

H₈-BINOL Chiral Imidodiphosphoric Acids Catalyzed Highly Enantioselective Aza-Friedel–Crafts Reactions of Pyrroles and Enamides/Imines

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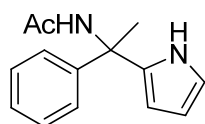
1. General information:

The solvents used were purified by distillation over the drying agents indicated and were transferred under argon: toluene (Na), 1,4-dioxane (Na). All other reagents were used without purification as commercially available. All reactions were monitored by thin layer chromatography. Purification of reaction products were carried out by flash chromatography on silica gel. Chemical yields refer to pure isolated substances. The melting point was recorded on a melting point apparatus (MPA100, Stanford Research Systems, Inc.). Optical rotations were measured using a 0.8 mL cell with a 1 cm path length on Jasco-P-2000 digital polarimeter and concentrations (c) were reported in $\text{g} \times (100 \text{ mL})^{-1}$. ^1H NMR, ^{13}C NMR and ^{31}P NMR spectra were recorded on Bruker 300 MHz. Chemical shifts of ^1H and ^{13}C signals were given in δ relative to the signal of tetramethylsilane (TMS). The following abbreviations are used: s, singlet, d, doublet, t, triplet, q, quartet, quint, quintuplet, m, multiplet, br, broad. IR spectra were recorded on Nicolet Avatar 360 FT-IR spectrometer. High-resolution mass spectral analysis (HRMS) data were measured on a Bruker ApexII mass spectrometer by means of the ESI technique. Analytical HPLC was recorded on a HPLC machine equipped with Agilent 1100 series quaternary pump with a UV diode array detector. The chiral stationary phase was Daicel Chiralpak AD-H, AS-H and OD-H column.

2. General Procedures and Characterization Data for Compounds 4a-4l

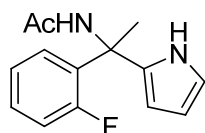
General procedure for enamides: Enamide **3** (0.1 mmol), imidodiphosphoric acid **1d** (2.4 mg, 0.002 mmol, 2 mol %), 5 Å molecular sieves (90 mg) and 1,4-dioxane (1 mL) were loaded into test tube, the solution was stirred for 5 minutes at room temperature and pyrrole **2** (0.11 mmol) was added. After the reaction was complete (monitored by TLC), the mixture was purified by silica gel chromatography (ethyl acetate/petroleum ether = 1/3) directly to afford the product **4**.

(R)-N-[1-phenyl-1-(pyrrol-2-yl)ethyl]acetamide (4a)



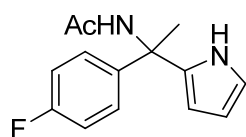
Reaction time: 2 h, white solid, 90% yield, mp: 159-160 °C, $[\alpha]_D^{20} = 45.4$ ($c = 1$, Acetone), 96% *ee* [Daicel Chiralpak AS-H column, *n*-hexane/ethanol = 95 : 5, 1.0 mL/min, $\lambda = 220$ nm, $t(\text{major}) = 9.82$ min, $t(\text{minor}) = 16.50$ min]; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 10.05 (br, 1H), 7.36 – 7.23 (m, 6H), 7.13 – 7.03 (m, 2H), 6.84 – 6.76 (m, 1H), 6.36 – 6.08 (m, 3H), 2.09 (s, 3H), 1.88 (s, 3H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 170.8, 145.4, 135.7, 128.1, 126.7, 125.1, 118.0, 106.3, 58.1, 30.7, 23.9. HRMS(ESI+) m/z calcd for $\text{C}_{14}\text{H}_{17}\text{N}_2\text{O}$ ($[\text{M}+\text{H}]^+$) : 229.1335, found: 229.1332.

(R)-N-[1-(2-fluorophenyl)-1-(pyrrol-2-yl)ethyl]acetamide (4b)



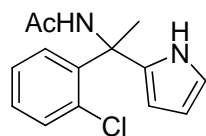
Reaction time: 8 h, white solid, 94% yield, mp: 162-163 °C, $[\alpha]_D^{20} = 146$ ($c = 1$, Acetone), 94% *ee* [Daicel Chiralpak AS-H column, *n*-hexane/ethanol = 93 : 7, 1.0 mL/min, $\lambda = 220$ nm, $t(\text{major}) = 7.57$ min, $t(\text{minor}) = 9.10$ min]; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 10.21 (br, 1H), 7.75 – 7.48 (m, 1H), 7.04 – 6.88 (m, 2H), 6.79 (s, 1H), 6.54 (t, $J = 8.4$ Hz, 1H), 6.35 (d, $J = 18.3$ Hz, 1H), 6.17 (s, 2H), 1.99 (s, 3H), 1.93 (s, 3H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 171.4, 135.2, 131.0, 129.4, 128.9, 124.0, 118.3, 116.2, 115.9, 106.6, 106.2, 56.7, 27.3, 23.7. HRMS(ESI+) m/z calcd for $\text{C}_{14}\text{H}_{16}\text{FN}_2\text{O}$ ($[\text{M}+\text{H}]^+$) : 247.1241, found: 247.1240.

(R)-N-[1-(4-fluorophenyl)-1-(pyrrol-2-yl)ethyl]acetamide (4c)



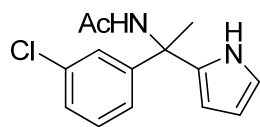
Reaction time: 8 h, white solid, 94% yield, mp: 126-127 °C, $[\alpha]_D^{20} = 48.8$ ($c = 1$, Acetone), 95% *ee* [Daicel Chiralpak AS-H column, *n*-hexane/ethanol = 93 : 7, 1.0 mL/min, $\lambda = 220$ nm, $t(\text{major}) = 6.80$ min, $t(\text{minor}) = 12.33$ min]; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 9.96 (br, 1H), 7.01 – 6.85 (m, 4H), 6.73 (s, 1H), 6.39 – 6.29 (m, 1H), 6.21 – 6.09 (m, 2H), 1.99 (s, 3H), 1.77 (s, 3H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 170.8, 163.2, 135.4, 126.9, 126.8, 118.3, 115.1, 114.8, 106.4, 57.8, 31.0, 24.0. HRMS(ESI+) m/z calcd for $\text{C}_{14}\text{H}_{16}\text{FN}_2\text{O}$ ($[\text{M}+\text{H}]^+$) : 247.1241, found: 247.1241.

(R)-N-[1-(2-chlorophenyl)-1-(pyrrol-2-yl)ethyl]acetamide (4d)



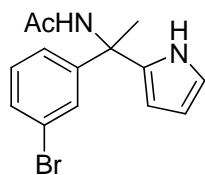
Reaction time: 8 h, white solid, 85% yield, mp: 175-176 °C, $[\alpha]_{\text{D}}^{20} = 202.8$ ($c = 1$, Acetone), 93% *ee* [Daicel Chiralpak AS-H column, *n*-hexane/ethanol = 93 : 7, 1.0 mL/min, $\lambda = 220$ nm, $t(\text{major}) = 6.15$ min, $t(\text{minor}) = 8.48$ min]; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 10.14 (br, 1H), 7.29 (dd, $J = 7.8, 1.5$ Hz, 1H), 7.16 – 6.98 (m, 2H), 6.81 – 6.72 (m, 1H), 6.54 (dd, $J = 7.8, 1.8$ Hz, 1H), 6.23 (br, 1H), 6.19 – 6.07 (m, 2H), 2.02 (s, 3H), 1.98 (s, 3H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 171.3, 141.4, 136.0, 131.5, 130.9, 130.0, 128.5, 127.0, 118.3, 106.6, 106.4, 58.6, 25.8, 23.5. HRMS(ESI+) m/z calcd for $\text{C}_{14}\text{H}_{16}\text{ClN}_2\text{O}$ ($[\text{M}+\text{H}]^+$): 263.0946, found: 263.0944.

(R)-N-[1-(3-chlorophenyl)-1-(pyrrol-2-yl)ethyl]acetamide (4e)



Reaction time: 8 h, white solid, 95% yield, mp: 123-124 °C, $[\alpha]_{\text{D}}^{20} = 55.2$ ($c = 1$, Acetone), 96% *ee* [Daicel Chiralpak AS-H column, *n*-hexane/ethanol = 95 : 5, 1.0 mL/min, $\lambda = 220$ nm, $t(\text{major}) = 9.08$ min, $t(\text{minor}) = 16.98$ min]; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 9.92 (br, 1H), 7.18 – 7.09 (m, 2H), 7.01 – 6.94 (m, 1H), 6.90 – 6.80 (m, 1H), 6.76 – 6.67 (m, 1H), 6.22 (br, 1H), 6.19 – 6.09 (m, 2H), 2.01 (s, 3H), 1.78 (s, 3H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 171.0, 139.9, 135.2, 133.1, 131.2, 130.8, 130.6, 126.8, 118.2, 106.4, 106.3, 57.9, 25.3, 23.0. HRMS(ESI+) m/z calcd for $\text{C}_{14}\text{H}_{16}\text{ClN}_2\text{O}$ ($[\text{M}+\text{H}]^+$): 263.0946, found: 263.0943.

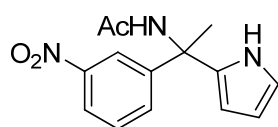
(R)-N-[1-(3-bromophenyl)-1-(pyrrol-2-yl)ethyl]acetamide(4f)



Reaction time: 5 h, white solid, 92% yield, m.p. 113-114 °C, $[\alpha]_{\text{D}}^{20} = 63.4$ ($c = 1$, Acetone), 95% *ee* [Daicel Chiralpak AS-H column, *n*-hexane/ethanol = 93 : 7, 1.0 mL/min, $\lambda = 220$ nm, $t(\text{major}) = 7.40$ min, $t(\text{minor}) = 13.62$ min]; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 9.94 (br, 1H), 7.39 – 7.30 (m, 1H), 7.20 – 7.07 (m, 2H), 6.97 – 6.89 (m, 1H), 6.79 – 6.73 (m, 1H), 6.27 (br, 1H),

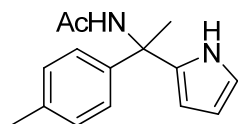
6.23 – 6.13 (m, 2H), 2.04 (s, 3H), 1.80 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 171.0, 148.1, 134.9, 130.1, 129.9, 128.4, 124.1, 122.6, 118.6, 106.8, 106.6, 57.9, 30.9, 24.1. HRMS(ESI+) m/z calcd for $\text{C}_{14}\text{H}_{16}\text{BrN}_2\text{O}$ ($[\text{M}+\text{H}]^+$) : 307.0441, found: 307.0439.

(R)-N-[1-(3-nitrophenyl)-1-(pyrrol-2-yl)ethyl]acetamide (4g)



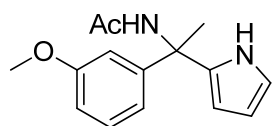
Reaction time: 8 h, yellow solid, 95% yield, mp: 135-136 °C, $[\alpha]_{\text{D}}^{20} = 64.2$ ($c = 1$, Acetone), 98% *ee* [Daicel Chiralpak AS-H column, *n*-hexane/ethanol = 93 : 7, 1.0 mL/min, $\lambda = 220$ nm, $t(\text{major}) = 12.08$ min, $t(\text{minor}) = 23.38$ min]; ^1H NMR (300 MHz, CDCl_3) δ 9.88 (br, 1H), 8.12 – 8.02 (m, 1H), 7.97 (t, $J = 2.1$ Hz, 1H), 7.41 (t, $J = 8.1$ Hz, 1H), 7.25 – 7.21 (m, 1H), 6.81 – 6.73 (m, 1H), 6.47 (s, 1H), 6.28 – 6.21 (m, 1H), 6.21 – 6.13 (m, 1H), 2.06 (s, 3H), 1.83 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 171.1, 148.3, 134.1, 131.6, 129.1, 121.8, 120.0, 118.7, 107.0, 106.7, 57.7, 30.5, 23.8. HRMS(ESI+) m/z calcd for $\text{C}_{14}\text{H}_{16}\text{N}_3\text{O}_3$ ($[\text{M}+\text{H}]^+$) : 274.1186, found: 274.1185.

(R)-N-[1-(4-methylphenyl)-1-(pyrrol-2-yl)ethyl]acetamide (4h)



Reaction time: 5 h, white solid, 86% yield, mp: 137-138 °C, $[\alpha]_{\text{D}}^{20} = 46.0$ ($c = 1$, Acetone), 91% *ee* [Daicel Chiralpak AS-H column, *n*-hexane/ethanol = 93 : 7, 1.0 mL/min, $\lambda = 220$ nm, $t(\text{major}) = 6.12$ min, $t(\text{minor}) = 12.23$ min]; ^1H NMR (300 MHz, CDCl_3) δ 9.99 (br, 1H), 7.07 (d, $J = 8.1$ Hz, 2H), 6.92 (d, $J = 8.4$ Hz, 2H), 6.73 (q, $J = 2.4$ Hz, 1H), 6.22 (br, 1H), 6.18 – 6.05 (m, 2H), 2.30 (s, 3H), 2.01 (s, 3H), 1.81 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 170.9, 142.6, 136.5, 136.0, 129.0, 125.2, 118.1, 106.4, 106.3, 58.2, 31.0, 24.2, 21.1. HRMS(ESI+) m/z calcd for $\text{C}_{15}\text{H}_{19}\text{N}_2\text{O}$ ($[\text{M}+\text{H}]^+$) : 243.1492, found: 243.1493.

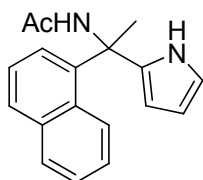
(R)-N-[1-(3-methoxyphenyl)-1-(pyrrol-2-yl)ethyl]acetamide (4i)



Reaction time: 5 h, white solid, 93% yield, mp: 133-134 °C, $[\alpha]_{\text{D}}^{20} = 54.8$ ($c = 1$, Acetone), 93% *ee* [Daicel Chiralpak AS-H

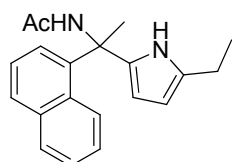
column, *n*-hexane/ethanol = 93 : 7, 1.0 mL/min, $\lambda = 220$ nm, $t(\text{major}) = 8.47$ min, $t(\text{minor}) = 13.72$ min]; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 10.01 (br, 1H), 7.19 (t, $J = 8.1$ Hz, 1H), 6.80 – 6.71 (m, 2H), 6.65 (dd, $J = 7.8, 1.8$ Hz, 1H), 6.60 – 6.55 (m, 1H), 6.34 – 6.24 (m, 1H), 6.22 – 6.10 (m, 2H), 3.74 (s, 3H), 2.02 (s, 3H), 1.83 (s, 3H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 170.5, 159.2, 147.1, 135.3, 128.9, 117.8, 117.5, 111.8, 111.1, 106.1, 57.9, 54.8, 30.5, 23.8. HRMS(ESI+) m/z calcd for $\text{C}_{15}\text{H}_{19}\text{N}_2\text{O}_2$ ($[\text{M}+\text{H}]^+$) : 259.1441, found: 259.1438.

(R)-N-[1-(naphthalene-1-yl)-1-(pyrrol-2-yl)ethyl]acetamide (4j)



Reaction time: 8 h, white solid, 96% yield, mp: 128-129 °C, $[\alpha]_{\text{D}}^{20} = 57.0$ ($c = 1$, Acetone), 94% *ee* [Daicel Chiralpak AS-H column, *n*-hexane/ethanol = 90 : 10, 1.0 mL/min, $\lambda = 220$ nm, $t(\text{minor}) = 5.63$ min, $t(\text{major}) = 6.97$ min]; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 10.01 (br, 1H), 8.06 (d, $J = 7.8$ Hz, 1H), 7.90 – 7.74 (m, 2H), 7.54 – 7.29 (m, 4H), 6.79 – 6.64 (m, 1H), 6.43 – 6.23 (m, 1H), 6.08 (q, $J = 3.0$ Hz, 1H), 5.90 (br, 1H), 2.19 (s, 3H), 1.96 (s, 3H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 170.5, 139.2, 136.9, 134.6, 130.1, 129.3, 128.8, 128.2, 125.3, 125.2, 125.1, 124.8, 117.5, 106.5, 105.7, 59.2, 27.6, 23.7. HRMS(ESI+) m/z calcd for $\text{C}_{18}\text{H}_{19}\text{N}_2\text{O}$ ($[\text{M}+\text{H}]^+$) : 279.1492, found: 279.1492.

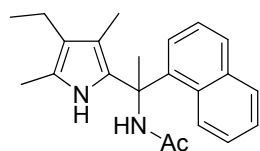
(R)-N-[1-(naphthalene-1-yl)-1-(5-ethyl-1H-pyrrol-2-yl)ethyl]acetamide (4k)



Reaction time: 12 h, white solid, 83% yield, mp: 70-71 °C, $[\alpha]_{\text{D}}^{20} = 46.0$ ($c = 1$, Acetone), 91% *ee* [Daicel Chiralpak AS-H column, *n*-hexane/ethanol = 98 : 2, 1.0 mL/min, $\lambda = 220$ nm, $t(\text{major}) = 16.42$ min, $t(\text{minor}) = 42.05$ min]; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 9.77 (br, 1H), 8.17 (d, $J = 8.0$ Hz, 1H), 7.89 (d, $J = 7.9$ Hz, 1H), 7.81 (d, $J = 7.4$ Hz, 1H), 7.43 (dd, $J = 21.5, 8.6$ Hz, 3H), 6.30 (s, 1H), 5.82 (s, 2H), 2.61 (q, 2H), 2.22 (s, 3H), 2.02 (s, 3H), 1.23 (t, $J = 7.6$ Hz, 3H); $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 170.54, 139.61, 135.38, 134.84, 134.53, 130.48, 129.50, 128.97, 125.52, 125.45, 125.27, 125.09,

124.98, 105.97, 102.57, 59.60, 27.97, 24.14, 20.97, 13.55. HRMS(ESI+) m/z calcd for $C_{20}H_{23}N_2O$ ($[M+H]^+$) : 307.1805, found: 307.1802.

(R)-N-[1-(naphthalene-1-yl)-1-(4-ethyl-3,5-dimethyl-1H-pyrrol-2-yl)ethyl]acetamide (4l)

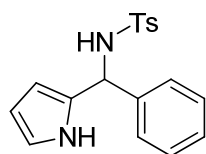


Reaction time: 12 h, white solid, 80% yield, mp: 71-72 °C, $[\alpha]_D^{20} = 4.0$ ($c = 1$, Acetone), 77% *ee* [Daicel Chiralpak AS-H column, *n*-hexane/ethanol = 97 : 3, 1.0 mL/min, $\lambda = 220$ nm, $t(\text{major}) = 8.35$ min, $t(\text{minor}) = 16.98$ min]; ^1H NMR (400 MHz, CDCl_3) δ 8.77 (br, 1H), 8.00 – 7.83 (m, 3H), 7.66 (d, $J = 6.7$ Hz, 1H), 7.57 – 7.41 (m, 2H), 7.40 – 7.31 (m, 1H), 6.26 (s, 1H), 2.36 (s, 3H), 2.29 (q, $J = 15.1, 8.2$ Hz, 2H), 2.13 (s, 3H), 2.00 (s, 3H), 1.40 (s, 3H), 0.97 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 170.26, 139.55, 134.92, 129.26, 129.16, 128.71, 125.65, 125.56, 125.22, 125.00, 121.09, 112.85, 60.86, 27.07, 24.61, 17.46, 15.78, 11.16, 9.80. HRMS(ESI+) m/z calcd for $C_{22}H_{27}N_2O$ ($[M+H]^+$) : 335.2118, found: 335.2115.

3. General Procedures and Characterization Data for Compounds 6a-6r

General procedure for aldimines: Aldimine **5** (0.1 mmol), imidodiphosphoric acid **1b** (100 μL of 4.6 mg/mL solution in toluene, 0.46 mg, 0.0003 mmol, 0.3 mol %), 5Å molecular sieves (5 mg) and toluene (0.3 mL) were loaded into test tube, the solution was stirred for 15 minutes at -60 °C and pyrrole **2** (0.11 mmol) was added. After the reaction was complete (monitored by TLC), the mixture was purified by silica gel chromatography (ethyl acetate/petroleum ether = 1/3) directly to afford the product **6**.

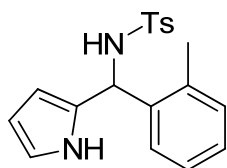
(R)-N-(pyrrol-2-yl-phenylmethyl)-4-methylbenzenesulfonamide (6a)



Reaction time: 9 h, white solid, 90% yield, mp: 120-121 °C, $[\alpha]_D^{20} = -8.8$ ($c = 1.0$, Acetone), 99% *ee* [Daicel Chiralpak AD-H column, *n*-hexane/*i*-PrOH = 80 : 20, 1.0 mL/min, $\lambda = 254$ nm, $t(\text{major}) =$

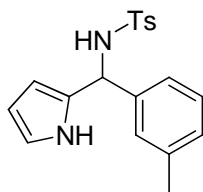
13.77 min, t(minor) = 21.35 min]; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 8.55 (br, 1H), 7.58 (d, $J = 8.4$ Hz, 1H), 7.34 – 7.03 (m, 8H), 6.79 – 6.65 (m, 1H), 6.11 – 5.98 (m, 1H), 5.65 – 5.58 (m, 1H), 5.53 (d, $J = 7.8$ Hz, 1H), 5.21 (d, $J = 7.5$ Hz, 1H), 2.38 (s, 3H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 143.6, 138.8, 137.1, 130.8, 129.6, 128.7, 128.1, 127.4, 127.3, 118.8, 108.4, 108.2, 56.0, 21.6. HRMS(ESI+) m/z calcd for $\text{C}_{18}\text{H}_{19}\text{N}_2\text{O}_2\text{S}$ ($[\text{M}+\text{H}]^+$) : 327.1162, found: 327.1161.

(R)-N-[pyrrol-2-yl-(2-methylphenyl)methyl]-4-methylbenzenesulfonamide (6b)



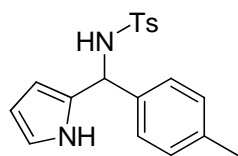
Reaction time: 24 h, colorless gum, 94% yield, $[\alpha]_{\text{D}}^{20} = -13.6$ (c 1.0, Acetone), 98% *ee* [Daicel Chiralpak AD-H column, *n*-hexane/*i*-PrOH = 60 : 40, 1.0 mL/min, $\lambda = 254$ nm, t(major) = 7.65 min, t(minor) = 14.12 min]; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 8.53 (br, 1H), 7.58 (d, $J = 8.4$ Hz, 2H), 7.19 – 7.00 (m, 6H), 6.76 – 6.66 (m, 1H), 6.05 – 5.93 (m, 1H), 5.72 (d, $J = 7.5$ Hz, 1H), 5.54 – 5.46 (m, 1H), 5.22 – 5.09 (m, 1H), 2.37 (s, 3H), 2.05 (s, 3H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 143.5, 137.1, 136.9, 135.5, 130.5, 130.2, 129.5, 127.8, 127.1, 127.0, 126.3, 118.7, 108.3, 108.1, 52.4, 21.6, 19.1. HRMS(ESI+) m/z calcd for $\text{C}_{19}\text{H}_{21}\text{N}_2\text{O}_2\text{S}$ ($[\text{M}+\text{H}]^+$) : 341.1318, found: 341.1316.

(R)-N-[pyrrol-2-yl-(3-methylphenyl)methyl]-4-methylbenzenesulfonamide (6c)



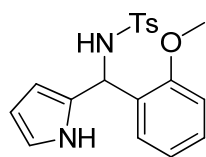
Reaction time: 24 h, colorless gum, 97% yield, $[\alpha]_{\text{D}}^{20} = -8.3$ (c 1.0, Acetone), 98% *ee* [Daicel Chiralpak AD-H column, *n*-hexane/*i*-PrOH = 60 : 40, 1.0 mL/min, $\lambda = 254$ nm, t(major) = 7.13 min, t(minor) = 9.27 min]; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 8.52 (br, 1H), 7.57 (d, $J = 8.2$ Hz, 2H), 7.16 (d, $J = 8.1$ Hz, 2H), 7.10 (d, $J = 7.5$ Hz, 1H), 7.03 (d, $J = 7.3$ Hz, 1H), 6.91 (d, $J = 7.4$ Hz, 1H), 6.84 (s, 1H), 6.72 (s, 1H), 6.04 (dd, $J = 5.7, 2.8$ Hz, 1H), 5.63 (s, 1H), 5.49 (d, $J = 7.8$ Hz, 1H), 5.17 (d, $J = 7.4$ Hz, 1H), 2.38 (s, 3H), 2.21 (s, 3H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 143.14, 138.43, 137.99, 137.08, 130.64, 129.28, 128.46, 128.31, 127.99, 127.07, 124.41, 118.51, 108.10, 107.89, 55.86, 21.41, 21.17. HRMS(ESI+) m/z calcd for $\text{C}_{19}\text{H}_{21}\text{N}_2\text{O}_2\text{S}$ ($[\text{M}+\text{H}]^+$) : 341.1318, found: 341.1316.

(R)-N-[pyrrol-2-yl-(4-methylphenyl)methyl]-4-methylbenzenesulfonamide (6d)



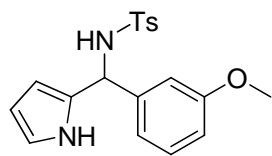
Reaction time: 5 h, colorless gum, 96% yield, $[\alpha]_D^{20} = -6.6$ (*c* 1.0, Acetone), >99% *ee* [Daicel Chiralpak AD-H column, *n*-hexane/*i*-PrOH = 90 : 10, 1.0 mL/min, $\lambda = 254$ nm, $t(\text{major}) = 23.90$ min, $t(\text{minor}) = 27.05$ min]; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 8.55 (br, 1H), 7.59 (d, $J = 8.4$ Hz, 2H), 7.18 (d, $J = 8.4$ Hz, 2H), 7.08 – 6.91 (m, 4H), 6.76 – 6.62 (m, 1H), 6.08 – 5.98 (m, 1H), 5.67 – 5.54 (m, 1H), 5.47 (d, $J = 7.8$ Hz, 1H), 5.08 (d, $J = 7.8$ Hz, 1H), 2.39 (s, 3H), 2.31 (s, 3H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 143.4, 137.7, 137.2, 135.8, 130.9, 129.5, 129.2, 127.3, 127.2, 118.6, 108.3, 108.0, 55.7, 21.6, 21.2. HRMS(ESI+) m/z calcd for $\text{C}_{19}\text{H}_{21}\text{N}_2\text{O}_2\text{S}$ ($[\text{M}+\text{H}]^+$) : 341.1318, found: 341.1318.

(R)-N-[pyrrol-2-yl-(2-methoxyphenyl)methyl]-4-methylbenzenesulfonamide (6e)



Reaction time: 24 h, white solid, 90% yield, mp: 150-151 °C, $[\alpha]_D^{20} = -22.0$ (*c* 1.0, Acetone), 97% *ee* [Daicel Chiralpak AD-H column, *n*-hexane/*i*-PrOH = 60 : 40, 1.0 mL/min, $\lambda = 254$ nm, $t(\text{major}) = 11.47$ min, $t(\text{minor}) = 22.18$ min]; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 8.66 (br, 1H), 7.52 (d, $J = 8.4$ Hz, 2H), 7.18 (td, $J = 7.8, 1.8$ Hz, 1H), 7.07 (d, $J = 8.1$ Hz, 2H), 6.98 (dd, $J = 7.5, 1.8$ Hz, 1H), 6.80 (td, $J = 7.5, 1.1$ Hz, 1H), 6.74 – 6.66 (m, 2H), 6.04 – 5.98 (m, 1H), 5.95 – 5.85 (m, 1H), 5.64 (d, $J = 9.3$ Hz, 1H), 5.60 – 5.54 (m, 1H), 3.66 (s, 3H), 2.33 (s, 3H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 156.6, 143.0, 137.3, 130.7, 129.6, 129.2, 129.2, 127.1, 126.3, 120.9, 118.1, 111.2, 108.1, 106.9, 55.5, 54.1, 21.5. HRMS(ESI+) m/z calcd for $\text{C}_{19}\text{H}_{21}\text{N}_2\text{O}_3\text{S}$ ($[\text{M}+\text{H}]^+$) : 357.1267, found: 357.1267.

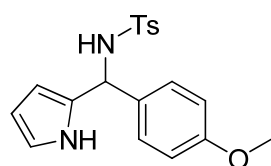
(R)-N-[pyrrol-2-yl-(3-methoxyphenyl)methyl]-4-methylbenzenesulfonamide(6f)



Reaction time: 24 h, white solid, 98% yield, mp: 126-127 °C, $[\alpha]_D^{20} = -13.6$ (*c* 1.0, Acetone), 99% *ee* [Daicel Chiralpak AD-H column, *n*-hexane/*i*-PrOH = 60 : 40, 1.0 mL/min, $\lambda = 254$ nm, $t(\text{major}) = 8.98$ min, $t(\text{minor}) = 11.57$ min]; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 8.53 (br,

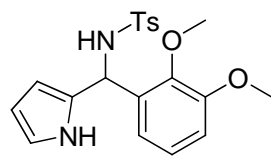
1H), 7.59 (d, $J = 8.4$ Hz, 2H), 7.20 – 7.09 (m, 3H), 6.80 – 6.67 (m, 3H), 6.63 – 6.58 (m, 1H), 6.07 – 5.98 (m, 1H), 5.68 – 5.60 (m, 1H), 5.51 (d, $J = 8.1$ Hz, 1H), 5.23 – 5.10 (m, 1H), 3.69 (s, 3H), 2.39 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 159.6, 143.3, 140.2, 137.1, 130.5, 129.5, 129.4, 127.2, 119.8, 118.7, 113.7, 112.7, 108.2, 108.1, 55.9, 55.1, 21.5. HRMS(ESI+) m/z calcd for $\text{C}_{19}\text{H}_{21}\text{N}_2\text{O}_3\text{S}$ ($[\text{M}+\text{H}]^+$) : 357.1267, found: 357.1266.

(R)-N-[pyrrol-2-yl-(4-methoxyphenyl)methyl]-4-methylbenzenesulfonamide (6g)



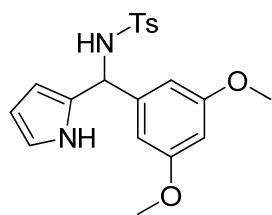
Reaction time: 15 h, white solid, 85% yield, mp: 161-162 °C, $[\alpha]_{\text{D}}^{20} = -3.2$ (c 1.0, Acetone), 98% *ee* [Daicel Chiralpak AD-H column, *n*-hexane/*i*-PrOH = 60 : 40, 1.0 mL/min, $\lambda = 254$ nm, $t(\text{minor}) = 9.62$ min, $t(\text{major}) = 10.78$ min]; ^1H NMR (300 MHz, CDCl_3) δ 8.58 (br, 1H), 7.56 (d, $J = 8.4$ Hz, 2H), 7.16 (d, $J = 8.1$ Hz, 2H), 7.00 (d, $J = 8.7$ Hz, 2H), 6.76 – 6.63 (m, 3H), 6.06 – 5.93 (m, 1H), 5.63 – 5.56 (m, 1H), 5.47 (d, $J = 7.8$ Hz, 1H), 5.25 (d, $J = 7.8$ Hz, 1H), 3.76 (s, 3H), 2.38 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 159.3, 143.4, 137.2, 131.1, 131.0, 129.6, 128.7, 127.3, 118.6, 113.9, 108.3, 108.0, 55.5, 55.4, 21.6. HRMS(ESI+) m/z calcd for $\text{C}_{19}\text{H}_{21}\text{N}_2\text{O}_3\text{S}$ ($[\text{M}+\text{H}]^+$) : 357.1267, found: 357.1266.

(R)-N-[pyrrol-2-yl-(2,3-dimethoxyphenyl)methyl]-4-methylbenzenesulfonamide(6h)



Reaction time: 15 h, white solid, 98% yield, m.p. 139-140 °C, $[\alpha]_{\text{D}}^{20} = -12.2$ (c 1.0, Acetone), 97% *ee* [Daicel Chiralpak AD-H column, *n*-hexane/*i*-PrOH = 60 : 40, 1.0 mL/min, $\lambda = 254$ nm, $t(\text{major}) = 7.52$ min, $t(\text{minor}) = 10.95$ min]; ^1H NMR (300 MHz, CDCl_3) δ 8.72 (br, 1H), 7.58 (d, $J = 8.4$ Hz, 2H), 7.13 – 7.04 (m, 2H), 6.91 – 6.82 (m, 1H), 6.78 (dd, $J = 8.4, 1.5$ Hz, 1H), 6.73 – 6.66 (m, 1H), 6.59 (dd, $J = 7.5, 1.8$ Hz, 1H), 6.05 – 5.94 (m, 2H), 5.63 – 5.48 (m, 2H), 3.81 (s, 3H), 3.53 (s, 3H), 2.33 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 152.6, 146.2, 143.0, 137.0, 132.4, 131.0, 129.2, 127.0, 123.9, 120.7, 117.8, 112.2, 108.1, 106.7, 60.4, 55.7, 53.5, 21.4. HRMS(ESI+) m/z calcd for $\text{C}_{20}\text{H}_{23}\text{N}_2\text{O}_4\text{S}$ ($[\text{M}+\text{H}]^+$) : 387.1373, found: 387.1370.

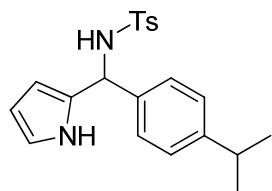
(R)-N-[pyrrol-2-yl-(3,5-dimethoxyphenyl)methyl]-4-methylbenzenesulfonamide(6i)



Reaction time: 48 h, colorless gum, 67% yield, $[\alpha]_D^{20} = -8.0$ (*c* 1.0, Acetone), >99% *ee* [Daicel Chiralpak AD-H column, *n*-hexane/*i*-PrOH = 60 : 40, 1.0 mL/min, $\lambda = 254$ nm, $t(\text{major}) = 8.52$ min, $t(\text{minor}) = 20.10$ min]; $^1\text{H NMR}$ (300 MHz, CDCl_3)

δ 8.52 (br, 1H), 7.59 (d, $J = 8.4$ Hz, 2H), 7.18 (d, $J = 8.1$ Hz, 2H), 6.78 – 6.66 (m, 1H), 6.31 (t, $J = 2.1$ Hz, 1H), 6.24 (d, $J = 2.1$ Hz, 2H), 6.10 – 5.99 (m, 1H), 5.76 – 5.65 (m, 1H), 5.46 (d, $J = 7.8$ Hz, 1H), 5.29 – 5.11 (m, 1H), 3.67 (s, 3H), 2.39 (s, 3H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 160.8, 143.4, 140.8, 137.2, 130.3, 129.5, 127.2, 118.7, 108.3, 108.0, 105.4, 100.2, 56.1, 55.3, 21.5. HRMS(ESI+) m/z calcd for $\text{C}_{20}\text{H}_{23}\text{N}_2\text{O}_4\text{S}$ ($[\text{M}+\text{H}]^+$) : 387.1373, found: 387.1371.

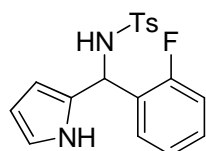
(R)-N-[pyrrol-2-yl-(4-isopropylphenyl)methyl]-4-methylbenzenesulfonamide (6j)



Reaction time: 15 h, colorless gum, 99% yield, $[\alpha]_D^{20} = -5.0$ (*c* 1.0, Acetone), 99% *ee* [Daicel Chiralpak OD-H column, *n*-hexane/*i*-PrOH = 90 : 10, 1.0 mL/min, $\lambda = 254$ nm, $t(\text{major}) = 6.45$ min, $t(\text{minor}) = 9.67$ min]; $^1\text{H NMR}$ (300 MHz, CDCl_3)

δ 8.50 (br, 1H), 7.57 (d, $J = 8.4$ Hz, 2H), 7.15 (d, $J = 7.8$ Hz, 2H), 7.09 – 6.93 (m, 4H), 6.77 – 6.68 (m, 1H), 6.07 – 5.99 (m, 1H), 5.65 (s, 1H), 5.52 (d, $J = 7.8$ Hz, 1H), 5.17 – 5.01 (m, 1H), 2.85 (p, $J = 7.2, 6.6$ Hz, 1H), 2.38 (s, 3H), 1.22 (d, $J = 6.9$ Hz, 6H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 148.7, 143.3, 137.2, 136.0, 130.9, 129.5, 127.4, 127.3, 126.6, 118.6, 108.3, 108.1, 55.9, 33.9, 24.1, 21.6. HRMS(ESI+) m/z calcd for $\text{C}_{21}\text{H}_{25}\text{N}_2\text{O}_2\text{S}$ ($[\text{M}+\text{H}]^+$) : 369.1631, found: 369.1631.

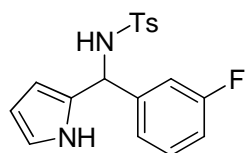
(R)-N-[pyrrol-2-yl-(2-fluorophenyl)methyl]-4-methylbenzenesulfonamide (6k)



Reaction time: 48 h, Pale yellow solid, 65% yield, mp: 124-125 °C, $[\alpha]_D^{20} = -12.2$ (*c* 1.0, Acetone), 96% *ee* [Daicel Chiralpak AD-H

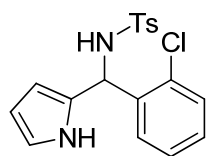
column, *n*-hexane/*i*-PrOH = 60 : 40, 1.0 mL/min, λ = 254 nm, $t(\text{major})$ = 8.22 min, $t(\text{minor})$ = 17.20 min]; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 8.61 (br, 1H), 7.58 (d, J = 8.4 Hz, 2H), 7.25 – 7.16 (m, 1H), 7.15 – 7.08 (m, 3H), 7.05 – 6.97 (m, 1H), 6.94 – 6.84 (m, 1H), 6.79 – 6.68 (m, 1H), 6.08 – 5.97 (m, 1H), 5.75 (d, J = 9.0 Hz, 1H), 5.63 – 5.53 (m, 1H), 5.44 – 5.25 (m, 1H), 2.35 (s, 3H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 143.3, 136.7, 129.6, 129.5, 129.3, 129.2, 129.2, 127.0, 124.2, 118.8, 115.7, 115.4, 108.2, 107.6, 51.1, 21.4. HRMS(ESI+) m/z calcd for $\text{C}_{18}\text{H}_{18}\text{FN}_2\text{O}_2\text{S}$ ($[\text{M}+\text{H}]^+$) : 345.1068, found: 345.1066.

(*R*)-*N*-[pyrrol-2-yl-(3-fluorophenyl)methyl]-4-methylbenzenesulfonamide (6l)



Reaction time: 24 h, white solid, 75% yield, mp: 127-128 °C, $[\alpha]_{\text{D}}^{20}$ = -37.8 (*c* 1.0, Acetone), 99% *ee* [Daicel Chiralpak AD-H column, *n*-hexane/*i*-PrOH = 60 : 40, 1.0 mL/min, λ = 254 nm, $t(\text{major})$ = 6.60 min, $t(\text{minor})$ = 7.38 min]; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 8.54 (br, 1H), 7.57 (d, J = 8.1 Hz, 2H), 7.23 – 7.10 (m, 3H), 6.97 – 6.87 (m, 2H), 6.80 (dt, J = 9.6, 2.1 Hz, 1H), 6.74 – 6.68 (m, 1H), 6.03 (q, J = 3.0 Hz, 1H), 5.68 – 5.49 (m, 2H), 5.45 – 5.31 (m, 1H), 2.38 (s, 3H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 143.7, 141.3, 137.0, 130.2, 130.1, 130.0, 129.6, 127.2, 123.1, 119.1, 115.0, 114.7, 114.4, 108.3, 55.5, 21.6. HRMS(ESI+) m/z calcd for $\text{C}_{18}\text{H}_{18}\text{FN}_2\text{O}_2\text{S}$ ($[\text{M}+\text{H}]^+$) : 345.1068, found: 345.1067.

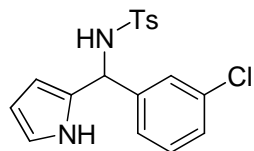
(*R*)-*N*-[pyrrol-2-yl-(2-chlorophenyl)methyl]-4-methylbenzenesulfonamide(6m)



Reaction time: 48 h, white solid, 60% yield, mp: 132-133 °C, $[\alpha]_{\text{D}}^{20}$ = -39.0 (*c* 1.0, Acetone), 99% *ee* [Daicel Chiralpak AD-H column, *n*-hexane/*i*-PrOH = 60 : 40, 1.0 mL/min, λ = 254 nm, $t(\text{major})$ = 7.72 min, $t(\text{minor})$ = 23.83 min]; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 8.50 (br, 1H), 7.60 (d, J = 8.1 Hz, 2H), 7.25 – 7.09 (m, 6H), 6.78 – 6.63 (m, 1H), 6.03 (q, J = 3.0 Hz, 1H), 5.95 (d, J = 8.4 Hz, 1H), 5.57 – 5.47 (m, 1H), 5.40 (d, J = 8.4 Hz, 1H), 2.36 (s, 3H); $^{13}\text{C NMR}$ (75 MHz, DMSO-d_6) δ 142.1, 138.3, 137.5, 131.5, 129.4, 129.0, 128.9, 128.7, 128.4, 126.9, 126.2, 118.3, 107.2, 51.8, 20.9. HRMS(ESI+) m/z calcd for

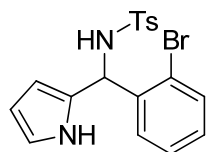
$C_{18}H_{18}ClN_2O_2S$ ($[M+H]^+$) : 361.0772, found: 361.0773.

(R)-N-[pyrrol-2-yl-(3-chlorophenyl)methyl]-4-methylbenzenesulfonamide (6n)



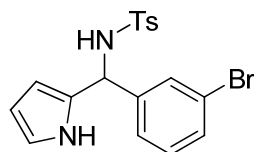
Reaction time: 10 h, pale yellow gum, 98% yield, $[\alpha]_D^{20} = -5.4$ (c 1.0, Acetone), 98% *ee* [Daicel Chiralpak AD-H column, *n*-hexane/*i*-PrOH = 60 : 40, 1.0 mL/min, $\lambda = 254$ nm, $t(\text{major}) = 6.15$ min, $t(\text{minor}) = 7.18$ min]; 1H NMR (300 MHz, $CDCl_3$) δ 8.57 (br, 1H), 7.54 (dd, $J = 8.4, 2.1$ Hz, 2H), 7.23 – 7.09 (m, 4H), 7.08 – 6.96 (m, 2H), 6.76 – 6.69 (m, 1H), 6.07 – 5.99 (m, 1H), 5.65 – 5.58 (m, 1H), 5.55 (d, $J = 8.1$ Hz, 1H), 5.50 – 5.32 (m, 1H), 2.39 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.6, 140.4, 136.7, 134.2, 129.7, 129.7, 129.4, 127.8, 127.5, 127.0, 125.6, 118.9, 108.3, 55.4, 21.4. HRMS(ESI+) m/z calcd for $C_{18}H_{18}ClN_2O_2S$ ($[M+H]^+$) : 361.0772, found: 361.0772.

(R)-N-[pyrrol-2-yl-(2-bromophenyl)methyl]-4-methylbenzenesulfonamide(6o)



Reaction time: 48 h, white solid, 80% yield, mp: 135-136 °C, $[\alpha]_D^{20} = -2.8$ (c 1.0, Acetone), 96% *ee* [Daicel Chiralpak AD-H column, *n*-hexane/*i*-PrOH = 60 : 40, 1.0 mL/min, $\lambda = 254$ nm, $t(\text{major}) = 7.82$ min, $t(\text{minor}) = 26.15$ min]; 1H NMR (300 MHz, $CDCl_3$) δ 8.57 (br, 1H), 7.60 (d, $J = 8.3$ Hz, 2H), 7.40 (d, $J = 7.9$ Hz, 1H), 7.34 – 7.02 (m, 5H), 6.72 (s, 1H), 6.19 – 5.84 (m, 2H), 5.77 – 5.49 (m, 2H), 2.34 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.45, 137.88, 136.64, 132.88, 129.42, 129.17, 129.11, 127.58, 127.14, 122.98, 118.79, 108.34, 107.96, 55.18, 21.44. HRMS(ESI+) m/z calcd for $C_{18}H_{18}BrN_2O_2S$ ($[M+H]^+$) : 405.0267, found: 405.0266.

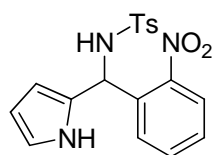
(R)-N-[pyrrol-2-yl-(3-bromophenyl)methyl]-4-methylbenzenesulfonamide(6p)



Reaction time: 20 h, pale yellow solid, 93% yield, mp: 126-127 °C, $[\alpha]_D^{20} = -2.8$ (c 1.0, Acetone), 97% *ee* [Daicel Chiralpak AD-H column, *n*-hexane/*i*-PrOH = 60 : 40, 1.0

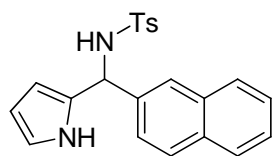
mL/min, $\lambda = 254$ nm, $t(\text{major}) = 6.25$ min, $t(\text{minor}) = 7.48$ min]; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 8.59 (br, 1H), 7.53 (d, $J = 8.4$ Hz, 2H), 7.36 – 7.28 (m, 1H), 7.18 – 6.99 (m, 5H), 6.77 – 6.65 (m, 1H), 6.03 (q, $J = 3.0$ Hz, 1H), 5.63 – 5.57 (m, 1H), 5.54 (d, $J = 8.4$ Hz, 1H), 5.45 (d, $J = 8.1$ Hz, 1H), 2.39 (s, 3H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 143.8, 140.8, 136.9, 130.9, 130.5, 130.1, 129.9, 129.6, 127.1, 126.2, 122.5, 119.1, 108.4, 55.5, 21.7. HRMS(ESI+) m/z calcd for $\text{C}_{18}\text{H}_{18}\text{BrN}_2\text{O}_2\text{S}$ ($[\text{M}+\text{H}]^+$) : 405.0267, found: 405.0267.

(R)-N-[pyrrol-2-yl-(2-nitrophenyl)methyl]-4-methylbenzenesulfonamide(6q)



Reaction time: 48 h, white solid, 70% yield, mp: 122-123 °C, $[\alpha]_{\text{D}}^{20} = -38.0$ (c 1.0, Acetone), 97% *ee* [Daicel Chiralpak AD-H column, *n*-hexane/*i*-PrOH = 60 : 40, 1.0 mL/min, $\lambda = 254$ nm, $t(\text{major}) = 10.08$ min, $t(\text{minor}) = 30.13$ min]; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 8.48 (br, 1H), 7.84 (dd, $J = 8.1, 1.3$ Hz, 1H), 7.62 (d, $J = 8.3$ Hz, 2H), 7.58 – 7.45 (m, 2H), 7.42 – 7.35 (m, 1H), 7.19 (d, $J = 8.1$ Hz, 2H), 6.67 (dd, $J = 4.1, 2.6$ Hz, 1H), 6.22 (d, $J = 7.6$ Hz, 1H), 5.98 (dd, $J = 5.9, 2.8$ Hz, 1H), 5.75 (d, $J = 7.8$ Hz, 1H), 5.54 (dd, $J = 3.4, 2.7$ Hz, 1H), 2.38 (s, 3H); $^{13}\text{C NMR}$ (75 MHz, DMSO-d_6) δ 142.79, 138.59, 131.37, 129.30, 128.22, 124.85, 124.50, 123.71, 123.41, 121.86, 119.67, 113.69, 103.22, 101.93, 46.66, 16.28. HRMS(ESI+) m/z calcd for $\text{C}_{18}\text{H}_{18}\text{N}_3\text{O}_4\text{S}$ ($[\text{M}+\text{H}]^+$) : 372.1013, found: 372.1011.

(R)-N-[pyrrol-2-yl-(2-naphthyl)methyl]-4-methylbenzenesulfonamide (6r)

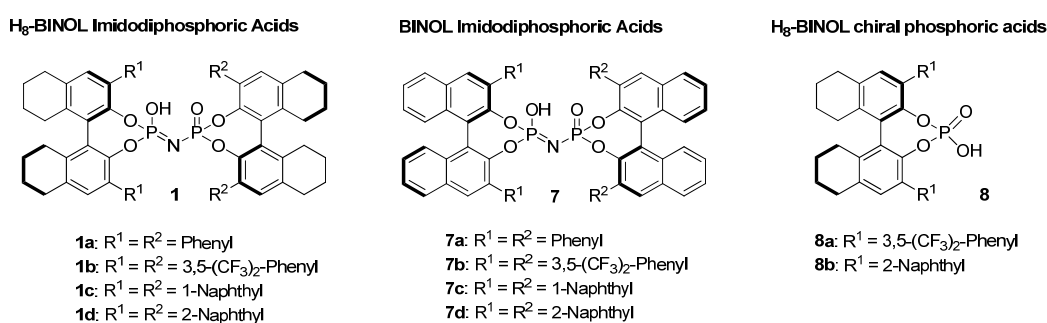


Reaction time: 12 h, yellow solid, 93% yield, mp: 114-115 °C, $[\alpha]_{\text{D}}^{20} = -10.6$ (c 1.0, Acetone), 98% *ee* [Daicel Chiralpak AD-H column, *n*-hexane/ethanol = 90 : 10, 1.0 mL/min, $\lambda = 254$ nm, $t(\text{major}) = 37.03$ min, $t(\text{minor}) = 40.42$ min]; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 8.53 (br, 1H), 7.83 – 7.73 (m, 1H), 7.74 – 7.62 (m, 2H), 7.59 – 7.52 (m, 2H), 7.51 – 7.43 (m, 3H), 7.22 (dd, $J = 8.4, 1.8$ Hz, 1H), 7.03 (d, $J = 8.1$ Hz, 2H), 6.78 – 6.70 (m, 1H), 6.06 (q, $J = 3.0$ Hz, 1H), 5.71 (d, $J = 8.1$ Hz, 1H), 5.67 – 5.61 (m, 1H), 5.25 – 5.10 (m, 1H), 2.25 (s, 3H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 143.4, 137.1, 135.8, 133.1, 132.9, 130.5, 129.4,

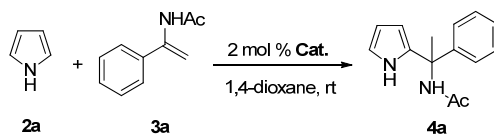
128.5, 128.1, 127.6, 127.2, 126.6, 126.3, 125.2, 118.8, 108.4, 108.3, 56.2, 21.4.
 HRMS(ESI+) m/z calcd for $C_{22}H_{21}N_2O_2S$ ($[M+H]^+$) : 377.1318, found: 377.1317.

4. Supplementary Data of the Comparison of the Catalysts and Molecular Sieves

Supplementary Table 1. The comparison of the catalysts



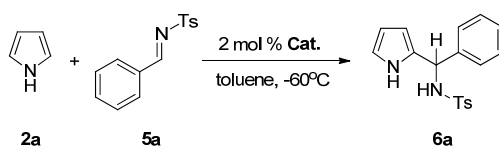
Part 1: Aza-Friedel–Crafts reaction of pyrrole **2a** and enamide **3a**



Entry	Cat.	Time [h]	Yield [%] ^b	ee[%] ^c
1	1c	1	60	87
2	1d	1	65	93
3	7c	1	63	20
4	7d	1	60	70
5	8a	12	65	7
6	8b	5	60	17

^a Reaction conditions: **3a** (0.1 mmol), **2a** (0.11 mmol), 1,4-dioxane (1 mL). ^b Isolated yield. ^c Determined by HPLC analysis on Chiralpak AS-H column.

Part 2: Aza-Friedel–Crafts reaction of pyrrole **2a** and imine **5a**



Entry	Cat.	Time [h]	Yield [%] ^b	ee [%] ^c
1	1a	32	58	57
2	1b	11	82	82
3	1c	20	45	81
4	7a	5	42	25
5	7b	1	80	30
6	7c	7	55	67
7	8a	6	65	4
8	8b	8	60	51

^a Reaction conditions: **5a** (0.1 mmol), **2a** (0.11 mmol), solvent (1 mL). ^b Isolated yield. ^c Determined by HPLC analysis on Chiralpak AD-H column.

Supplementary Table 2. The comparison of the effect of the molecular sieves

Part 1: Aza-Friedel–Crafts reaction of pyrrole **2a** and enamide **3a**

Entry	M.S.	Time [h]	Yield [%] ^b	ee [%] ^c
1	3Å	2	90	95
2	4Å	2	89	95
3	5Å	2	90	96

^a Reaction conditions: **3a** (0.1 mmol), **2a** (0.11 mmol), 90 mg M.S., 1,4-dioxane (1 mL). ^b Isolated yield. ^c Determined by HPLC analysis on Chiralpak AS-H column.

Part 2: Aza-Friedel–Crafts reaction of pyrrole **2a** and imine **5a**

Entry	Cat.	Time [h]	Yield [%] ^b	ee [%] ^c
1	3Å	3	92	97
2	4Å	3	91	95
3	5Å	3	90	98

^a Reaction conditions: **5a** (0.1 mmol), **2a** (0.11 mmol), 5 mg M.S., toluene (1 mL). ^b Isolated yield. ^c Determined by HPLC analysis on Chiralpak AD-H column.

5. Experimental Procedures for Gram-Scale Reactions

Gram-scale Aza-Friedel-Crafts reaction of 3f and 2a: Enamide **3f** (1.20 g, 5 mmol), imidodiphosphoric acid **1d** (120 mg, 0.1 mmol, 2 mol %), 5Å molecular sieves (4.5 g) and 1,4-dioxane (50 mL) were loaded into test tube, the solution was stirred for 5 minutes at room temperature and pyrrole **2a** (0.11 mmol) was added. The reaction was complete in 8 hours. The resulting mixture was purified by silica gel chromatography (ethyl acetate/petroleum ether = 1/3) directly to afford the product **4f** (1.32 g, 88 % yield, 97% ee).

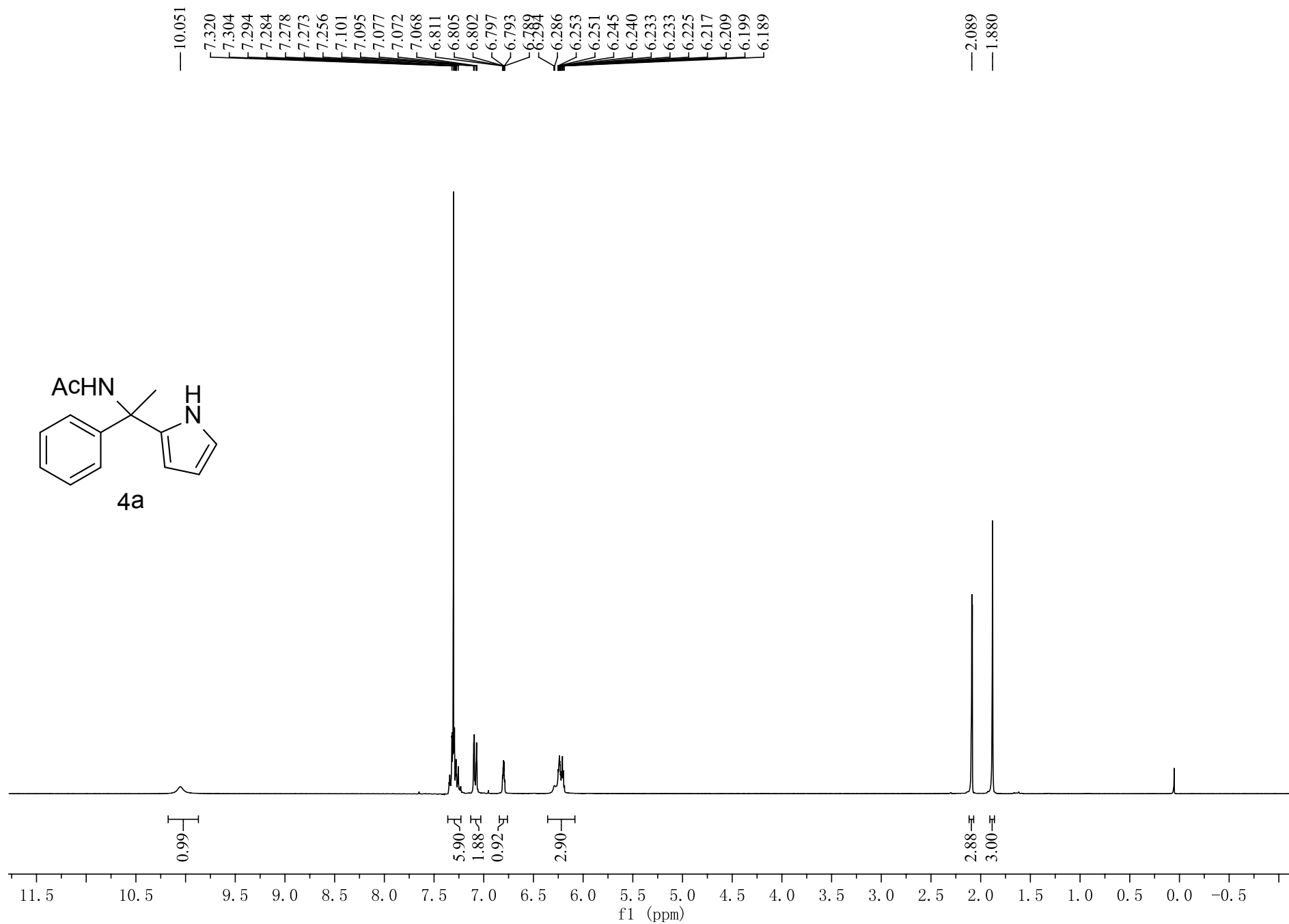
Gram-scale Aza-Friedel-Crafts reaction of 5d and 2a: Aldimine **5d** (1.37 g, 5 mmol), imidodiphosphoric acid **1b** (23 mg, 0.015 mmol, 0.3 mol %), 5Å molecular sieves (250 mg) and toluene (15 mL) were loaded into test tube, the solution was stirred for 15 minutes at -60 °C and pyrrole **2a** (0.55 mmol) was added. The reaction was complete in 6 hours. The resulting mixture was purified by silica gel chromatography (ethyl acetate/petroleum ether = 1/3) directly to afford the product **6d** (1.70 g, 93 % yield, 98% ee).

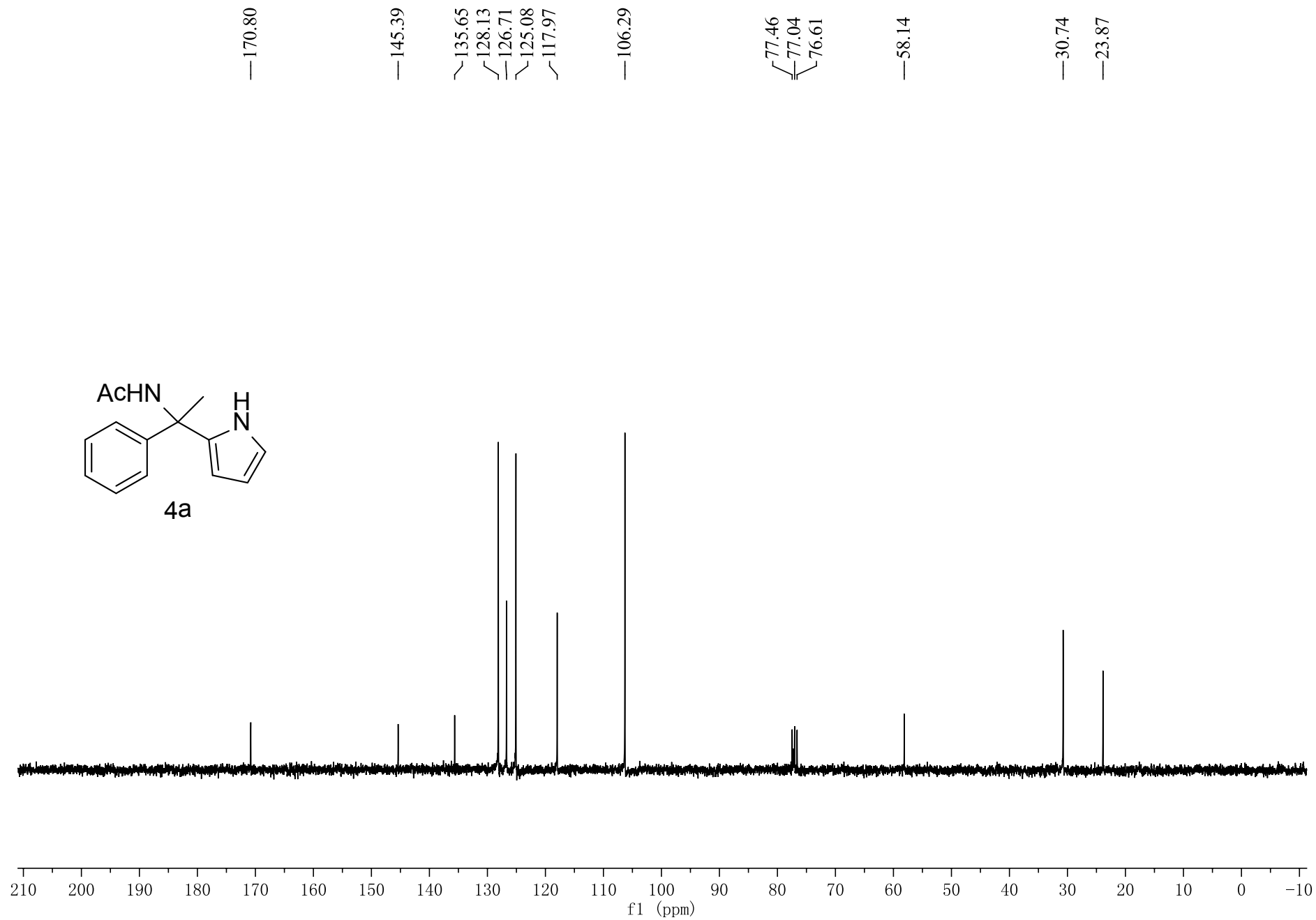
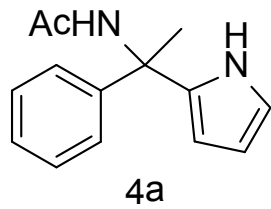
The synthesis of enamide **3** and aldimine **5** has been previously reported in literature^{1,2}.

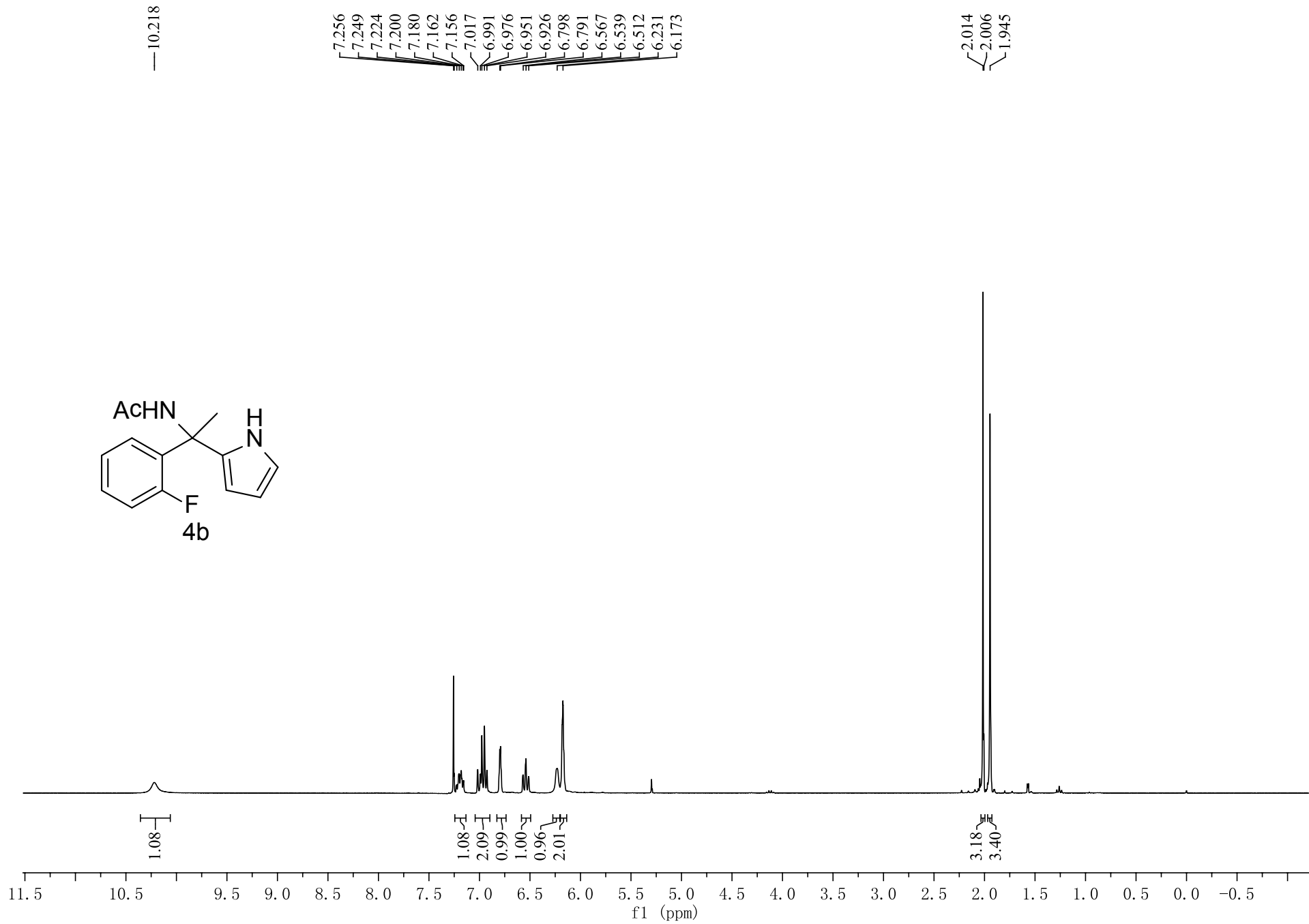
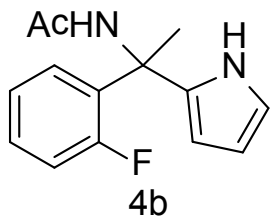
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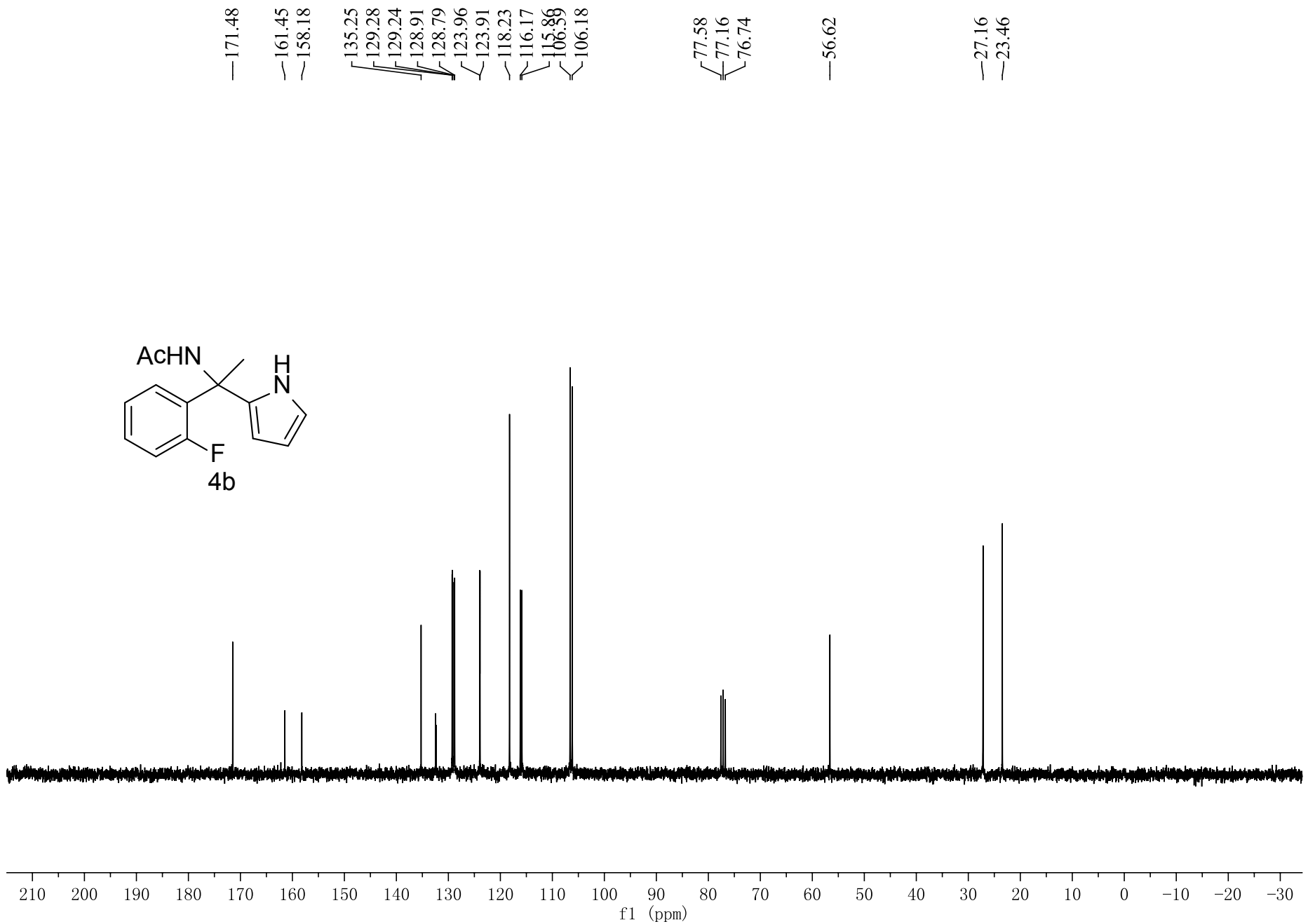
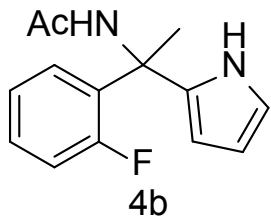
- [1] Reeves, J. T.; Tan, Z.; Han, Z. S.; Li, G.; Zhang, Y.; Xu, Y.; Reeves, D. C.; Gonnella, N. C.; Ma, S.; Lee, H.; Lu, B. Z.; Senanayake, C. H. *Angew. Chem., Int. Ed.* 2012, **51**, 1400-1404
- [2] Chemla, F.; Hebbe, V.; Normant, J.-F. *Synthesis* 2000, 75-77

6. Copies of NMR Spectra





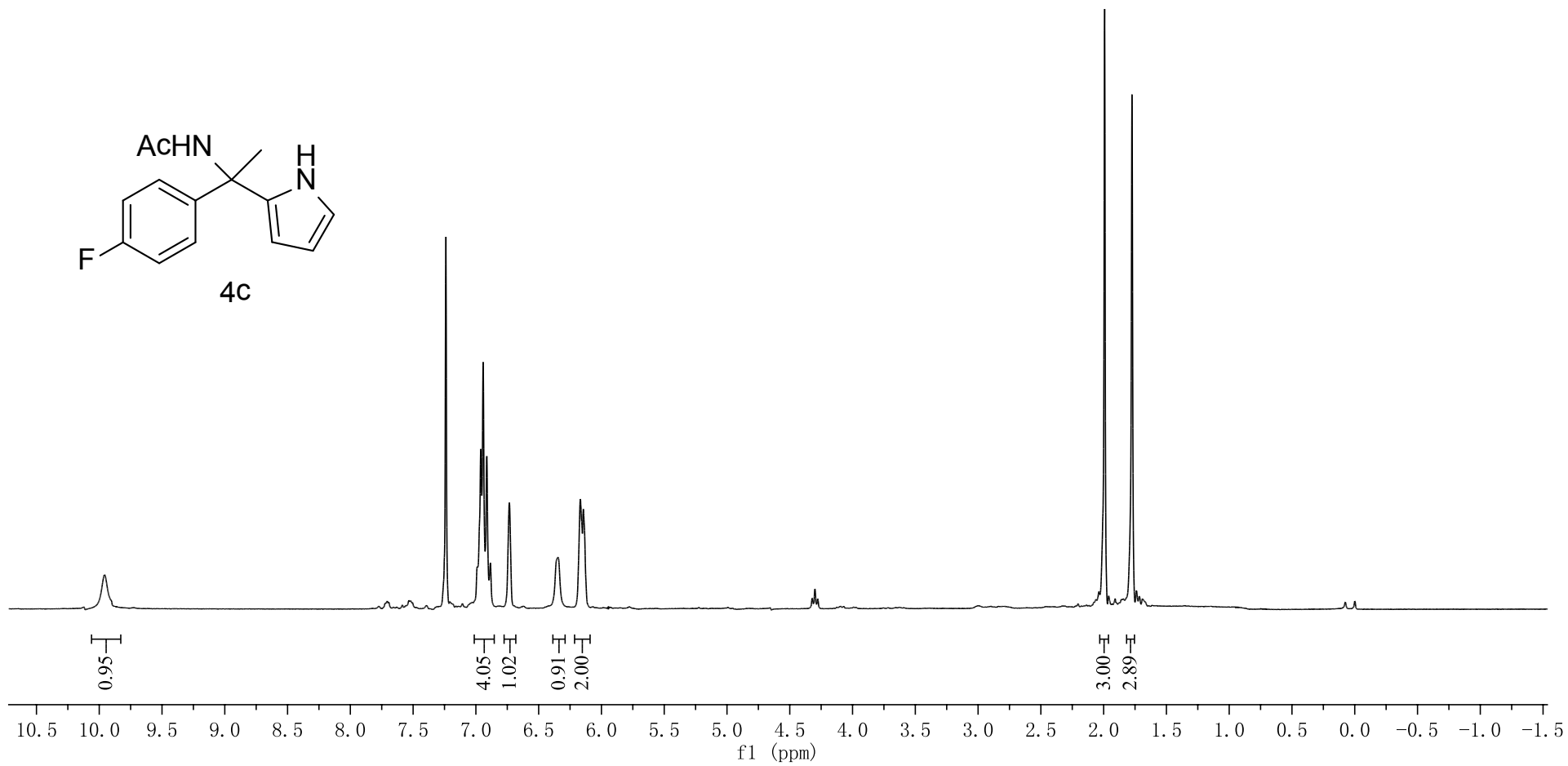
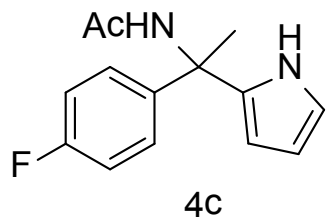


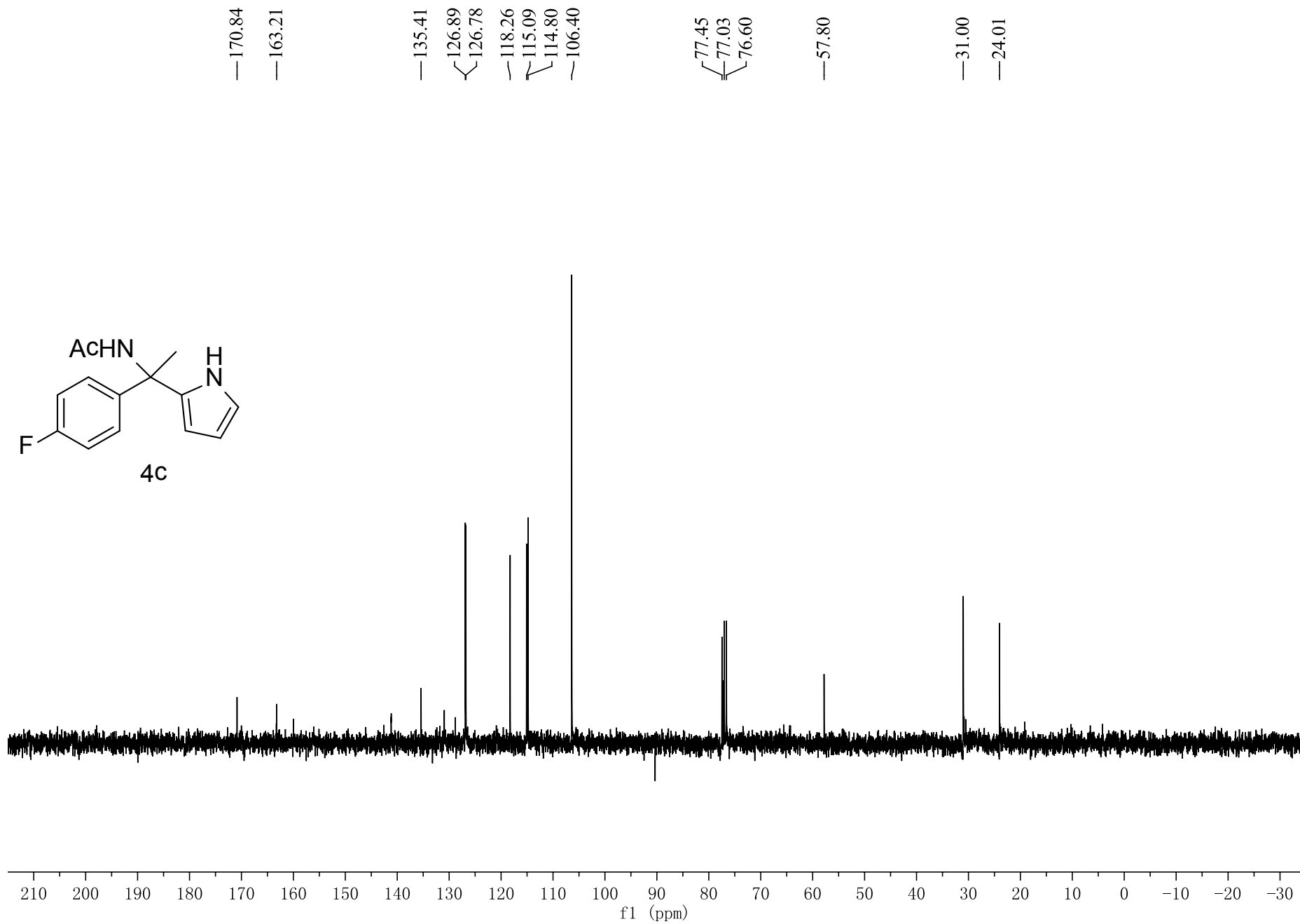
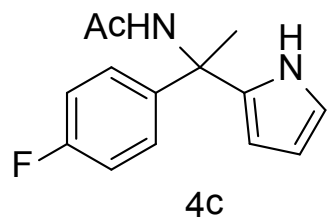


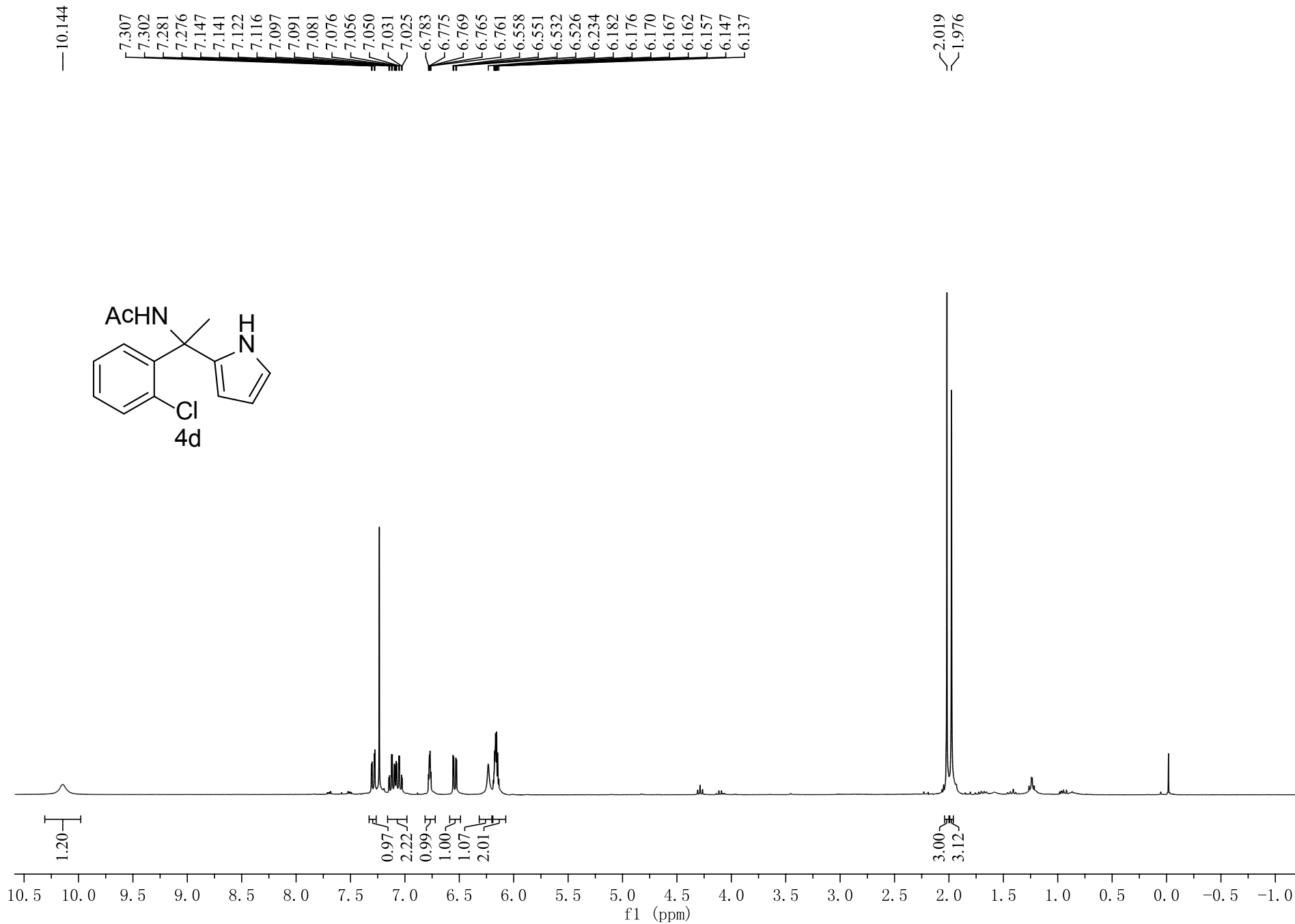
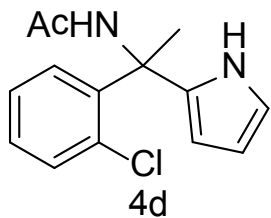
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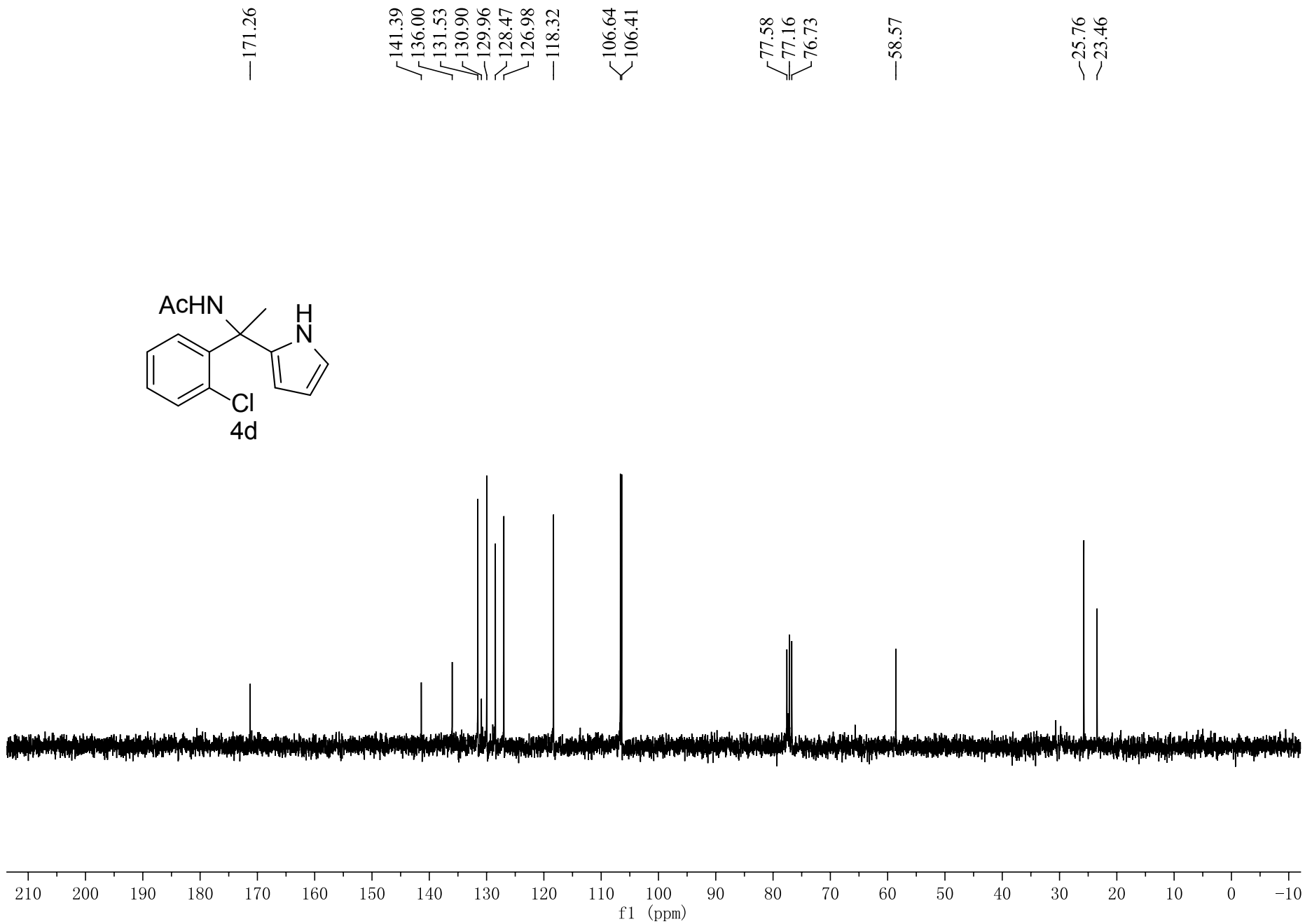
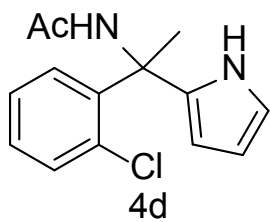
6.990
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6.961
6.942
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6.884
6.734
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6.349
6.340
6.169
6.156
6.144
6.134

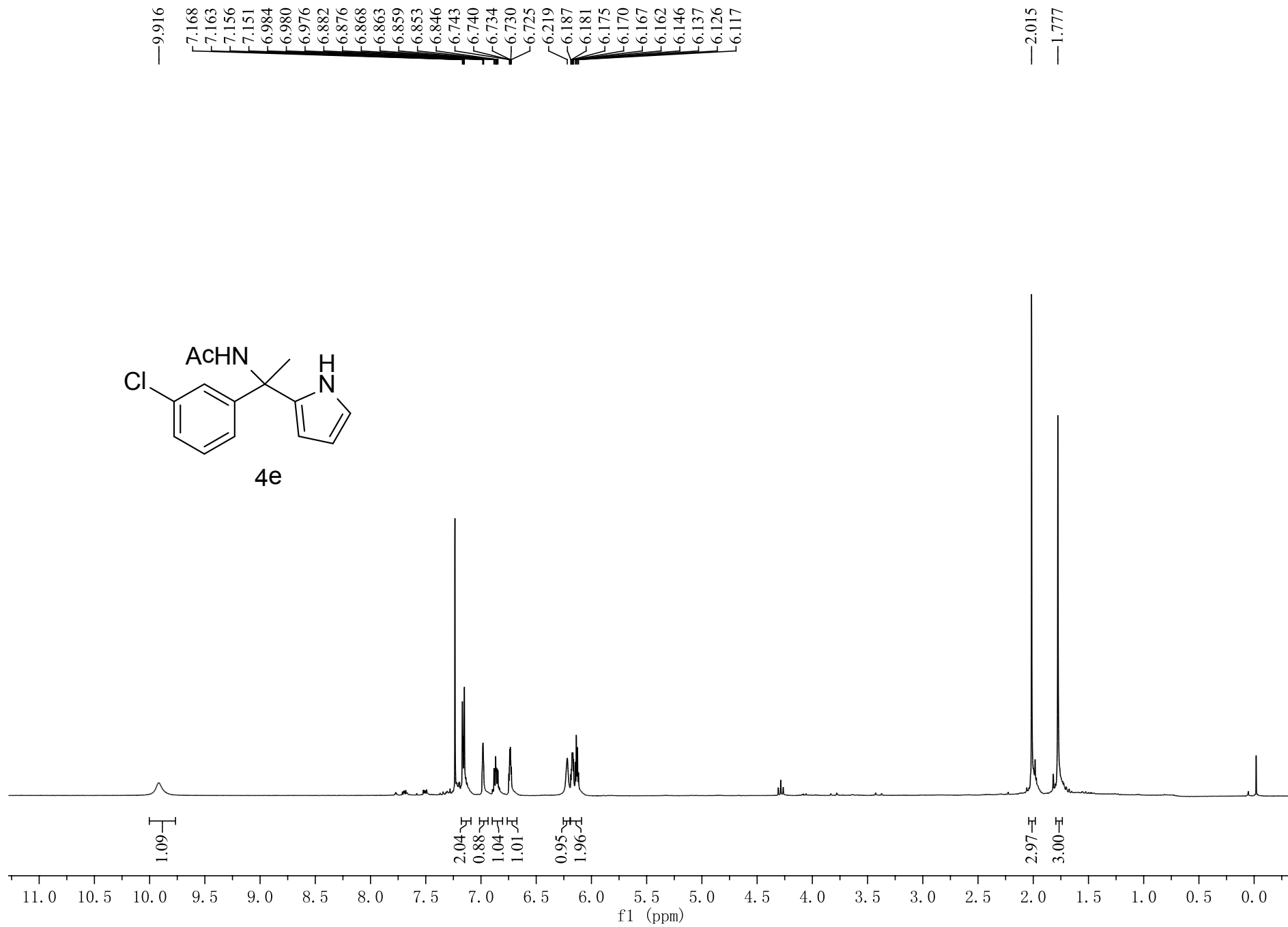
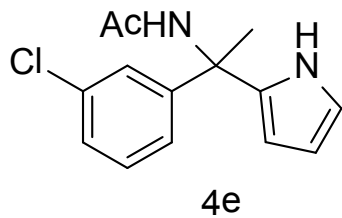
—1.992
—1.774

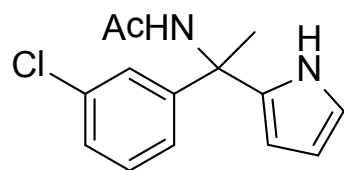




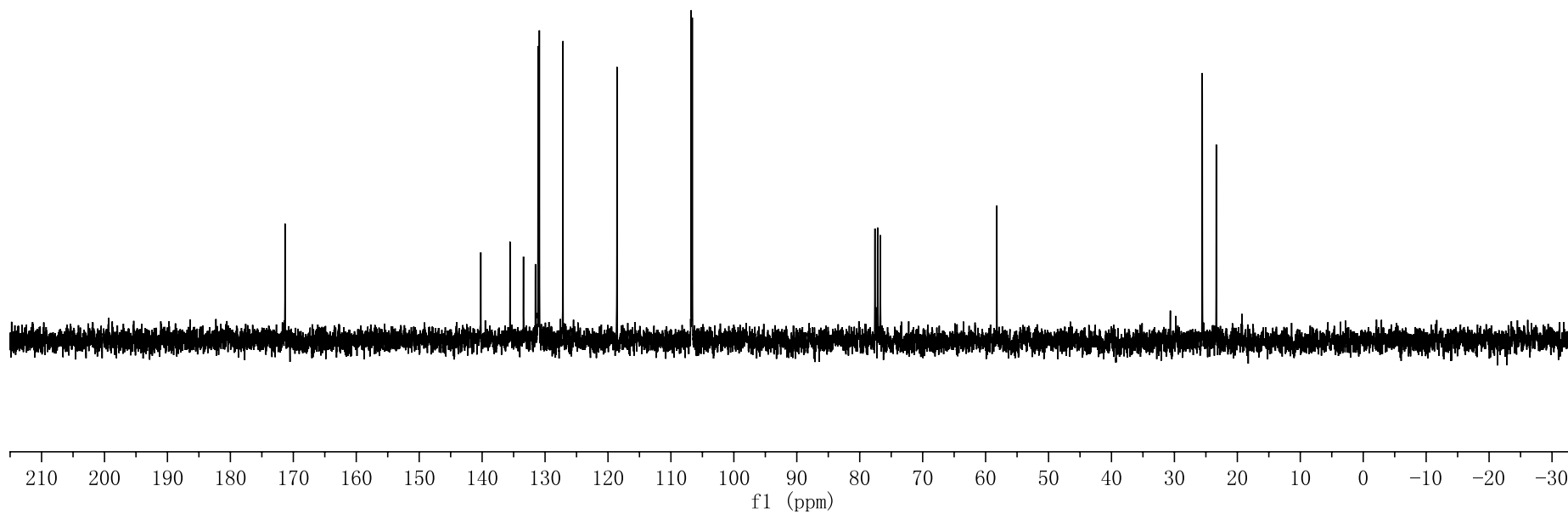


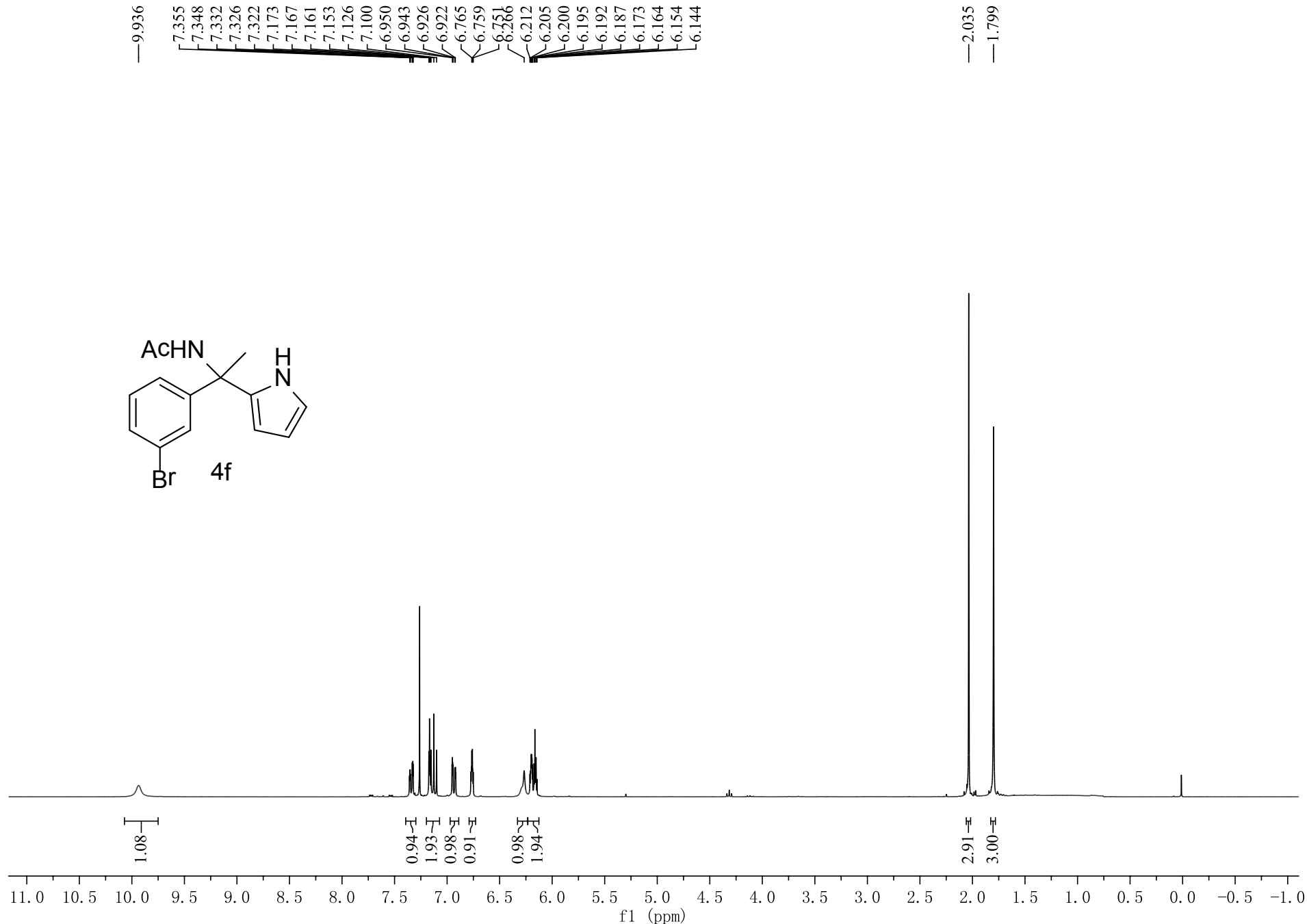
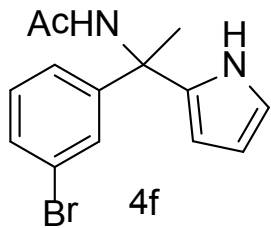


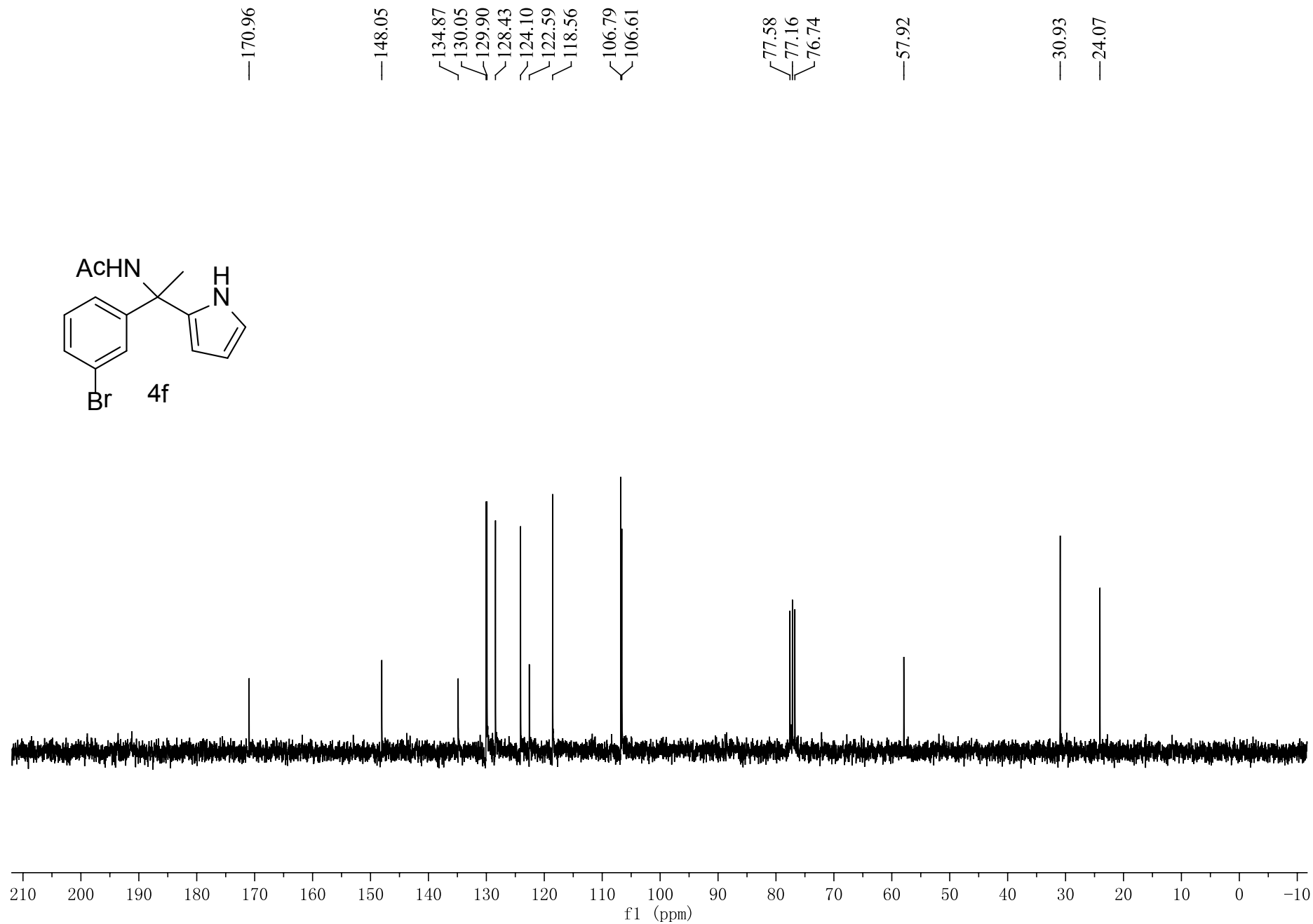
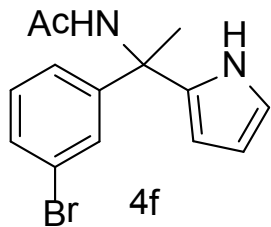


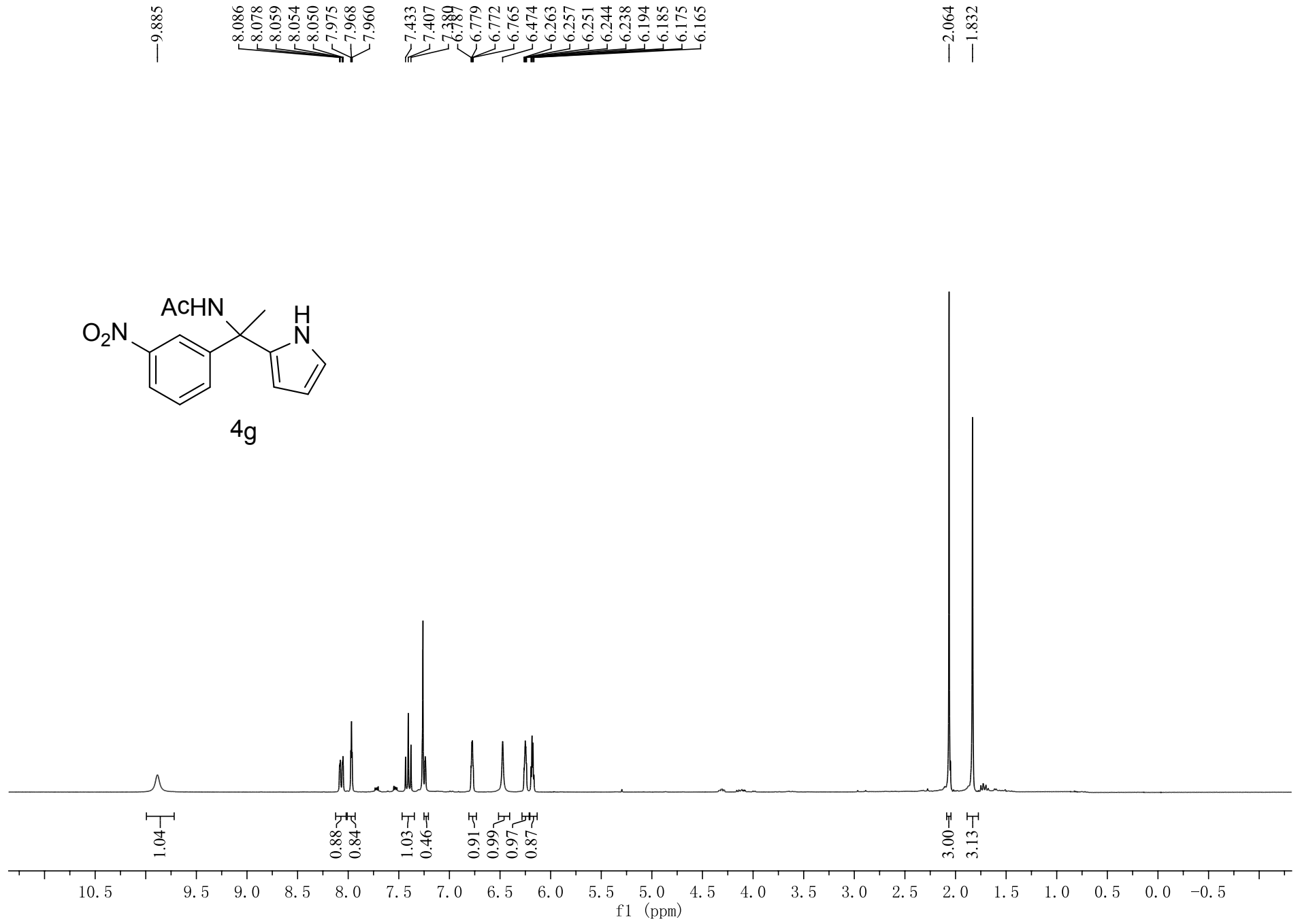
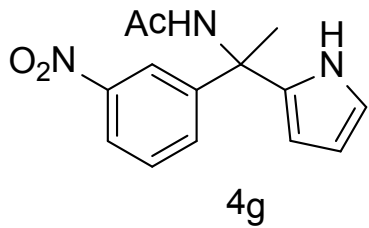


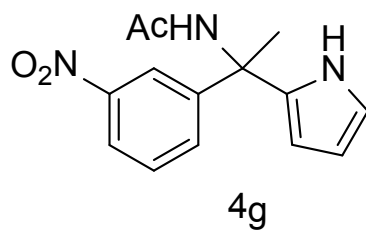
4e











—171.31

—148.18

134.27

131.76

129.31

122.02

120.23

118.90

107.20

106.86

77.58

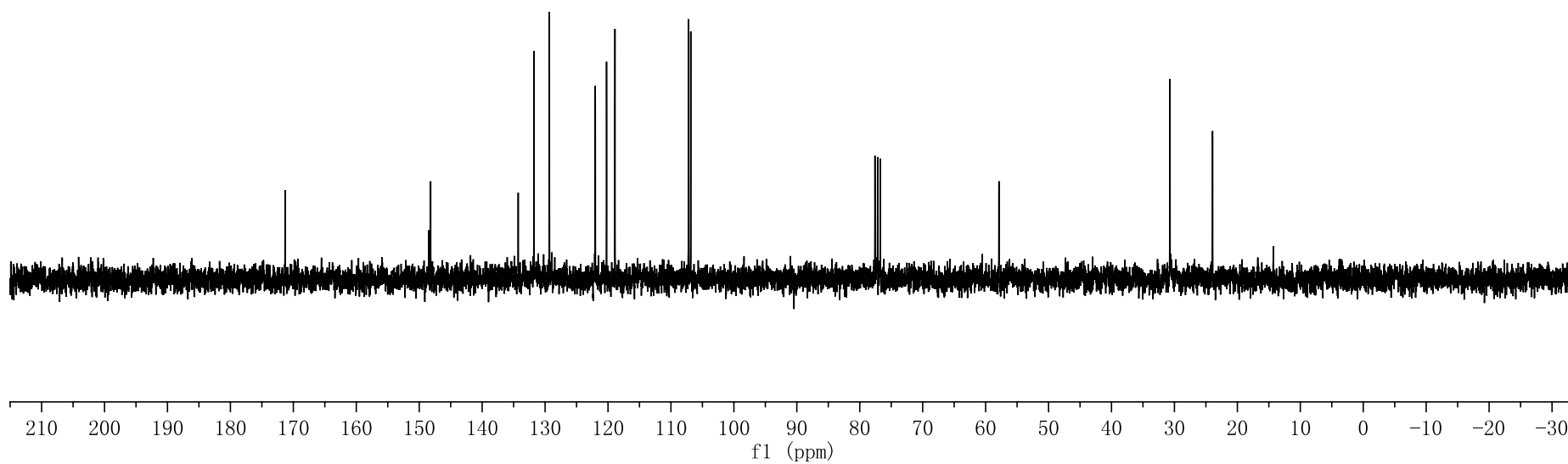
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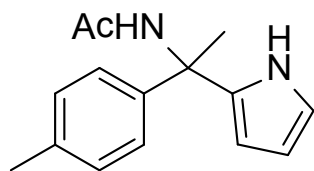
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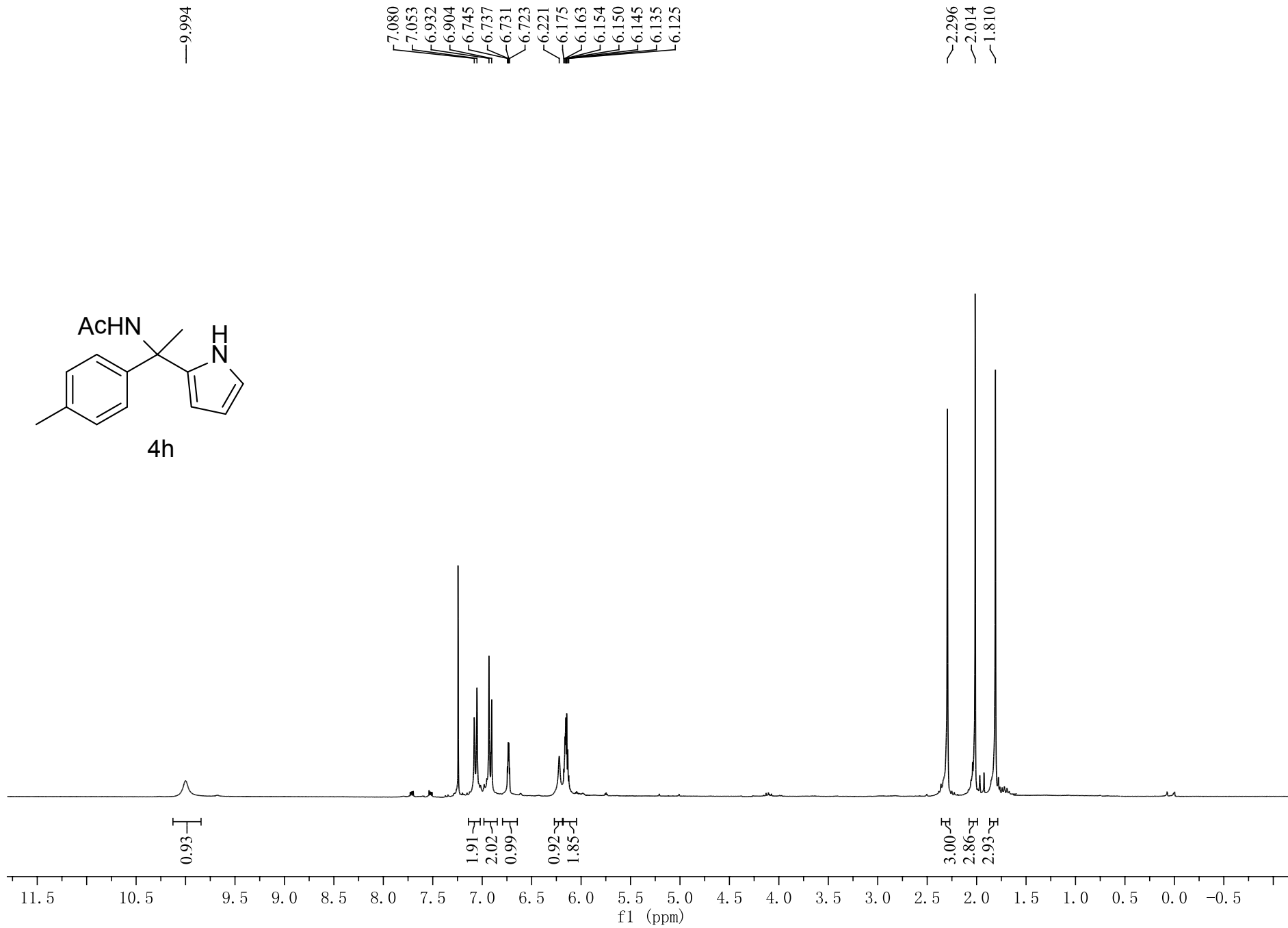
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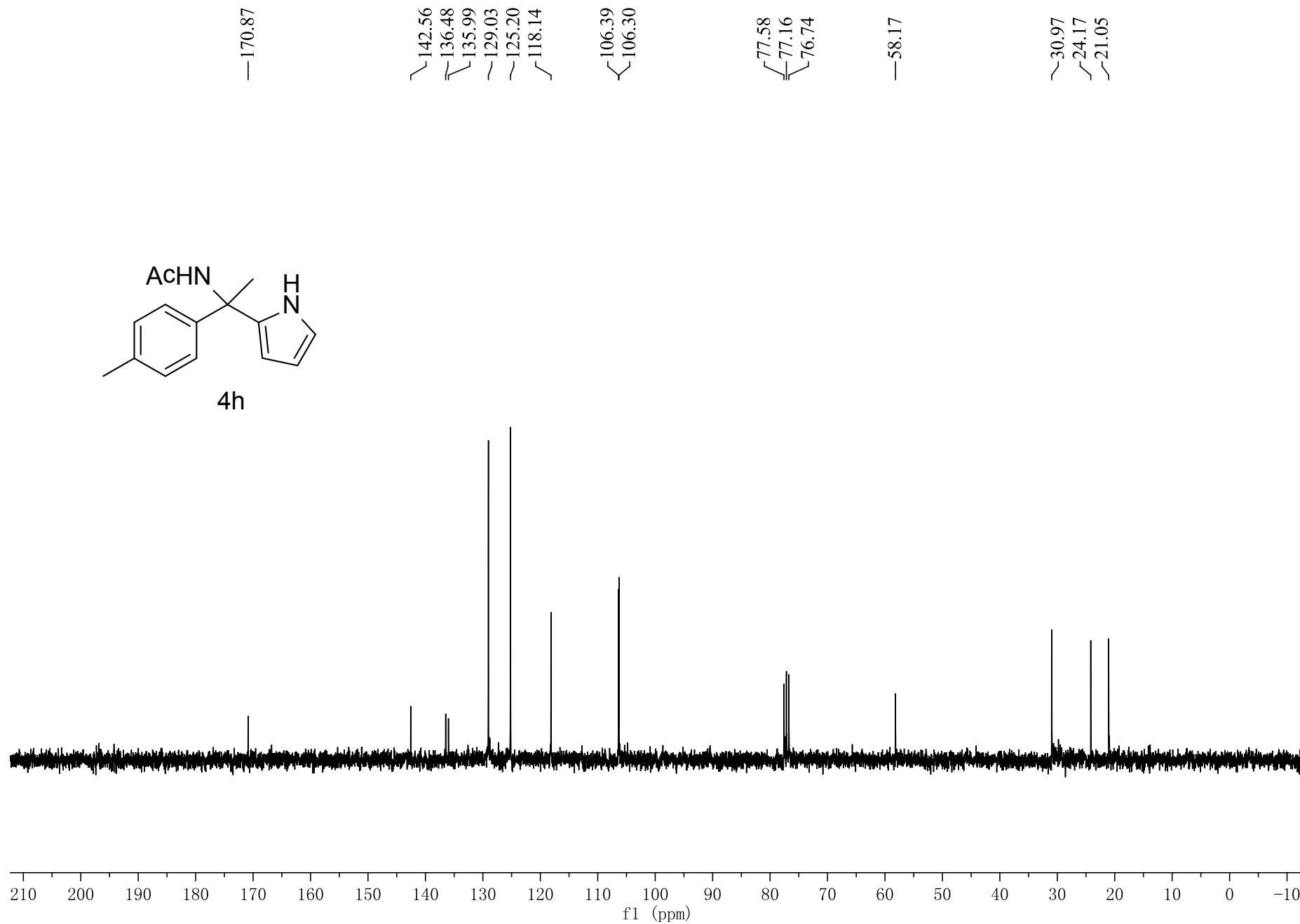
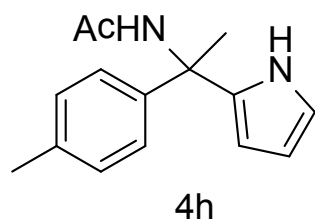
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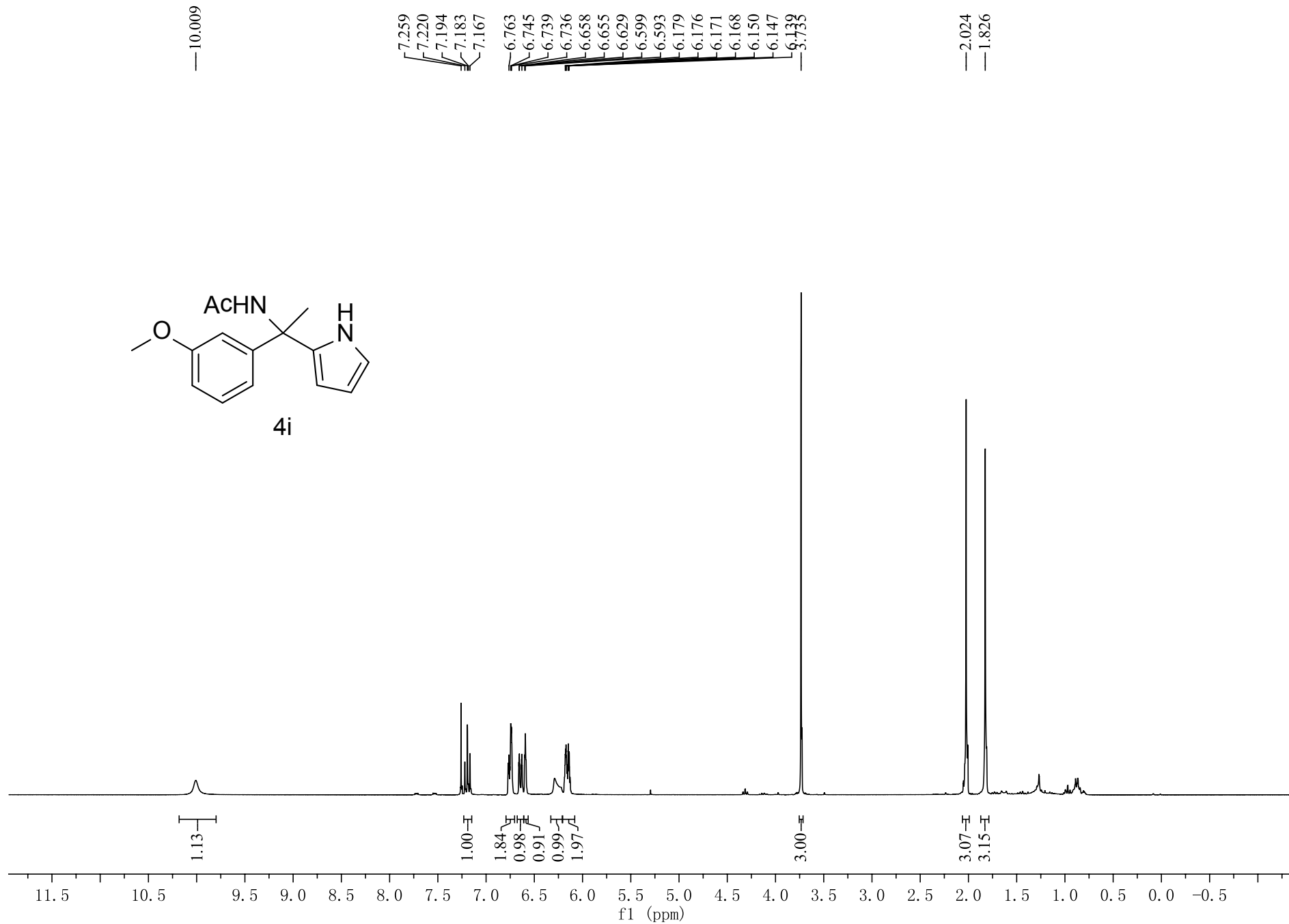
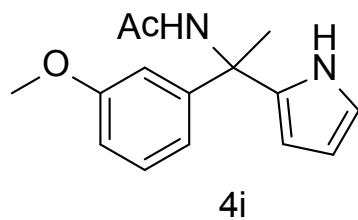


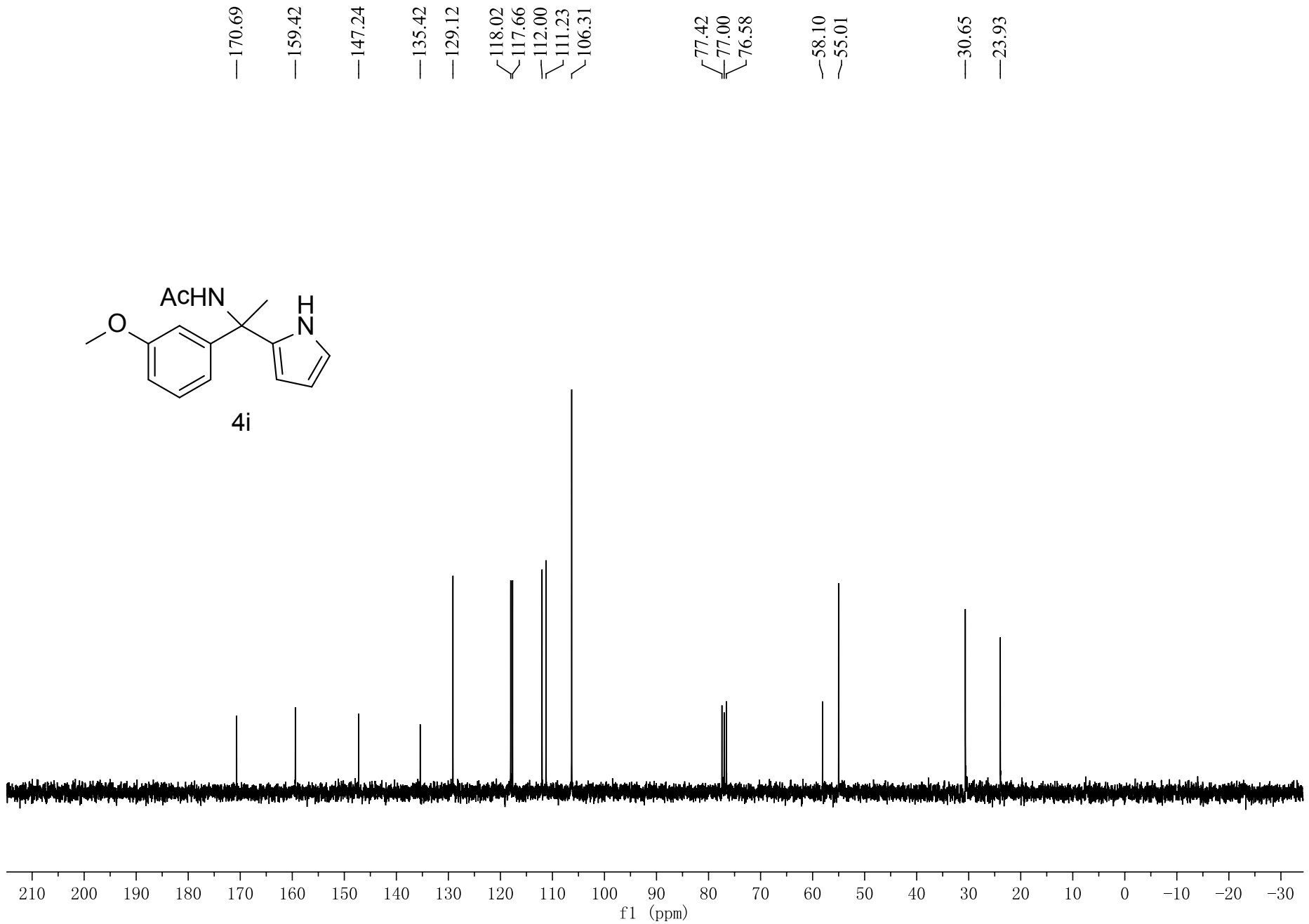
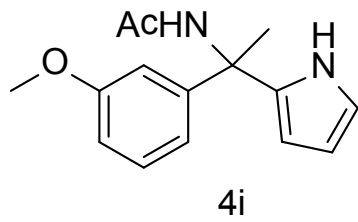


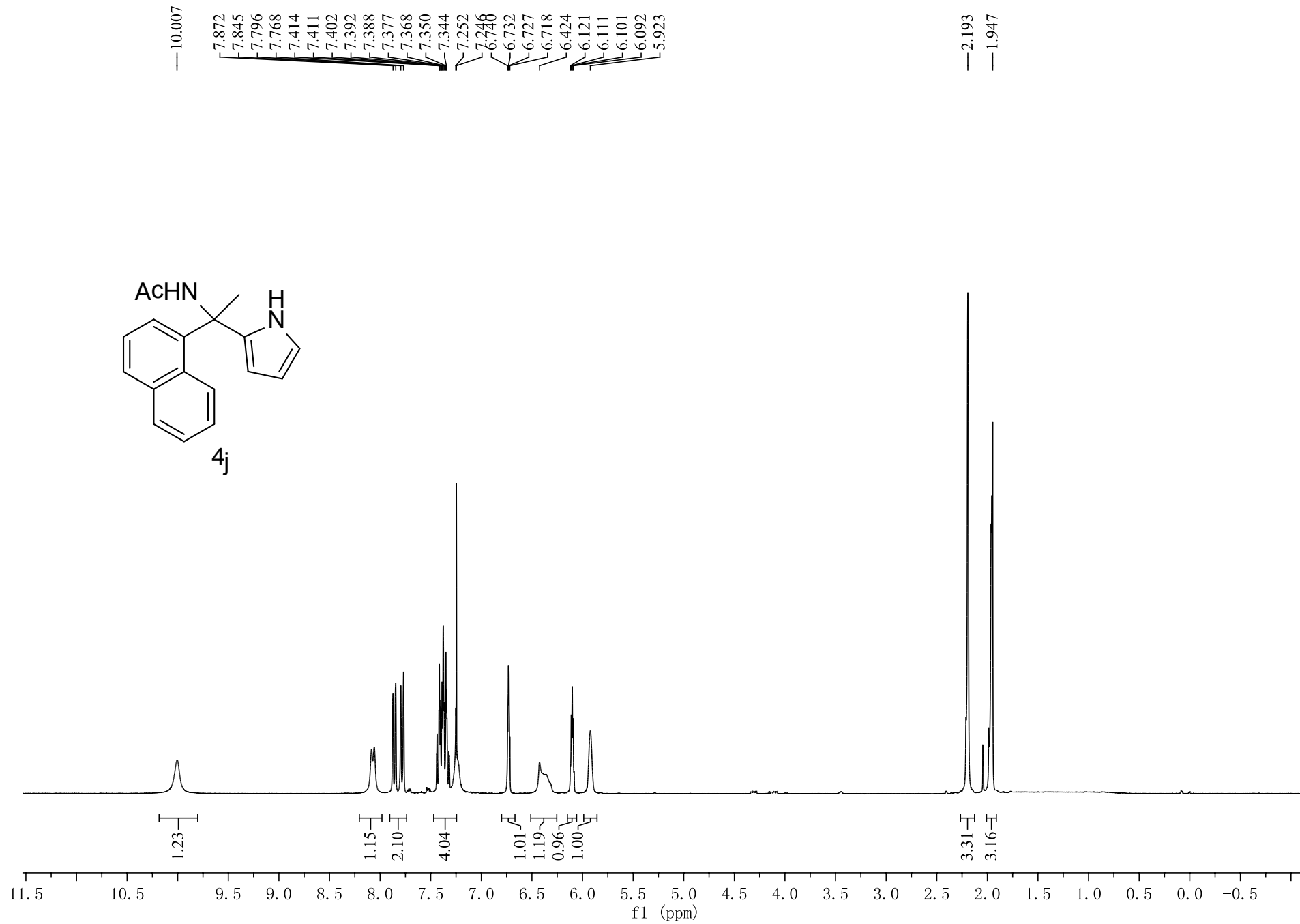
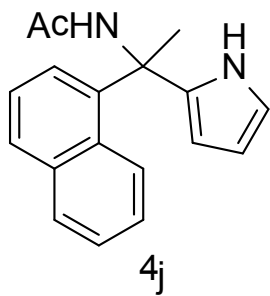
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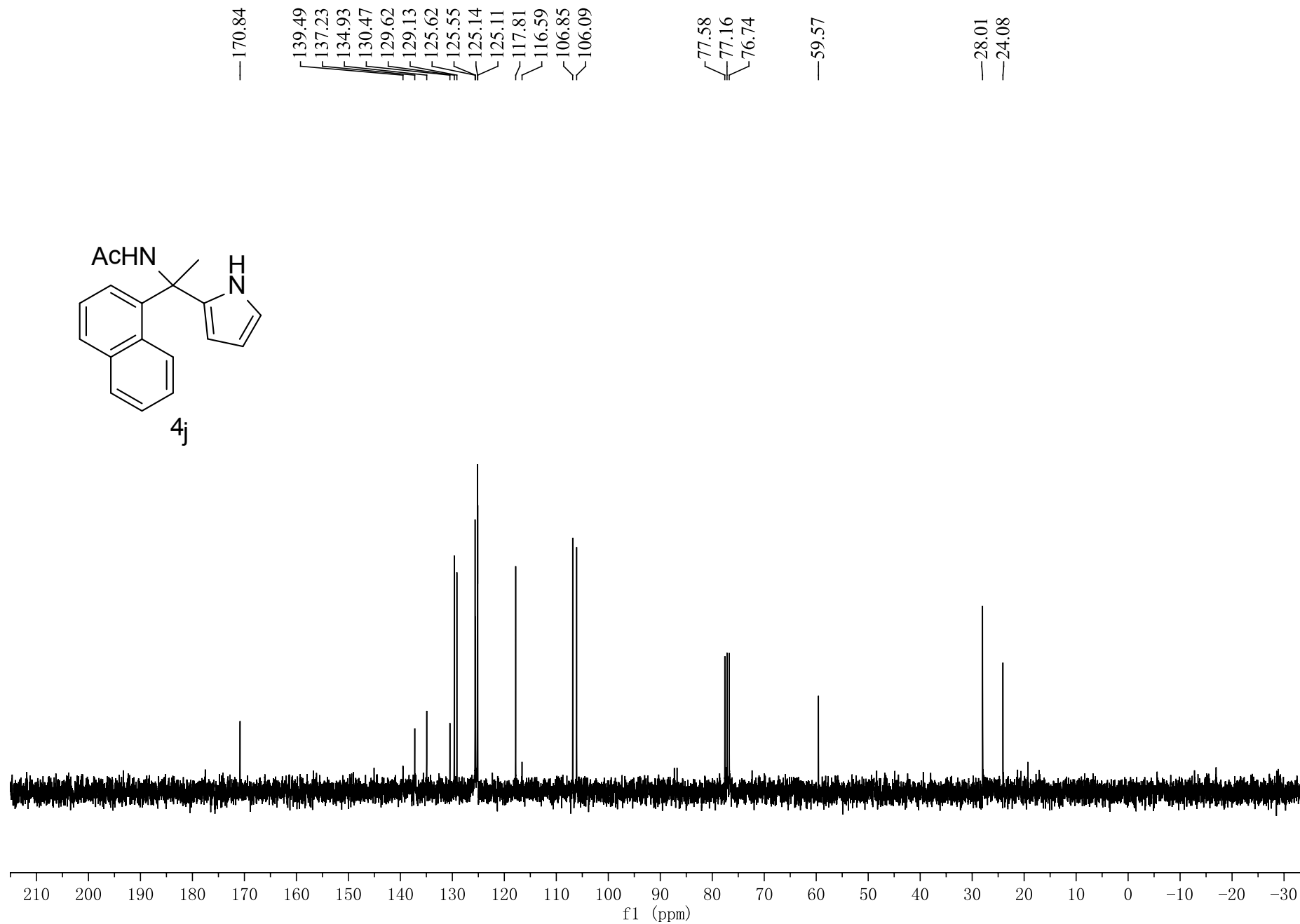
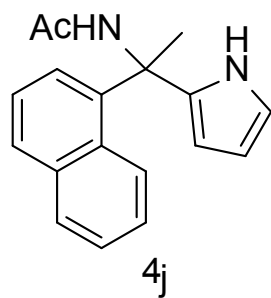


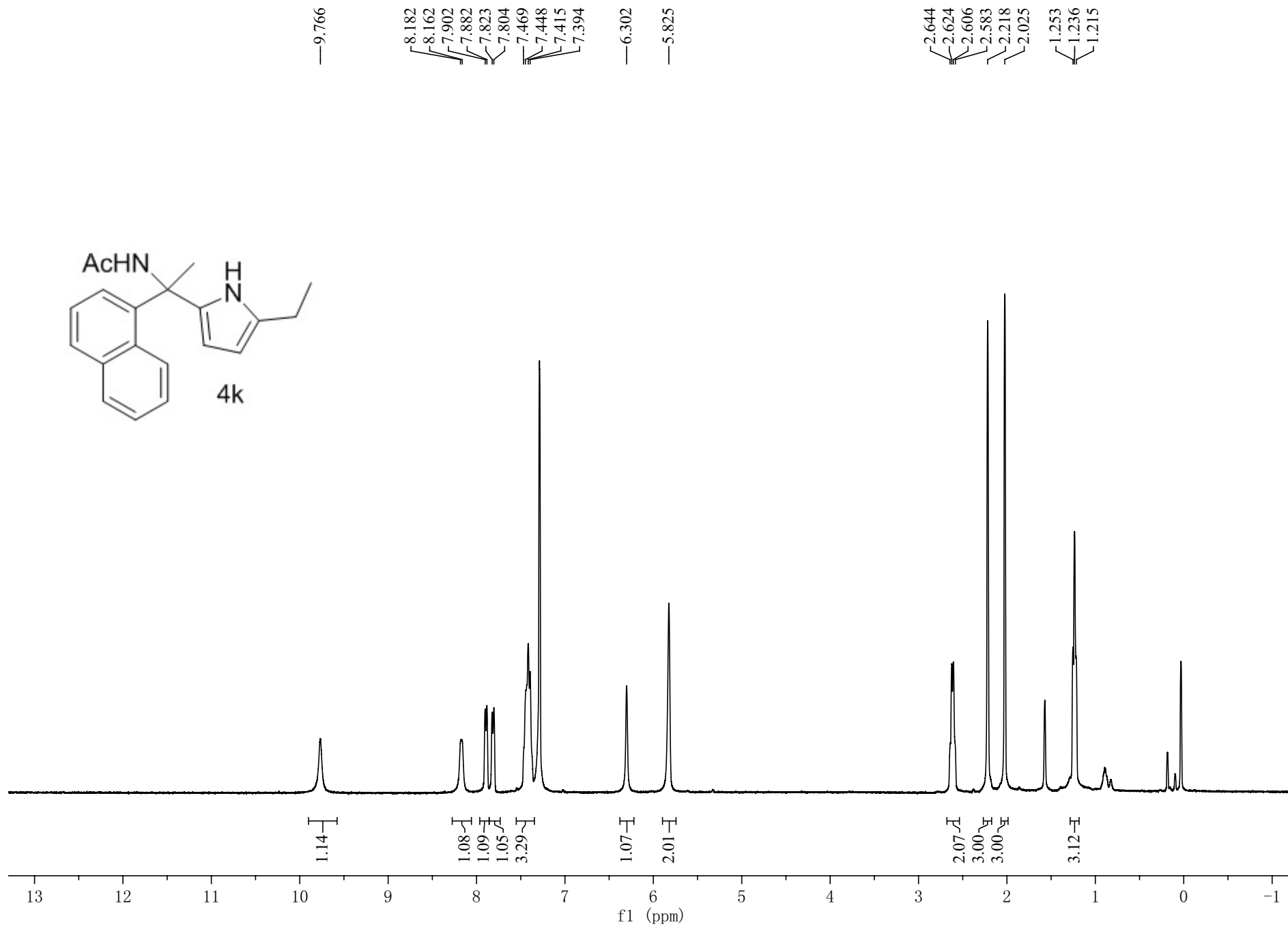
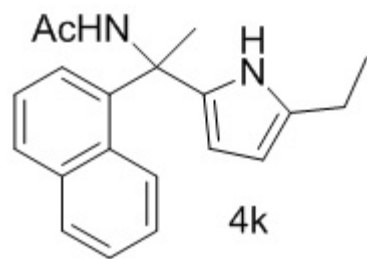


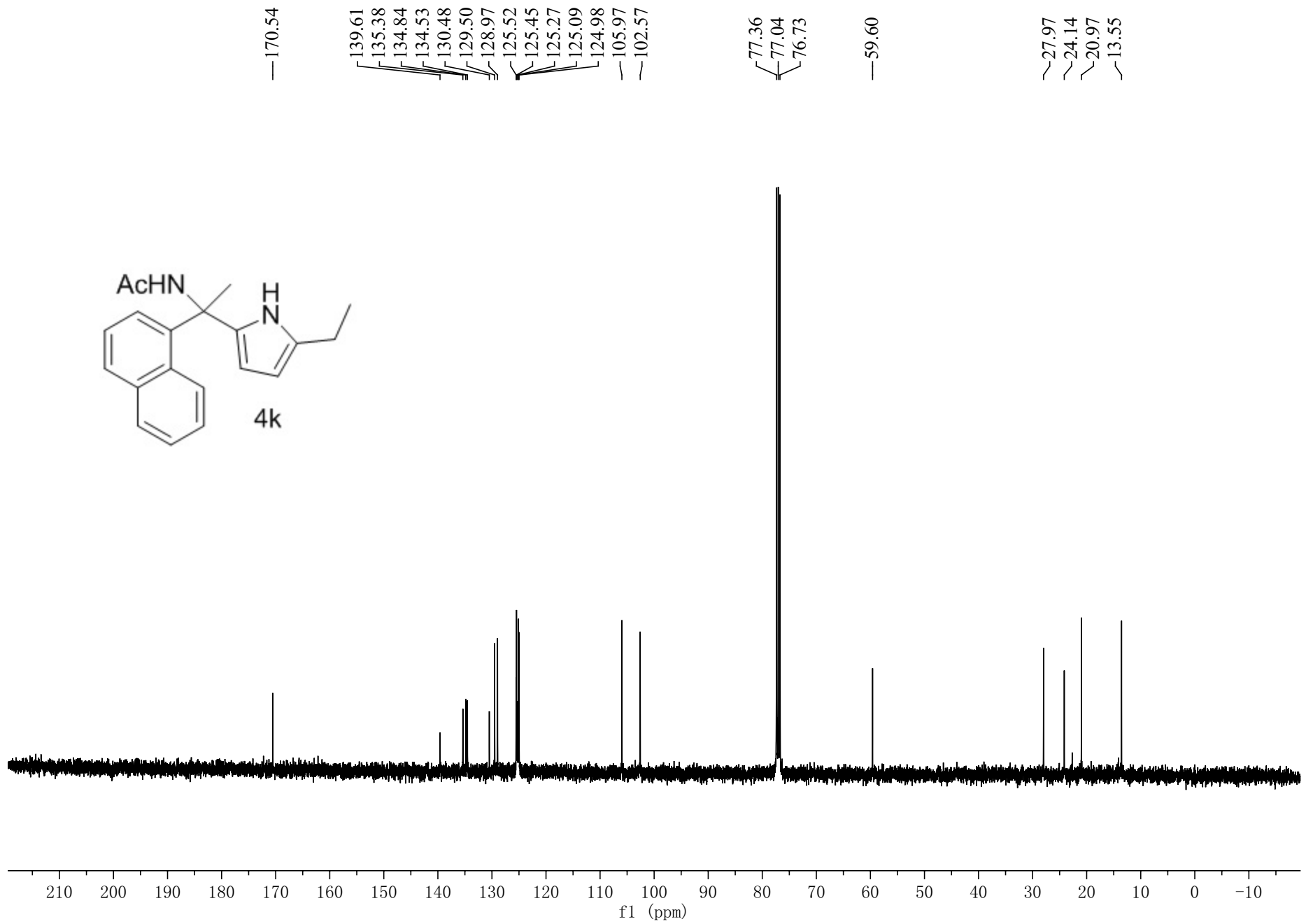
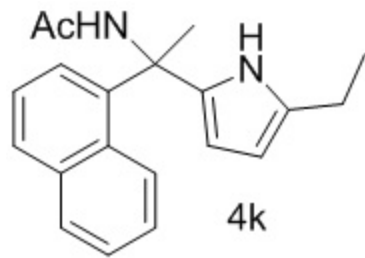


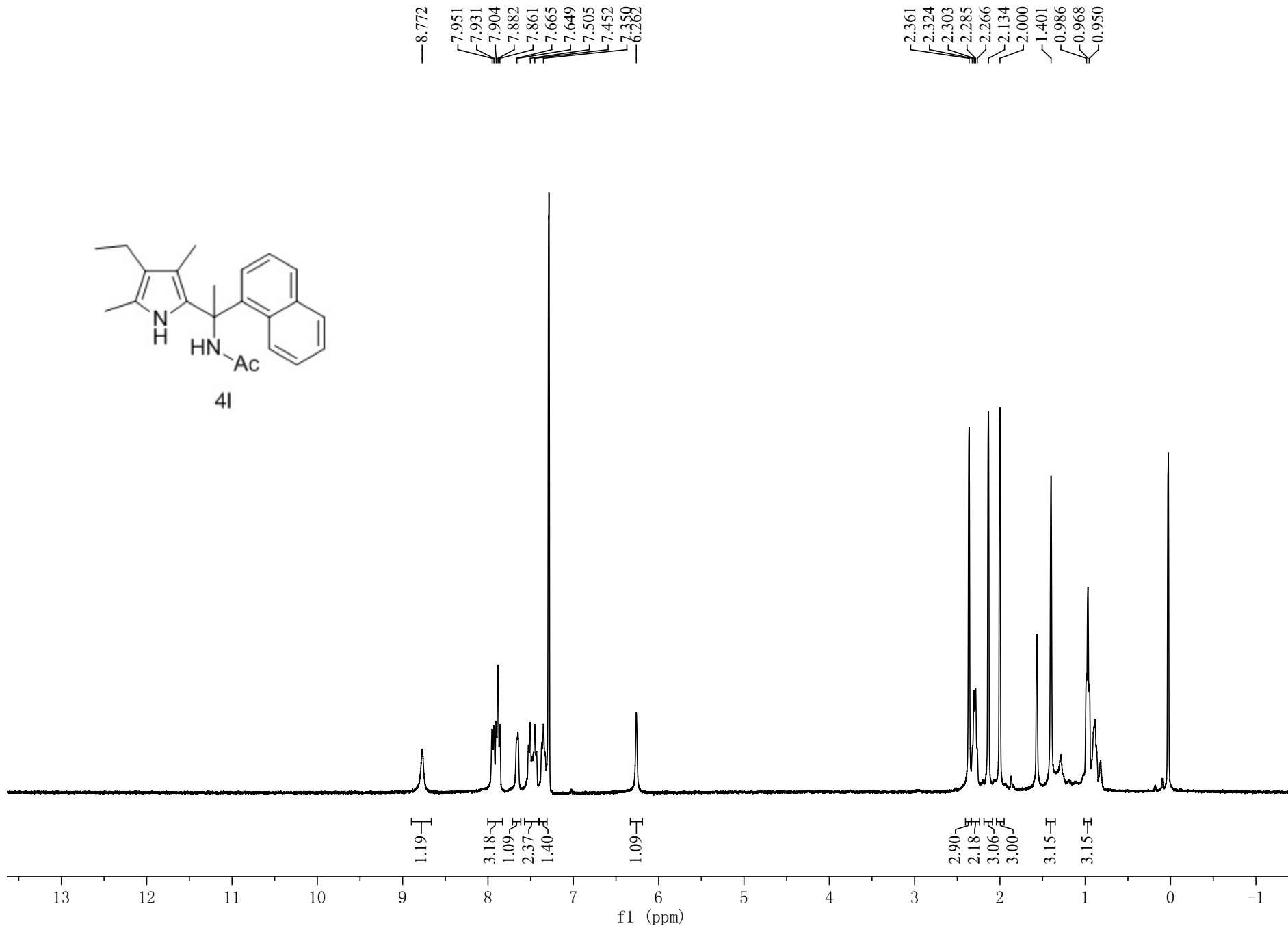
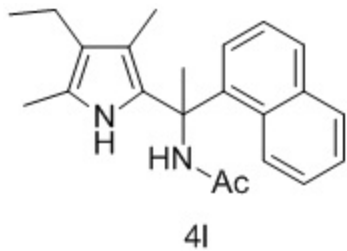


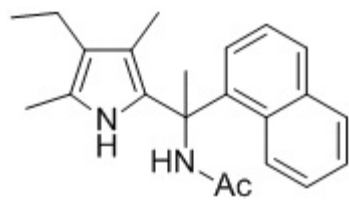




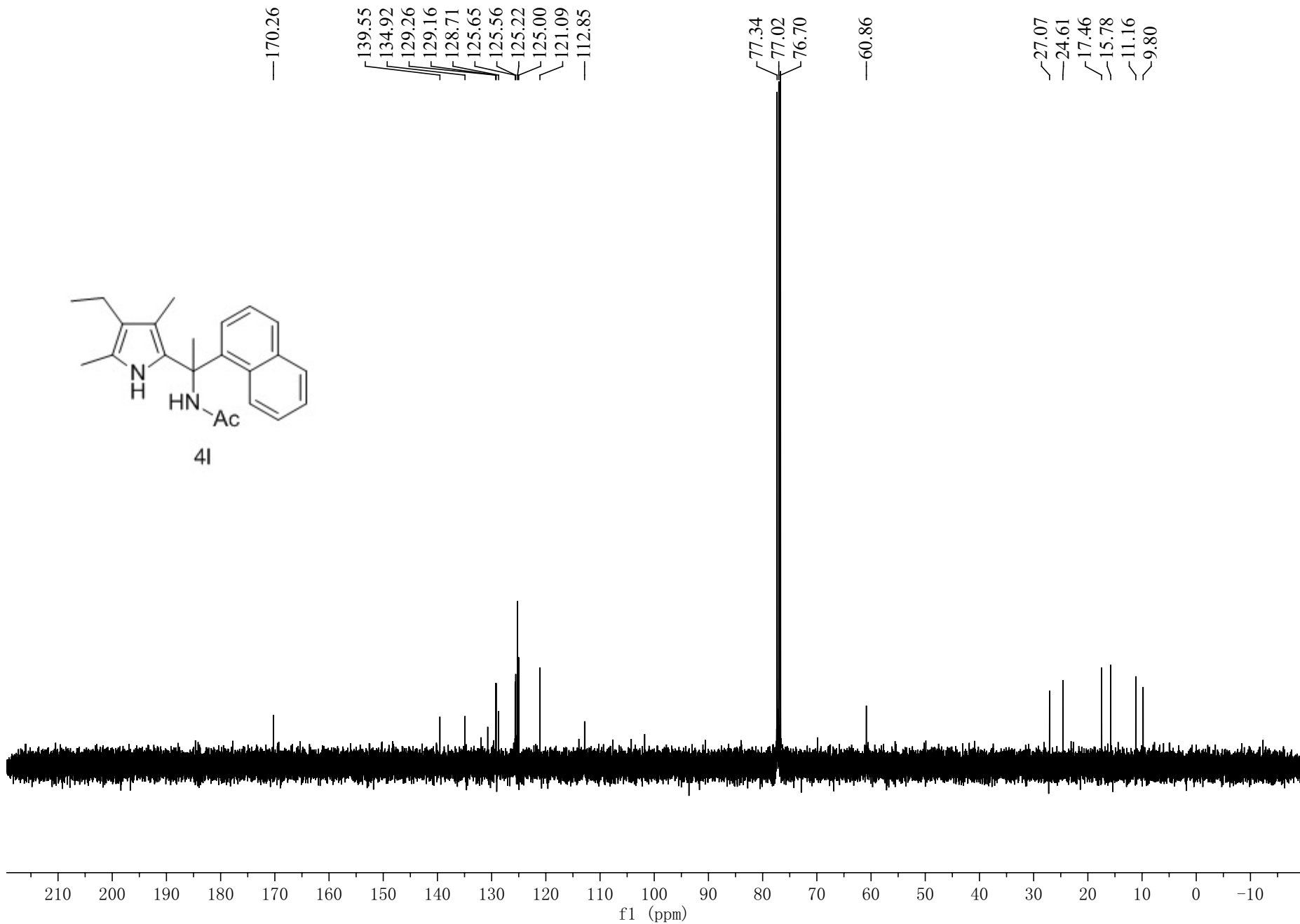


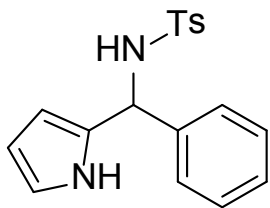




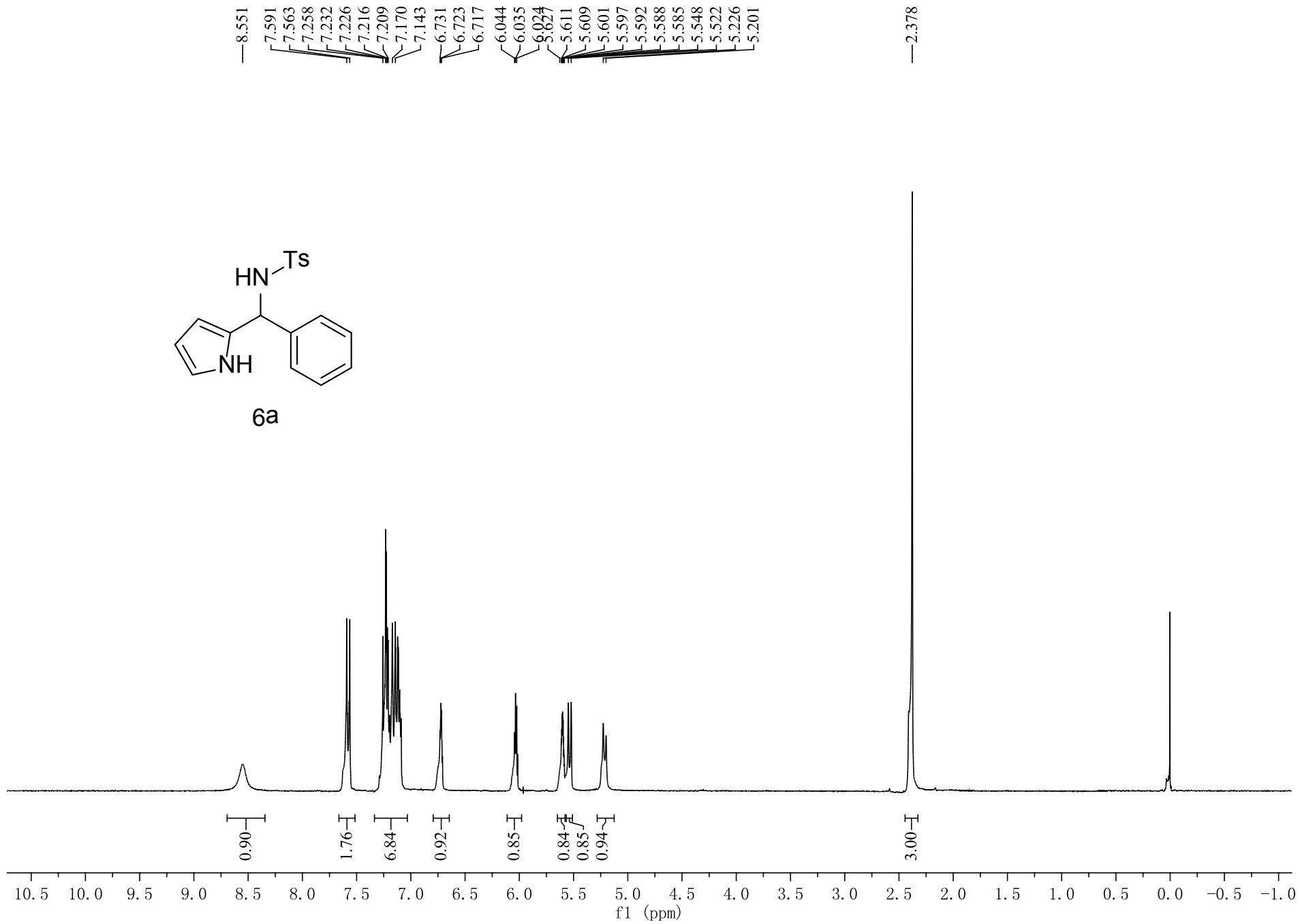


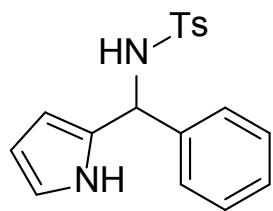
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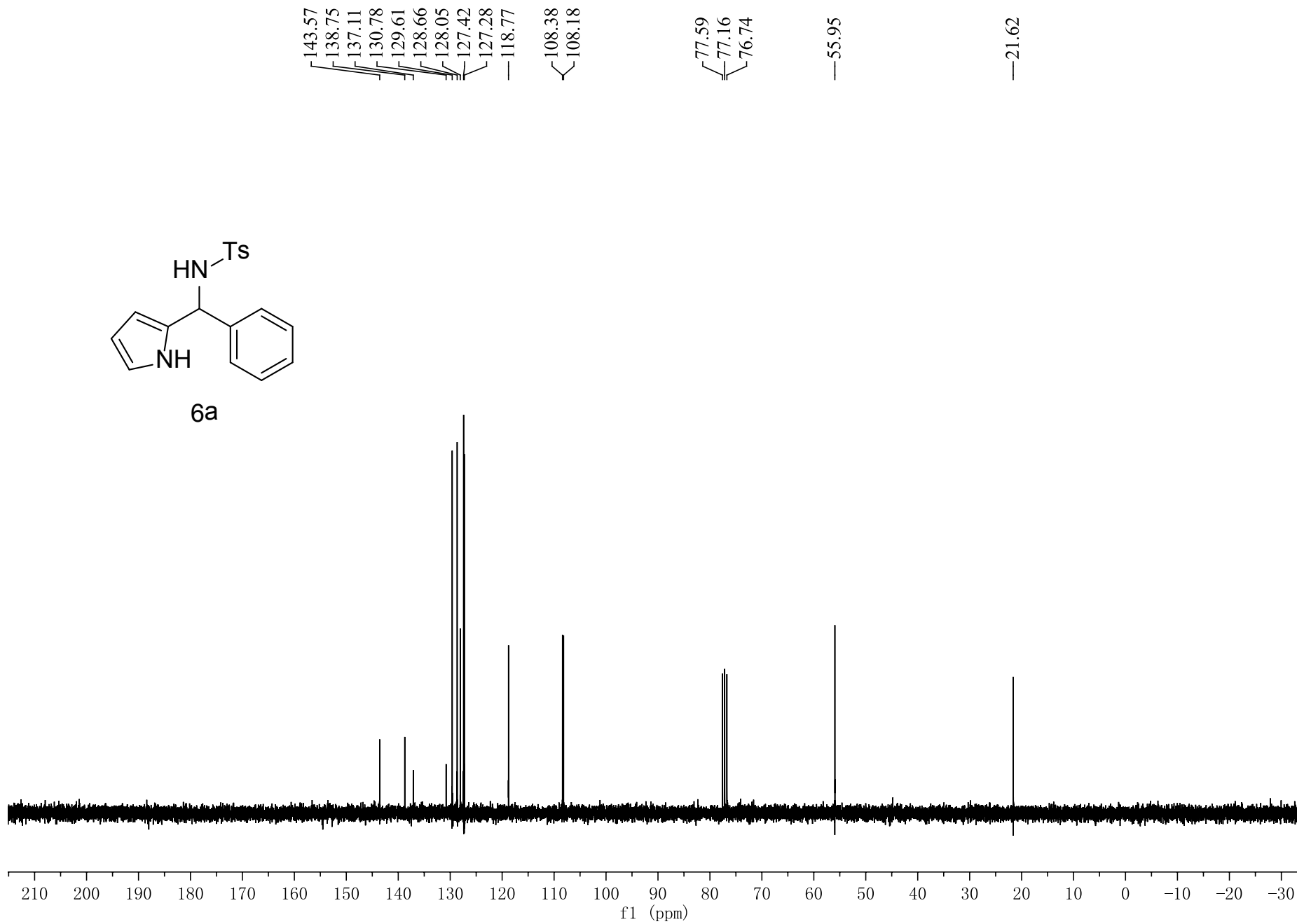


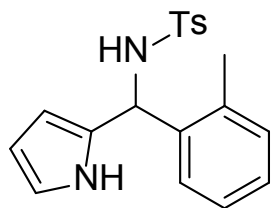
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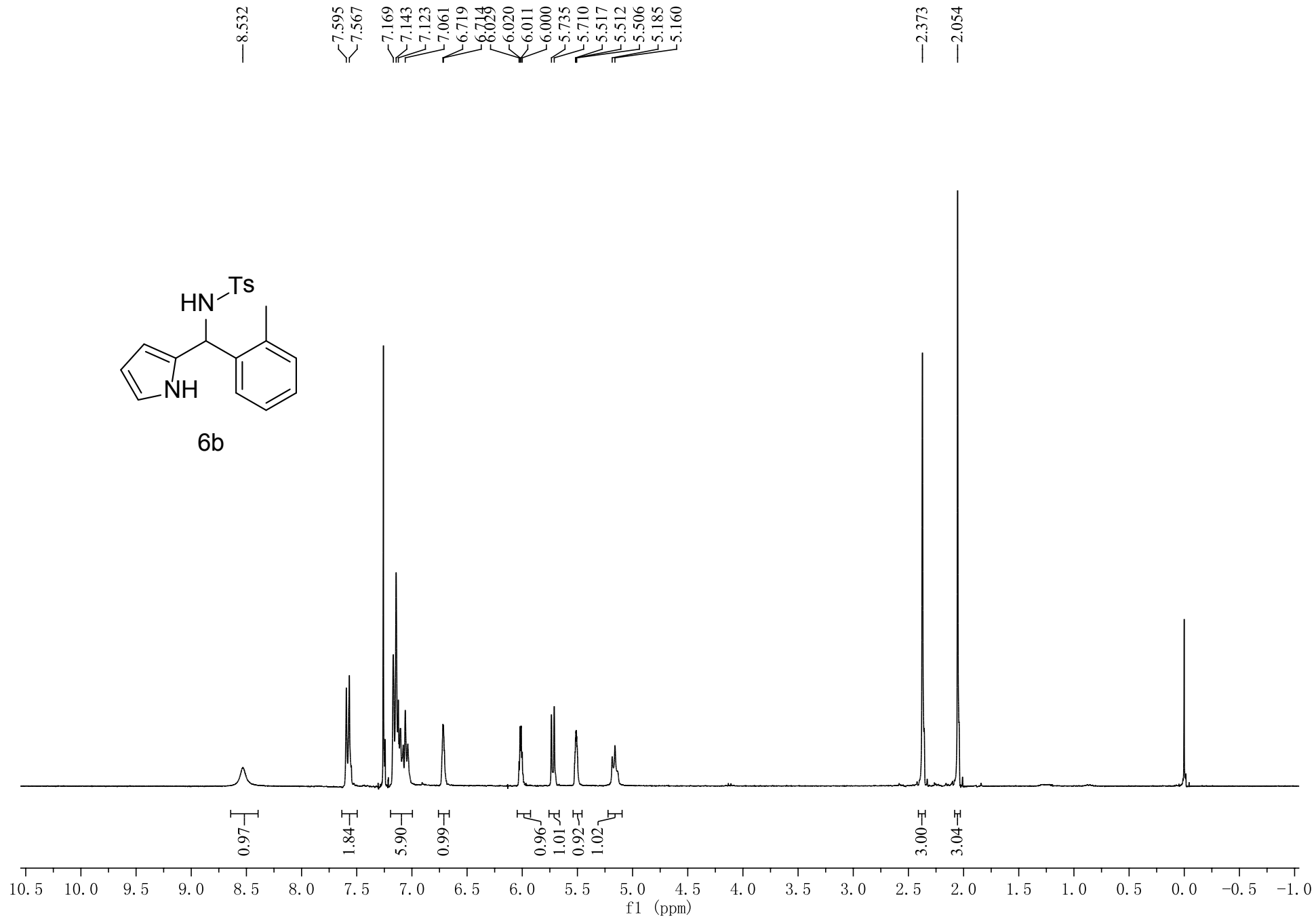


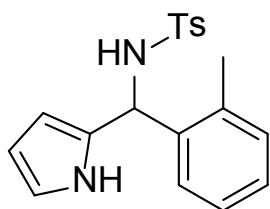
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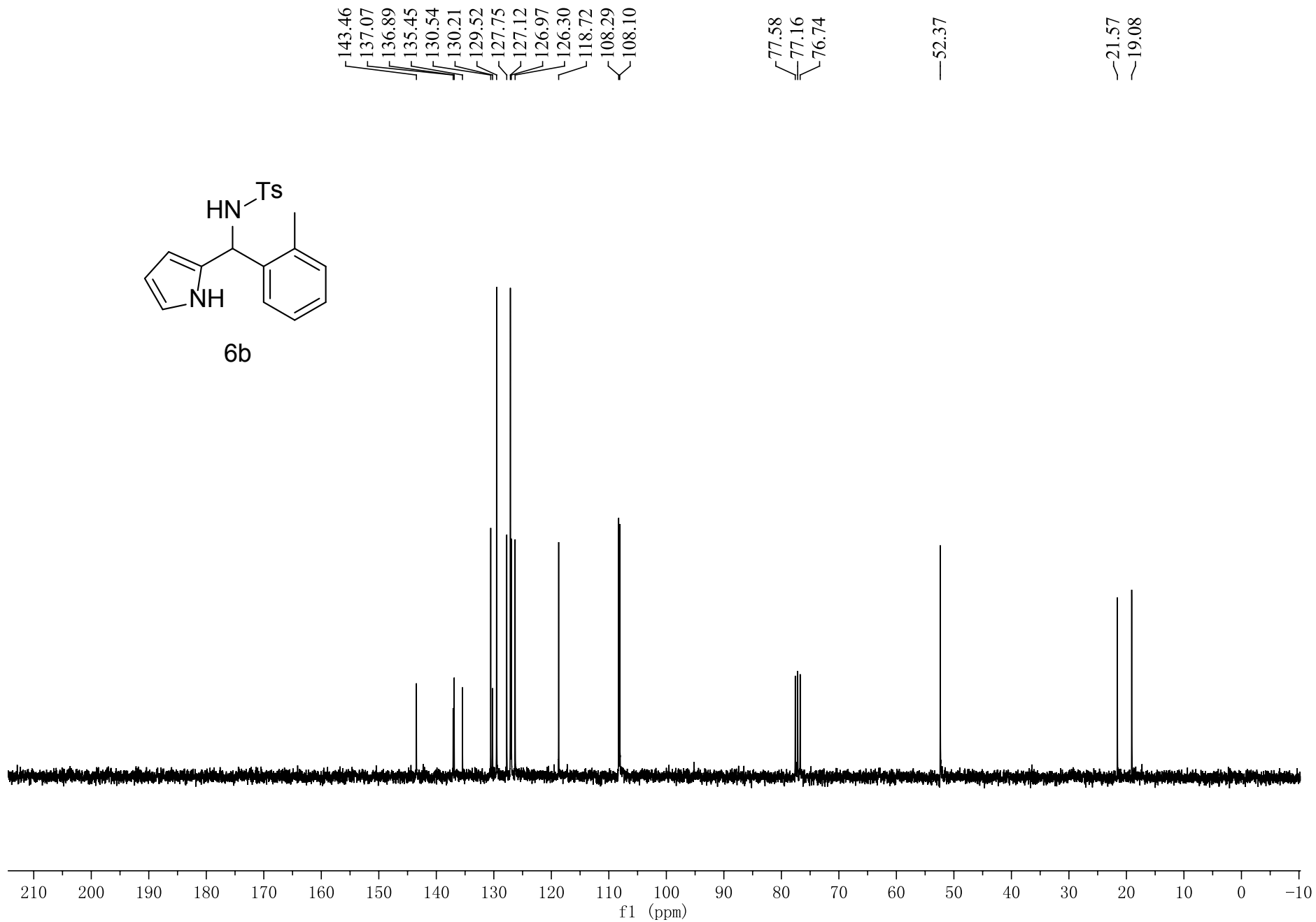


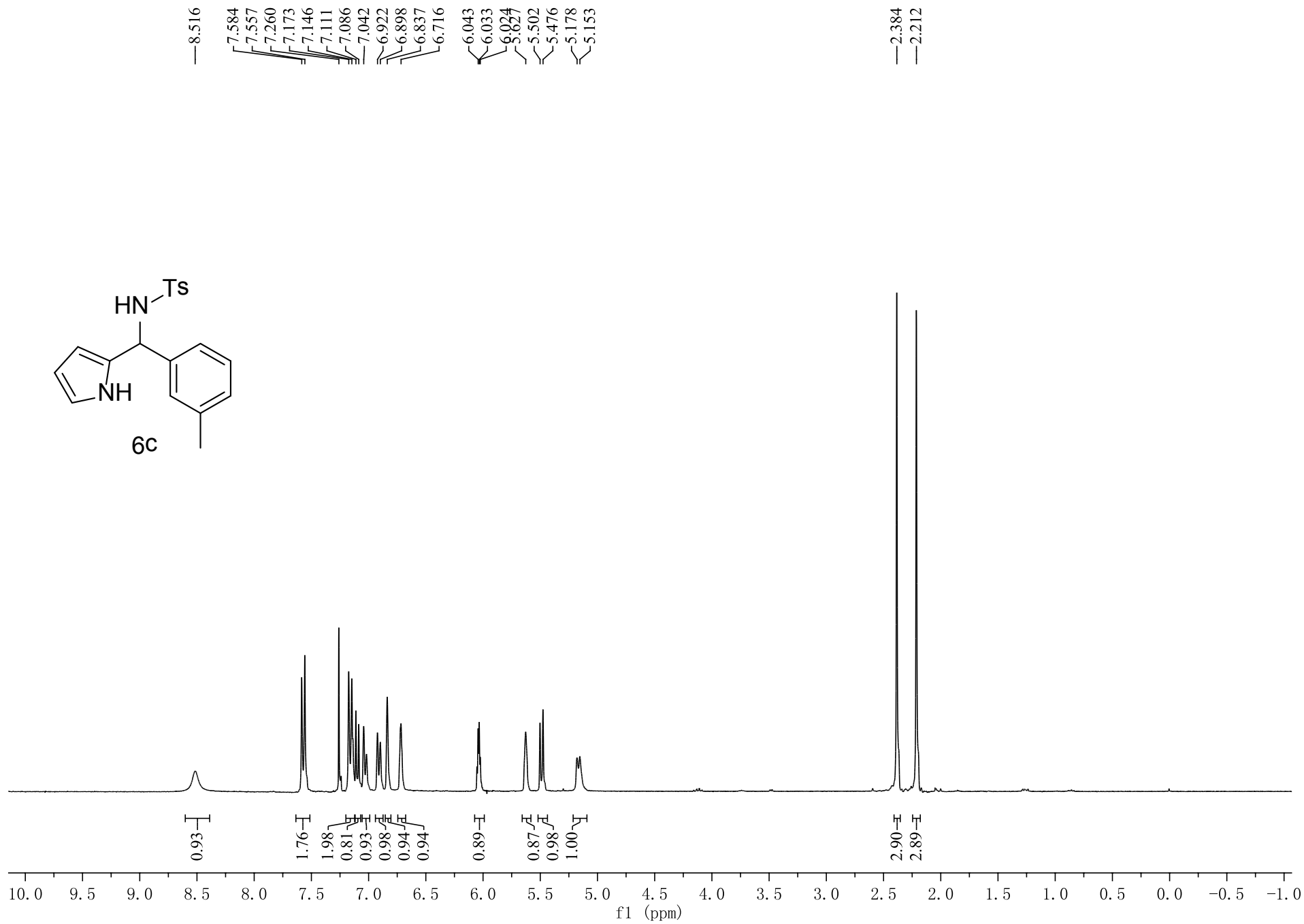
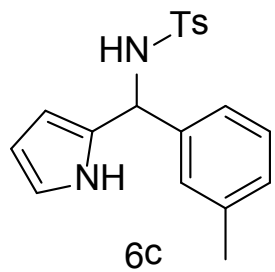
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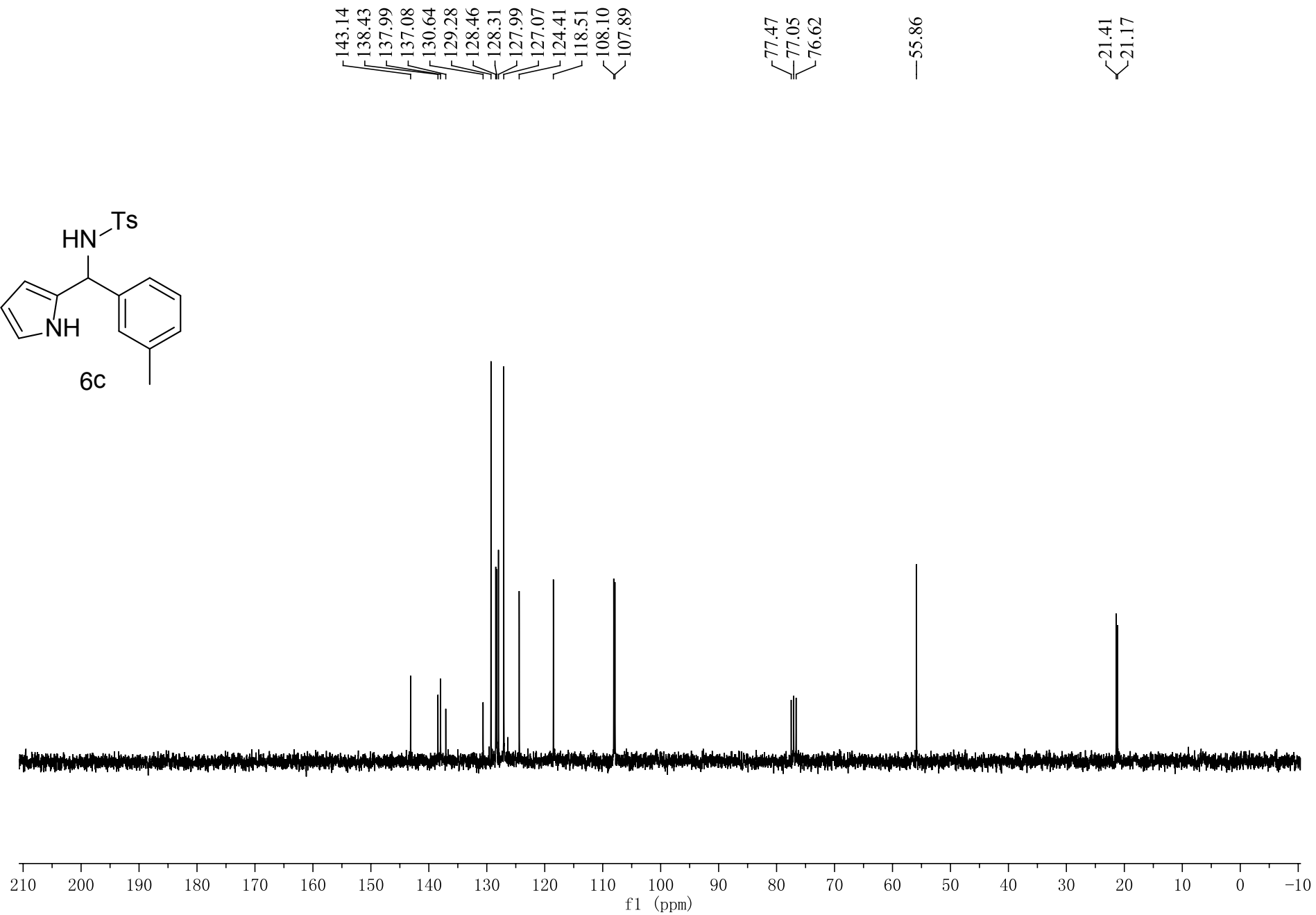
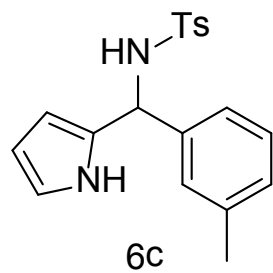


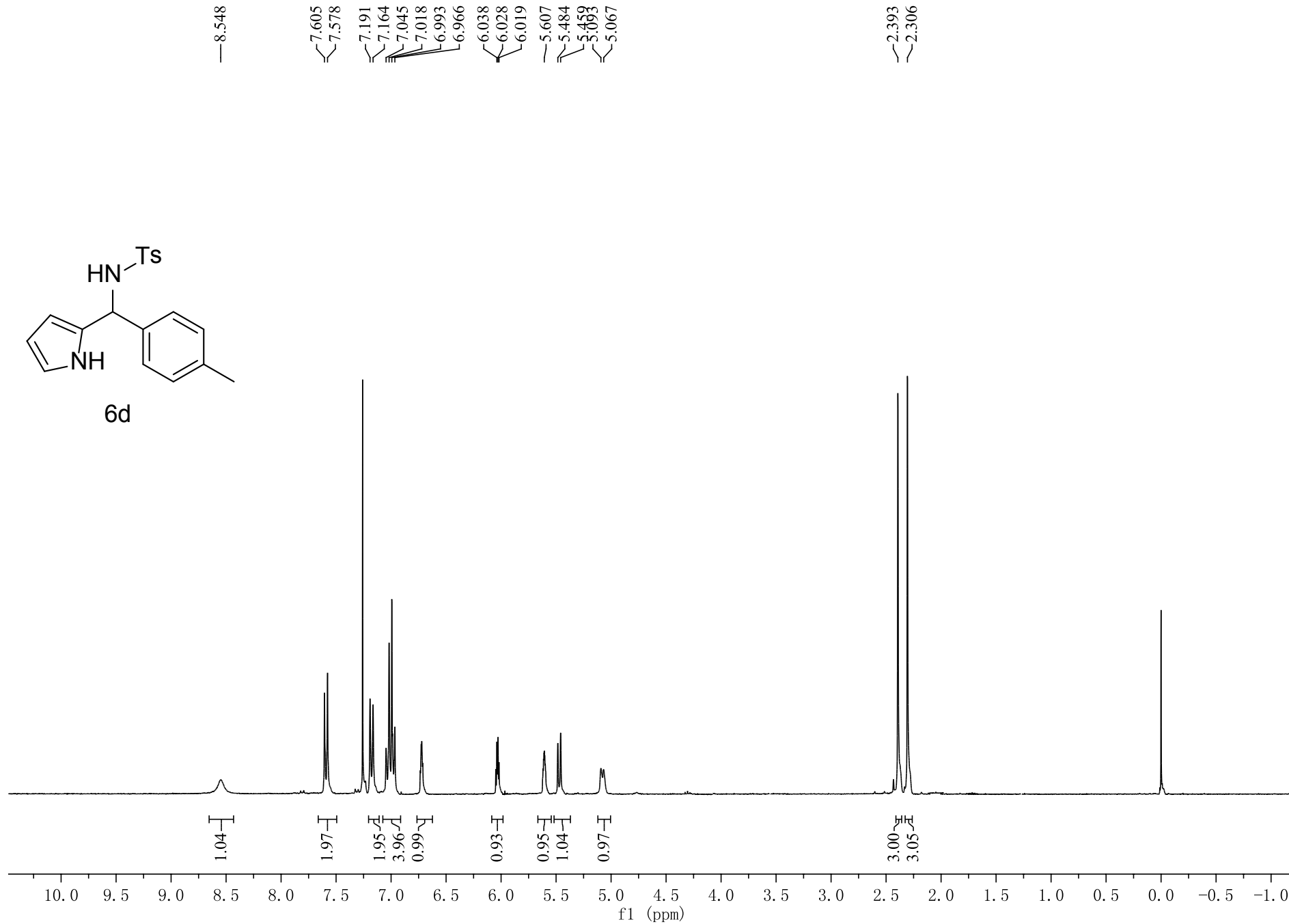
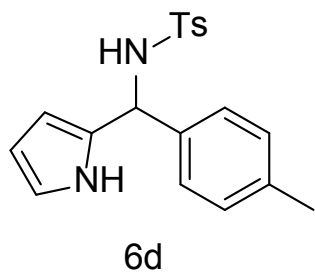


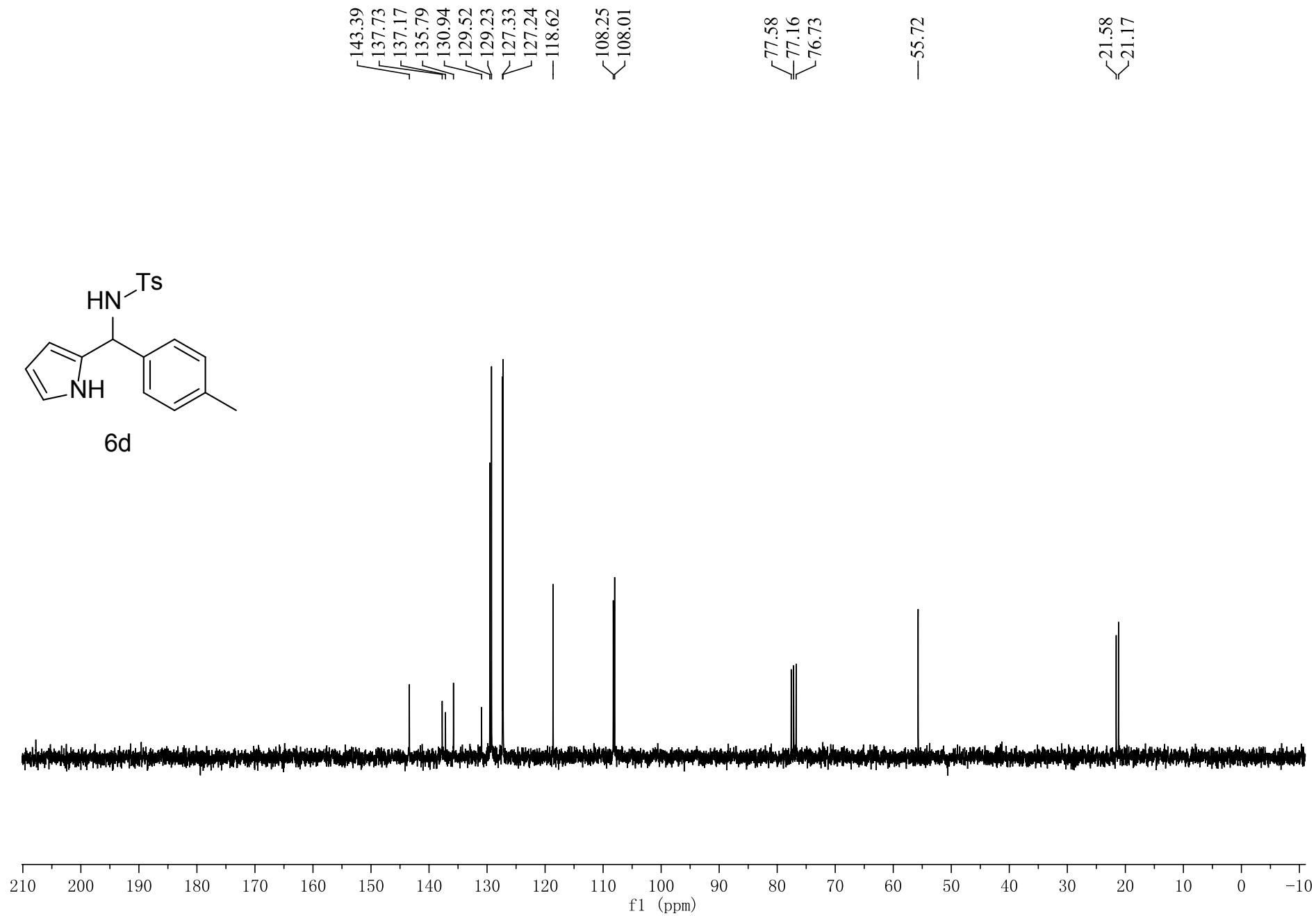
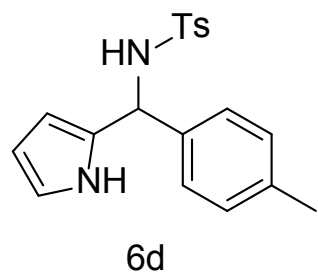
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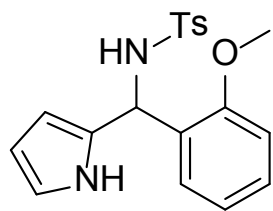




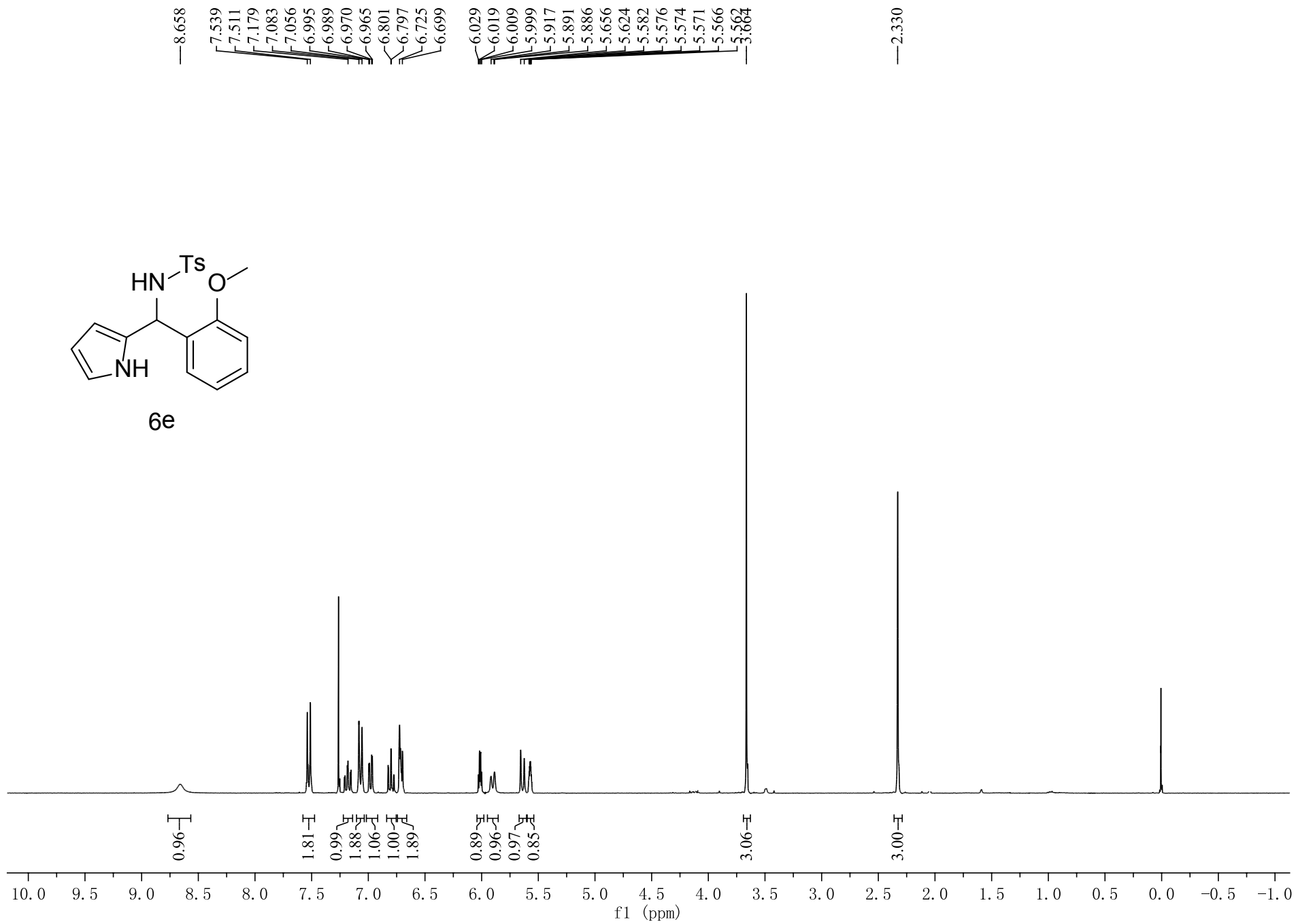


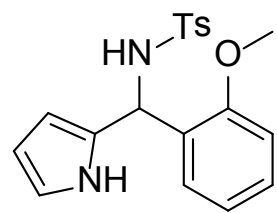




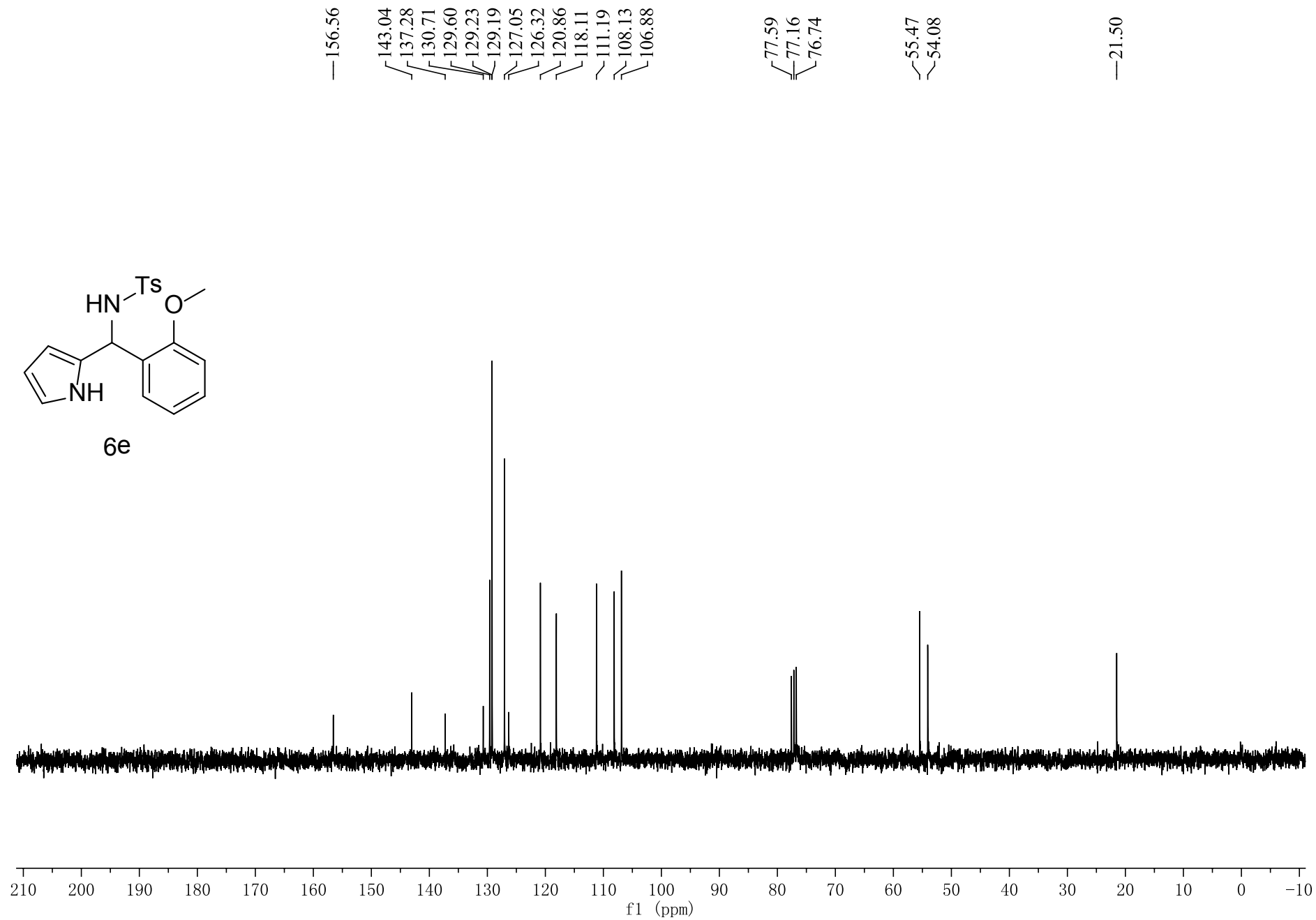


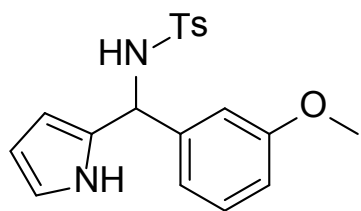
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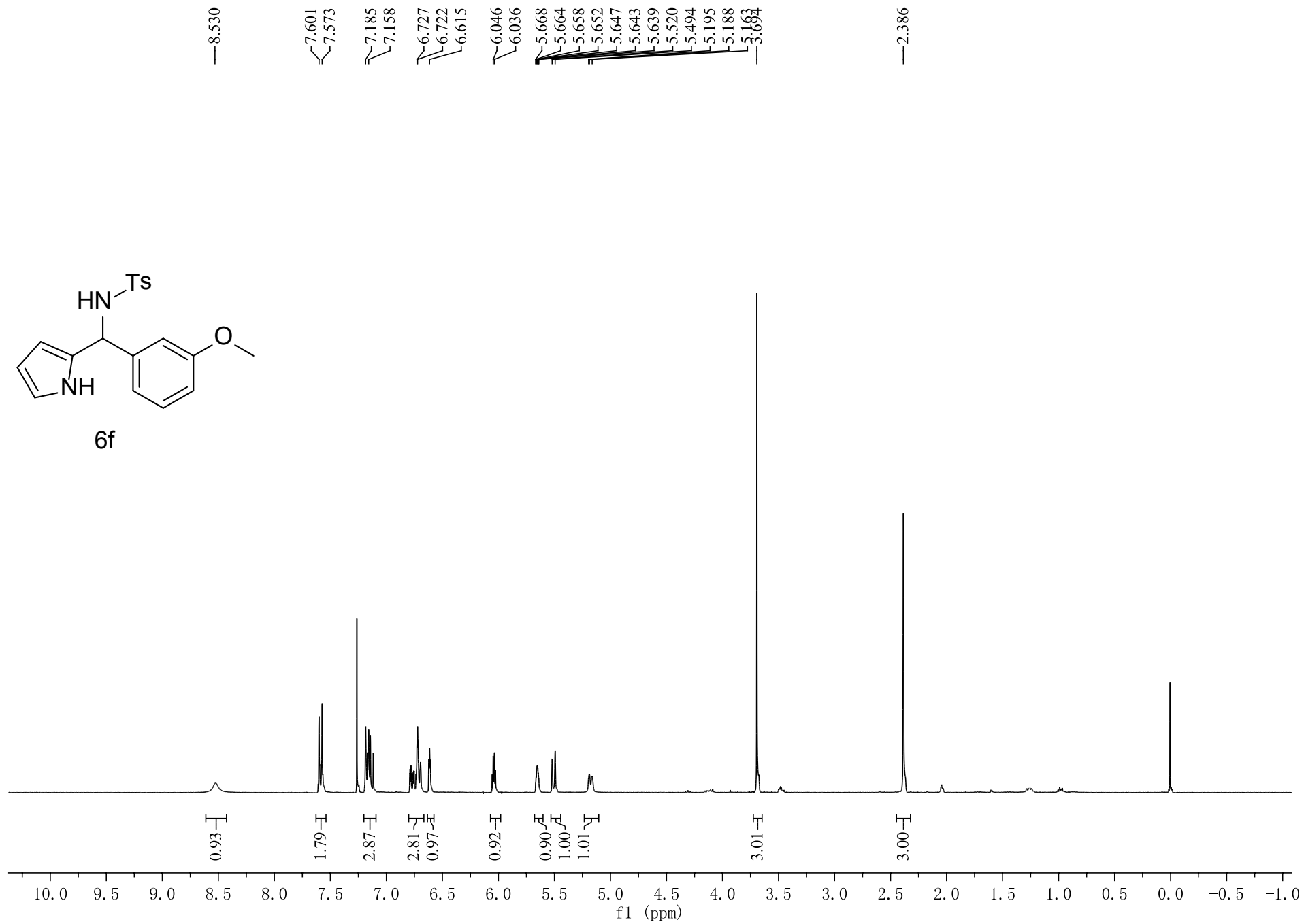


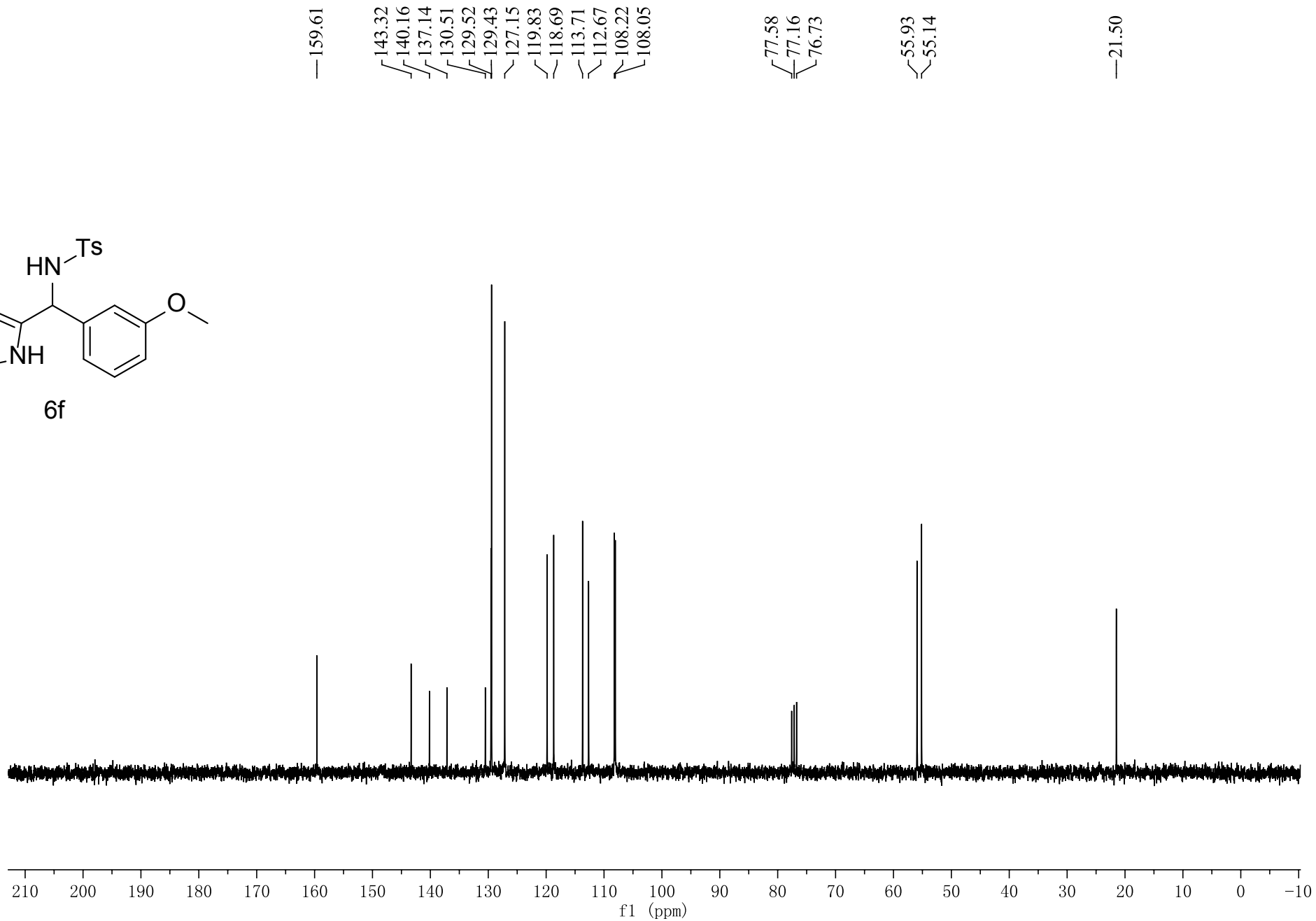
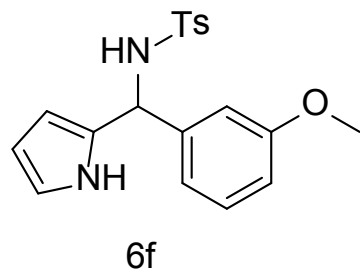
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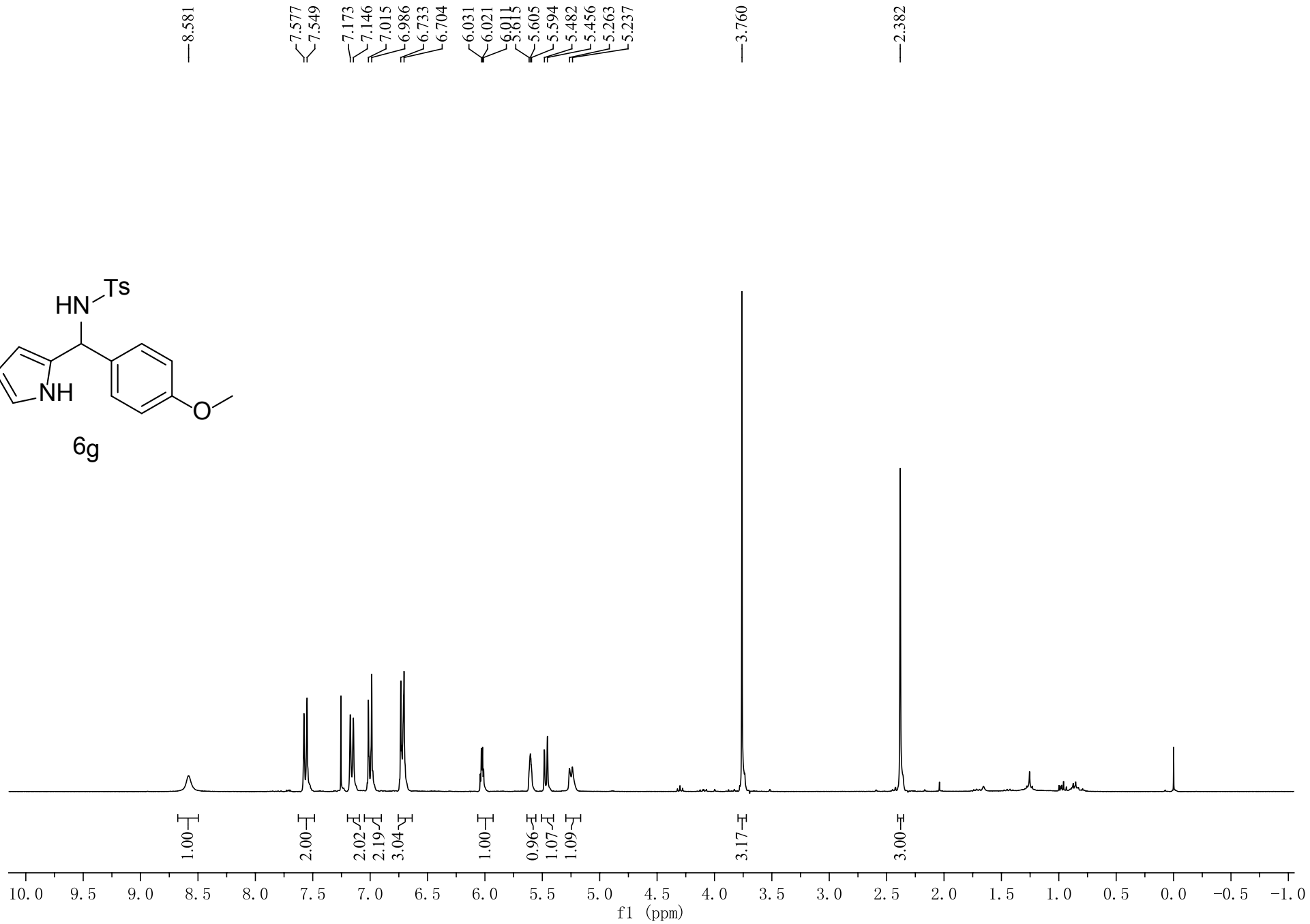
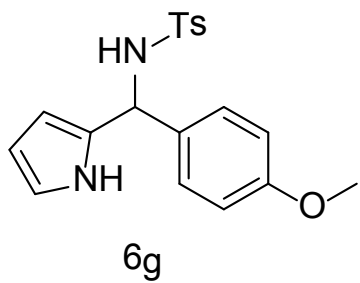


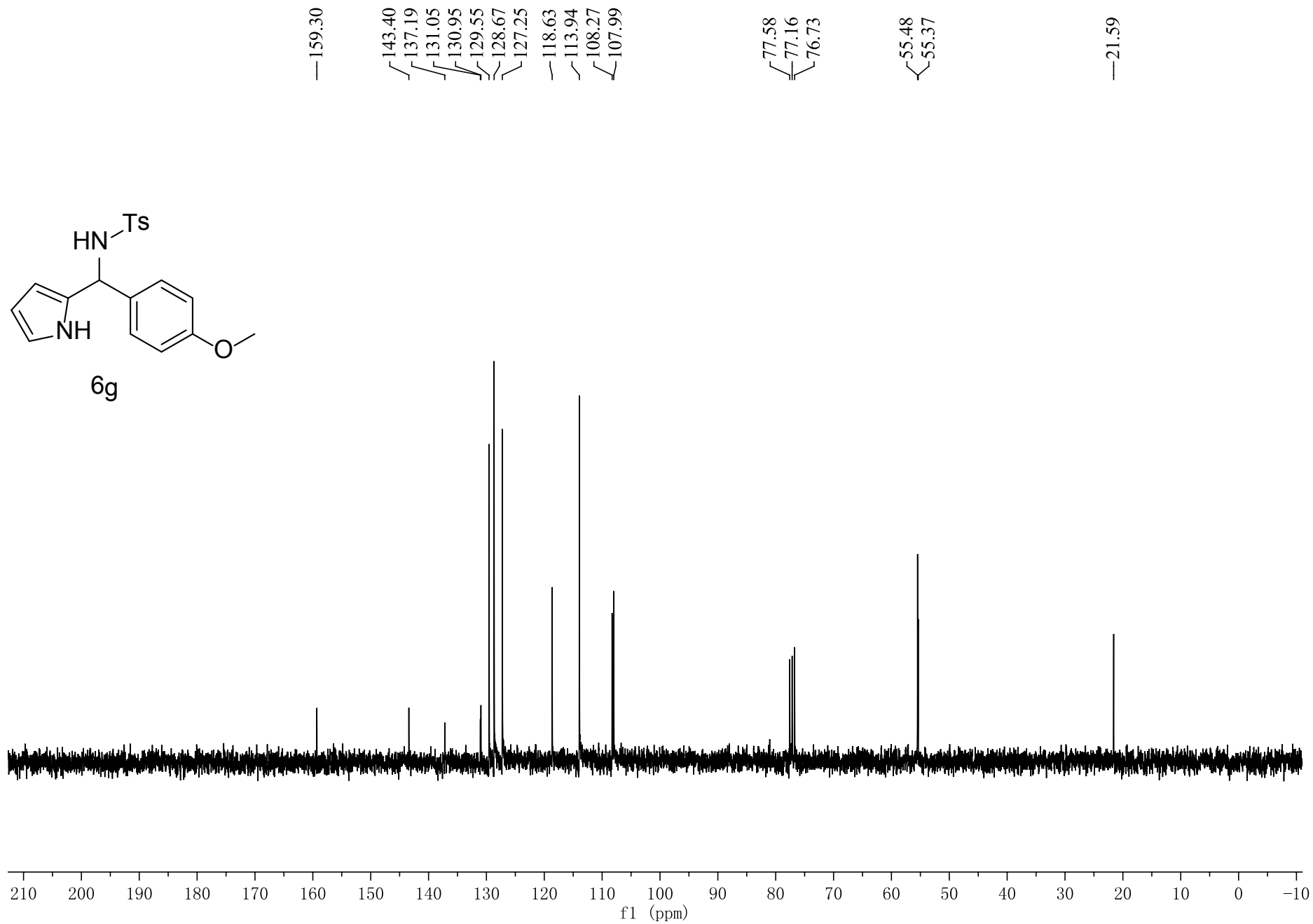
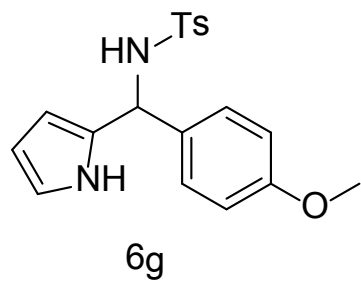


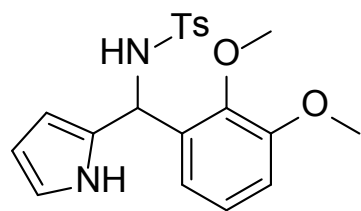
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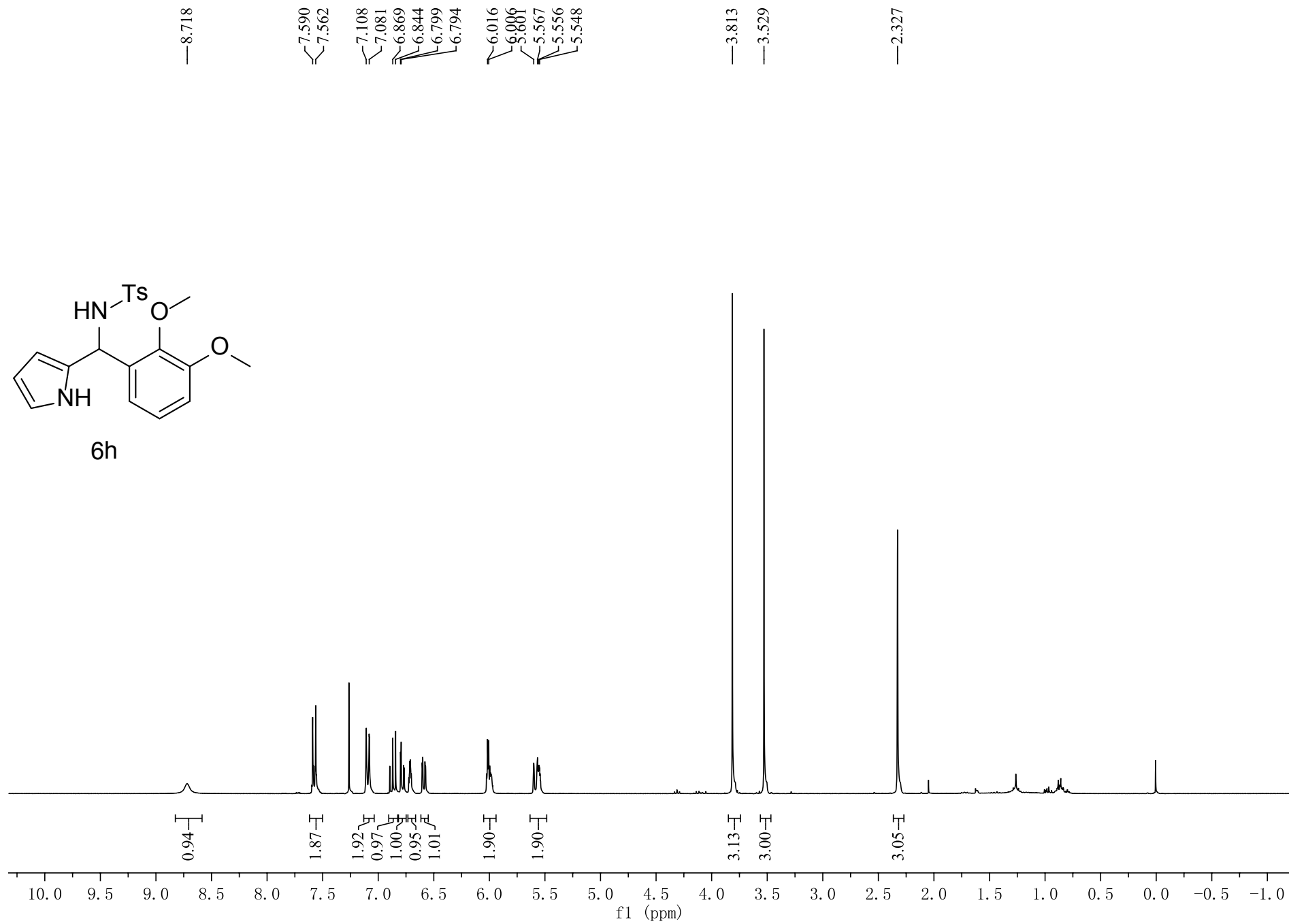


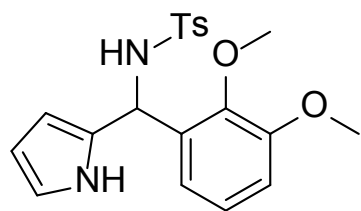




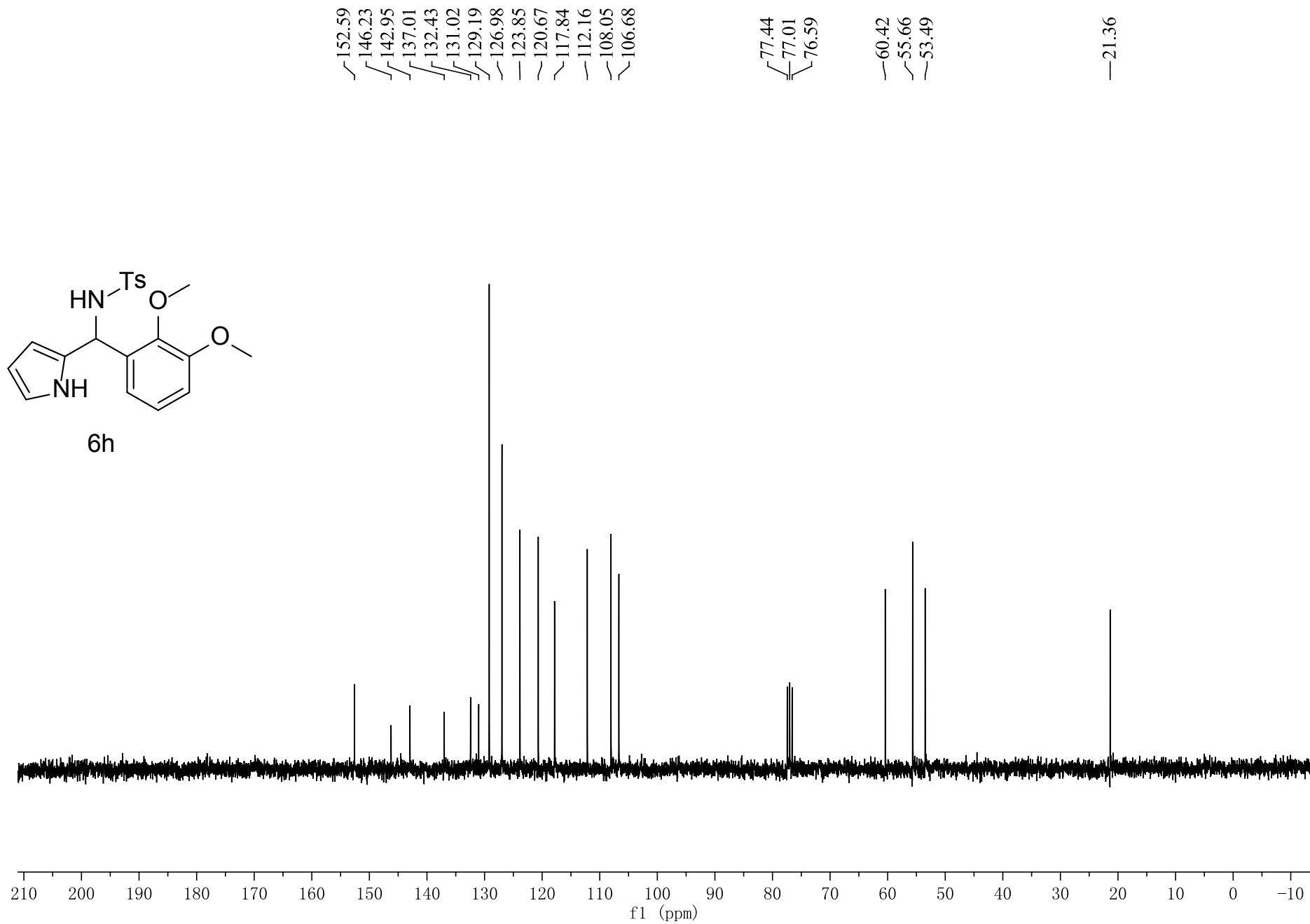


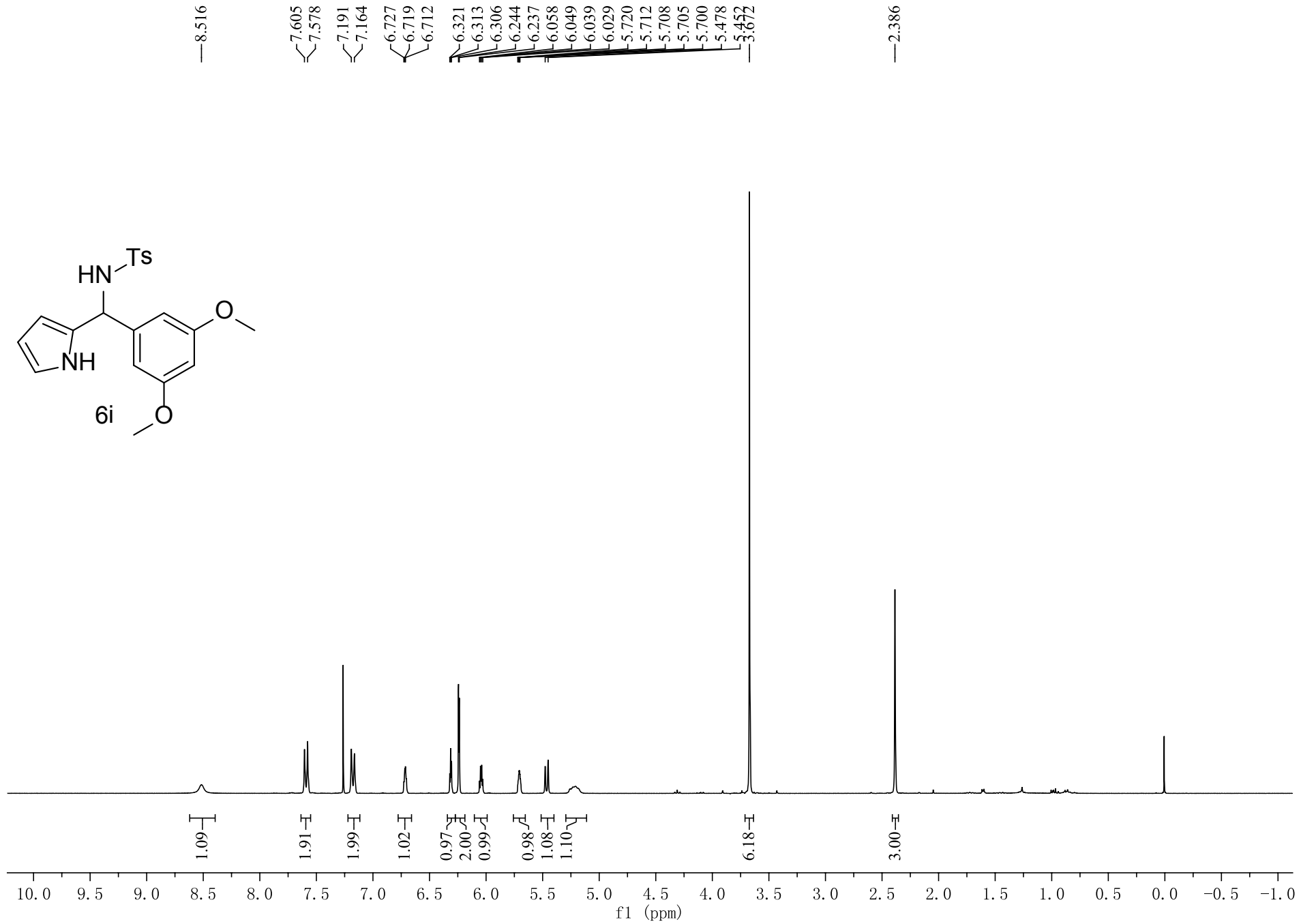
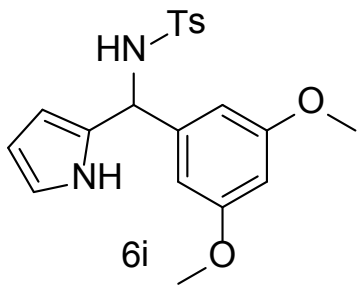
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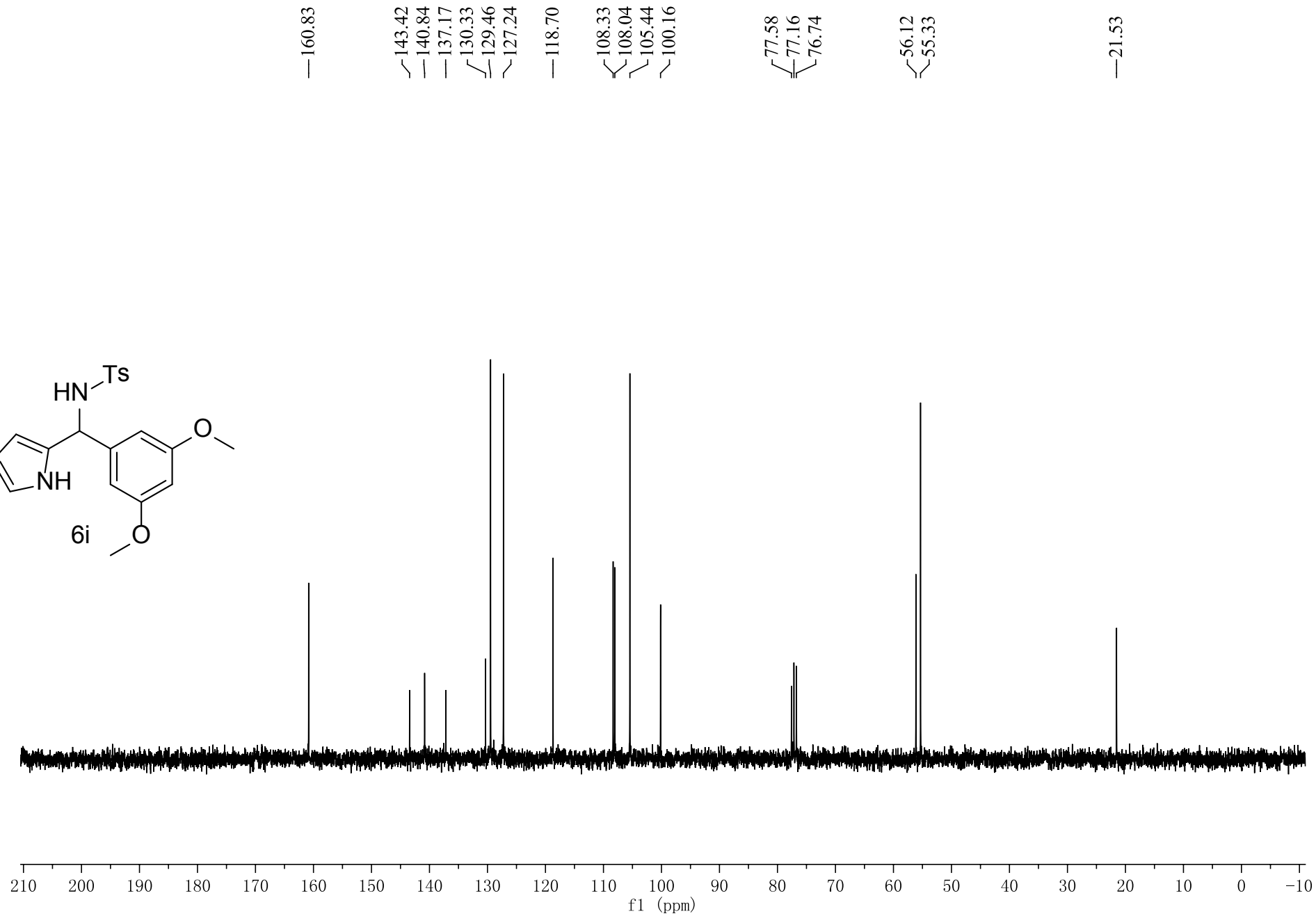
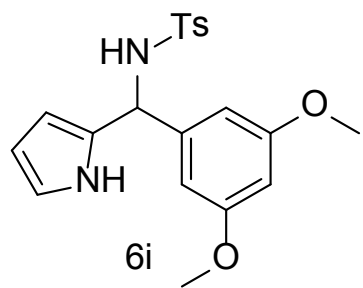


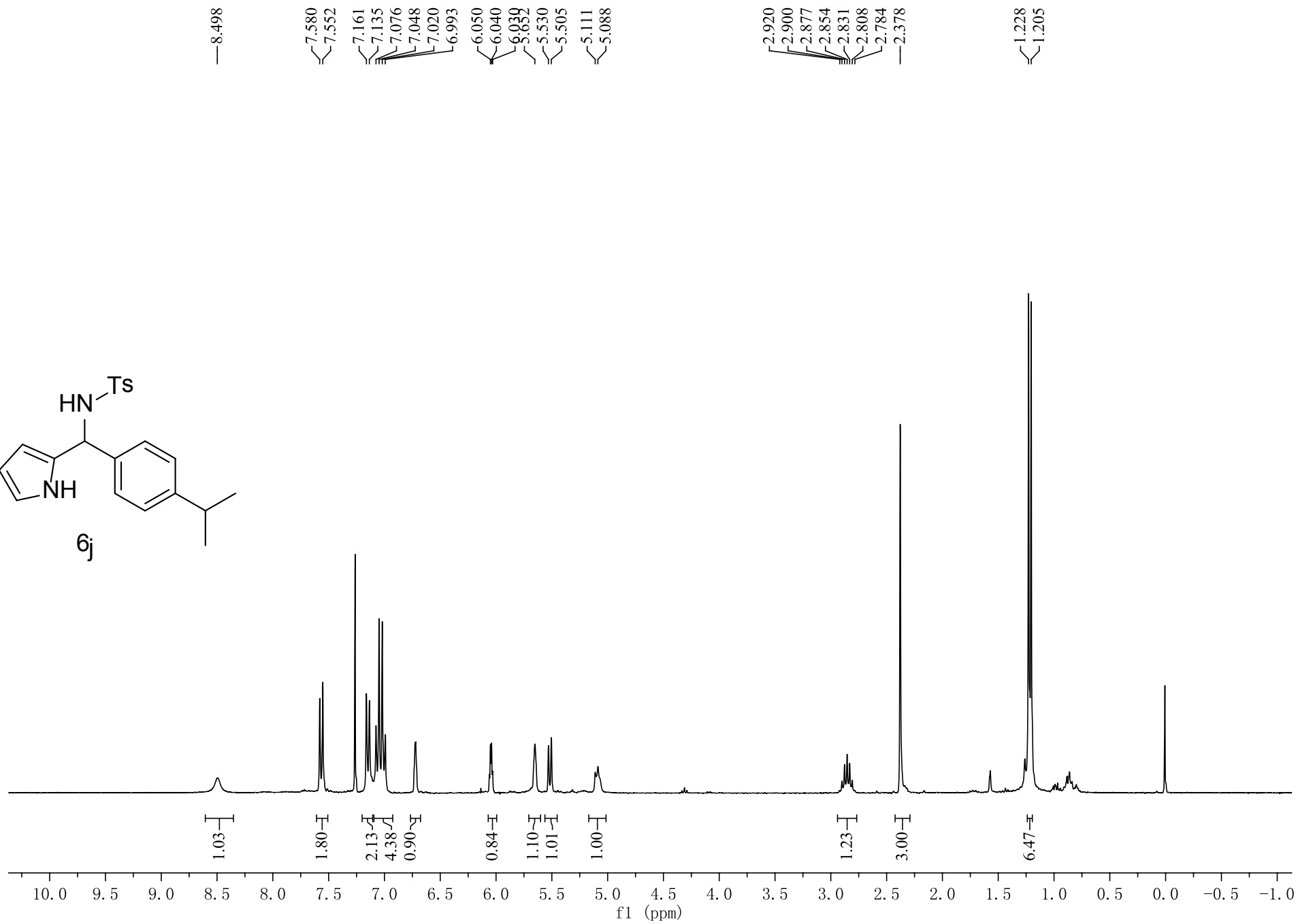
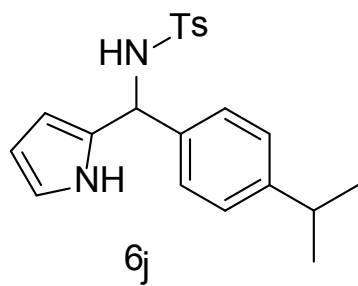


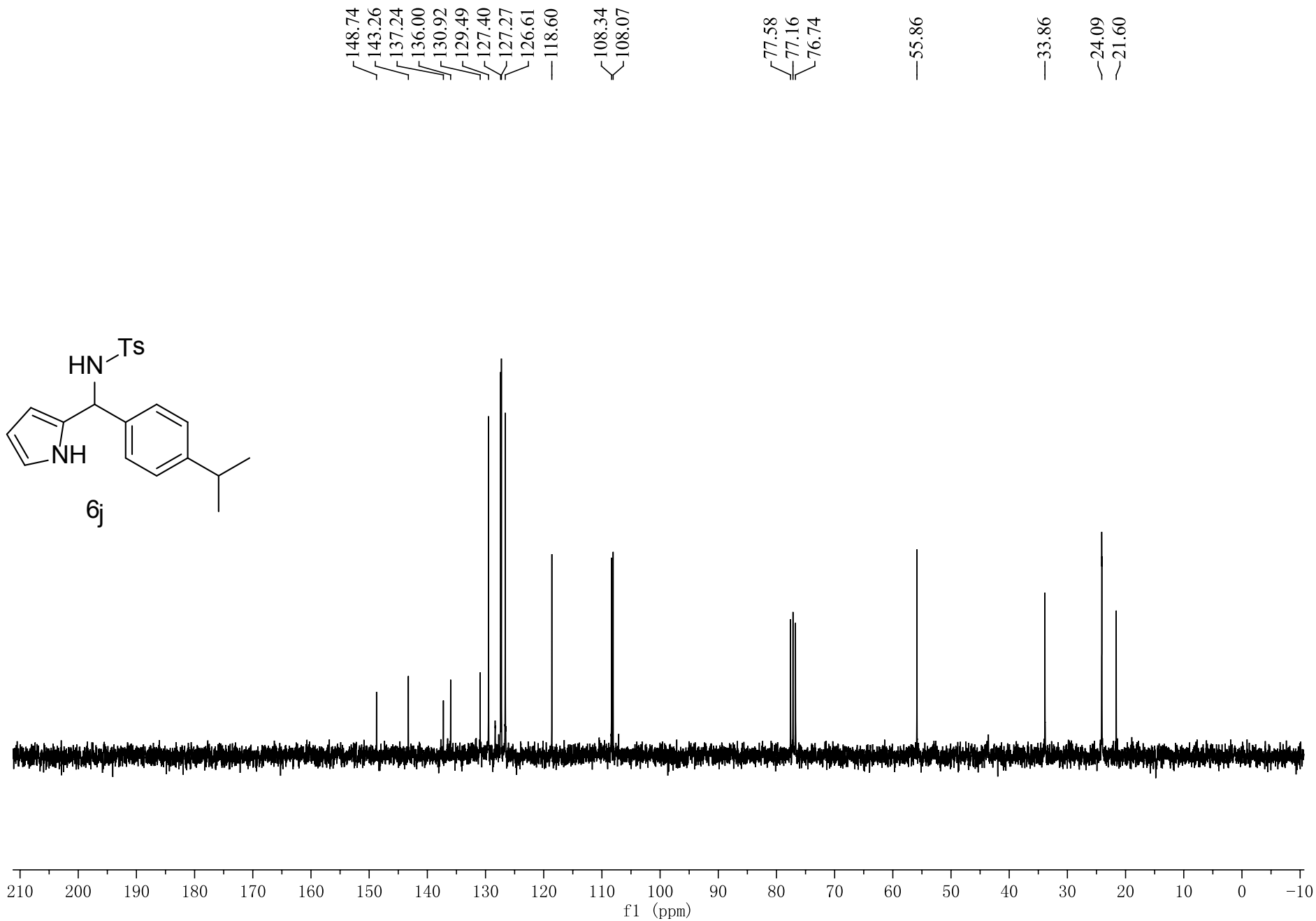
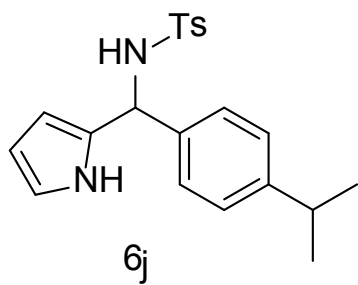
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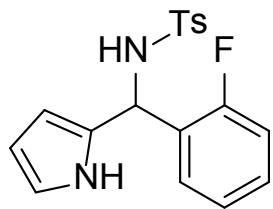




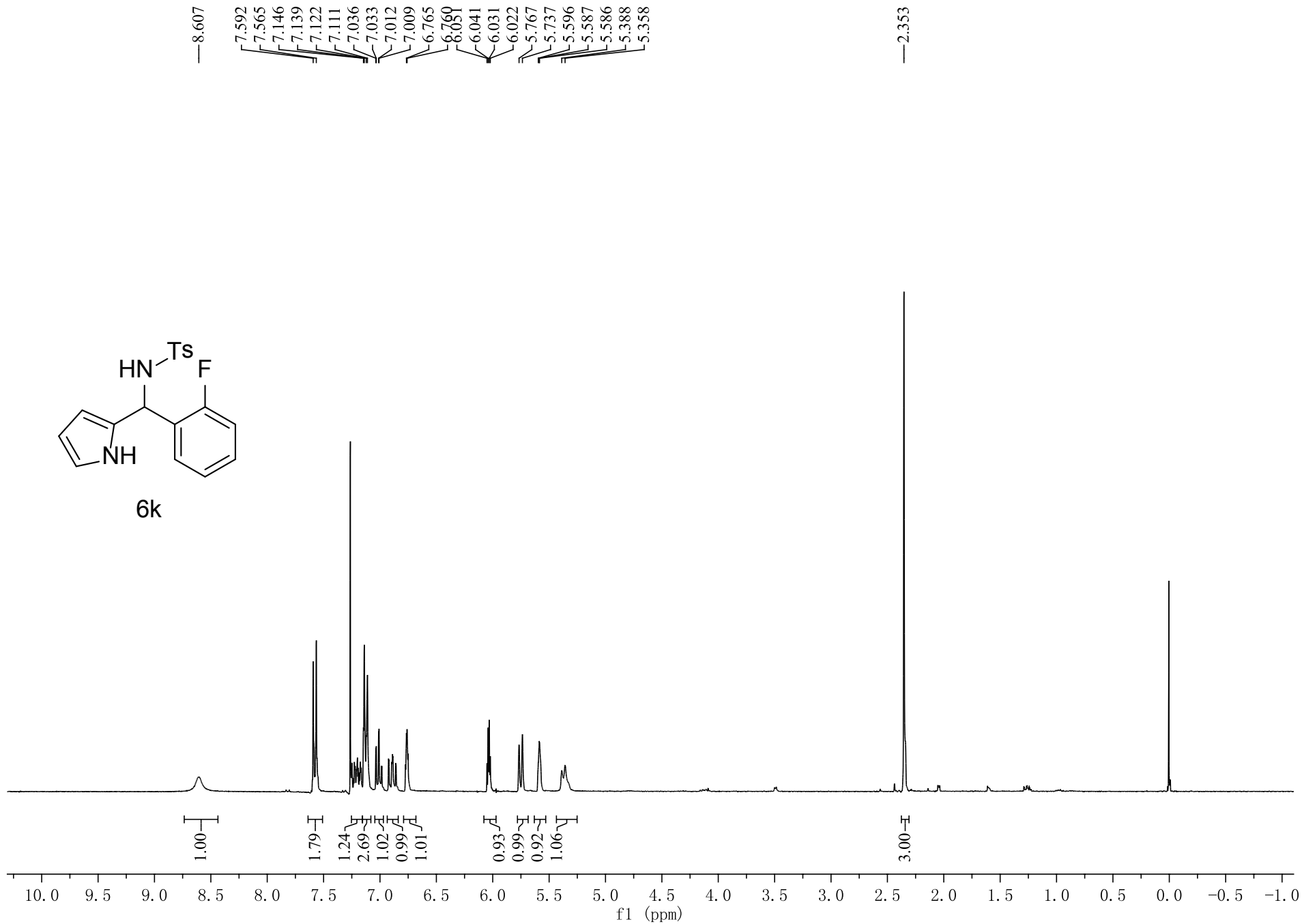


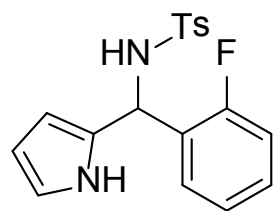




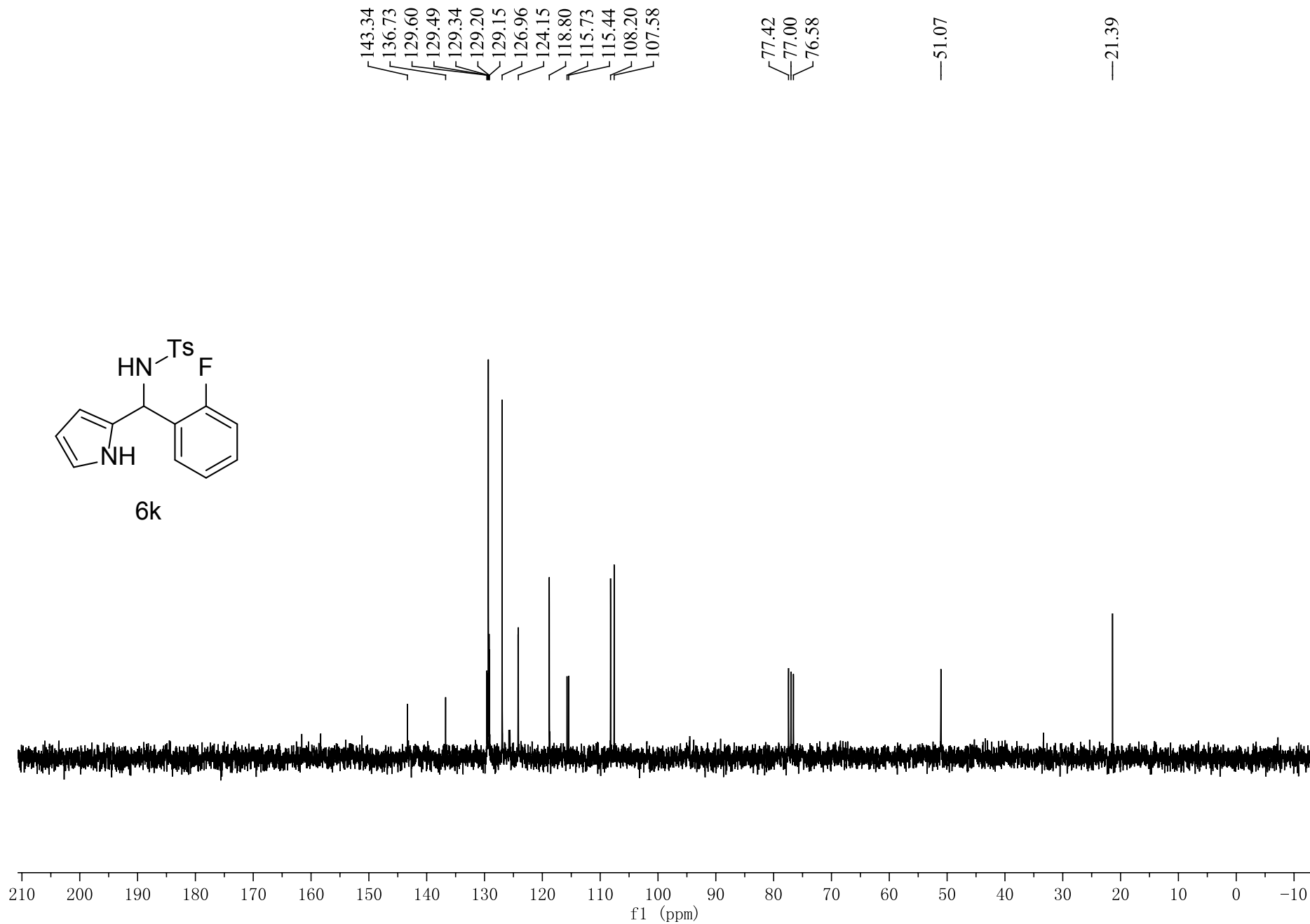


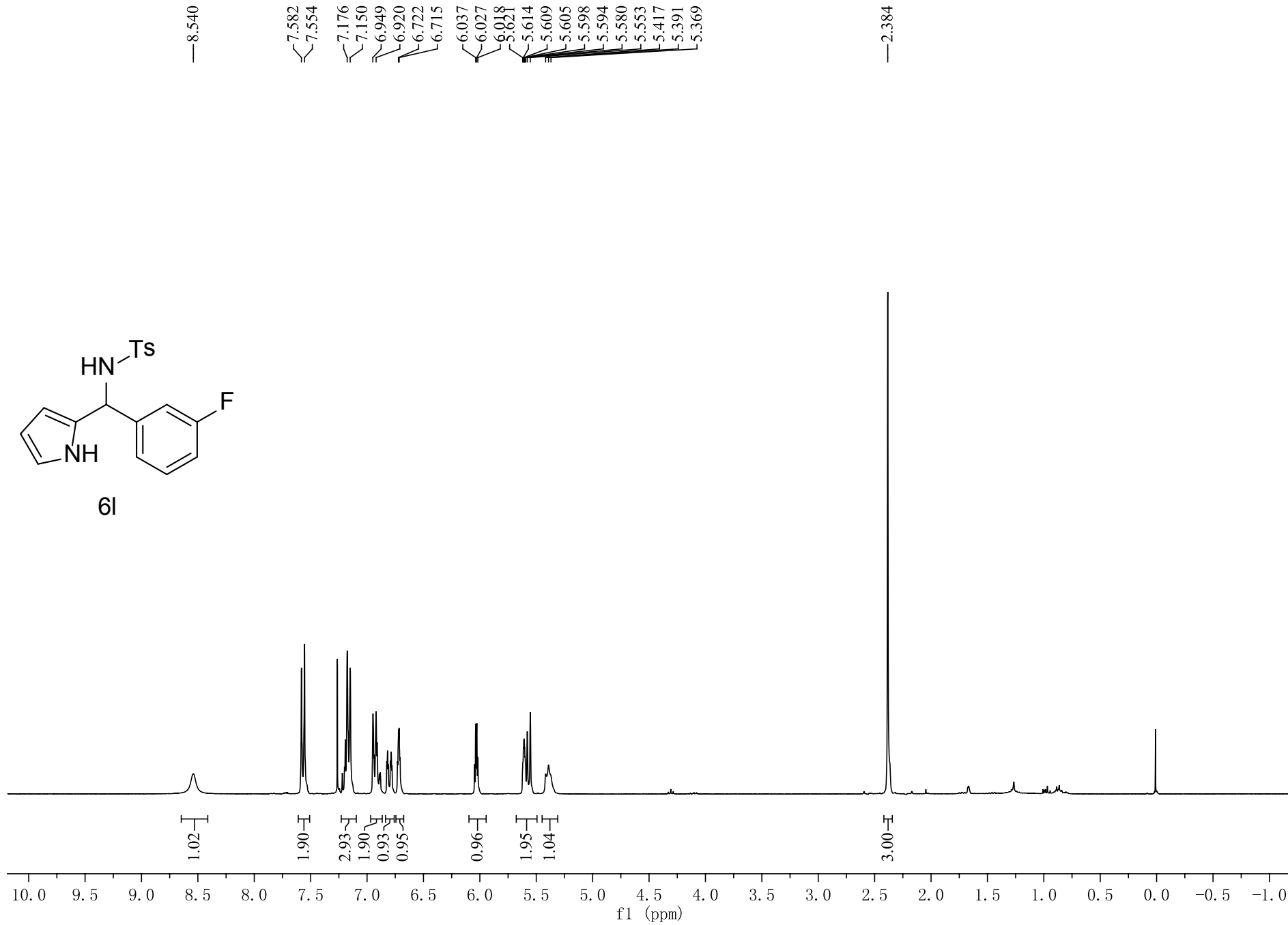
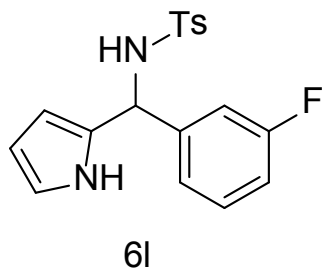
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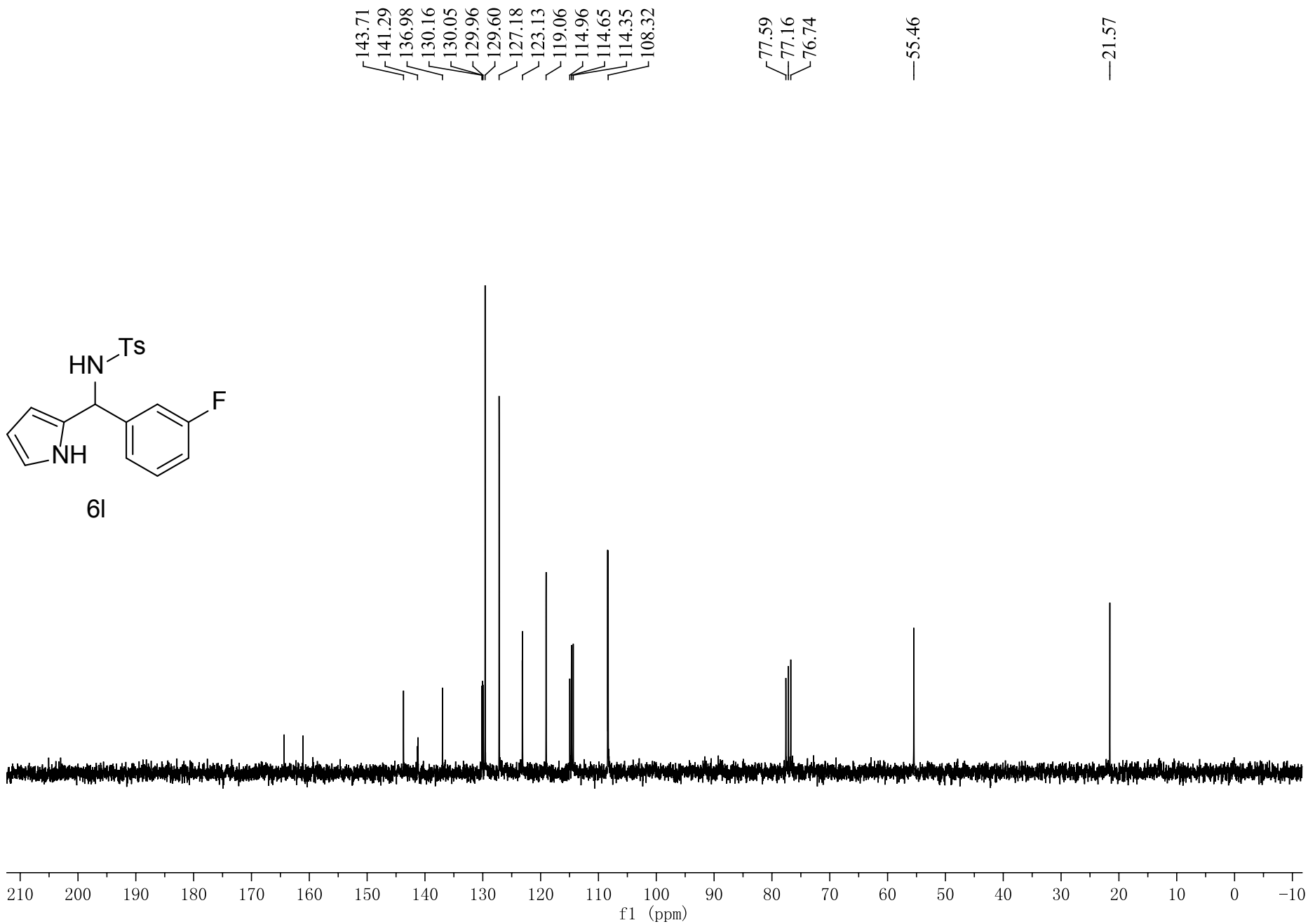
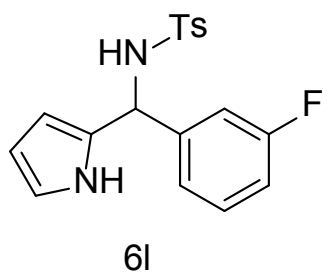


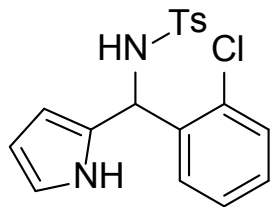


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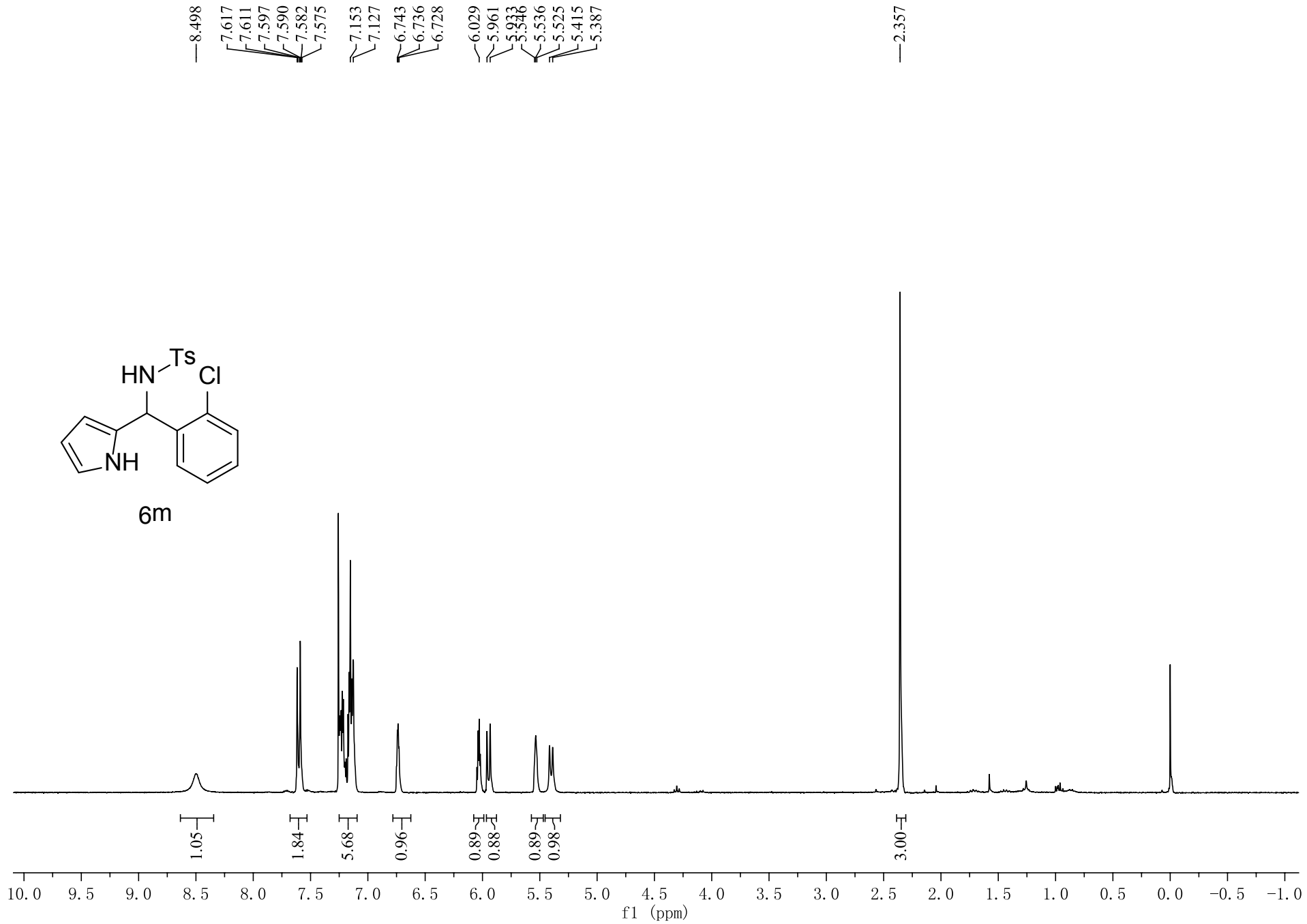


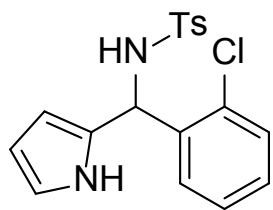




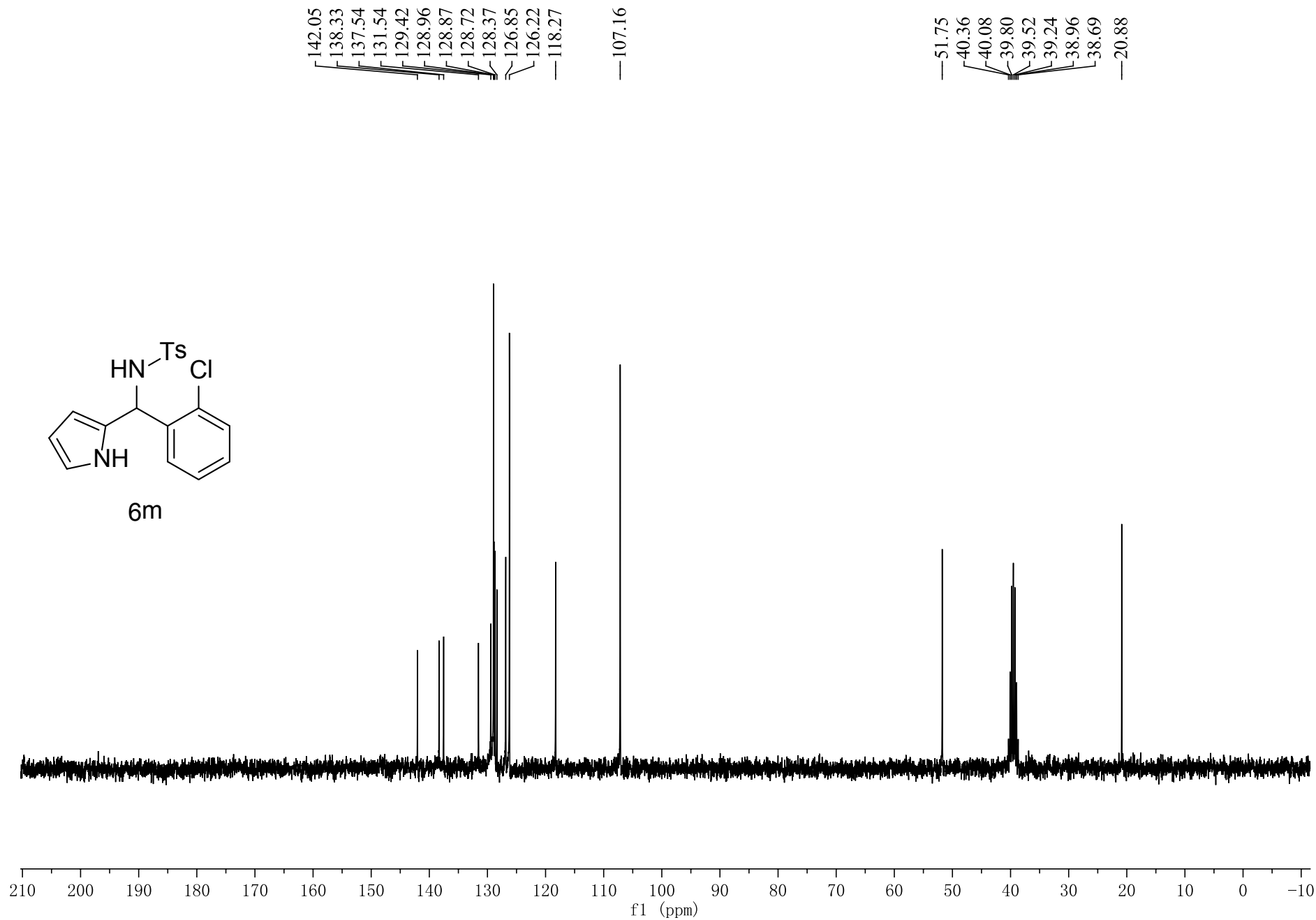


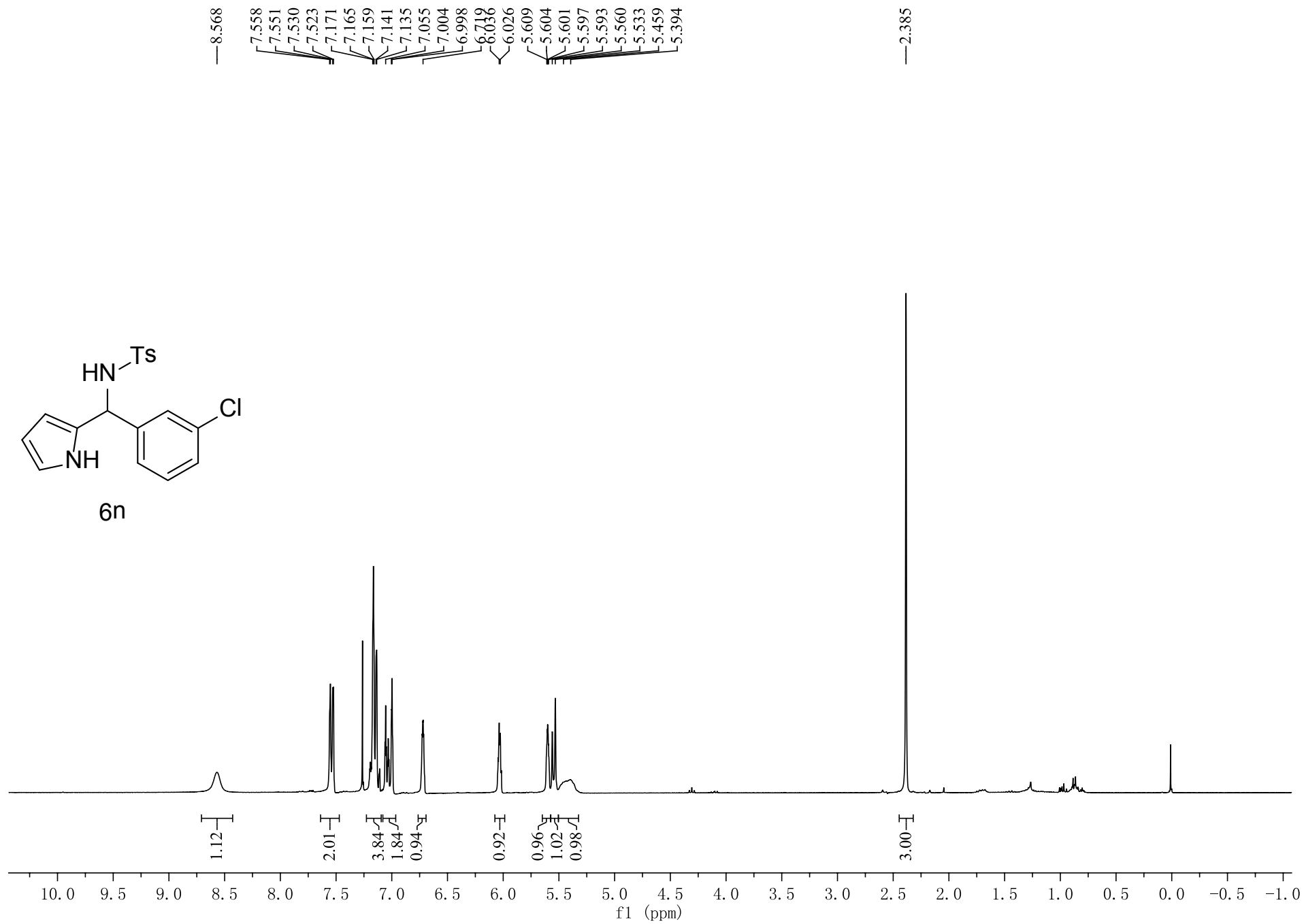
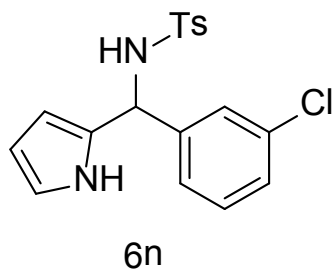
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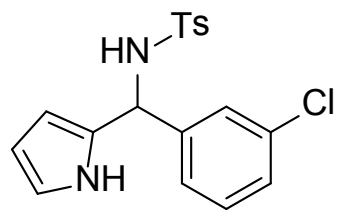




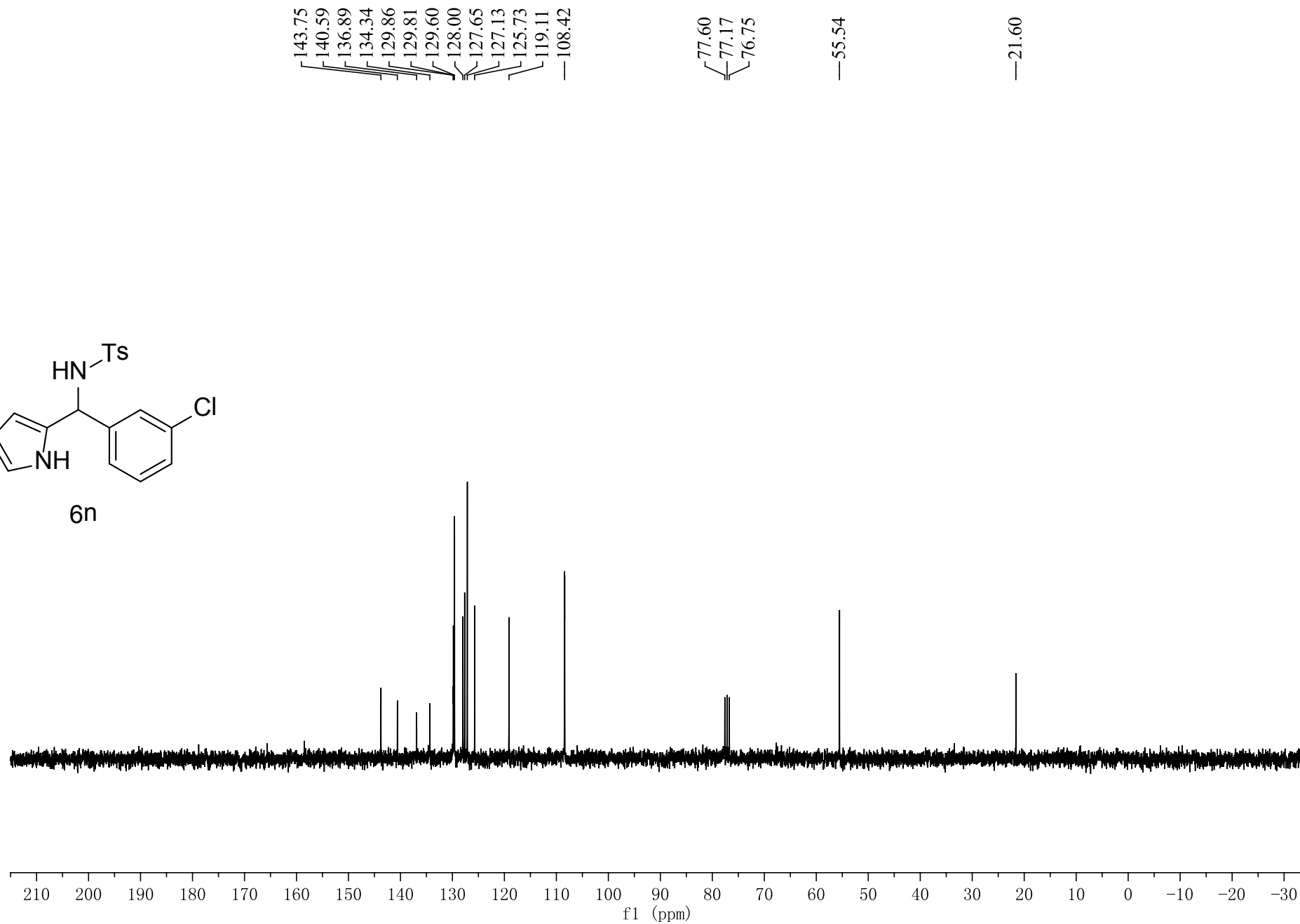
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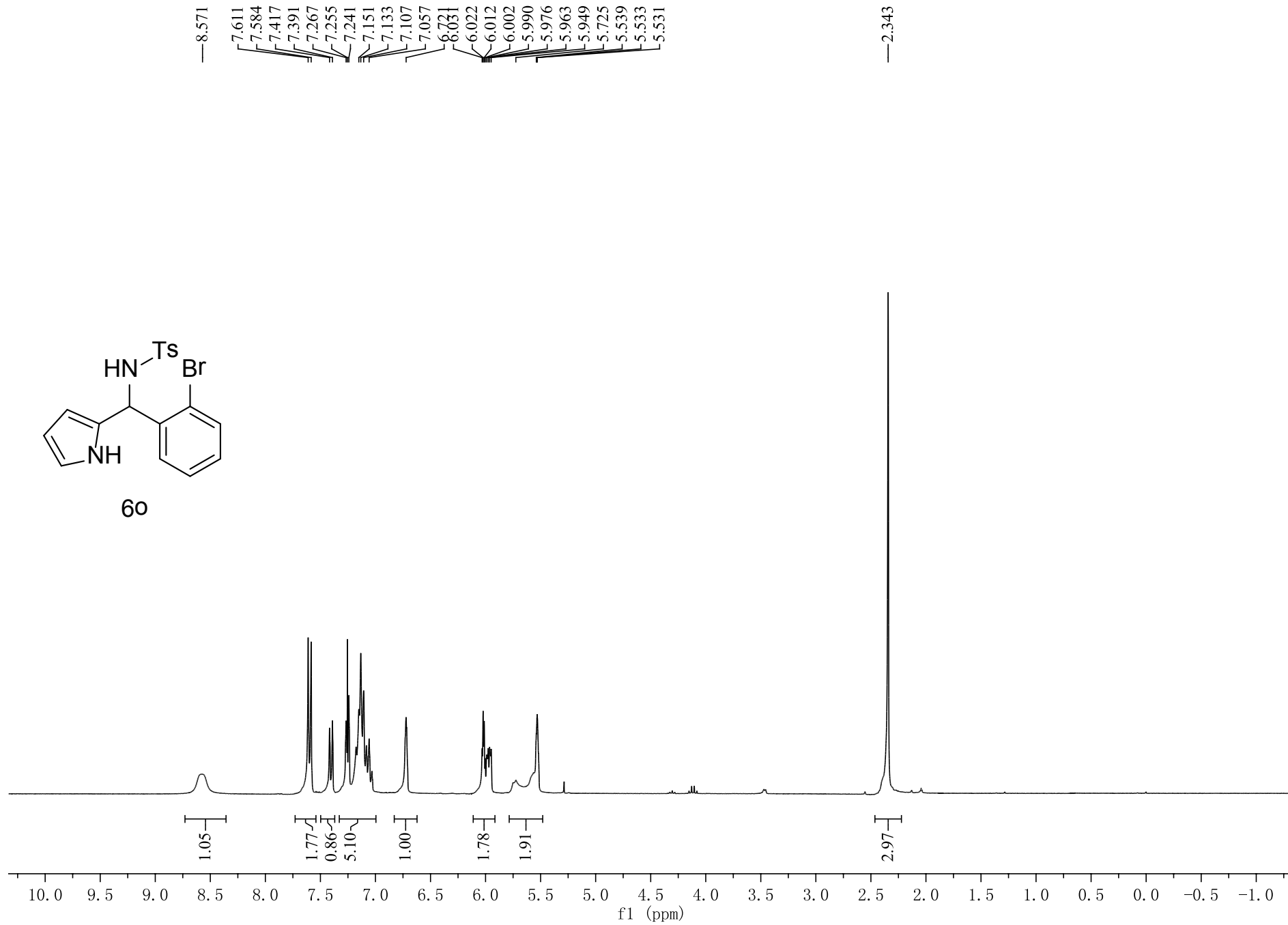
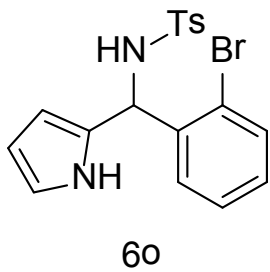


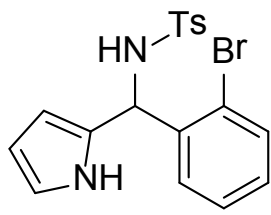




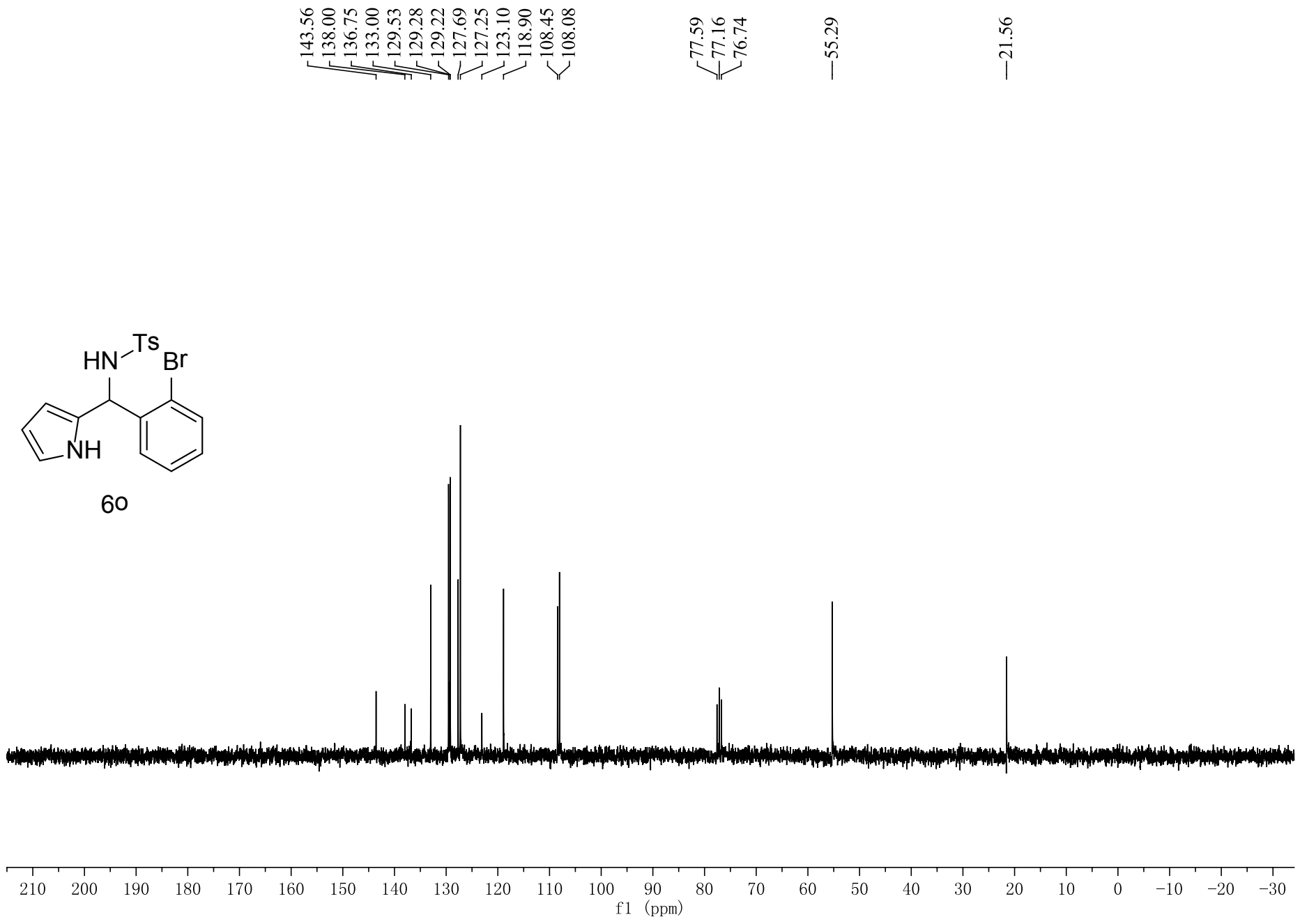
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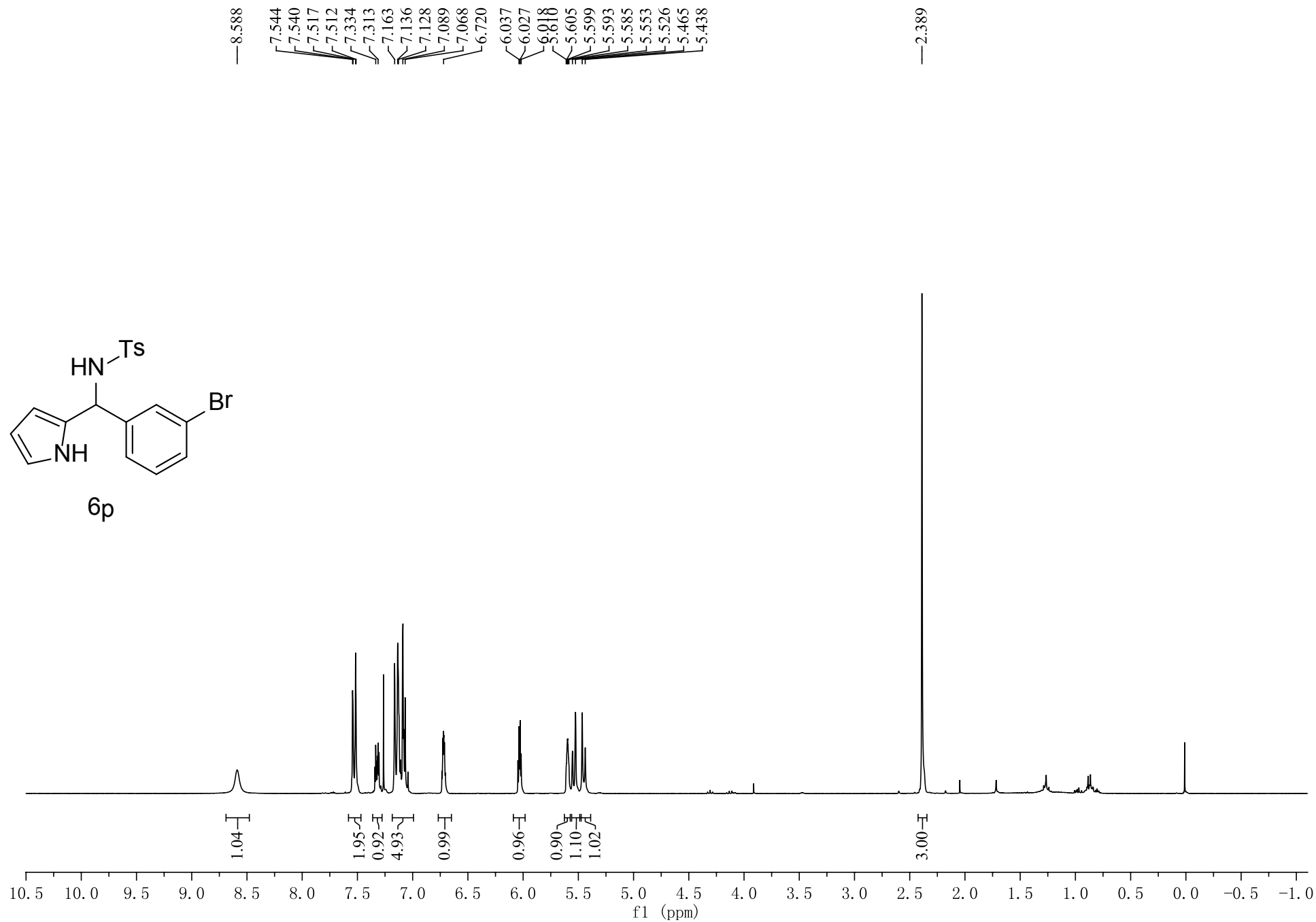
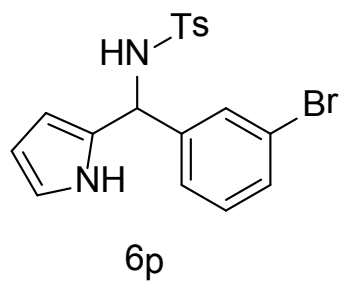


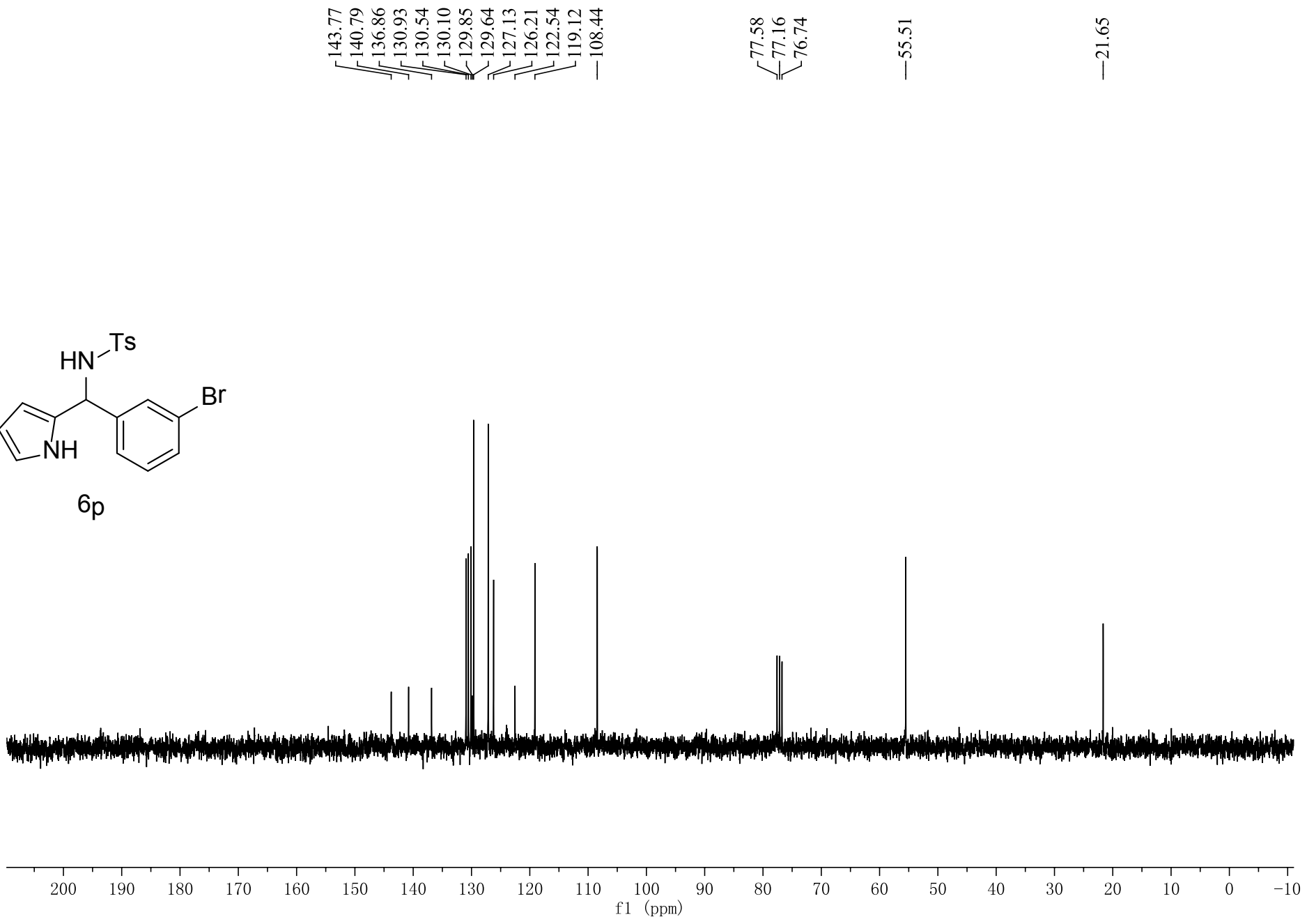
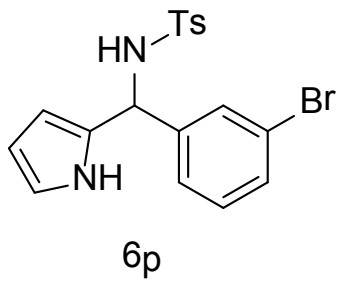


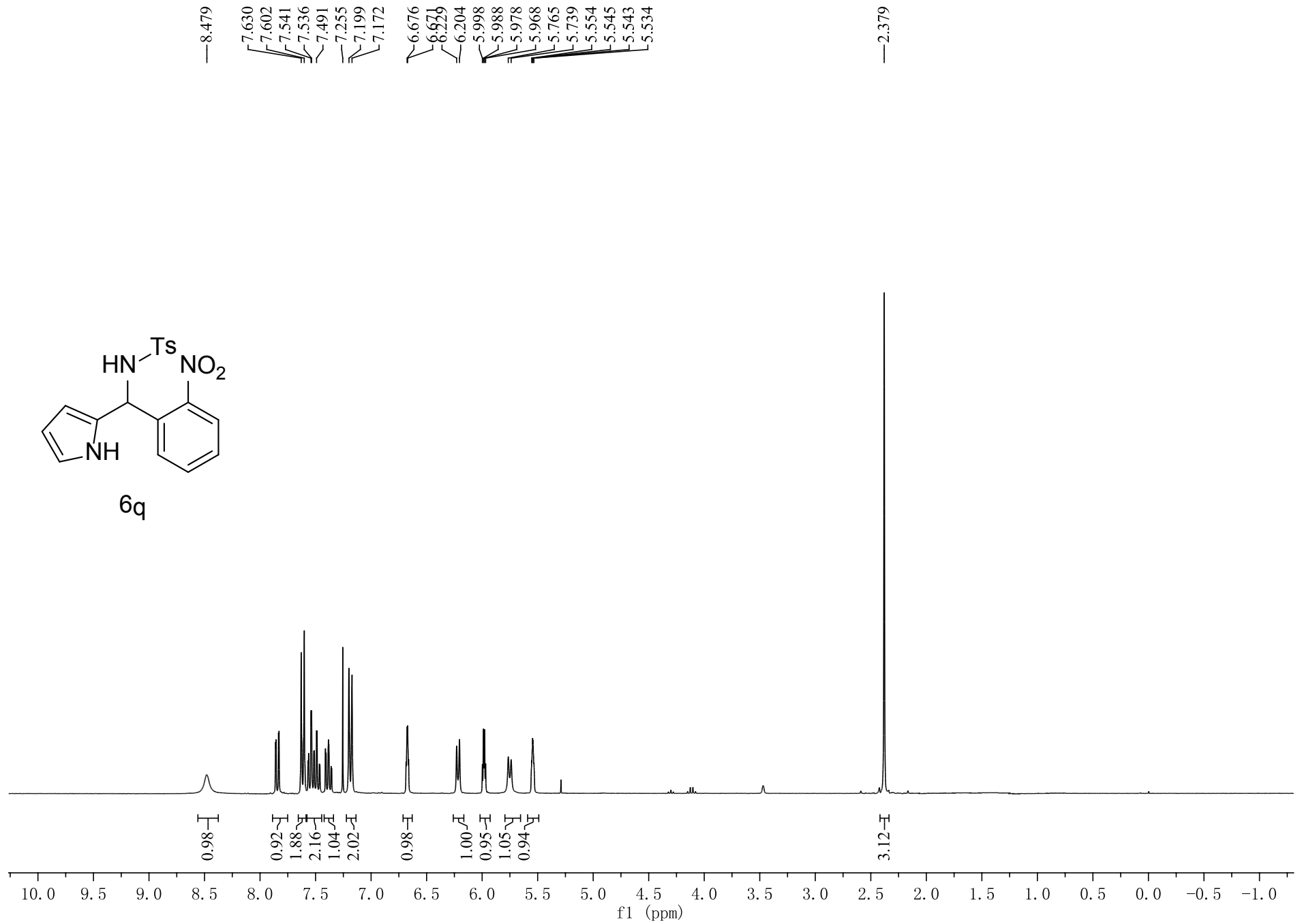
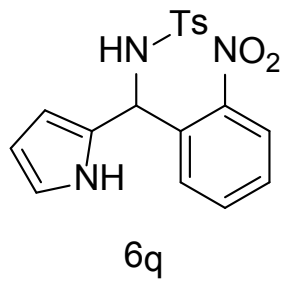


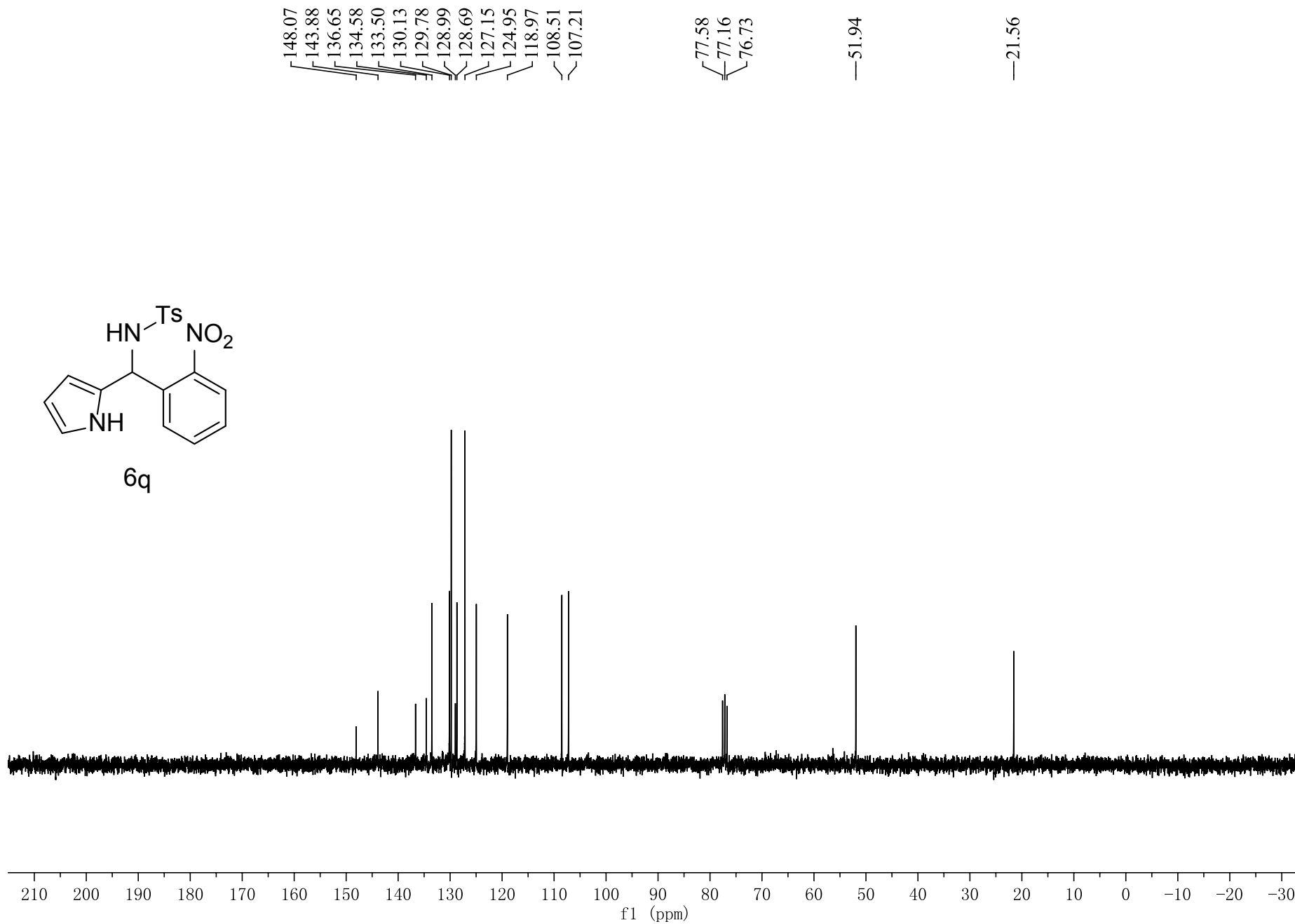
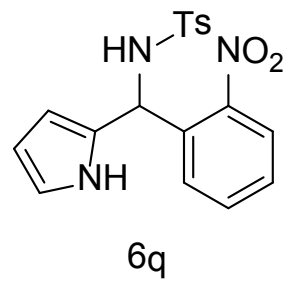
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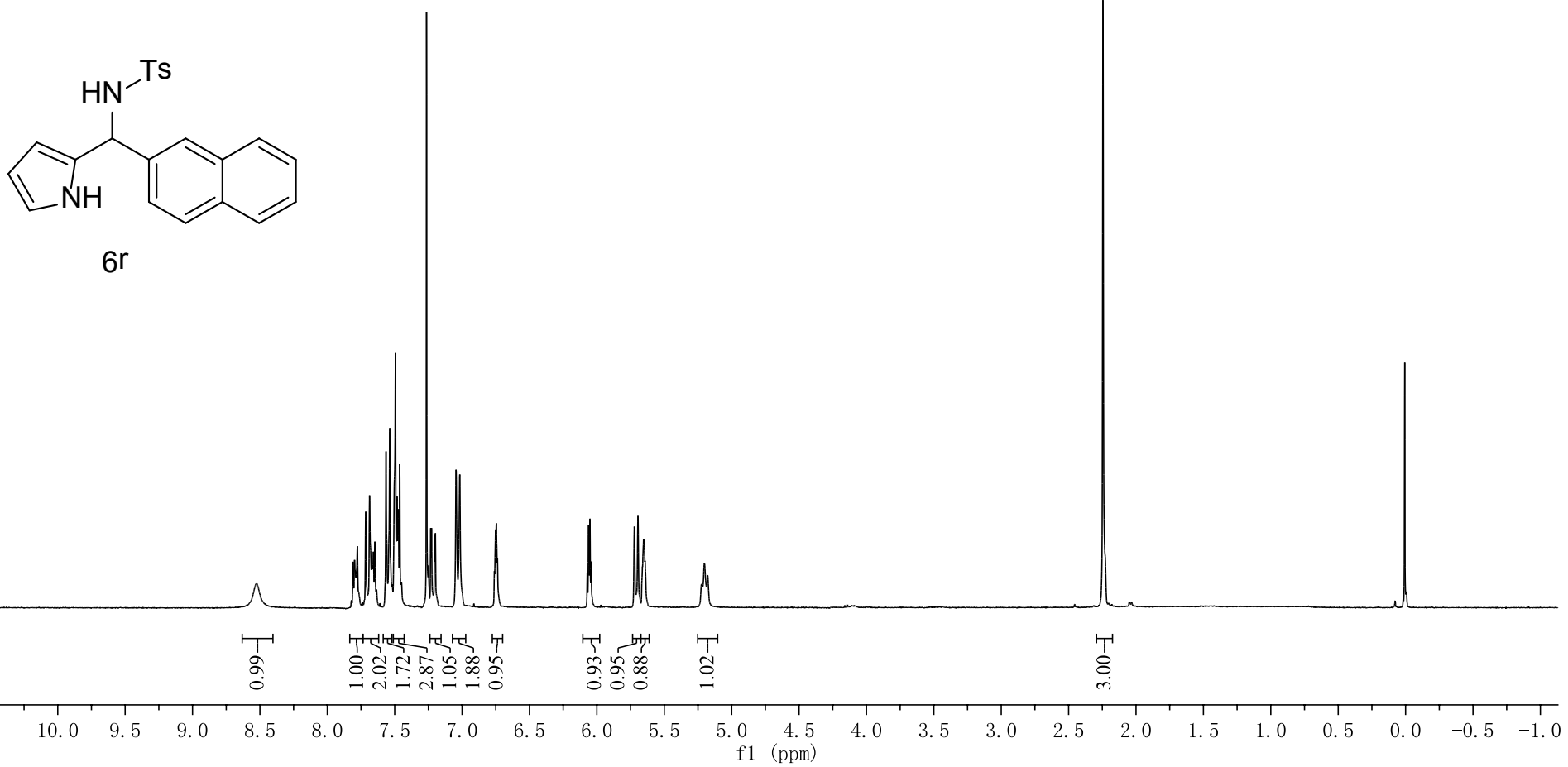


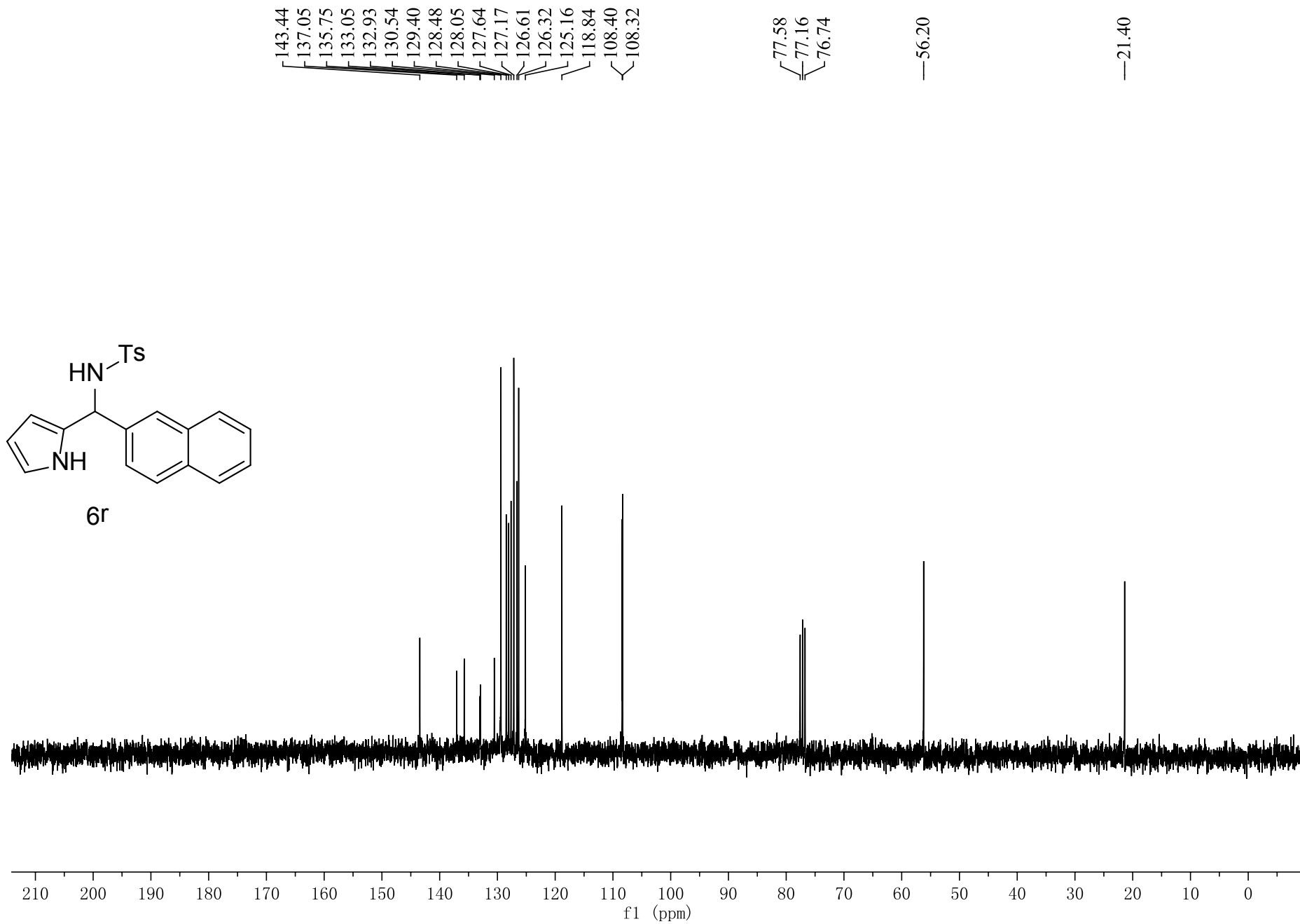
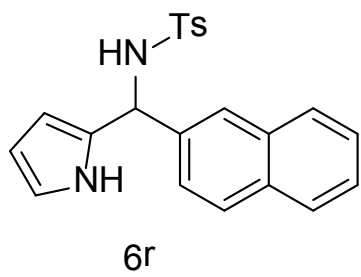




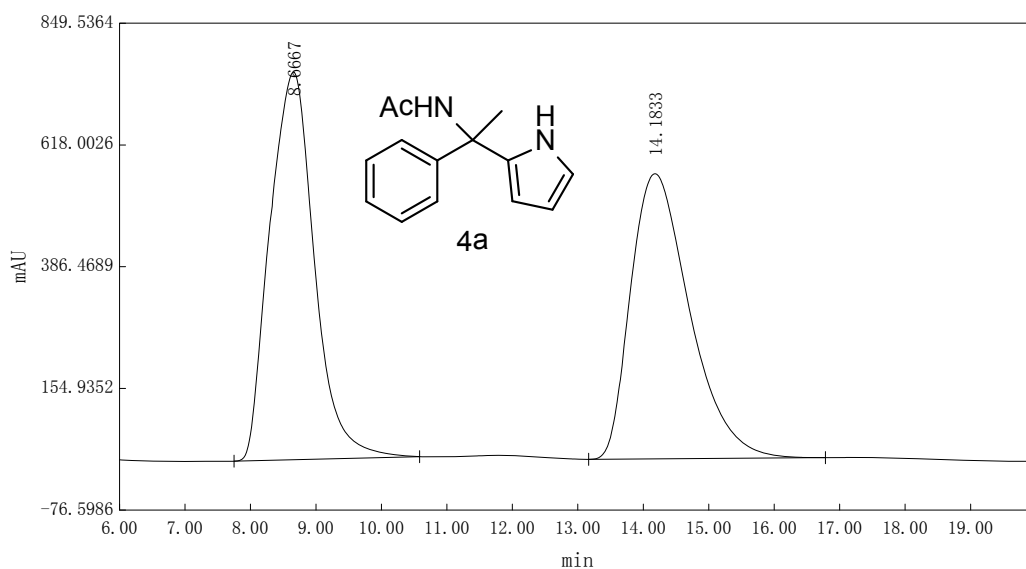




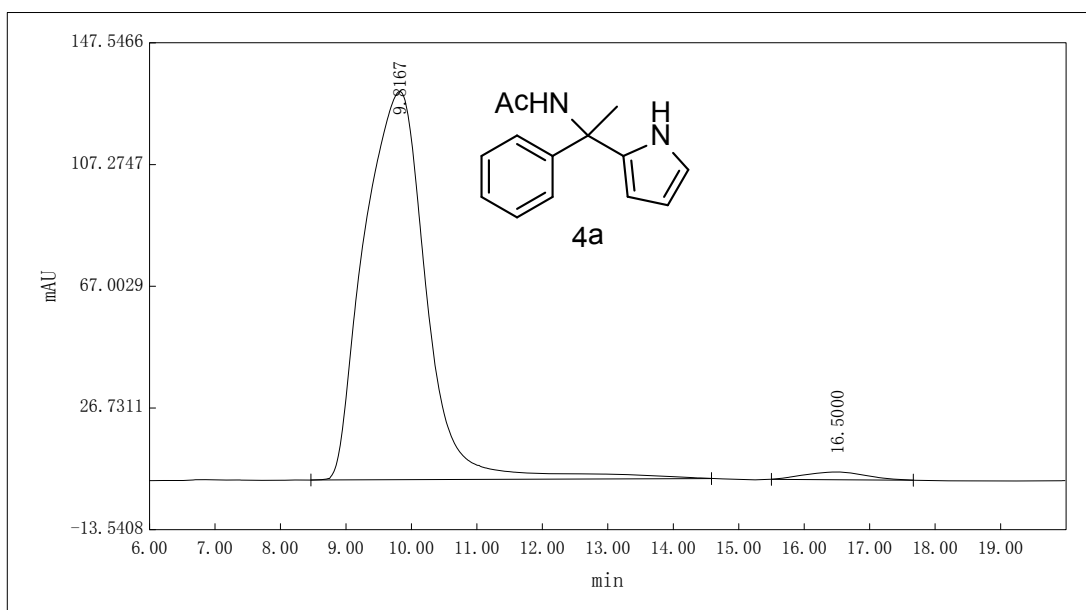




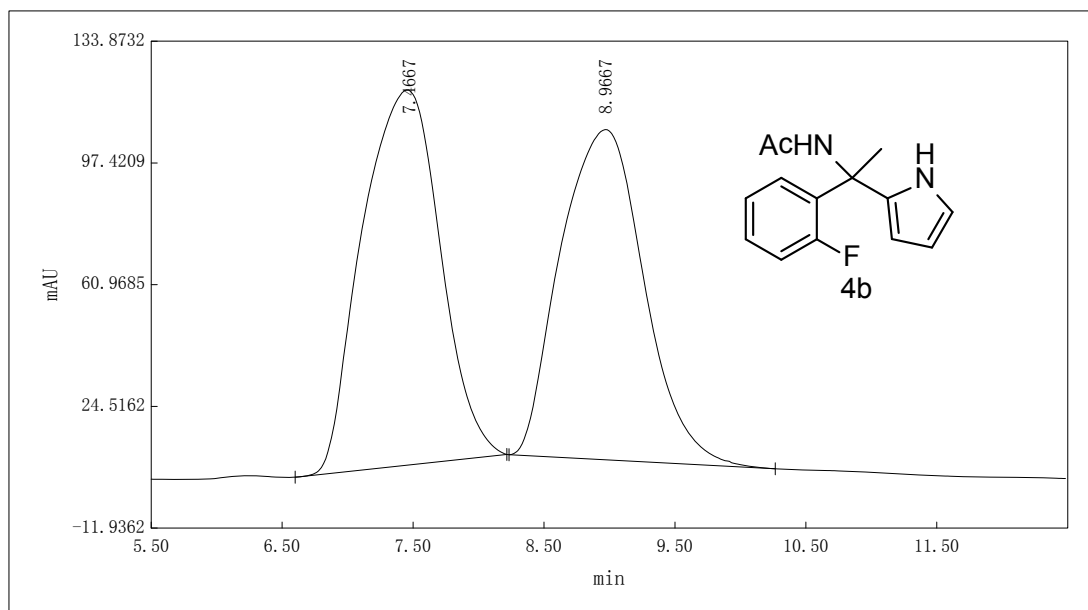
7. Copies of HPLC Spectra



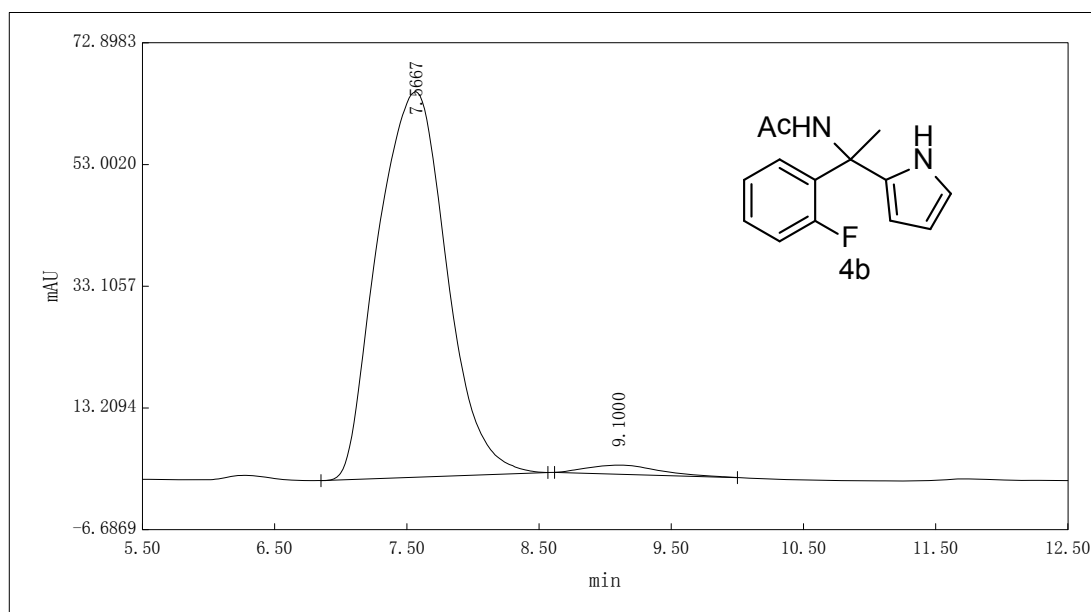
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	8.6667	36315.0336	BB	170.0000	50.6609
2	14.1833	35367.5301	FF	217.0000	49.3391



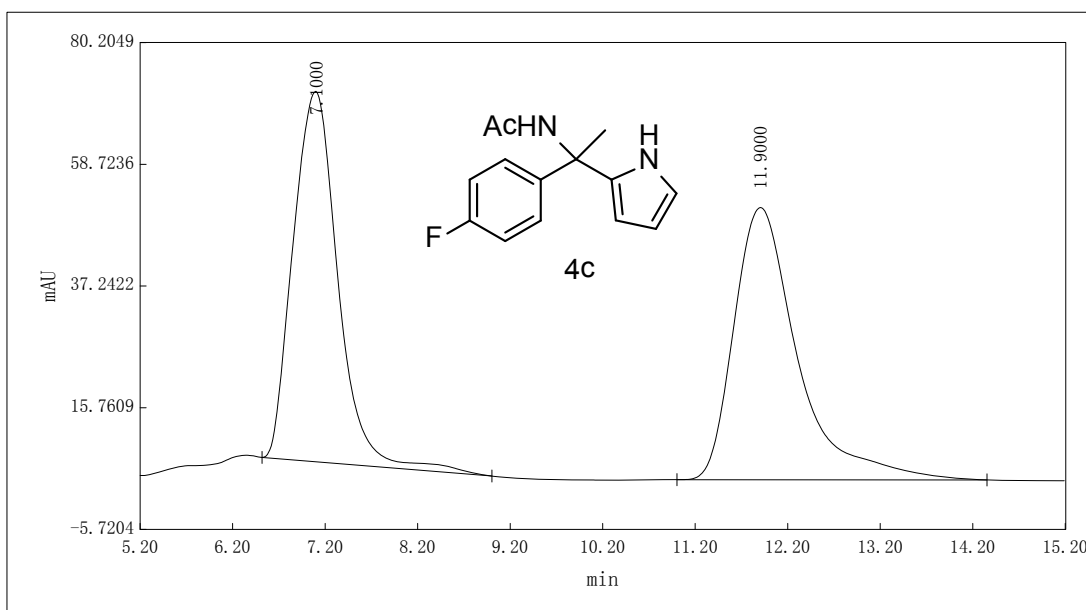
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	9.8167	8830.2375	FF	367.0000	98.0719
2	16.5000	173.6024	FF	130.0000	1.9281



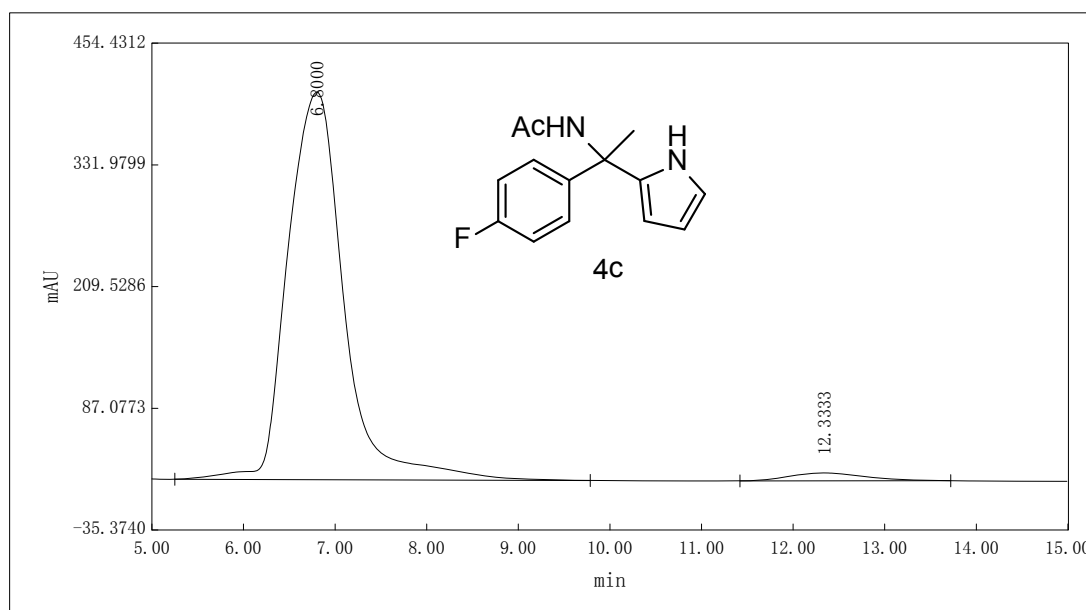
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	7.4667	4865.7272	FF	97.0000	51.5130
2	8.9667	4579.9033	FF	122.0000	48.4870



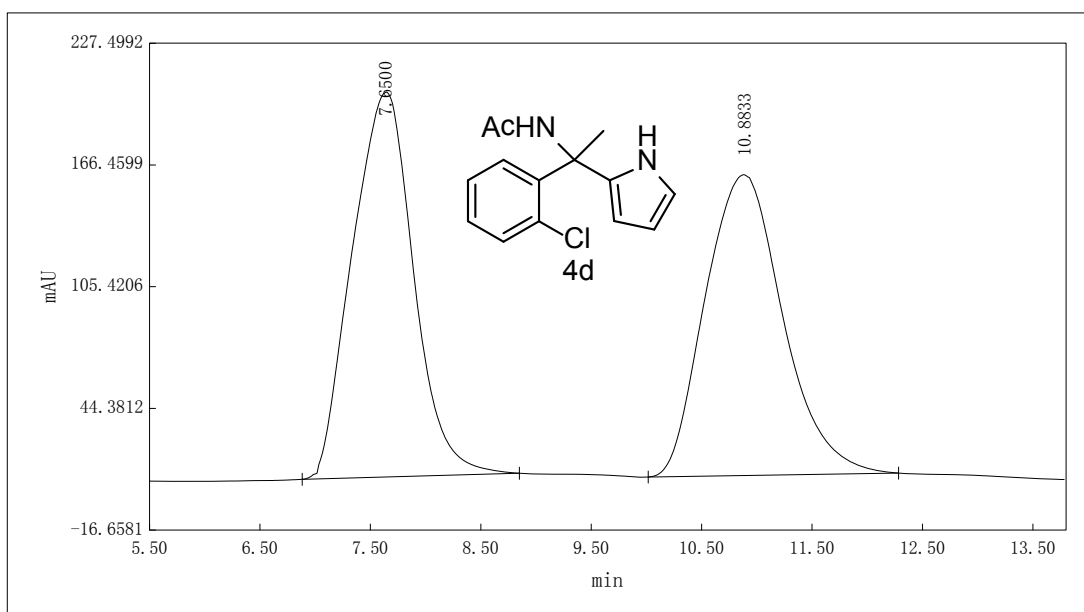
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	7.5667	2384.0139	FF	103.0000	97.5879
2	9.1000	58.9253	FF	83.0000	2.4121



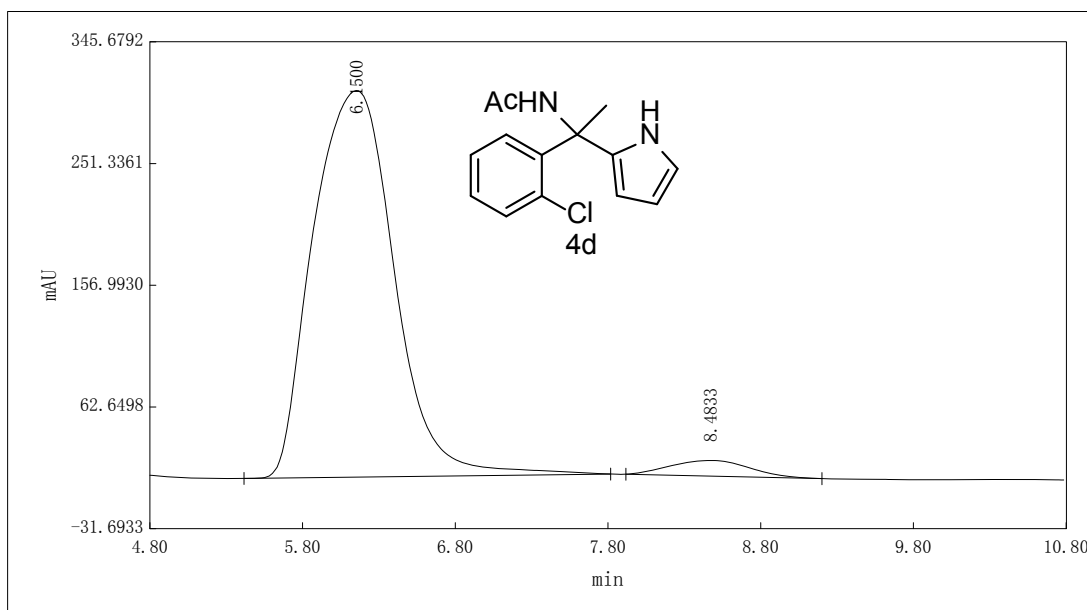
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	7.1000	2293.4337	FF	149.0000	49.0152
2	11.9000	2385.5946	FF	201.0000	50.9848



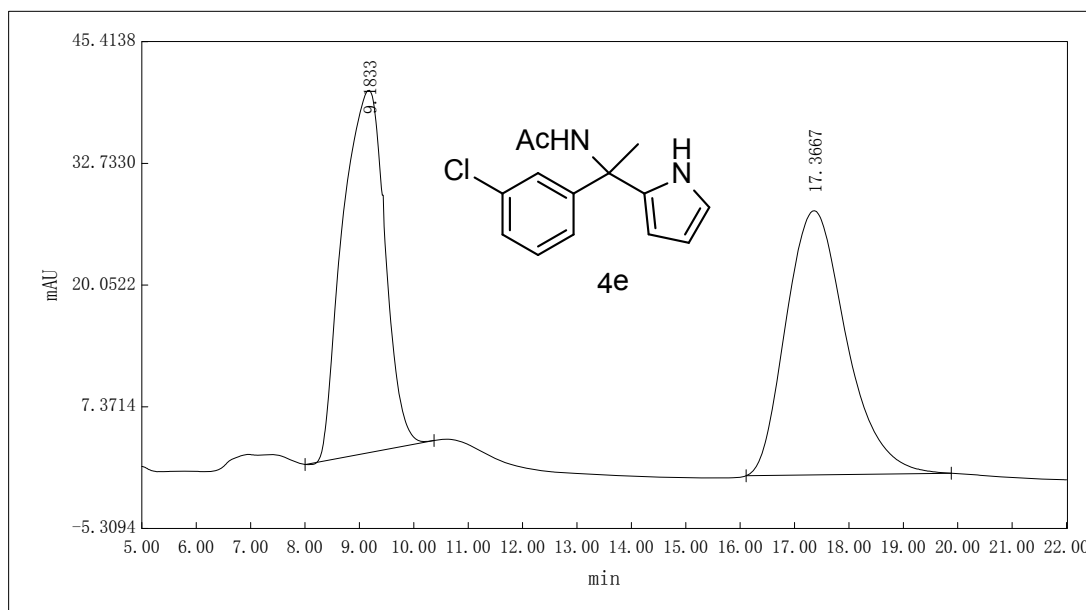
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	6.8000	17301.3755	FF	272.0000	97.3378
2	12.3333	473.2022	FF	138.0000	2.6622



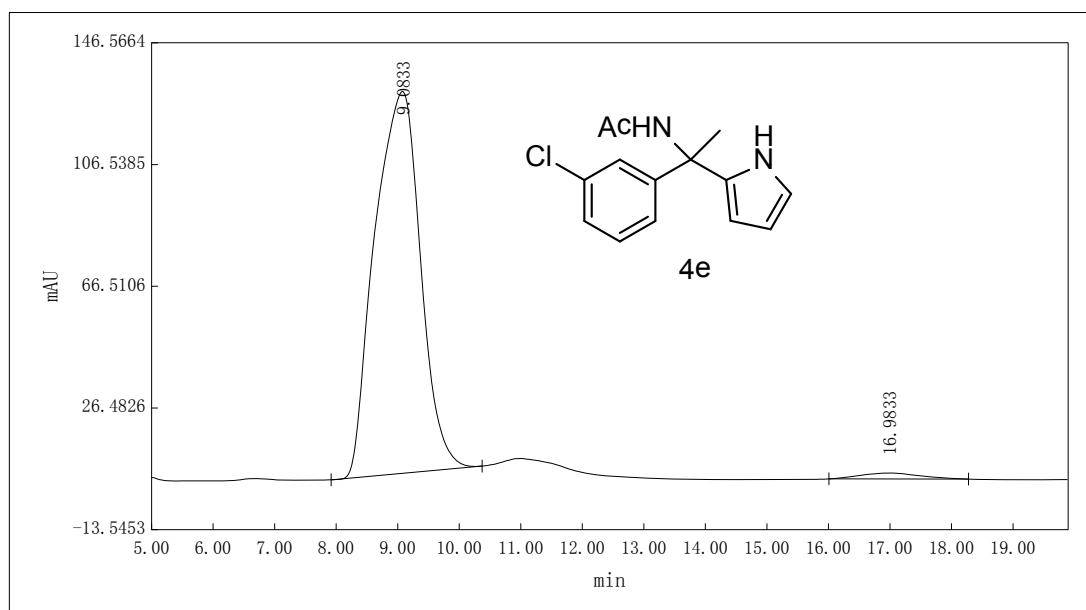
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	7.6500	7756.7320	BB	118.0000	50.2576
2	10.8833	7677.2097	BB	136.0000	49.7424



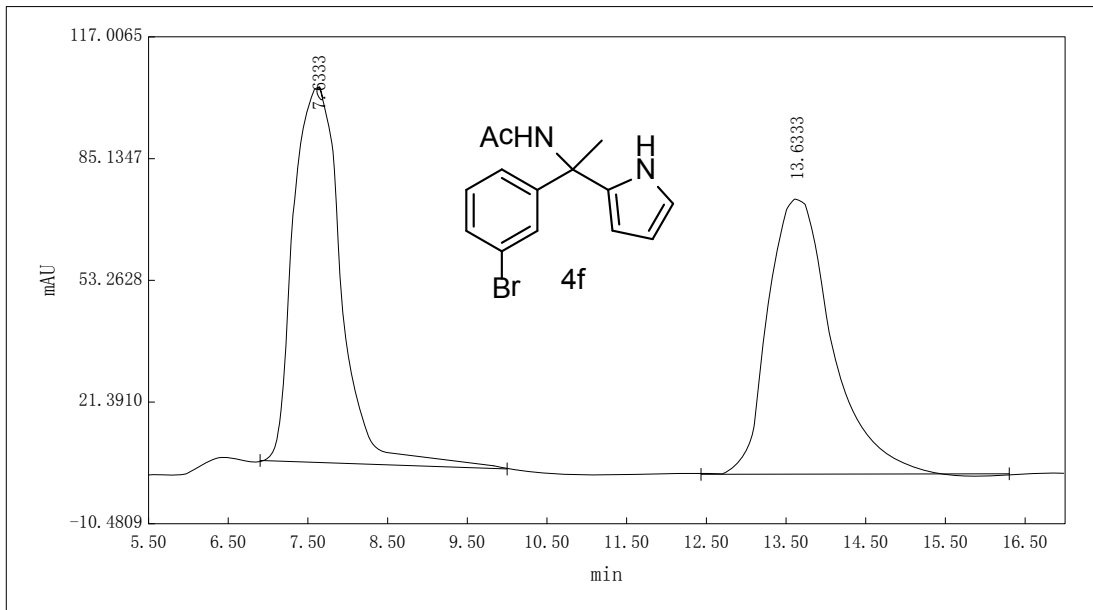
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	6.1500	11554.9260	FF	144.0000	96.2227
2	8.4833	453.5994	BB	77.0000	3.7773



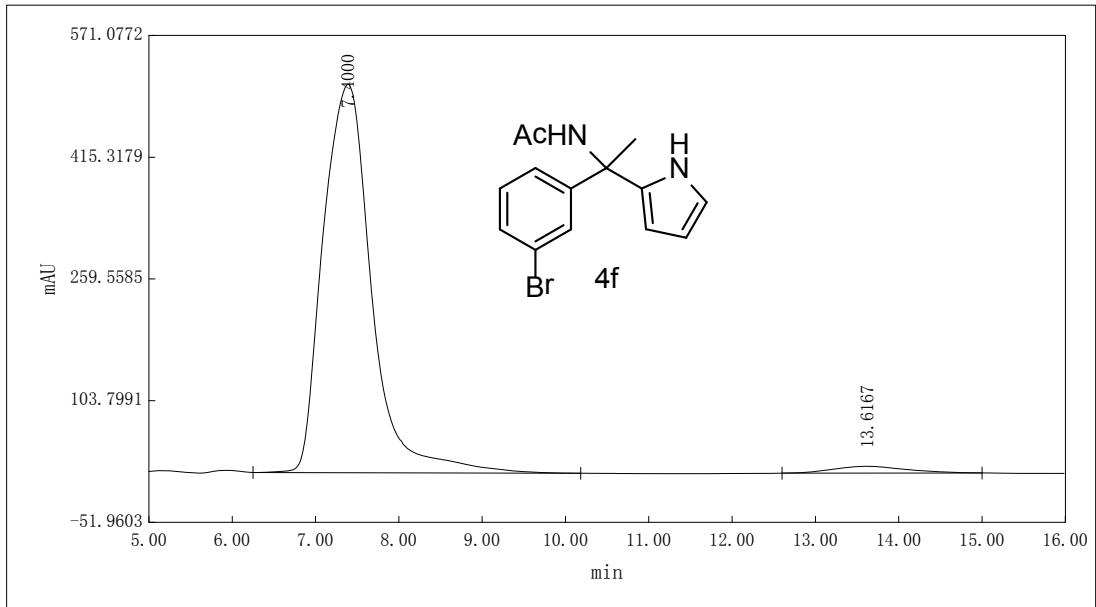
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	9.1833	2123.3289	FF	142.0000	49.1152
2	17.3667	2199.8328	FF	226.0000	50.8848



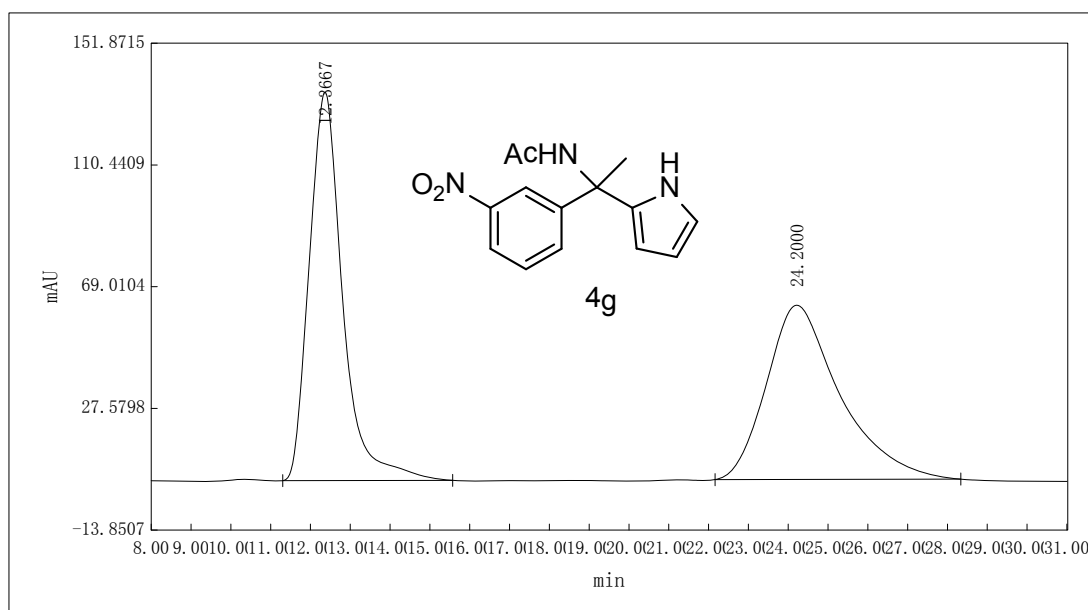
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	9.9833	6756.4068	FF	147.0000	98.0310
2	16.9833	135.7033	FF	136.0000	1.9690



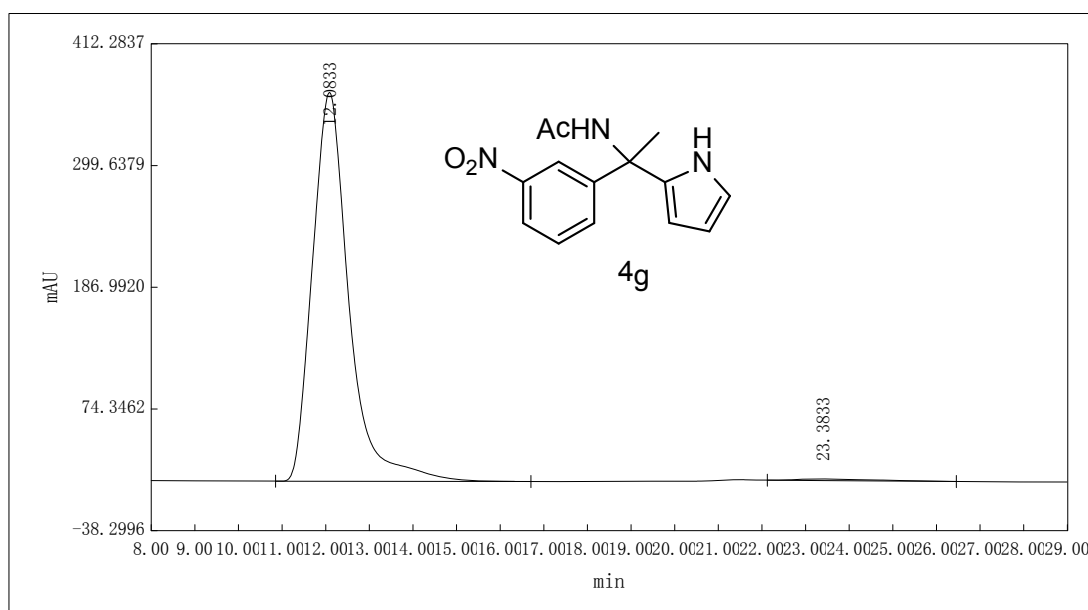
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	7.6333	4469.8395	FF	186.0000	50.8938
2	13.6333	4312.8406	FF	232.0000	49.1062



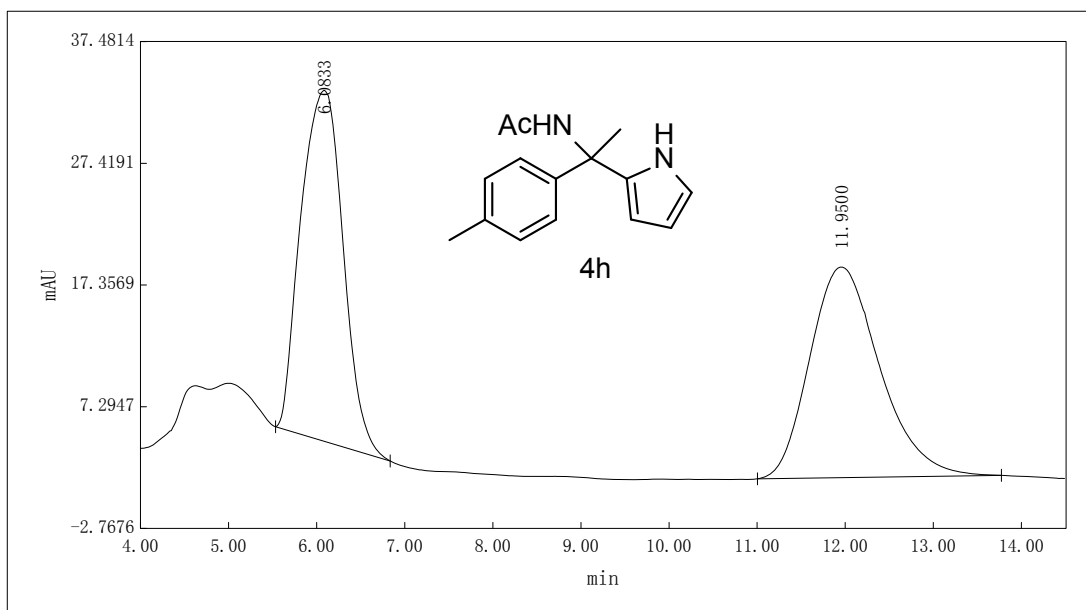
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	7.4000	20988.5592	FF	236.0000	97.4216
2	13.6167	555.4973	FF	144.0000	2.5784



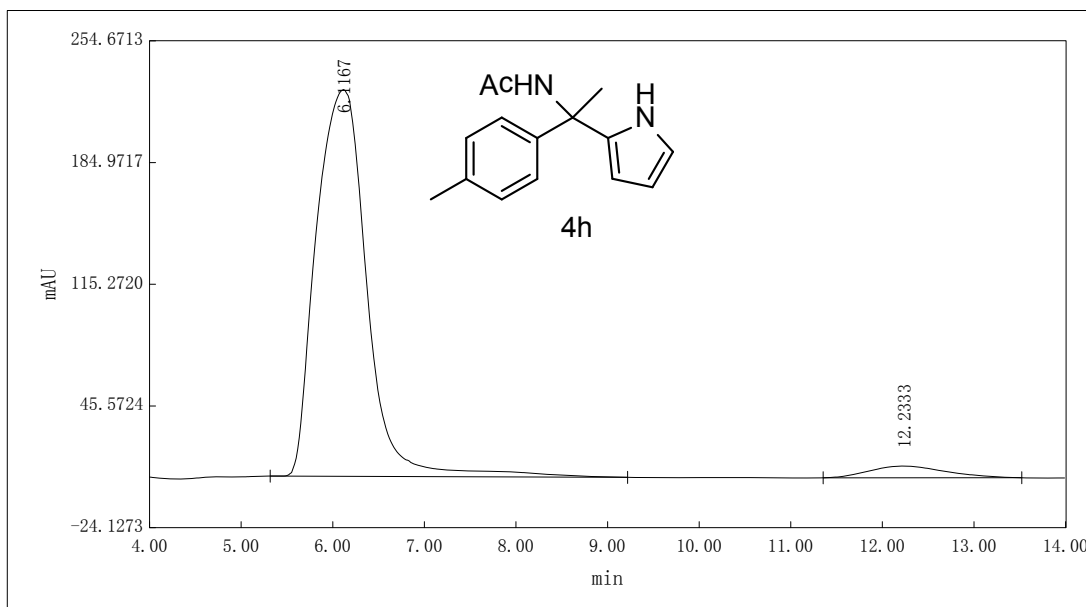
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	12.3667	8019.7583	FF	256.0000	50.3891
2	24.2000	7895.9139	FF	370.0000	49.6109



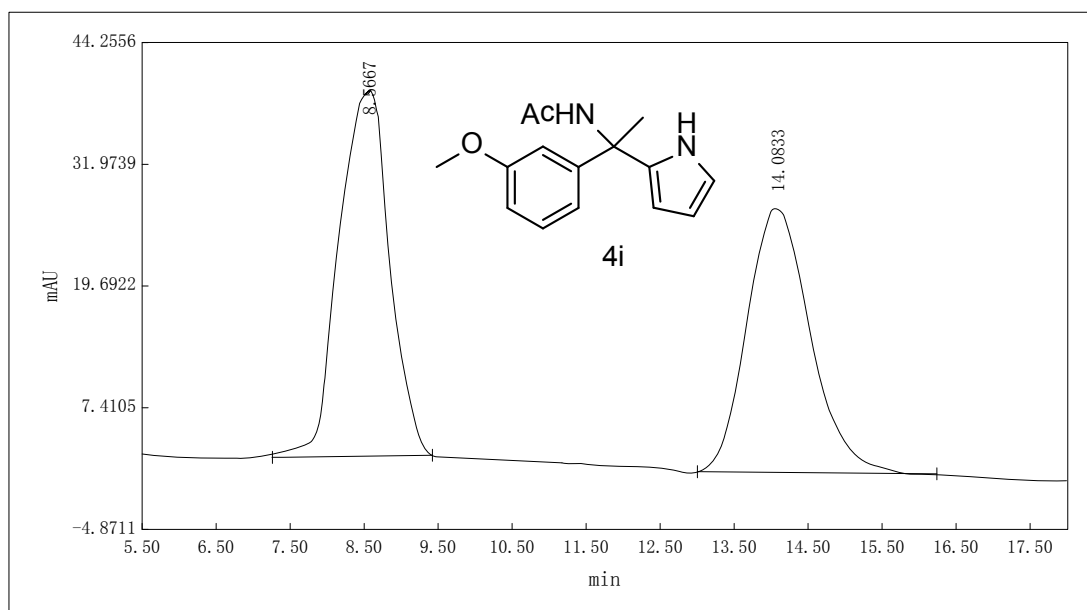
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	12.0833	21796.3444	FF	351.0000	99.0956
2	23.3833	198.9202	FF	260.0000	0.9044



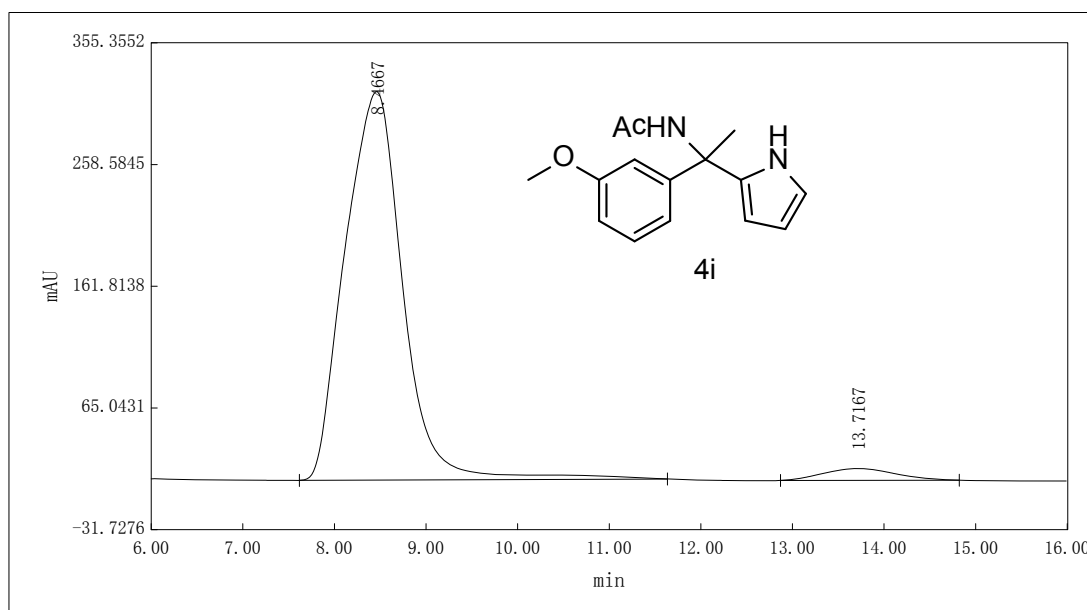
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	6.0833	1006.9461	BB	78.0000	50.2011
2	11.9500	998.8804	FF	166.0000	49.7989



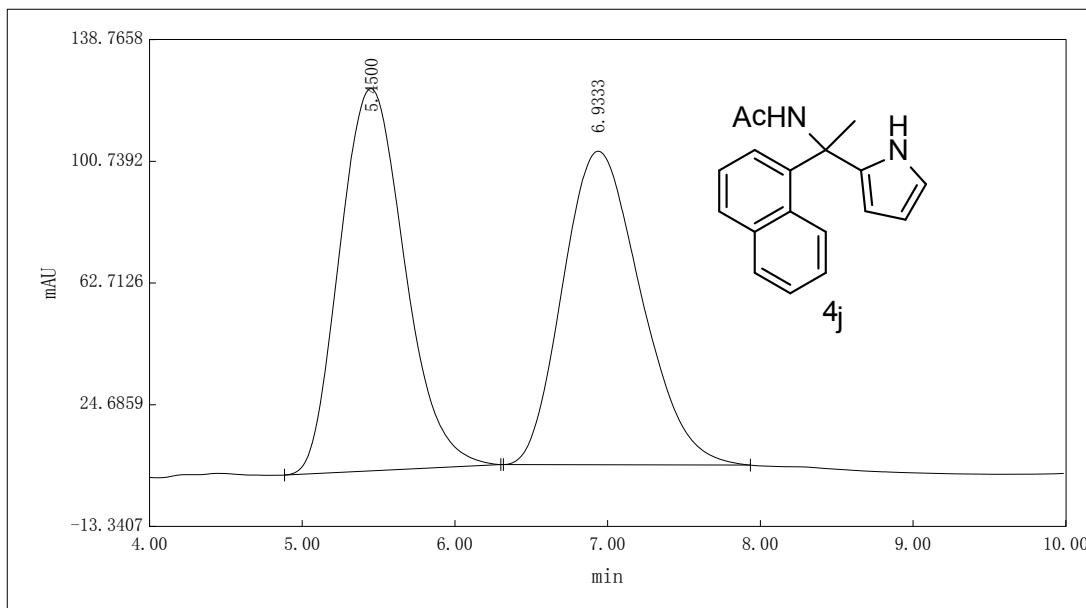
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	6.1167	8882.0603	FF	234.0000	95.6603
2	12.2333	402.9449	FF	130.0000	4.3397



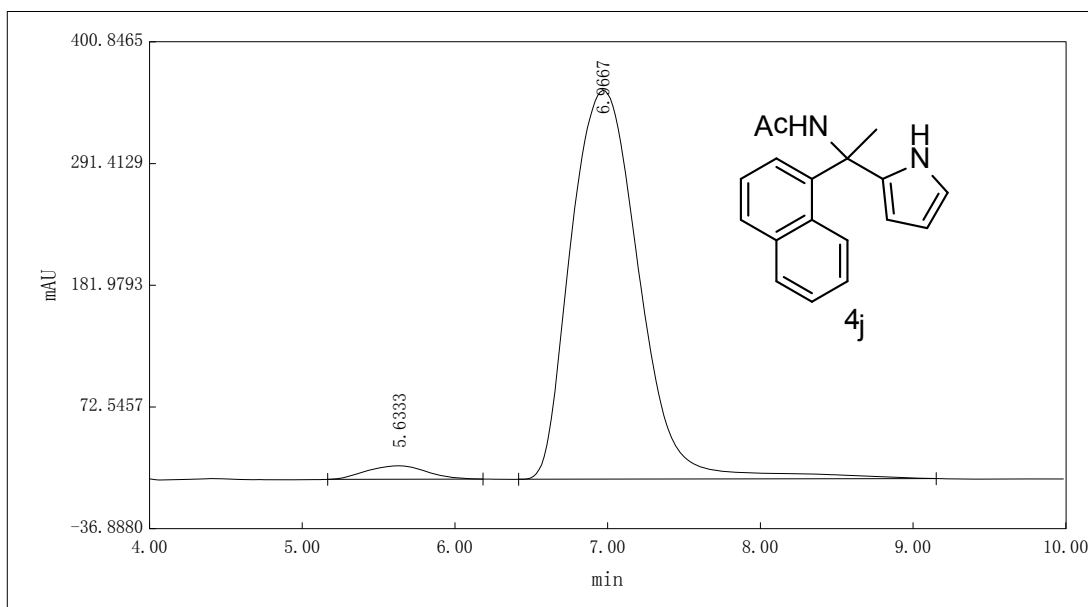
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	8.5667	1692.6249	BB	121.0000	50.6680
2	14.0833	1647.9953	FF	194.0000	49.3320



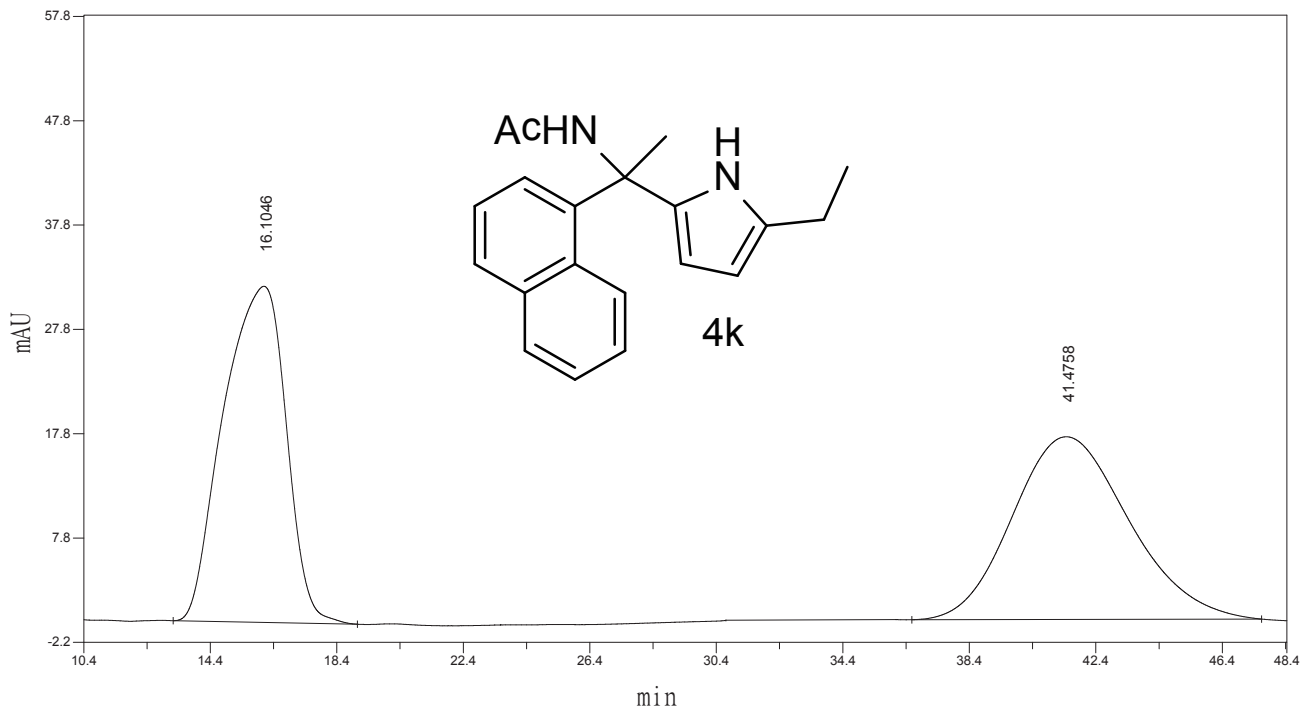
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	8.4667	14110.3012	FF	241.0000	96.3525
2	13.7167	534.1516	FF	117.0000	3.6475



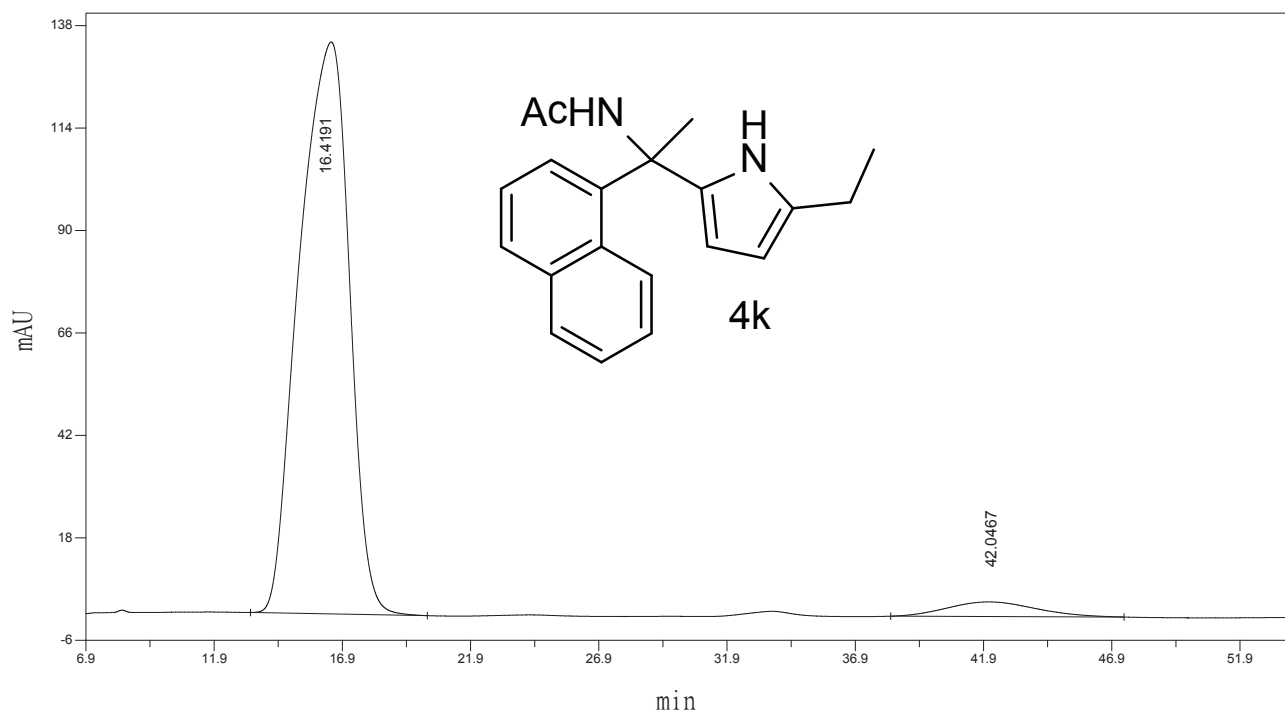
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	5.4500	3676.3929	BB	85.0000	50.3413
2	6.9333	3626.5395	BB	97.0000	49.6587



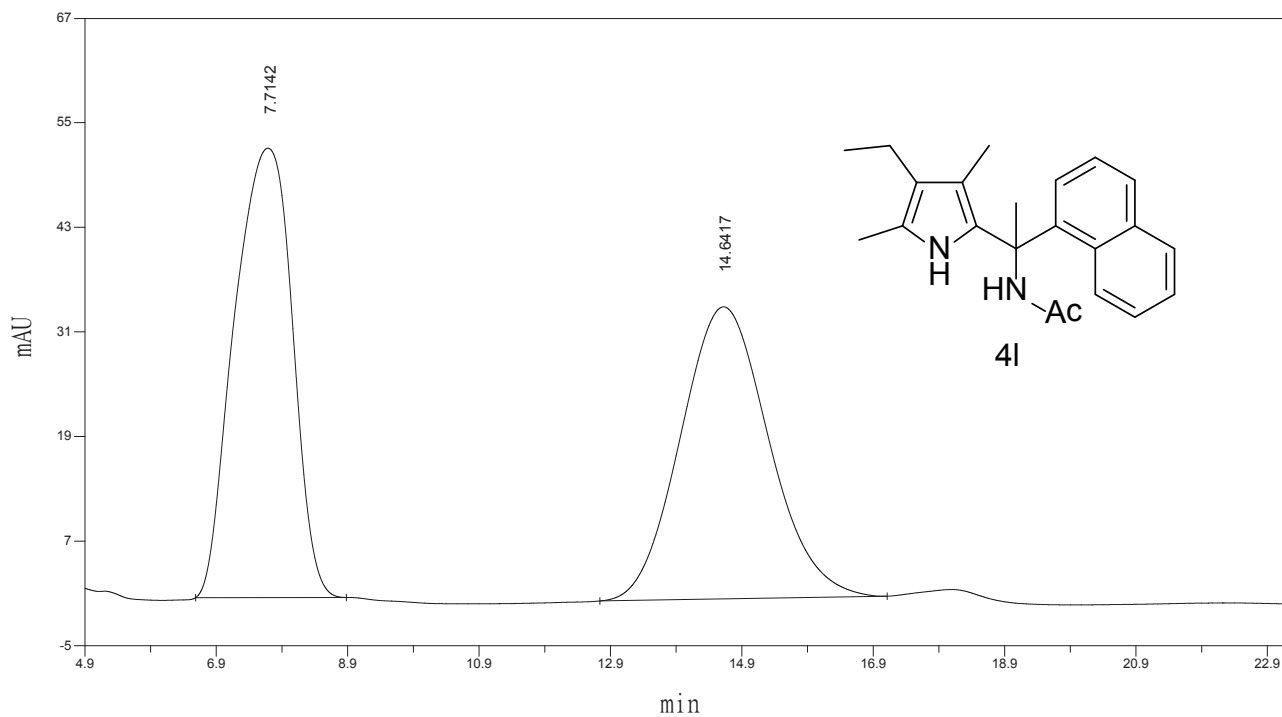
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	5.6333	344.6895	BB	61.0000	2.9432
2	6.9667	11366.8492	FF	164.0000	97.0568



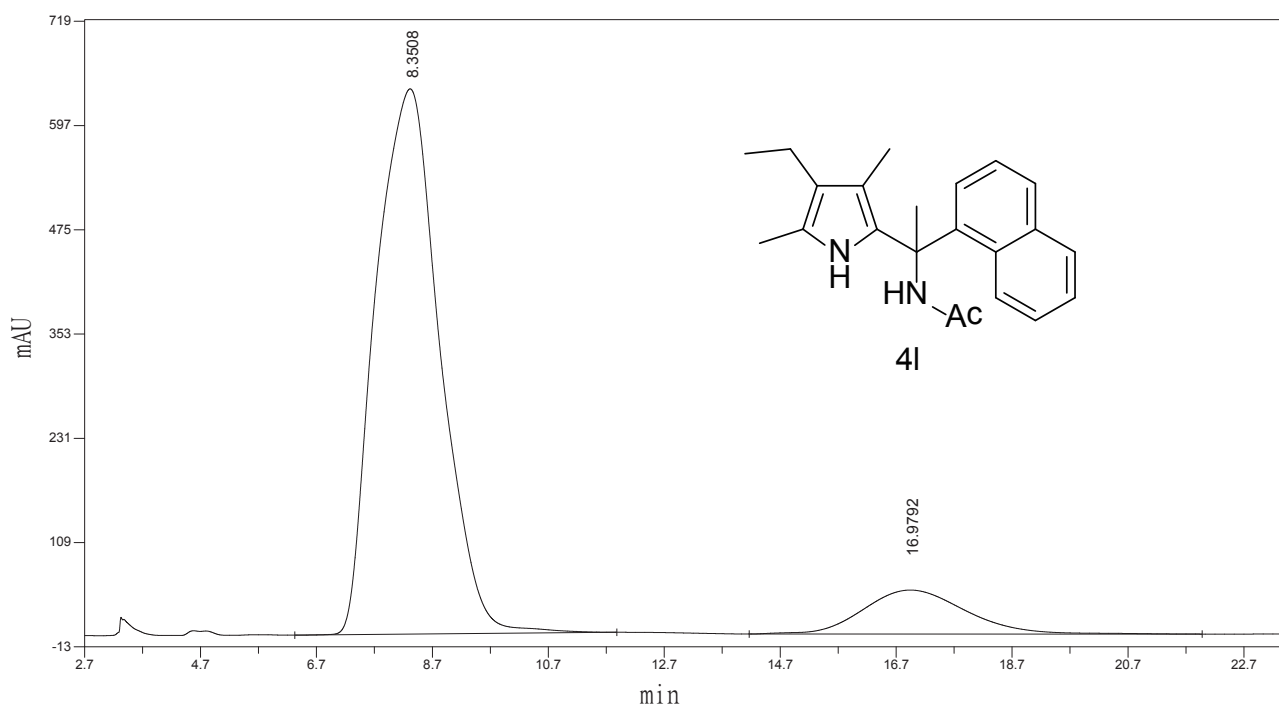
Peak #	RetTime [min]	Area [mAU*s]	type	Area %
1	16.1046	4435.60	BB	49.7584
2	41.4758	4478.67	FF	50.2416



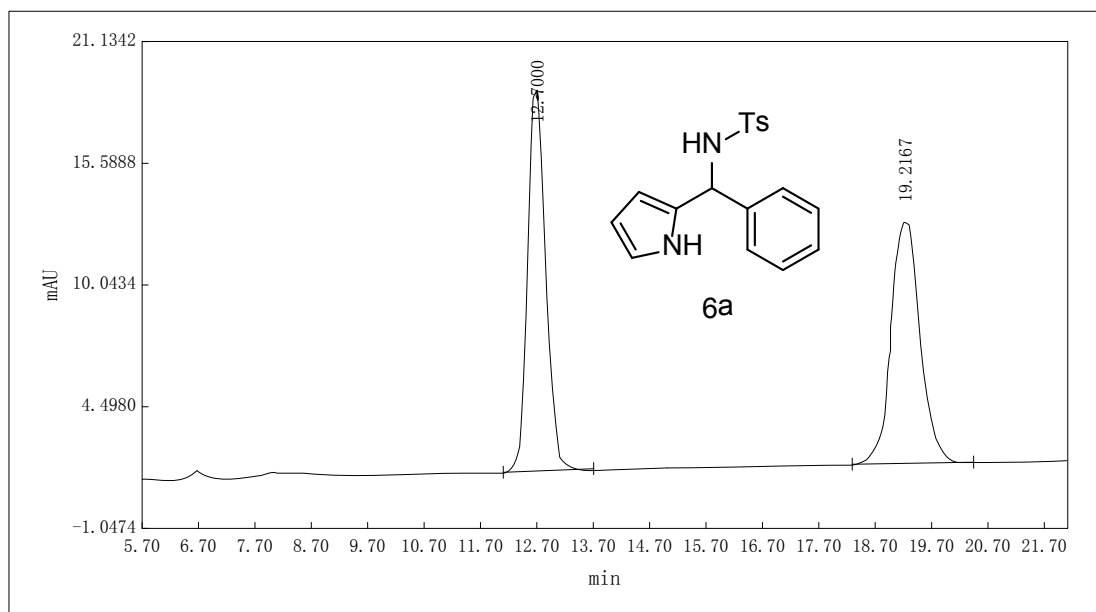
Peak #	RetTime [min]	Area [mAU*s]	type	Area %
1	16.4191	18119.06	BB	95.6555
2	42.0467	822.93	FF	4.3445



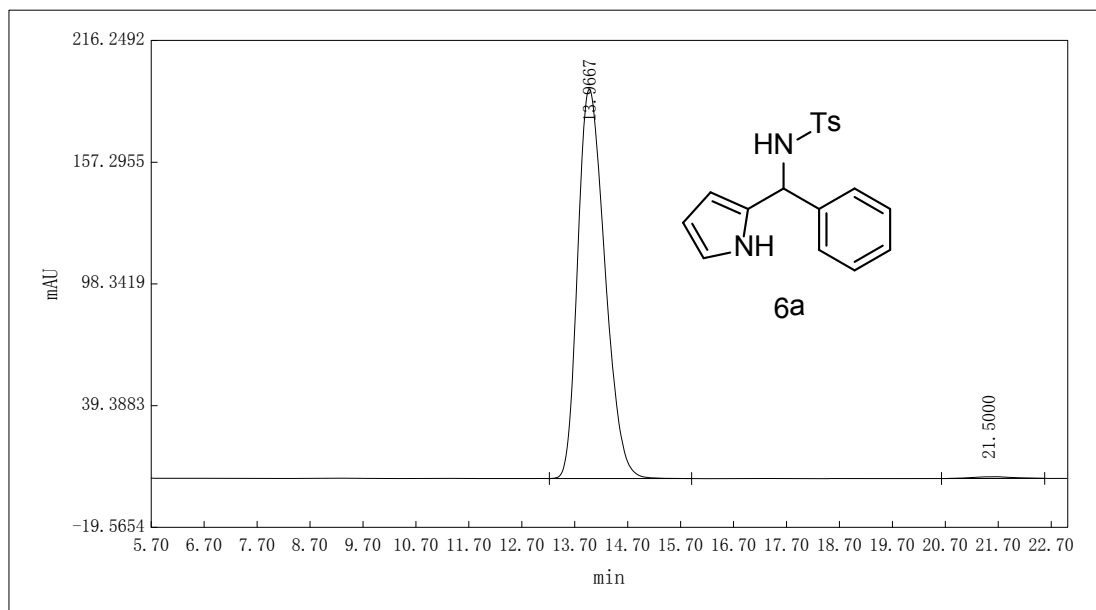
Peak #	RetTime [min]	Area [mAU*s]	type	Area %
1	7.7142	3159.76	BB	50.3210
2	14.6417	3119.45	FF	49.6790



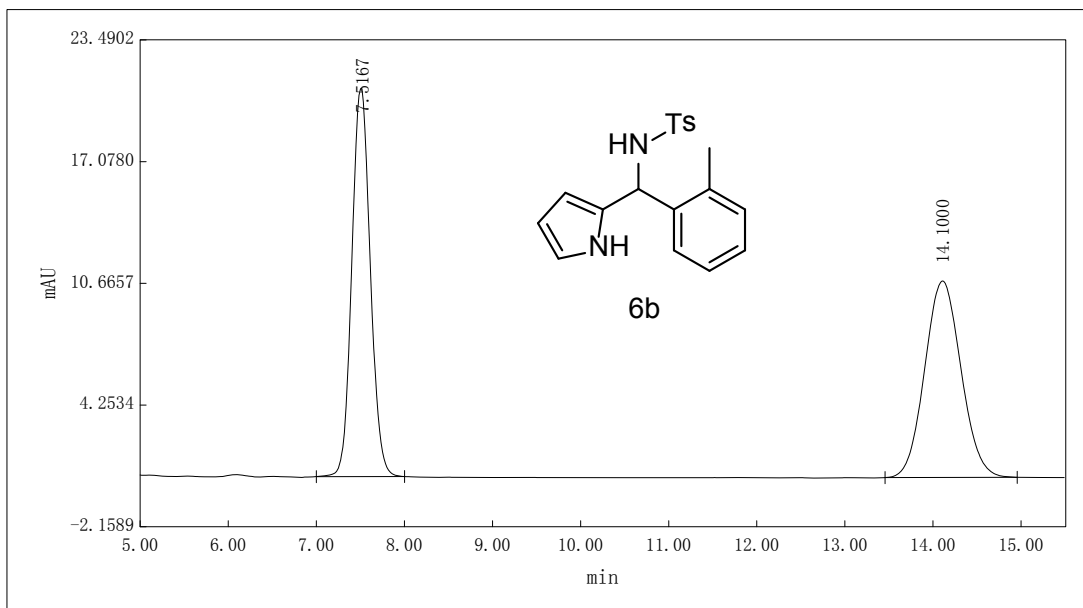
Peak #	RetTime [min]	Area [mAU*s]	type	Area %
1	8.3508	49168.64	BB	88.5291
2	16.9792	6370.87	FF	11.4709



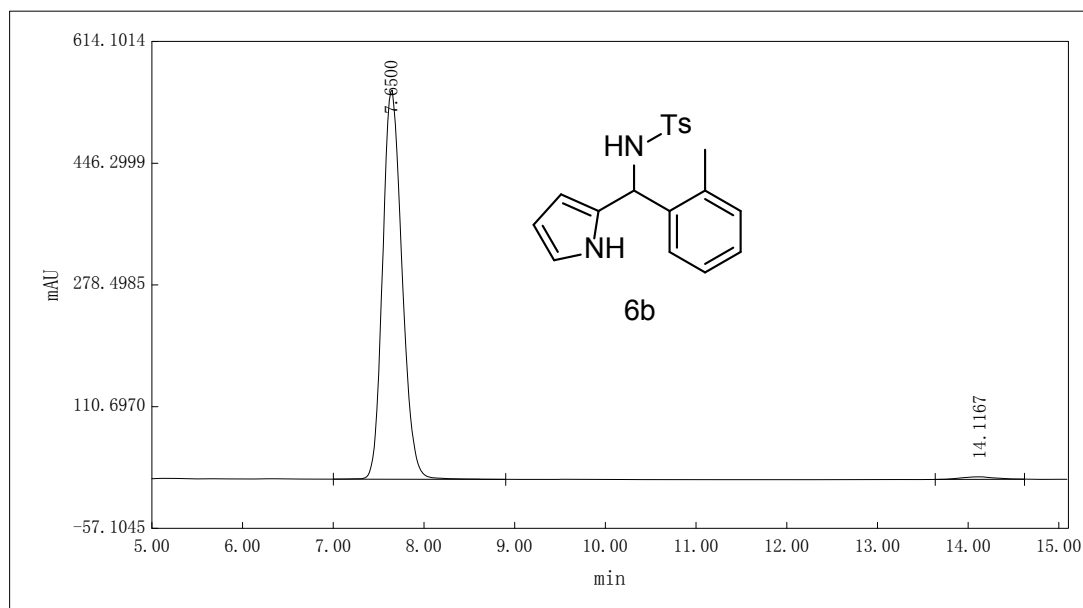
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	12.7000	423.9400	FF	96.0000	50.1078
2	19.2167	422.1161	FF	129.0000	49.8922



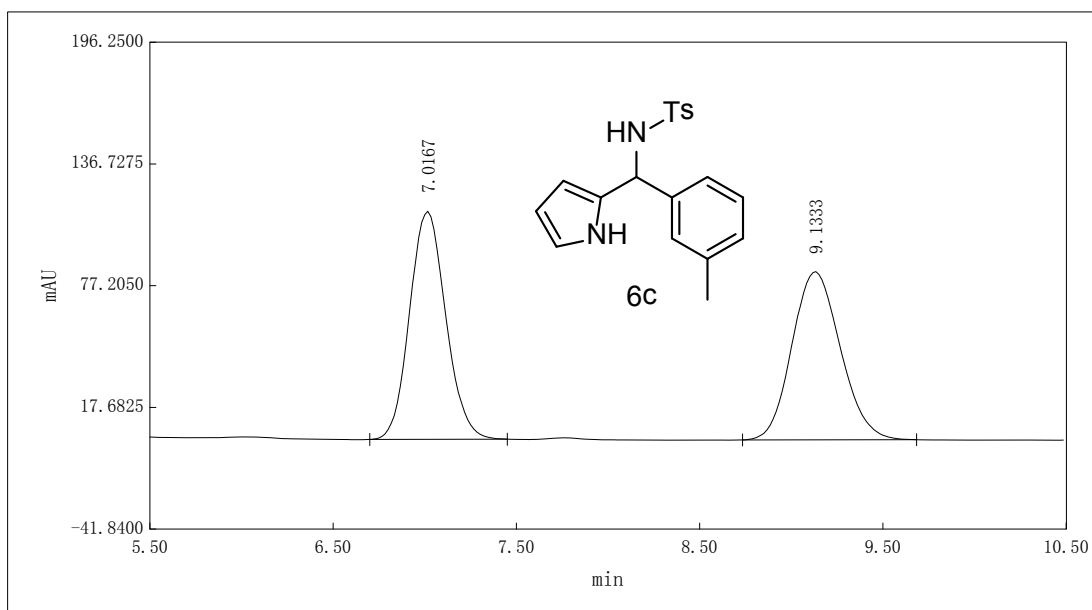
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	13.7667	4305.0681	BB	78.0000	99.2954
2	21.3500	30.5486	FF	69.0000	0.7046



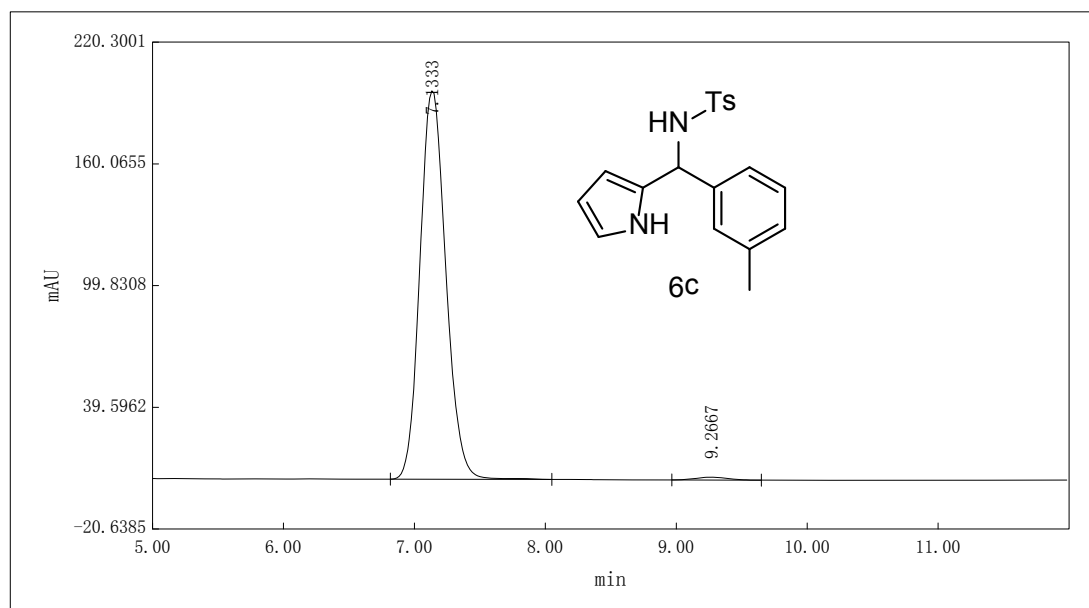
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	7.5167	310.2642	FF	60.0000	50.1424
2	14.1000	308.5023	FF	90.0000	49.8576



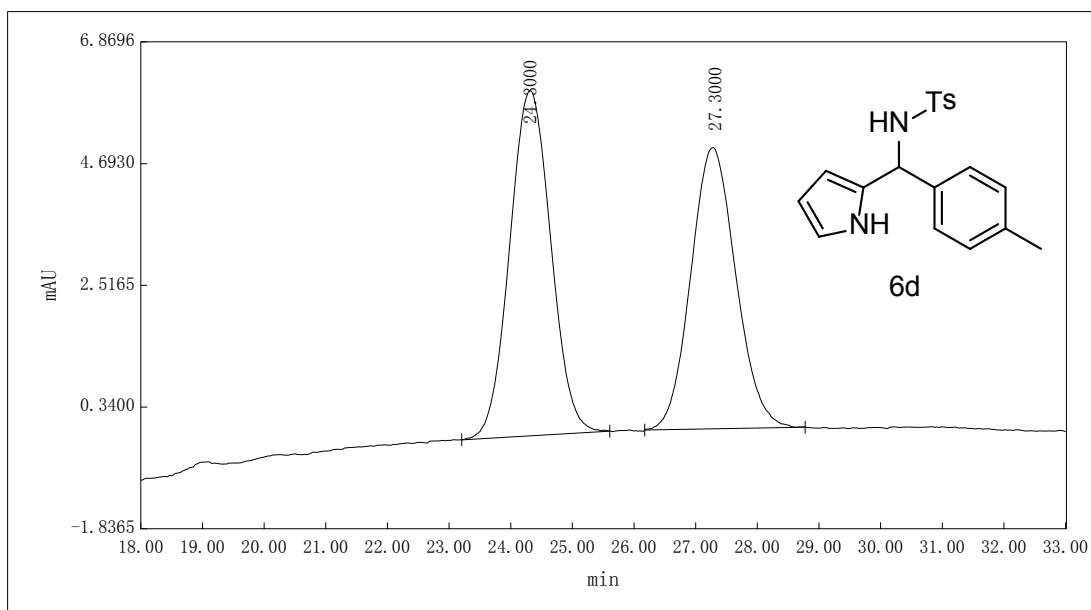
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	7.6500	8310.3791	FF	114.0000	98.8526
2	14.1167	96.4620	FF	59.0000	1.1474



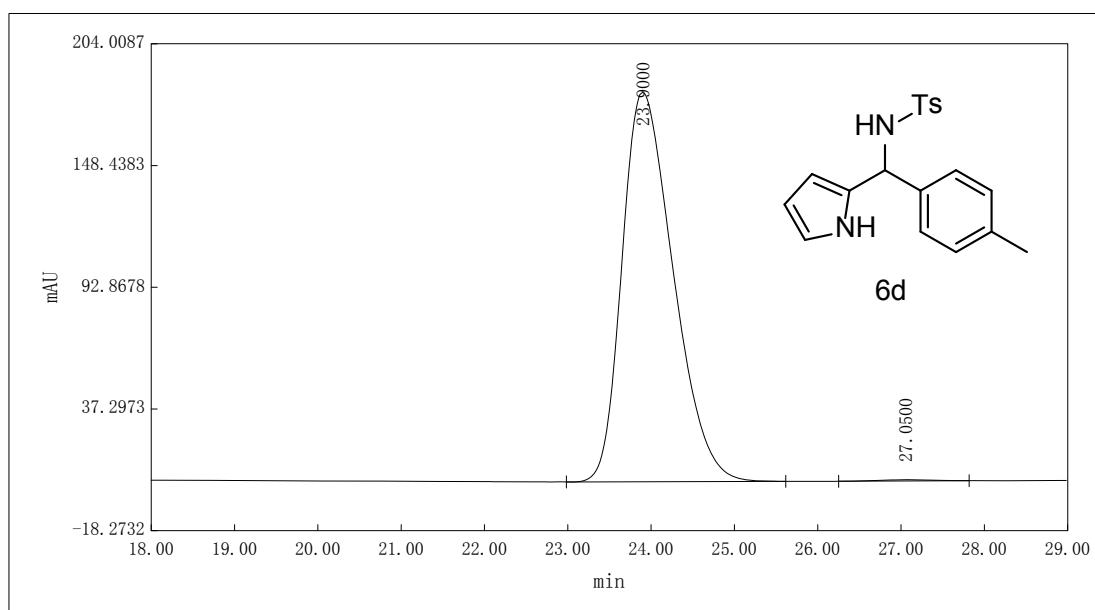
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	7.0167	1529.4819	BB	45.0000	50.3440
2	9.1333	1508.5797	BB	57.0000	49.6560



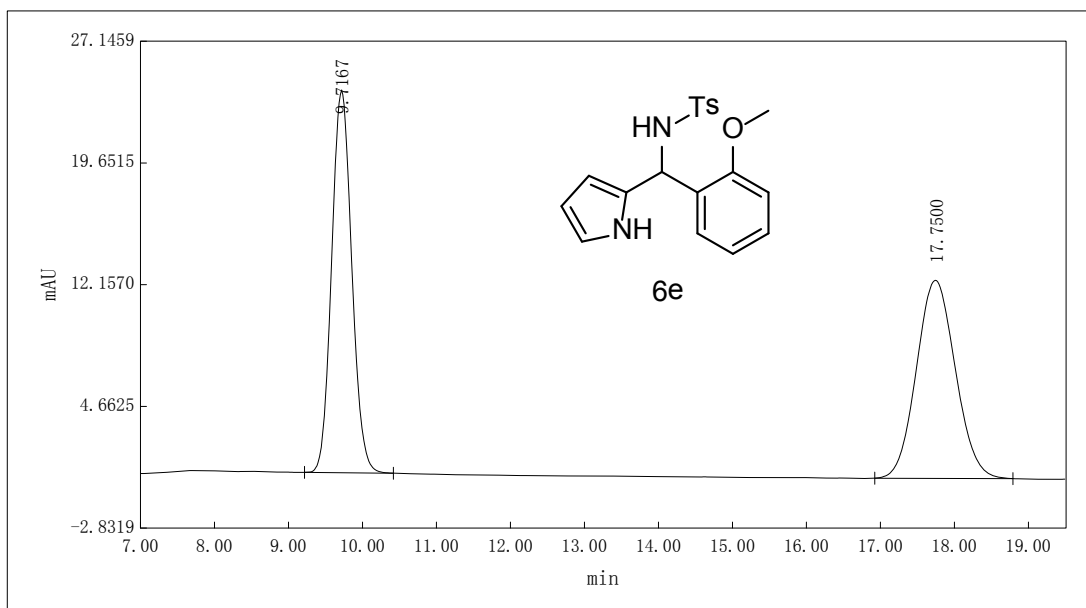
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	7.1333	2759.6622	FF	74.0000	99.0940
2	9.2667	25.2319	FF	41.0000	0.9060



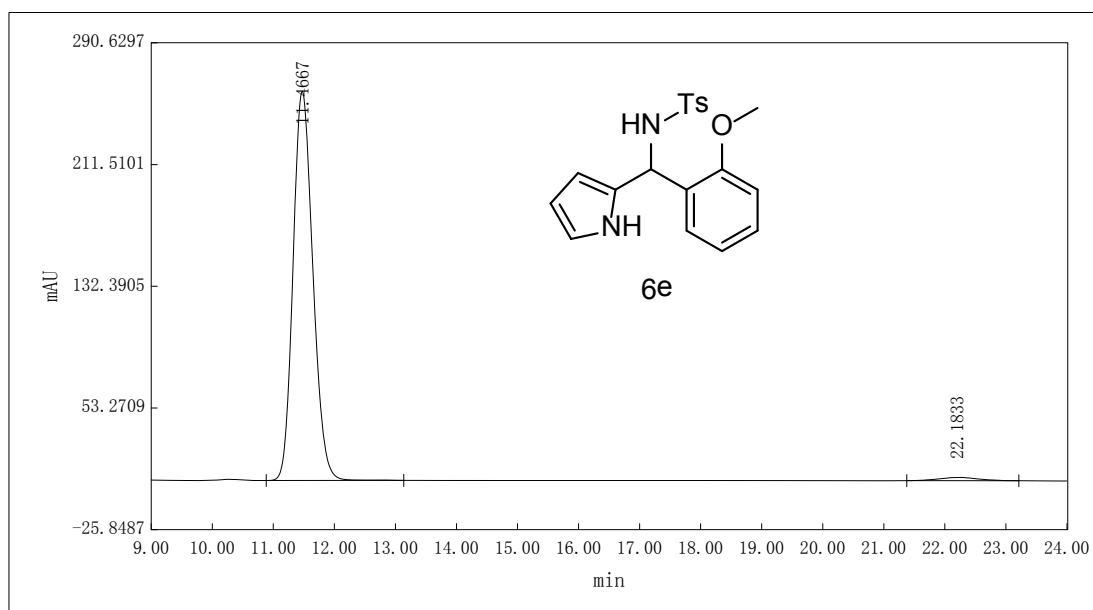
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	24.3000	297.9284	FF	144.0000	52.8605
2	27.3000	265.6846	FF	156.0000	47.1395



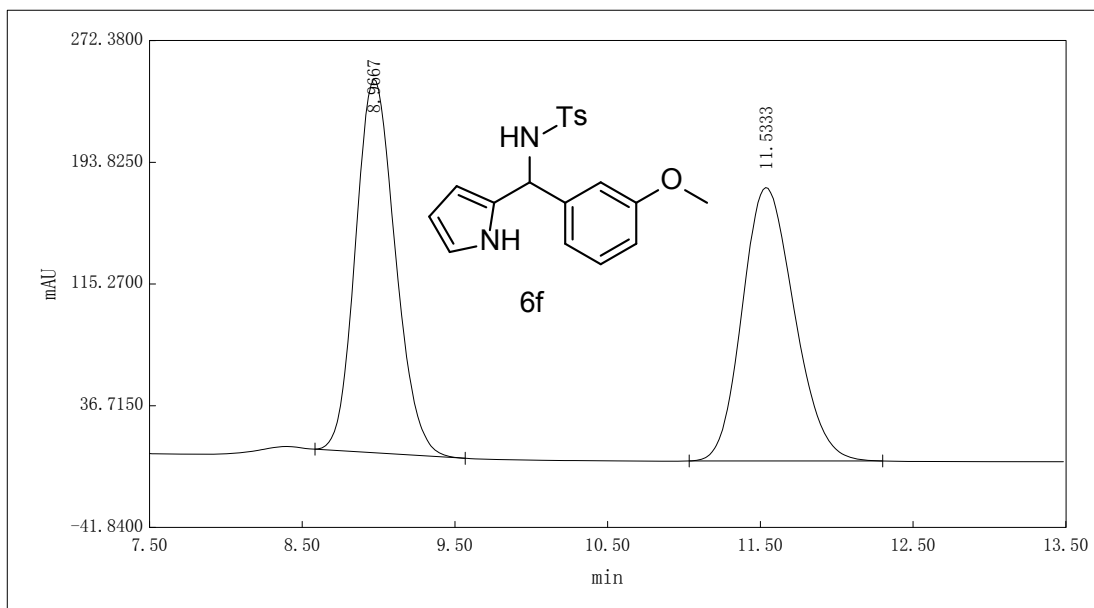
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	23.9000	7895.0508	FF	158.0000	99.6514
2	27.0500	27.6208	FF	94.0000	0.3486



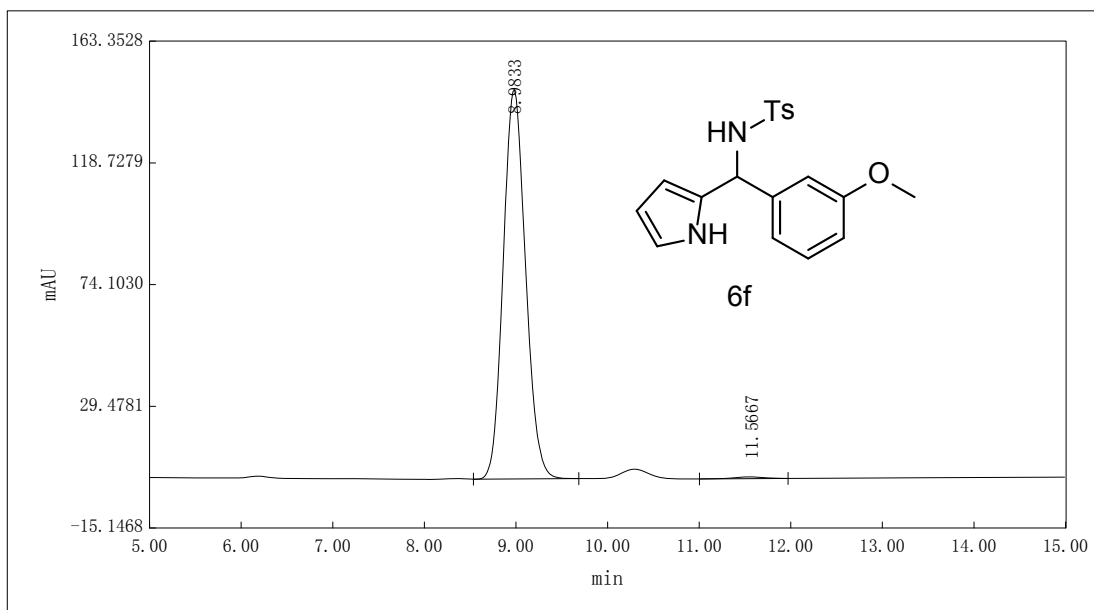
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	9.7167	463.4055	FF	72.0000	49.9806
2	17.7500	463.7656	FF	112.0000	50.0194



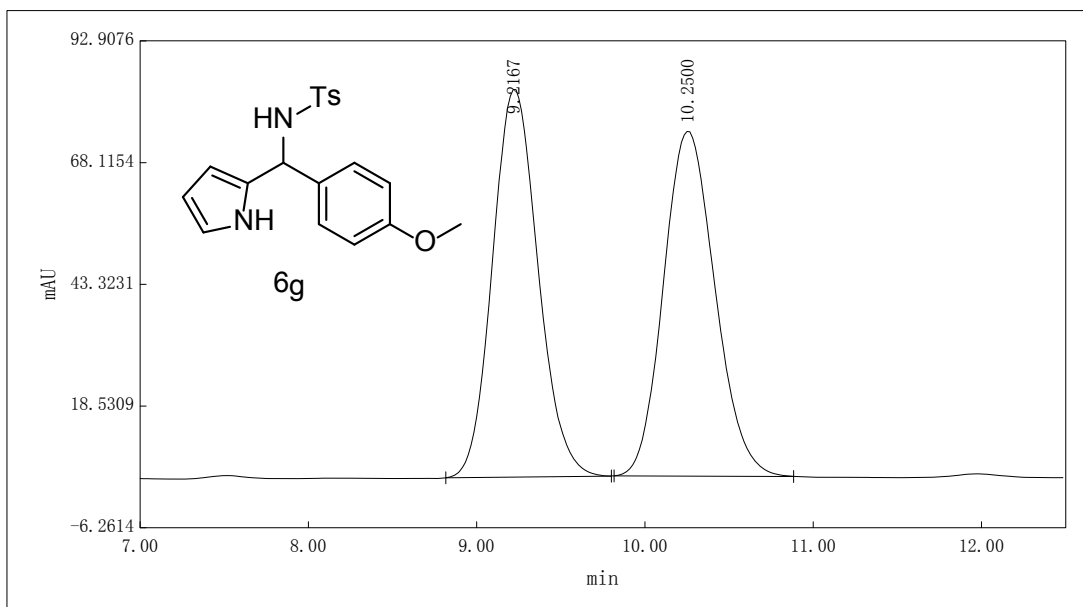
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	11.4667	5926.0362	FF	135.0000	98.3337
2	22.1833	100.4173	FF	110.0000	1.6663



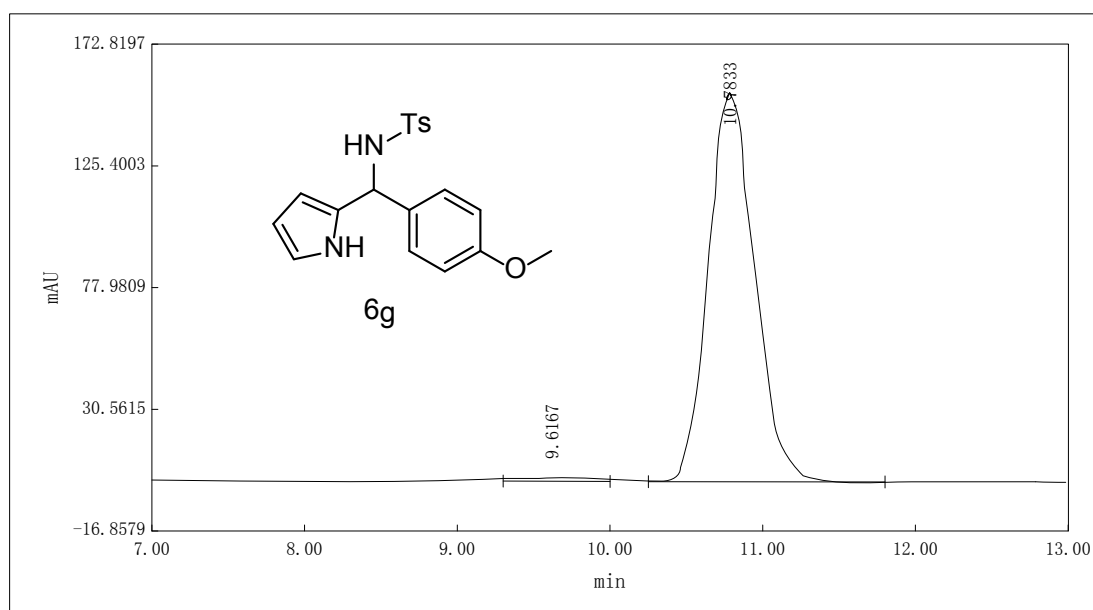
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	8.9667	4348.6797	BB	59.0000	50.9795
2	11.5333	4181.5766	BB	76.0000	49.0205



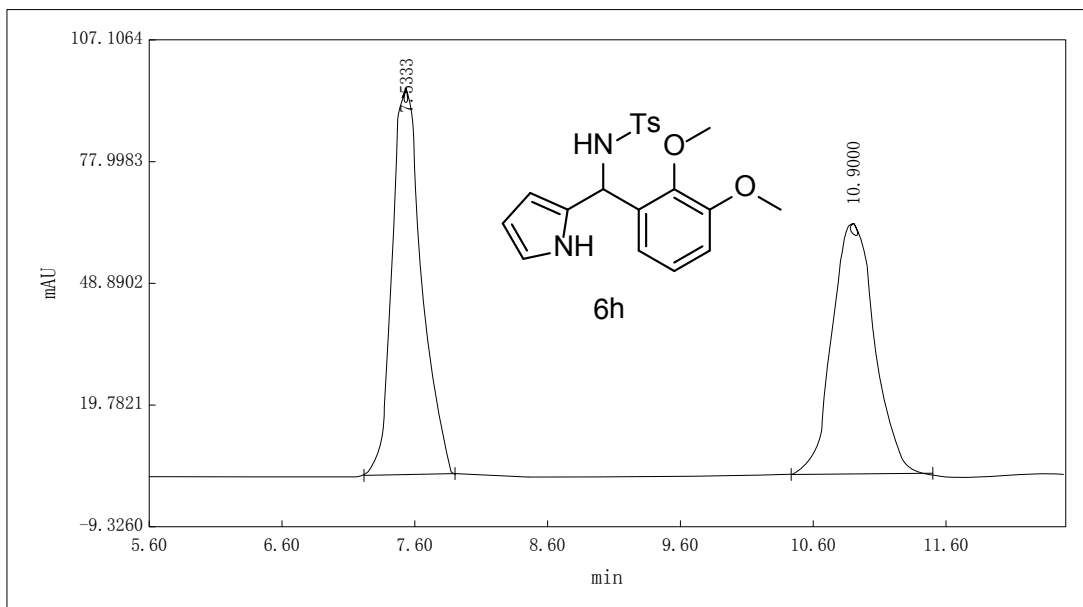
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	8.9833	2516.2268	FF	69.0000	99.4132
2	11.5667	14.8535	FF	58.0000	0.5868



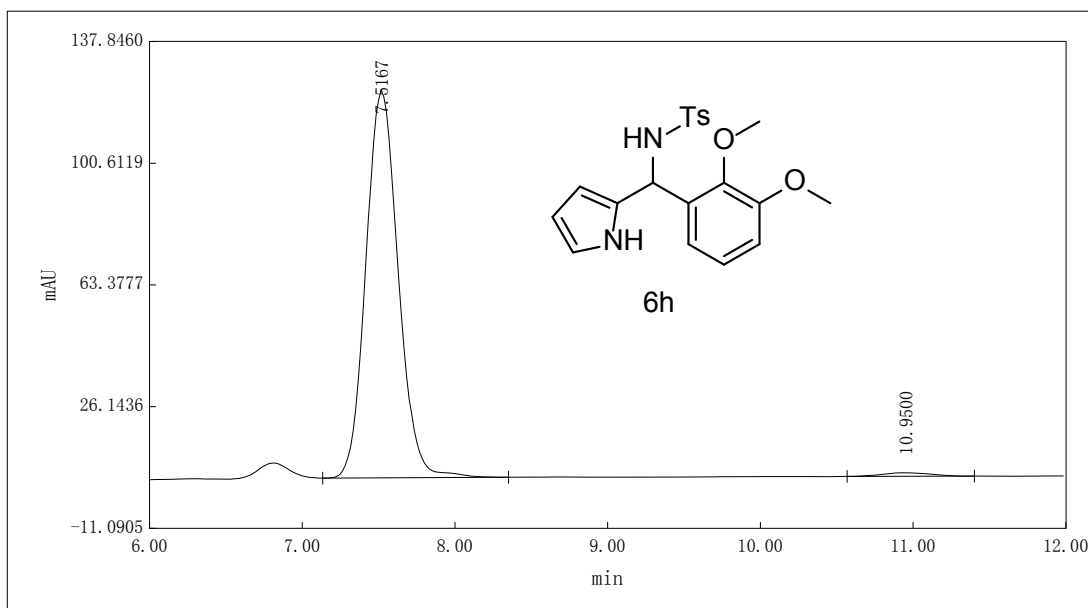
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	9.2167	1549.3971	BB	59.0000	49.9038
2	10.2500	1555.3695	BB	64.0000	50.0962



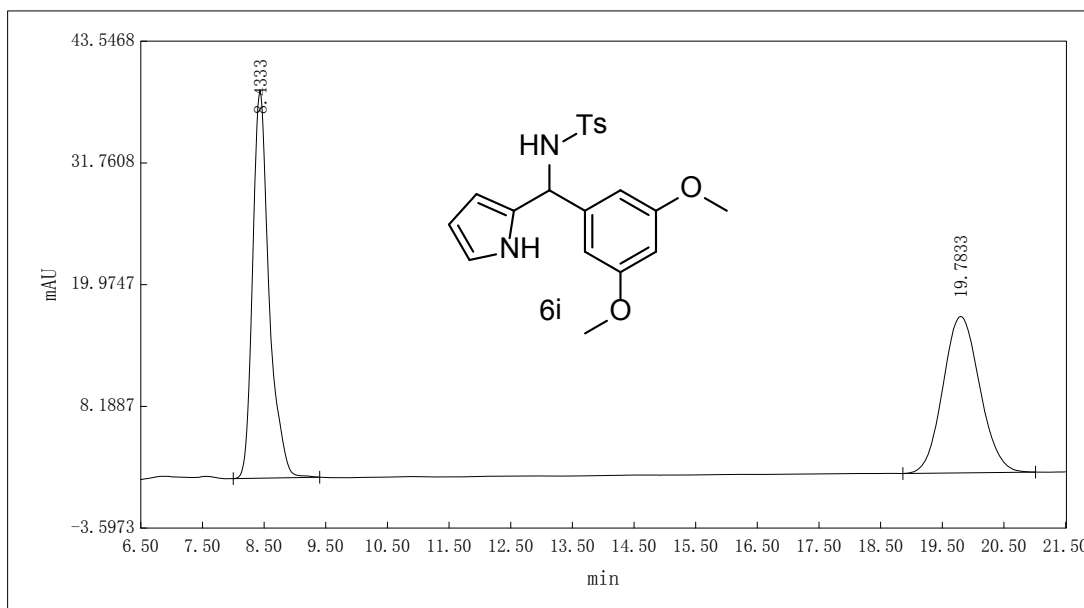
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	9.6167	34.5541	FF	42.0000	1.0226
2	10.7833	3344.5947	FF	93.0000	98.9774



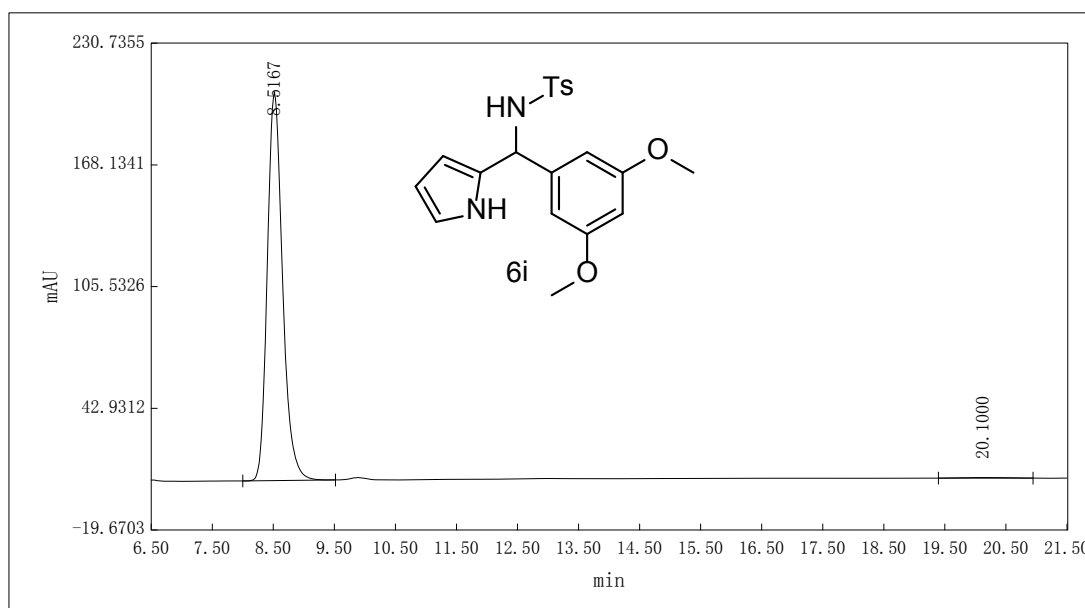
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	7.5333	1314.6259	BB	37.0000	48.4488
2	10.9000	1398.8092	BB	64.0000	51.5512



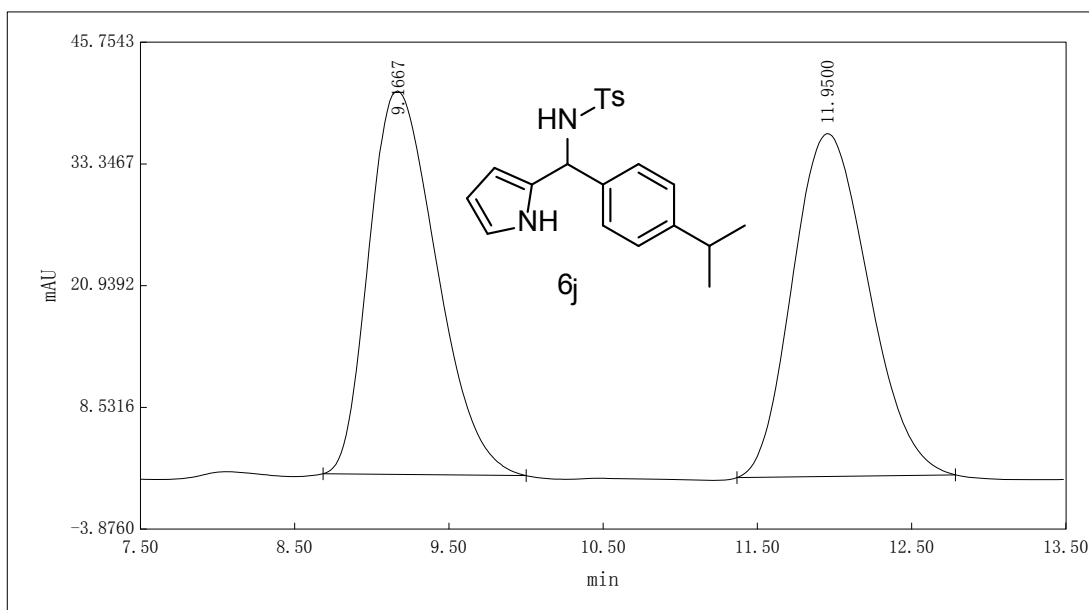
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	7.5167	1814.8695	FF	73.0000	98.6311
2	10.9500	25.1890	FF	50.0000	1.3689



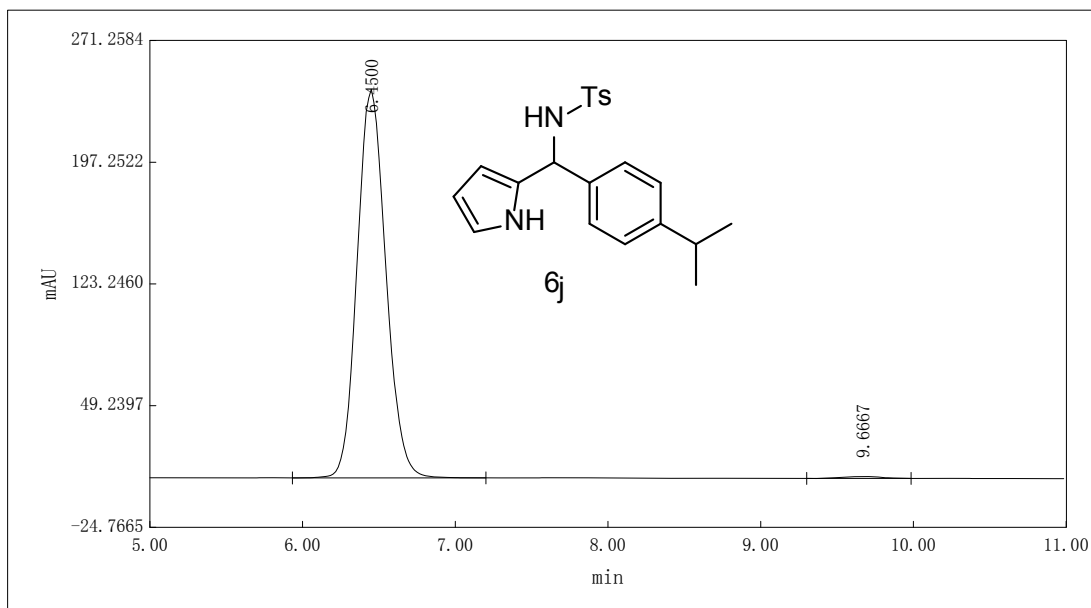
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	8.4333	724.0309	FF	84.0000	52.5378
2	19.7833	654.0835	FF	129.0000	47.4622



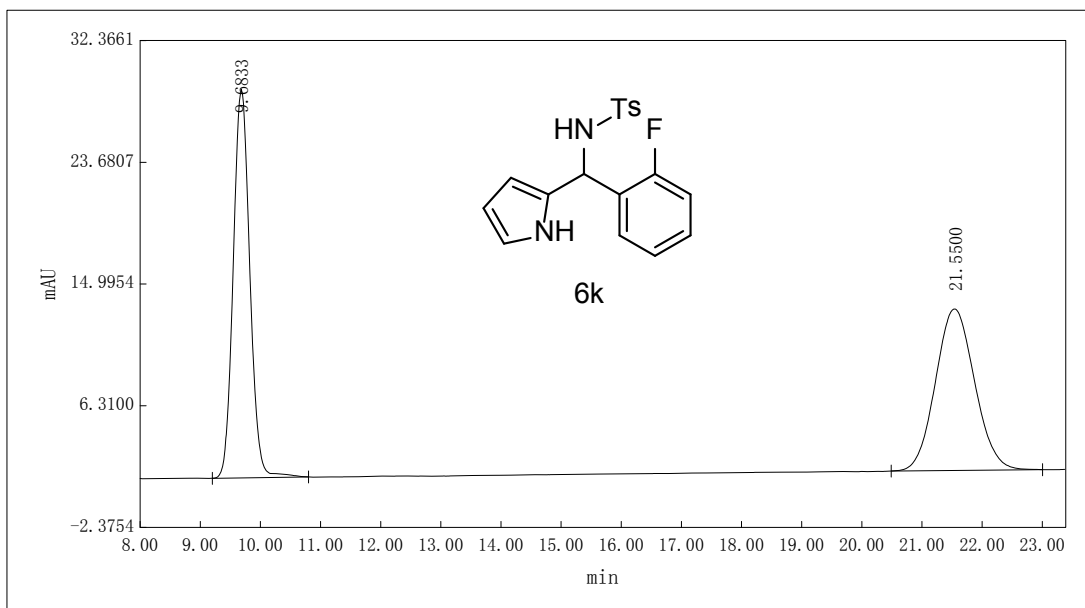
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	8.5167	3607.8903	FF	91.0000	99.5713
2	20.1000	15.5354	FF	93.0000	0.4287



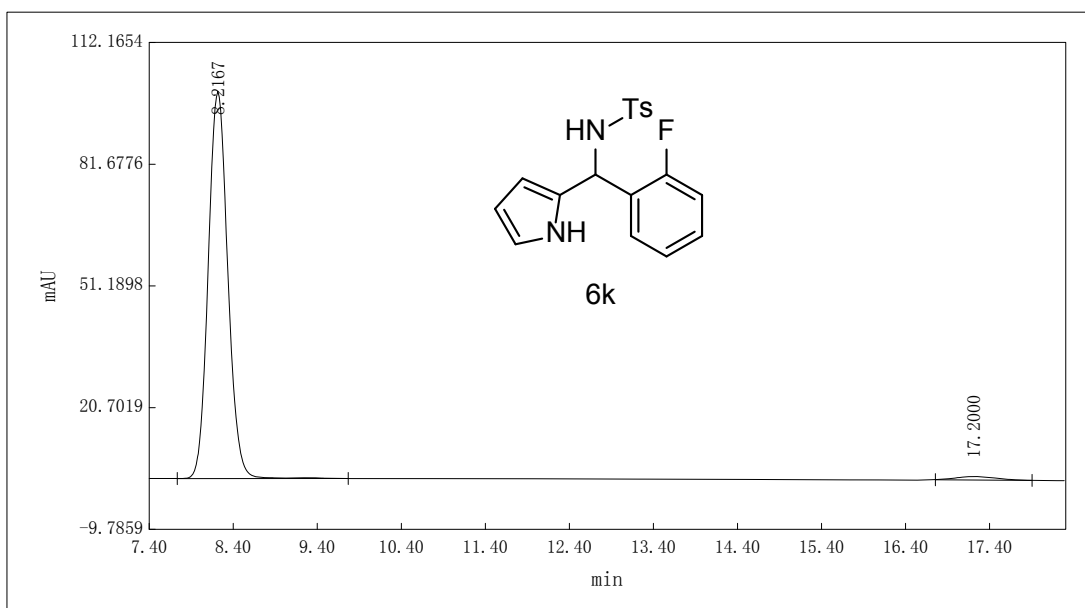
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	9.1667	1240.2820	BB	79.0000	49.8624
2	11.9500	1247.1294	BB	85.0000	50.1376



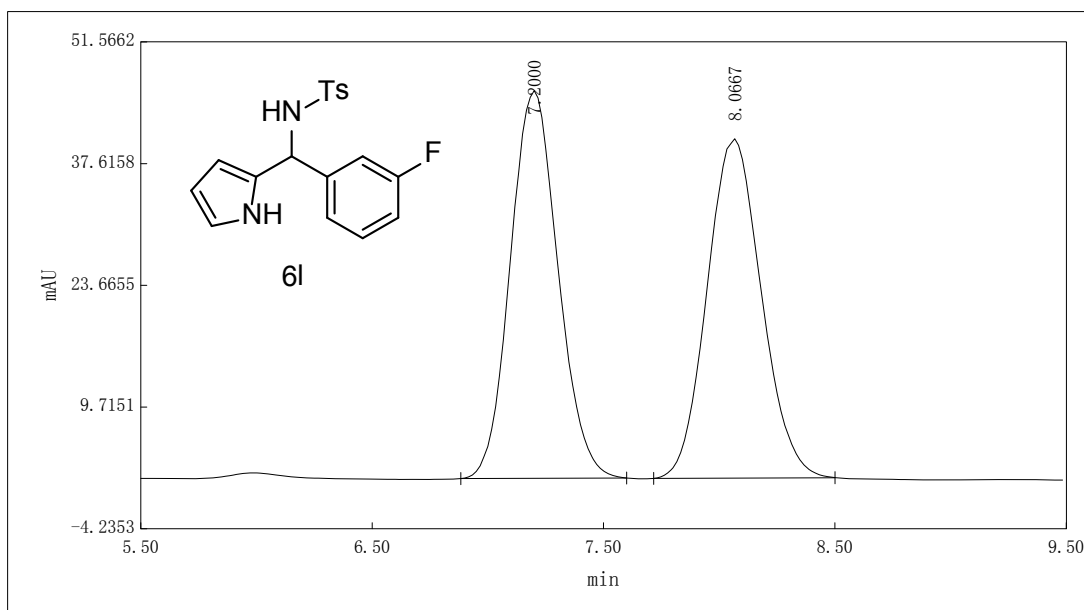
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	6.4500	3218.1324	FF	76.0000	99.3869
2	9.6667	19.8508	FF	41.0000	0.6131



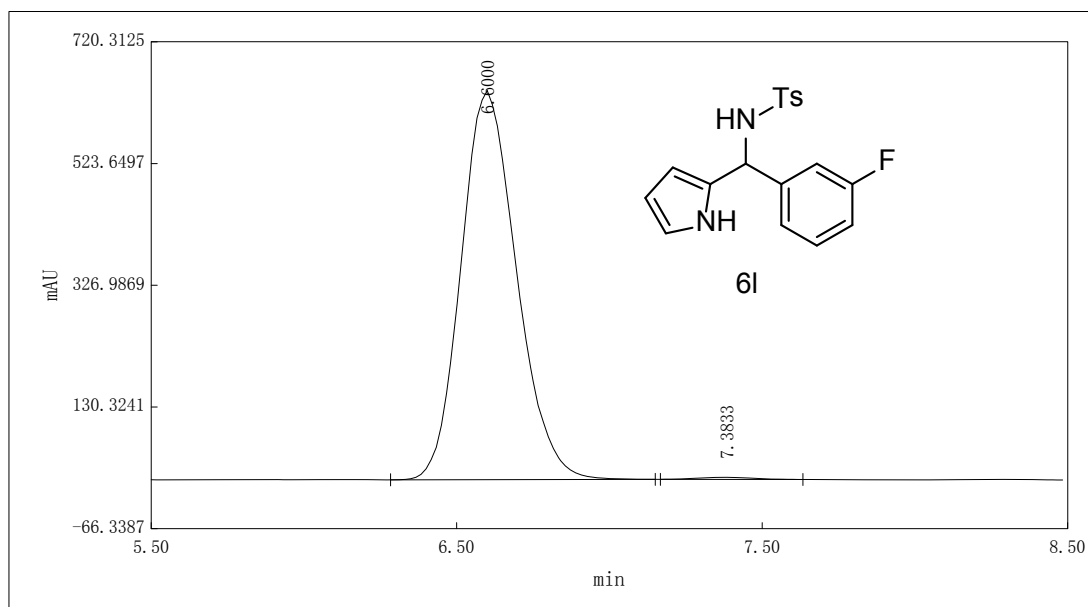
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	9.6833	552.2262	FF	96.0000	50.4667
2	21.5500	542.0123	FF	151.0000	49.5333



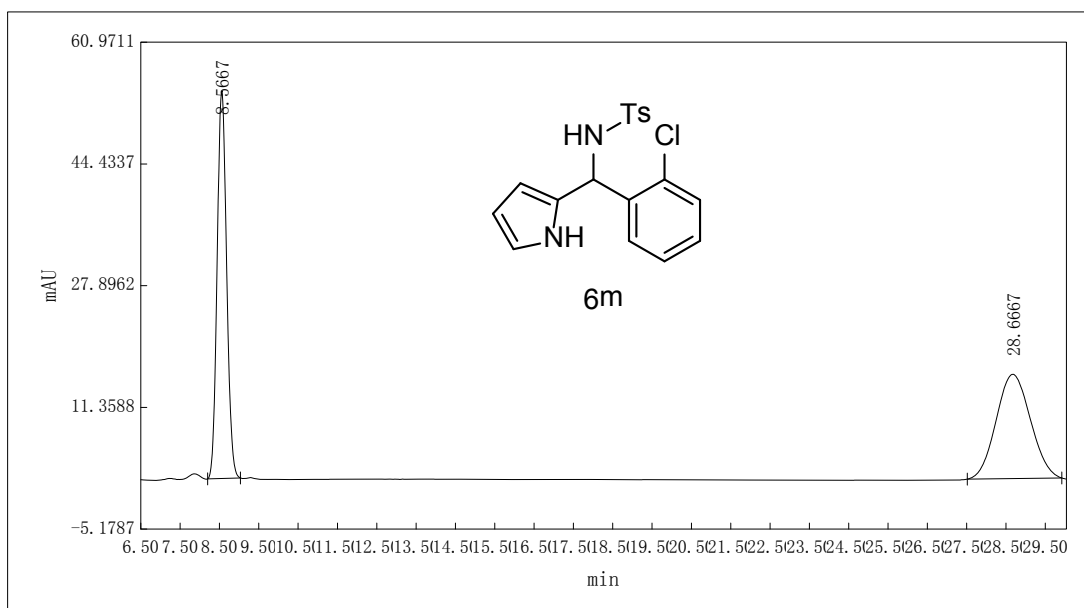
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	8.2167	1628.6571	FF	122.0000	98.2355
2	17.2000	29.2540	FF	69.0000	1.7645



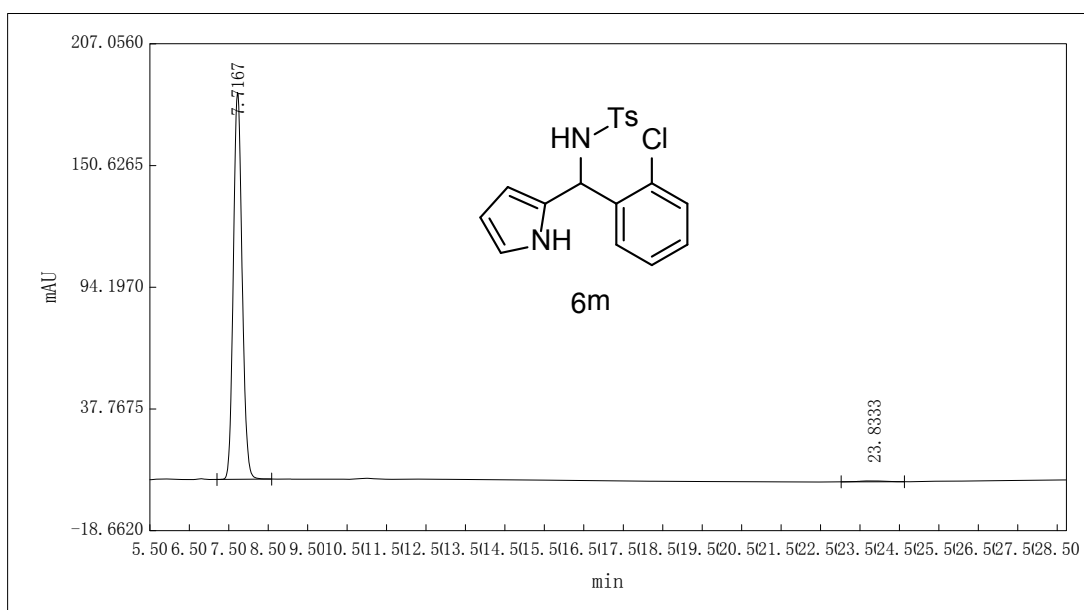
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	7.2000	662.3114	BB	43.0000	50.1193
2	8.0667	659.1571	BB	47.0000	49.8807



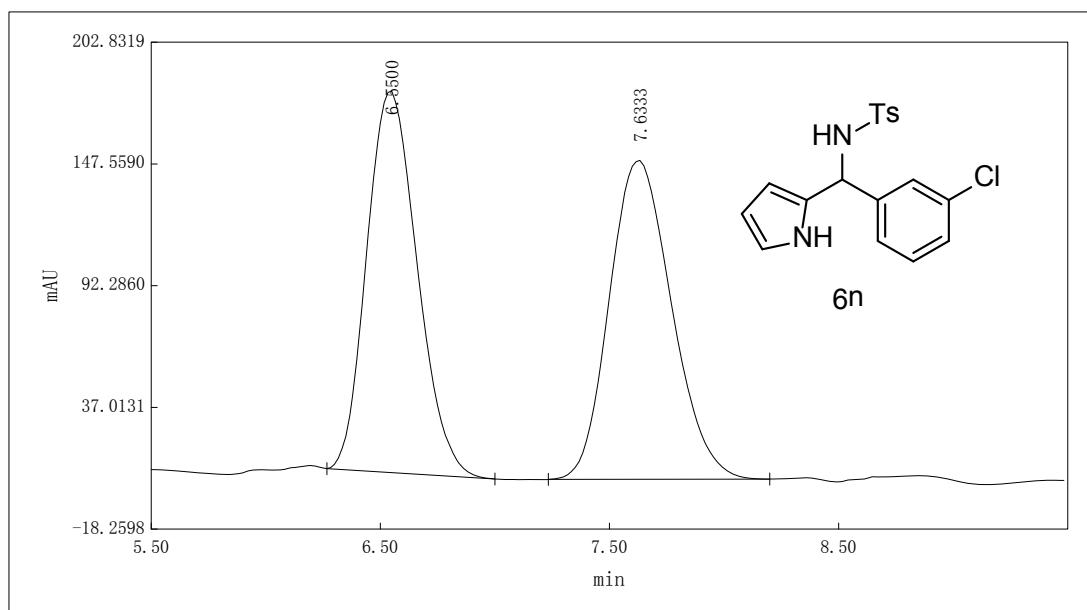
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	6.6000	8245.2907	BB	52.0000	99.4740
2	7.3833	43.5973	BB	28.0000	0.5260



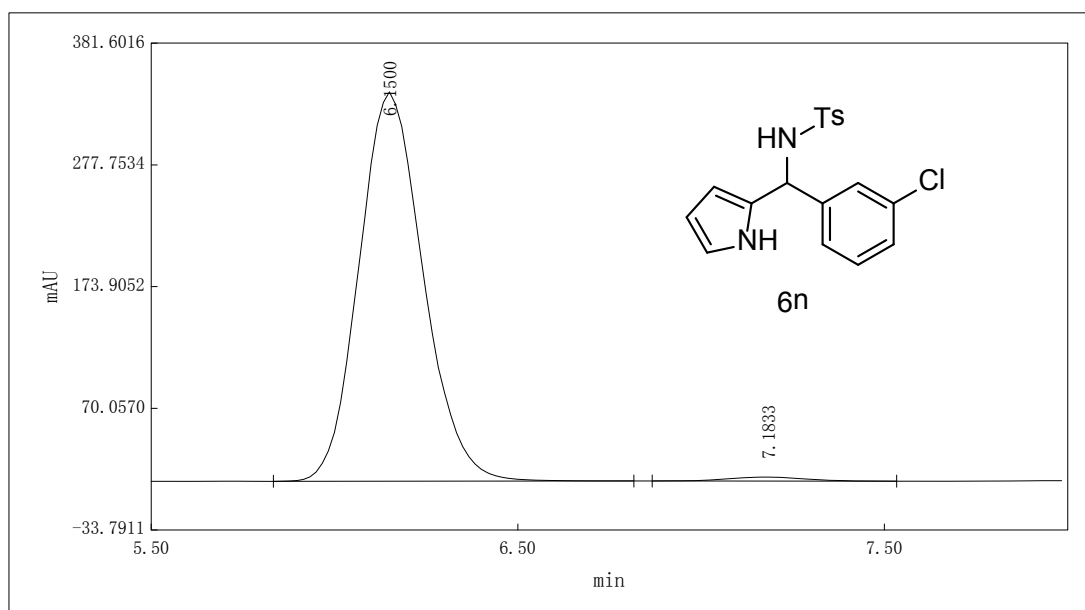
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	8.5667	920.5383	BB	50.0000	50.7761
2	28.6667	892.3977	FF	144.0000	49.2239



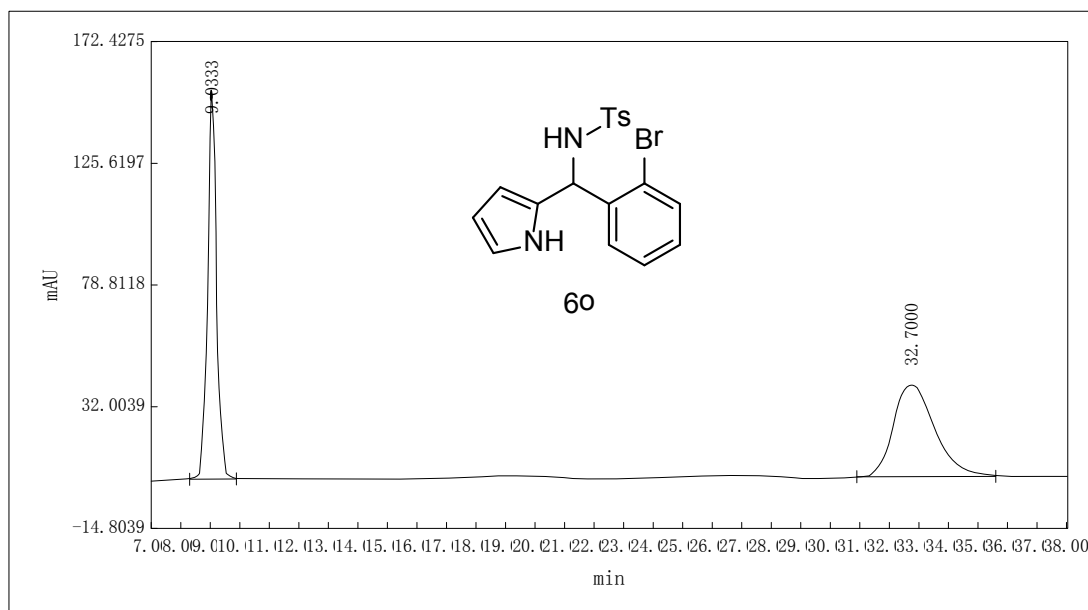
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	7.7167	2893.3366	FF	83.0000	99.3606
2	23.8333	18.6181	BB	96.0000	0.6394



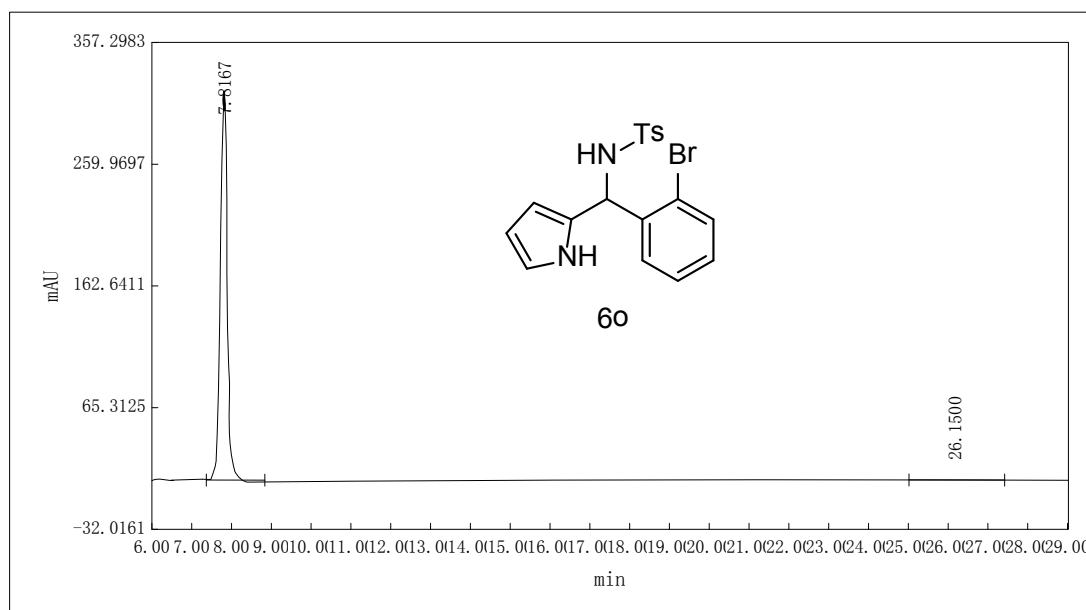
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	6.5500	2758.4248	BB	44.0000	49.2558
2	7.6333	2841.7737	BB	58.0000	50.7442



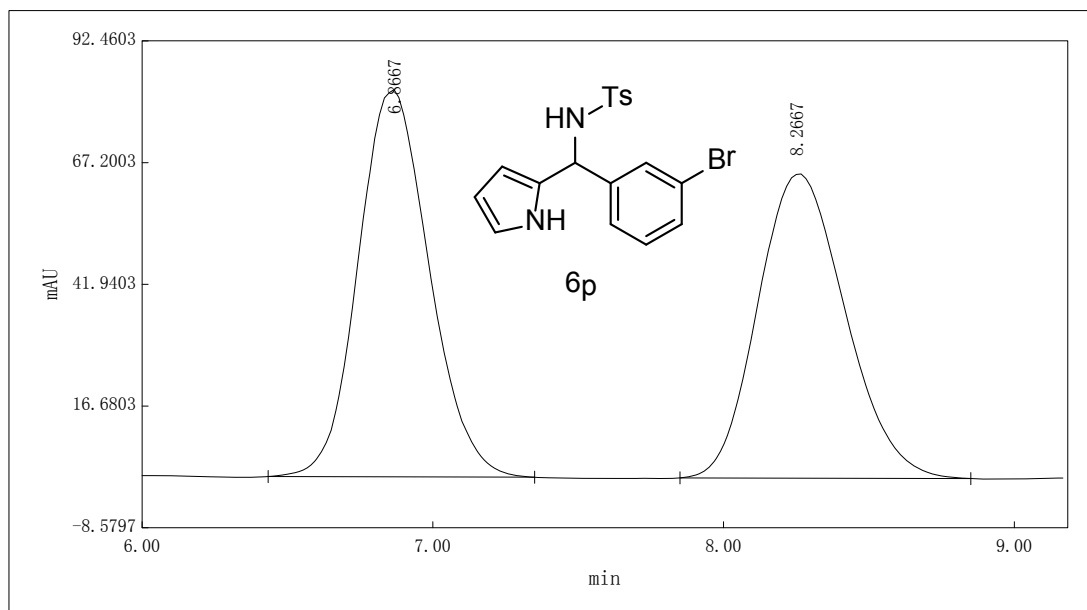
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	6.1500	4204.7581	FF	59.0000	98.7911
2	7.1833	51.4532	FF	40.0000	1.2089



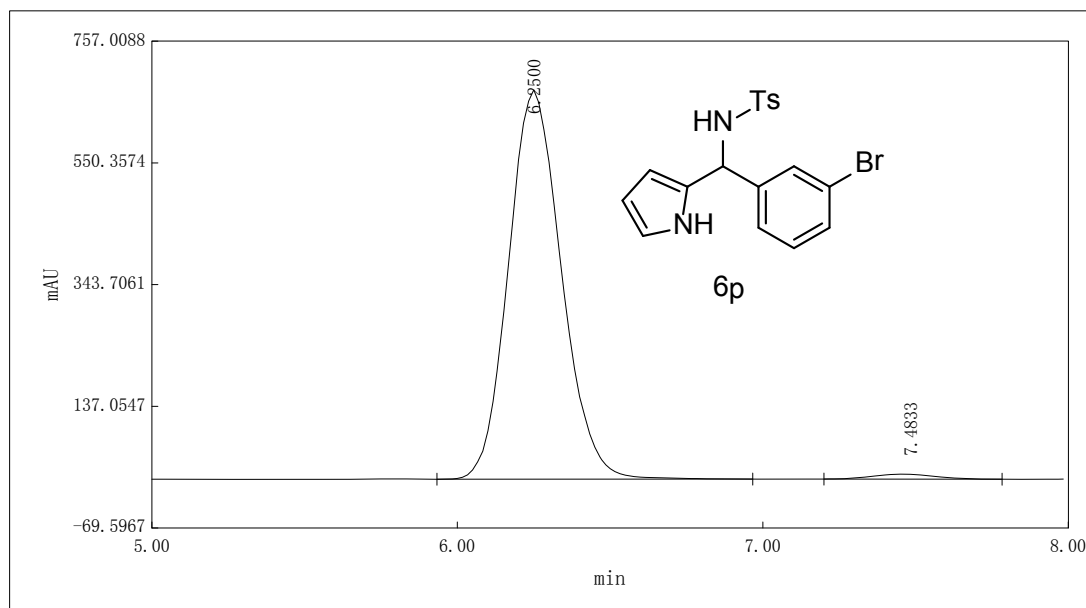
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	9.0333	3629.9775	FF	95.0000	50.2667
2	32.7000	3591.4561	FF	282.0000	49.7333



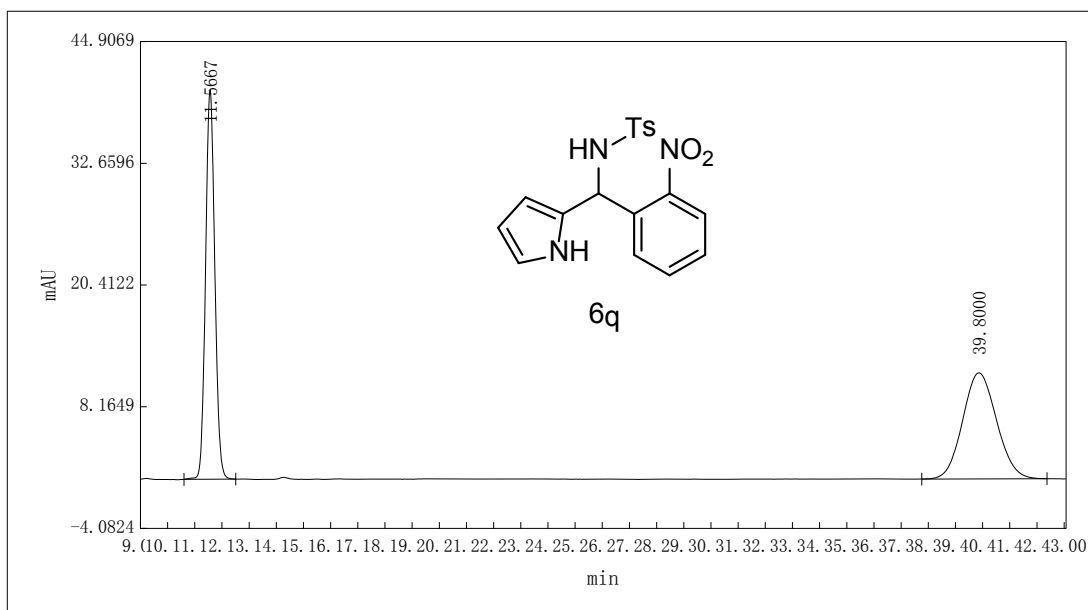
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	7.8167	4877.5885	FF	88.0000	98.2233
2	26.1500	88.2270	FF	144.0000	1.7767



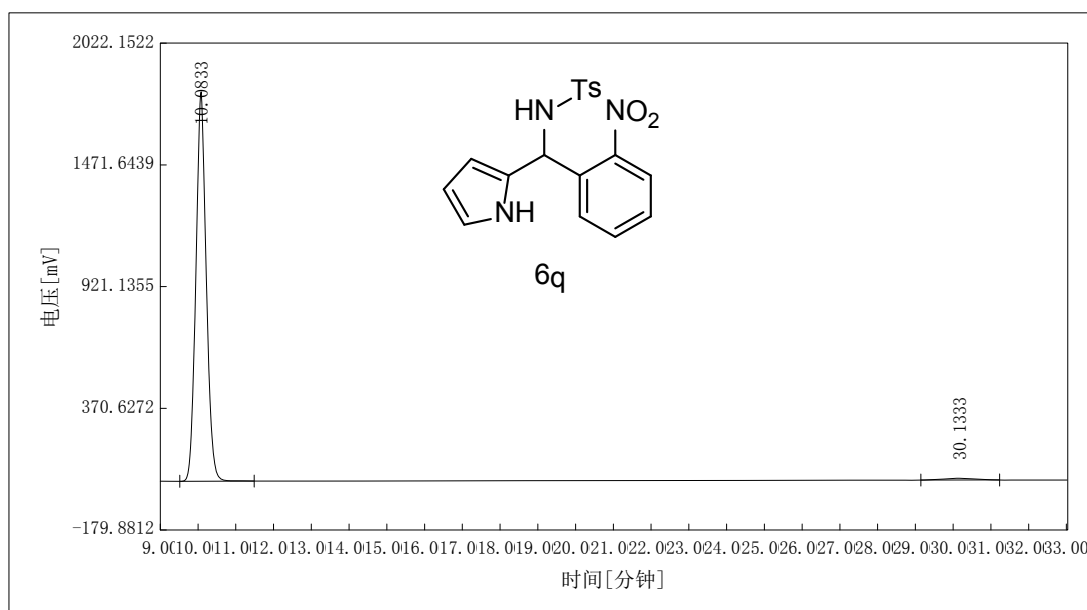
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	6.8667	1420.0285	BB	55.0000	50.8260
2	8.2667	1373.8730	BB	60.0000	49.1740



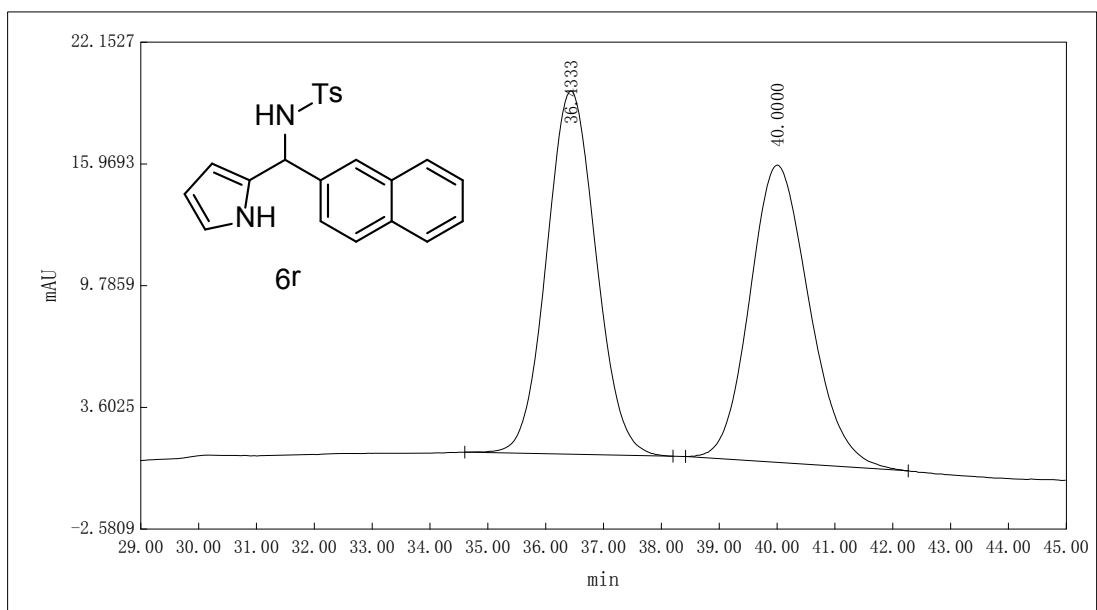
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	6.2500	8277.3009	FF	62.0000	98.5176
2	7.4833	124.5477	BB	35.0000	1.4824



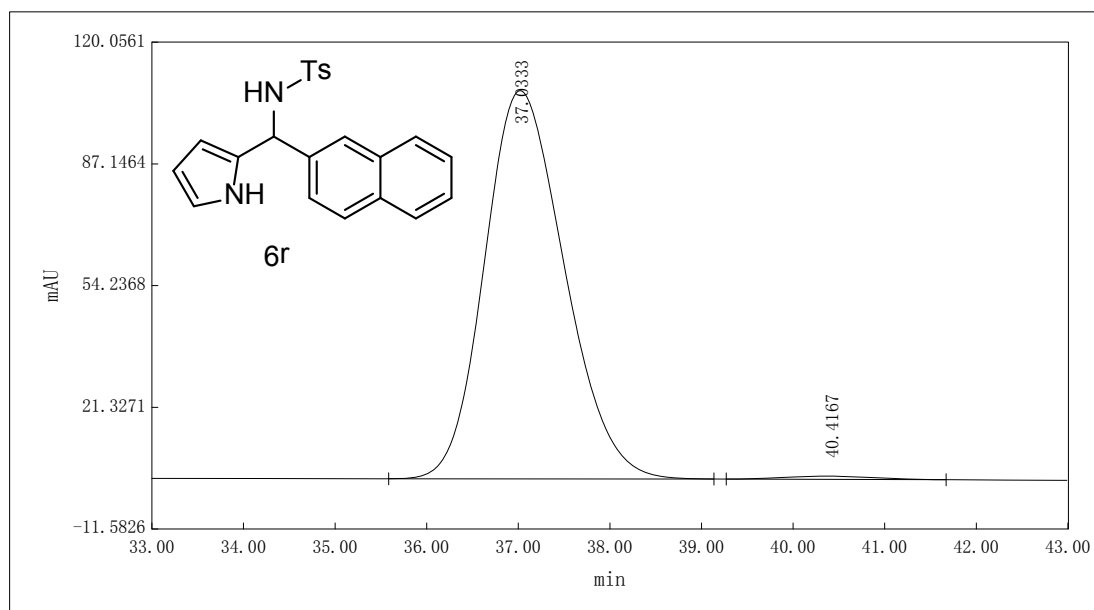
Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	11.5667	962.4428	FF	114.0000	50.0456
2	39.8000	960.6880	FF	276.0000	49.9544



Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	10.0833	34896.7765	FF	118.0000	98.6567
2	30.1333	475.1477	FF	125.0000	1.3433



Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	36.4333	1141.4287	FF	216.0000	50.3029
2	40.0000	1127.6815	FF	231.0000	49.6971



Peak #	RetTime [min]	Area [mAU*s]	type	Peak width [s]	Area %
1	37.0333	6625.0331	FF	213.0000	99.1005
2	40.4167	60.1364	FF	144.0000	0.8995