

FACILE AND EFFICIENT N-BOC DEPROTECTION OF AMIDE AND SULFONAMIDE UNDER MICROWAVE IRRADIATION IN WATER

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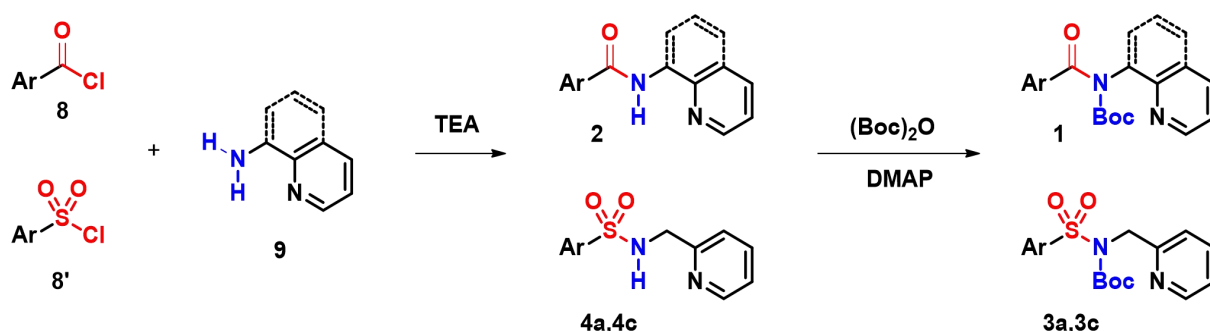
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1. General information

Solvents and reagents were commercially available and used without further purification. The concentration of water in the CH₂Cl₂ was less than 30 ppm. Thin-layer chromatography (TLC) analysis was used to monitor reaction. Flash column chromatography was performed using silica gel (200-300 mesh). ¹H spectra were recorded in DMSO-*d*₆ or CDCl₃ on 600 or 400 MHz NMR spectrometers and resonances (•) are given in parts per million relative to tetramethylsilane. Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet), coupling constants (Hz) and integration. ¹³C spectra were recorded in CDCl₃ or DMSO-*d*₆ on 100 or 150 MHz NMR spectrometers and resonances (•) are given in ppm. **The version of microwave instruments was WX6000.** Melting points were taken on a SGW X-4 melting point apparatus without correcting.

2. The general procedure of synthesis.

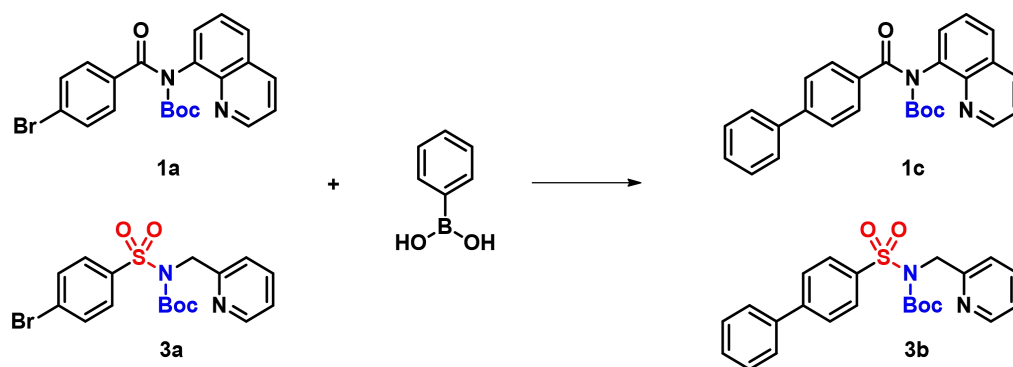


Scheme S1 The total synthesis of 1, 3a and 3c

2.1 General procedure for the synthesis of *N*-Boc amide and sulfonamide

1). The synthesis of **2** is representative: **9** (amino, 4 mmol) and DMAP (0.4 mmol) were placed in a 50 mL two-necked reaction flask. Dichloromethane (20 mL), **8** (acyl chloride, 4.3 mmol) and triethylamine (6.0 mmol) were added, and the mixture was stirred at room temperature for 12 h. The resulting mixture was then quenched with water. The mixture was extracted with dichloromethane, and the combined organic layer was dried over sodium sulfate. Concentration in vacuum followed by silica gel column purification gave **2**.¹⁻³ The method to **4a**, **4c** was similar to the synthesis **2**.

2). The synthesis of **1**, **3a** and **3c** is representative: Boc-anhydride (4 mmol, (Boc)₂O) was added to a solution of **2**, **4a** and **4c** (2 mmol) and DMAP (3 mmol) in CH₂Cl₂ (10 mL) and the reaction mixture was stirred overnight. The reaction mixture was quenched with sat. aq NH₄Cl (10 mL) and extracted with CH₂Cl₂ (3 x 20 mL). The combined organic extracts were dried (Na₂SO₄), concentrated under reduced pressure and purified by column chromatography to give the **1**, **3a** and **3c**.⁴⁻⁶



Scheme S2 The total synthesis of 1a and 3b

2.2 Typical $\text{Pd}(\text{PPh}_3)_4$ -Catalyzed to biphenyl compound (**1c** and **3b**).

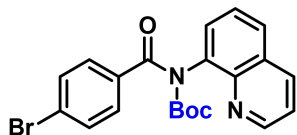
1a, **3a** (0.5 mmol) and phenylboronic acids (**4**, 0.5 mmol) were dissolved in dioxane (3 mL) and H_2O (1 mL) in a microwave tube under a nitrogen atmosphere. $\text{Pd}(\text{PPh}_3)_4$ (2 mol%) and base (NaOAc 1.0 mmol) were added, the reaction mixture was irradiated in a microwave apparatus at 80 °C for 20 minutes. After the reaction mixture was cooled to ambient temperature, the product was concentrated, and the crude mixture was purified by column chromatography on silica gel to the desired product (**1c** and **3b**).⁷⁻⁸

2.3 The microwave-assisted *N*-Boc deprotection of amide (**2**) and sulfonamide (**4**) in water

The compound **1** or **3** (0.5 mmol) and water (3.0 mL) were added to the tank, the reaction mixture was irradiated in a microwave apparatus at 120°C for 8 minutes. After the reaction mixture was cooled to ambient temperature, 10 g trash ice was added to the reaction tank which was placed in an ice bath. 30 minutes later, the product was obtained after filtration (The solid was washed with acetone and petroleum ether) and dry in excellent yields.

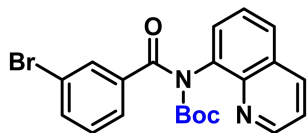
3. Analytical data of the products

tert-butyl (4-bromobenzoyl)(quinolin-8-yl)carbamate (**1a**)⁷.



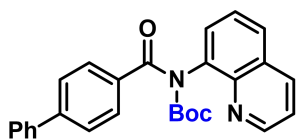
White solid, yield 92%; mp 159 - 160 °C. ¹H NMR (600 MHz, DMSO) δ 8.90 (d, *J* = 4.2 Hz, 1H), 8.47 (d, *J* = 8.4 Hz, 1H), 8.06 (d, *J* = 8.4 Hz, 1H), 7.82 (d, *J* = 7.2 Hz, 1H), 7.76 (s, 4H), 7.68 (t, *J* = 7.8 Hz, 1H), 7.61 (dd, *J* = 8.4, 4.2 Hz, 1H), 1.14 (s, 9H). ¹³C NMR (150 MHz, DMSO) δ 171.7, 152.4, 150.7, 143.3, 136.5, 136.2, 131.3, 130.0, 129.3, 128.6, 128.5, 126.4, 125.0, 122.0, 82.7, 27.0.

tert-butyl (3-bromobenzoyl)(quinolin-8-yl)carbamate (**1b**)⁷.



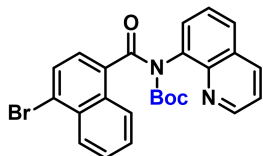
White solid, yield 90 %; mp 125 - 126 °C. ¹H NMR (600 MHz, DMSO) δ 8.91 (d, *J* = 3.0 Hz, 1H), 8.48 (d, *J* = 7.8 Hz, 1H), 8.07 (d, *J* = 7.8 Hz, 1H), 8.00 (s, 1H), 7.86 (d, *J* = 7.2 Hz, 1H), 7.80 (m, 2H), 7.69 (t, *J* = 7.8 Hz, 1H), 7.61 (dd, *J* = 8.4, 4.2 Hz, 1H), 7.51 (t, *J* = 7.8 Hz, 1H), 1.14 (s, 9H). ¹³C NMR (150 MHz, DMSO) δ 171.1, 152.3, 150.8, 143.3, 139.3, 136.5, 136.3, 134.0, 130.6, 130.4, 129.5, 128.7, 128.6, 126.8, 126.4, 122.1, 121.3, 82.9, 27.0.

tert-butyl [1,1'-biphenyl]-4-carbonyl(quinolin-8-yl)carbamate (**1c**)⁷.



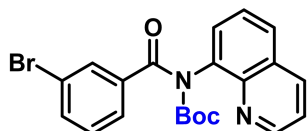
White solid, yield 91 %; mp 155 - 156 °C. ¹H NMR (600 MHz, DMSO) δ 8.93 (d, *J* = 4.2 Hz, 1H), 8.48 (d, *J* = 8.4 Hz, 1H), 8.06 (d, *J* = 8.4 Hz, 1H), 7.91 (d, *J* = 7.2 Hz, 2H), 7.85 (d, *J* = 7.2 Hz, 2H), 7.82 (d, *J* = 7.2 Hz, 1H), 7.77 (d, *J* = 7.2 Hz, 2H), 7.69 (t, *J* = 7.8 Hz, 1H), 7.65 – 7.59 (m, 1H), 7.52 (t, *J* = 7.2 Hz, 2H), 7.44 (t, *J* = 7.2 Hz, 1H), 1.15 (s, 9H). ¹³C NMR (150 MHz, DMSO) δ 172.3, 152.7, 150.7, 143.5, 143.1, 139.1, 136.8, 136.4, 135.8, 129.3, 129.1, 128.8, 128.7, 128.4, 128.2, 126.9, 126.4, 122.0, 82.5, 27.0.

tert-butyl (4-bromo-1-naphthoyl)(quinolin-8-yl)carbamate (**1d**)⁷.



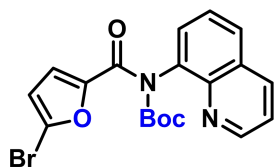
White solid, yield 90%; mp 159 - 160 °C. ¹H NMR (600 MHz, CDCl₃) δ 9.00 (s, 1H), 8.65 (s, 1H), 8.33 (s, 1H), 8.24 (d, *J* = 7.8 Hz, 1H), 7.95 (s, 1H), 7.91 (d, *J* = 7.8 Hz, 1H), 7.86 (d, *J* = 7.8 Hz, 1H), 7.80 (d, *J* = 7.2 Hz, 1H), 7.65 (d, *J* = 6.6 Hz, 4H), 7.48 (s, 1H), 0.90 (s, 9H). ¹³C NMR (150 MHz, CDCl₃) δ 171.8, 152.6, 150.5, 136.3, 131.8, 131.6, 129.3, 129.2, 128.9, 128.6, 127.8, 127.7, 127.4, 126.3, 126.2, 125.4, 125.0, 121.7, 83.5, 27.1.

tert-butyl (4-bromobenzoyl)(pyridin-2-ylmethyl)carbamate (**1e**)⁷.



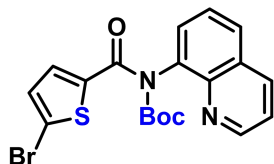
White solid, yield 92%; mp 82-83 °C. ¹H NMR (600 MHz, DMSO) δ 8.50 (d, *J* = 4.8 Hz, 1H), 7.79 (t, *J* = 7.8 Hz, 1H), 7.71 (d, *J* = 8.4 Hz, 2H), 7.56 (d, *J* = 8.4 Hz, 2H), 7.35 (d, *J* = 7.8 Hz, 1H), 7.30 – 7.25 (m, 1H), 5.01 (s, 2H), 1.10 (s, 9H). ¹³C NMR (150 MHz, CDCl₃) δ 172.2, 156.8, 153.0, 149.2, 136.4, 136.3, 131.1, 129.3, 125.4, 122.0, 120.8, 83.3, 50.1, 27.4.

tert-butyl (5-bromofuran-2-carbonyl)(quinolin-8-yl)carbamate (**1f**)⁷.



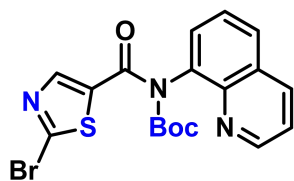
White solid, yield 87%; mp 111 - 112 °C. ¹H NMR (600 MHz, DMSO) δ 8.88 (s, 1H), 8.47 (d, *J* = 8.4 Hz, 1H), 8.05 (d, *J* = 8.4 Hz, 1H), 7.75 (d, *J* = 7.2 Hz, 1H), 7.66 (t, *J* = 7.8 Hz, 1H), 7.60 (dd, *J* = 8.4, 4.2 Hz, 1H), 7.25 (d, *J* = 3.0 Hz, 1H), 6.90 (d, *J* = 3.0 Hz, 1H), 1.32 (s, 9H). ¹³C NMR (150 MHz, DMSO) δ 162.4, 155.0, 151.9, 150.9, 145.8, 143.2, 136.5, 135.8, 135.7, 129.4, 129.1, 128.6, 126.4, 122.2, 83.4, 27.2.

tert-butyl (5-bromothiophene-2-carbonyl)(quinolin-8-yl)carbamate (**1g**)⁷.



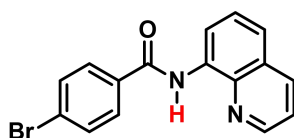
White solid, yield 86%; mp 92 - 93 °C. ¹H NMR (600 MHz, DMSO) δ 8.91 (d, *J* = 4.2 Hz, 1H), 8.47 (d, *J* = 8.4 Hz, 1H), 8.05 (d, *J* = 8.4 Hz, 1H), 7.77 (d, *J* = 7.2 Hz, 1H), 7.66 (t, *J* = 7.8 Hz, 1H), 7.61 (dd, *J* = 8.4, 4.2 Hz, 1H), 7.39 (d, *J* = 4.2 Hz, 1H), 7.28 (d, *J* = 4.2 Hz, 1H), 1.26 (s, 9H). ¹³C NMR (150 MHz, CDCl₃) δ 164.4, 153.0, 150.4, 143.9, 140.4, 136.8, 136.1, 133.1, 130.0, 129.0, 128.9, 128.4, 126.2, 121.7, 119.9, 83.4, 27.6.

tert-butyl (5-bromothiazole-2-carbonyl)(quinolin-8-yl)carbamate (**1h**)⁷.



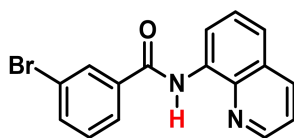
White solid, yield 83%; mp 111-112 °C. ¹H NMR (600 MHz, DMSO) δ 8.92 (d, *J* = 4.2 Hz, 1H), 8.49 (d, *J* = 8.4 Hz, 1H), 8.15 (s, 1H), 8.09 (d, *J* = 8.4 Hz, 1H), 7.86 (d, *J* = 7.2 Hz, 1H), 7.69 (t, *J* = 7.8 Hz, 1H), 7.63 (dd, *J* = 8.4, 4.2 Hz, 1H), 1.26 (s, 9H). ¹³C NMR (150 MHz, DMSO) δ 162.3, 155.0, 151.9, 150.9, 145.8, 143.2, 136.5, 135.8, 135.7, 129.4, 129.1, 128.6, 126.4, 122.2, 83.3, 27.2.

4-bromo-1-(quinolin-8-yl)benzamide (**2a**)⁷.



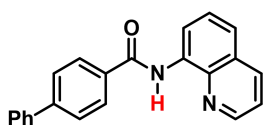
White solid, yield 97%; mp 128 - 129 °C.³¹ ¹H NMR (600 MHz, DMSO) δ 10.67 (s, 1H), 8.99 (dd, *J* = 4.2, 1.2 Hz, 1H), 8.71 (d, *J* = 7.8 Hz, 1H), 8.47 (dd, *J* = 8.4, 1.2 Hz, 1H), 7.99 (d, *J* = 8.4 Hz, 2H), 7.84 (d, *J* = 8.4 Hz, 2H), 7.77 (d, *J* = 8.2 Hz, 1H), 7.71 – 7.65 (m, 2H). ¹³C NMR (150 MHz, DMSO) δ 163.5, 149.1, 138.2, 136.6, 133.8, 133.4, 131.9, 129.1, 127.7, 126.9, 125.9, 122.4, 122.2, 116.7.

3-bromo-1-(quinolin-8-yl)benzamide(**2b**)⁷.



White solid, yield 99%; mp 99 - 100 °C.³² ¹H NMR (600 MHz, DMSO) δ 10.71 (s, 1H), 9.04 (s, 1H), 8.72 (d, *J* = 7.8 Hz, 1H), 8.52 (d, *J* = 8.4 Hz, 1H), 8.24 (s, 1H), 8.09 (d, *J* = 7.2 Hz, 1H), 7.93 (d, *J* = 7.8 Hz, 1H), 7.83 (d, *J* = 7.8 Hz, 1H), 7.76 – 7.70 (m, 2H), 7.65 (t, *J* = 7.8 Hz, 1H). ¹³C NMR (150 MHz, DMSO) δ 163.0, 149.1, 138.4, 136.7, 136.6, 134.7, 133.7, 131.0, 130.0, 127.8, 126.9, 125.9, 122.7, 122.3, 122.2, 117.2.

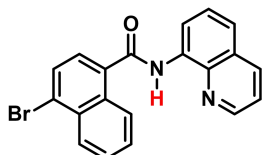
N-(quinolin-8-yl)-[1,1'-biphenyl]-4-carboxamide (**2c**)⁷.



White solid, yield 99%; mp 153 - 154 °C. ¹H NMR (600 MHz, DMSO) δ 10.74 (s, 1H), 9.02 (s, 1H), 8.77 (d, *J* = 7.8 Hz, 1H), 8.49 (d, *J* = 8.4 Hz, 1H), 8.14 (d, *J* = 7.2 Hz, 2H), 7.94 (d, *J* = 7.2 Hz, 2H), 7.78 (m,

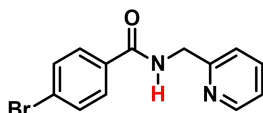
3H), 7.74 – 7.65 (m, 2H), 7.54 (t, $J = 7.2$ Hz, 2H), 7.46 (t, $J = 7.2$ Hz, 1H). ^{13}C NMR (150 MHz, DMSO) δ 164.1, 149.2, 143.6, 138.9, 138.3, 136.8, 134.0, 133.1, 129.1, 128.3, 127.9, 127.7, 127.2, 127.1, 127.0, 122.4, 116.6.

*4-bromo-1-(quinolin-8-yl)-1-naphthamide(2d)*⁷.



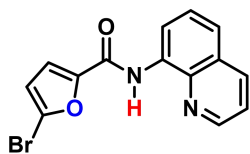
White solid, yield 97%; mp 198 - 199 °C. ^1H NMR (600 MHz, CDCl_3) δ 10.41 (s, 1H), 9.04 (s, 1H), 8.76 (s, 1H), 8.54 (s, 1H), 8.36 (d, $J = 7.8$ Hz, 1H), 8.23 (s, 1H), 7.91 (s, 1H), 7.78 (s, 1H), 7.65 (m, 4H), 7.49 (s, 1H). ^{13}C NMR (150 MHz, CDCl_3) δ 166.9, 148.3, 138.3, 136.4, 134.4, 134.3, 132.1, 131.2, 129.0, 128.0, 127.8, 127.4, 127.3, 126.0, 125.9, 125.5, 122.2, 121.7, 116.9.

*5-bromo-N-(pyridin-2-ylmethyl)benzamide (2e)*⁷.



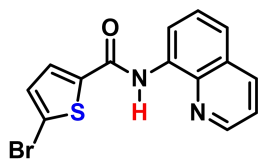
White solid, yield 99%; mp 112-113 °C. ^1H NMR (600 MHz, CDCl_3) δ 8.57 (d, $J = 4.2$ Hz, 1H), 7.76 (d, $J = 8.4$ Hz, 2H), 7.72 (m, 2H), 7.59 (d, $J = 8.4$ Hz, 2H), 7.35 (d, $J = 7.8$ Hz, 1H), 7.25 (s, 1H), 4.76 (d, $J = 4.8$ Hz, 2H). ^{13}C NMR (150 MHz, DMSO) δ 165.5, 158.6, 148.9, 136.7, 133.3, 131.4, 129.4, 125.1, 122.1, 121.0, 44.8.

*5-bromo-1-(quinolin-8-yl)furan-2-carboxamide (2f)*⁷.



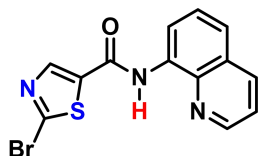
White solid, yield 99%; mp 109 - 110 °C. ^1H NMR (600 MHz, DMSO) δ 10.54 (s, 1H), 9.02 (dd, $J = 4.2, 1.2$ Hz, 1H), 8.68 (d, $J = 7.8$ Hz, 1H), 8.48 (dd, $J = 8.4, 1.2$ Hz, 1H), 7.76 (d, $J = 8.4$ Hz, 1H), 7.70 (dd, $J = 8.4, 4.2$ Hz, 1H), 7.65 (t, $J = 7.8$ Hz, 1H), 7.43 (d, $J = 3.6$ Hz, 1H), 6.94 (d, $J = 3.6$ Hz, 1H). ^{13}C NMR (150 MHz, DMSO) δ 154.3, 149.3, 149.1, 137.8, 136.8, 133.3, 127.8, 127.0, 125.6, 122.5, 122.4, 118.0, 116.4, 115.1.

*5-bromo-N-(quinolin-8-yl)thiophene-2-carboxamide (2g)*⁷.



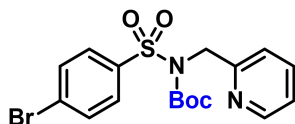
White solid, yield 94 %; mp 156 - 157 °C. ¹H NMR (600 MHz, DMSO) δ 10.58 (s, 1H), 9.00 (d, *J* = 4.2 Hz, 1H), 8.50 (d, *J* = 7.8 Hz, 2H), 7.90 (d, *J* = 4.2 Hz, 1H), 7.80 (d, *J* = 8.4 Hz, 1H), 7.70 (dd, *J* = 8.4, 4.2 Hz, 1H), 7.66 (t, *J* = 7.8 Hz, 1H), 7.43 (d, *J* = 4.2 Hz, 1H).

*5-bromo-N-(quinolin-8-yl)thiazole-2-carboxamide (2h)*⁷.



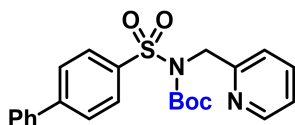
White solid, yield 98 %; mp 137 - 138 °C. ¹H NMR (600 MHz, CDCl₃) δ 10.55 (s, 1H), 8.87 (d, *J* = 4.2 Hz, 1H), 8.79 – 8.72 (m, 1H), 8.22 (d, *J* = 8.4 Hz, 2H), 7.61 – 7.56 (m, 2H), 7.52 (dd, *J* = 8.4, 4.2 Hz, 1H). ¹³C NMR (150 MHz, CDCl₃) δ 157.2, 156.0, 148.5, 141.3, 138.1, 137.9, 136.4, 133.4, 127.8, 127.2, 122.4, 121.9, 116.7. HRMS (ESI): *m/z* [M+H]⁺ calcd for C₁₃H₉BrN₃OS 333.9650 ; found 333.9641.

*tert-butyl (4-bromophenyl)sulfonyl(pyridin-2-ylmethyl)carbamate (3a)*⁸



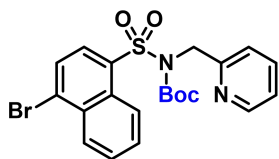
White solid; yield 92%; m.p. 124-125 °C; ¹H NMR (600 MHz, CDCl₃) δ 8.53 (d, *J* = 4.8 Hz, 1H), 7.87 (d, *J* = 8.4 Hz, 2H), 7.68 (d, *J* = 7.8 Hz, 1H), 7.64 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 7.8 Hz, 1H), 7.23 – 7.17 (m, 1H), 5.14 (s, 2H), 1.30 (s, 9H). ¹³C NMR (150 MHz, CDCl₃) δ 156.45, 150.59, 149.05, 138.69, 136.60, 131.60, 130.05, 128.29, 122.21, 120.82, 84.69, 51.16, 27.69.

*tert-butyl [1,1'-biphenyl]-4-ylsulfonyl(pyridin-2-ylmethyl)carbamate (3b)*⁸



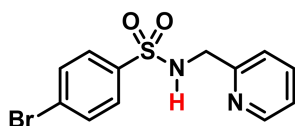
White solid; yield 91%; m.p. 116-117 °C; ¹H NMR (600 MHz, CDCl₃) δ 8.56 (d, *J* = 4.2 Hz, 1H), 8.04 (d, *J* = 8.4 Hz, 2H), 7.71 (t, *J* = 9.0 Hz, 3H), 7.63 (d, *J* = 7.2 Hz, 2H), 7.49 (t, *J* = 7.8 Hz, 2H), 7.44 (d, *J* = 7.2 Hz, 1H), 7.37 (d, *J* = 7.8 Hz, 1H), 7.20 (d, *J* = 6.6 Hz, 1H), 5.19 (s, 2H), 1.31 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 156.83, 150.78, 149.08, 146.02, 139.00, 138.22, 136.62, 128.95, 128.79, 128.50, 127.22, 126.96, 122.11, 120.65, 84.50, 51.33, 27.68.

tert-butyl (4-bromonaphthalen-1-yl)sulfonyl(pyridin-2-ylmethyl)carbamate (**3c**)⁸



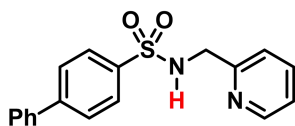
White solid; yield 93%; m.p. 168-169 °C; ¹H NMR (600 MHz, CDCl₃) δ 8.53 (d, *J* = 4.2 Hz, 1H), 7.87 (d, *J* = 8.4 Hz, 2H), 7.68 (t, *J* = 7.8 Hz, 1H), 7.64 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 7.8 Hz, 1H), 7.22 – 7.17 (m, 1H), 5.14 (s, 2H), 1.30 (s, 9H). ¹³C NMR (150 MHz, CDCl₃) δ 156.67, 150.46, 149.10, 136.78, 134.43, 132.22, 132.04, 130.57, 129.18, 128.85, 128.36, 128.18, 128.12, 124.08, 122.16, 120.34, 84.79, 51.43, 27.55.

4-bromo-*N*-(pyridin-2-ylmethyl)benzenesulfonamide (**4a**)⁸



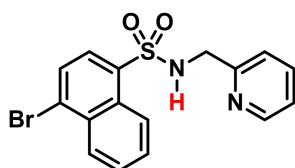
White solid; yield 97%; m.p. 104.5-104.9 °C; ¹H NMR (600 MHz, CDCl₃) δ 8.44 (d, *J* = 4.2 Hz, 1H), 7.70 (d, *J* = 8.4 Hz, 2H), 7.62 (t, *J* = 7.8 Hz, 1H), 7.57 (d, *J* = 8.4 Hz, 2H), 7.17 (dd, *J* = 12.0, 7.8 Hz, 2H), 6.26 (s, 1H), 4.27 (d, *J* = 4.8 Hz, 2H). ¹³C NMR (150 MHz, CDCl₃) δ 154.65, 148.80, 138.81, 136.89, 132.02, 128.50, 127.20, 122.63, 122.14, 47.36.

N-(pyridin-2-ylmethyl)-[1,1'-biphenyl]-4-sulfonamide (**4b**)⁸



White solid; yield 96%; m.p. 90-91 °C; ¹H NMR (600 MHz, CDCl₃) δ 8.46 (d, *J* = 4.2 Hz, 1H), 7.91 (d, *J* = 8.4 Hz, 2H), 7.68 – 7.59 (m, 3H), 7.56 (d, *J* = 7.2 Hz, 2H), 7.47 (t, *J* = 7.8 Hz, 2H), 7.42 (d, *J* = 7.2 Hz, 1H), 7.26 – 7.14 (m, 2H), 6.14 (s, 1H), 4.33 (d, *J* = 5.4 Hz, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 154.88, 148.76, 145.30, 139.22, 138.23, 136.92, 128.94, 128.34, 127.56, 127.49, 127.17, 122.59, 122.13, 47.44.

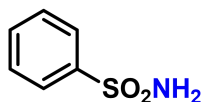
4-bromo-*N*-(pyridin-2-ylmethyl)naphthalene-1-sulfonamide (**4c**)⁸



White solid; yield 96%; m.p. 86.0-86.6 °C; ¹H NMR (600 MHz, CDCl₃) δ 8.70 (d, *J* = 9.0 Hz, 1H), 8.30 (d, *J* = 8.4 Hz, 1H), 8.21 (d, *J* = 4.8 Hz, 1H), 8.08 (d, *J* = 7.8 Hz, 1H), 7.80 (d, *J* = 7.8 Hz, 1H), 7.73 –

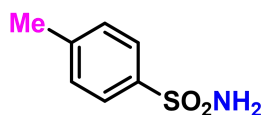
7.63 (m, 2H), 7.51 – 7.43 (m, 1H), 7.02 (dd, $J = 7.2, 5.4$ Hz, 1H), 6.97 (d, $J = 7.8$ Hz, 1H), 6.48 (s, 1H), 4.23 (d, $J = 5.4$ Hz, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 154.43, 148.58, 136.44, 134.70, 132.32, 129.47, 129.27, 129.00, 128.81, 128.19, 128.06, 125.02, 122.32, 121.83, 47.44.

benzenesulfonamide (4d)⁹



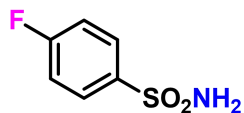
White solid; yield 98%; m.p. 149 - 150 °C; ^1H NMR (400 MHz, DMSO) δ 7.84 (dd, $J = 8.0$ Hz, 1.6 Hz, 2H), 7.64 – 7.54 (m, 3H), 7.37 (br, 2H).

4-methylbenzenesulfonamide (4e)⁹



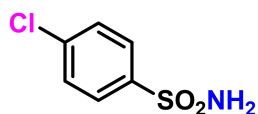
White solid; yield 99%; m.p. 135 - 136 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.81 (m, 2H), 7.64 – 7.31 (m, 2H), 4.87 (br, 2H), 2.43 (s, 3H).

4-fluorobenzenesulfonamide (4f)⁹



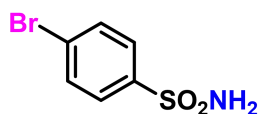
White solid; yield 99%; m.p. 100-101 °C; ^1H NMR (400 MHz, DMSO) δ 7.89 (dd, $J = 8.0$ Hz, 5.6 Hz, 2H), 7.44 – 7.39 (m, 4H).

4-chlorobenzenesulfonamide (4g)⁹



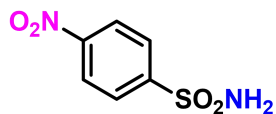
White solid; yield 98%; m.p. 135-136 °C; ^1H NMR (400 MHz, DMSO) δ 7.84-7.81 (dd, $J = 8.0$ Hz, 5.6 Hz, 2H), 7.67 – 7.65 (m, 2H), 7.47 (br, 2H).

4-bromobenzenesulfonamide (4h)⁹



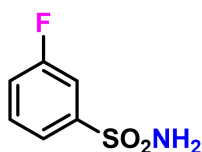
White solid; yield 97%; m.p. 159-160 °C; ^1H NMR (600 MHz, DMSO) δ 7.78 ($J = 8.0$ Hz, 5.6 Hz, 4H), 7.47 (br, 2H).

4-nitrobenzenesulfonamide (4i)¹⁰



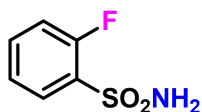
White solid; yield 98%; m.p. 181-182 °C; ¹H NMR (400 MHz, DMSO) δ 8.46-8.40 (m, 2H), 8.12-8.06 (m, 2H), 7.75 (br, 2H) .

3-fluorobenzenesulfonamide (4j)¹¹



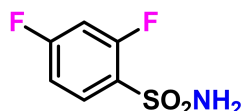
White solid; yield 99%; m.p. 128-129 °C; ¹H NMR (400 MHz, DMSO) δ 7.71-7.58 (m, 3H), 7.56 – 7.44 (m, 3H).

2-fluorobenzenesulfonamide (4k)¹¹



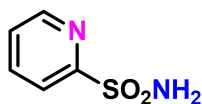
White solid; yield 99%; m.p. 159-160 °C; ¹H NMR (400 MHz, DMSO) δ 7.84 (td, *J* = 8.0 Hz, 2.0 Hz, 4H), 7.69 (m, 3H), 7.45 (dd, *J* = 10.8 Hz, 8.4 Hz, 1H), 7.39 (t, *J* = 8.0 Hz, 1H).

2,4-difluorobenzenesulfonamide (4l)¹¹



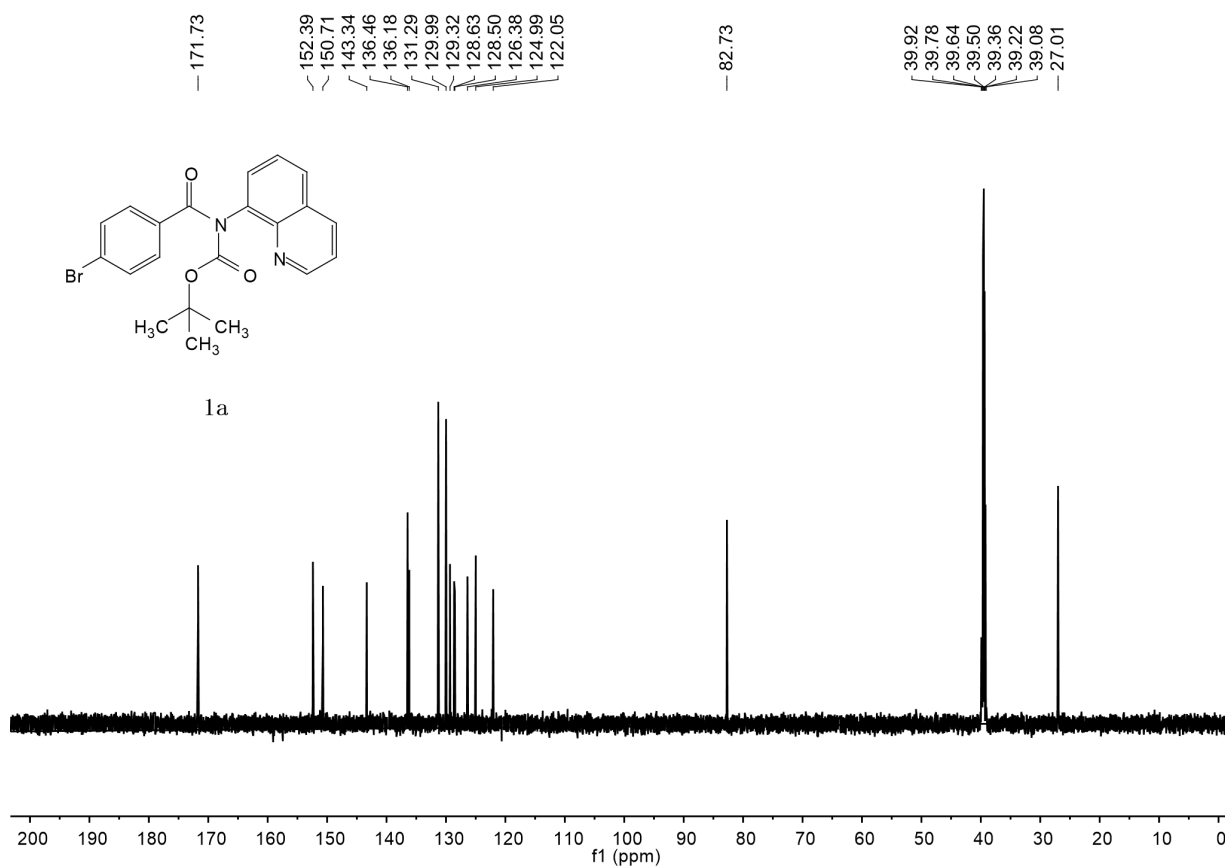
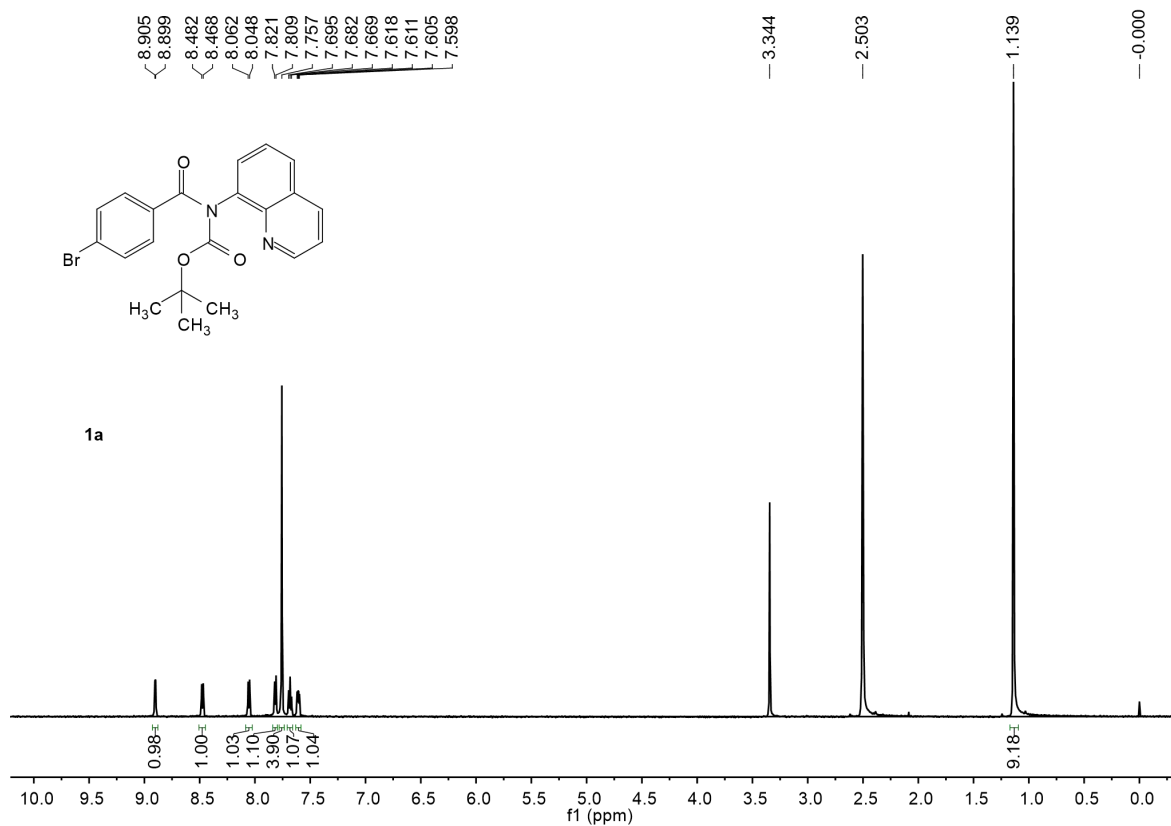
White solid; yield 99%; m.p. 161-162 °C; ¹H NMR (400 MHz, , DMSO) δ 7.87 (td, *J* = 9.0 Hz, 6.4 Hz, 1H), 7.73 (br, 2H), 7.56-7.49 (m, 1H), 7.29-7.23 (m, 1H).

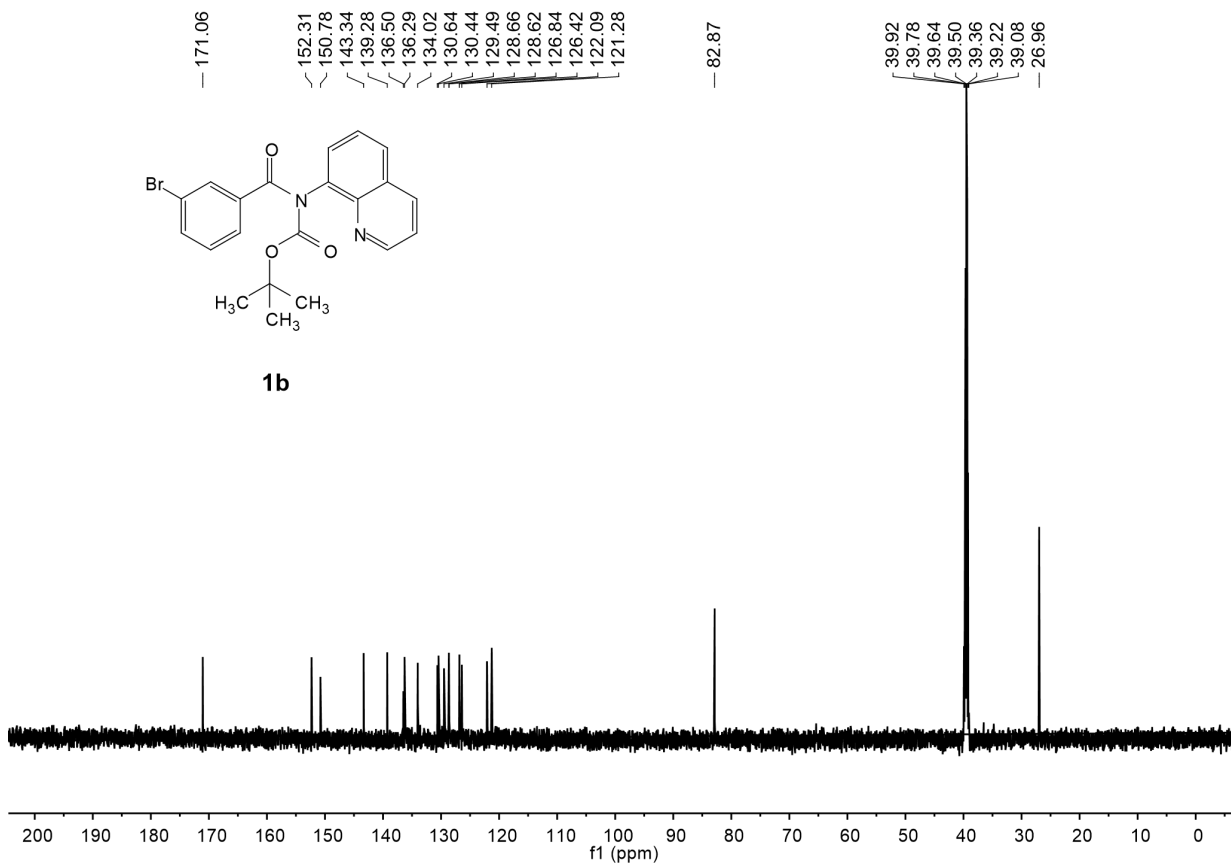
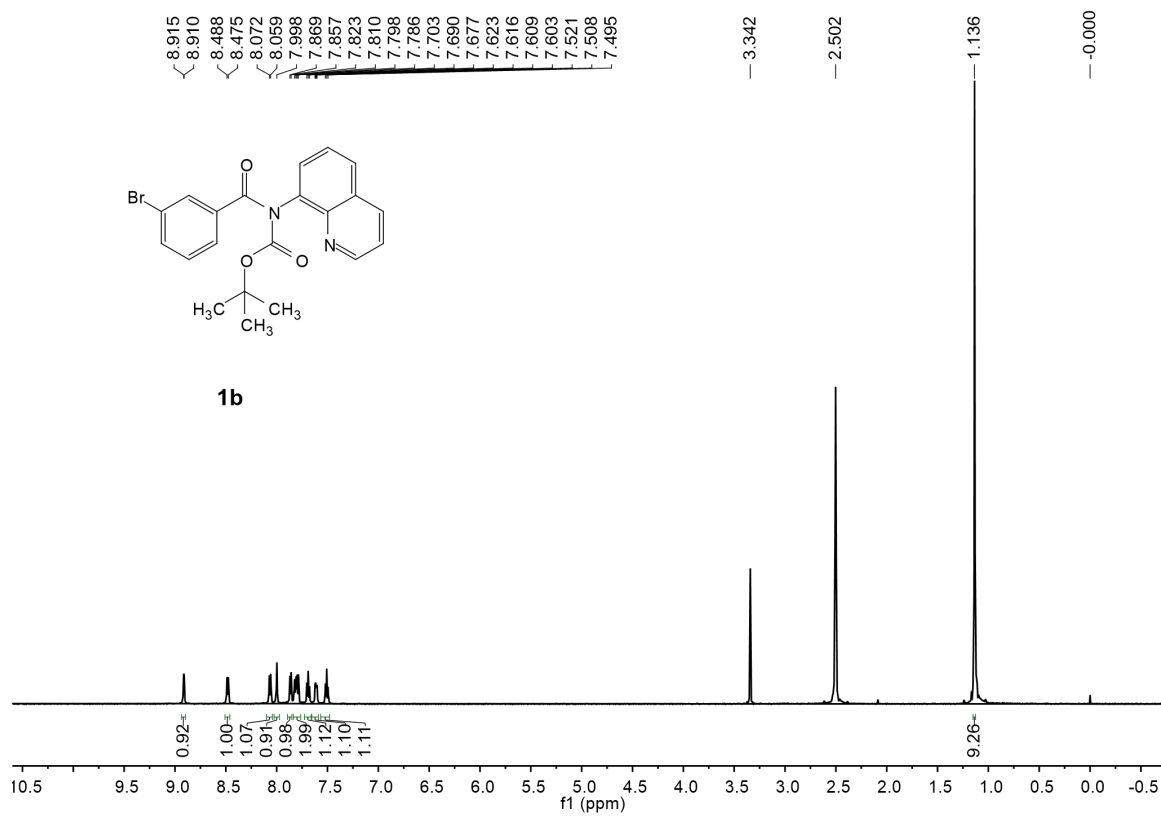
pyridine-2-sulfonamide (4m)¹⁰

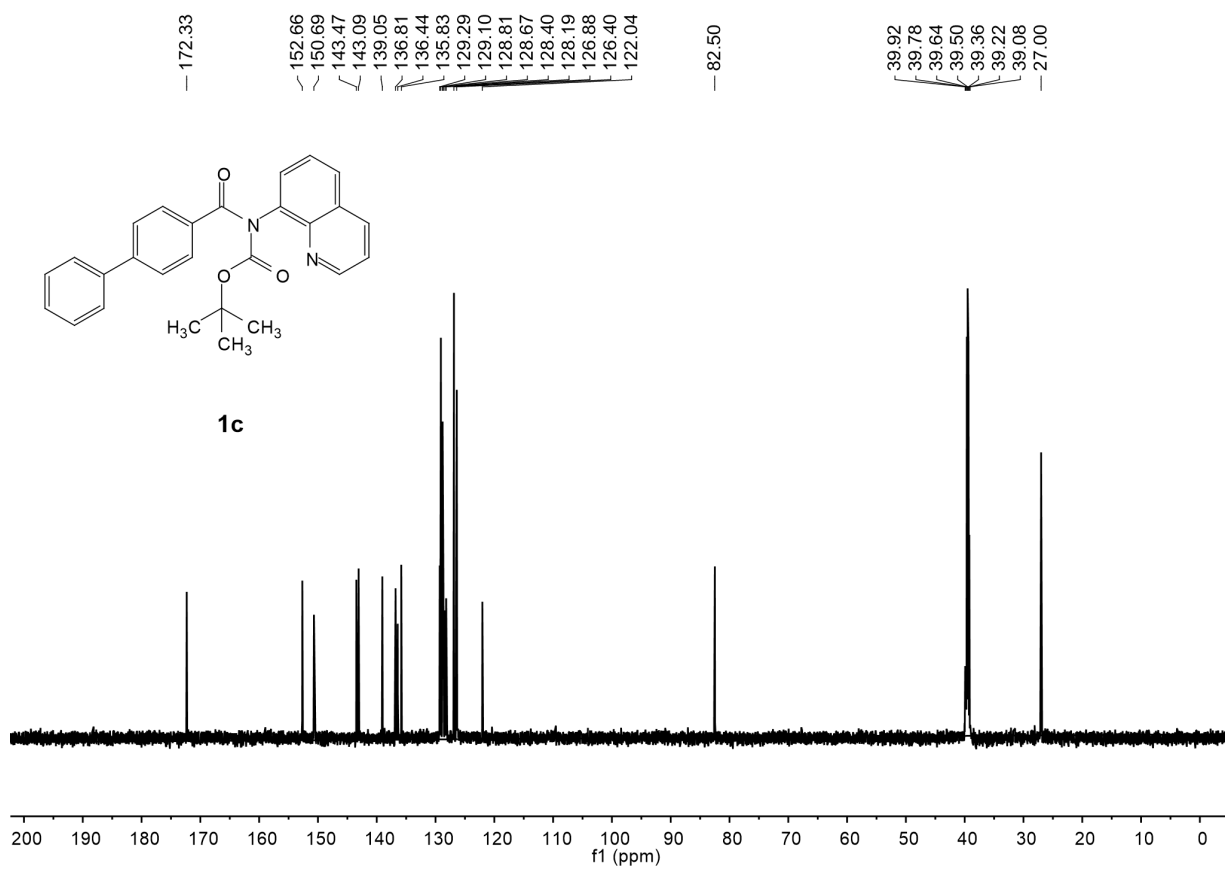
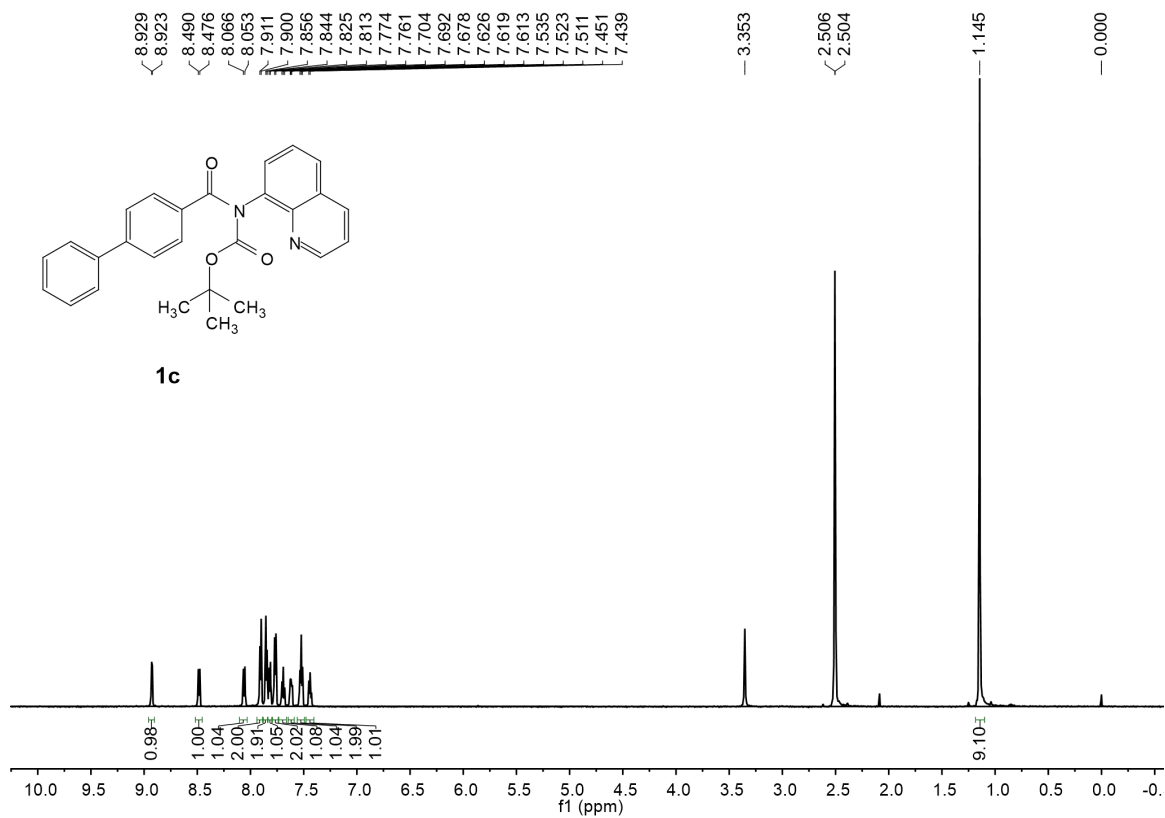


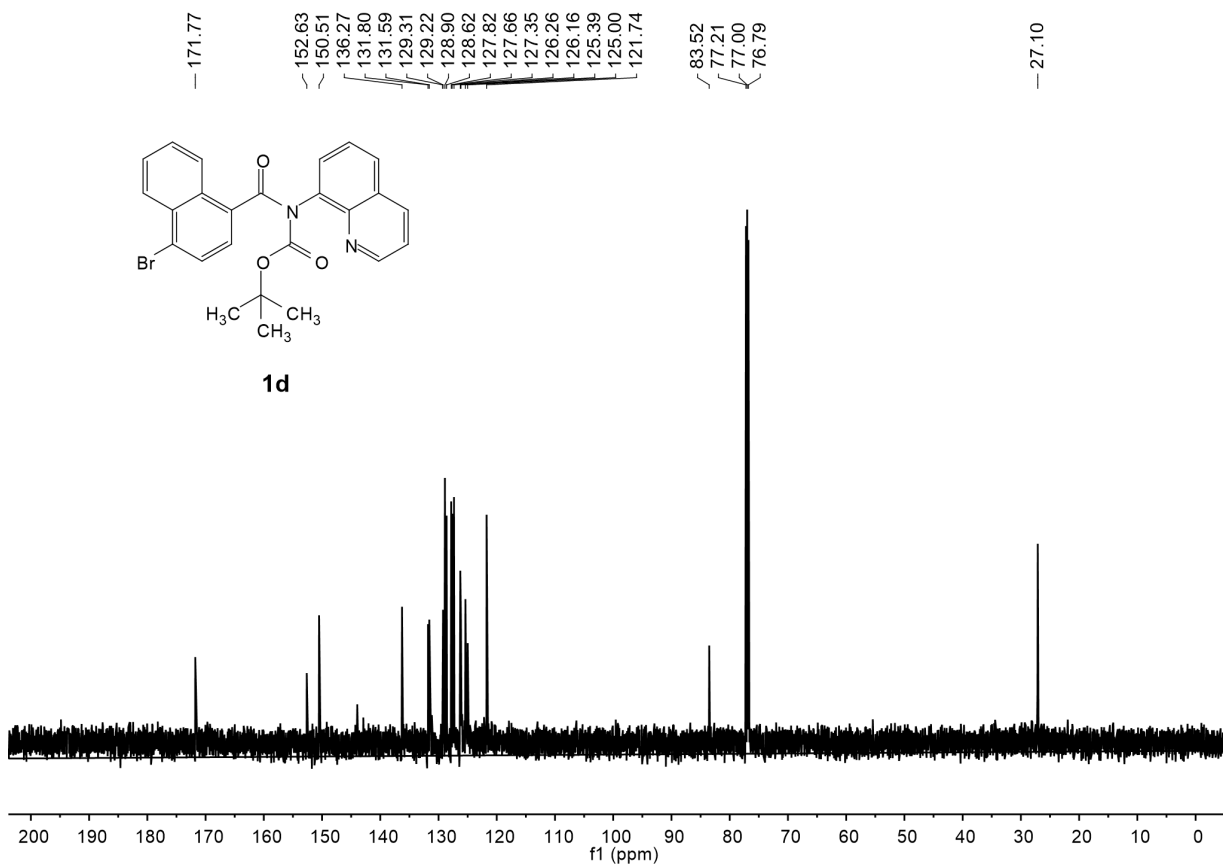
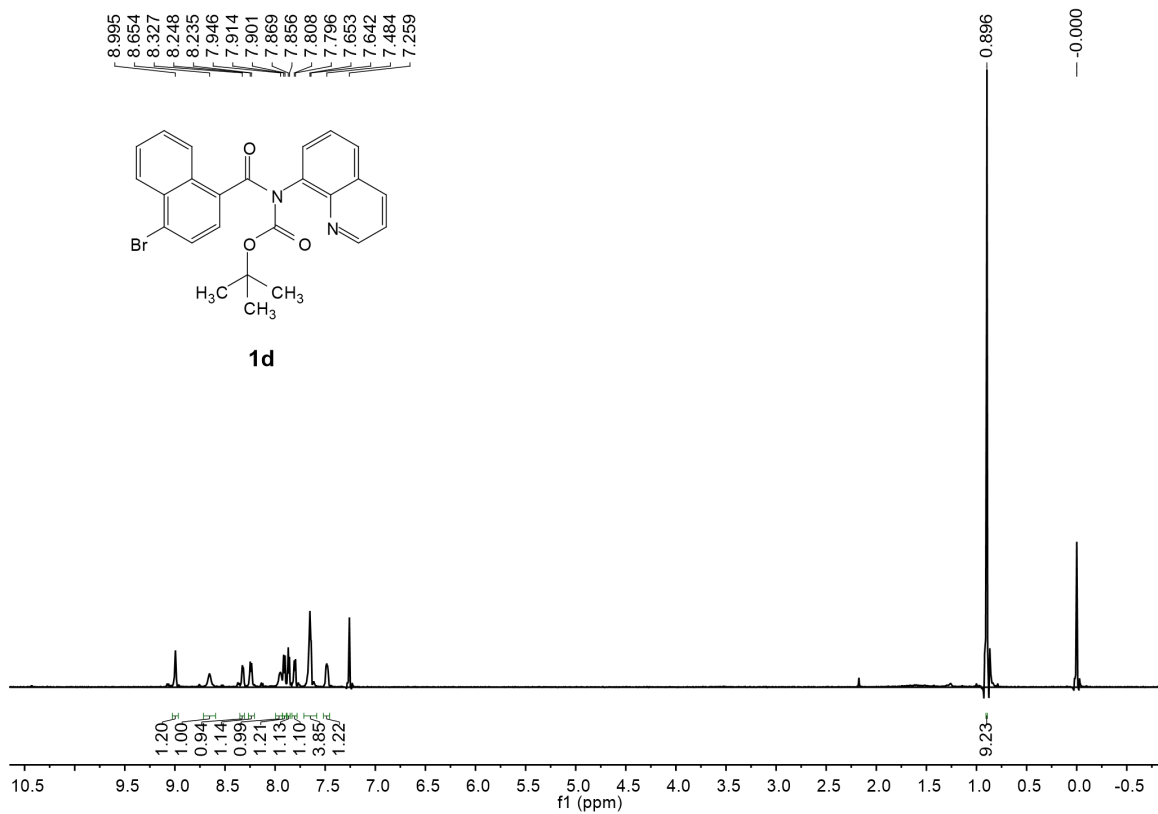
White solid; yield 99%; m.p. 144-145 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.81 (m, 2H), 7.64 – 7.31 (m, 2H), 4.87 (br, 2H) , 2.43 (s, 3H).

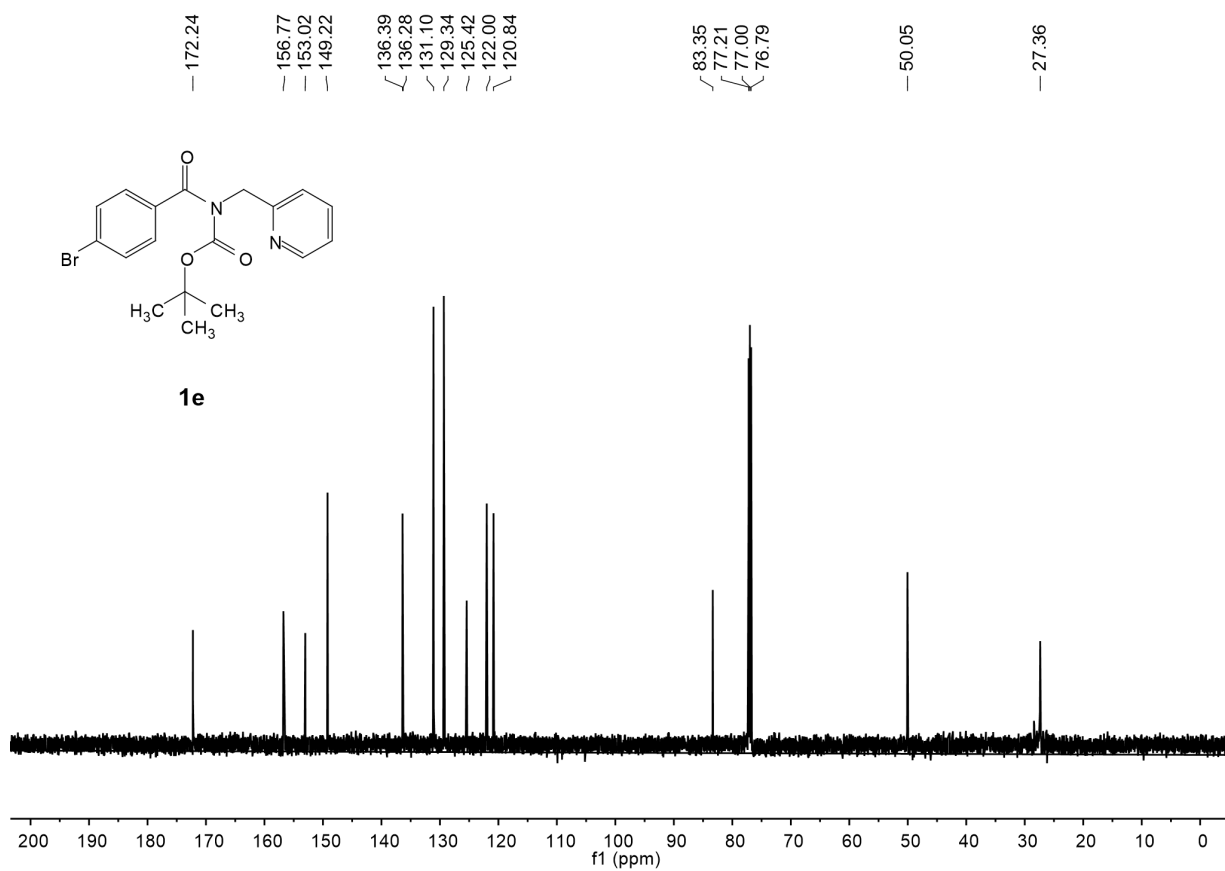
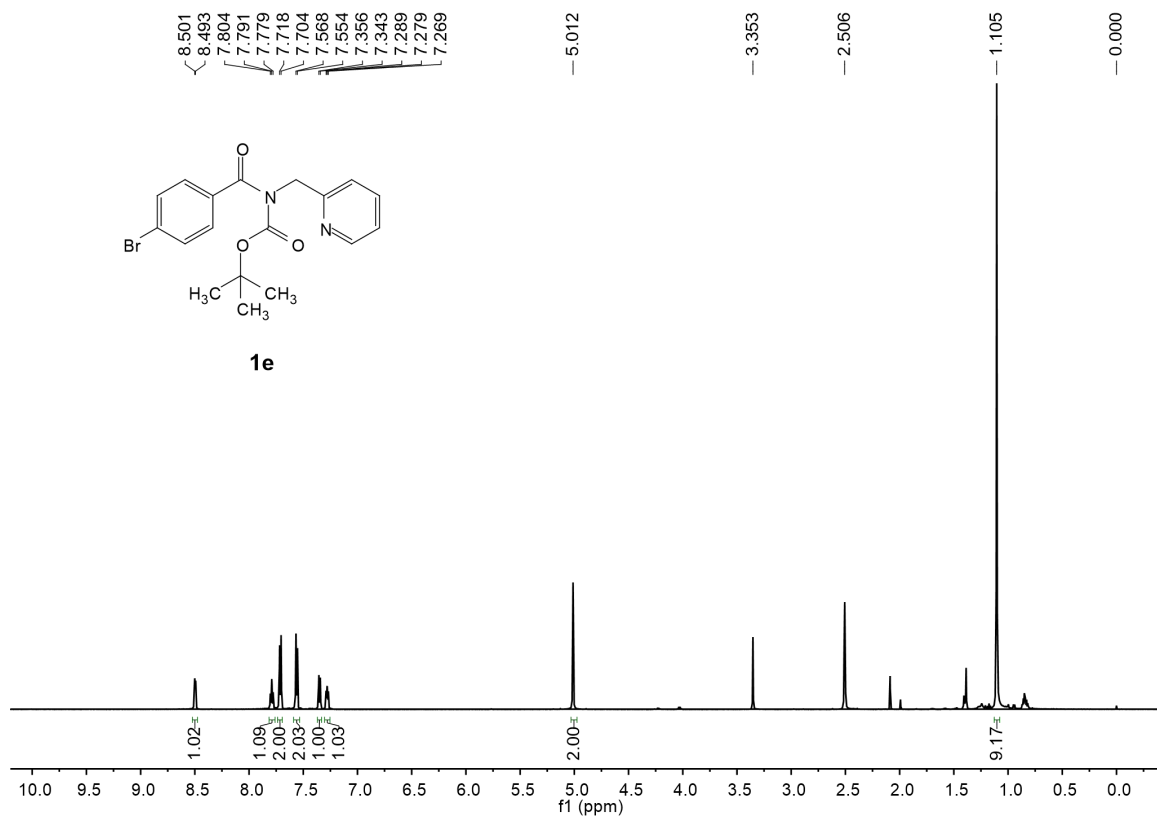
4. Copies of NMR Spectra

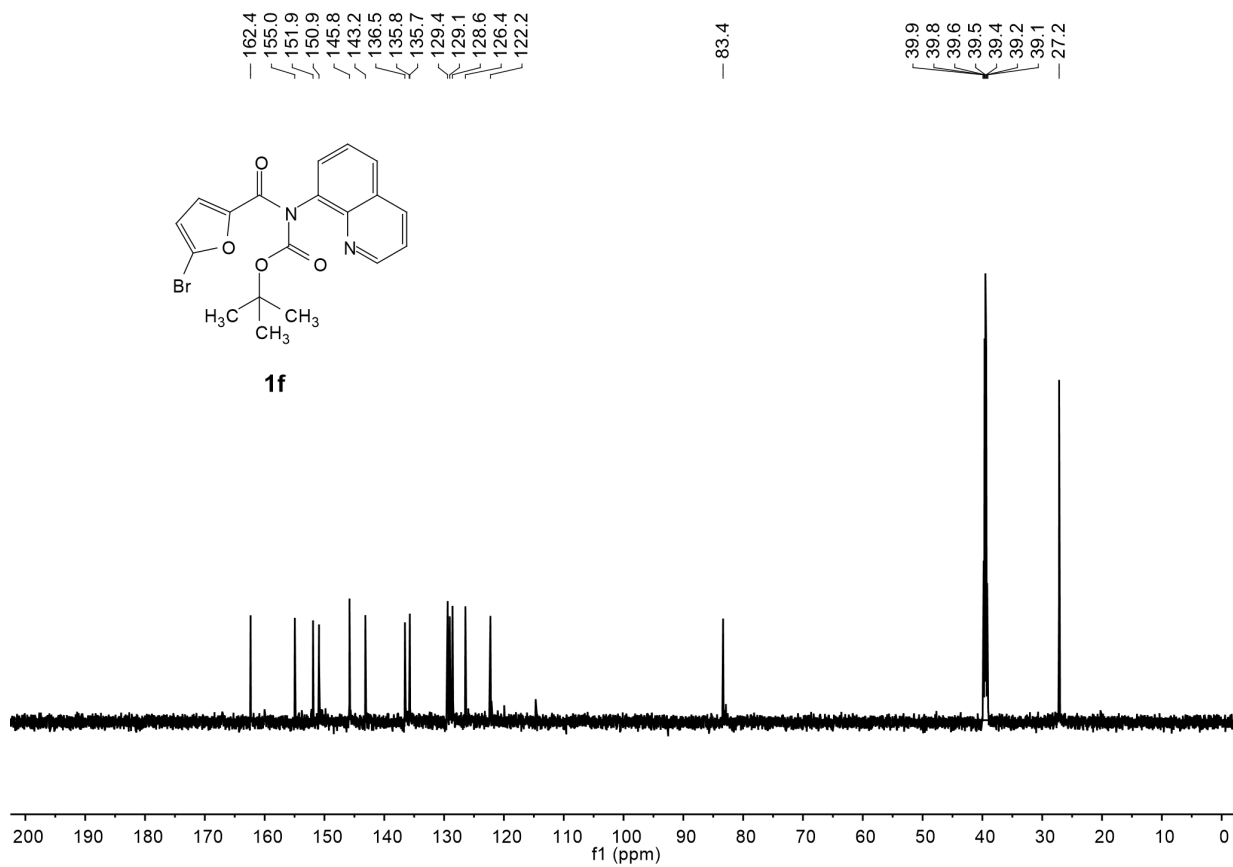
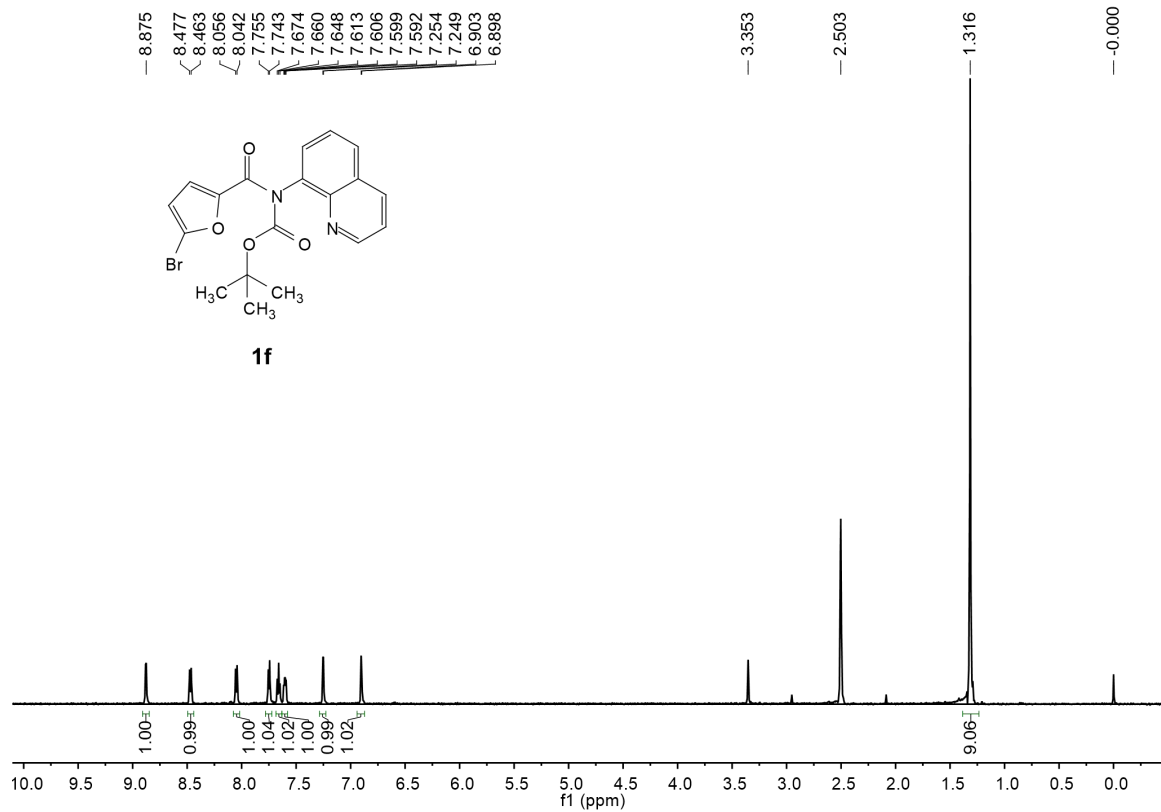


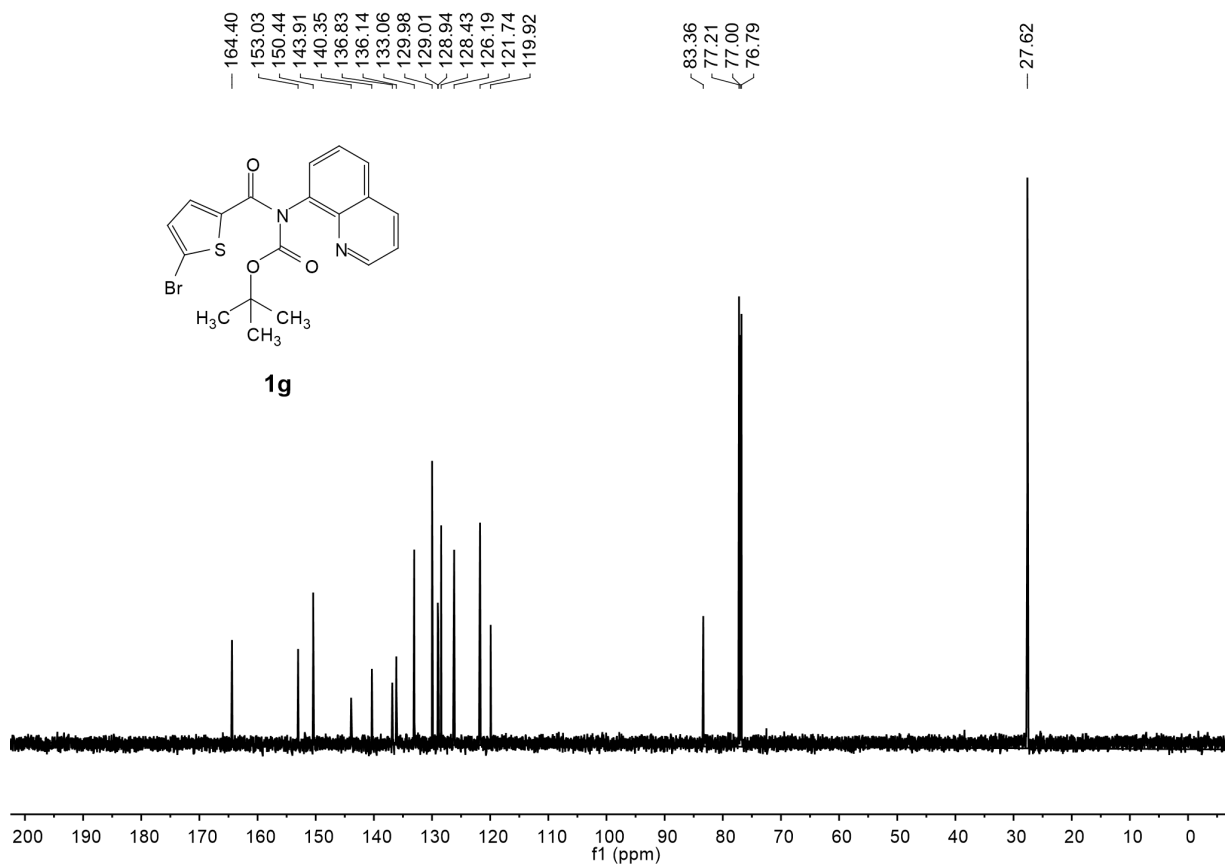
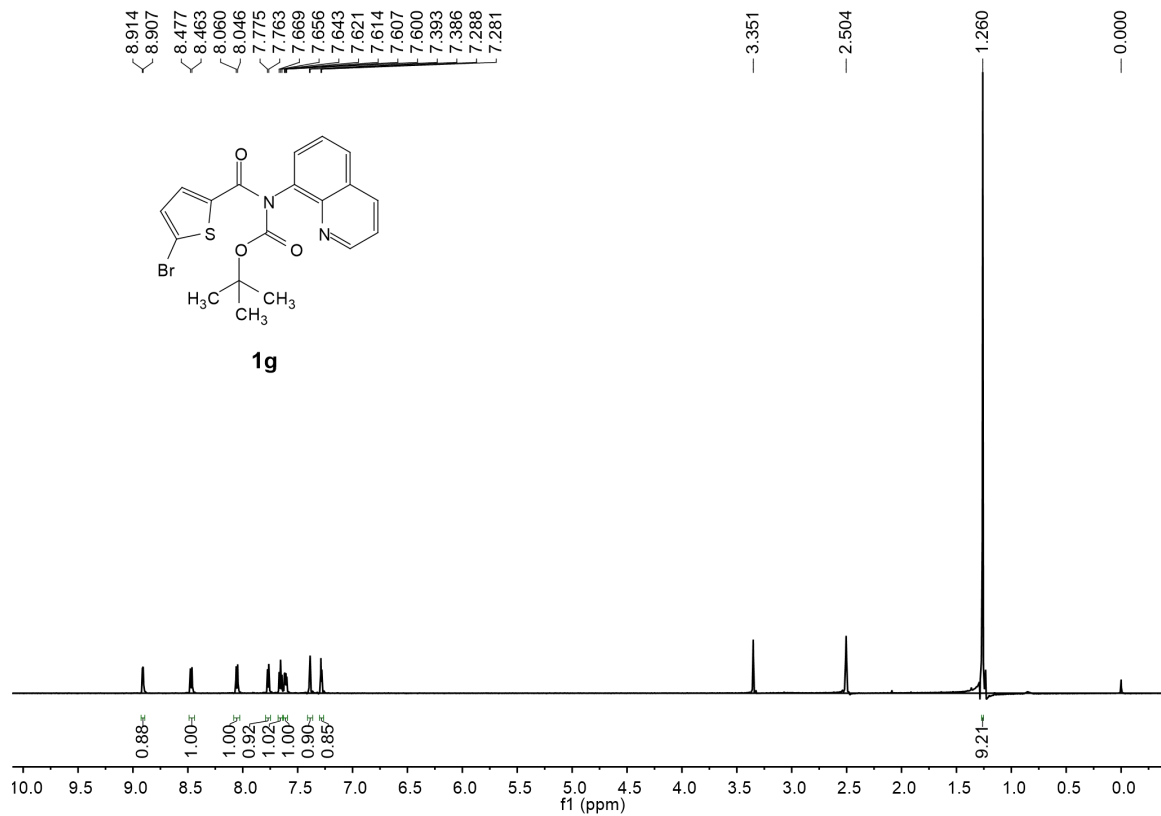


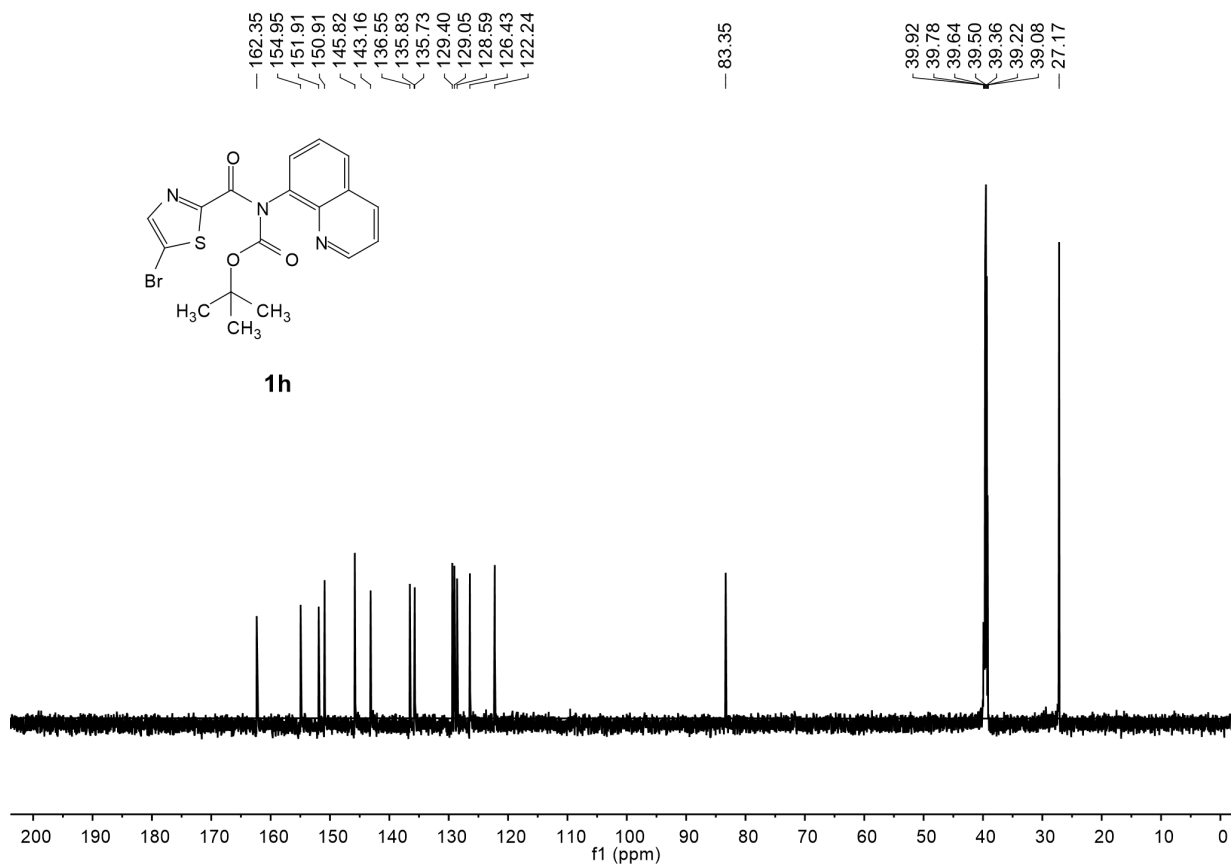
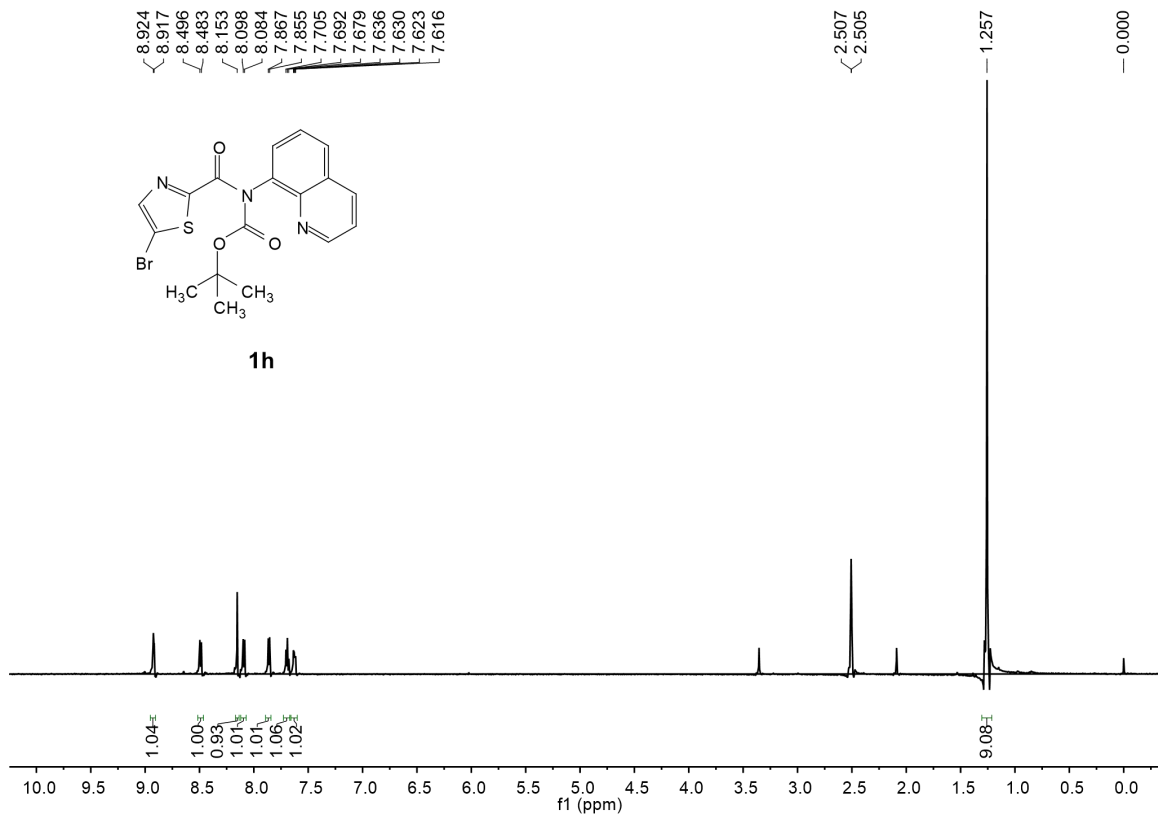


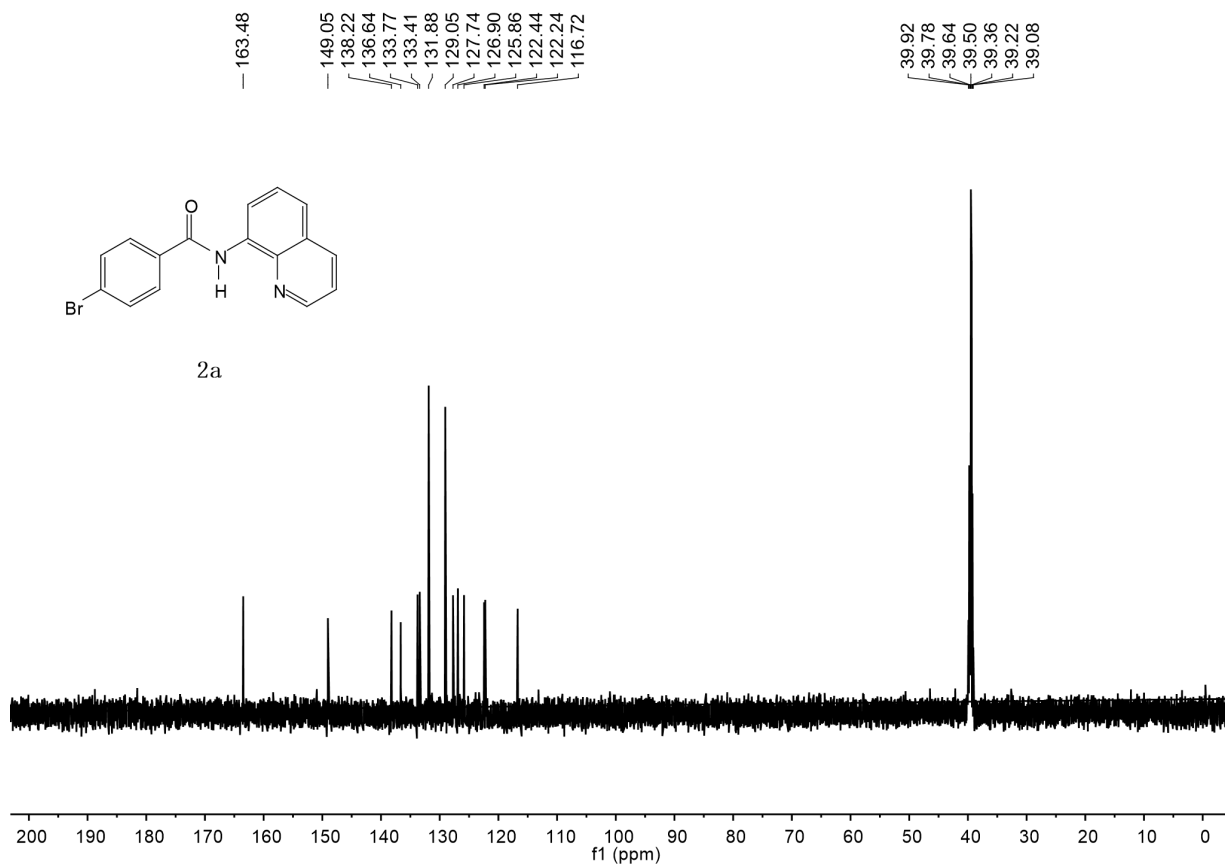
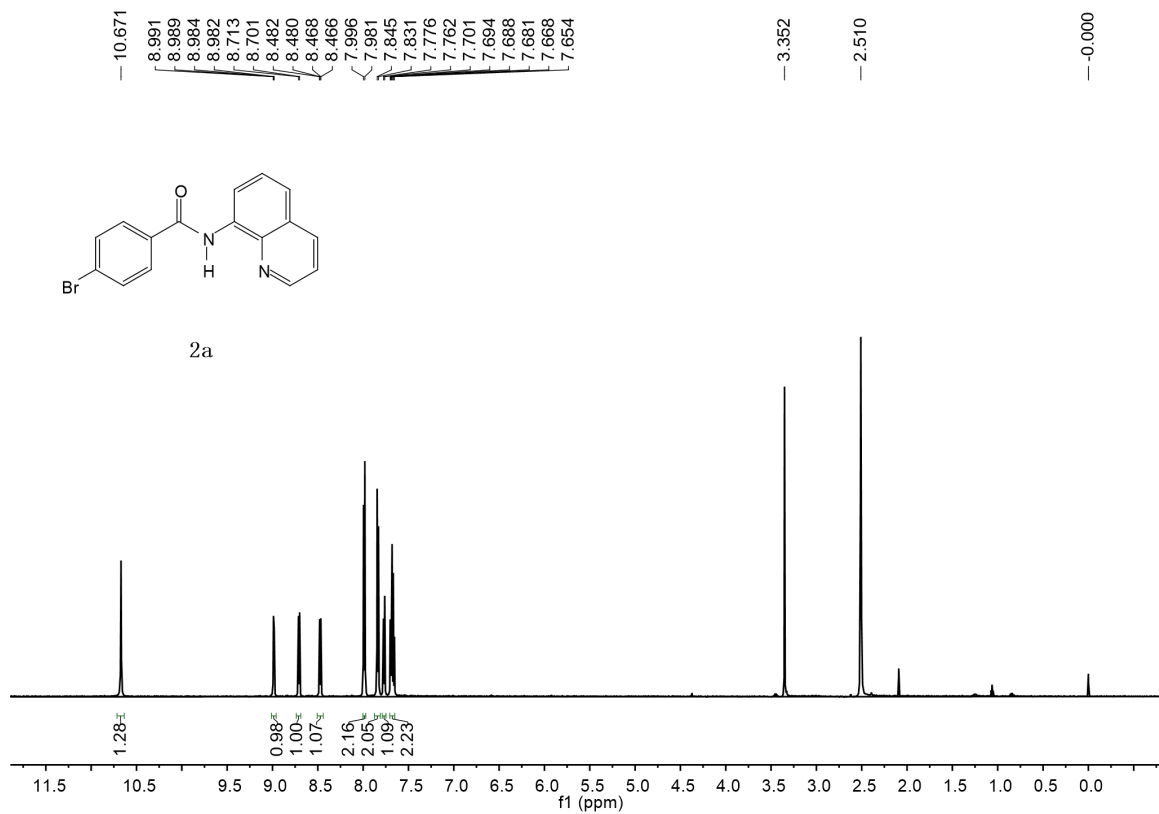


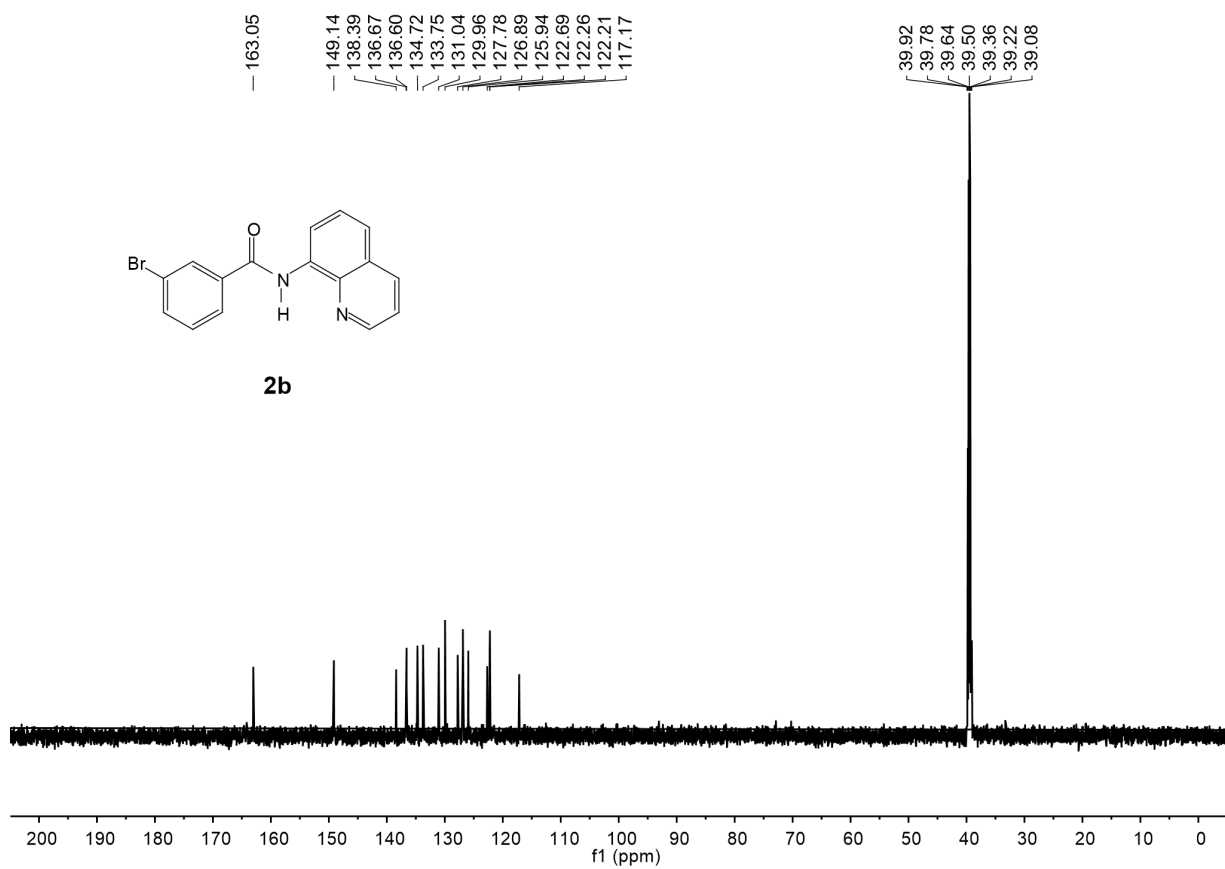
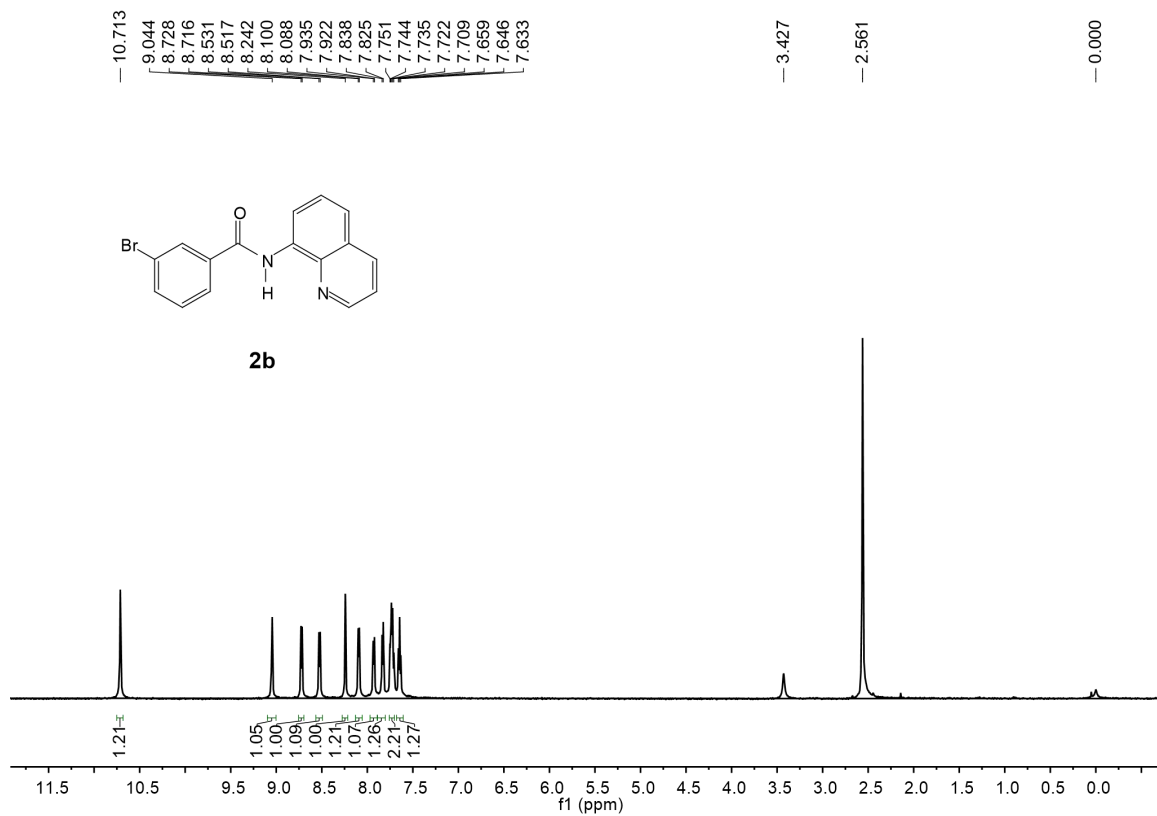


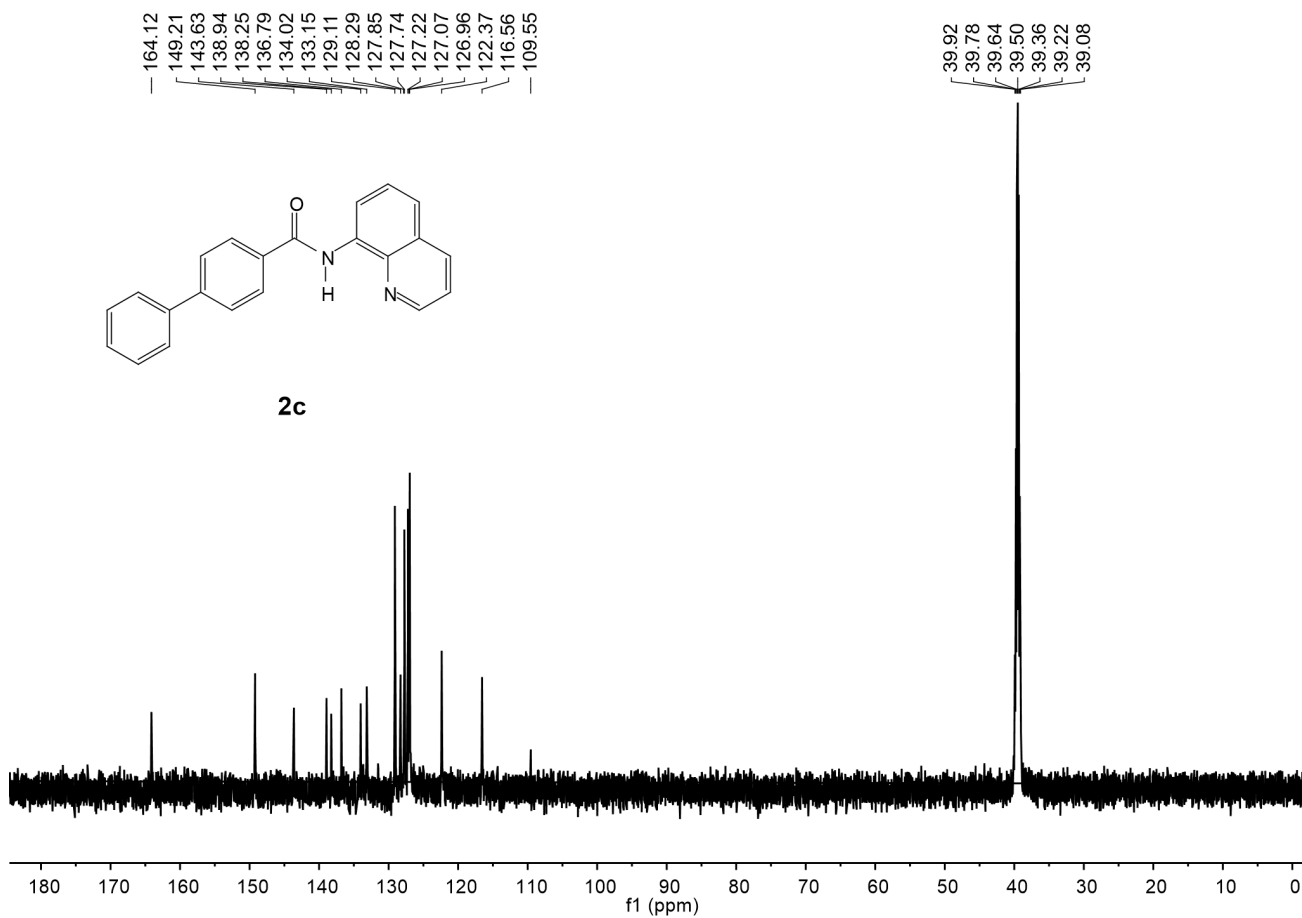
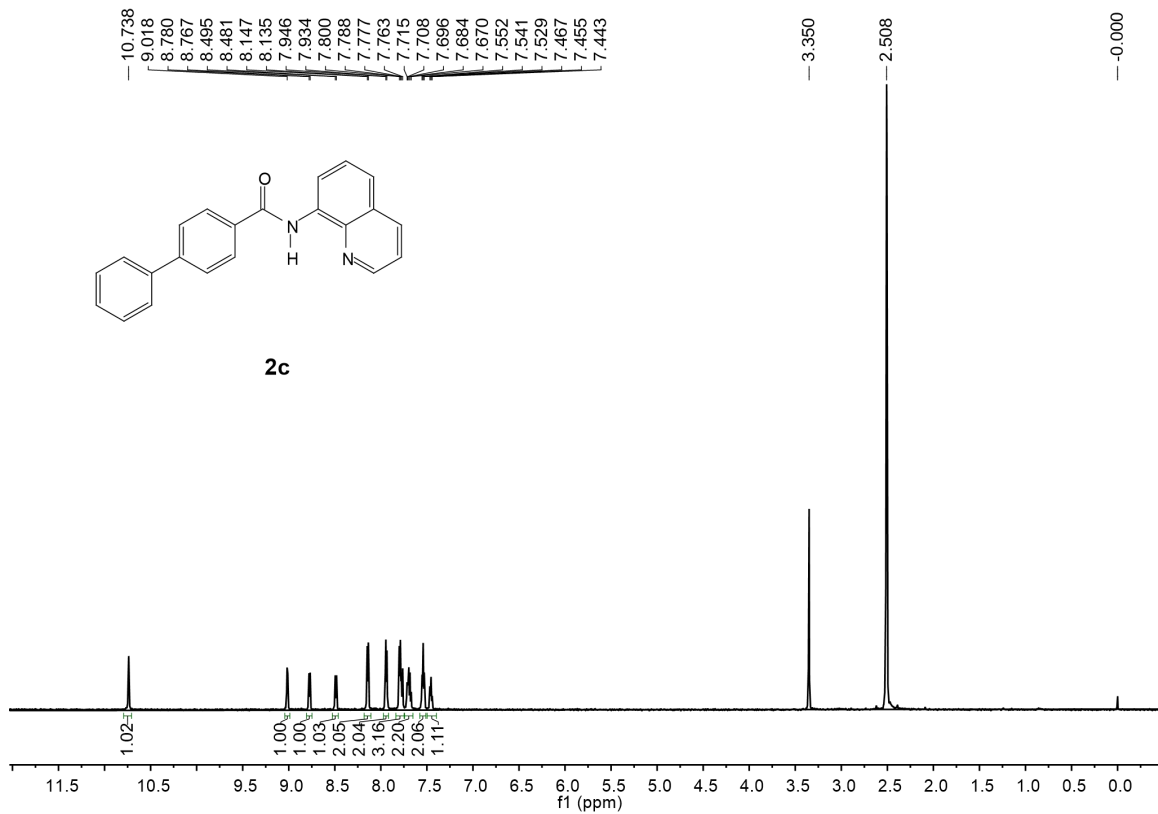


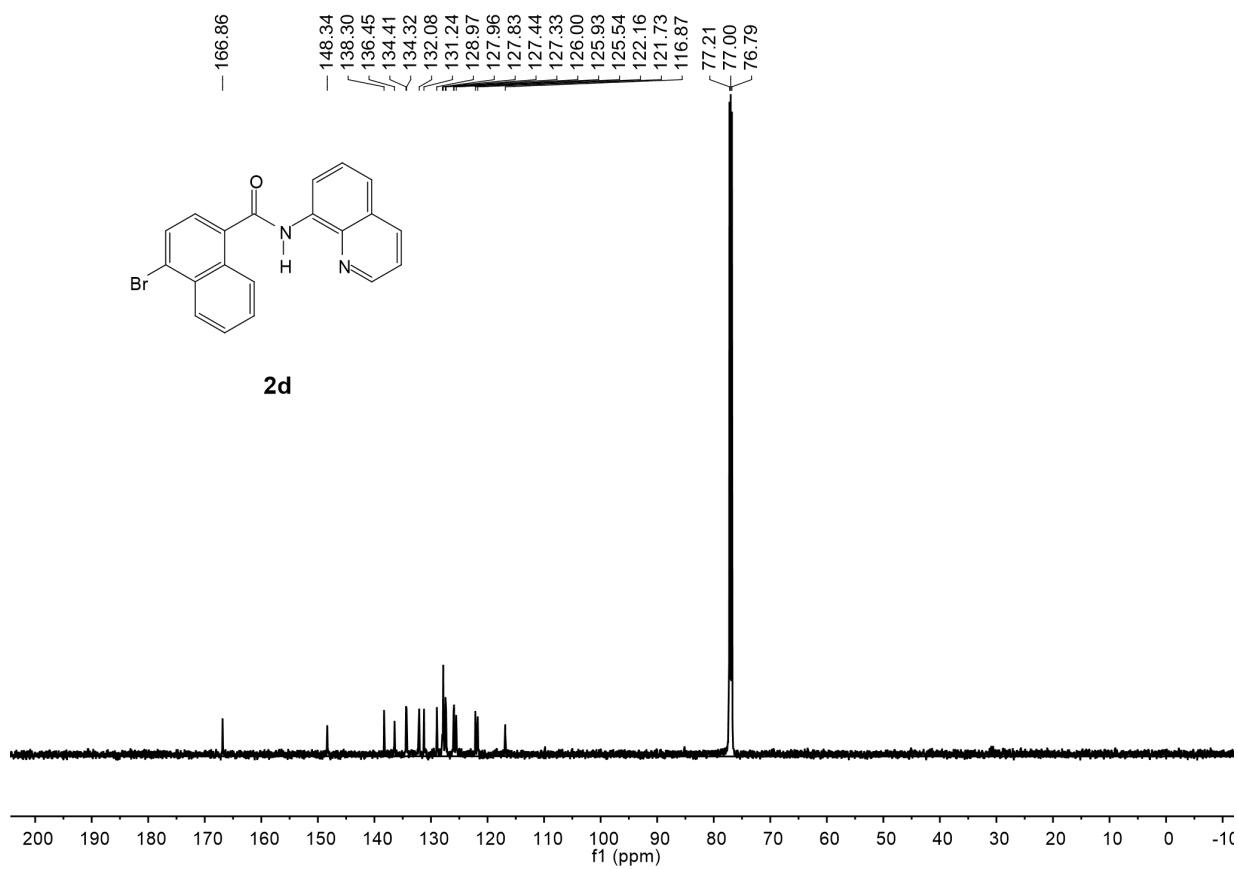
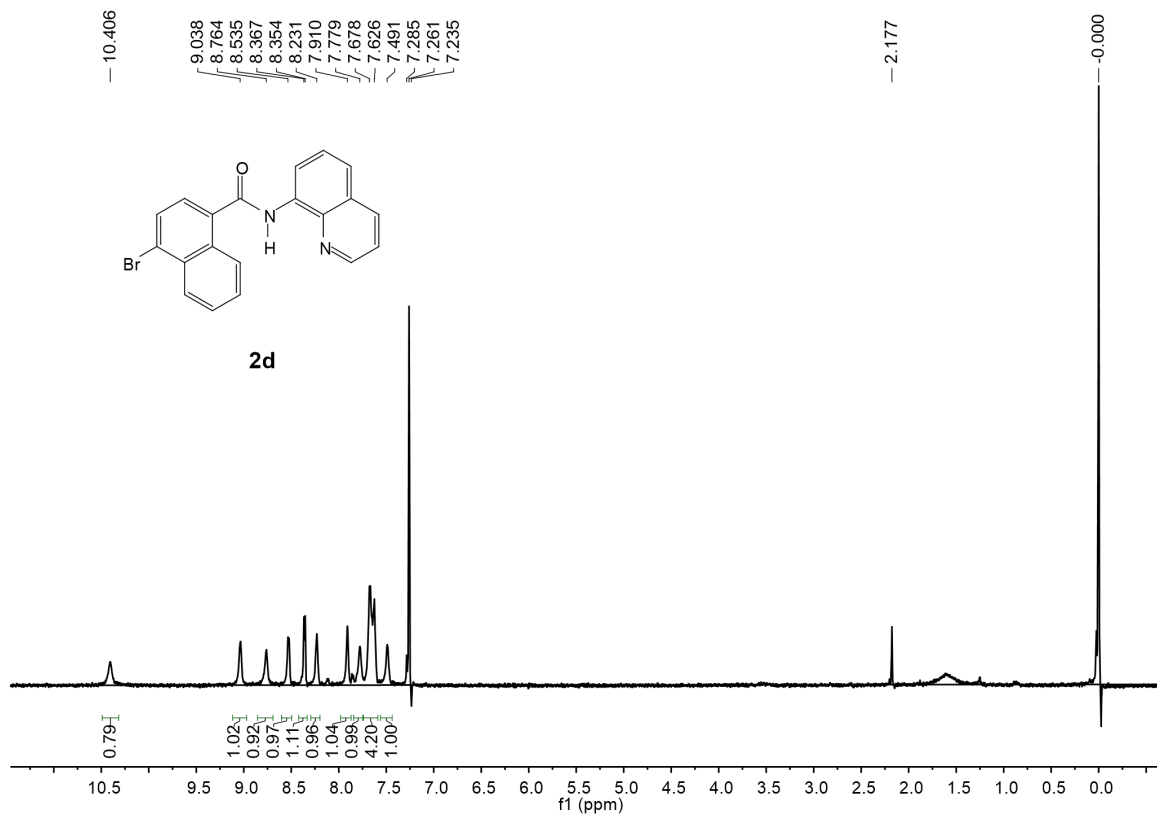


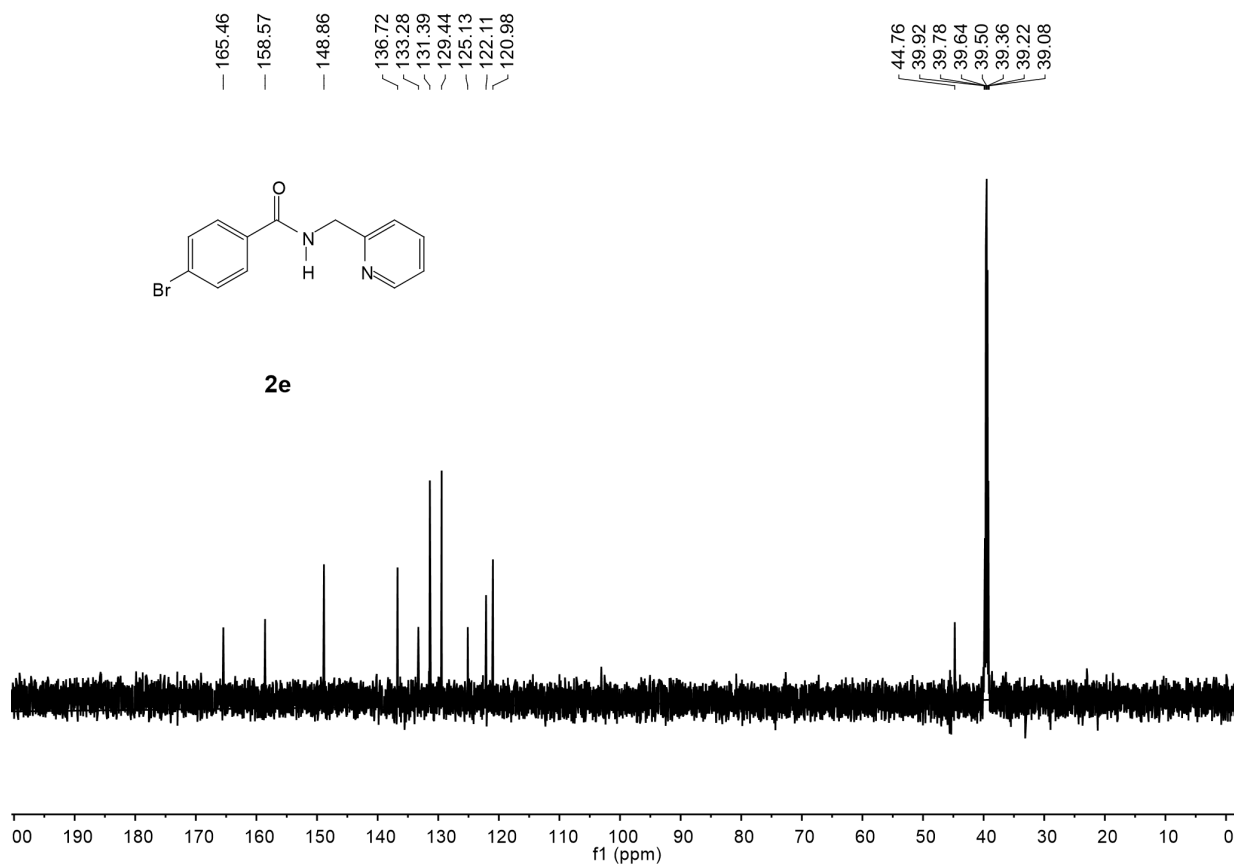
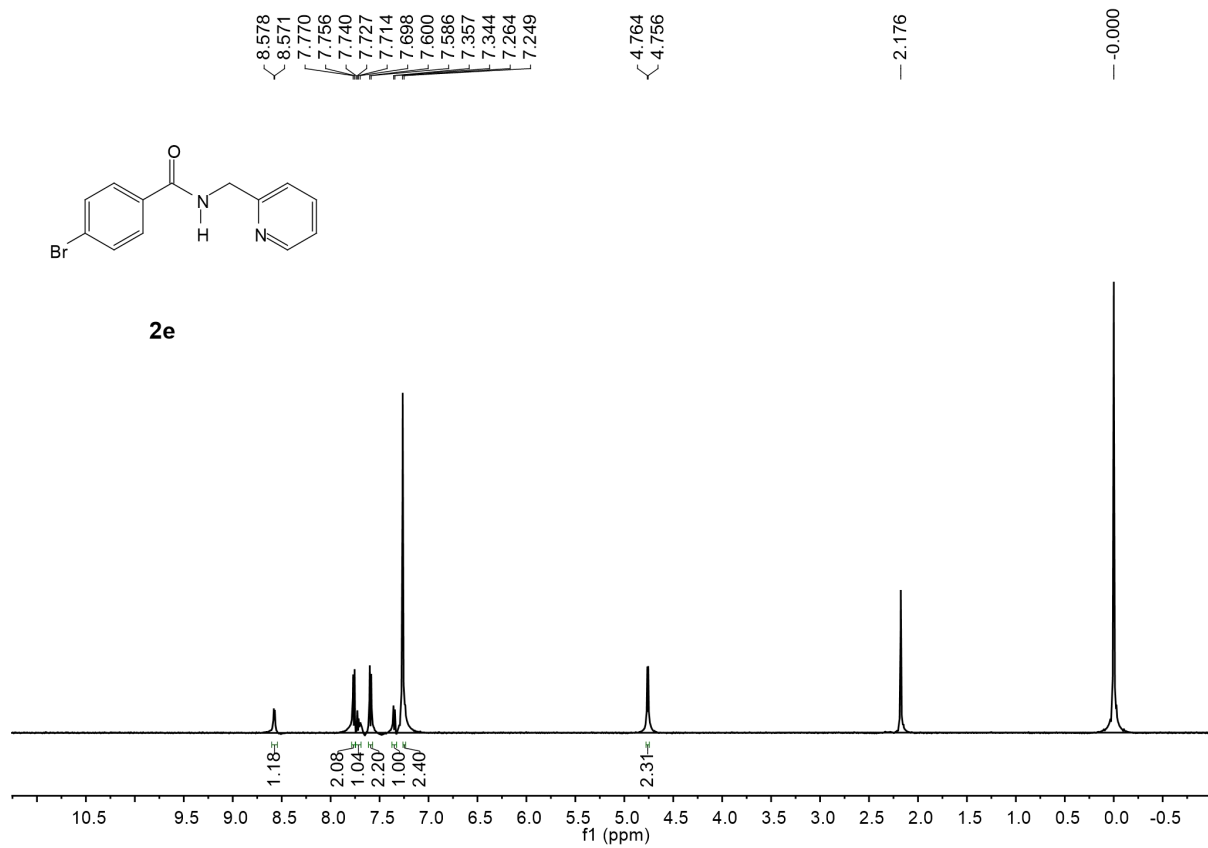


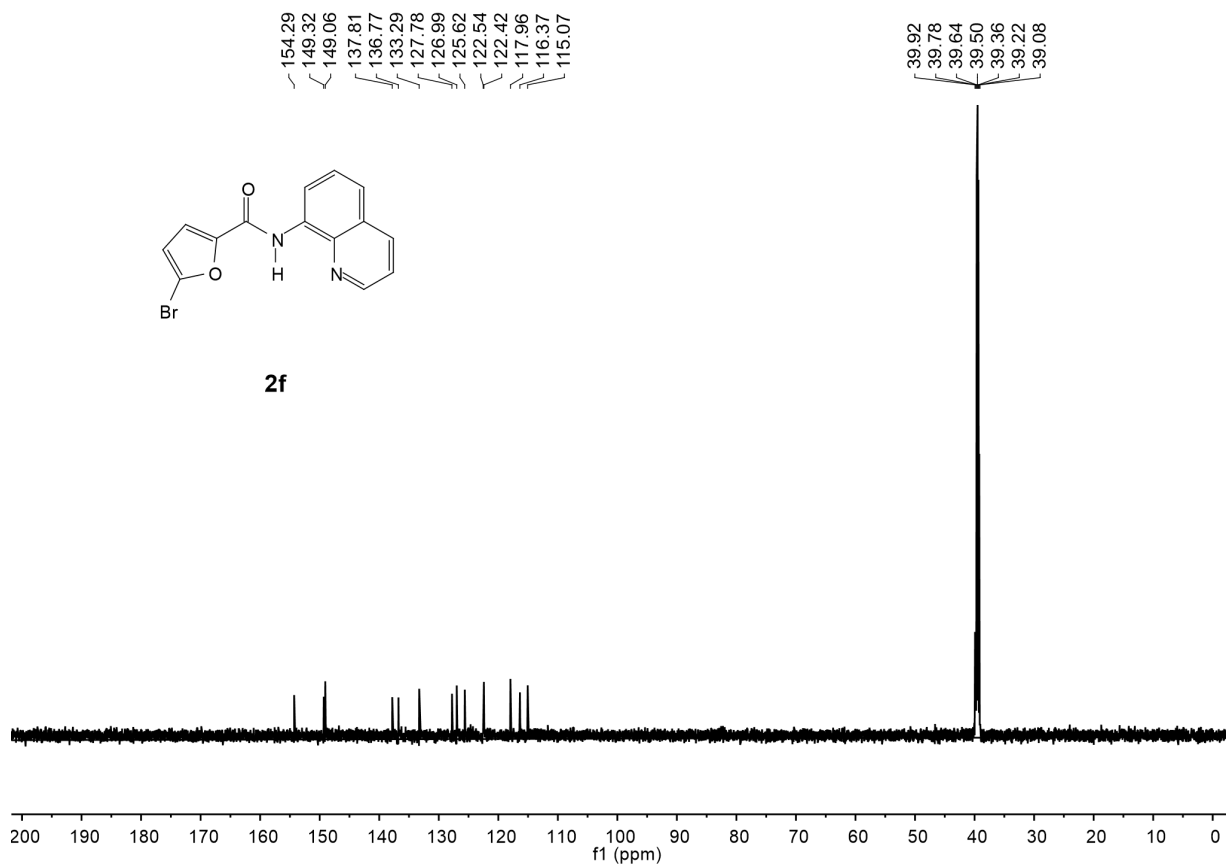
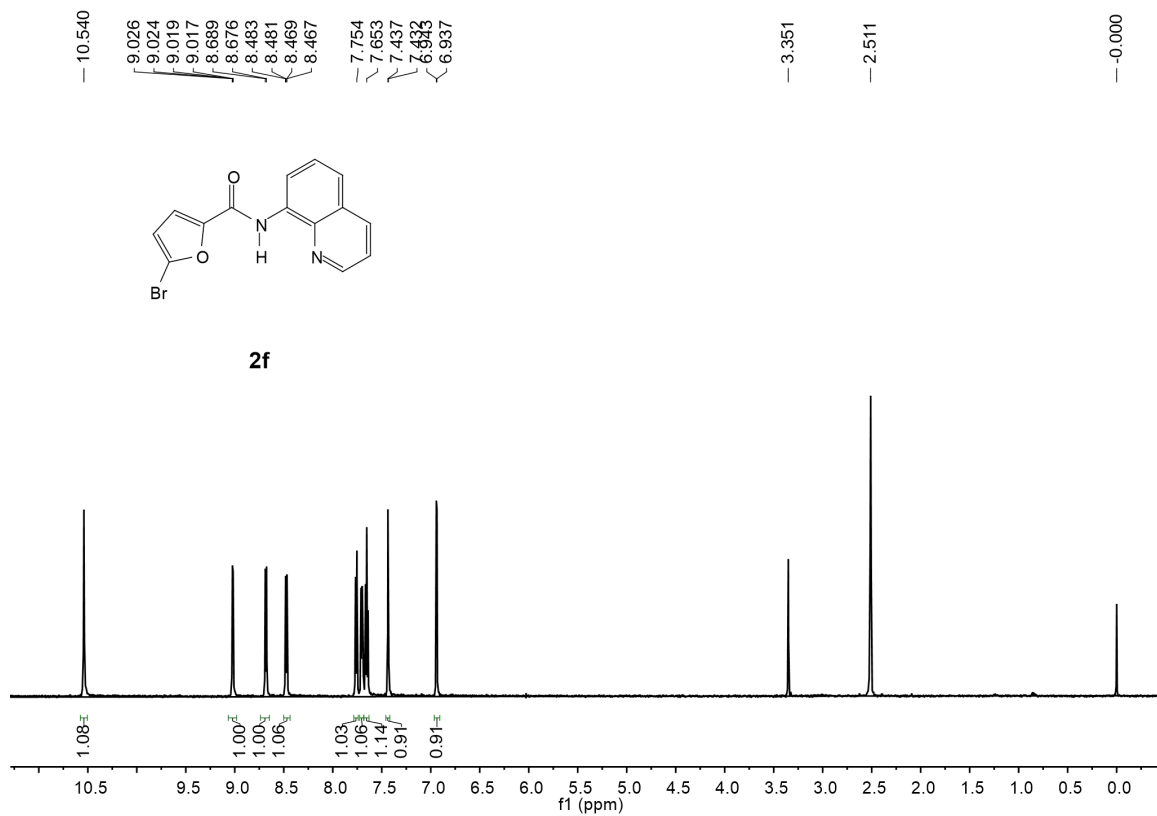


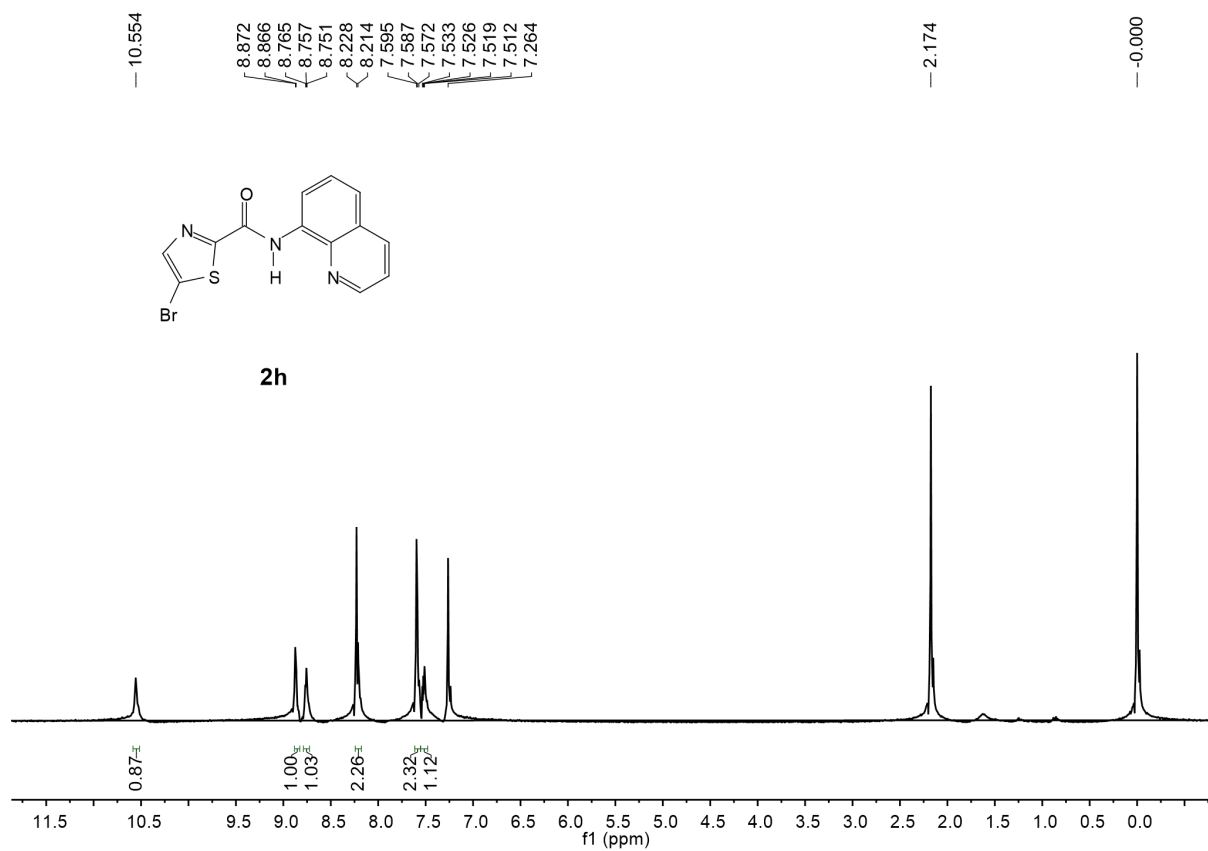
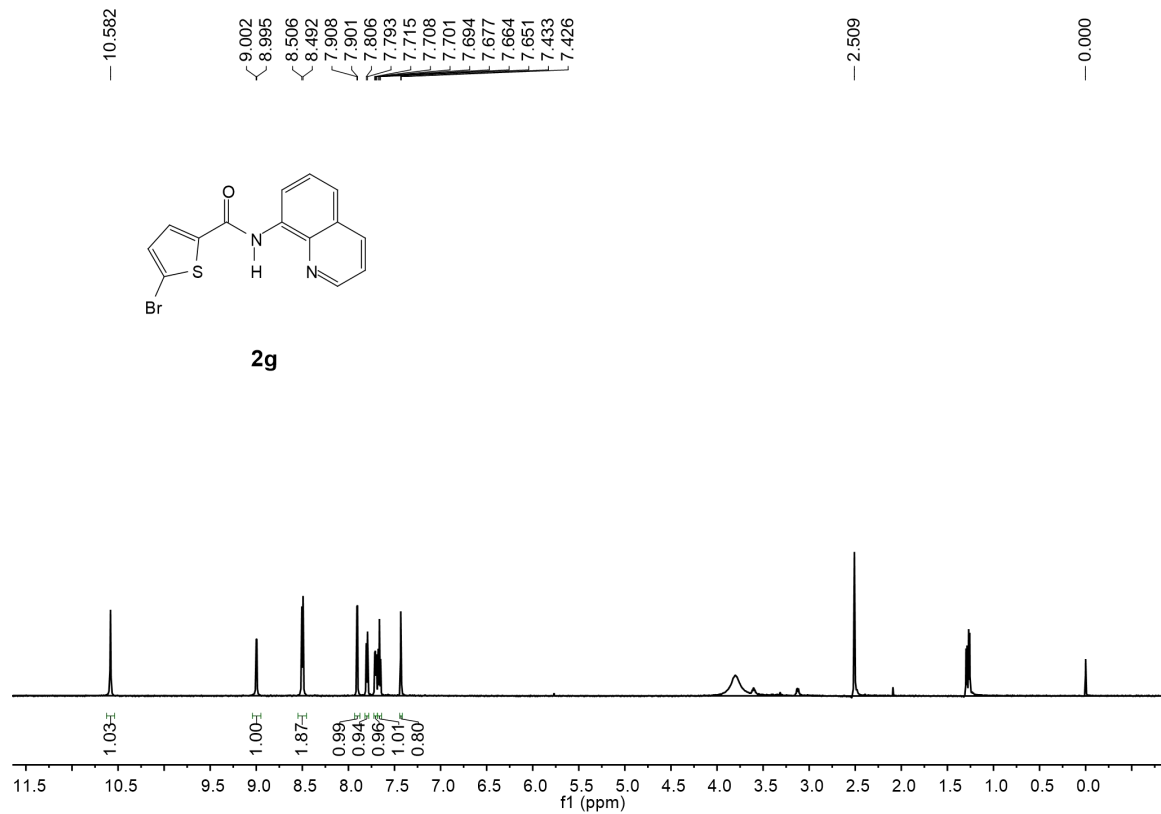


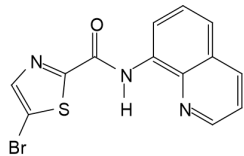




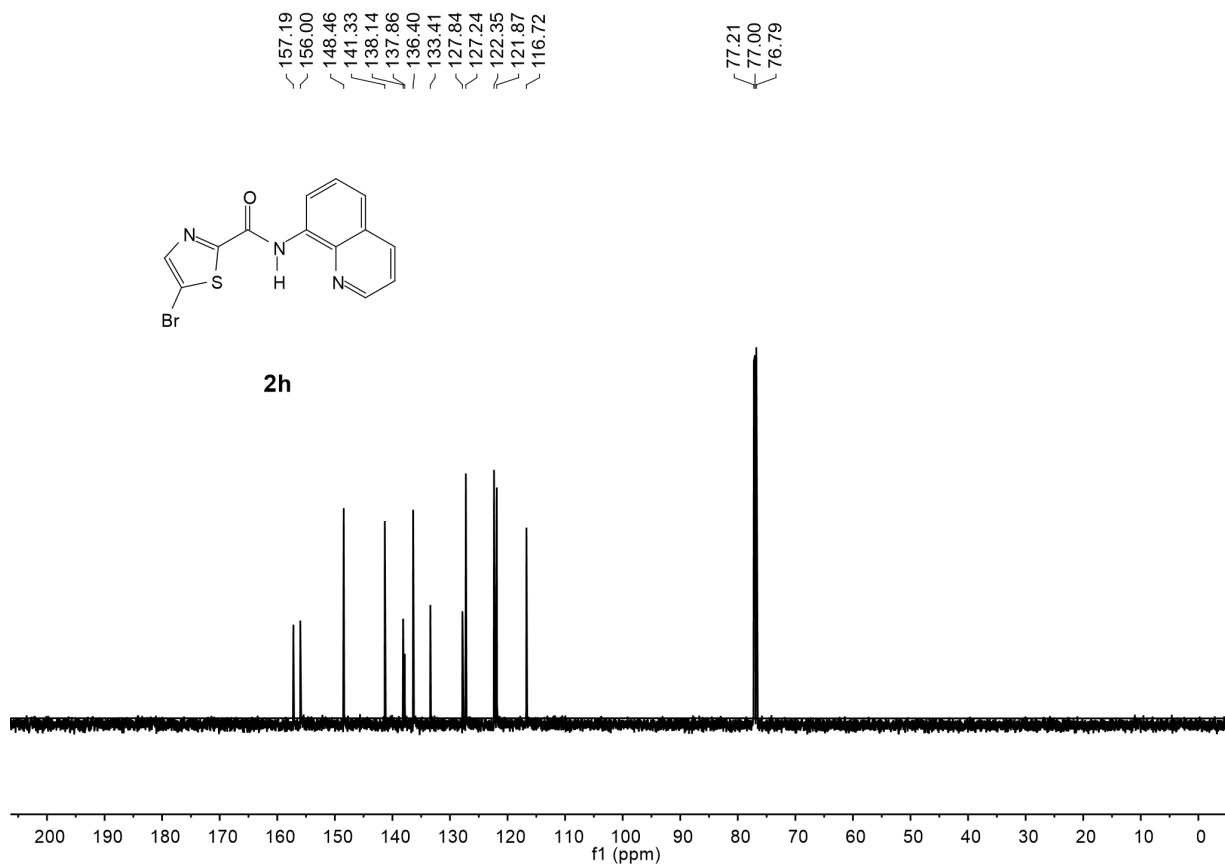


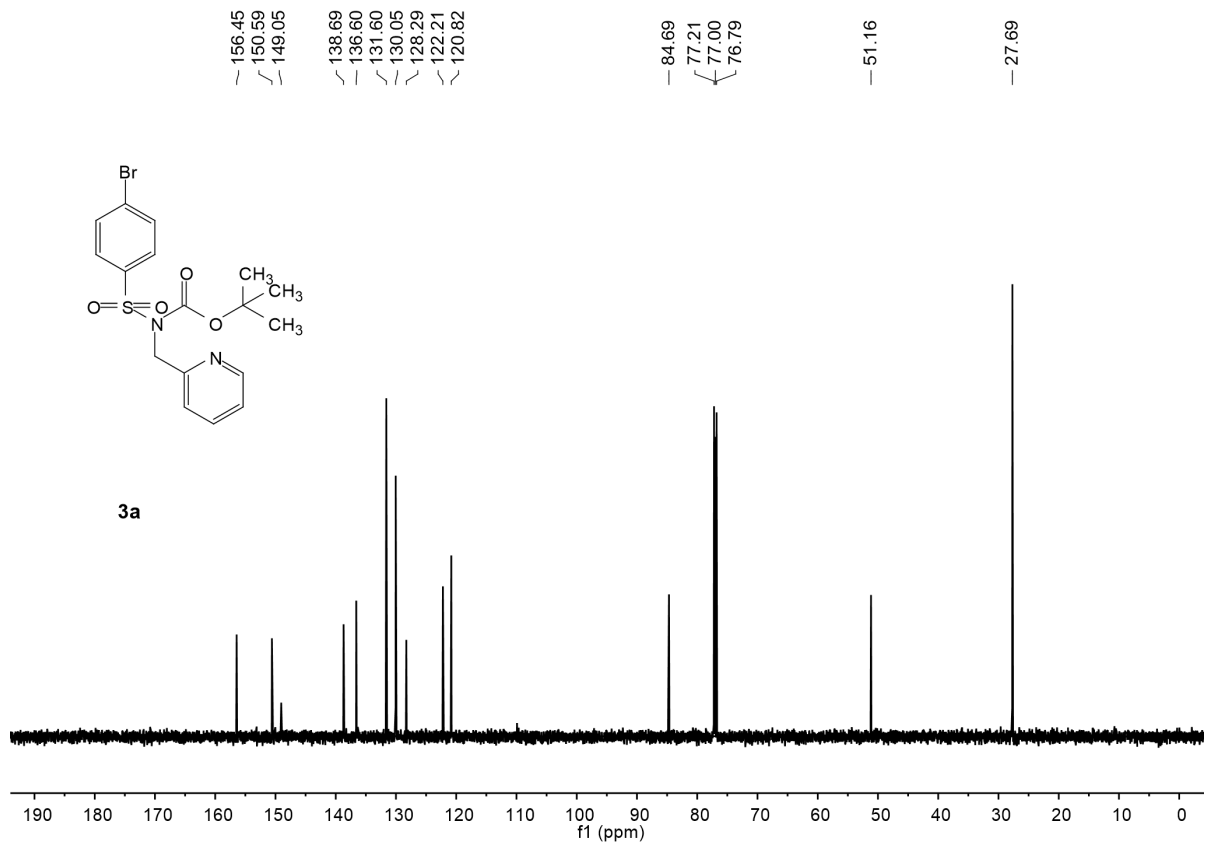
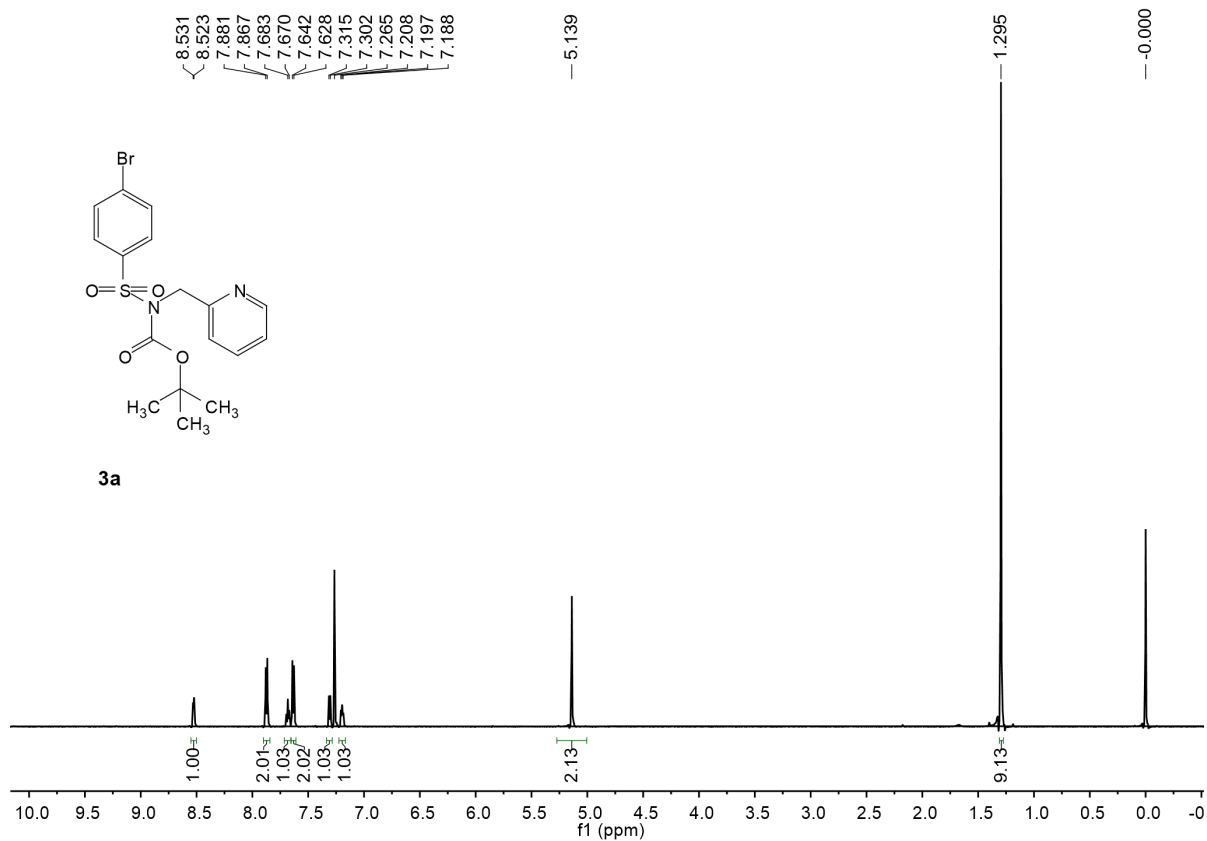


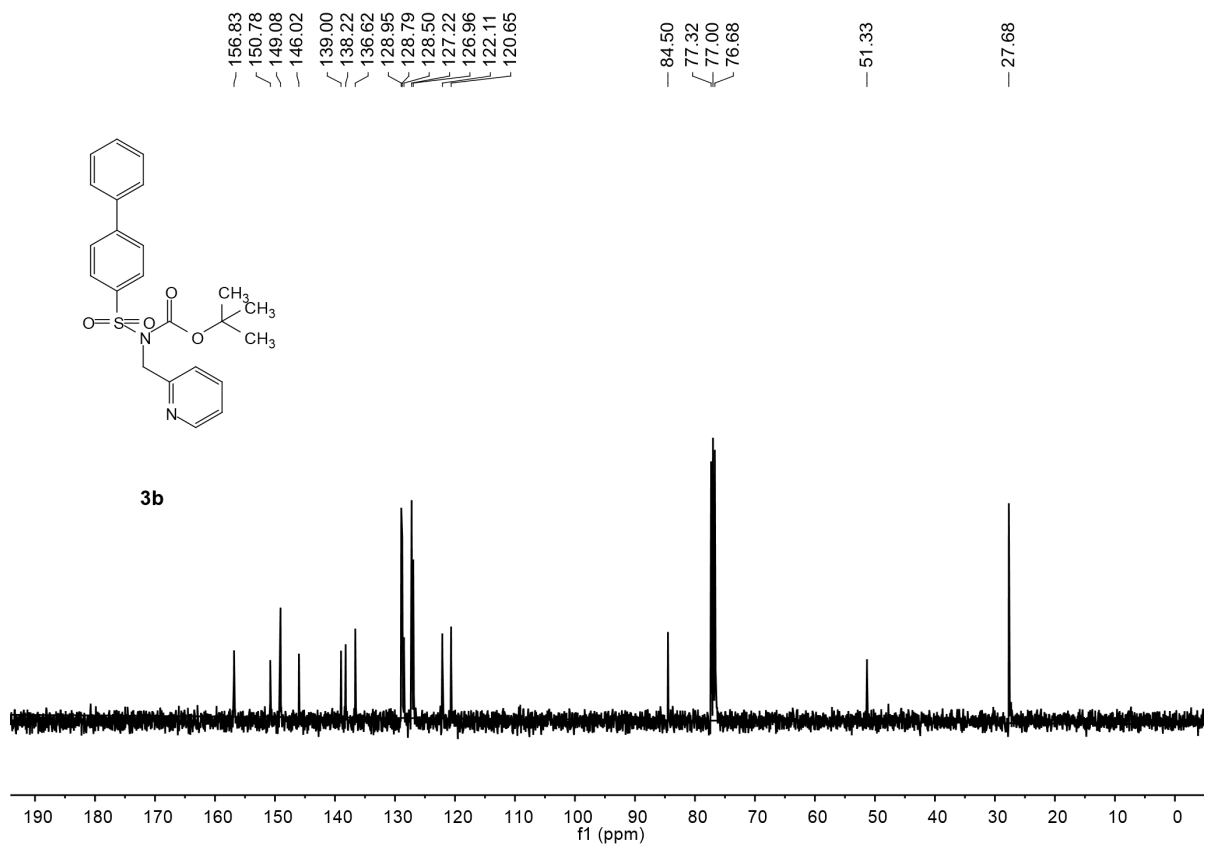
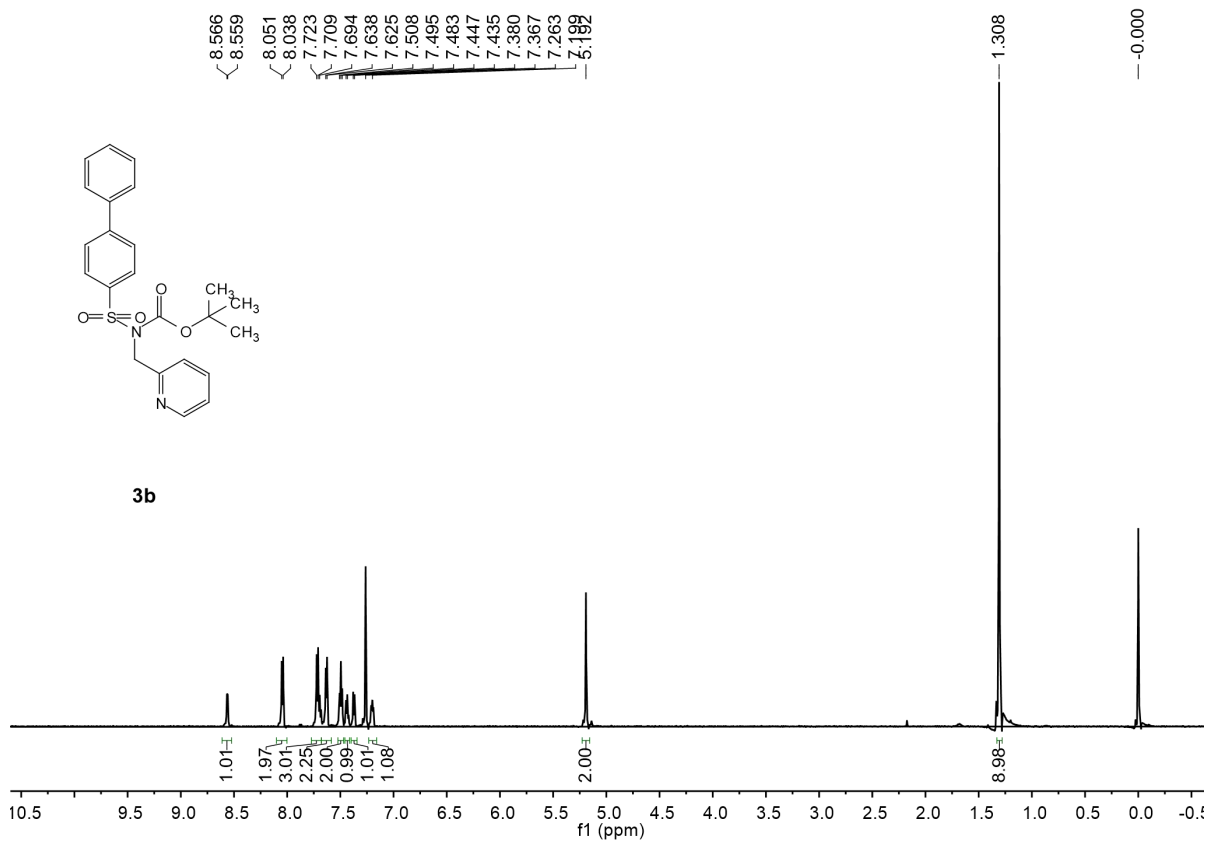


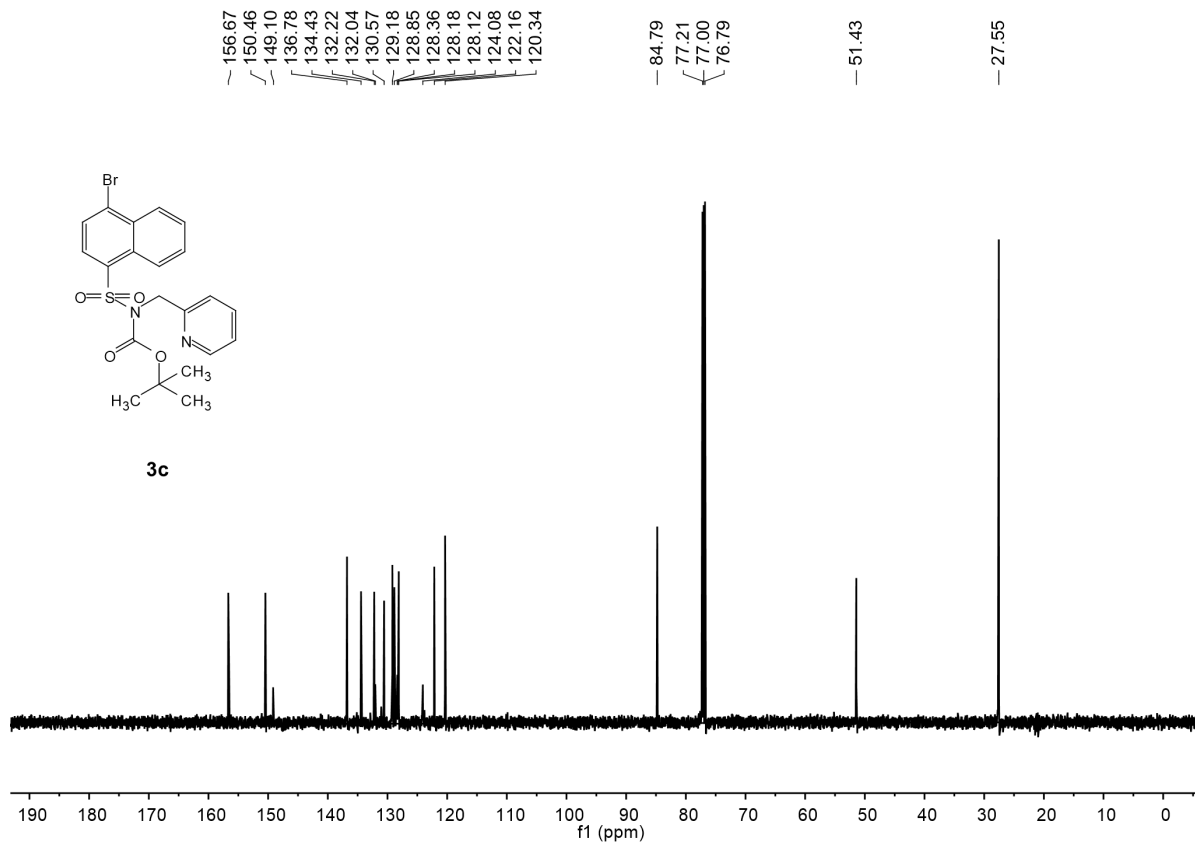
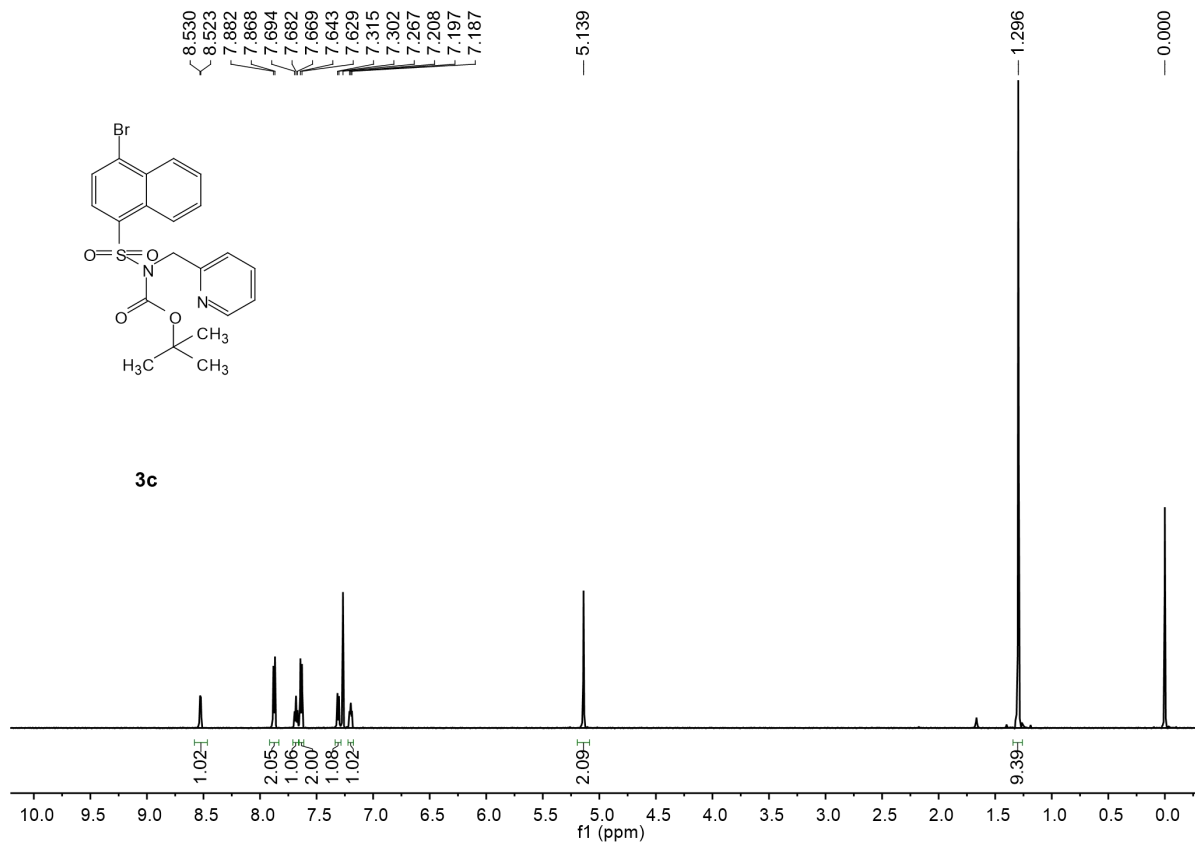


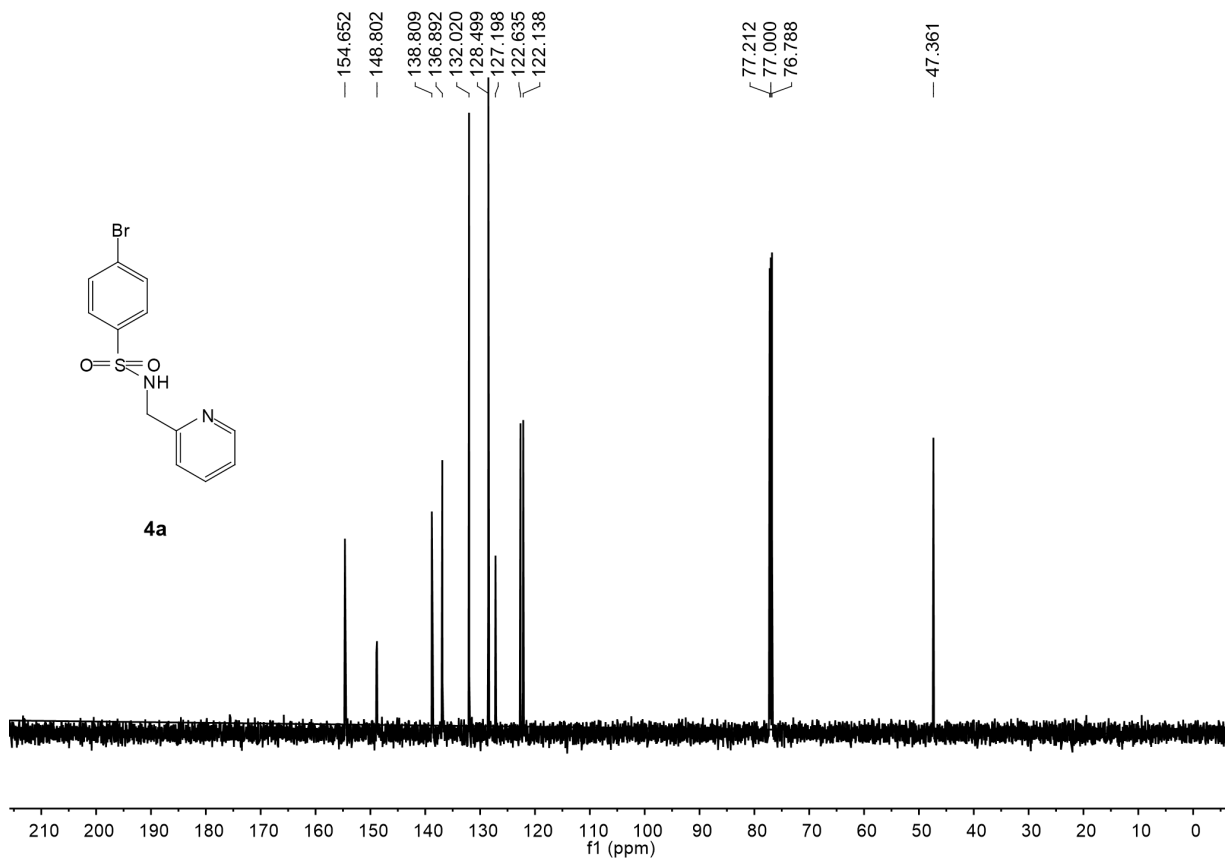
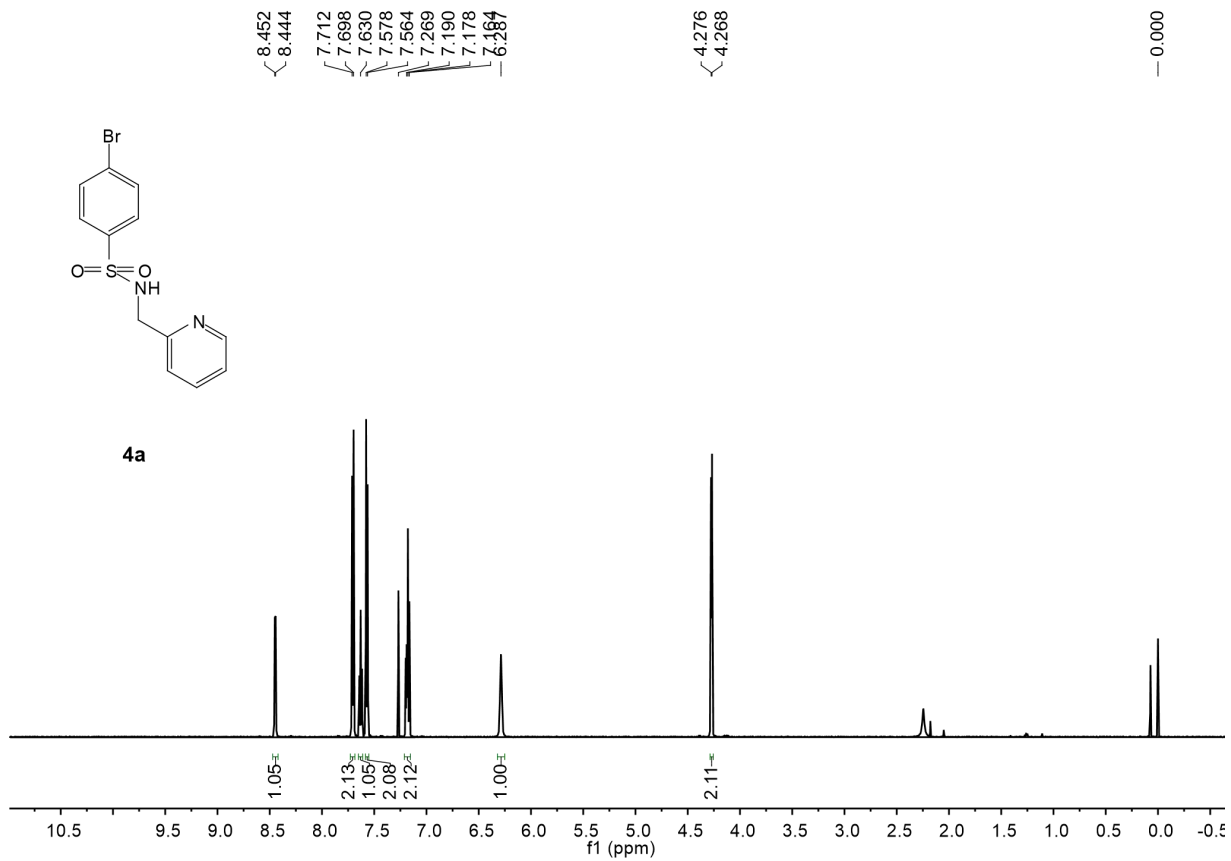
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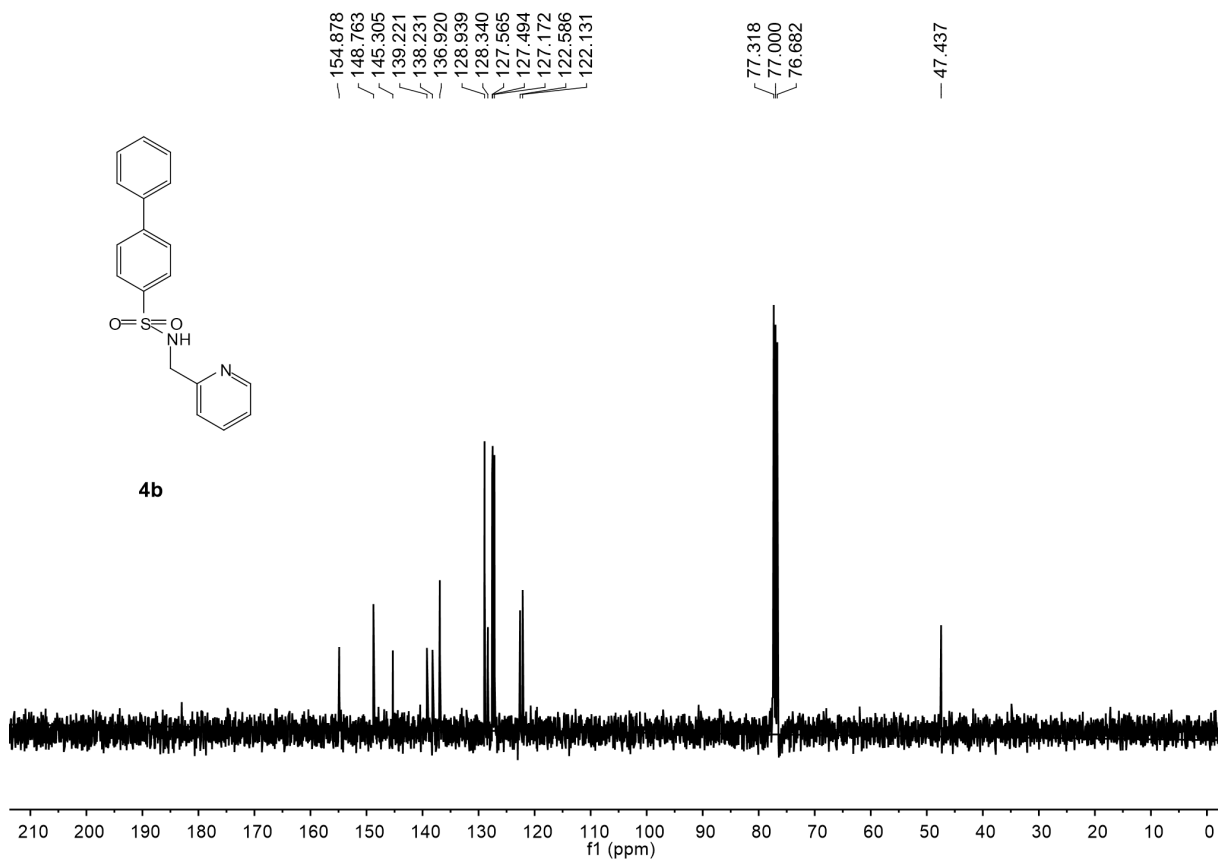
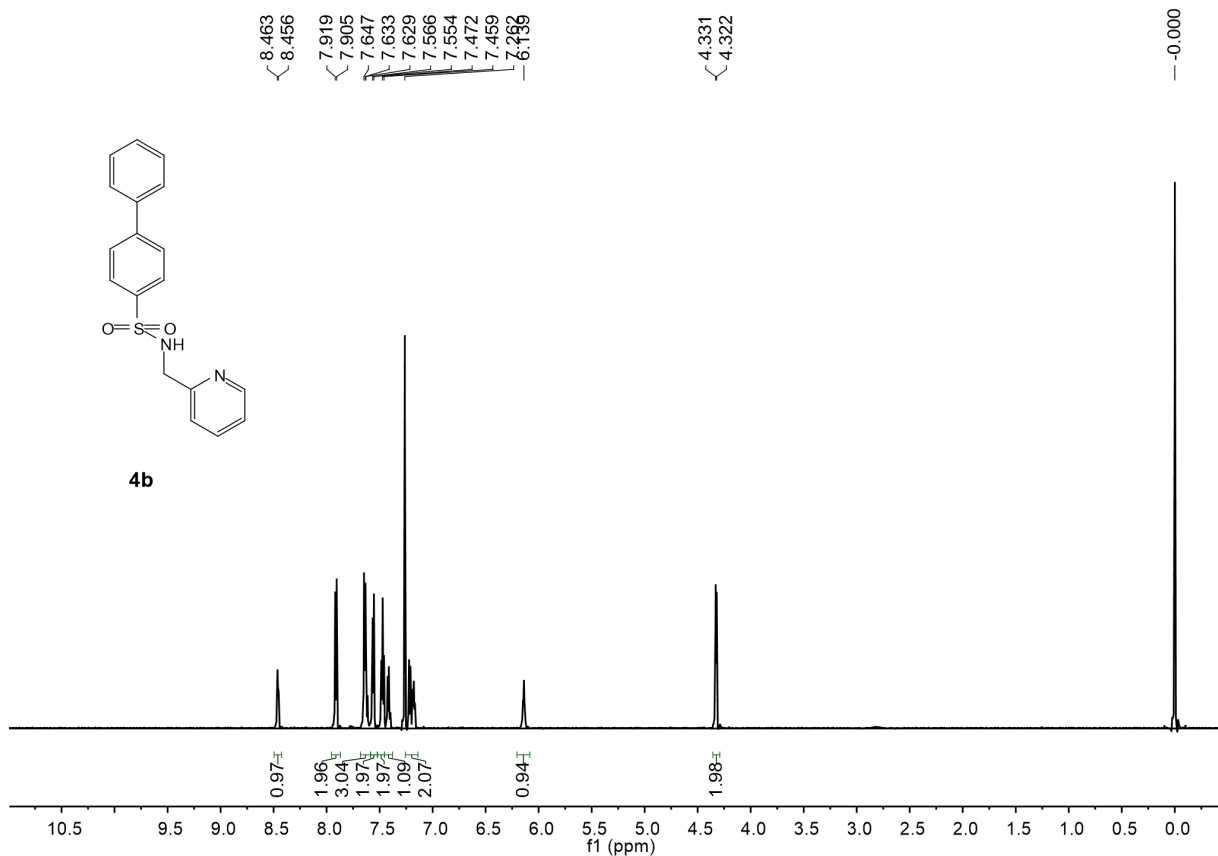


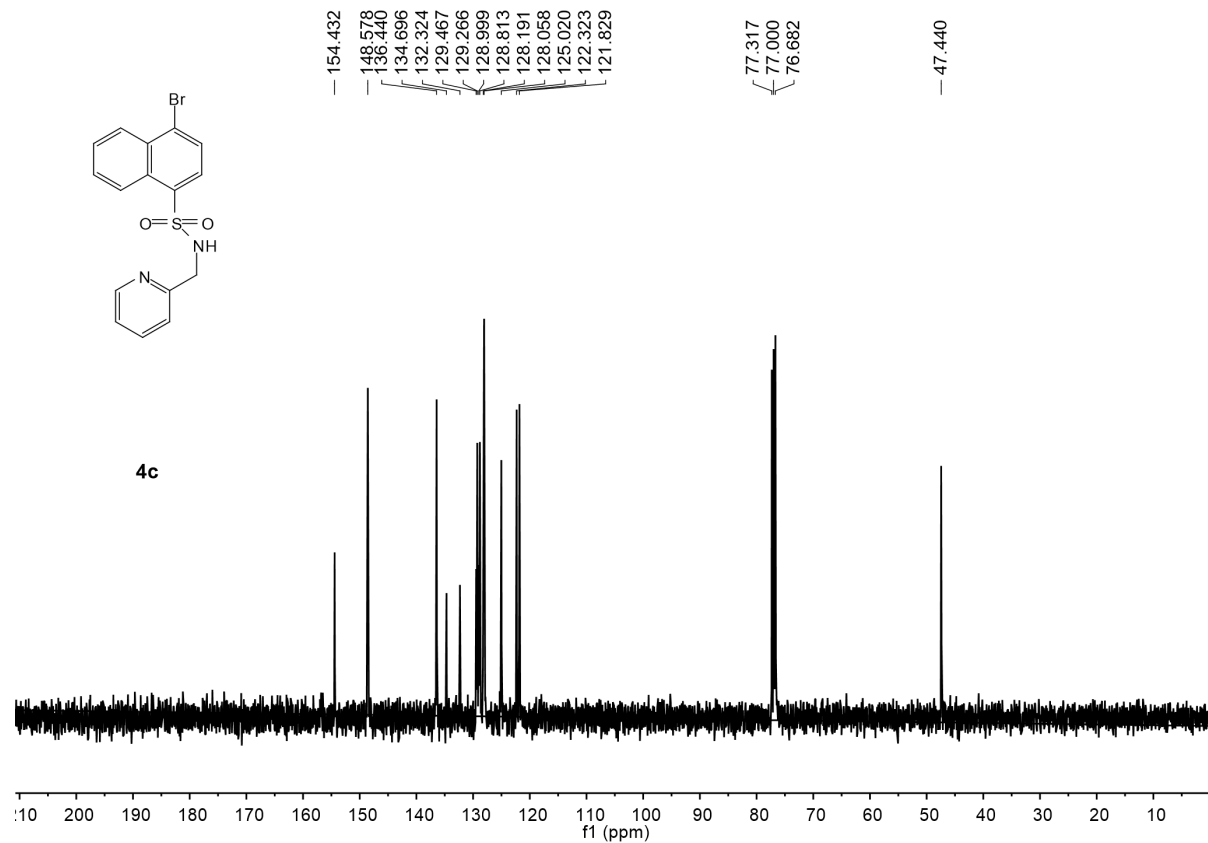
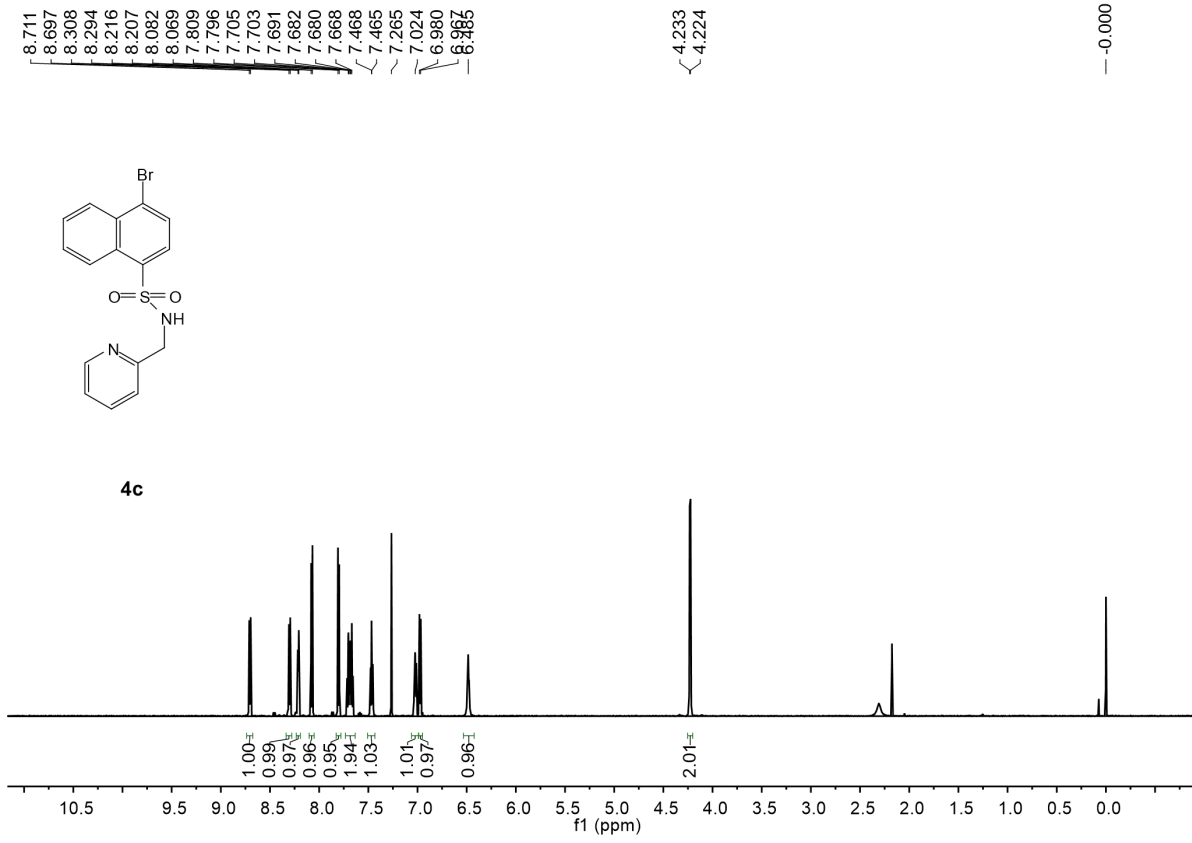


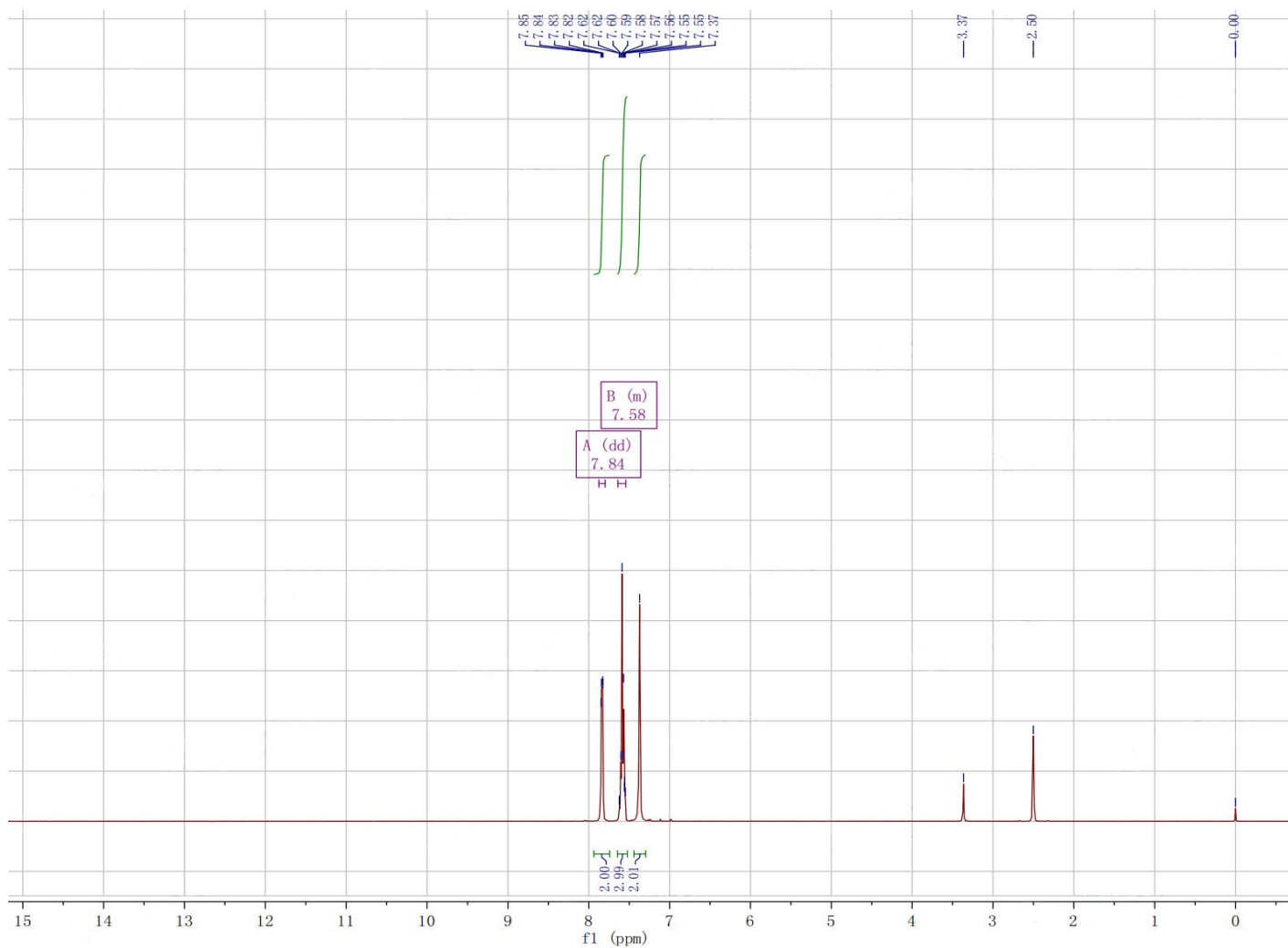




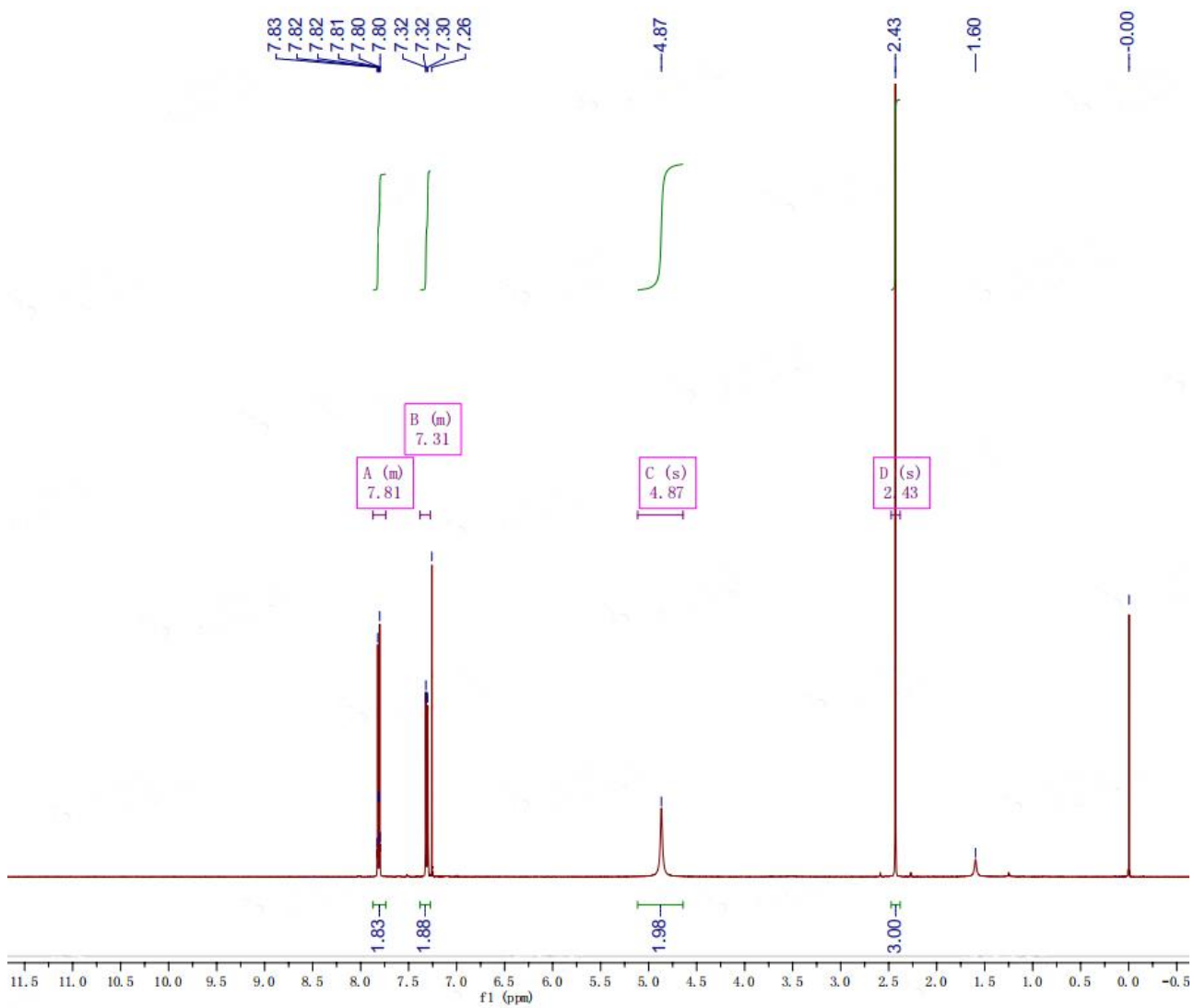




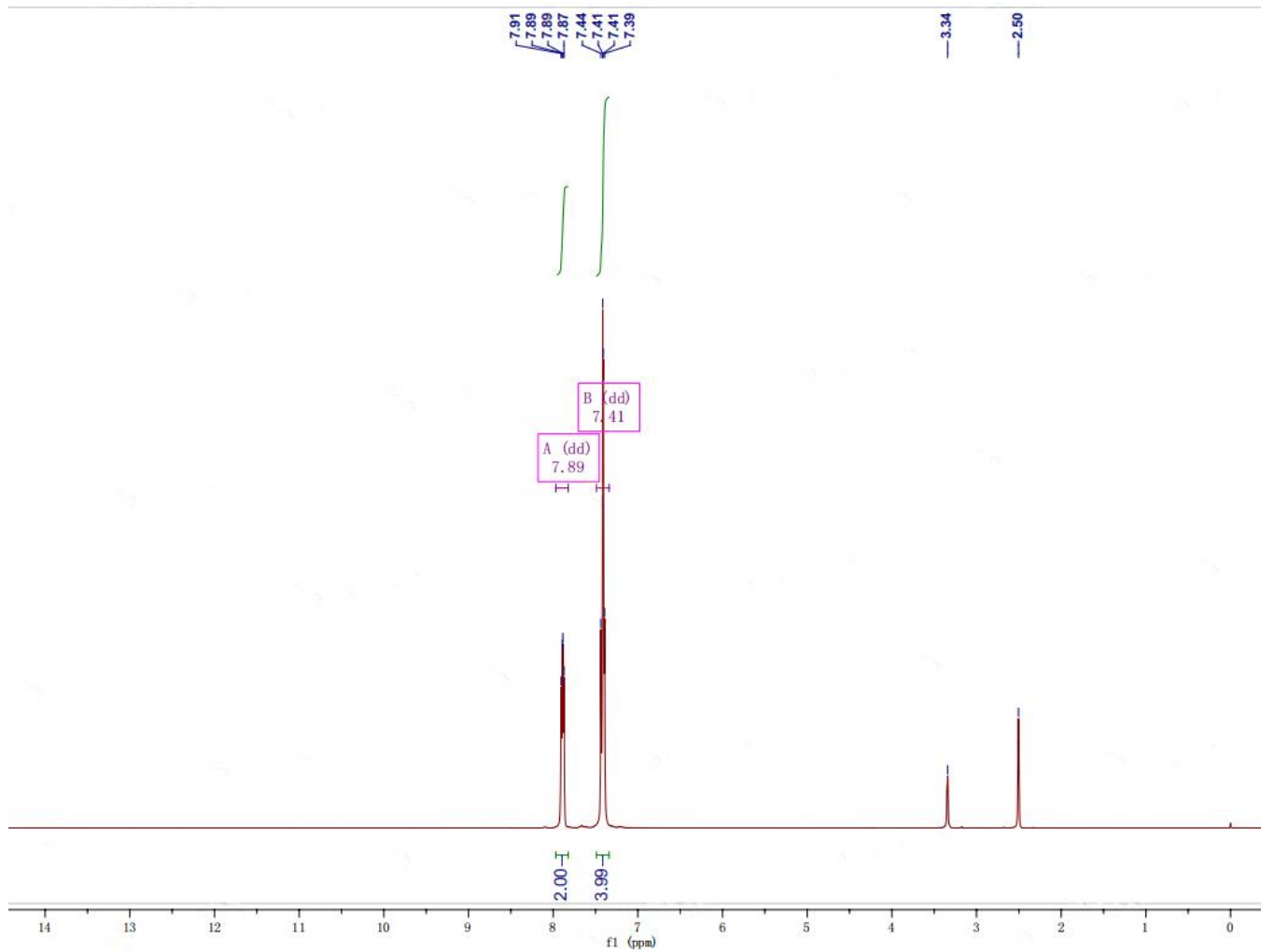




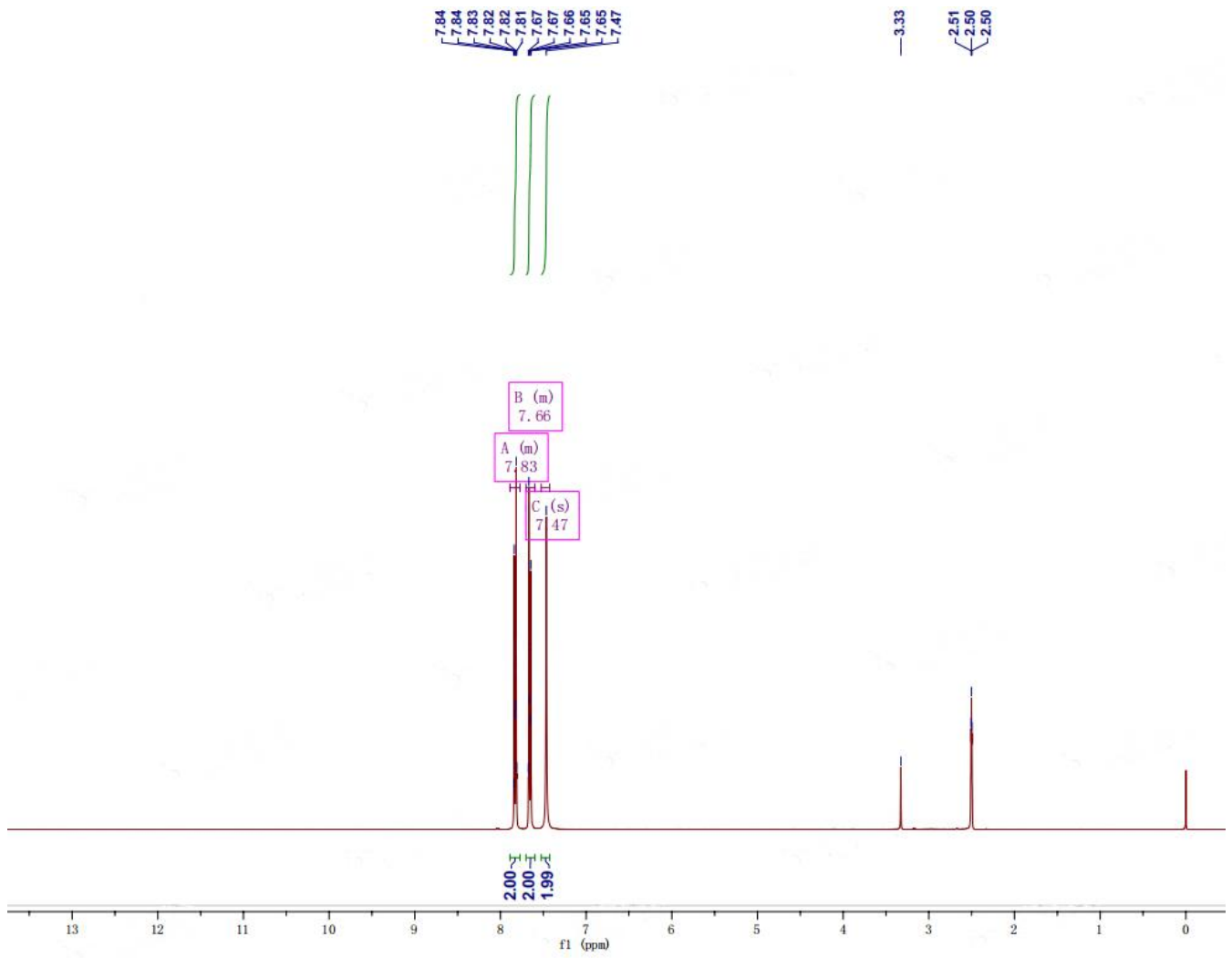
4d, $^1\text{H-NMR}$



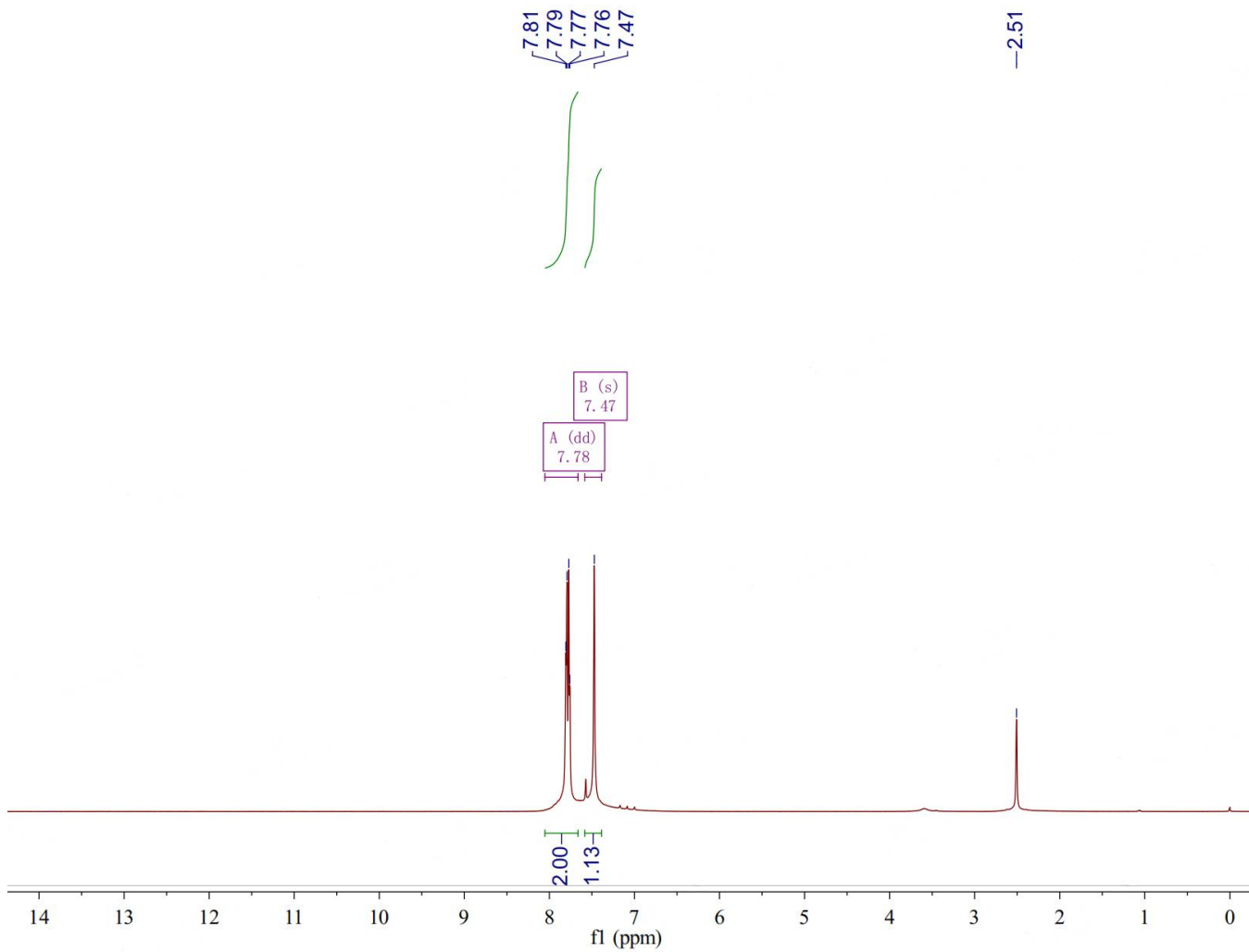
4e, $^1\text{H-NMR}$



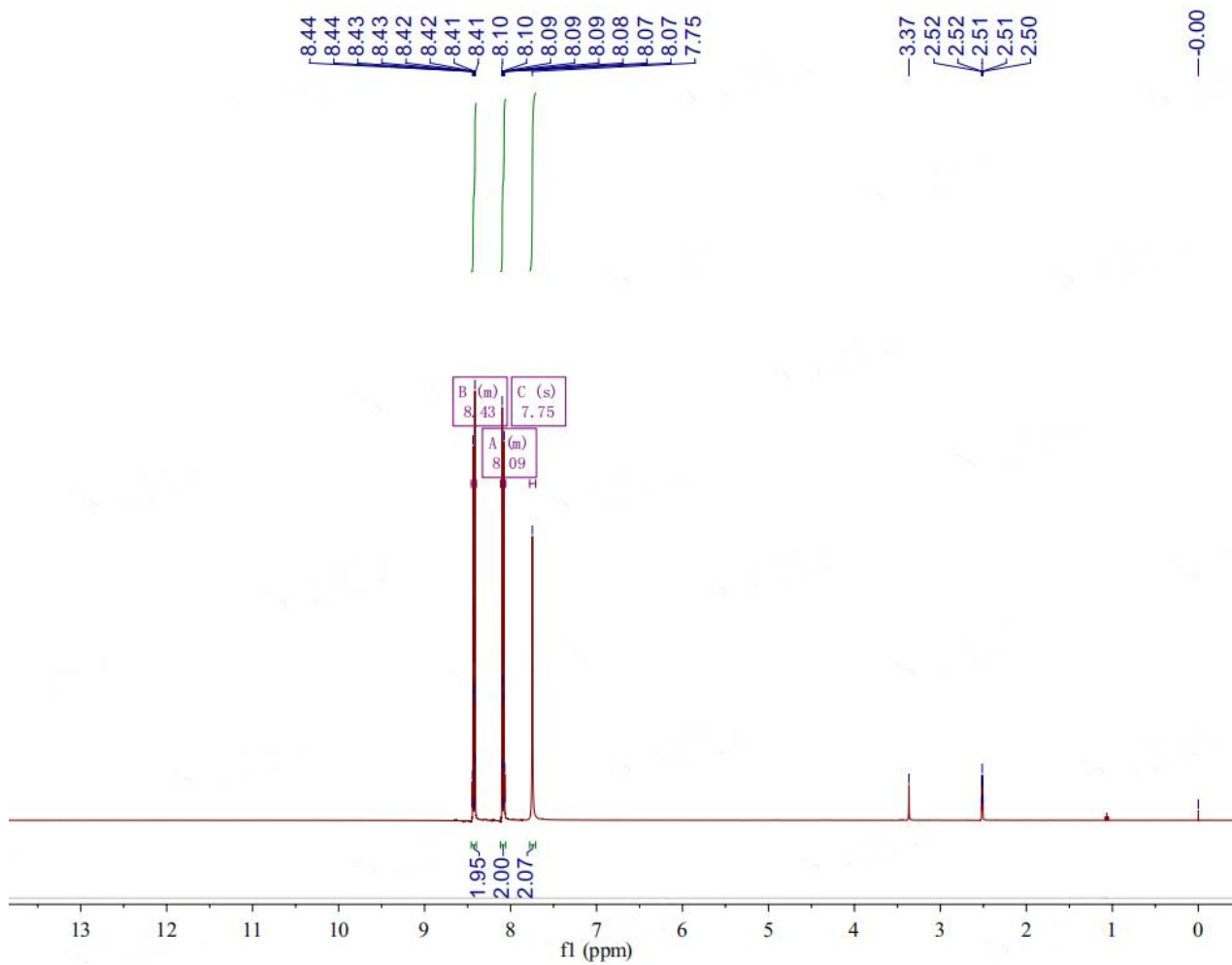
4f, $^1\text{H-NMR}$



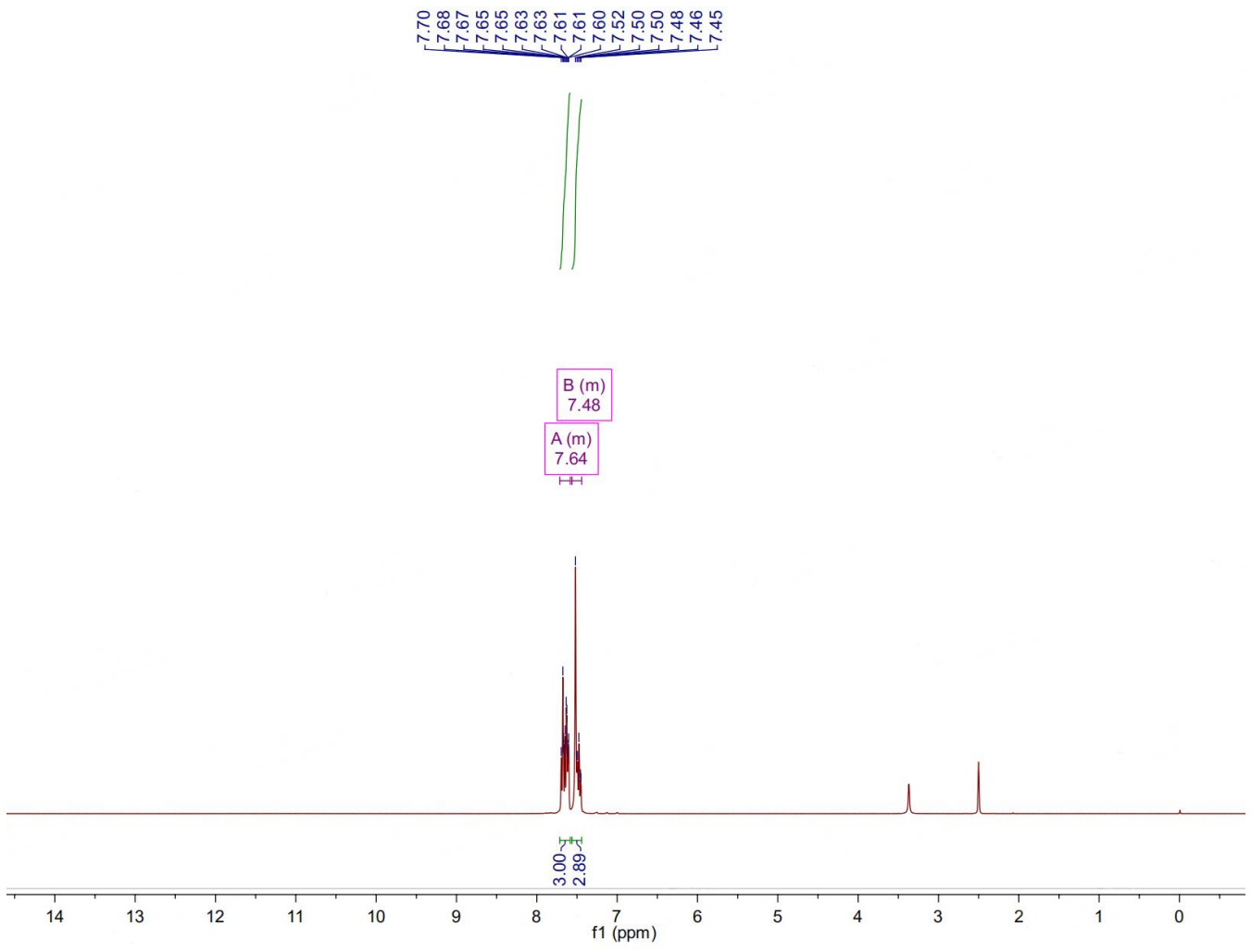
4g, ¹H-NMR



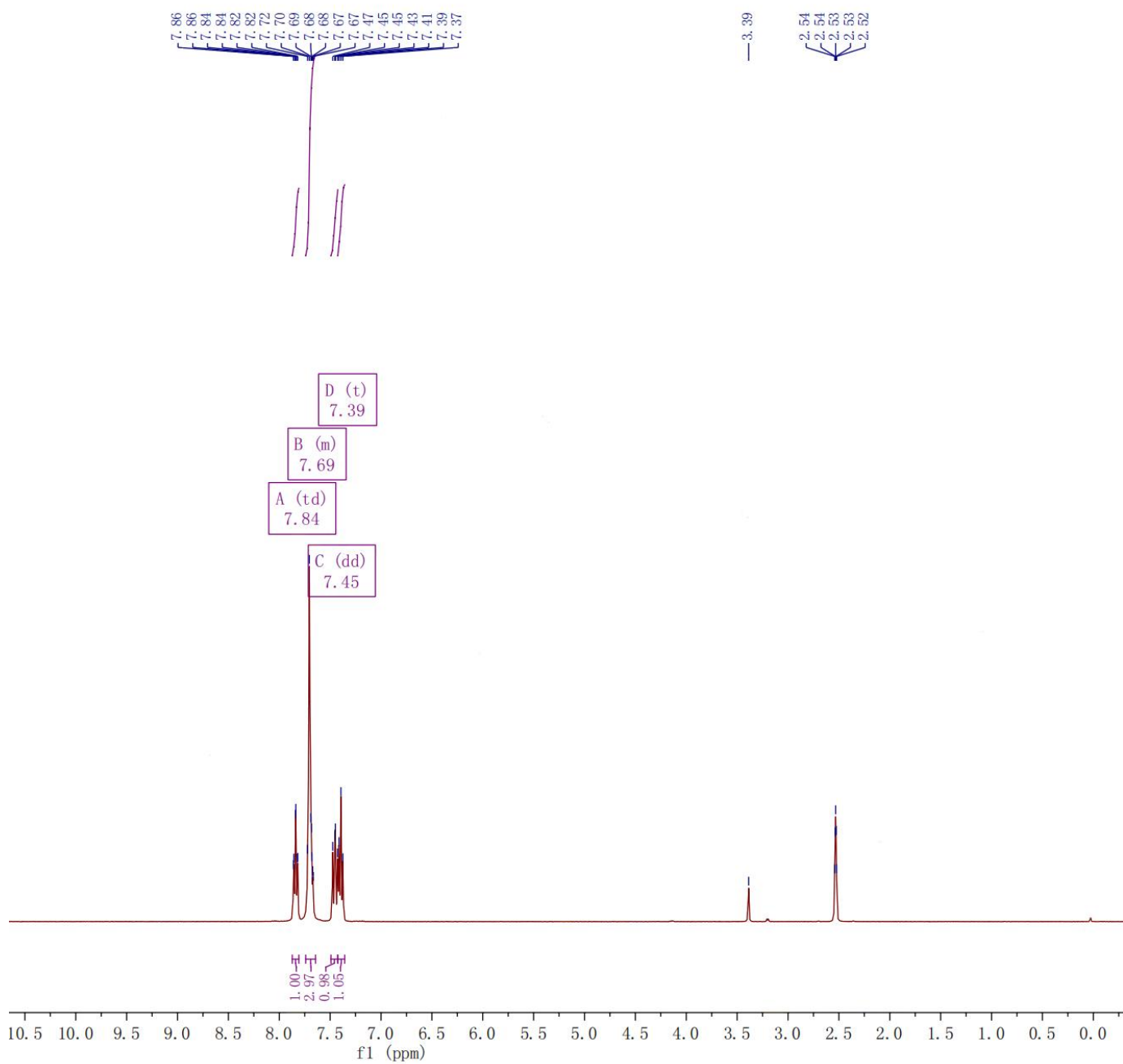
4h, ¹H-NMR



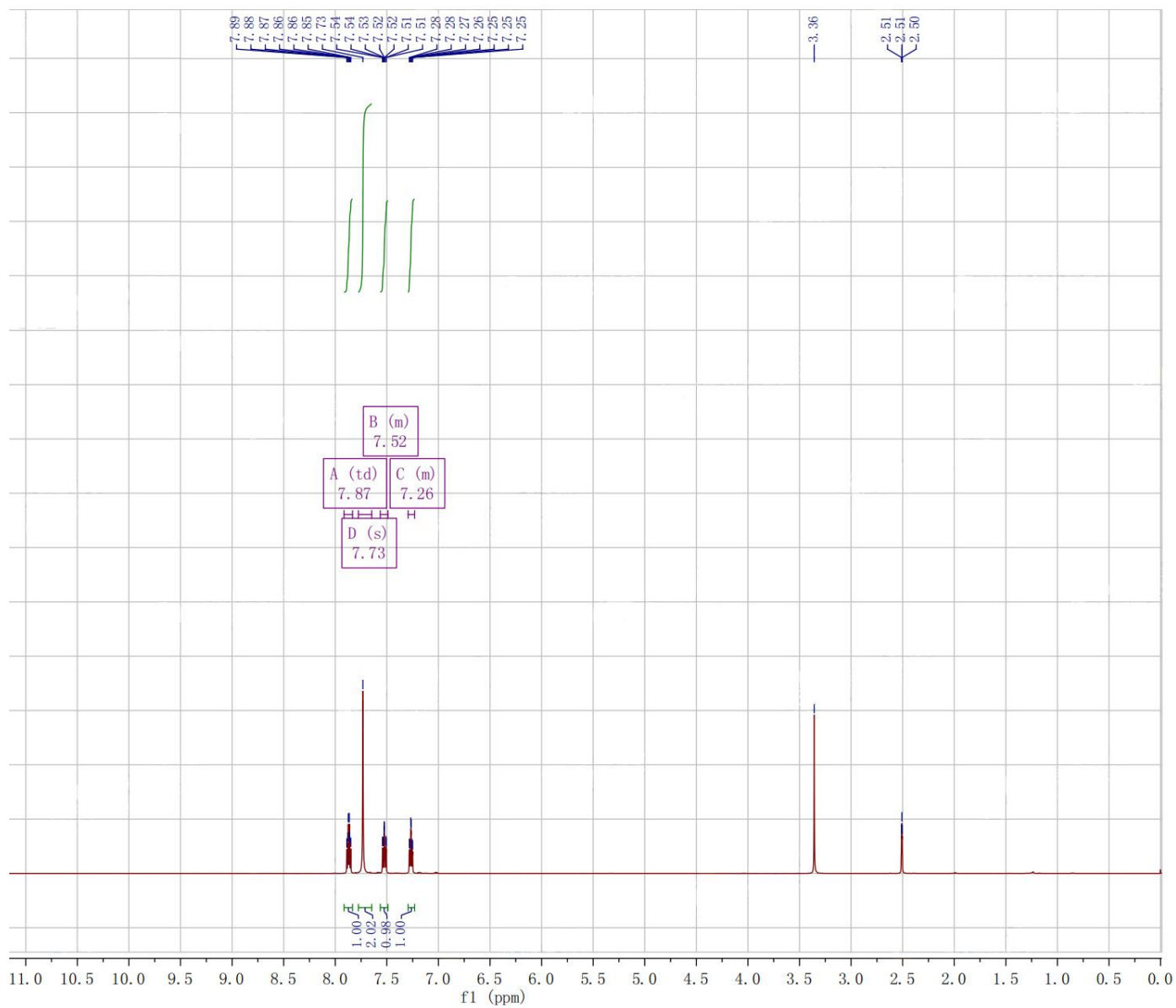
4i, ¹H-NMR



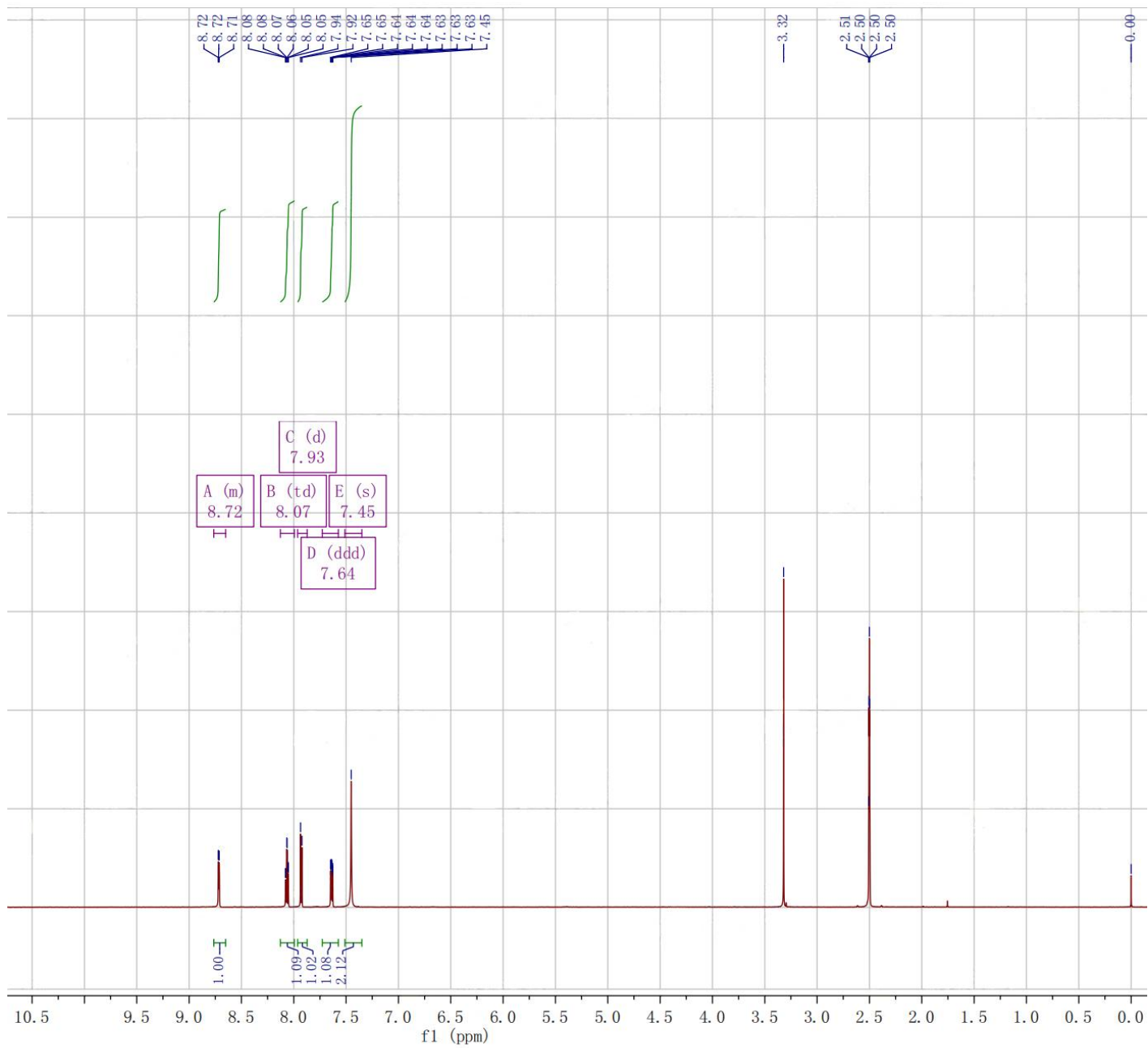
4j, ¹H-NMR



4k, $^1\text{H-NMR}$



41, $^1\text{H-NMR}$



4m, $^1\text{H-NMR}$

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