

Supporting information
Facile approach for C(sp³)-H bond thioetherification of
isochroman

Jie Feng^a Guoping Lu^a and Chun Cai^{a*}

^a Chemical Engineering College, Nanjing University of Science & Technology, Nanjing,
Jiangsu 210094, P. R. China

* Corresponding Author E-mail: c.cai@mail.njust.edu.cn

Table of Contents

1. General information	2
2. General procedures	2
3. Characterization Data	2
4. Copies of ¹H NMR, ¹³C NMR and ¹⁹F NMR Spectra	9

1 General information

All commercial materials were used without further purification. All known compounds are identified by appropriate technique such as ^1H NMR, ^{13}C NMR and compared with previously reported data. All unknown compounds are characterized by ^1H NMR, ^{13}C NMR, MS and elemental analyses. Analytical thin-layer chromatography are performed on glass plates precoated with silica gel impregnated with a fluorescent indicator (254 nm), and the plates are visualized by exposure to ultraviolet light. GC-MS analyses were performed on an Agilent 7890A-5975C instrument (Column: DB-5 MS). Mass spectra are taken on a Finnigan TSQ Quantum - MS instrument in the electrospray ionization (ESI) mode. ^1H NMR and ^{13}C NMR spectra are recorded on an AVANCE 500 Bruker spectrometer operating at 500 MHz and 125 MHz in CDCl_3 , respectively, and chemical shifts are reported in ppm. Elemental analyses are performed on a Yanagimoto MT3CHN recorder. GC analyses are performed on an Agilent 7890A instrument (Column: Agilent 19091J-413: 30 m \times 320 μm \times 0.25 μm , carrier gas: H_2 , FID detection).

2 General procedures

2.1 General procedures for the oxidative C-N formation

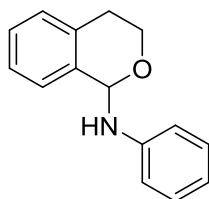
A sealed tube was charged with isochroman(or benzylic ether)(1 mmol), DTBP(1.5 mmol), amine (or amide)(1.3 mmol). The reaction mixture was stirred at 120 $^\circ\text{C}$ for 16 h. Upon completion, The reaction mixture was then cooled to obtain brown liquid, The organic solutions could be purified directly by column chromatography on silica gel to give the pure product (hexane/ethyl acetate=20/1).

2.1 General procedures for the oxidative C=N formation

A sealed tube was charged with isochroman(or benzylic ether)(1 mmol), DTBP(3 mmol), amine (or amide)(1.3 mmol). The reaction mixture was stirred at 120 $^\circ\text{C}$ for 24 h. Upon completion, The reaction mixture was then cooled to obtain dark brown liquid, The organic solutions could be purified directly by column chromatography on silica gel to give the pure product (hexane/ethyl acetate=20/1).

3 Characterization Data

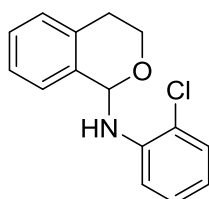
^1H NMR, ^{13}C NMR, ^{19}F NMR, Elemental analyses and MS data of all the isolated products **3a-3d**, **3f-3i**, **3l**, **3n**, **3o**, **3t**, **3x**, **4a-4c**, **4e** were given as below. The ^1H NMR of **3u** and **3v** mixture was also given.

Chemical Formula: C₁₅H₁₅NO

Exact Mass: 225.12

Elemental Analysis: C, 79.97; H, 6.71; N, 6.22; O, 7.10

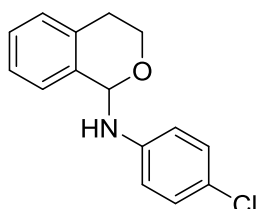
N-phenylisochroman-1-amine **3a** ¹H NMR (500 MHz, CDCl₃) δ 7.30 (t, *J* = 7.1 Hz, 1H), 7.28 – 7.22 (m, 4H), 7.17 (d, *J* = 7.4 Hz, 1H), 6.88 (d, *J* = 8.2 Hz, 2H), 6.83 (t, *J* = 7.3 Hz, 1H), 6.07 (d, *J* = 7.7 Hz, 1H), 4.57 (d, *J* = 7.4 Hz, 1H), 4.16 (ddd, *J* = 12.0, 9.7, 4.0 Hz, 1H), 3.96 – 3.87 (m, 1H), 3.02 – 2.93 (m, 1H), 2.77 (dt, *J* = 16.5, 3.9 Hz, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 145.82, 135.55, 135.08, 129.43, 129.00, 128.04, 126.88, 126.54, 119.02, 114.02, 80.37, 77.38, 77.13, 76.87, 58.45, 28.39. Anal. Calcd for C₁₅H₁₅NO: C, 79.97; H, 6.71. Found: C, 79.68; H, 6.54. MS (ESI) *m/z*: 225.

Chemical Formula: C₁₅H₁₄ClNO

Exact Mass: 259.08

Elemental Analysis: C, 69.37; H, 5.43; Cl, 13.65; N, 5.39; O, 6.16

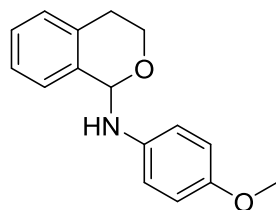
N-(2-chlorophenyl)isochroman-1-amine **3b** ¹H NMR (500 MHz, CDCl₃) δ 7.37 – 7.27 (m, 4H), 7.26 – 7.20 (m, 3H), 6.84 – 6.75 (m, 1H), 6.11 (d, *J* = 7.3 Hz, 1H), 5.22 (d, *J* = 7.0 Hz, 1H), 4.24 – 4.14 (m, 1H), 3.95 (ddd, *J* = 11.6, 5.5, 3.6 Hz, 1H), 3.11 – 2.99 (m, 1H), 2.81 (dt, *J* = 16.5, 3.6 Hz, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 141.04, 133.99, 133.93, 128.21, 127.99, 127.16, 126.91, 125.84, 125.67, 118.68, 118.08, 112.66, 78.95, 76.34, 76.08, 75.83, 57.34, 27.26. Anal. Calcd for C₁₅H₁₄ClNO: C, 69.37; H, 6.43. Found: C, 69.66; H, 6.14. MS (ESI) *m/z*: 259.

Chemical Formula: C₁₅H₁₄ClNO

Exact Mass: 259.08

Elemental Analysis: C, 69.37; H, 5.43; Cl, 13.65; N, 5.39; O, 6.16

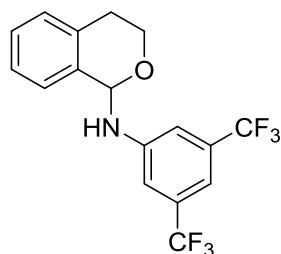
N-(4-chlorophenyl)isochroman-1-amine **3c** ¹H NMR (500 MHz, CDCl₃) δ 7.30 – 7.22 (m, 3H), 7.17 (t, *J* = 7.9 Hz, 3H), 6.80 (d, *J* = 8.8 Hz, 2H), 6.00 (d, *J* = 7.6 Hz, 1H), 4.57 (d, *J* = 7.4 Hz, 1H), 4.18 – 4.06 (m, 1H), 3.94 – 3.86 (m, 1H), 3.01 – 2.91 (m, 1H), 2.77 (dt, *J* = 16.6, 3.9 Hz, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 143.30, 134.06, 133.94, 128.15, 127.95, 127.09, 125.67, 125.50, 122.63, 114.18, 79.33, 76.27, 76.02, 75.76, 57.46, 27.23. Anal. Calcd for C₁₅H₁₄ClNO: C, 69.37; H, 5.43. Found: C, 69.64; H, 5.55. MS (ESI) *m/z*: 259.

Chemical Formula: C₁₆H₁₇NO₂

Exact Mass: 255.13

Elemental Analysis: C, 75.27; H, 6.71; N, 5.49; O, 12.53

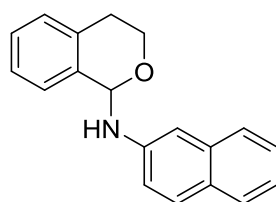
N-(4-methoxyphenyl)isochroman-1-amine **3d** ¹H NMR (500 MHz, CDCl₃) δ 7.34 (d, *J* = 7.4 Hz, 1H), 7.27 – 7.21 (m, 2H), 7.17 (d, *J* = 7.3 Hz, 1H), 6.85 (s, 4H), 6.01 (s, 1H), 4.24 – 4.11 (m, 1H), 3.92 (dt, *J* = 11.5, 4.8 Hz, 1H), 3.79 (s, 3H), 3.01 – 2.92 (m, 1H), 2.79 (dt, *J* = 16.5, 4.1 Hz, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 158.62, 133.77, 133.00, 132.81, 127.76, 126.62, 126.11, 125.00, 113.53, 85.95, 76.28, 76.02, 75.77, 57.07, 54.36, 26.83. Anal. Calcd for C₁₆H₁₇NO₂: C, 75.27; H, 6.71. Found: C, 75.04; H, 6.53. MS (ESI) *m/z*: 255.

Chemical Formula: C₁₇H₁₃F₆NO

Exact Mass: 361.09

Elemental Analysis: C, 56.52; H, 3.63; F, 31.55; N, 3.88; O, 4.43

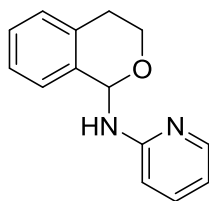
N-(3,5-bis(trifluoromethyl)phenyl)isochroman-1-amine **3f** ¹H NMR (500 MHz, CDCl₃) δ 7.31 (dd, *J* = 9.3, 5.1 Hz, 2H), 7.28 – 7.23 (m, 4H), 7.19 (d, *J* = 7.4 Hz, 1H), 6.06 (d, *J* = 7.3 Hz, 1H), 4.97 (d, *J* = 7.0 Hz, 1H), 4.16 – 4.03 (m, 1H), 4.01 – 3.89 (m, 1H), 3.08 – 2.94 (m, 1H), 2.77 (dt, *J* = 16.6, 3.5 Hz, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 146.66, 134.99, 134.15, 132.98, 132.72, 132.46, 132.20, 129.20, 128.54, 126.80, 126.62, 124.68, 122.51, 113.64, 112.10, 79.82, 58.62, 28.15. Anal. Calcd for C₁₇H₁₃F₆NO: C, 56.52; H, 3.63. Found: C, 56.34; H, 3.50. MS (ESI) *m/z*: 361.

Chemical Formula: C₁₉H₁₇NO

Exact Mass: 275.13

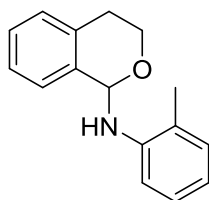
Elemental Analysis: C, 82.88; H, 6.22; N, 5.09; O, 5.81

N-(naphthalen-2-yl)isochroman-1-amine **3g** ¹H NMR (500 MHz, CDCl₃) δ 7.83 (d, *J* = 8.0 Hz, 1H), 7.75 (d, *J* = 8.4 Hz, 1H), 7.51 – 7.36 (m, 5H), 7.30 (dd, *J* = 13.3, 6.8 Hz, 2H), 7.22 (d, *J* = 7.3 Hz, 2H), 6.24 (d, *J* = 7.3 Hz, 1H), 5.20 (d, *J* = 7.2 Hz, 1H), 4.22 (ddd, *J* = 11.7, 9.7, 4.0 Hz, 1H), 3.97 (ddd, *J* = 9.5, 4.5, 3.0 Hz, 1H), 3.07 – 2.98 (m, 1H), 2.86 – 2.78 (m, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 140.02, 134.68, 134.12, 133.36, 127.97, 127.79, 127.33, 127.06, 125.97, 125.63, 125.20, 124.67, 123.93, 122.52, 118.86, 118.06, 106.38, 79.63, 76.26, 76.01, 75.76, 57.61, 27.36. Anal. Calcd for C₁₉H₁₇NO: C, 82.88; H, 6.22. Found: C, 82.54; H, 6.57. MS (ESI) *m/z*: 275.



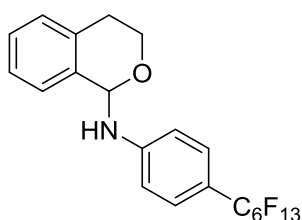
Chemical Formula: C₁₄H₁₄N₂O
 Exact Mass: 226.11
 Elemental Analysis: C, 74.31; H, 6.24; N, 12.38; O, 7.07

N-(isochroman-1-yl)pyridin-2-amine **3h** ¹H NMR (500 MHz, CDCl₃) δ 8.04 – 7.90 (m, 1H), 7.50 (dd, *J* = 10.7, 4.9 Hz, 1H), 7.29 (d, *J* = 7.6 Hz, 1H), 7.26 (t, *J* = 7.3 Hz, 1H), 7.23 – 7.19 (m, 1H), 7.15 (d, *J* = 7.5 Hz, 1H), 6.74 (d, *J* = 8.4 Hz, 1H), 6.70 – 6.61 (m, 1H), 6.27 (d, *J* = 8.1 Hz, 1H), 5.85 (d, *J* = 8.0 Hz, 1H), 4.13 – 4.08 (m, 1H), 3.97 – 3.86 (m, 1H), 3.02 – 2.90 (m, 1H), 2.75 (dd, *J* = 16.5, 2.3 Hz, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 156.35, 147.18, 136.85, 133.92, 127.86, 126.95, 125.87, 125.49, 113.61, 106.53, 77.66, 57.85, 27.27. Anal. Calcd for C₁₄H₁₄N₂O: C, 74.31; H, 6.24. Found: C, 74.62; H, 6.07. MS (ESI) *m/z*: 226.



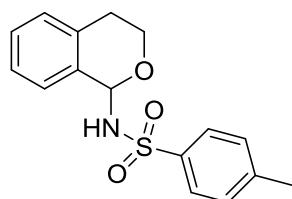
Chemical Formula: C₁₆H₁₇NO
 Exact Mass: 239.13
 Elemental Analysis: C, 80.30; H, 7.16; N, 5.85; O, 6.69

N-(o-tolyl)isochroman-1-amine **3i** ¹H NMR (500 MHz, CDCl₃) δ 7.34 (d, *J* = 7.5 Hz, 1H), 7.28 (dt, *J* = 6.9, 4.5 Hz, 2H), 7.20 (d, *J* = 7.4 Hz, 2H), 7.15 (d, *J* = 8.0 Hz, 1H), 7.11 (d, *J* = 7.4 Hz, 1H), 6.79 (t, *J* = 7.3 Hz, 1H), 6.11 (d, *J* = 7.5 Hz, 1H), 4.42 (d, *J* = 7.3 Hz, 1H), 4.23 – 4.13 (m, 1H), 3.99 – 3.89 (m, 1H), 3.07 – 2.95 (m, 1H), 2.83 (s, 1H), 2.15 (s, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 142.88, 134.76, 134.14, 129.29, 127.93, 126.95, 126.26, 125.81, 125.55, 121.29, 117.66, 111.11, 79.28, 76.30, 76.04, 75.79, 57.44, 27.38, 16.65. Anal. Calcd for C₁₆H₁₇NO: C, 80.30; H, 7.16. Found: C, 80.67; H, 6.97. MS (ESI) *m/z*: 239.



Chemical Formula: C₂₁H₁₄F₁₃NO
 Exact Mass: 543.09
 Elemental Analysis: C, 46.42; H, 2.60; F, 45.46; N, 2.58; O, 2.94

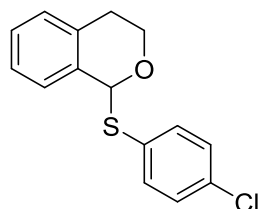
N-(4-(6,6,6,6,6,6,6,6,6,6,6,6,6,6-tridecafluoro-6l16-hexa-1,3,5-triyn-1-yl)phenyl)isochroman-1-amine **3l** ¹H NMR (500 MHz, CDCl₃) δ 7.39 (d, *J* = 8.6 Hz, 2H), 7.27 – 7.22 (m, 2H), 7.20 (d, *J* = 7.4 Hz, 1H), 7.14 (d, *J* = 7.4 Hz, 1H), 6.88 (d, *J* = 8.6 Hz, 2H), 6.04 (d, *J* = 6.6 Hz, 1H), 4.84 (d, *J* = 6.8 Hz, 1H), 4.13 – 4.03 (m, 1H), 3.89 (ddd, *J* = 11.6, 5.3, 3.9 Hz, 1H), 3.01 – 2.89 (m, 1H), 2.73 (dt, *J* = 16.5, 3.7 Hz, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 148.66, 135.03, 134.68, 129.14, 128.35, 126.72, 118.63, 118.44, 114.30, 113.48, 79.72, 77.36, 77.11, 76.85, 58.62, 28.23. ¹⁹F NMR (470 MHz, CDCl₃) δ -80.73, -80.75, -80.77, -109.45, -109.47, -121.25, -121.85, -122.69, -126.09. Anal. Calcd for C₂₁H₁₄F₁₃NO: C, 46.42; H, 2.60. Found: C, 46.53; H, 2.99. MS (ESI) *m/z*: 543.

Chemical Formula: C₁₆H₁₇NO₃S

Exact Mass: 303.09

Elemental Analysis: C, 63.35; H, 5.65; N, 4.62; O, 15.82; S, 10.57

N-(isochroman-1-yl)-4-methylbenzenesulfonamide **3n** ¹H NMR (500 MHz, CDCl₃) δ 7.85 (d, *J* = 8.2 Hz, 2H), 7.31 (d, *J* = 8.2 Hz, 2H), 7.21 (dt, *J* = 11.5, 3.8 Hz, 3H), 7.08 (d, *J* = 7.1 Hz, 1H), 6.10 (d, *J* = 8.5 Hz, 1H), 5.43 (d, *J* = 8.0 Hz, 1H), 3.73 – 3.58 (m, 2H), 2.89 – 2.79 (m, 1H), 2.61 (dt, *J* = 16.6, 3.6 Hz, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 143.45, 138.90, 134.61, 132.94, 129.57, 128.93, 128.53, 127.32, 126.90, 126.83, 80.01, 58.87, 27.67, 21.67. Anal. Calcd for C₁₆H₁₇NO₃S: C, 63.35; H, 5.65. Found: C, 63.71; H, 6.43. MS (ESI) *m/z*: 303.

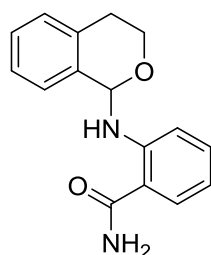


1-((4-chlorophenyl)thio)isochromane

Exact Mass: 276.04

Elemental Analysis: C, 65.09; H, 4.73; Cl, 12.81; O, 5.78; S, 11.58

1-((4-chlorophenyl)thio)isochromane **3o**. ¹H NMR (500 MHz, CDCl₃) δ 7.56 (d, *J* = 8.3 Hz, 2H), 7.40 – 7.30 (m, 3H), 7.28 – 7.21 (m, 2H), 7.20 – 7.11 (m, 1H), 6.49 (s, 1H), 4.55 (td, *J* = 11.5, 3.3 Hz, 1H), 4.03 (dd, *J* = 11.3, 6.2 Hz, 1H), 3.20 – 3.09 (m, 1H), 2.72 (dd, *J* = 16.5, 2.4 Hz, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 133.56, 132.90, 132.48, 132.27, 131.64, 128.06, 127.89, 126.93, 126.13, 125.16, 85.06, 76.39, 76.14, 75.89, 57.36, 26.75. Anal. Calcd for C₁₅H₁₃ClOS: C, 65.09; H, 4.73. Found: C, 64.88; H, 4.64. MS (ESI) *m/z*: 276.

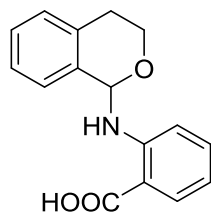
Chemical Formula: C₁₆H₁₆N₂O₂

Exact Mass: 268.12

Elemental Analysis: C, 71.62; H, 6.01; N, 10.44; O, 11.93

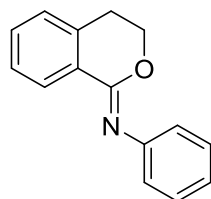
2-(isochroman-1-ylamino)benzamide **3t**

¹H NMR (500 MHz, CDCl₃) δ 8.51 (d, *J* = 6.5 Hz, 1H), 7.41 (dd, *J* = 13.5, 4.7 Hz, 2H), 7.32 – 7.21 (m, 4H), 7.15 (d, *J* = 7.1 Hz, 1H), 6.82 – 6.71 (m, 1H), 6.08 (d, *J* = 6.8 Hz, 1H), 5.83 (s, 2H), 4.21 – 4.10 (m, 1H), 3.90 (ddd, *J* = 11.5, 5.6, 3.4 Hz, 1H), 2.99 (ddd, *J* = 15.9, 9.9, 5.6 Hz, 1H), 2.75 (dt, *J* = 16.5, 3.5 Hz, 1H). ¹³C NMR (126 MHz, CDCl₃) δ 171.92, 148.42, 135.16, 134.79, 133.63, 128.91, 128.13, 127.97, 126.86, 126.67, 116.84, 114.53, 114.34, 79.60, 77.40, 77.15, 76.89, 58.53, 28.39. Anal. Calcd for C₁₆H₁₆N₂O₂: C, 71.62; H, 6.01. Found: C, 71.95; H, 5.76. MS (ESI) *m/z*: 268.



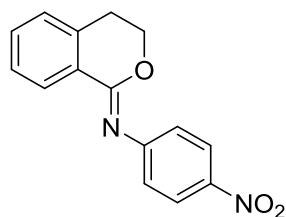
Chemical Formula: C₁₆H₁₅NO₃
 Exact Mass: 269.11
 Elemental Analysis: C, 71.36; H, 5.61; N, 5.20; O, 17.82

2-(isochroman-1-ylamino)benzoic acid 3x ¹H NMR (500 MHz, DMSO) δ 12.89 (s, 1H), 8.58 (d, *J* = 7.1 Hz, 1H), 7.92 (d, *J* = 7.8 Hz, 1H), 7.44 (t, *J* = 7.6 Hz, 1H), 7.28 (d, *J* = 6.6 Hz, 1H), 7.23 (d, *J* = 8.5 Hz, 2H), 7.18 (d, *J* = 7.1 Hz, 1H), 6.75 (t, *J* = 7.5 Hz, 1H), 6.15 (d, *J* = 7.0 Hz, 1H). ¹³C NMR (126 MHz, DMSO) δ 169.46, 148.62, 134.51, 134.11, 133.80, 130.98, 128.32, 127.35, 125.97, 125.83, 116.02, 113.12, 110.99, 77.84, 57.25, 27.10. Anal. Calcd for C₁₆H₁₅NO₃: C, 71.36; H, 5.61. Found: C, 71.77; H, 5.41. MS (ESI) *m/z*: 269.



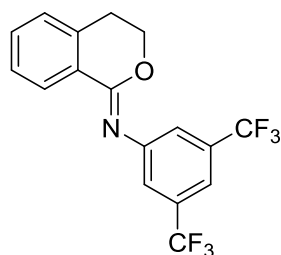
Chemical Formula: C₁₅H₁₃NO
 Exact Mass: 223.10
 Elemental Analysis: C, 80.69; H, 5.87; N, 6.27; O, 7.17

(Z)-N-phenylisochroman-1-imine 4a ¹H NMR (500 MHz, CDCl₃) δ 8.30 (d, *J* = 7.8 Hz, 1H), 7.45 (td, *J* = 7.5, 1.1 Hz, 1H), 7.39 (t, *J* = 7.6 Hz, 1H), 7.35 (t, *J* = 7.8 Hz, 2H), 7.24 (d, *J* = 7.4 Hz, 1H), 7.13 (d, *J* = 7.4 Hz, 2H), 7.09 (t, *J* = 7.4 Hz, 1H), 4.36 (t, *J* = 5.8 Hz, 2H), 3.03 (t, *J* = 5.7 Hz, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 151.65, 146.28, 140.18, 136.01, 130.17, 127.64, 127.54, 126.37, 122.42, 121.63, 65.51, 27.54. Anal. Calcd for C₁₆H₁₆N₂O₂: C, 80.69; H, 5.87. Found: C, 80.49; H, 5.61. MS (ESI) *m/z*: 223.



Chemical Formula: C₁₅H₁₂N₂O₃
 Exact Mass: 268.08
 Elemental Analysis: C, 67.16; H, 4.51; N, 10.44; O, 17.89

(Z)-N-(4-nitrophenyl)isochroman-1-imine 4b ¹H NMR (500 MHz, CDCl₃) δ 8.25 (d, *J* = 7.8 Hz, 1H), 8.21 (d, *J* = 8.9 Hz, 2H), 7.50 (t, *J* = 7.5 Hz, 1H), 7.41 (t, *J* = 7.6 Hz, 1H), 7.25 (s, 1H), 7.16 (d, *J* = 8.9 Hz, 2H), 4.38 (t, *J* = 5.8 Hz, 2H), 3.06 (t, *J* = 5.8 Hz, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 153.37, 152.95, 142.60, 136.27, 130.93, 127.86, 126.57, 126.13, 123.50, 121.97, 65.95, 27.30. Anal. Calcd for C₁₆H₁₆N₂O₂: C, 67.16; H, 4.51. Found: C, 66.88; H, 4.66. MS (ESI) *m/z*: 268.



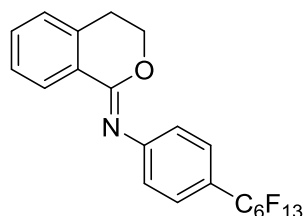
Chemical Formula: C₁₇H₁₁F₆NO

Exact Mass: 359.07

Elemental Analysis: C, 56.83; H, 3.09; F, 31.73; N, 3.90; O, 4.45

(Z)-N-(3,5-bis(trifluoromethyl)phenyl)isochroman-1-imine **4c**

¹H NMR (500 MHz, CDCl₃) δ 8.25 (d, *J* = 7.8 Hz, 1H), 7.55 (d, *J* = 3.8 Hz, 3H), 7.49 (t, *J* = 7.4 Hz, 1H), 7.40 (t, *J* = 7.6 Hz, 1H), 7.25 (s, 1H), 4.39 (t, *J* = 5.8 Hz, 2H), 3.06 (t, *J* = 5.8 Hz, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 154.65, 148.90, 137.25, 132.07, 131.92, 131.66, 128.87, 127.67, 127.22, 124.71, 123.36, 122.54, 116.87, 67.03, 28.38. ¹⁹F NMR (470 MHz, CDCl₃) δ -62.82. Anal. Calcd for C₁₇H₁₁F₆NO: C, 56.83; H, 3.09. Found: C, 57.04; H, 3.22. MS (ESI) *m/z*: 359.



Chemical Formula: C₂₁H₁₂F₁₃NO

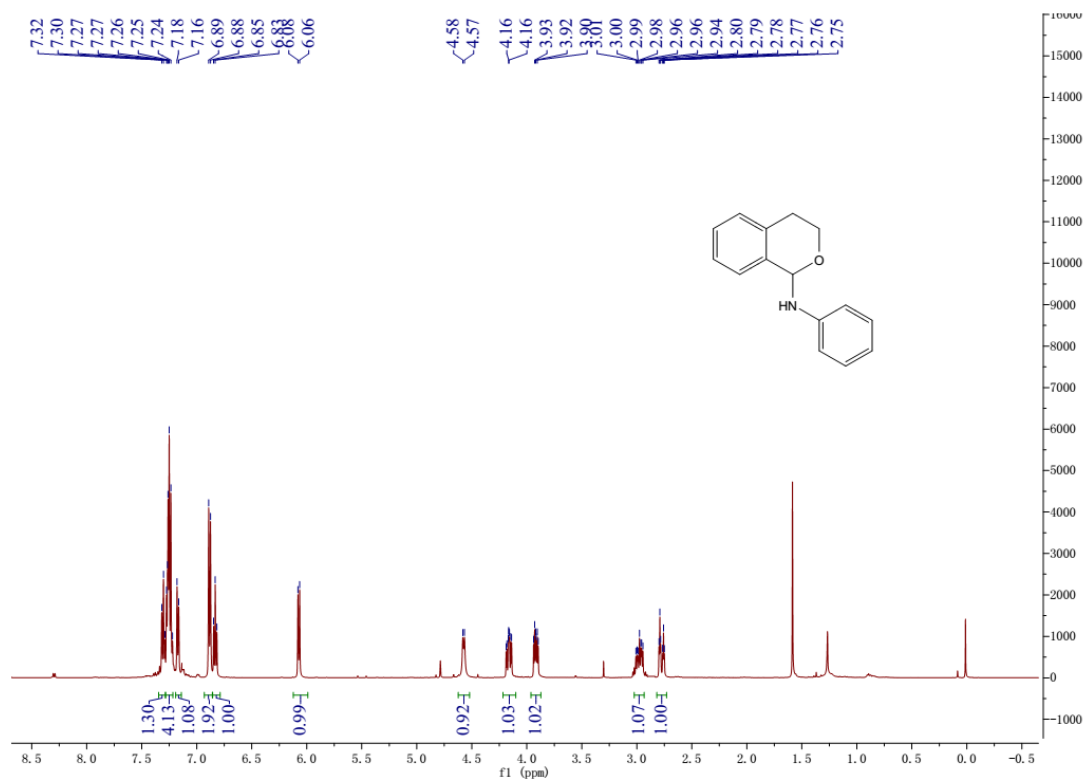
Exact Mass: 541.07

Elemental Analysis: C, 46.60; H, 2.23; F, 45.63; N, 2.59; O, 2.96

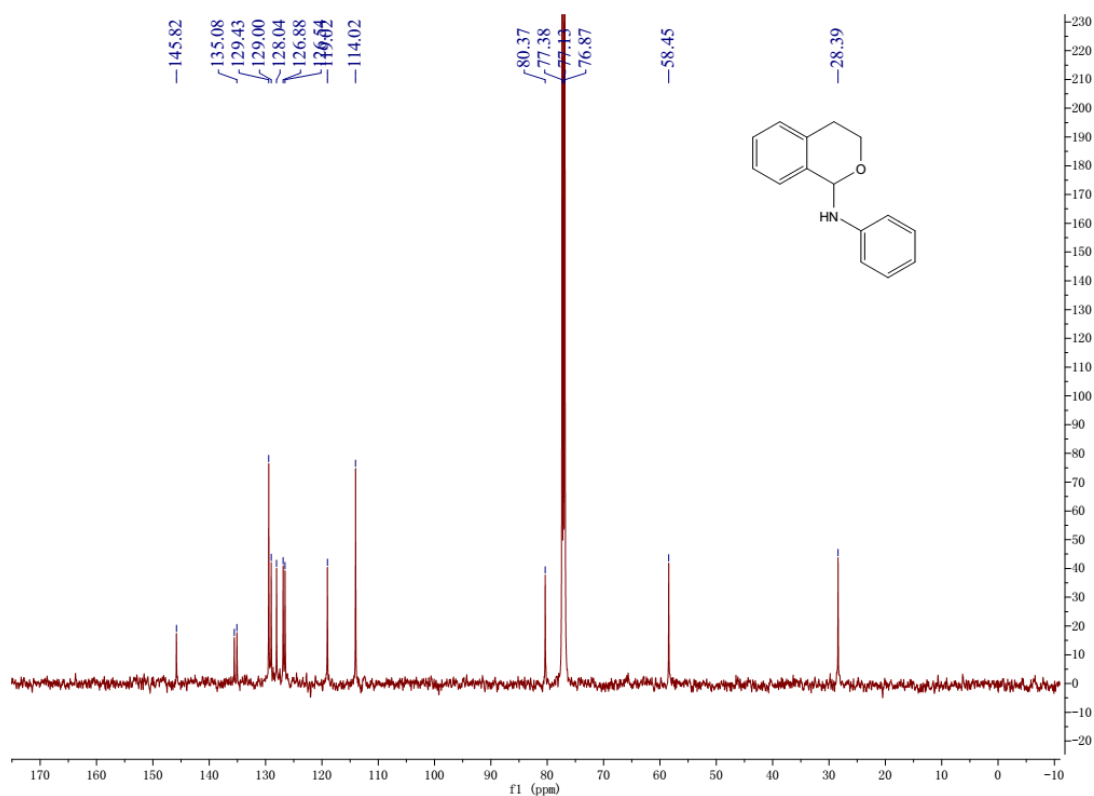
(Z)-N-(4-(6,6,6,6,6,6,6,6,6,6,6,6,6,6-tridecafluoro-6H-hexa-1,3,5-triyn-1-yl)phenyl)isochroman-1-imine **4e**

¹H NMR (500 MHz, CDCl₃) δ 8.22 (d, *J* = 7.8 Hz, 1H), 7.48 (d, *J* = 8.3 Hz, 2H), 7.42 (t, *J* = 7.3 Hz, 1H), 7.34 (t, *J* = 7.6 Hz, 1H), 7.20 (d, *J* = 7.6 Hz, 1H), 7.14 (d, *J* = 8.3 Hz, 2H), 4.32 (s, 2H), 3.00 (s, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 153.56, 151.15, 137.20, 131.73, 128.85, 127.57, 127.40, 123.40, 122.80, 116.38, 66.83, 28.48. ¹⁹F NMR (470 MHz, CDCl₃) δ -80.72, -80.74, -80.76, -109.91, -109.94, -109.97, -121.24, -121.79, -121.88, -122.69, -126.09. Anal. Calcd for C₁₆H₁₆N₂O₂: C, 46.60; H, 2.23. Found: C, 46.93; H, 2.00. MS (ESI) *m/z*: 541.

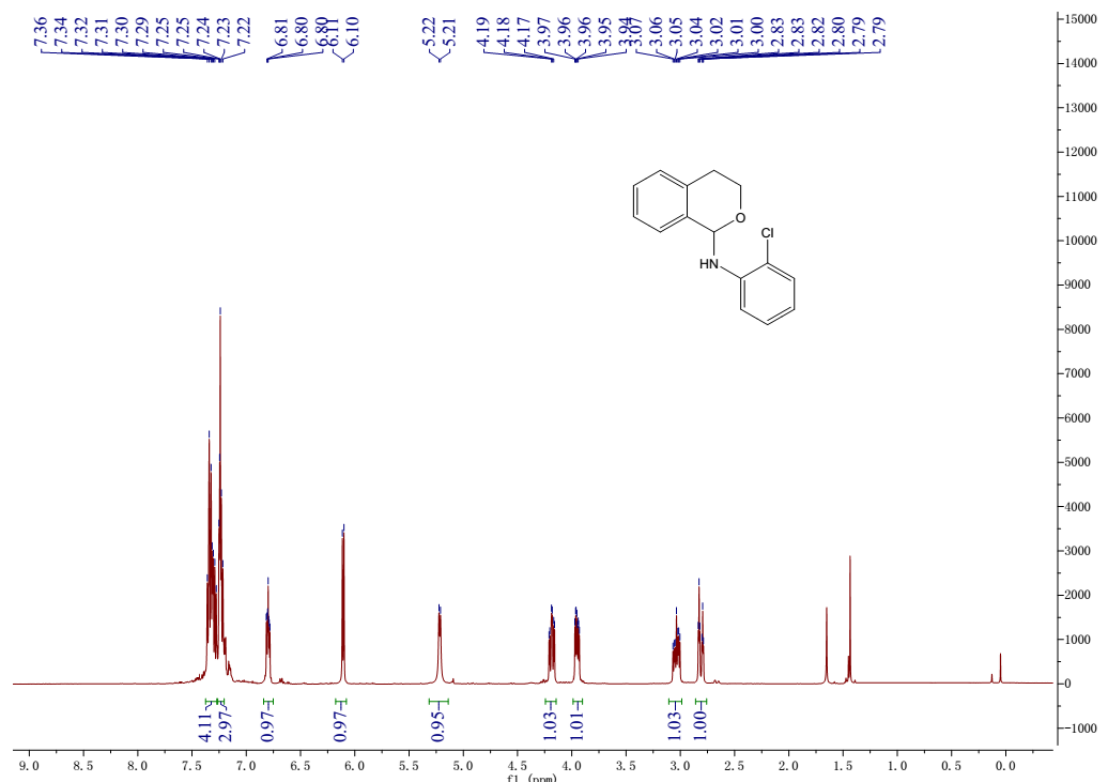
4 NMR Spectra of All Products



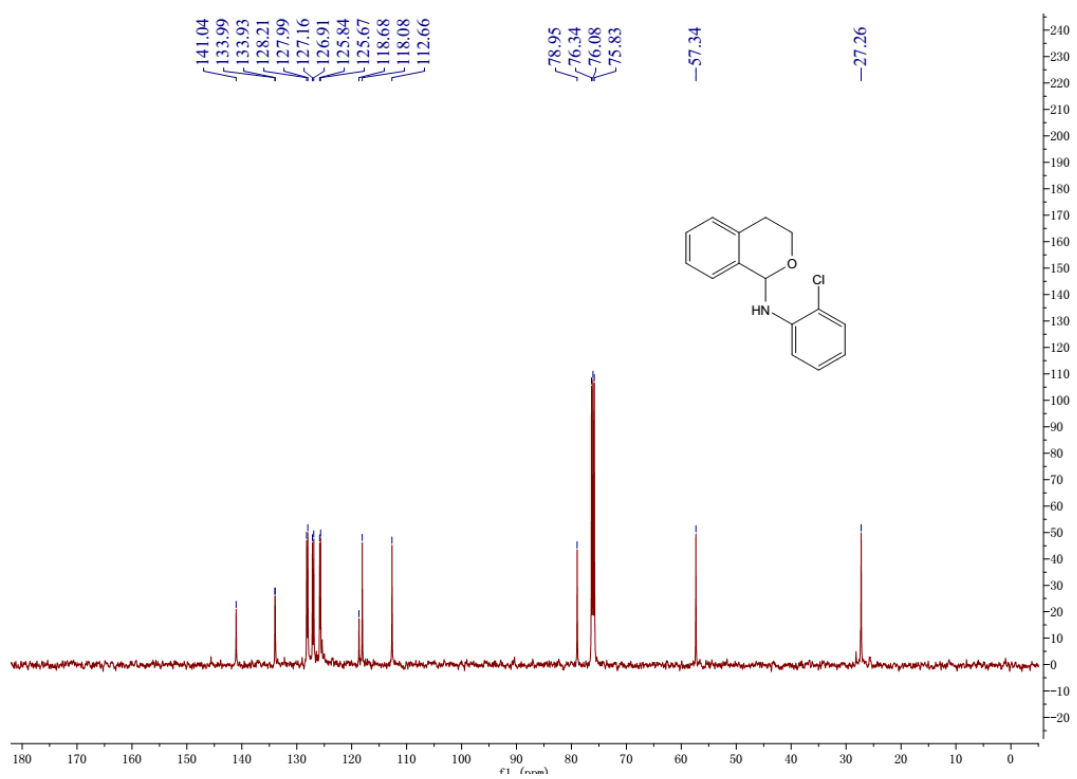
¹H NMR spectrum (500 MHz, CDCl₃) of **3a**



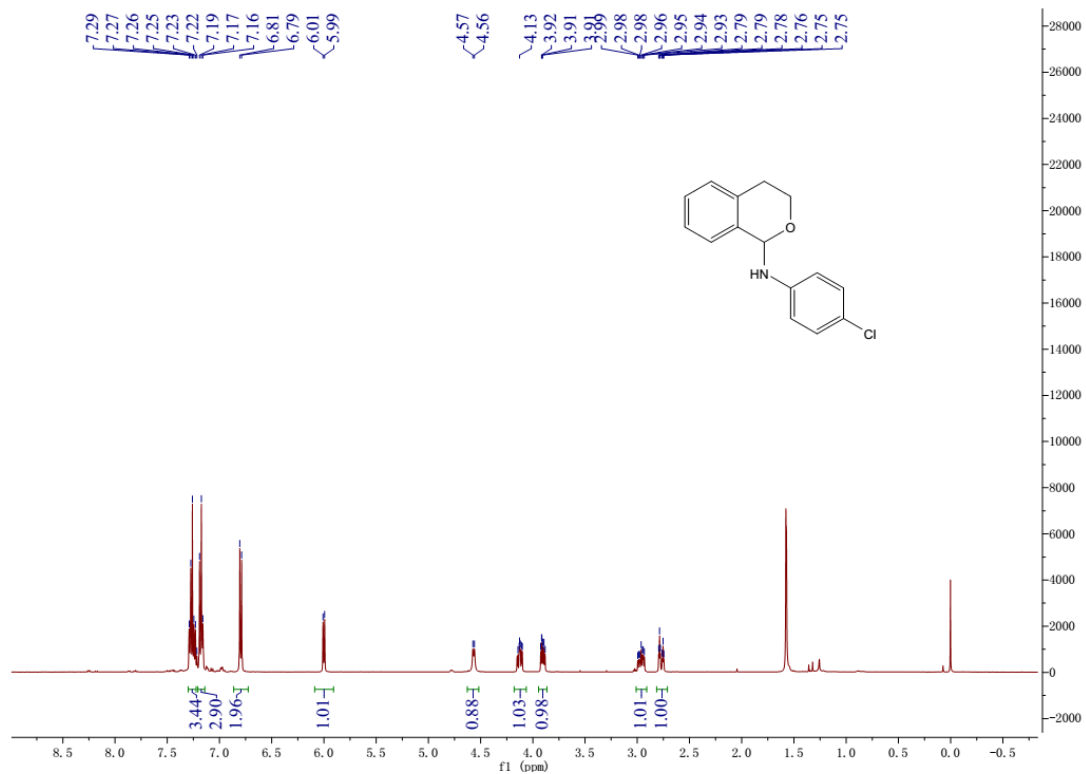
¹³C NMR spectrum (125 MHz, CDCl₃) of **3a**



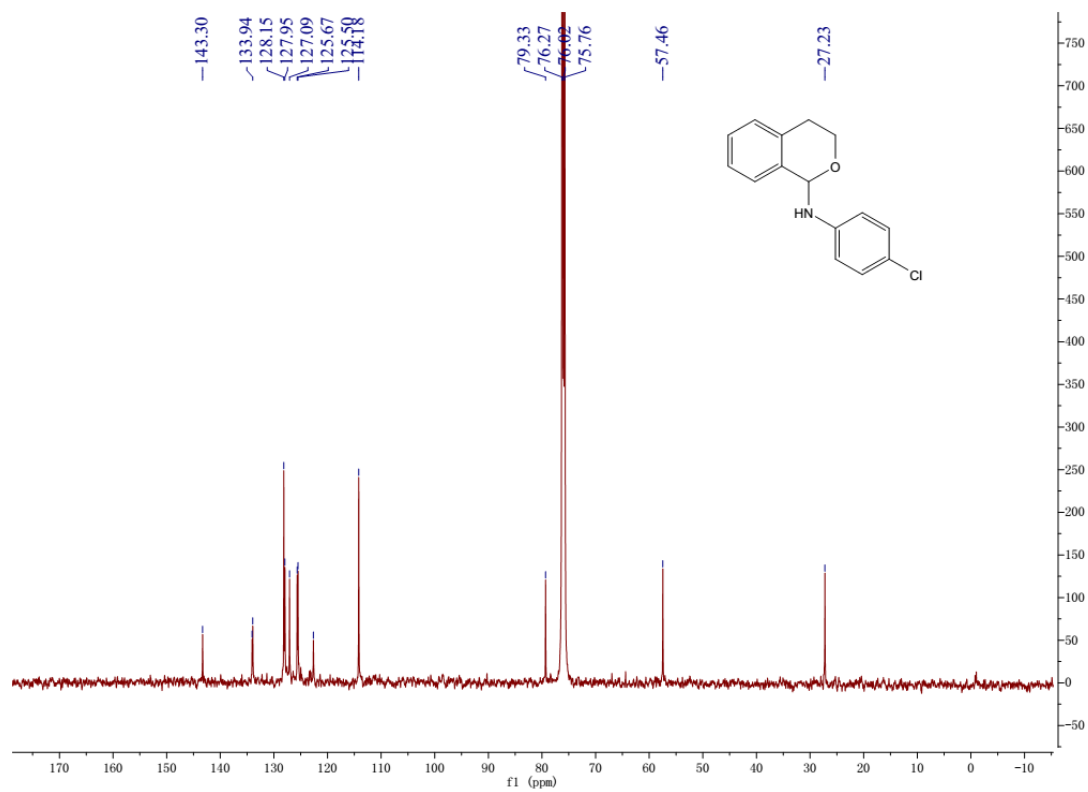
¹H NMR spectrum (500 MHz, CDCl₃) of **3b**



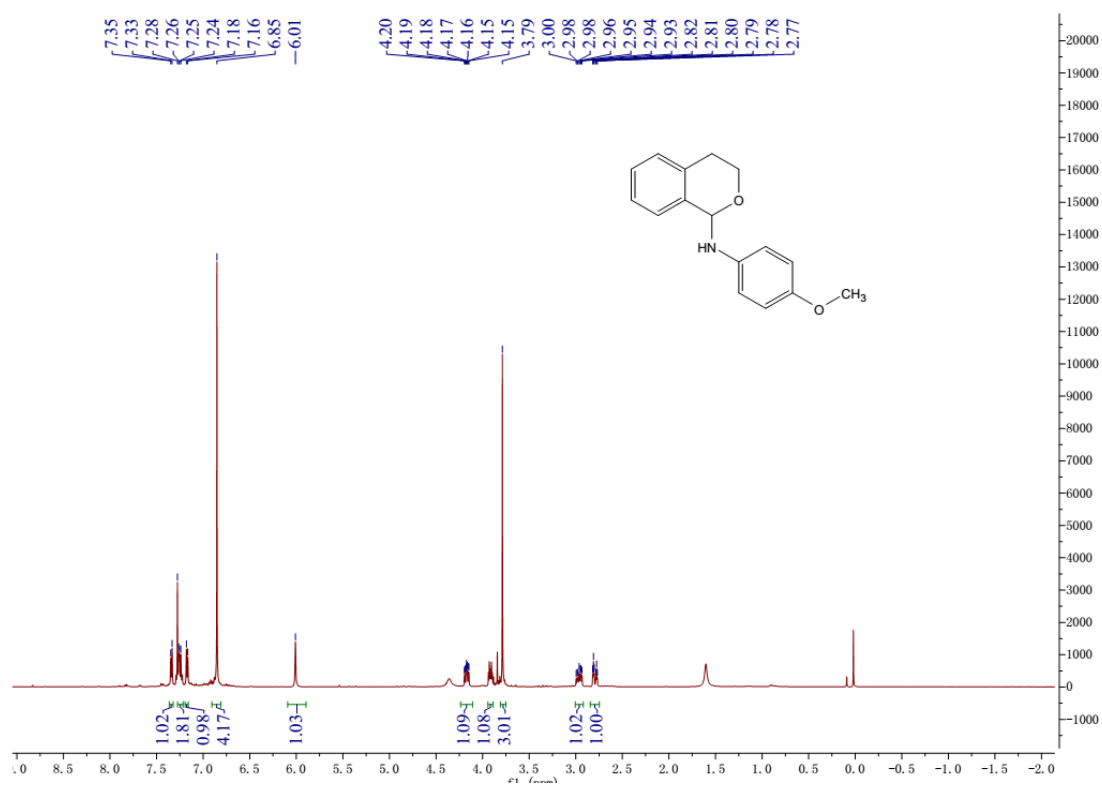
¹³C NMR spectrum (125 MHz, CDCl₃) of **3b**



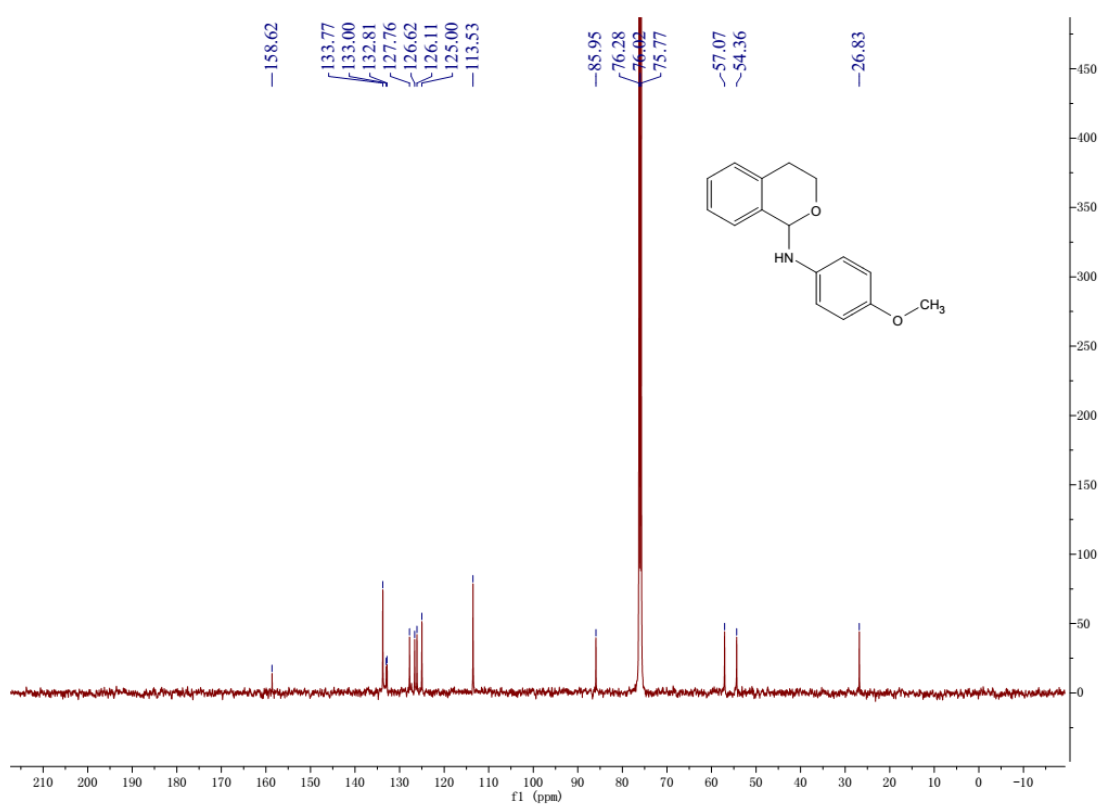
¹H NMR spectrum (500 MHz, CDCl₃) of **3c**



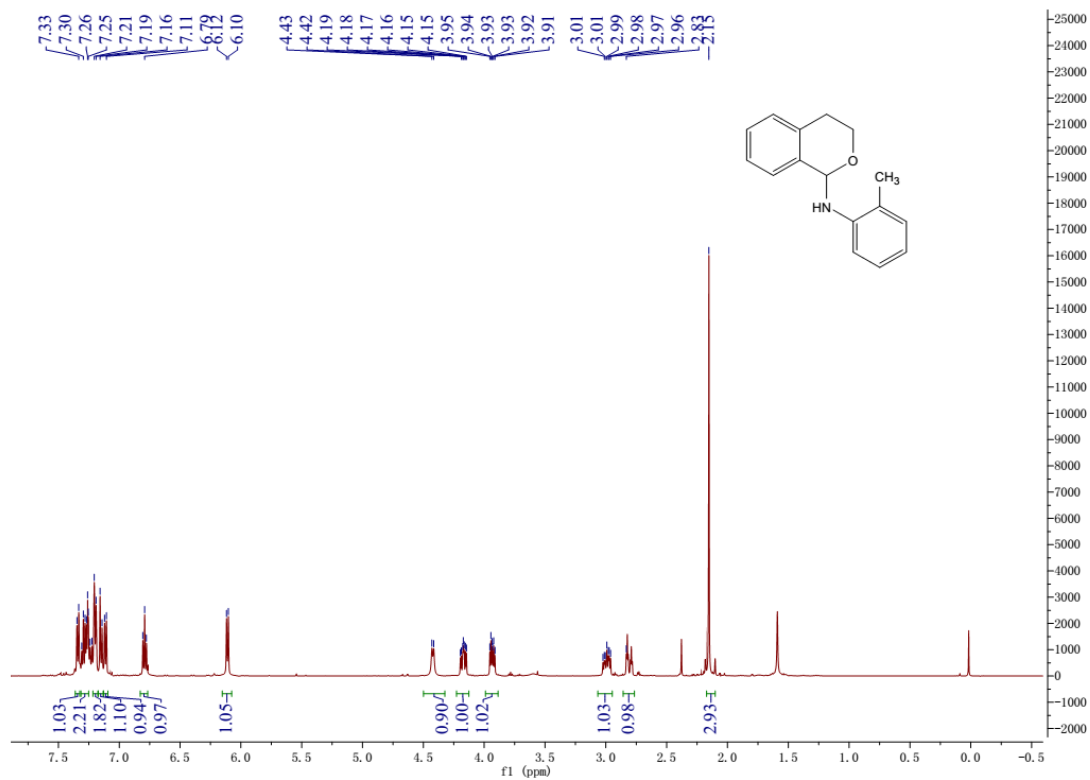
¹³C NMR spectrum (125 MHz, CDCl₃) of **3c**



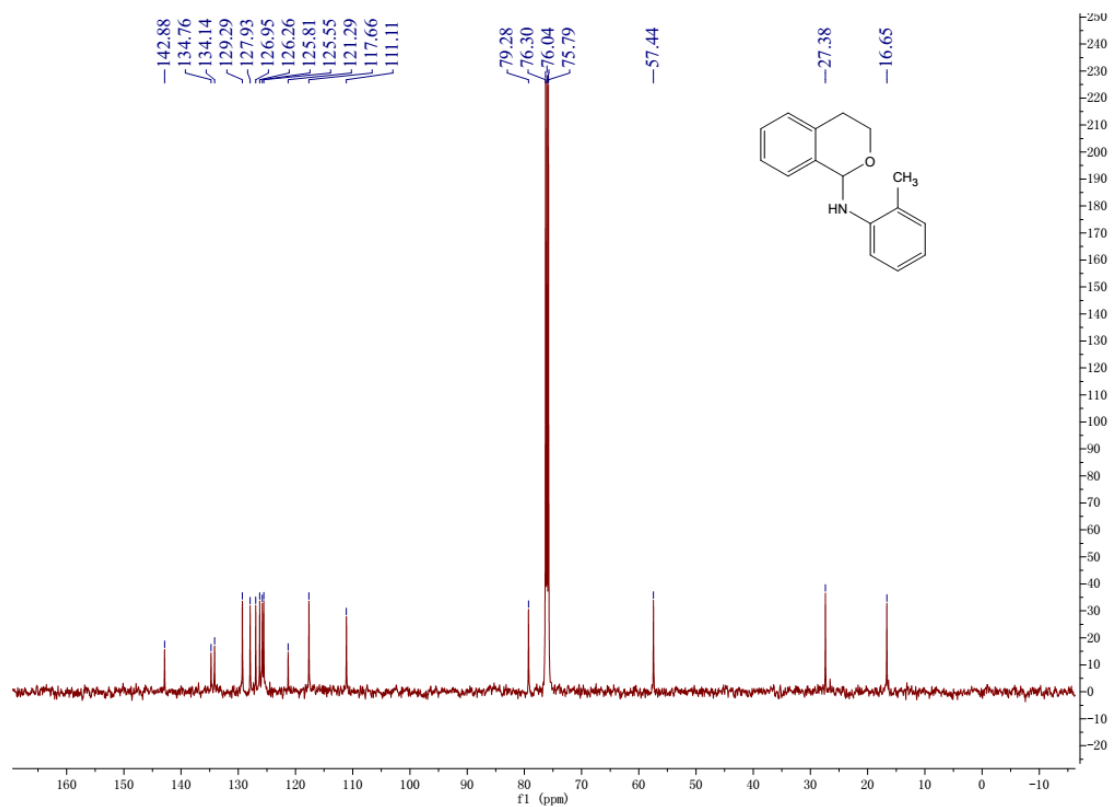
¹H NMR spectrum (500 MHz, CDCl₃) of **3d**



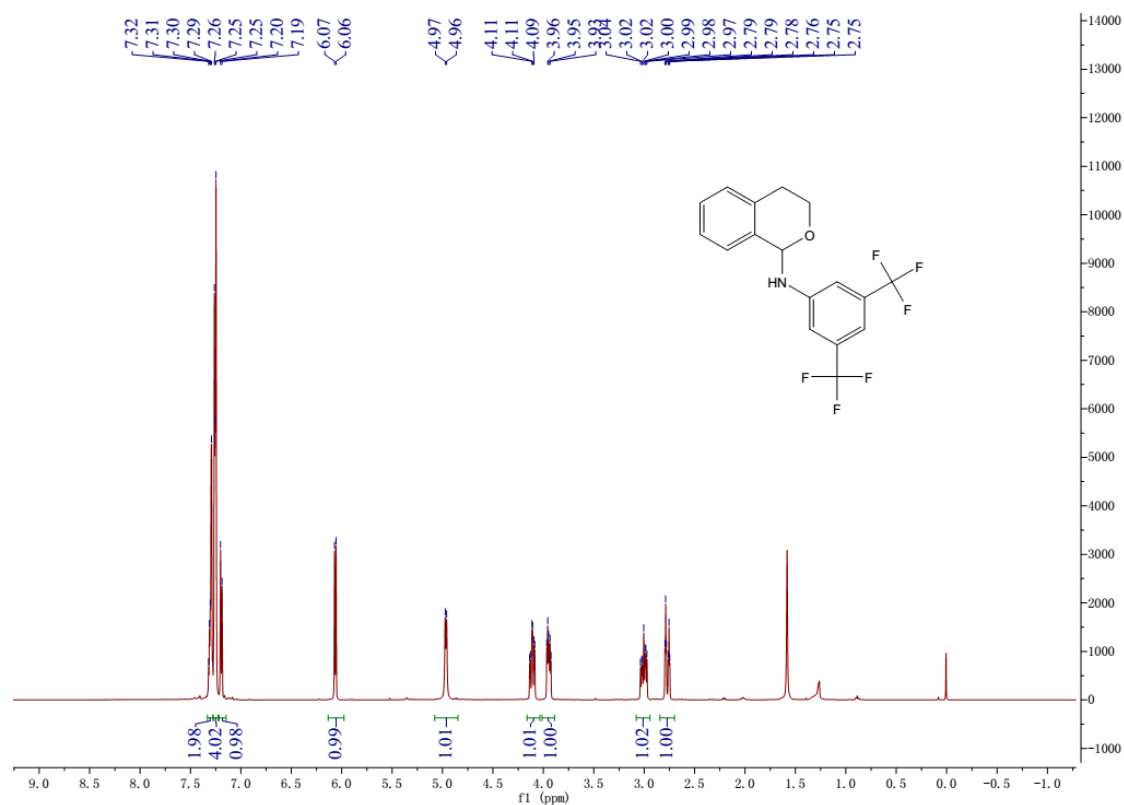
¹³C NMR spectrum (125 MHz, CDCl₃) of **3d**



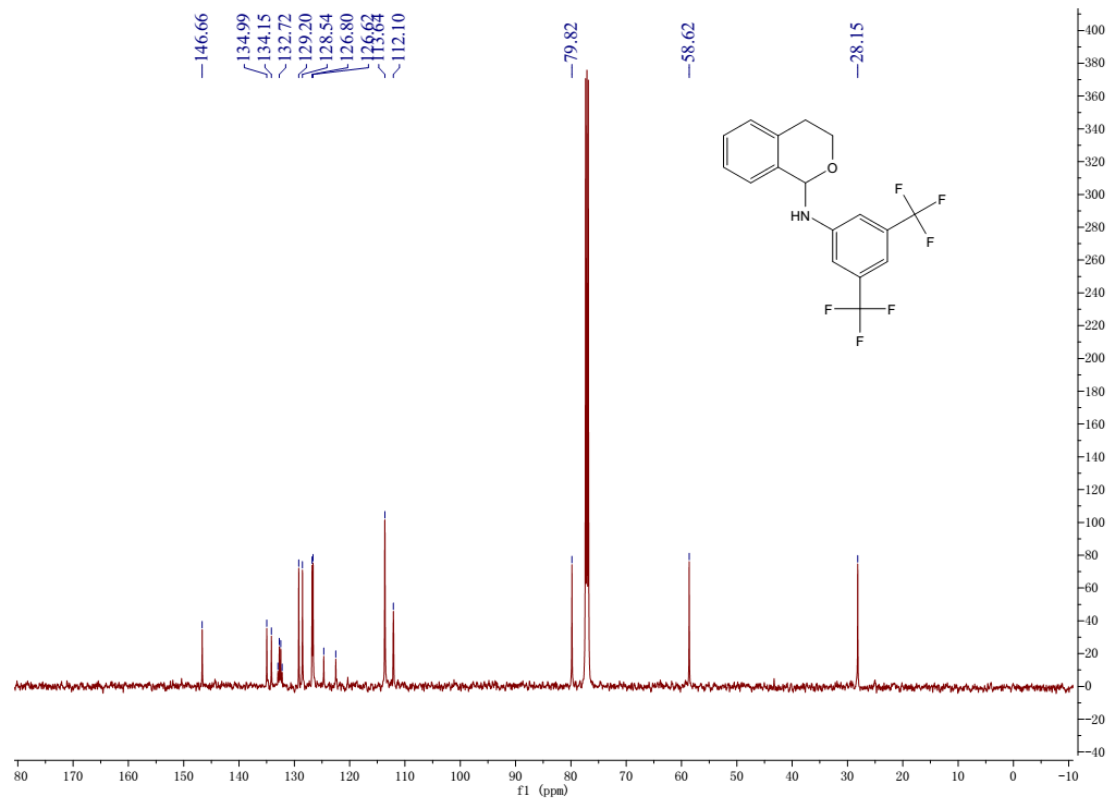
¹H NMR spectrum (500 MHz, CDCl₃) of **3e**



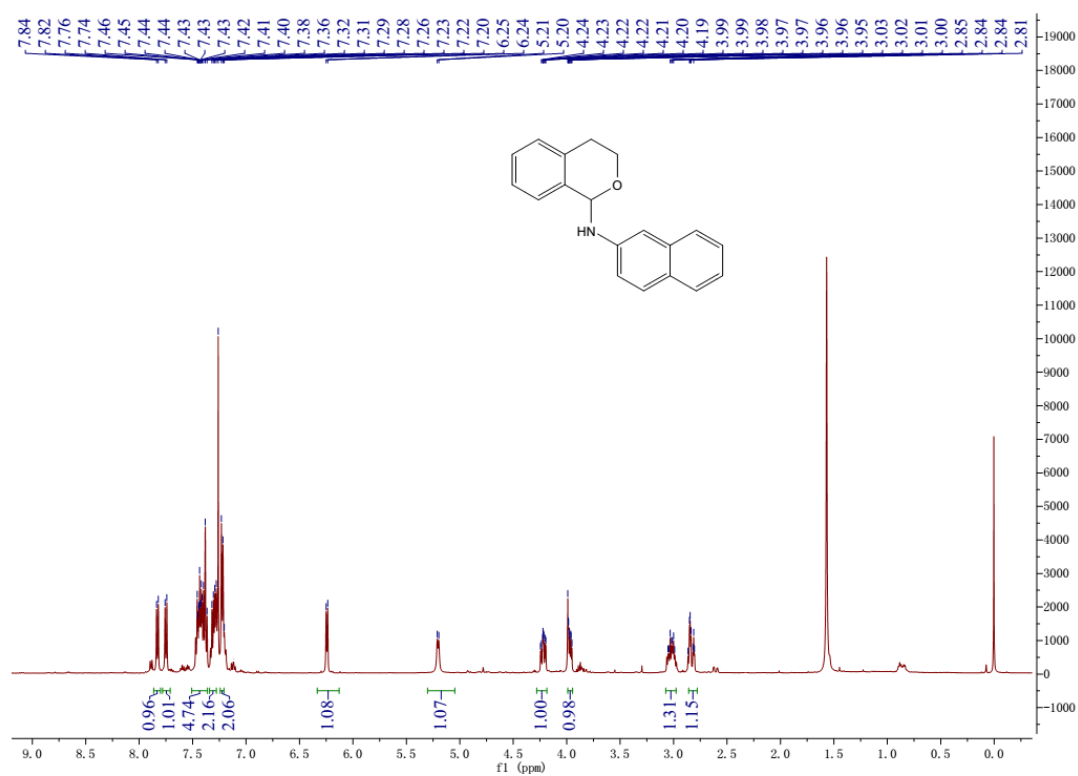
¹³C NMR spectrum (500 MHz, CDCl₃) of **3e**



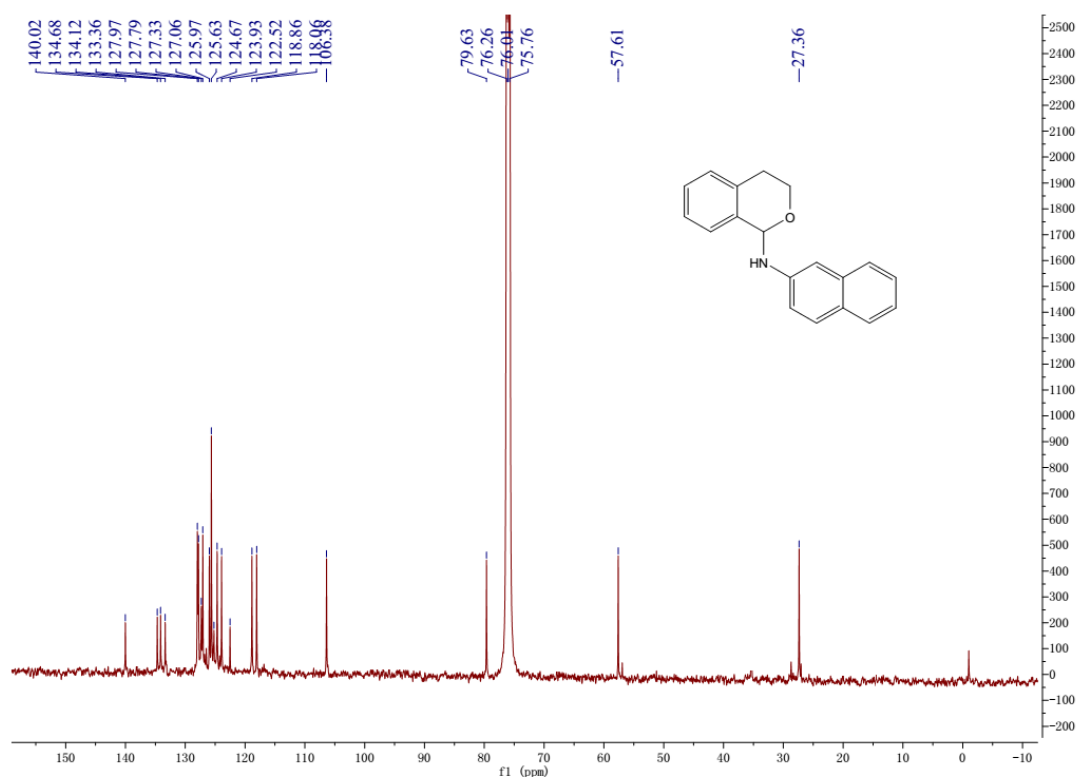
¹H NMR spectrum (500 MHz, CDCl₃) of **3f**



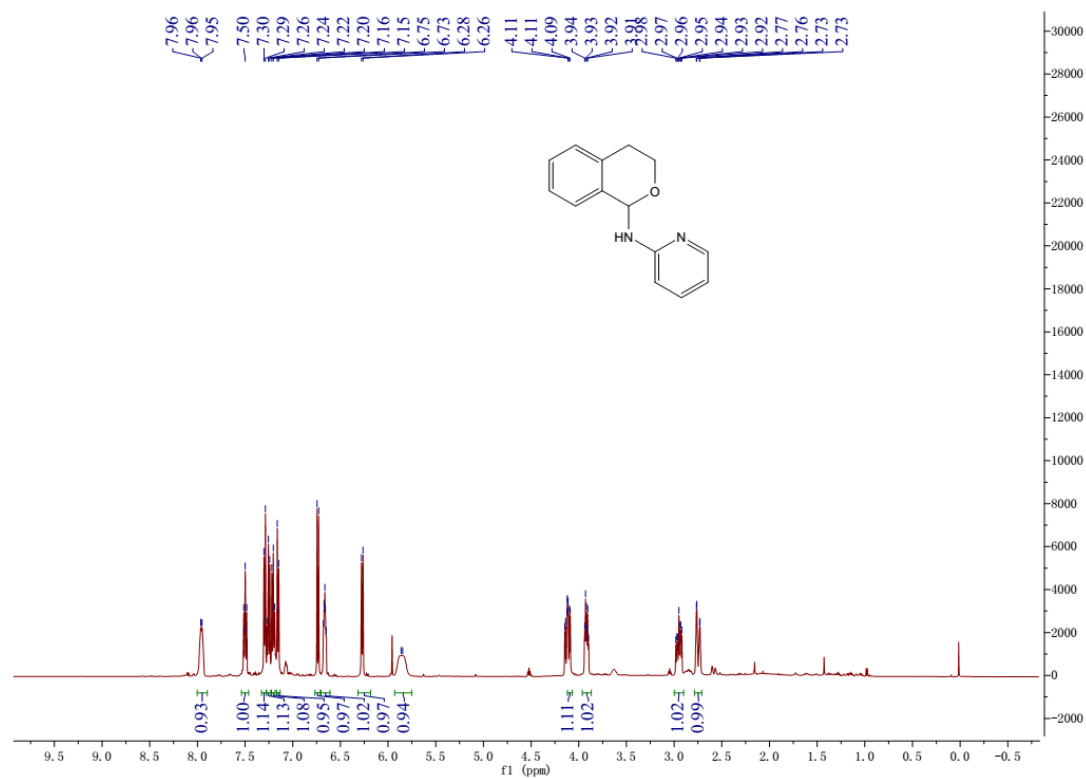
¹³C NMR spectrum (500 MHz, CDCl₃) of **3f**



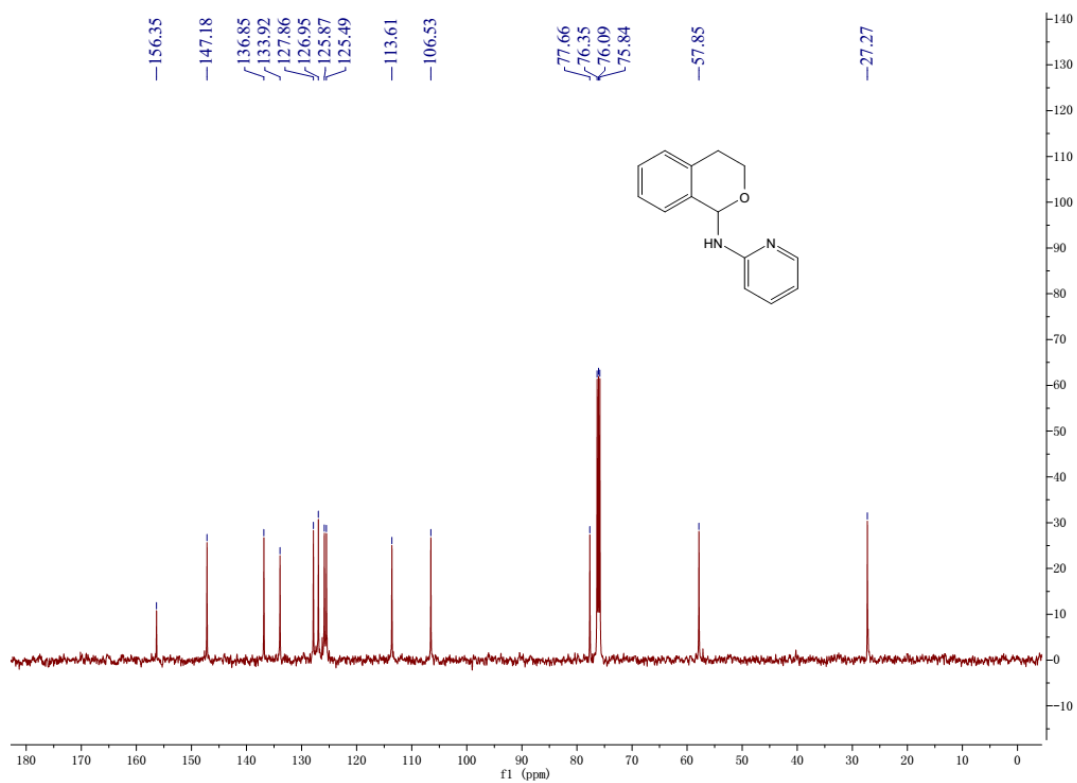
¹H NMR spectrum (500 MHz, CDCl₃) of **3g**



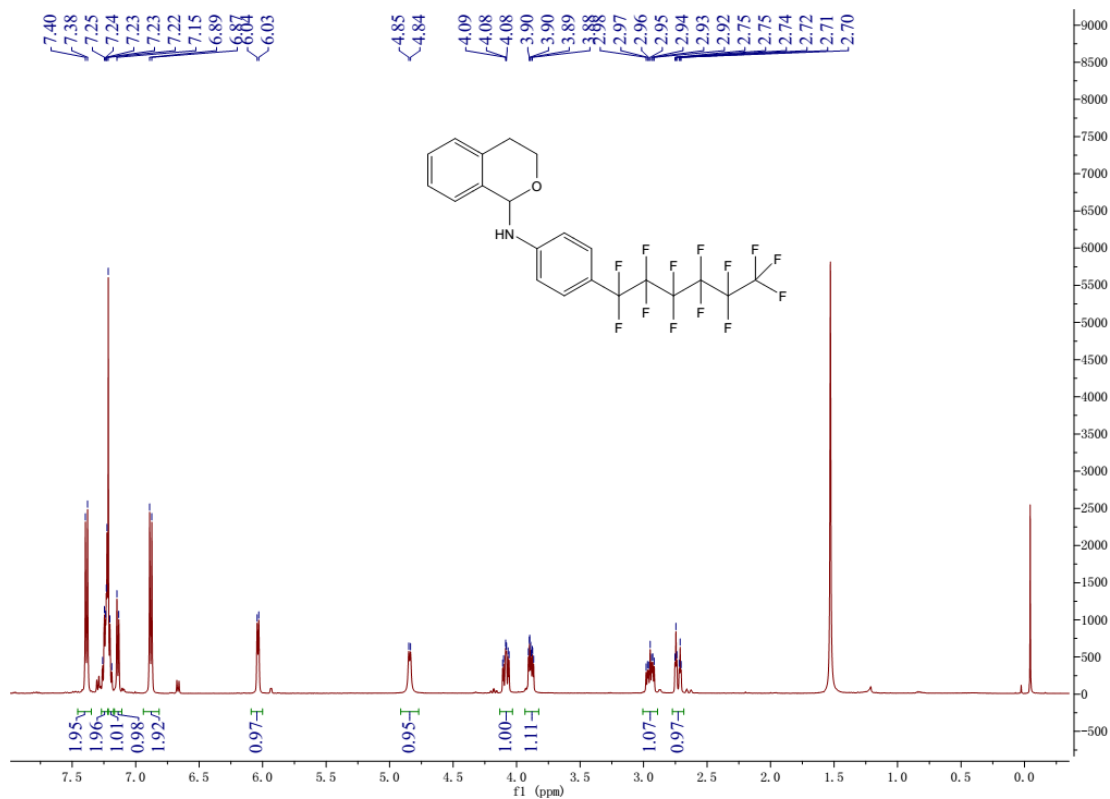
¹³C NMR spectrum (125 MHz, CDCl₃) of **3g**



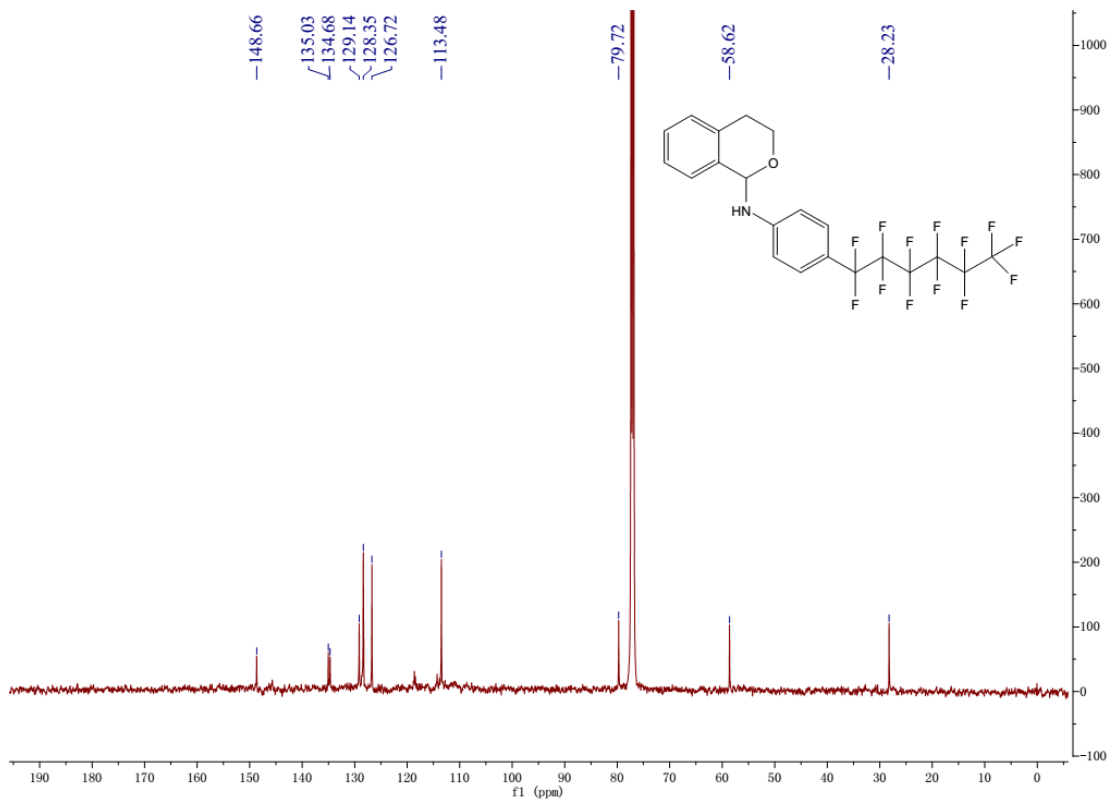
¹H NMR spectrum (500 MHz, CDCl₃) of **3h**



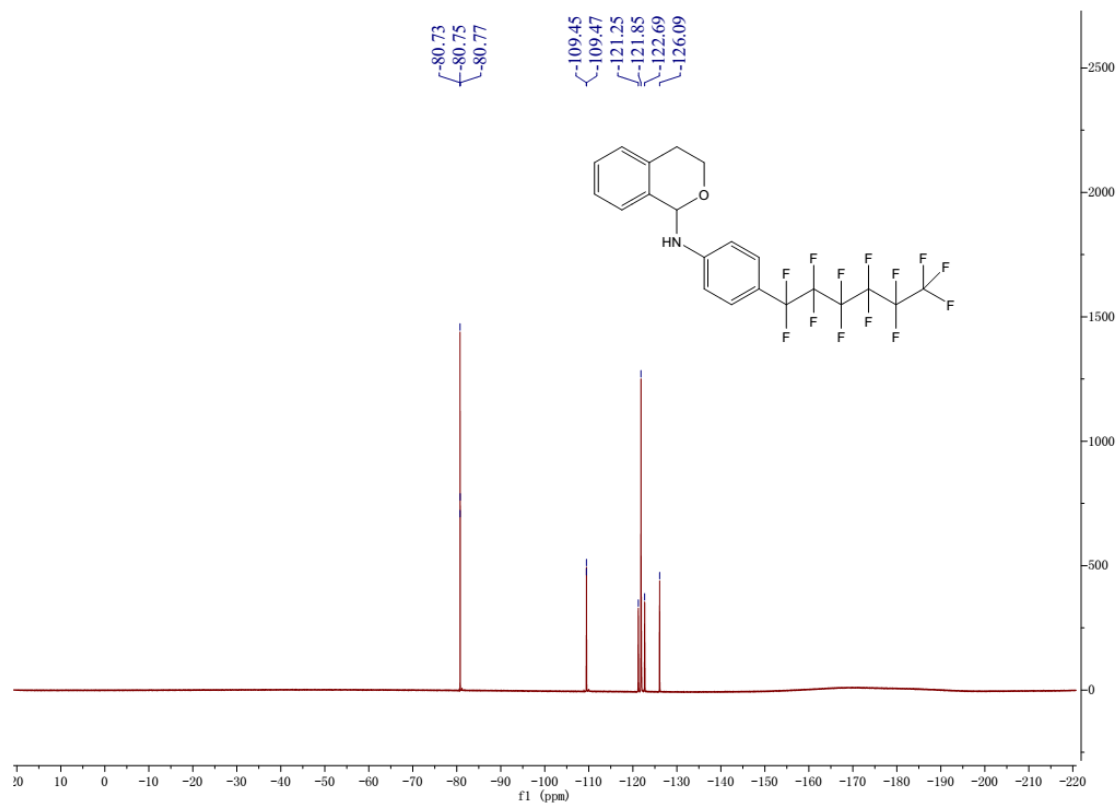
¹³C NMR spectrum (125 MHz, CDCl₃) of **3h**

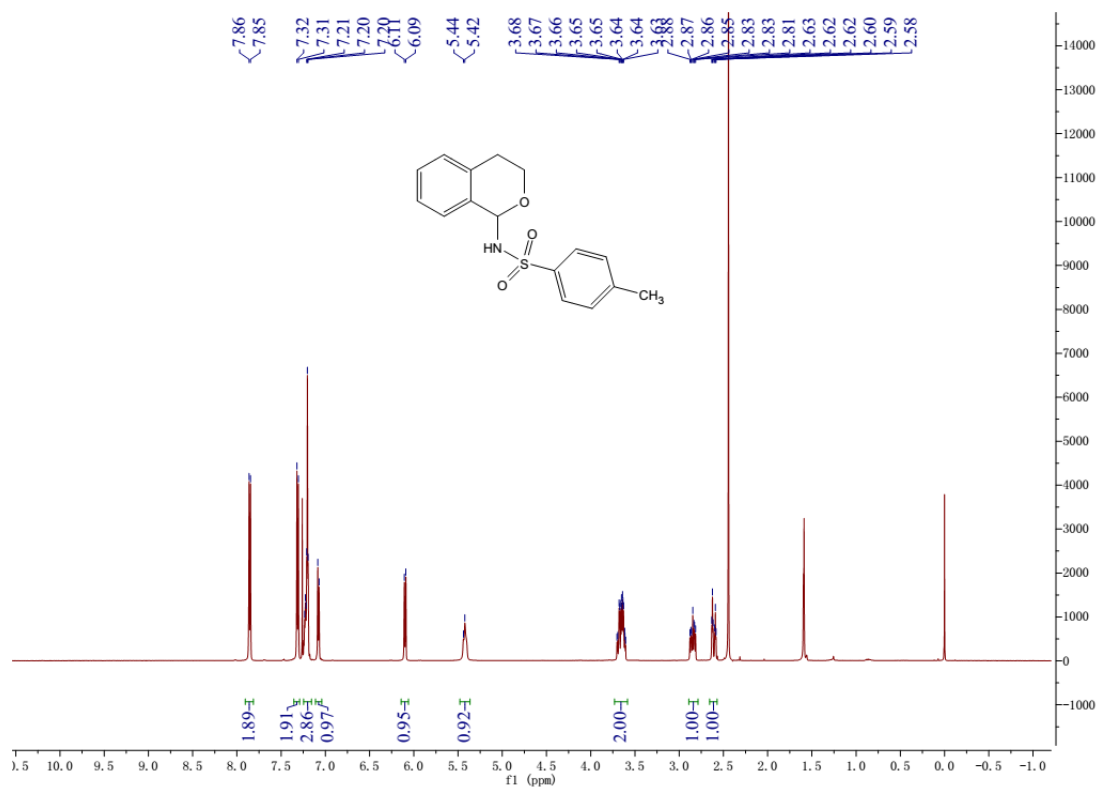


¹H NMR spectrum (500 MHz, CDCl₃) of **31**

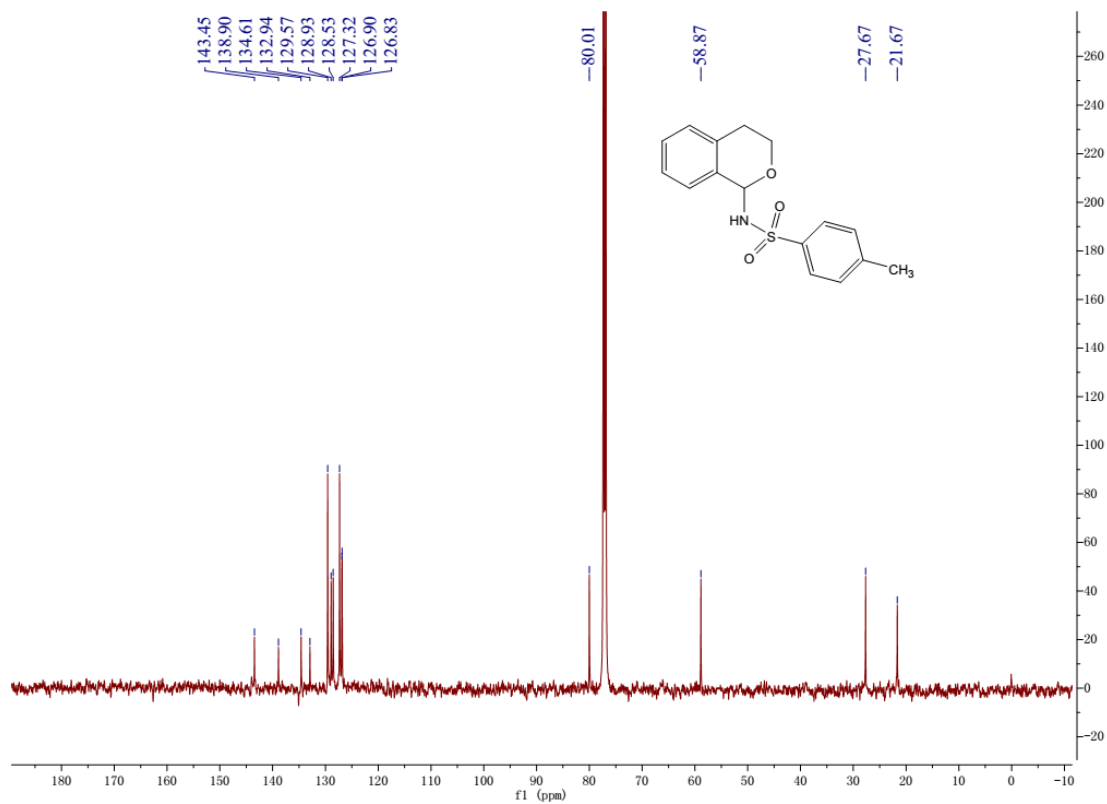


¹³C NMR spectrum (125 MHz, CDCl₃) of **31**

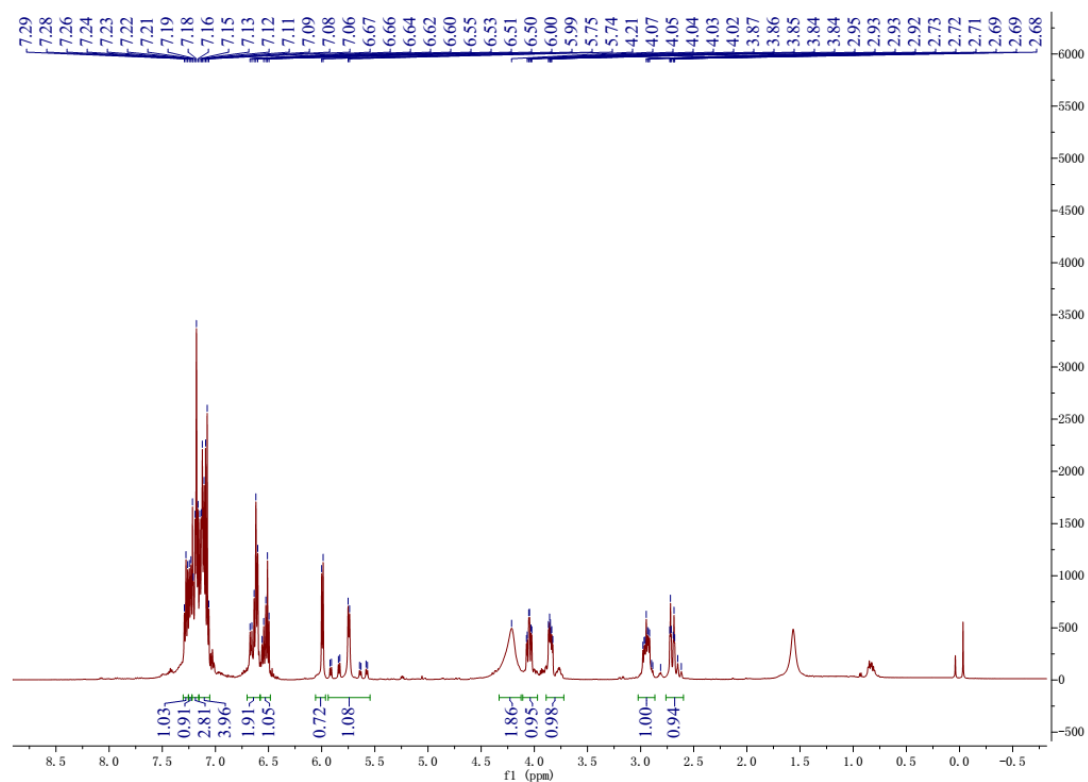


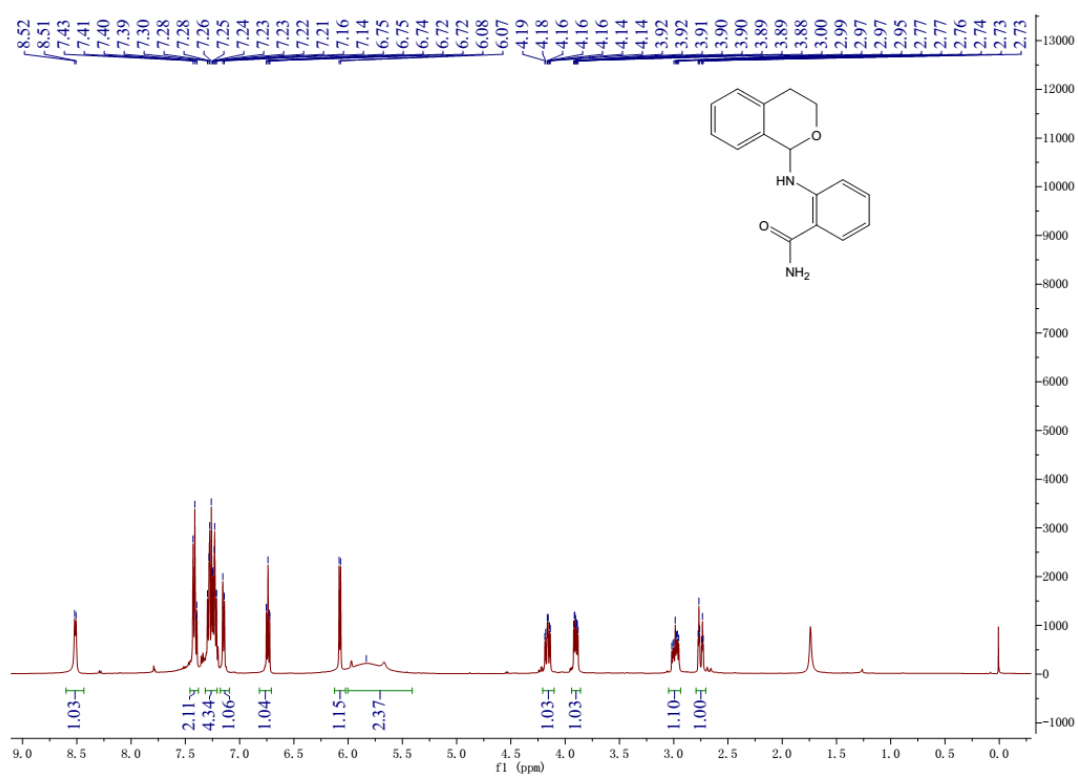


¹H NMR spectrum (500 MHz, CDCl₃) of **3n**

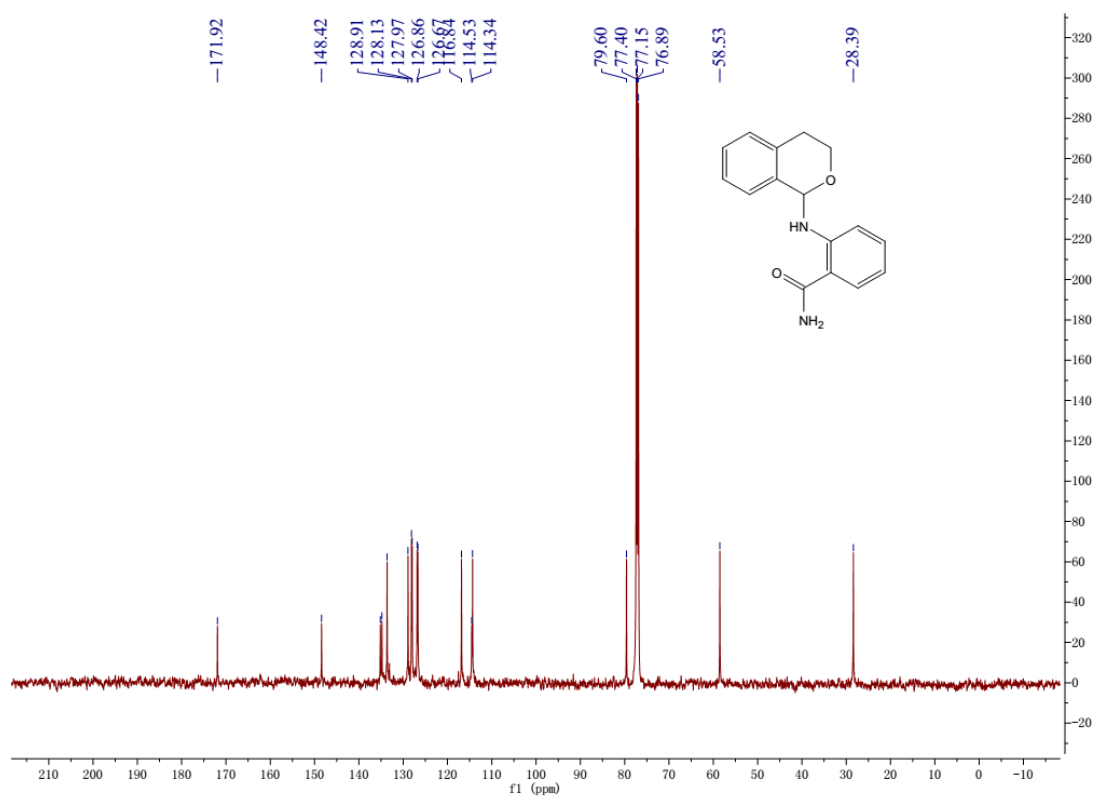


¹³C NMR spectrum (125 MHz, CDCl₃) of **3n**

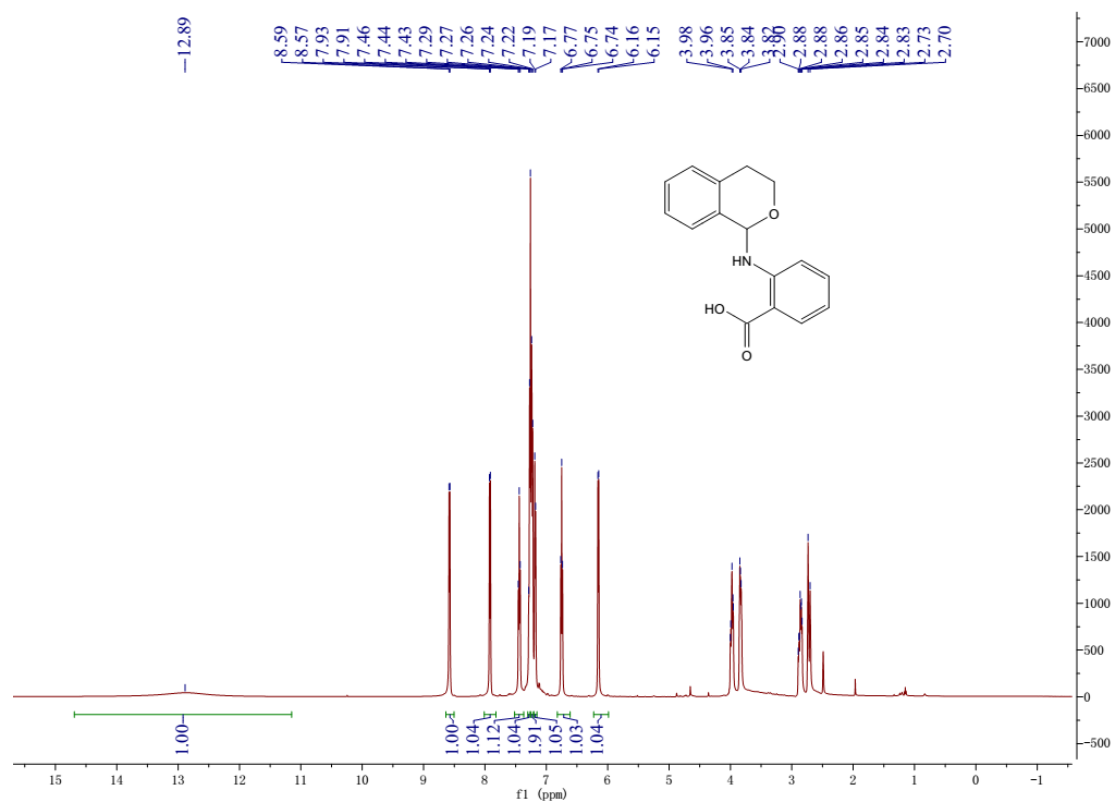
Mixture of **3u** and **3v**



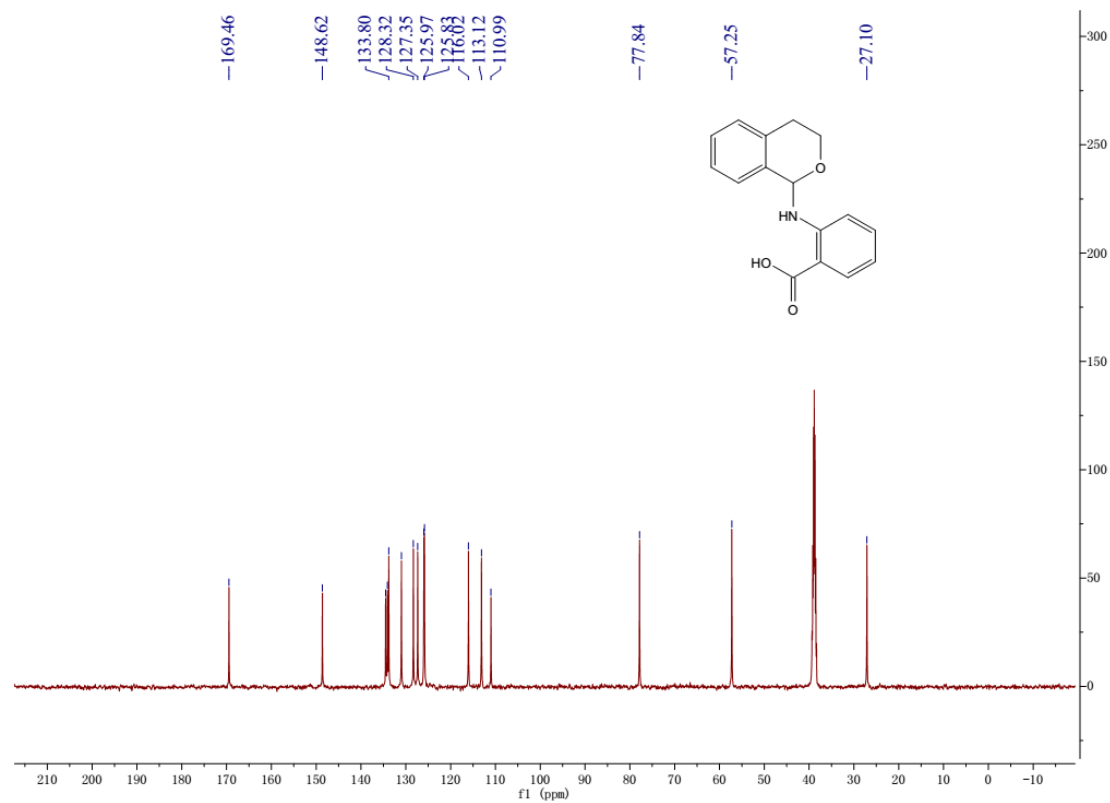
¹H NMR spectrum (500 MHz, CDCl₃) of **3t**



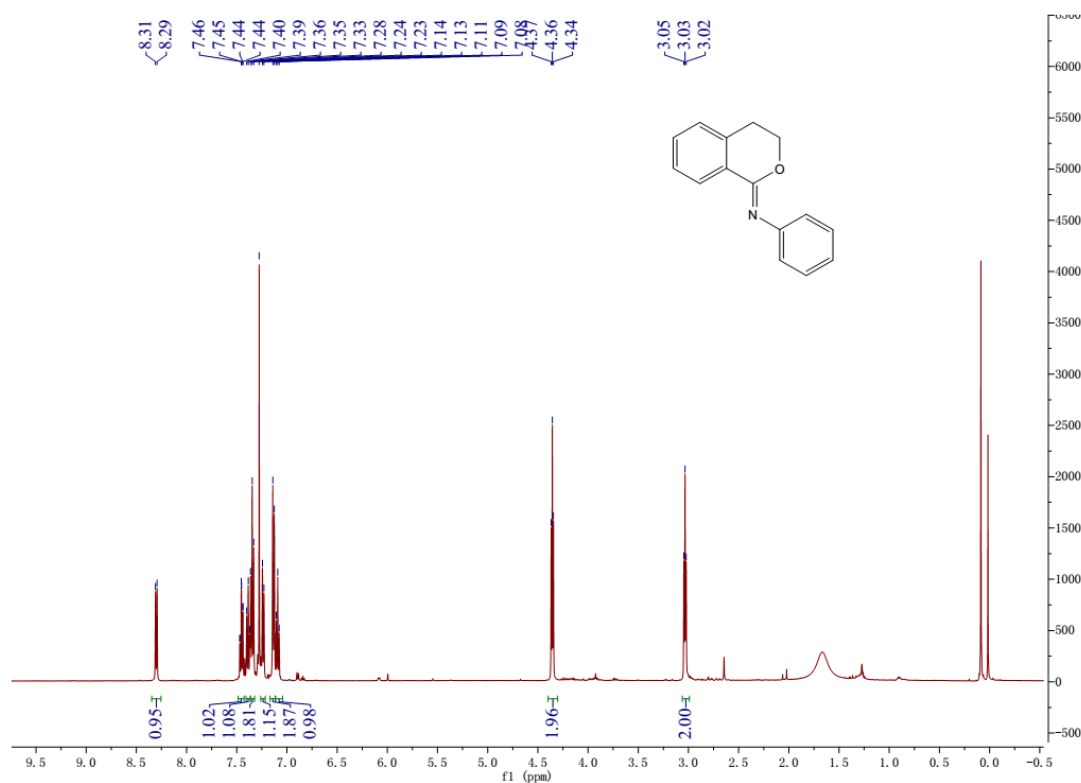
¹³C NMR spectrum (125 MHz, CDCl₃) of **3t**



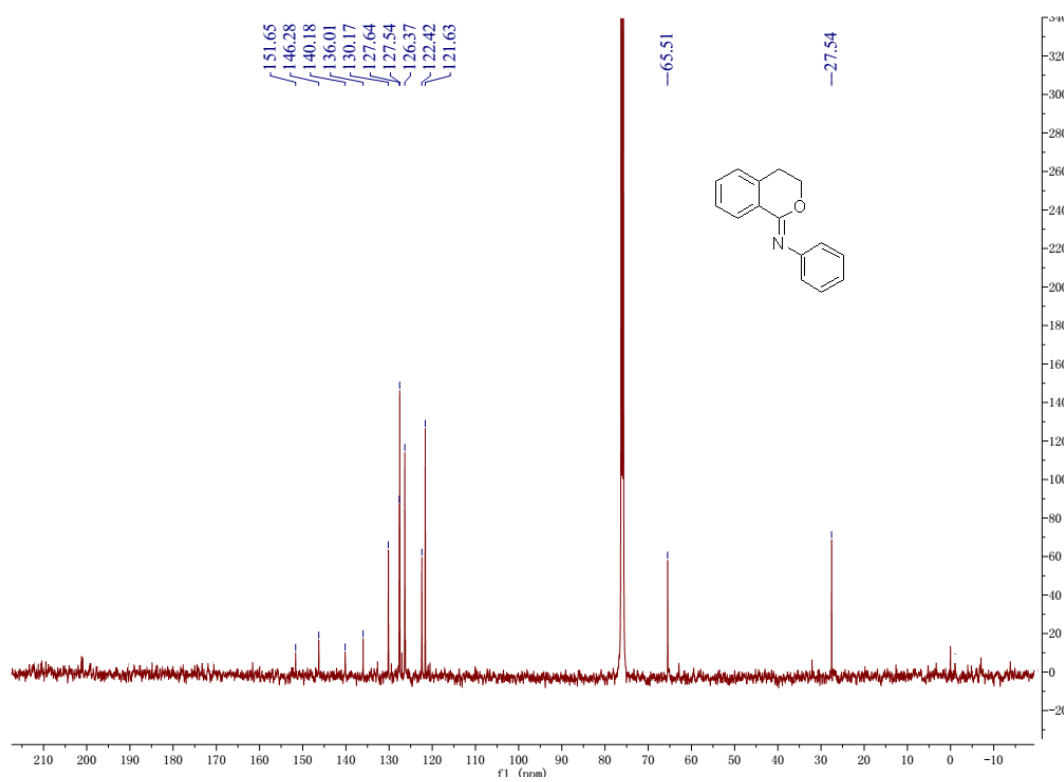
¹H NMR spectrum (500 MHz, CDCl₃) of **3x**



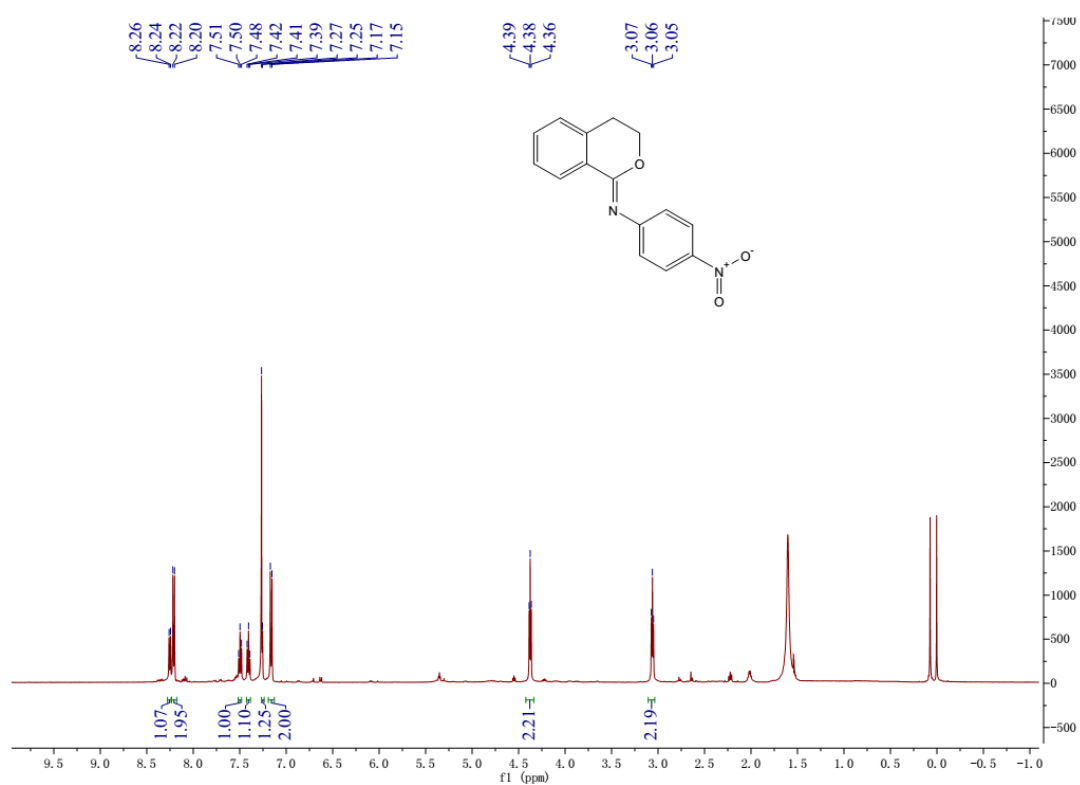
¹³C NMR spectrum (125 MHz, CDCl₃) of **3x**



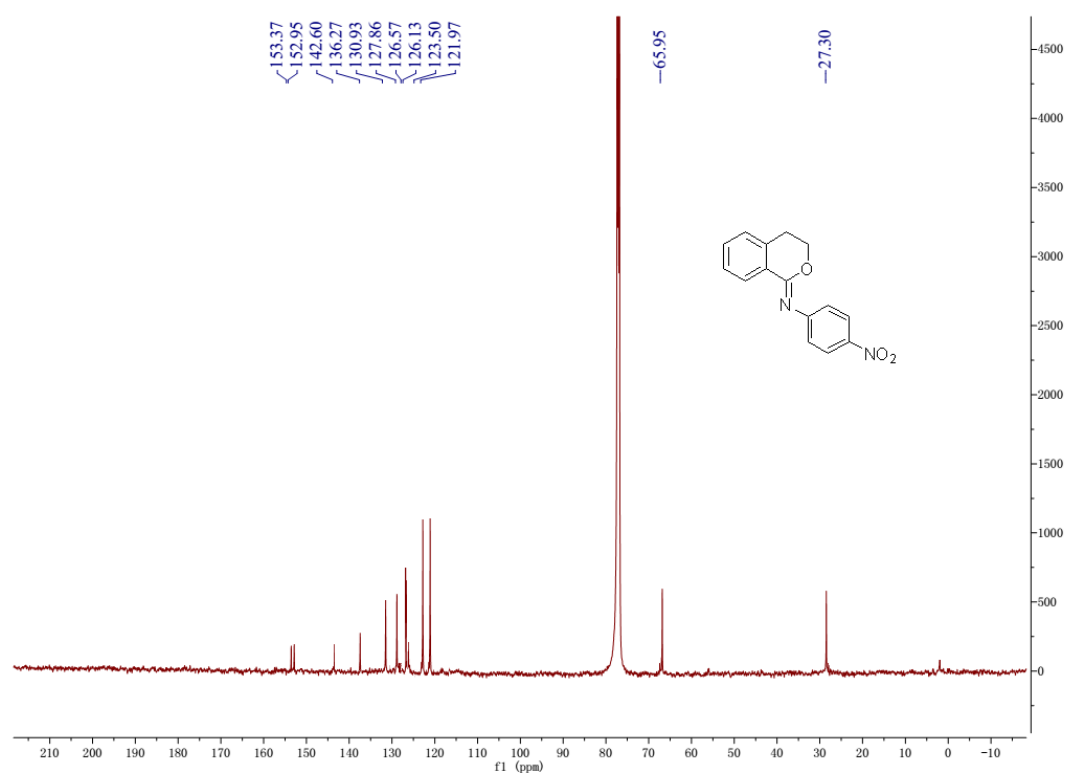
¹H NMR spectrum (500 MHz, CDCl₃) of **4a**



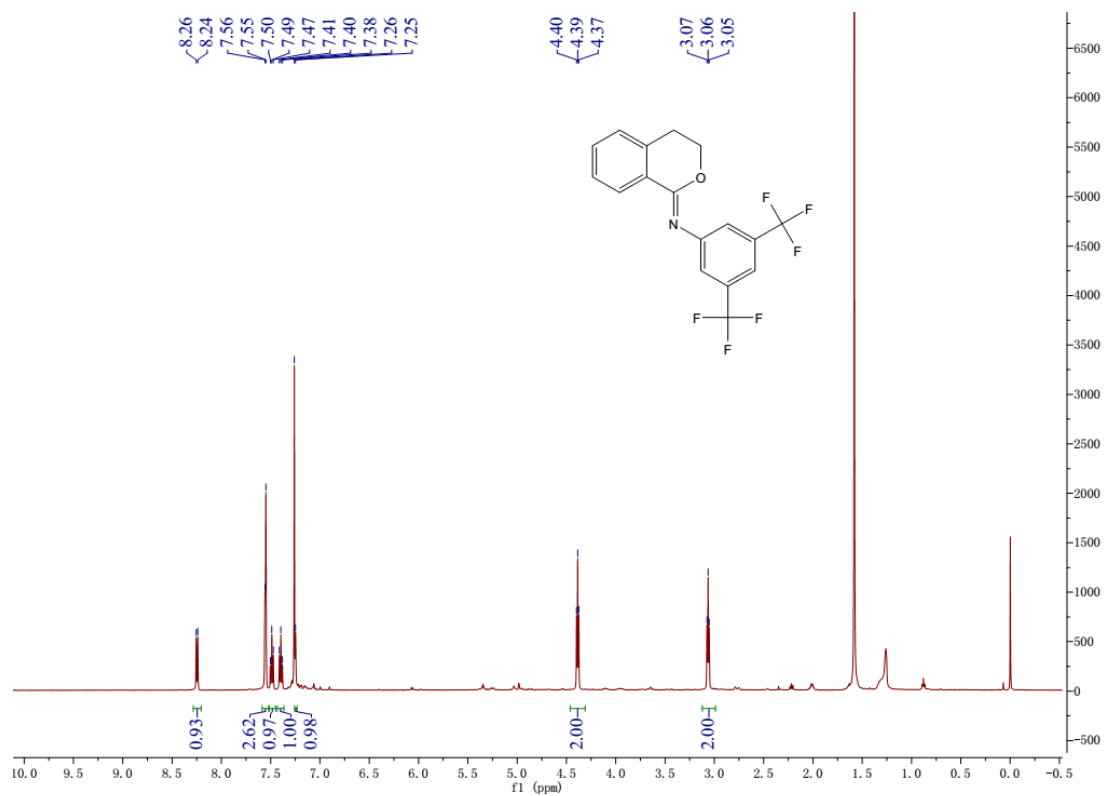
¹³C NMR spectrum (125 MHz, CDCl₃) of **4a**



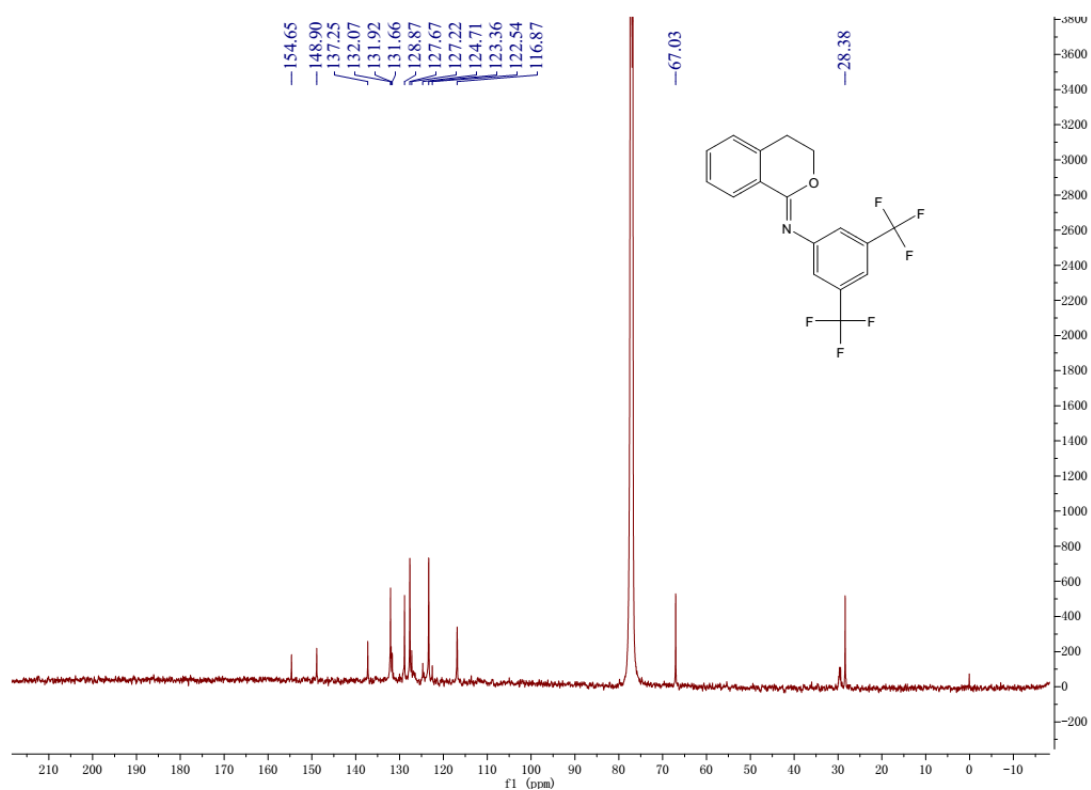
¹H NMR spectrum (500 MHz, CDCl₃) of **4b**



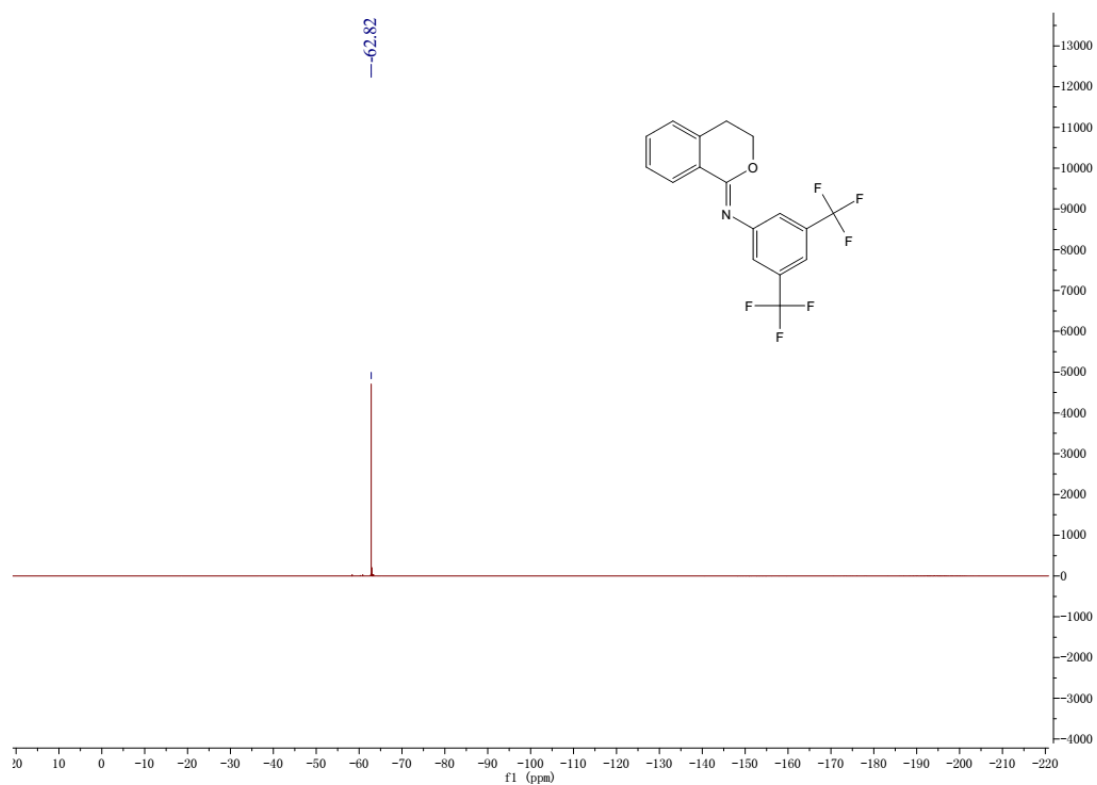
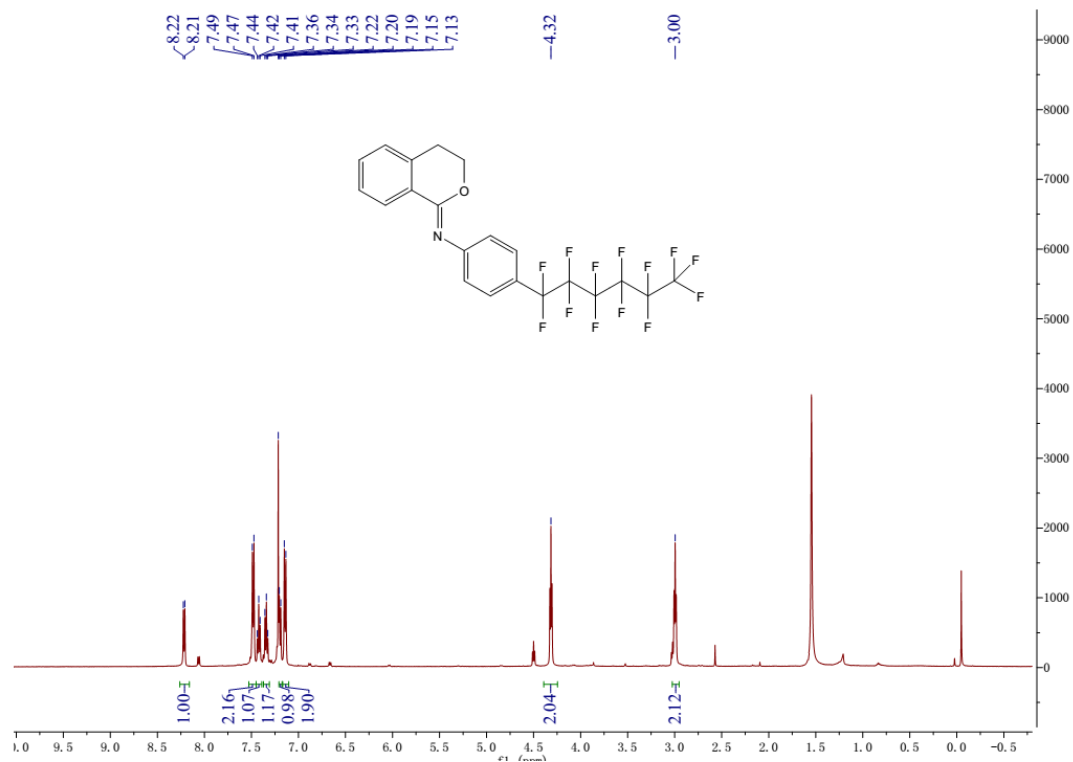
¹³C NMR spectrum (500 MHz, CDCl₃) of **4b**

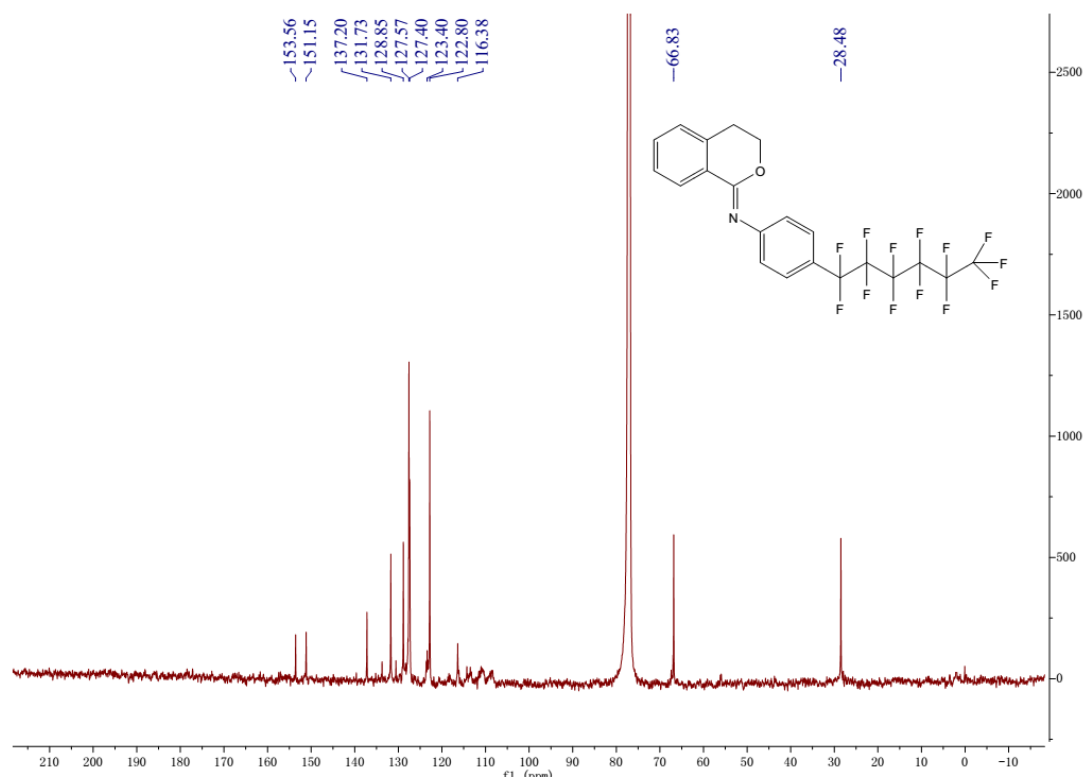


¹H NMR spectrum (500 MHz, CDCl₃) of **4c**

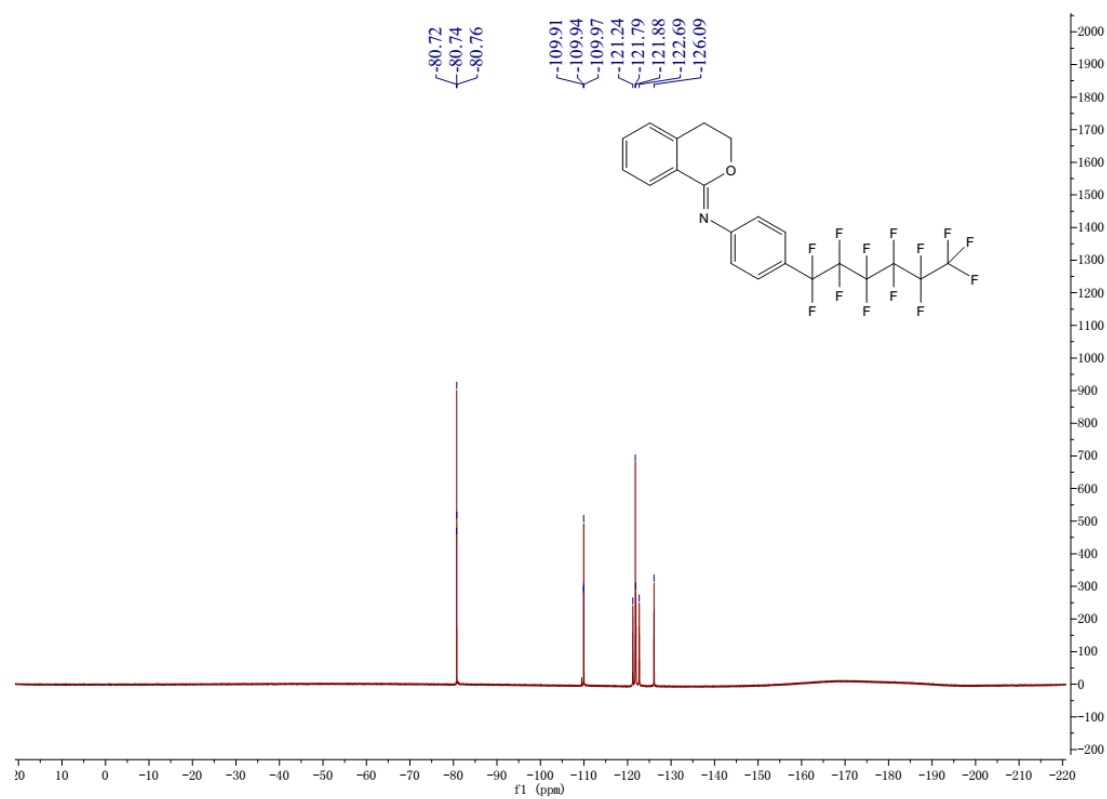


¹³C NMR spectrum (500 MHz, CDCl₃) of **4c**

 ^{19}F NMR spectrum (470 MHz, CDCl_3) of **4c** ^1H NMR spectrum (500 MHz, CDCl_3) of **4e**



¹³C NMR spectrum (500 MHz, CDCl₃) of **4e**



¹⁹F NMR spectrum (470 MHz, CDCl₃) of **4e**