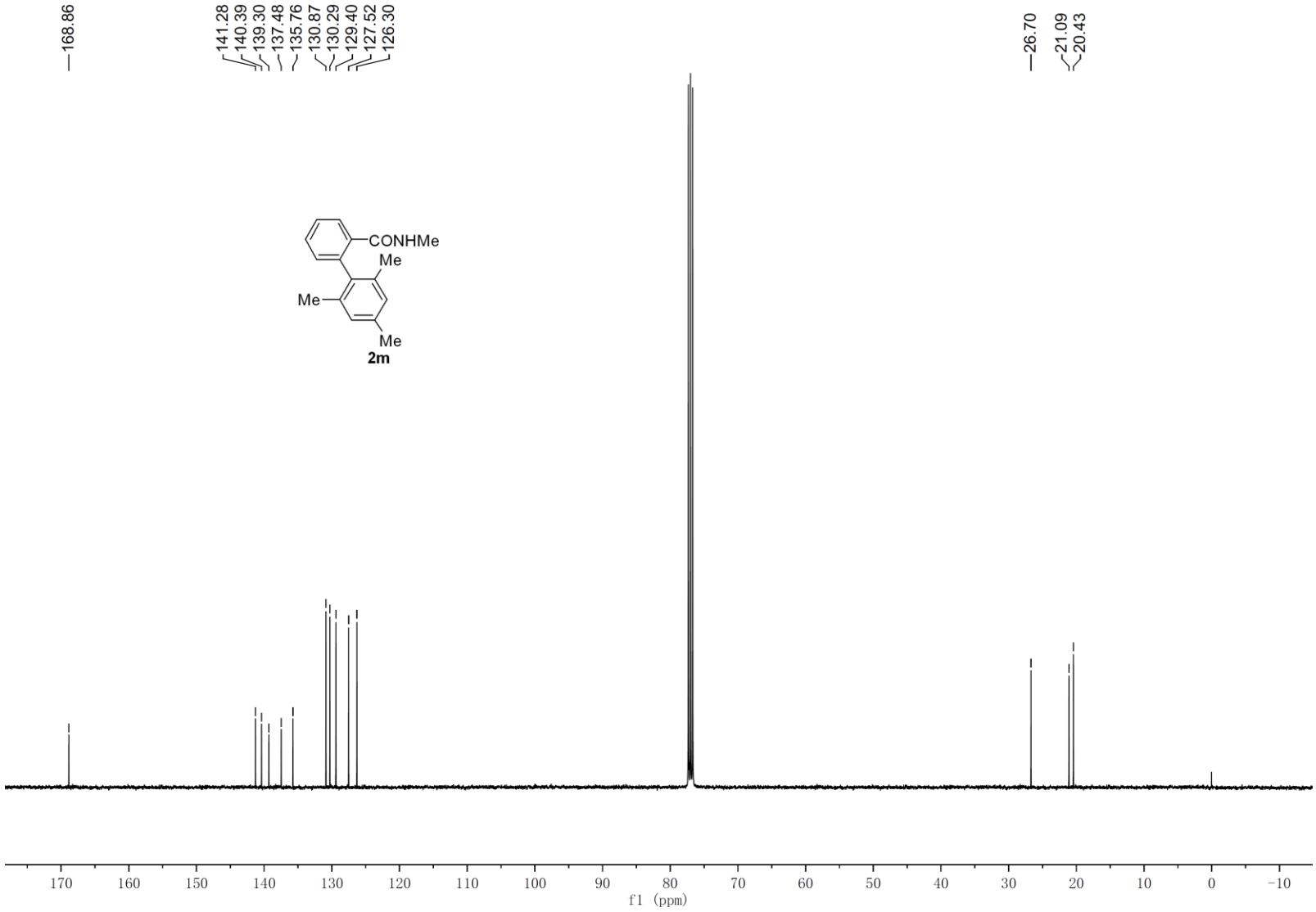
3.01
6.00
f1 (ppm)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20.43
CONHMe
Me
Me
Me
2m
170
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30
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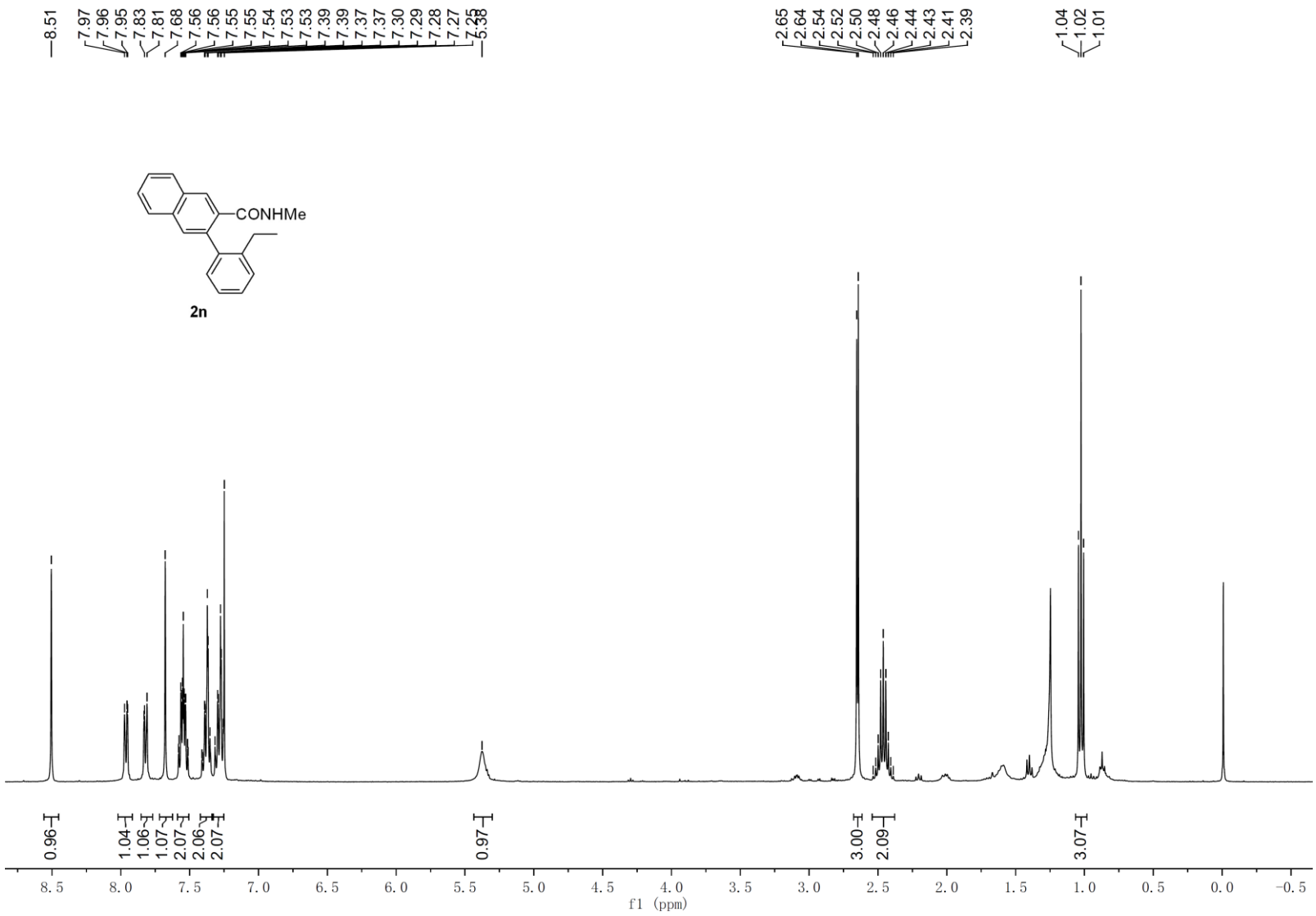
f1 (ppm)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7.28
7.27
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2.54
2.52
2.50
2.48
2.46
2.44
2.43
2.41
2.39
1.04
1.02
1.01
CONHMe
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1.04
1.06
1.07
2.07
2.06
2.07
0.97
3.00
2.09
3.07
8.5
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7.0
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6.0
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5.0
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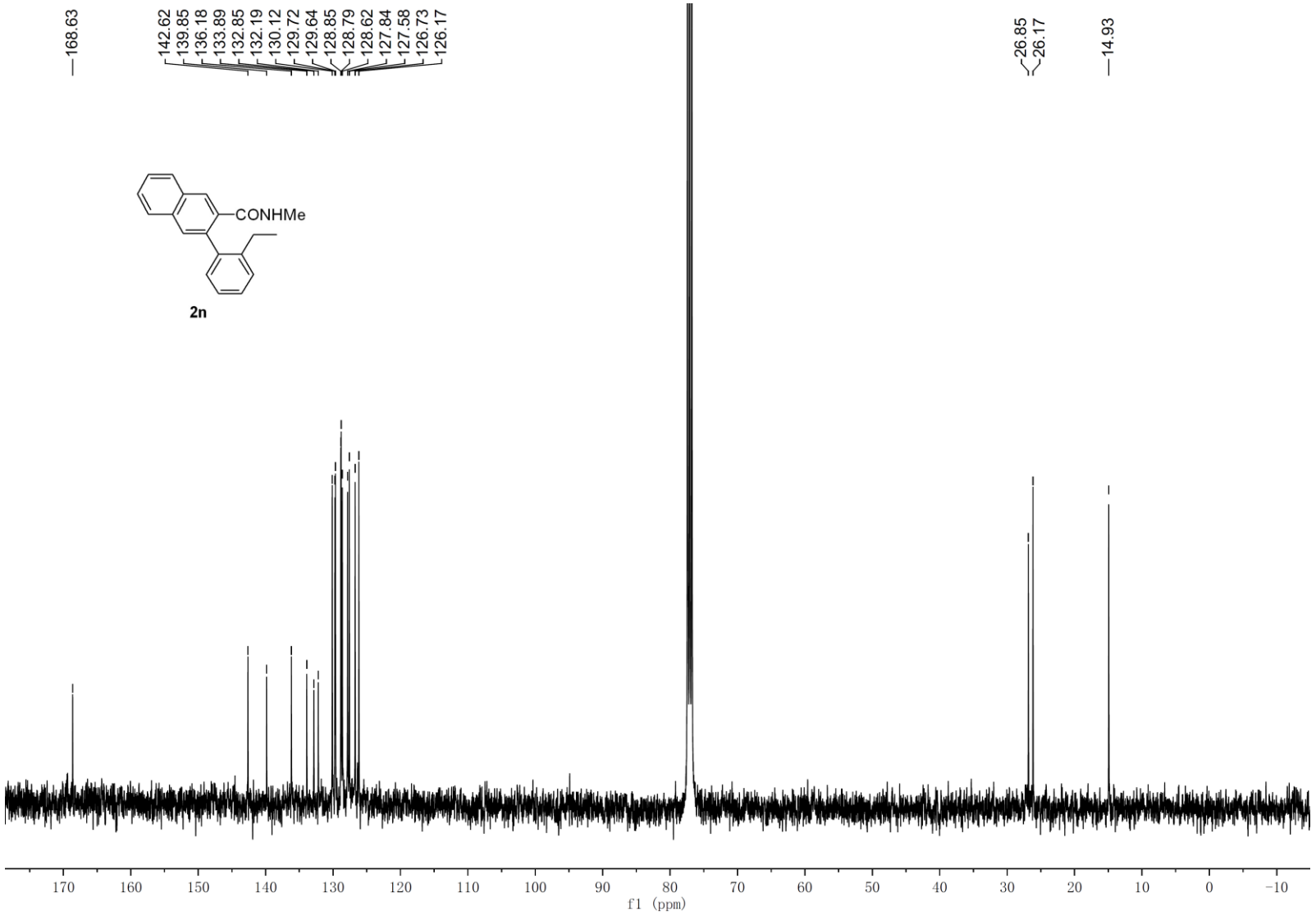
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127.58
126.73
126.17
26.85
26.17
14.93
CONHMe
2n
170
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80
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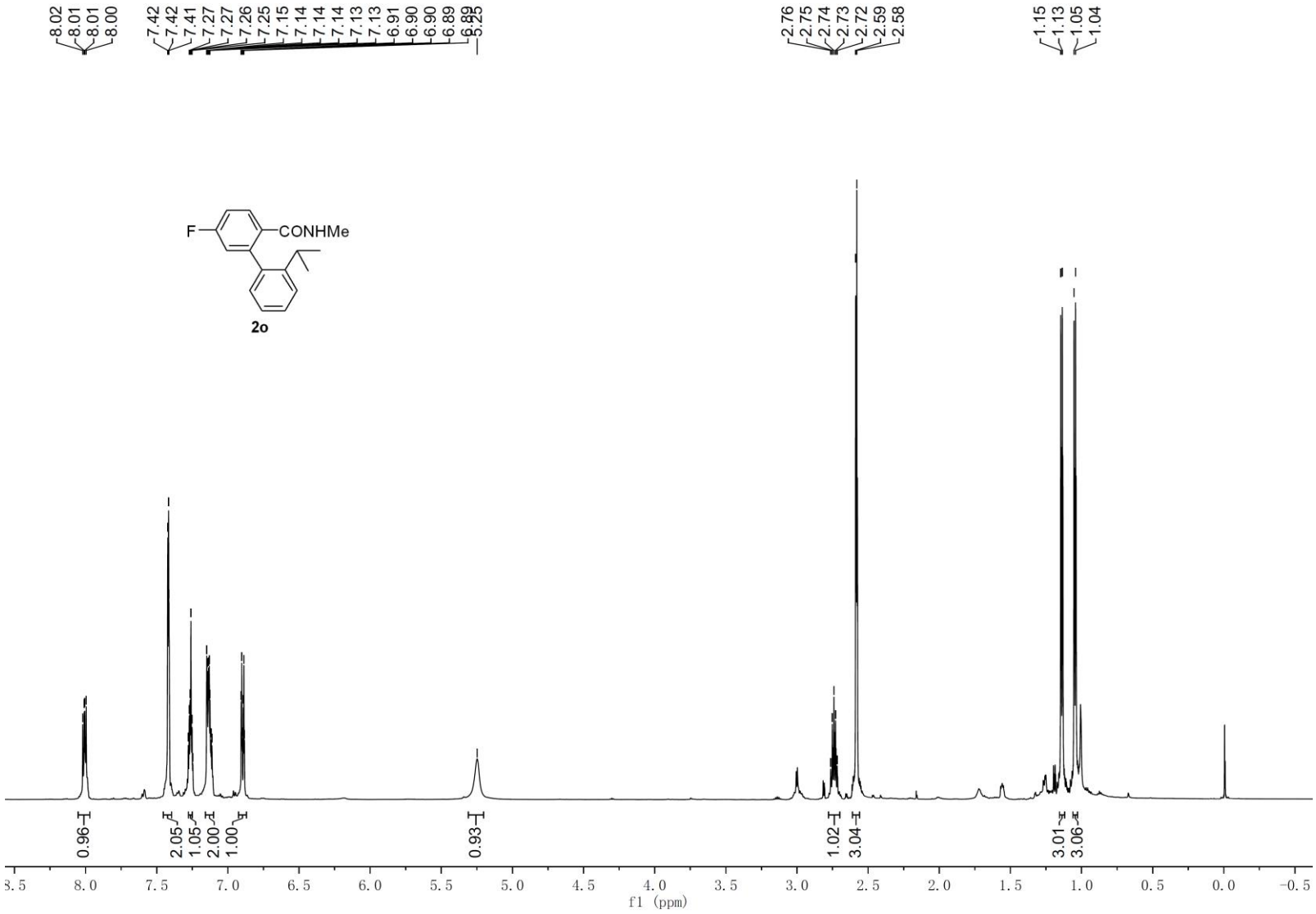
f1 (ppm)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7.14
7.14
7.14
7.13
7.13
6.91
6.90
6.90
6.89
3.25
2.76
2.75
2.74
2.73
2.72
2.59
2.58
1.15
1.13
1.05
1.04
F
CONHMe
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1.00
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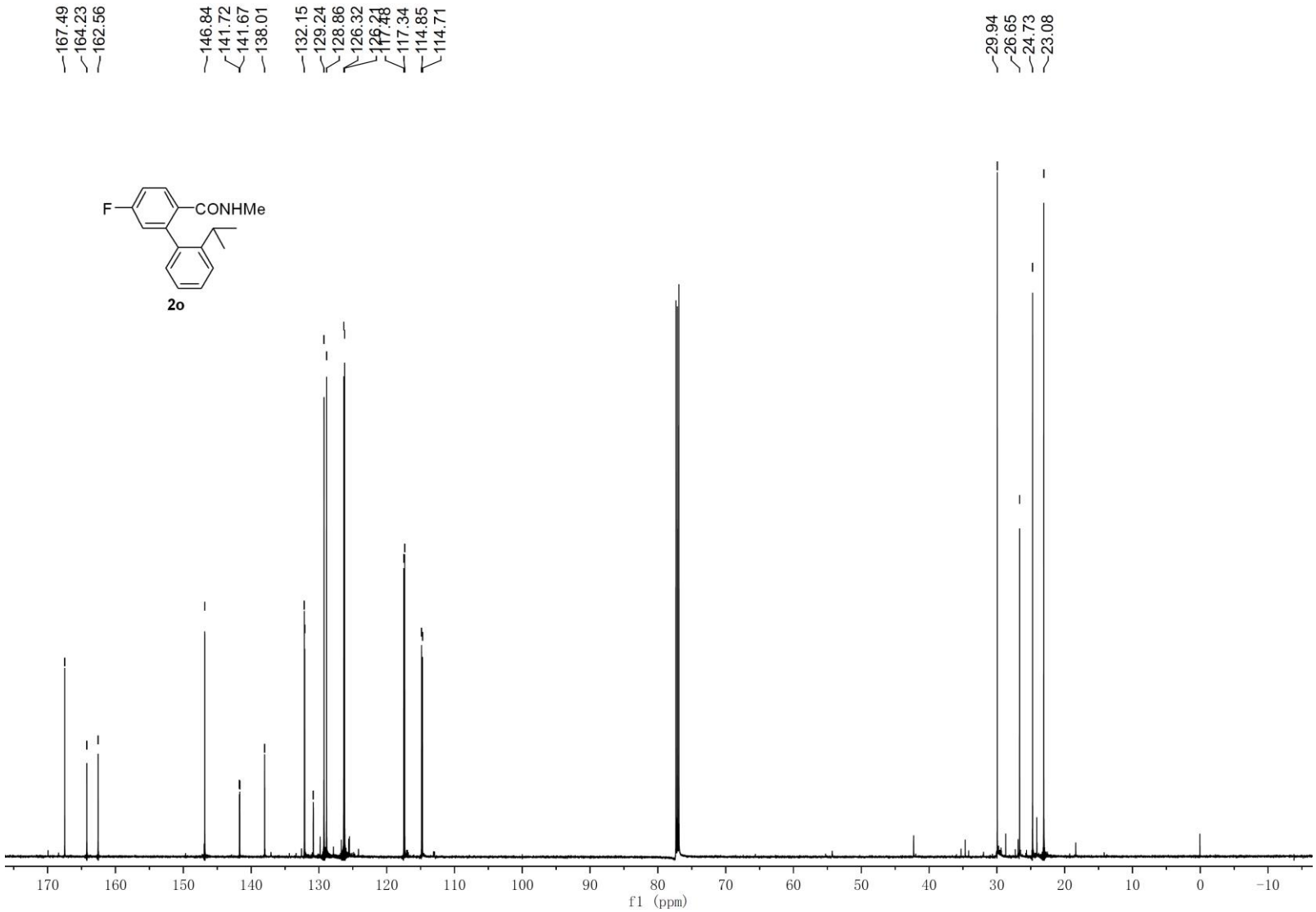
3.04
3.01
3.06
f1 (ppm)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114.85
114.71
29.94
26.65
24.73
23.08
F
CONHMe
2o
170
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100
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80
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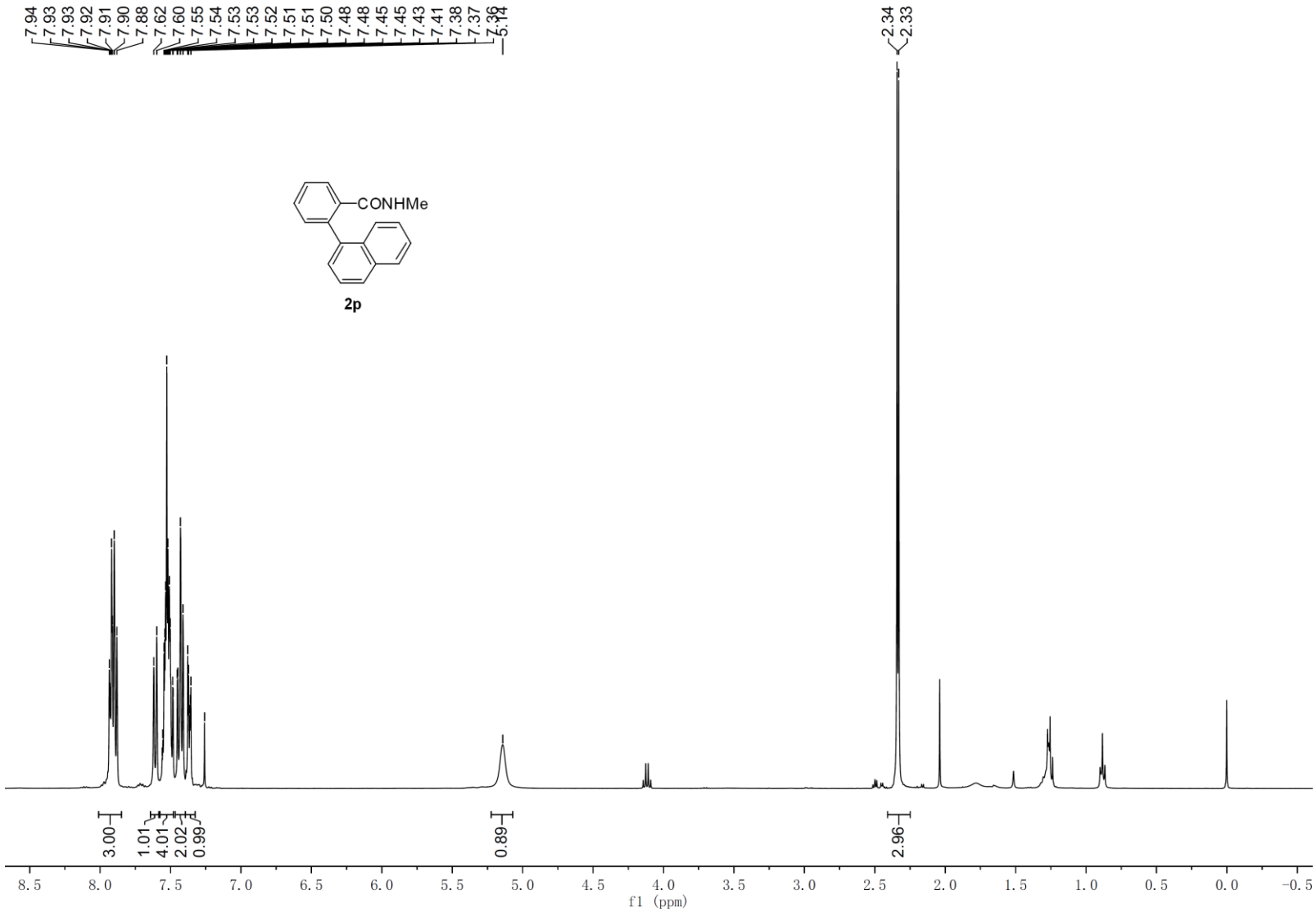
f1 (ppm)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7.51
7.51
7.50
7.48
7.48
7.45
7.45
7.43
7.41
7.38
7.37
7.36
5.14
2.34
2.33
CONHMe
2p
3.00
1.01
4.01
2.02
0.99
0.89
2.96
8.5
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7.0
6.5
6.0
5.5
5.0
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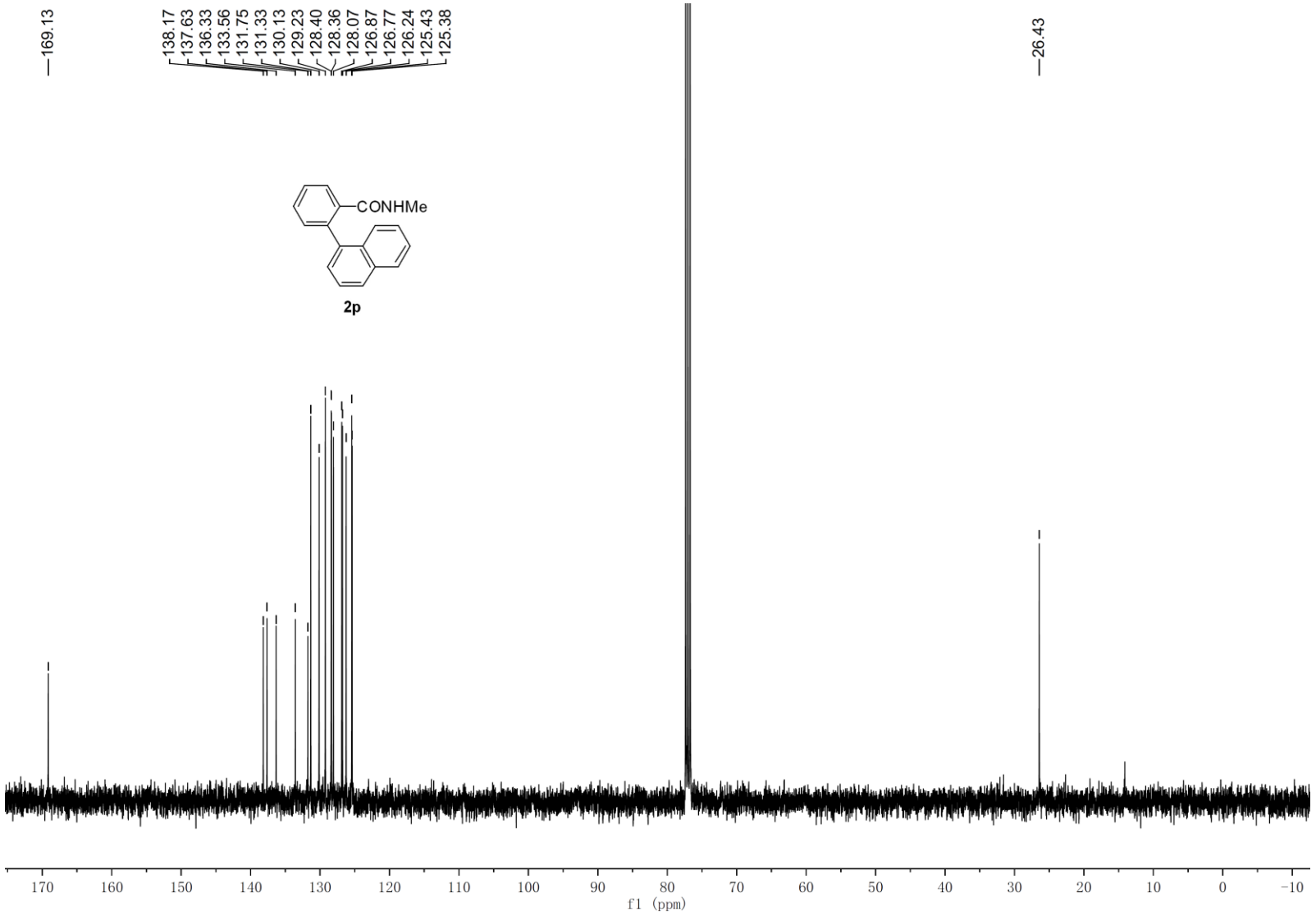
f1 (ppm)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126.77
126.24
125.43
125.38
26.43
CONHMe
2p
170
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140
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120
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100
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80
70
60
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40
30
20
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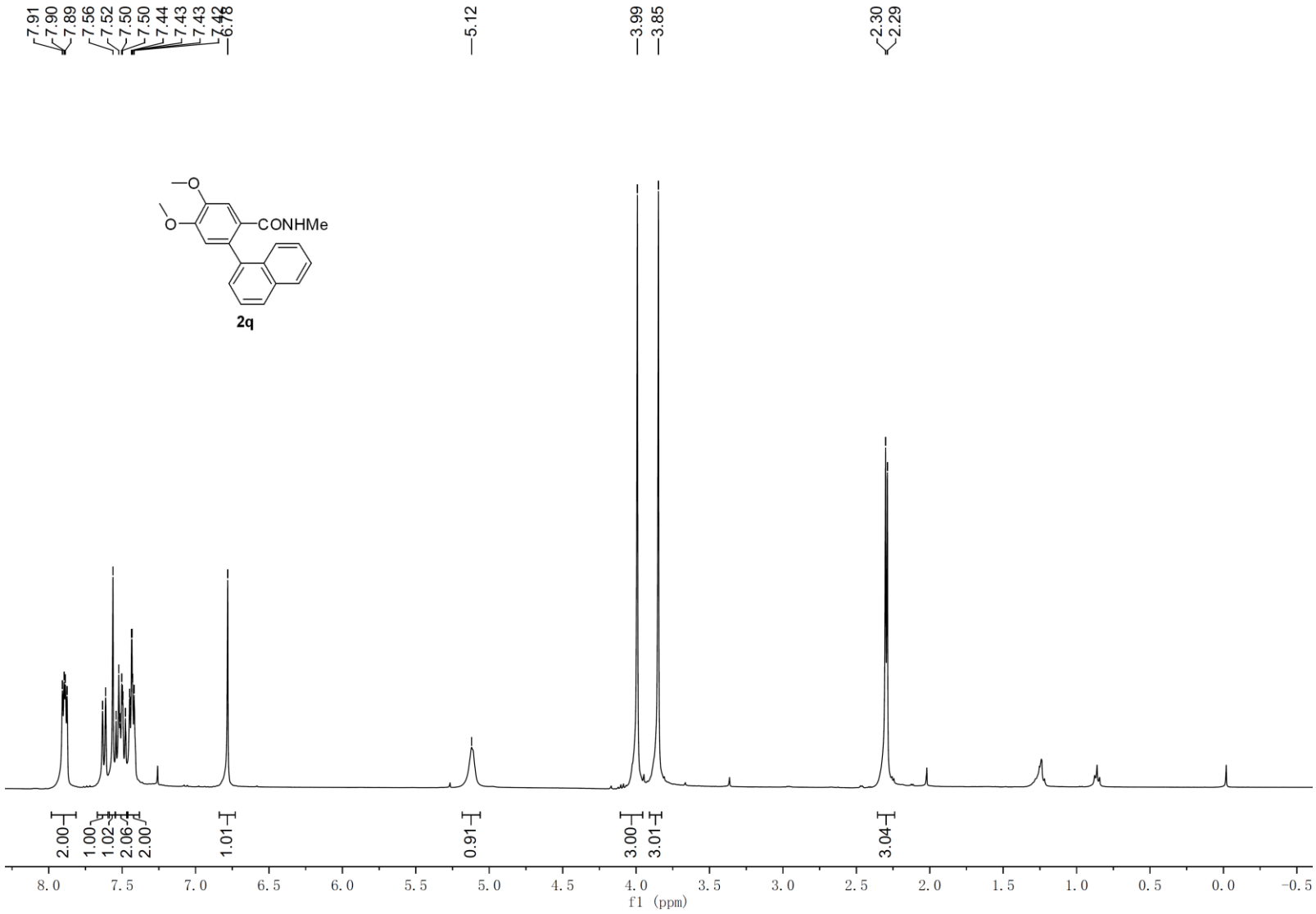
−10
f1 (ppm)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7.42
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5.12
3.99
3.85
2.30
2.29
CONHMe
2q
2.00
1.00
1.02
2.06
2.00
1.01
0.91
3.00
3.01
3.04
8.0
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6.0
5.5
5.0
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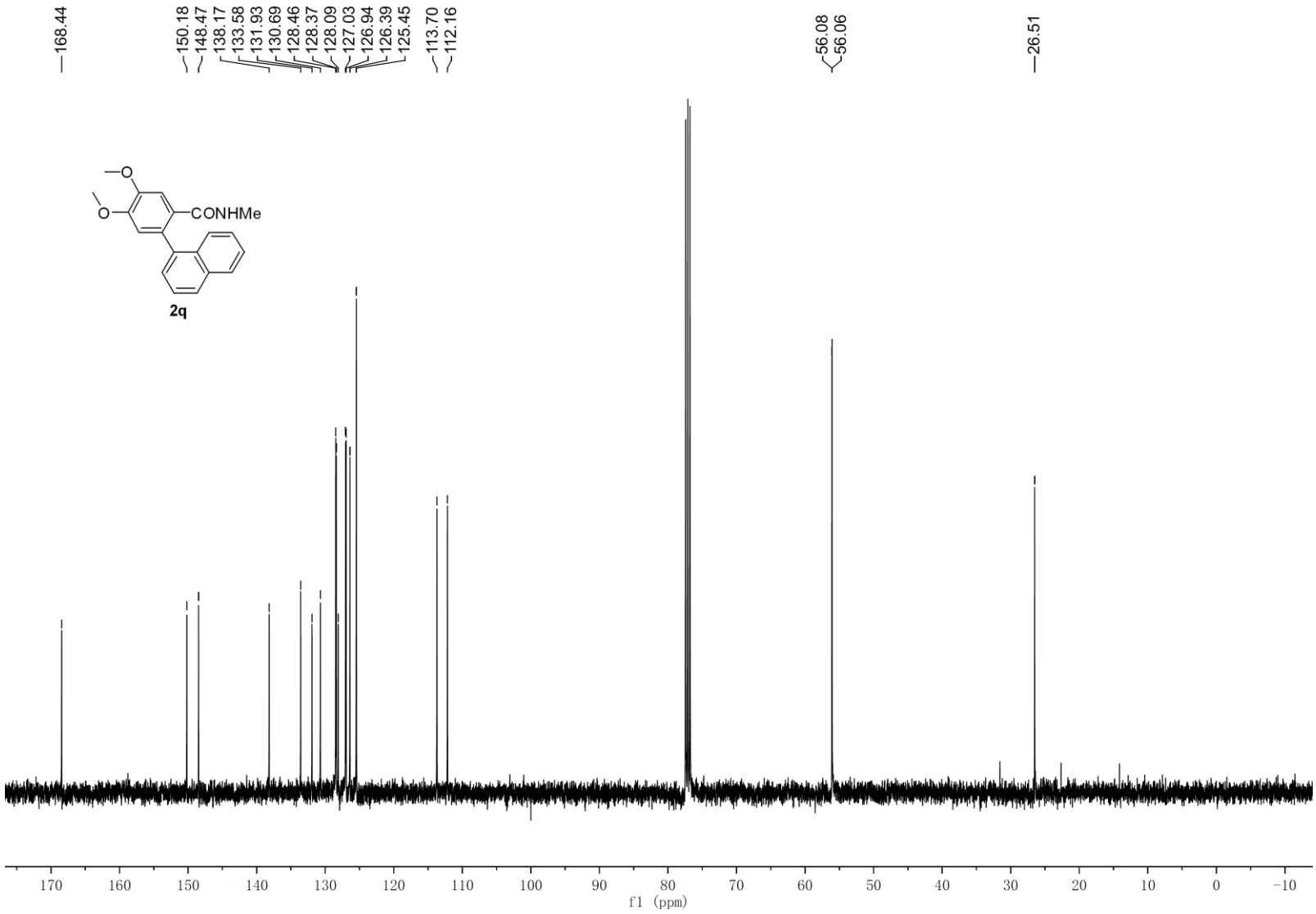
f1 (ppm)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113.70
112.16
56.08
56.06
26.51
O
O
CONHMe
2q
170
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100
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80
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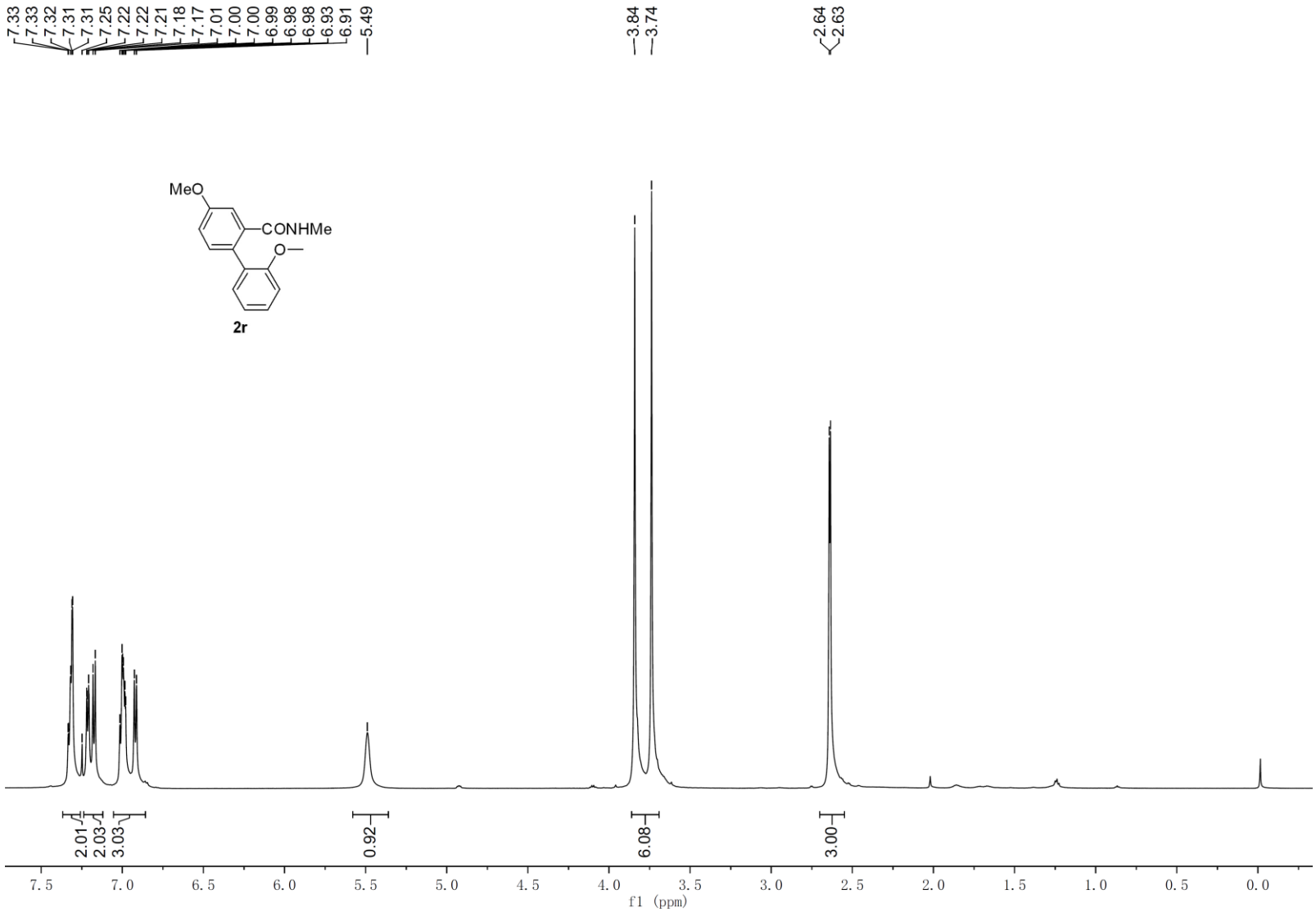
0
−10
f1 (ppm)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6.98
6.93
6.91
5.49
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3.74
2.64
2.63
MeO
CONHMe
O
2r
2.01
2.03
3.03
0.92
6.08
3.00
7.5
7.0
6.5
6.0
5.5
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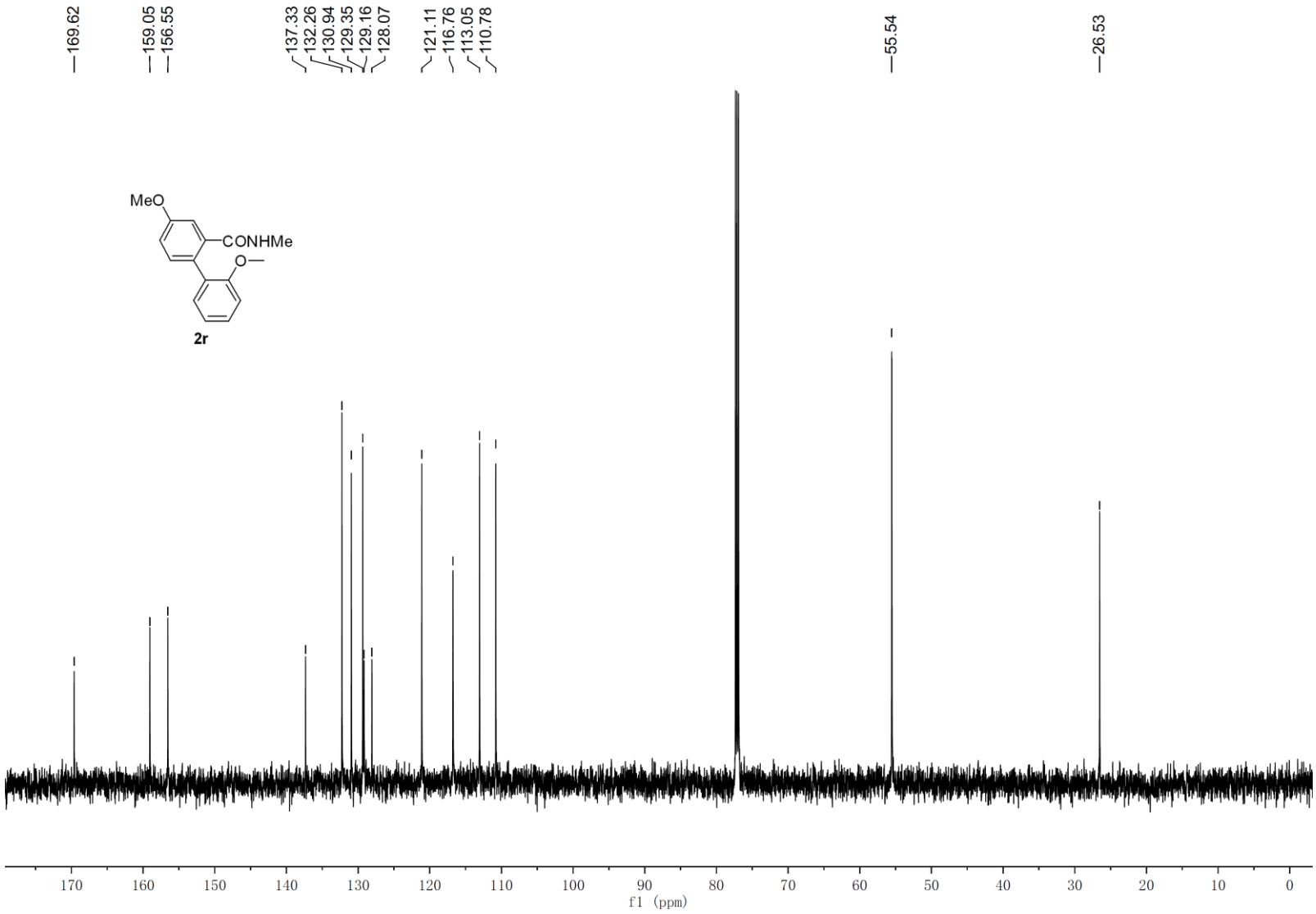
0.0
f1 (ppm)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113.05
110.78
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26.53
MeO
CONHMe
O
2r
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130
120
110
100
90
80
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50
40
30
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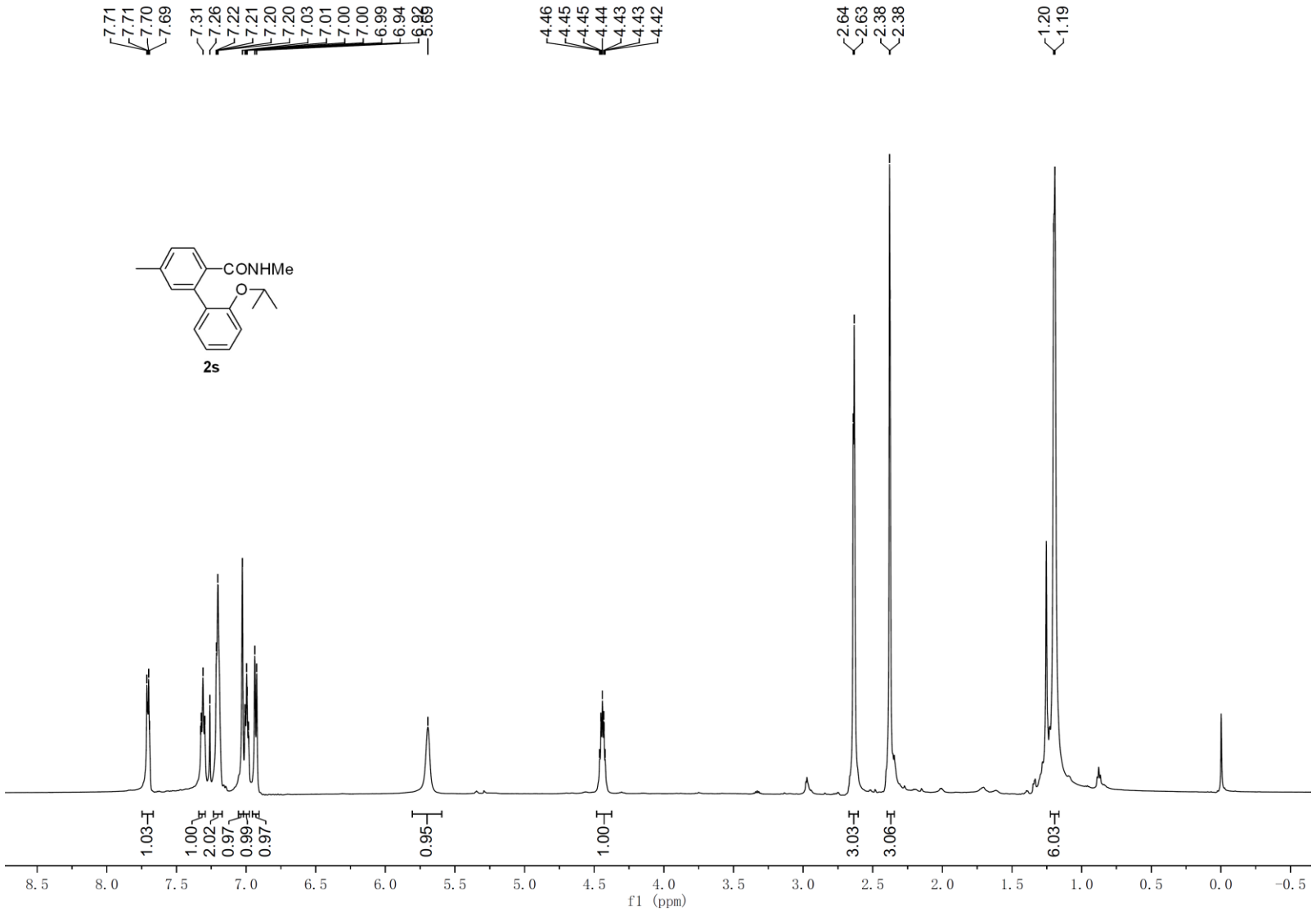
10
0
f1 (ppm)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7.00
6.99
6.94
6.92
6.89
4.46
4.45
4.45
4.44
4.43
4.43
4.42
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2.63
2.38
2.38
1.20
1.19
CONHMe
O
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0.99
0.97
0.95
1.00
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3.06
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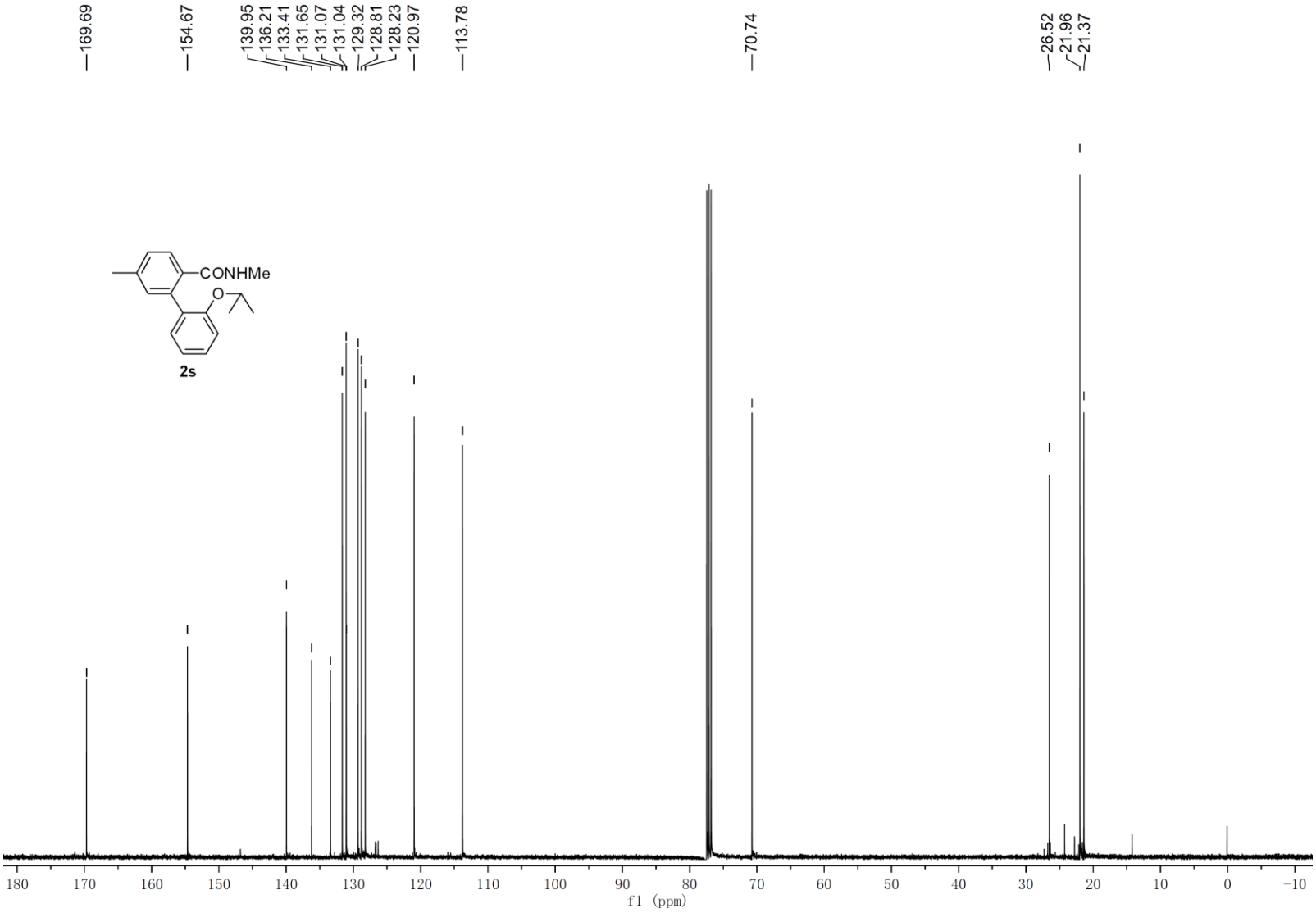
f1 (ppm)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113.78
70.74
26.52
21.96
21.37
CONHMe
O
2s
180
170
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100
90
80
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60
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40
30
20
10
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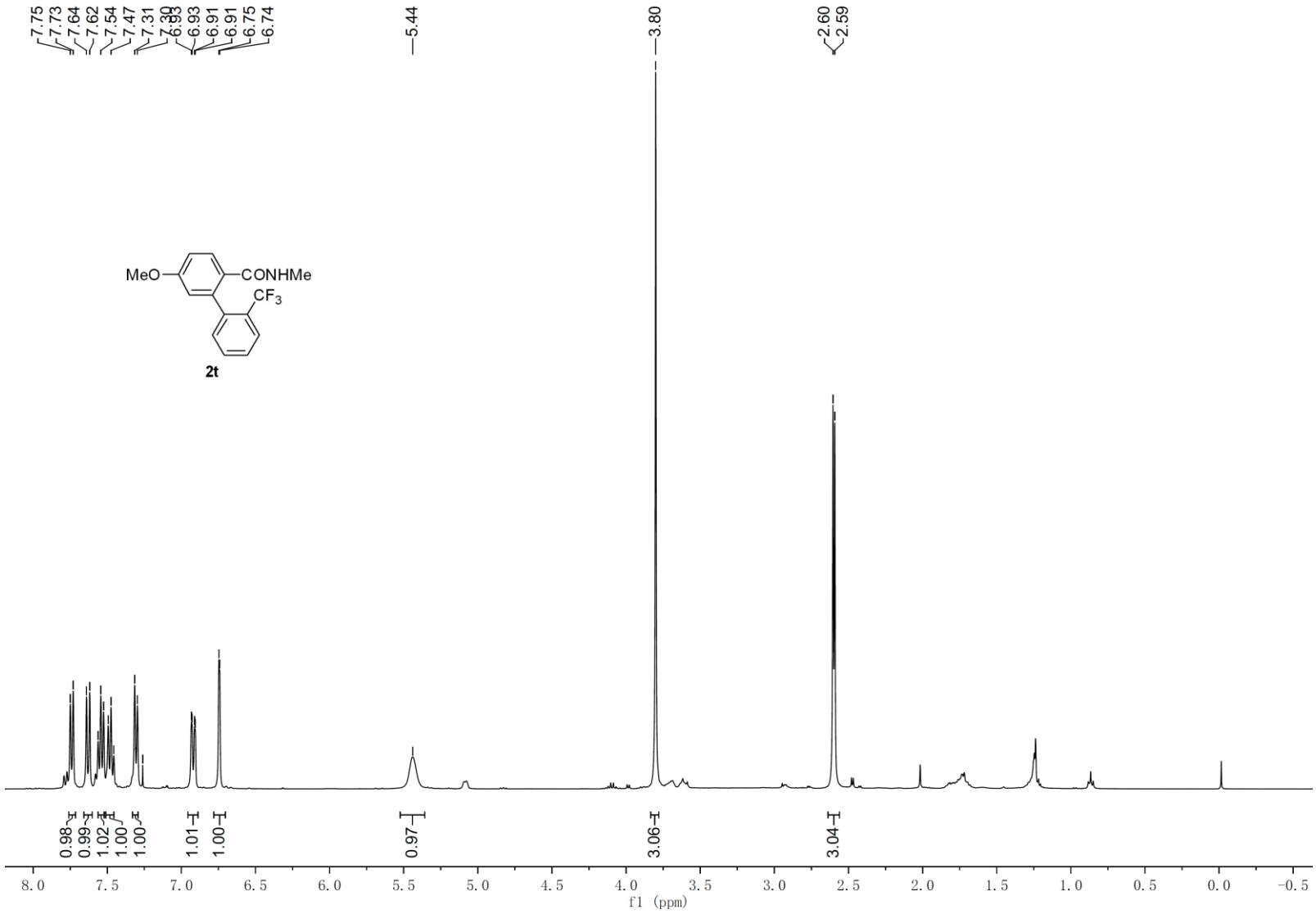
−10
f1 (ppm)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CONHMe
CF3
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1.00
1.00
1.01
1.00
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3.06
3.04
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7.5
7.0
6.5
6.0
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5.0
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2.0
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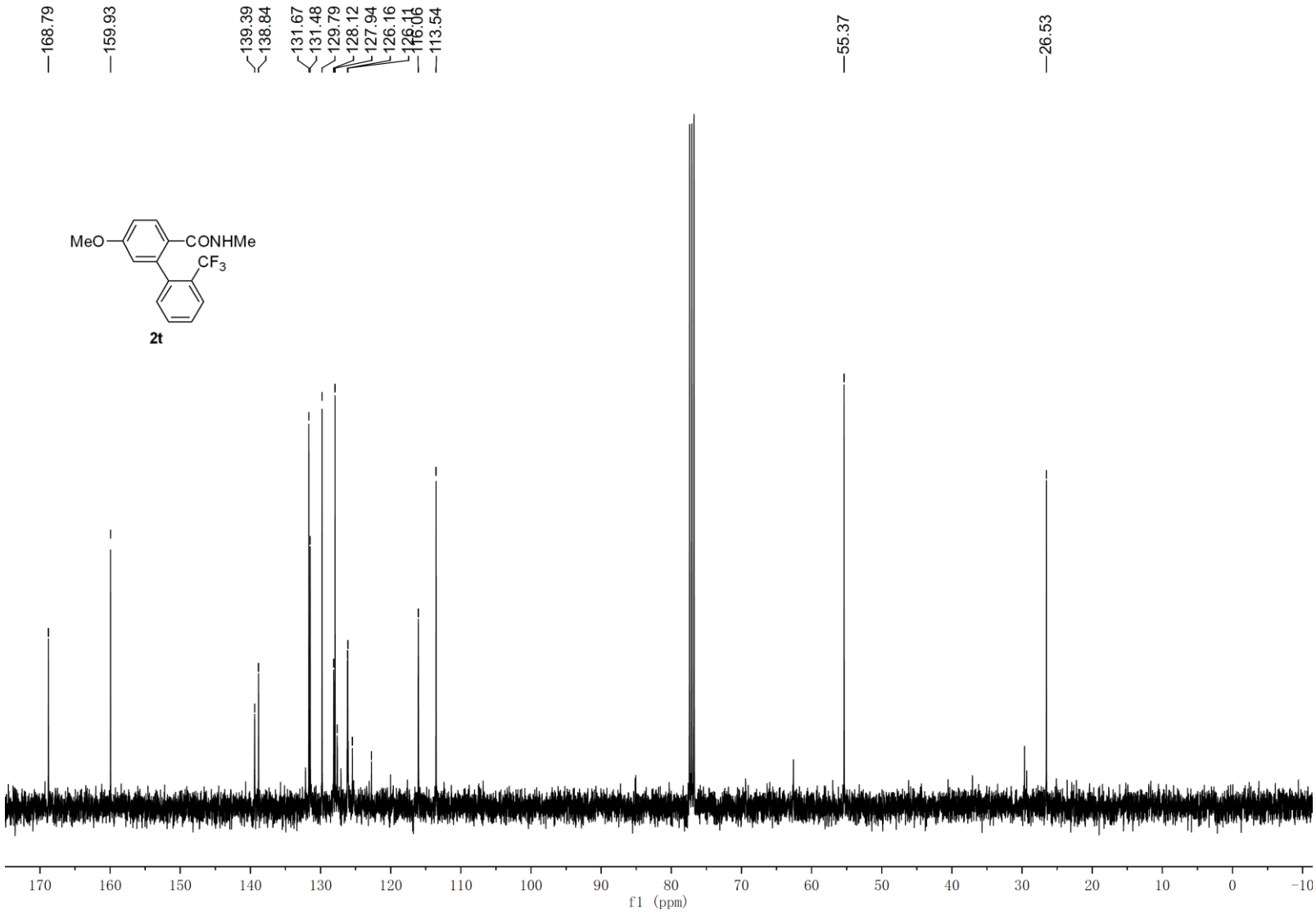
f1 (ppm)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55.37
26.53
MeO
CONHMe
CF3
2t
170
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120
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100
90
80
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60
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40
30
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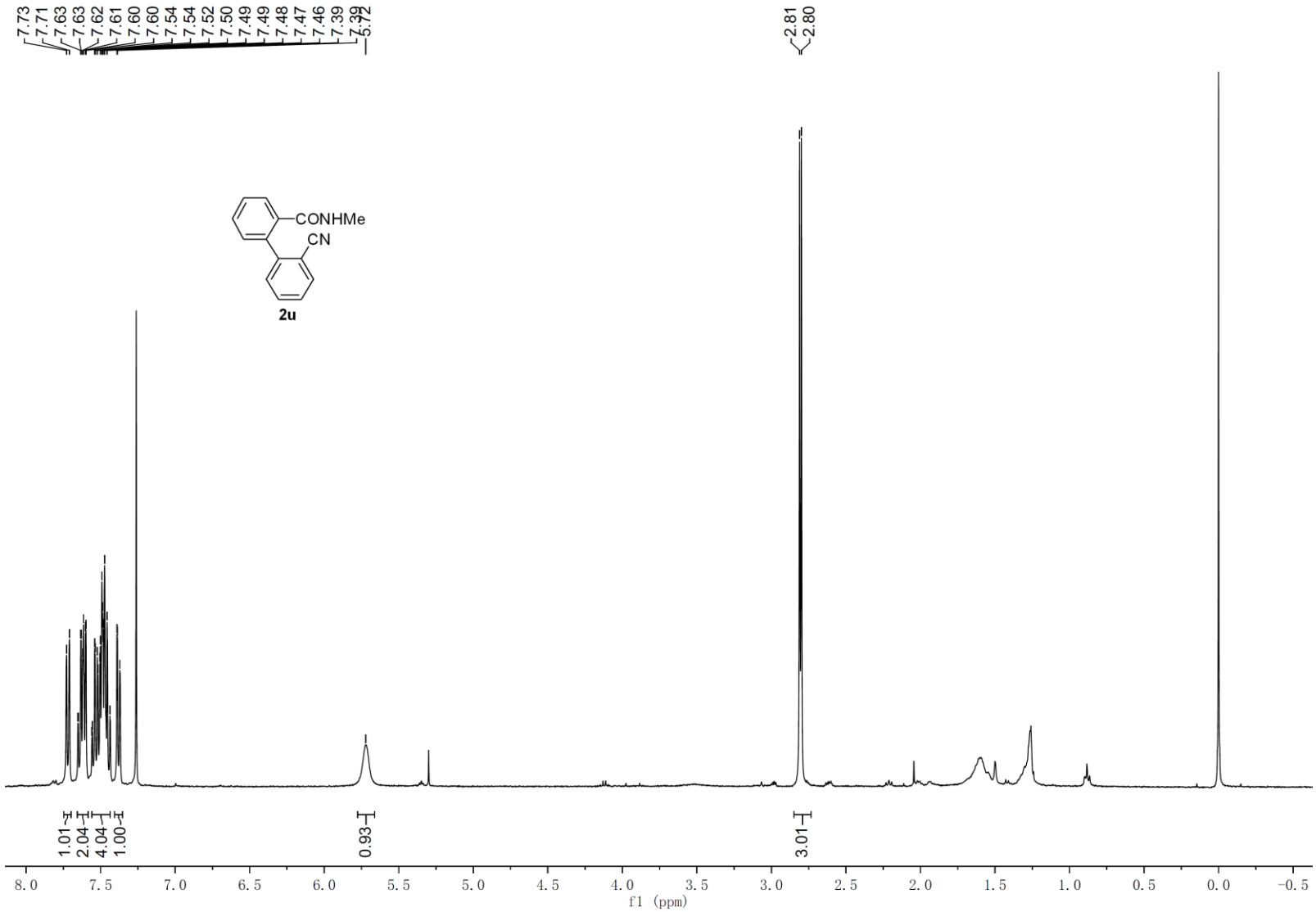
f1 (ppm)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7.46
7.39
7.39
7.32
2.81
2.80
CONHMe
CN
2u
1.01
2.04
4.04
1.00
0.93
3.01
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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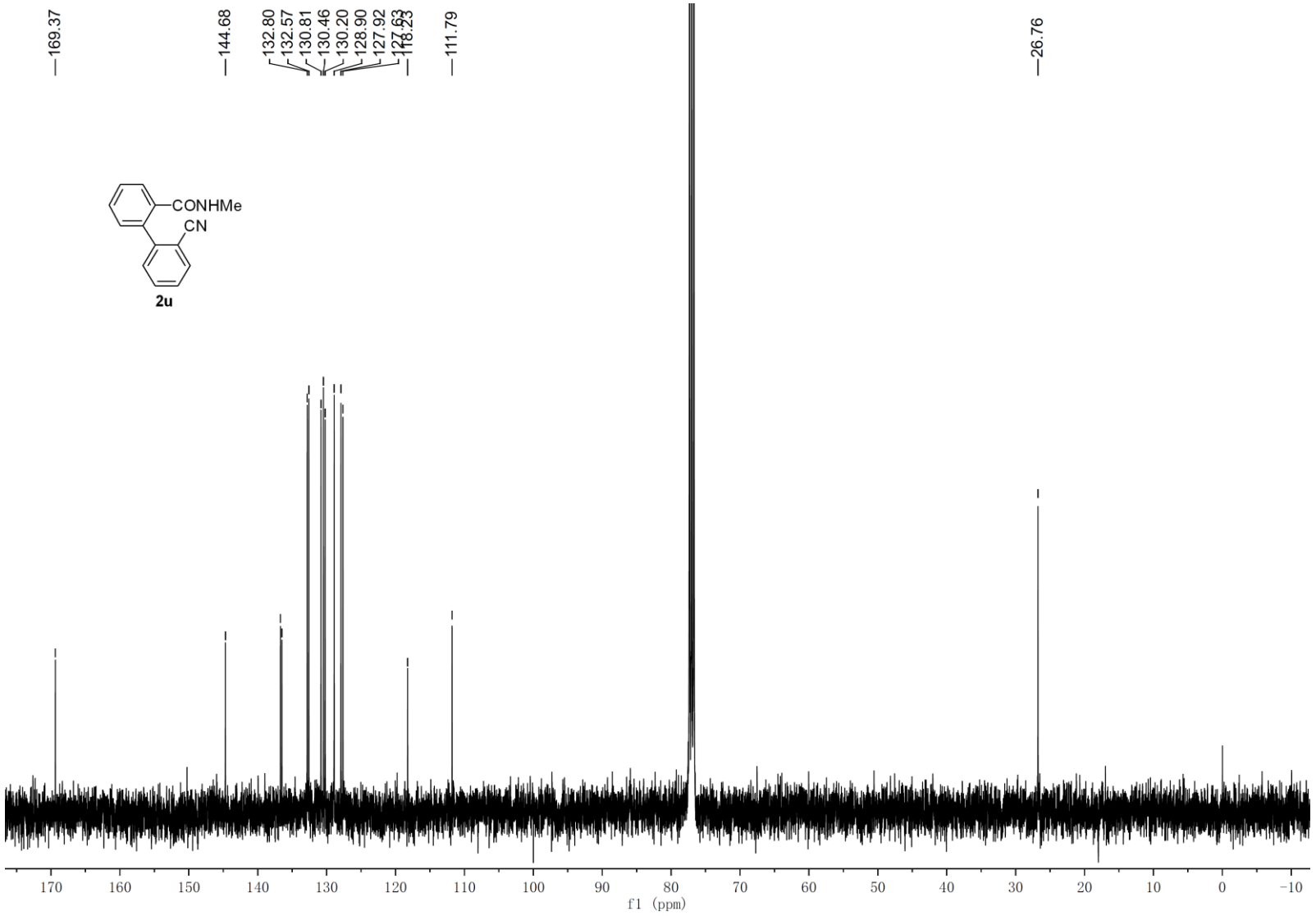
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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CN
2u
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100
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80
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40
30
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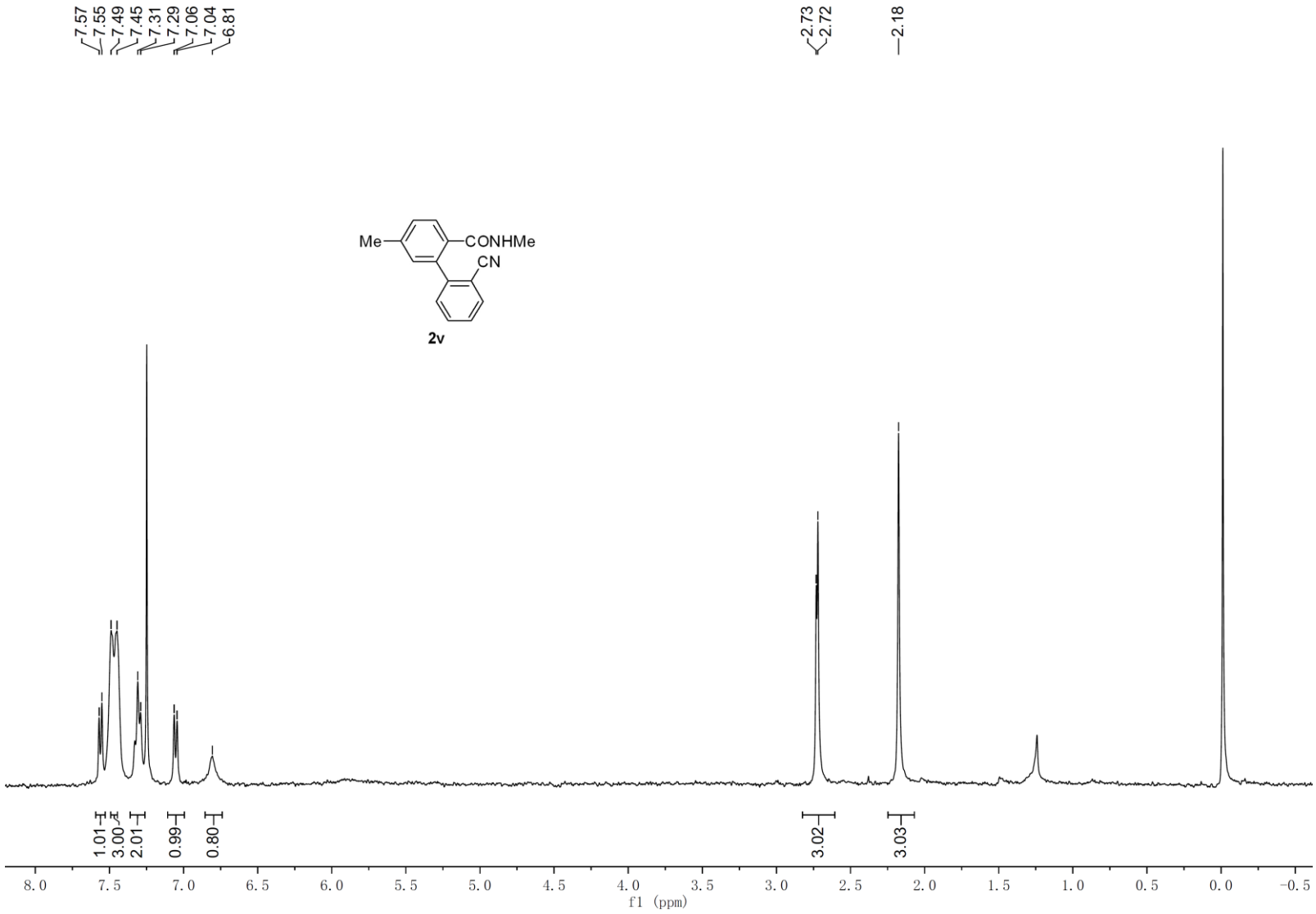
−10
f1 (ppm)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CN
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2.01
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0.80
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3.03
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5.0
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2.0
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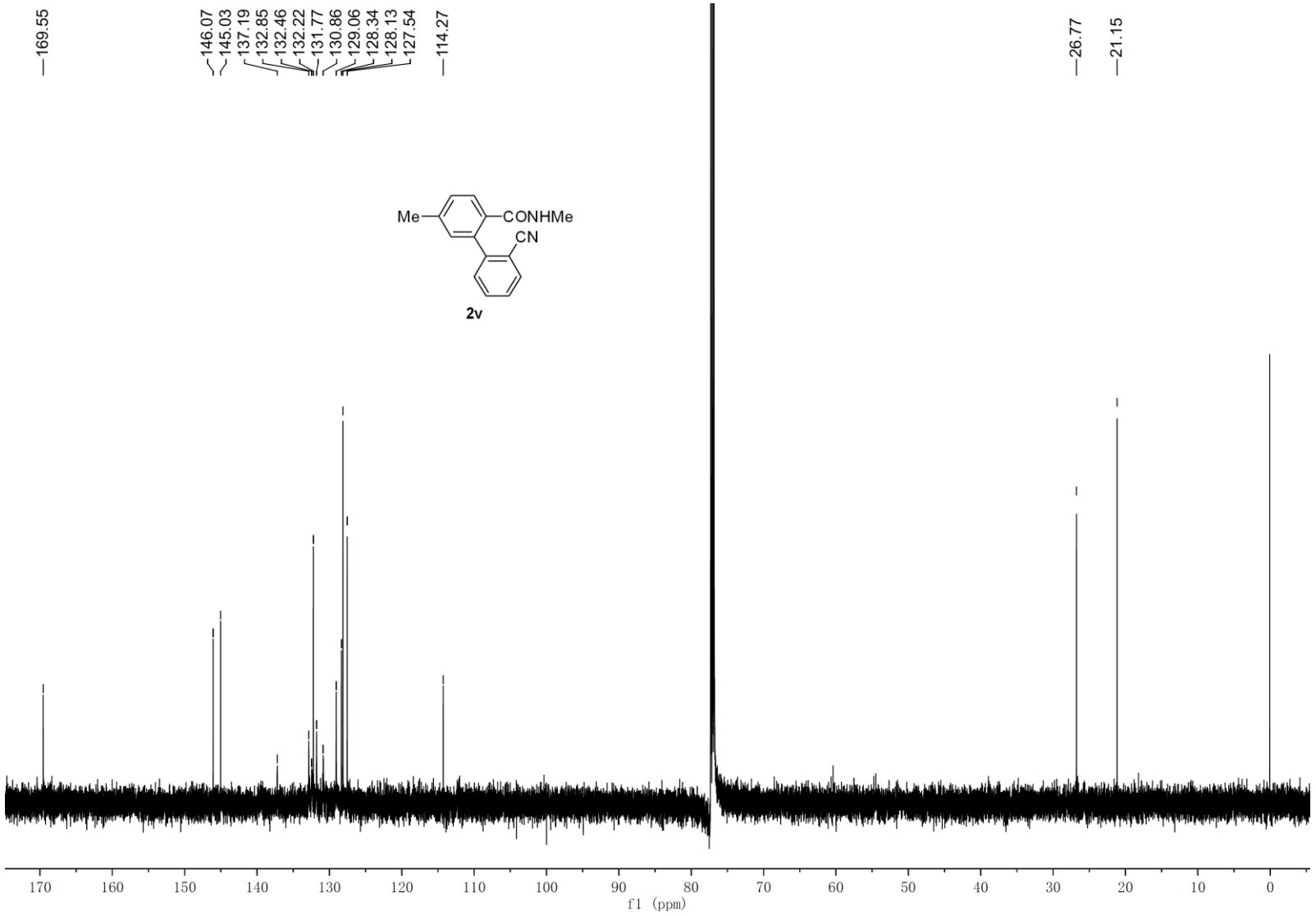
f1 (ppm)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114.27
26.77
21.15
Me
CONHMe
CN
2v
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110
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90
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50
40
30
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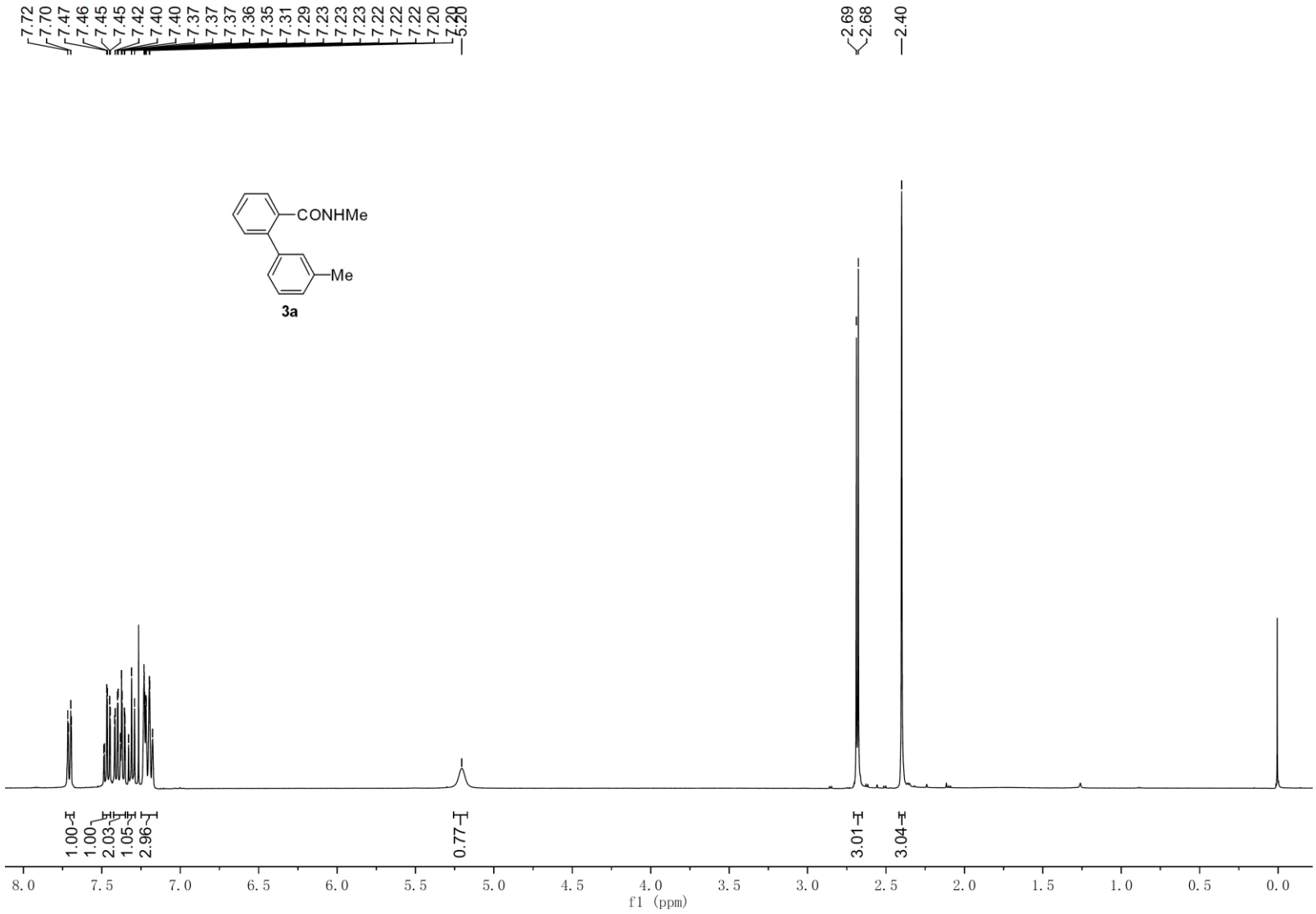
10
0
f1 (ppm)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7.23
7.23
7.22
7.22
7.22
7.20
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2.68
2.40
CONHMe
Me
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1.00
2.03
1.05
2.96
0.77
3.01
3.04
8.0
7.5
7.0
6.5
6.0
5.5
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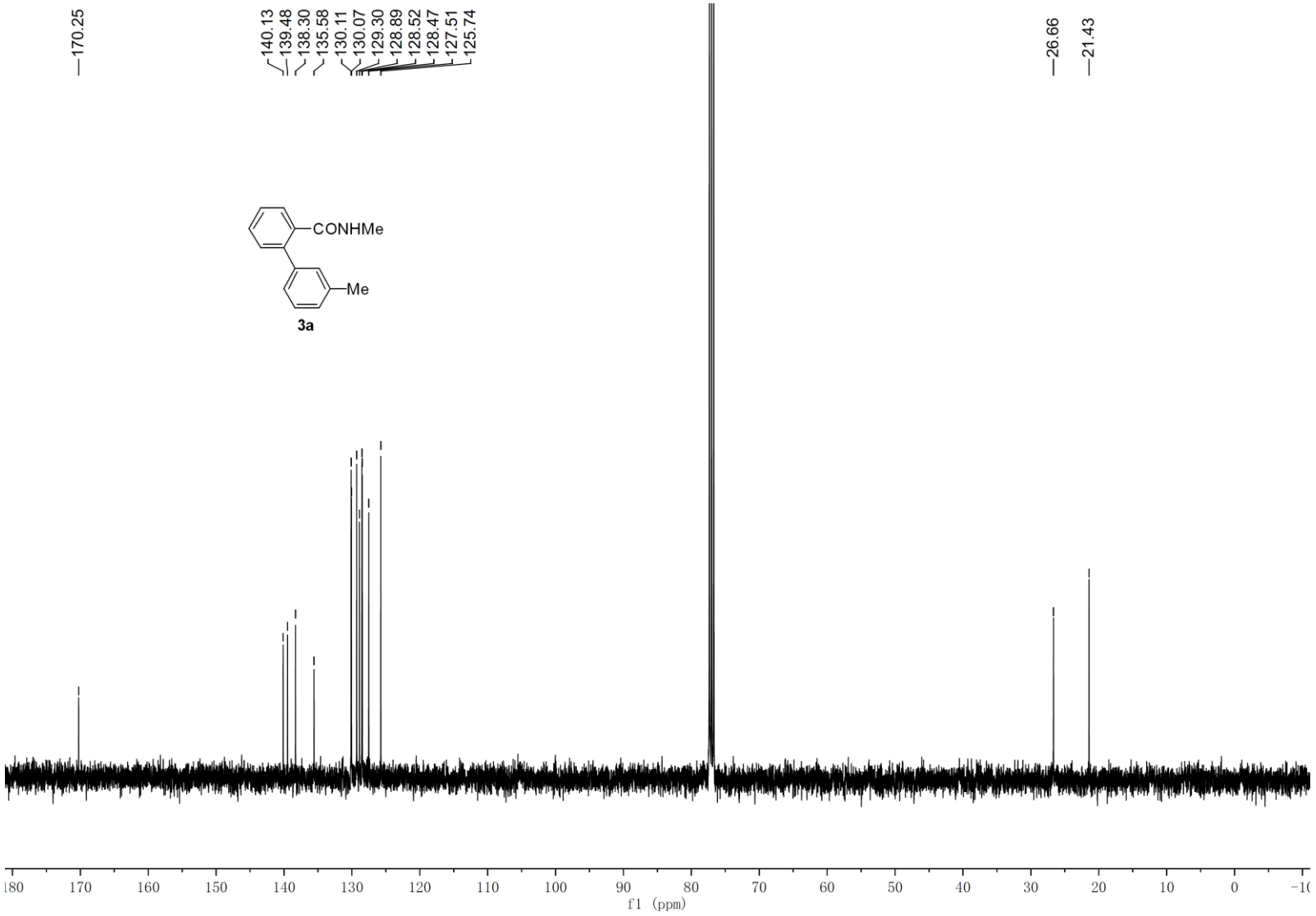
f1 (ppm)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26.66
21.43
CONHMe
Me
3a
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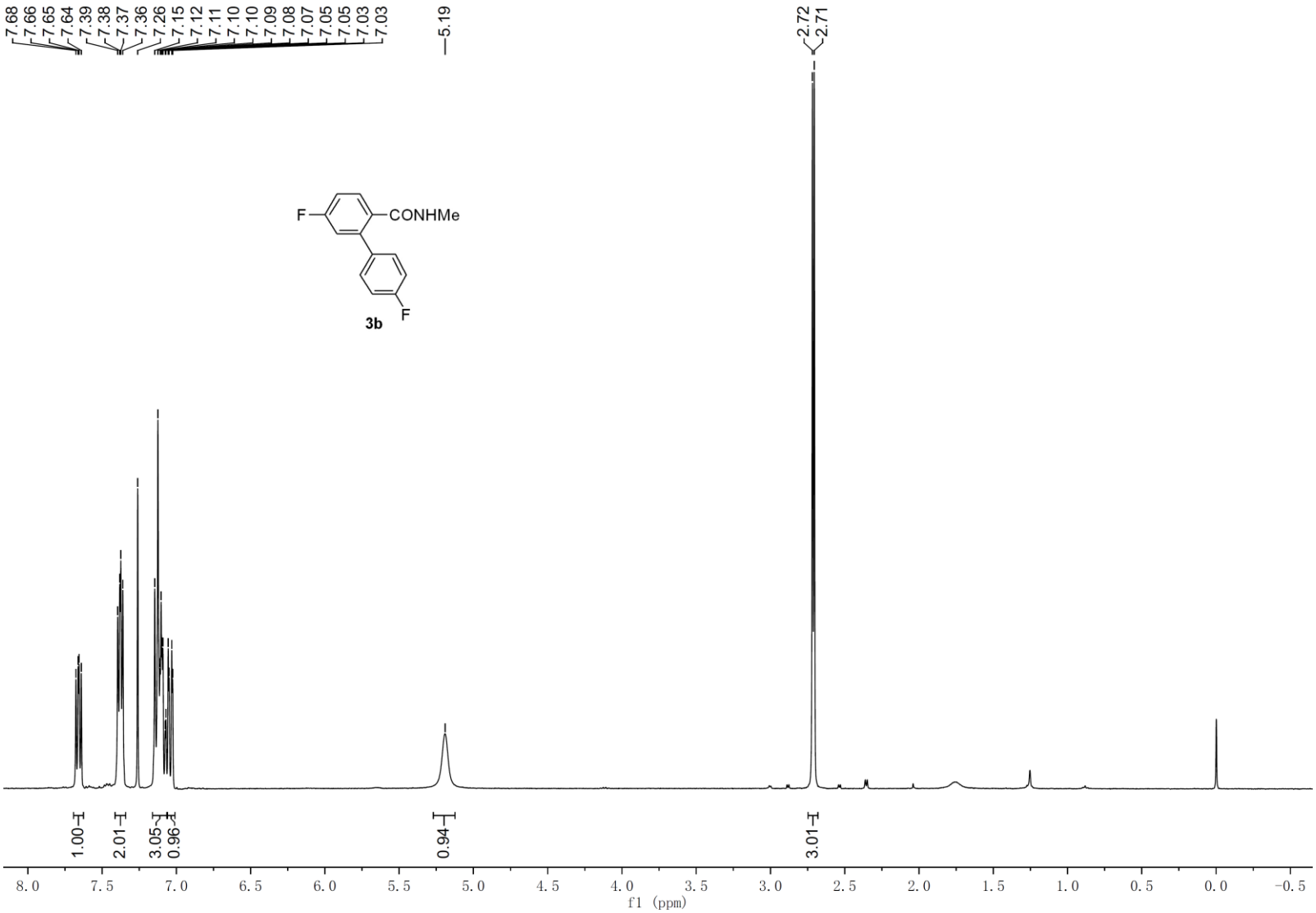
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7.05
7.03
7.03
5.19
2.72
2.71
F
CONHMe
F
3b
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3.05
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0.94
3.01
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7.5
7.0
6.5
6.0
5.5
5.0
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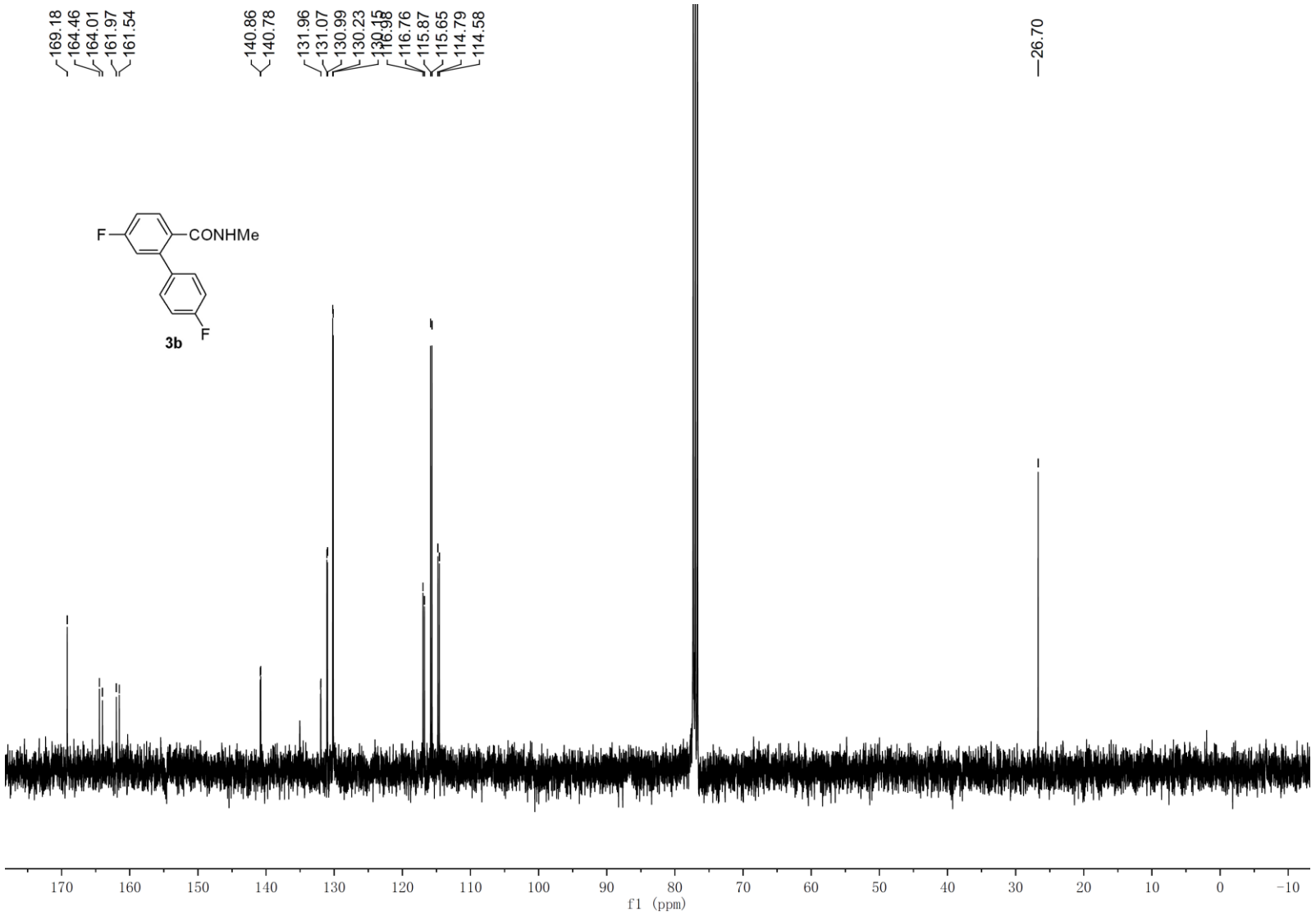
f1 (ppm)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114.58
26.70
F
CONHMe
F
3b
170
160
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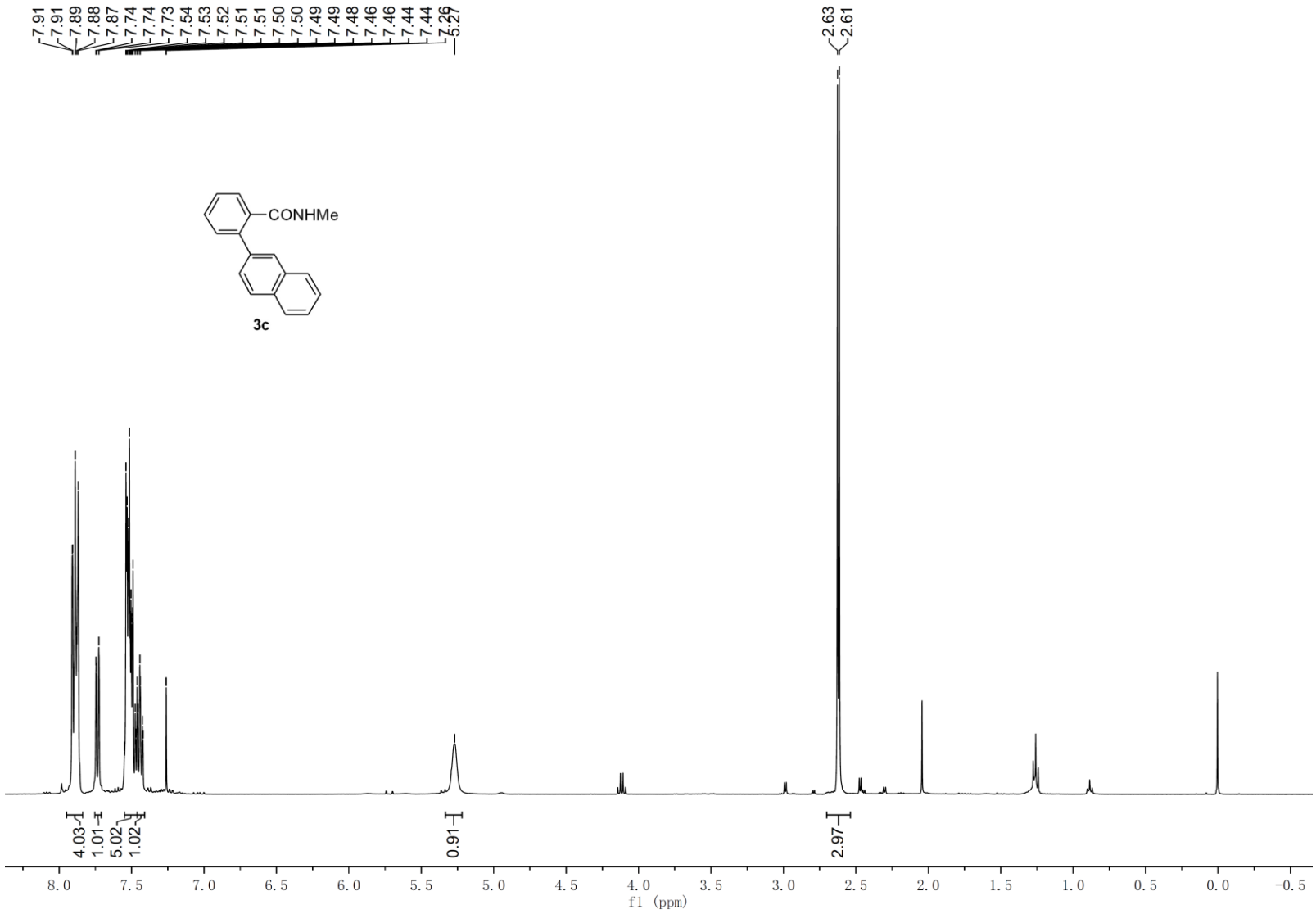
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7.50
7.49
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7.48
7.46
7.46
7.44
7.44
7.26
5.27
2.63
2.61
CONHMe
3c
4.03
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5.02
1.02
0.91
2.97
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5.5
5.0
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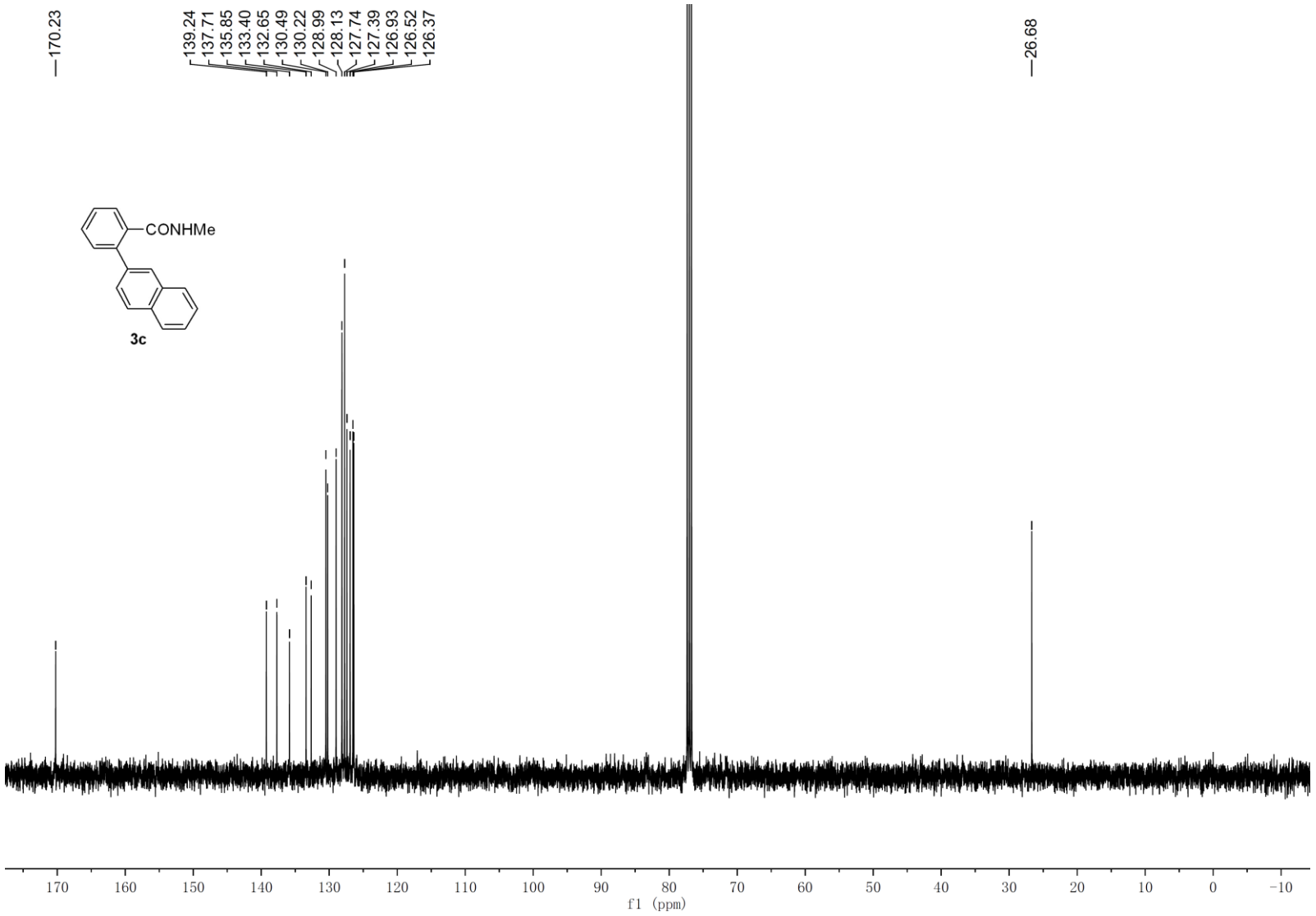
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126.93
126.52
126.37
26.68
CONHMe
3c
170
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80
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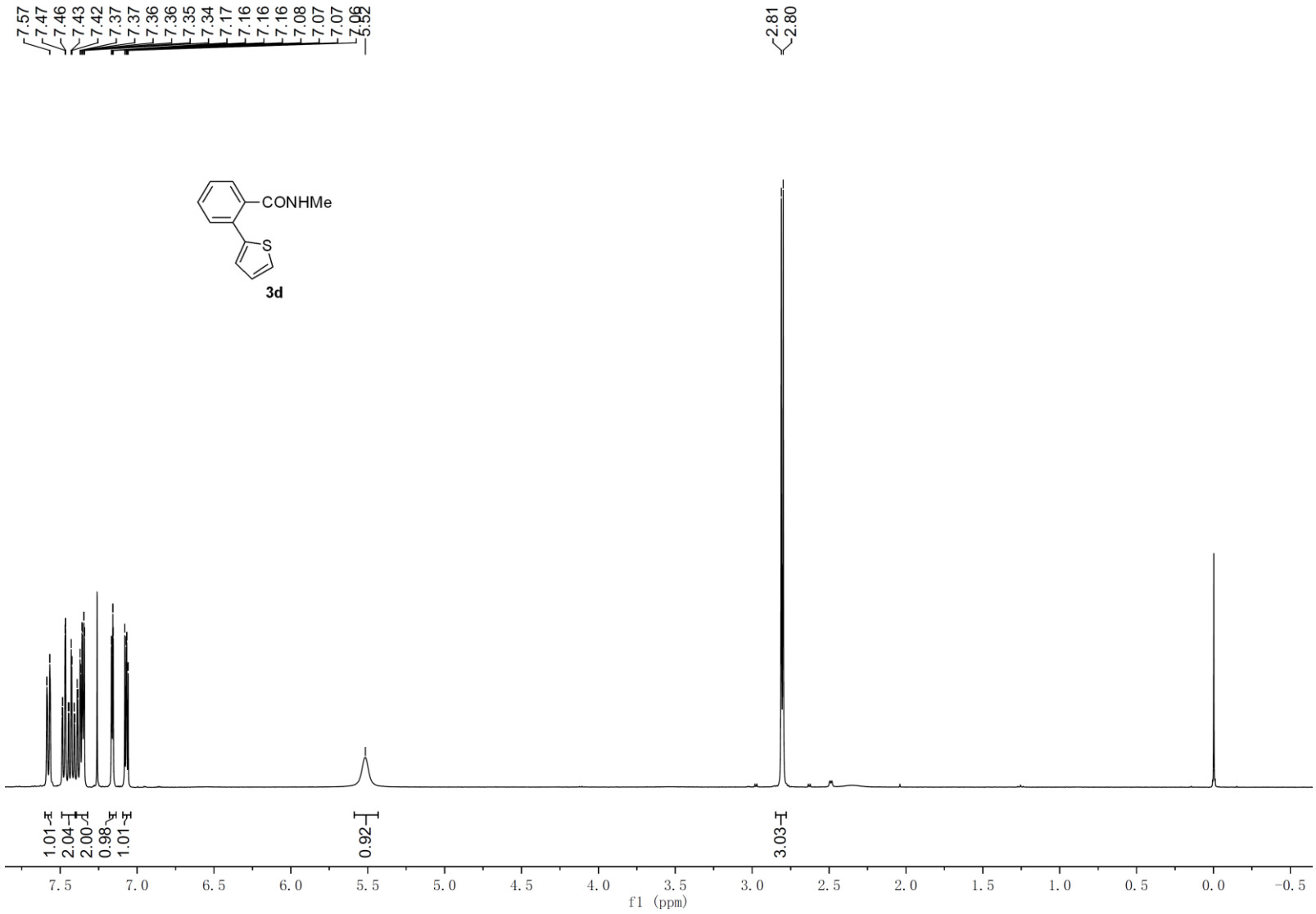
−10
f1 (ppm)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7.08
7.07
7.07
7.06
5.52
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2.80
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S
3d
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2.04
2.00
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1.01
0.92
3.03
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6.0
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5.0
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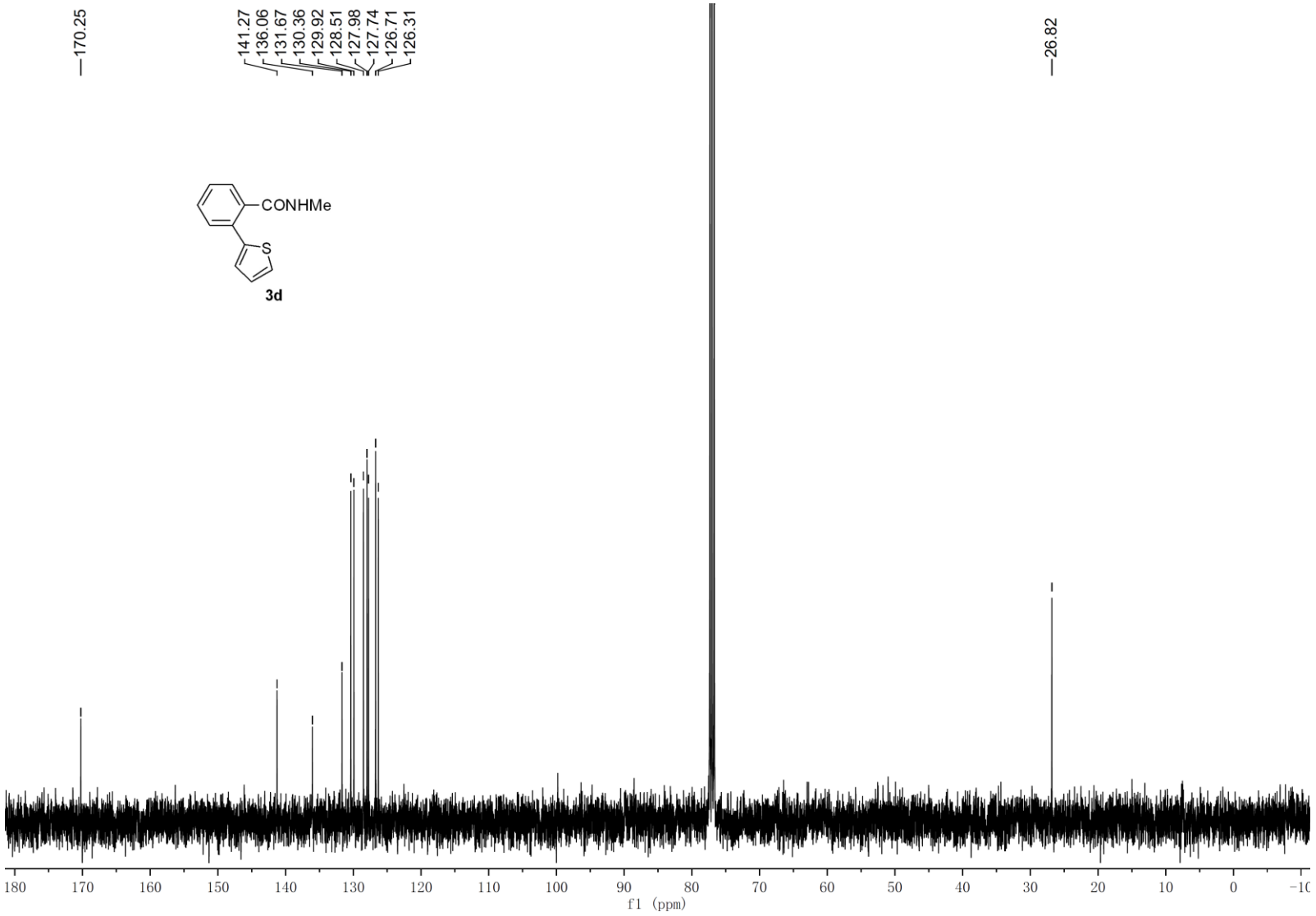
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CONHMe
S
3d
180
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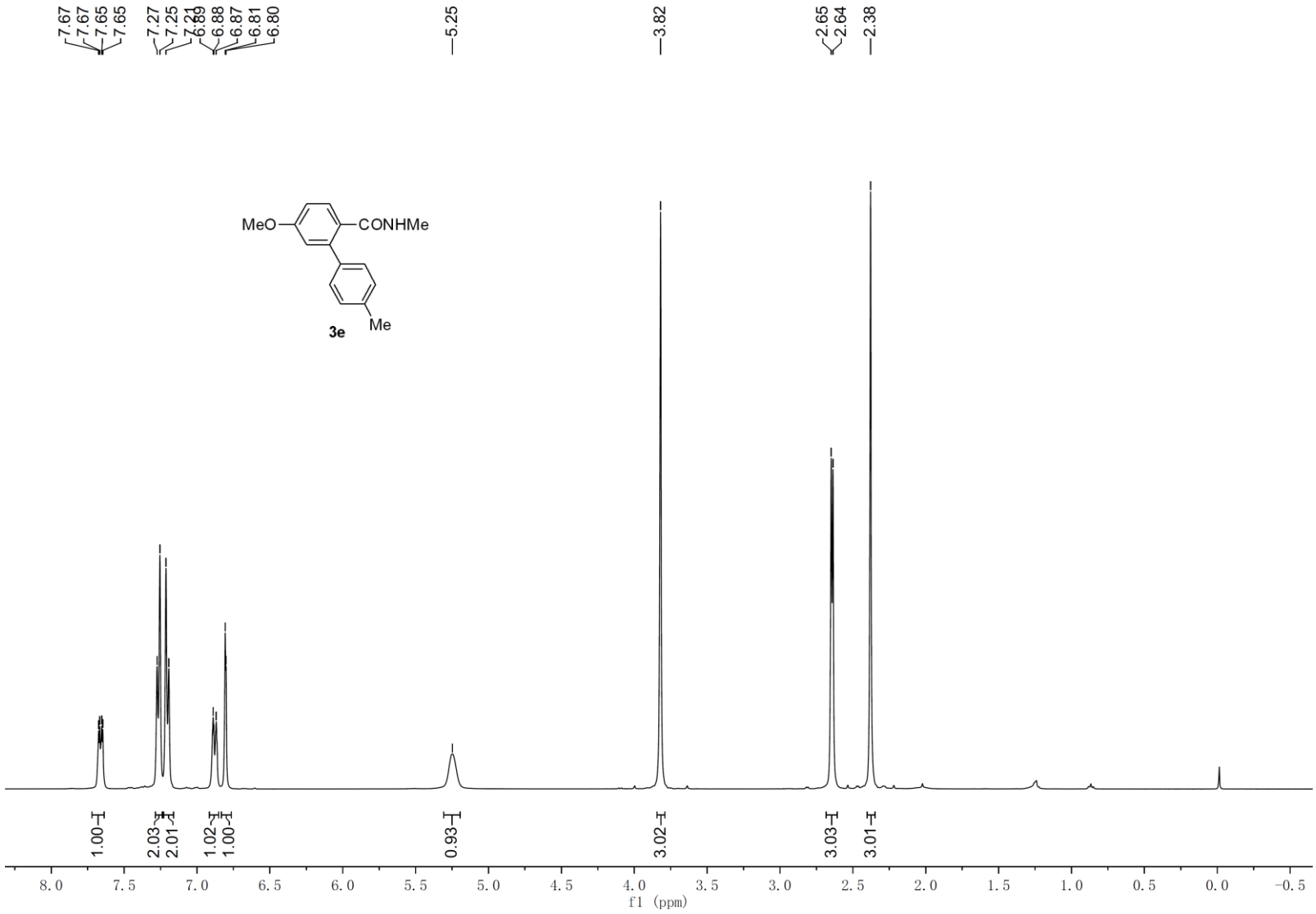
f1 (ppm)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3.82
2.65
2.64
2.38
MeO
CONHMe
Me
3e
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2.03
2.01
1.02
1.00
0.93
3.02
3.03
3.01
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7.5
7.0
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6.0
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5.0
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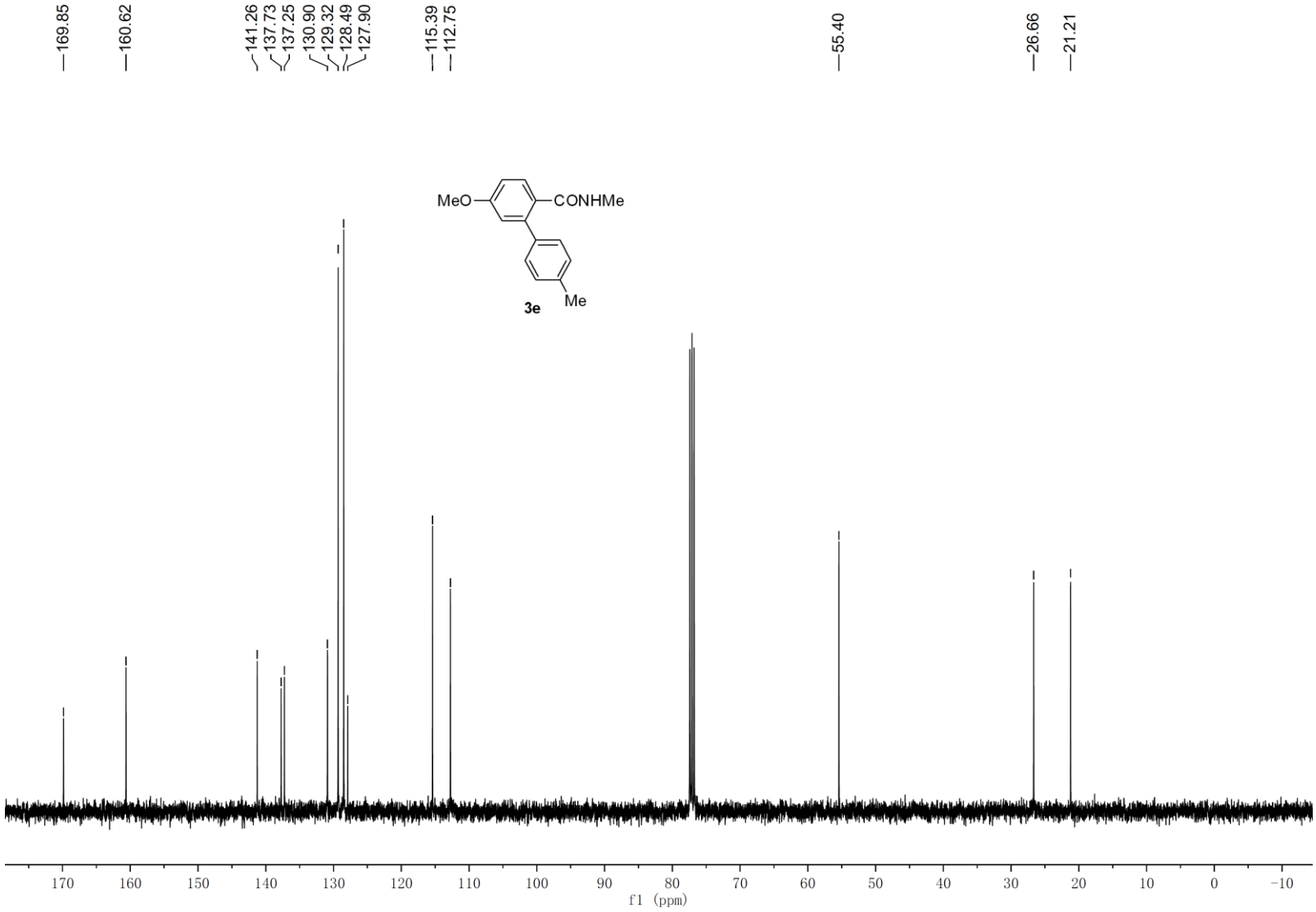
f1 (ppm)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21.21
MeO
CONHMe
Me
3e
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100
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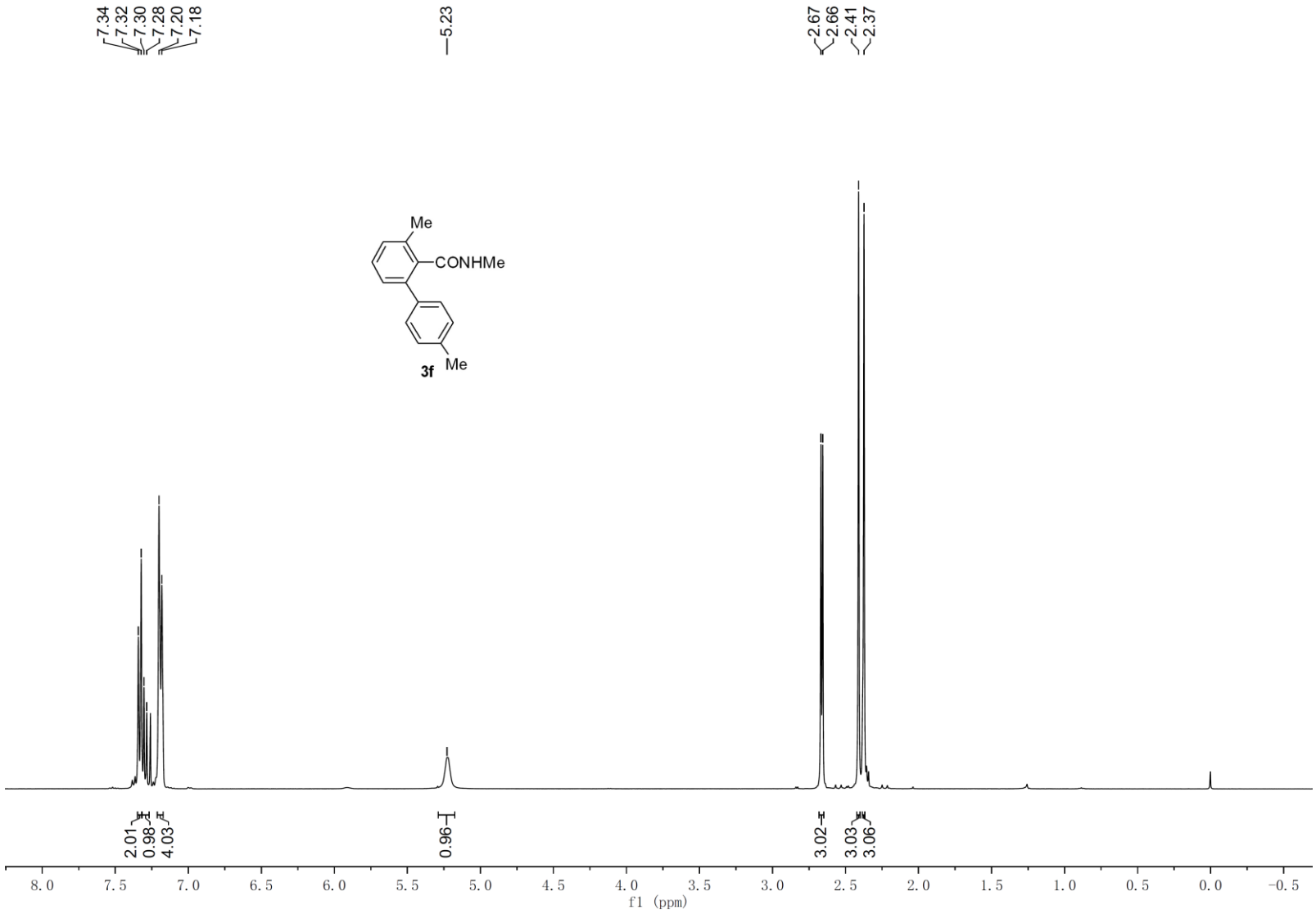
f1 (ppm)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2.37
Me
CONHMe
Me
3f
2.01
0.98
4.03
0.96
3.02
3.03
3.06
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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2.0
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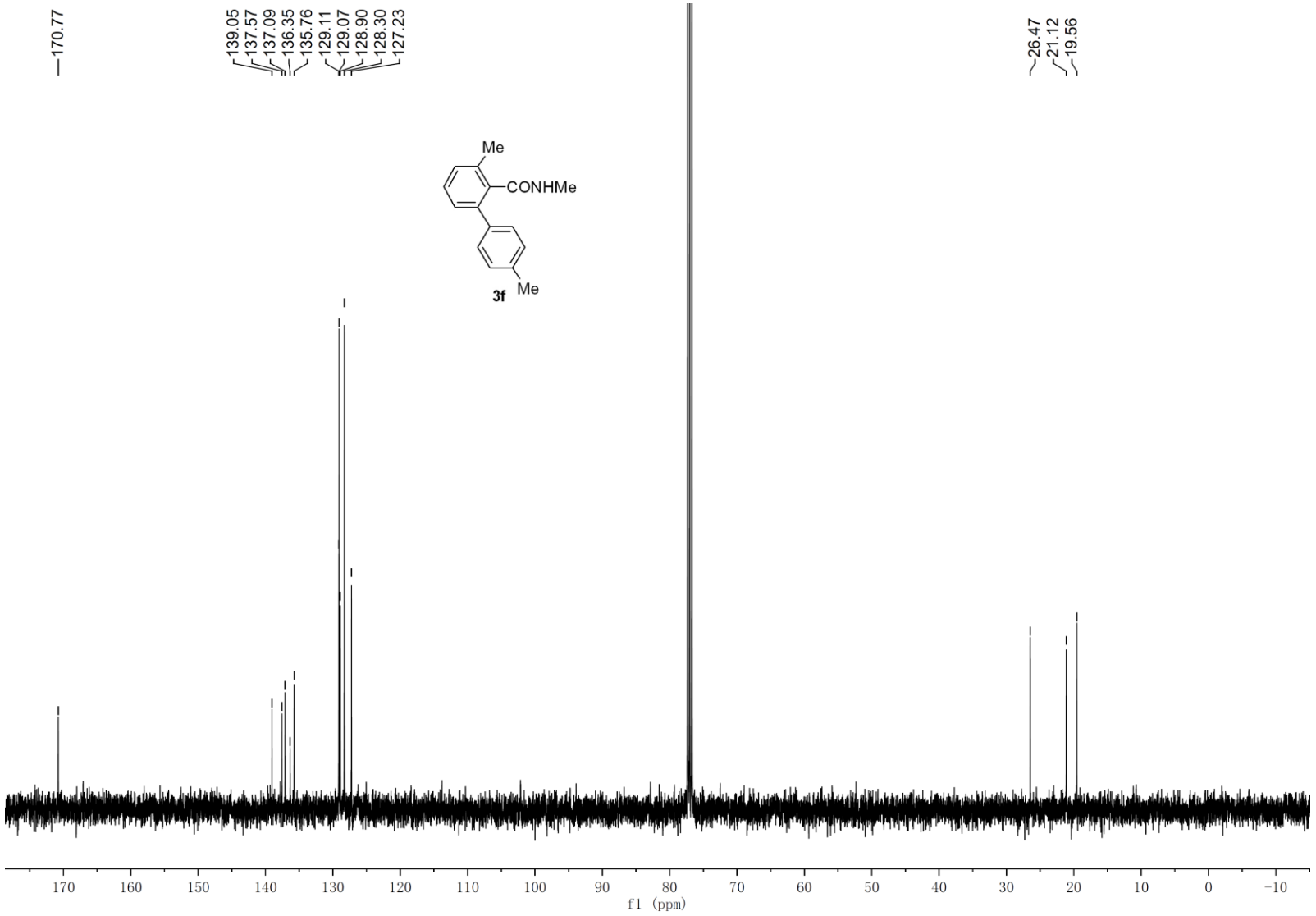
f1 (ppm)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21.12
19.56
Me
CONHMe
Me
3f
170
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140
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120
110
100
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80
70
60
50
40
30
20
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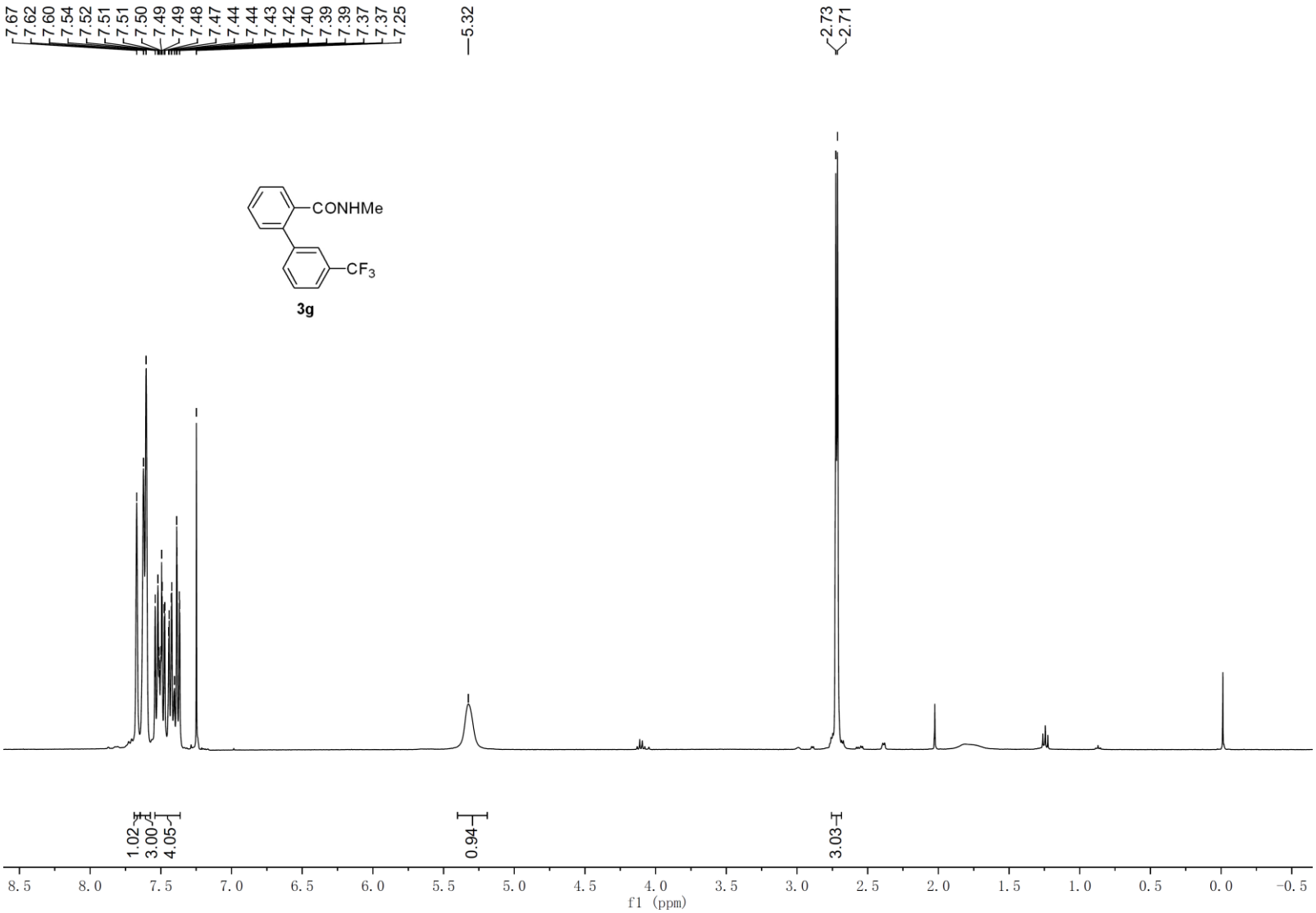
0
−10
f1 (ppm)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7.39
7.39
7.37
7.37
7.25
5.32
2.73
2.71
CONHMe
CF3
3g
1.02
3.00
4.05
0.94
3.03
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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4.0
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3.0
2.5
2.0
1.5
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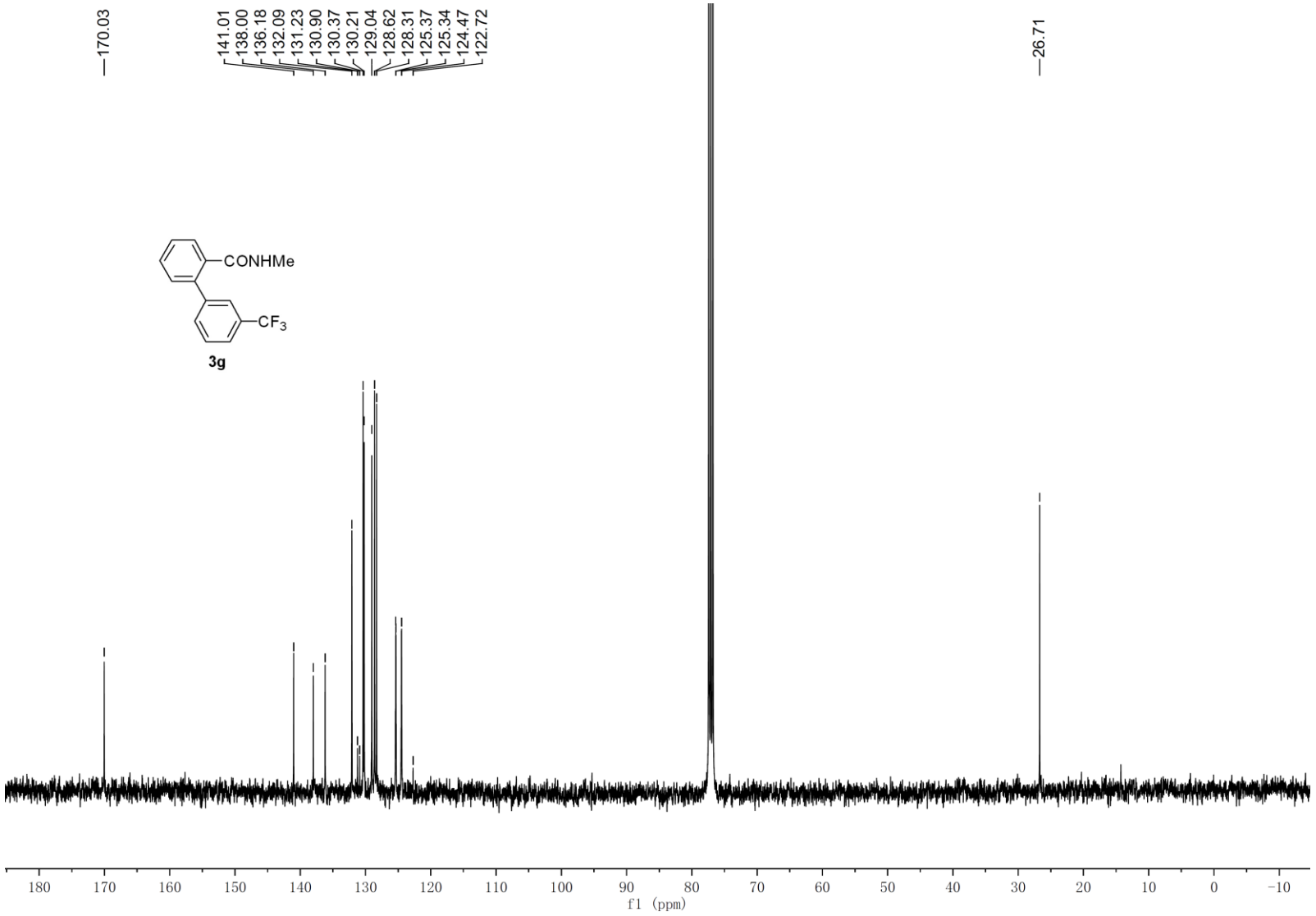
-0.5
f1 (ppm)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125.37
125.34
124.47
122.72
26.71
CONHMe
CF3
3g
180
170
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140
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120
110
100
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80
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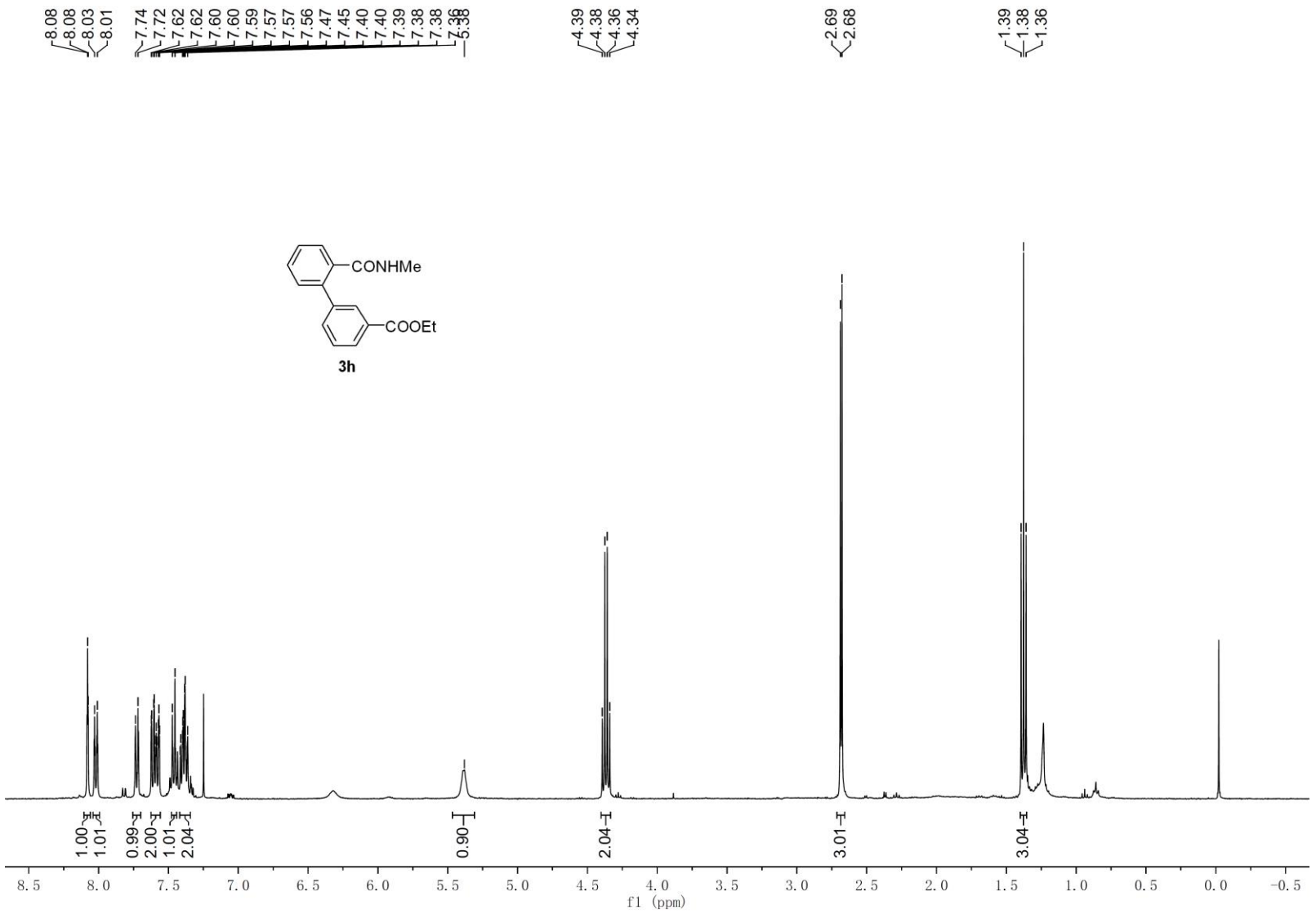
60
50
40
30
20
10
0
−10
f1 (ppm)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7.39
7.38
7.38
7.36
7.36
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4.38
4.36
4.34
2.69
2.68
1.39
1.38
1.36
CONHMe
COOEt
3h
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1.01
0.99
2.00
1.01
2.04
0.90
2.04
3.01
3.04
8.5
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7.0
6.5
6.0
5.5
5.0
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2.0
1.5
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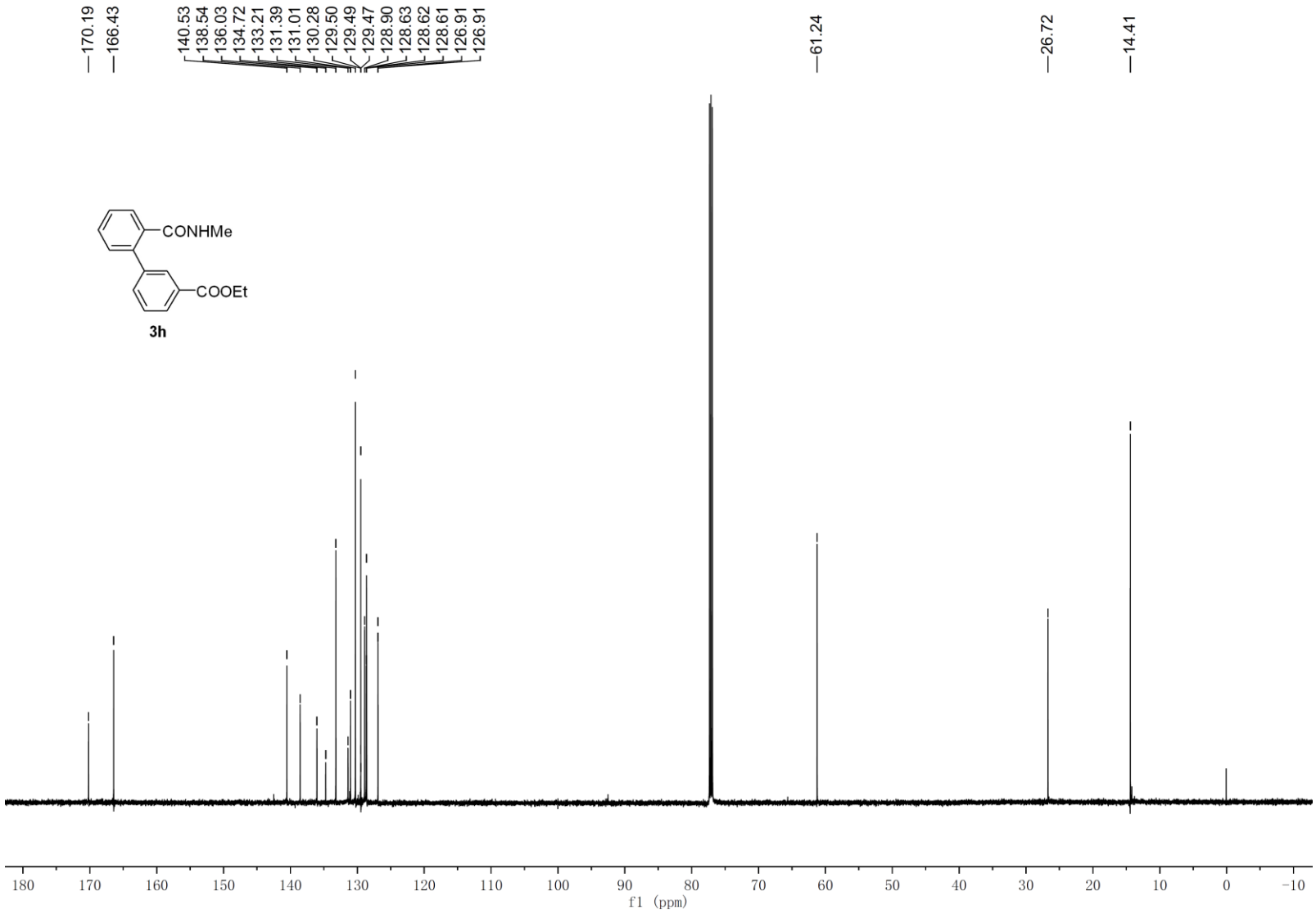
0.0
-0.5
f1 (ppm)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129.47
128.90
128.63
128.62
128.61
126.91
126.91
61.24
26.72
14.41
CONHMe
COOEt
3h
180
170
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120
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100
90
80
70
60
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40
30
20
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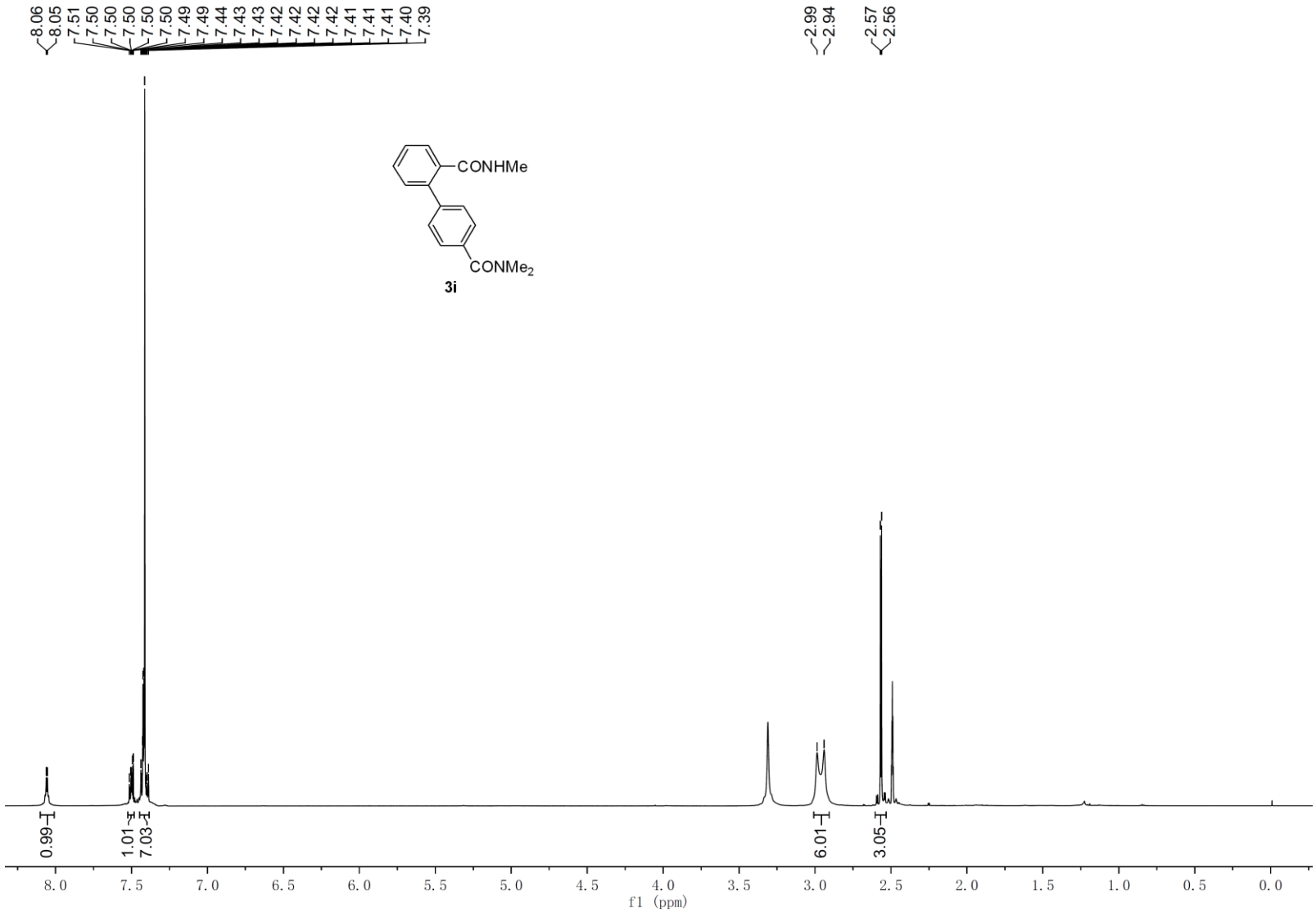
0
−10
f1 (ppm)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7.42
7.42
7.42
7.41
7.41
7.41
7.40
7.39
2.99
2.94
2.57
2.56
CONHMe
CONMe2
3i
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1.01
7.03
6.01
3.05
8.0
7.5
7.0
6.5
6.0
5.5
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1.0
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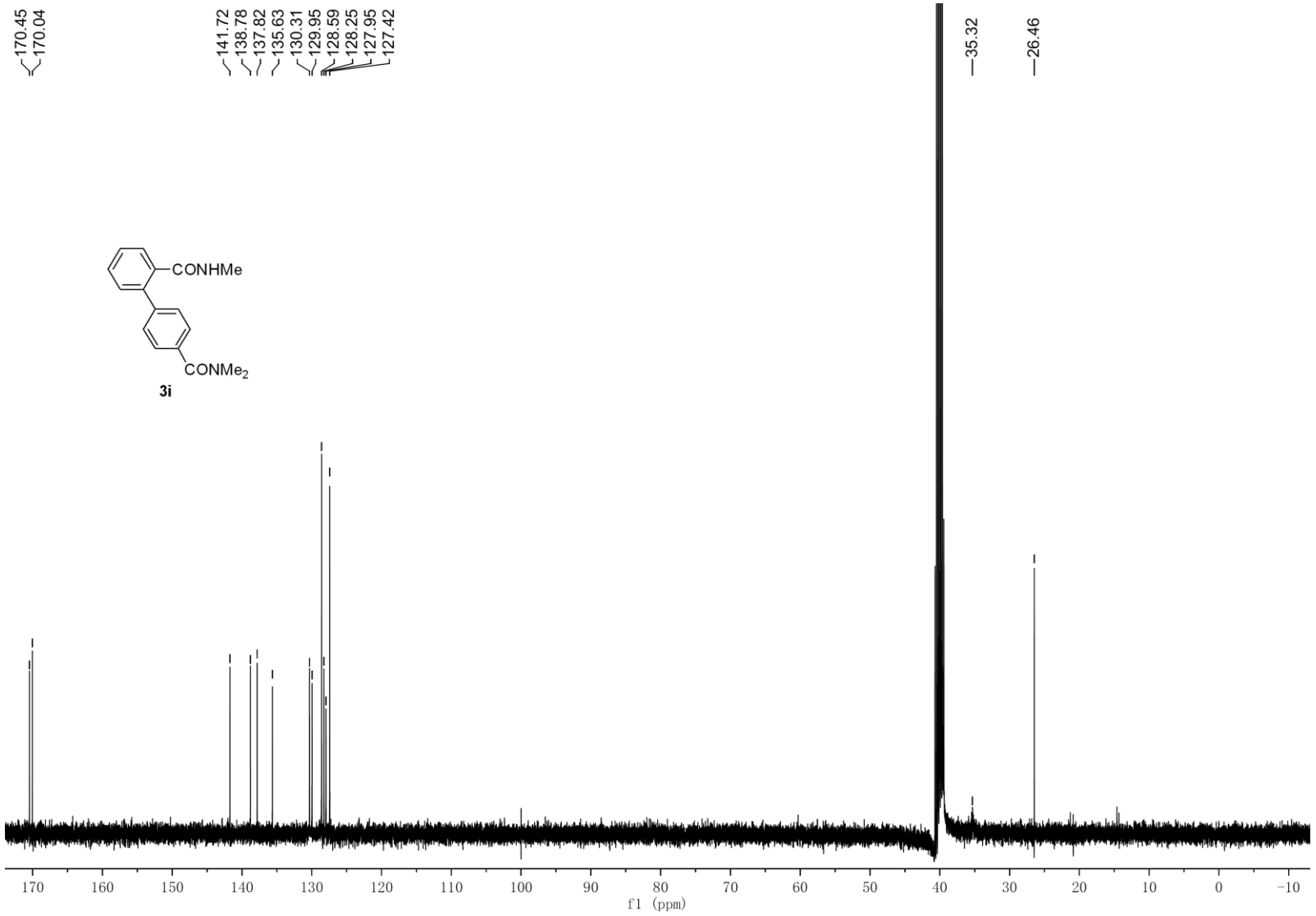
0.0
f1 (ppm)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35.32
26.46
CONHMe
CONMe2
3i
170
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120
110
100
90
80
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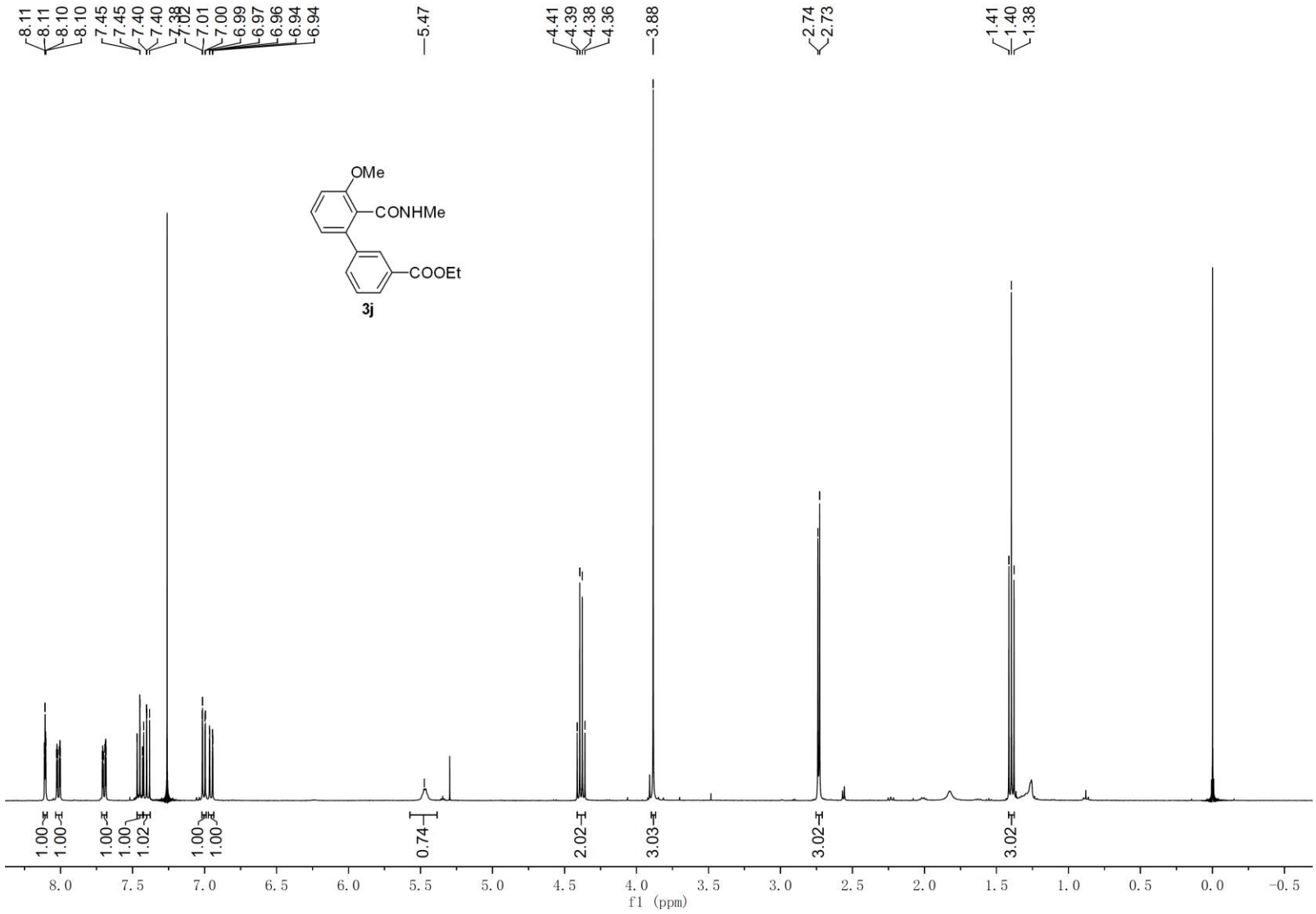
−10
f1 (ppm)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6.99
6.97
6.96
6.94
6.94
5.47
4.41
4.39
4.38
4.36
3.88
2.74
2.73
1.41
1.40
1.38
OMe
CONHMe
COOEt
3j
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1.00
1.00
1.00
1.02
1.00
1.00
0.74
2.02
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3.02
3.02
8.0
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7.0
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6.0
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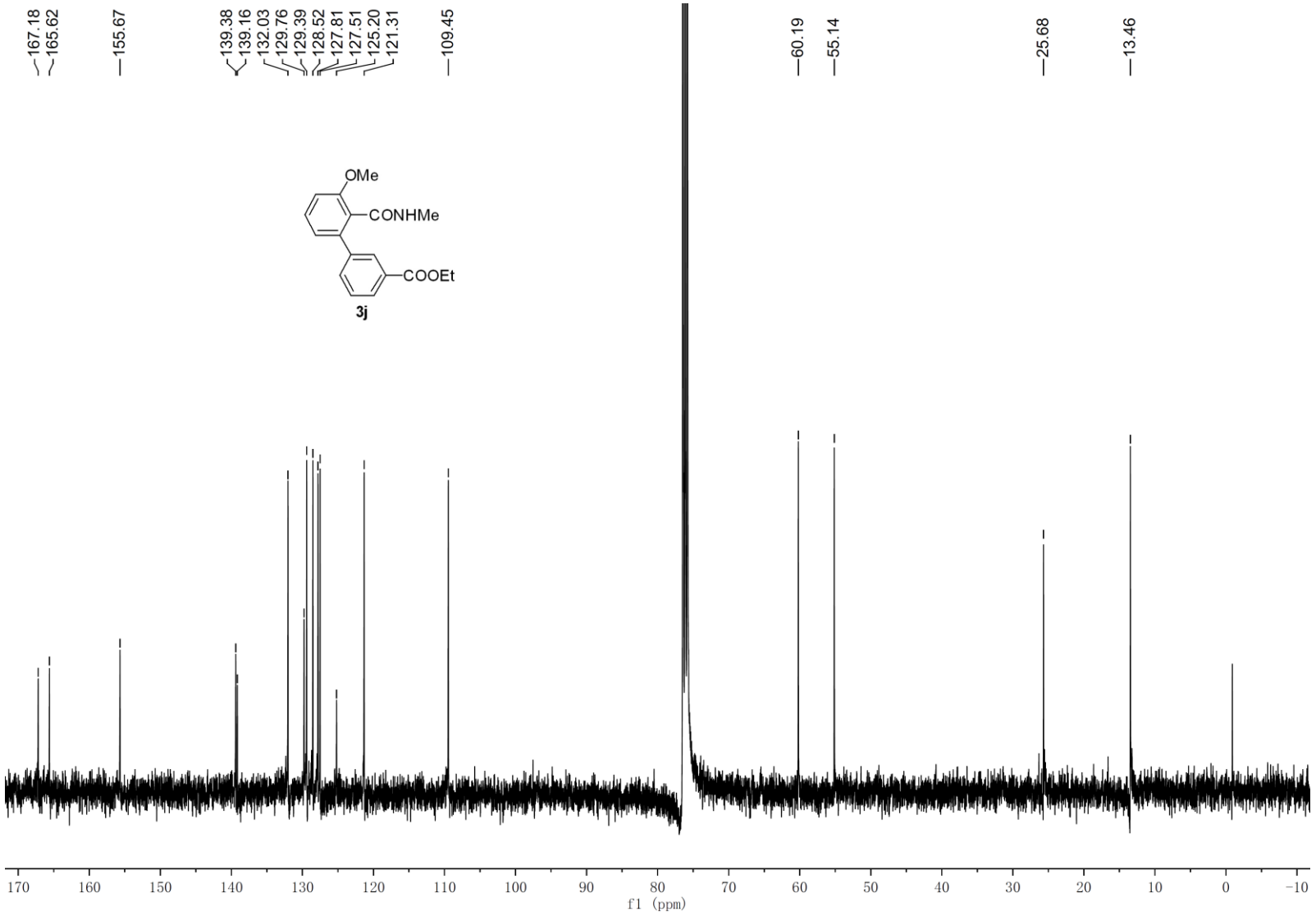
f1 (ppm)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121.31
109.45
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55.14
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13.46
OMe
CONHMe
COOEt
3j
170
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120
110
100
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80
70
60
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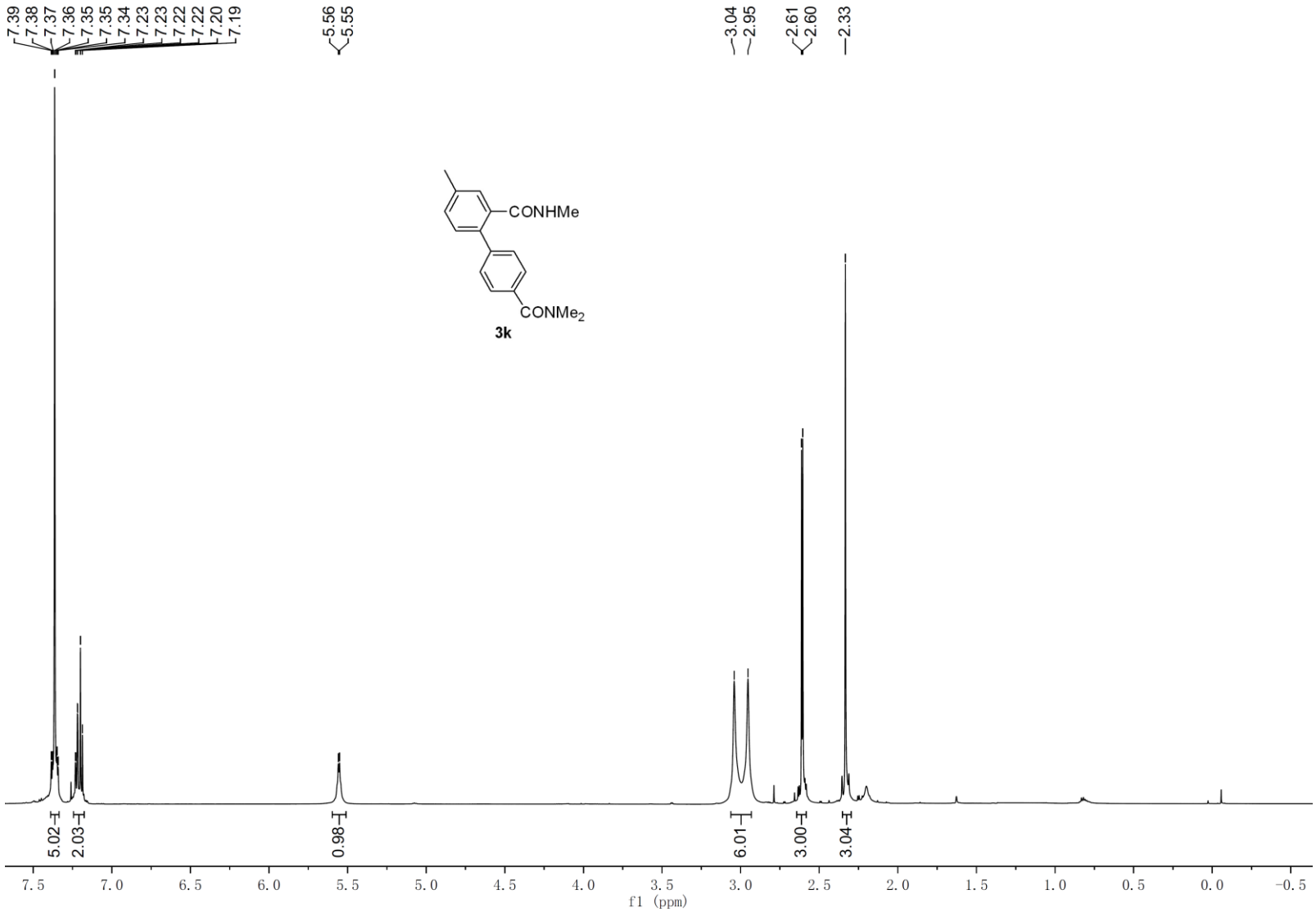
40
30
20
10
0
−10
f1 (ppm)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7.19
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5.55
3.04
2.95
2.61
2.60
2.33
CONHMe
CONMe2
3k
5.02
2.03
0.98
6.01
3.00
3.04
7.5
7.0
6.5
6.0
5.5
5.0
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2.0
1.5
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0.5
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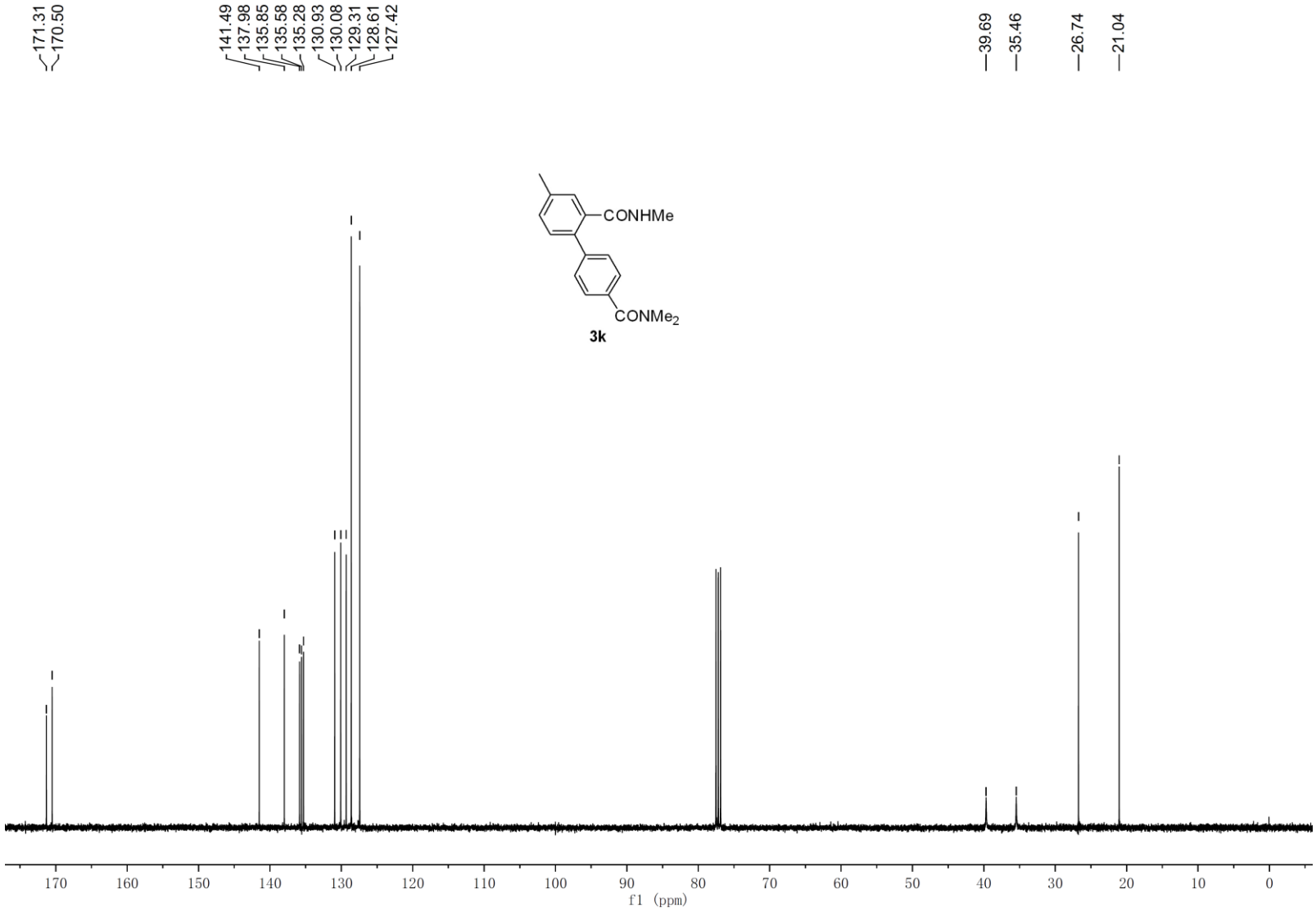
f1 (ppm)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39.69
35.46
26.74
21.04
CONHMe
CONMe2
3k
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110
100
90
80
70
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50
40
30
20
10
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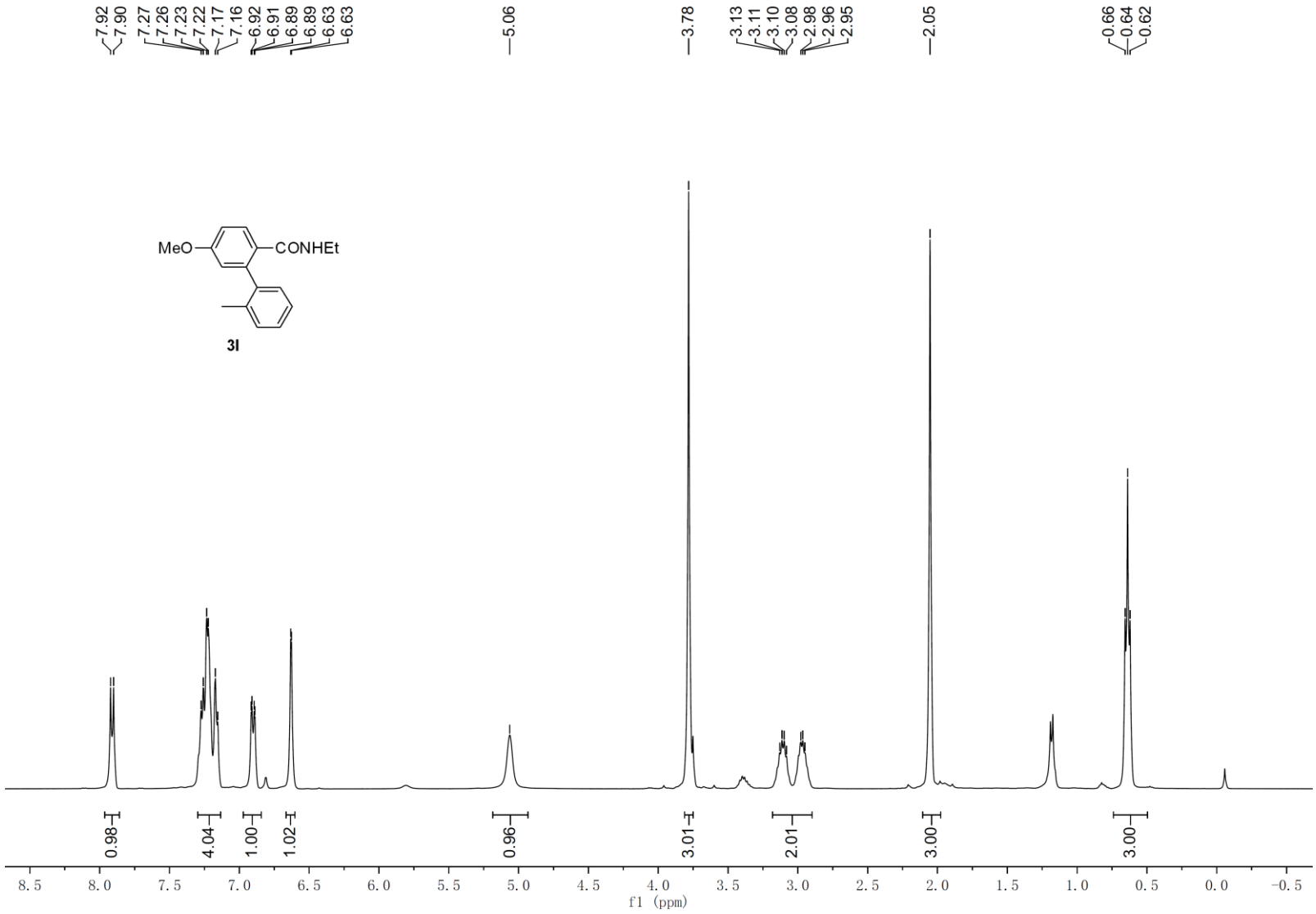
f1 (ppm)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5.06
3.78
3.13
3.11
3.10
3.08
2.98
2.96
2.95
2.05
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0.64
0.62
MeO
CONHEt
3l
0.98
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1.02
0.96
3.01
2.01
3.00
3.00
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7.5
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5.0
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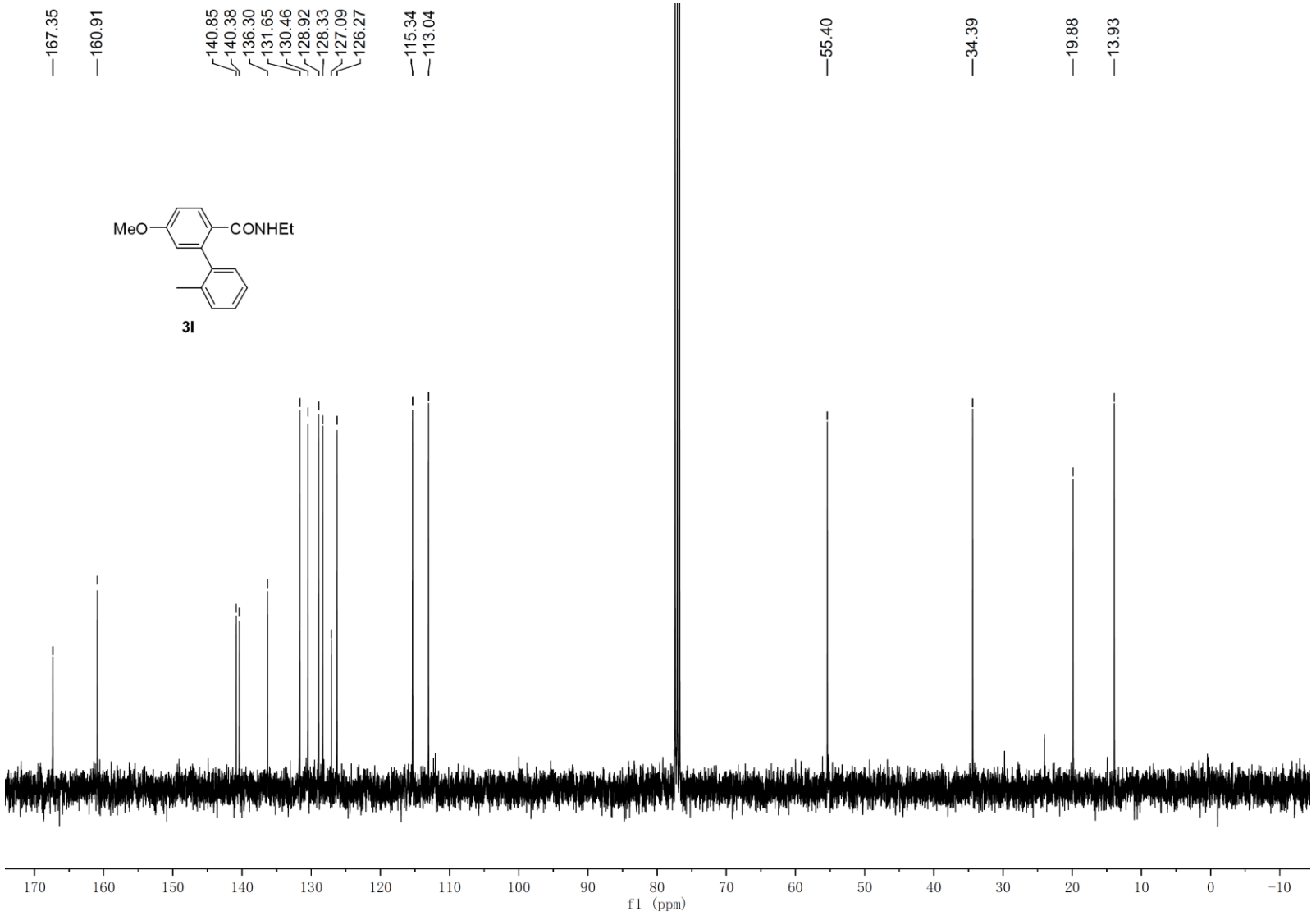
f1 (ppm)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113.04
55.40
34.39
19.88
13.93
MeO
CONHEt
3l
170
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140
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120
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100
90
80
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60
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40
30
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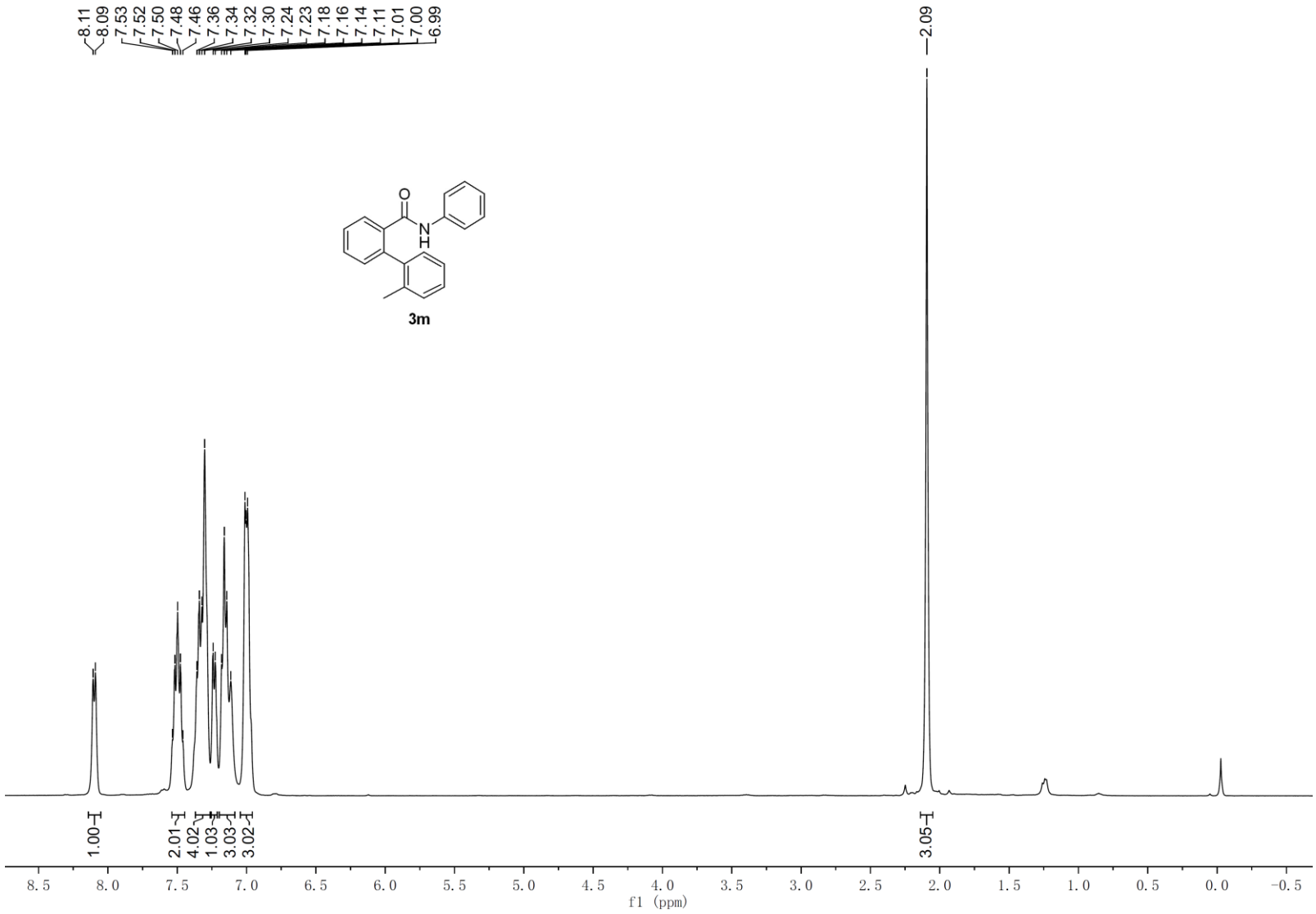
0
−10
f1 (ppm)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7.23
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7.16
7.14
7.11
7.01
7.00
6.99
2.09
O
N
H
3m
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2.01
4.02
1.03
3.03
3.02
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6.5
6.0
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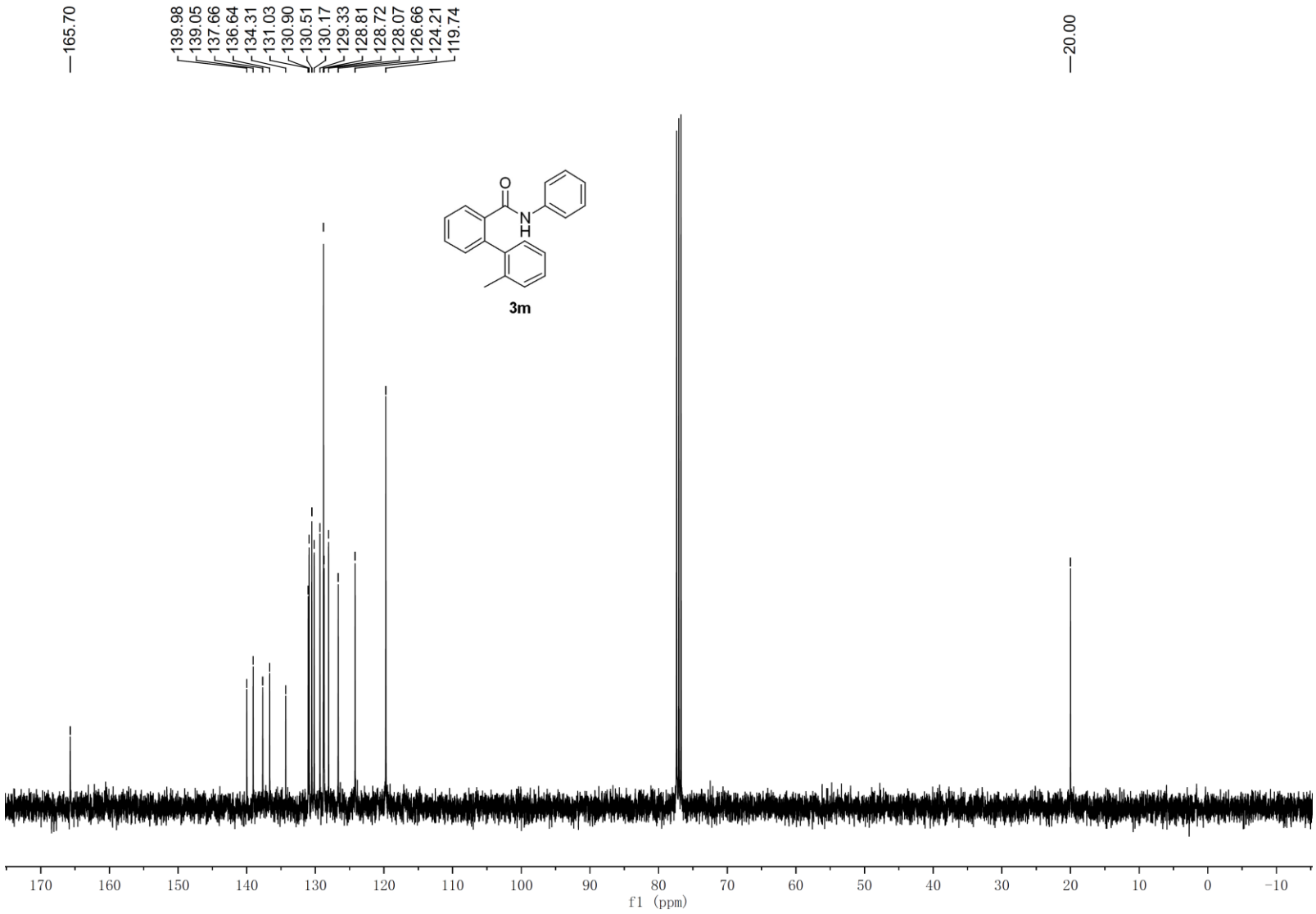
f1 (ppm)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128.81
128.72
128.07
126.66
124.21
119.74
20.00
O
N
H
3m
170
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140
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120
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100
90
80
70
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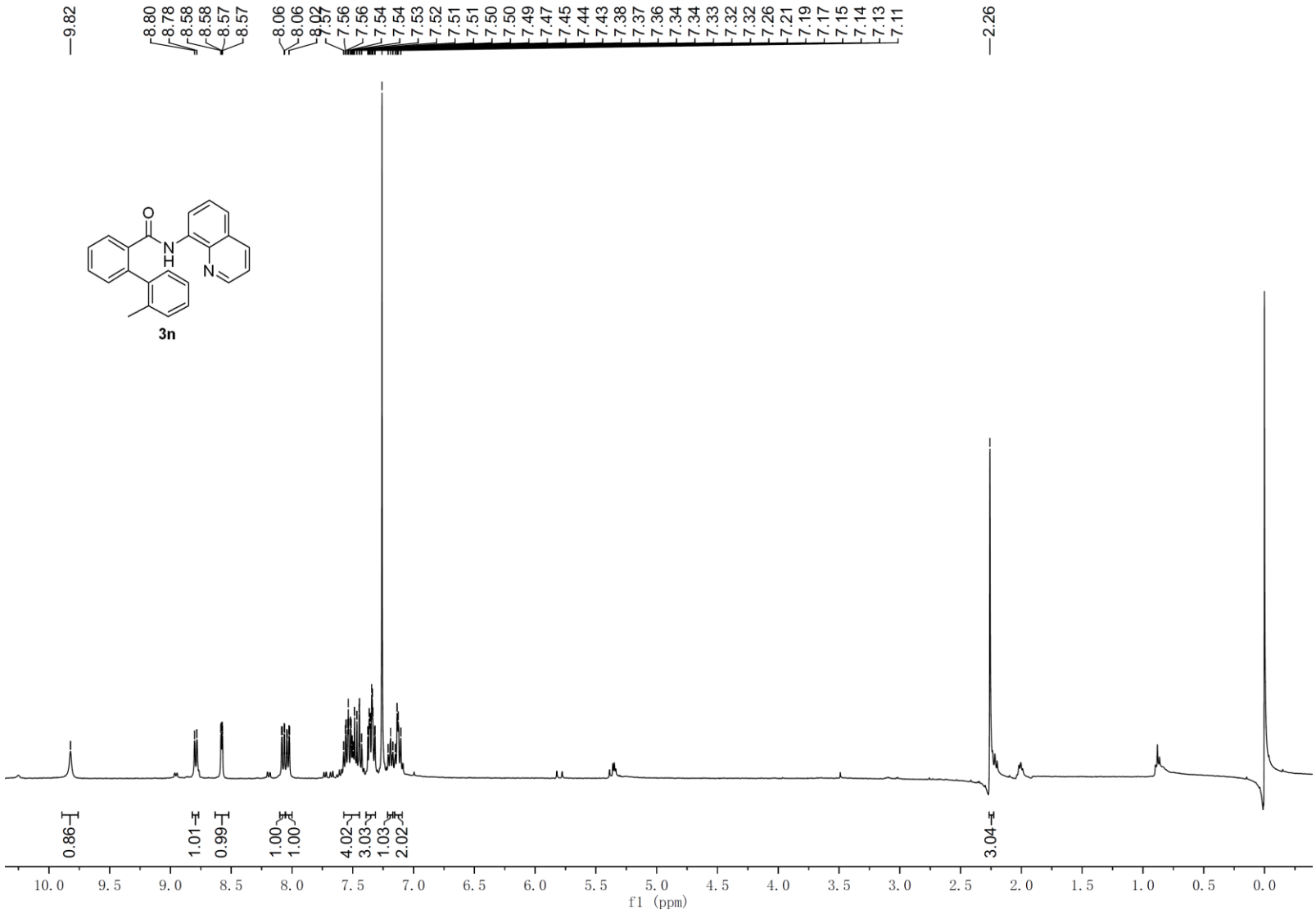
0
−10
f1 (ppm)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2.02
3.04
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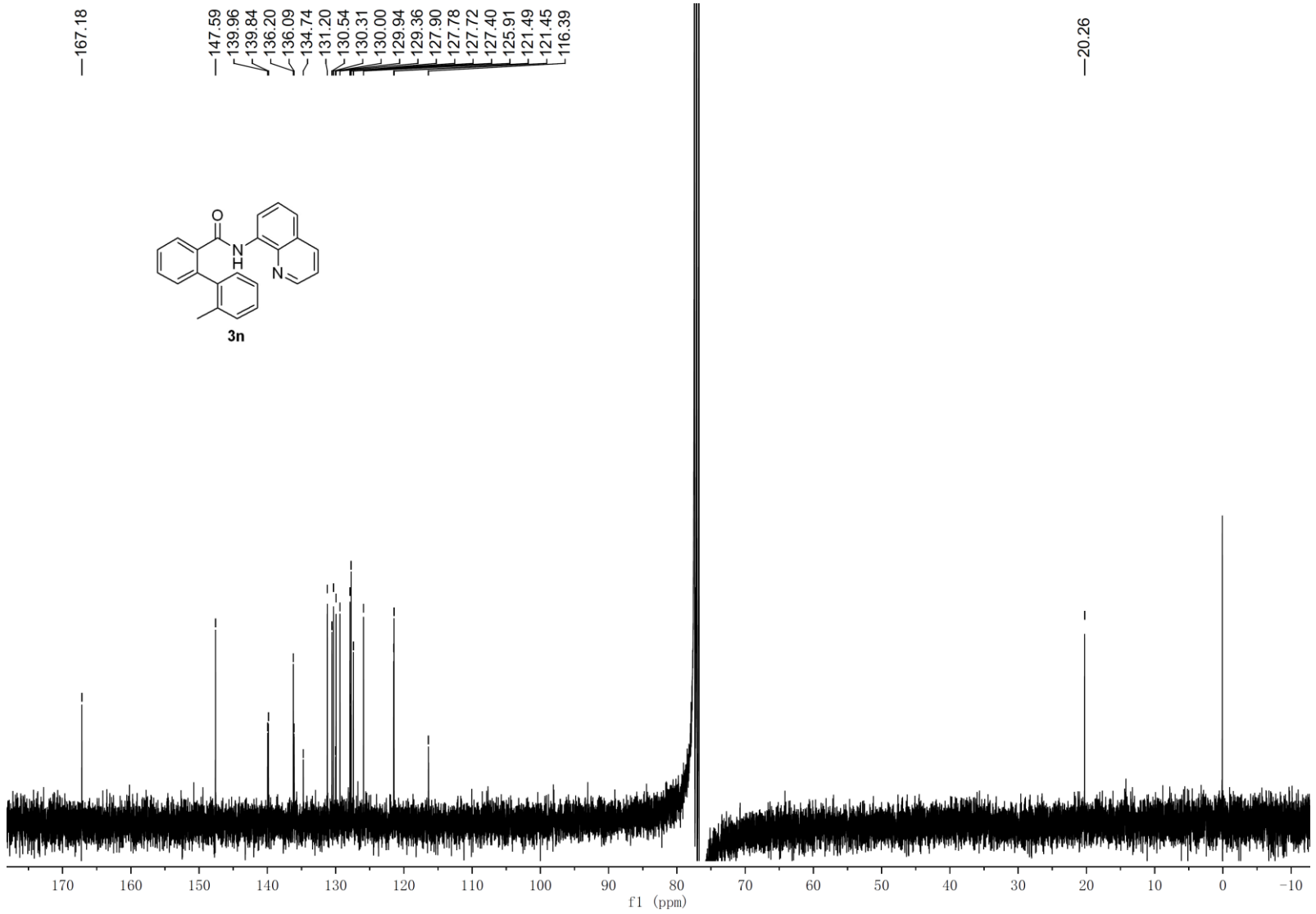
f1 (ppm)


167.18
147.59
139.96
139.84
136.20
136.09
134.74
131.20
130.54
130.31
130.00
129.94
129.36
127.90
127.78
127.72
127.40
125.91
121.49
121.45
116.39
20.26
O
N
H
N
3n
170
160
150
140
130
120
110
100
90
80
70
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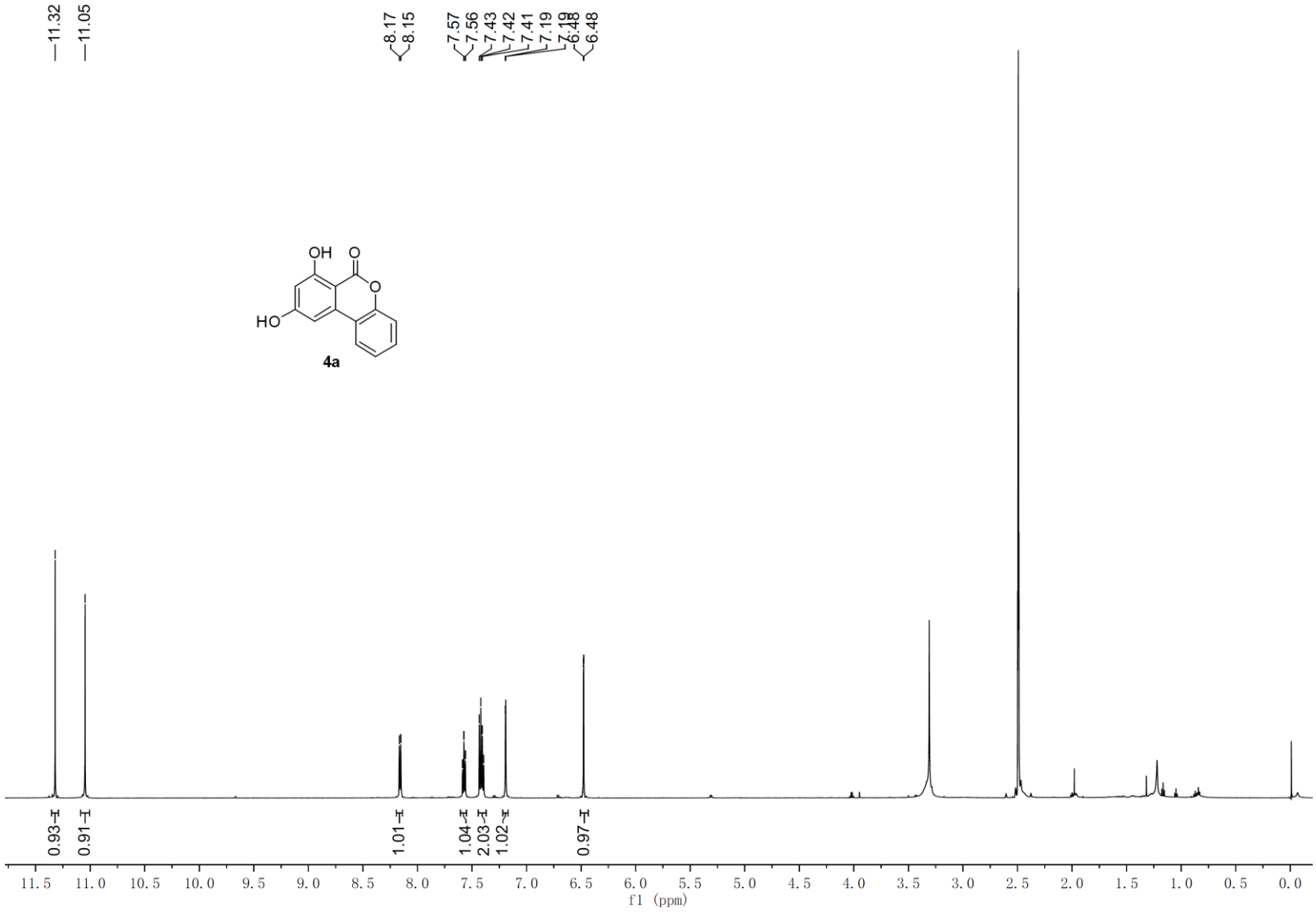
50
40
30
20
10
0
−10
f1 (ppm)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11.05
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8.15
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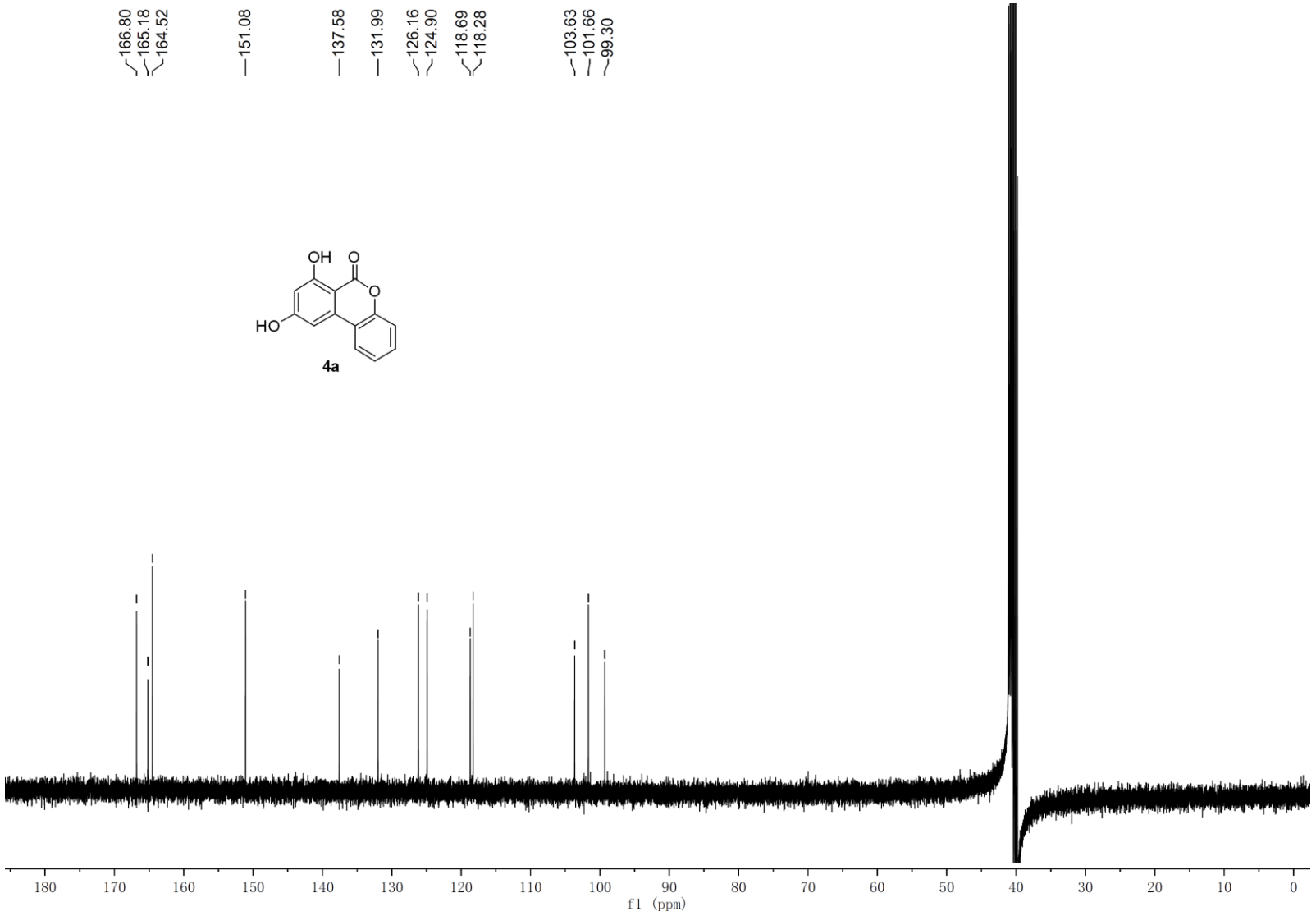
7.56
7.43
7.42
7.41
7.19
7.19
6.48
6.48
OH
O
O
HO
4a
0.93
0.91
1.01
1.04
2.03
1.02
0.97
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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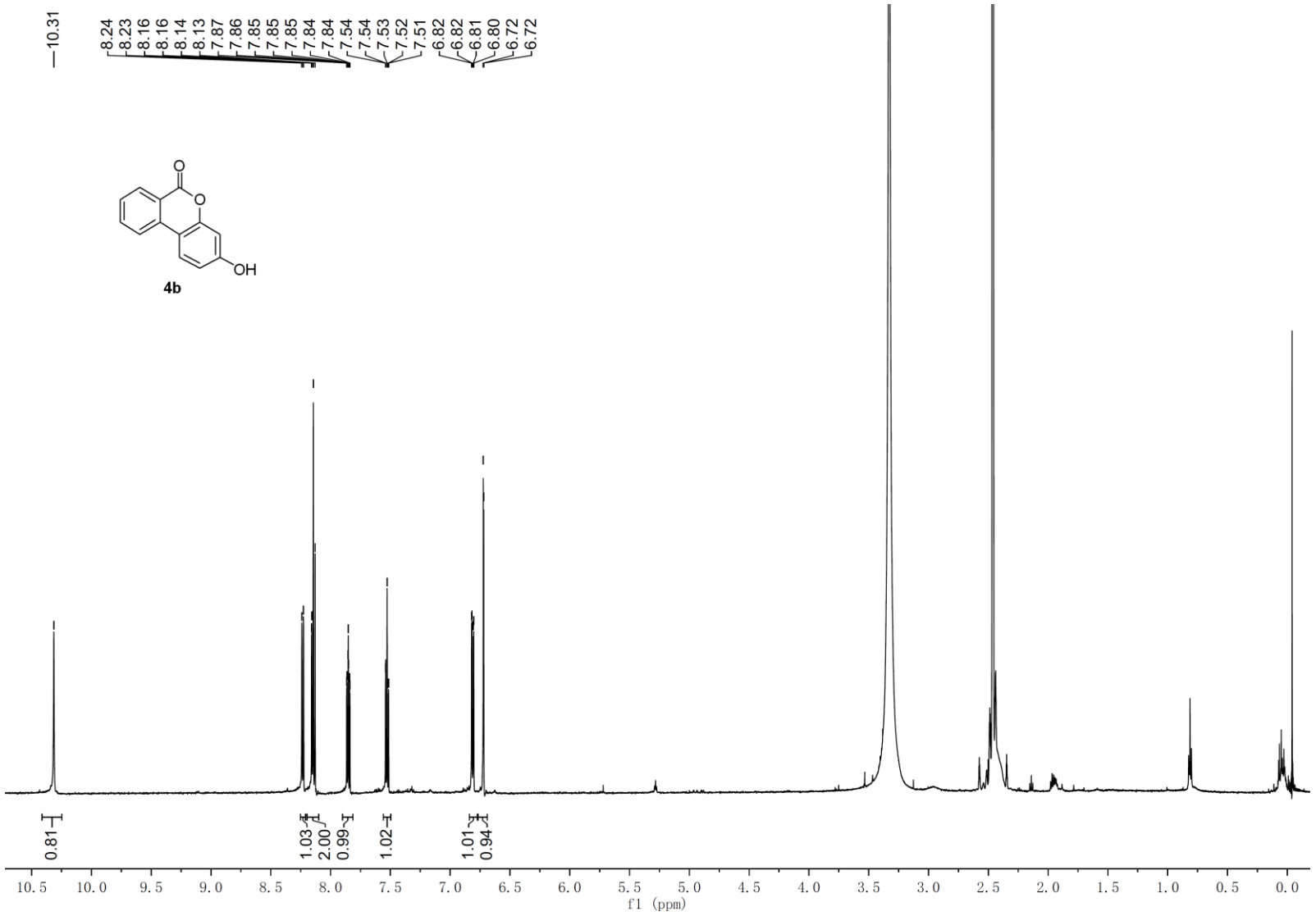
165.18
164.52
151.08
137.58
131.99
126.16
124.90
118.69
118.28
103.63
101.66
99.30
OH
O
O
HO
4a
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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8.23
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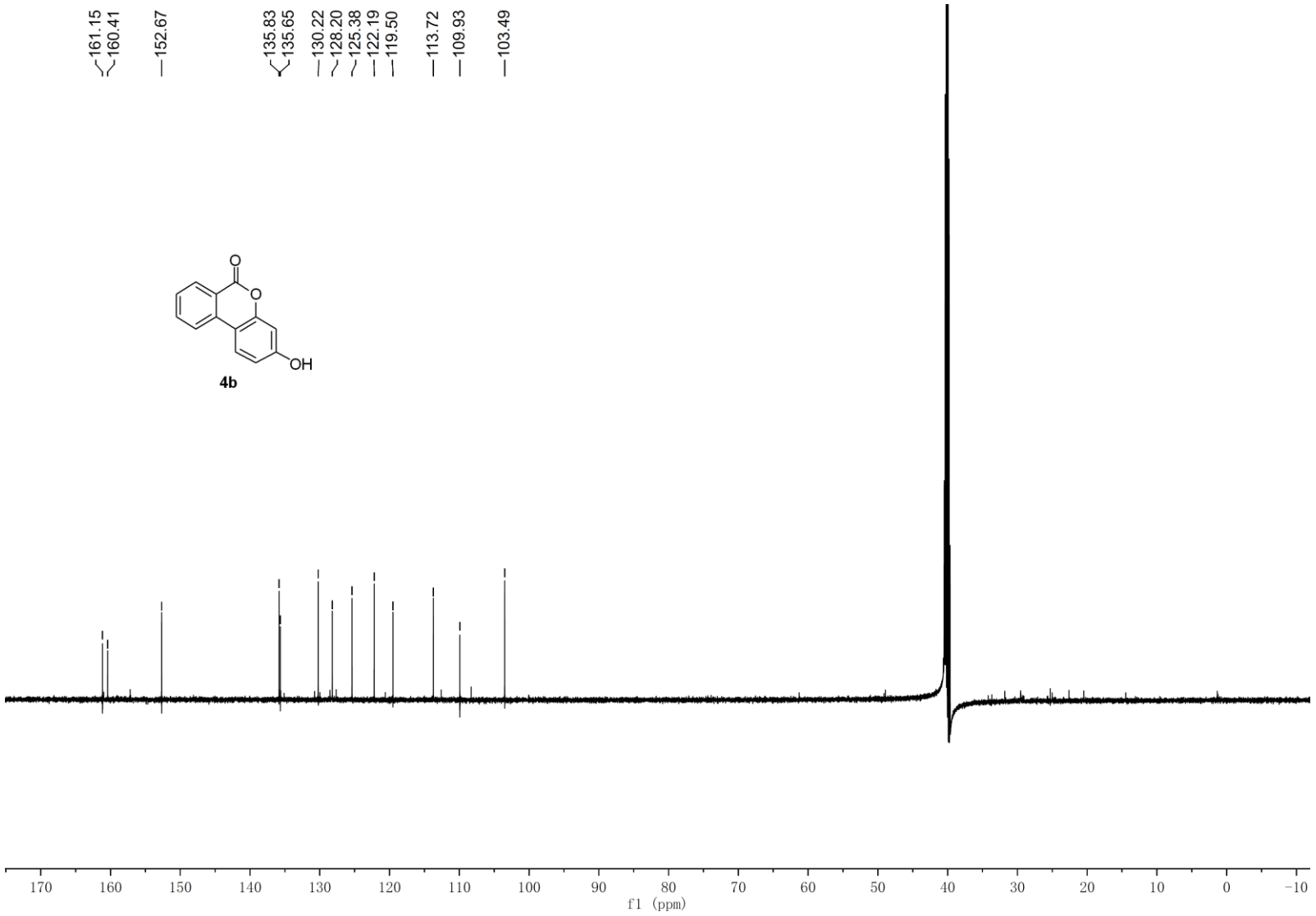
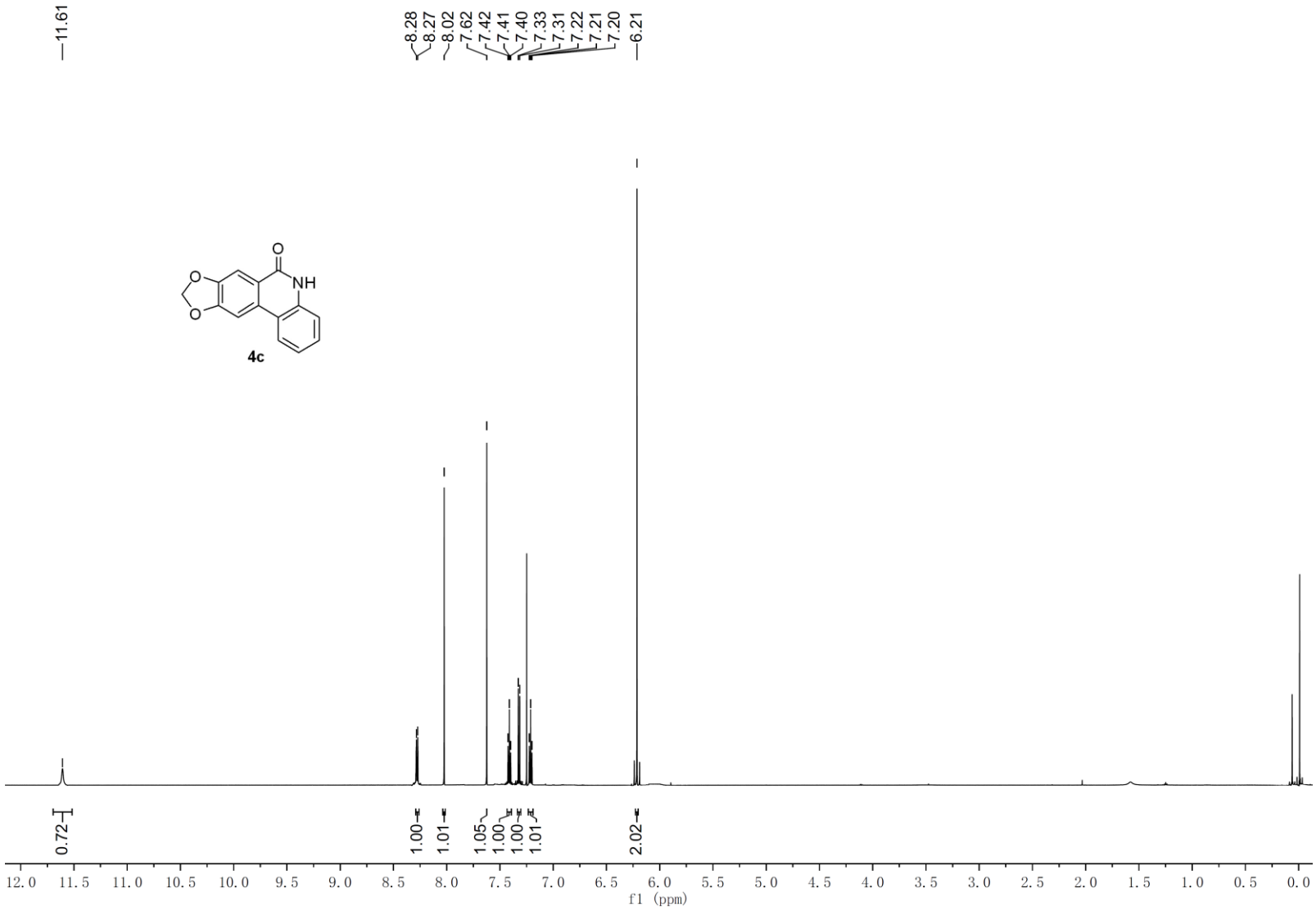
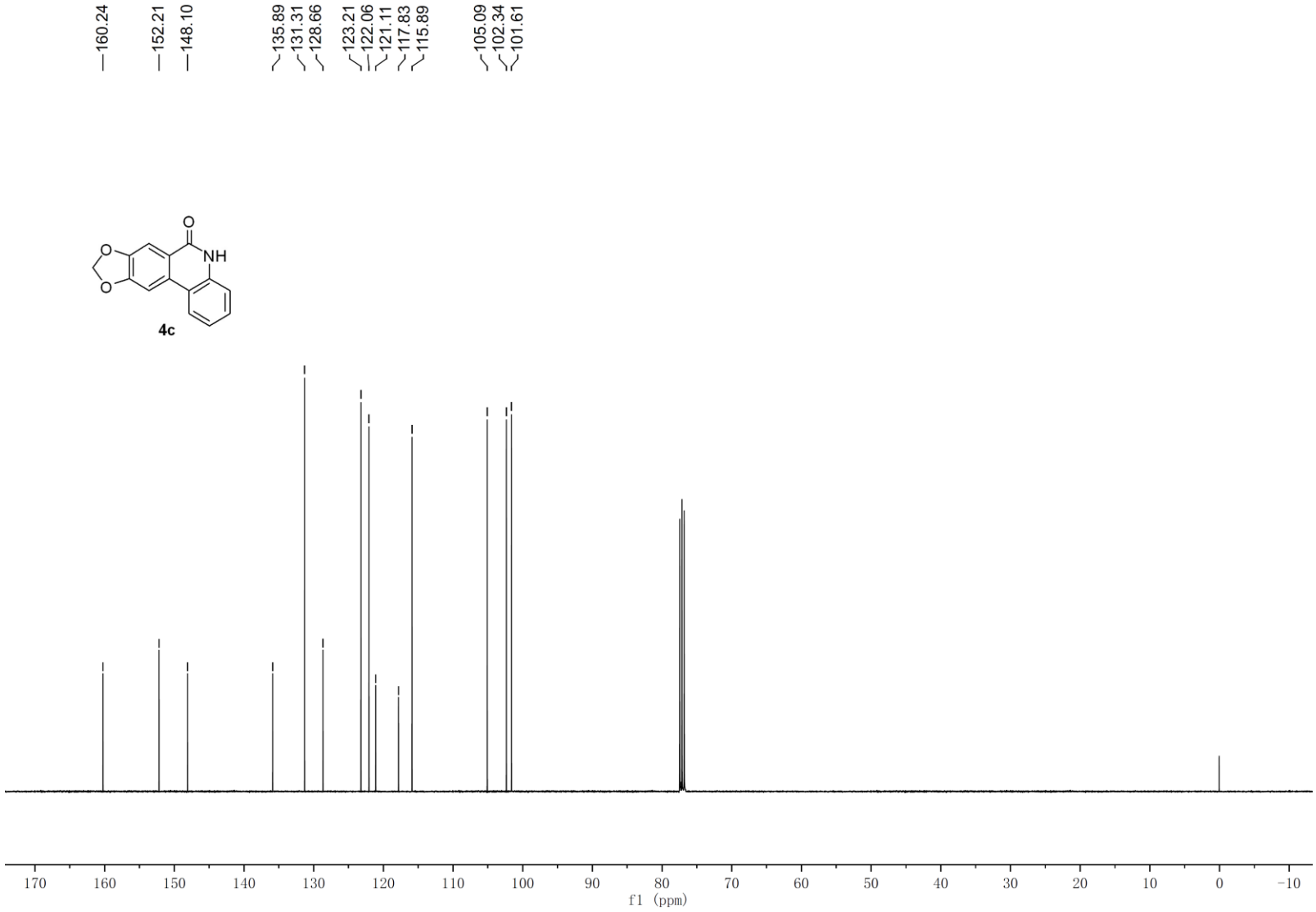
8.16
8.14
8.13
7.87
7.86
7.85
7.85
7.85
7.84
7.84
7.54
7.54
7.53
7.52
7.51
6.82
6.82
6.81
6.80
6.72
6.72
O
O
OH
4b
0.81
1.03
2.00
0.99
1.02
1.01
0.94
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


161.15
160.41
152.67
135.83
135.65
130.22
128.20
125.38
122.19
119.50
113.72
109.93
103.49
O
O
OH
4b
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)


11.61
8.28
8.27
8.02
7.62
7.42
7.41
7.40
7.33
7.31
7.22
7.21
7.20
6.21
O
NH
O
O
4c
0.72
1.00
1.01
1.05
1.00
1.00
1.01
2.02
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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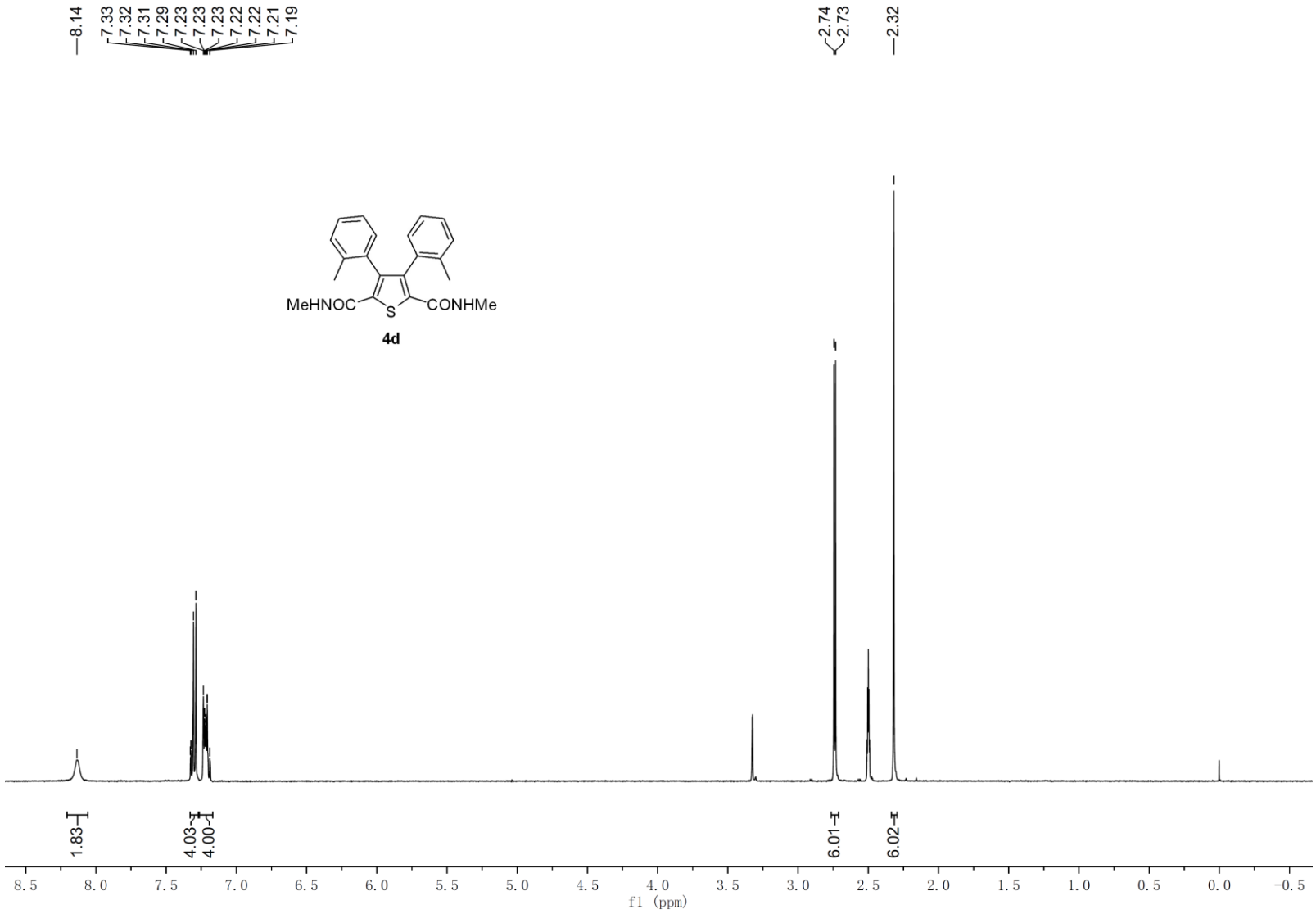
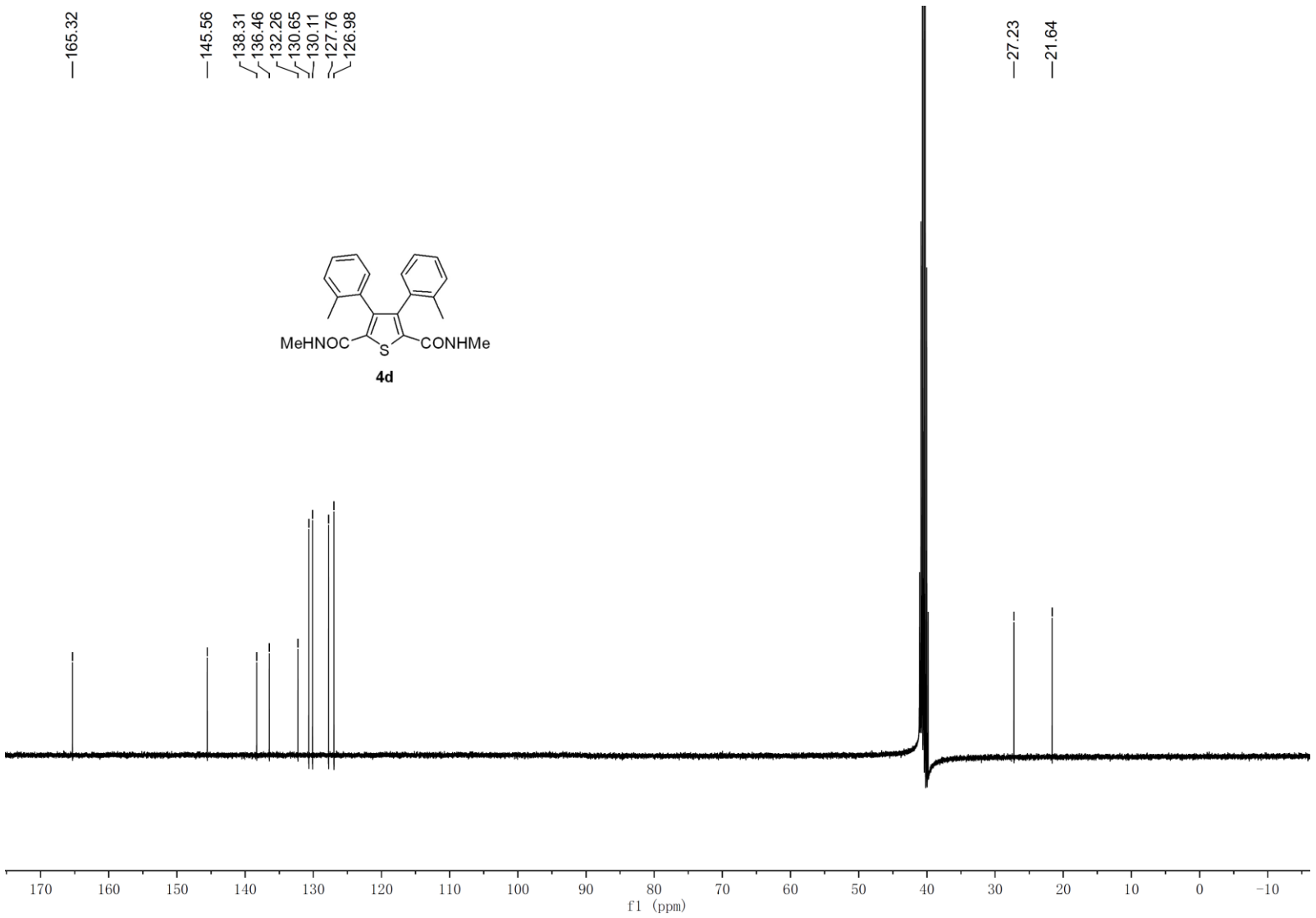
152.21
148.10
135.89
131.31
128.66
123.21
122.06
121.11
117.83
115.89
105.09
102.34
101.61
O
NH
O
O
4c
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)


8.14
7.33
7.32
7.31
7.29
7.23
7.23
7.23
7.22
7.22
7.21
7.19
2.74
2.73
2.32
MeHNOC
S
CONHMe
4d
1.83
4.03
4.00
6.01
6.02
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


165.32
145.56
138.31
136.46
132.26
130.65
130.11
127.76
126.98
27.23
21.64
MeHNOC
S
CONHMe
4d
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)