

Electronic Supplementary Information

Thiosuccinimide enabled S–N bond formation to access *N*-sulfenylated sulfonamide derivatives with synthetic diversity

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1. General information

The commercially available starting materials, *e.g.*, sulfa drugs, thiols, *N*-chlorosuccinimide, solvents, and various additives, were purchased from Adamas-beta, Energy chemical, and Heowns, *etc.*, and were used as received unless otherwise noted. The sulfenylating reagents, such as thiosulfonate (**Ts-2a**), thiophthalimide (**Phth-2a**), and thiosuccinimides (**Su-2a–Su-2o**), were prepared according to literature procedures.^{1–3} All air- and moisture-sensitive manipulations were conducted with a standard Schlenk technique under nitrogen. Flash column chromatography was performed using Qingdao Haiyang silica gel (300–400) with distilled solvents. Analytical thin layer chromatography (TLC) was performed on Haiyang TLC silica gel GF254 (0.25 mm) plates. Melting points were determined using the XT-4 micro melting point apparatus and were uncorrected. The ¹H, ¹³C NMR, and ¹⁹F NMR spectra were recorded on a Bruker ADVANCE III spectrometer operating at 400 MHz, 101 MHz, and 376 MHz, respectively, and chemical shifts are reported in ppm (δ) relative to internal tetramethylsilane (TMS). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), and coupling constants (*J*) were reported in hertz. High-resolution mass spectra were measured on a Thermo Fisher Scientific Exactive Orbitrap Mass Spectrometer under Electron Spray Ionization conditions. Single crystal X-ray diffraction analysis was performed on a Bruker D8 Quest diffractometer. High-temperature NMR spectrum of **Su-2a** was measured using a Bruker ADVANCE III HD 400 instrument.

2. Synthesis of starting materials

Synthesis of thiosulfonate **Ts-2a**

Thiosulfonate **Ts-2a** was synthesized following a one-step procedure from 4-methylbenzenethiol **2a**, according to previous literature.¹ To a 100 mL Schlenk tube equipped with a magnetic stir bar, **2a** (5 mmol), sodium *p*-tolylsulfinate (10 mmol), FeCl₃ (1 mmol), and DMF (20 mL) were added under open-flask conditions, and the reaction mixture was vigorously stirred at rt for 3 h. Upon completion, the reaction mixture was washed with saturated NH₄Cl solution and extracted by dichloromethane. The combined organic layer was dried over anhydrous Na₂SO₄, filtered, and

concentrated *in vacuo*. The resulting residue was purified by flash column chromatography on silica gel, eluting with EA/PE, to afford the desired product.

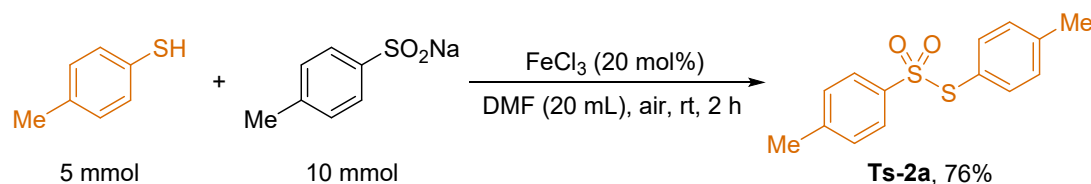


Fig. S1 Synthetic route for preparing thiophthalimide **Ts-2a**.

Synthesis of thiophthalimide **Phth-2a**

Thiophthalimide **Phth-2a** was synthesized following a two-step procedure from 4-methylbenzenethiol **2a**, according to previous literature.² To a cooled (0 °C) solution of **2a** (10 mmol) and a catalytic amount of Et₃N (1 mmol) in CH₂Cl₂ (10 mL), a solution of sulfonyl chloride (10 mmol) in anhydrous CH₂Cl₂ was added slowly under N₂ with vigorous stirring, and the reaction temperature was maintained at 0 °C for 0.5 h. Then, the reaction mixture was gradually warmed to rt over a period of 2 h, and then added dropwise to a solution of phthalimide (10 mmol) and Et₃N (12 mmol) in dry CH₂Cl₂ at 0 °C, followed by stirring at rt for 2 h. Upon completion, the reaction mixture was washed with deionized water and extracted by dichloromethane. The combined organic layer was dried over anhydrous Na₂SO₄, filtered, and concentrated *in vacuo*. The resulting residue was purified by flash column chromatography on silica gel, eluting with EA/PE, to afford the desired product.

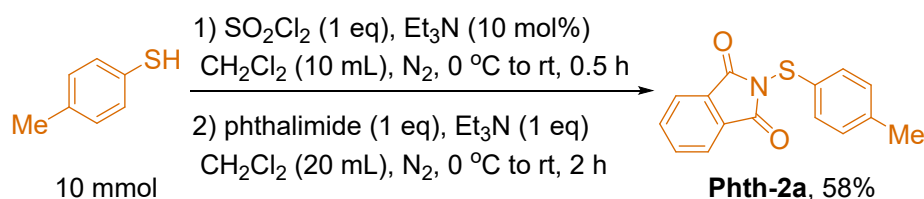


Fig. S2 Synthetic route for preparing thiophthalimide **Phth-2a**.

General procedure for the synthesis of thiosuccinimides **Su-2a–Su-2o**

According to previous literature,³ thiosuccinimides were synthesized following a one-step procedure from the corresponding thiols. To a cooled (0 °C) suspension of NCS (20 mmol) in anhydrous toluene (30 mL), thiol (20 mmol) was added under N₂. After being stirred at 0 °C for 0.5 h, Et₃N (20 mmol) in dry toluene was added dropwise, followed by reacting at room temperature for approximately 12 h. Upon completion, the reaction mixture was washed with deionized water and extracted by

dichloromethane. The combined organic layer was dried over anhydrous Na_2SO_4 , filtered, and concentrated *in vacuo*. The resulting residue was purified by flash column chromatography on silica gel, eluting with EA/PE, to afford the desired product.

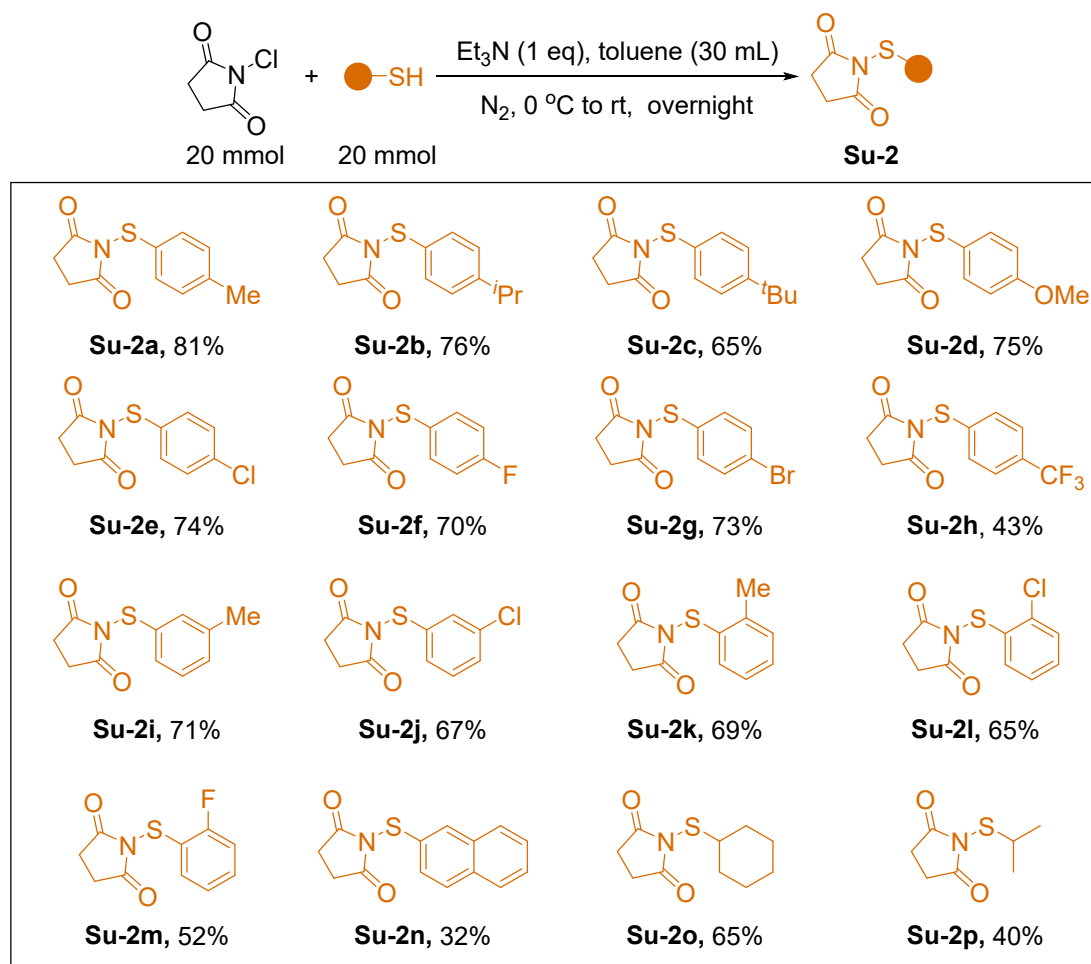
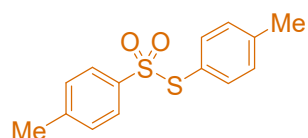


Fig. S3 Synthetic route for preparing thiosuccinimides **Su-2a–Su-2p**.

3. Analytical data

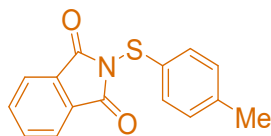
S-(*p*-tolyl) 4-methylbenzenesulfonothioate (**Ts-2a**)¹



White solid; Yield: 76% (1.05 g); ^1H NMR (400 MHz, CDCl_3) δ 7.45 (dd, $J = 6.7$ Hz, 2H), 7.26–7.15 (m, 4H), 7.15–7.13 (m, 2H), 2.42 (s, 3H), 2.38 (s, 3H). ^{13}C NMR (101

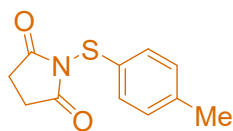
MHz, CDCl₃) δ 144.6, 142.0, 140.4, 136.5, 130.2, 129.3, 127.6, 124.5, 21.6, 21.5.
HRMS (ESI) calcd for C₁₁H₁₁O₂NS, [M+H]⁺: 279.0513; found: 279.0508.

2-(*p*-tolylthio)isoindoline-1,3-dione (Phth-2a)²



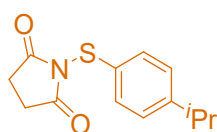
White solid; Yield: 58% (1.56 g); ¹H NMR (400 MHz, CDCl₃) δ 7.90 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.75 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.59 (dd, *J* = 6.3, 1.9 Hz, 2H), 7.13 (dd, *J* = 8.6 Hz, 2H), 2.31 (s, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 167.8, 140.3, 134.6, 132.7, 132.0, 131.4, 130.0, 123.9, 21.3; HRMS (ESI) calcd for C₁₁H₁₁O₂NS, [M+H]⁺: 270.0589; found: 270.0595.

1-(*p*-tolylthio)pyrrolidine-2,5-dione (Su-2a)³



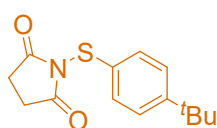
White solid; Yield: 81% (3.58 g); ¹H NMR (400 MHz, CDCl₃) δ 7.56 (dd, *J* = 8.2, 1.7 Hz, 2H), 7.12 (d, *J* = 7.8 Hz, 2H), 2.76 (d, *J* = 0.9 Hz, 2H), 2.31 (s, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 176.3, 140.3, 132.7, 130.1, 129.7, 28.2, 20.9; HRMS (ESI) calcd for C₁₁H₁₁O₂NS, [M+H]⁺: 222.0589; found: 222.0599.

1-((4-isopropylphenyl)thio)pyrrolidine-2,5-dione (Su-2b)⁴



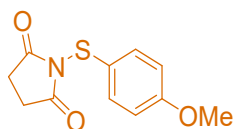
White solid; Yield: 76% (3.78 g); ¹H NMR (400 MHz, CDCl₃) δ 7.57 (dd, *J* = 6.3 Hz, 2H), 7.15 (dd, *J* = 6.3 Hz, 2H), 2.85 (m, 1H), 2.75 (s, 4H), 1.17 (d, *J* = 6.9 Hz, 6H); ¹³C NMR (101 MHz, CDCl₃) δ 176.5, 151.4, 133.5, 130.4, 127.3, 33.8, 28.4, 23.6; HRMS (ESI) calcd for C₁₃H₁₅O₂NS, [M+H]⁺: 250.0902; found: 250.0901.

1-((4-*tert*-butylphenyl)thio)pyrrolidine-2,5-dione (Su-2c)³



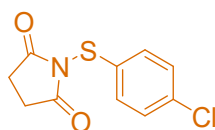
White solid; Yield: 65% (3.42 g); ^1H NMR (400 MHz, CDCl_3) δ 7.54 (dd, $J = 6.6$ Hz, 2H), 7.28 (dd, $J = 6.5$ Hz, 2H), 2.72 (s, 4H), 1.21 (s, 9H); ^{13}C NMR (101 MHz, CDCl_3) δ 176.5, 153.7, 133.3, 130.2, 126.3, 34.7, 31.0, 28.5; HRMS (ESI) calcd for $\text{C}_{14}\text{H}_{17}\text{O}_2\text{NS}$, $[\text{M}+\text{H}]^+$: 264.1058; found: 264.1061.

1-((4-methoxyphenyl)thio)pyrrolidine-2,5-dione (Su-2d)⁴



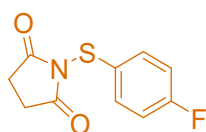
Pink solid; Yield: 75% (3.55 g); ^1H NMR (400 MHz, CDCl_3) δ 7.72 (dd, $J = 6.7$ Hz, 2H), 6.83 (dd, $J = 6.7$ Hz, 2H), 3.78 (s, 3H), 2.74 (s, 4H); ^{13}C NMR (101 MHz, CDCl_3) δ 176.6, 161.7, 137.3, 124.3, 114.6, 55.4, 28.5; HRMS (ESI) calcd for $\text{C}_{11}\text{H}_{11}\text{O}_3\text{NS}$, $[\text{M}+\text{H}]^+$: 238.0538; found: 238.0535.

1-((4-chlorophenyl)thio)pyrrolidine-2,5-dione (Su-2e)³



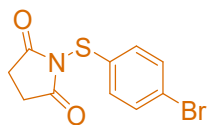
White solid; Yield: 74% (3.58 g); ^1H NMR (400 MHz, CDCl_3) δ 7.58 (dd, $J = 6.5, 2.0$ Hz, 2H), 7.31 (dd, $J = 6.6, 2.1$ Hz, 2H), 2.83 (s, 4H); ^{13}C NMR (101 MHz, CDCl_3) δ 176.2, 136.2, 133.9, 132.1, 129.4, 28.4; HRMS (ESI) calcd for $\text{C}_{10}\text{H}_8\text{O}_2\text{ClNS}$, $[\text{M}+\text{H}]^+$: 242.0043; found: 242.0020.

1-((4-fluorophenyl)thio)pyrrolidine-2,5-dione (Su-2f)³



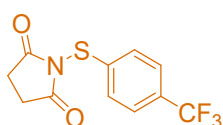
White solid; Yield: 70% (3.15 g); ^1H NMR (400 MHz, CDCl_3) δ 7.71–7.66 (m, 2H), 7.01–6.96 (m, 2H), 2.76 (s, 4H); ^{13}C NMR (101 MHz, CDCl_3) δ 176.3, 163.8 (d, $J = 253.1$ Hz), 136.4 (d, $J = 8.8$ Hz), 128.9 (d, $J = 3.5$ Hz), 116.4 (d, $J = 22.4$ Hz), 28.4; ^{19}F NMR (376 MHz, CDCl_3) δ -108.7; HRMS (ESI) calcd for $\text{C}_{10}\text{H}_8\text{O}_2\text{FNS}$, $[\text{M}+\text{H}]^+$: 226.0338; found: 226.0339.

1-((4-bromophenyl)thio)pyrrolidine-2,5-dione (Su-2g)³



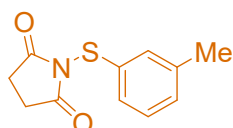
White solid; Yield: 73% (4.17 g); ¹H NMR (400 MHz, CDCl₃) δ 7.51 (dd, *J* = 6.4 Hz, 2H), 7.46 (dd, *J* = 6.6 Hz, 2H), 2.82 (s, 4H); ¹³C NMR (101 MHz, CDCl₃) δ 176.2, 134.3, 132.7, 132.5, 124.8, 28.5; HRMS (ESI) calcd for C₁₀H₈O₂BrNS, [M+H]⁺: 285.9537; found: 285.9539.

1-((4-(trifluoromethyl)phenyl)thio)pyrrolidine-2,5-dione (Su-2h)



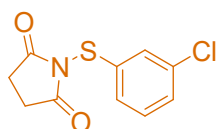
White solid; Yield: 43% (2.36 g); ¹H NMR (400 MHz, CDCl₃) δ 7.56–7.50 (m, 4H), 2.87 (s, 4H); ¹³C NMR (101 MHz, CDCl₃) δ 176.0, 130.7 (q, *J* = 33.0 Hz), 129.3, 126.1 (q, *J* = 3.6 Hz), 123.5 (q, *J* = 273.1 Hz), 28.5; ¹⁹F NMR (376 MHz, CDCl₃) δ -62.9; HRMS (ESI) calcd for C₁₁H₈O₂F₃NS, [M+H]⁺: 276.0306; found: 276.0316.

1-(*m*-tolylthio)pyrrolidine-2,5-dione (Su-2i)⁴



Yellow solid; Yield: 71% (3.14 g); ¹H NMR (400 MHz, CDCl₃) δ 7.37 (s, 1H), 7.34 (d, *J* = 7.8 Hz, 1H), 7.19 (t, *J* = 7.6 Hz, 1H), 7.12 (d, *J* = 7.6 Hz, 1H), 2.79 (s, 4H), 2.31 (s, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 176.3, 138.9, 133.4, 131.7, 130.2, 128.8, 128.3, 28.3, 20.9; HRMS (ESI) calcd for C₁₁H₁₁O₂NS, [M+H]⁺: 222.0589; found: 222.0599.

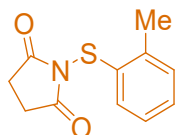
1-((3-chlorophenyl)thio)pyrrolidine-2,5-dione (Su-2j)⁵



Pink solid; Yield: 67% (3.24 g); ¹H NMR (400 MHz, CDCl₃) δ 7.51 (t, *J* = 1.9 Hz, 1H), 7.43 (dt, *J* = 7.5, 1.6 Hz, 1H), 7.29 (dt, *J* = 8.1, 1.6 Hz, 1H), 7.23 (t, *J* = 2.9 Hz, 1H), 2.83 (s, 4H); ¹³C NMR (101 MHz, CDCl₃) δ 176.1, 135.6, 135.0, 131.1, 130.4,

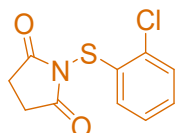
129.9, 129.5, 28.6; HRMS (ESI) calcd for C₁₀H₈O₂CINS, [M+H]⁺: 242.0043; found: 242.0020.

1-(*o*-tolylthio)pyrrolidine-2,5-dione (Su-2k)⁴



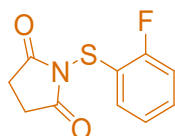
Yellow solid; Yield: 69% (3.05 g); ¹H NMR (400 MHz, CDCl₃) δ 7.40 (d, *J* = 7.2 Hz, 1H), 7.22–7.17 (m, 2H), 7.13–7.09 (m, 1H), 2.80 (s, 4H), 2.56 (s, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 176.4, 139.2, 133.0, 131.6, 130.6, 129.4, 126.7, 28.5, 20.2; HRMS (ESI) calcd for C₁₁H₁₁O₂NS, [M+H]⁺: 222.0589; found: 222.0599.

1-((2-chlorophenyl)thio)pyrrolidine-2,5-dione (Su-2l)⁵



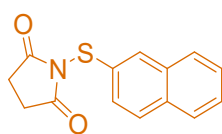
White solid; Yield: 65% (3.14 g); ¹H NMR (400 MHz, CDCl₃) δ 7.36–7.33 (m, 1H), 7.22–7.14 (m, 2H), 6.92–6.89 (m, 1H), 2.93 (s, 4H); ¹³C NMR (101 MHz, CDCl₃) δ 175.9, 133.1, 130.0, 129.8, 128.1, 127.4, 125.4, 28.6; HRMS (ESI) calcd for C₁₀H₈O₂CINS, [M+H]⁺: 242.0043; found: 242.0052.

1-((2-fluorophenyl)thio)pyrrolidine-2,5-dione (Su-2m)³



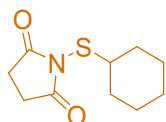
White solid; Yield: 52% (2.34 g); ¹H NMR (400 MHz, CDCl₃) δ 7.56–7.52 (m, 1H), 7.37–7.31 (m, 1H), 7.13–7.05 (m, 2H), 2.83 (s, 4H); ¹³C NMR (101 MHz, CDCl₃) δ 176.0, 160.8, (d, *J* = 251 Hz), 133.7, 131.9 (d, *J* = 8.0 Hz), 124.8 (d, *J* = 3.8 Hz), 120.7 (d, *J* = 17.5 Hz), 116.4 (d, *J* = 21.9 Hz), 28.5; ¹⁹F NMR (376 MHz, CDCl₃) δ -107.7; HRMS (ESI) calcd for C₁₀H₈O₂FNS, [M+H]⁺: 226.0338; found: 226.0339.

1-(naphthalen-2-ylthio)pyrrolidine-2,5-dione (Su-2n)⁴



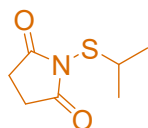
White solid; Yield: 32% (1.65 g); ^1H NMR (400 MHz, CDCl_3) δ 8.16 (s, 1H), 7.82–7.76 (m, 3H), 7.64 (dd, $J = 8.6, 1.8$ Hz, 1H), 7.53–7.47 (m, 2H), 2.77 (s, 4H); ^{13}C NMR (101 MHz, CDCl_3) δ 176.5, 133.4, 133.1, 132.6, 130.9, 129.2, 128.8, 128.1, 127.6, 127.5, 126.9, 28.5; HRMS (ESI) calcd for $\text{C}_{14}\text{H}_{11}\text{O}_2\text{NS}$, $[\text{M}+\text{H}]^+$: 258.0589; found: 258.0581.

1-(cyclohexylthio)pyrrolidine-2,5-dione (Su-2o)³



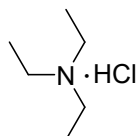
White solid; Yield: 65% (2.77 g); ^1H NMR (400 MHz, CDCl_3) δ 3.17–3.13 (m, 1H), 2.81 (s, 4H), 1.80–1.73 (m, 4H), 1.60–1.56 (m, 1H), 1.26–1.21 (m, 5H); ^{13}C NMR (101 MHz, CDCl_3) δ 177.4, 48.3, 30.8, 28.4, 25.3; HRMS (ESI) calcd for $\text{C}_{10}\text{H}_{15}\text{O}_2\text{ClNS}$, $[\text{M}+\text{H}]^+$: 214.0902; found: 214.0891.

1-(isopropylthio)pyrrolidine-2,5-dione (Su-2p)³



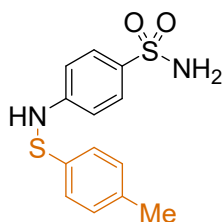
Colorless oil; Yield: 40% (1.38 g); ^1H NMR (400 MHz, CDCl_3) δ 3.21–3.16 (m, 1H), 2.65 (s, 4H), 0.96 (d, $J = 7.4$ Hz, 6H); ^{13}C NMR (101 MHz, CDCl_3) δ 177.1, 39.8, 28.1, 20.2; HRMS (ESI) calcd for $\text{C}_7\text{H}_{11}\text{O}_2\text{NS}$, $[\text{M}+\text{H}]^+$: 174.0589; found: 174.0562.

Triethylamine hydrochloride⁶



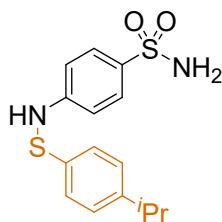
White solid; ^1H NMR (400 MHz, CDCl_3) δ 10.44 (s, 1H), 2.94 (q, $J = 14.6$ Hz, 6H), 1.20 (t, $J = 7.4$ Hz, 9H); ^{13}C NMR (101 MHz, CDCl_3) δ 45.5, 8.3.

4-((p-tolylthio)amino)benzenesulfonamide (3a)



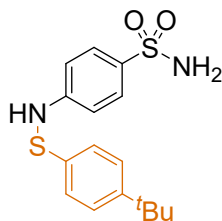
White solid; Yield: 84% (123.4 mg, *additive-free synthesis*), 71% (104.5 mg, *one-pot synthesis*); Mp: 123–124 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.48 (s, 1H), 7.64 (d, *J* = 8.7 Hz, 2H), 7.14 (d, *J* = 8.1 Hz, 2H), 7.15–7.07 (m, 6H), 2.23 (s, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 150.7, 136.7, 135.2, 134.6, 129.8, 127.5, 123.0, 113.9, 20.6; HRMS (ESI) calcd for C₁₃H₁₄O₂N₂S₂Na, [M+Na]⁺: 317.0394; found: 317.0391.

4-(((4-isopropylphenyl)thio)amino)benzenesulfonamide (3b)



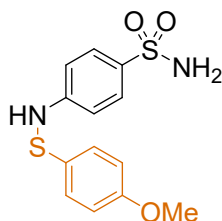
White solid; Yield: 83% (133.6 mg, *additive-free synthesis*), 63% (101.5 mg, *one-pot synthesis*); Mp: 133–135 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.47 (s, 1H), 7.63 (d, *J* = 8.7 Hz, 2H), 7.20 (d, *J* = 8.3 Hz, 2H), 7.11–7.08 (m, 6H), 2.87–2.77 (m, 1H), 1.14 (d, *J* = 6.8 Hz, 6H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 150.7, 146.2, 137.1, 134.6, 127.4, 127.2, 123.0, 113.8, 33.0, 23.8; HRMS (ESI) calcd for C₁₅H₁₉O₂N₂S₂, [M+H]⁺: 323.0888; found: 323.0879.

4-(((4-(*tert*-butyl)phenyl)thio)amino)benzenesulfonamide (3c)



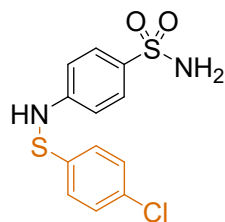
White solid; Yield: 86% (144.5 mg, *additive-free synthesis*), 67% (112.5 mg, *one-pot synthesis*); Mp: 125–127 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.49 (s, 1H), 7.65 (d, *J* = 8.7 Hz, 2H), 7.35 (d, *J* = 8.4 Hz, 2H), 7.13–7.10 (m, 6H), 1.22 (s, 9H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 150.7, 148.5, 136.9, 134.6, 127.5, 126.1, 122.7, 113.9, 34.2, 31.1; HRMS (ESI) calcd for C₁₆H₂₁O₂N₂S₂, [M+H]⁺: 337.1044; found: 337.1032.

4-(((4-methoxyphenyl)thio)amino)benzenesulfonamide (3d)



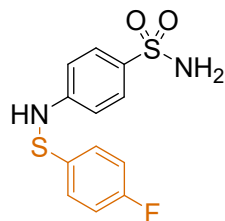
White solid; Yield: 35% (54.3 mg, *additive-free synthesis*); Mp: 107–109 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.44 (s, 1H), 7.61 (dd, *J* = 7.1 Hz, 2H), 7.21 (dd, *J* = 6.7 Hz, 2H), 7.09 (dd, *J* = 7.4 Hz, 4H), 6.93 (d, *J* = 6.8 Hz, 2H), 3.70 (s, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 158.3, 150.7, 134.4, 130.3, 127.4, 126.4, 114.9, 113.9, 55.2; HRMS (ESI) calcd for C₁₃H₁₅O₃N₂S₂Na, [M+Na]⁺: 333.0344; found: 333.0349.

4-(((4-chlorophenyl)thio)amino)benzenesulfonamide (3e)



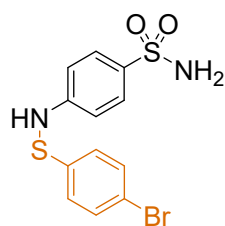
White solid; Yield: 78% (122.0 mg, *additive-free synthesis*), 70% (109.6 mg, *one-pot synthesis*); Mp: 111–113 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.57 (s, 1H), 7.66 (d, *J* = 8.0 Hz, 2H), 7.40 (dd, *J* = 6.6 Hz, 2H), 7.18 (dd, *J* = 6.7 Hz, 2H), 7.12–7.08 (m, 4H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 150.2, 139.6, 135.0, 130.1, 129.1, 127.5, 124.1, 113.9; HRMS (ESI) calcd for C₁₂H₁₂O₂ClN₂S₂, [M+H]⁺: 315.0029; found: 315.0028.

4-(((4-fluorophenyl)thio)amino)benzenesulfonamide (3f)



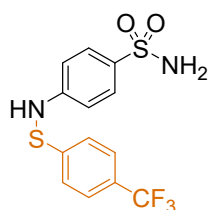
White solid; Yield: 65% (96.9 mg, *additive-free synthesis*), 56% (83.5 mg, *one-pot synthesis*); Mp: 137–139 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.54 (s, 1H), 7.64 (dd, *J* = 6.7 Hz, 2H), 7.25–7.18 (m, 4H), 7.11–7.08 (m, 4H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 160.7 (d, *J* = 243.5 Hz), 150.4, 135.7 (d, *J* = 2.8 Hz), 134.8, 127.5, 125.0, (d, *J* = 8.1 Hz), 116.3 (d, *J* = 22.2 Hz), 113.9; ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -117.0; HRMS (ESI) calcd for C₁₂H₁₁O₂FN₂S₂Na, [M+Na]⁺: 321.0144; found: 321.0140.

4-(((4-bromophenyl)thio)amino)benzenesulfonamide (3g)



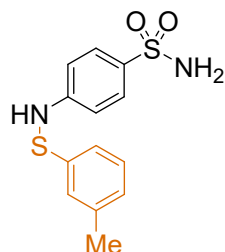
White solid; Yield: 62% (110.6 mg, *additive-free synthesis*), 51% (91.1 mg, *one-pot synthesis*); Mp: 135–136 °C; $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 8.57 (s, 1H), 7.65 (dd, $J = 6.8$ Hz, 2H), 7.52 (dd, $J = 6.6$ Hz, 2H), 7.13–7.12 (m, 3H), 7.11–7.09 (m, 2H), 7.08 (d, $J = 2.0$ Hz, 1H); $^{13}\text{C NMR}$ (101 MHz, $\text{DMSO-}d_6$) δ 150.2, 140.2, 135.0, 132.0, 127.5, 124.4, 118.3, 113.9; HRMS (ESI) calcd for $\text{C}_{12}\text{H}_{12}\text{O}_2\text{BrN}_2\text{S}_2$, $[\text{M}+\text{H}]^+$: 358.9524; found: 358.9518.

4-(((4-(trifluoromethyl)phenyl)thio)amino)benzenesulfonamide (3h)



White solid; Yield: 64% (111.4 mg, *additive-free synthesis*), 57% (99.1 mg, *one-pot synthesis*); $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 8.64 (s, 1H), 7.70–7.65 (m, 4H), 7.35 (d, $J = 8.2$ Hz, 2H), 7.16–7.08 (m, 4H); $^{13}\text{C NMR}$ (101 MHz, $\text{DMSO-}d_6$) δ 149.9, 146.7, 135.2, 127.6, 126.0 (q, $J = 3.7$ Hz), 126.4–125.5 (m), 122.9, 122.2, 113.9; $^{19}\text{F NMR}$ (376 MHz, $\text{DMSO-}d_6$) δ -60.7; HRMS (ESI) calcd for $\text{C}_{13}\text{H}_{11}\text{O}_2\text{F}_3\text{N}_2\text{S}_2$, $[\text{M}+\text{H}]^+$: 349.0292; found: 349.0253.

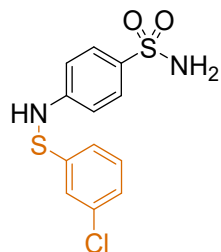
4-(((*m*-tolylthio)amino)benzenesulfonamide (3i)



White solid; Yield: 65% (95.7 mg, *additive-free synthesis*), 40% (58.9 mg, *one-pot synthesis*); Mp: 153–155 °C; $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 8.48 (s, 1H), 7.65 (dd, $J = 6.7$ Hz, 2H), 7.21 (t, $J = 7.6$ Hz, 1H), 7.11–7.09 (m, 4H), 7.01 (s, 1H), 6.95 (d, $J = 6.8$ Hz, 2H), 2.25 (s, 3H); $^{13}\text{C NMR}$ (101 MHz, $\text{DMSO-}d_6$) δ 150.7, 140.2, 138.5,

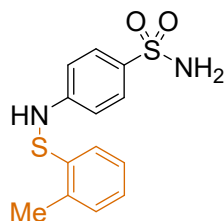
134.7, 129.0, 127.5, 126.5, 122.7, 119.5, 113.9, 21.1; HRMS (ESI) calcd for $C_{13}H_{14}O_2N_2S_2Na$, $[M+Na]^+$: 317.0394; found: 317.0391.

4-(((3-chlorophenyl)thio)amino)benzenesulfonamide (3j)



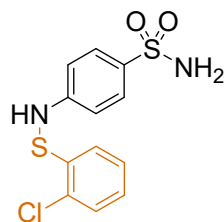
White solid; Yield: 72% (112.7 mg, *additive-free synthesis*), 64% (100.2 mg, *one-pot synthesis*); Mp: 145–147 °C; 1H NMR (400 MHz, DMSO- d_6) δ 8.57 (s, 1H), 7.66 (d, $J = 8.0$ Hz, 2H), 7.40 (dd, $J = 6.6$ Hz, 2H), 7.18 (d, $J = 6.6$ Hz, 2H), 7.12–7.08 (m, 4H); ^{13}C NMR (101 MHz, DMSO- d_6) δ 150.0, 143.4, 135.10, 134.0, 131.0, 127.6, 125.5, 121.5, 120.8, 114.0; HRMS (ESI) calcd for $C_{12}H_{12}O_2ClN_2S_2$, $[M+H]^+$: 315.0029; found: 315.0028.

4-(((*o*-tolylthio)amino)benzenesulfonamide (3k)



White solid; Yield: 80% (117.6 mg, *additive-free synthesis*), 68% (99.8 mg, *one-pot synthesis*); Mp: 133–135 °C; 1H NMR (400 MHz, DMSO- d_6) δ 8.38 (s, 1H), 7.64 (d, $J = 8.3$ Hz, 2H), 7.18–7.03 (m, 7H), 6.98 (d, $J = 7.7$ Hz, 1H), 2.26 (s, 3H); ^{13}C NMR (101 MHz, DMSO- d_6) δ 150.5, 138.6, 134.7, 131.7, 130.3, 127.5, 126.5, 125.1, 121.3, 113.9, 18.1; HRMS (ESI) calcd for $C_{13}H_{14}O_2N_2S_2Na$, $[M+Na]^+$: 317.0394; found: 317.0391.

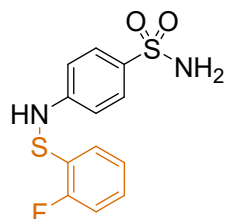
4-(((2-chlorophenyl)thio)amino)benzenesulfonamide (3l)



White solid; Yield: 82% (128.3 mg, *additive-free synthesis*), 63% (98.5 mg, *one-pot synthesis*); Mp: 141–143 °C; 1H NMR (400 MHz, DMSO- d_6) δ 8.53 (s, 1H), 7.67 (d,

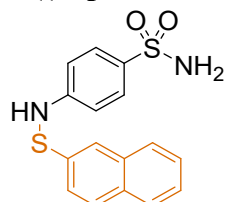
$J = 8.8$ Hz, 2H), 7.45 (dd, $J = 8.1, 1.2$ Hz, 1H), 7.33–7.28 (m, 1H), 7.20–7.16 (m, 1H), 7.13 (s, 2H), 7.10 (dd, $J = 6.8, 2.0$ Hz, 2H), 7.01 (dd, $J = 6.4, 1.6$ Hz, 1H); ^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) δ 149.9, 138.7, 135.2, 129.7, 127.8, 127.6, 126.8, 126.5, 123.1, 114.0. HRMS (ESI) calcd for $\text{C}_{12}\text{H}_{12}\text{O}_2\text{ClN}_2\text{S}_2$, $[\text{M}+\text{H}]^+$: 315.0029; found: 315.0015.

4-(((2-fluorophenyl)thio)amino)benzenesulfonamide (3m)



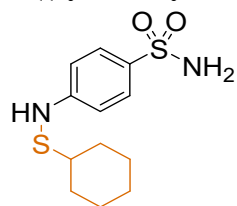
White solid; Yield: 61% (90.9 mg, *additive-free synthesis*), 48% (71.6 mg, *one-pot synthesis*); Mp: 126–128 °C; ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 8.45 (s, 1H), 7.65 (dd, $J = 6.8, 2.0$ Hz, 2H), 7.27–7.07 (m, 8H), 7.09 (s, 1H); ^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) δ 157.0 (d, $J = 241.0$ Hz) 150.0, 135.1, 127.6 (d, $J = 7.1$ Hz), 127.5, 127.1 (d, $J = 16.3$ Hz), 125.3 (d, $J = 2.9$ Hz), 124.8 (d, $J = 3.0$ Hz), 115.7 (d, $J = 19.9$ Hz), 114.1; ^{19}F NMR (376 MHz, $\text{DMSO-}d_6$) δ -115.9; HRMS (ESI) calcd for $\text{C}_{12}\text{H}_{11}\text{O}_2\text{FN}_2\text{S}_2\text{Na}$, $[\text{M}+\text{Na}]^+$: 321.0144; found: 321.0140.

4-(((naphthalen-2-ylthio)amino)benzenesulfonamide (3n)



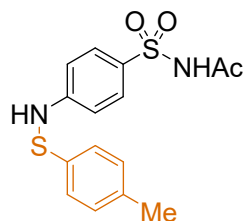
White solid; Yield: 87% (143.5 mg, *additive-free synthesis*), 67% (110.7 mg, *one-pot synthesis*); Mp: 163–165 °C; ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 8.63 (s, 1H), 7.89 (d, $J = 8.7$ Hz, 1H), 7.85 (d, $J = 7.9$ Hz, 1H), 7.77 (d, $J = 7.8$ Hz, 1H), 7.64 (d, $J = 8.7$ Hz, 3H), 7.46 (s, 2H), 7.36–7.42 (m, 2H), 7.34 (dd, $J = 8.6, 1.9$ Hz, 1H), 7.14 (d, $J = 8.7$ Hz), 7.09 (s, 2H); ^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) δ 161.9, 159.5, 150.4, 135.7 (d, $J = 2.9$ Hz), 134.8, 127.5, 125.1, 125.0, 116.4, 116.2, 113.9; HRMS (ESI) calcd for $\text{C}_{16}\text{H}_{15}\text{O}_2\text{N}_2\text{S}_2$, $[\text{M}+\text{H}]^+$: 331.0575; found: 331.0558.

4-((cyclohexylthio)amino)benzenesulfonamide (3o)



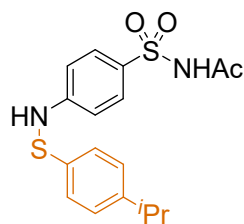
White solid; Yield: 21% (30.1 mg, *additive-free synthesis*); Mp: 112–114 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.75 (s, 1H), 7.59 (d, *J* = 8.7 Hz, 2H), 7.10–7.05 (m, 4H), 2.85–2.80 (m, 1H), 1.86–1.82 (m, 2H), 1.71–1.68 (m, 2H), 1.57–1.51 (m, 1H), 1.29–1.14 (m, 5H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 152.5, 133.4, 127.2, 113.4, 47.9, 30.6, 25.3, 25.2; HRMS (ESI) calcd for C₁₂H₁₈O₂N₂S₂, [M+H]⁺: 287.0888; found: 287.0895.

N-((4-((*p*-tolylthio)amino)phenyl)sulfonyl)acetamide (4a)



Yellow oil; Yield: 86% (144.5 mg, *additive-free synthesis*), 64% (107.5 mg, *one-pot synthesis*); ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.82 (s, 1H), 8.68 (s, 1H), 7.75–7.70 (m, 2H), 7.17–7.08 (m, 6H), 2.24 (s, 3H), 1.90 (s, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 168.7, 152.5, 136.4, 135.5, 129.9, 129.7, 129.0, 123.3, 113.9, 23.3, 20.6; HRMS (ESI) calcd for C₁₅H₁₇O₃N₂S₂, [M+H]⁺: 337.0681; found: 337.0695.

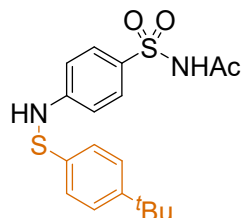
N-((4-(((4-isopropylphenyl)thio)amino)phenyl)sulfonyl)acetamide (4b)



White solid; Yield: 82% (149.2 mg, *additive-free synthesis*), 63% (114.7 mg, *one-pot synthesis*); Mp: 126–128 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.84 (s, 1H), 8.68 (s, 1H), 7.72 (d, *J* = 8.8 Hz, 2H), 7.21 (d, *J* = 8.4 Hz, 2H), 7.14 (d, *J* = 1.4 Hz, 2H), 7.12 (d, *J* = 0.8 Hz, 2H), 2.86–2.76 (m, 1H), 1.88 (s, 3H), 1.14 (d, *J* = 7.0 Hz, 6H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 168.7, 152.5, 146.4, 136.8, 129.7, 129.1, 127.3, 123.2,

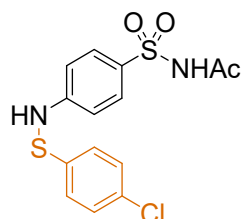
113.8, 33.0, 23.8, 23.3; HRMS (ESI) calcd for C₁₇H₂₁O₃N₂S₂, [M+H]⁺: 365.0994; found: 365.0969.

***N*-((4-(((4-*tert*-butyl)phenyl)thio)amino)phenyl)sulfonyl)acetamide (4c)**



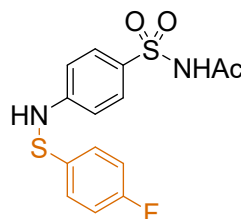
White solid; Yield: 80% (151.2 mg, *additive-free synthesis*), 65% (122.8 mg, *one-pot synthesis*); Mp: 133–135 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.86 (s, 1H), 8.69 (s, 1H), 7.72 (d, *J* = 8.9 Hz, 2H), 7.36 (d, *J* = 8.5 Hz, 2H), 7.14 (d, *J* = 2.0 Hz, 2H), 7.12 (d, *J* = 2.0 Hz, 2H), 1.88 (s, 3H), 1.22 (s, 9H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 168.6, 152.5, 148.7, 136.6, 129.7, 129.0, 126.2, 122.9, 113.8, 34.2, 31.1, 23.3; HRMS (ESI) calcd for C₁₈H₂₃O₃N₂S₂, [M+H]⁺: 379.1150; found: 379.1152.

***N*-((4-(((4-chlorophenyl)thio)amino)phenyl)sulfonyl)acetamide (4d)**



Colorless oil; Yield: 84% (149.5 mg, *additive-free synthesis*), 71% (126.4 mg, *one-pot synthesis*); ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.83 (s, 1H), 8.76 (s, 1H), 7.74 (d, *J* = 8.8 Hz, 2H), 7.40 (d, *J* = 8.6 Hz, 2H), 7.20 (d, *J* = 8.6 Hz, 2H), 7.13 (d, *J* = 8.9 Hz, 2H), 1.88 (s, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 168.7, 152.0, 139.3, 130.3, 129.7, 129.5, 129.2, 124.3, 114.0, 23.3; HRMS (ESI) calcd. for C₁₄H₁₄O₃ClN₂S₂, [M+H]⁺: 357.0134; found: 357.0128.

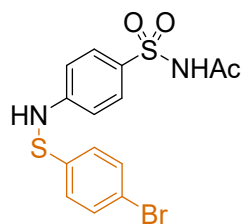
***N*-((4-(((4-fluorophenyl)thio)amino)phenyl)sulfonyl)acetamide (4e)**



White solid; Yield: 84% (142.8 mg, *additive-free synthesis*), 70% (119.1 mg, *one-pot synthesis*); Mp: 137–139 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.87 (s, 1H), 8.75 (s,

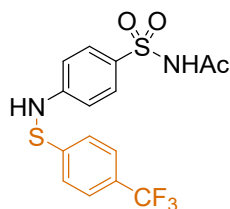
1H), 7.74 (d, $J = 8.8$ Hz, 2H), 7.27–7.24 (m, 2H), 7.22–7.17 (m, 2H), 7.16–7.13 (m, 2H), 1.89 (s, 3H); ^{13}C NMR (101 MHz, DMSO- d_6) δ 168.7, 162.0, 159.6, 152.2, 135.5 (d, $J = 2.6$ Hz), 129.7, 129.3, 125.4, 125.3, 116.5, 116.3, 113.9, 23.2; ^{19}F NMR (376 MHz, DMSO- d_6) δ -116.7; HRMS (ESI) calcd for $\text{C}_{14}\text{H}_{14}\text{O}_3\text{FN}_2\text{S}_2$, $[\text{M}+\text{H}]^+$: 341.0430; found: 341.0407.

***N*-((4-(((4-bromophenyl)thio)amino)phenyl)sulfonyl)acetamide (4f)**



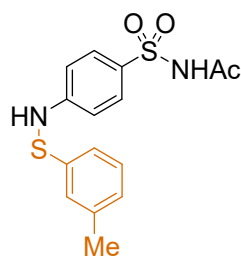
White solid; Mp: 129–130 °C; Yield: 87% (173.6 mg, *additive-free synthesis*), 82% (163.5 mg, *one-pot synthesis*); ^1H NMR (400 MHz, DMSO- d_6) δ 11.87 (s, 1H), 8.76 (s, 1H), 7.74 (d, $J = 8.9$ Hz, 2H), 7.52 (d, $J = 8.6$ Hz, 2H), 7.15–7.10 (m, 4H), 1.89 (s, 3H); ^{13}C NMR (101 MHz, DMSO- d_6) δ 168.7, 152.0, 139.9, 132.0, 129.8, 129.4, 124.5, 118.5, 114.0, 23.3; HRMS (ESI) calcd for $\text{C}_{14}\text{H}_{14}\text{O}_3\text{BrN}_2\text{S}_2$, $[\text{M}+\text{H}]^+$: 400.9629; found: 400.9633.

***N*-((4-(((4-(trifluoromethyl)phenyl)thio)amino)phenyl)sulfonyl)acetamide (4g)**



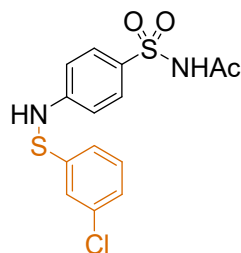
White solid; Yield: 41% (79.9 mg, *additive-free synthesis*); ^1H NMR (400 MHz, DMSO- d_6) δ 11.89 (s, 1H), 8.83 (s, 1H), 7.81–7.67 (m, 4H), 7.36 (d, $J = 8.6$ Hz, 2H), 7.14 (dd, $J = 9.0, 2.1$ Hz, 2H), 1.88 (s, 3H); ^{13}C NMR (101 MHz, DMSO- d_6) δ 168.6, 151.7, 146.4, 129.8, 126.5–125.6 (m), 126.0 (d, $J = 4.4$ Hz), 122.9, 122.3, 114.0, 23.2; ^{19}F NMR (376 MHz, DMSO- d_6) δ -60.7. HRMS (ESI) calcd for $\text{C}_{15}\text{H}_{13}\text{O}_3\text{F}_3\text{N}_2\text{S}_2$, $[\text{M}+\text{H}]^+$: 391.0398; found: 391.0418.

***N*-((4-((*m*-tolylthio)amino)phenyl)sulfonyl)acetamide (4h)**



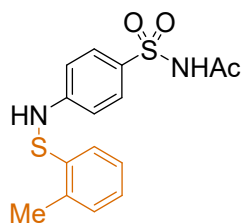
White solid; Yield: 77% (129.3 mg, *additive-free synthesis*), 56% (94.1 mg, *one-pot synthesis*); Mp: 133–135 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.82 (s, 1H), 8.68 (s, 1H), 7.72 (d, *J* = 8.9 Hz, 2H), 7.21 (t, *J* = 7.7 Hz, 1H), 7.13–7.11 (m, 2H), 7.02 (s, 1H), 6.98–6.97 (m, 2H), 2.25 (s, 3H), 1.88 (s, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 168.7, 152.4, 139.9, 138.6, 129.7, 129.2, 129.1, 126.7, 122.9, 119.7, 113.9, 23.3, 21.1; HRMS (ESI) calcd. for C₁₅H₁₇O₃N₂S₂ [M+H]⁺: 337.0681; found: 337.0673.

***N*-((4-(((3-chlorophenyl)thio)amino)phenyl)sulfonyl)acetamide (4i)**



White solid; Yield: 66% (117.5 mg, *additive-free synthesis*), 43% (76.6 mg, *one-pot synthesis*); Mp: 145–146 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.88 (s, 1H), 8.77 (s, 1H), 7.76 (dd, *J* = 9.0, 1.1 Hz, 2H), 7.37–7.33 (m, 1H), 7.22–7.19 (m, 2H), 7.16–7.13 (m, 3H), 1.89 (s, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 168.6, 151.7, 143.0, 133.9, 130.9, 129.6, 129.5, 125.6, 121.5, 120.8, 113.9, 23.2; HRMS (ESI) calcd for C₁₄H₁₄O₃ClN₂S₂, [M+H]⁺: 357.0134; found: 357.0128.

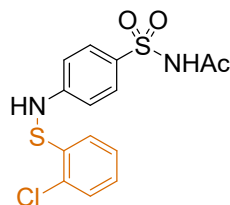
***N*-((4-((*o*-tolylthio)amino)phenyl)sulfonyl)acetamide (4j)**



White solid; Yield: 81% (136.1 mg, *additive-free synthesis*), 65% (109.2 mg, *one-pot synthesis*); Mp: 141–143 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.87 (s, 1H), 8.76 (s, 1H), 7.73 (d, *J* = 8.9 Hz, 2H), 7.40 (d, *J* = 8.6 Hz, 2H), 7.20 (d, *J* = 8.7 Hz, 2H), 7.12

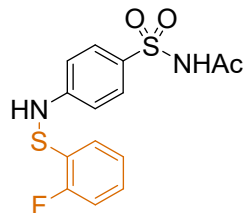
(d, $J = 8.9$ Hz, 2H), 1.88 (s, 3H); ^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) δ 168.6, 152.4, 138.3, 131.9, 130.4, 129.7, 129.1, 126.6, 125.3, 121.5, 113.9, 23.3, 18.2; HRMS (ESI) calcd. for $\text{C}_{15}\text{H}_{17}\text{O}_3\text{N}_2\text{S}_2$ $[\text{M}+\text{H}]^+$: 337.0681; found: 337.0695.

***N*-((4-(((2-chlorophenyl)thio)amino)phenyl)sulfonyl)acetamide (4k)**



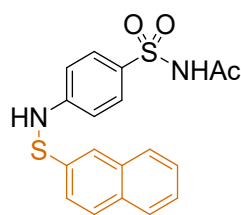
White solid; Yield: 90% (160.2 mg, *additive-free synthesis*), 84% (149.5 mg, *one-pot synthesis*); Mp: 137–139 °C; ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 11.87 (s, 1H), 8.72 (s, 1H), 7.75 (d, $J = 8.8$ Hz, 2H), 7.45 (dd, $J = 7.9, 1.3$ Hz, 1H), 7.33–7.29 (m, 1H), 7.20–7.16 (m, 1H), 7.14–7.12 (m, 2H), 7.03 (dd, $J = 7.9, 1.5$ Hz, 1H), 1.89 (s, 3H); ^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) δ 168.7, 151.7, 138.5, 129.8, 129.8, 129.7, 128.0, 126.9, 126.6, 123.1, 114.1, 23.3; HRMS (ESI) calcd for $\text{C}_{14}\text{H}_{14}\text{O}_3\text{ClN}_2\text{S}_2$, $[\text{M}+\text{H}]^+$: 357.0134; found: 357.0128.

***N*-((4-(((2-fluorophenyl)thio)amino)phenyl)sulfonyl)acetamide (4l)**



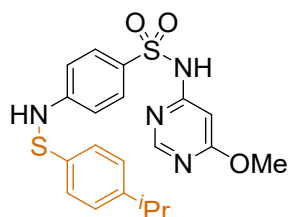
White solid; Yield: 72% (122.4 mg, *additive-free synthesis*), 58% (98.6 mg, *one-pot synthesis*); Mp: 158–160 °C; ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 11.80 (s, 1H), 8.64 (s, 1H), 7.75 (d, $J = 8.9$ Hz, 2H), 7.25–7.22 (m, 2H), 7.19–7.10 (m, 4H), 1.88 (s, 3H); ^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) δ 168.7, 158.2, 155.8, 151.9, 129.7, 129.6, 127.9, 127.8, 126.9, 126.7, 125.3, 125.0, 124.9, 115.8, 115.6, 114.0, 23.2; ^{19}F NMR (376 MHz, $\text{DMSO-}d_6$) δ -115.6. HRMS (ESI) calcd for $\text{C}_{14}\text{H}_{14}\text{O}_3\text{FN}_2\text{S}_2$, $[\text{M}+\text{H}]^+$: 341.0430; found: 341.0445.

***N*-((4-((naphthalen-2-ylthio)amino)phenyl)sulfonyl)acetamide (4m)**



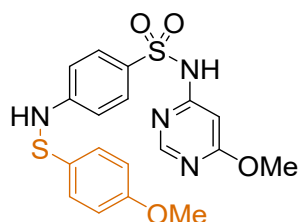
White solid; Yield: 65% (121.0 mg, additive-free synthesis), 56% (104.1 mg, one-pot synthesis); Mp: 155–157 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.86 (s, 1H), 8.85 (s, 1H), 7.90 (d, *J* = 8.7 Hz, 1H), 7.85 (d, *J* = 8.5 Hz, 1H), 7.78 (d, *J* = 8.5 Hz, 1H), 7.75 (d, *J* = 8.9 Hz, 2H), 7.67 (s, 1H), 7.49–7.42 (m, 2H), 7.37 (dd, *J* = 8.6, 1.9 Hz, 1H), 7.20 (d, *J* = 9.0 Hz, 2H), 1.88 (s, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 168.7, 152.4, 137.8, 133.2, 131.4, 129.7, 129.3, 129.0, 127.9, 127.0, 125.7, 121.2, 120.1, 114.0, 23.3; HRMS (ESI) calcd for C₁₈H₁₇O₃N₂S₂, [M+H]⁺: 373.0681; found: 373.0660.

4-(((4-isopropylphenyl)thio)amino)-*N*-(6-methoxypyrimidin-4-yl) benzenesulfonamide (5a)



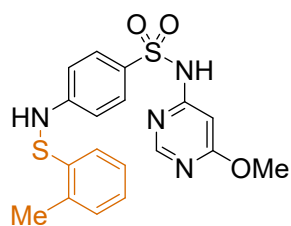
White solid; Yield: 63% (135.5 mg, additive-free synthesis), 56% (120.4 mg, one-pot synthesis); Mp: 166–168 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.47 (s, 1H), 8.35 (s, 1H), 7.81 (d, *J* = 2.2 Hz, 1H), 7.62 (dd, *J* = 8.7, 2.3 Hz, 1H), 7.13 (d, *J* = 8.3 Hz, 2H), 6.99 (d, *J* = 8.3 Hz, 2H), 6.82 (d, *J* = 8.7 Hz, 1H), 6.37 (s, 2H), 6.27 (s, 1H), 3.83 (s, 3H), 2.87–2.76 (m, 1H), 1.15 (d, *J* = 6.9 Hz, 6H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 169.8, 158.8, 153.9, 146.8, 136.6, 132.1, 129.9, 127.7, 127.3, 114.1, 112.5, 90.8, 54.0, 33.0, 23.8; HRMS (ESI) calcd for C₂₀H₂₃O₃N₄S₂, [M+H]⁺: 431.1212; found: 431.1226.

4-(((4-methoxyphenyl)thio)amino)-*N*-(6-methoxypyrimidin-4-yl) benzenesulfonamide (5b)



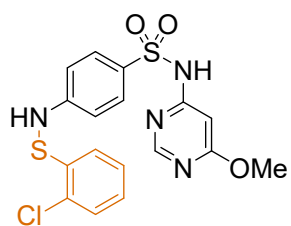
White solid; Yield: 31% (64.8 mg, *additive-free synthesis*); Mp: 122–123 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.68 (s, 1H), 8.59 (s, 1H), 8.38 (d, *J* = 1.0 Hz, 1H), 7.72 (d, *J* = 8.8 Hz, 2H), 7.21 (d, *J* = 8.8 Hz, 2H), 7.10 (d, *J* = 8.8 Hz, 2H), 6.91 (d, *J* = 8.8 Hz, 2H), 6.30 (d, *J* = 1.0 Hz, 1H), 3.82 (s, 3H), 3.70 (s, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 169.8, 158.7, 158.4, 152.1, 130.0, 128.9, 126.7, 114.9, 114.0, 90.7, 56.0, 55.2, 54.0, 18.6; HRMS (ESI) calcd for C₁₈H₁₇O₄N₄S₂, [M+H]⁺: 419.0848; found: 419.0867.

***N*-(6-methoxypyrimidin-4-yl)-4-((*o*-tolylthio)amino)benzenesulfonamide (5c)**



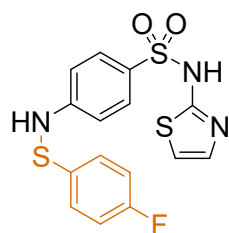
White solid; Yield: 70% (140.7 mg, *additive-free synthesis*), 62% (124.6 mg, *one-pot synthesis*); Mp: 132–134 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.72 (s, 1H), 8.53 (s, 1H), 8.39 (d, *J* = 0.9 Hz, 1H), 7.75 (d, *J* = 8.8 Hz, 2H), 7.17 (d, *J* = 7.0 Hz, 1H), 7.12–7.08 (m, 3H), 7.06–7.02 (m, 1H), 6.96 (dd, *J* = 7.8, 1.4 Hz, 1H), 6.31 (d, *J* = 0.9 Hz, 1H), 3.82 (s, 3H), 2.24 (s, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 169.9, 158.7, 151.9, 138.3, 131.8, 130.4, 129.0, 126.5, 125.3, 121.4, 114.1, 90.8, 54.1, 18.1; HRMS (ESI) calcd for C₁₈H₁₉O₃N₄S₂, [M+H]⁺: 403.0899; found: 403.0918.

4-(((2-chlorophenyl)thio)amino)-*N*-(6-methoxypyrimidin-4-yl)benzenesulfonamide (5d)



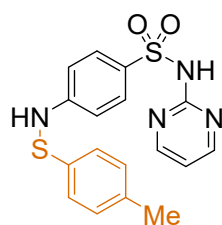
White solid; Yield: 83% (175.1 mg, *additive-free synthesis*), 69% (145.6 mg, *one-pot synthesis*); Mp: 124–126 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.75 (s, 1H), 8.66 (s, 1H), 8.39 (s, 1H), 7.77 (d, *J* = 8.8 Hz, 2H), 7.45 (d, *J* = 6.9 Hz, 1H), 7.30–7.26 (m, 1H), 7.20–7.15 (m, 1H), 7.10 (d, *J* = 8.9 Hz, 2H), 6.99 (dd, *J* = 8.0, 1.6 Hz, 1H), 6.30 (s, 1H), 3.83 (s, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 169.8, 158.8, 151.2, 138.5, 129.7, 129.1, 127.9, 126.8, 126.5, 123.0, 114.2, 90.8, 54.1, 29.5; HRMS (ESI) calcd for C₁₈H₁₆O₃ClN₄S₂, [M+H]⁺: 423.0352; found: 423.0363.

4-(((4-fluorophenyl)thio)amino)-N-(thiazol-2-yl)benzenesulfonamide (5e)



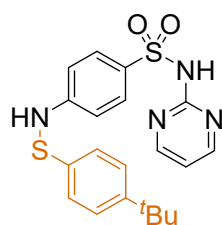
Yellow solid; Yield: 20% (38.1 mg, *additive-free synthesis*); Mp: 118–120 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 12.57 (s, 1H), 8.54 (s, 1H), 7.62 (d, *J* = 8.7 Hz, 2H), 7.24–7.16 (m, 5H), 7.07 (d, *J* = 8.7 Hz, 2H), 6.77 (d, *J* = 4.6 Hz, 1H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 168.4, 161.9, 159.5, 150.7, 135.8 (d, *J* = 2.9 Hz), 132.9, 127.7, 125.1, 125.0, 116.4, 116.2, 113.9, 107.9; ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -117.0; HRMS (ESI) calcd for C₁₅H₁₃O₂FN₃S₃, [M+H]⁺: 382.0154; found: 382.0161.

N-(pyrimidin-2-yl)-4-(*p*-tolylthio)amino)benzenesulfonamide (5f)



White solid; Yield: 66% (122.7 mg, *additive-free synthesis*), 57% (105.9 mg, *one-pot synthesis*); Mp: 138–140 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.53 (s, 1H), 8.58 (s, 1H), 8.48 (d, *J* = 4.8 Hz, 2H), 7.80 (d, *J* = 8.9 Hz, 2H), 7.12 (d, *J* = 8.1 Hz, 2H), 7.10–7.06 (m, 4H), 7.03–7.00 (m, 1H), 2.21 (s, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 158.4, 157.1, 151.9, 136.5, 135.3, 130.1, 129.8, 129.7, 123.1, 115.7, 113.7, 20.5; HRMS (ESI) calcd for C₁₇H₁₇O₂N₄S₂, [M+H]⁺: 373.0793; found: 373.0798.

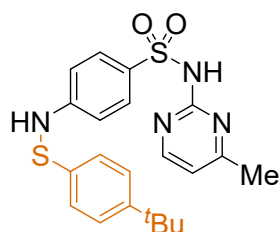
4-(((4-*tert*-butyl)phenyl)thio)amino)-N-(pyrimidin-2-yl)benzenesulfonamide (5g)



White solid; Yield: 61% (126.2 mg, *additive-free synthesis*), 53% (109.7 mg, *one-pot synthesis*); Mp: 129–131 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.53 (s, 1H), 8.59 (s, 1H), 8.48 (d, *J* = 4.8 Hz, 2H), 7.81 (d, *J* = 8.9 Hz, 2H), 7.33 (d, *J* = 8.6 Hz, 2H), 7.11–7.09 (m, 4H), 7.01–7.00 (m, 1H), 1.21 (s, 9H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ

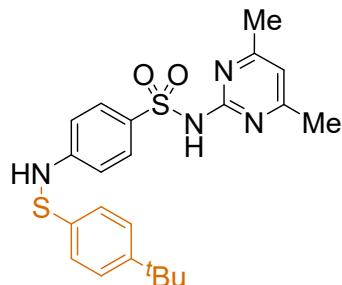
158.4, 157.1, 151.9, 148.6, 136.7, 130.1, 129.7, 126.1, 122.8, 115.7, 113.7, 34.2, 31.1; HRMS (ESI) calcd for C₂₀H₂₃O₂N₄S₂, [M+H]⁺: 415.1262; found: 415.1265.

4-(((4-(*tert*-butyl)phenyl)thio)amino)-*N*-(4-methylpyrimidin-2-yl) benzenesulfonamide (5h)



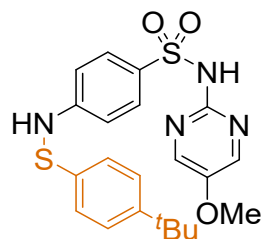
White solid; Yield: 45% (96.4 mg, *additive-free synthesis*); Mp: 165–167 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.42 (s, 1H), 8.57 (s, 1H), 8.29 (d, *J* = 5.1 Hz, 1H), 7.82 (d, *J* = 9.1 Hz, 2H), 7.32 (d, *J* = 8.3 Hz, 2H), 7.09 (d, *J* = 8.0 Hz, 4H), 6.86 (d, *J* = 5.1 Hz, 1H), 2.27 (s, 3H), 1.20 (s, 9H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 168.2, 157.7, 156.8, 151.9, 148.6, 136.7, 130.2, 130.0, 126.1, 122.9, 114.9, 113.6, 34.2, 31.1, 23.4; HRMS (ESI) calcd for C₂₀H₂₃O₂N₄S₂, [M+H]⁺: 429.1419; found: 429.1457.

4-(((4-(*tert*-butyl)phenyl)thio)amino)-*N*-(4,6-dimethylpyrimidin-2-yl) benzenesulfonamide (5i)



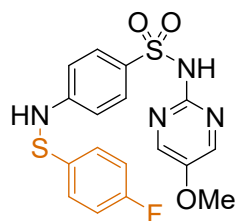
White solid; Yield: 29% (64.1 mg, *additive-free synthesis*); Mp: 172–174 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.37 (s, 1H), 8.55 (s, 1H), 7.85–7.81 (m, 2H), 7.33–7.29 (m, 2H), 7.10–7.08 (m, 4H), 6.71–6.68 (m, 1H), 2.21 (s, 6H), 1.19 (s, 9H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 167.3, 156.4, 151.7, 148.5, 136.6, 130.3, 130.1, 126.0, 122.8, 113.7, 113.3, 34.1, 31.0, 23.0; HRMS (ESI) calcd for C₂₂H₂₇O₂N₄S₂, [M+H]⁺: 443.1575; found: 443.1565.

4-(((4-(*tert*-butyl)phenyl)thio)amino)-*N*-(5-methoxypyrimidin-2-yl) benzenesulfonamide (5j)



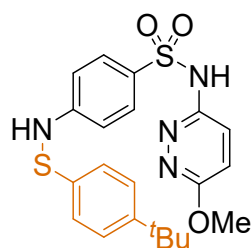
White solid; Yield: 61% (135.4 mg, *additive-free synthesis*), 45% (100.1 mg, *one-pot synthesis*); Mp: 130–131 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.24 (s, 1H), 8.57 (s, 1H), 8.26 (s, 2H), 7.80–7.78 (m, 2H), 7.34–7.31 (m, 2H), 7.11–7.08 (m, 4H), 3.76 (s, 3H), 1.19 (s, 9H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 151.8, 151.3, 149.5, 148.6, 144.6, 136.7, 130.5, 129.6, 126.1, 122.9, 113.7, 56.3, 34.2, 31.1; HRMS (ESI) calcd. for C₂₁H₂₅O₃N₄S₂ [M+H]⁺: 445.1368; found: 445.1394.

4-(((4-fluorophenyl)thio)amino)-*N*-(5-methoxypyrimidin-2-yl) benzenesulfonamide (5k)



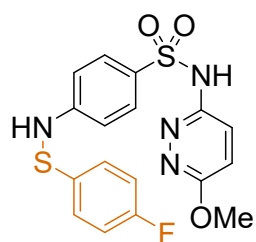
Yellow solid; Yield: 68% (138.0 mg, *additive-free synthesis*), 35% (71.1 mg, *one-pot synthesis*); Mp: 155–157 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.24 (s, 1H), 8.62 (s, 1H), 8.26 (s, 2H), 7.78 (d, *J* = 8.6 Hz, 2H), 7.24–7.16 (m, 4H), 7.08 (d, *J* = 8.8 Hz, 2H), 3.78 (s, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 162.0, 159.5, 151.3, 149.4, 144.6, 135.6, 135.6, 130.8, 129.5, 125.2, 125.1, 116.4, 116.2, 113.7, 56.3; ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -116.9; HRMS (ESI) calcd for C₁₇H₁₆O₃FN₄S₂, [M+H]⁺: 407.0648; found: 407.0686.

4-(((4-(*tert*-butyl)phenyl)thio)amino)-*N*-(6-methoxypyridazin-3-yl) benzenesulfonamide (5l)



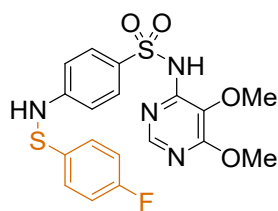
White solid; Yield: 85% (188.7 mg, *additive-free synthesis*), 66% (146.5 mg, *one-pot synthesis*); Mp: 142–144 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 12.61 (s, 1H), 8.50 (s, 1H), 7.66 (d, *J* = 8.8 Hz, 3H), 7.34–7.28 (m, 3H), 7.11–7.06 (m, 4H), 3.82 (s, 3H), 1.20 (s, 9H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 158.9, 152.7, 151.2, 148.6, 136.8, 132.4, 128.2, 126.1, 125.8, 124.4, 122.8, 114.0, 54.5, 34.2, 31.1; HRMS (ESI) calcd for C₂₁H₂₅O₃N₄S₂, [M+H]⁺: 445.1368; found: 445.1394.

4-(((4-fluorophenyl)thio)amino)-*N*-(6-methoxypyridazin-3-yl)benzenesulfonamide (5m)



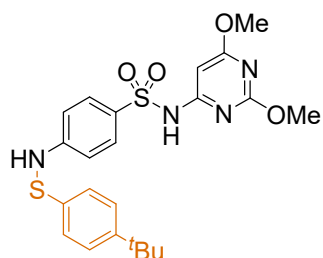
Colorless oil; Yield: 40% (81.2 mg, *additive-free synthesis*); ¹H NMR (400 MHz, DMSO-*d*₆) δ 12.65 (s, 1H), 8.57 (s, 1H), 7.66 (d, *J* = 8.8 Hz, 3H), 7.30 (d, *J* = 9.8 Hz, 1H), 7.24–7.26 (m, 4H), 7.07 (d, *J* = 8.8 Hz, 2H), 3.83 (s, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 162.0, 159.5, 152.7, 150.9, 135.7 (d, *J* = 2.8 Hz), 132.7, 128.2, 125.9, 125.2, 125.1, 116.4, 116.2, 114.1, 54.5; ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -117.0; HRMS (ESI) calcd for C₁₇H₁₆O₃FN₄S₂, [M+H]⁺: 407.0648; found: 407.0645.

***N*-(5,6-dimethoxypyrimidin-4-yl)-4-(((4-fluorophenyl)thio)amino)benzenesulfonamide (5n)**



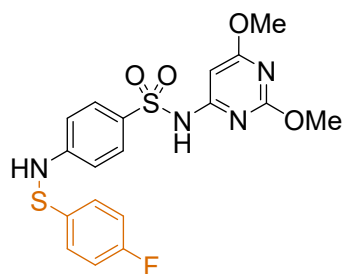
White solid; Yield: 70% (152.6 mg, *additive-free synthesis*), 60% (130.8 mg, *one-pot synthesis*); Mp: 158–160 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.93 (s, 1H), 8.65 (s, 1H), 8.10 (s, 1H), 7.82 (d, *J* = 8.9 Hz, 2H), 7.25–7.18 (m, 4H), 7.09 (d, *J* = 9.0 Hz, 2H), 3.89 (s, 3H), 3.66 (s, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 162.0, 161.5, 159.5, 151.51, 150.7, 135.6, 135.5, 129.6, 127.0, 125.2, 125.1, 116.5, 116.2, 113.7, 60.2, 54.1; ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -117.0; HRMS (ESI) calcd for C₁₈H₁₈O₄FN₄S₂, [M+H]⁺: 437.0754; found: 437.0761.

4-(((4-(*tert*-butyl)phenyl)thio)amino)-*N*-(2,6-dimethoxypyrimidin-4-yl) benzenesulfonamide (5o)



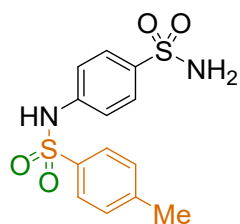
White solid; Yield: 69% (163.5 mg, *additive-free synthesis*), 57% (135.1 mg, *one-pot synthesis*); Mp: 170–172 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.35 (s, 1H), 8.65 (s, 1H), 7.75 (d, *J* = 9.1 Hz, 2H), 7.33 (dd, *J* = 8.5, 2.2 Hz, 2H), 7.13–7.09 (m, 4H), 5.93 (s, 1H), 3.77 (s, 3H), 3.71 (s, 3H), 1.20 (s, 9H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 171.7, 164.4, 160.1, 152.2, 148.6, 136.5, 129.6, 129.2, 126.1, 122.9, 114.0, 84.4, 54.5, 53.8, 34.2, 31.0; HRMS (ESI) calcd. for C₂₂H₂₇O₄N₄S₂ [M+H]⁺: 475.1474; found: 475.1470.

***N*-(2,6-dimethoxypyrimidin-4-yl)-4-(((4-fluorophenyl)thio)amino) benzenesulfonamide (5p)**



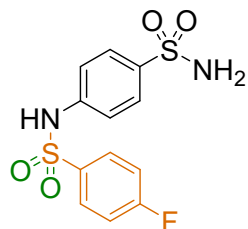
White solid; Yield: 61% (132.9 mg, *additive-free synthesis*), 43% (93.7 mg, *one-pot synthesis*); Mp: 165–167 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.36 (s, 1H), 8.71 (s, 1H), 7.76 (d, *J* = 8.8 Hz, 2H), 7.24–7.16 (m, 4H), 7.14–7.10 (m, 2H), 5.92 (s, 1H), 3.78 (s, 3H), 3.72 (s, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 171.66, 164.37, 161.97, 160.09, 159.57, 151.9, 135.41 (d, *J* = 3.2 Hz), 129.9, 129.3, 125.3, 125.2, 116.4, 116.2, 114.1, 84.4, 54.5, 53.8; ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -116.8; HRMS (ESI) calcd for C₁₈H₁₈O₄FN₄S₂, [M+H]⁺: 437.0754; found: 437.0761.

4-methyl-*N*-(4-sulfamoylphenyl)benzenesulfonamide (6)



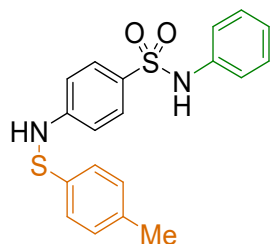
White solid; Yield: 91% (88.9 mg); Mp: 133–135 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.83 (s, 1H), 7.76–7.70(m, 4H), 7.36 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 9.1 Hz, 4H), 2.30 (s, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 143.9, 141.1, 138.8, 136.4, 130.0, 127.3, 126.9, 118.4, 21.1; HRMS (ESI) calcd for C₁₃H₁₄O₄N₂S₂, [M+H]⁺: 327.0473; found: 327.2471.

4-fluoro-*N*-(4-sulfamoylphenyl)benzenesulfonamide (7)



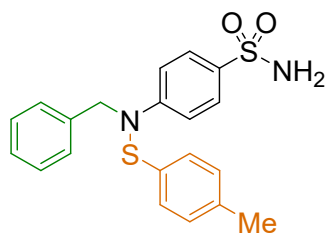
White solid; Yield: 87% (86.1 mg); Mp: 149–151 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.90 (s, 1H), 7.92–7.88 (m, 2H), 7.70–7.68 (m, 2H), 7.45–7.40 (m, 2H), 7.27–7.24 (m, 4H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 164.6 (d, *J* = 252.3 Hz), 140.7, 139.1, 135.5 (d, *J* = 3.2 Hz), 129.9 (d, *J* = 9.6 Hz), 127.3, 118.7, 116.8 (d, *J* = 23.0 Hz); ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -105.0; HRMS (ESI) calcd for C₁₂H₁₁O₄FN₂S₂, [M+H]⁺: 331.0223; found: 331.0208.

N-phenyl-4-((*p*-tolylthio)amino)benzenesulfonamide (8)



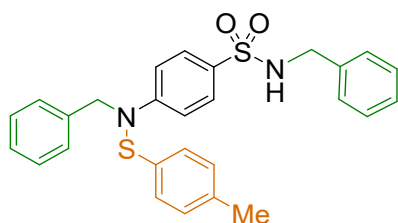
White solid; Yield: 53% (58.8 mg); Mp: 147–149 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.06 (s, 1H), 8.55 (s, 1H), 7.57 (d, *J* = 8.7 Hz, 2H), 7.21–7.17 (m, 2H), 7.12 (d, *J* = 7.9 Hz, 2H), 7.06–6.96 (m, 7H), 2.22 (s, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 151.7, 138.1, 136.4, 135.3, 129.8, 129.43, 129.1, 128.6, 123.6, 123.1, 119.6, 114.1, 20.5; HRMS (ESI) calcd for C₁₉H₁₈O₂N₂S₂, [M+H]⁺: 371.0888; found: 371.0873.

4-(benzyl(*p*-tolylthio)amino)benzenesulfonamide (9)



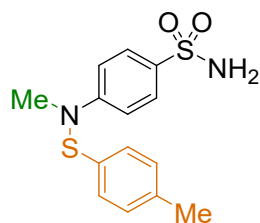
White solid; Yield: 56% (107.5 mg); Mp: 138–140 °C; ^1H NMR (400 MHz, DMSO- d_6) δ 7.70 (d, $J = 9.0$ Hz, 2H), 7.37–7.26 (m, 7H), 7.21–7.18 (m, 4H), 7.12 (d, $J = 8.2$ Hz, 2H), 5.10 (s, 2H), 2.27 (s, 3H); ^{13}C NMR (101 MHz, DMSO- d_6) δ 151.4, 137.4, 136.1, 134.8, 134.7, 130.1, 128.7, 127.3, 126.5, 123.7, 114.9, 59.6, 20.6; HRMS (ESI) calcd for $\text{C}_{20}\text{H}_{20}\text{O}_2\text{N}_2\text{S}_2$, $[\text{M}+\text{H}]^+$: 385.1044; found: 385.1025.

N-benzyl-4-(benzyl(*p*-tolylthio)amino)benzenesulfonamide (9')



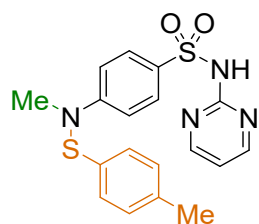
White solid; Yield: 22% (52.1 mg); Mp: 153–155 °C; ^1H NMR (400 MHz, DMSO- d_6) δ 7.94 (t, $J = 6.3$ Hz, 1H), 7.67–7.59 (m, 2H), 7.36 (dd, $J = 7.9, 6.6$ Hz, 2H), 7.32–7.16 (m, 12H), 7.15–7.09 (m, 2H), 5.08 (s, 2H), 3.95 (d, $J = 6.3$ Hz, 2H), 2.28 (s, 3H). ^{13}C NMR (101 MHz, DMSO- d_6) δ 151.9, 137.8, 137.3, 136.1, 134.6, 131.1, 130.1, 128.7, 128.1, 127.5, 127.3, 127.0, 126.5, 123.7, 115.0, 59.5, 46.1, 20.6. HRMS (ESI) calcd for $\text{C}_{27}\text{H}_{27}\text{O}_2\text{N}_2\text{S}_2$, $[\text{M}+\text{H}]^+$: 475.1514; found: 475.1528.

4-(methyl(*p*-tolylthio)amino)benzenesulfonamide (10)

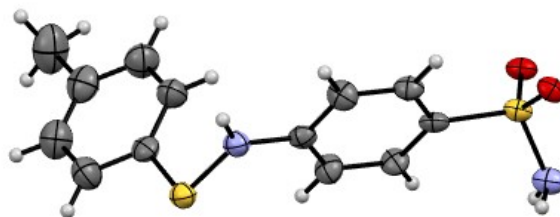


White solid; Yield: 72% (110.9 mg); Mp: 162–163 °C; ^1H NMR (400 MHz, DMSO- d_6) δ 7.74 (d, $J = 9.0$ Hz, 2H), 7.30 (dd, $J = 7.0, 2.1$ Hz, 2H), 7.20–7.15 (m, 4H), 7.03 (dd, $J = 6.4, 2.0$ Hz, 2H), 3.49 (s, 3H), 2.24 (s, 3H); ^{13}C NMR (101 MHz, DMSO- d_6) δ 152.1, 136.0, 134.8, 134.6, 130.1, 127.2, 123.8, 114.5, 44.9, 20.6; HRMS (ESI) calcd for $\text{C}_{14}\text{H}_{16}\text{O}_2\text{N}_2\text{S}_2$, $[\text{M}+\text{H}]^+$: 309.0731; found: 309.0721.

4-(methyl(*p*-tolylthio)amino)-*N*-(pyrimidin-2-yl)benzenesulfonamide (11)



White solid; Yield: 61% (117.7 mg); Mp: 162–164 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 11.55 (s, 1H), 8.48(dd, *J* = 4.8, 1.8 Hz, 2H), 7.85 (dd, *J* = 8.8, 2.0 Hz, 2H), 7.27 (dd, *J* = 8.9, 2.0 Hz, 2H), 7.15 (d, *J* = 7.9 Hz, 2H), 7.09–6.98 (m, 3H), 3.47 (s, 3H), 2.24 (s, 3H); ¹³C NMR (101 MHz, DMSO-*d*₆) δ 158.3, 157.0, 153.0, 136.1, 134.5, 130.0, 129.3, 127.8, 123.9, 115.8, 114.3, 44.9, 20.5; HRMS (ESI) calcd for C₁₈H₁₈O₂N₄S₂, [M+H]⁺: 387.0949; found: 387.0960.

Table S1 Crystal data and structure refinement for **3a**.

CCDC number	2304488
Empirical formula	C ₁₃ H ₁₄ N ₂ O ₂ S ₂
Formula weight	294.38
Temperature/K	150.00(10)
Crystal system	orthorhombic
Space group	P2 ₁ 2 ₁ 2 ₁
a/Å	4.9499(3)
b/Å	13.8952(10)
c/Å	20.1686(16)
α/°	90
β/°	90
γ/°	90
Volume/Å ³	1387.21(17)
Z	4
ρ _{calc} /cm ³	1.410
μ/mm ⁻¹	3.481
F(000)	616.0
Crystal size/mm ³	0.15 × 0.12 × 0.11
Radiation	Cu Kα (λ = 1.54184)
2θ range for data collection/°	8.768 to 133.192
Index ranges	-5 ≤ h ≤ 5, -9 ≤ k ≤ 16, -24 ≤ l ≤ 22
Reflections collected	2903
Independent reflections	2049 [R _{int} = 0.0461, R _{sigma} = 0.0629]
Data/restraints/parameters	2049/133/173
Goodness-of-fit on F ²	1.069
Final R indexes [I ≥ 2σ (I)]	R ₁ = 0.1028, wR ₂ = 0.2746
Final R indexes [all data]	R ₁ = 0.1147, wR ₂ = 0.2893
Largest diff. peak/hole / e Å ⁻³	0.55/-0.52

4. Stability experiment

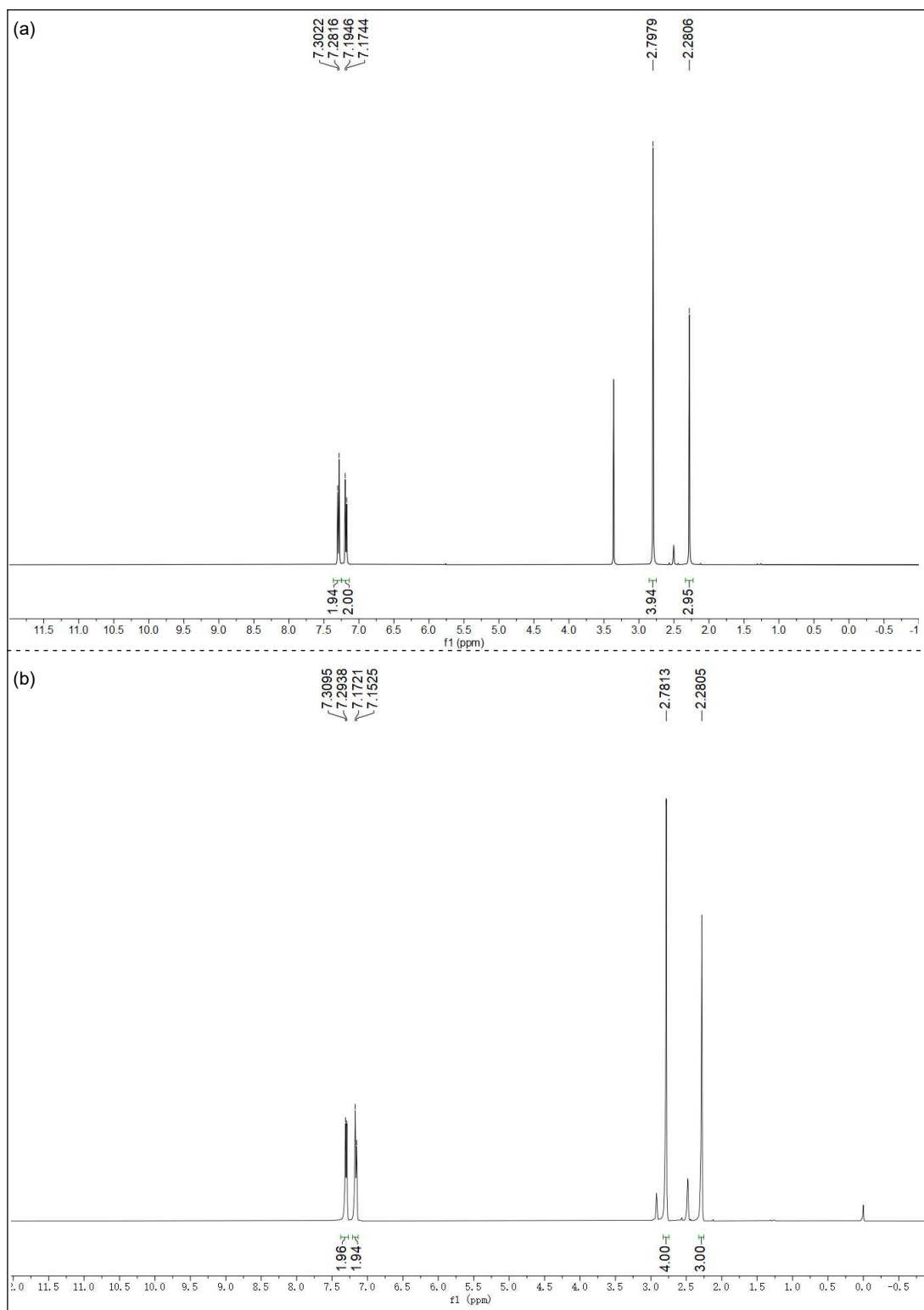
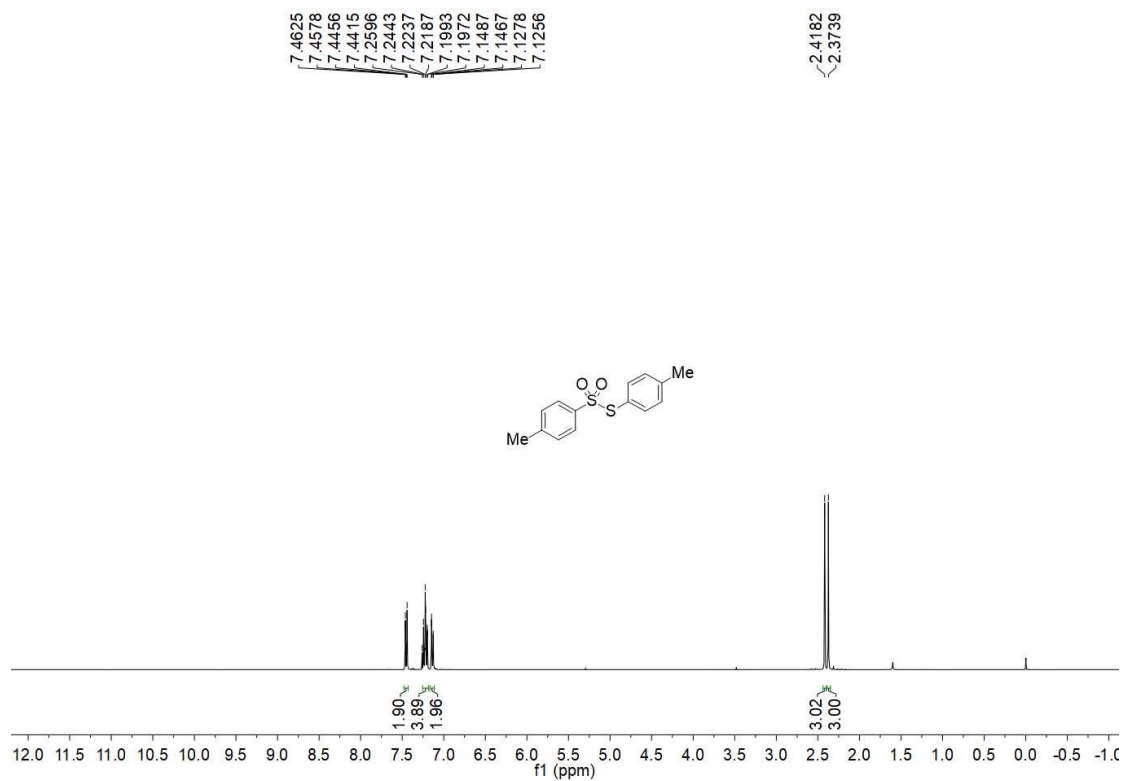


Fig. S4 ^1H NMR (400 MHz, $\text{DMSO-}d_6$) spectra of thiosuccinimide **Su-2a** measured at different temperatures. (a) Performed at room temperature. (b) Performed at 100 °C.

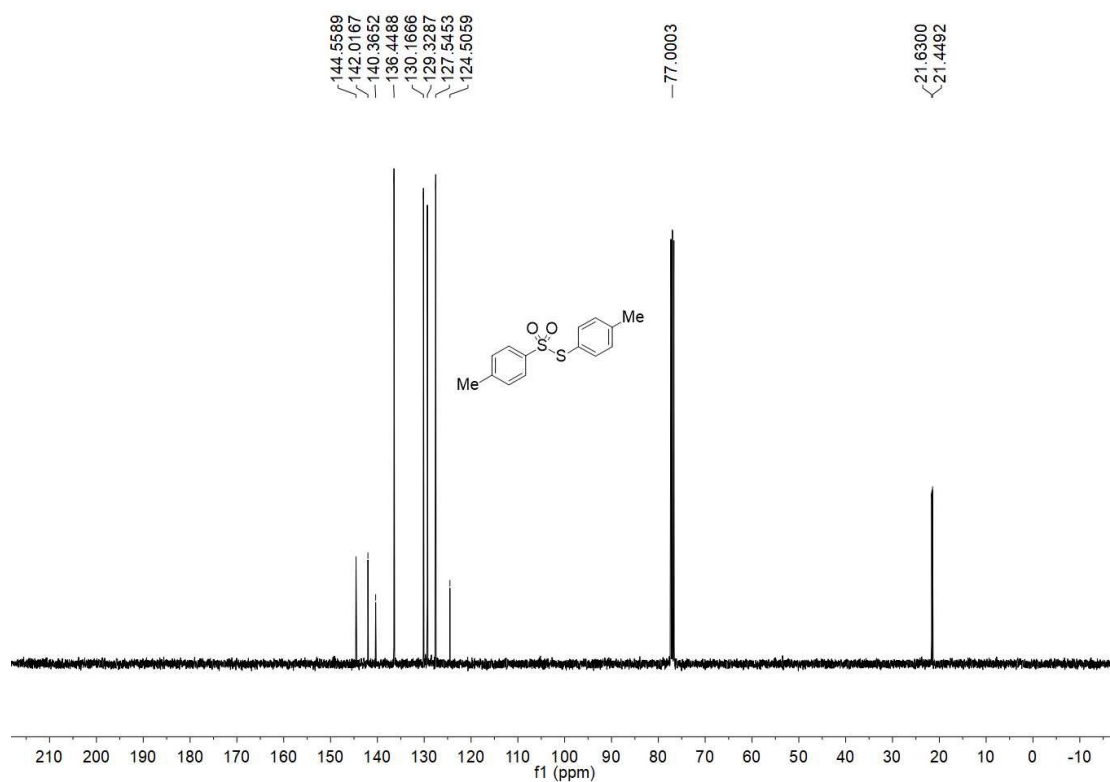
5. References

- 1 T. Keshari , R. Kapoor and L. D. S. Yadav, *Synlett*, 2016, **27**, 1878–1882.
- 2 Y. Li, Y. Wang, F. Fang, Y. Zhang, C. Li, T. Yu, Q. Chen, J. Wang and H. Liu, *Org. Lett.*, 2023, **25**, 6018–6023.
- 3 A. Kesavan, A. K. Sahu and P. Anbarasan, *Org. Lett.*, 2023, **25**, 4765–4769.
- 4 S. Kanikarapu, M. P. Gogoi, S. Dutta and A. K. Sahoo, *Org. Lett.*, 2022, **24**, 8289–8294.
- 5 J. Grover, G. Prakash, C. Teja, G. K. Lahiri and D. Maiti, *Green Chem.*, 2023, **25**, 3431–3436.
- 6 G. S. Kumar, J. Bhattacharjee, K. Kumari, S. Moorthy, A. Bandyopadhyay, Saurabh K. Singh and T. K. Panda, *Polyhedron*, 2022, **219**, 115784.

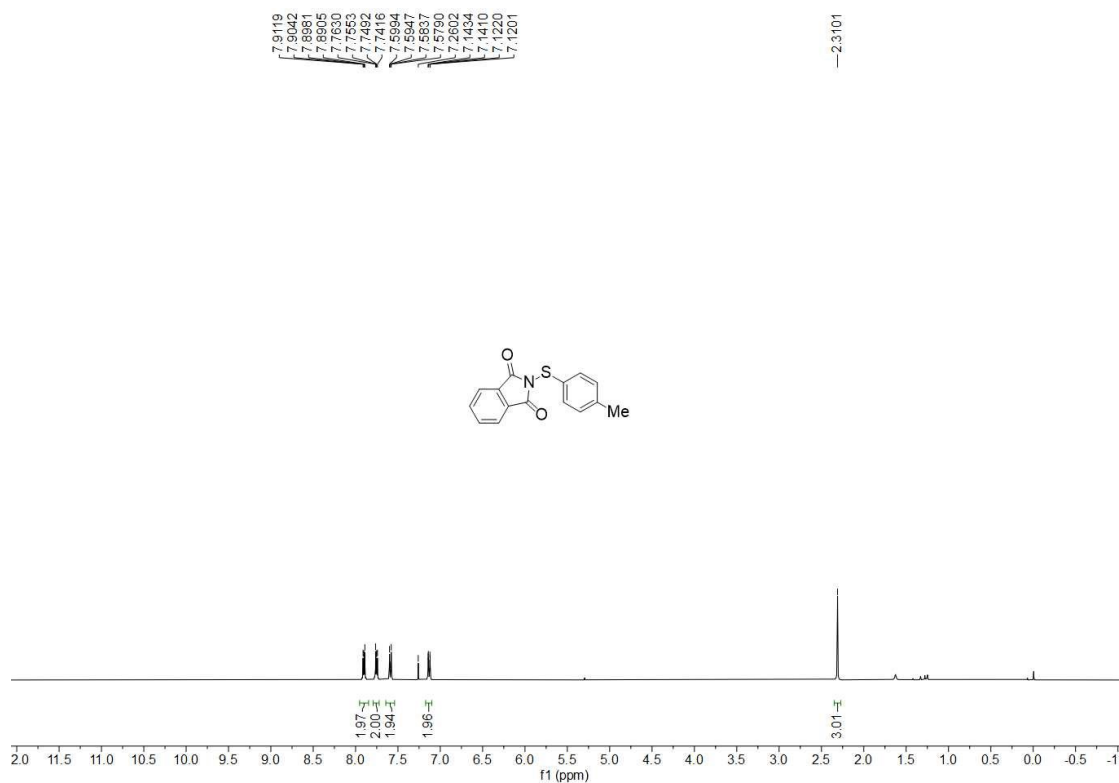
6. NMR spectra



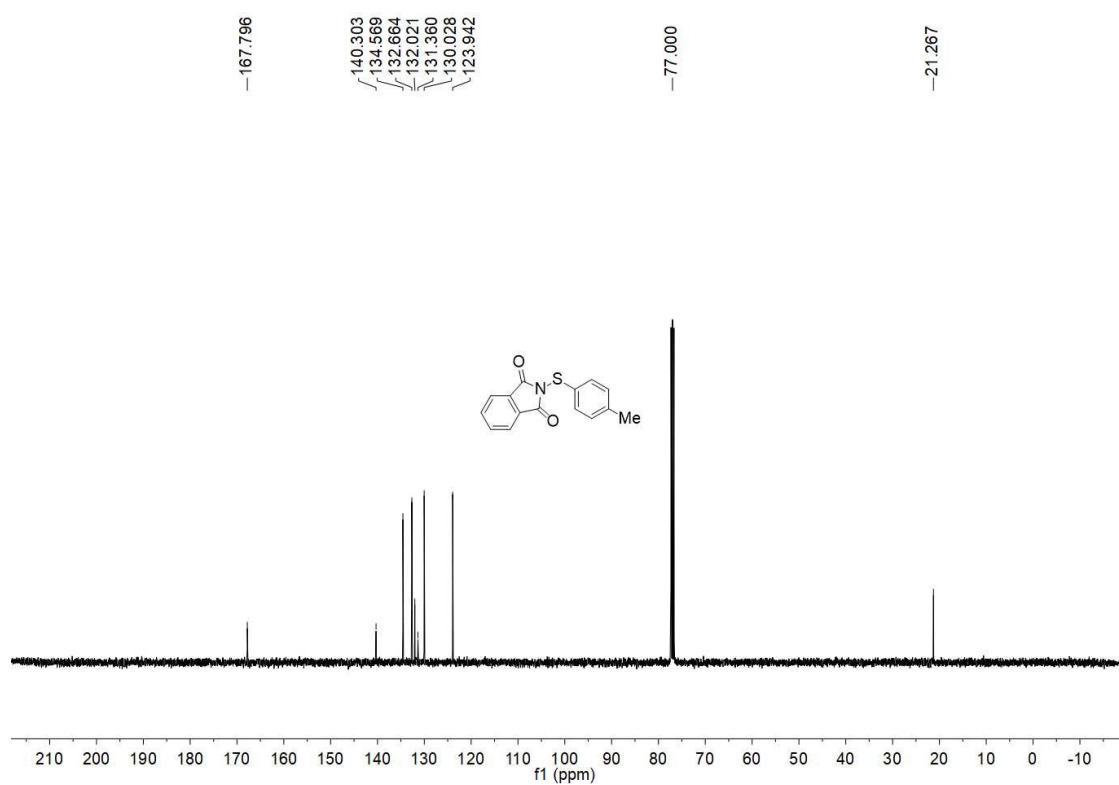
¹H NMR (400 MHz, CDCl₃) spectrum of Compound Ts-2a



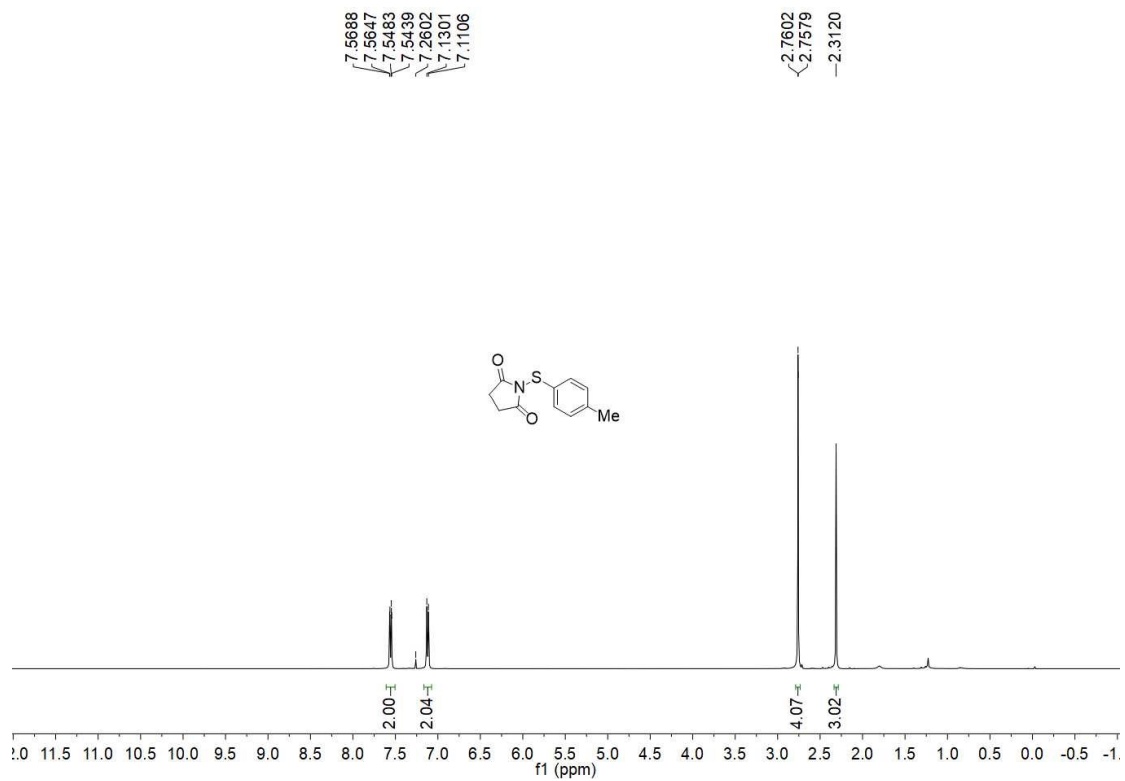
¹³C NMR (101 MHz, CDCl₃) spectrum of Compound Ts-2a



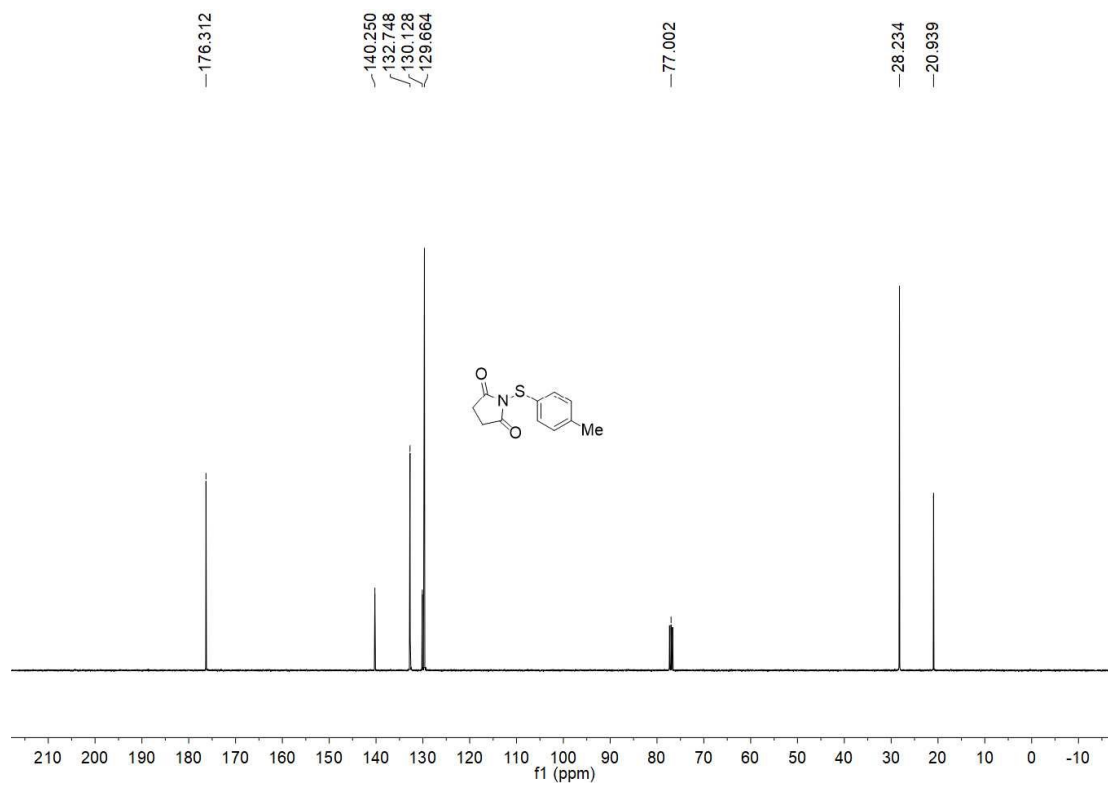
¹H NMR (400 MHz, CDCl₃) spectrum of Compound **Phth-2a**



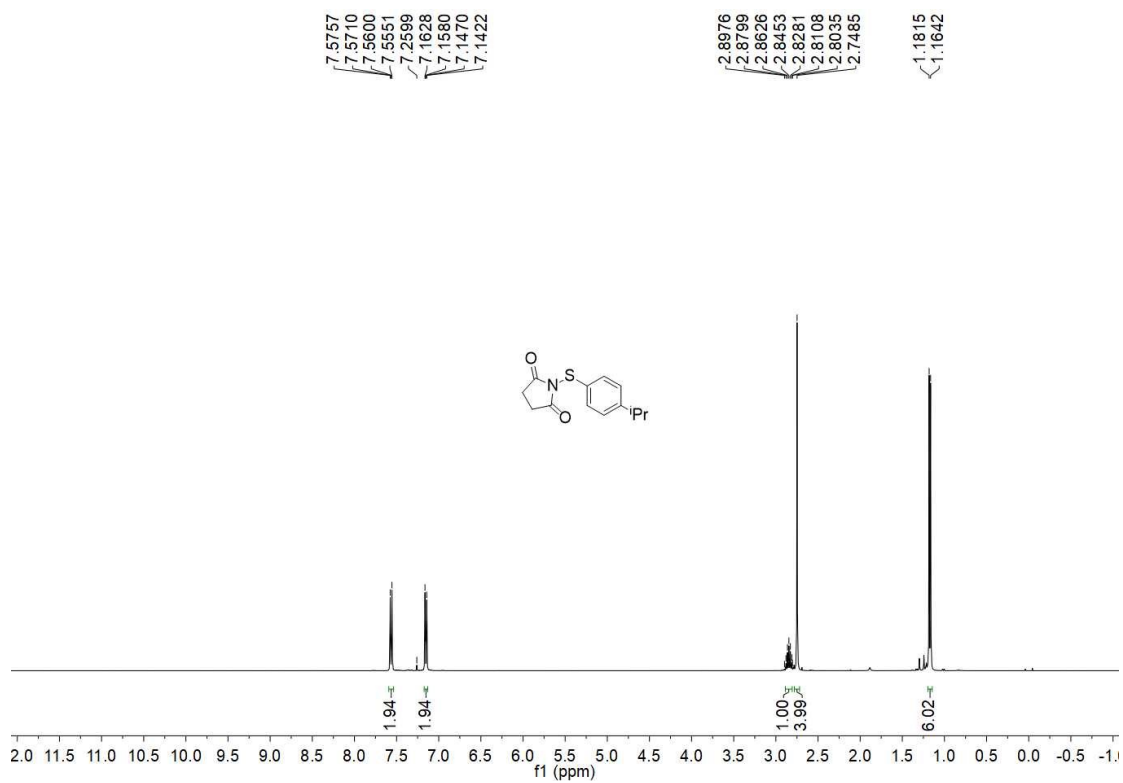
¹³C NMR (101 MHz, CDCl₃) spectrum of Compound **Phth-2a**



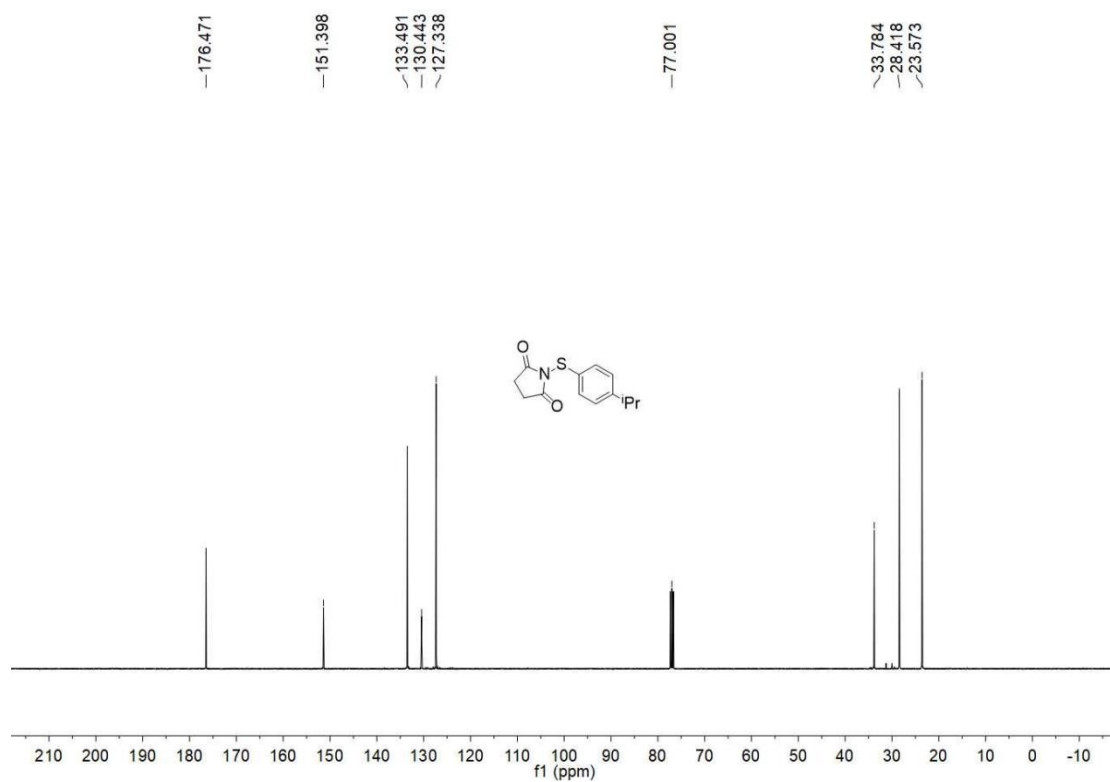
¹H NMR (400 MHz, CDCl₃) spectrum of Compound Su-2a



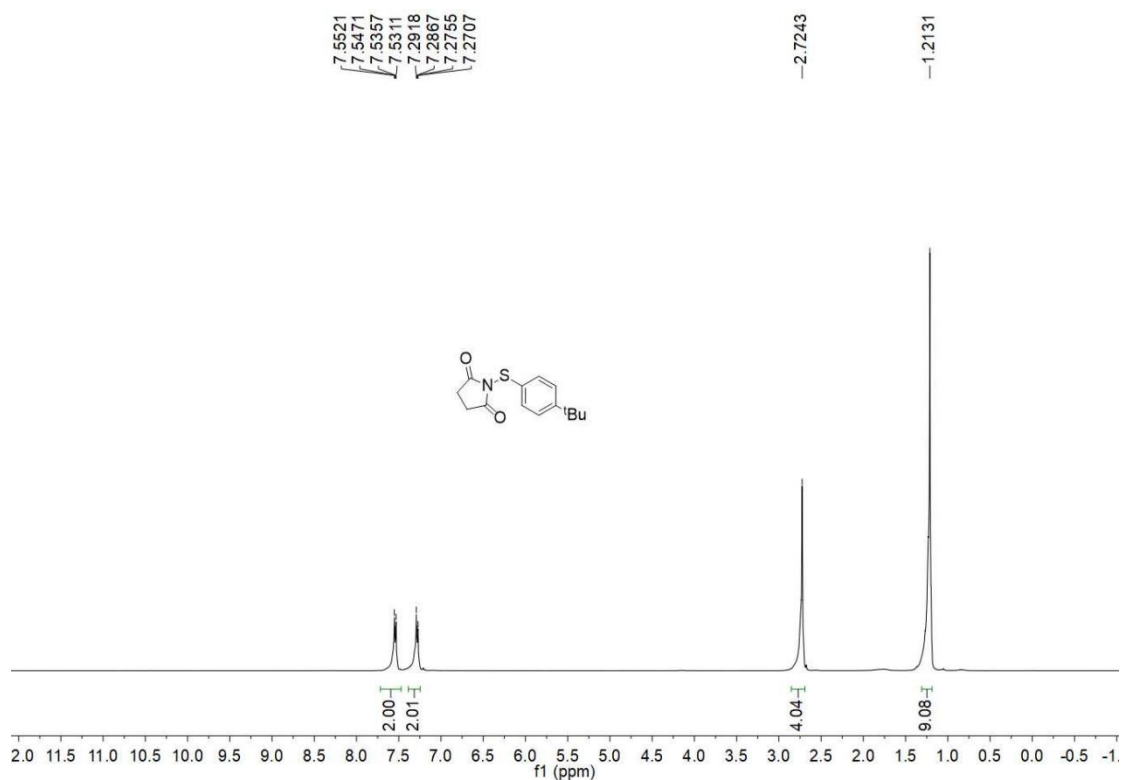
¹³C NMR (101 MHz, CDCl₃) spectrum of Compound Su-2a



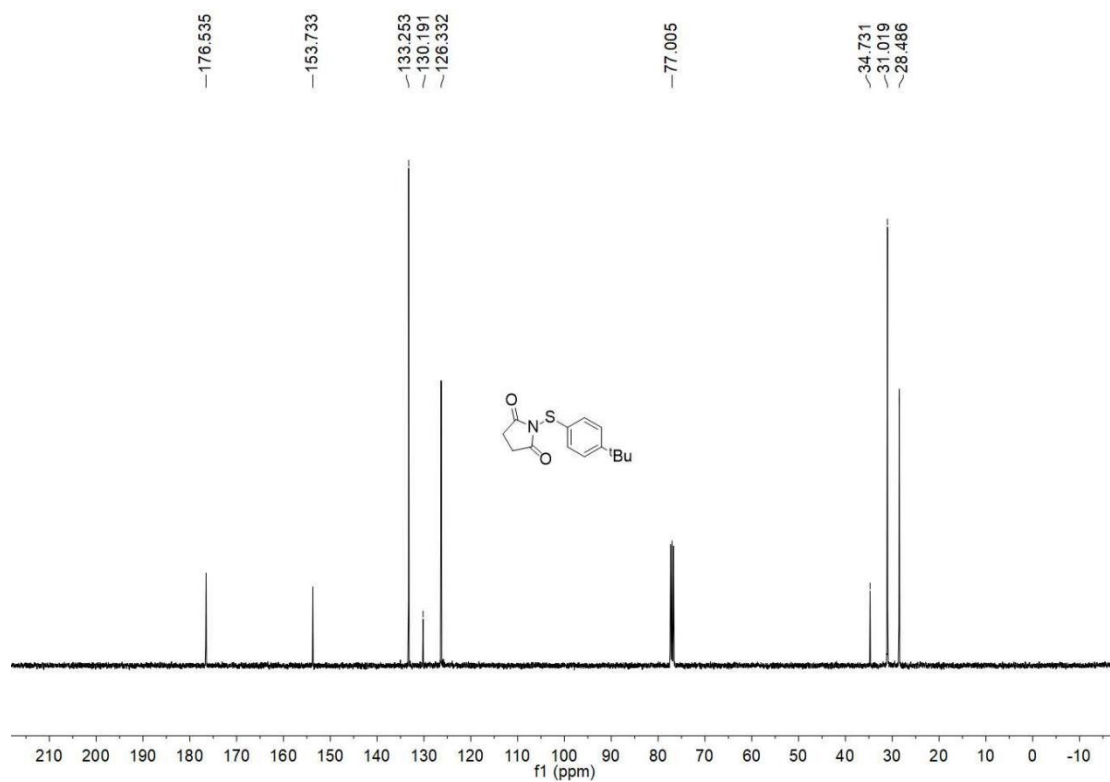
¹H NMR (400 MHz, CDCl₃) spectrum of Compound Su-2b



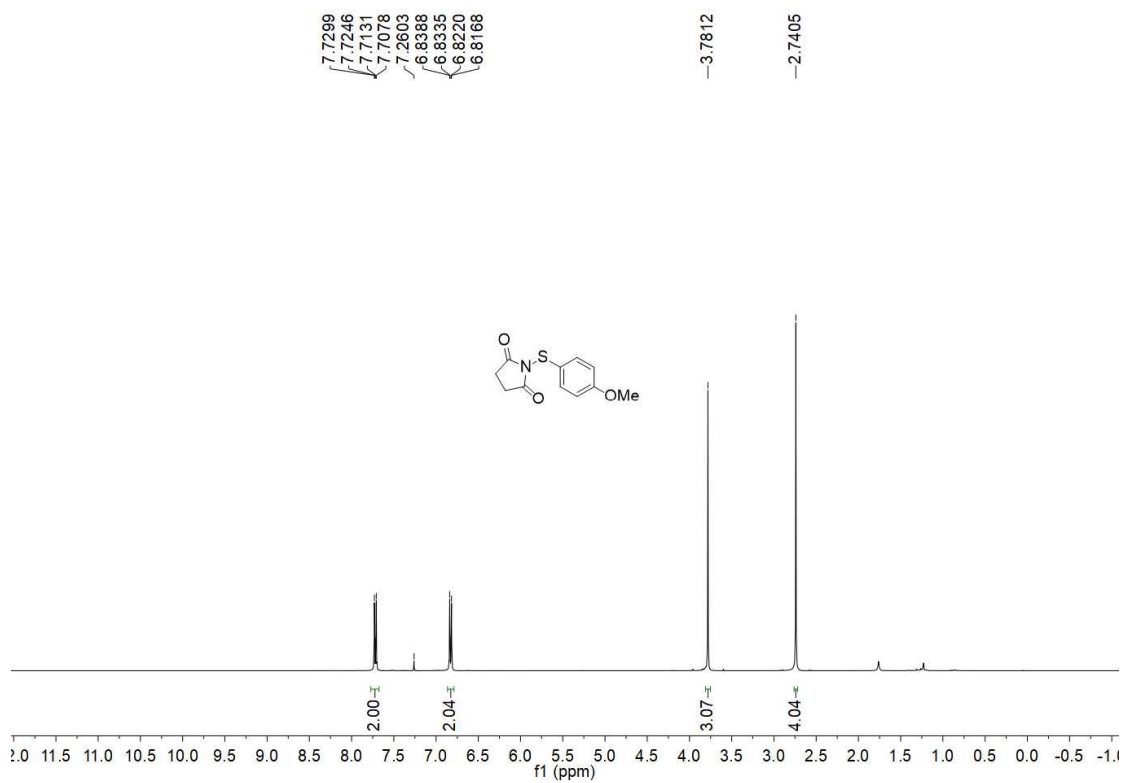
¹³C NMR (101 MHz, CDCl₃) spectrum of Compound Su-2b



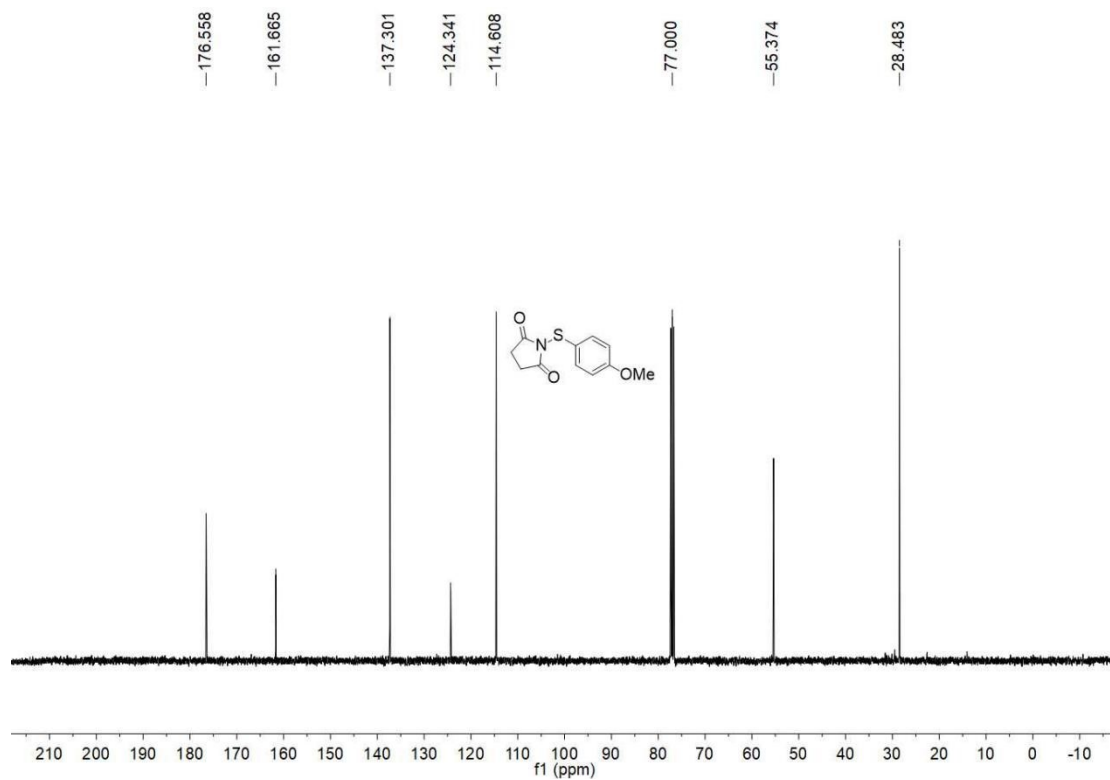
¹H NMR (400 MHz, CDCl₃) spectrum of Compound Su-2c



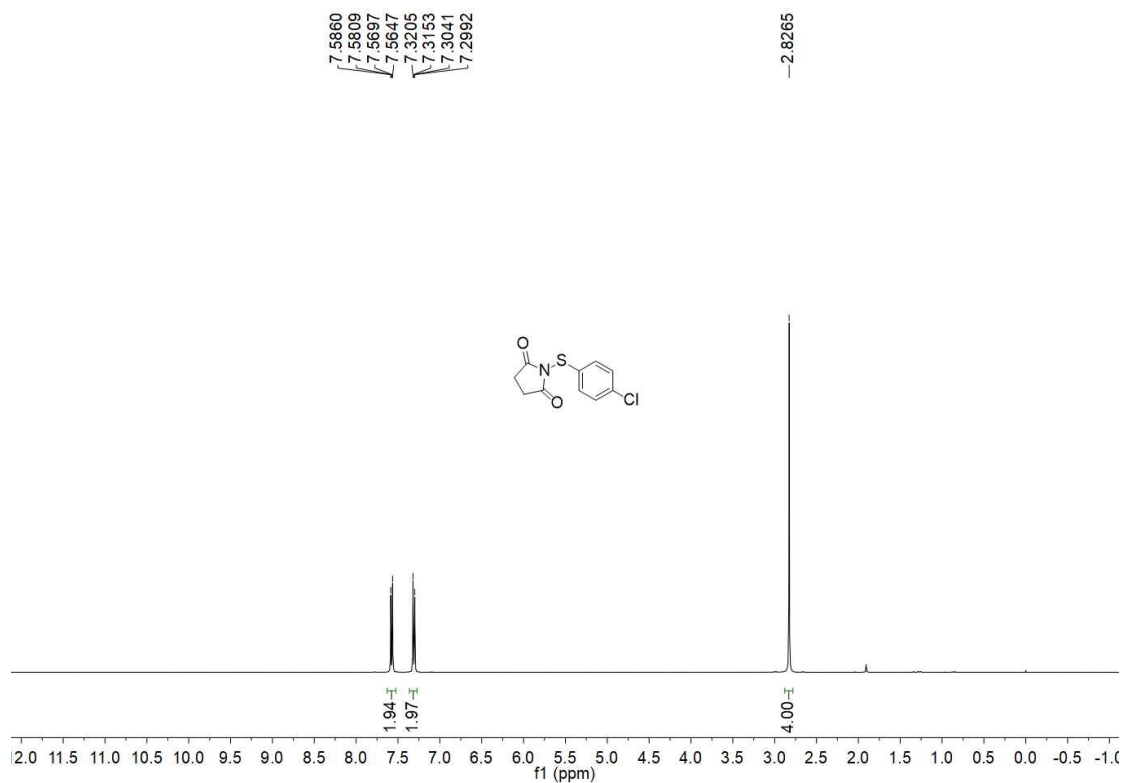
¹³C NMR (101 MHz, CDCl₃) spectrum of Compound Su-2c



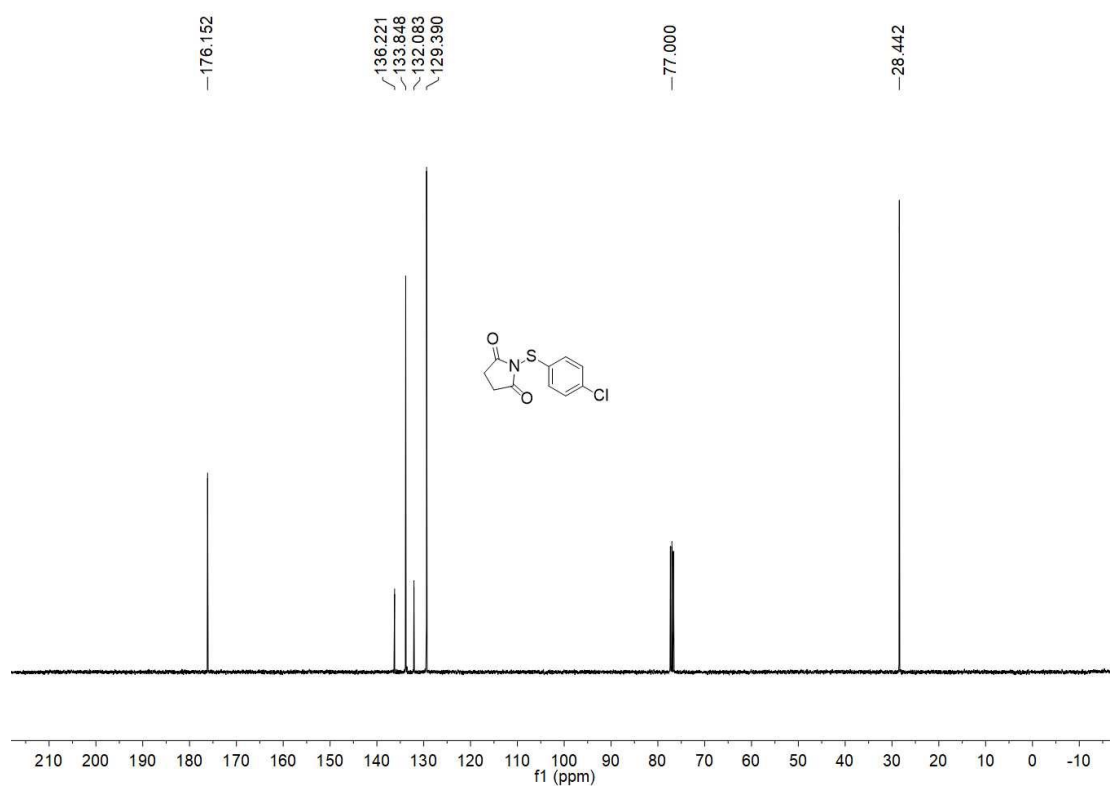
^1H NMR (400 MHz, CDCl_3) spectrum of Compound **Su-2d**



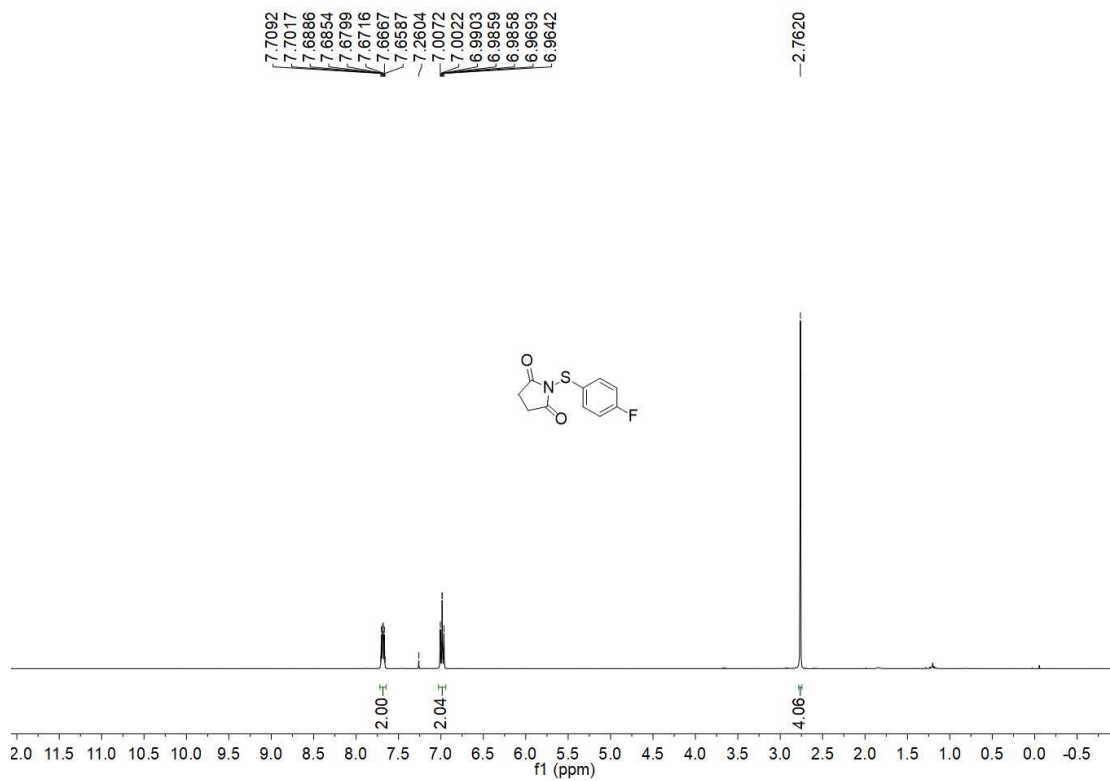
^{13}C NMR (101 MHz, CDCl_3) spectrum of Compound **Su-2d**



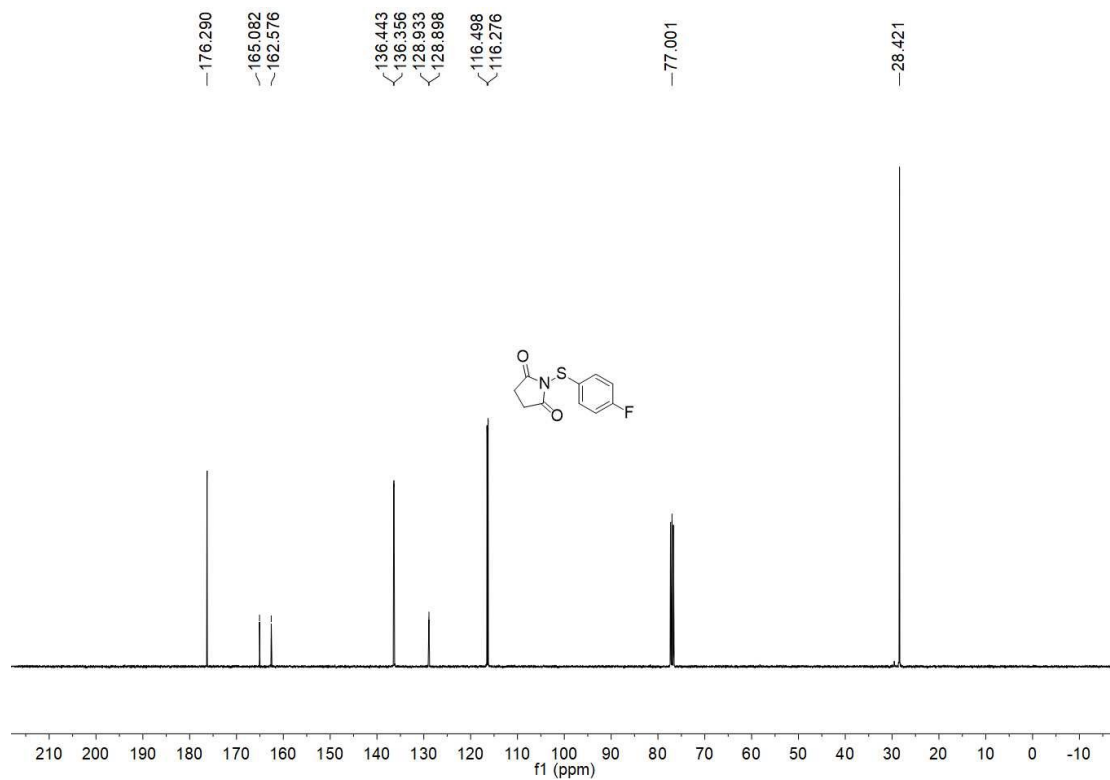
¹H NMR (400 MHz, CDCl₃) spectrum of Compound Su-2e



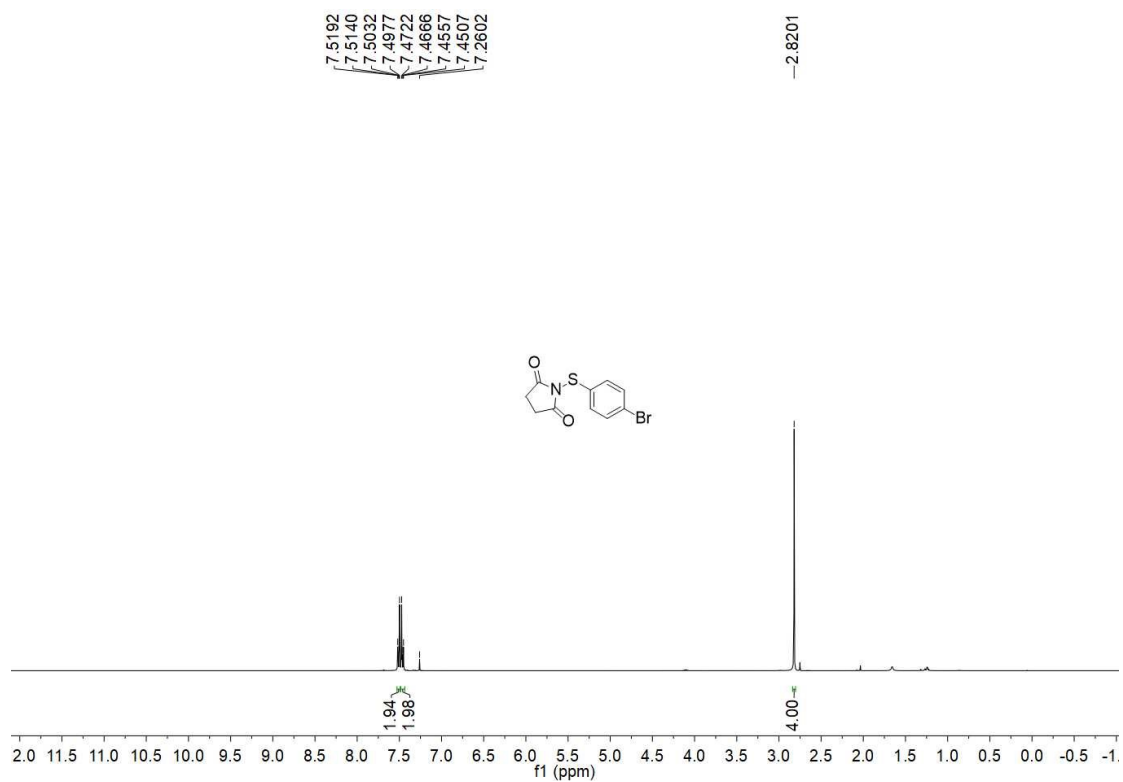
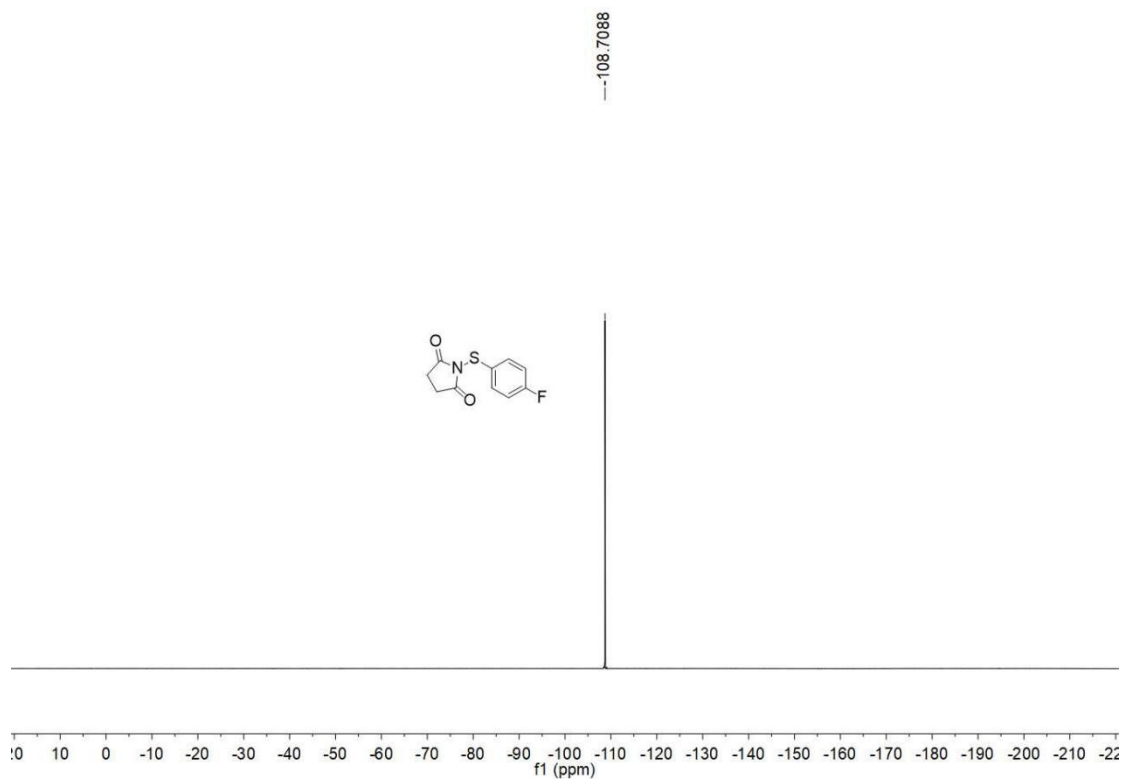
¹³C NMR (101 MHz, CDCl₃) spectrum of Compound Su-2e

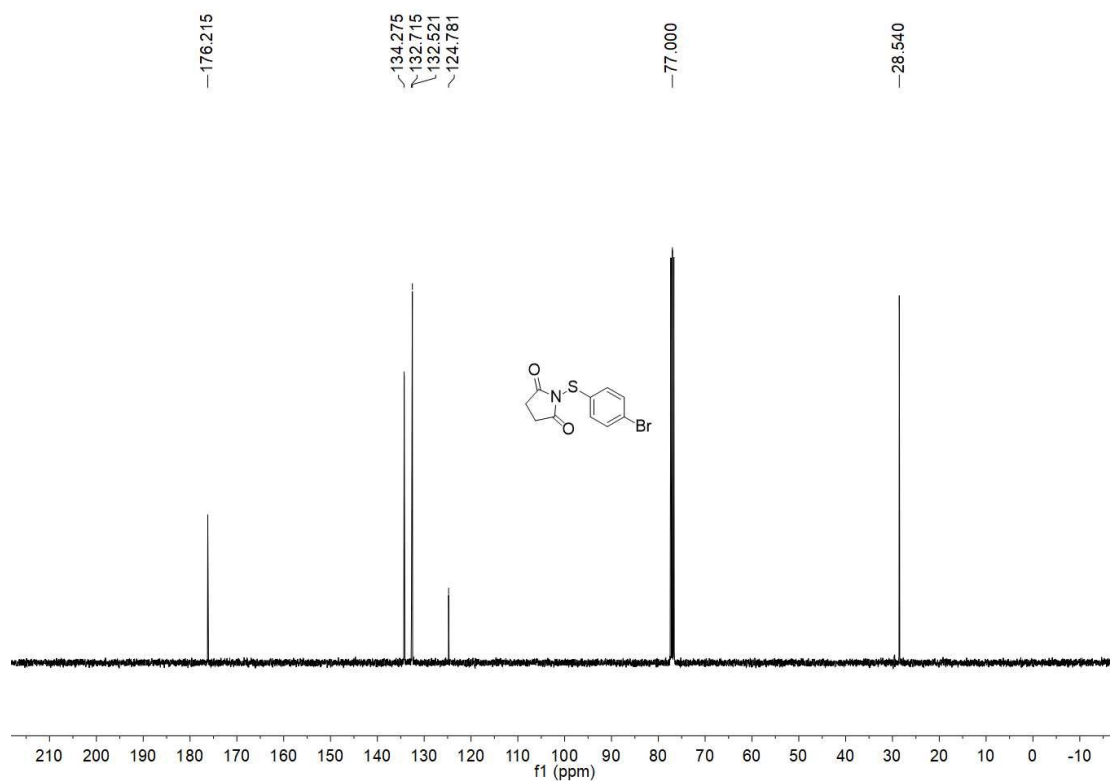


¹H NMR (400 MHz, CDCl₃) spectrum of Compound **Su-2f**

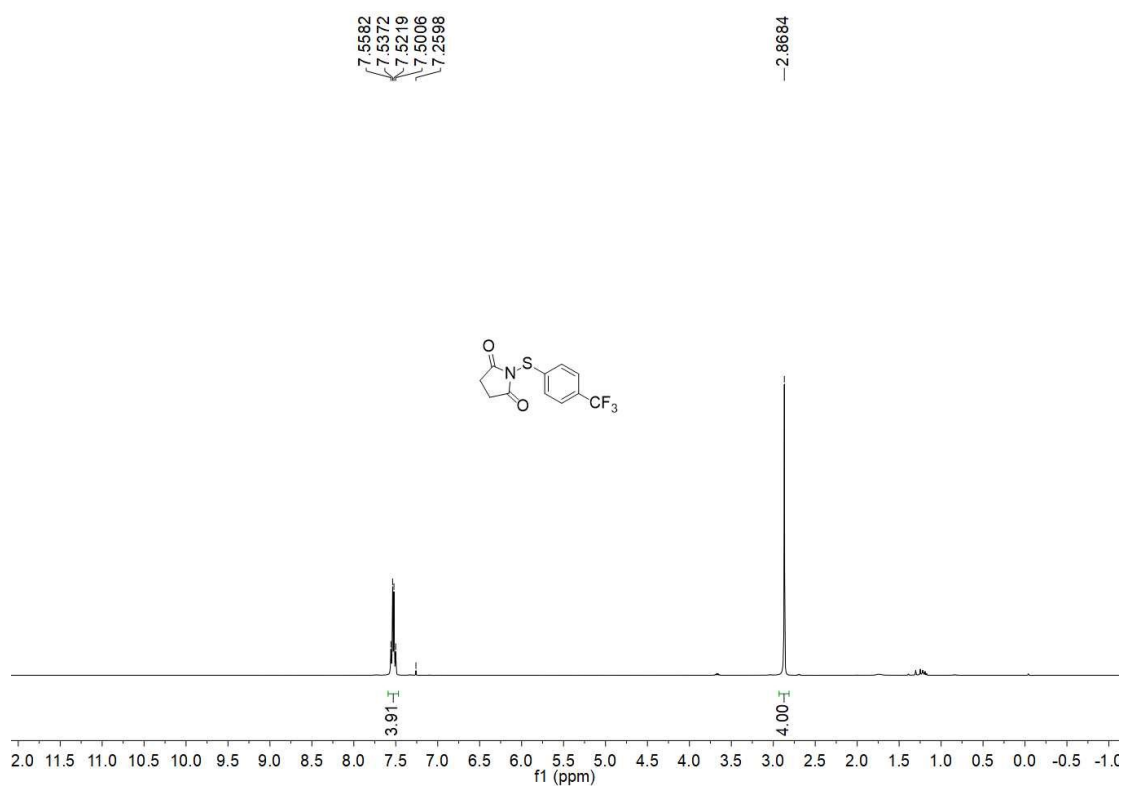


¹³C NMR (101 MHz, CDCl₃) spectrum of Compound **Su-2f**

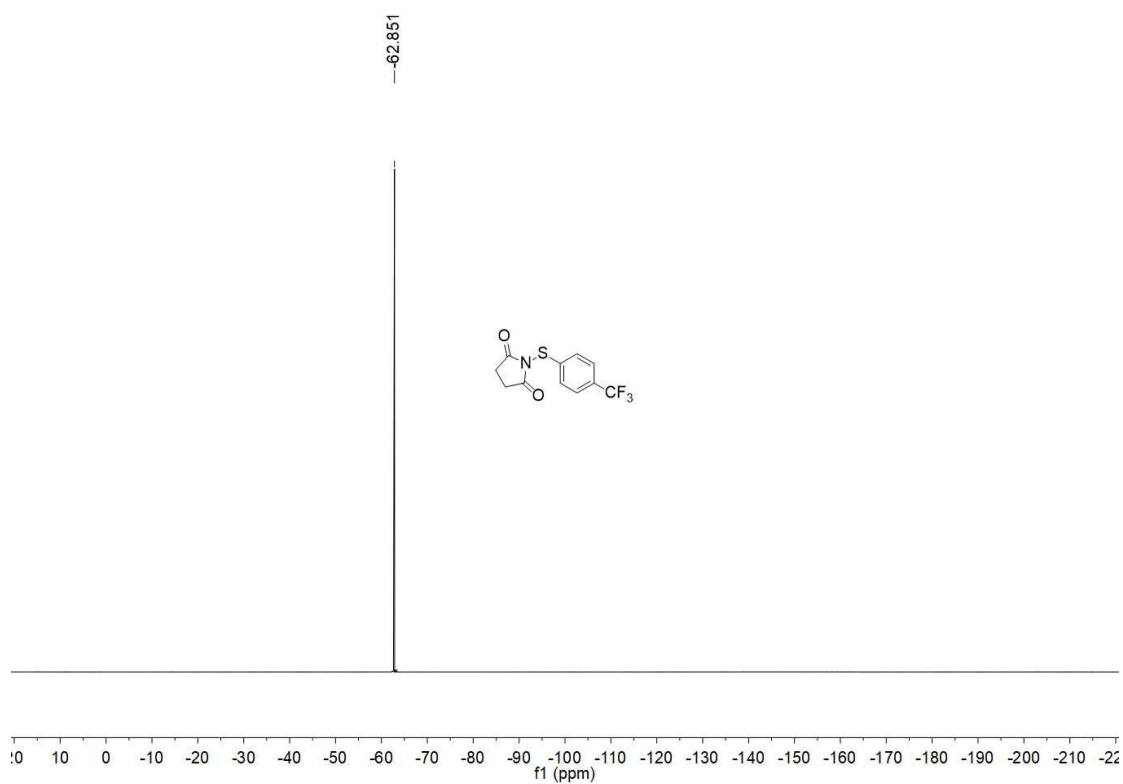
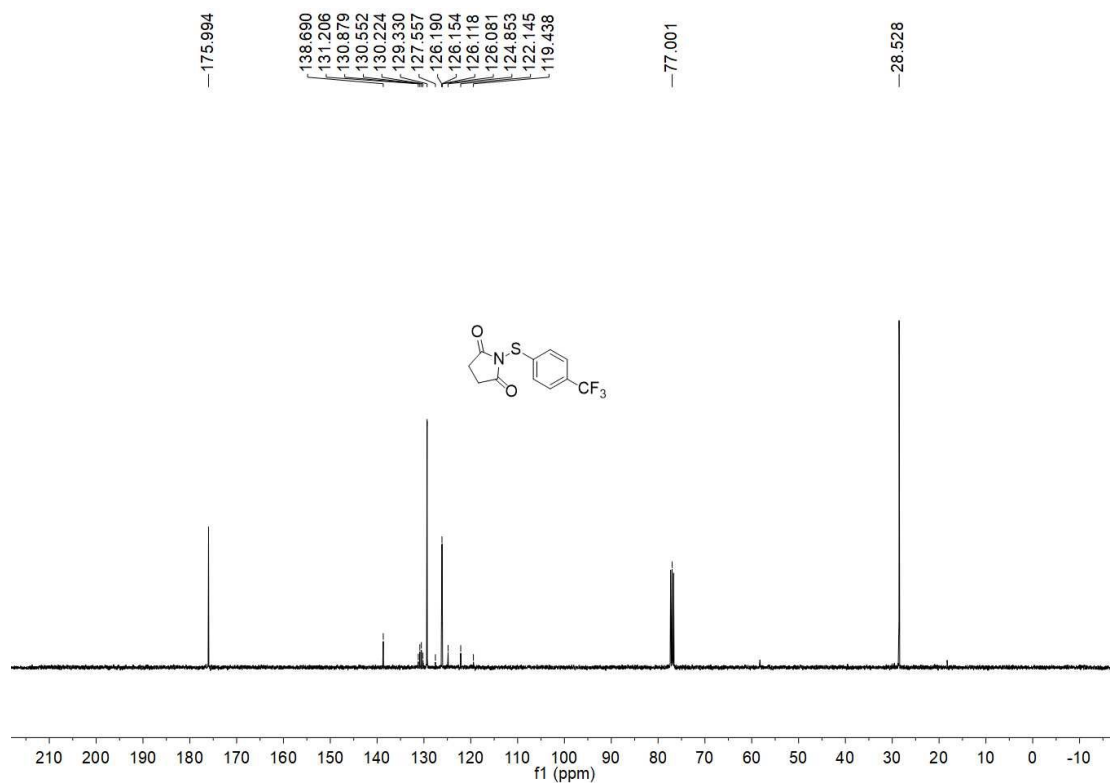




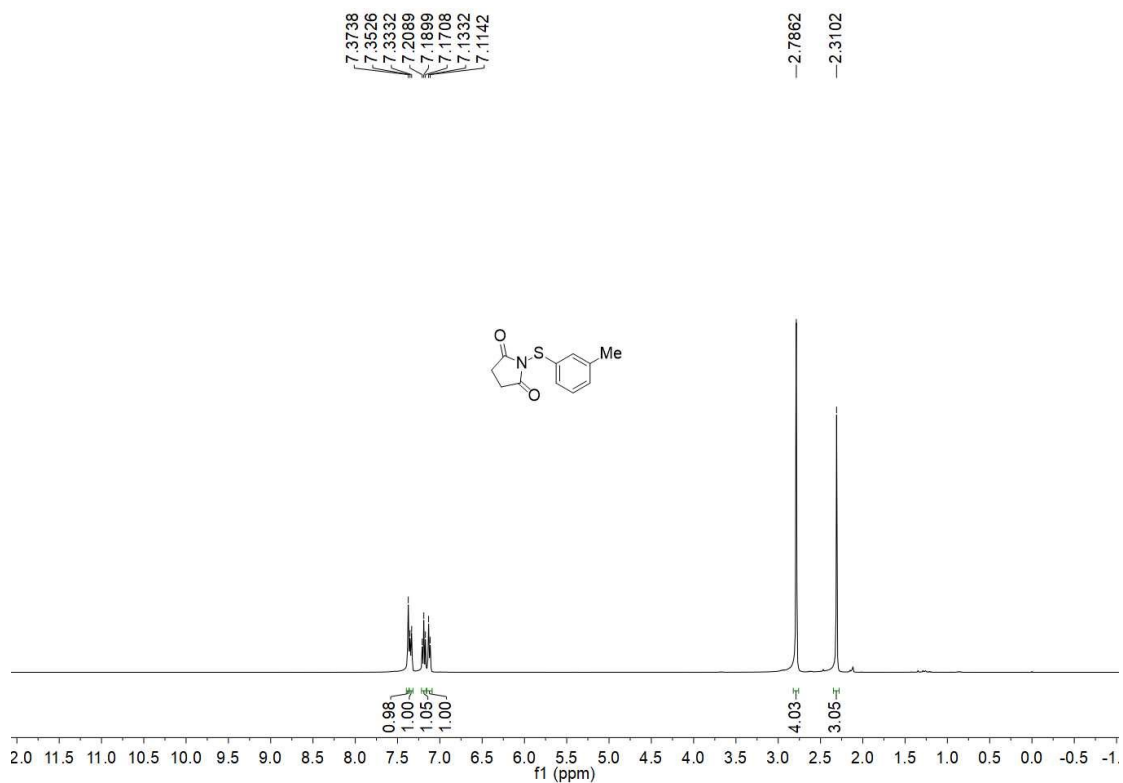
¹³C NMR (101 MHz, CDCl₃) spectrum of Compound Su-2g



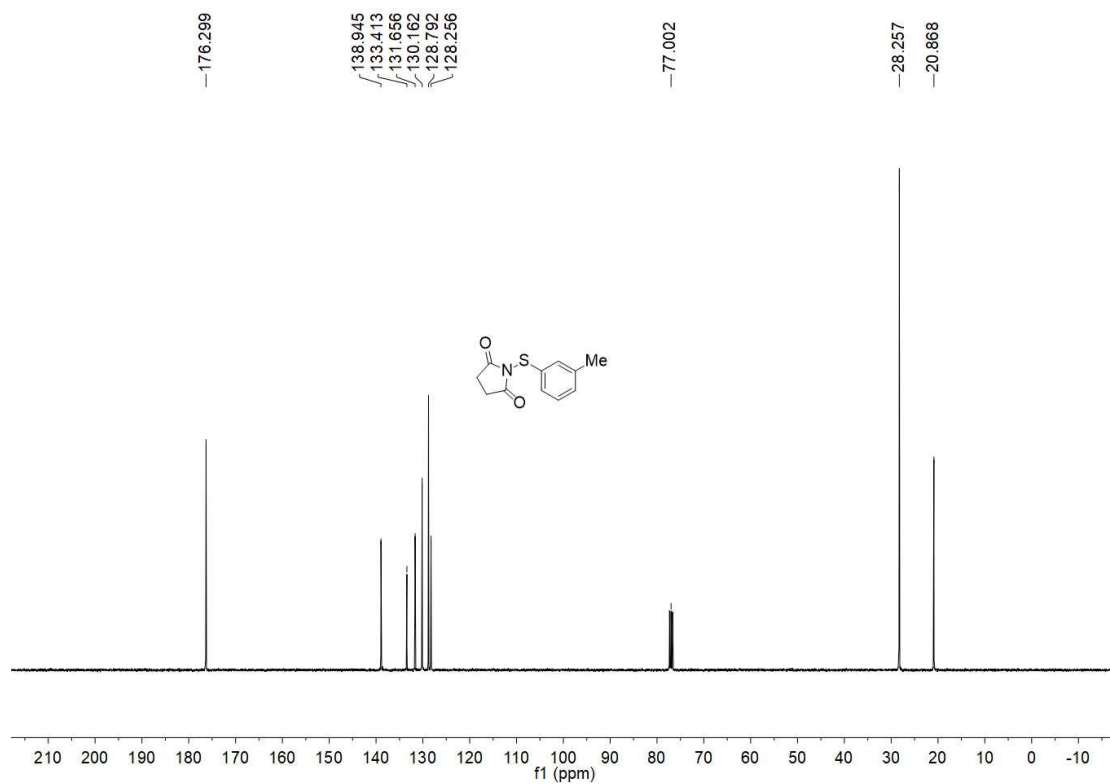
¹H NMR (400 MHz, CDCl₃) spectrum of Compound Su-2h



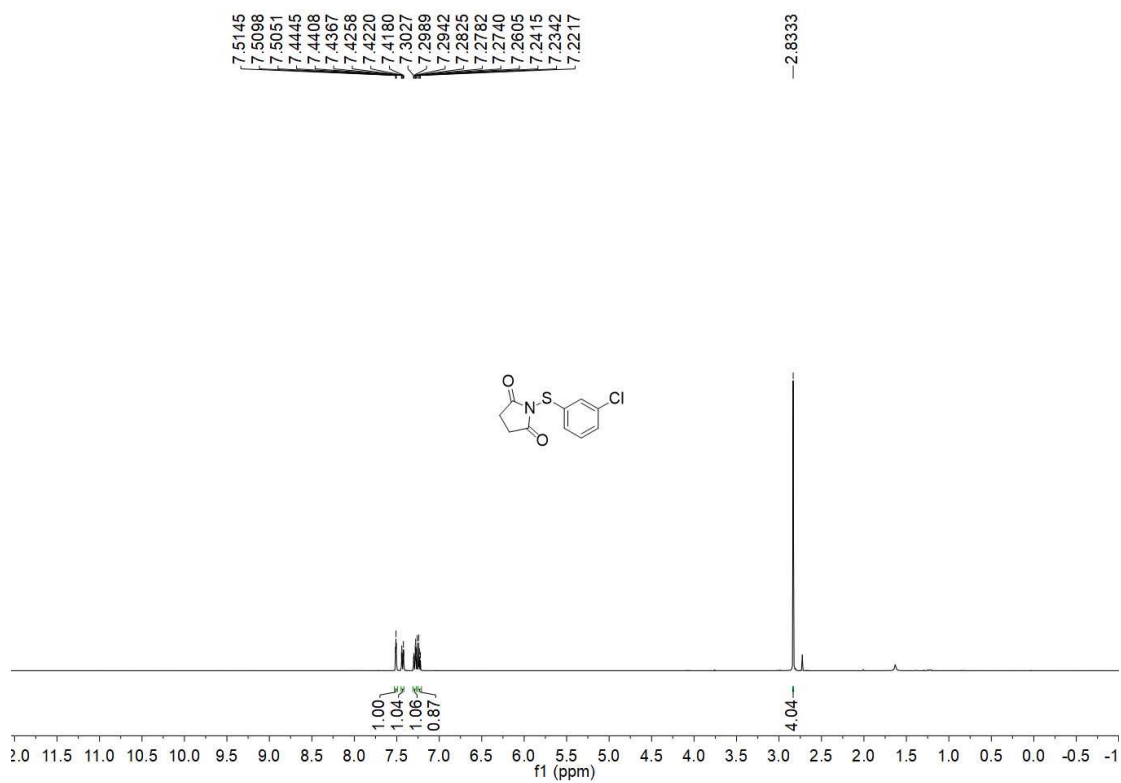
¹⁹F NMR (376 MHz, CDCl₃) spectrum of Compound Su-2h



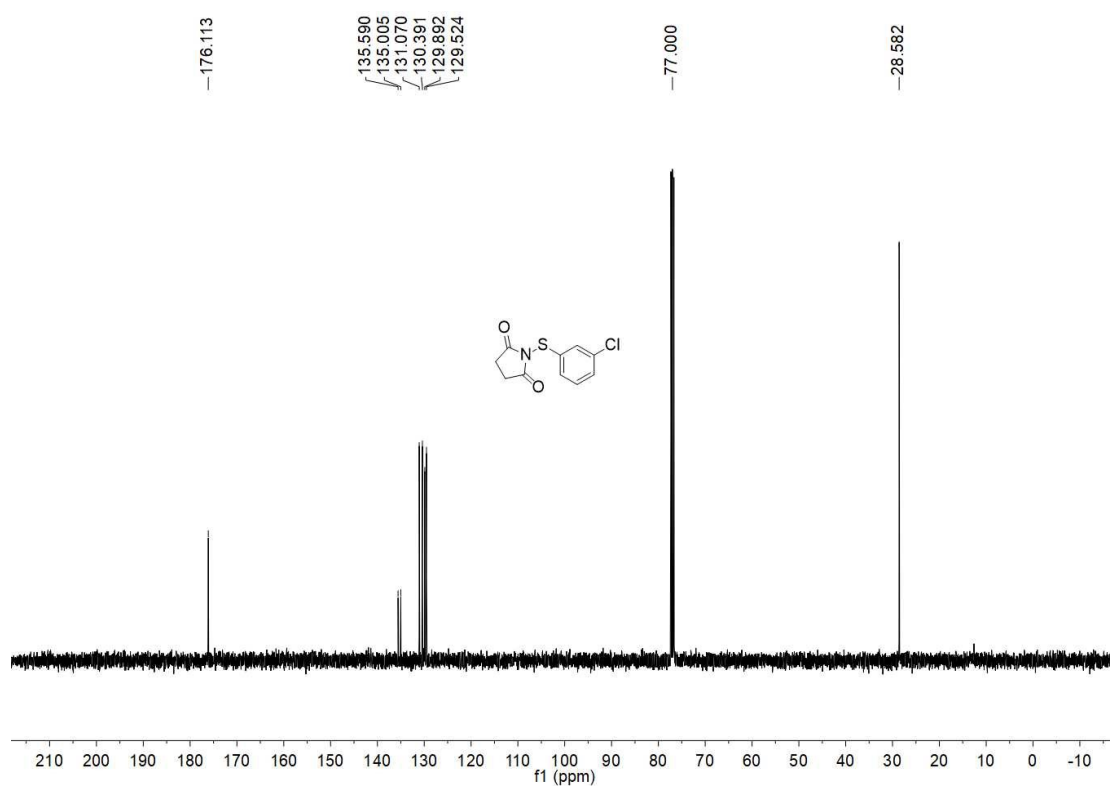
^1H NMR (400 MHz, CDCl_3) spectrum of Compound **Su-2i**



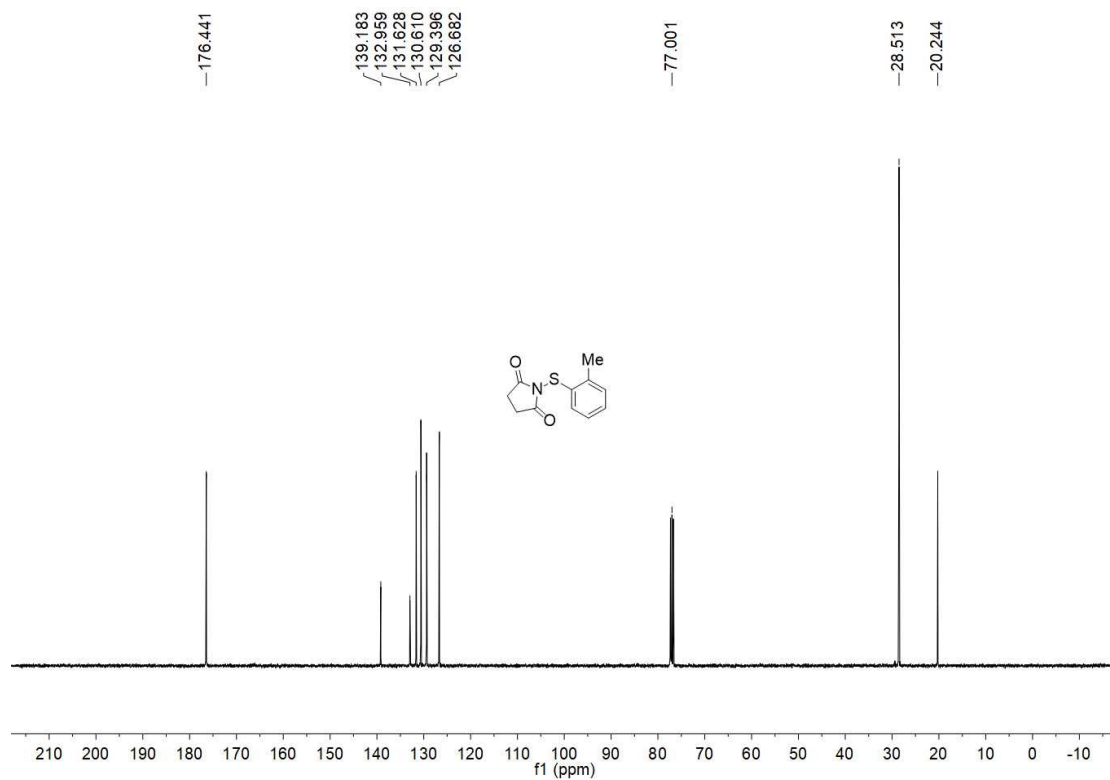
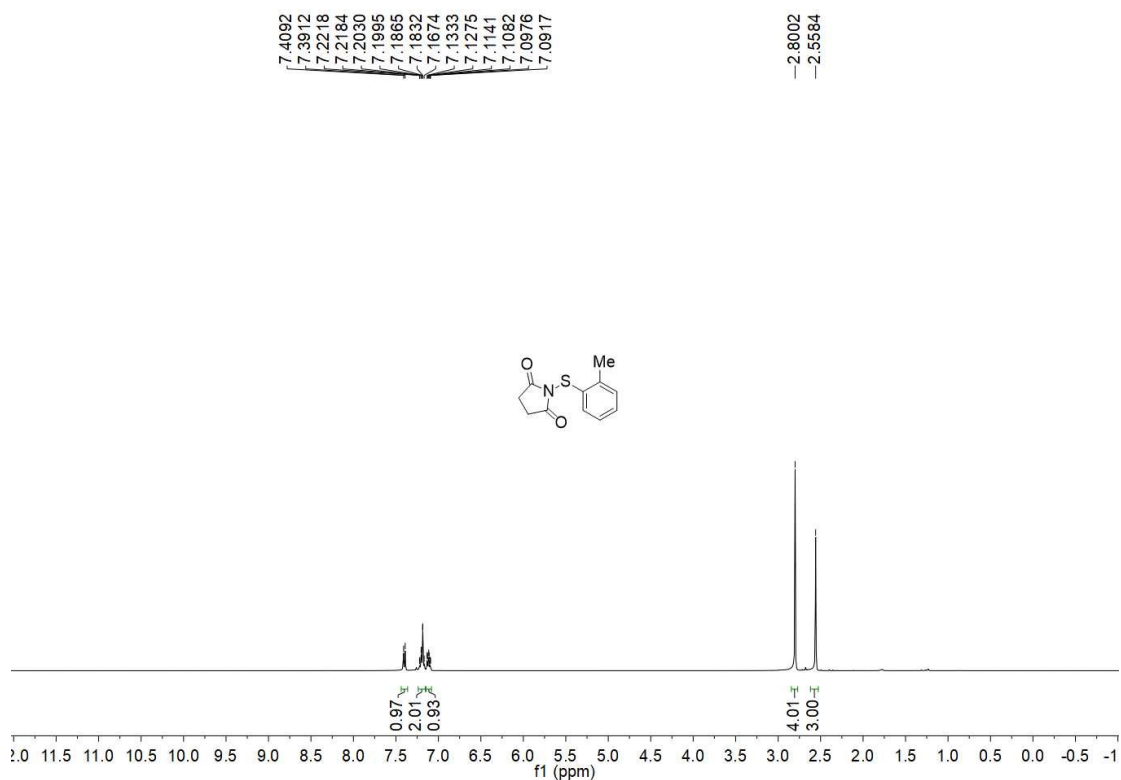
^{13}C NMR (101 MHz, CDCl_3) spectrum of Compound **Su-2i**

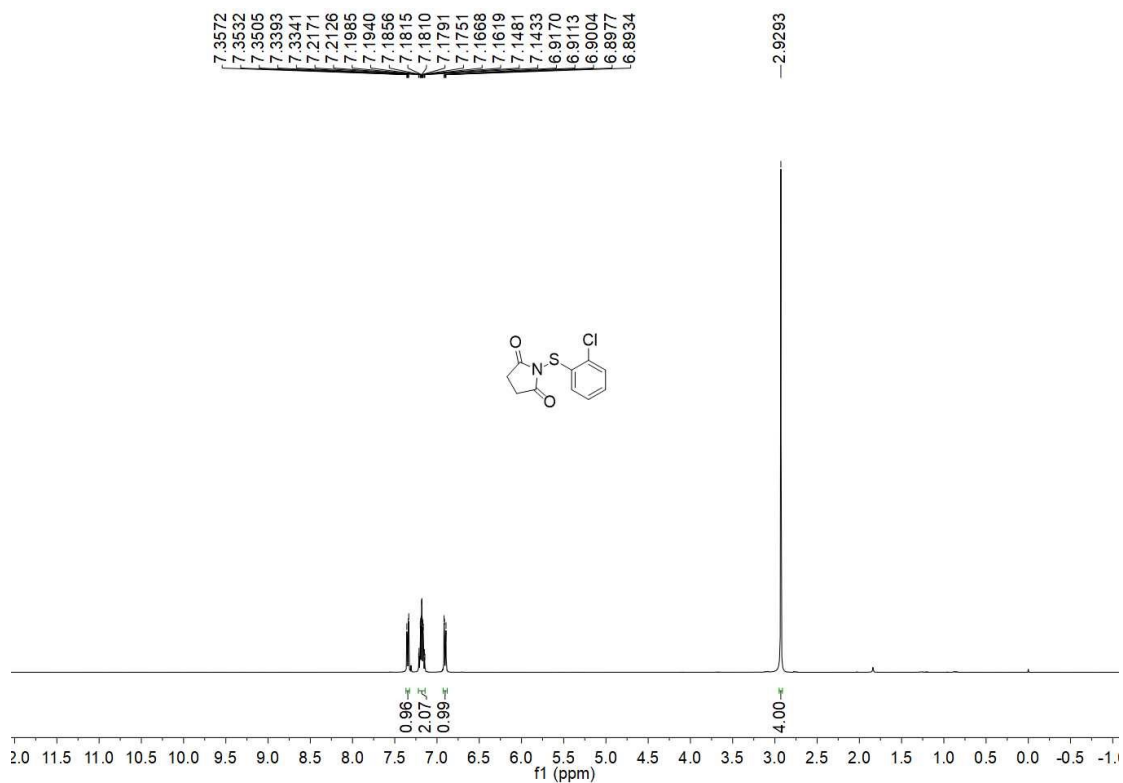


¹H NMR (400 MHz, CDCl₃) spectrum of Compound Su-2j

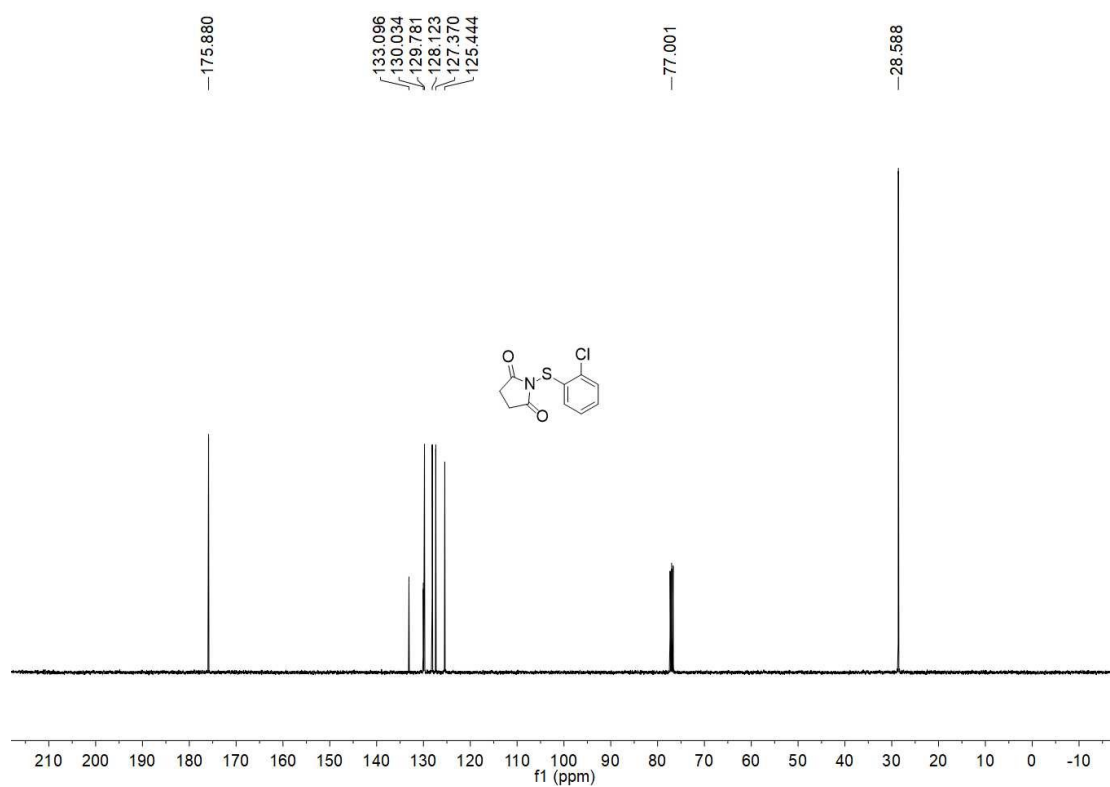


¹³C NMR (101MHz, CDCl₃) spectrum of compound Su-2j

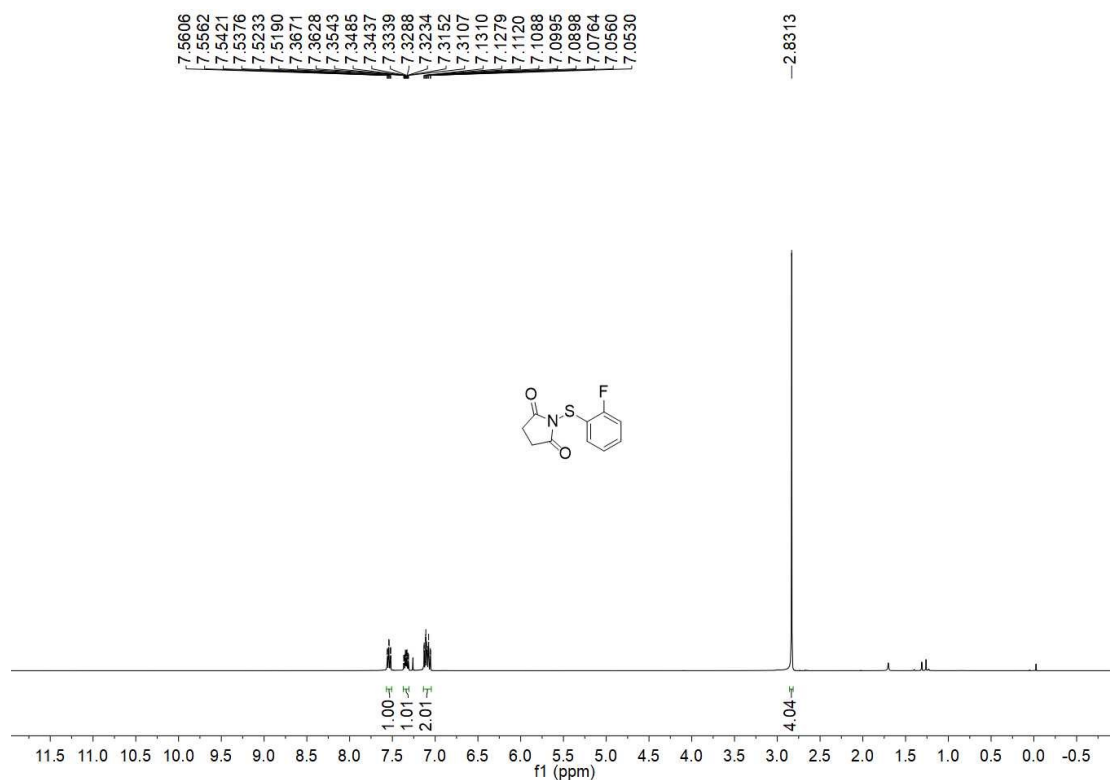




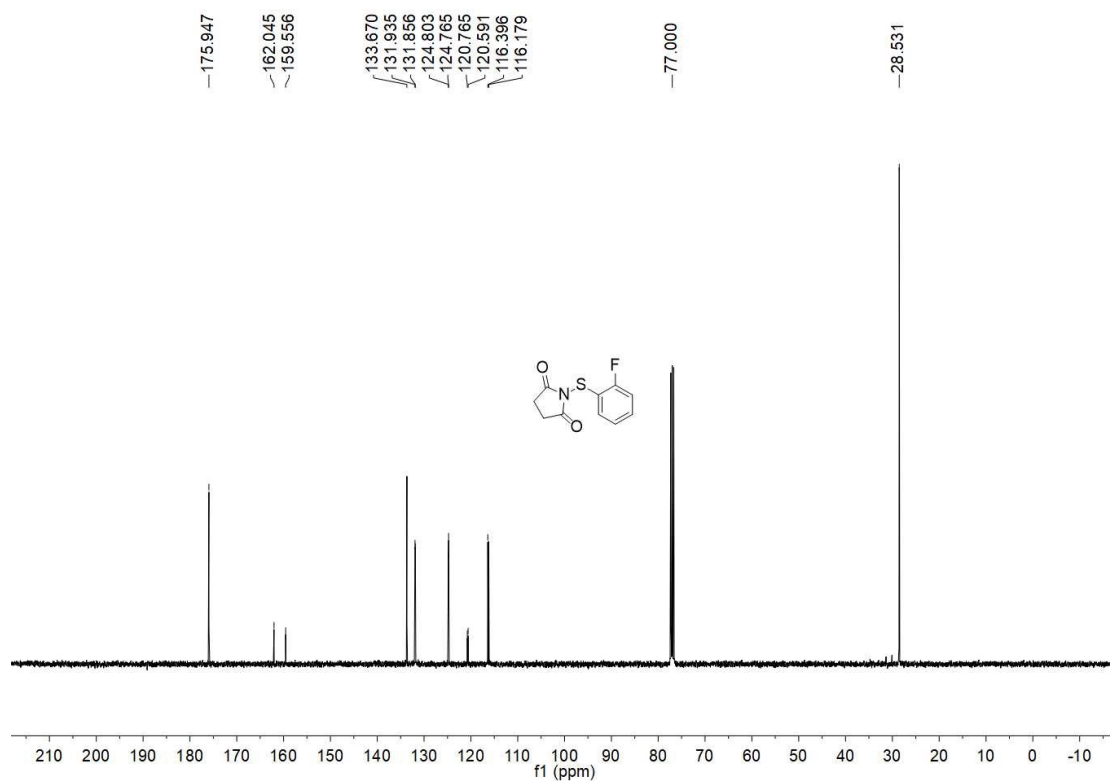
^1H NMR (400 MHz, CDCl_3) spectrum of Compound **Su-21**



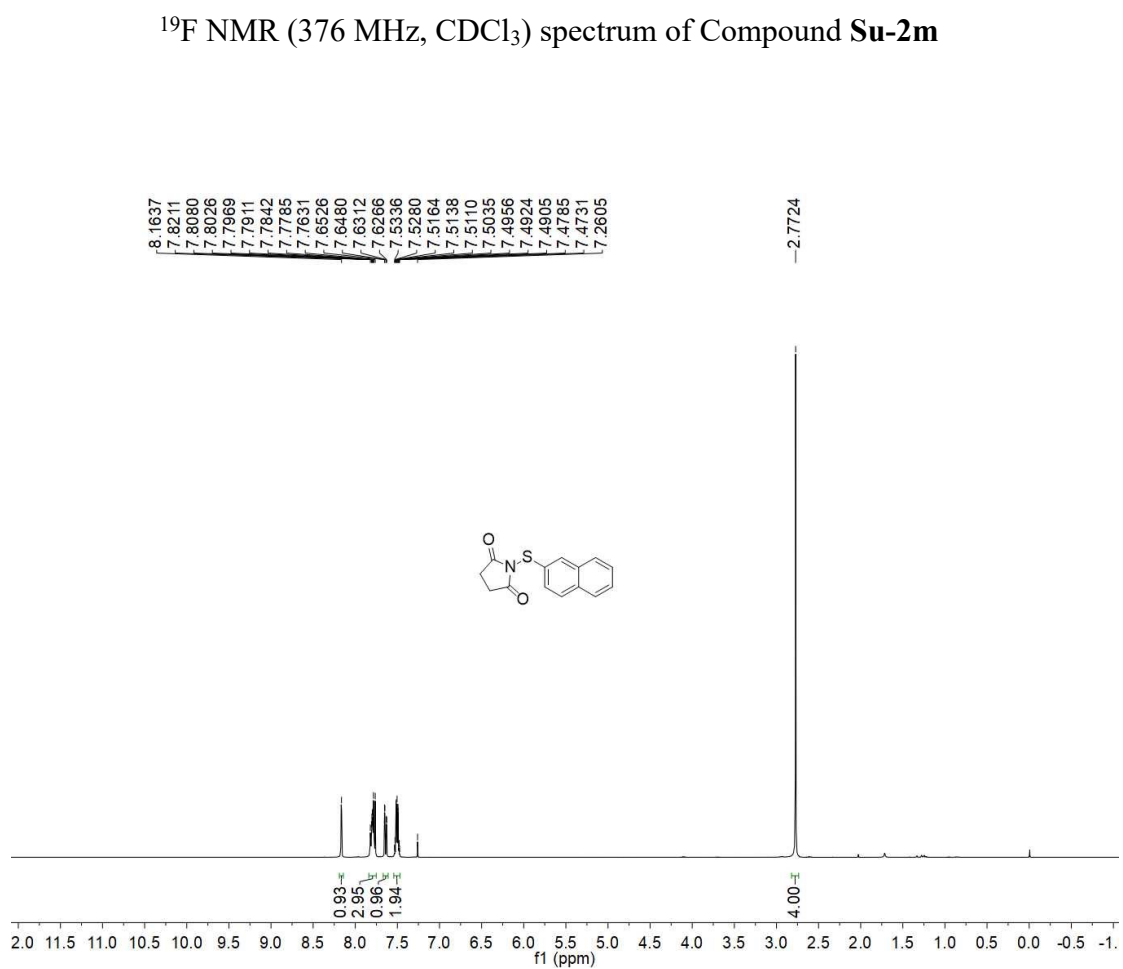
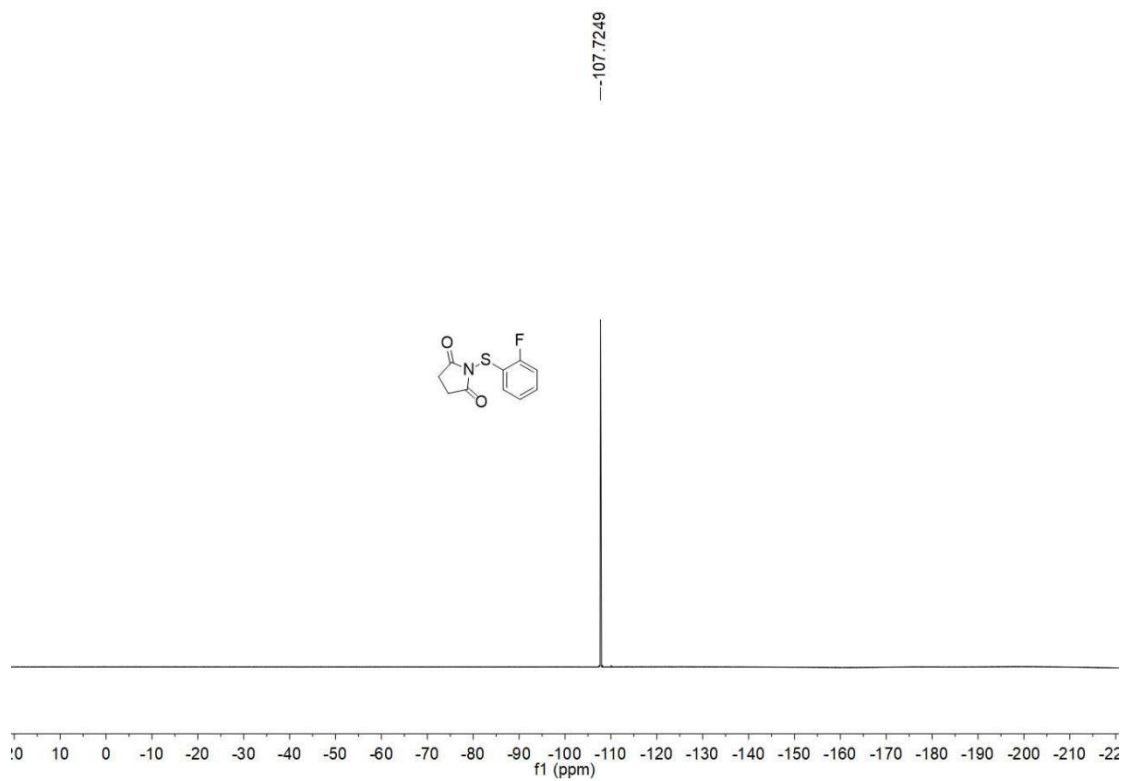
^{13}C NMR (101 MHz, CDCl_3) spectrum of Compound **Su-21**

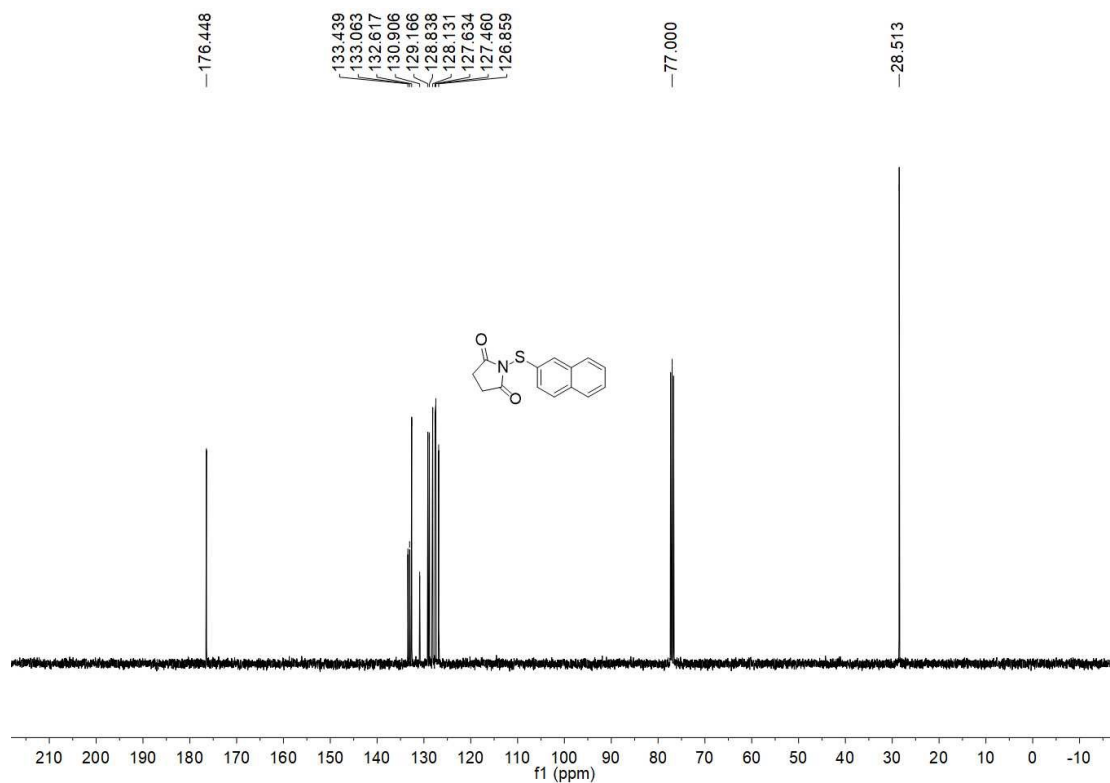


^1H NMR (400 MHz, CDCl_3) spectrum of Compound **Su-2m**

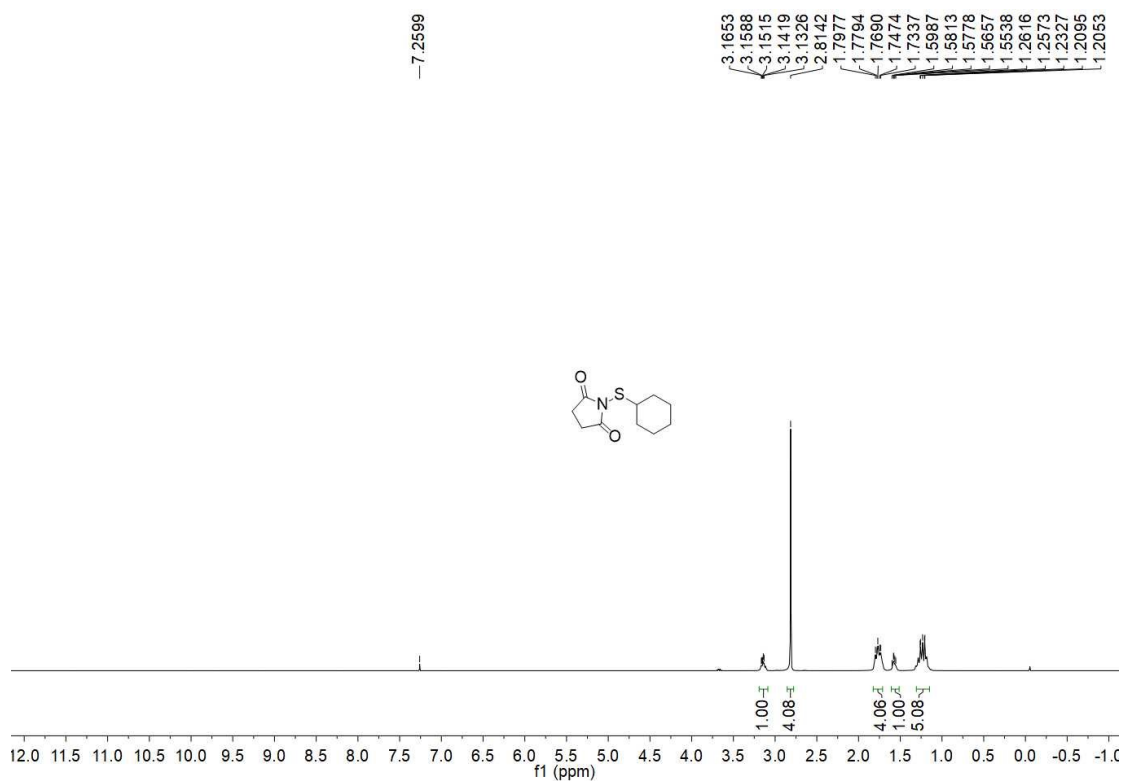


^{13}C NMR (101 MHz, CDCl_3) spectrum of Compound **Su-2m**

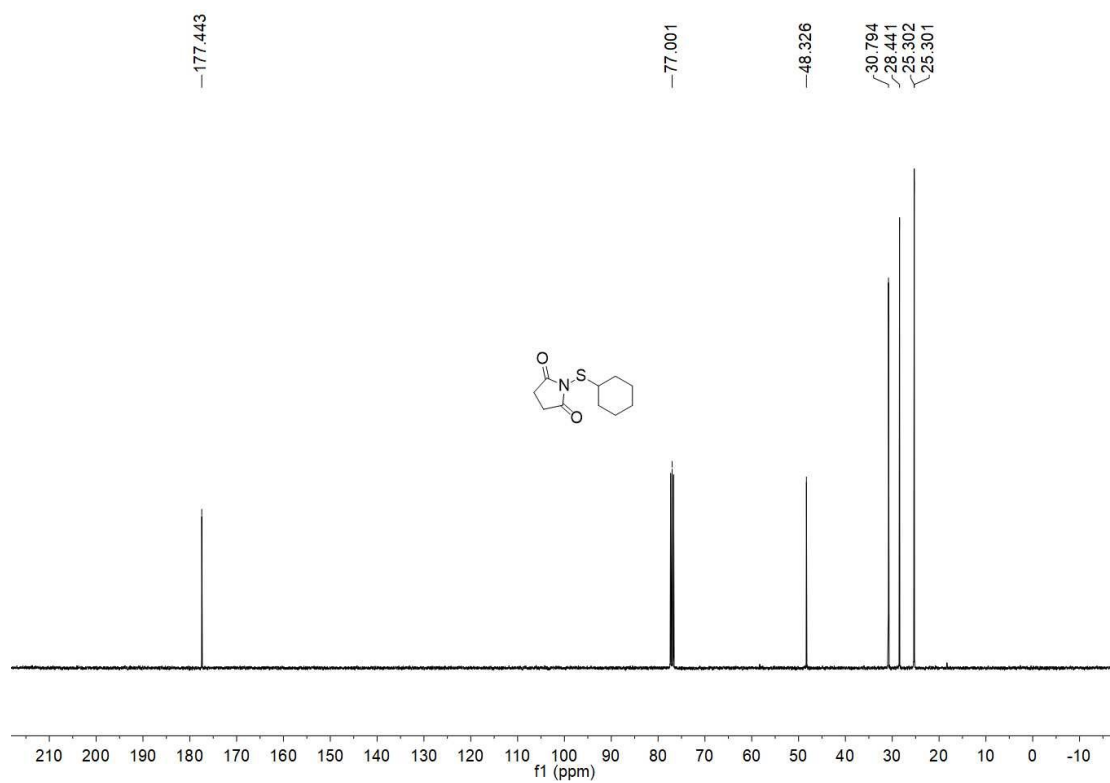




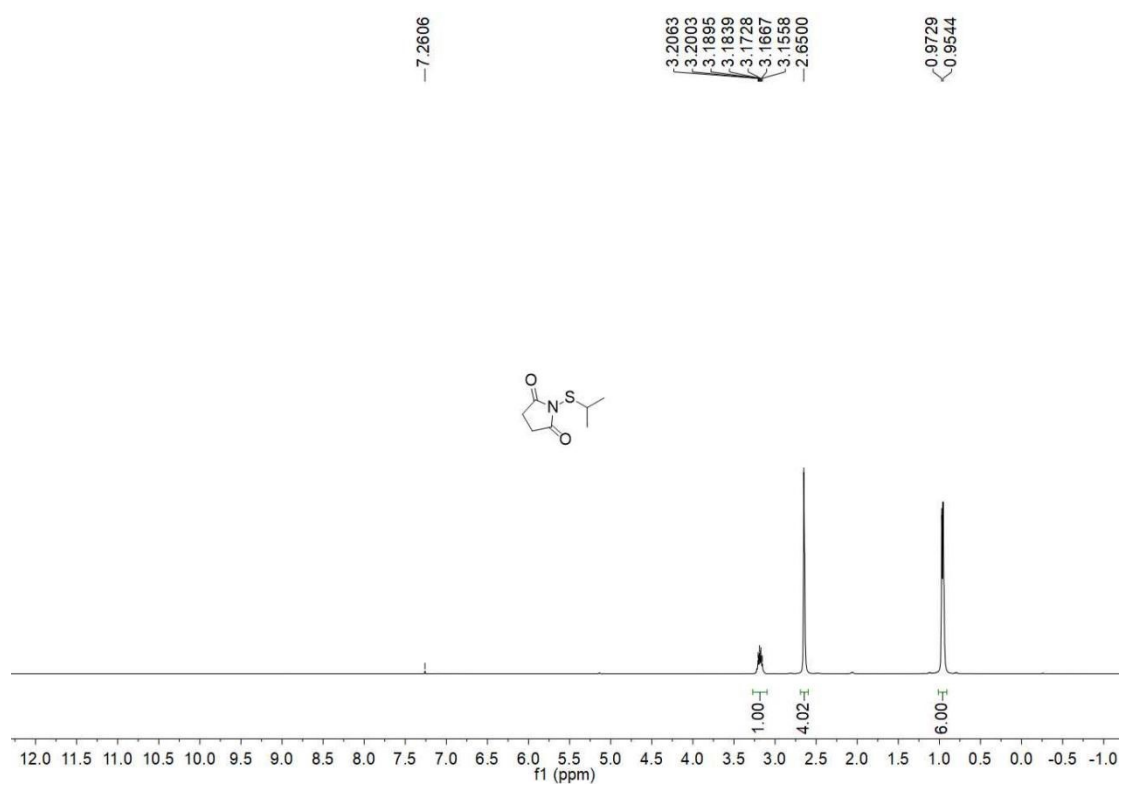
^{13}C NMR (101 MHz, CDCl_3) spectrum of Compound Su-2n



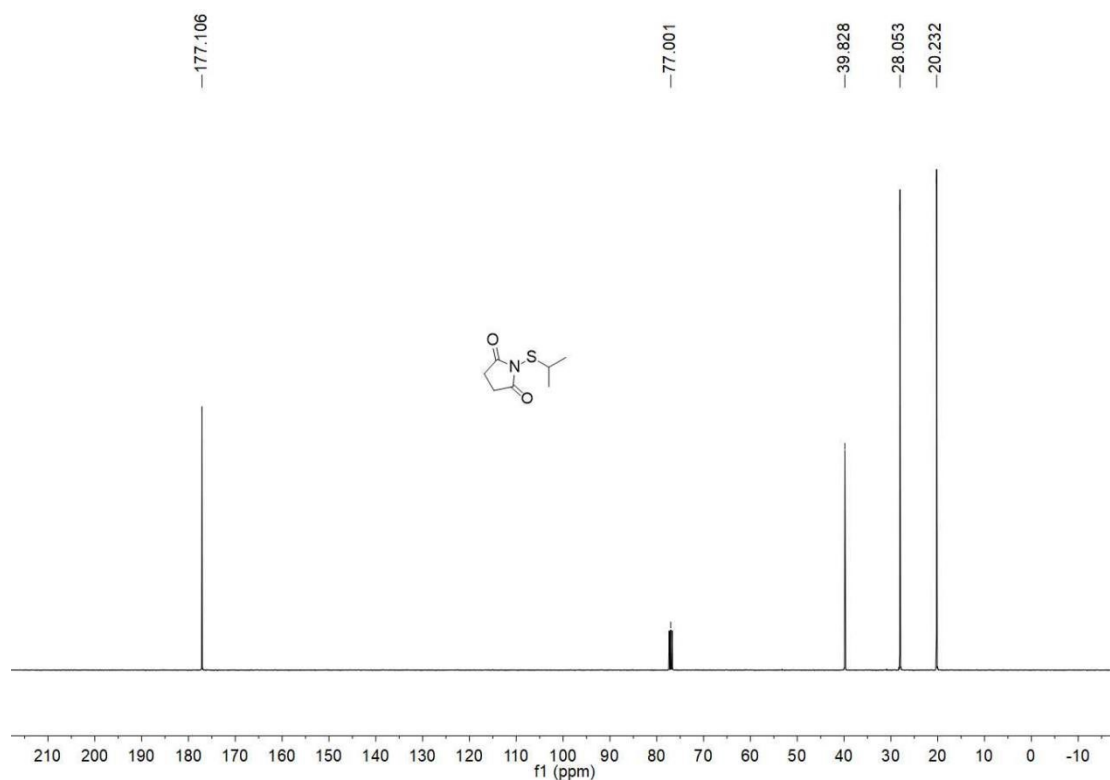
^1H NMR (400 MHz, CDCl_3) spectrum of Compound Su-2o



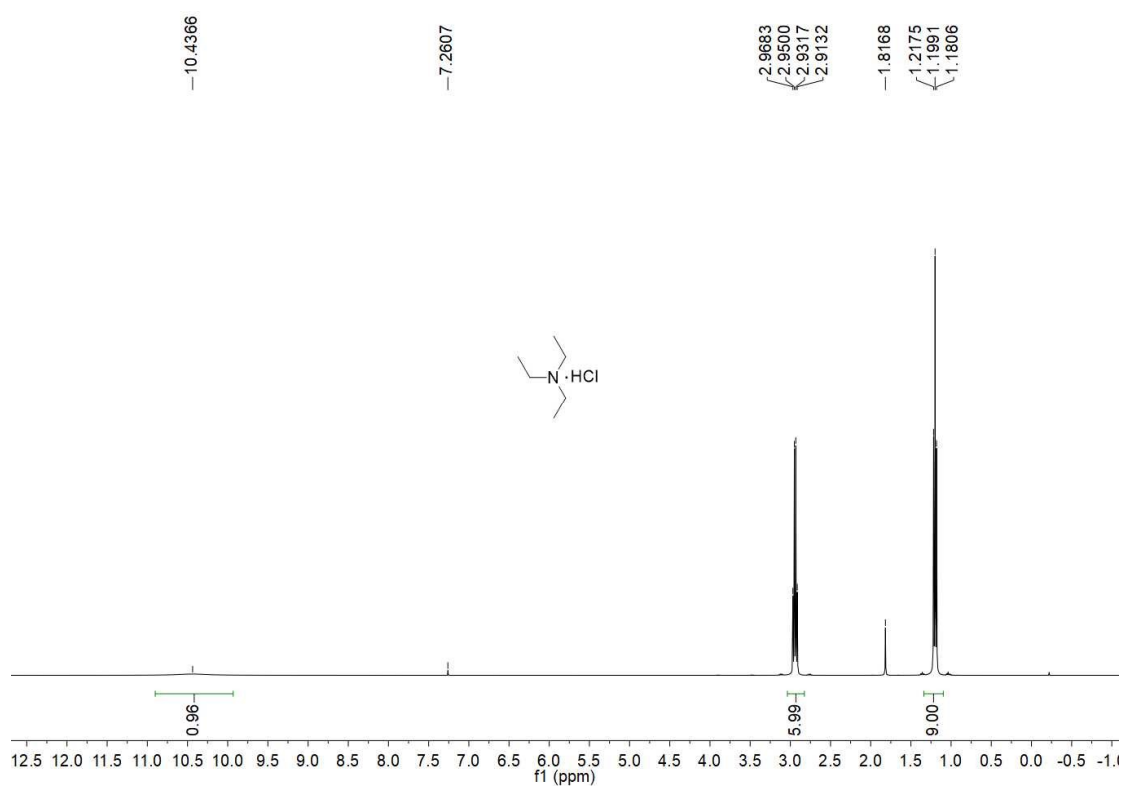
¹³C NMR (101 MHz, CDCl₃) spectrum of Compound Su-2o



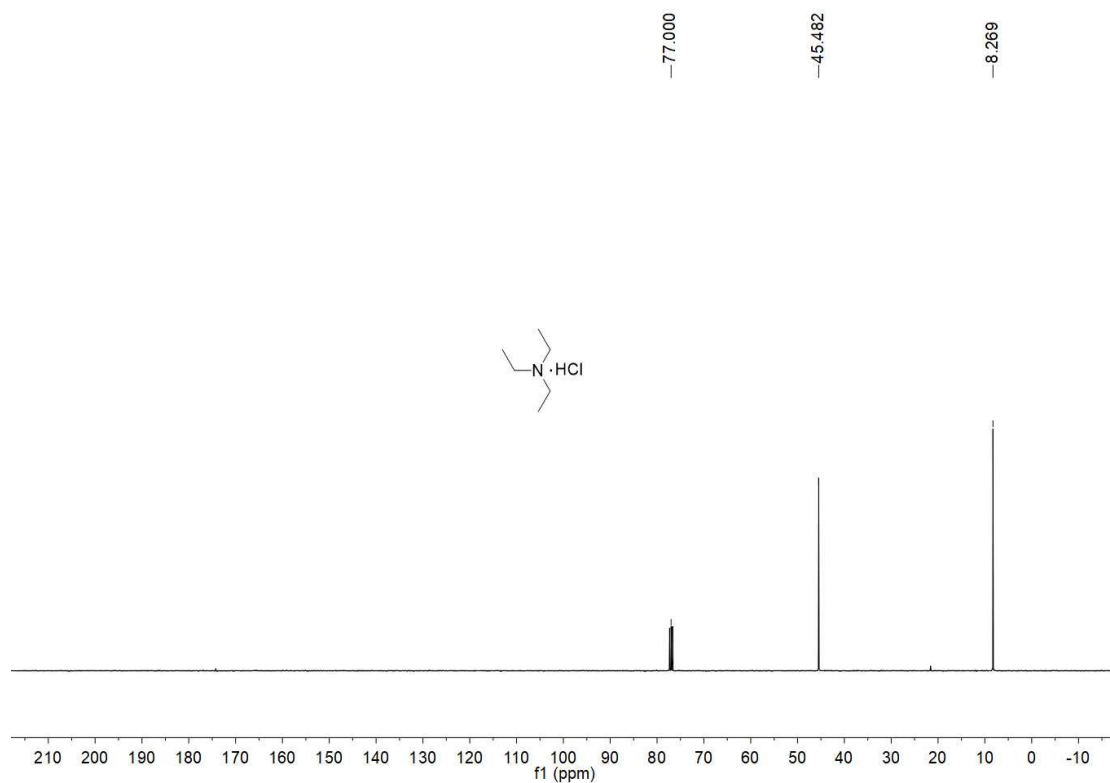
¹H NMR (400 MHz, CDCl₃) spectrum of Compound Su-2p



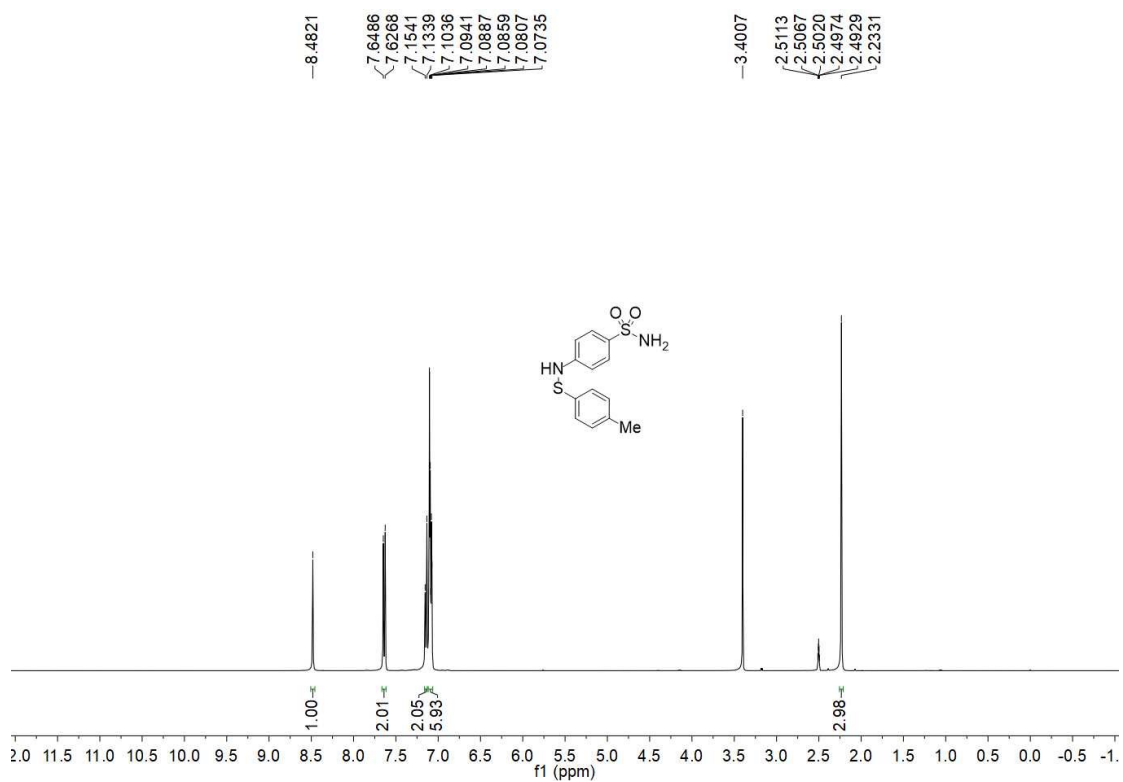
^{13}C NMR (101 MHz, CDCl_3) spectrum of Compound Su-2p



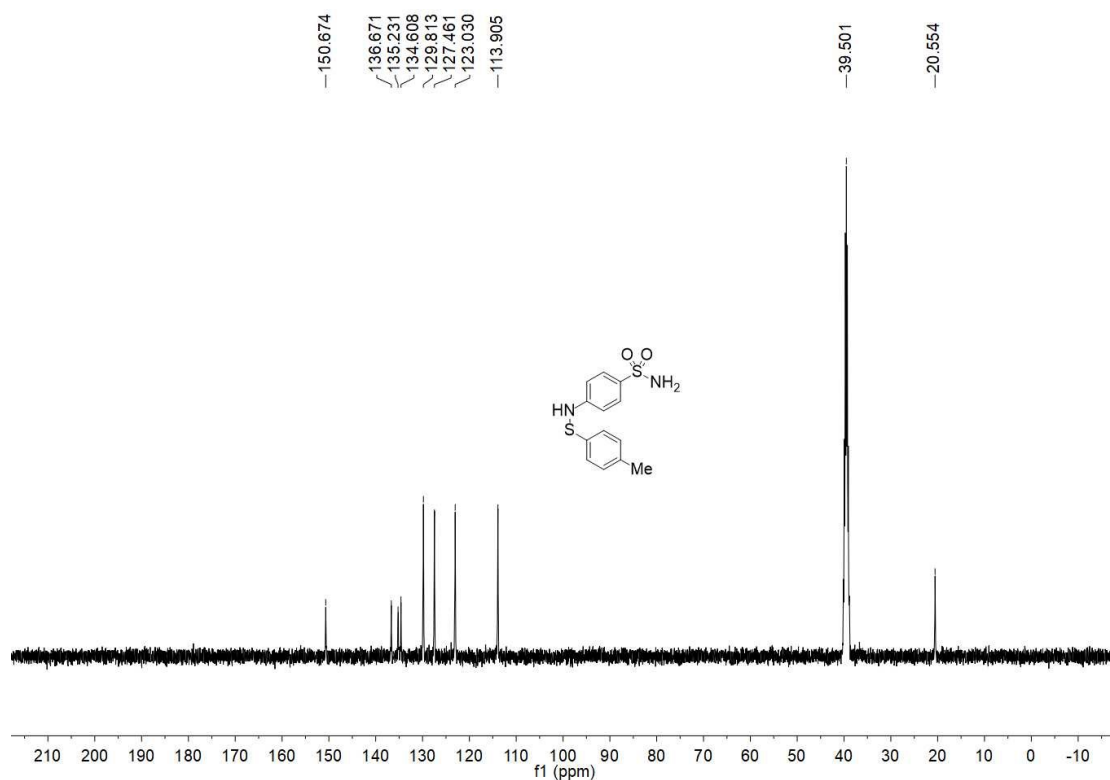
^1H NMR (400 MHz, CDCl_3) spectrum of Et_3NHCl



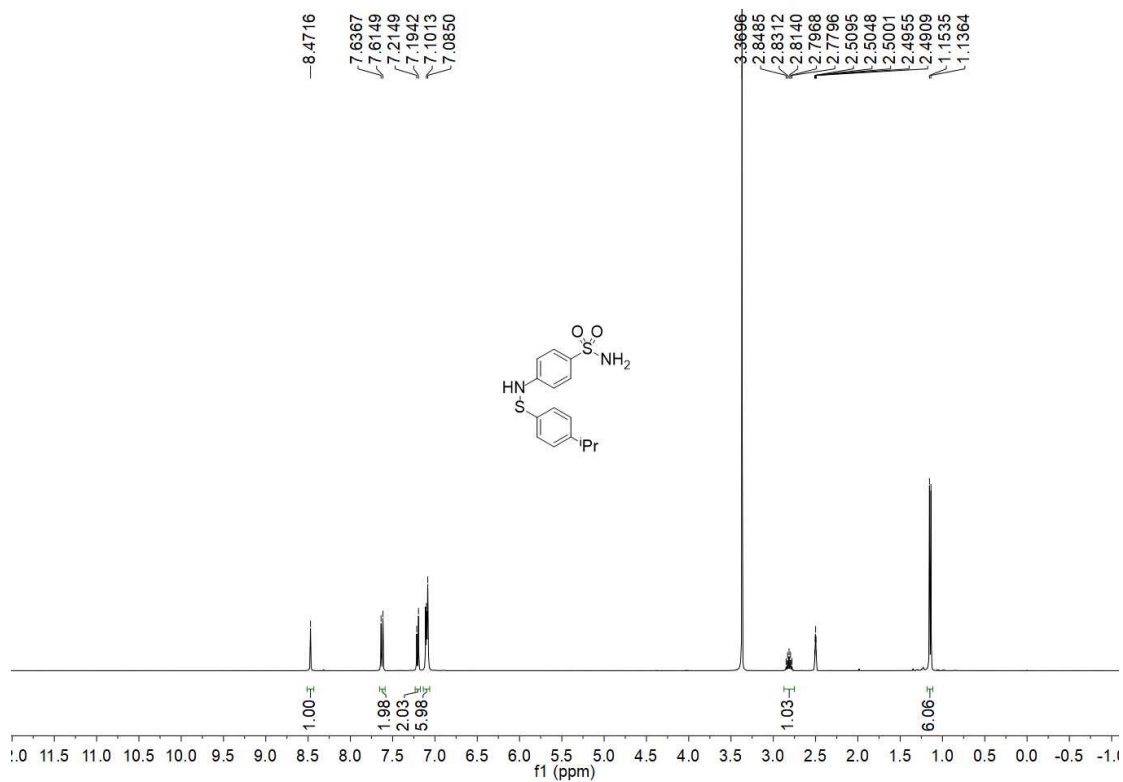
^{13}C NMR (101 MHz, CDCl_3) spectrum of Et_3NHCl



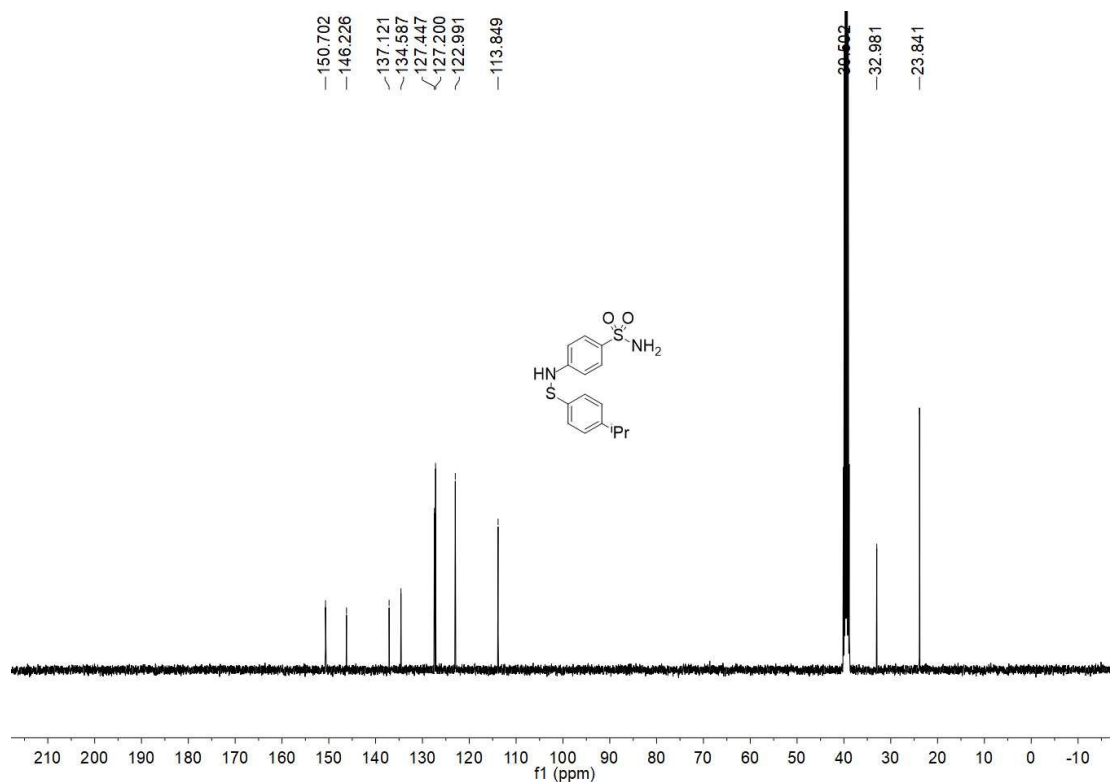
^1H NMR (400 MHz, $\text{DMSO}-d_6$) spectrum of Compound **3a**



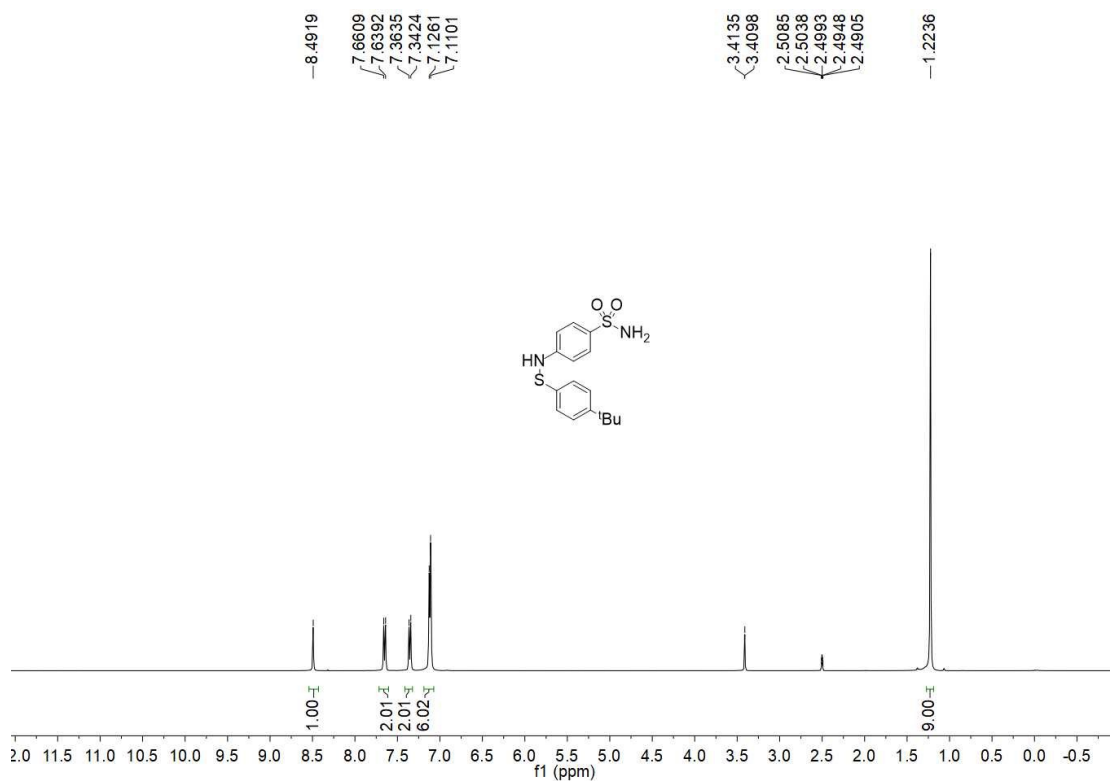
^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) spectrum of Compound 3a



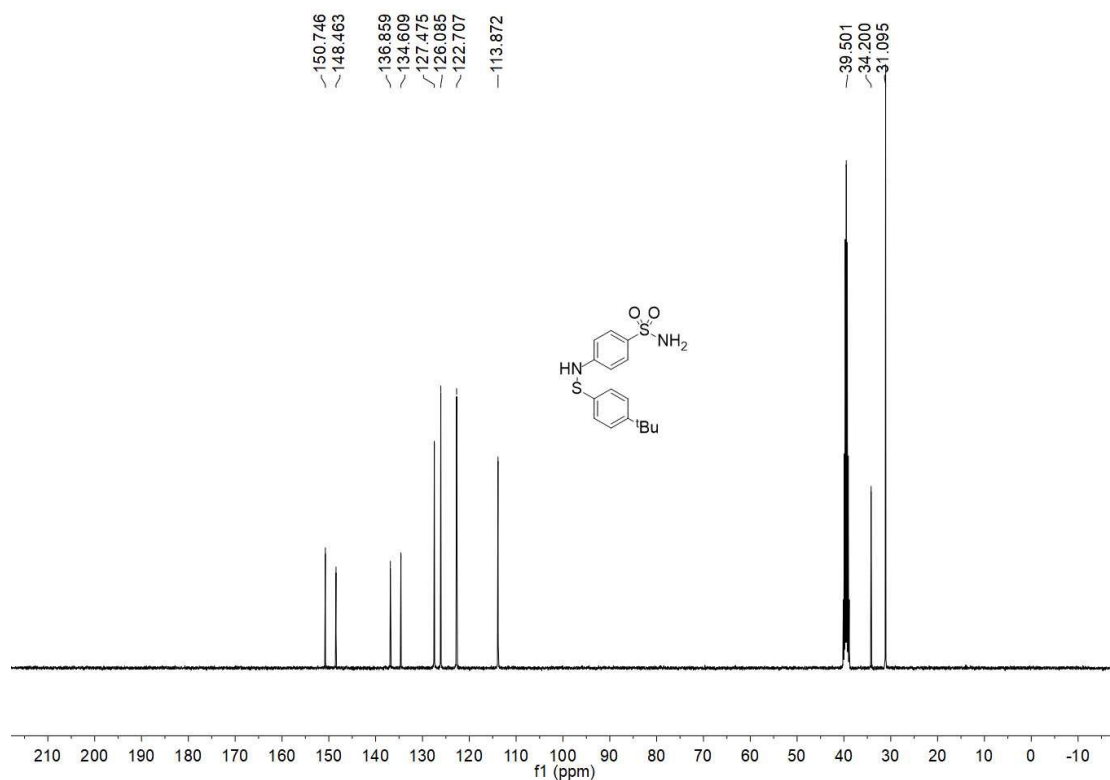
^1H NMR (400 MHz, $\text{DMSO-}d_6$) spectrum of Compound 3b



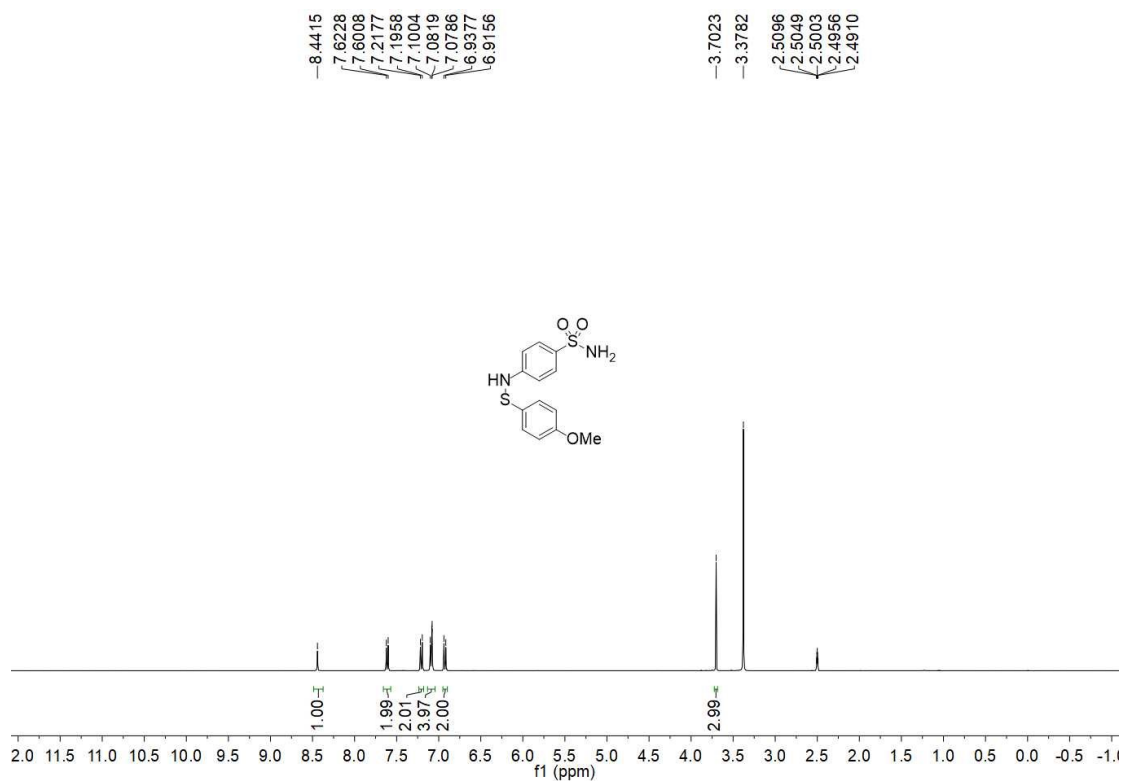
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 3b



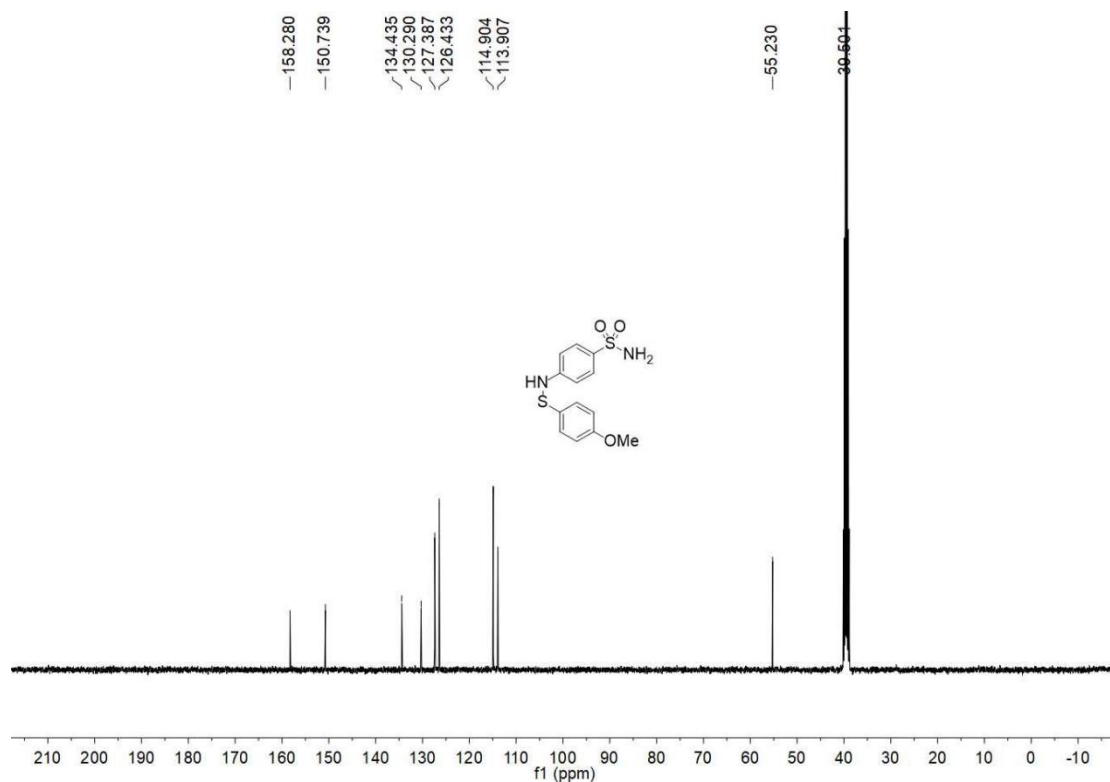
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 3c



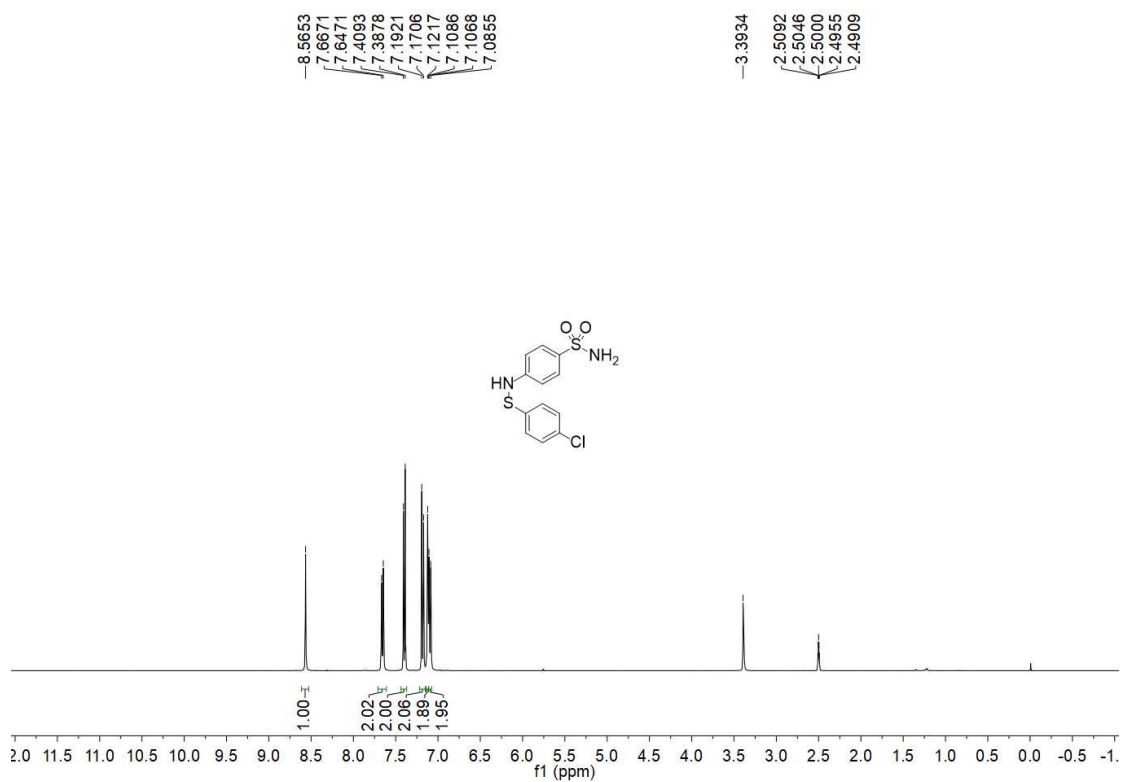
^{13}C NMR (101 MHz, DMSO- d_6) spectrum of Compound 3c



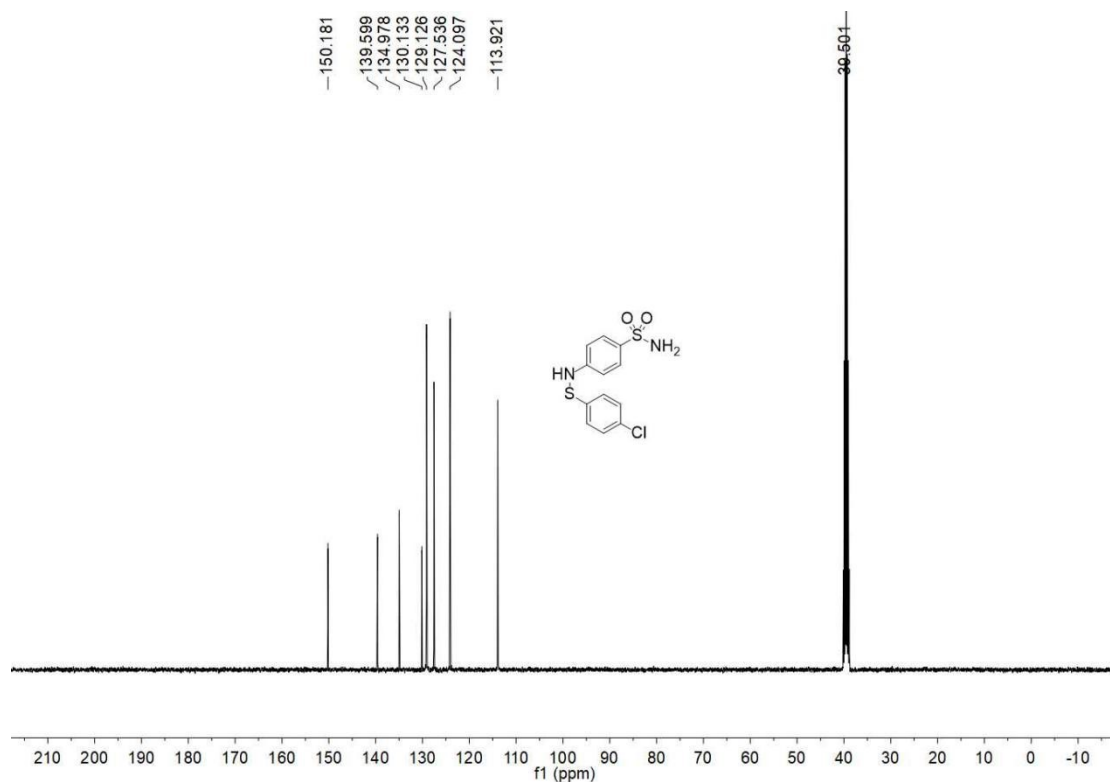
^1H NMR (400 MHz, DMSO- d_6) spectrum of Compound 3d



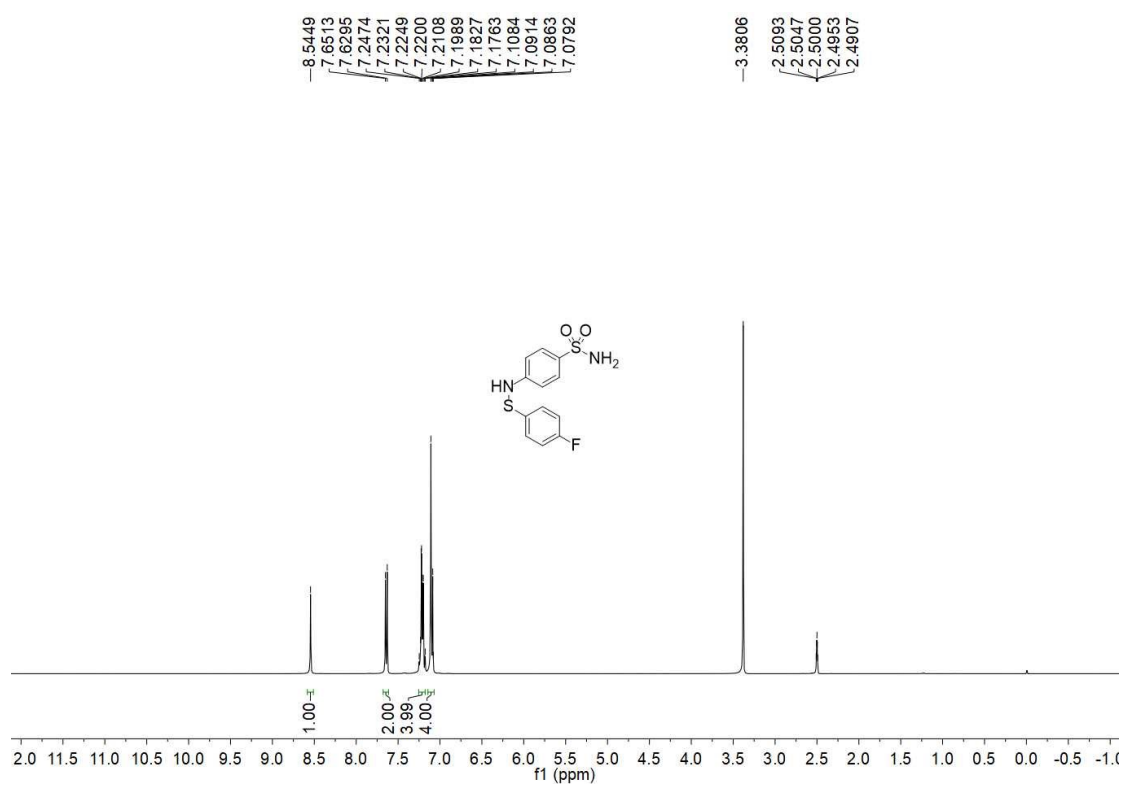
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 3d



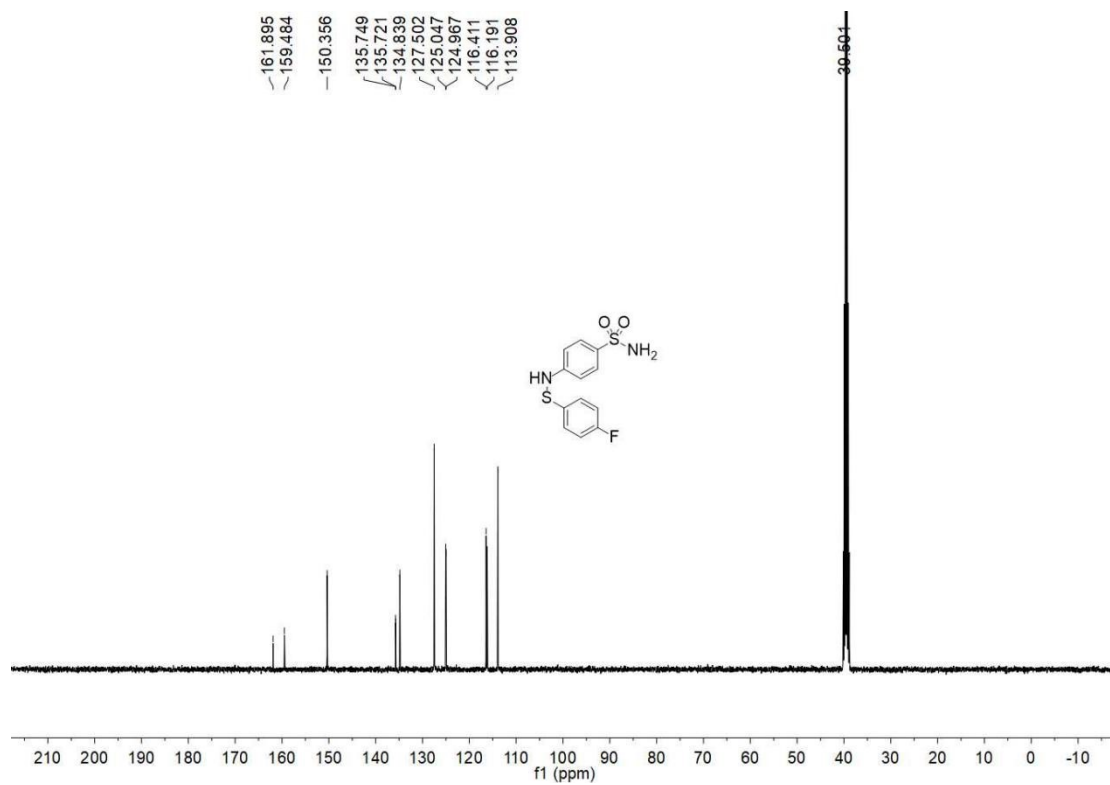
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 3e



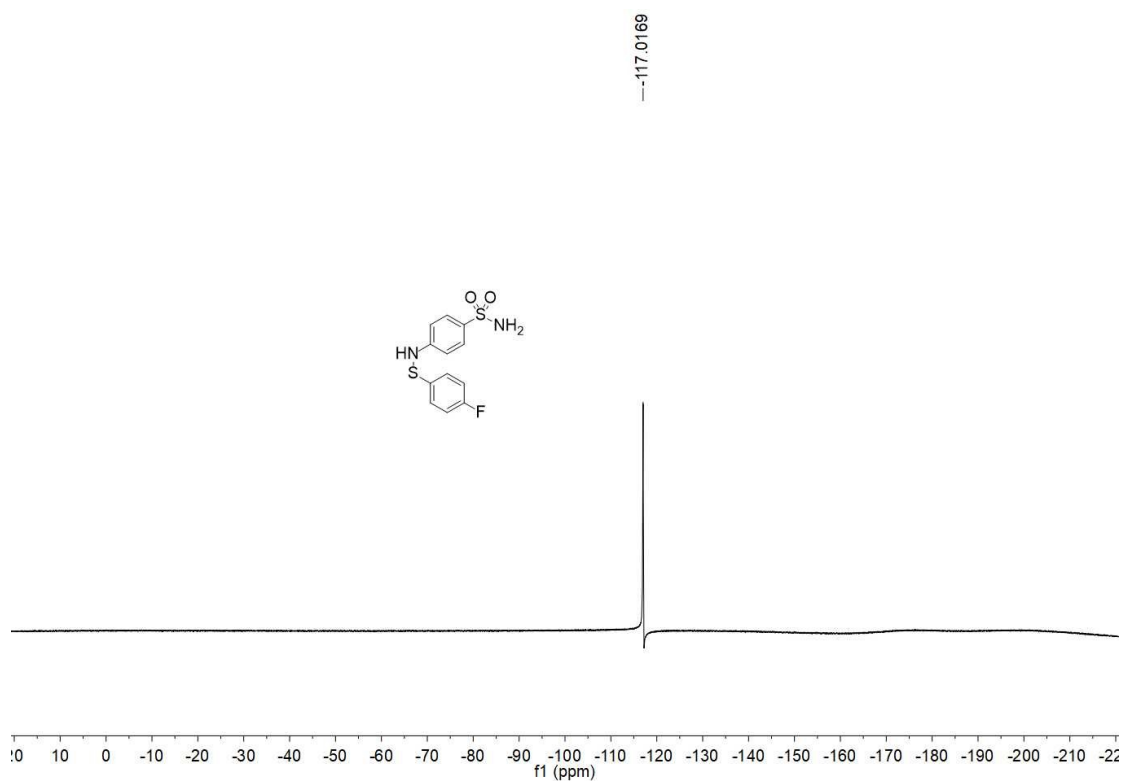
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 3e



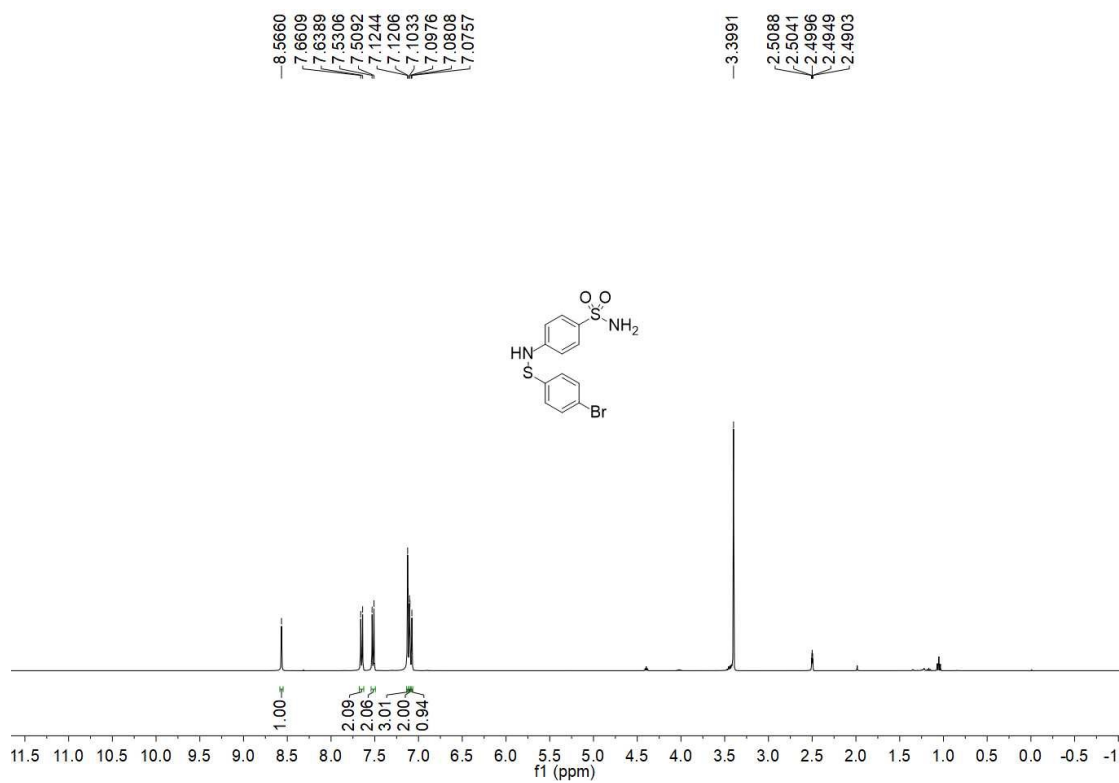
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 3f



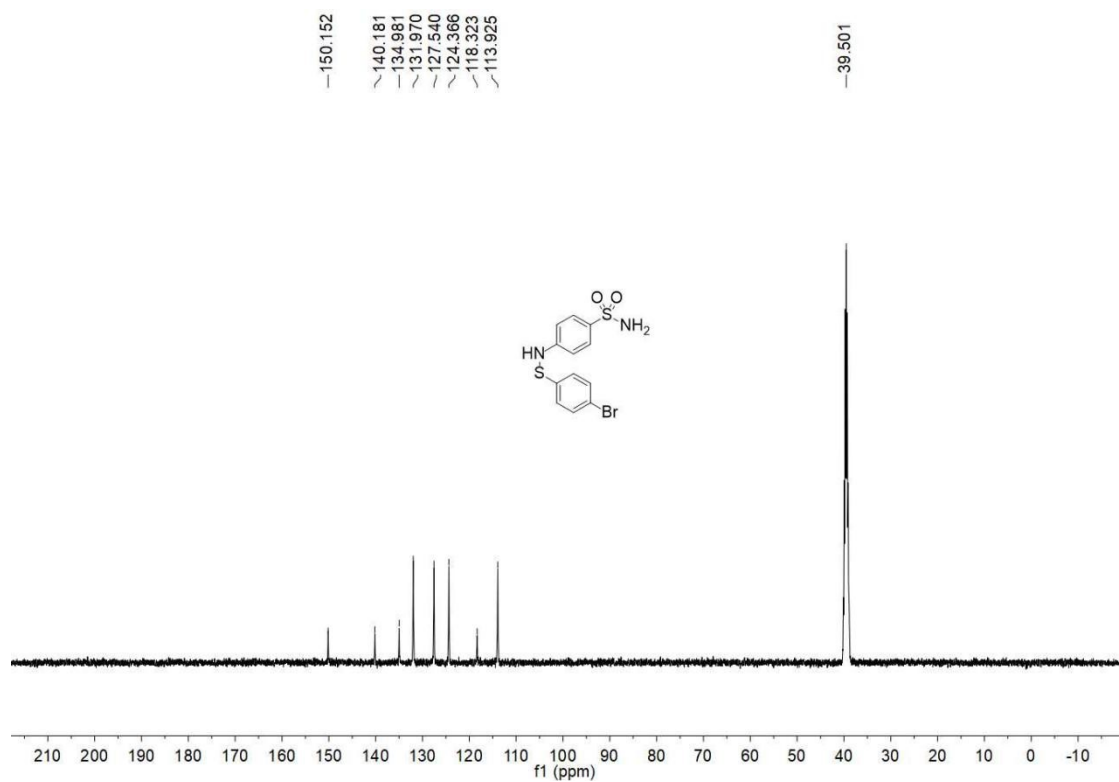
^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) spectrum of Compound **3f**



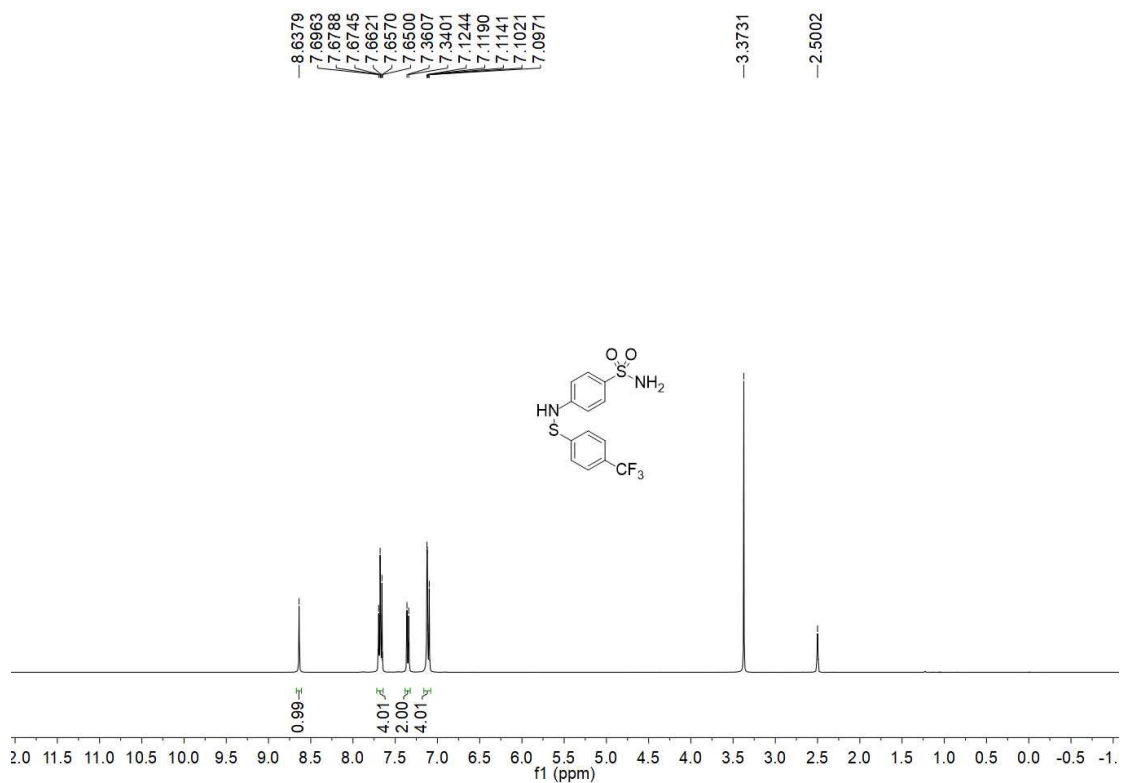
^{19}F NMR (376 MHz, $\text{DMSO-}d_6$) spectrum of Compound **3f**



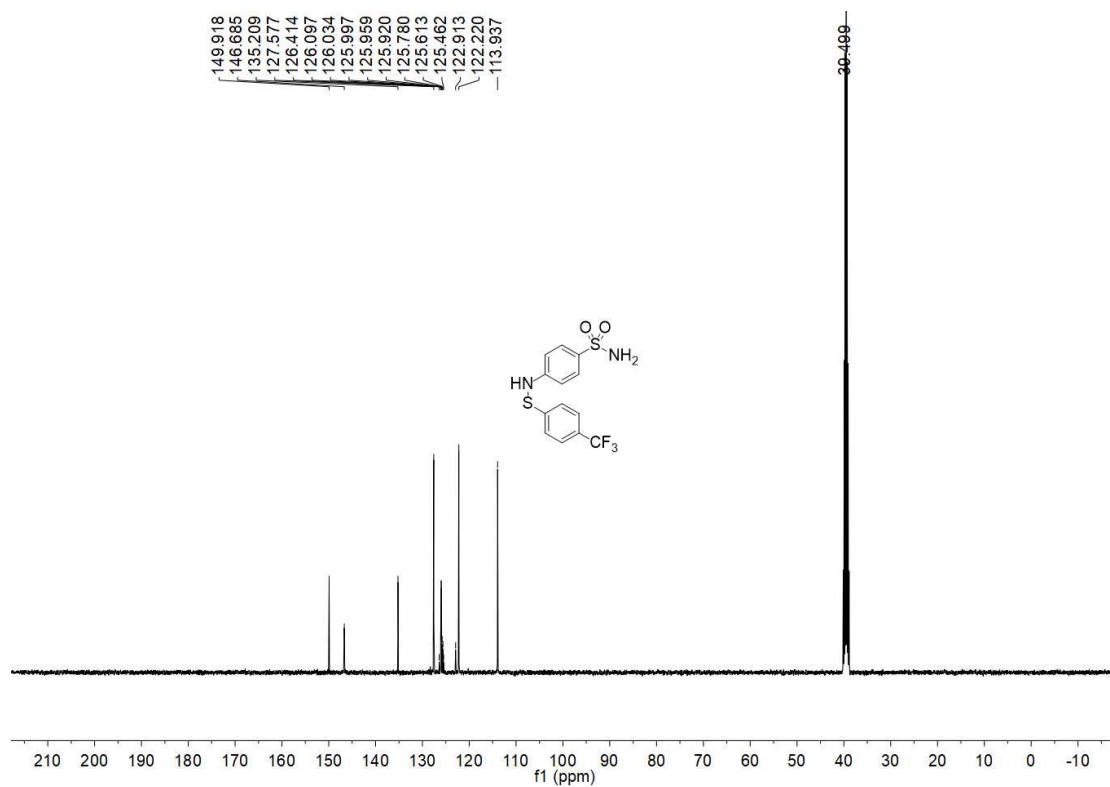
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound **3g**



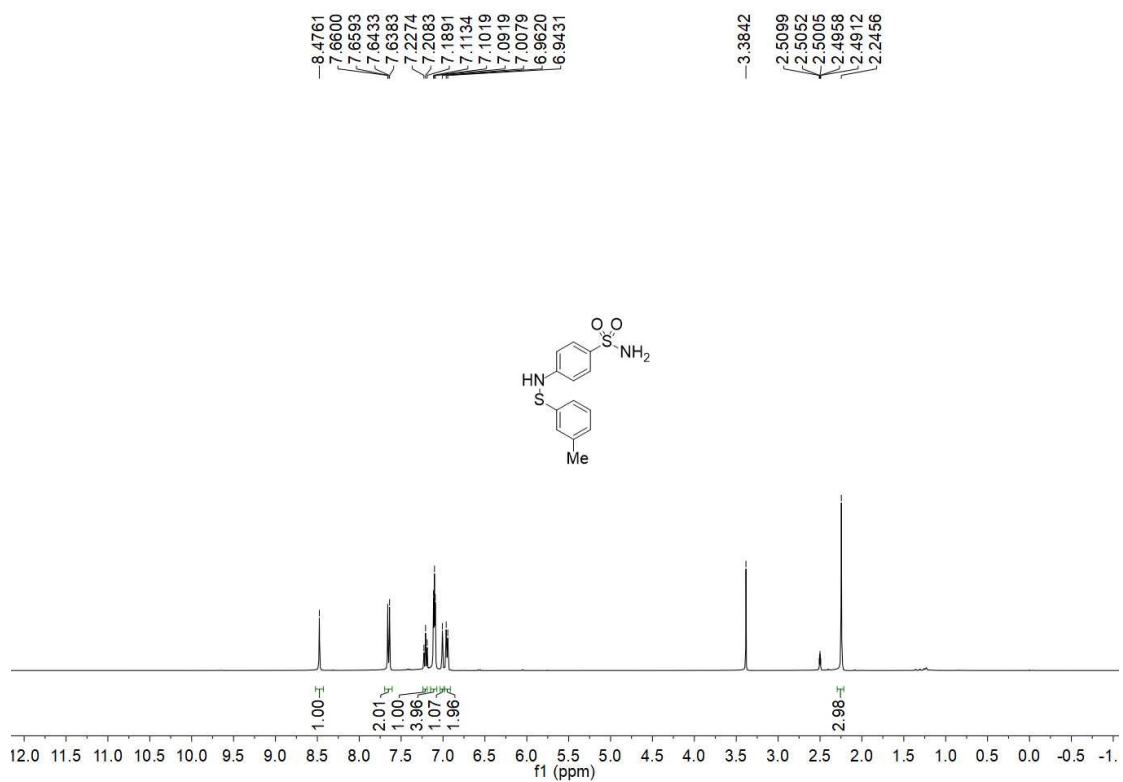
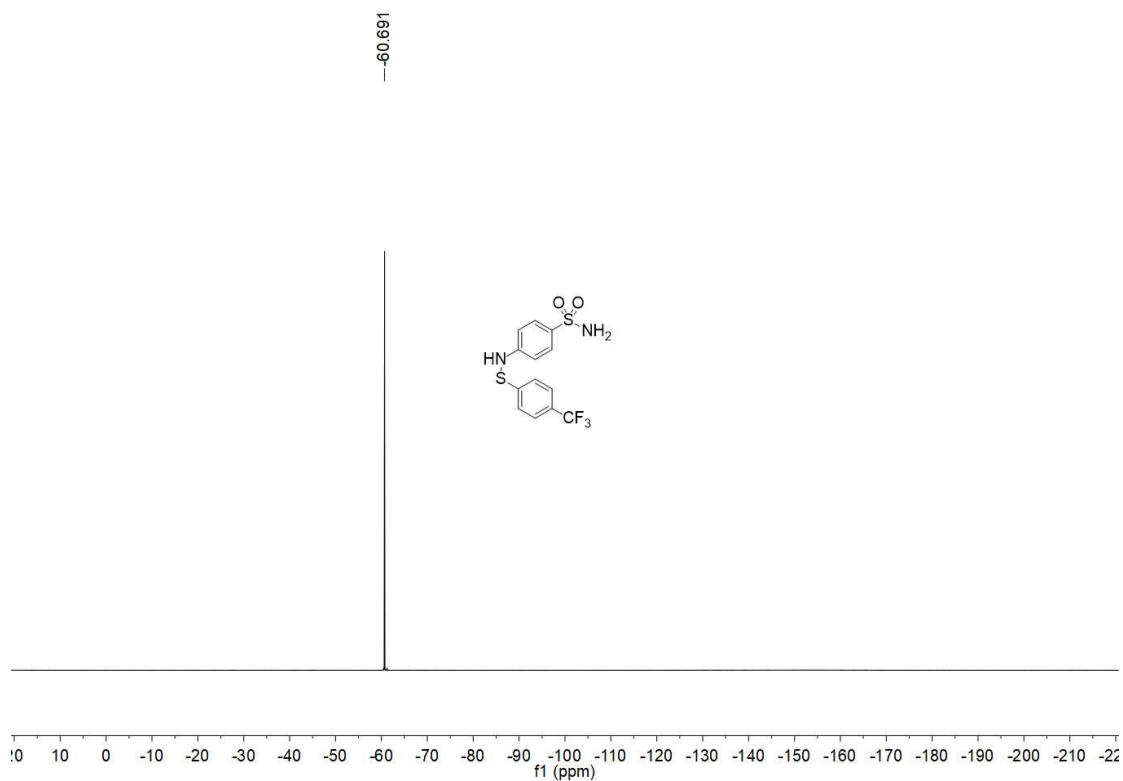
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound **3g**



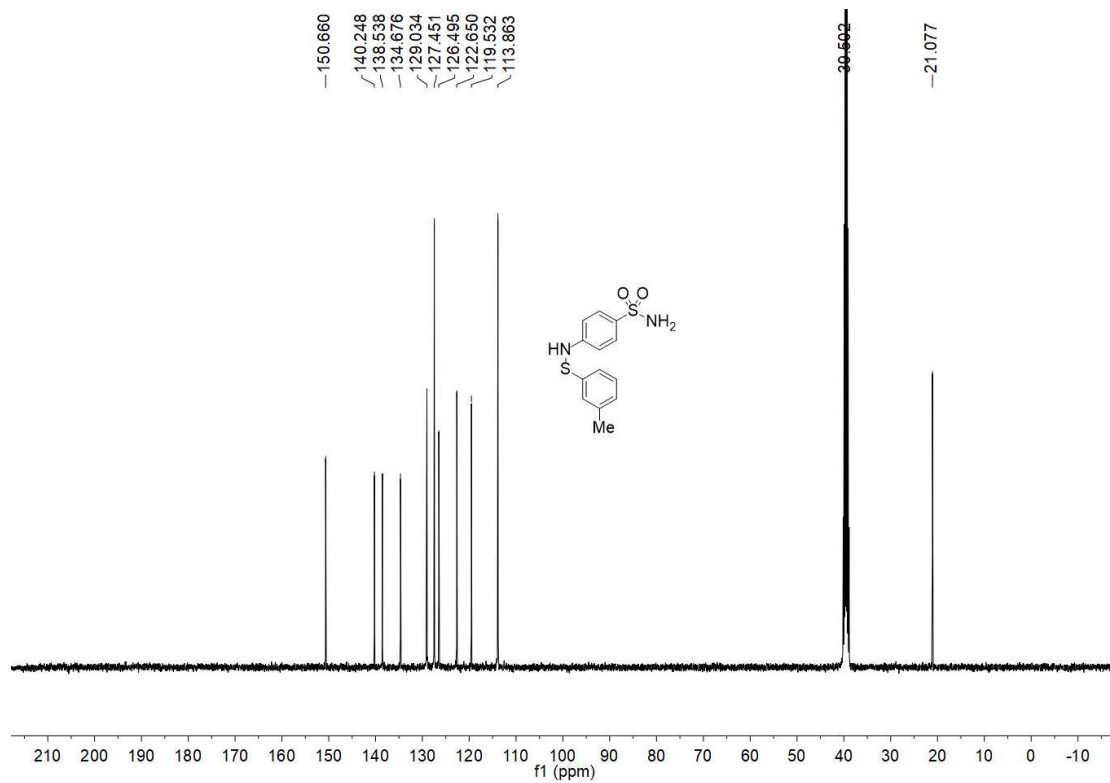
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 3h



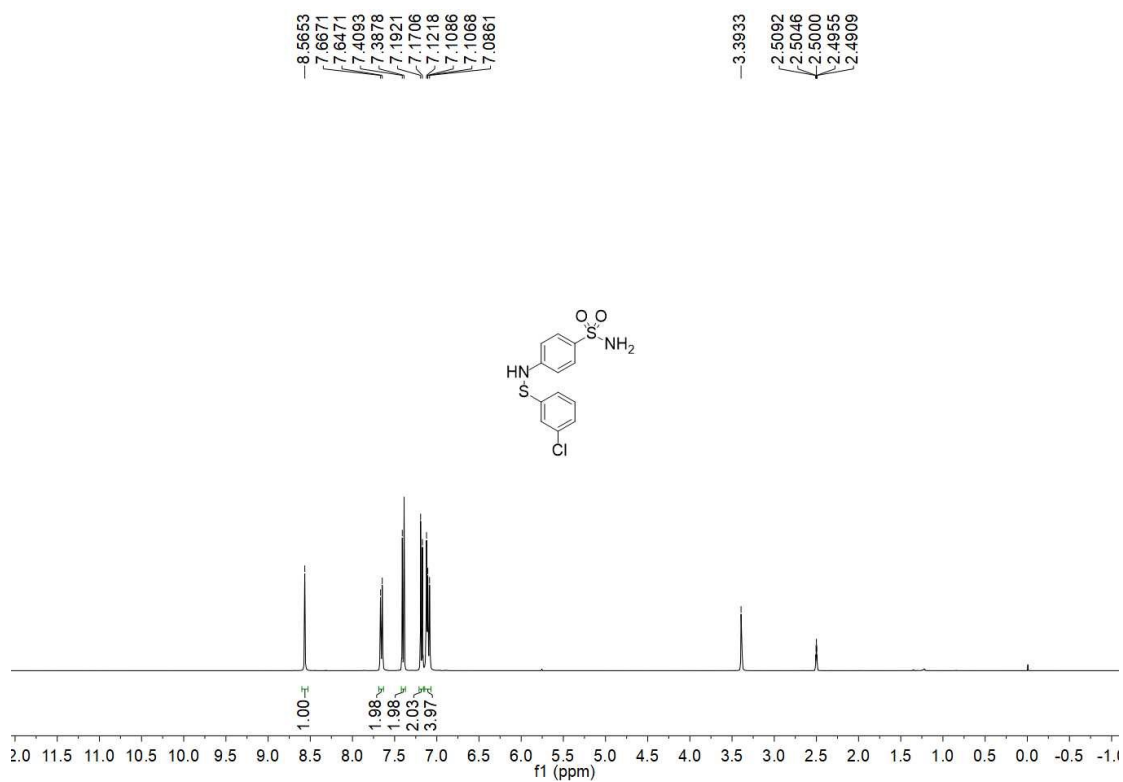
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 3h



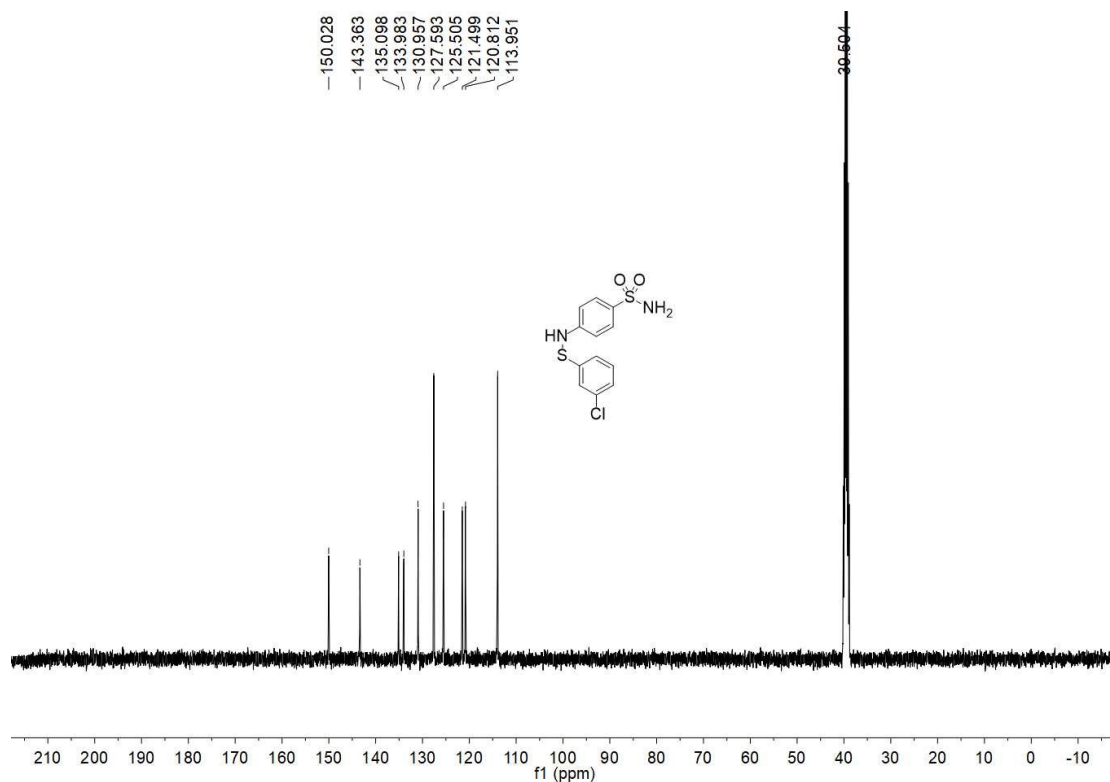
^1H NMR (400 MHz, $\text{DMSO-}d_6$) spectrum of Compound 3i



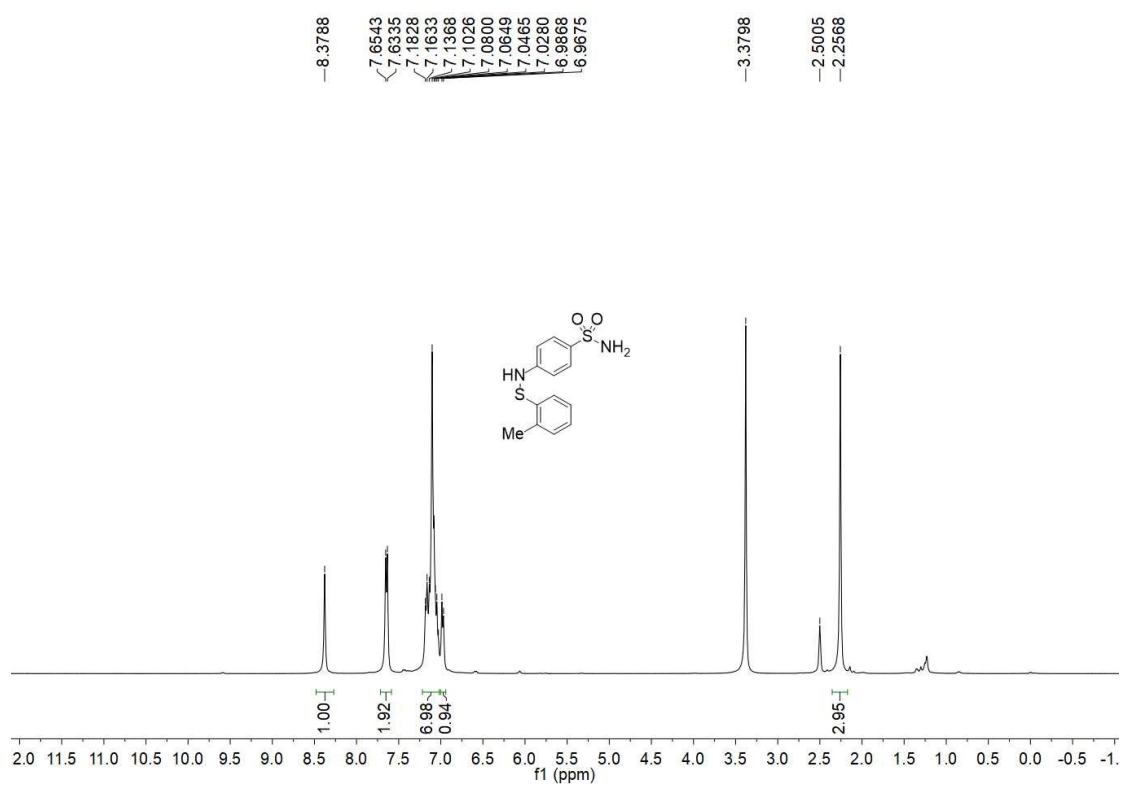
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 3i



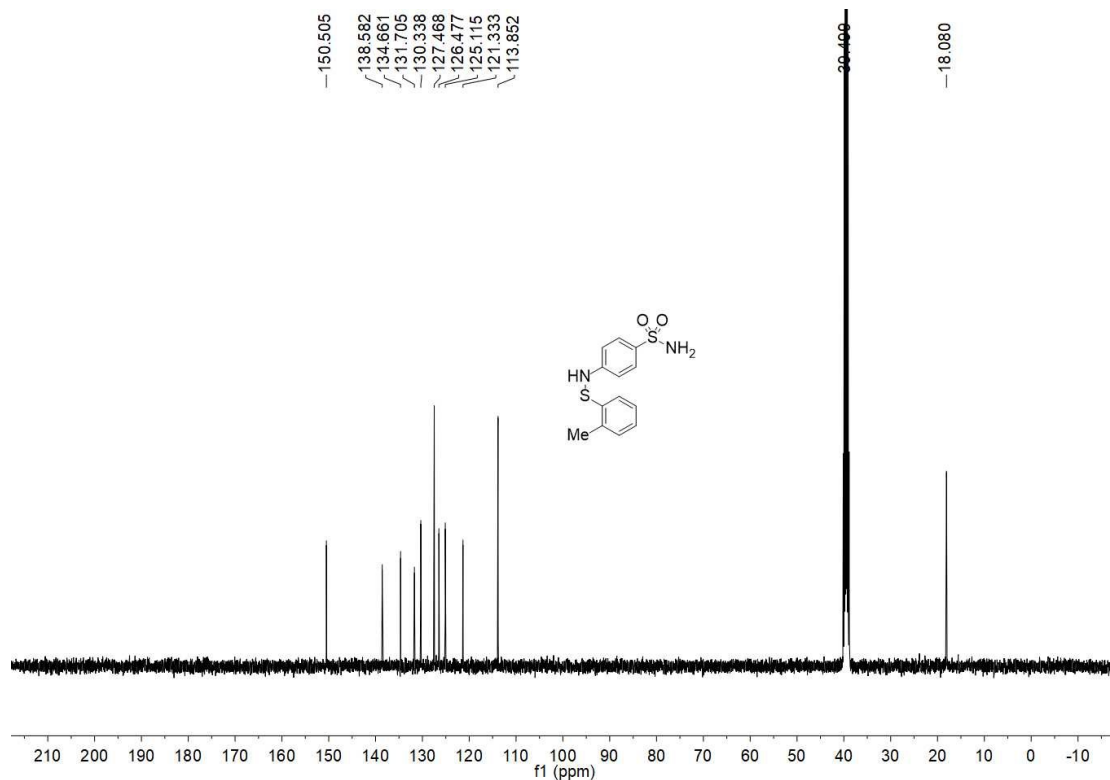
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 3j



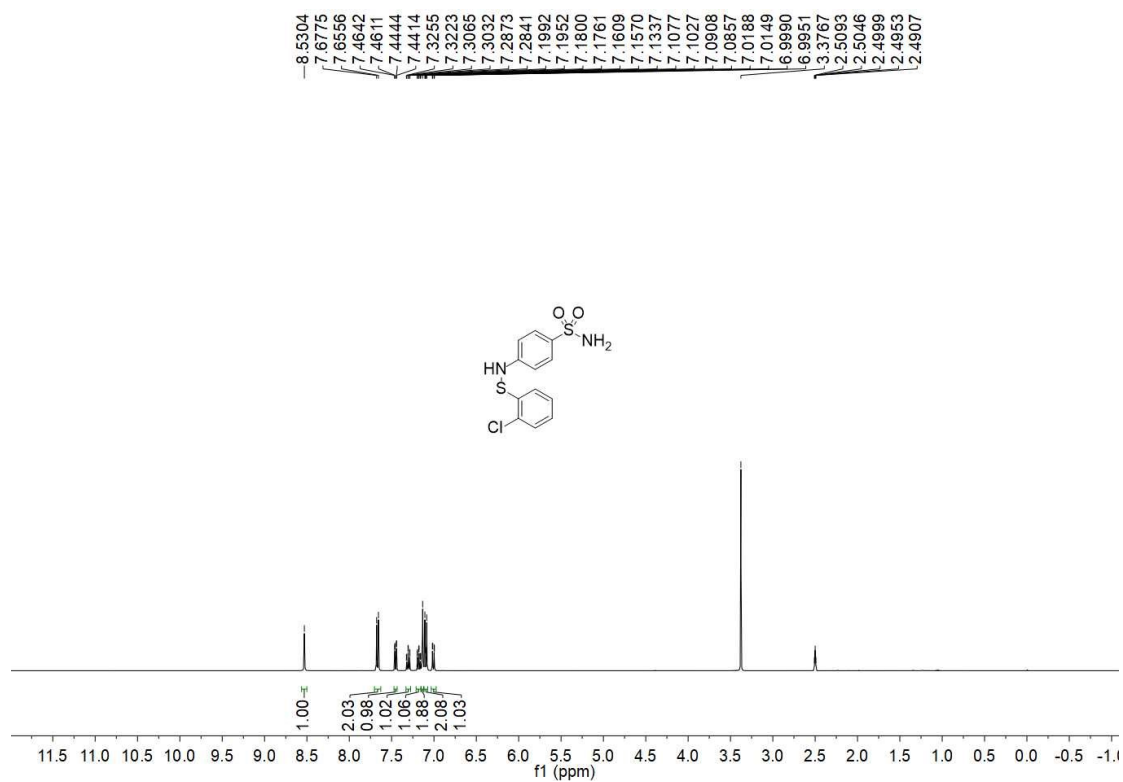
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 3j



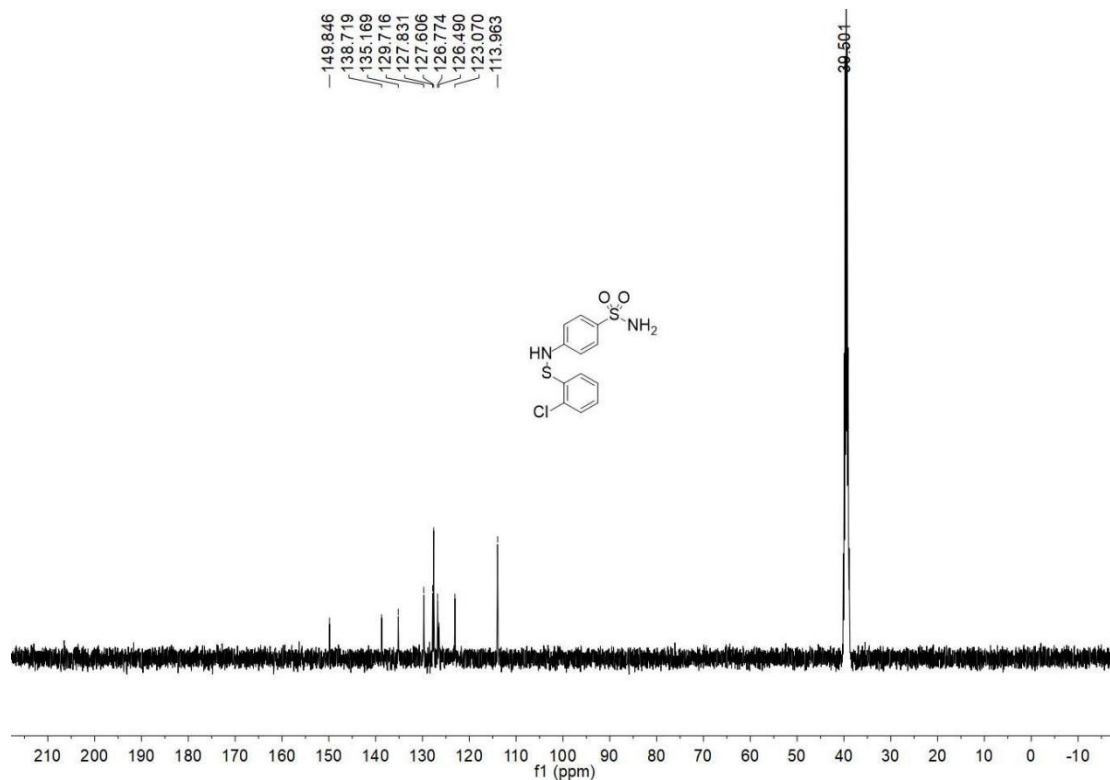
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 3k



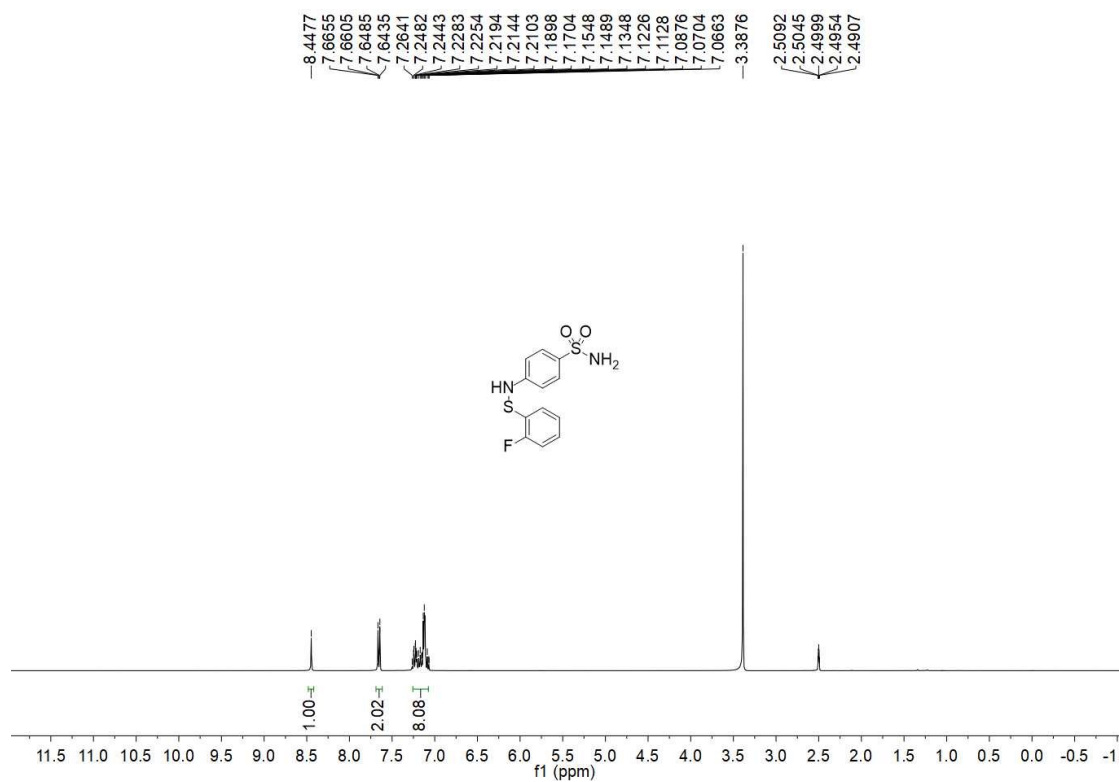
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 3k



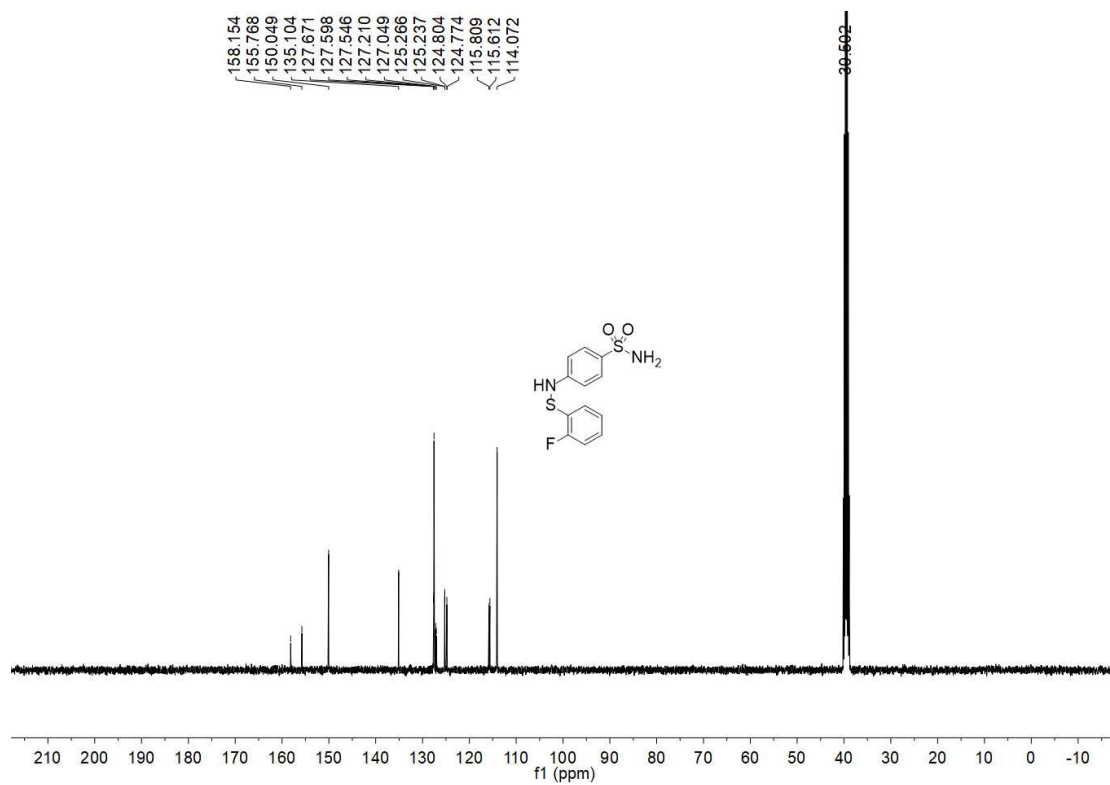
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 3l



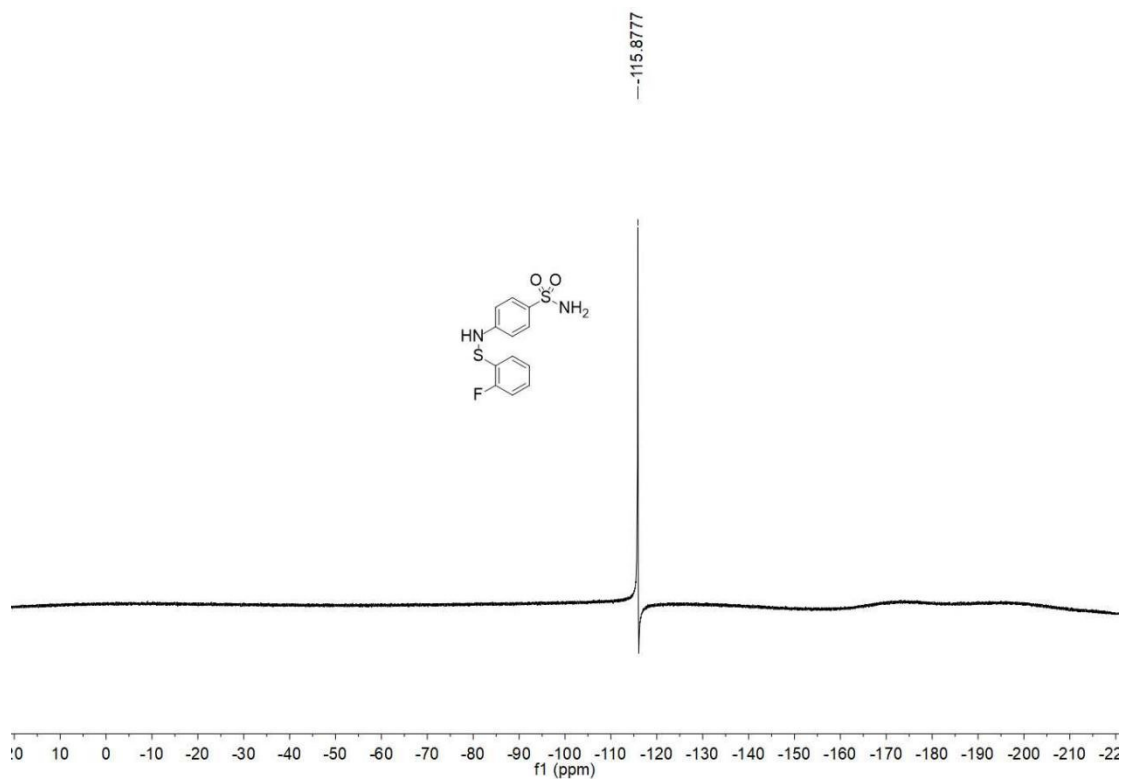
^{13}C NMR (101 MHz, DMSO- d_6) spectrum of Compound 3l



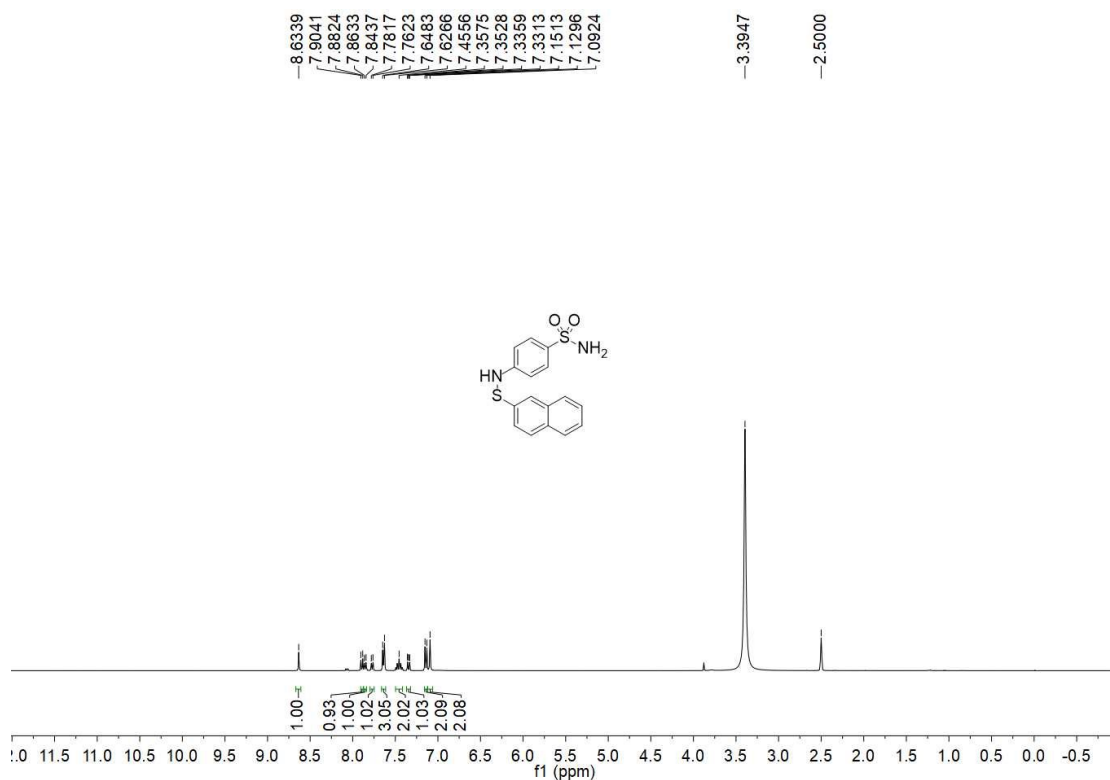
^1H NMR (400 MHz, DMSO- d_6) spectrum of Compound 3m



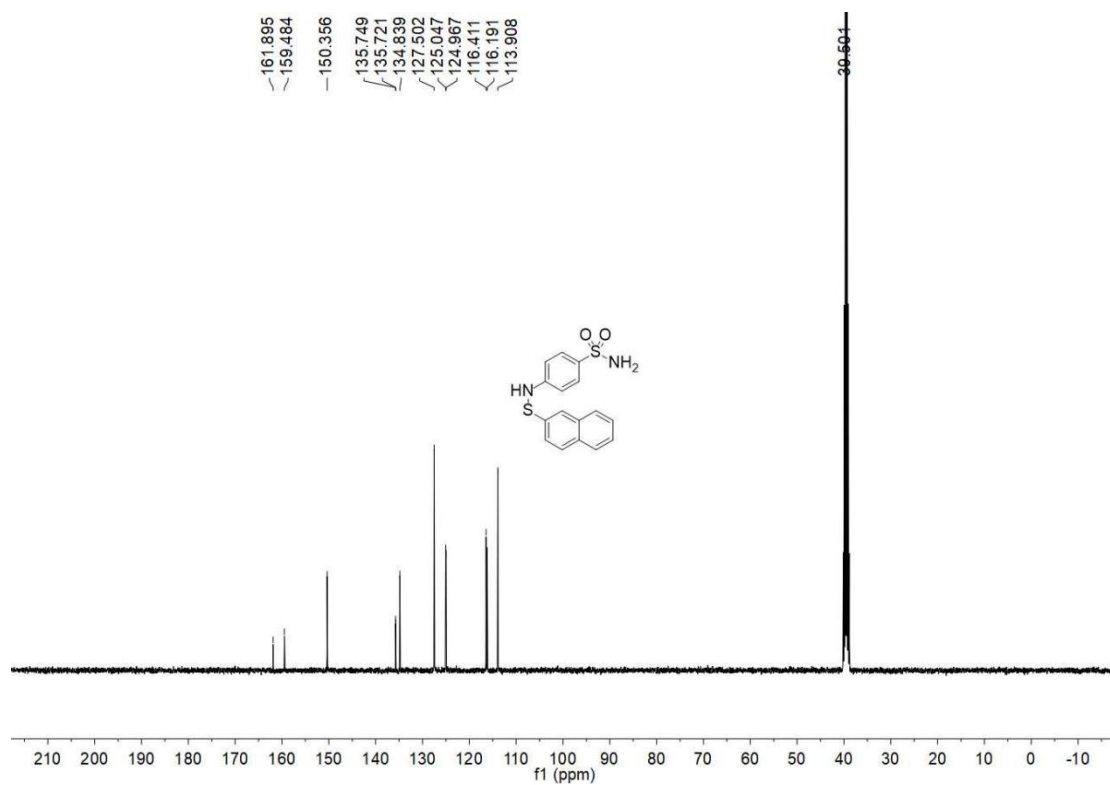
^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) spectrum of Compound **3m**



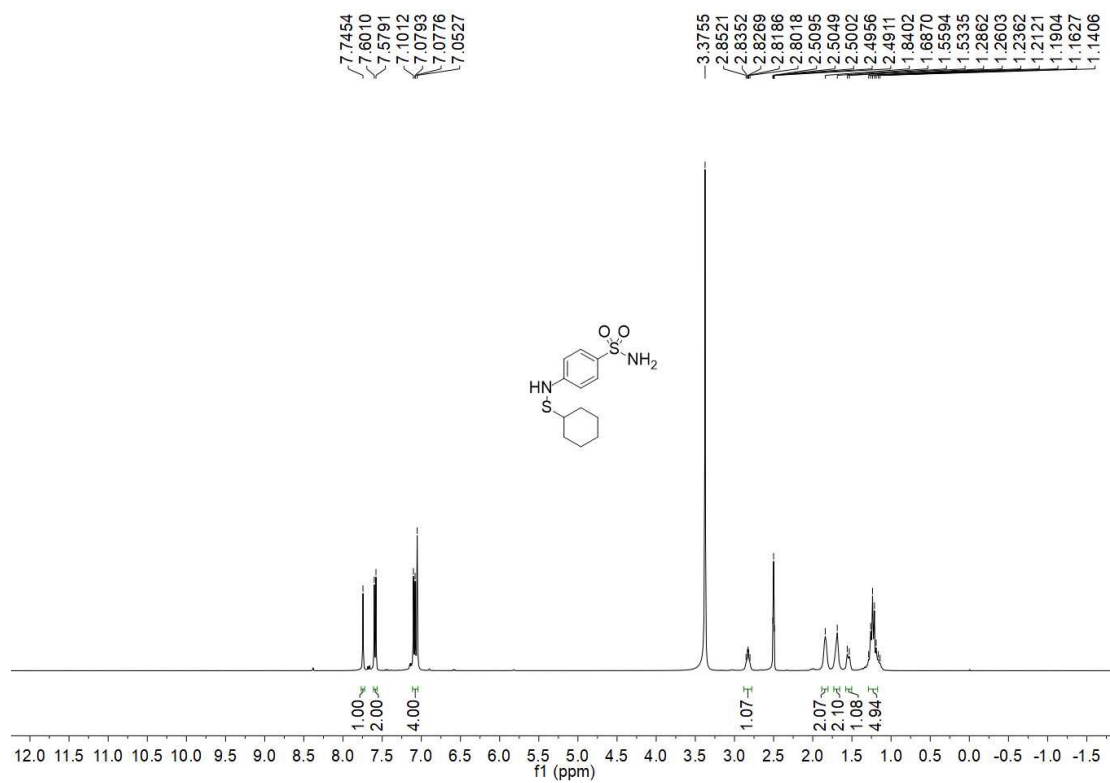
^{19}F NMR (376 MHz, $\text{DMSO-}d_6$) spectrum of Compound **3m**



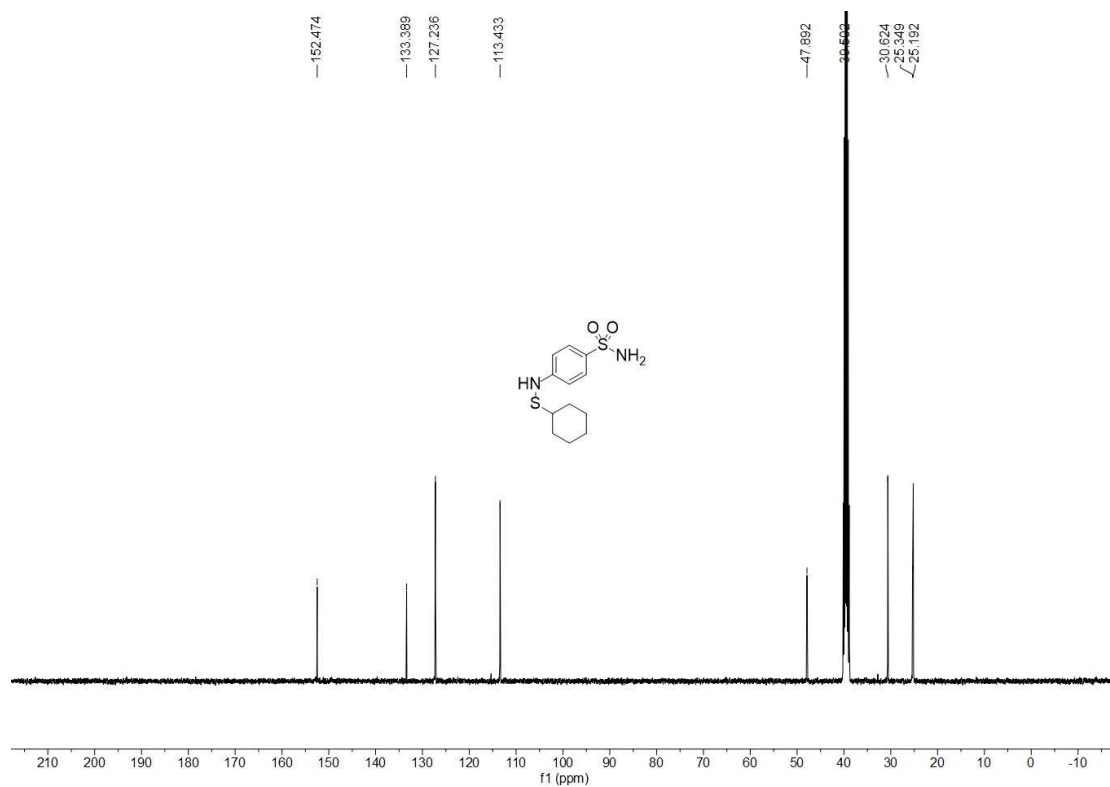
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 3n



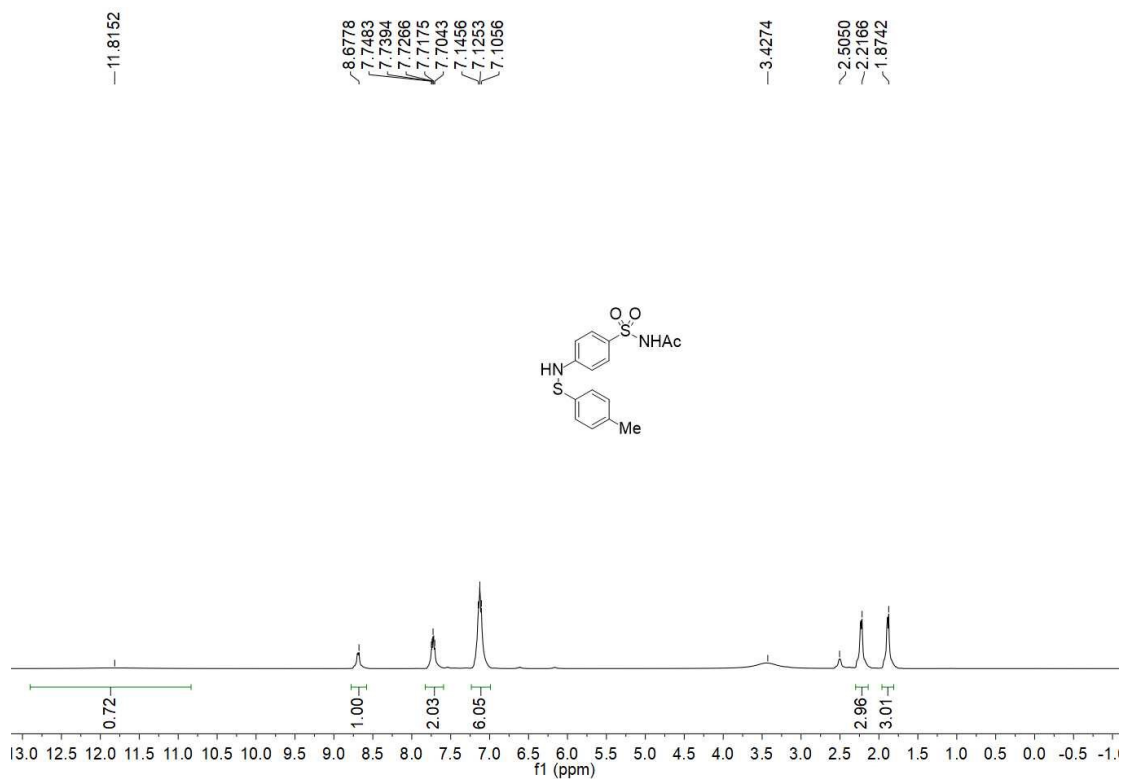
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 3n



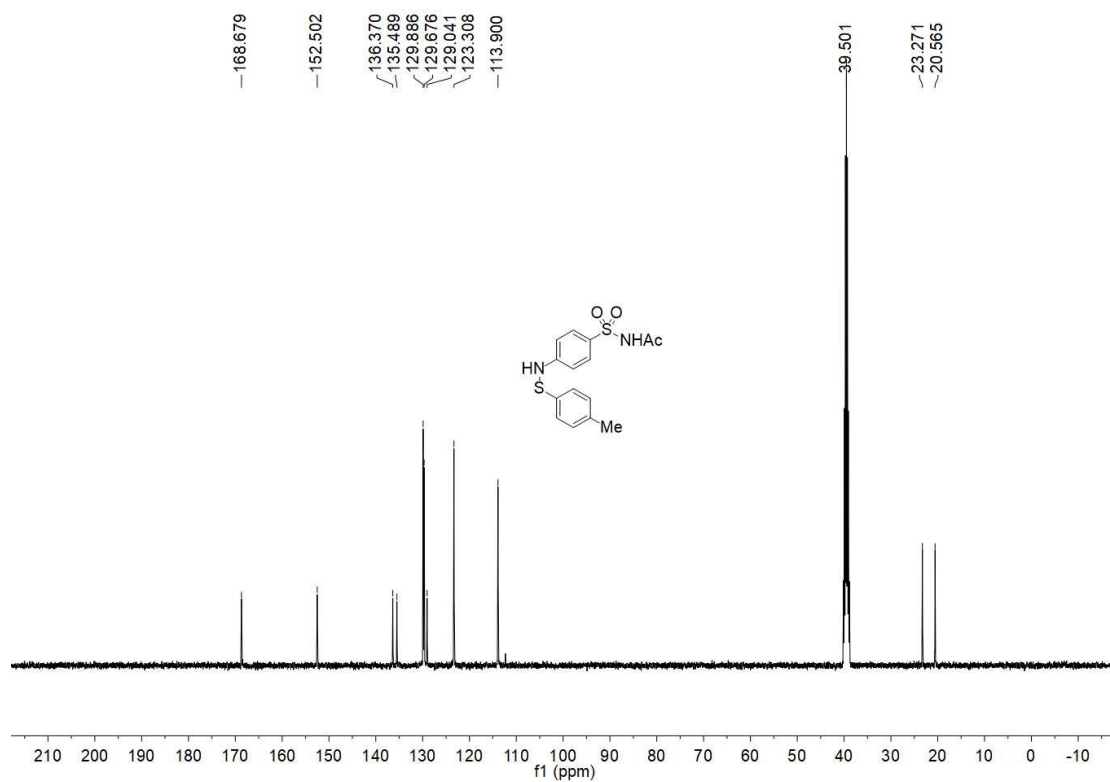
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 30



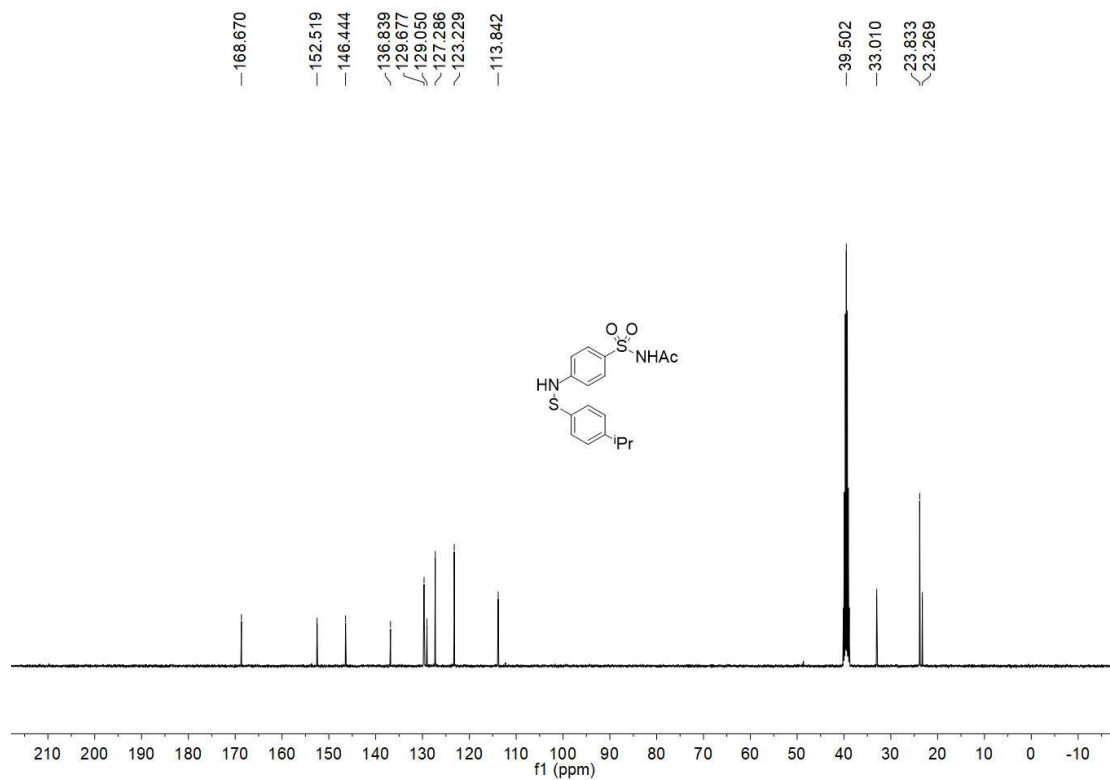
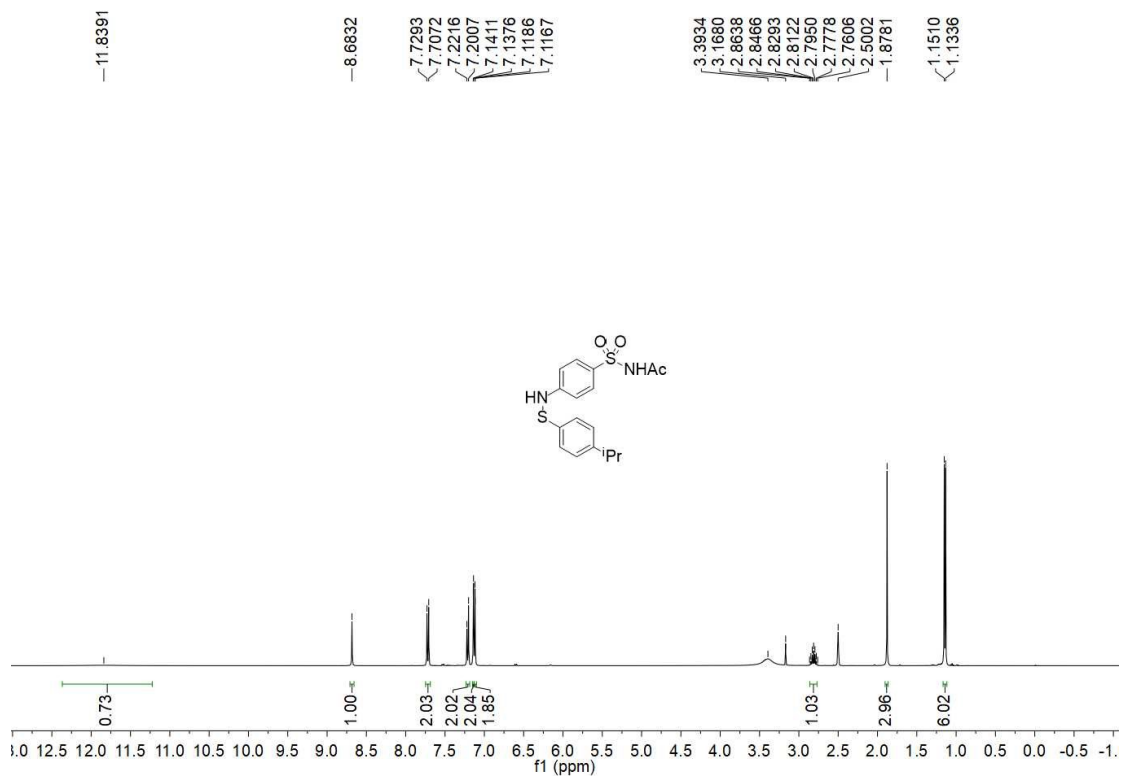
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 30

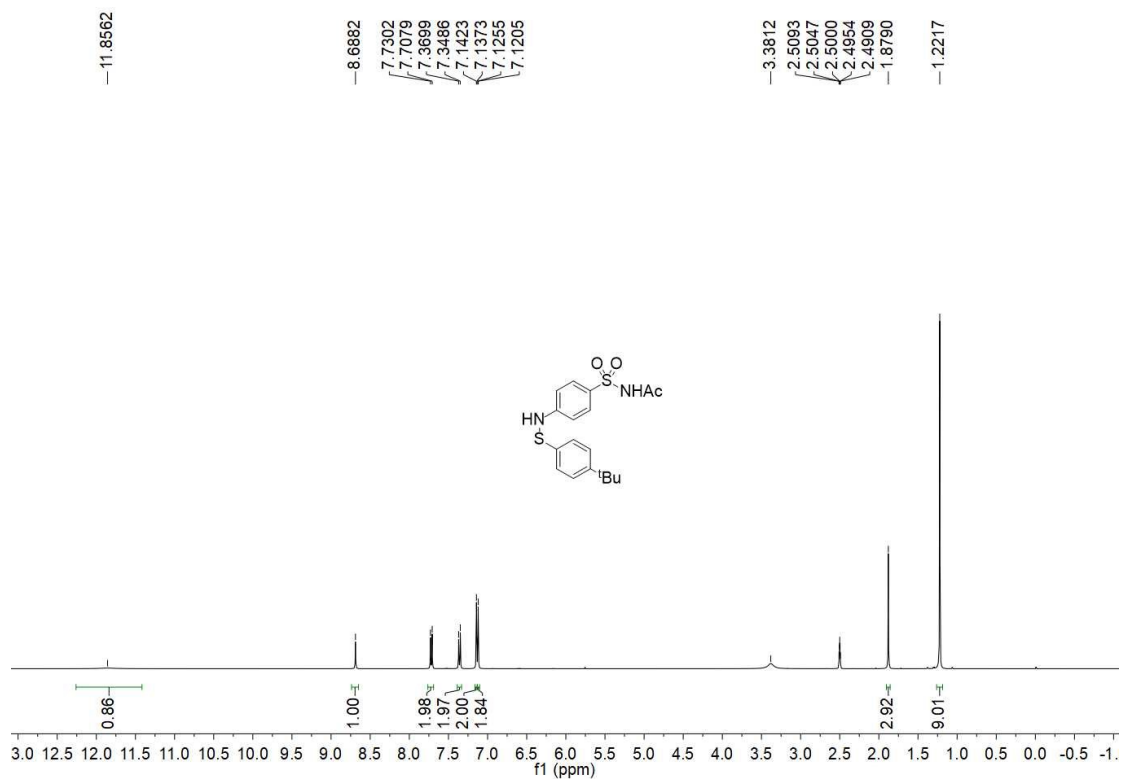


¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 4a

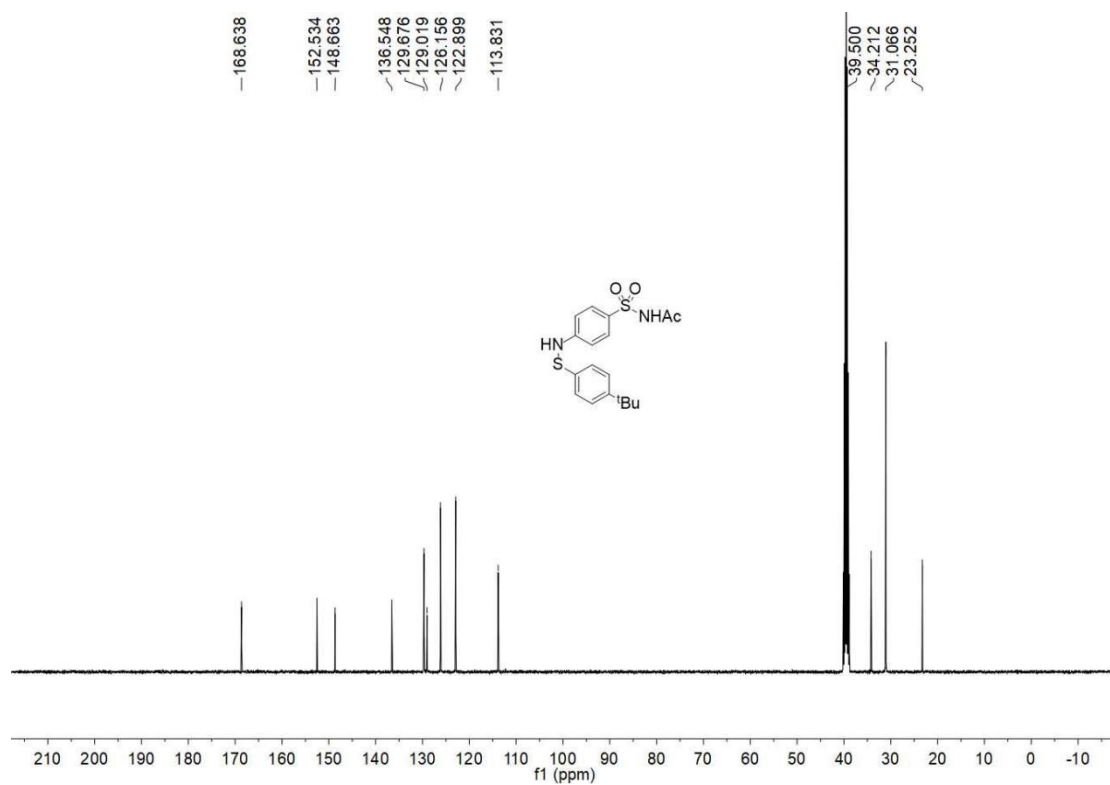


¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 4a

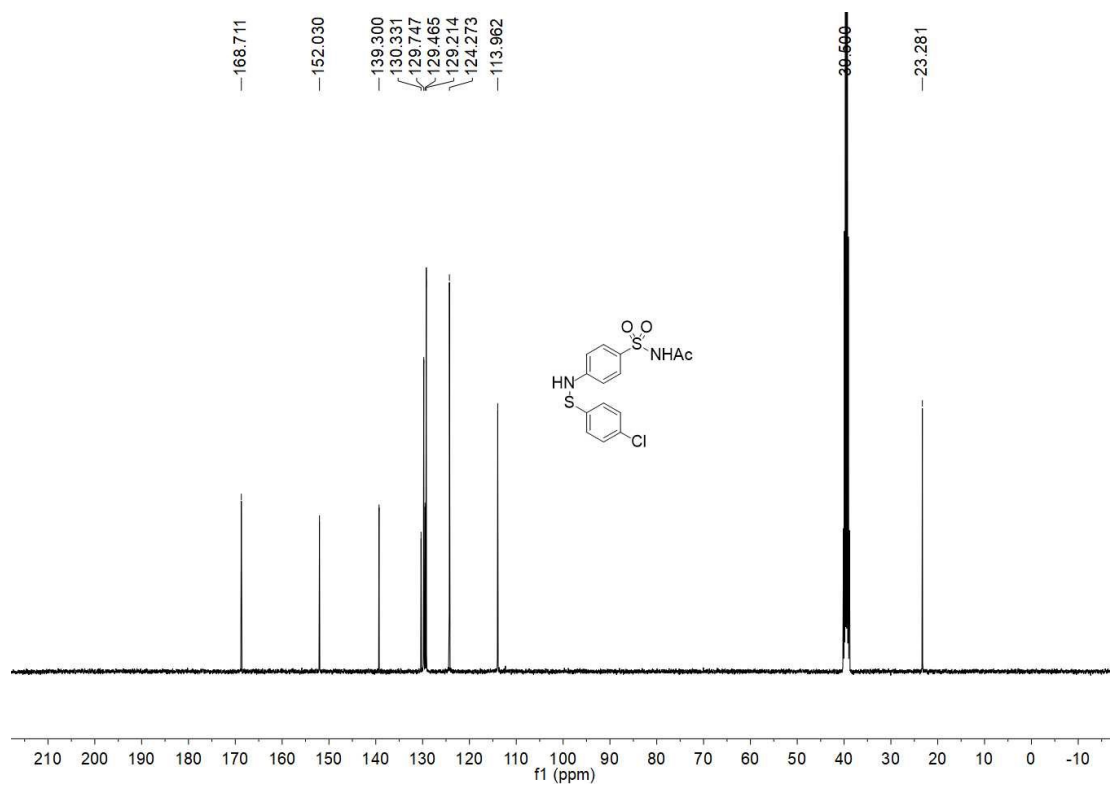
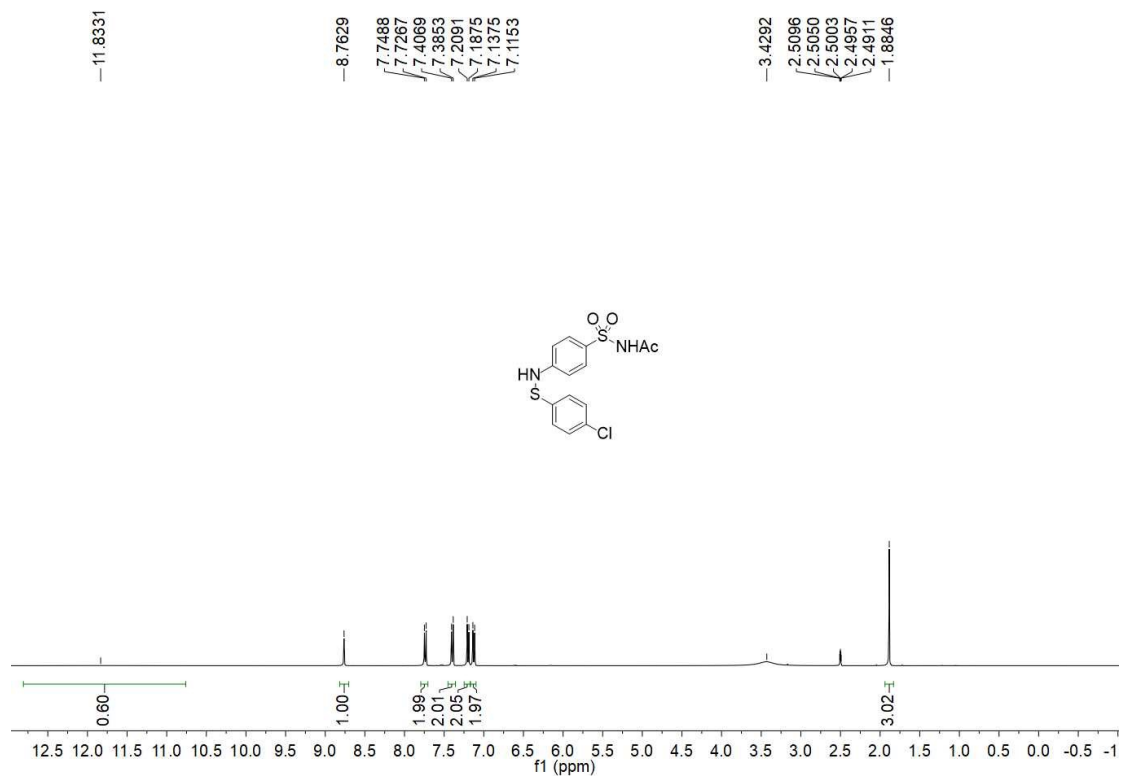


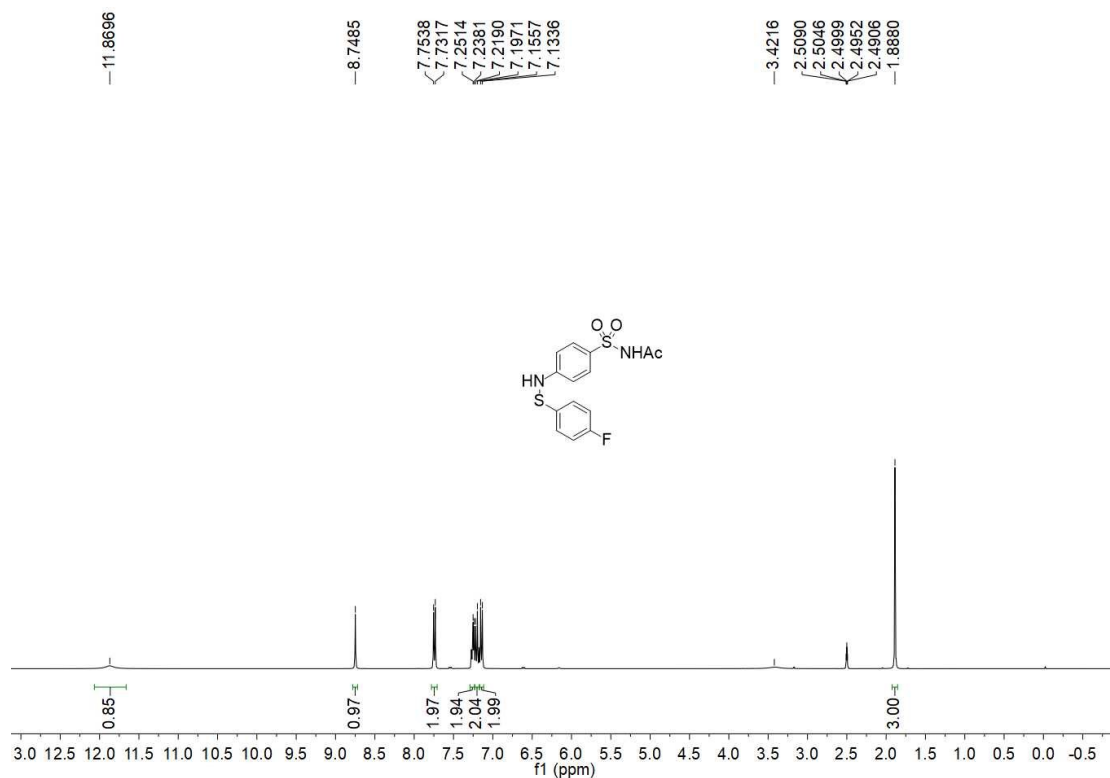


¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 4c

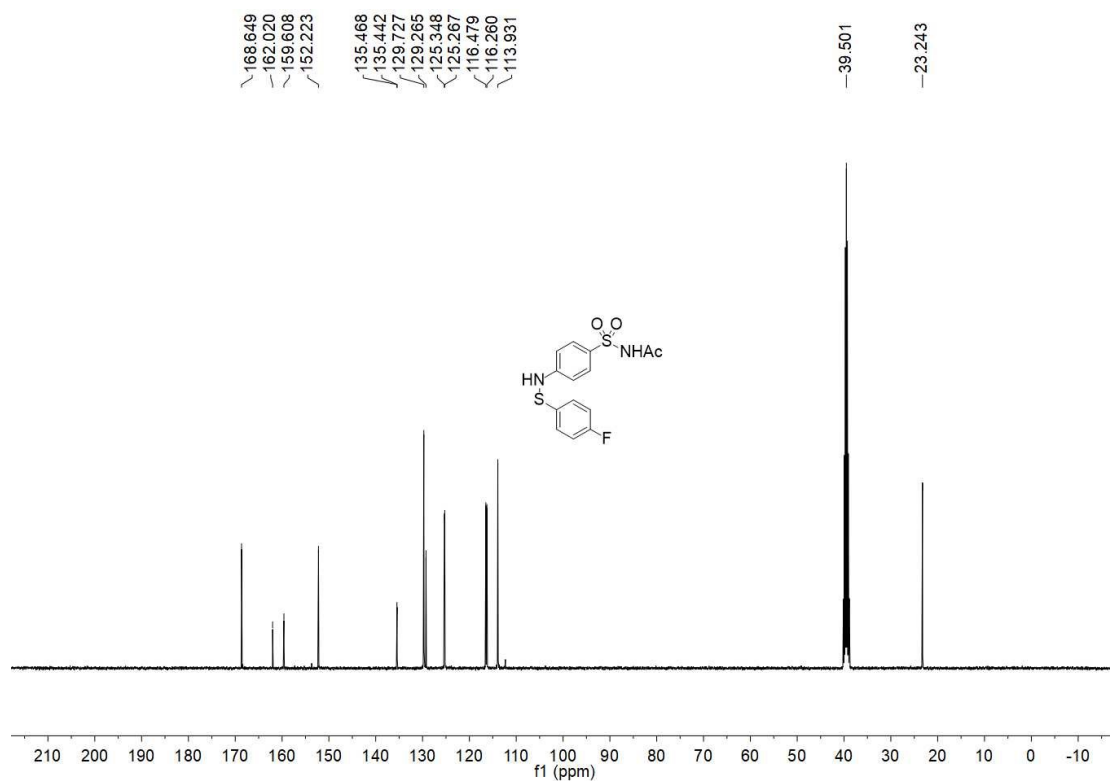


¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 4c

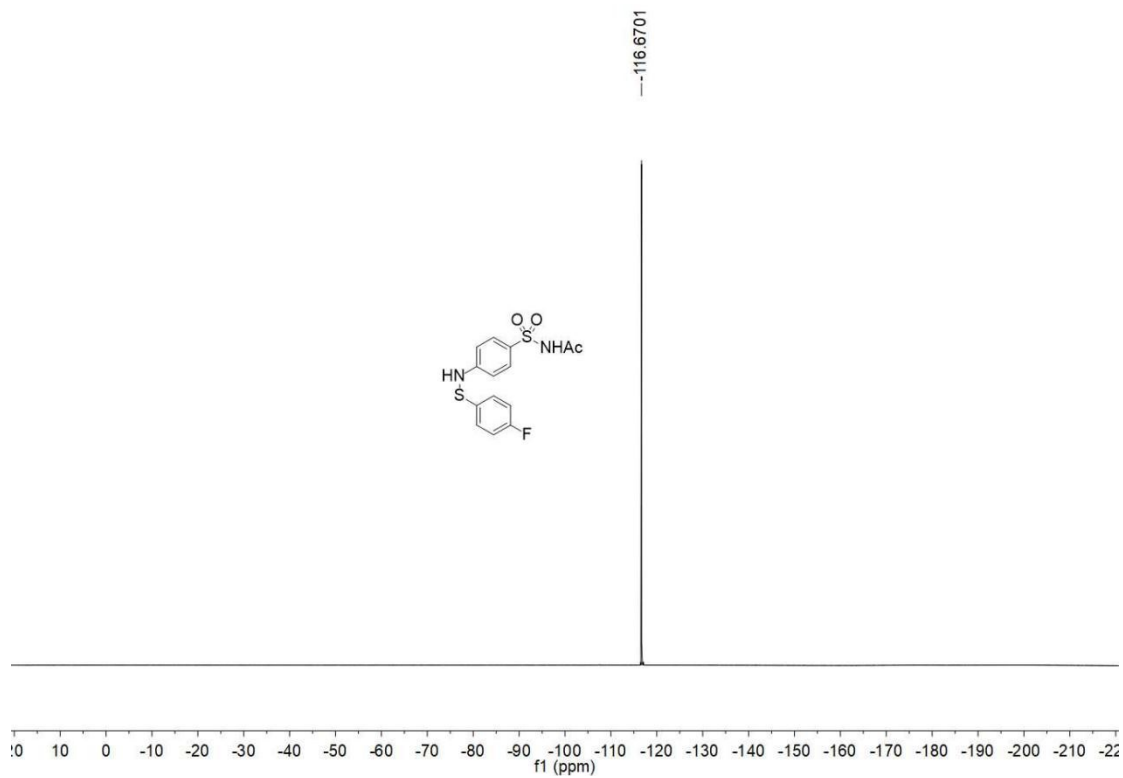




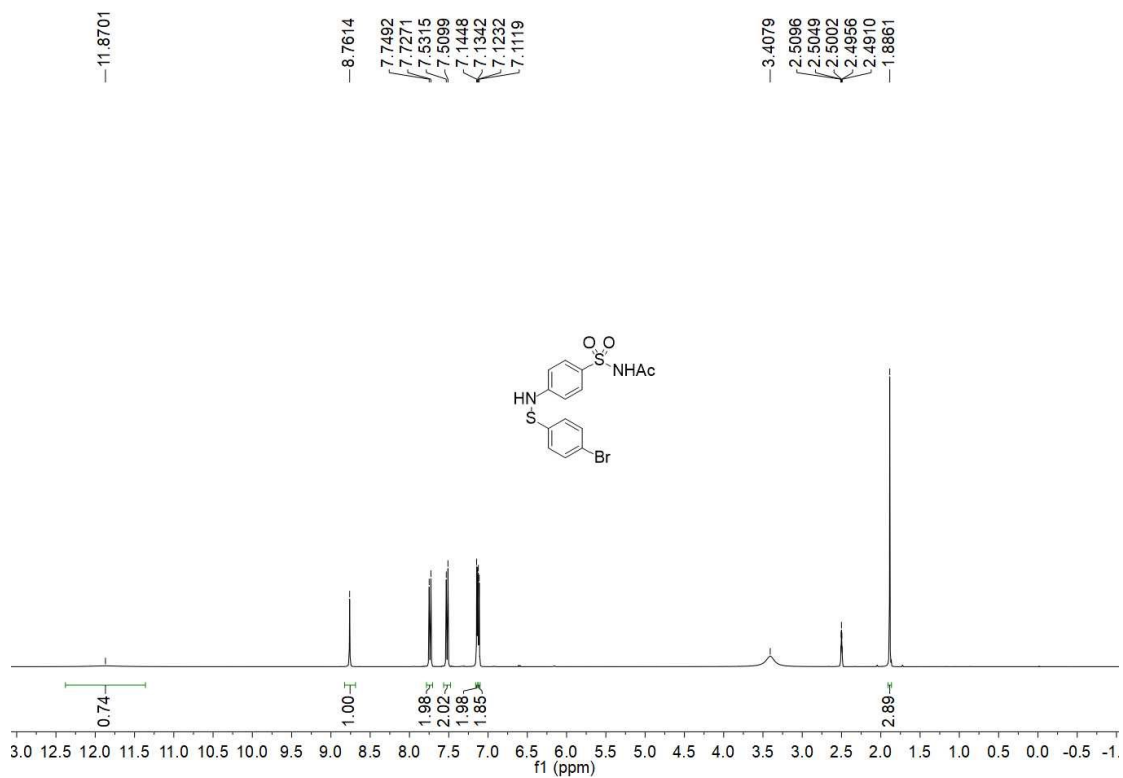
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 4e



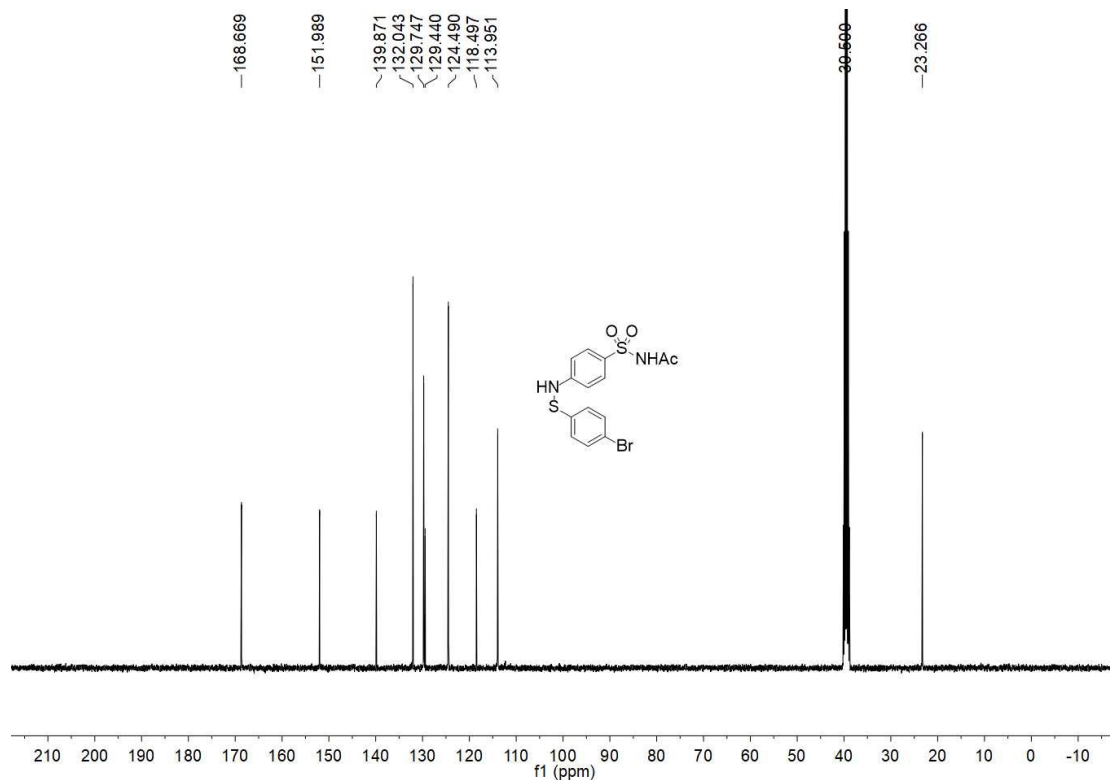
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 4e



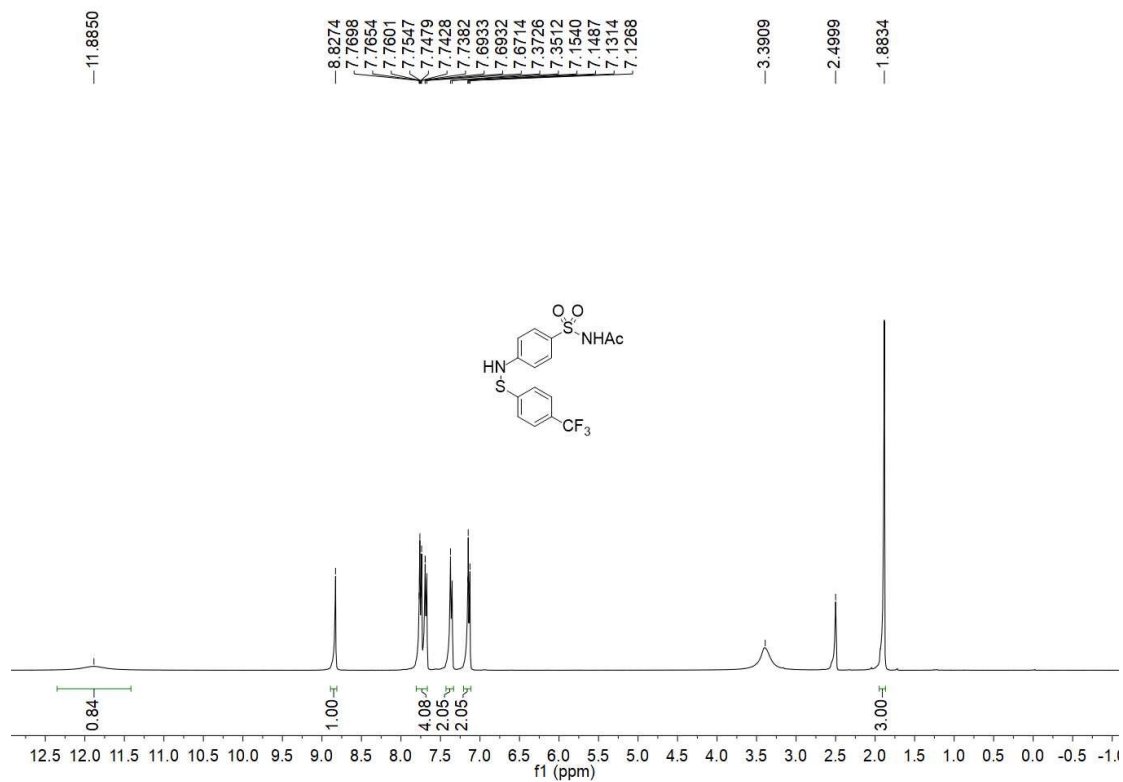
^{19}F NMR (376 MHz, $\text{DMSO-}d_6$) spectrum of Compound 4e



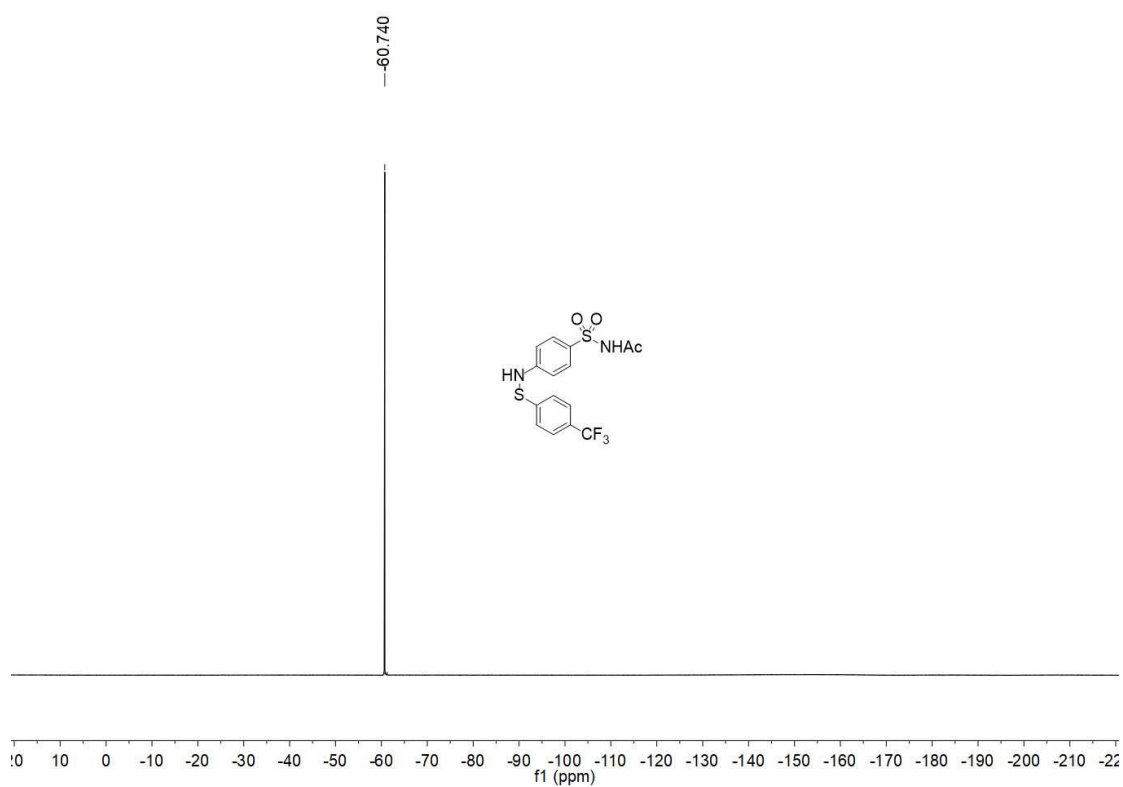
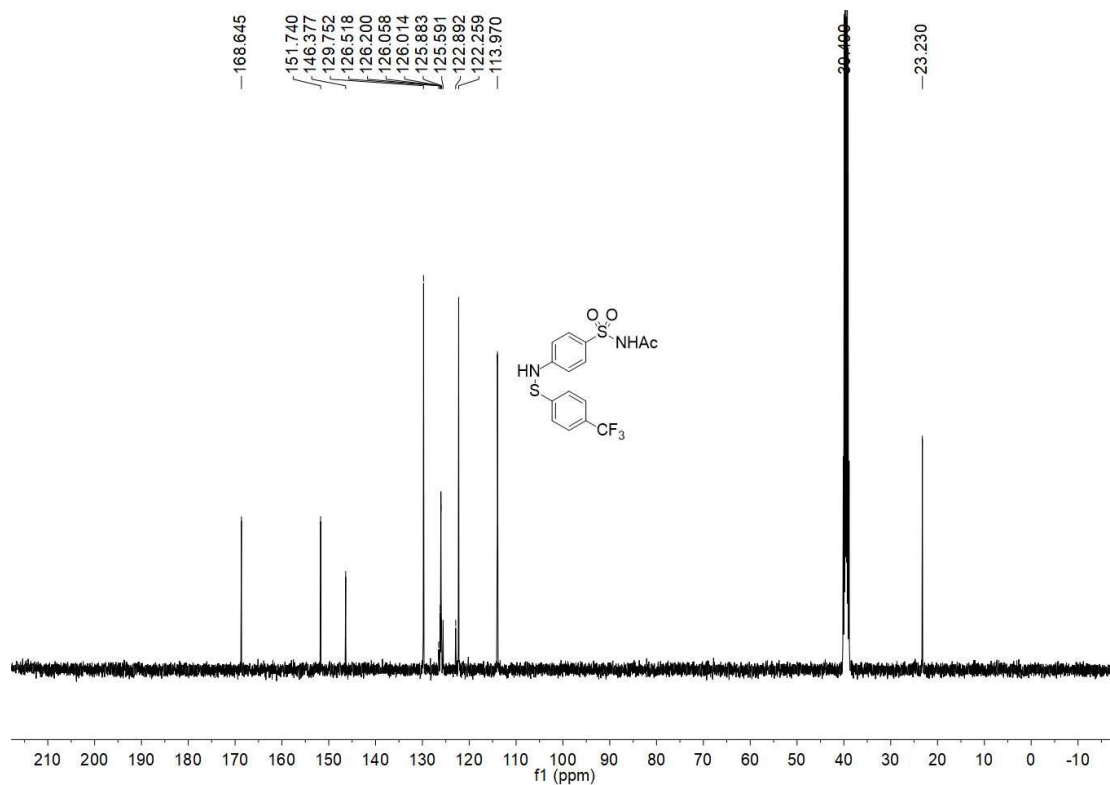
^1H NMR (400 MHz, $\text{DMSO-}d_6$) spectrum of Compound 4f

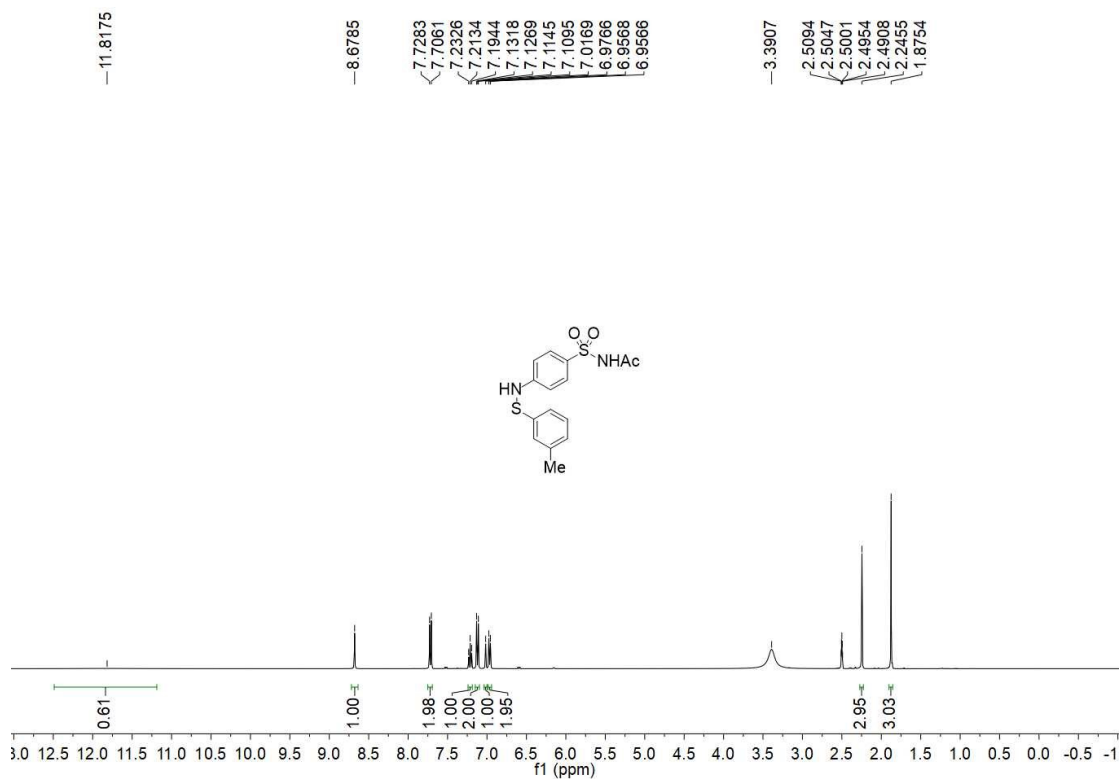


^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) spectrum of Compound 4f

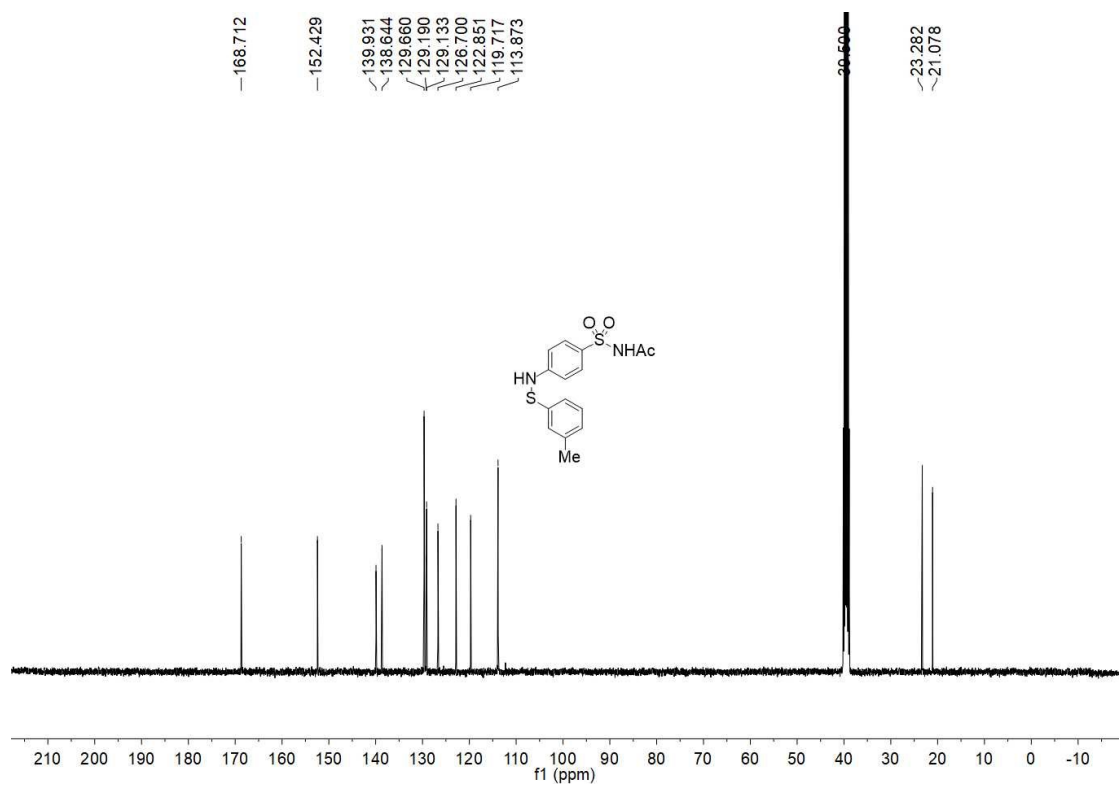


^1H NMR (400 MHz, $\text{DMSO-}d_6$) spectrum of Compound 4g

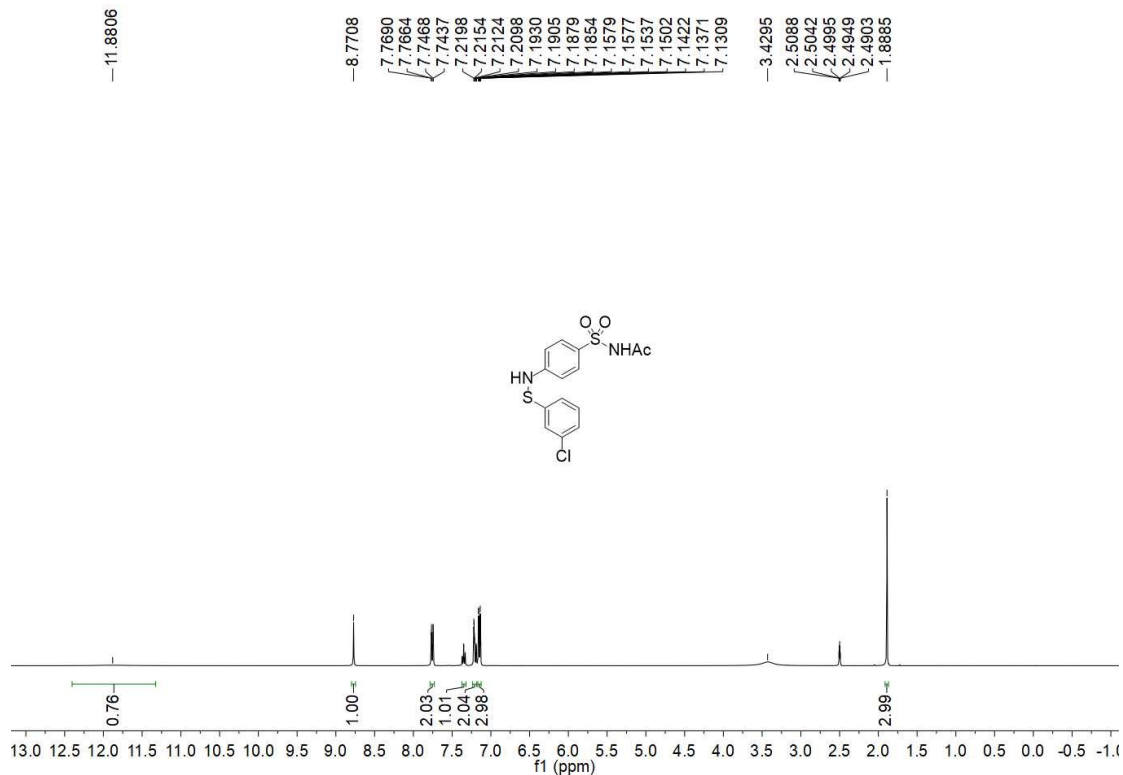




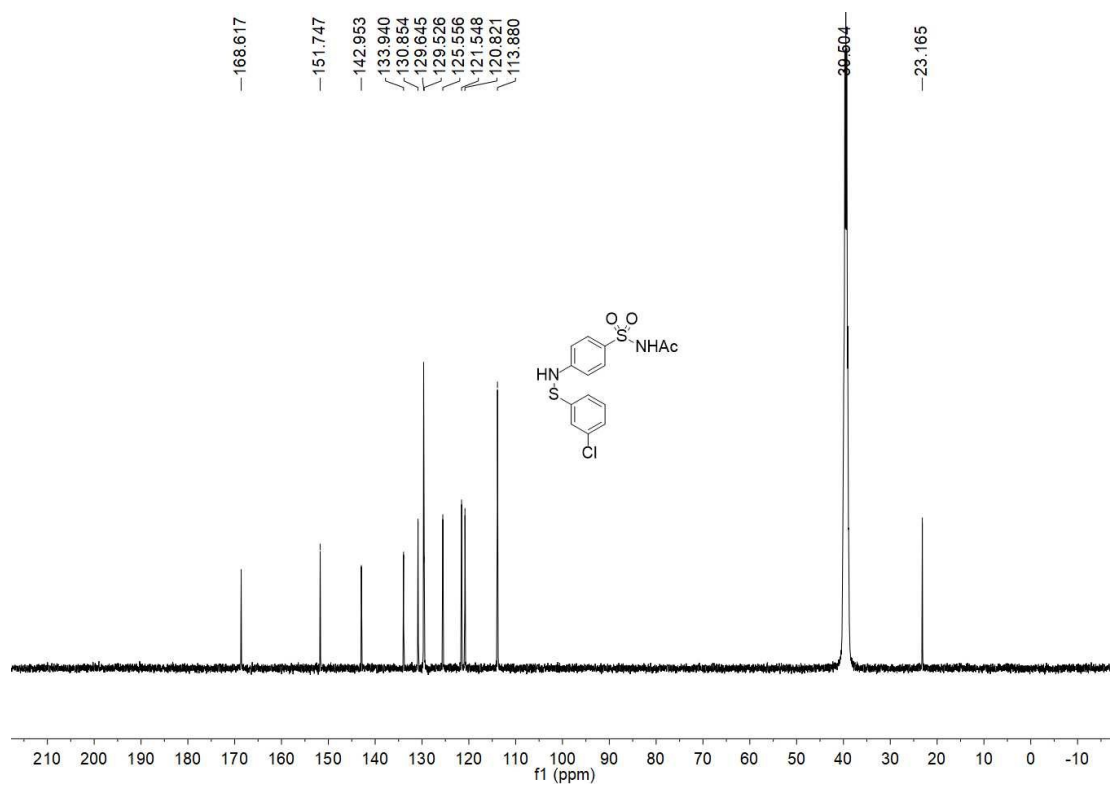
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 4h



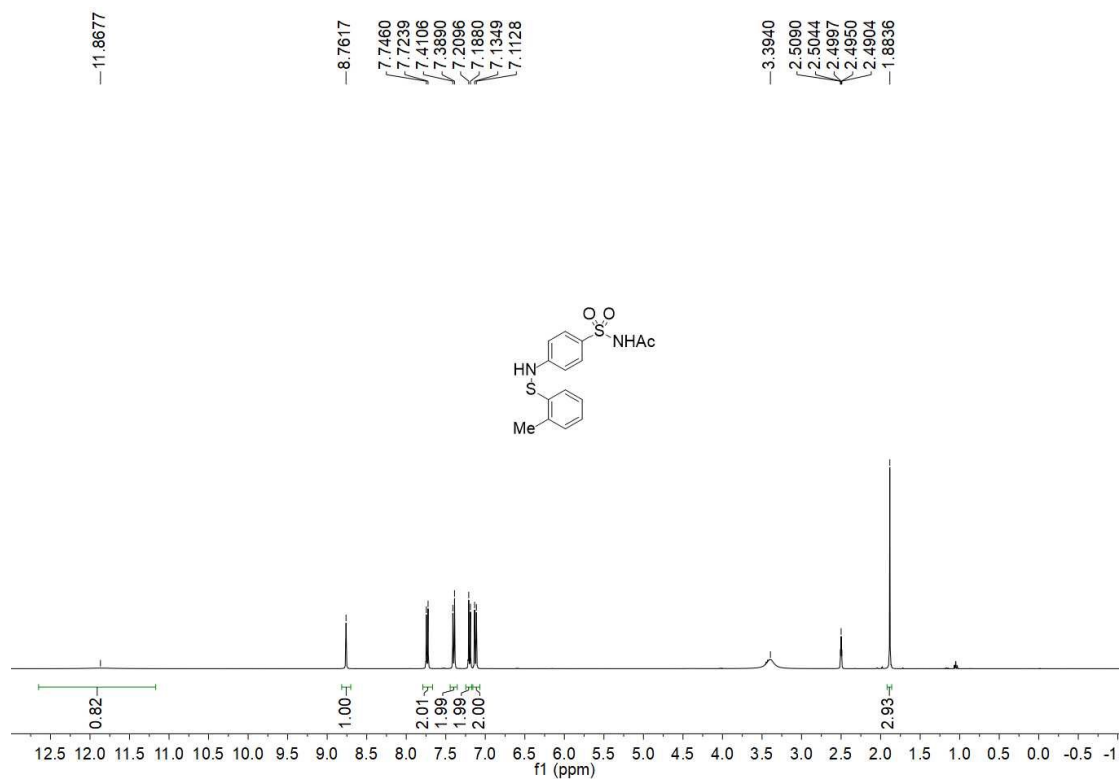
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 4h



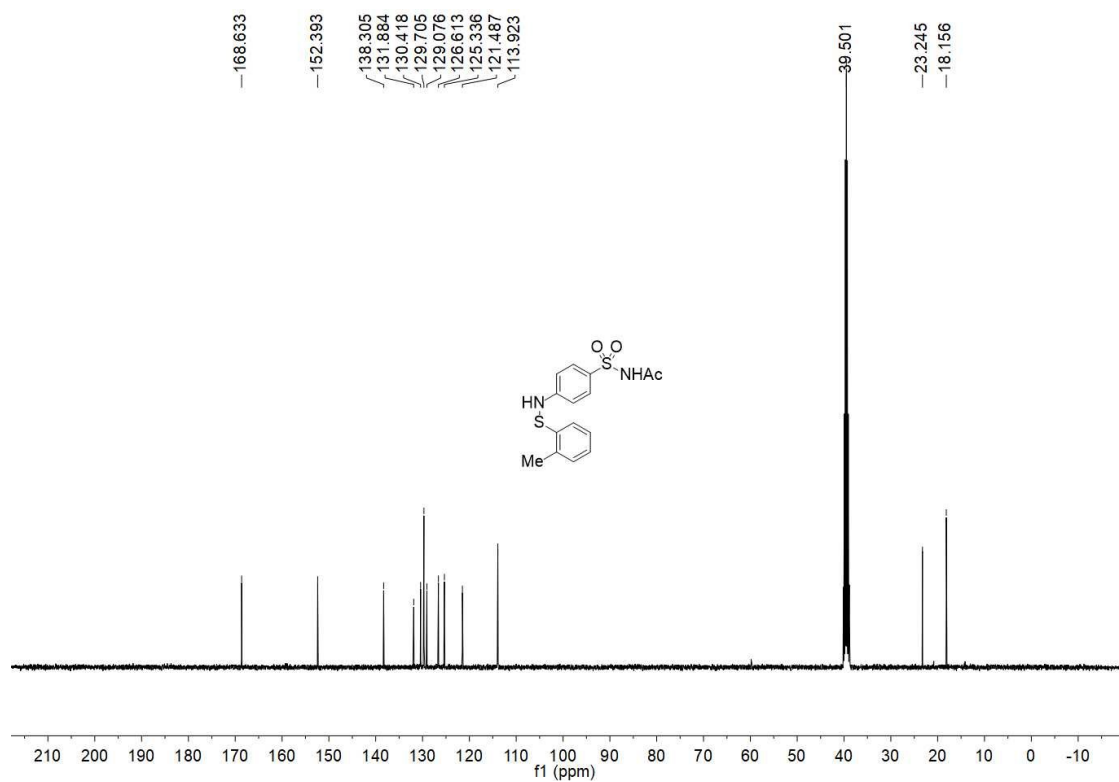
^1H NMR (400 MHz, $\text{DMSO-}d_6$) spectrum of Compound **4i**



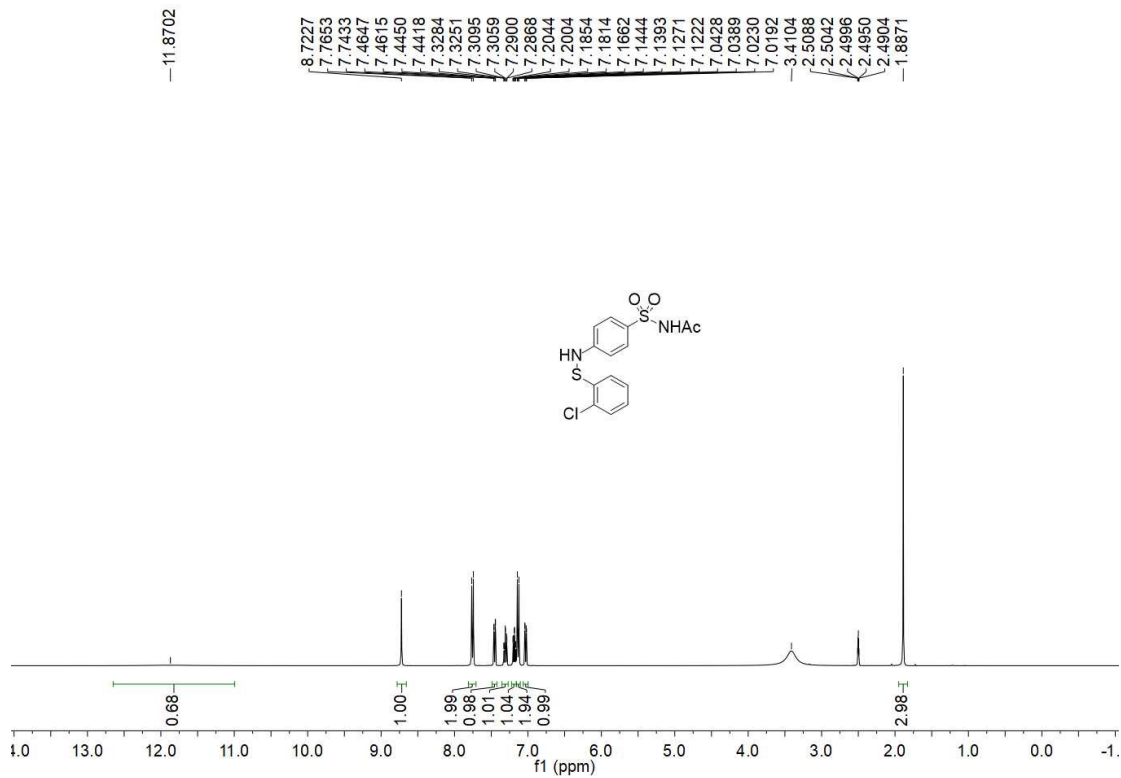
^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) spectrum of Compound **4i**



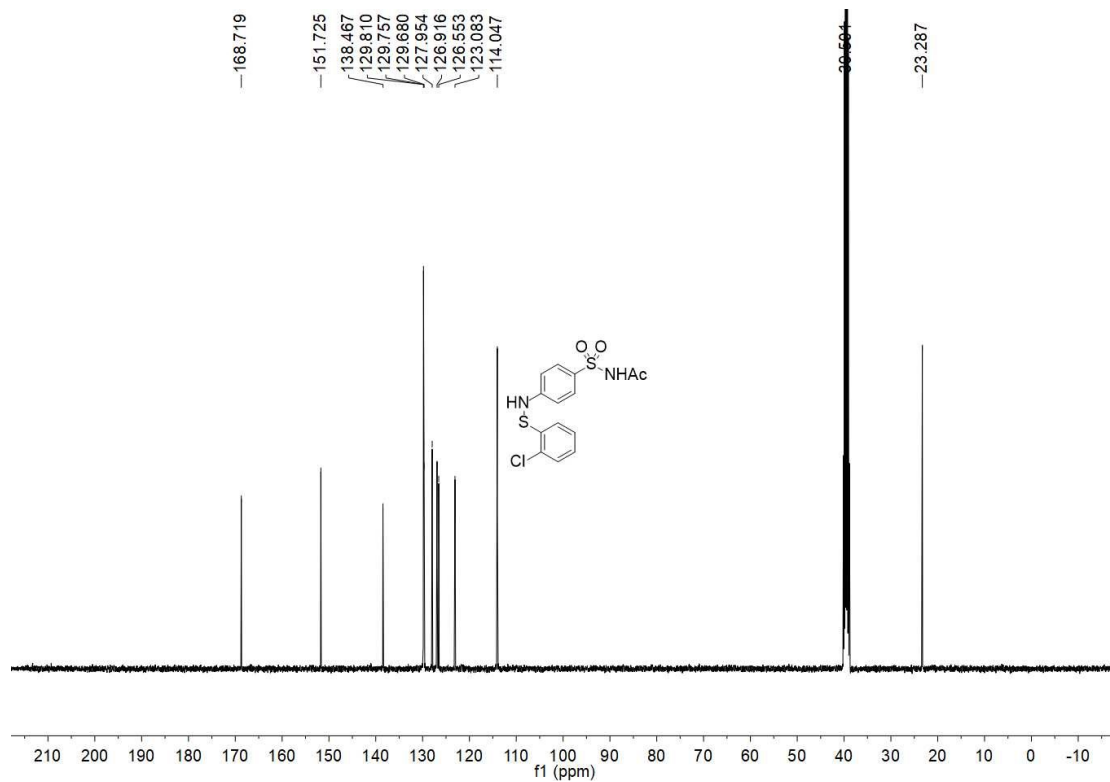
^1H NMR (400 MHz, $\text{DMSO-}d_6$) spectrum of Compound 4j



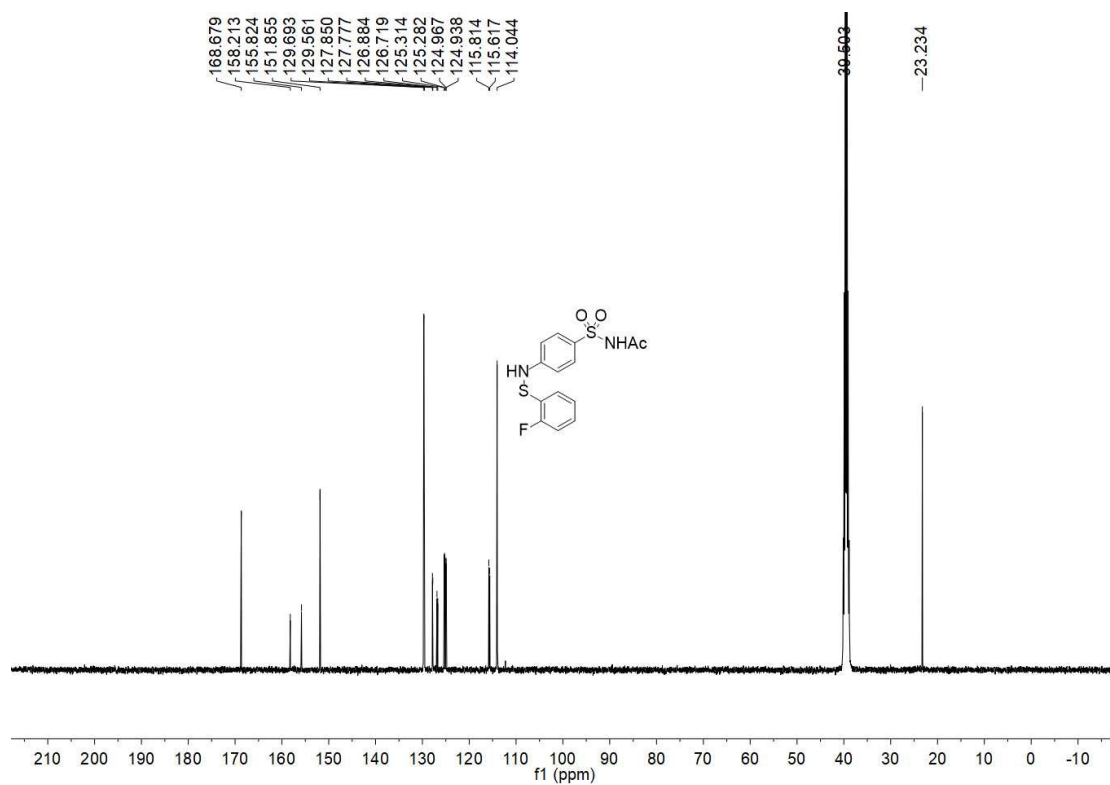
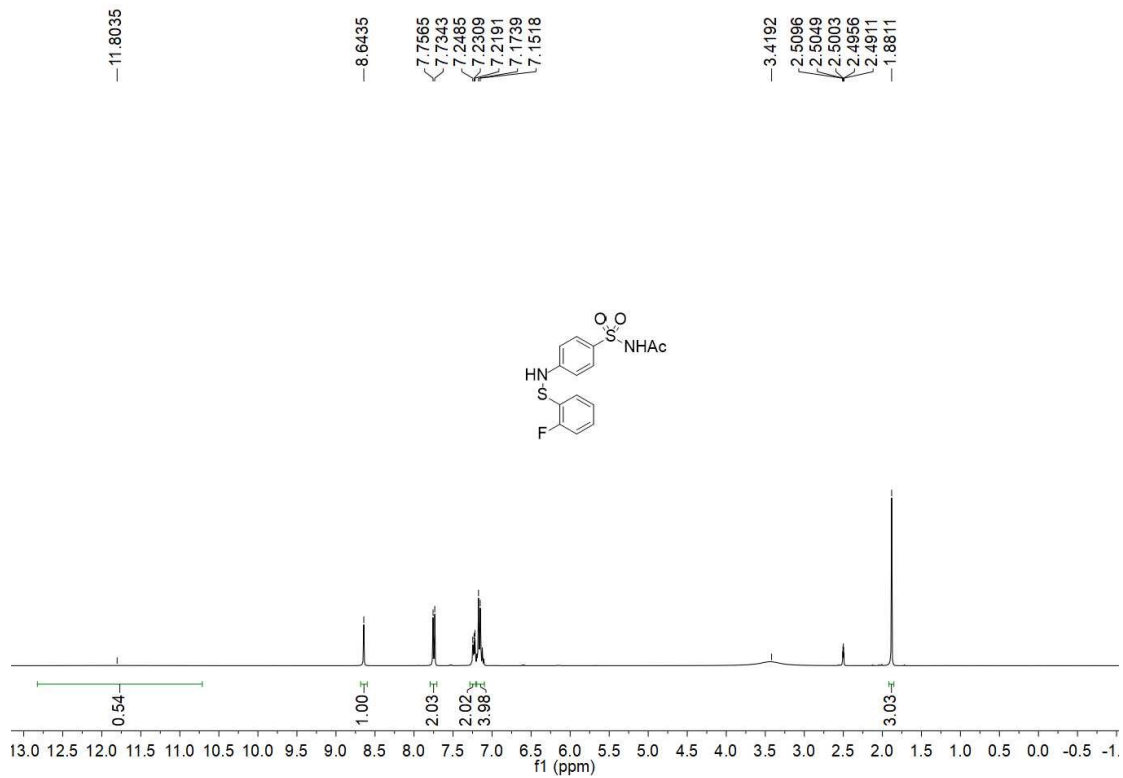
^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) spectrum of Compound 4j

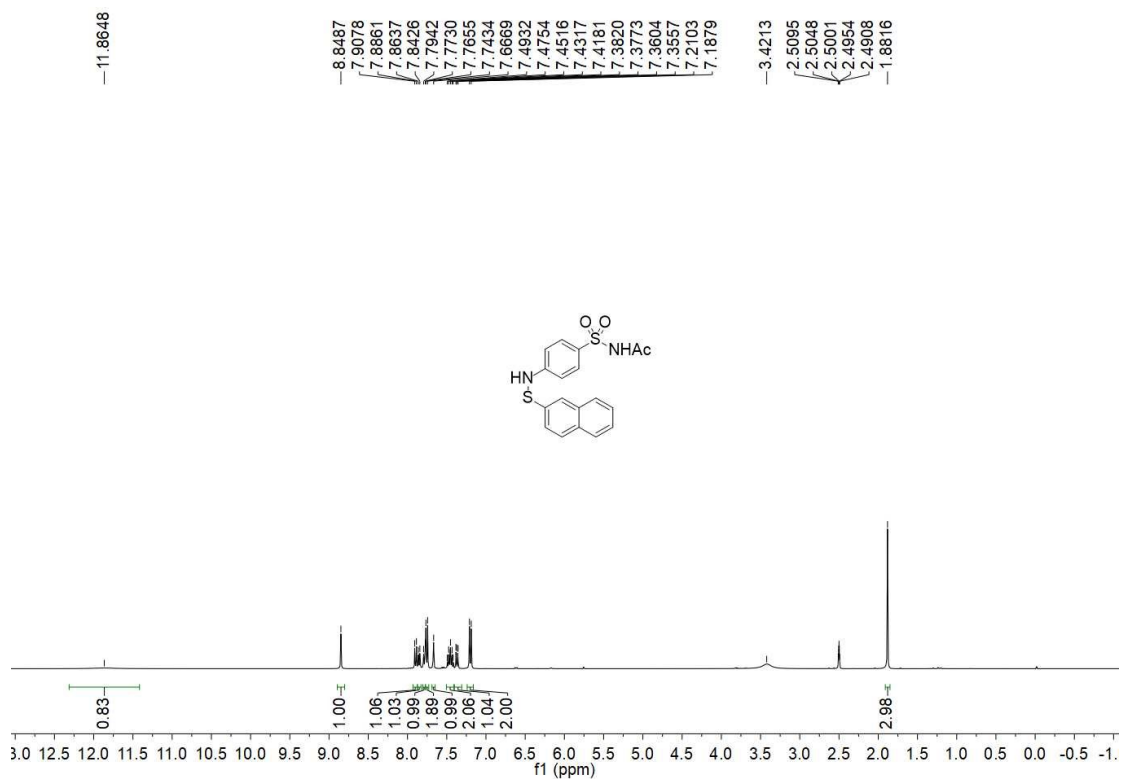
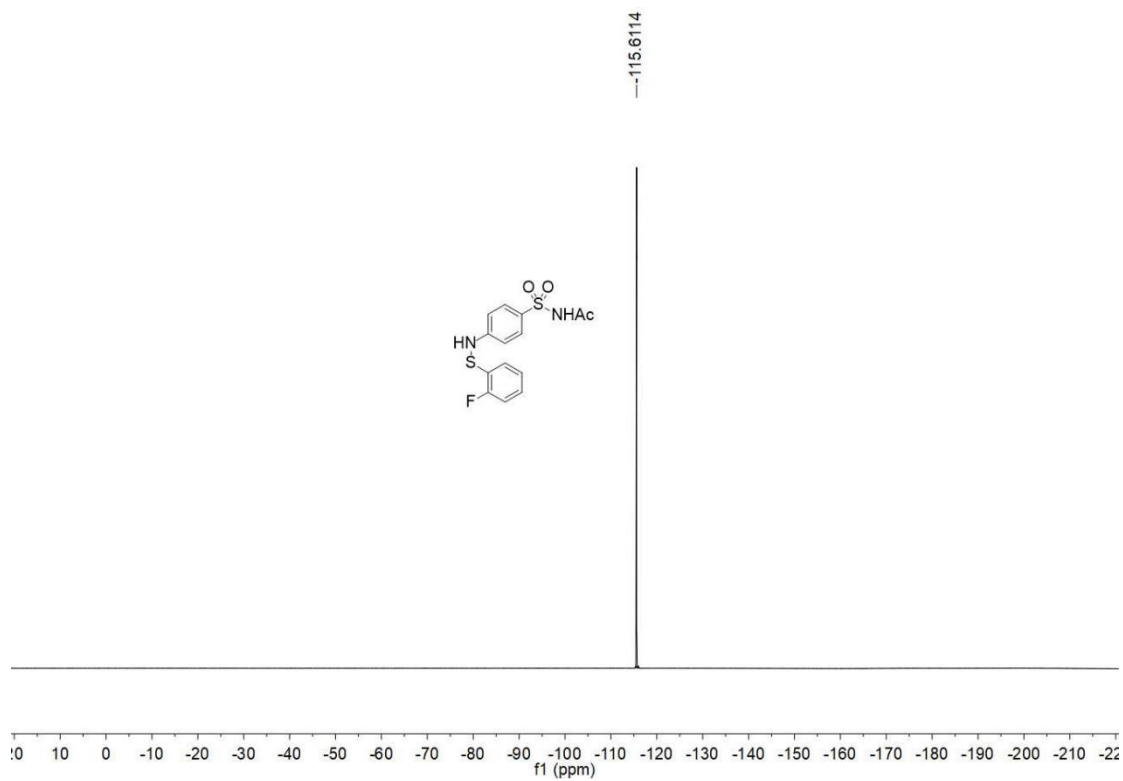


^1H NMR (400 MHz, $\text{DMSO-}d_6$) spectrum of Compound **4k**

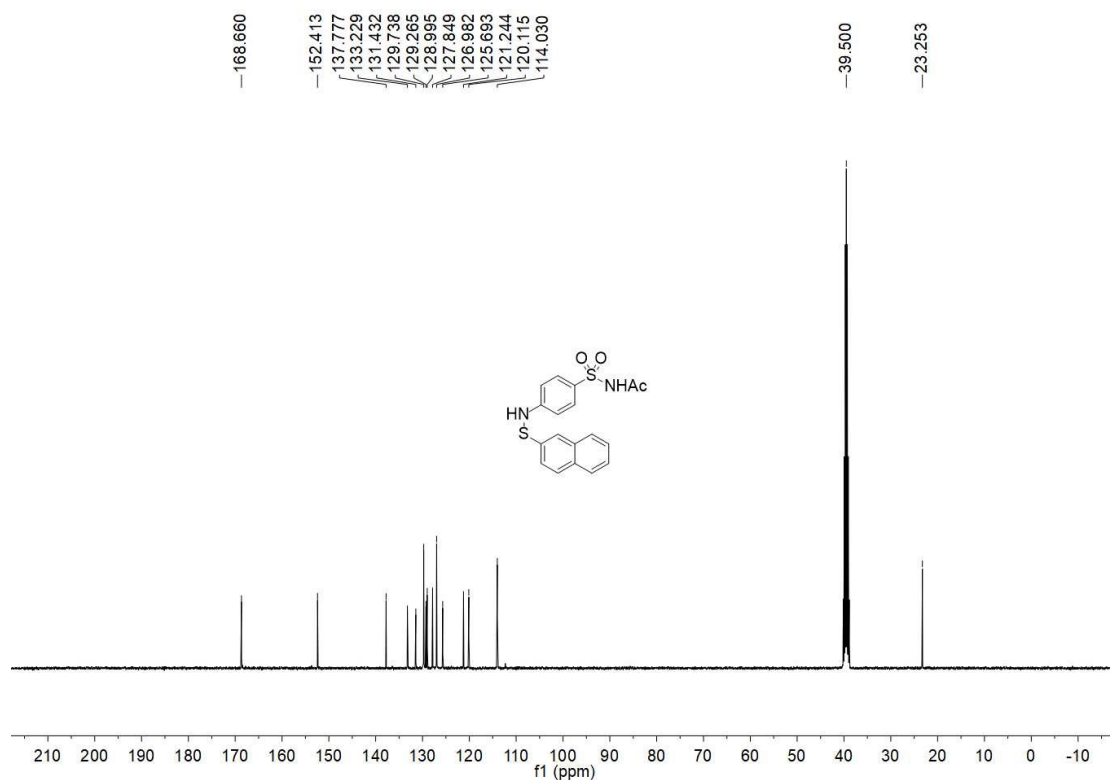


^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) spectrum of Compound **4k**

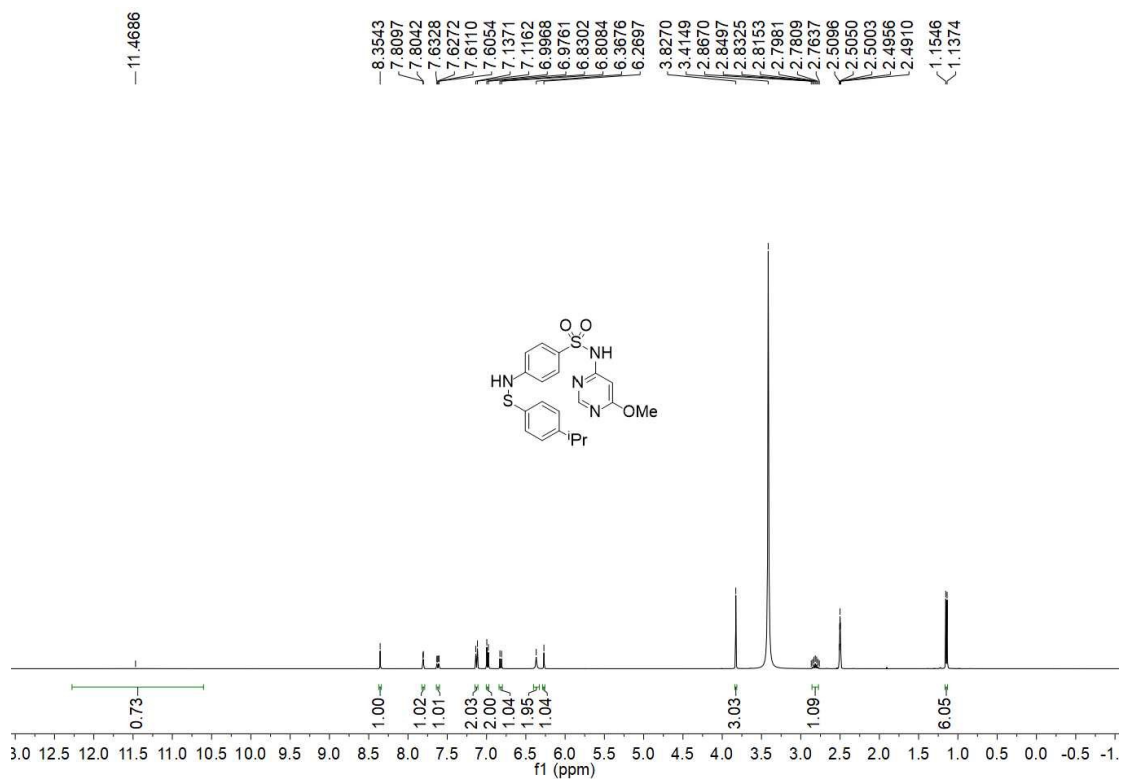




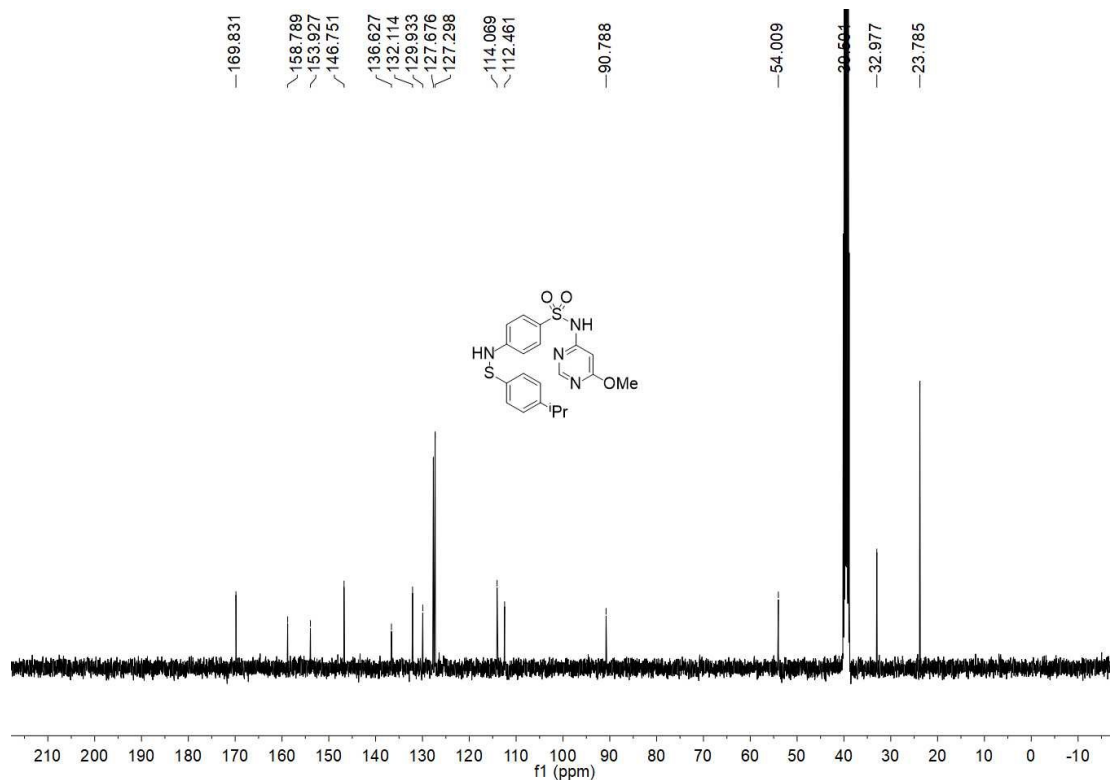
^1H NMR (400 MHz, $\text{DMSO-}d_6$) spectrum of Compound 4m



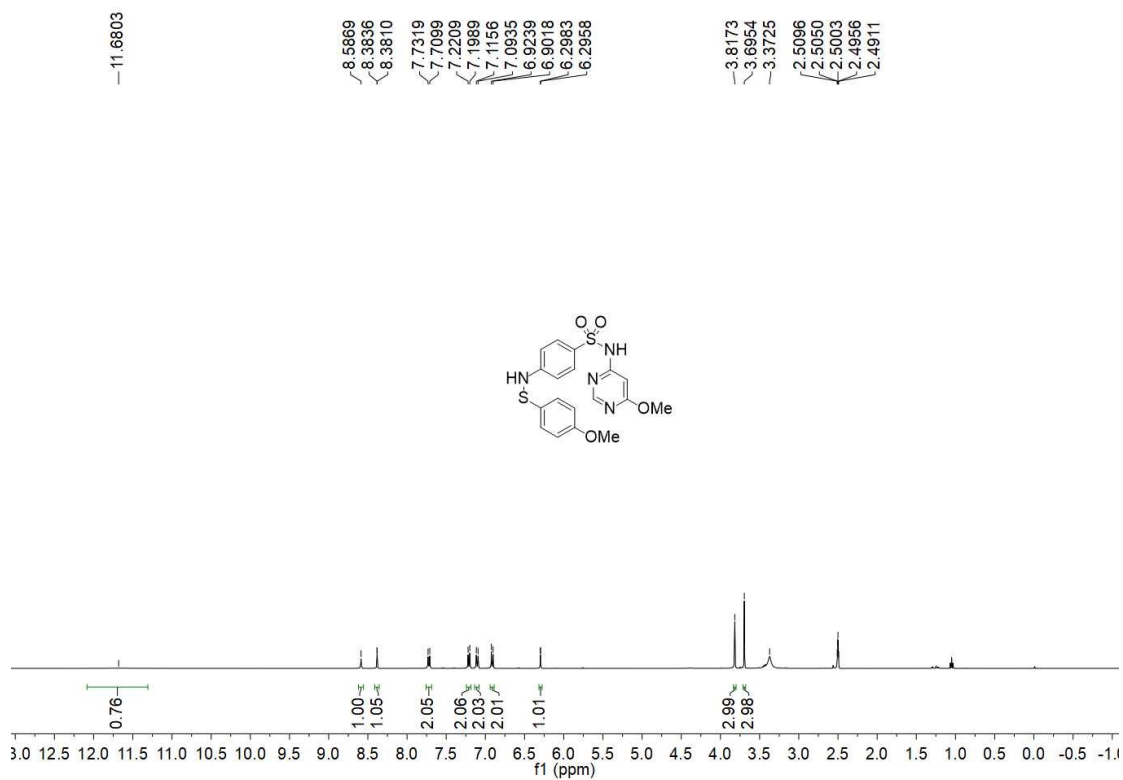
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 4m



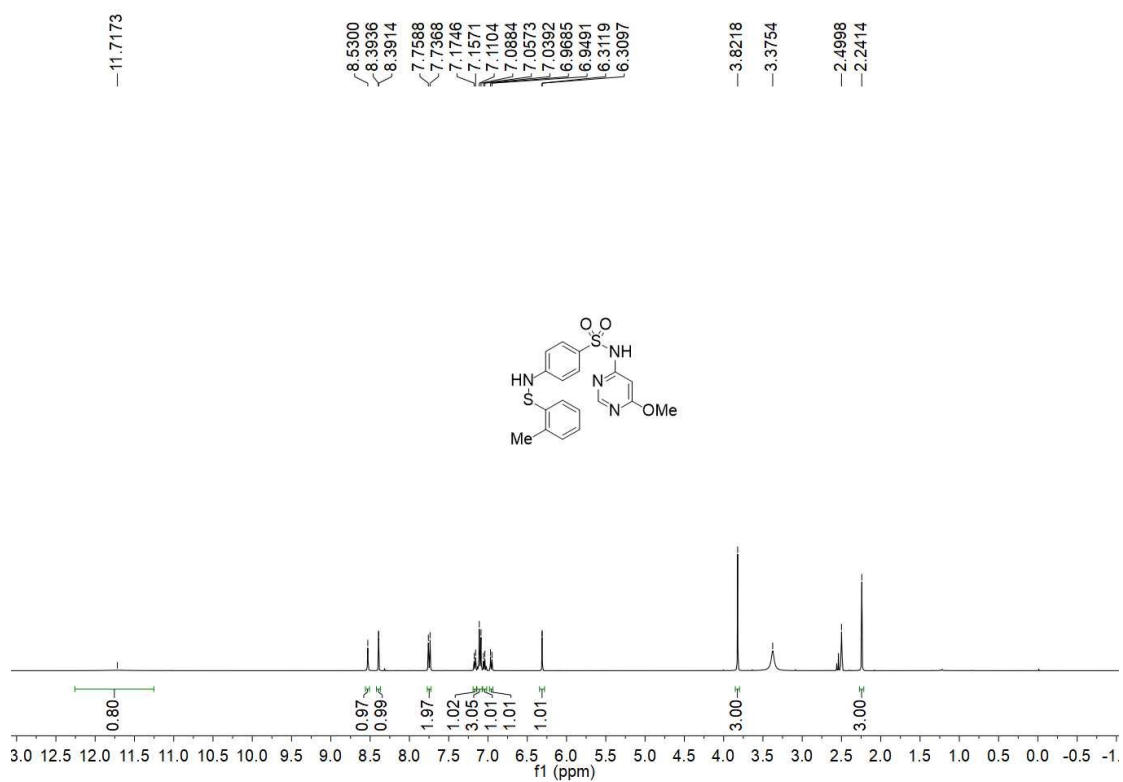
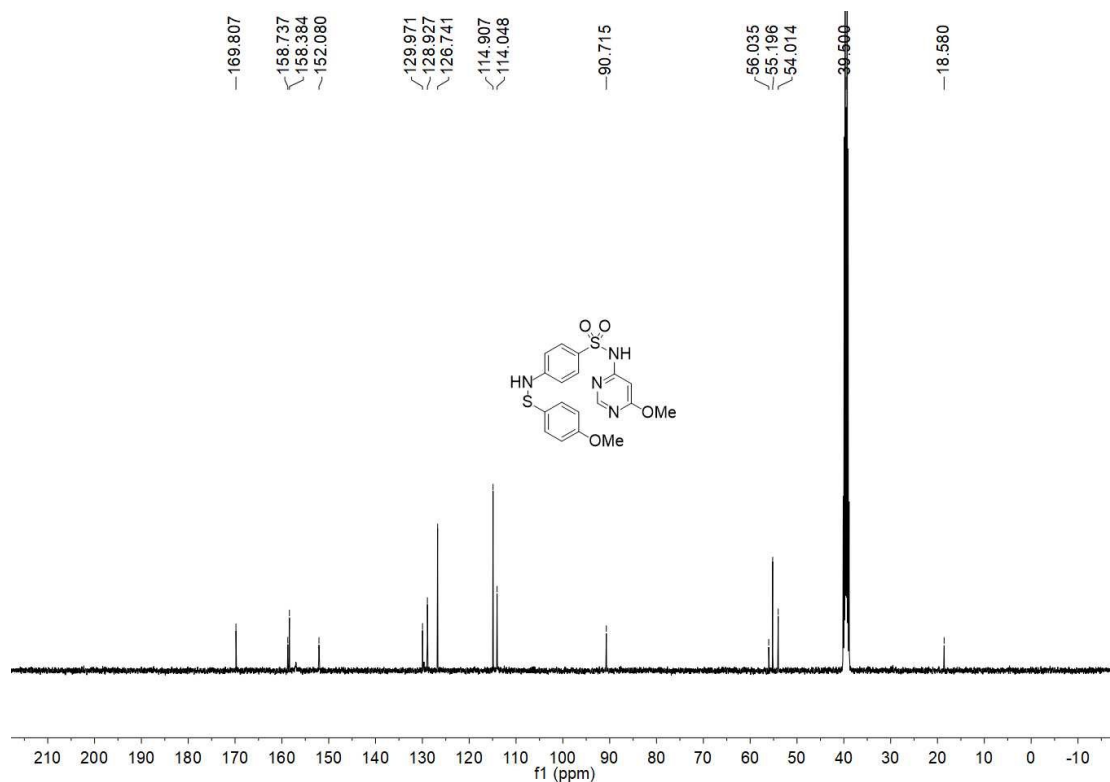
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 5a

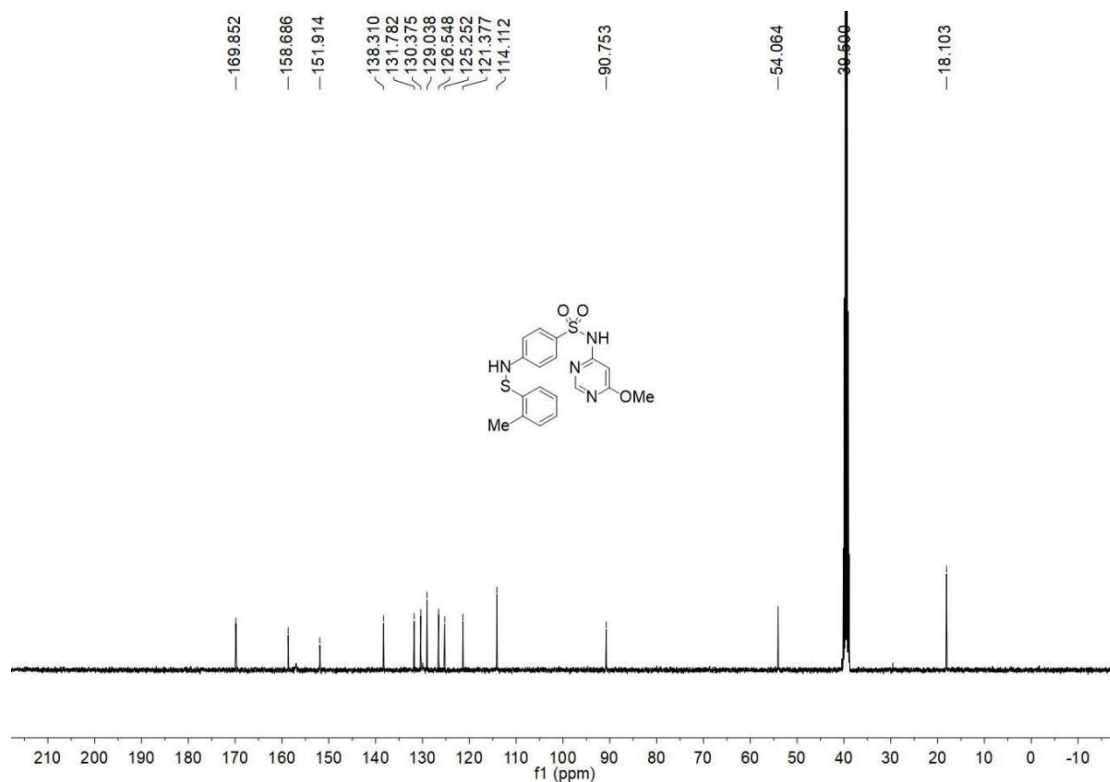


¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 5a

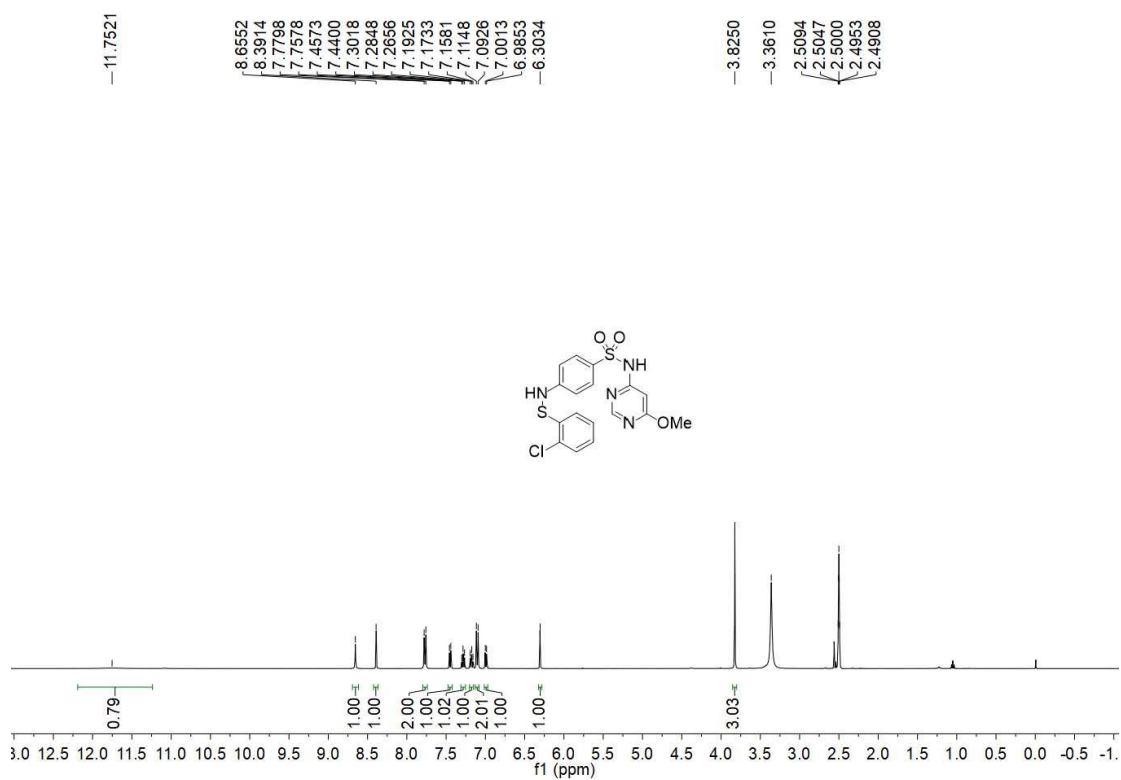


¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 5b

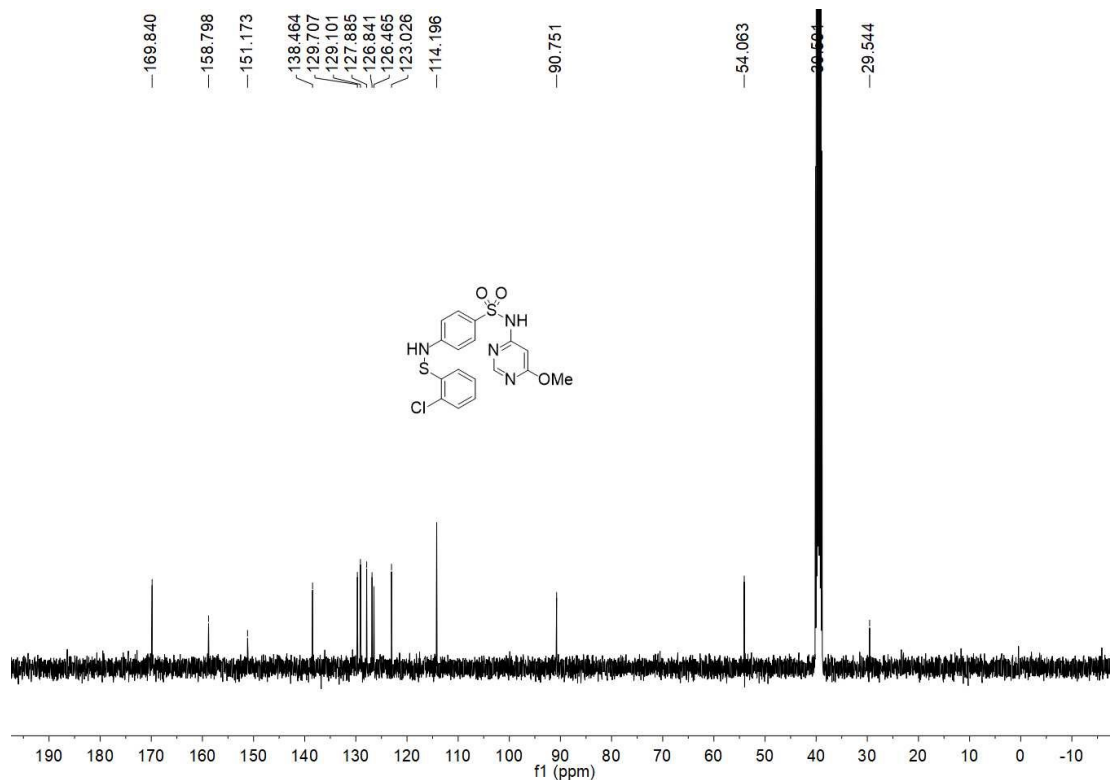




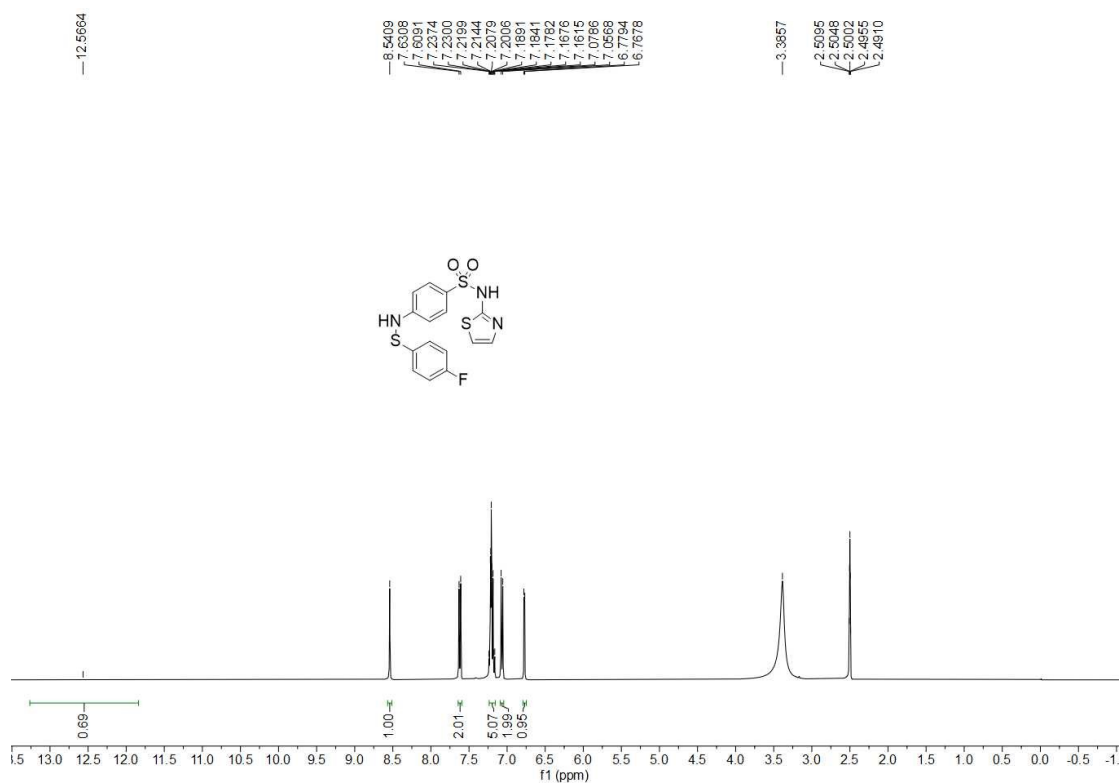
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 5c



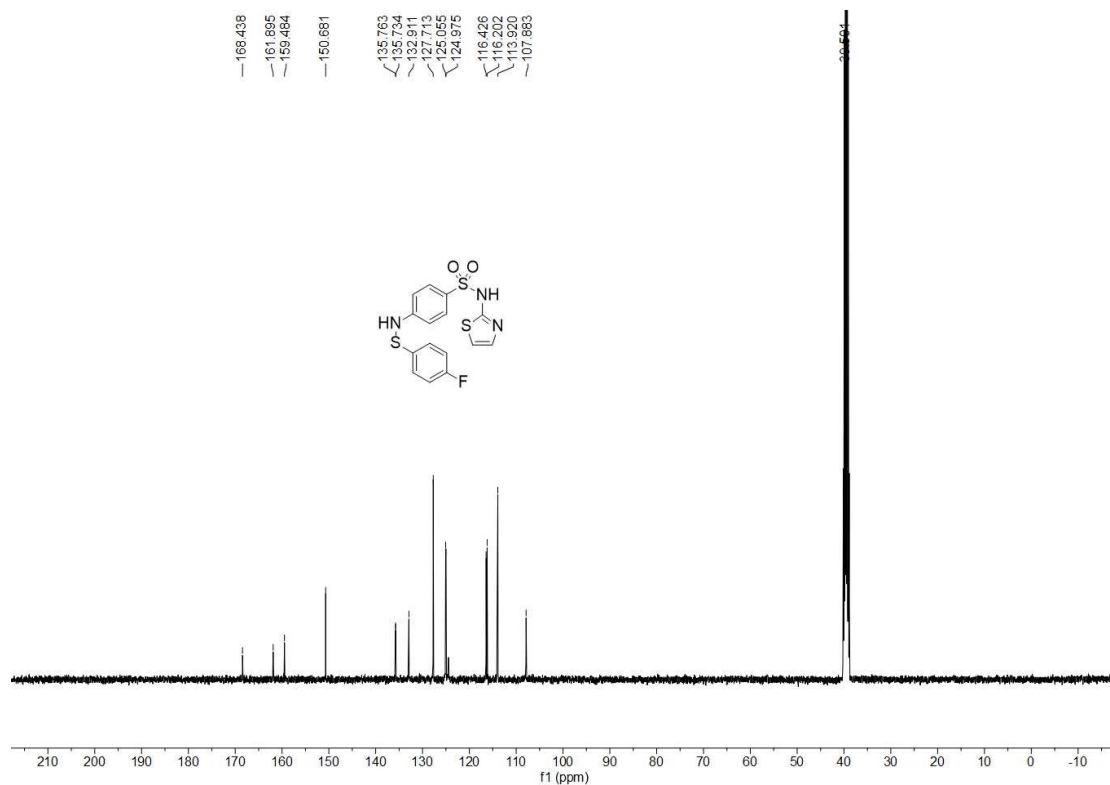
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 5d



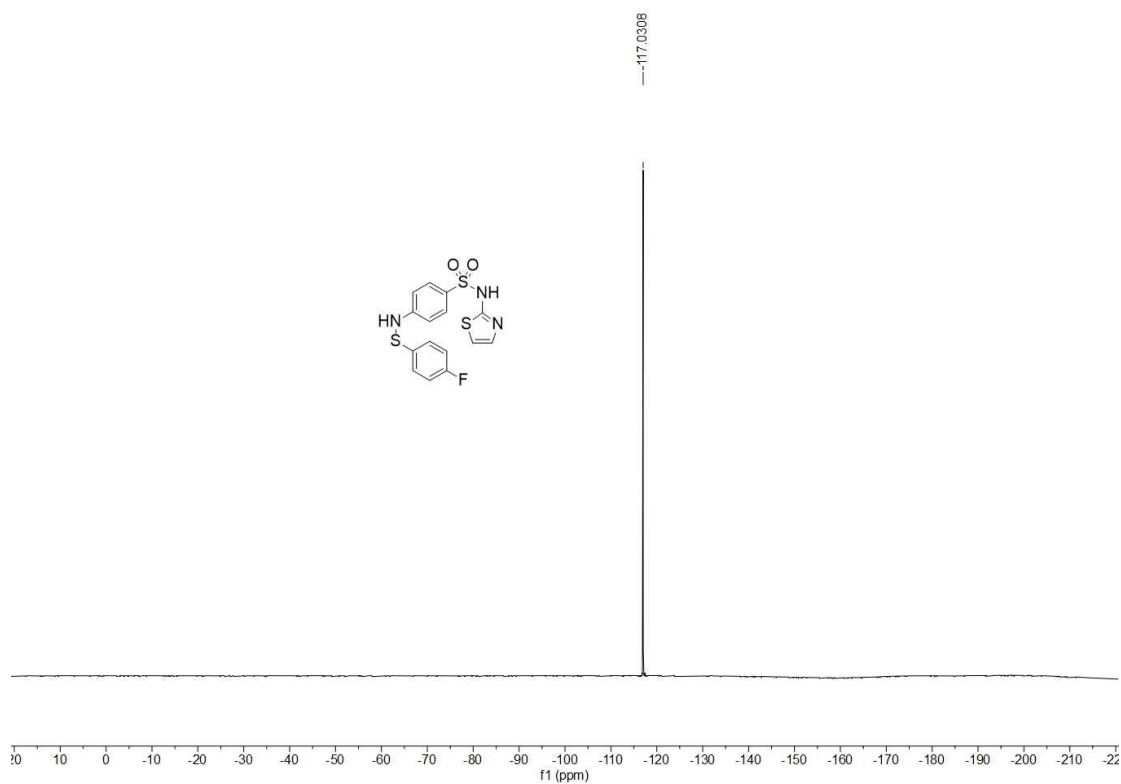
^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) spectrum of Compound **5d**



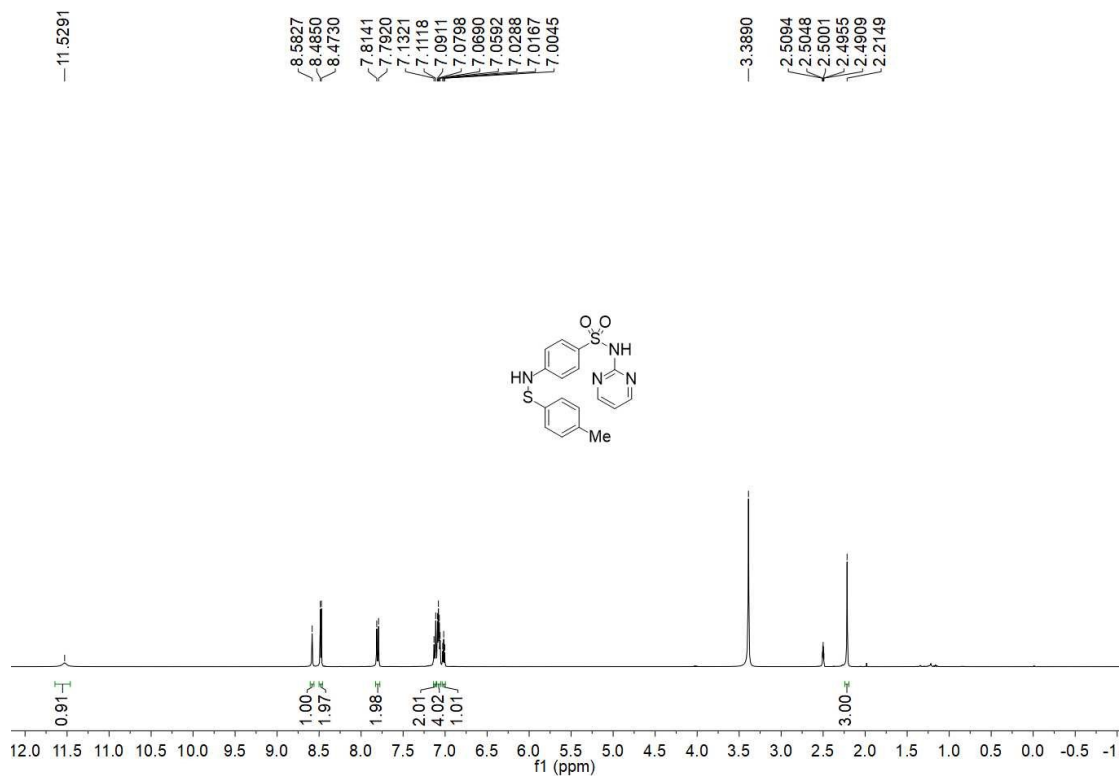
^1H NMR (400 MHz, $\text{DMSO-}d_6$) spectrum of Compound **5e**



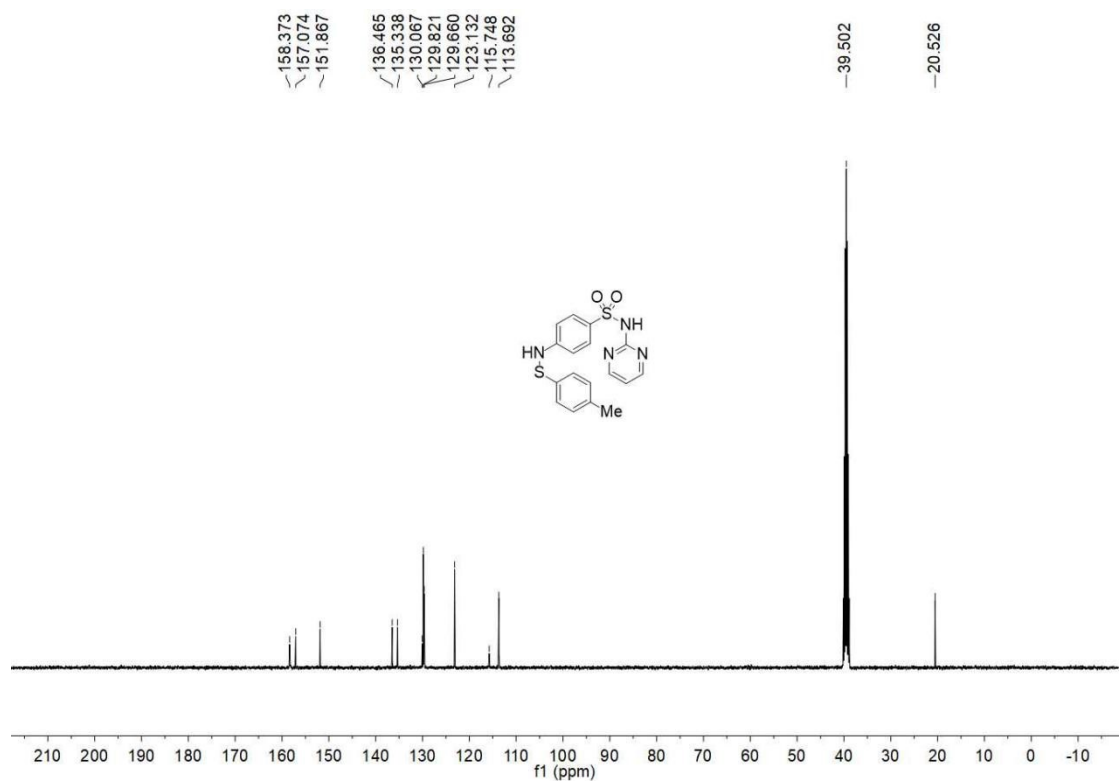
^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) spectrum of Compound 5e



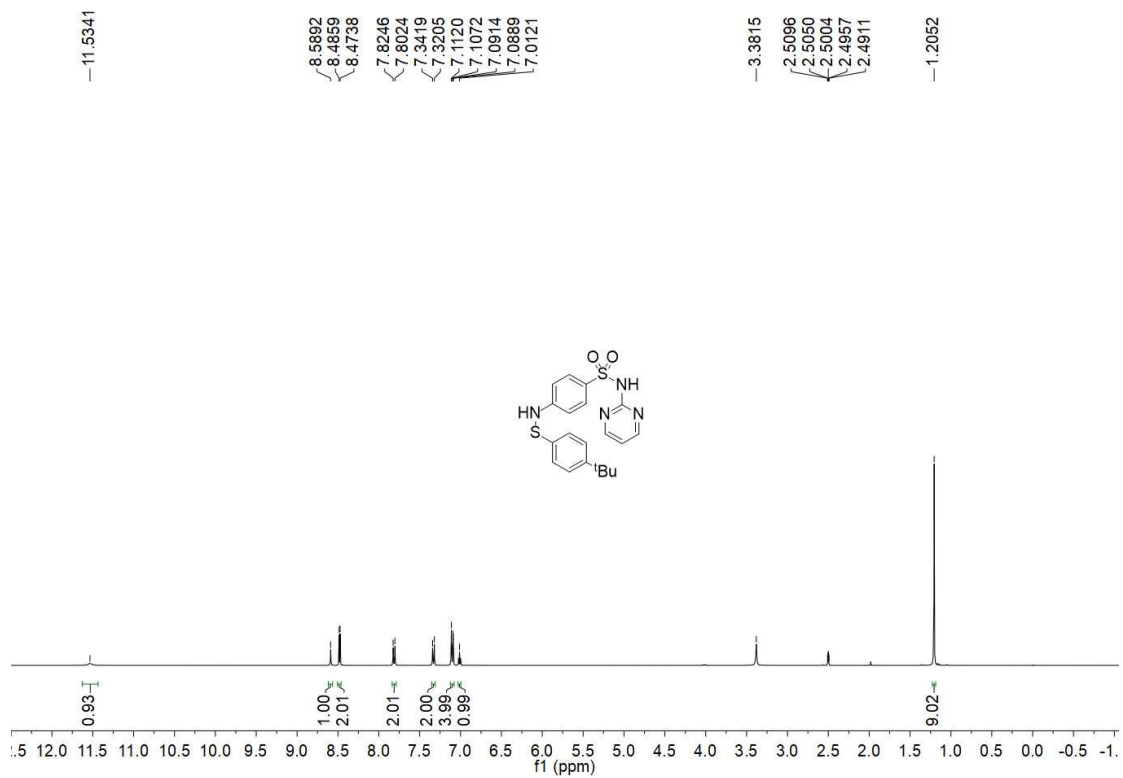
^{19}F NMR (376 MHz, $\text{DMSO-}d_6$) spectrum of Compound 5e



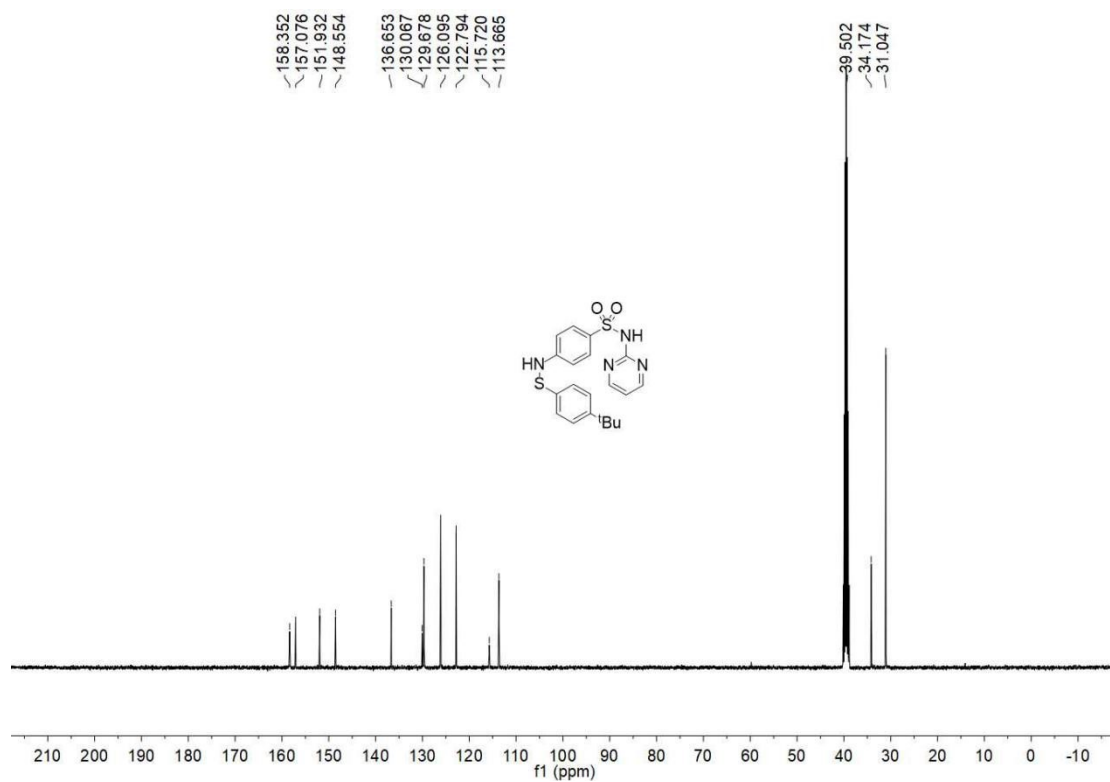
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 5f



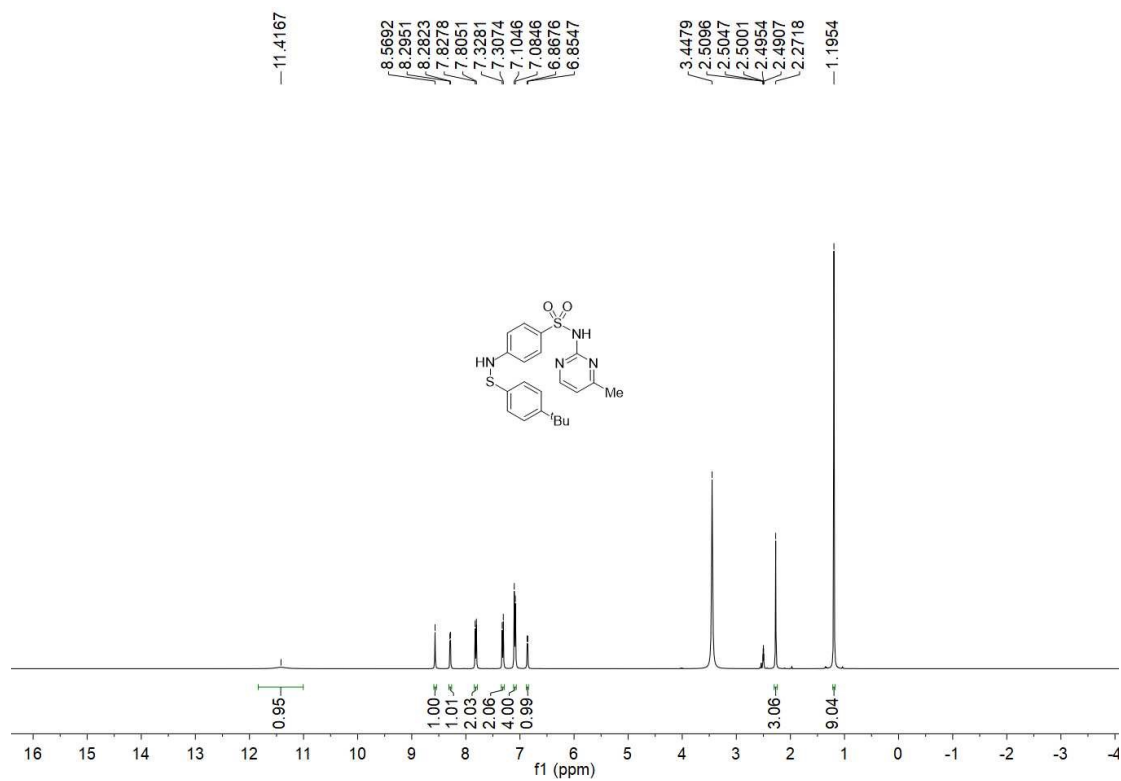
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 5f



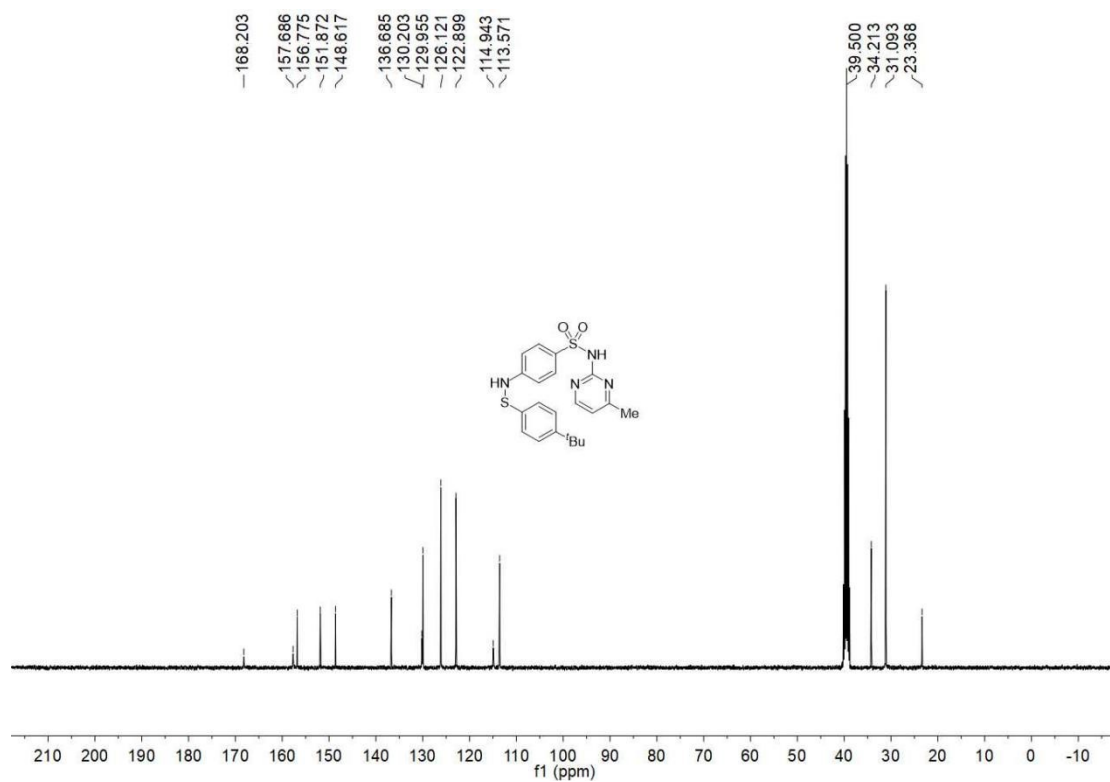
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound **5g**



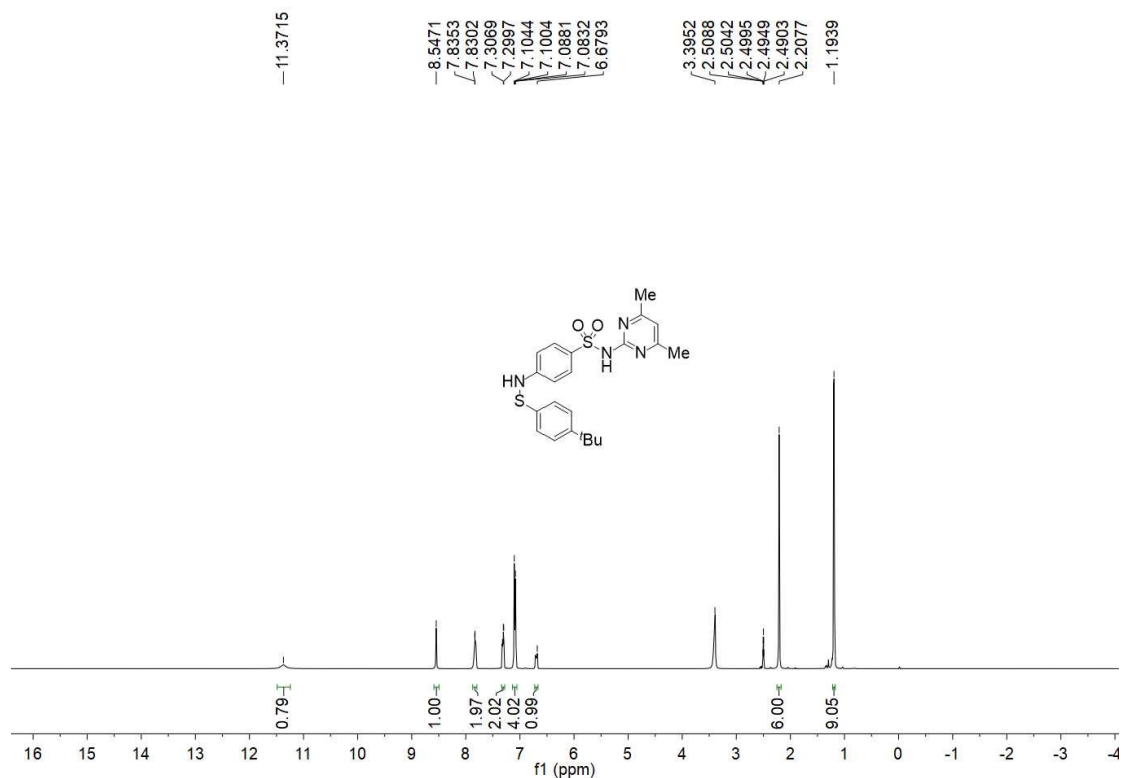
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound **5g**



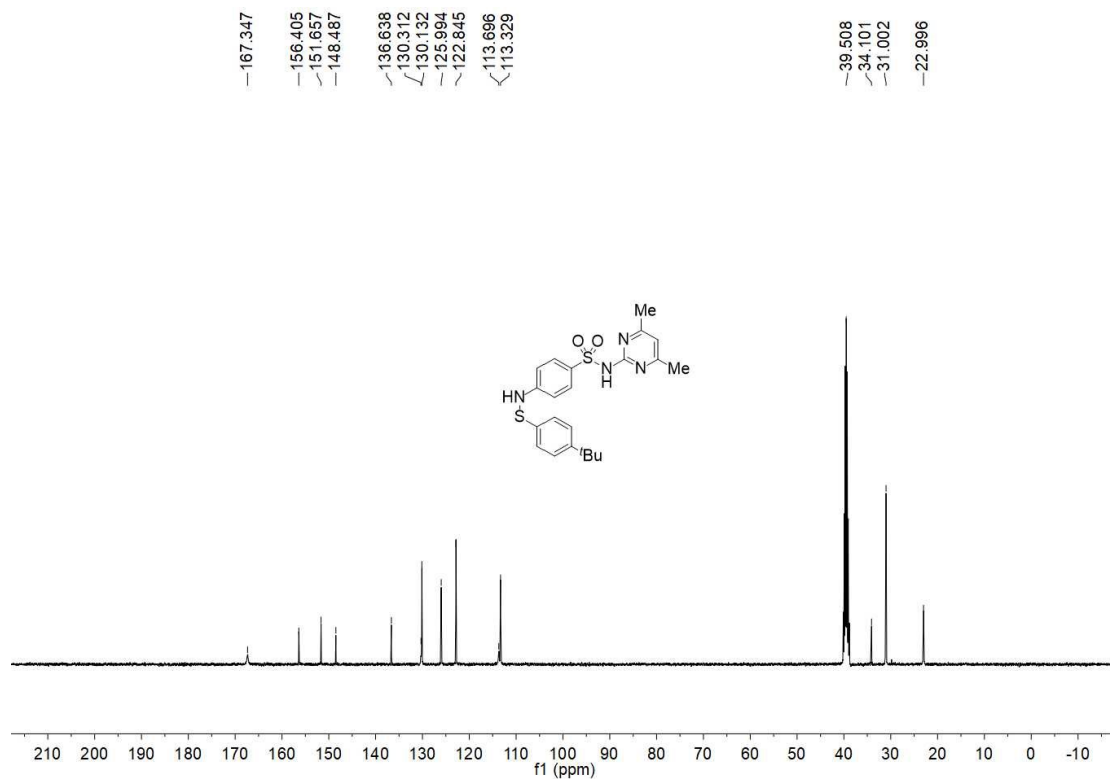
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 5h



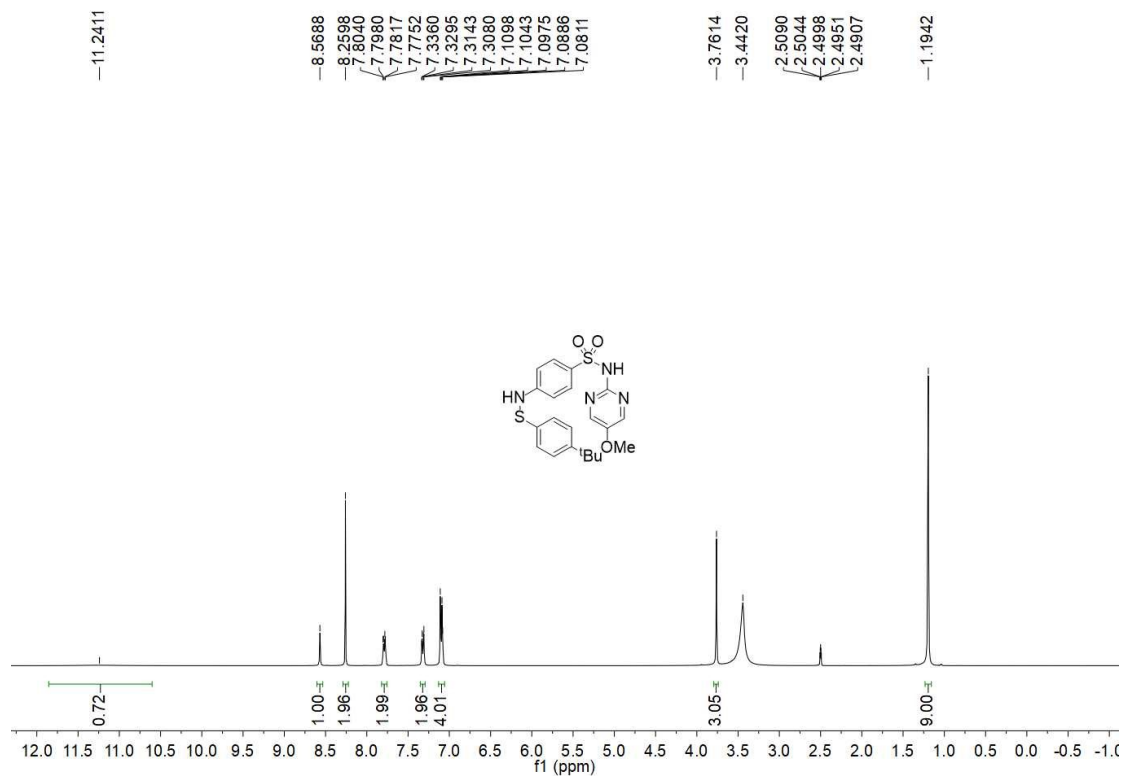
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 5h



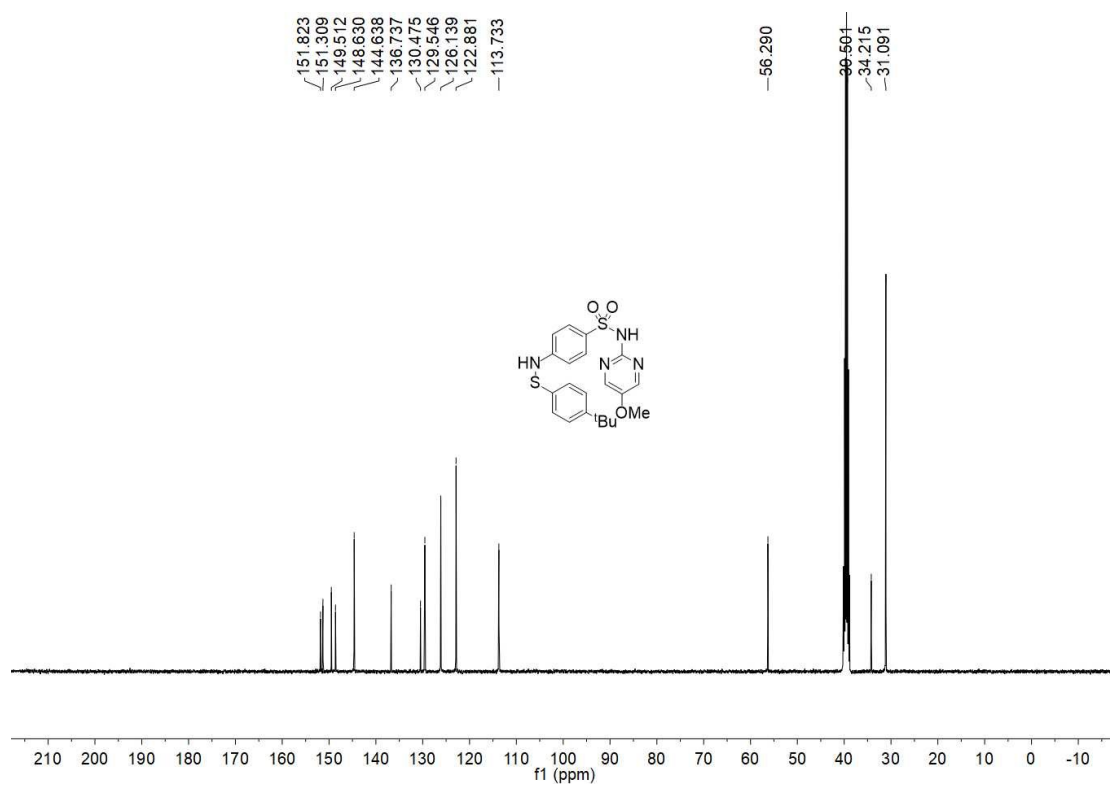
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound **5i**



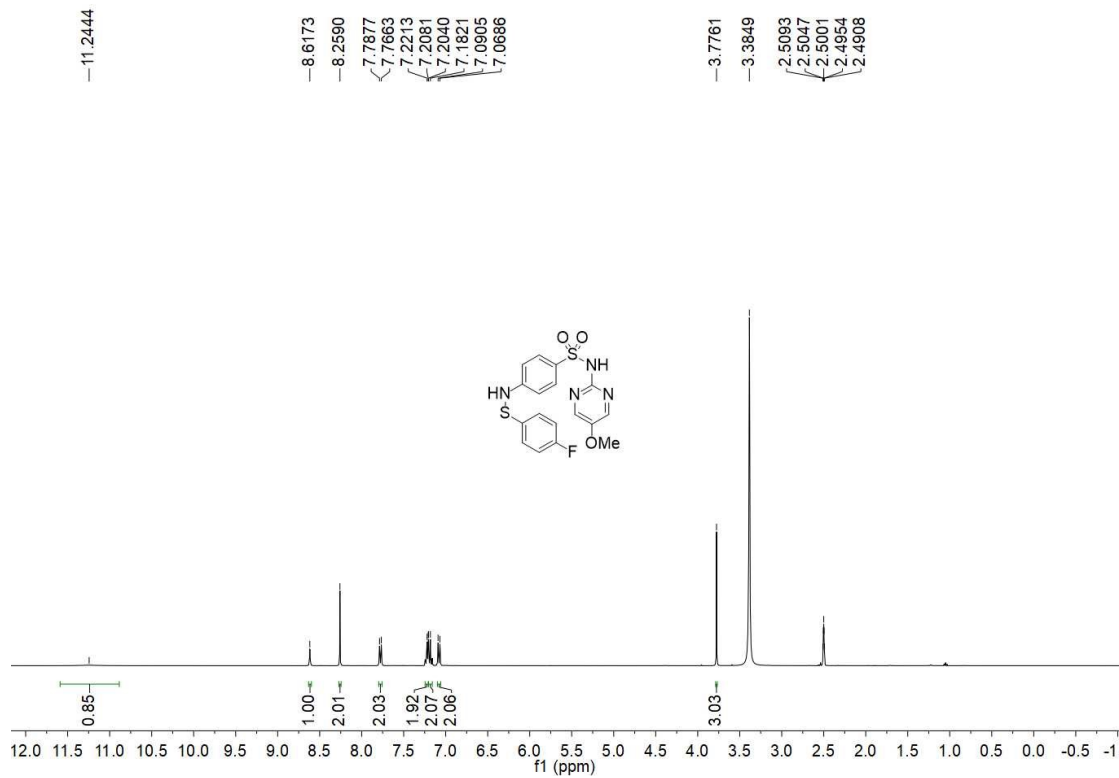
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound **5i**



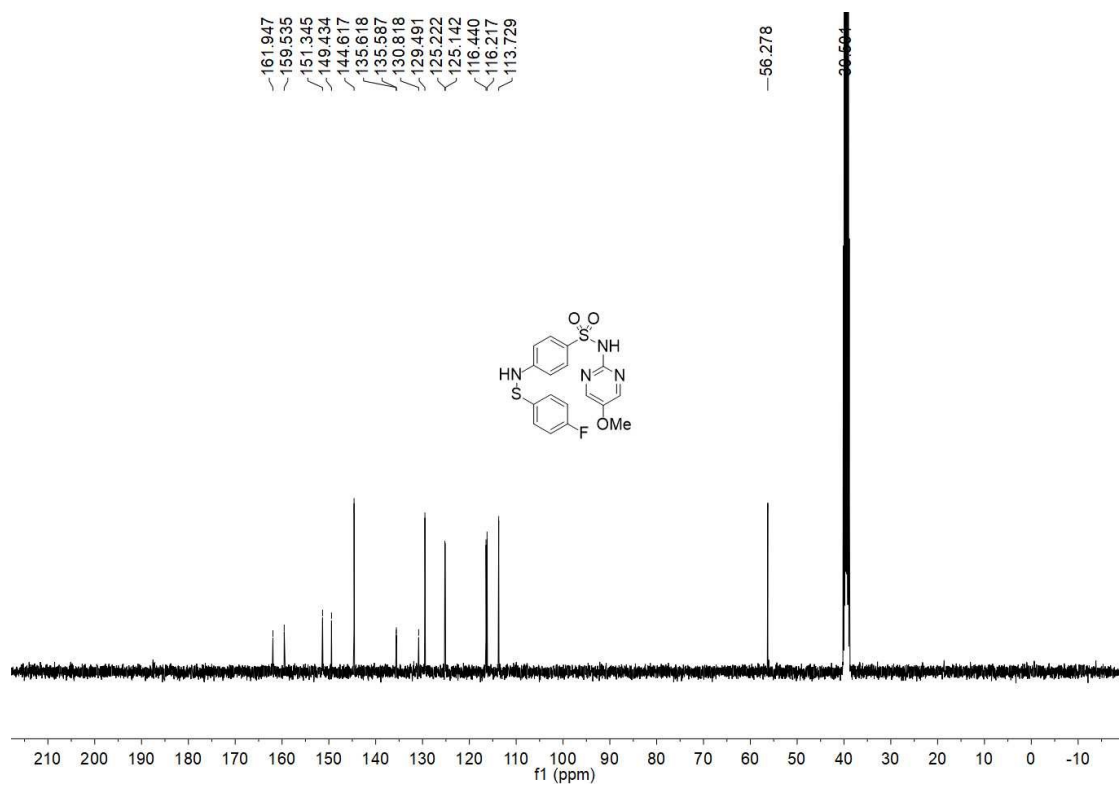
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound **5j**



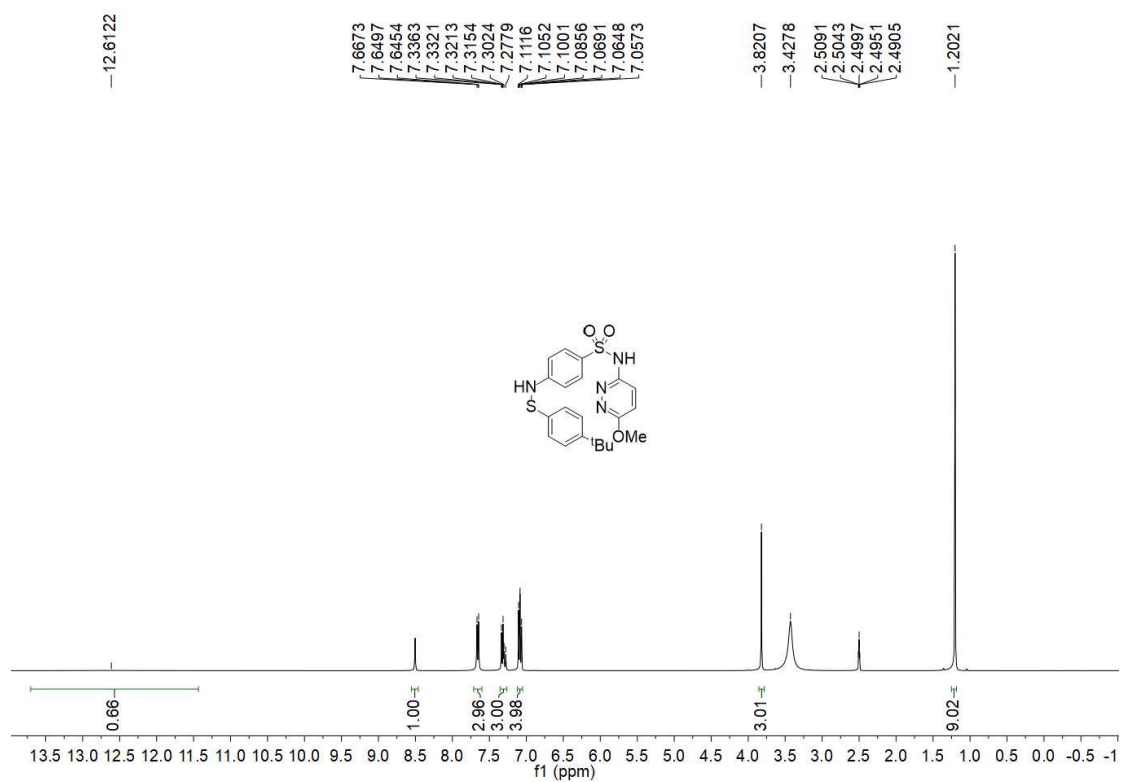
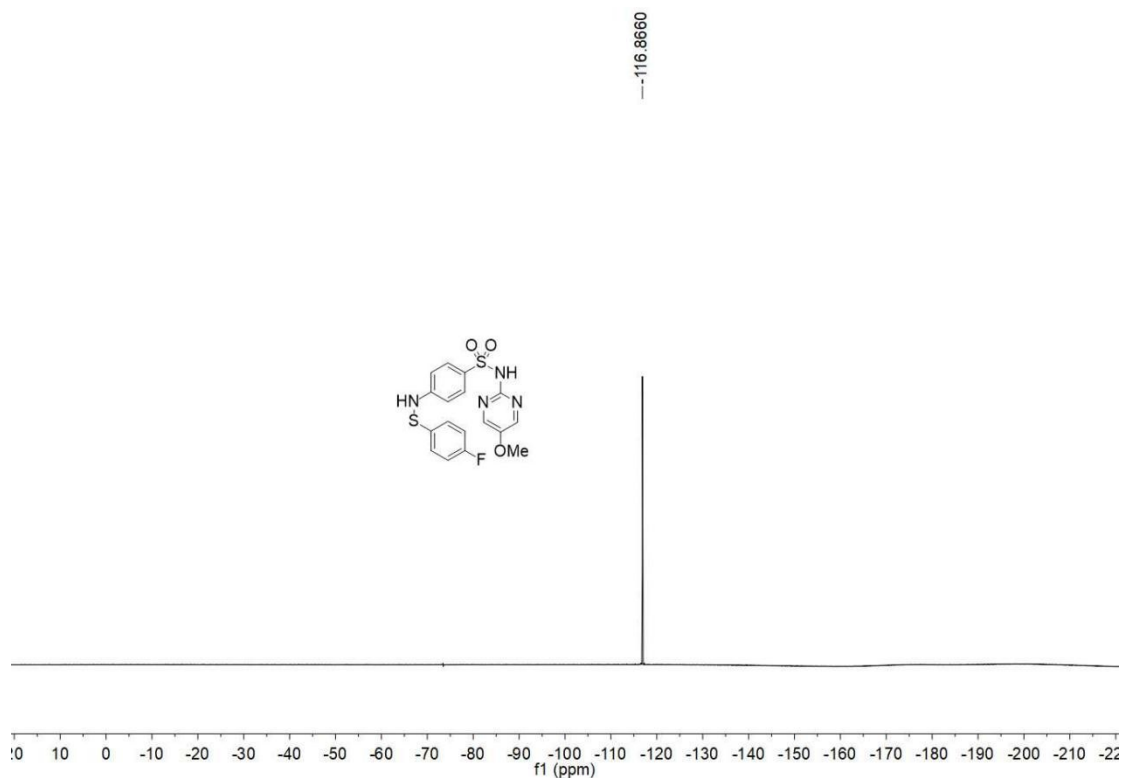
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound **5j**



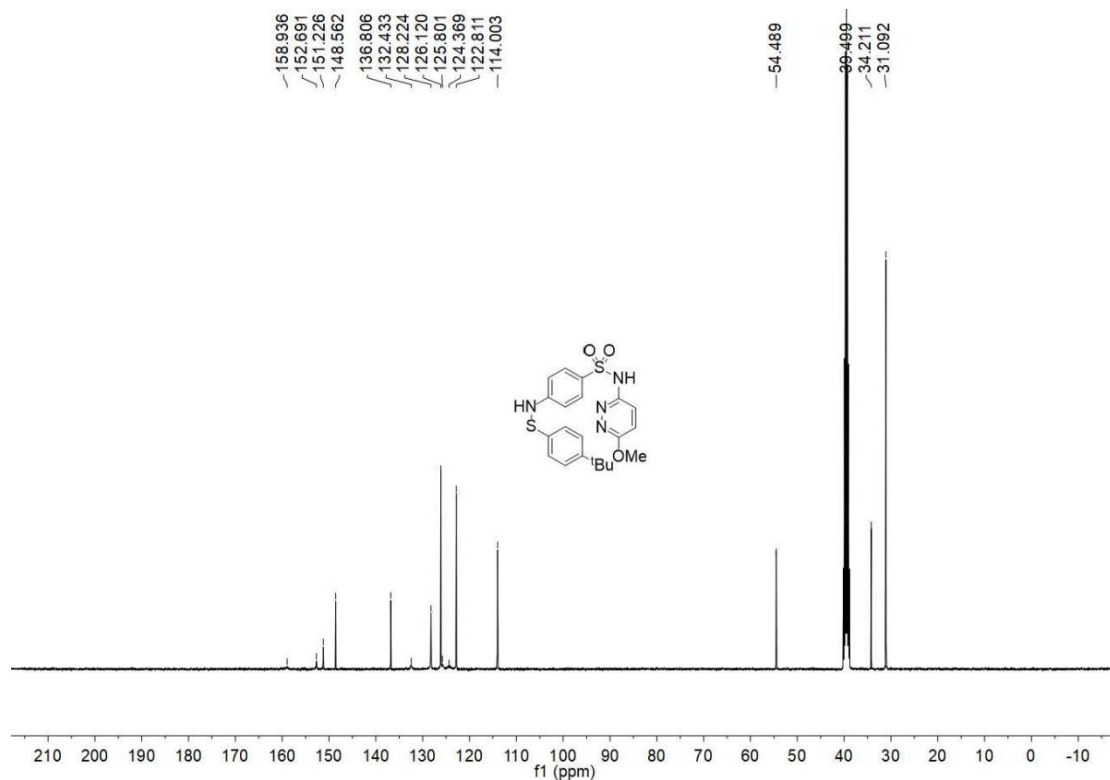
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 5k



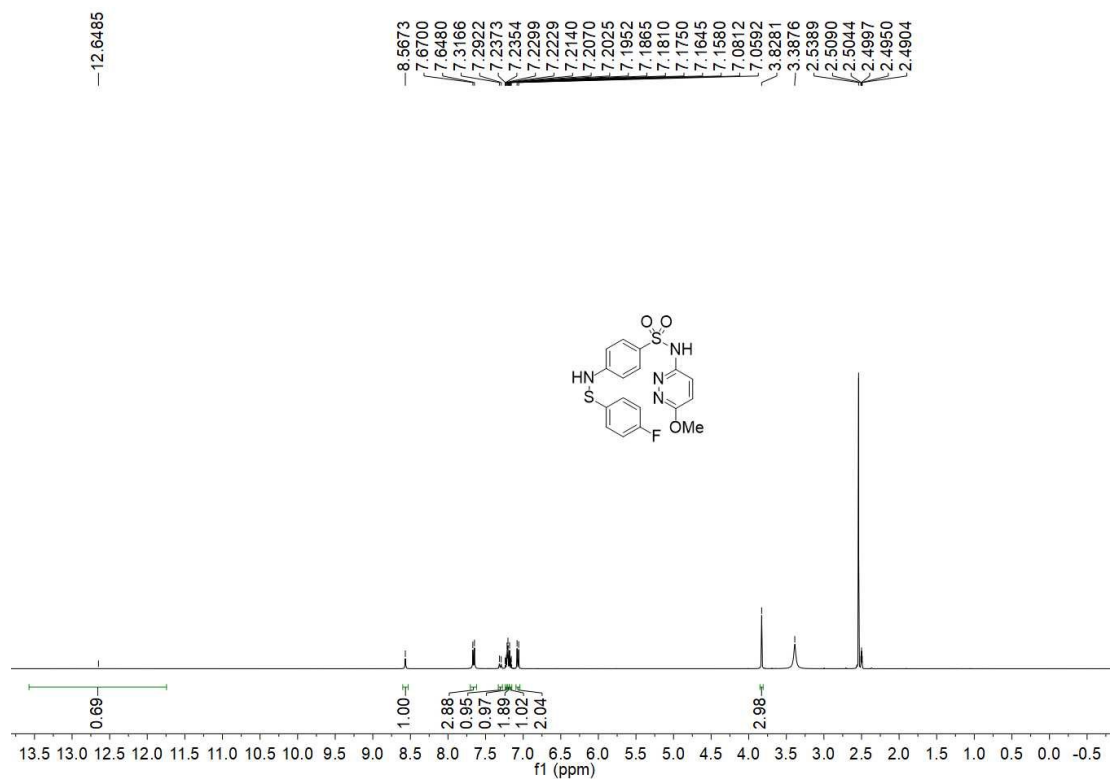
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 5k



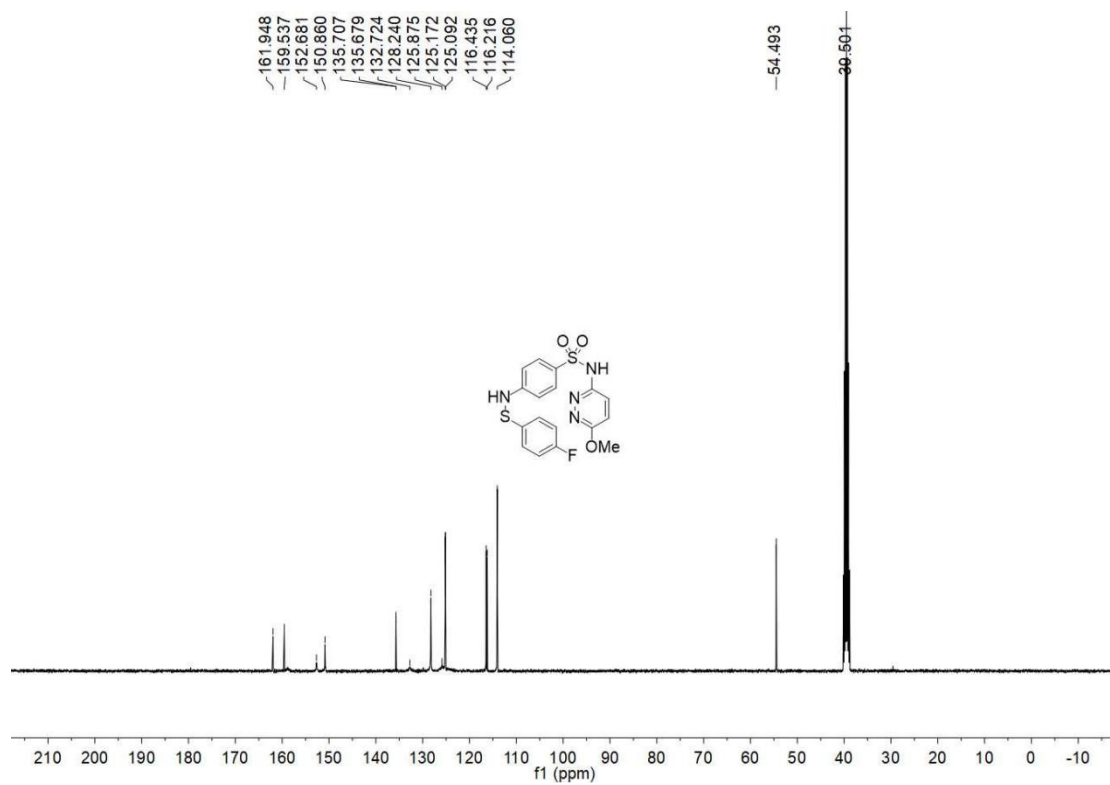
^1H NMR (400 MHz, $\text{DMSO-}d_6$) spectrum of Compound 5l



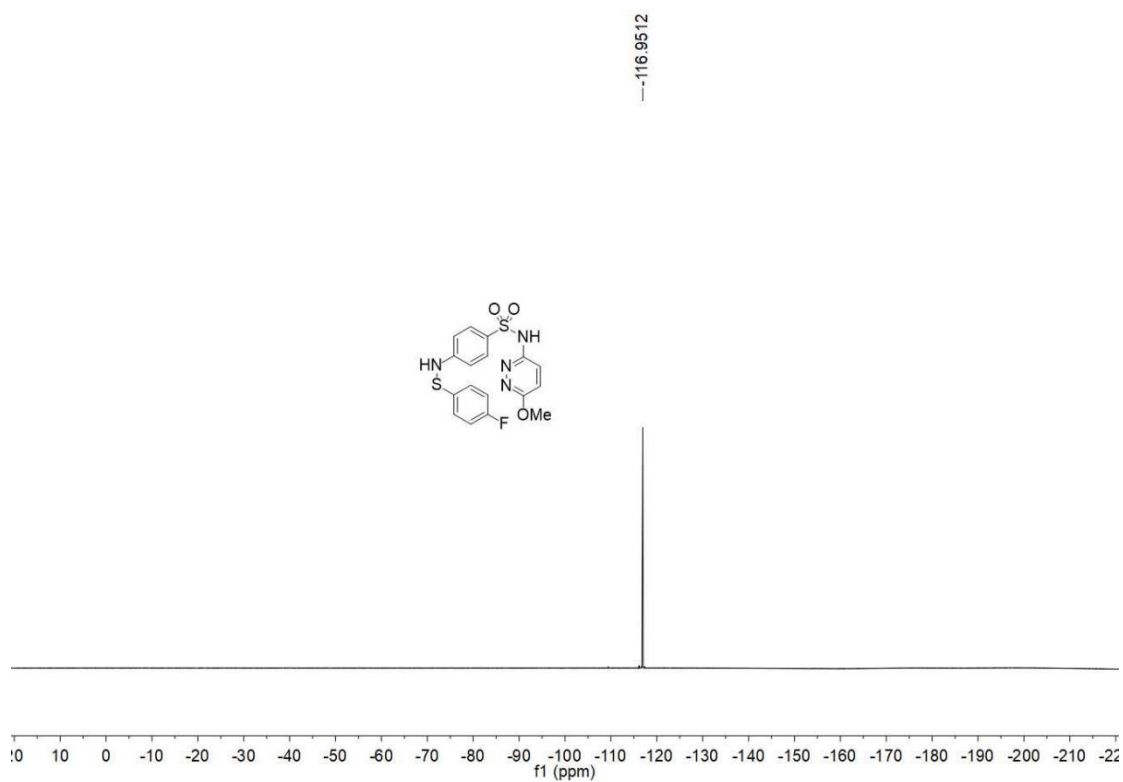
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 5l



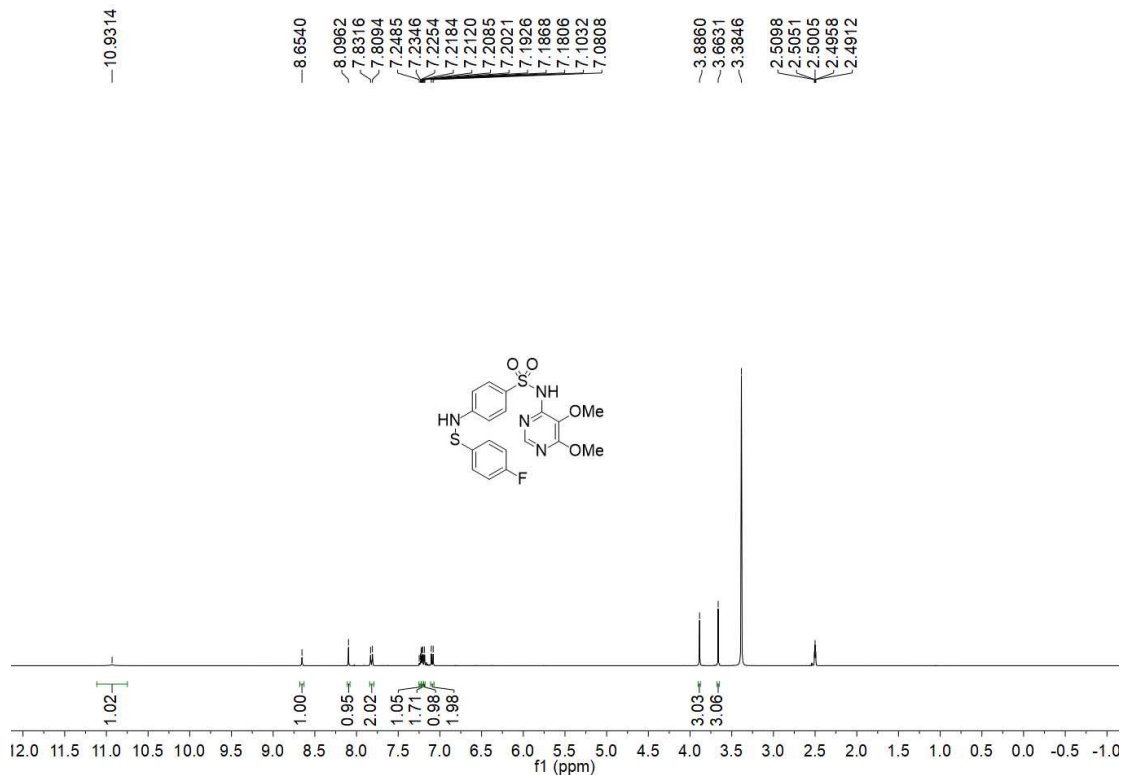
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 5m



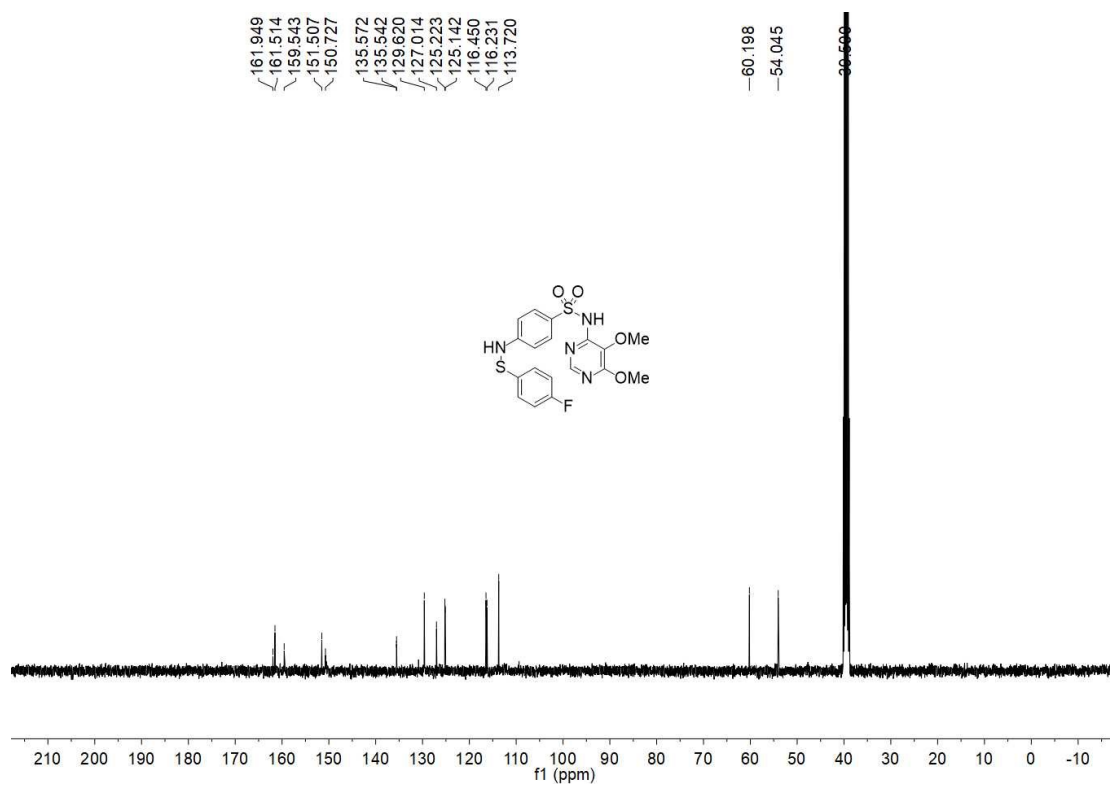
^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) spectrum of Compound **5m**



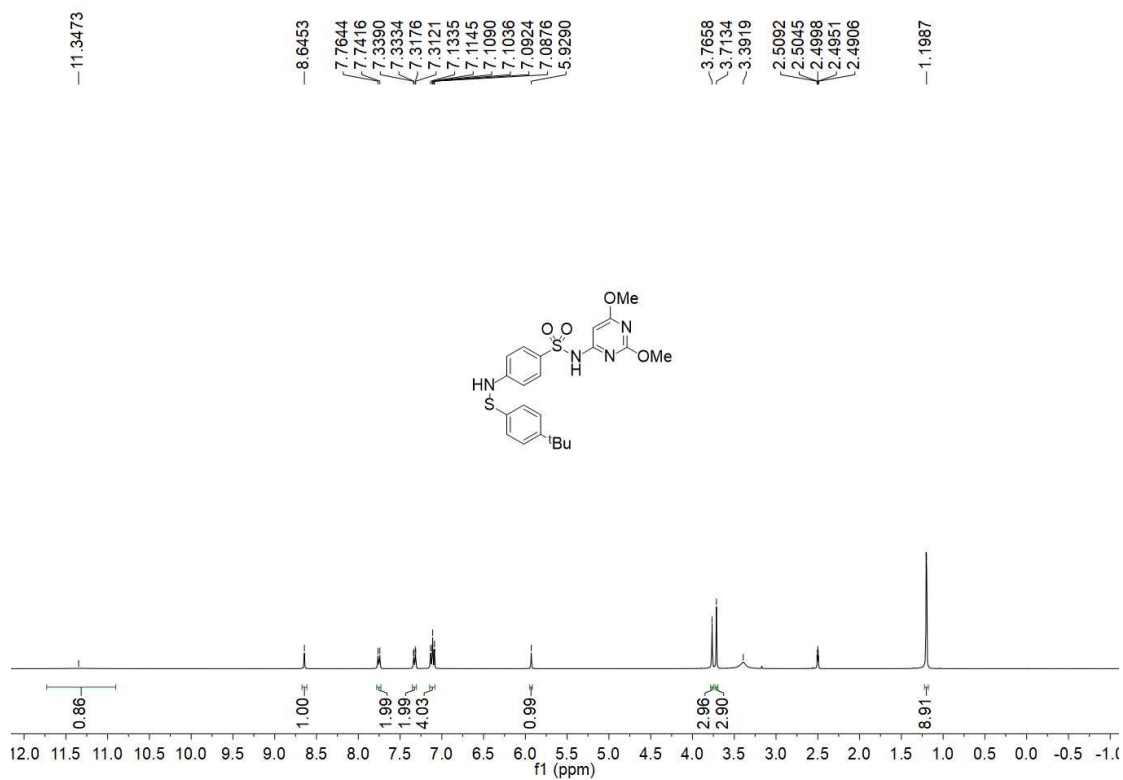
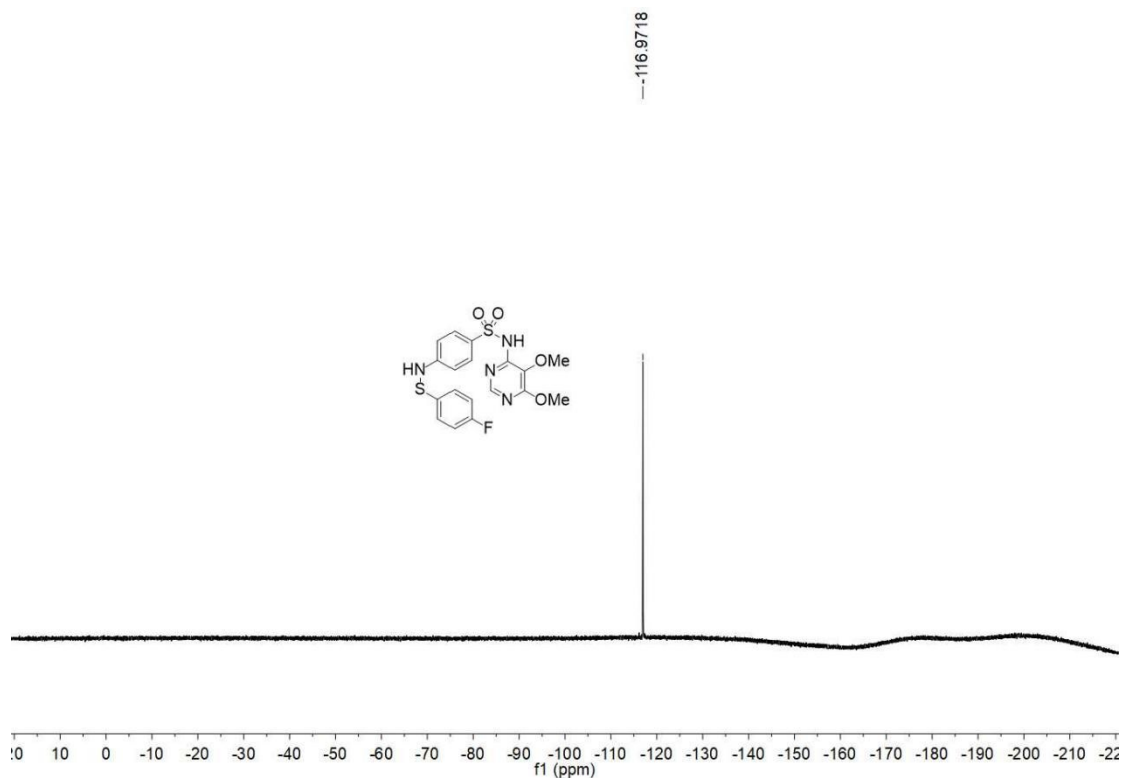
^{19}F NMR (376 MHz, $\text{DMSO-}d_6$) spectrum of Compound **5m**

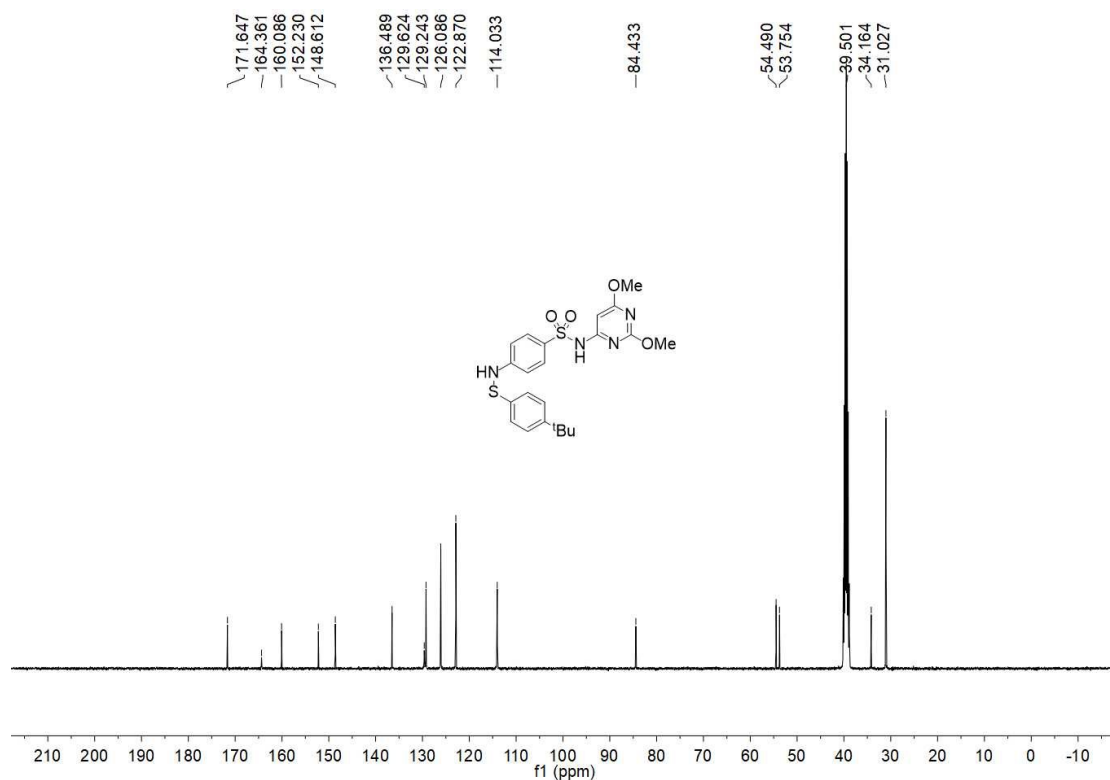


^1H NMR (400 MHz, $\text{DMSO-}d_6$) spectrum of Compound 5n

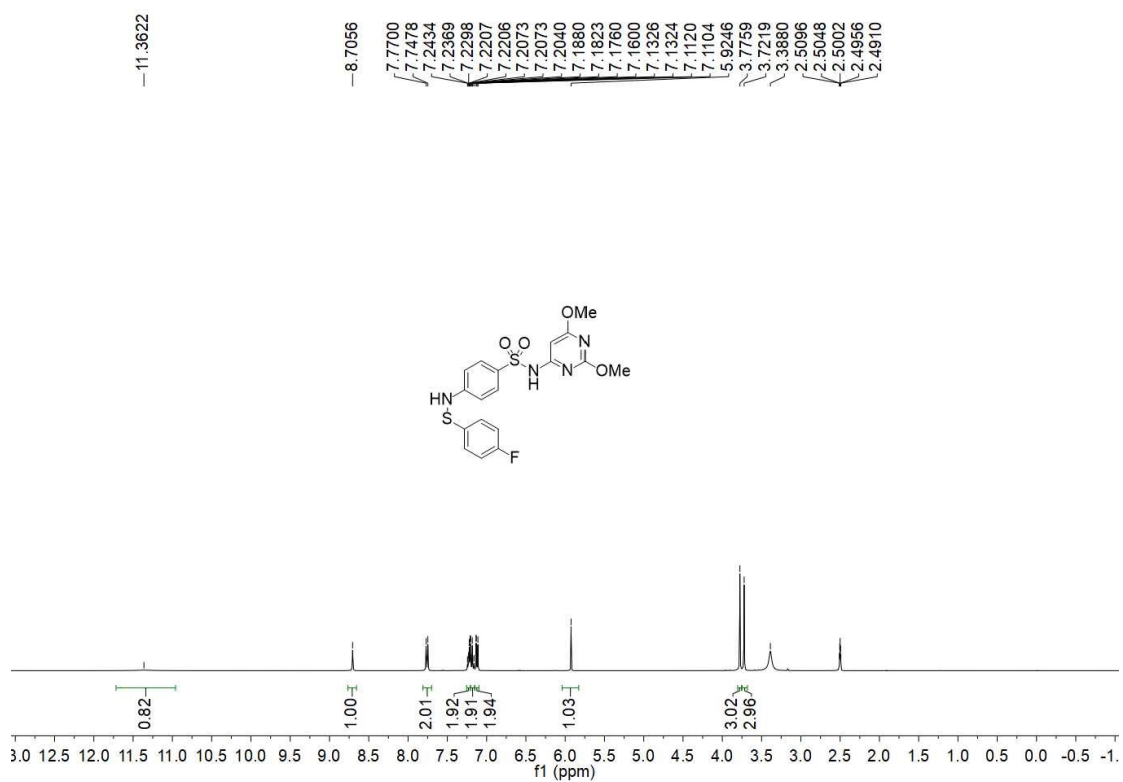


^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) spectrum of Compound 5n

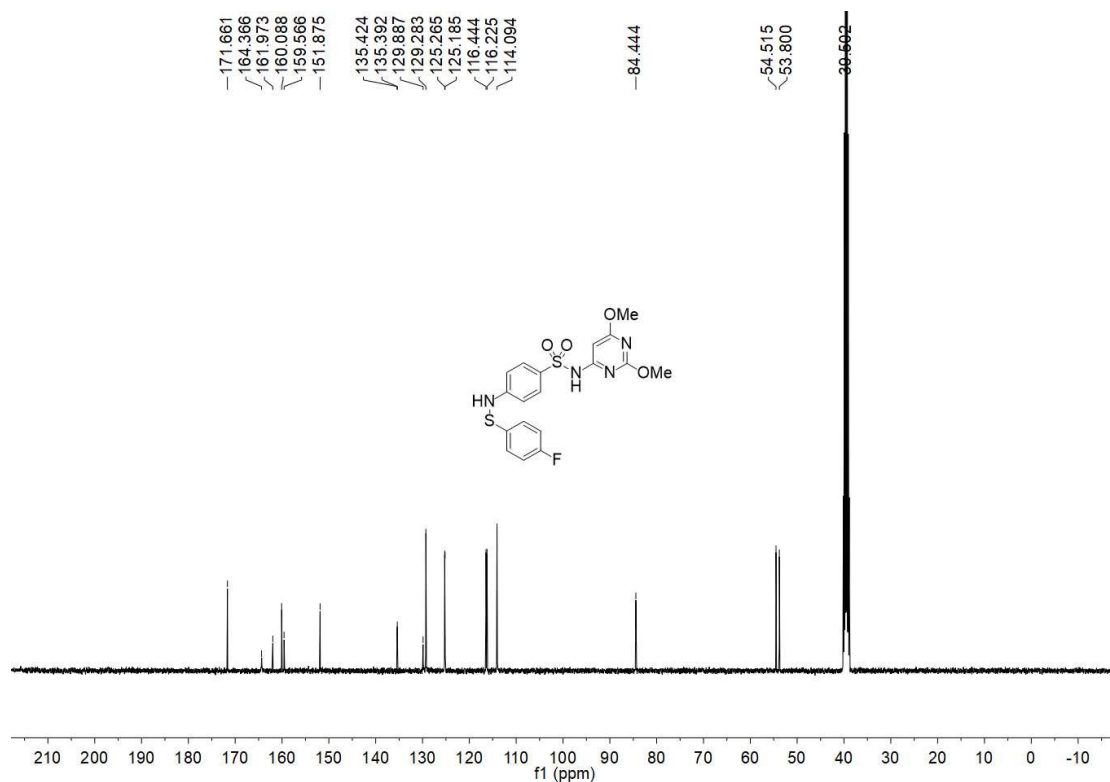




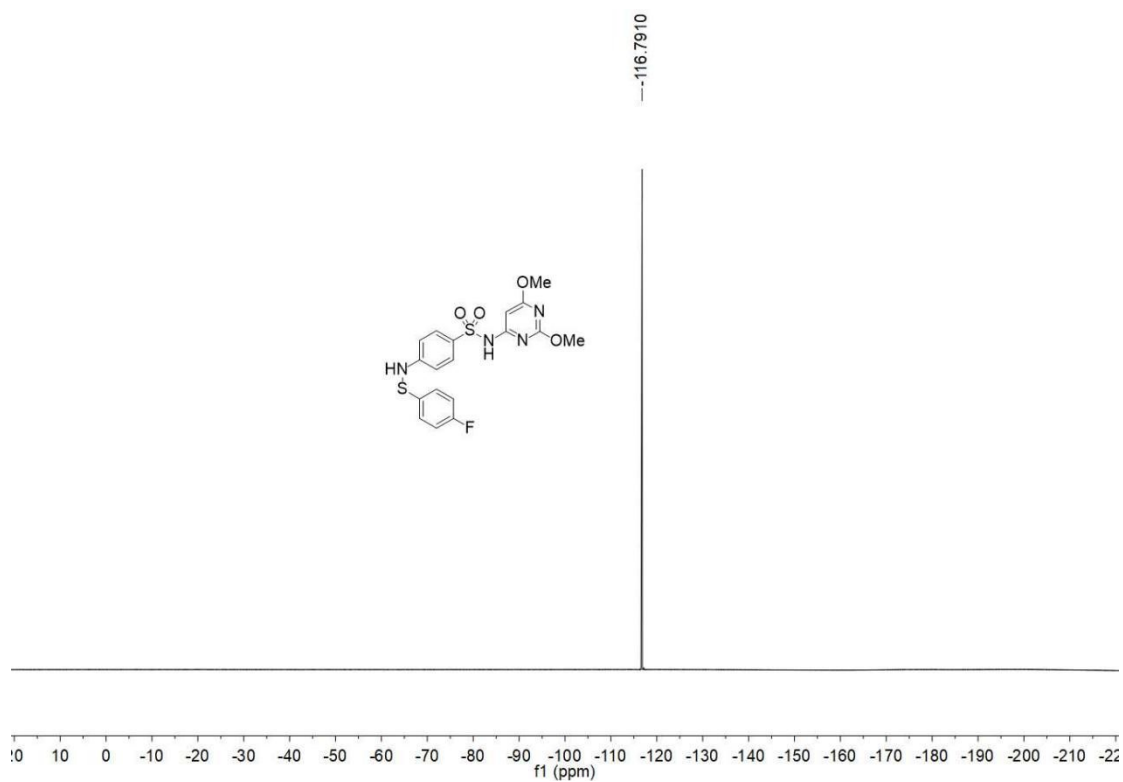
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 5o



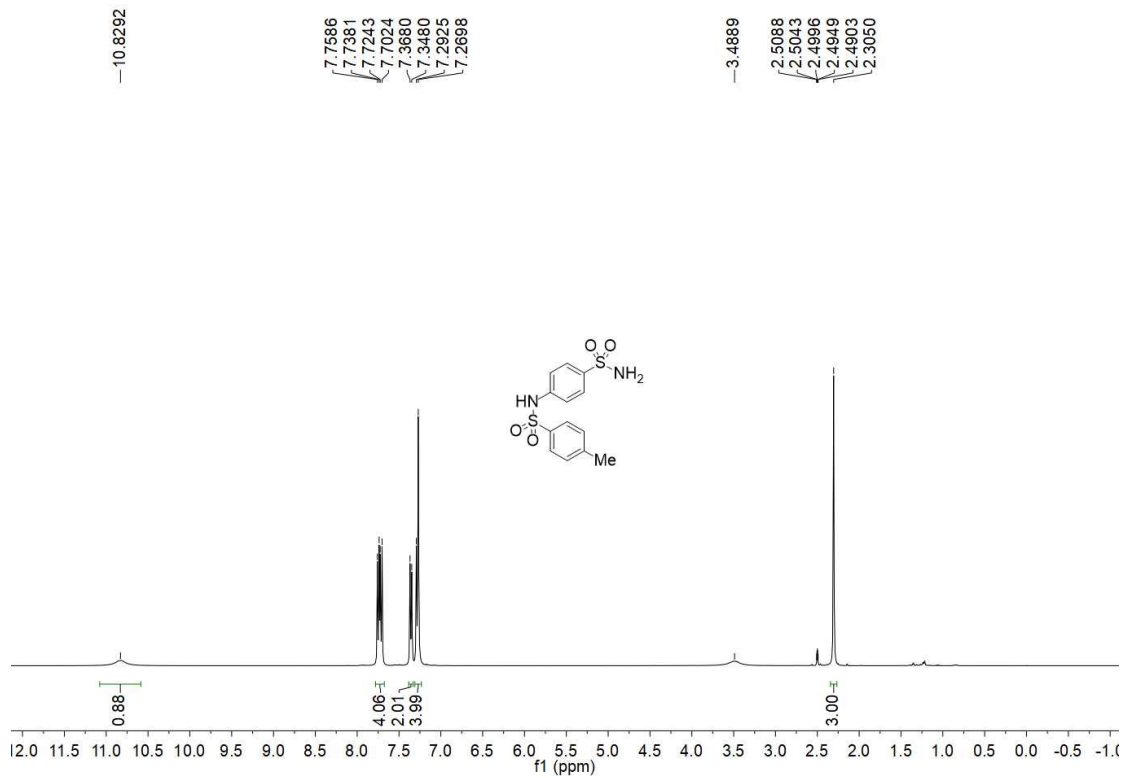
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 5p



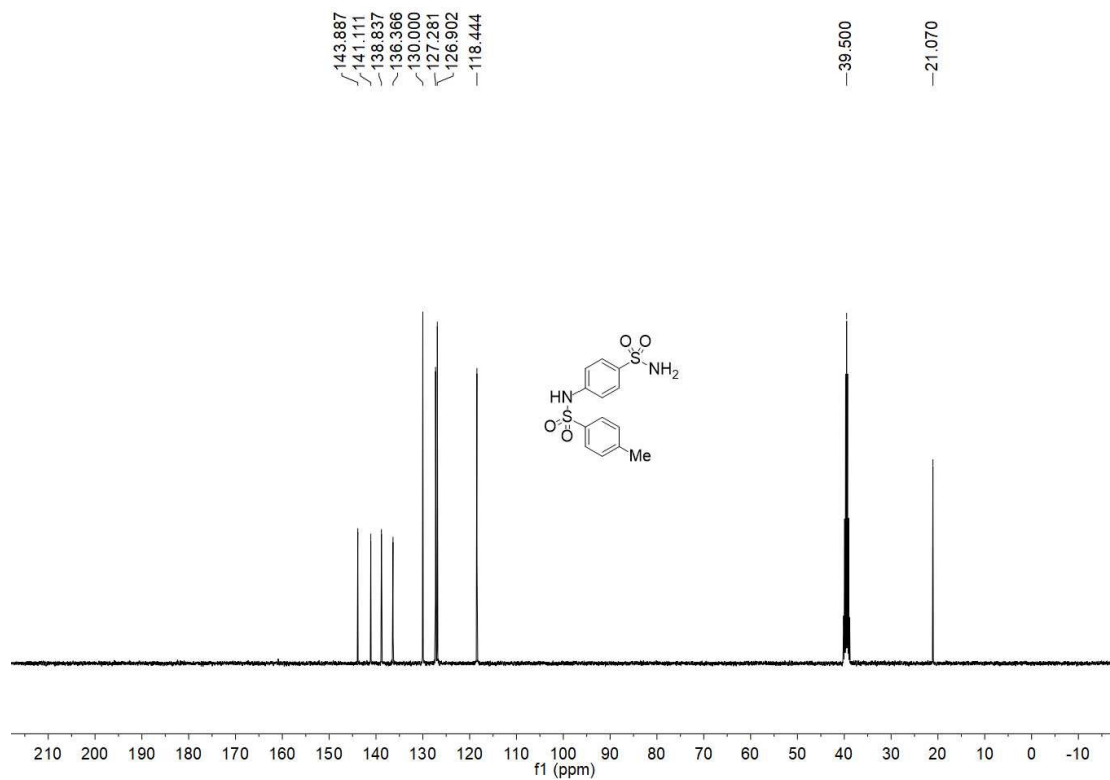
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 5p



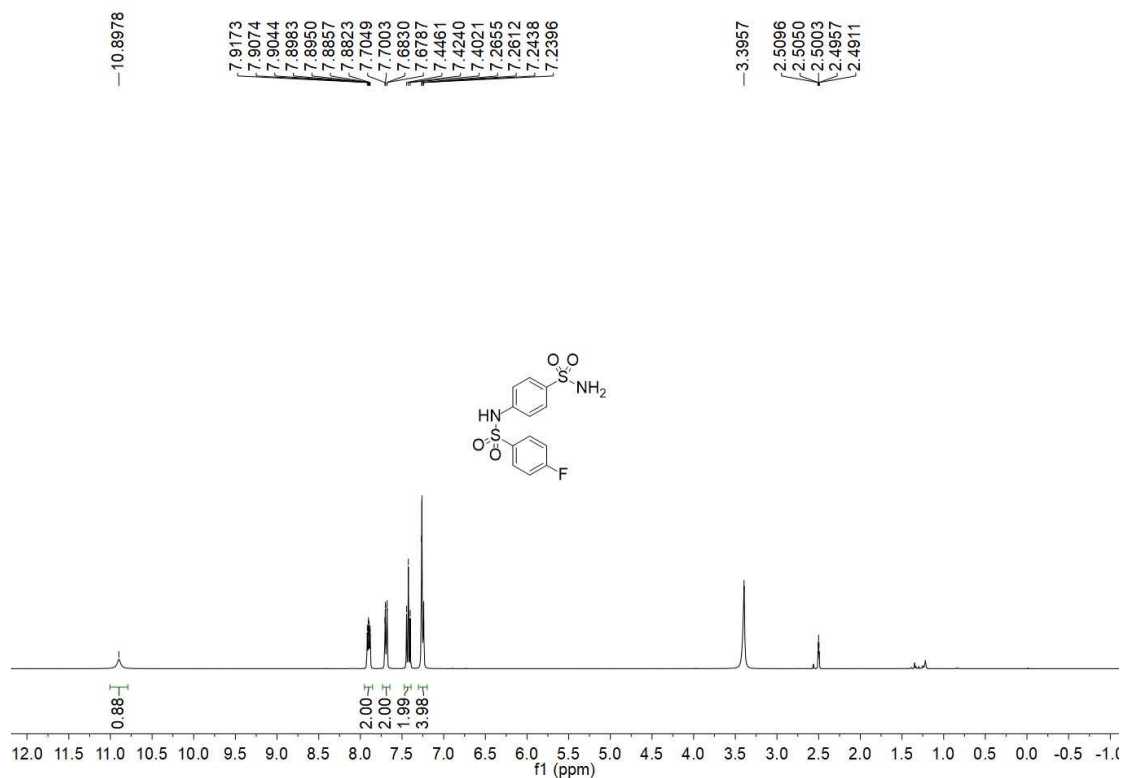
¹⁹F NMR (376 MHz, DMSO-*d*₆) spectrum of Compound 5p



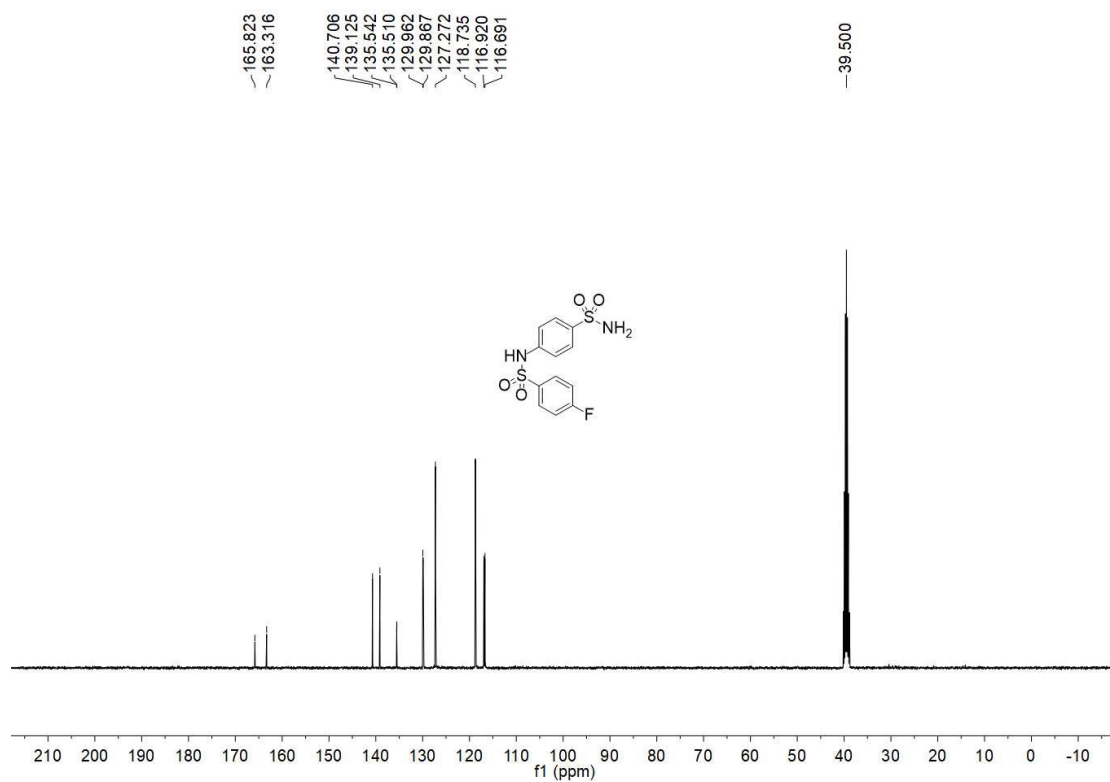
^1H NMR (400 MHz, $\text{DMSO-}d_6$) spectrum of Compound 6



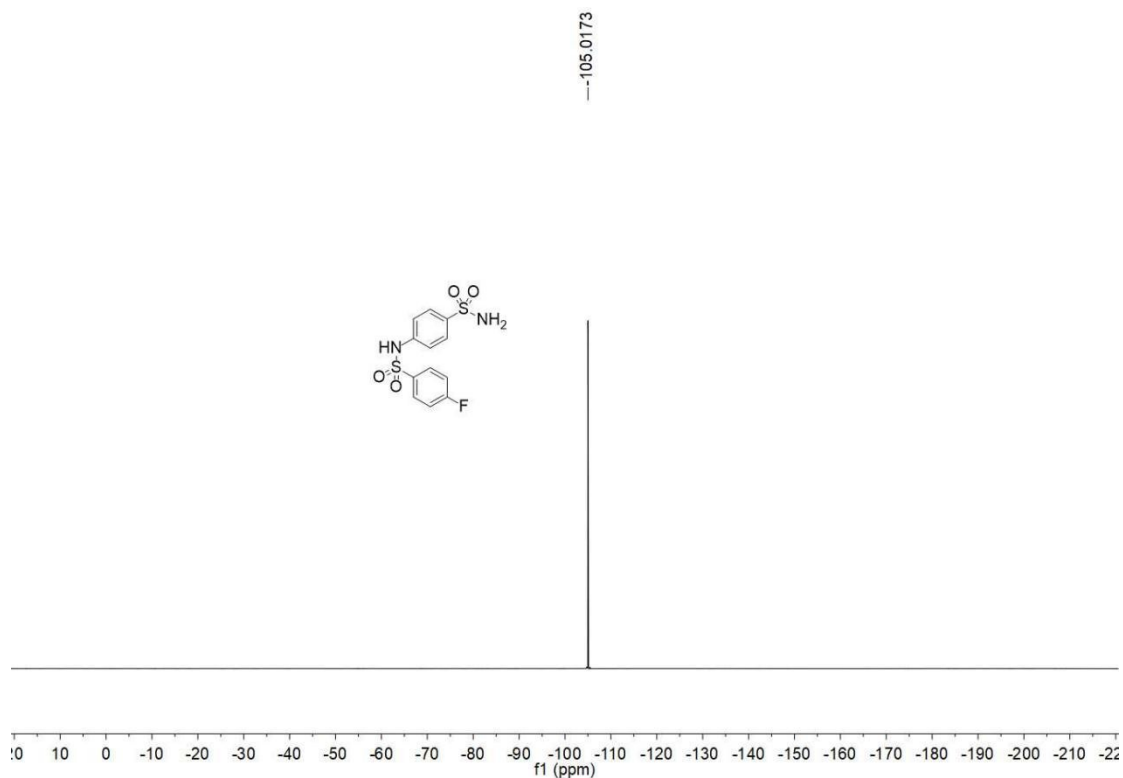
^{13}C NMR (101MHz, $\text{DMSO-}d_6$) spectrum of Compound 6



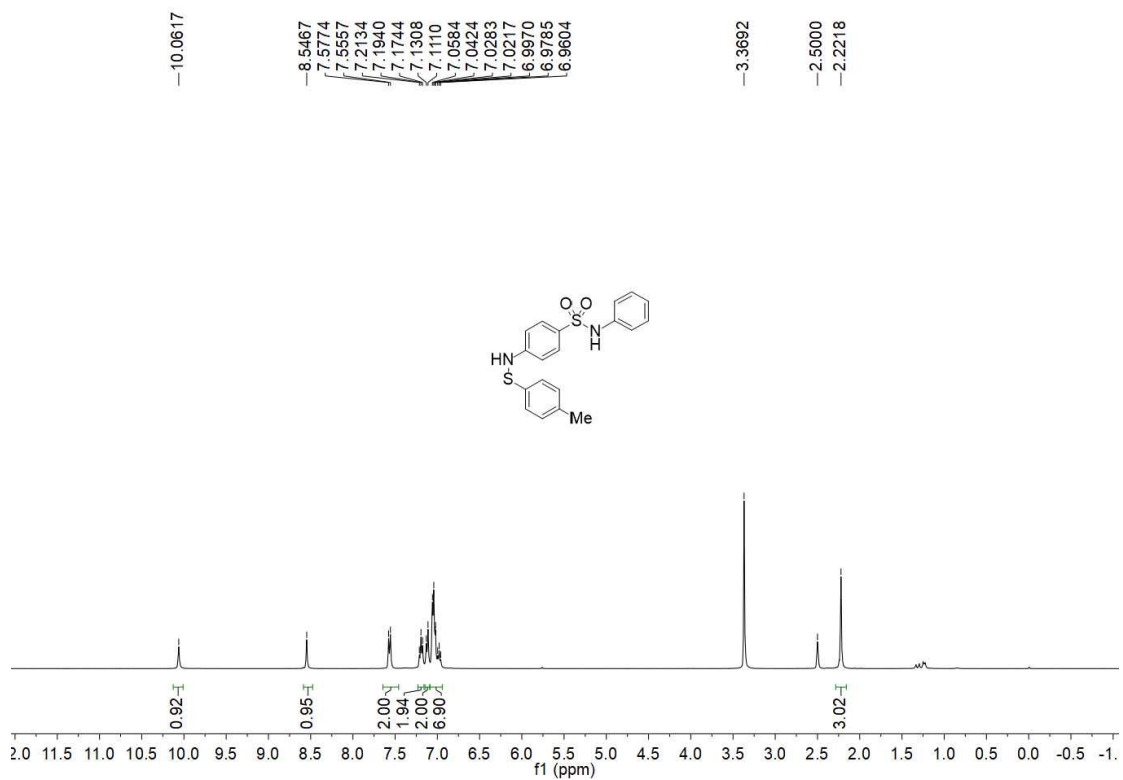
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 7



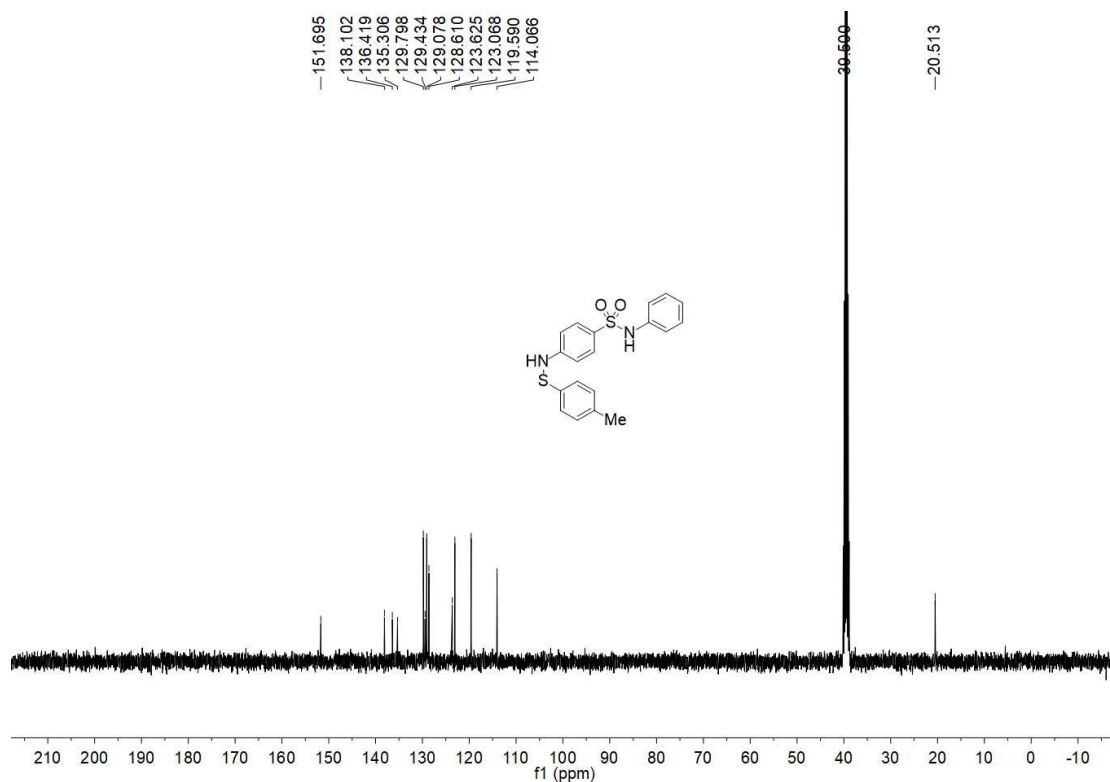
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 7



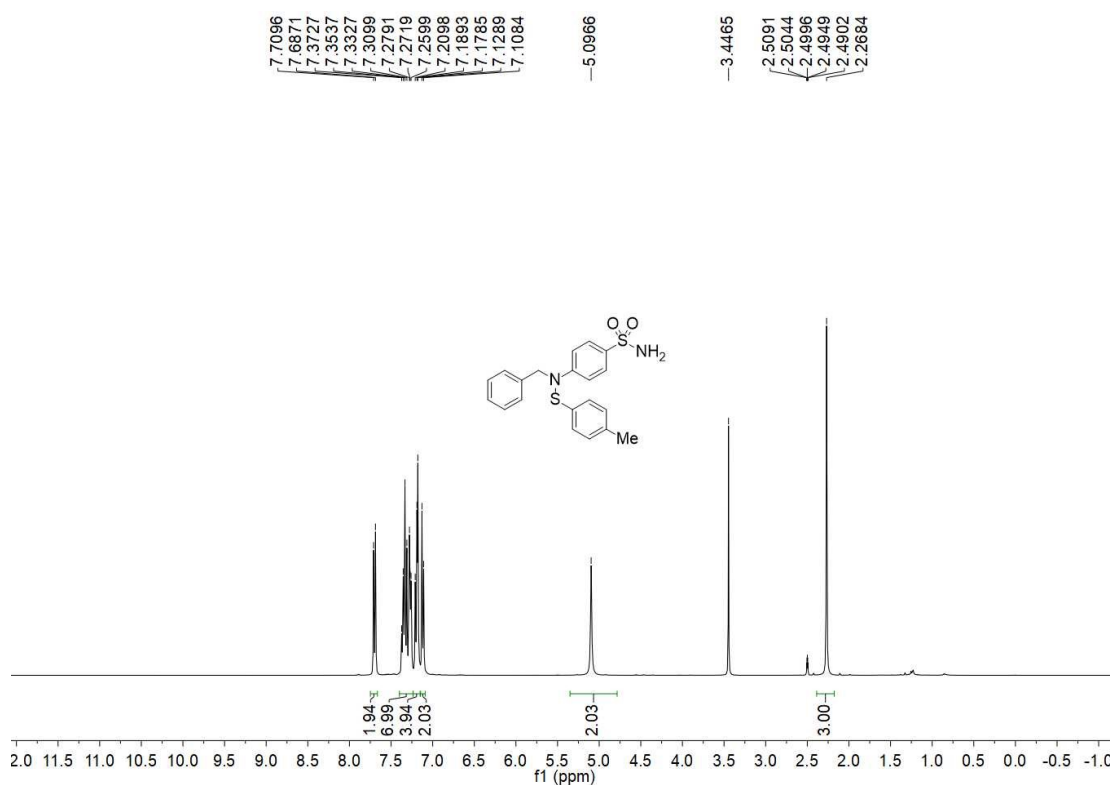
^{19}F NMR (376 MHz, $\text{DMSO-}d_6$) spectrum of Compound 7



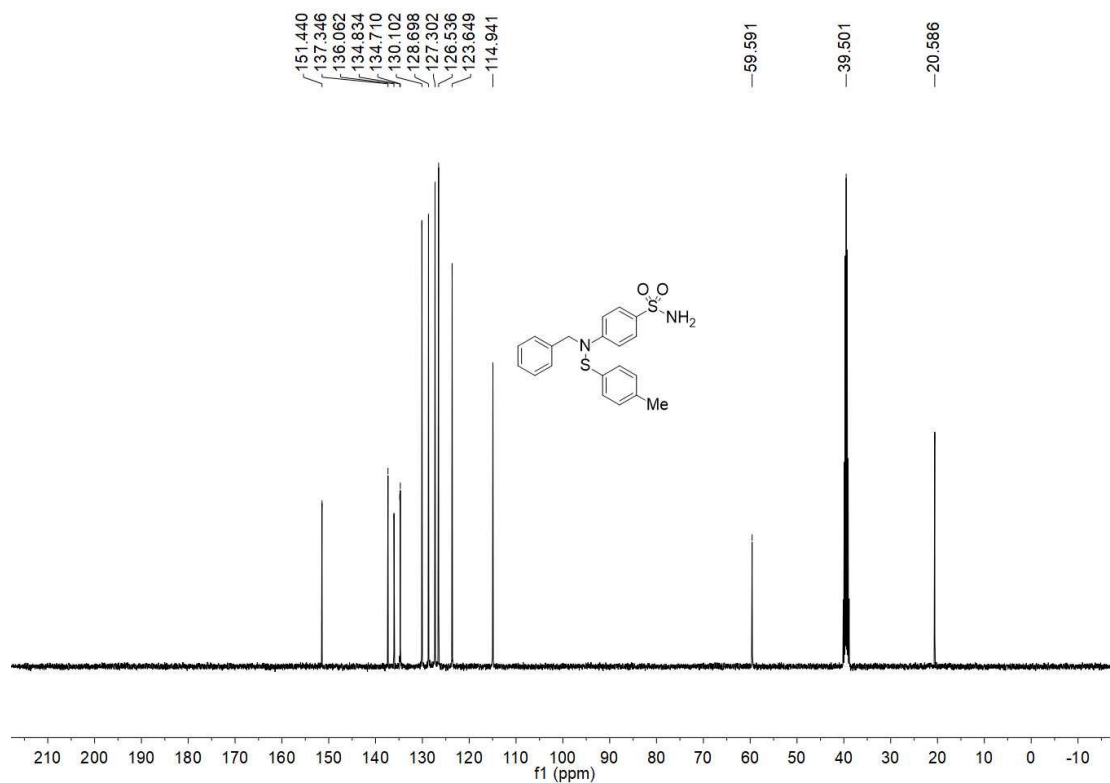
^1H NMR (400 MHz, $\text{DMSO-}d_6$) spectrum of Compound 8



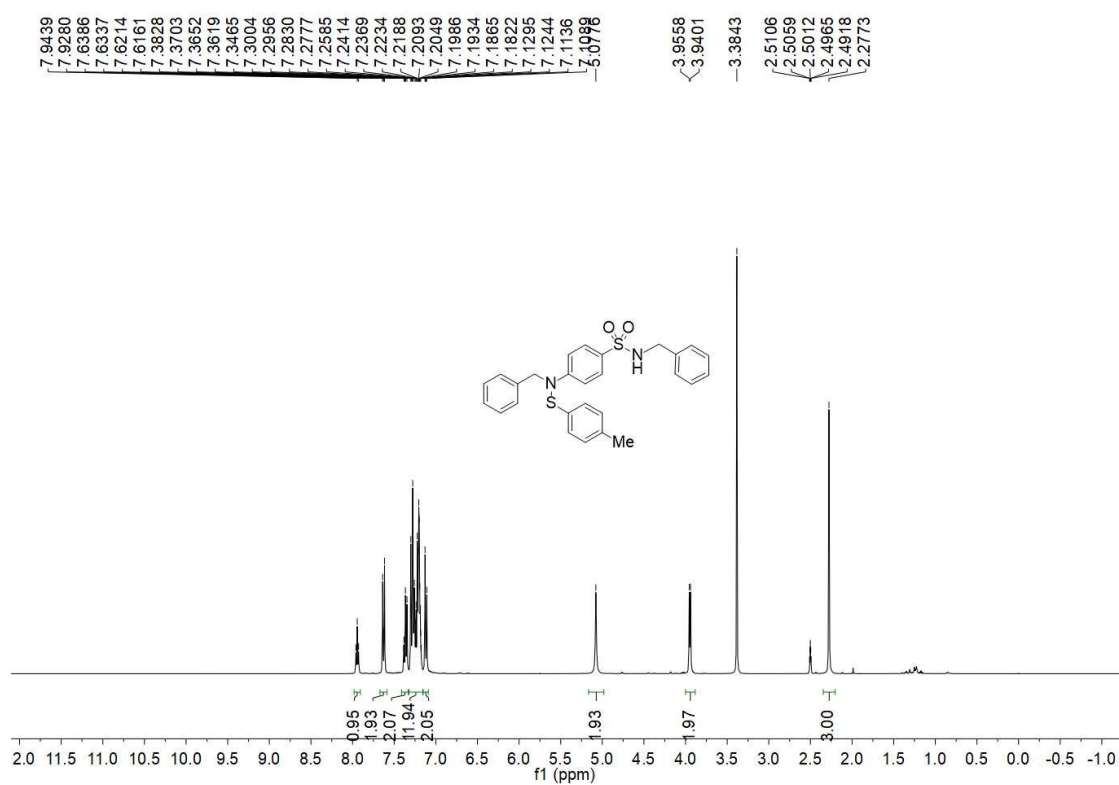
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 8



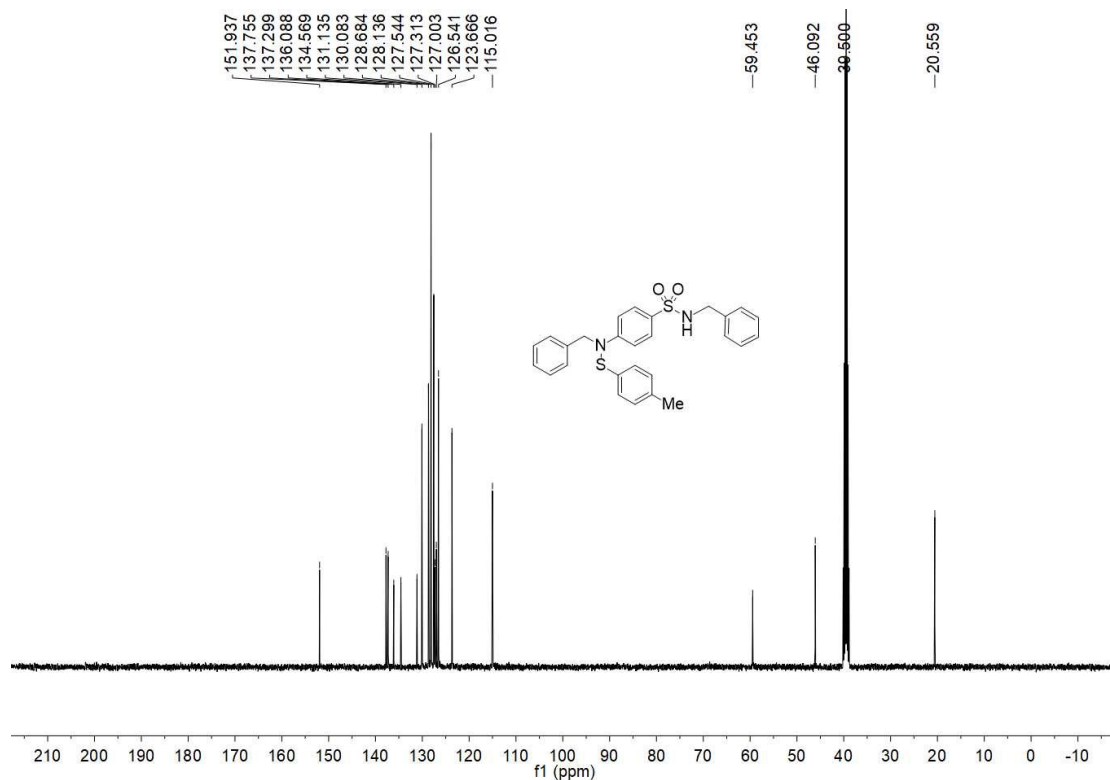
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 9



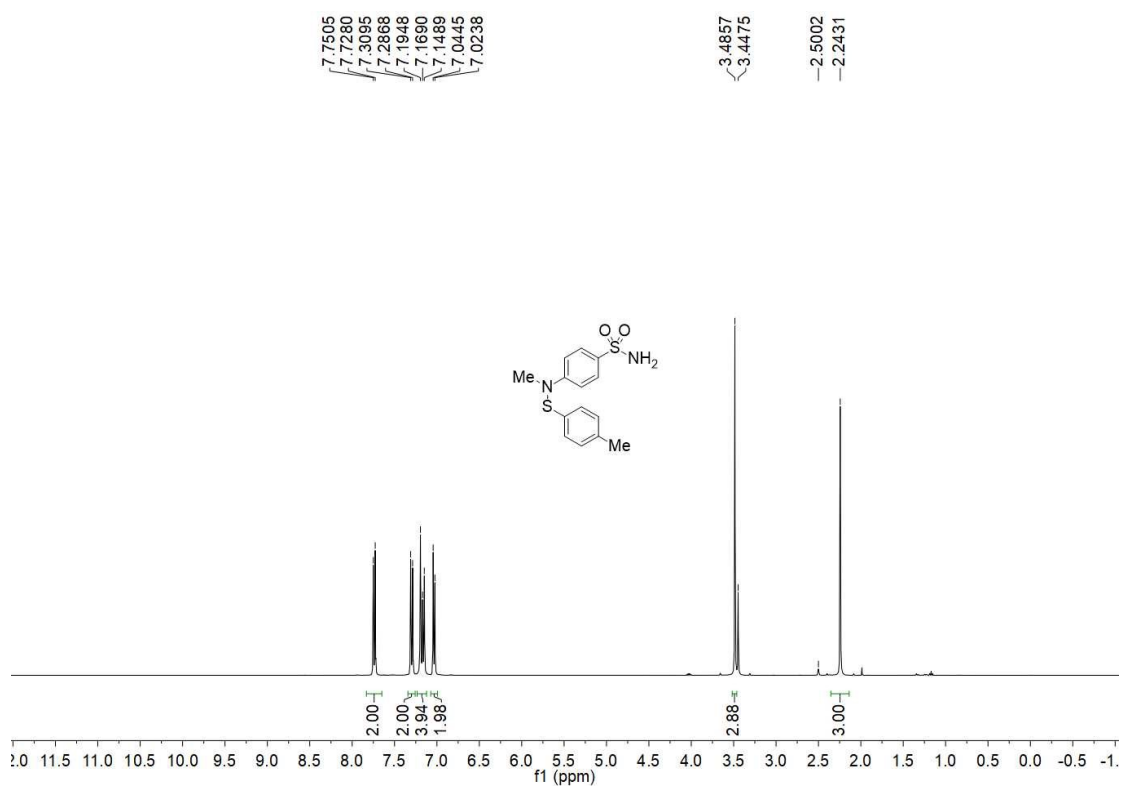
¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 9



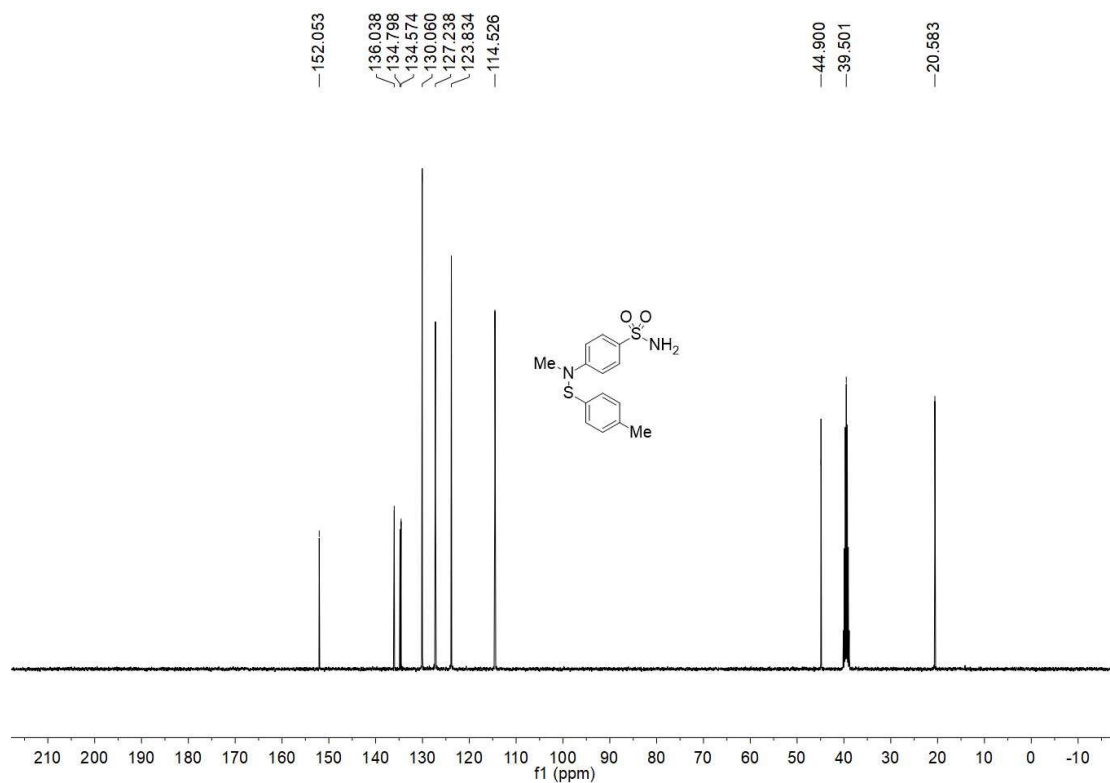
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of Compound 9'



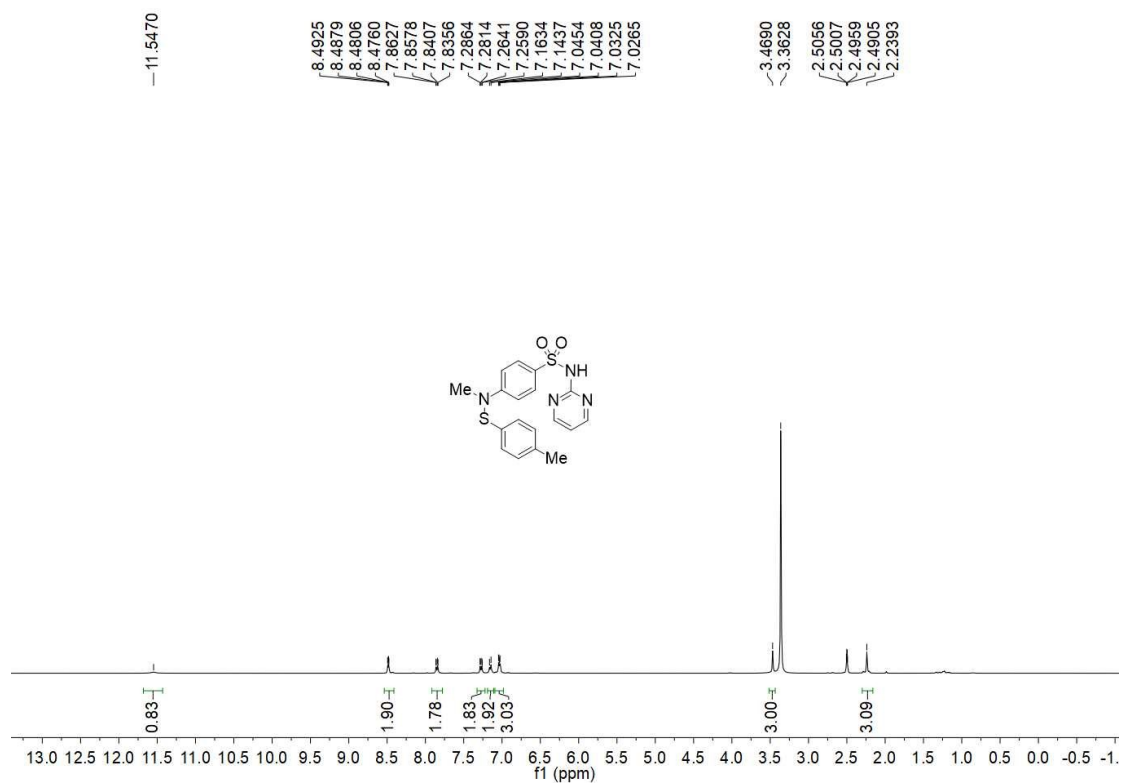
^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) spectrum of Compound 9'



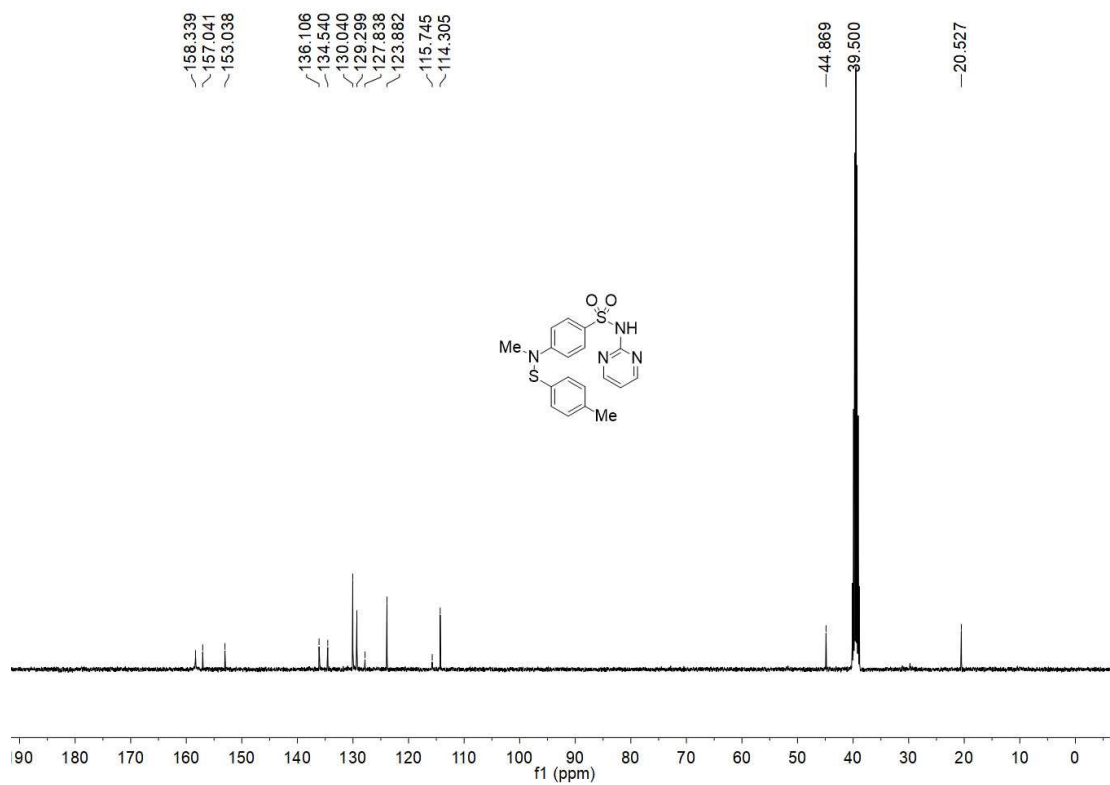
^1H NMR (400 MHz, $\text{DMSO-}d_6$) spectrum of Compound 10



^{13}C NMR (101 MHz, $\text{DMSO-}d_6$) spectrum of Compound 10



^1H NMR (400 MHz, $\text{DMSO-}d_6$) spectrum of Compound 11



¹³C NMR (101 MHz, DMSO-*d*₆) spectrum of Compound 11