

An Efficient Synthesis of Isoquinolines via Rhodium-Catalyzed Direct C-H Functionalization of Arylhydrazines

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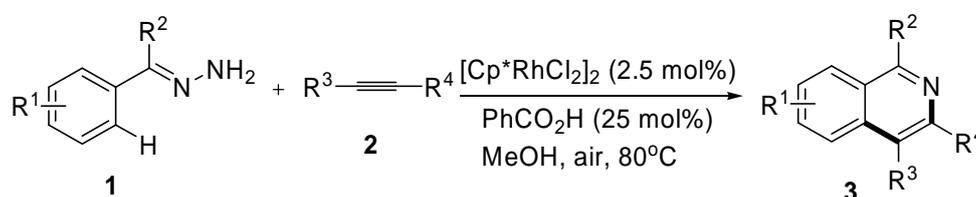
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General Information:

All of the reactions were carried out in a flame-dried tube. Solvents were dried prior to use. For column chromatography, 200-300 mesh silica gel was used. ^1H NMR were recorded on Bruker 400 MHz spectrometer and ^{13}C NMR were recorded on Bruker 100 MHz or 125 MHz spectrometer in CDCl_3 . HRMS were performed on Agilent 6540 Q-TOF mass spectrometer. Melting points were determined on a SGW X-4B melting point apparatus. The arylhydrazines were known compounds and prepared according to the literature procedures ¹. 1,2-diphenylethyne and oct-4-yne were commercial available; the other alkynes were known products and prepared according to the literature procedures ².

General procedure for synthesis of isoquinolines via Rhodium-Catalyzed direct C-H functionalization of arylhydrazines for table 2



To a 10 mL schlenk tube was added $[\text{Cp}^*\text{RhCl}_2]_2$ (15.4 mg, 0.025 mmol), arylhydrazine **1** (1 mmol), alkyne **2** (1.2 mmol), PhCO_2H (30.5 mg, 0.25 mmol) and MeOH (3 mL) under air, the tube was sealed and heated to 80°C for 2h. The reaction solution was cooled and concentrated; the crude residue was purified by column chromatography (silica gel; EtOAc: Petroleum ether= 1: 50 ~ 1: 10) to give the title compound **3**.

1,3,4-triphenylisoquinoline (3a) ³

The title compound was obtained as a white solid (314 mg, Yield: 88%) via the general procedure. ^1H NMR (400 MHz, CDCl_3) δ 8.18 (d, $J = 8.3$ Hz, 1H), 7.86 – 7.79 (m, 2H), 7.72 (d, $J = 8.3$ Hz, 1H), 7.62 – 7.46 (m, 5H), 7.45 – 7.34 (m, 5H), 7.32 – 7.27 (m, 2H), 7.22 – 7.12 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 159.85, 149.69, 140.94, 139.87, 137.60, 137.02, 131.39, 130.49, 130.27, 129.98, 129.81, 128.57, 128.36, 128.34, 127.58, 127.55, 127.33, 127.02, 126.62, 126.07, 125.48.

1-phenyl-3,4-dip-tolylisoquinoline (3b) ⁴

The title compound was obtained as a white solid (354 mg, Yield: 92%) via the general procedure. ^1H NMR (400 MHz, CDCl_3) δ 8.16 (d, $J = 8.1$ Hz, 1H), 7.85 – 7.77 (m, 2H), 7.71 (d, $J = 8.1$ Hz, 1H), 7.60 – 7.44 (m, 5H), 7.34 (d, $J = 8.0$ Hz, 2H), 7.23 – 7.15 (m, 4H), 7.00 (d, $J = 8.0$ Hz, 2H), 2.41 (s, 3H), 2.27 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 159.53, 149.62, 139.99, 138.17, 137.27, 136.86, 136.60, 134.68, 131.20, 130.38, 130.30, 129.77, 129.50, 129.13, 128.48, 128.34, 128.28, 127.46, 126.37, 126.11, 125.35, 21.38, 21.22.

3,4-bis(4-chlorophenyl)-1-phenylisoquinoline (3c) ³

The title compound was obtained as a white solid (349 mg, Yield: 82%) via the general procedure. ^1H NMR (400 MHz, CDCl_3) δ 8.19 (d, $J = 8.3$ Hz, 1H), 7.82 – 7.76 (m, 2H), 7.71 – 7.59 (m, 2H), 7.59 – 7.48 (m, 4H), 7.40 (d, $J = 8.3$ Hz, 2H), 7.35 (d, $J = 8.5$ Hz, 2H), 7.23 (d, $J = 8.3$ Hz, 2H),

7.19 (d, $J = 8.5$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 160.37, 148.50, 139.52, 139.11, 136.79, 135.80, 133.68, 133.37, 132.63, 131.77, 130.37, 130.18, 128.90, 128.78, 128.63, 128.41, 127.99, 127.74, 127.00, 125.68, 125.57.

3,4-bis(4-fluorophenyl)-1-phenylisoquinoline (3d)⁵

The title compound was obtained as a white solid (306 mg, Yield: 78%) via the general procedure. ^1H NMR (400 MHz, CDCl_3) δ 8.19 (d, $J = 8.3$ Hz, 1H), 7.80 (d, $J = 7.1$ Hz, 2H), 7.72 – 7.60 (m, 2H), 7.60 – 7.46 (m, 4H), 7.44 – 7.33 (m, 2H), 7.25 (t, $J = 6.5$ Hz, 2H), 7.11 (t, $J = 8.4$ Hz, 2H), 6.90 (t, $J = 8.4$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 163.40 (d, $J = 11.3$ Hz), 160.94 (d, $J = 11.3$ Hz), 160.12, 153.80, 148.73, 140.64, 139.39, 137.06, 133.21, 132.93 (d, $J = 8.0$ Hz), 132.19 (d, $J = 8.1$ Hz), 132.15, 130.40, 130.22, 128.42, 127.77, 126.91, 125.75, 125.54, 115.66 (d, $J = 21.5$ Hz), 114.69 (d, $J = 21.4$ Hz).

1-phenyl-3,4-dim-tolyisoquinoline (3e)

The title compound was obtained as a white solid (346 mg, Yield: 90%) via the general procedure, Mp: 159-160 °C. ^1H NMR (400 MHz, CDCl_3) δ 8.15 (d, $J = 8.3$ Hz, 1H), 7.81 (d, $J = 7.0$ Hz, 2H), 7.72 (d, $J = 8.3$ Hz, 1H), 7.53 (dd, $J = 14.2, 7.1$ Hz, 3H), 7.49 – 7.41 (m, 2H), 7.35 (s, 1H), 7.25 (t, $J = 7.5$ Hz, 1H), 7.14 (dd, $J = 7.9, 3.2$ Hz, 3H), 7.07 (d, $J = 7.5$ Hz, 1H), 7.02 (t, $J = 7.6$ Hz, 1H), 6.95 (d, $J = 7.5$ Hz, 1H), 2.32 (s, 3H), 2.23 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 159.68, 149.73, 140.86, 139.99, 137.83, 137.62, 137.14, 137.06, 132.03, 131.30, 130.34, 129.96, 129.89, 128.56, 128.49, 128.37, 128.24, 128.03, 127.82, 127.61, 127.51, 127.31, 126.53, 126.23, 125.46, 21.54, 21.53. HRMS (ESI) calcd. for $\text{C}_{29}\text{H}_{24}\text{N}$ $[\text{M}+\text{H}]^+$: 386.1903, found: 386.1897.

1-phenyl-3,4-dipropylisoquinoline (3f)³

The title compound was obtained as a white solid (260 mg, Yield: 90%) via the general procedure. ^1H NMR (400 MHz, CDCl_3) δ 8.02 (dd, $J = 8.2, 5.7$ Hz, 2H), 7.70 – 7.61 (m, 3H), 7.54 – 7.37 (m, 4H), 3.11 – 2.94 (m, 4H), 1.85 (dd, $J = 15.4, 7.6$ Hz, 2H), 1.74 (dd, $J = 15.4, 7.6$ Hz, 2H), 1.14 (t, $J = 7.3$ Hz, 3H), 1.05 (t, $J = 7.3$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 158.12, 152.28, 140.19, 136.14, 130.07, 129.46, 128.28, 128.18, 128.10, 127.17, 125.33, 125.27, 123.35, 37.45, 30.01, 24.18, 23.70, 14.72, 14.39.

4-methyl-1,3-diphenylisoquinoline (3g)³

The title compound was obtained as a white solid (250 mg, Yield: 85%) via the general procedure. ^1H NMR (400 MHz, CDCl_3) δ 8.13 (d, $J = 8.5$ Hz, 2H), 7.78 – 7.69 (m, 3H), 7.67 – 7.59 (m, 2H), 7.56 – 7.42 (m, 6H), 7.38 (t, $J = 7.4$ Hz, 1H), 2.70 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 158.28, 151.12, 141.56, 139.94, 137.11, 130.23, 130.14, 129.94, 128.32, 128.25, 128.11, 128.06, 127.50, 126.35, 125.44, 123.95, 123.16, 15.74.

(1,3-diphenylisoquinolin-4-yl)methanol (3h)³

The title compound was obtained as a yellow solid (270 mg, Yield: 87%) via the general procedure. ^1H NMR (400 MHz, CDCl_3) δ 8.35 (d, $J = 8.5$ Hz, 1H), 8.14 (d, $J = 8.5$ Hz, 1H), 7.87 – 7.65 (m, 5H), 7.63 – 7.36 (m, 7H), 5.11 (s, 2H), 1.97 (brs, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 160.64, 152.17, 140.48, 139.61, 136.80, 130.65, 130.18, 129.94, 128.65, 128.31, 128.26, 128.23, 128.09, 126.76, 125.98, 124.73, 124.20, 59.59.

1,4-diphenyl-3-(thiophen-3-yl)isoquinoline and 1,3-diphenyl-4-(thiophen-3-yl)isoquinoline (3i and 3i')⁵

The specific structure of **3i** and **3i'** were not determined. The title compound **3i** was obtained as a white solid (152 mg, Yield: 42%) via the general procedure. ¹H NMR (400 MHz, CDCl₃) δ 8.16 (d, *J* = 8.3 Hz, 1H), 7.83 (d, *J* = 6.8 Hz, 2H), 7.62 – 7.44 (m, 9H), 7.37 (dd, *J* = 7.4, 1.6 Hz, 2H), 7.21 (d, *J* = 2.0 Hz, 1H), 7.17 (d, *J* = 5.0 Hz, 1H), 7.09 (dd, *J* = 5.0, 3.0 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 159.63, 144.40, 142.06, 139.84, 138.05, 137.43, 130.87, 130.29, 129.92, 129.41, 128.95, 128.69, 128.61, 128.31, 127.87, 127.46, 126.45, 126.08, 126.02, 125.21, 123.89. The other title compound **3i'** was isolated as a white solid (152 mg, Yield: 42%). ¹H NMR (400 MHz, CDCl₃) δ 8.17 (d, *J* = 8.4 Hz, 1H), 7.83 (dd, *J* = 21.1, 7.7 Hz, 3H), 7.63 (t, *J* = 7.3 Hz, 1H), 7.59 – 7.43 (m, 6H), 7.39 (dd, *J* = 4.8, 3.0 Hz, 1H), 7.27 – 7.18 (m, 3H), 7.15 (d, *J* = 1.9 Hz, 1H), 7.05 (d, *J* = 4.9 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 159.90, 150.31, 141.03, 139.81, 137.31, 137.28, 130.55, 130.25, 130.11, 130.09, 128.59, 128.34, 127.67, 127.59, 127.17, 126.71, 125.89, 125.53, 125.45, 124.91.

6-fluoro-1-(4-fluorophenyl)-3,4-diphenylisoquinoline (3j)⁶

The title compound was obtained as a white solid (298 mg, Yield: 76%) via the general procedure. ¹H NMR (400 MHz, CDCl₃) δ 8.15 (dd, *J* = 8.8, 5.9 Hz, 1H), 7.88 – 7.70 (m, 2H), 7.48 – 7.35 (m, 5H), 7.35 – 7.09 (m, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 164.50, 161.98, 158.56, 150.63, 140.48, 139.11 (d, *J* = 9.8 Hz), 137.05, 135.67 (d, *J* = 3.4 Hz), 131.99 (d, *J* = 8.3 Hz), 131.16, 130.54, 130.45, 130.38, 128.60, 127.67, 127.34, 122.73, 117.04 (d, *J* = 25.3 Hz), 115.48 (d, *J* = 21.6 Hz), 109.80 (d, *J* = 22.5 Hz), 99.99.

6-fluoro-1-(4-fluorophenyl)-3,4-dip-tolylisoquinoline (3k)⁵

The title compound was obtained as a white solid (336 mg, Yield: 80%) via the general procedure. ¹H NMR (400 MHz, CDCl₃) δ 8.13 (dd, *J* = 9.2, 5.8 Hz, 1H), 7.82 – 7.74 (m, 2H), 7.35 – 7.29 (m, 3H), 7.28 – 7.18 (m, 5H), 7.15 (d, *J* = 8.0 Hz, 2H), 7.00 (d, *J* = 8.0 Hz, 2H), 2.42 (s, 3H), 2.28 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 164.39, 161.89, 158.26, 150.54, 139.36 (d, *J* = 9.7 Hz), 137.72, 137.24, 137.00, 135.79 (d, *J* = 3.2 Hz), 134.13, 132.05, 131.96, 130.96, 130.38 (d, *J* = 9.6 Hz), 130.29, 129.36, 128.42, 122.59, 116.78 (d, *J* = 25.2 Hz), 115.41 (d, *J* = 21.6 Hz), 109.81 (d, *J* = 22.2 Hz), 21.37, 21.22.

3,4-bis(4-chlorophenyl)-6-fluoro-1-(4-fluorophenyl)isoquinoline (3l)

The title compound was obtained as a white solid (332 mg, Yield: 72%) via the general procedure, Mp: 124-126 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.16 (dd, *J* = 8.9, 5.7 Hz, 1H), 7.82 – 7.71 (m, 2H), 7.42 (d, *J* = 8.1 Hz, 2H), 7.37 – 7.24 (m, 6H), 7.24 – 7.15 (m, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 164.37 (d, *J* = 10.2 Hz), 162.38 (d, *J* = 6.2 Hz), 159.06, 149.48, 138.88 (d, *J* = 9.9 Hz), 138.64, 135.32 (d, *J* = 3.4 Hz), 135.23, 134.03, 133.71, 132.40, 131.92 (d, *J* = 8.2 Hz), 131.66, 130.71 (d, *J* = 9.4 Hz), 129.14, 128.41 (d, *J* = 5.4 Hz), 128.08, 122.77, 117.40 (d, *J* = 25.2 Hz), 115.56 (d, *J* = 21.6 Hz), 109.51 (d, *J* = 22.5 Hz). HRMS (ESI) calcd. for C₂₇H₁₆Cl₂F₂N [M+H]⁺: 430.1213, found: 430.1217.

6-fluoro-1,3,4-tris(4-fluorophenyl)isoquinoline (3m)

The title compound was obtained as a white solid (300 mg, Yield: 70%) via the general procedure, Mp: 156-158 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.16 (dd, *J* = 9.0, 5.8 Hz, 1H), 7.82 – 7.69 (m, 2H), 7.42 – 7.33 (m, 2H), 7.33 – 7.18 (m, 6H), 7.13 (t, *J* = 8.7 Hz, 2H), 6.91 (t, *J* = 8.7 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 164.34 (d, *J* = 8.4 Hz), 163.26 (d, *J* = 15.0 Hz), 162.34 (d, *J* = 4.6 Hz), 161.29 (d, *J* = 14.8 Hz), 158.87, 149.84, 139.10 (d, *J* = 9.9 Hz), 136.35 (d, *J* = 3.3 Hz), 135.42 (d, *J* = 3.3 Hz), 132.79, 132.74 (d, *J* = 7.9 Hz), 132.08 (d, *J* = 8.2 Hz), 131.92 (d, *J* = 8.3 Hz), 130.67 (d, *J* = 9.5 Hz), 128.50 (d, *J* = 5.1 Hz), 122.73, 117.23 (d, *J* = 25.2 Hz), 115.91 (d, *J* = 21.6 Hz), 115.54 (d, *J* = 21.6 Hz), 114.77 (d, *J* = 21.5 Hz), 109.53 (d, *J* = 22.5 Hz). HRMS (ESI) calcd. for C₂₇H₁₆F₄N [M+H]⁺: 386.1903, found: 386.1897.

6-methyl-3,4-diphenyl-1-p-tolylisoquinoline (3n)⁶

The title compound was obtained as a white solid (342 mg, Yield: 89%) via the general procedure. ¹H NMR (400 MHz, CDCl₃) δ 8.09 (d, *J* = 8.6 Hz, 1H), 7.71 (d, *J* = 8.0 Hz, 2H), 7.46 (s, 1H), 7.43 – 7.24 (m, 10H), 7.19 – 7.10 (m, 3H), 2.45 (s, 3H), 2.41 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 159.55, 149.82, 141.18, 140.23, 138.34, 137.86, 137.25, 137.16, 131.46, 130.51, 130.18, 129.10, 129.00, 128.73, 128.31, 127.53, 127.49, 127.19, 126.88, 124.86, 123.90, 22.17, 21.41.

6-methyl-1,3,4-trip-tolylisoquinoline (3o)

The title compound was obtained as a white solid (376 mg, Yield: 91%) via the general procedure, Mp: 183-184 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.07 (d, *J* = 8.6 Hz, 1H), 7.70 (d, *J* = 7.9 Hz, 2H), 7.46 (s, 1H), 7.37 – 7.25 (m, 5H), 7.18 (q, *J* = 8.1 Hz, 4H), 6.98 (d, *J* = 7.9 Hz, 2H), 2.45 (s, 3H), 2.41 (s, 6H), 2.26 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 159.25, 149.75, 139.99, 138.43, 138.23, 137.52, 137.31, 136.68, 136.43, 134.93, 131.27, 130.41, 130.23, 129.11, 128.96, 128.80, 128.51, 128.26, 127.46, 124.90, 123.78, 22.16, 21.42, 21.23. HRMS (ESI) calcd. for C₃₁H₂₈N [M+H]⁺: 414.2216, found: 414.2230.

3,4-bis(4-chlorophenyl)-6-methyl-1-p-tolylisoquinoline (3p)

The title compound was obtained as a white solid (372 mg, Yield: 82%) via the general procedure, Mp: 188-190 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.10 (d, *J* = 8.3 Hz, 1H), 7.68 (d, *J* = 7.6 Hz, 2H), 7.44 – 7.29 (m, 8H), 7.19 (dd, *J* = 14.3, 8.3 Hz, 4H), 2.47 (s, 3H), 2.45 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 160.04, 148.58, 140.72, 139.33, 138.60, 137.03, 136.80, 136.06, 133.48, 133.19, 132.67, 131.77, 130.07, 129.05, 128.84, 127.88, 127.67, 124.49, 123.96, 22.16, 21.38. HRMS (ESI) calcd. for C₂₉H₂₂Cl₂N [M+H]⁺: 454.1124, found: 454.1128.

3,4-bis(4-fluorophenyl)-6-methyl-1-p-tolylisoquinoline (3q)

The title compound was obtained as a white solid (349 mg, Yield: 83%) via the general procedure, Mp: 184-186 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.09 (d, *J* = 8.6 Hz, 1H), 7.69 (d, *J* = 8.0 Hz, 2H), 7.40 (s, 1H), 7.38 – 7.31 (m, 5H), 7.27 – 7.19 (m, 2H), 7.15 – 7.06 (m, 2H), 6.92 – 6.84 (m, 2H), 2.46 (s, 3H), 2.44 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 159.86, 148.99, 146.61 (d, *J* = 5.8 Hz), 140.58, 138.53, 137.27, 137.07 (d, *J* = 1.4 Hz), 136.92, 133.58 (d, *J* = 3.3 Hz), 133.00, 132.92, 132.15 (d, *J* = 8.1 Hz), 130.09, 129.06, 128.92, 127.99, 127.66, 124.56, 123.93, 115.59 (d, *J* = 21.3 Hz), 114.56 (d, *J* = 21.3 Hz), 22.18, 21.40. HRMS (ESI) calcd. for C₂₉H₂₂F₂N [M+H]⁺: 422.1715, found: 422.1719.

1-(4-chlorophenyl)-3,4-diphenylisoquinoline (3r)⁵

The title compound was obtained as a white solid (176 mg, Yield: 45%) via the general procedure. ¹H NMR (400 MHz, CDCl₃) δ 8.13 (d, *J* = 8.1 Hz, 1H), 7.75 (dd, *J* = 17.0, 8.1 Hz, 3H), 7.61 (t, *J* = 7.4 Hz, 1H), 7.53 (d, *J* = 7.4 Hz, 3H), 7.46 – 7.34 (m, 5H), 7.29 (d, *J* = 5.9 Hz, 2H), 7.23 – 7.11 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 158.51, 149.76, 140.75, 138.28, 137.42, 137.06, 134.75, 131.62, 131.34, 130.43, 130.11, 128.60, 128.39, 127.62, 127.41, 127.13, 127.09, 126.84, 126.22, 125.32.

6-chloro-1,3,4-triphenylisoquinoline (3r')⁵

The title compound was obtained as a white solid (176 mg, Yield: 45%) via the general procedure. ¹H NMR (400 MHz, CDCl₃) δ 8.13 (d, *J* = 9.0 Hz, 1H), 7.80 (d, *J* = 7.7 Hz, 2H), 7.69 (s, 1H), 7.59 – 7.49 (m, 3H), 7.48 – 7.36 (m, 6H), 7.28 (d, *J* = 7.7 Hz, 2H), 7.19 (d, *J* = 4.9 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 159.76, 150.84, 140.54, 139.40, 138.05, 136.88, 136.52, 131.26, 130.40, 130.17, 129.39, 128.98, 128.81, 128.56, 128.44, 127.53, 127.26, 124.90, 123.71.

1,6-dimethyl-3,4-diphenylisoquinoline (3s)⁷

The title compound was obtained as a white solid (290 mg, Yield: 91%) via the general procedure. ¹H NMR (400 MHz, CDCl₃) δ 8.09 (d, *J* = 8.8 Hz, 1H), 7.41 (d, *J* = 8.1 Hz, 2H), 7.38 – 7.28 (m, 5H), 7.24 – 7.12 (m, 5H), 3.04 (s, 3H), 2.43 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 157.41, 149.63, 141.22, 140.23, 137.80, 136.25, 131.48, 130.28, 128.76, 128.72, 128.18, 127.59, 127.04, 126.85, 125.49, 125.12, 124.58, 22.72, 22.16.

1-benzyl-3,4-diphenylisoquinoline (3t)⁸

The title compound was obtained as a white solid (334 mg, Yield: 90%) via the general procedure. ¹H NMR (400 MHz, CDCl₃) δ 8.22 (d, *J* = 7.8 Hz, 1H), 7.65 (d, *J* = 8.2 Hz, 1H), 7.57 – 7.46 (m, 2H), 7.42 (t, *J* = 6.0 Hz, 4H), 7.38 – 7.26 (m, 5H), 7.26 – 7.15 (m, 6H), 4.79 (s, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 159.20, 149.41, 140.99, 139.73, 137.59, 136.74, 131.39, 130.41, 129.79, 129.65, 128.76, 128.50, 128.27, 127.62, 127.22, 127.02, 126.65, 126.42, 126.25, 125.78, 42.47.

2,3-diphenyl-8,9-dihydro-7H-benz[de]quinoline (3u)⁷

The title compound was obtained as a white solid (263 mg, Yield: 82%) via the general procedure. ¹H NMR (400 MHz, CDCl₃) δ 7.48 (t, *J* = 4.3 Hz, 2H), 7.37 – 7.28 (m, 6H), 7.24 – 7.12 (m, 5H), 3.40 (t, *J* = 6.2 Hz, 2H), 3.20 (t, *J* = 6.2 Hz, 2H), 2.34 – 2.21 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 159.31, 153.30, 149.29, 138.62, 137.77, 136.31, 131.39, 130.31, 130.13, 129.16, 128.19, 127.64, 127.08, 126.96, 124.84, 123.91, 123.61, 34.69, 30.76, 23.43.

7-methyl-4,5-dip-tolylthieno[2,3-c]pyridine (3v)

The title compound was obtained as a white solid (253 mg, Yield: 77%) via the general procedure, Mp: 138-140 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.57 (d, *J* = 5.4 Hz, 1H), 7.26 (d, *J* = 7.5 Hz, 2H), 7.20 (d, *J* = 5.4 Hz, 1H), 7.12 (s, 4H), 7.01 (d, *J* = 7.5 Hz, 2H), 2.88 (s, 3H), 2.36 (s, 3H), 2.29 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 151.12, 150.94, 145.92, 137.72, 136.70, 136.63, 135.44, 133.98, 130.63, 130.39, 130.18, 129.02, 128.47, 128.06, 124.40, 23.64, 21.29, 21.21. HRMS (ESI) calcd. for C₂₂H₂₀NS [M+H]⁺: 330.1311, found: 330.1307.

1,7-dimethyl-3,4-diphenylisoquinoline (3w) ⁷

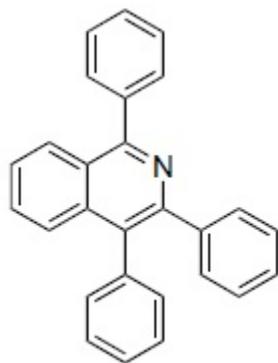
The title compound was obtained as a white solid (268 mg, Yield: 87%) via the general procedure. ¹H NMR (400 MHz, CDCl₃) δ 7.95 (s, 1H), 7.55 (d, *J* = 8.5 Hz, 1H), 7.42 (d, *J* = 8.5 Hz, 1H), 7.38 – 7.27 (m, 5H), 7.19 (dd, *J* = 15.6, 7.1 Hz, 5H), 3.05 (s, 3H), 2.57 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 157.05, 148.62, 141.05, 137.77, 136.45, 134.22, 132.13, 131.42, 130.30, 129.12, 128.19, 127.61, 127.08, 126.85, 126.36, 126.14, 124.55, 22.76, 21.92.

Dimethyl 2-(2-(dip-tolylmethylene)hydrazono)succinate (3x)

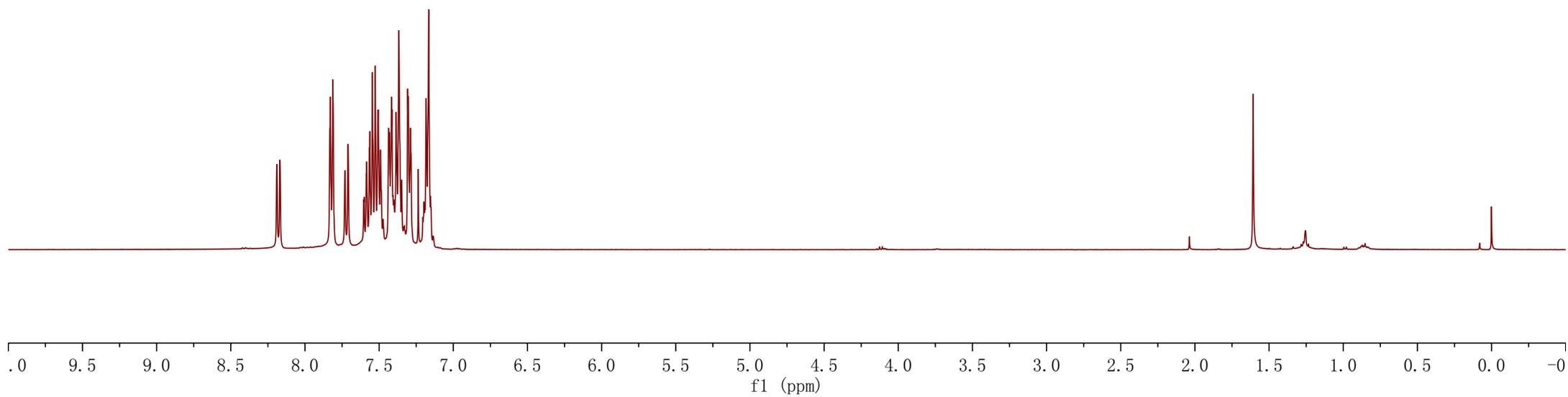
The title compound was obtained as colorless sticky oil (311 mg, Yield: 85%) via the general procedure. ¹H NMR (400 MHz, CDCl₃) δ 7.55 (d, *J* = 8.1 Hz, 2H), 7.24 – 7.17 (m, 6H), 3.85 (s, 2H), 3.83 (s, 3H), 3.63 (s, 3H), 2.40 (s, 3H), 2.39 (s, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 168.93, 164.58, 161.64, 148.88, 140.92, 140.06, 134.93, 131.23, 130.45, 129.50, 128.94, 128.48, 52.95, 52.24, 34.04, 21.51, 21.46. HRMS (ESI) calcd. for C₂₁H₂₃N₂O₄ [M+H]⁺: 367.1652, found: 367.1654.

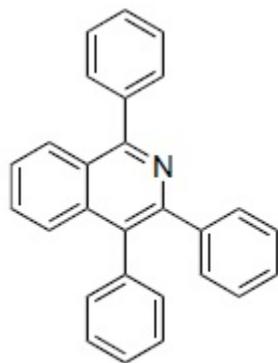
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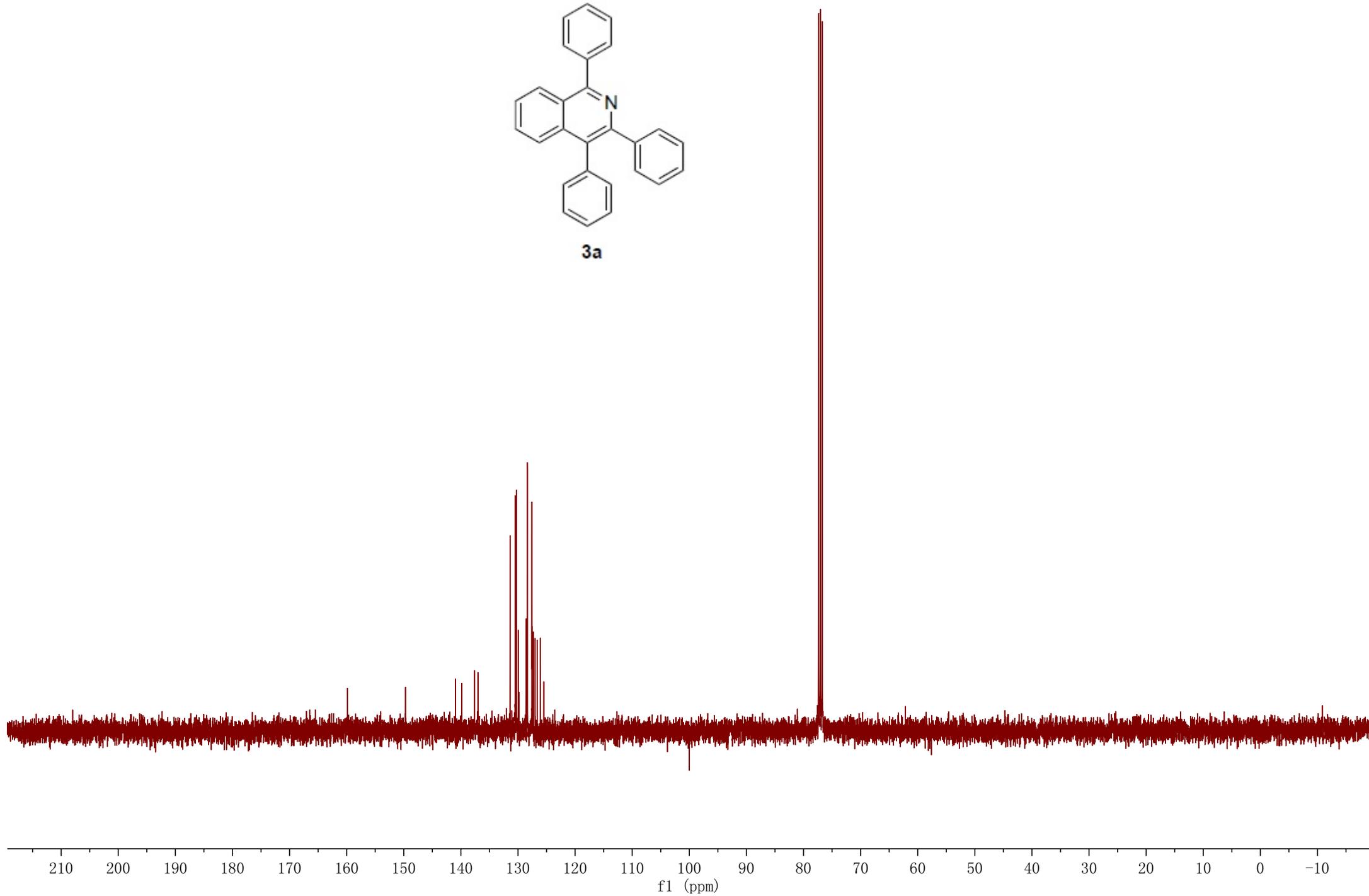


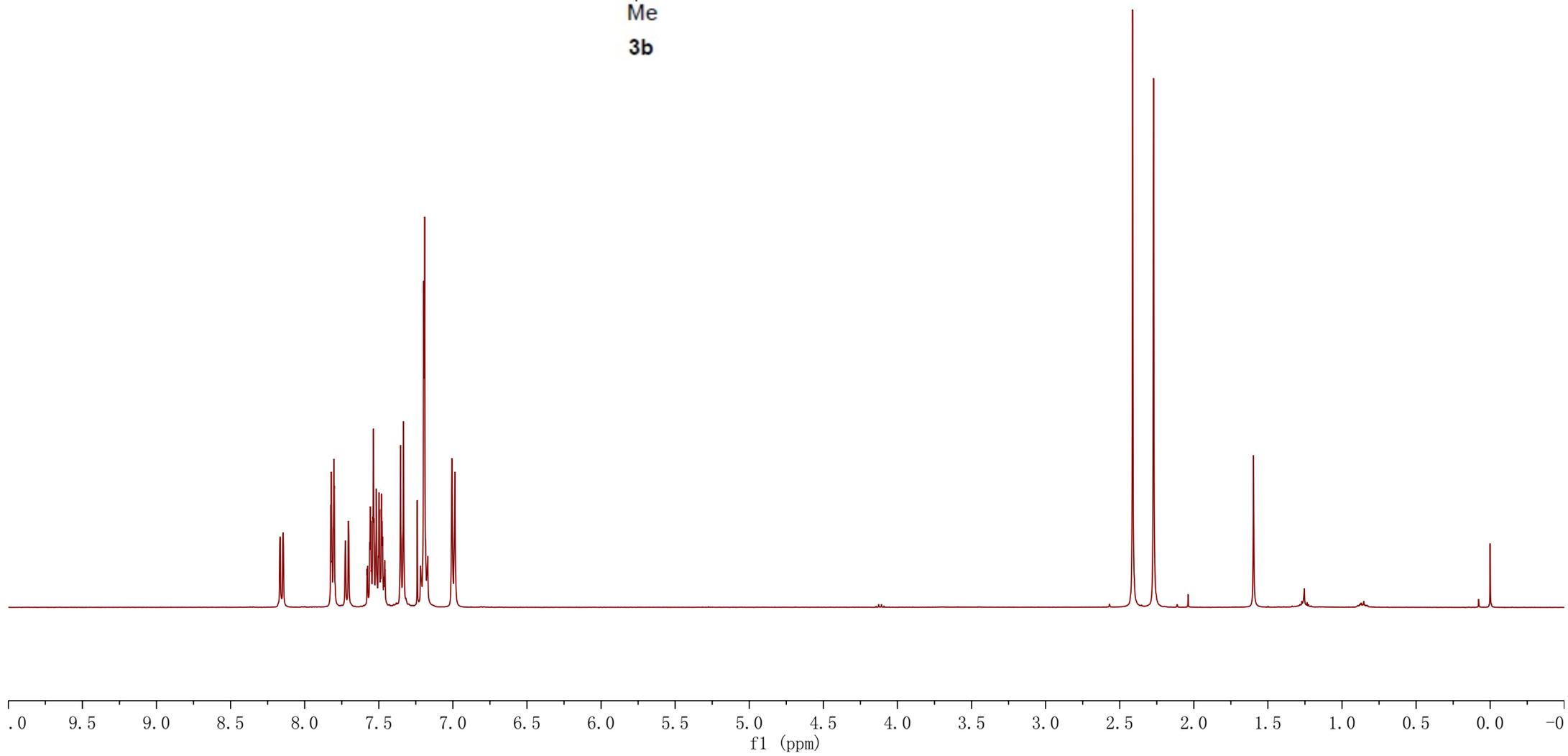
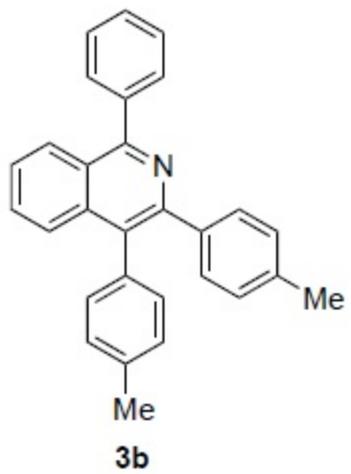
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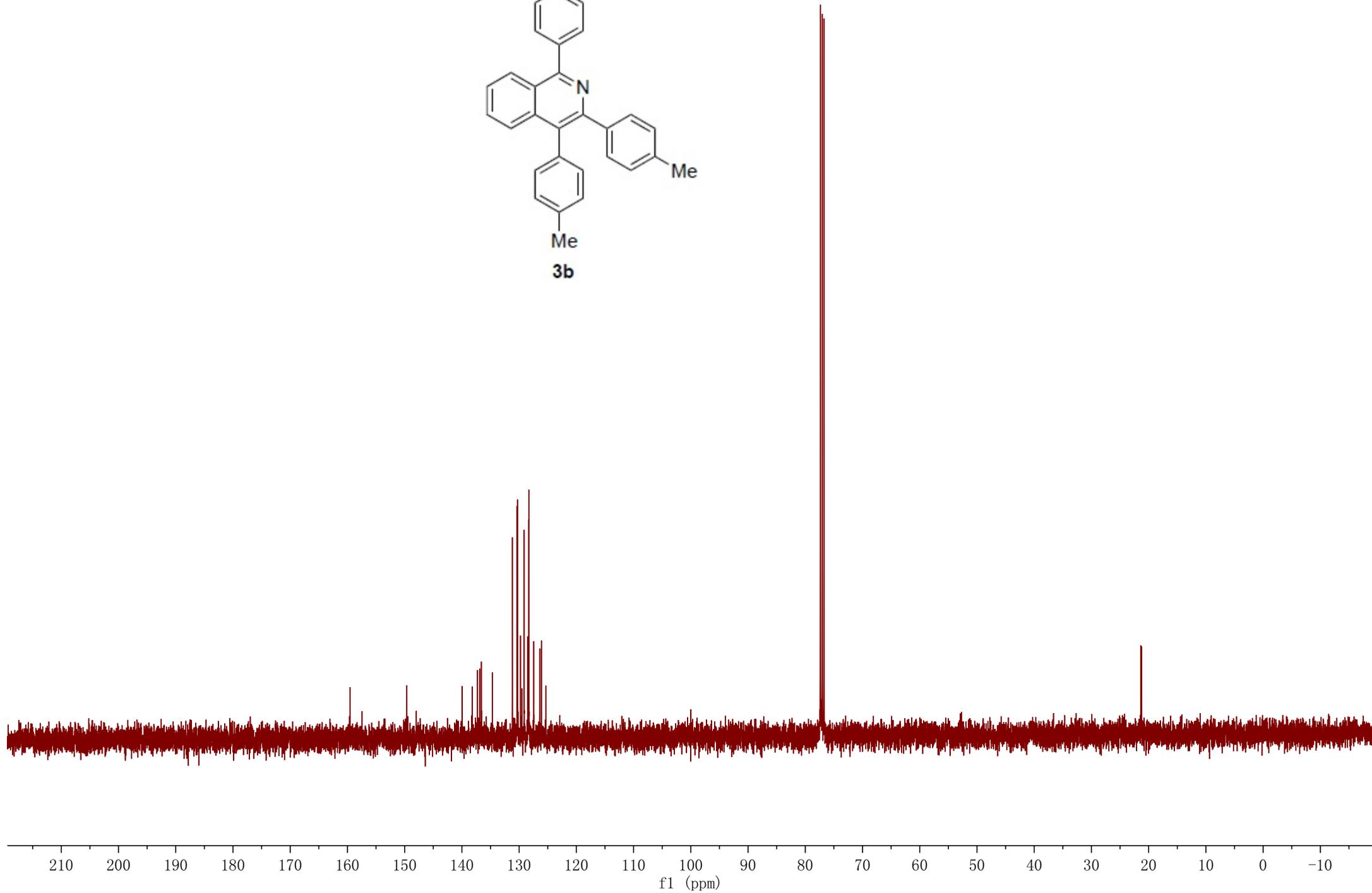
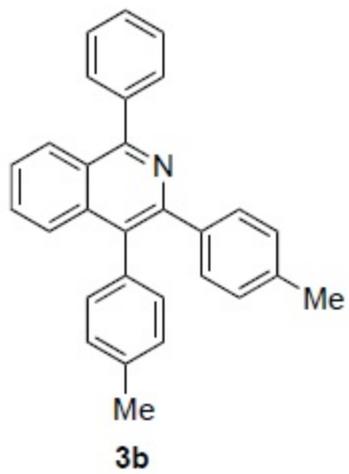


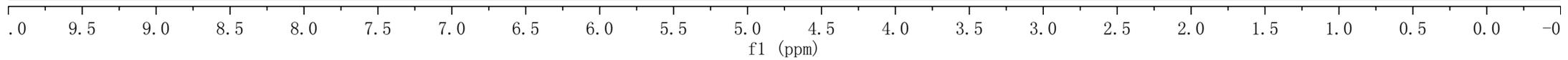
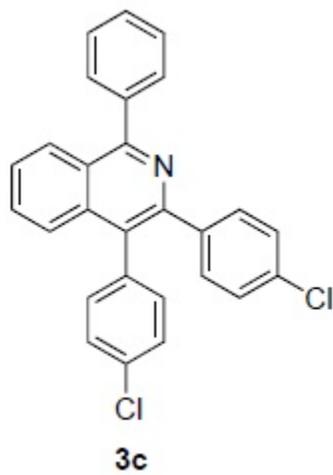


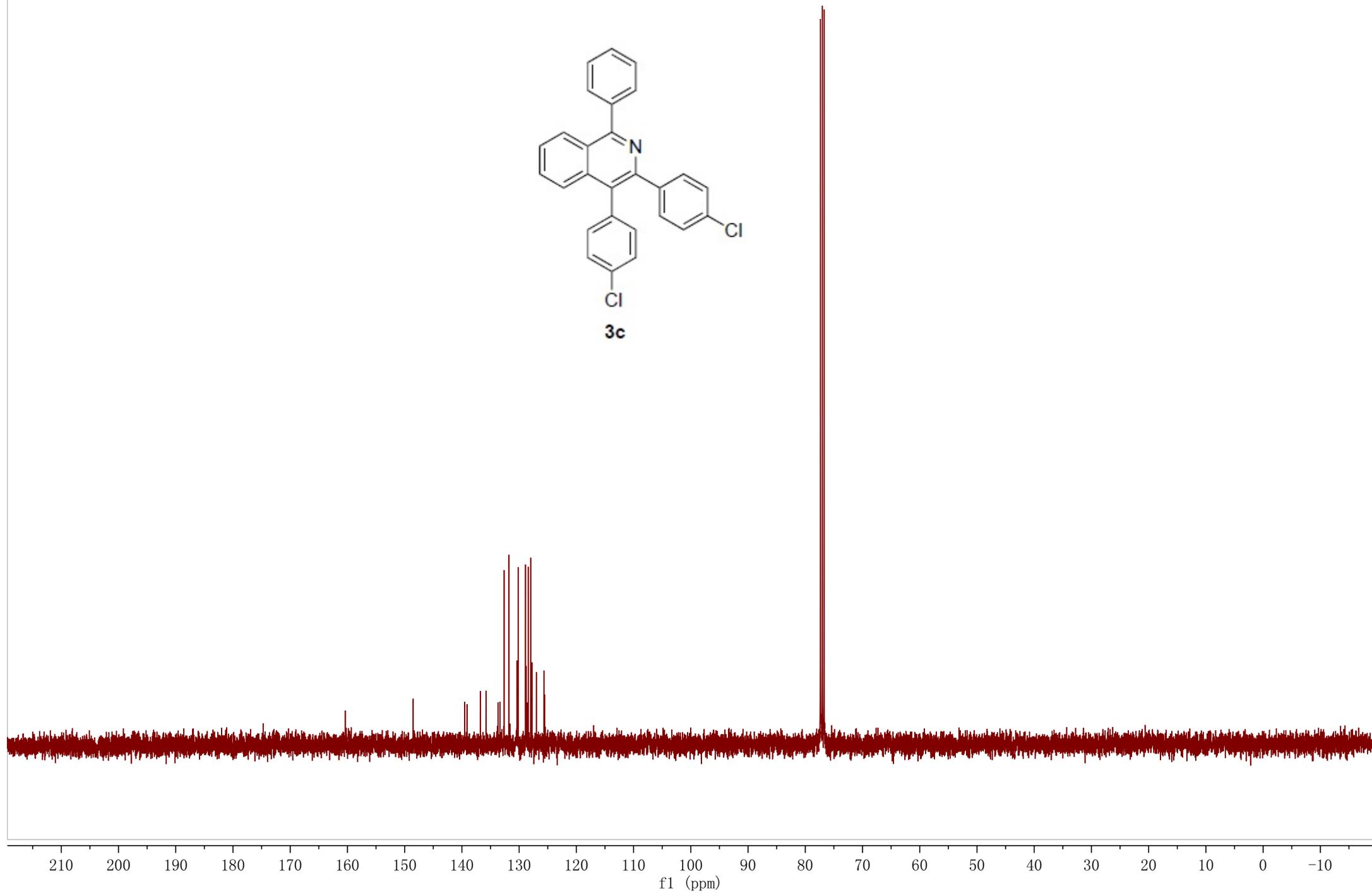
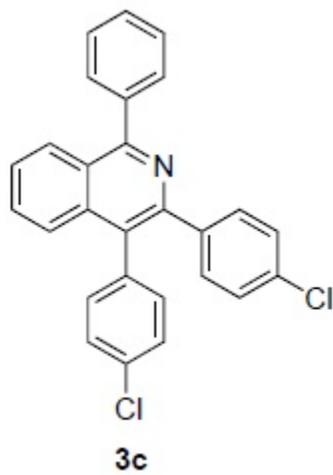
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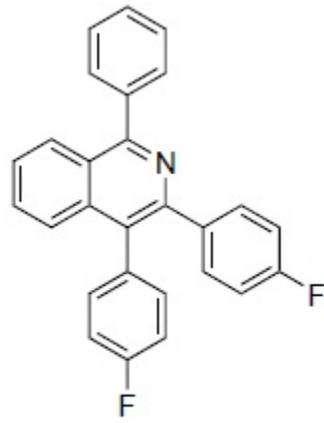




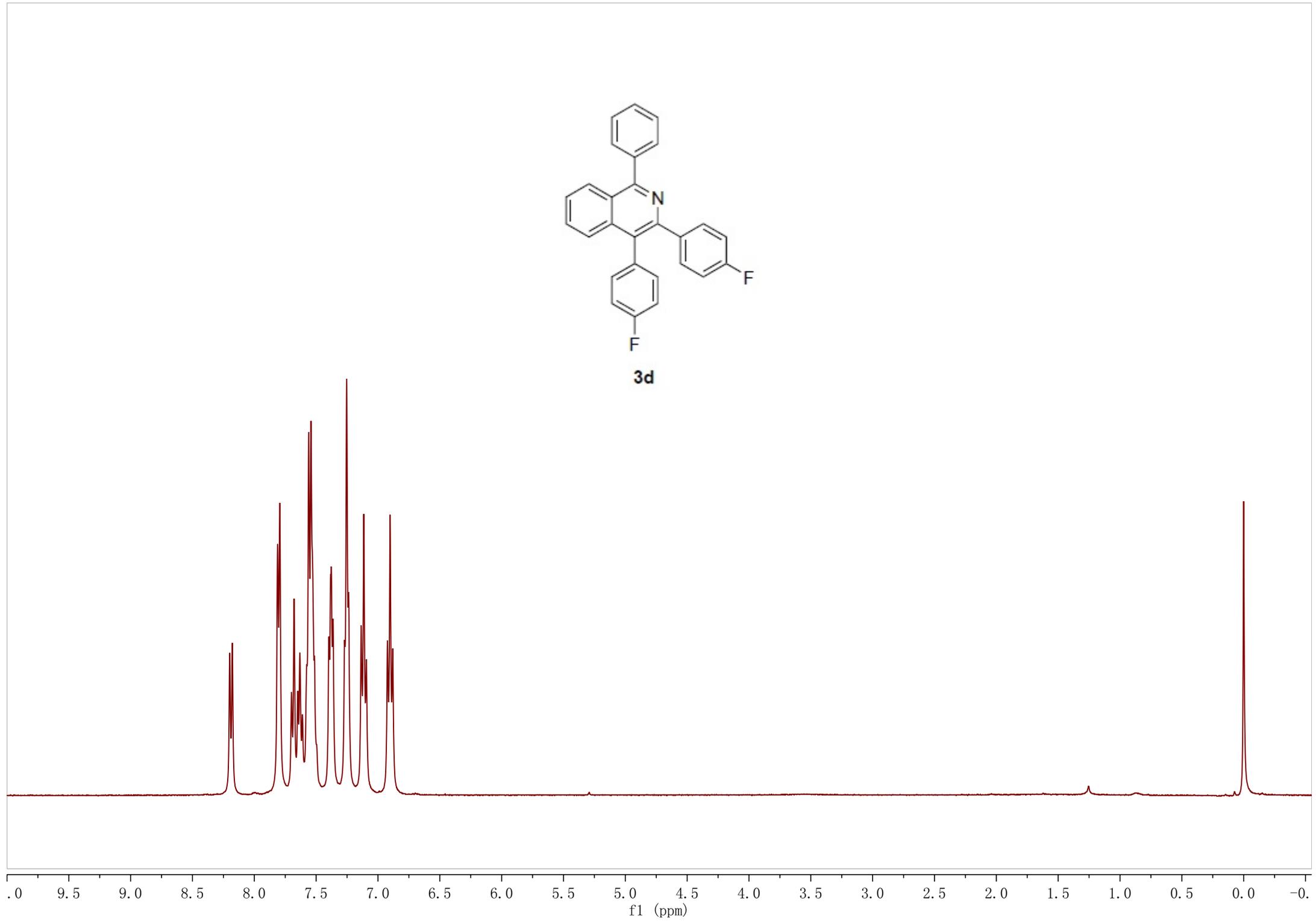


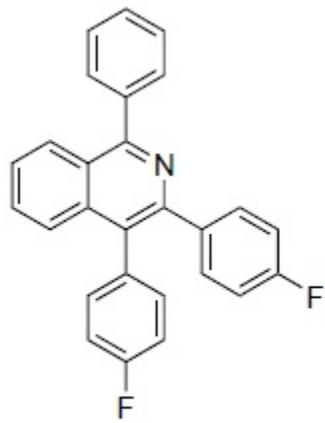




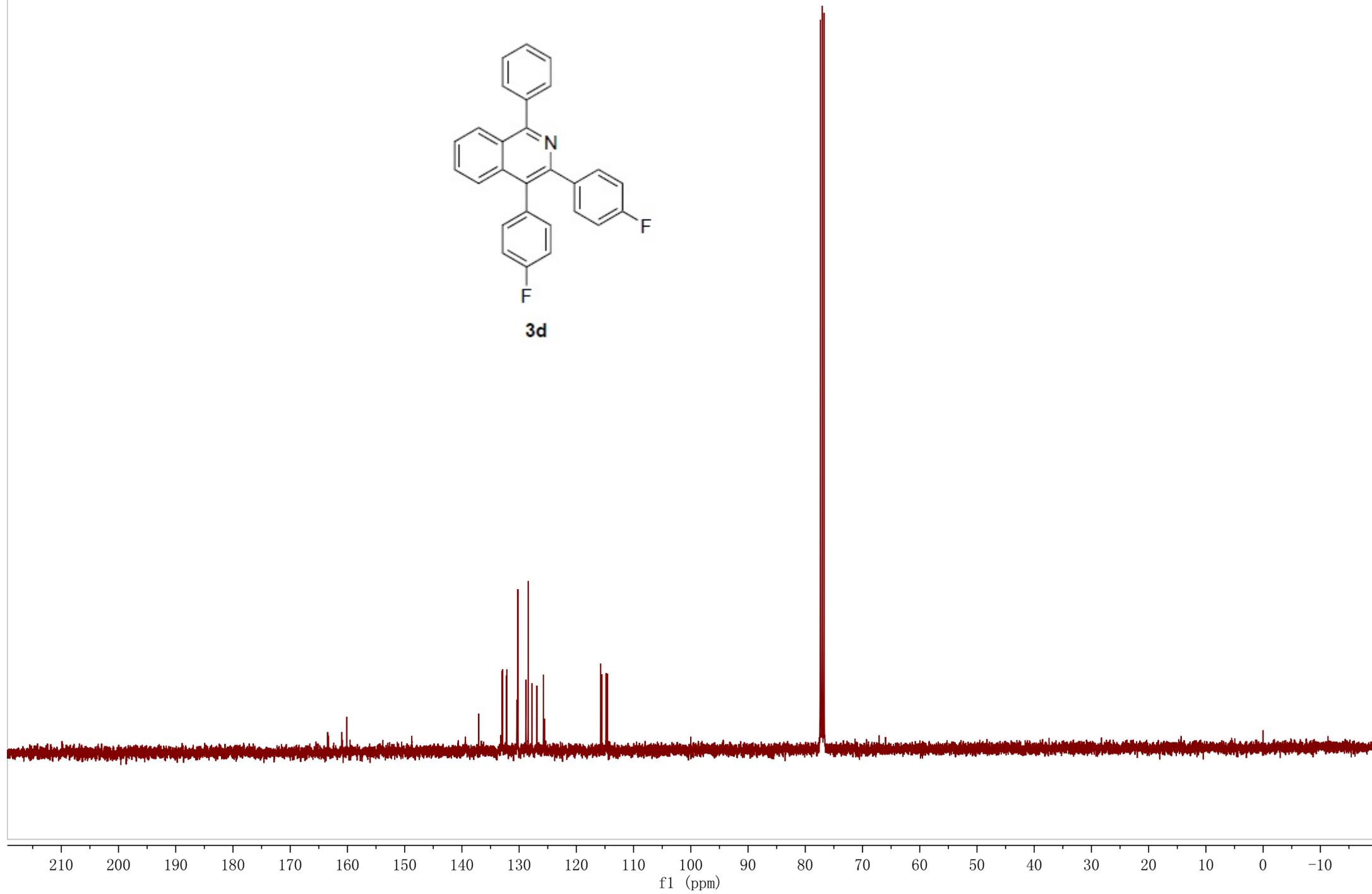


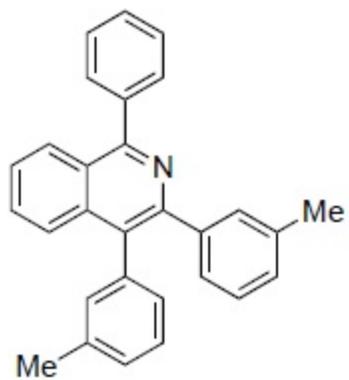
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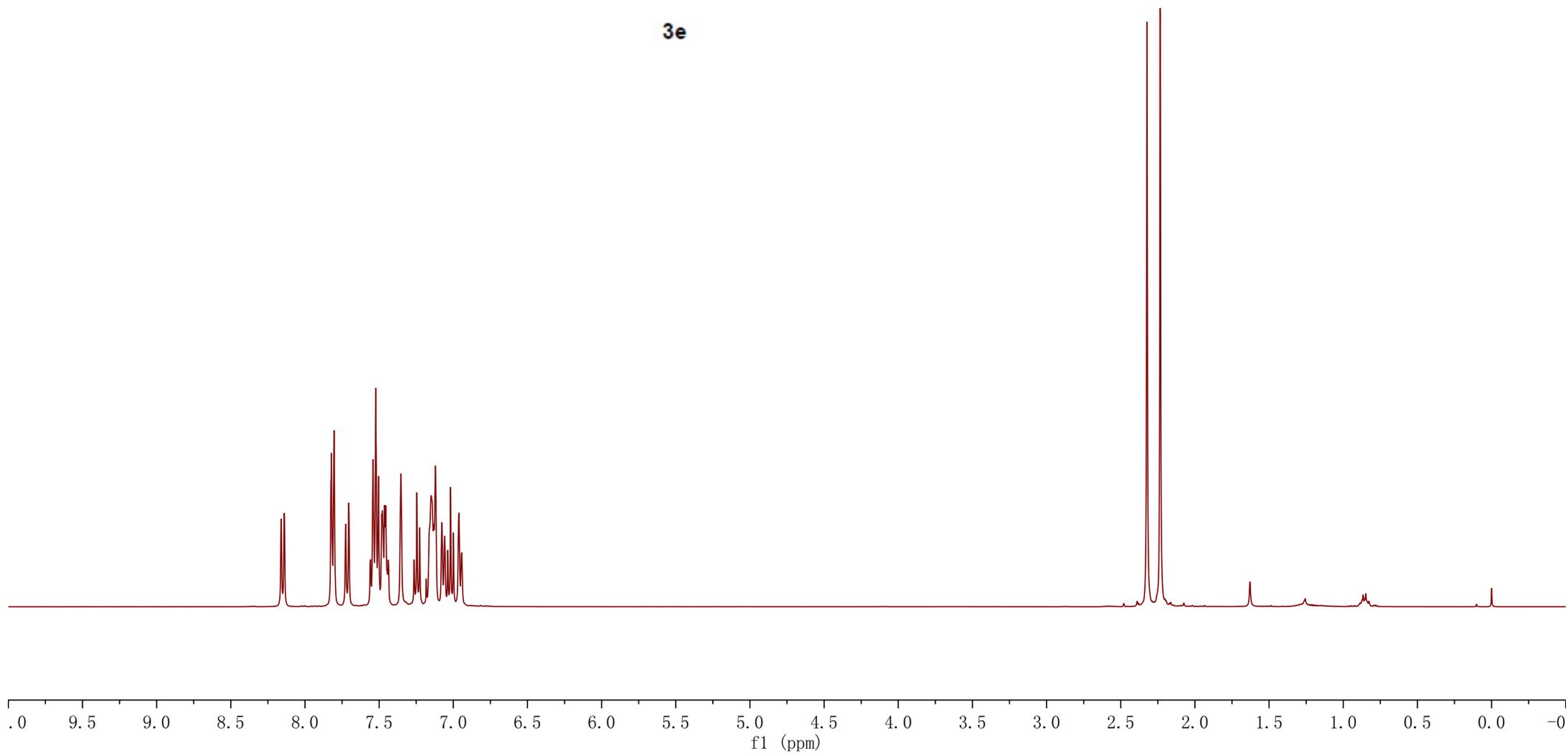


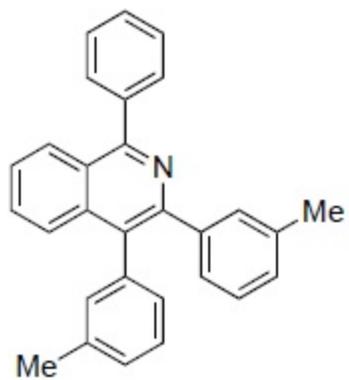
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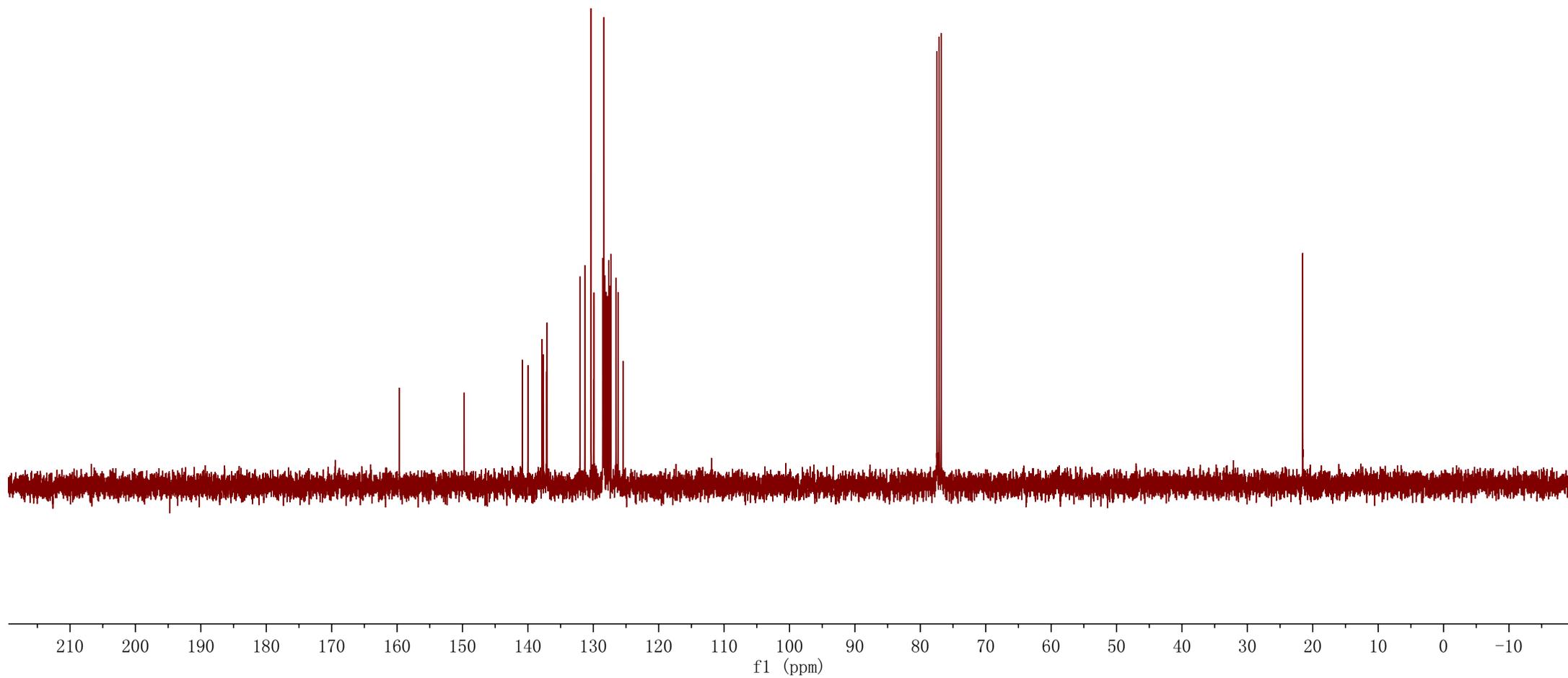


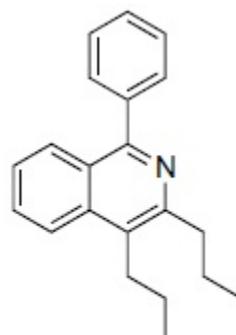
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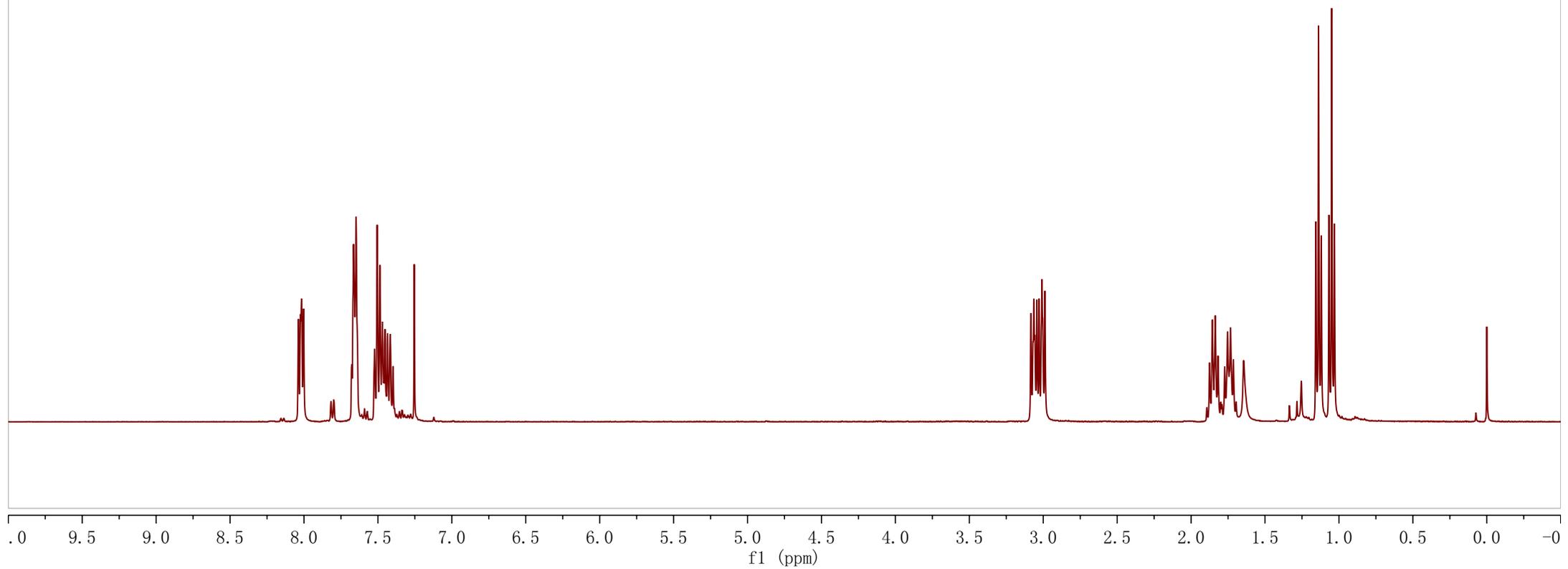


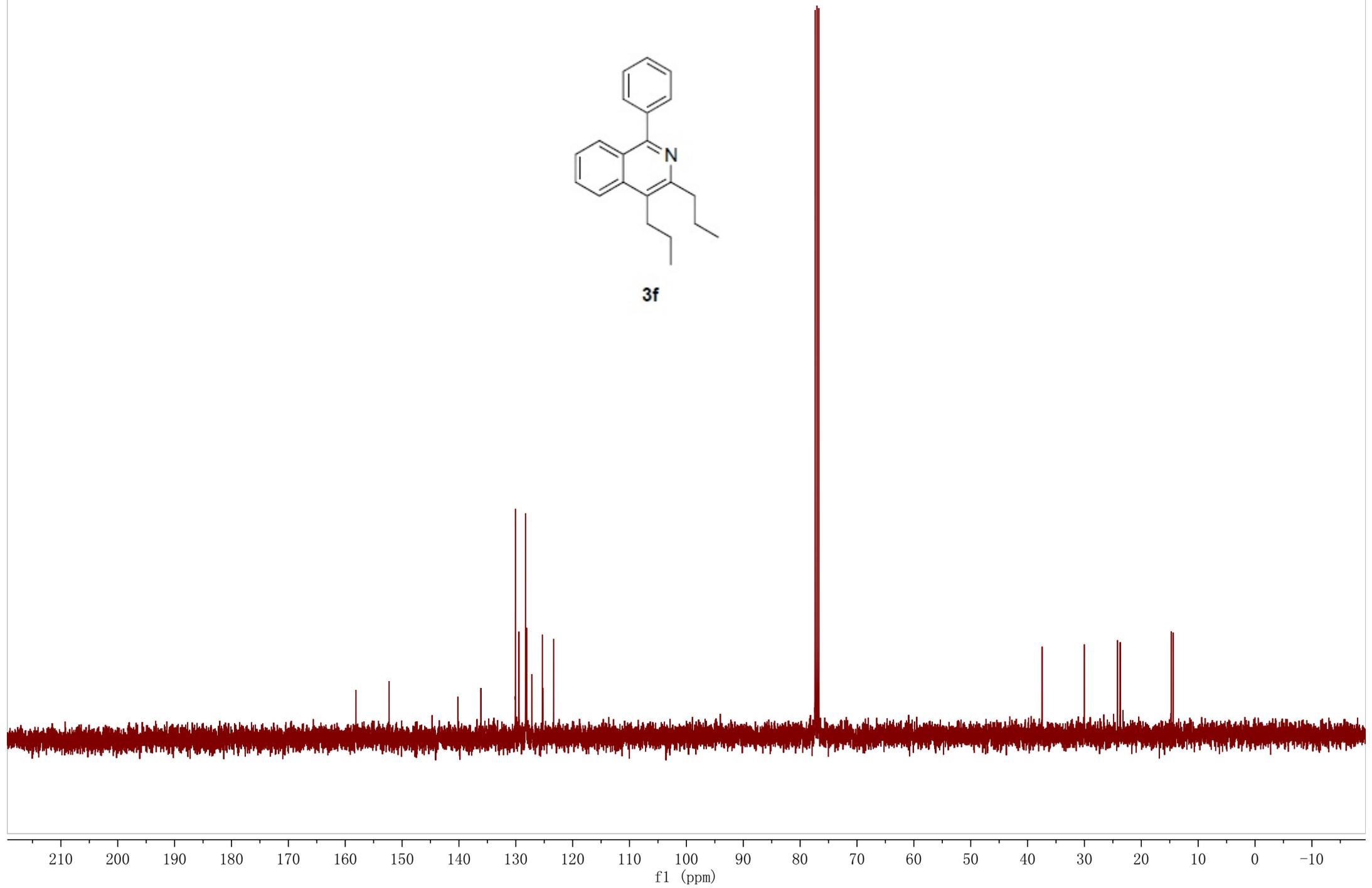
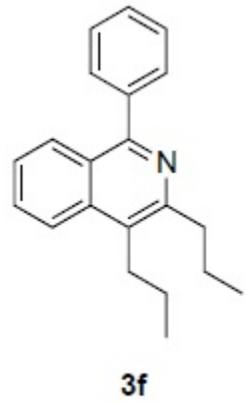
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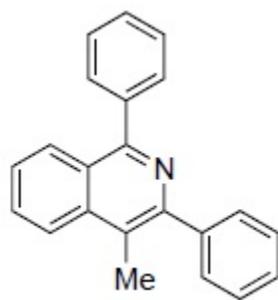




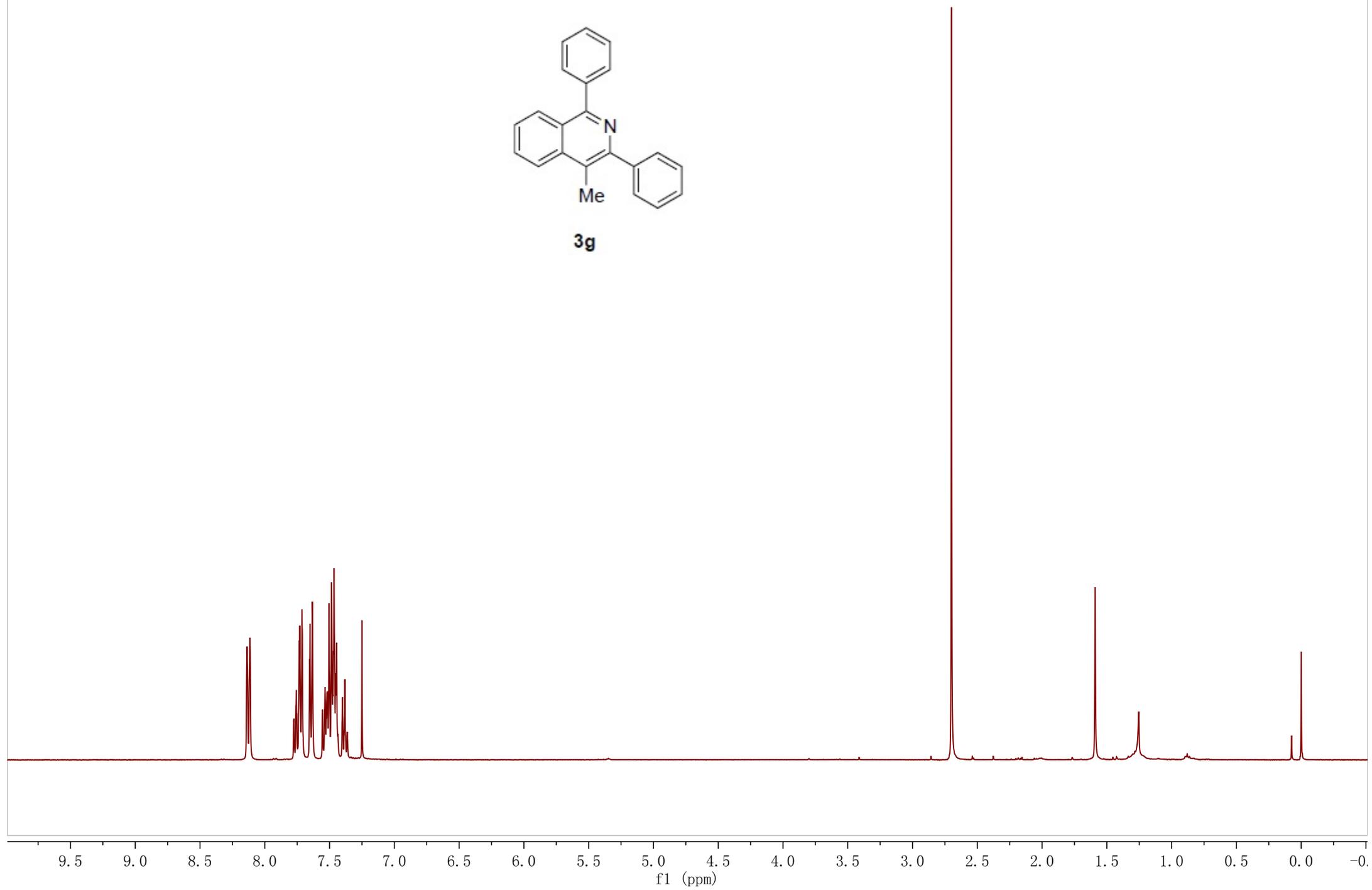
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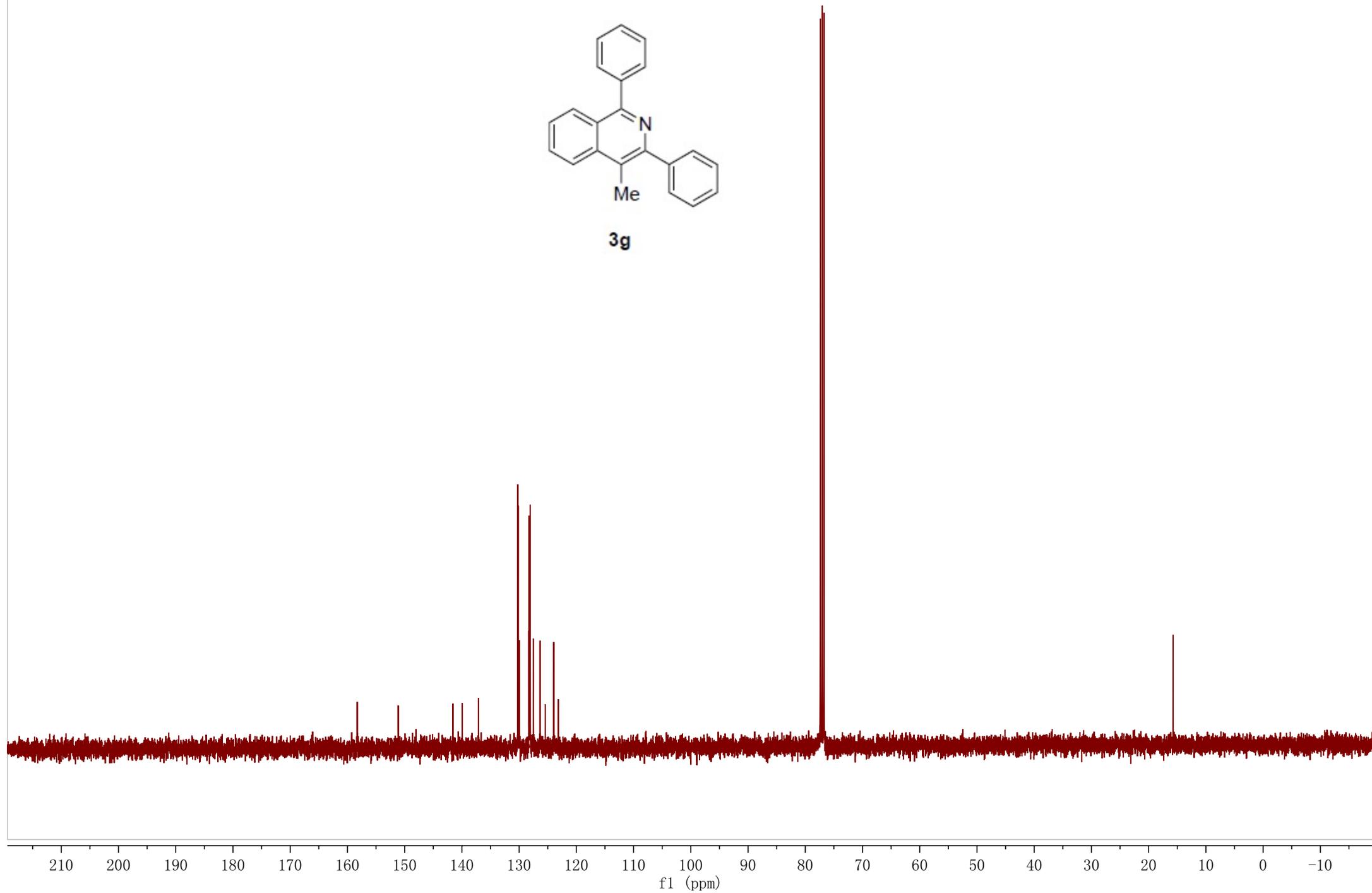
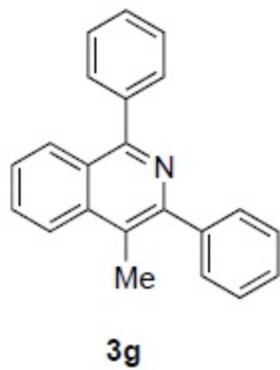


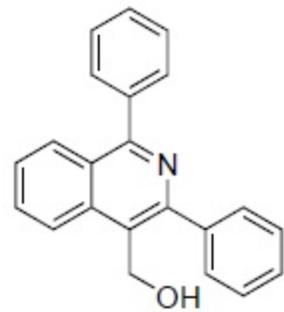




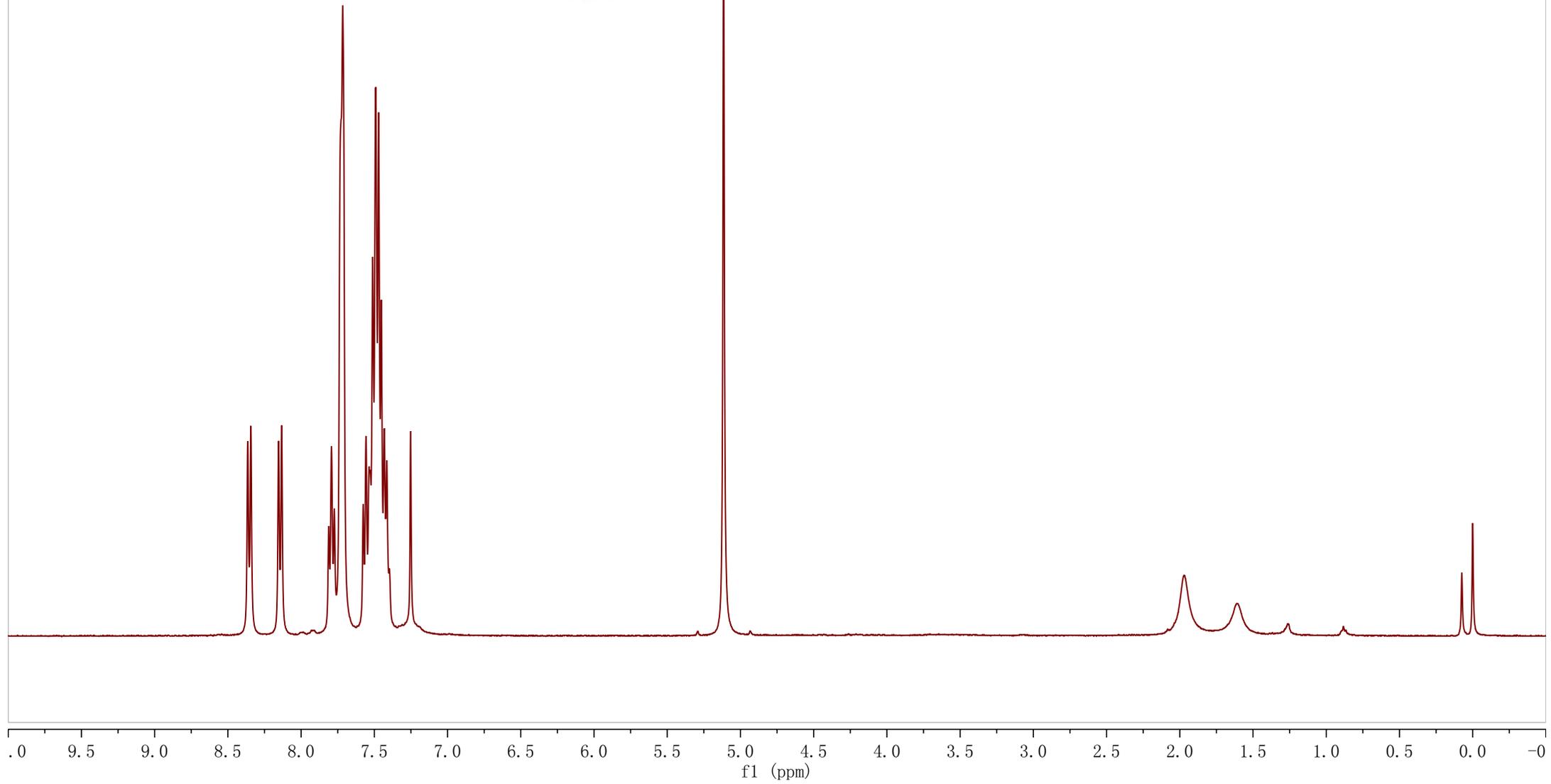
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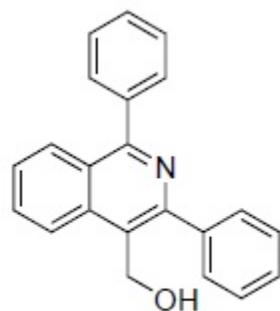




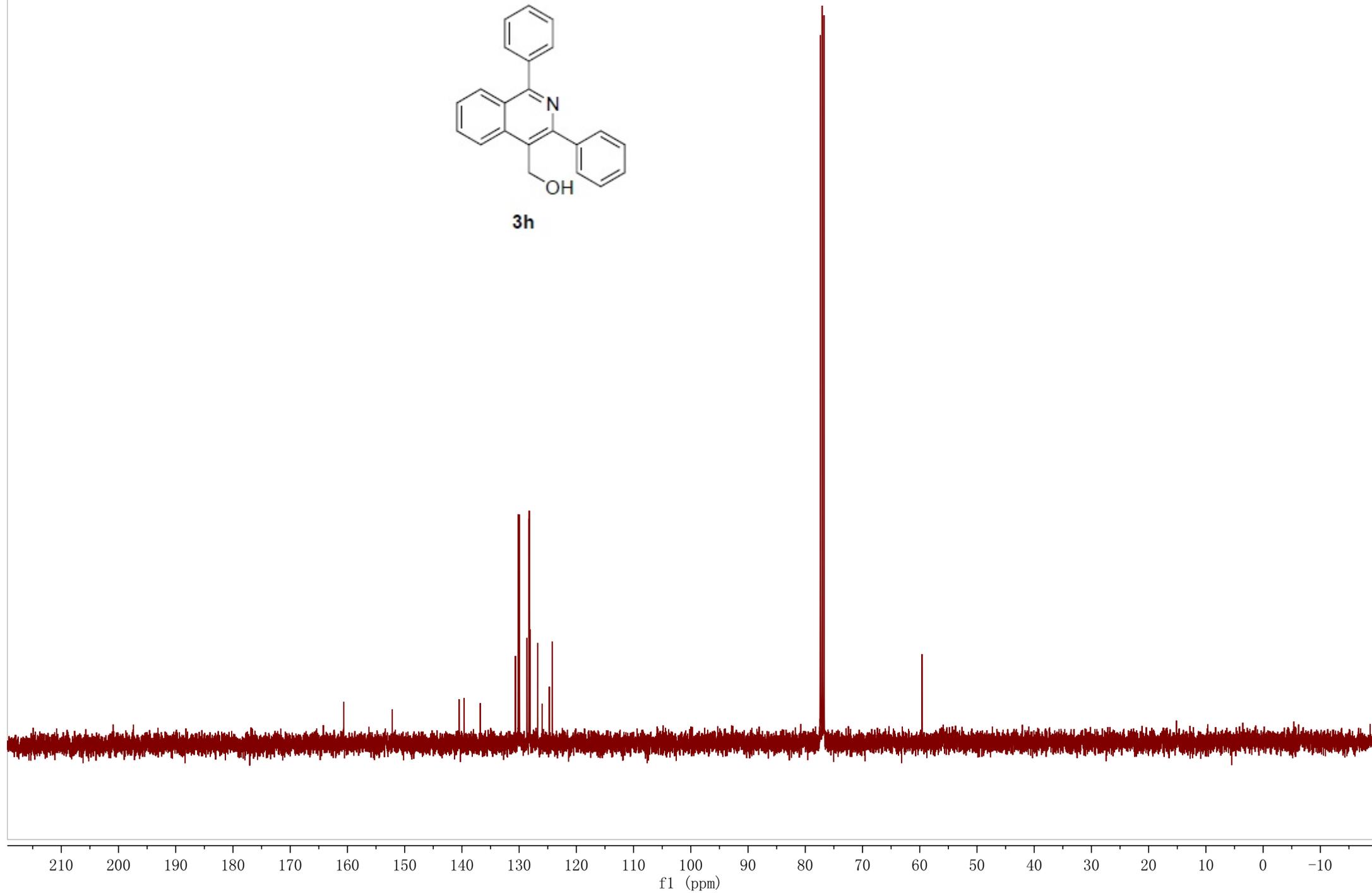


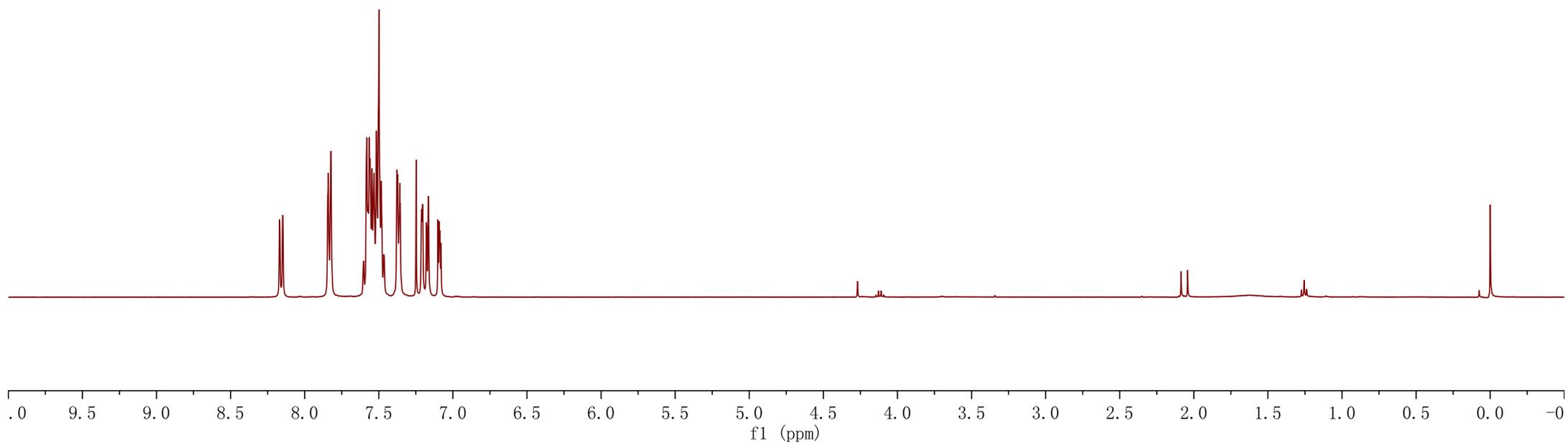
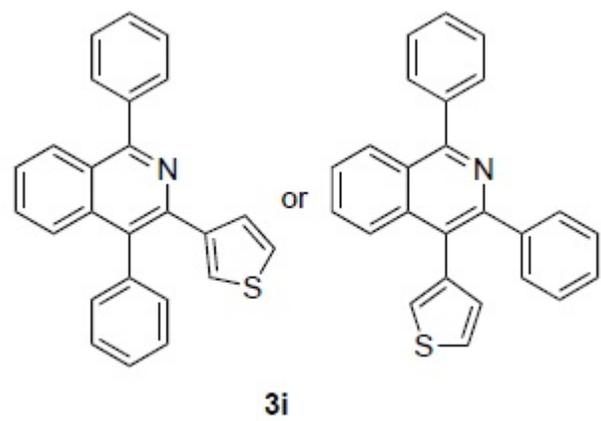
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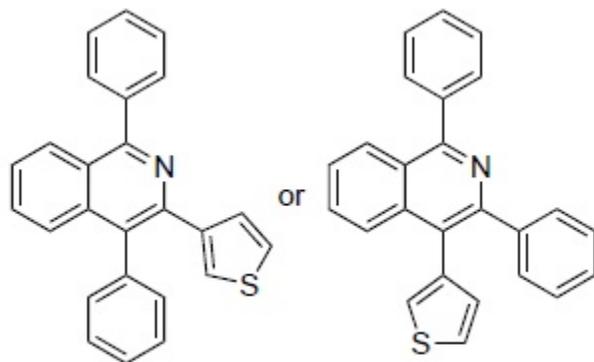




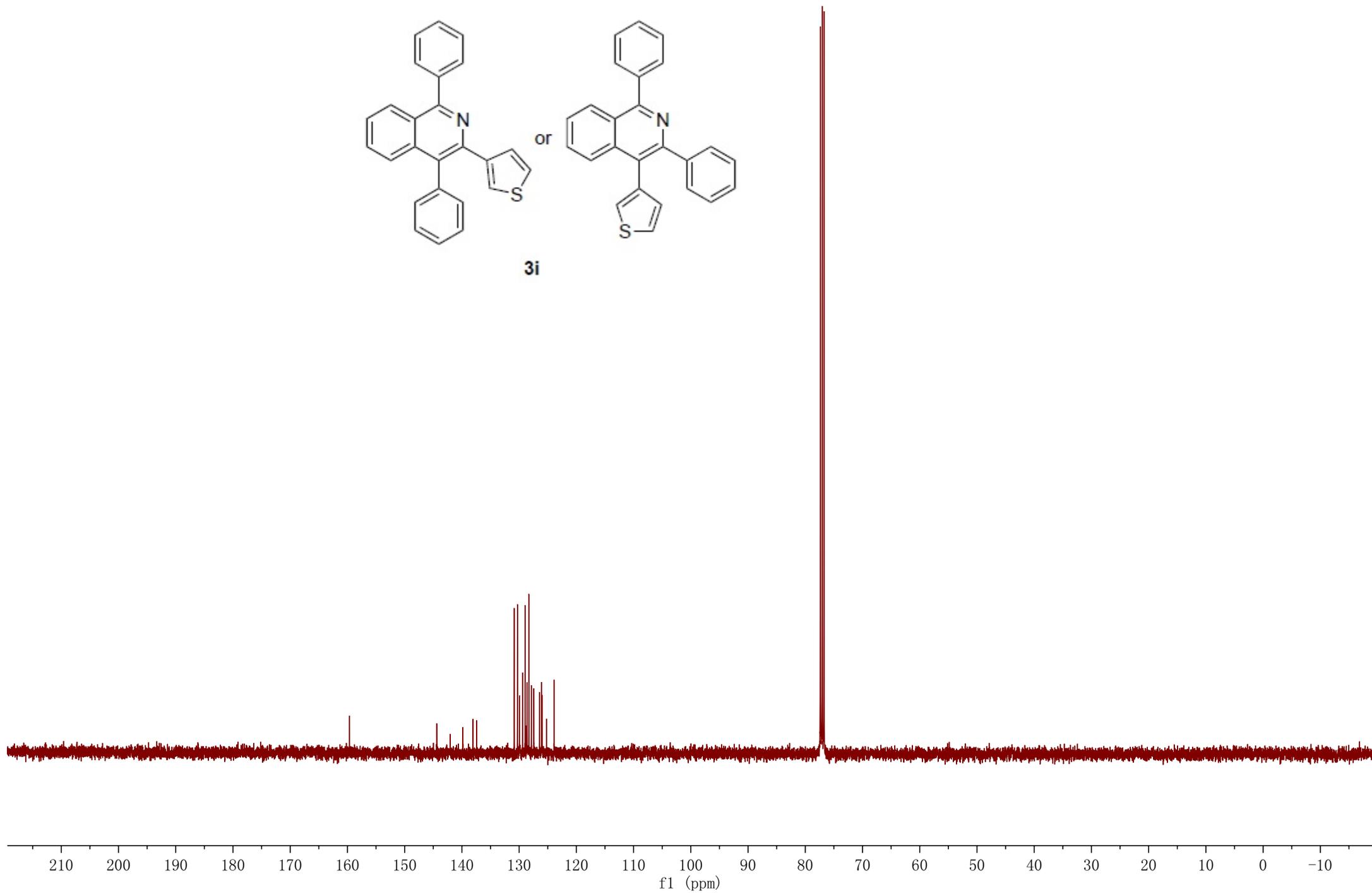
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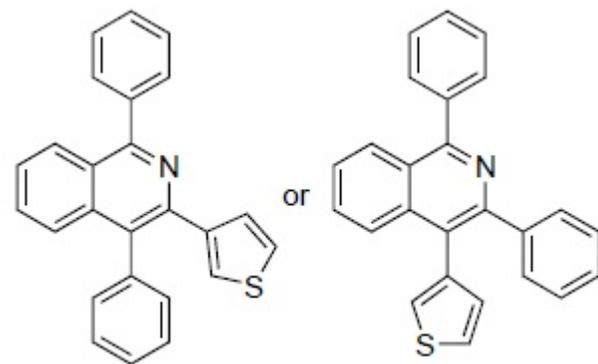




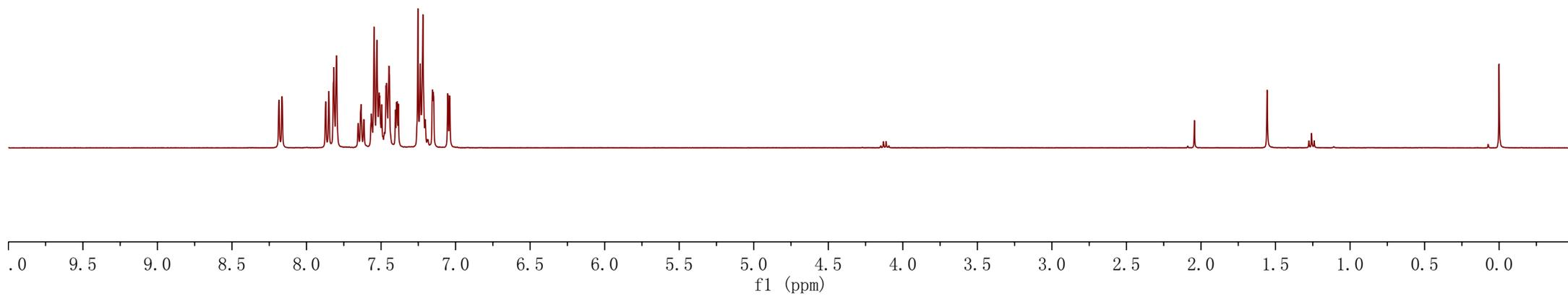


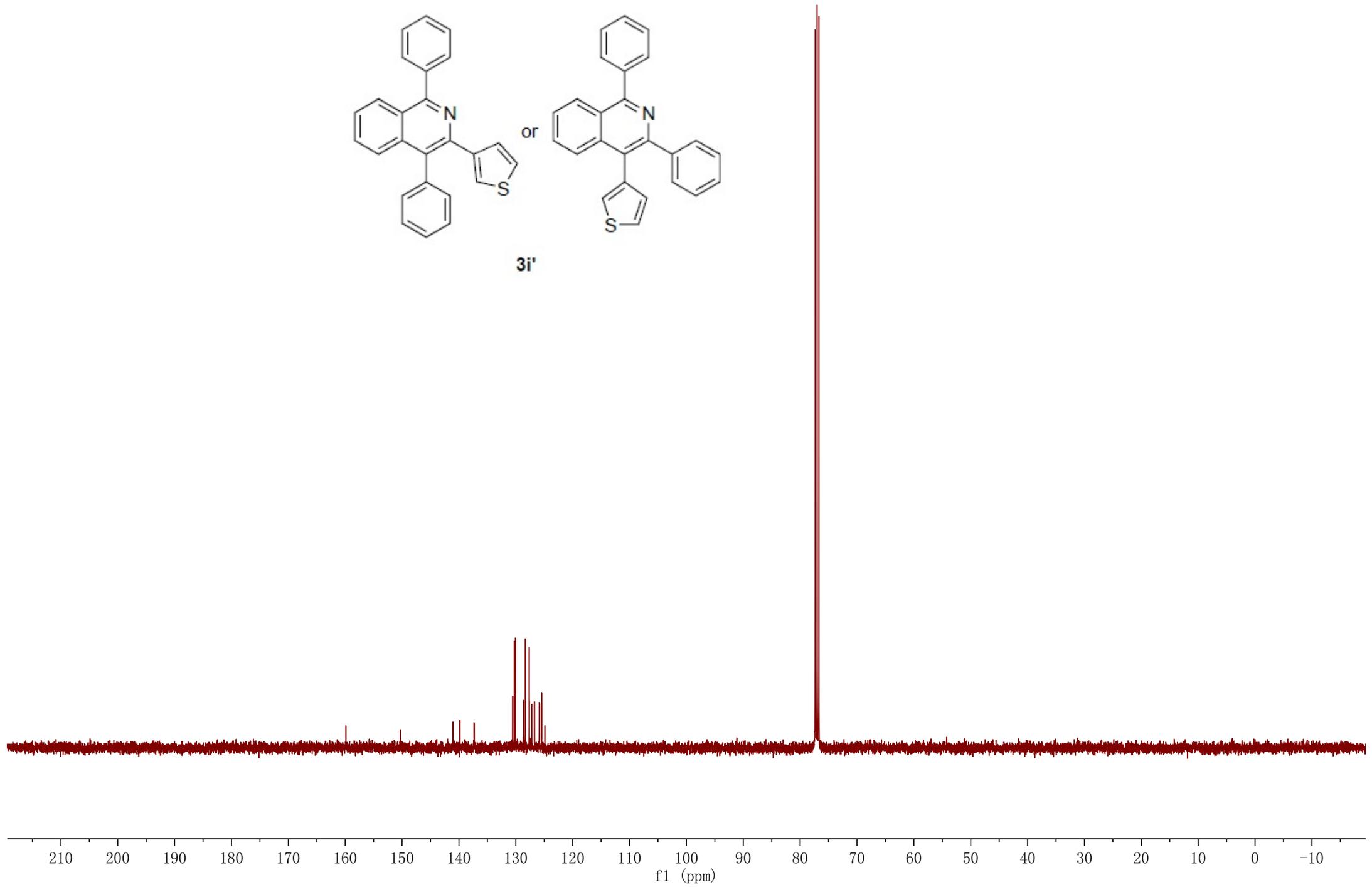
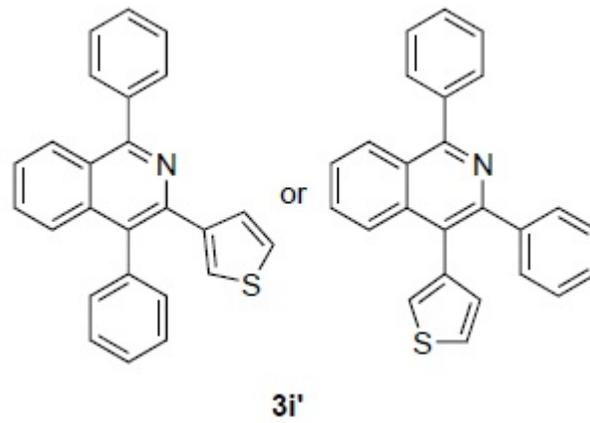
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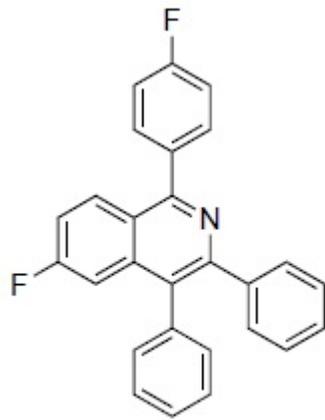




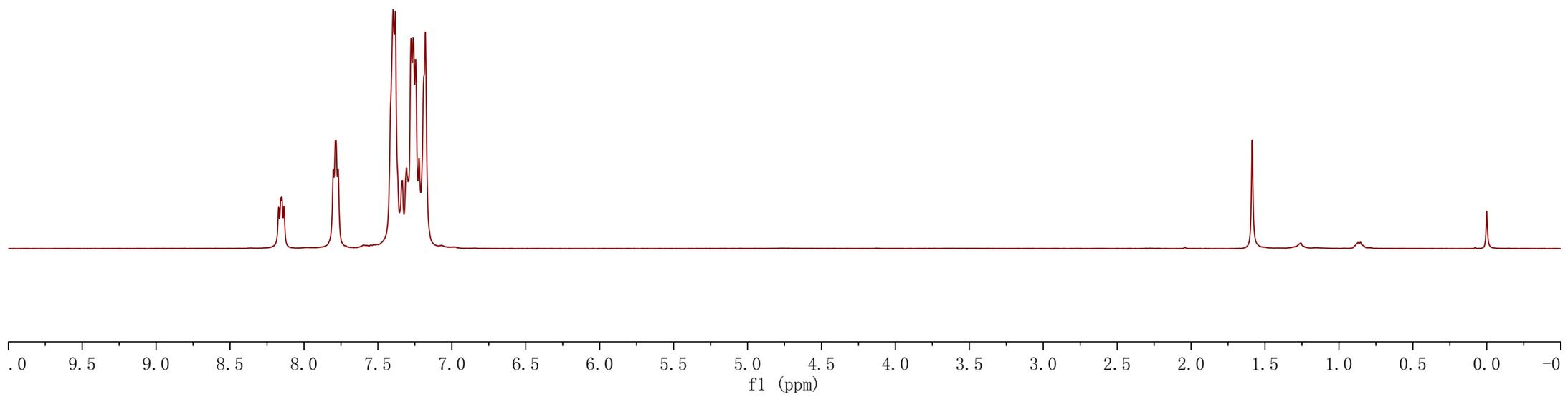
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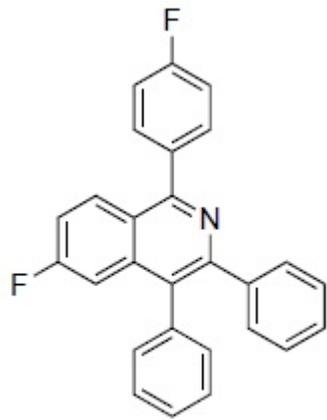




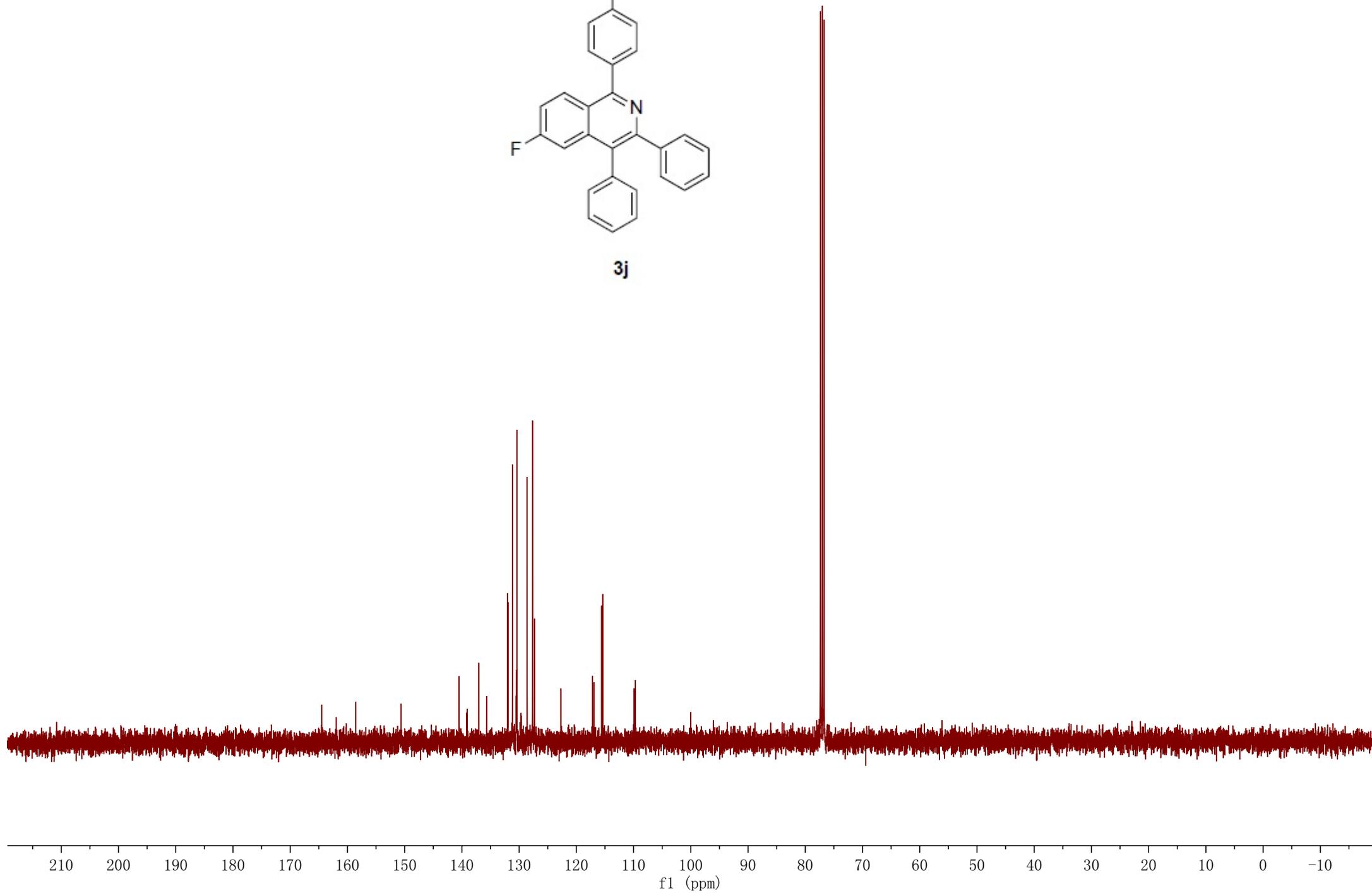


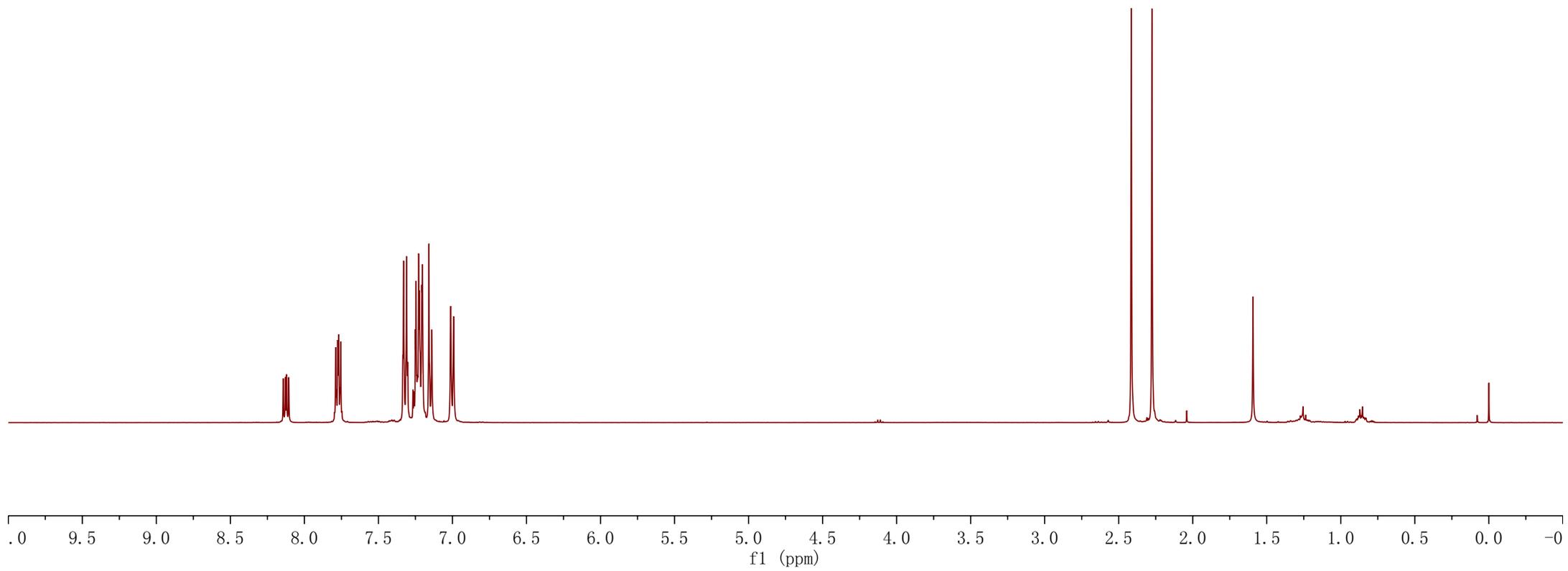
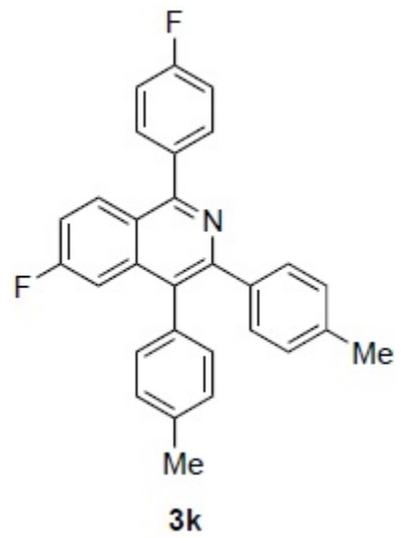
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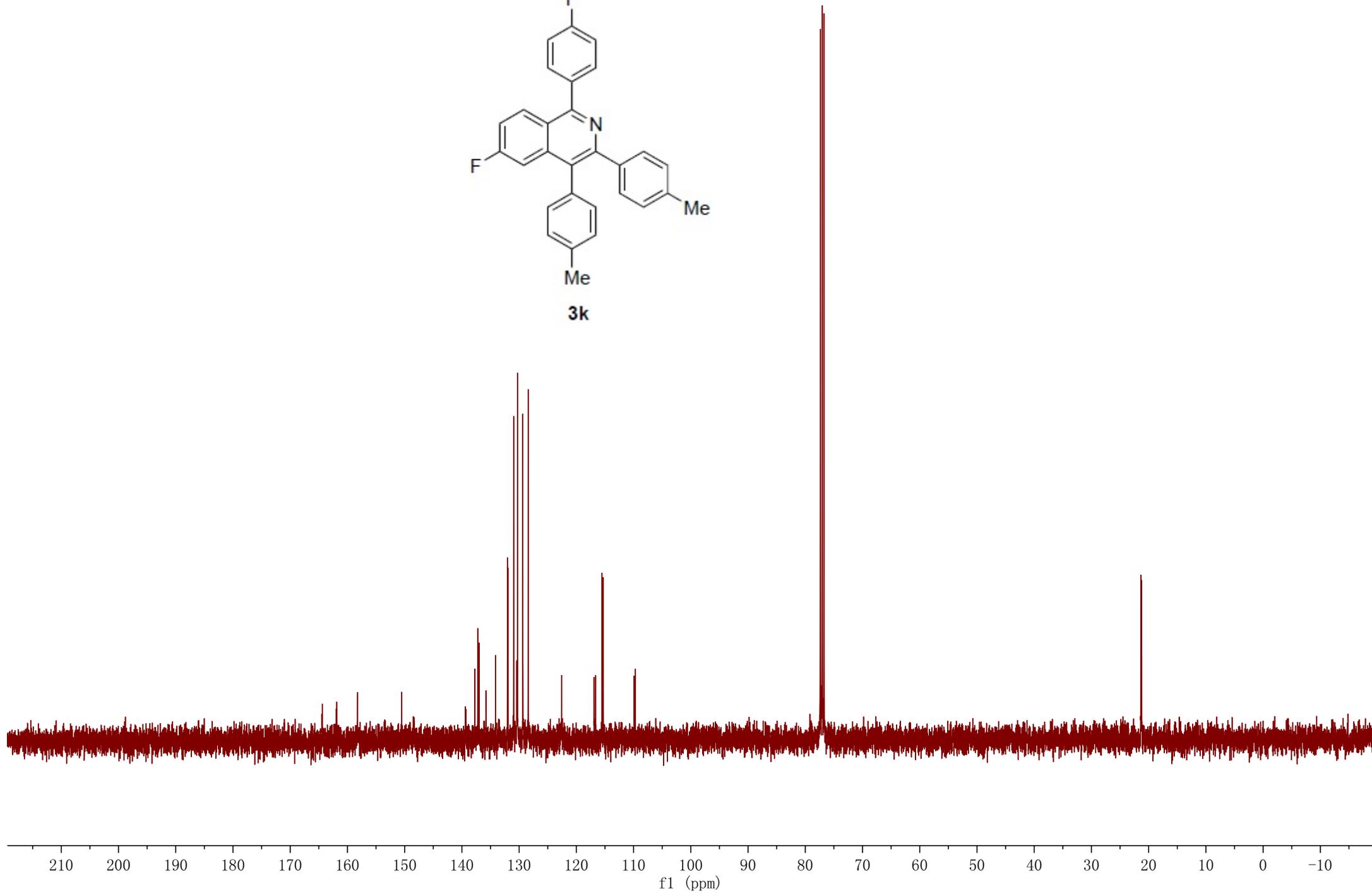
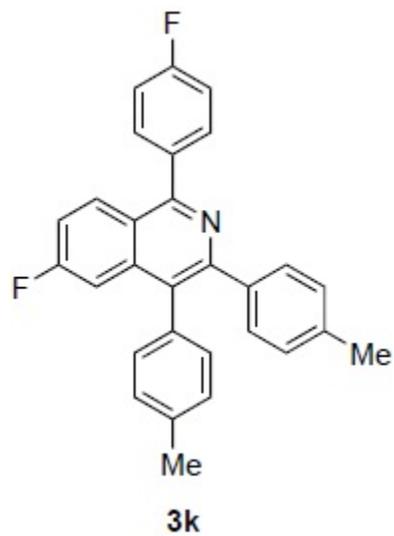


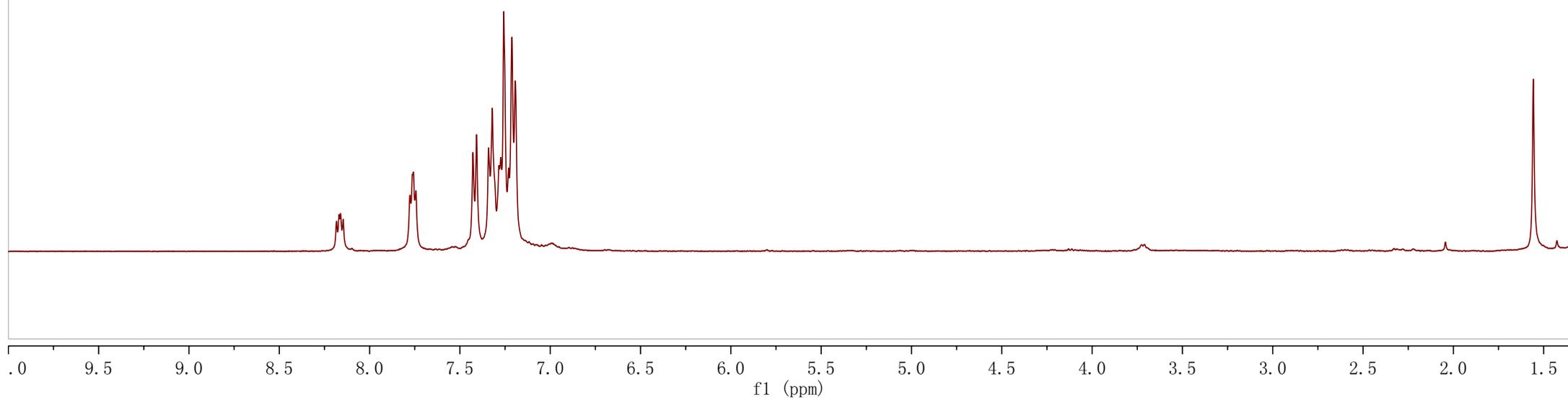
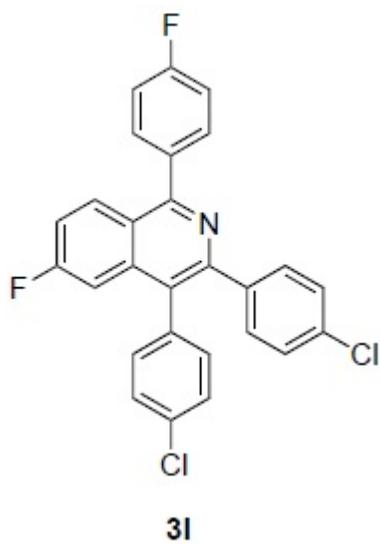


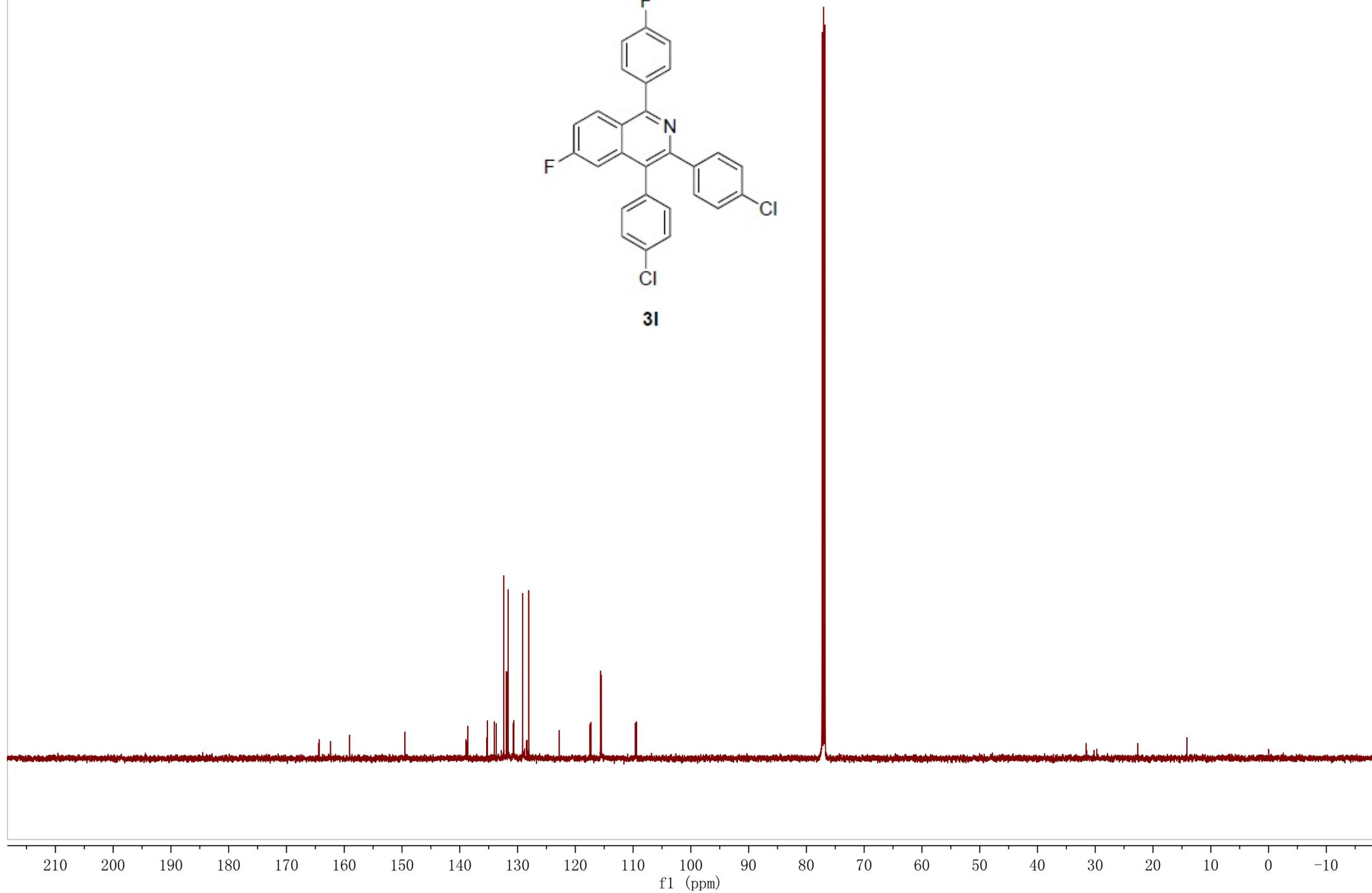
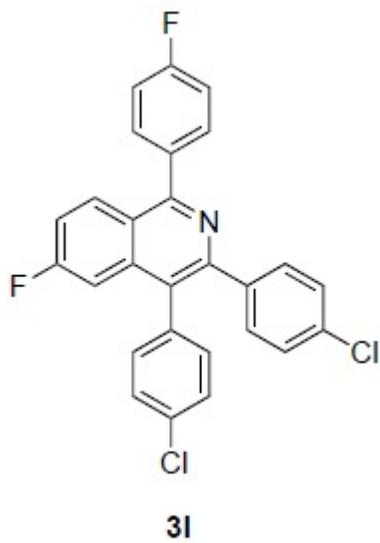
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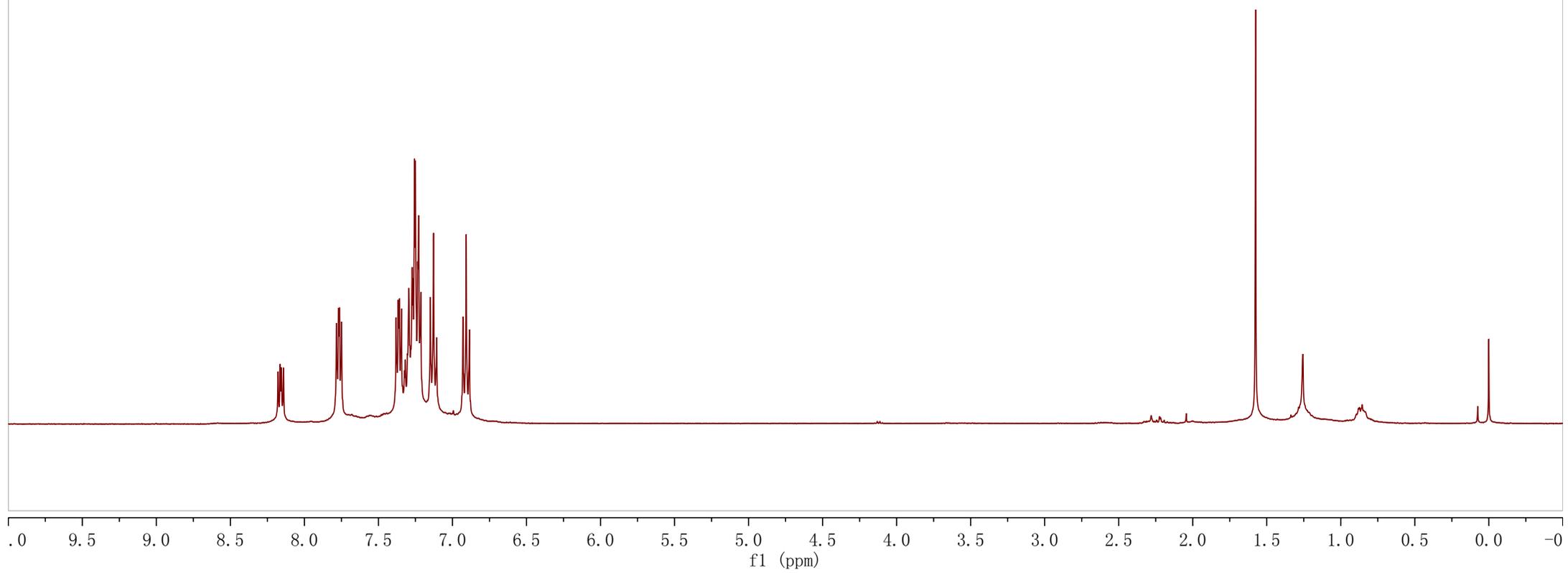
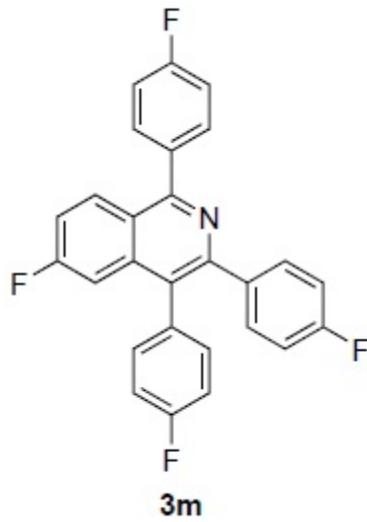


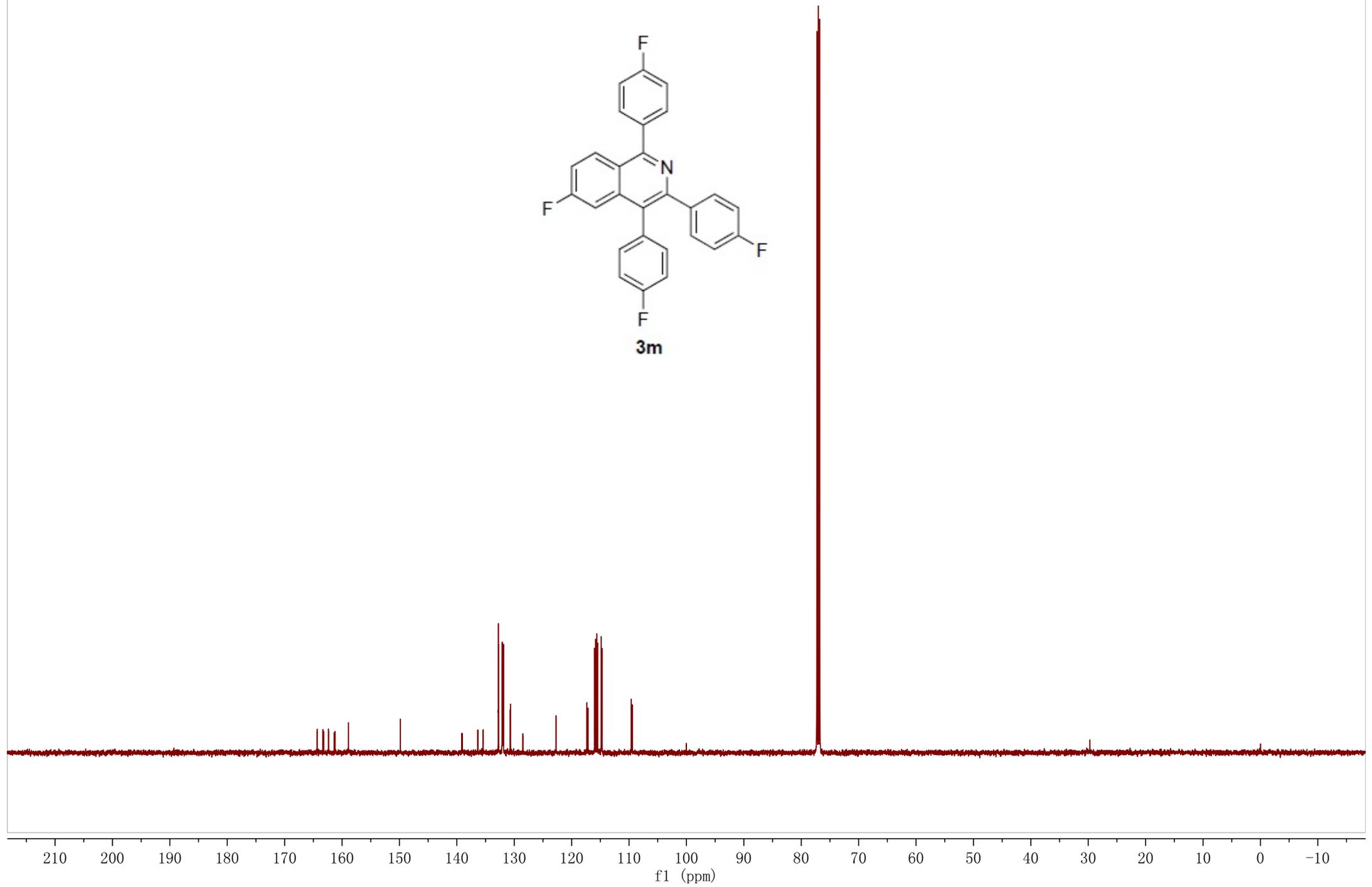
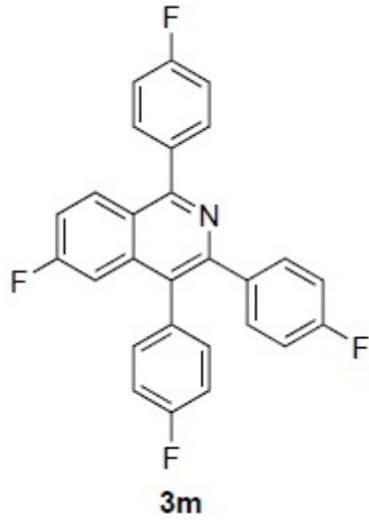


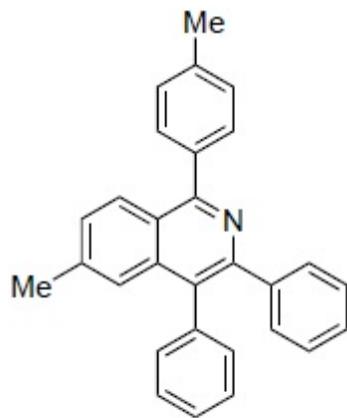




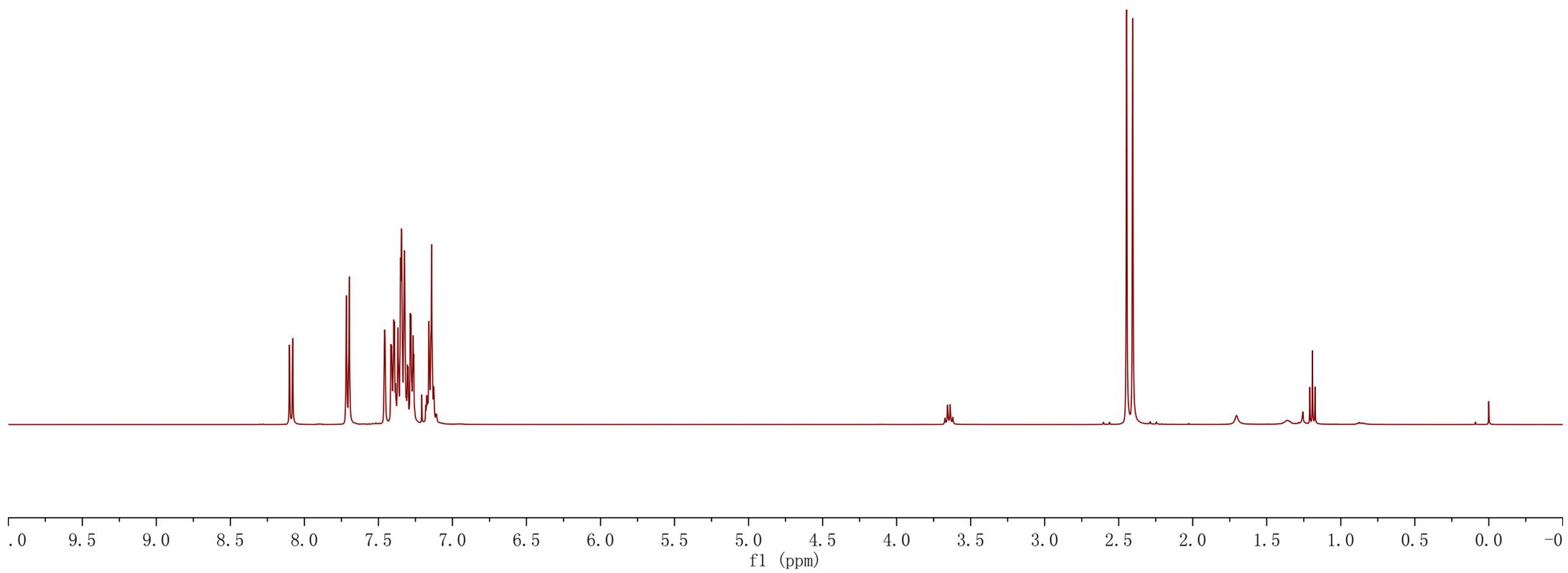


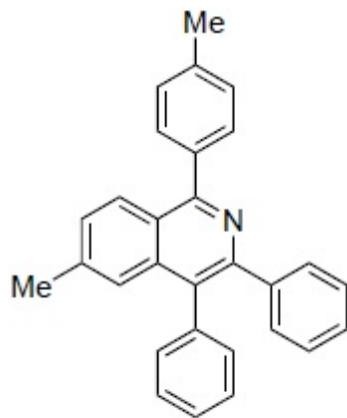




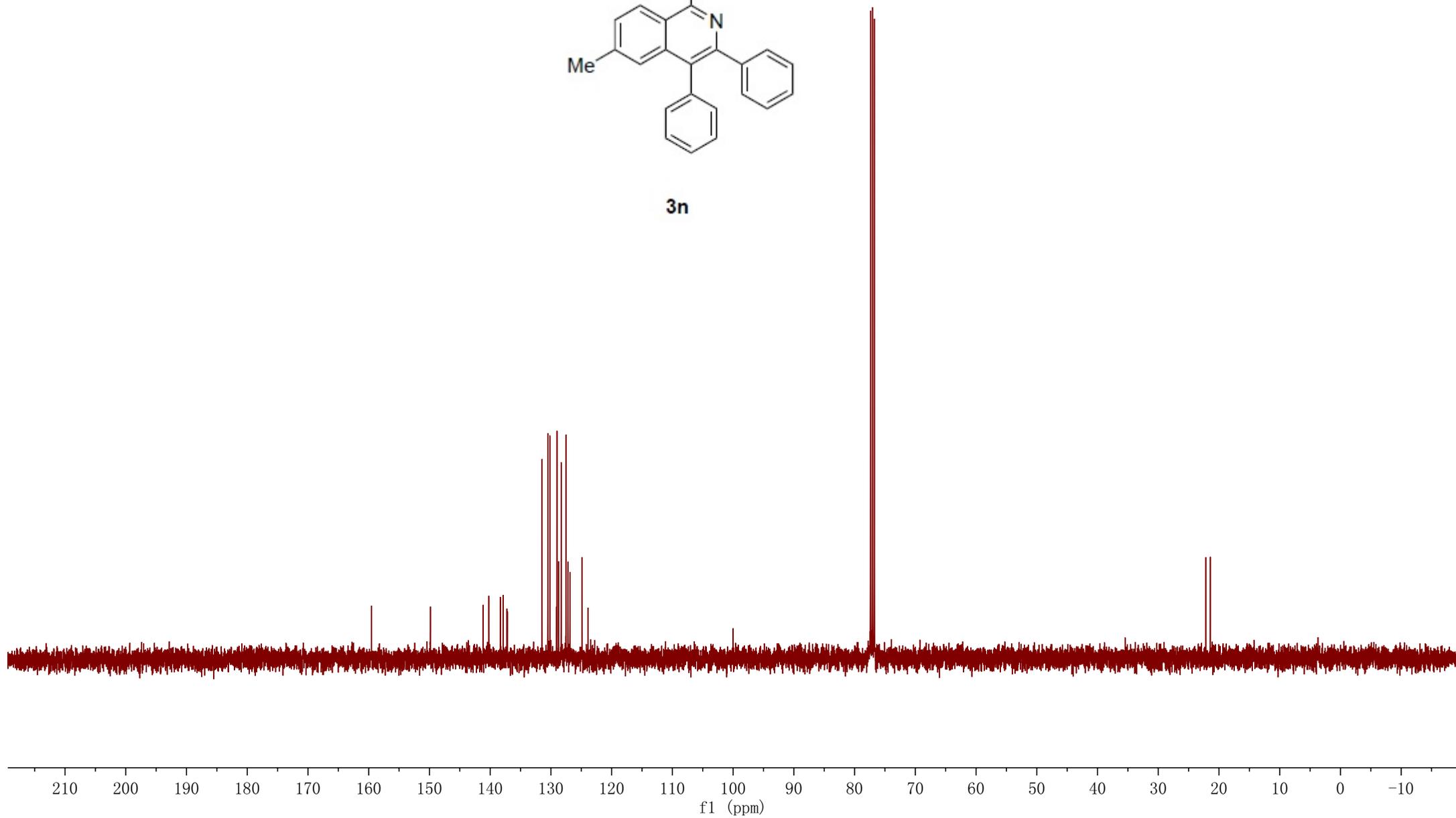


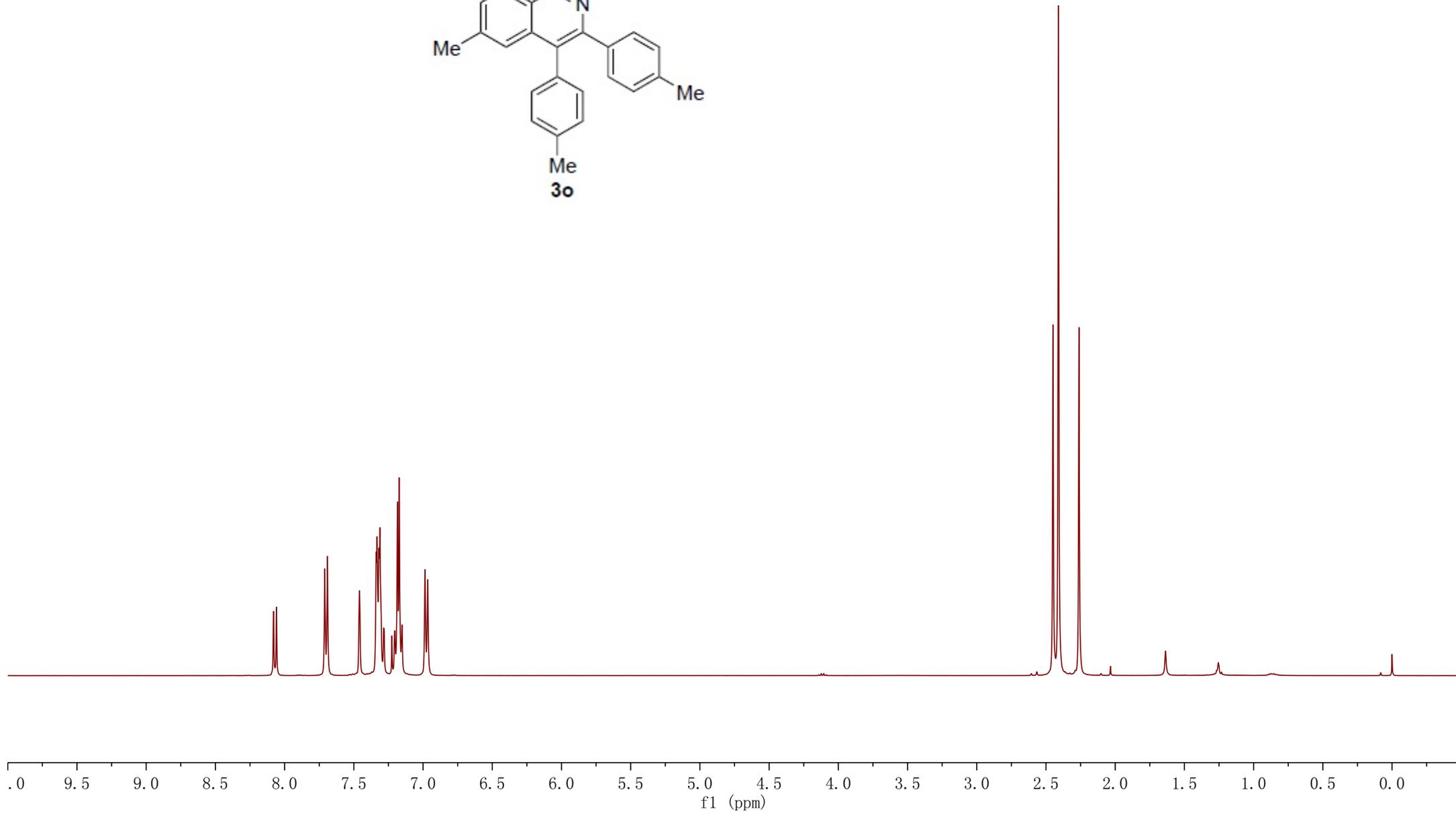
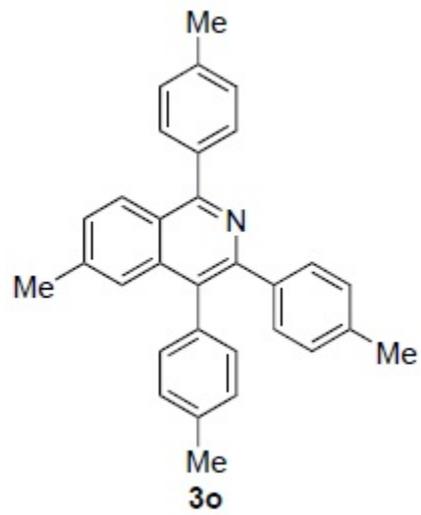
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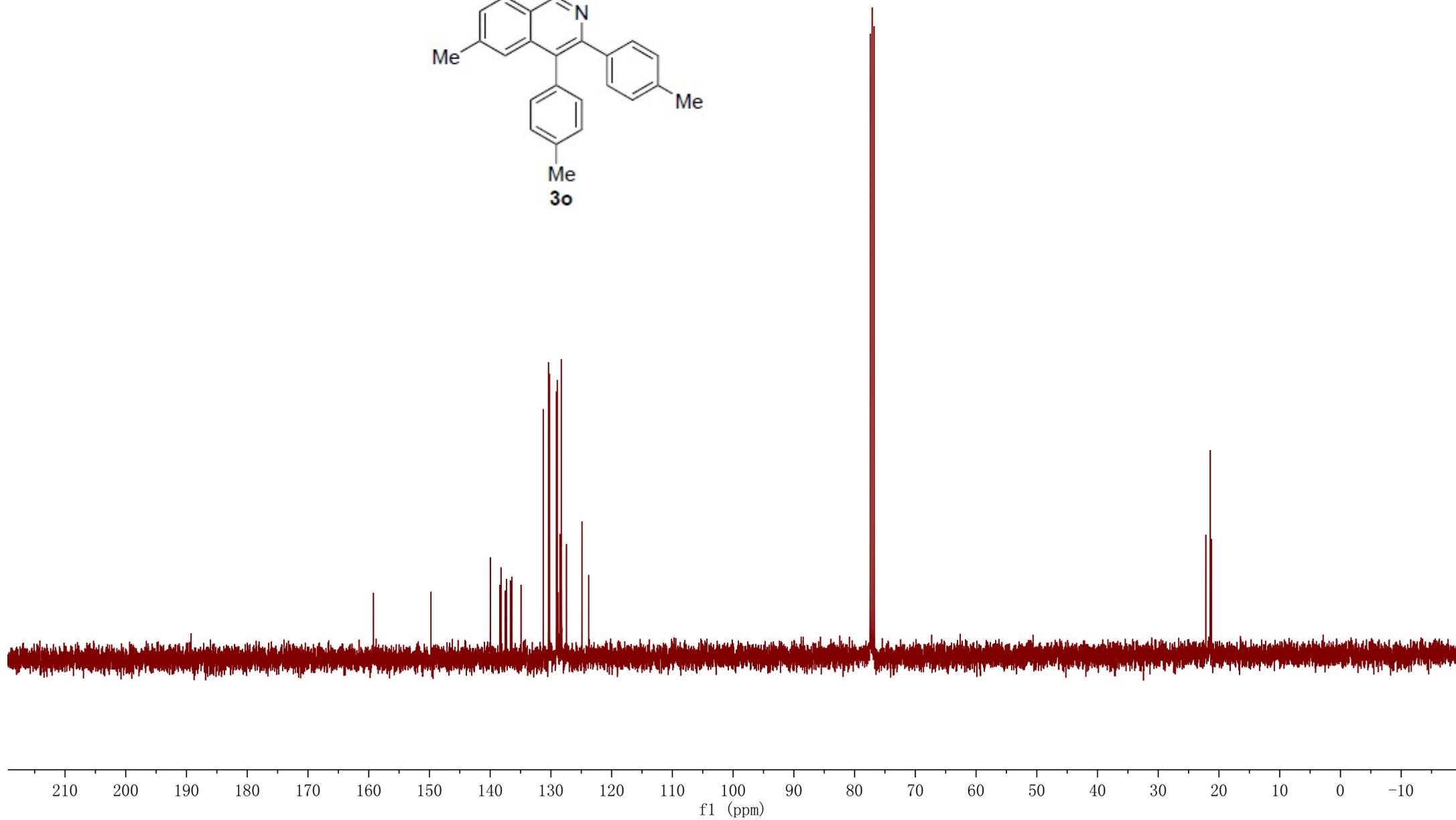
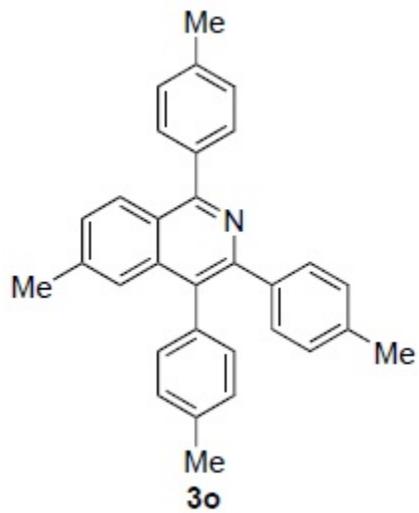


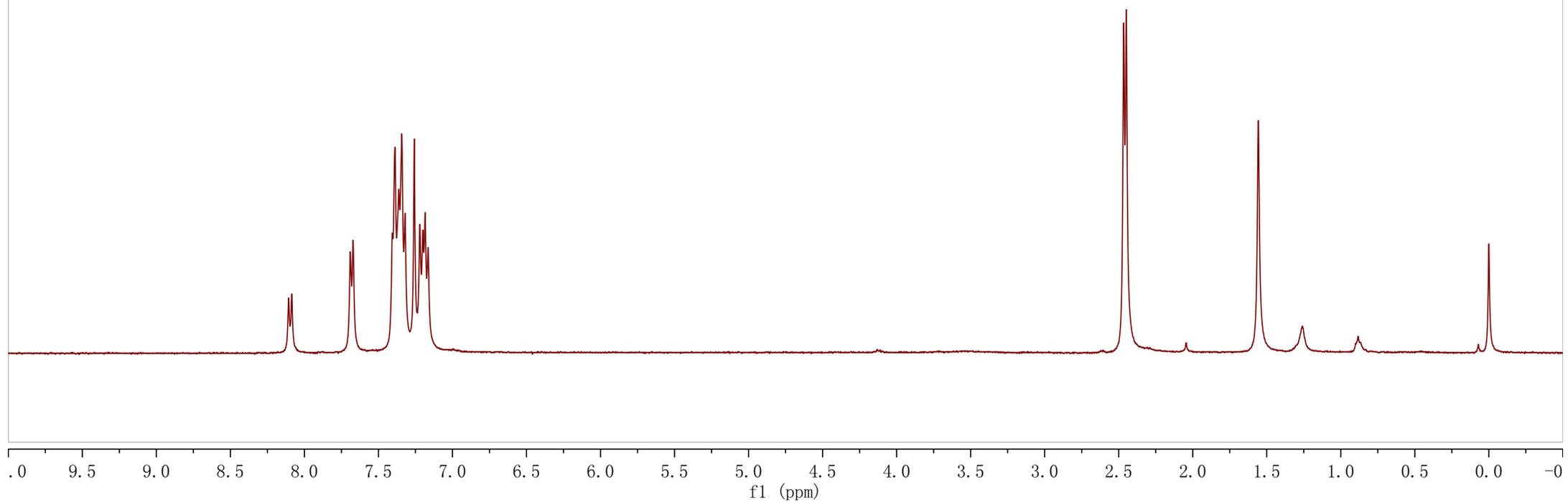
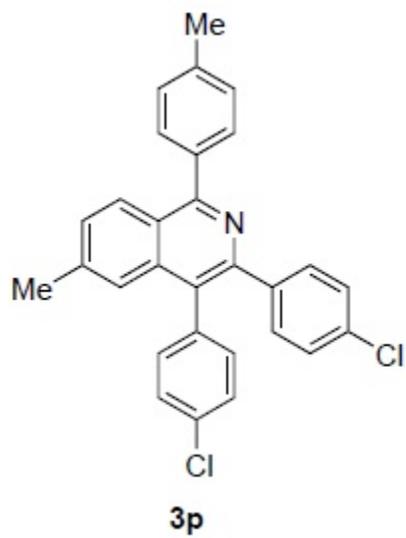


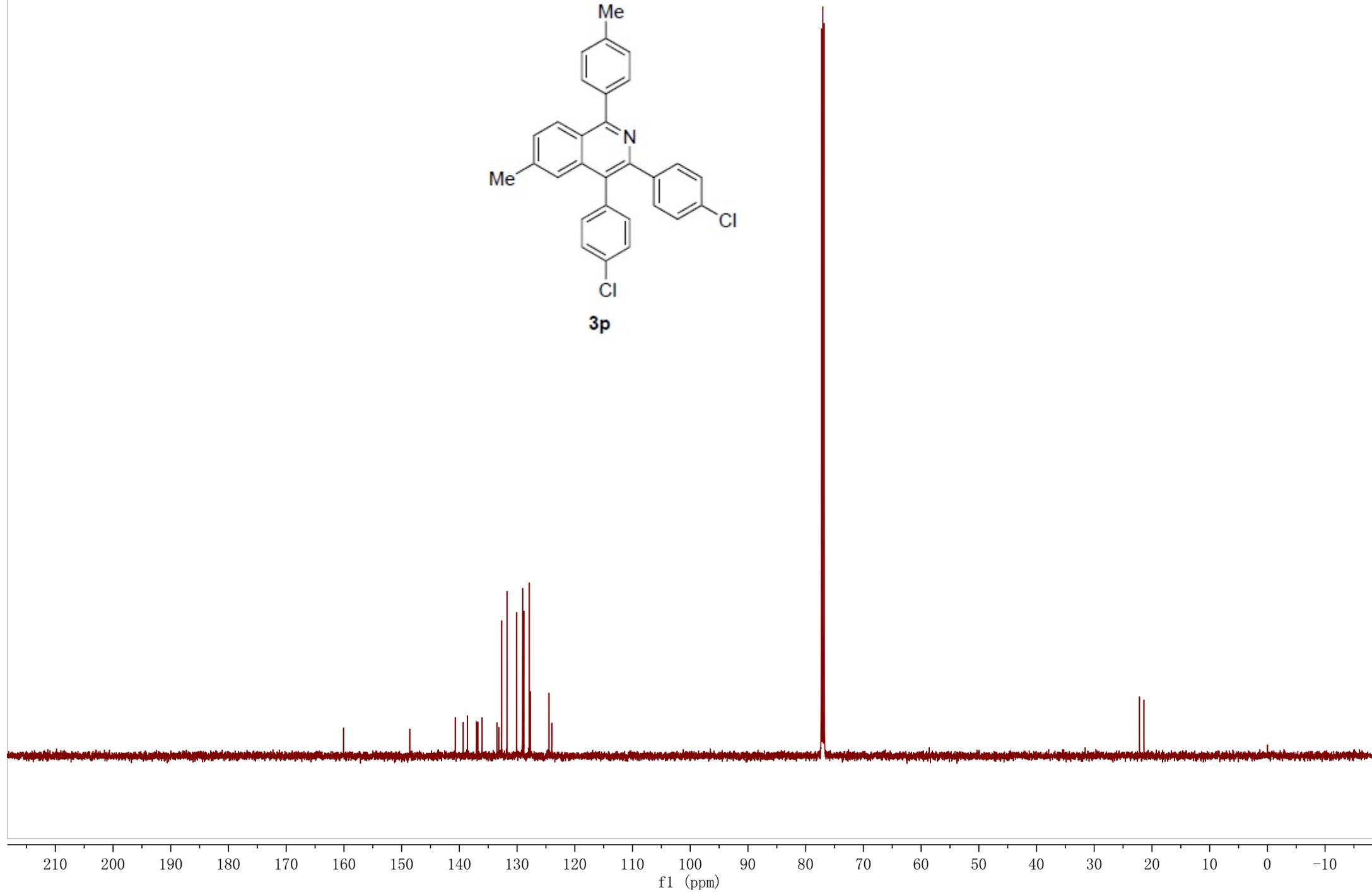
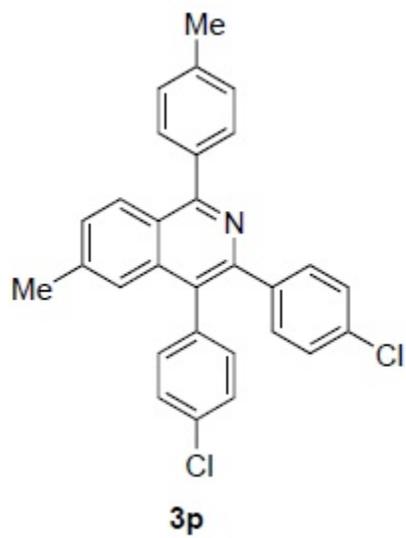
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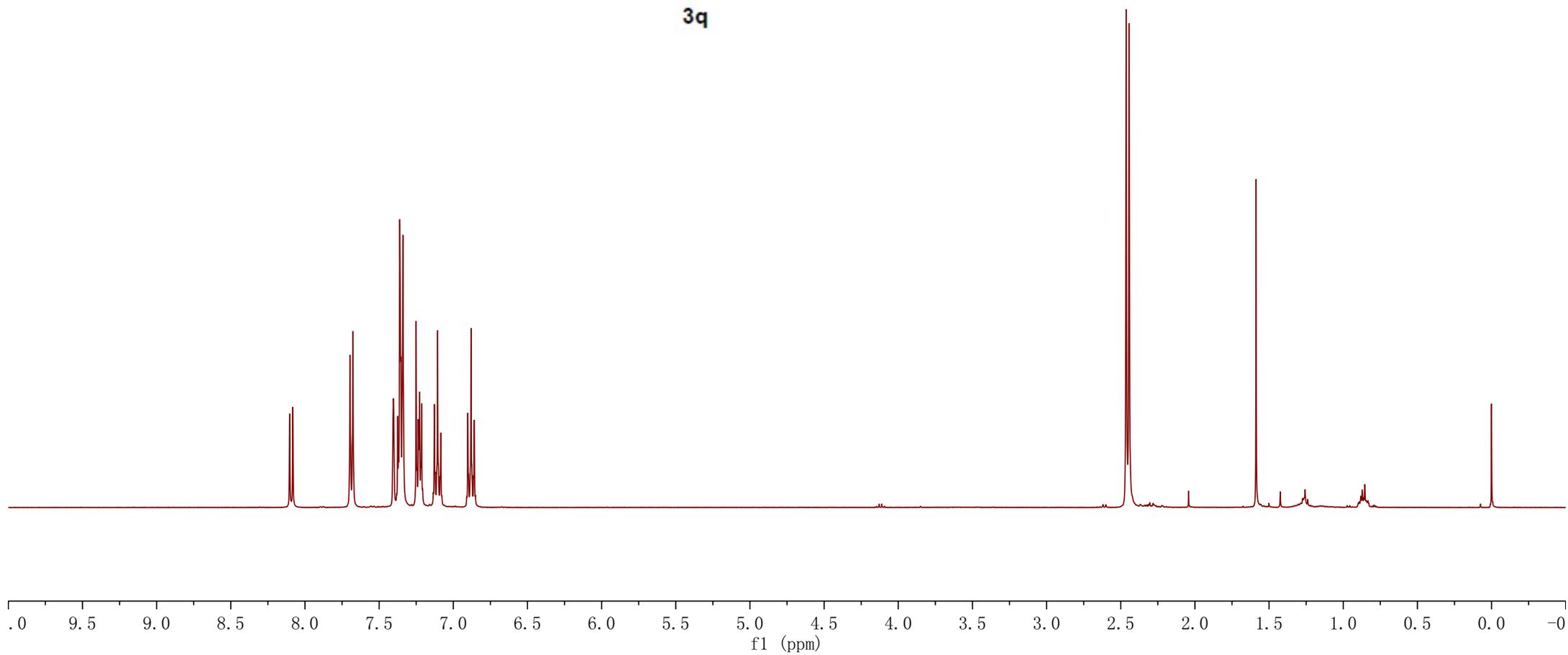
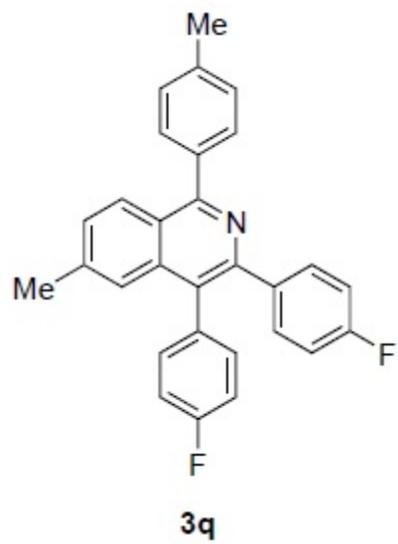


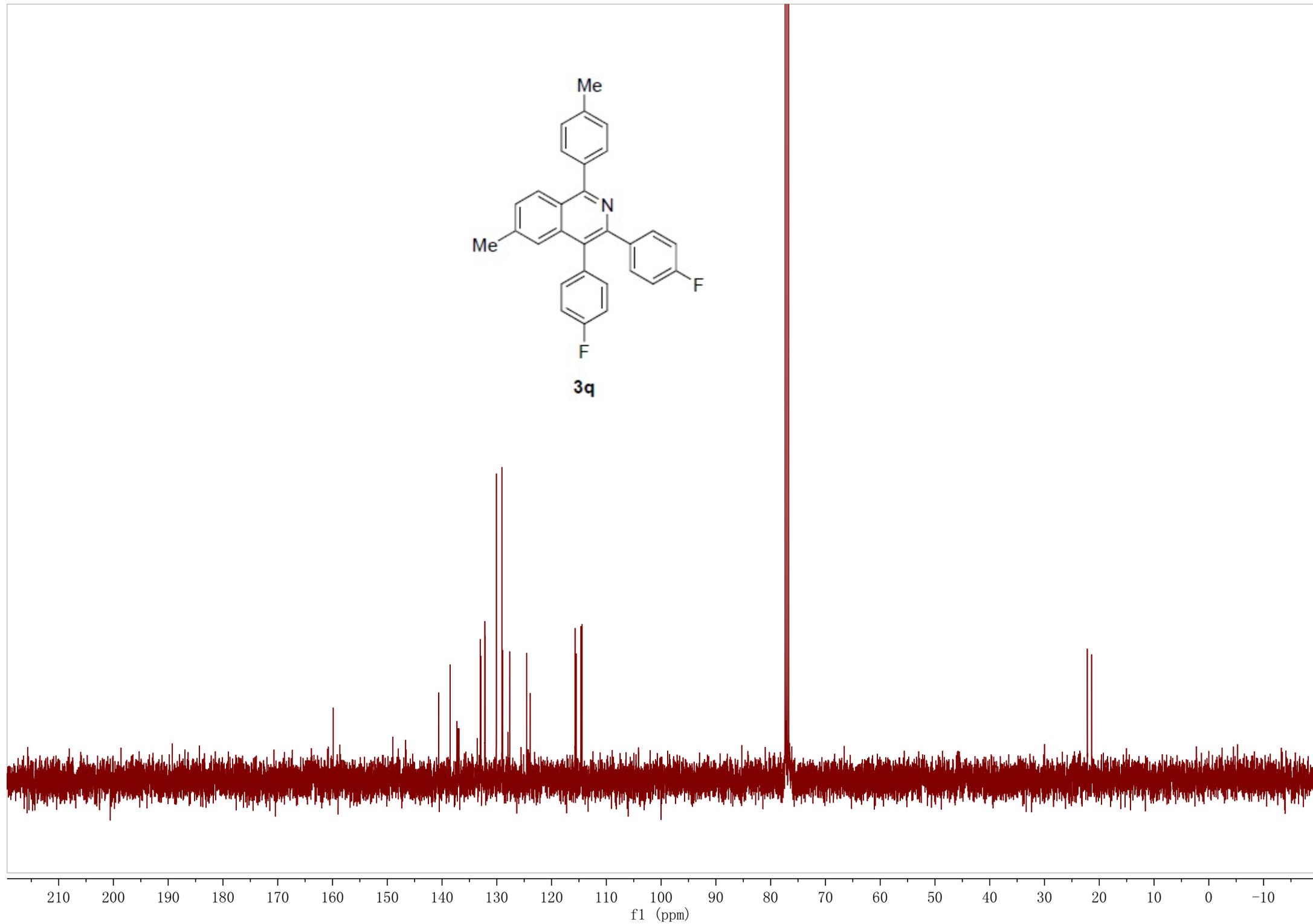
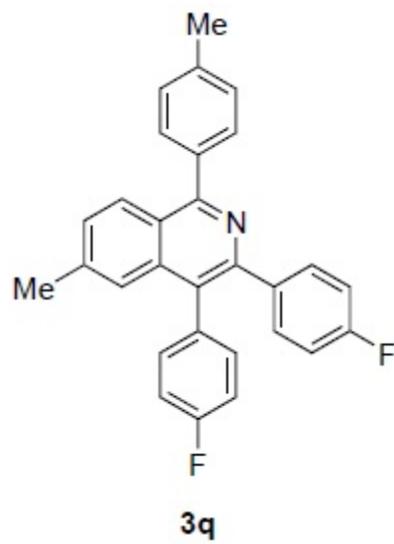


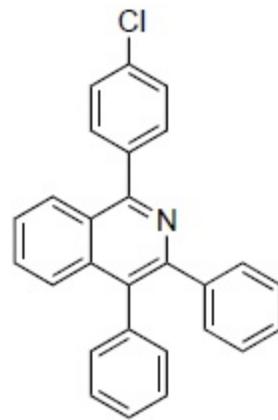




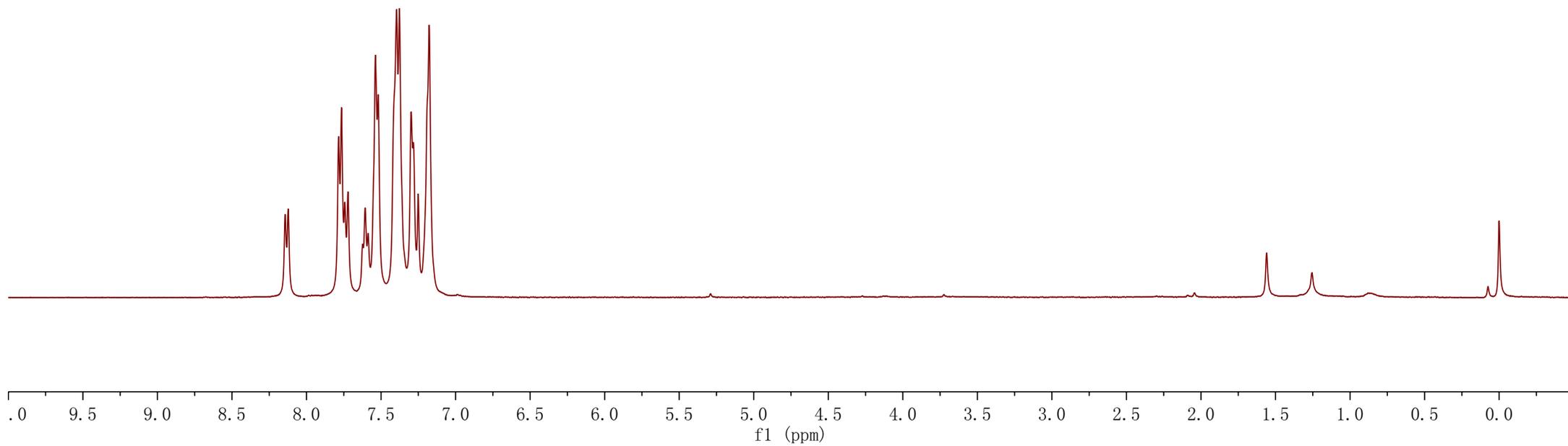


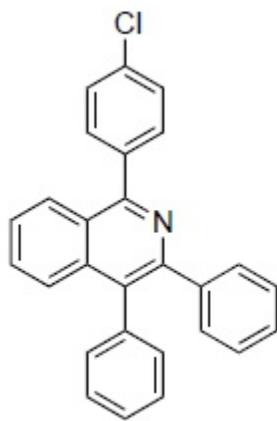




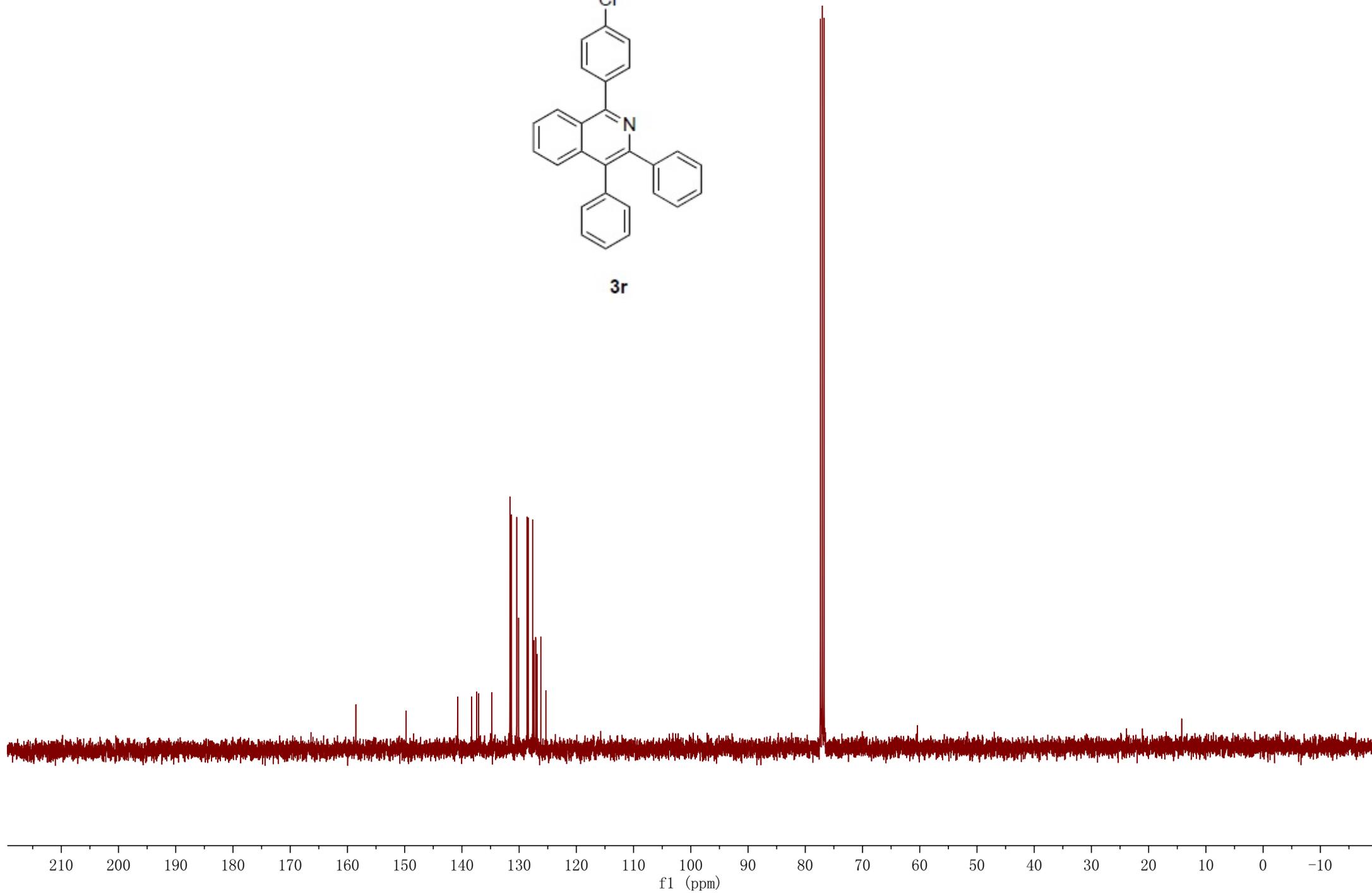


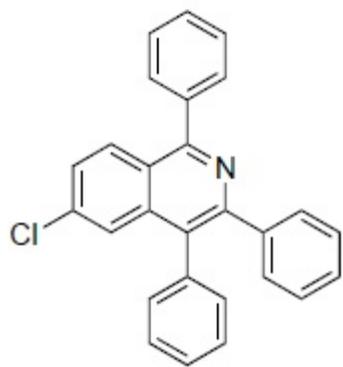
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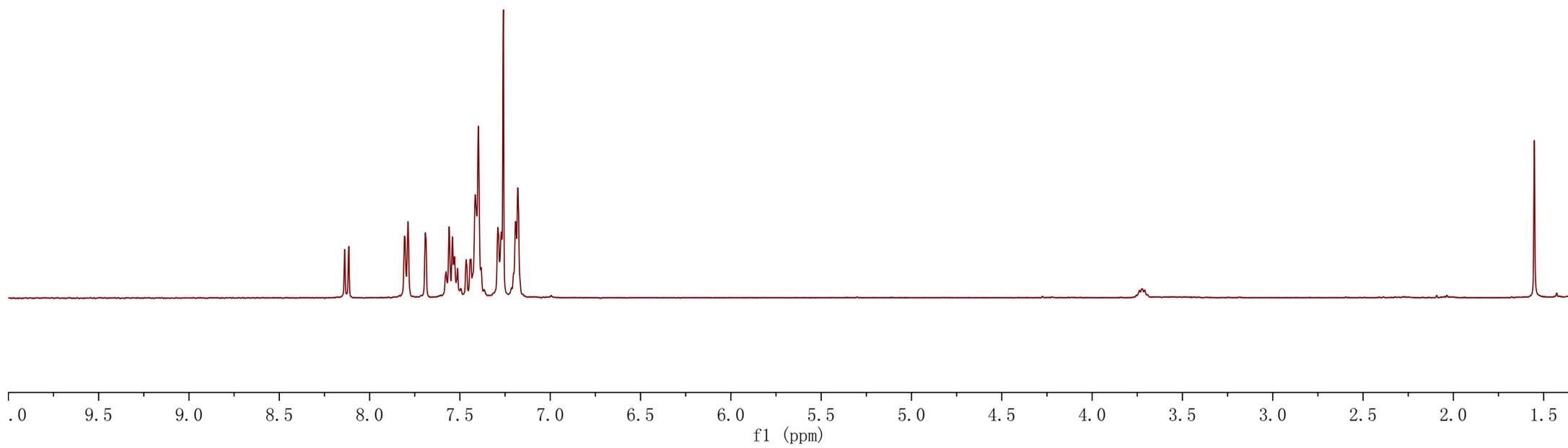


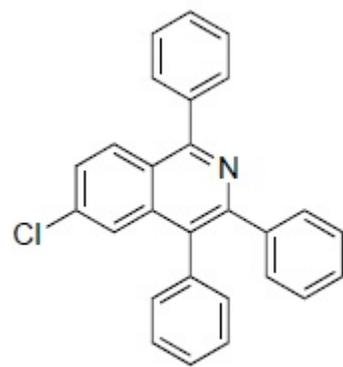
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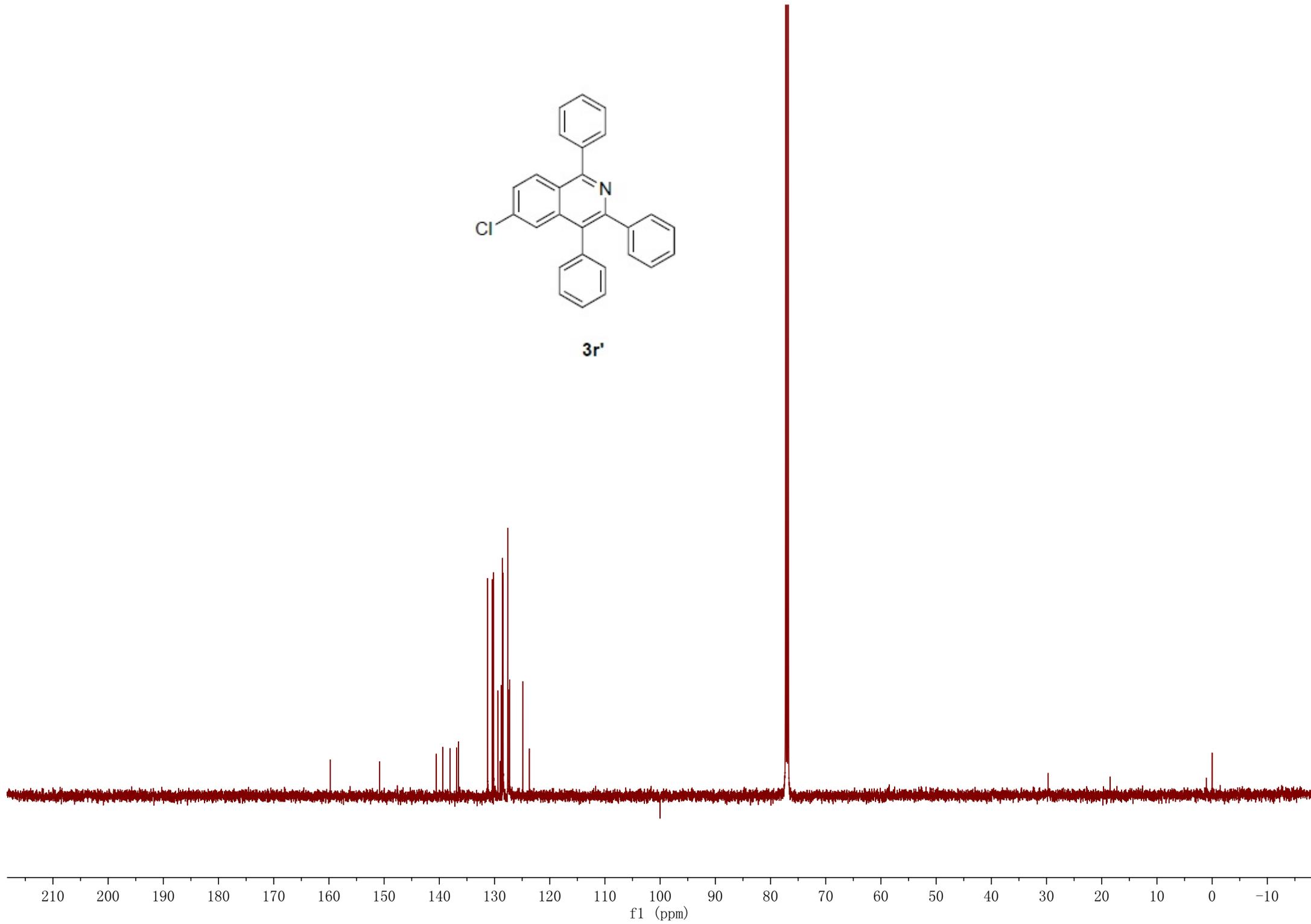


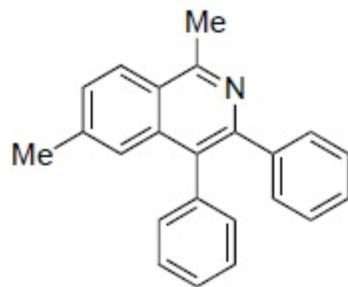
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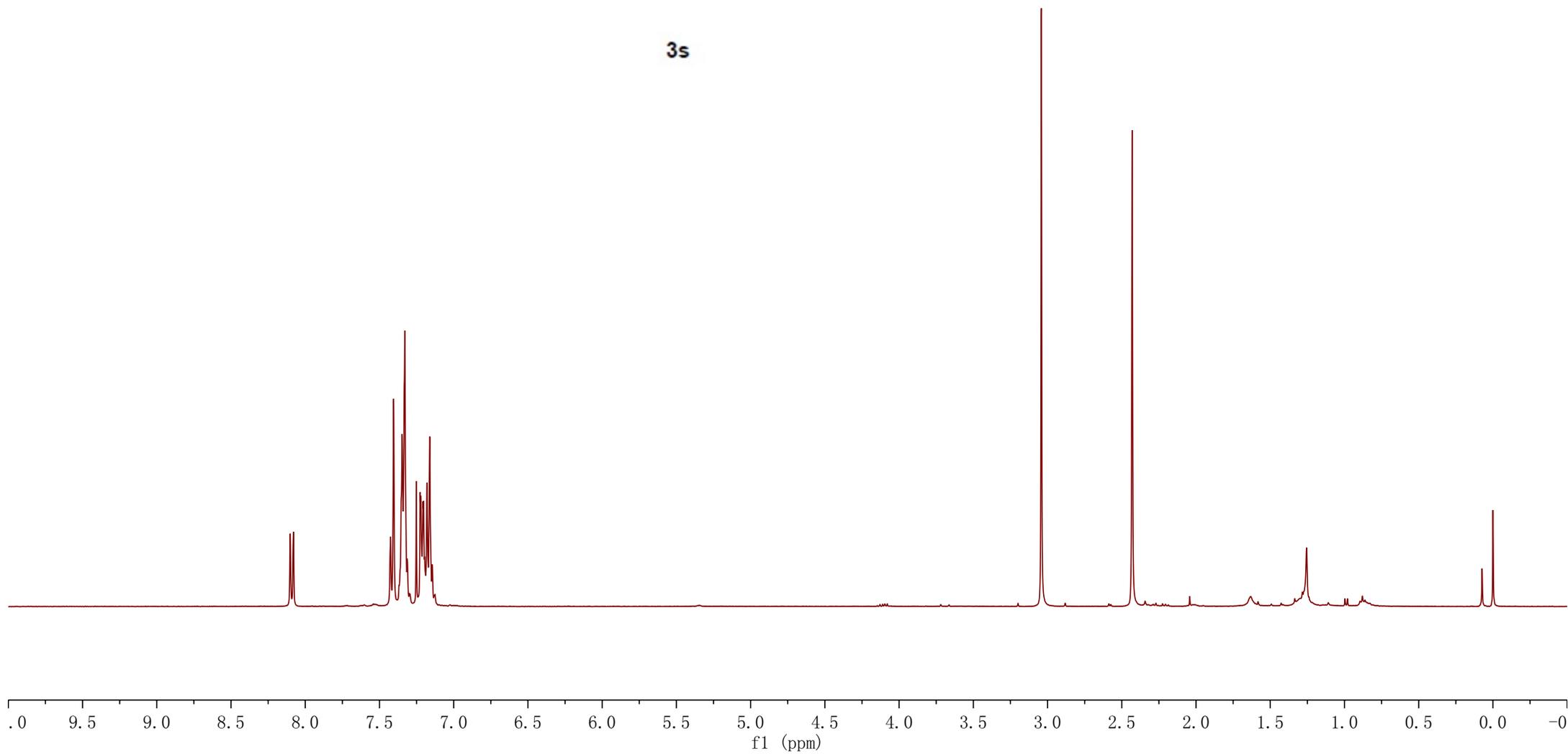


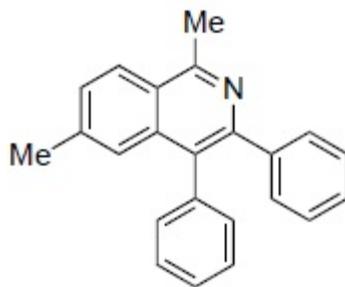
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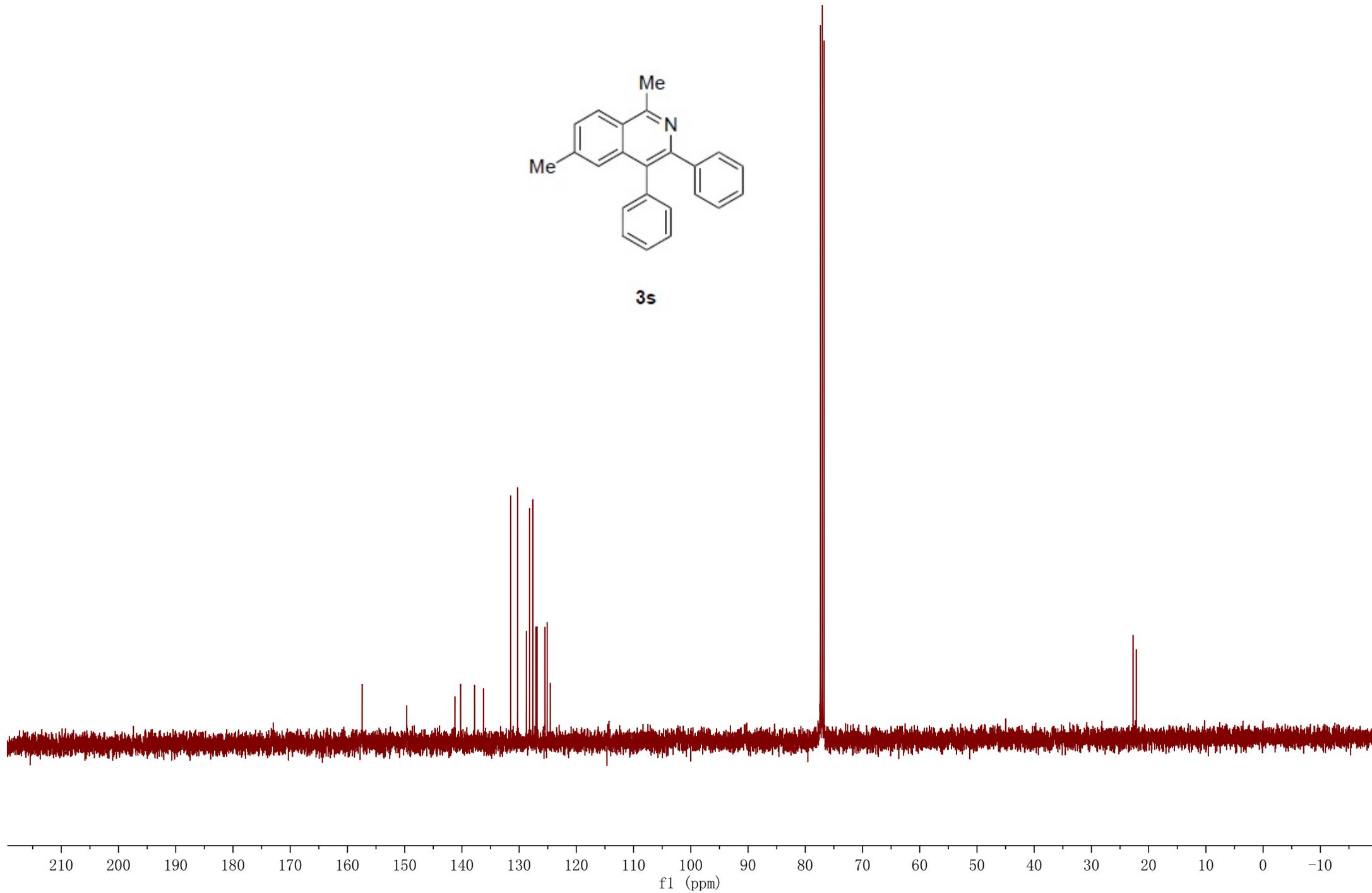


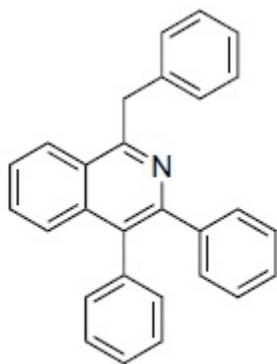
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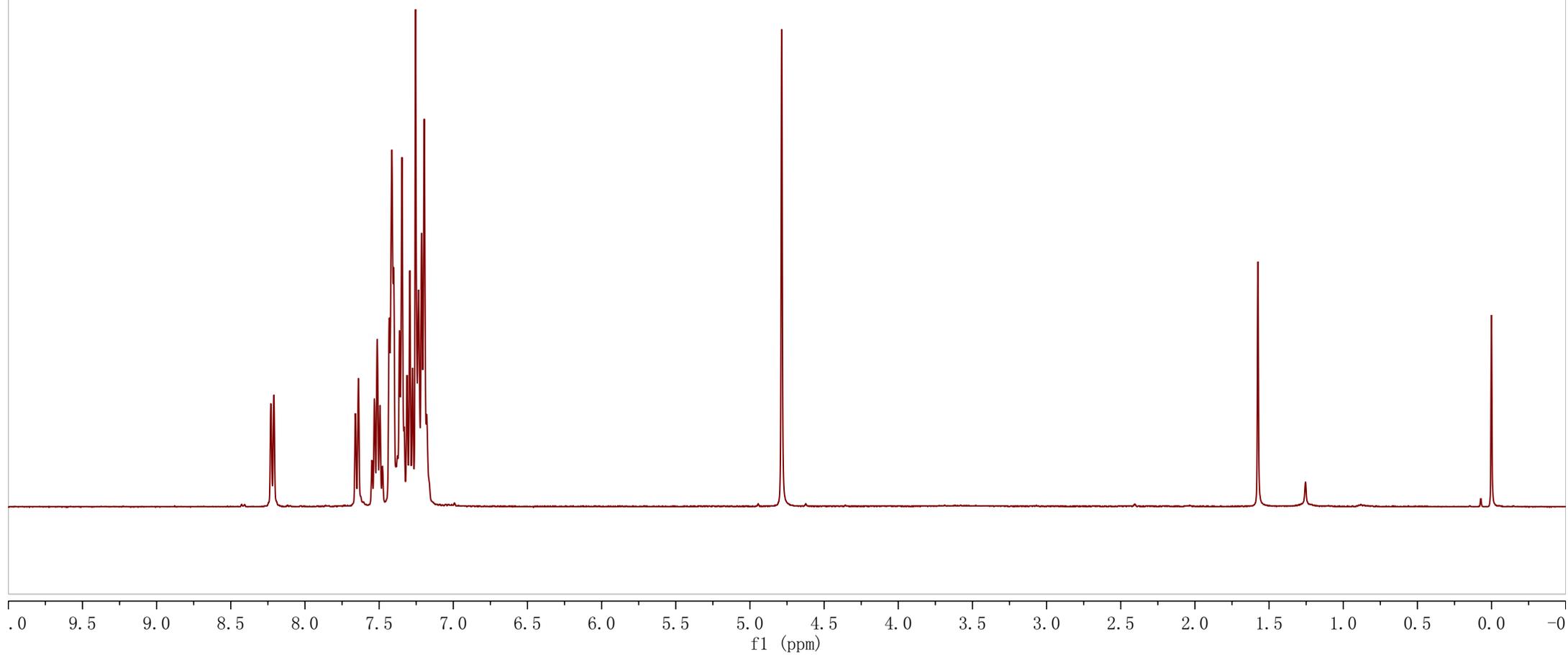


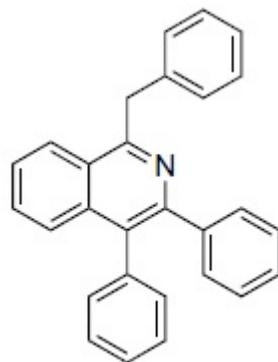
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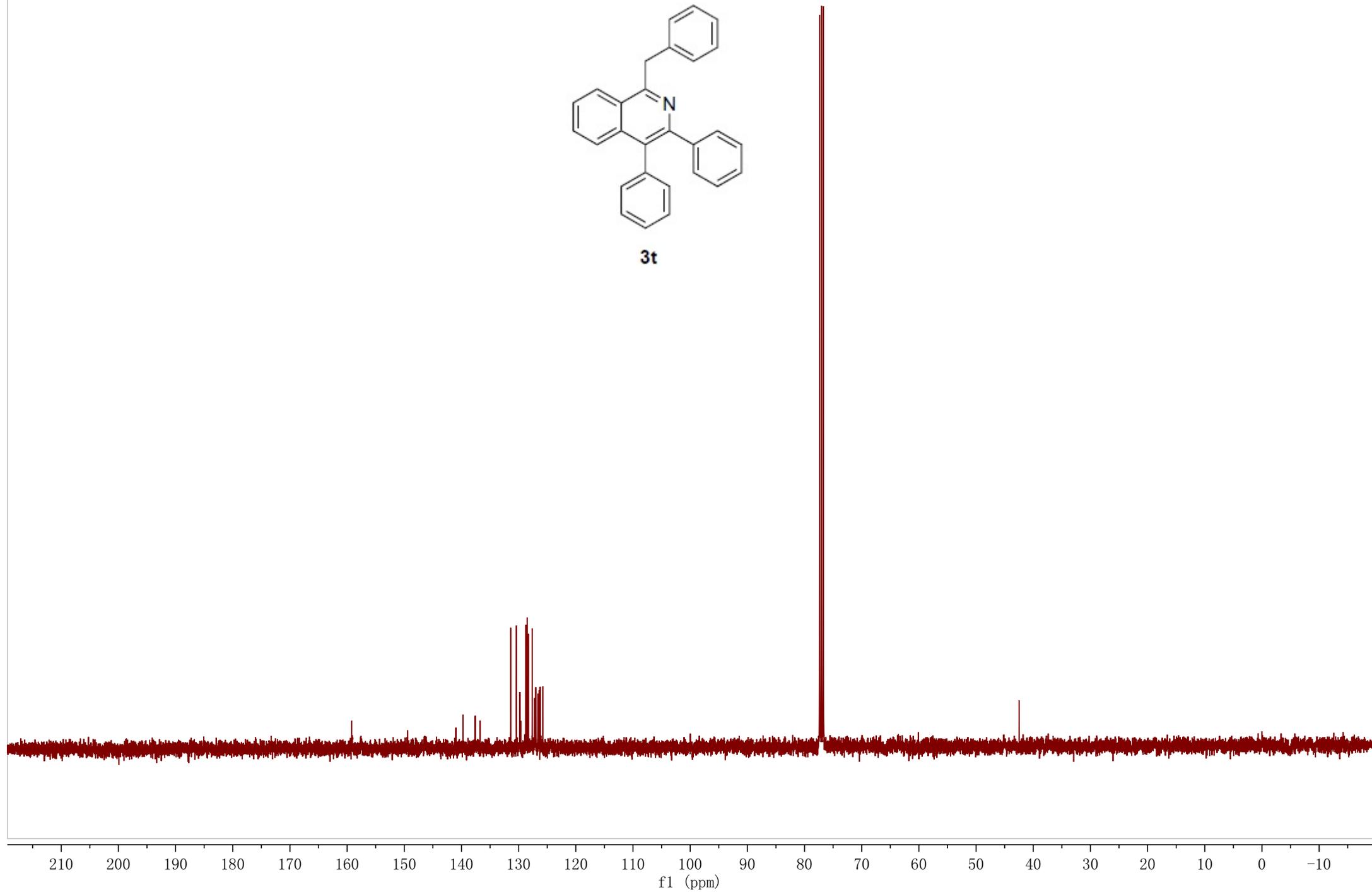


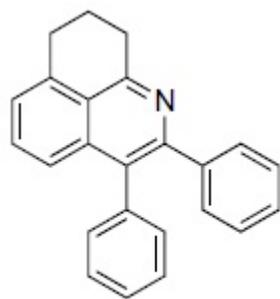
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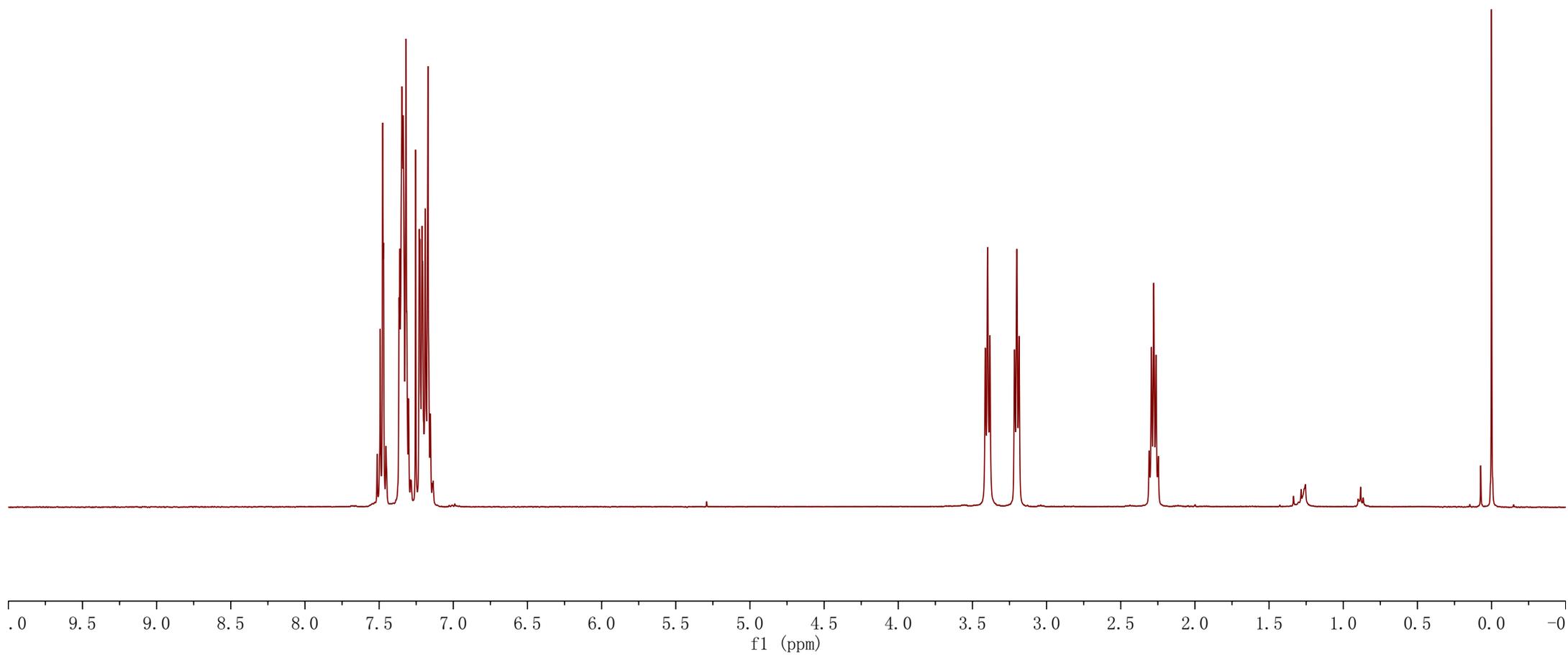


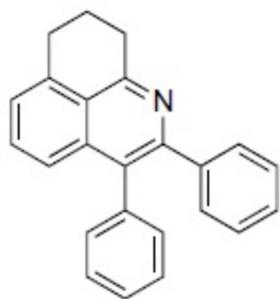
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3u





3u

