

Supporting Information

Transition-metal-free oxidative reaction of hydrazines and potassium metabisulfite for preparation of sulfonohydrazide

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1. General information

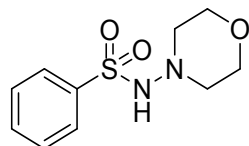
Except the special statement, all chemicals were purchased without further purification before use (Hydrazines were obtained by using NaOH (1 M) as base to neutralize the corresponding hydrazine hydrochlorides in water and extracted with ethyl acetate.). Solvents were dried by the standard method in solvent encliridion or treated with molecular sieves. General operation were conducted in oven-dried schlenk under dry-air balloom. Melting points were recorded on a melting points apparatus and were uncorrected. Thin layer chromatography (TLC) was performed on silica gel GF-254 plates and visualized by fluorescence quenching under UV light. For column chromatography, 200-300 mesh of silica gel was used and performed by standard technique.

¹H-NMR and ¹³C-NMR spectra were recorded on a Bruker Avance 400 spectrometer in CDCl₃ [using (CH₃)₄Si (for ¹H, δ = 0.00; for ¹³C, δ = 77.00) as internal standard] at room temperature. All coupling constants were given in Hertz (Hz). Infrared (IR) spectra was performed on Bruker Vector 22 in KBr pellets. High resolution mass spectra (HR MS) were obtained on a LTQ-Orbitrap XL with the ESI technique.

2. Typical procedure metal-free coupling reaction

Hydrazines **1** (0.25 mmol), K₂S₂O₅ (0.5 mmol), diamine (0.3 mmol), were added to a 25 mL schlenk tube under dry air balloom, followed by addition of CH₃CN (2.5 ml). The mixture was stirred at 40 °C for 12 h, then filtered and the solid was washed with ethyl acetate. The organic solution was dried over Na₂SO₄ and concentrated under vacuo. Then, crude product was purified by flash chromatography to afford the desired product **3**.

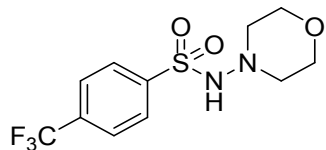
3. Characterization data for **3**, **4**



N-morpholinobenzenesulfonamide¹ (**3aa**)

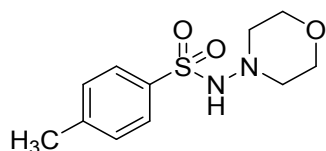
White solid, m.p. 121–122°C. ¹H NMR (400 MHz, Chloroform-d) δ 8.01 – 7.96 (m, 2H), 7.64 – 7.57 (m, 1H), 7.57 – 7.50 (m, 2H), 5.93 (s, 1H), 3.60 (t, 4H), 2.62 (t, 4H); ¹³C NMR (101 MHz,

Chloroform-d) δ 138.62 , 133.16 , 128.85 , 128.12 , 66.62 , 56.63; IR ν_{\max} (neat)/cm⁻¹ 3134, 2858, 1451, 1366, 1335, 1264, 1156, 1102, 856, 724.



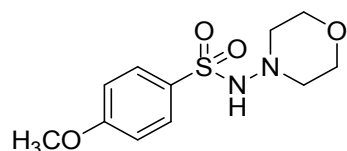
N-morpholino-4-(trifluoromethyl)benzenesulfonamide² (3ba)

White solid, m.p. 163–164°C. ¹H NMR (400 MHz, Chloroform-d) δ 8.12 (d, J = 8.2 Hz, 2H), 7.80 (d, J = 8.2 Hz, 2H), 5.81 (s, 1H), 3.63 (t, 4H), 2.67 (t, 4H); ¹³C NMR (101 MHz, Chloroform-d) δ 142.30 , 134.84 (q, J = 33.1 Hz), 128.65 , 126.00 (q, J = 3.7 Hz), 123.19 (q, J = 273.1 Hz), 66.58 , 56.80; IR ν_{\max} (neat)/cm⁻¹ 3196, 2860, 1403, 1322, 1165, 1107, 1061, 1012, 869, 840, 712.



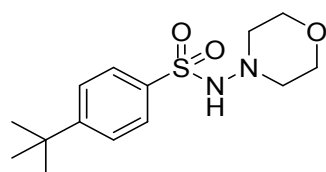
4-methyl-N-morpholinobenzenesulfonamide¹ (3ca)

White solid, m.p. 102–103°C. ¹H NMR (400 MHz, Chloroform-d) δ 7.92 – 7.65 (m, 2H), 7.24 (d, J = 8.1 Hz, 2H), 5.55 (s, 1H), 3.53 (t, 4H), 2.55 (t, 4H), 2.37 (s, 3H); ¹³C NMR (101 MHz, Chloroform-d) δ 144.04 , 135.66 , 129.49 , 128.17 , 66.66 , 56.72 , 21.64; IR ν_{\max} (neat)/cm⁻¹ 3217, 2922, 2847, 1594, 1497, 1325, 1259, 1181, 1097, 1022, 795.



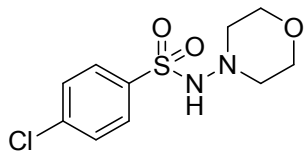
4-methoxy-N-morpholinobenzenesulfonamide¹ (3da)

Yellow solid, m.p. 167–168°C. ¹H NMR (400 MHz, Chloroform-d) δ 8.00 – 7.82 (m, 2H), 7.09 – 6.89 (m, 2H), 5.67 (s, 1H), 3.88 (s, 3H), 3.61 (t, 4H), 2.62 (t, 4H); ¹³C NMR (101 MHz, Chloroform-d) δ 163.27 , 130.32 , 130.08 , 114.01 , 66.68 , 56.69 , 55.63; IR ν_{\max} (neat)/cm⁻¹ 3199, 2962, 2839, 1593, 1579, 1495, 1456, 1335, 1253, 1104, 864, 803, 719.



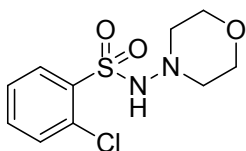
4-(tert-butyl)-N-morpholinobenzenesulfonamide¹ (3ea)

White solid, m.p. 173 – 174°C. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.98 – 7.79 (m, 2H), 7.62 – 7.46 (m, 2H), 5.74 (s, 1H), 3.61 (t, 4H), 2.64 (t, 4H), 1.35 (s, 9H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 157.04 , 135.64 , 127.96 , 125.81 , 66.62 , 56.75 , 35.21 , 31.10; IR ν_{max} (neat)/cm⁻¹ 13156, 2871, 2161, 1335, 1157, 1106, 863, 665.



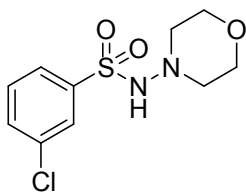
4-chloro-N-morpholinobenzenesulfonamide¹ (3fa)

White solid, m.p.173–174°C.¹H NMR (400 MHz, Chloroform-*d*) δ 8.00 – 7.84 (m, 2H), 7.63 – 7.42 (m, 2H), 5.73 (s, 1H), 3.62 (t, 4H), 2.65 (t, 4H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 139.75 , 137.13 , 129.59 , 129.18 , 66.61 , 56.73; IR ν_{max} (neat)/cm⁻¹13180, 2922, 2855, 1647, 1574, 1468, 1331, 1158, 1083, 866, 755.



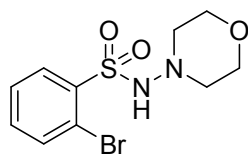
2-chloro-N-morpholinobenzenesulfonamide¹ (3ga)

White solid, m.p. 163–164°C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.24 – 8.17 (m, 1H), 7.59 – 7.50 (m, 2H), 7.49 – 7.43 (m, 1H), 6.21 (s, 1H), 3.57 (t, 4H), 2.70 (t, 4H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 136.28 , 134.24 , 132.99 , 131.66 , 131.52 , 127.17 , 66.44 , 56.58; IR ν_{max} (neat)/cm⁻¹3145, 2157, 2037, 1570, 1435, 1338, 1174, 1099, 1040, 858, 774, 665.



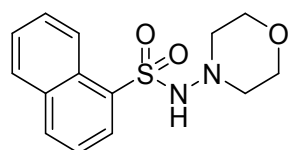
3-chloro-N-morpholinobenzenesulfonamide¹ (3ha)

Light yellow oil.¹H NMR (400 MHz, Chloroform-*d*) δ 7.98 (t, J = 2.0 Hz, 1H), 7.90 – 7.84 (m, 1H), 7.62 – 7.56 (m, 1H), 7.48 (t, J = 7.9 Hz, 1H), 5.96 (d, J = 8.4 Hz, 1H), 3.63 (t, 4H), 2.66 (t, 5H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 140.34 , 135.04 , 133.28 , 130.16 , 128.18 , 126.18 , 66.59 , 56.68; IR ν_{max} (neat)/cm⁻¹2855, 1458, 1362, 1264, 1160, 1107, 864, 789, 669.



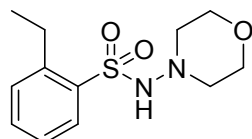
2-bromo-N-morpholinobenzenesulfonamide³ (3ia)

Yellow solid, m.p. 145–146°C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.25 (dd, *J* = 7.7, 1.9 Hz, 1H), 7.74 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.55 – 7.42 (m, 2H), 6.22 (s, 1H), 3.57 (t, 4H), 2.71 (t, 4H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 137.90 , 135.00 , 134.22 , 133.31 , 127.77 , 120.04 , 66.45 , 56.55; IR ν_{max} (neat)/cm⁻¹ 3160, 2920, 2847, 2155, 1430, 1338, 1170, 1099, 860, 772, 733.



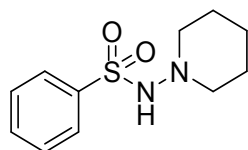
N-morpholinonaphthalene-1-sulfonamide⁴ (3ja)

Brown solid, m.p. 148–149°C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.80 (d, *J* = 8.7 Hz, 1H), 8.39 (dd, *J* = 7.4, 1.1 Hz, 1H), 8.09 (d, *J* = 8.2 Hz, 1H), 7.93 (d, *J* = 8.1 Hz, 1H), 7.72 – 7.64 (m, 1H), 7.63 – 7.53 (m, 2H), 5.97 (s, 1H), 3.47 (t, 4H), 2.53 (t, 4H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 134.80 , 134.07 , 133.57 , 131.40 , 128.91 , 128.55 , 128.18 , 126.85 , 125.14 , 124.20 , 66.50 , 56.75; IR ν_{max} (neat)/cm⁻¹ 3189, 2848, 1507, 1280, 1261, 1161, 1108, 864, 764, 676.



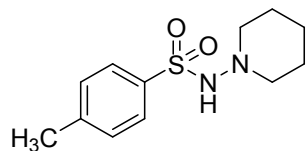
2-ethyl-N-morpholinobenzenesulfonamide (3ka)

Light yellow oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.11 – 7.85 (m, 1H), 7.46 (td, *J* = 7.5, 1.4 Hz, 1H), 7.35 – 7.19 (m, 2H), 5.62 (s, 1H), 3.65 – 3.40 (m, 4H), 3.04 (q, *J* = 7.5 Hz, 2H), 2.73 – 2.44 (m, 4H), 1.25 (t, *J* = 7.5 Hz, 4H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 144.22 , 136.13, 133.39 , 131.17 , 130.55, 125.91, 66.61, 56.76, 26.25, 15.53. HRMS (TOF MS ESI): calcd for C₁₂H₁₉N₂O₃S⁺ [M+H]⁺ 271.1111, found 271.1110. IR ν_{max} (neat)/cm⁻¹ 3184, 2966, 2864, 1363, 1262, 1108, 864, 763, 693, 596.



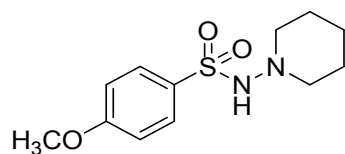
N-(piperidin-1-yl)benzenesulfonamide¹ (3ab)

Light yellow solid, m.p. 93–94°C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.05 – 7.90 (m, 2H), 7.63 – 7.56 (m, 1H), 7.55 – 7.48 (m, 2H), 5.48 (s, 1H), 2.53 (t, *J* = 5.4 Hz, 4H), 1.56 – 1.43 (m, 4H), 1.35 – 1.21 (m, 2H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 138.79 , 132.89 , 128.68 , 128.12 , 57.77 , 25.62 , 23.02; IR ν_{max} (neat)/cm⁻¹ 3206, 2933, 2850, 2159, 1448, 1326, 1158, 863, 761, 721.



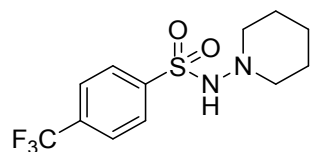
4-methyl-N-(piperidin-1-yl)benzenesulfonamide² (3ac)

Brown solid, m.p. 124–125°C. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.99 – 7.73 (m, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 5.42 (s, 1H), 2.53 (t, *J* = 5.4 Hz, 4H), 2.44 (s, 3H), 1.60 – 1.41 (m, 4H), 1.35 – 1.27 (m, 2H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 143.66 , 135.86 , 129.32 , 128.14 , 57.78 , 25.64 , 23.05 , 21.63; IR ν_{max} (neat)/cm⁻¹ 3199, 2921, 2851, 1597, 1468, 1455, 1329, 1161, 1036, 864, 794.



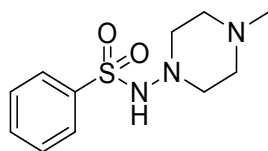
4-methoxy-N-(piperidin-1-yl)benzenesulfonamide² (3ad)

White solid, m.p. 179–180°C. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.92 – 7.87 (m, 2H), 7.02 – 6.93 (m, 2H), 5.35 (s, 1H), 3.88 (s, 3H), 2.53 (t, *J* = 5.4 Hz, 4H), 1.57 – 1.45 (m, 4H), 1.35 – 1.27 (m, 2H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 163.08 , 130.39 , 130.27 , 113.84 , 57.78 , 55.59 , 25.66 , 23.06; IR ν_{max} (neat)/cm⁻¹ 3197, 2960, 1595, 1457, 1333, 1266, 1161, 866, 812, 756.



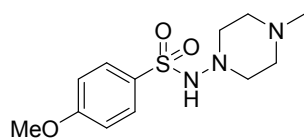
N-(piperidin-1-yl)-4-(trifluoromethyl)benzenesulfonamide (3ae)

Light yellow solid, m.p. 151–152°C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.11 (d, *J* = 8.2 Hz, 2H), 7.79 (d, *J* = 8.3 Hz, 2H), 5.55 (s, 1H), 2.56 (t, 4H), 1.59 – 1.45 (m, 4H), 1.39 – 1.27 (m, 2H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 142.43 , 134.56 (q, *J* = 33.0 Hz), 128.63 , 125.81 (q, *J* = 3.7 Hz), 123.28 (q, *J* = 272.9 Hz); HRMS (TOF MS ESI): calcd for C₁₂H₁₆F₃N₂O₂S⁺ [M+H]⁺ 309.0879, found 309.0876. IR ν_{max} (neat)/cm⁻¹ 3199, 2161, 1320, 1165, 1061, 795, 635.



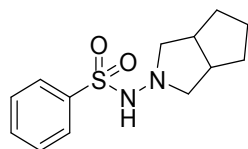
N-(4-methylpiperazin-1-yl)benzenesulfonamide (3af)

Light yellow solid, m.p. 153–154°C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.00 – 7.94 (m, 2H), 7.64 – 7.57 (m, 1H), 7.56 – 7.49 (m, 2H), 5.80 (s, 1H), 2.65 (t, *J* = 4.7 Hz, 4H), 2.37 (s, 4H), 2.22 (s, 3H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 138.71 , 133.01 , 128.78 , 128.12 , 55.93 , 54.53 , 45.55; HRMS (TOF MS ESI): calcd for C₁₁H₁₈N₃O₂S⁺ [M+H]⁺256.1114, found 256.1111. IR ν_{max} (neat)/cm⁻¹ 3676, 2955, 2823, 2160, 1447, 1315, 1277, 1153, 1078, 1012, 829, 757.



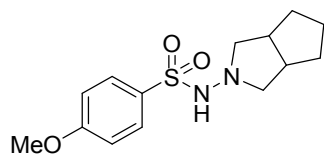
4-methoxy-N-(4-methylpiperazin-1-yl)benzenesulfonamide⁴ (3ag)

Yellow oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.95 – 7.81 (m, 2H), 7.06 – 6.90 (m, 2H), 5.48 (s, 1H), 3.88 (s, 3H), 2.68 (t, *J* = 4.9 Hz, 4H), 2.42 (s, 4H), 2.26 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 163.20, 130.31, 130.19, 55.79, 55.62, 54.52, 45.46. IR ν_{max} (neat)/cm⁻¹ 3194, 2962, 1594, 1336, 1255, 1155, 851, 686, 558.



N-(hexahydrocyclopenta[c]pyrrol-2(1H)-yl)benzenesulfonamide (3ah)

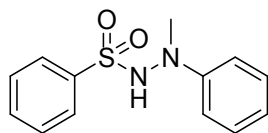
Light yellow oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.06 – 7.83 (m, 2H), 7.64 – 7.57 (m, 1H), 7.56 – 7.46 (m, 2H), 5.29 (s, 1H), 2.77 – 2.56 (m, 2H), 2.50 – 2.26 (m, 4H), 1.82 (s, 2H), 1.61 (td, *J* = 11.3, 9.7, 6.7 Hz, 4H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 138.68, 132.92, 128.76, 128.18, 63.18 , 40.06, 33.17, 26.19. HRMS (TOF MS ESI): calcd for C₁₃H₁₉N₂O₂S⁺ [M+H]⁺267.1162, found 267.1154. IR ν_{max} (neat)/cm⁻¹ 2949, 2862, 1447, 1329, 1165, 1093, 876, 723, 567.



N-(hexahydrocyclopenta[c]pyrrol-2(1H)-yl)-4-methoxybenzenesulfonamide (3ai)

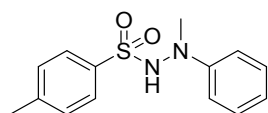
Light yellow oil. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.92 – 7.66 (m, 2H), 6.98 – 6.74 (m, 2H), 5.02

(s, 1H), 3.81 (s, 3H), 2.76 – 2.49 (m, 2H), 2.44 – 2.20 (m, 4H), 1.75 – 1.46 (m, 6H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 163.14, 130.31, 130.26, 113.93, 63.28, 55.62, 40.10, 33.17, 26.19. HRMS (TOF MS ESI): calcd for C₁₄H₂₁N₂O₃S⁺ [M+H]⁺297.1267, found 297.1273. IR ν_{max} (neat)/cm⁻¹ 3410, 2946, 2861, 1652, 1580, 1259, 1159, 1027, 833, 569.



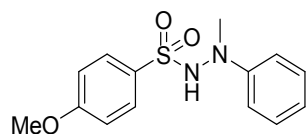
N'-methyl-N'-phenylbenzenesulfonohydrazide¹ (3aj)

Light yellow solid, m.p. 134–135°C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.03 – 7.91 (m, 2H), 7.70 – 7.55 (m, 1H), 7.54 – 7.41 (m, 2H), 7.22 – 7.11 (m, 2H), 6.94 – 6.76 (m, 3H), 6.37 (s, 1H), 2.95 (s, 3H); ¹³C NMR (101 MHz, Chloroform-*d*) δ 149.62, 138.54, 133.38, 129.11, 128.92, 128.17, 120.98, 114.37, 42.74; IR ν_{max} (neat)/cm⁻¹ 3196, 2918, 2850, 1599, 1501, 1446, 1339, 1285, 1156, 1087, 748.



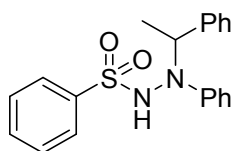
N',4-dimethyl-N'-phenylbenzenesulfonohydrazide⁵ (3ak)

brown solid, m.p. 93–94°C. ¹H NMR (400 MHz, Chloroform-*d*) δ 7.83 (d, *J* = 8.2 Hz, 2H), 7.37 – 7.21 (m, 2H), 7.22 – 7.12 (m, 2H), 6.91 – 6.77 (m, 3H), 6.21 (s, 1H), 2.95 (s, 3H), 2.42 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 149.74, 144.32, 135.55, 129.73, 128.91, 128.22, 120.92, 114.38, 42.68, 21.65. IR ν_{max} (neat)/cm⁻¹ 3196, 1599, 1501, 1335, 1157, 1090, 811, 749, 678, 531.



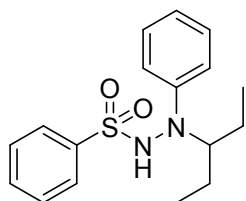
4-methoxy-N'-methyl-N'-phenylbenzenesulfonohydrazide² (3al)

Brown solid, m.p. 120–121°C. ¹H NMR (400 MHz, Chloroform-*d*) δ 8.00 – 7.69 (m, 2H), 7.22 – 7.06 (m, 2H), 7.02 – 6.91 (m, 2H), 6.92 – 6.76 (m, 3H), 6.20 (s, 1H), 3.85 (s, 3H), 2.96 (s, 3H). ¹³C NMR (101 MHz, Chloroform-*d*) δ 163.48, 149.76, 130.40, 129.96, 128.93, 120.89, 114.36, 114.27, 55.65, 42.65. IR ν_{max} (neat)/cm⁻¹ 3224, 1597, 1441, 1341, 1258, 1152, 1093, 1025, 754, 692, 534.



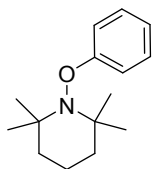
N'-phenyl-N'-(1-phenylethyl)benzenesulfonohydrazide (3ap)

Light yellow oil. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.87 – 7.62 (m, 2H), 7.51 – 7.36 (m, 1H), 7.33 – 7.15 (m, 5H), 7.07 – 6.92 (m, 4H), 6.84 (t, $J = 7.3$ Hz, 1H), 6.64 (d, $J = 7.5$ Hz, 2H), 6.02 (s, 1H), 4.69 (q, $J = 7.0$ Hz, 1H), 1.78 – 1.37 (m, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 148.49 , 138.81 , 132.76 , 128.55 , 128.52 , 128.30 , 128.09 , 127.95 , 127.81 , 122.87 , 119.41 , 65.68 , 18.15 . HRMS (TOF MS ESI): calcd for $\text{C}_{20}\text{H}_{21}\text{N}_2\text{O}_2\text{S}^+ [\text{M}+\text{H}]^+$ 353.1318, found 353.1310. IR ν_{max} (neat)/ cm^{-1} 3422, 2925, 1621, 1447, 1384, 1140, 752, 689, 596.



N'-(pentan-3-yl)-N'-phenylbenzenesulfonohydrazide (3ar)

Light yellow oil. ^1H NMR (400 MHz, Chloroform-*d*) δ 7.96 – 7.64 (m, 2H), 7.48 – 7.35 (m, 1H), 7.32 – 7.24 (m, 2H), 7.09 – 6.97 (m, 2H), 6.80 – 6.68 (m, 3H), 6.49 (s, 1H), 3.15 (tt, $J = 8.1, 5.5$ Hz, 1H), 1.56 (td, $J = 14.7, 7.4$ Hz, 2H), 1.42 (dq, $J = 14.5, 7.3, 5.5$ Hz, 2H), 1.11 – 0.55 (m, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 149.76 , 138.94 , 132.79 , 128.60 , 128.49 , 128.16 , 121.42 , 117.18 , 71.22 , 11.93. HRMS (TOF MS ESI): calcd for $\text{C}_{17}\text{H}_{22}\text{N}_2\text{O}_2\text{SNa}^+ [\text{M}+\text{Na}]^+$ 341.1294, found 341.1309. IR ν_{max} (neat)/ cm^{-1} 3442, 2923, 2852, 2361, 1633, 1401, 1143, 752, 618.



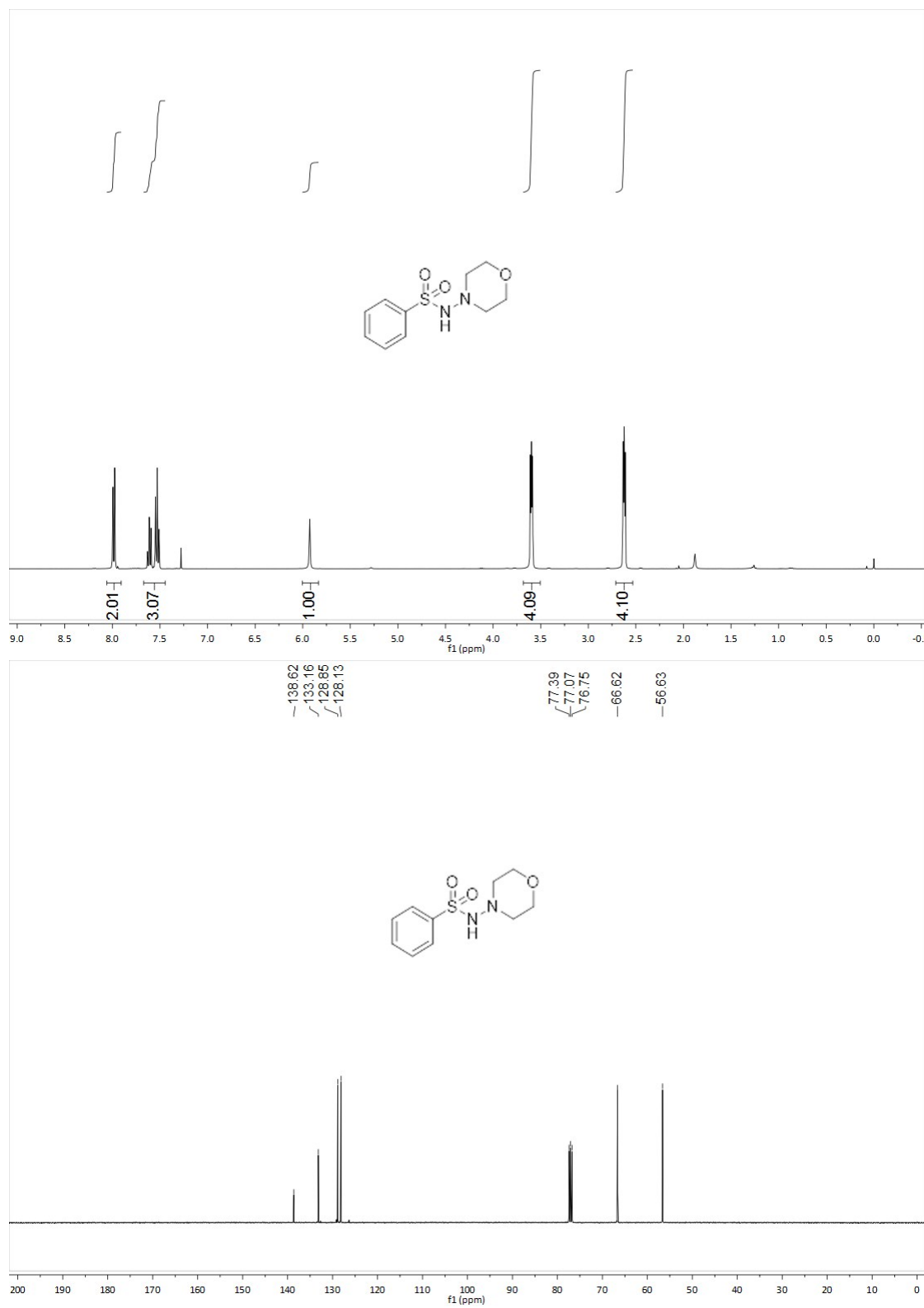
2,2,6,6-tetramethyl-1-phenoxy-piperidine¹(4)

Colourless oil. ^1H NMR (400 MHz, CDCl_3) δ 7.24 – 7.13 (m, 4H), 6.84 (tt, $J = 6.8, 1.8$ Hz, 1H), 1.62 – 1.52 (m, 6H), 1.23 (s, 6H), 1.01 (s, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 163.59, 128.65, 119.84, 113.89, 60.31, 39.79, 32.60, 20.45, 17.07.

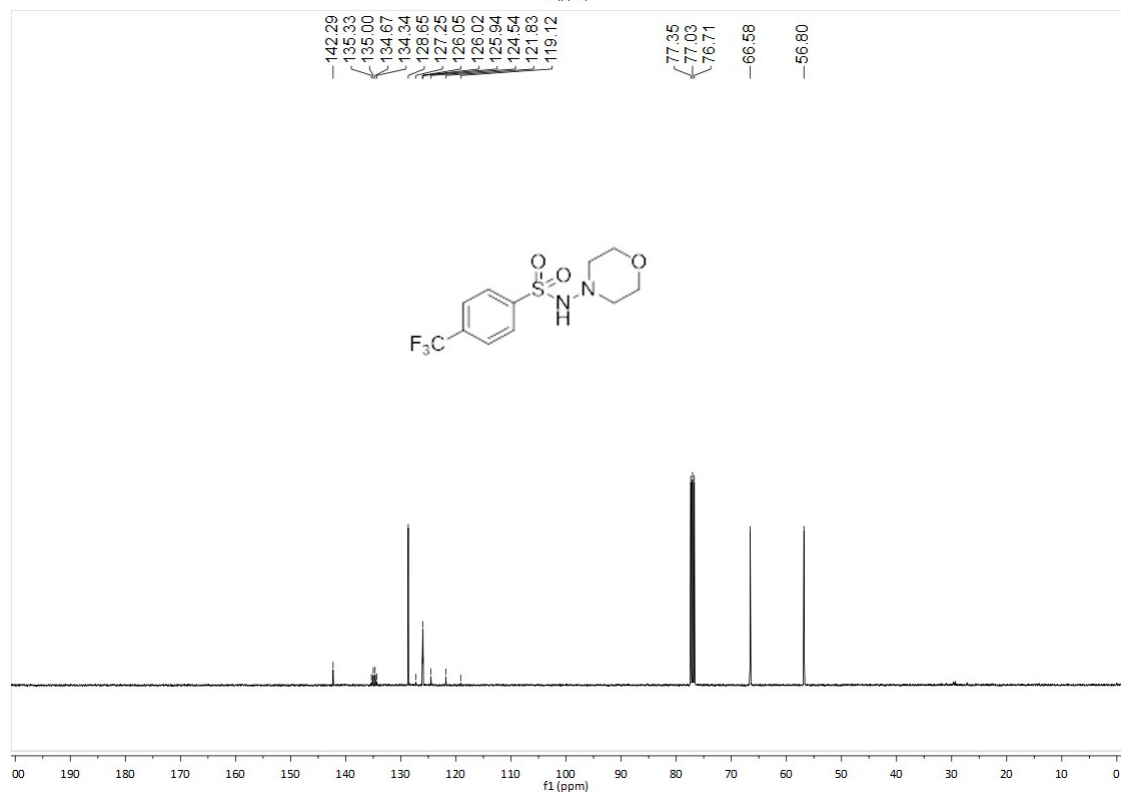
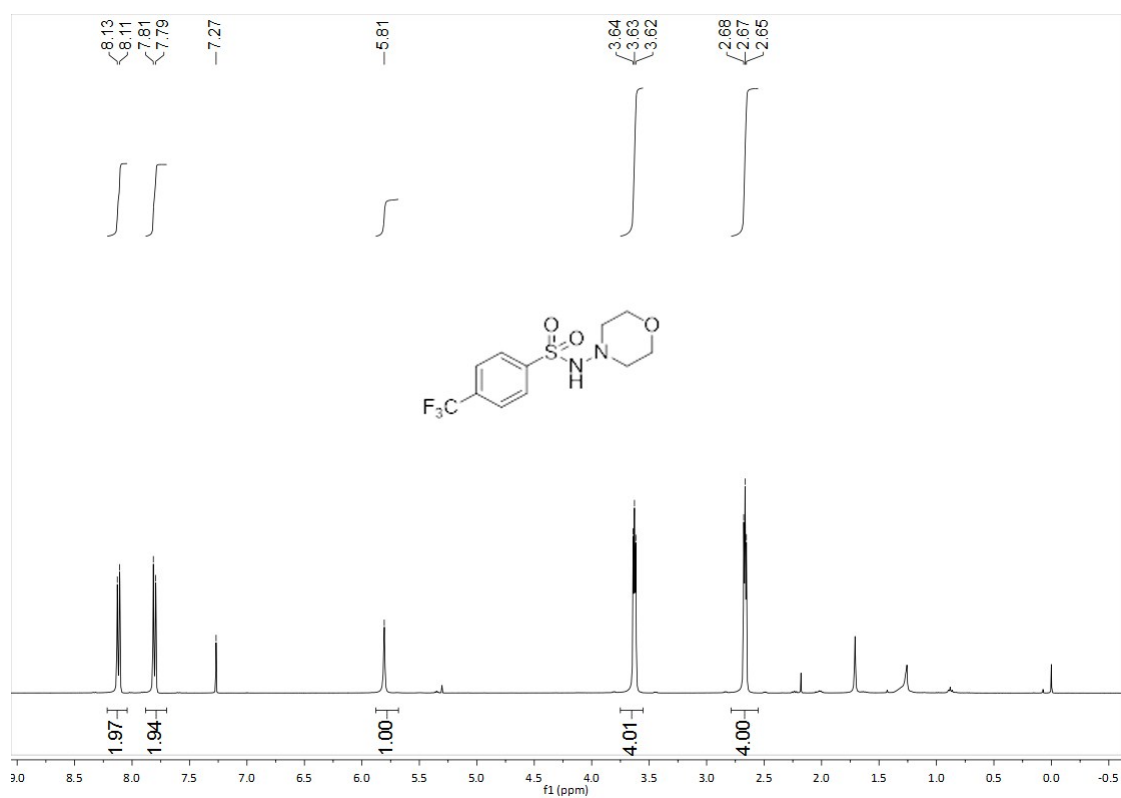
1. Zheng, D.; An, Y.; Li, Z.; Wu, J. *Angew. Chem. Int. Ed.* **2014**, *53*, 2451.
2. Ye, S.; Wu, J. *Chem. Commun.* **2012**, *48*, 10037.
3. Li, W.; Beller, M.; Wu, X. F. *Chem. Commun.* **2014**, *50*, 9513.
4. Li, W.; Li, H.; Langer, P.; Beller, M.; Wu, X.-F. *Eur. J. Org. Chem.* **2014**, 3101.
5. Emmett, E. J.; Richards-Taylor, C. S.; Nguyen, B.; Garcia-Rubia, A.; Hayter, B. R.; Willis, M. *C. Org. Biomol. Chem.* **2012**, *10*, 4007.

4. NMR spectra for compounds 3 and 4

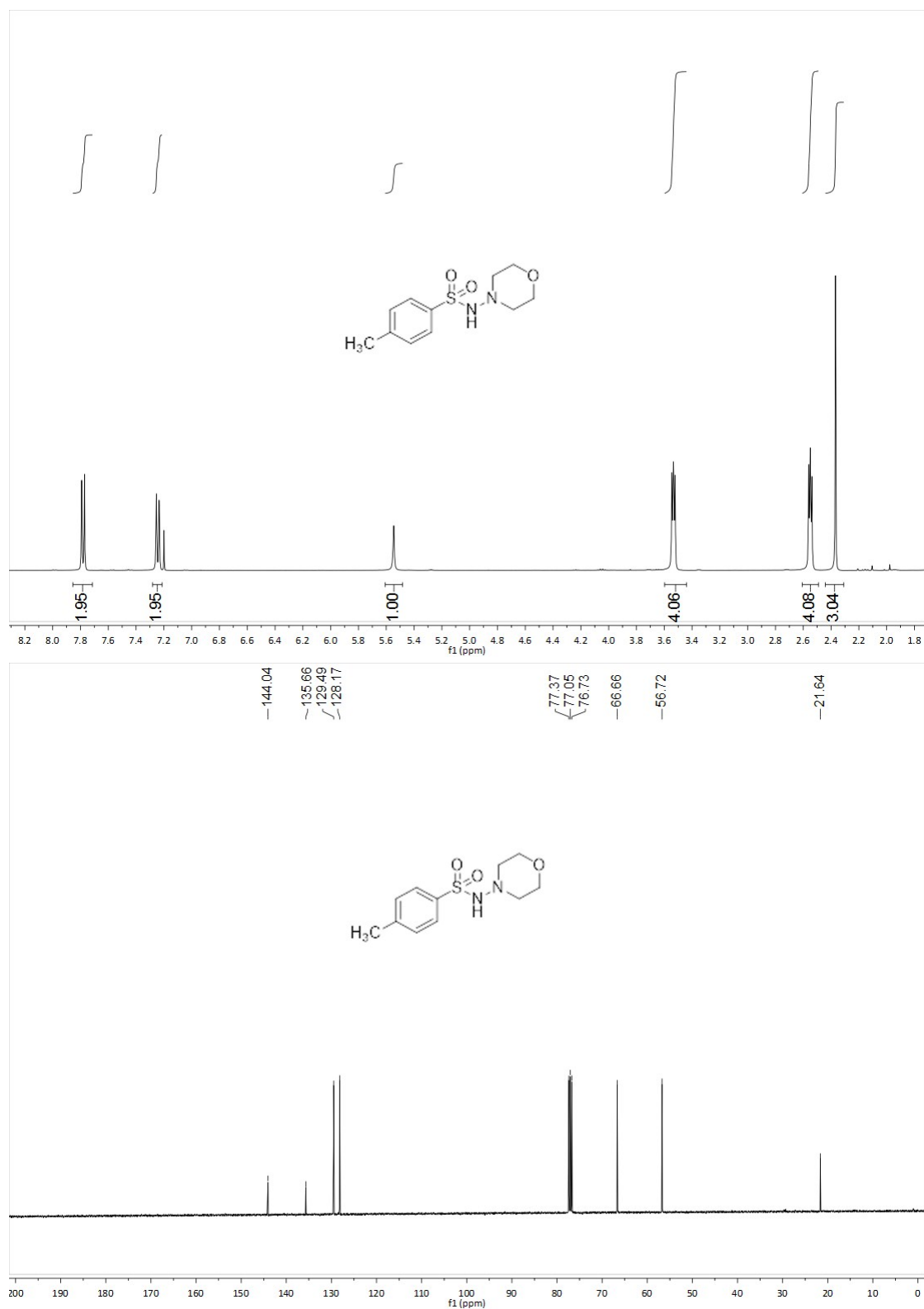
^1H and ^{13}C NMR for 3aa



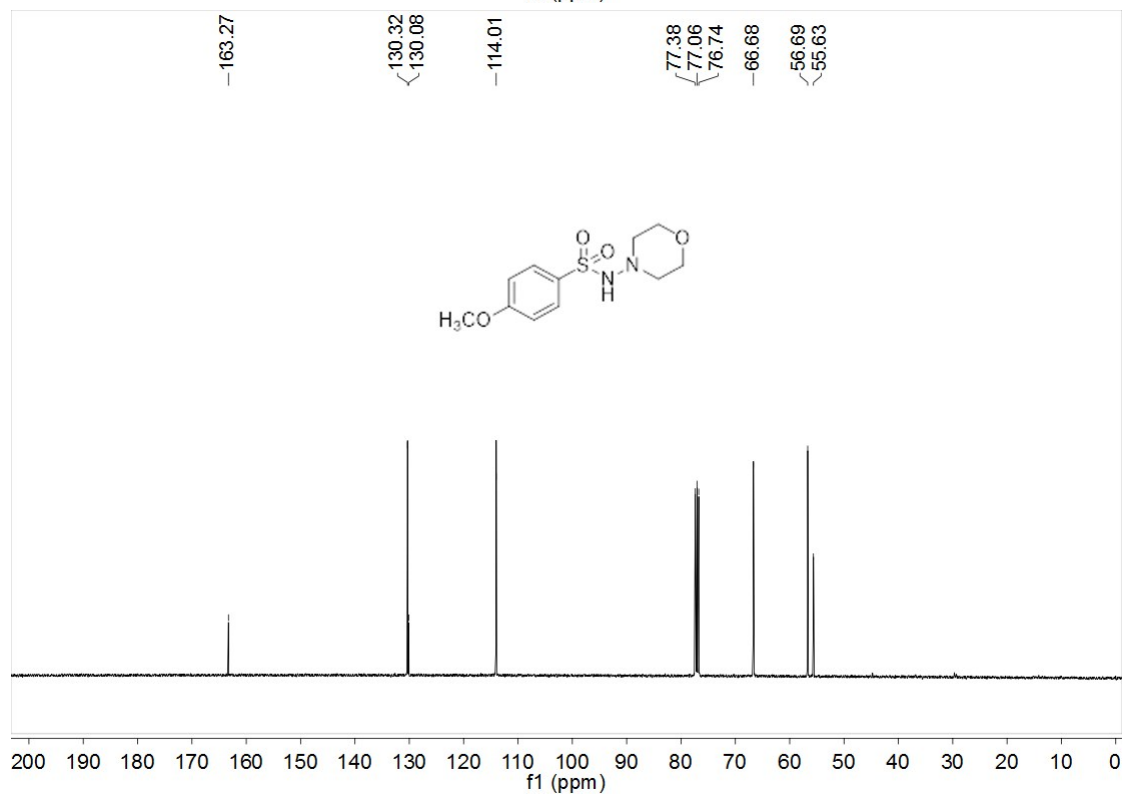
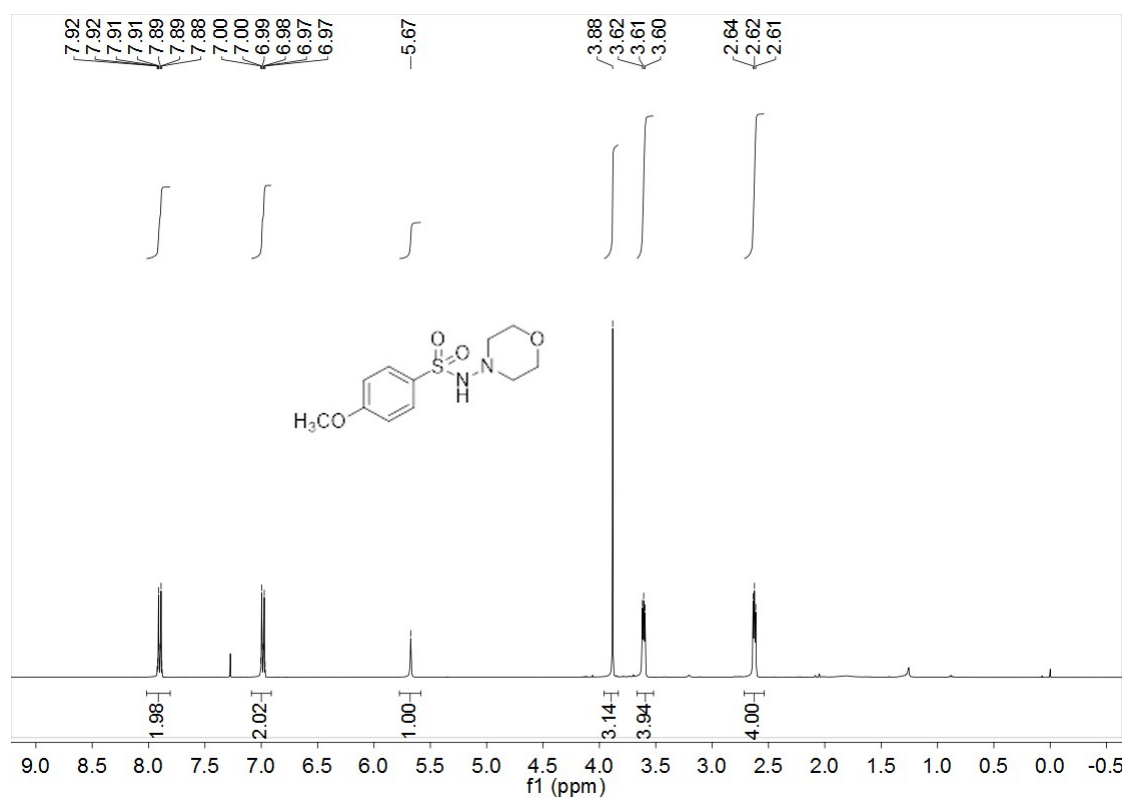
¹H and ¹³C NMR for **3ba**



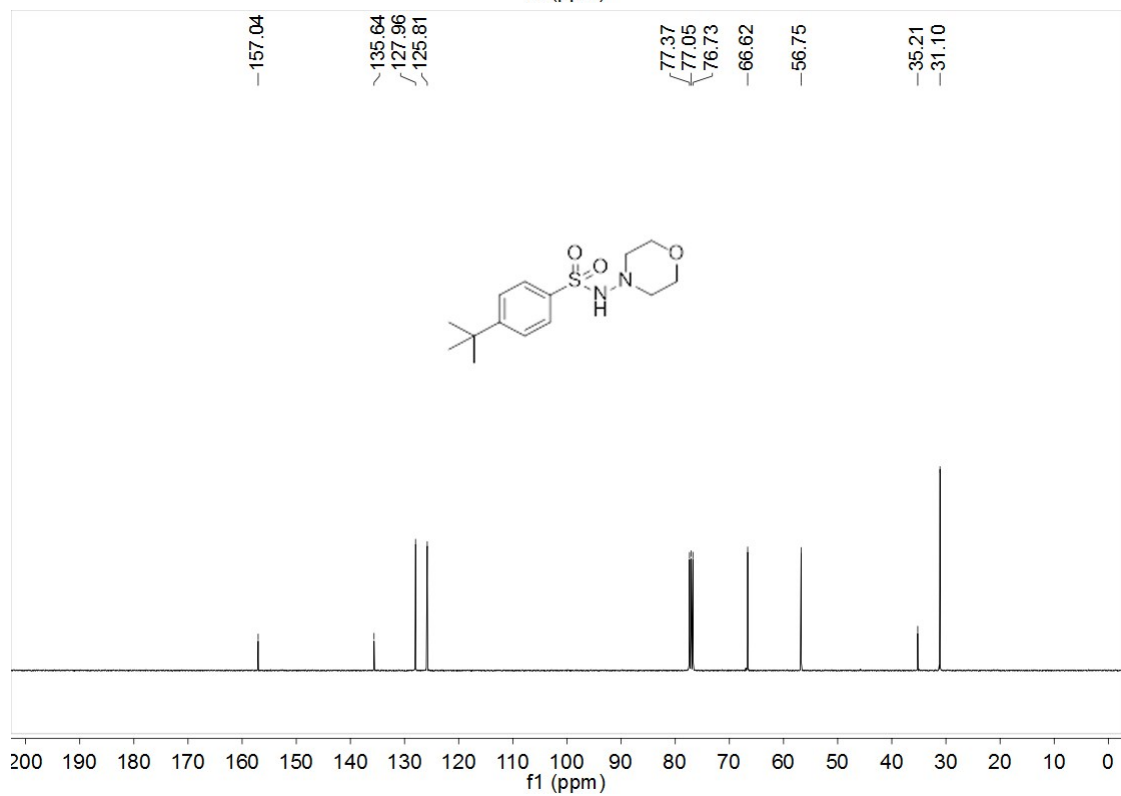
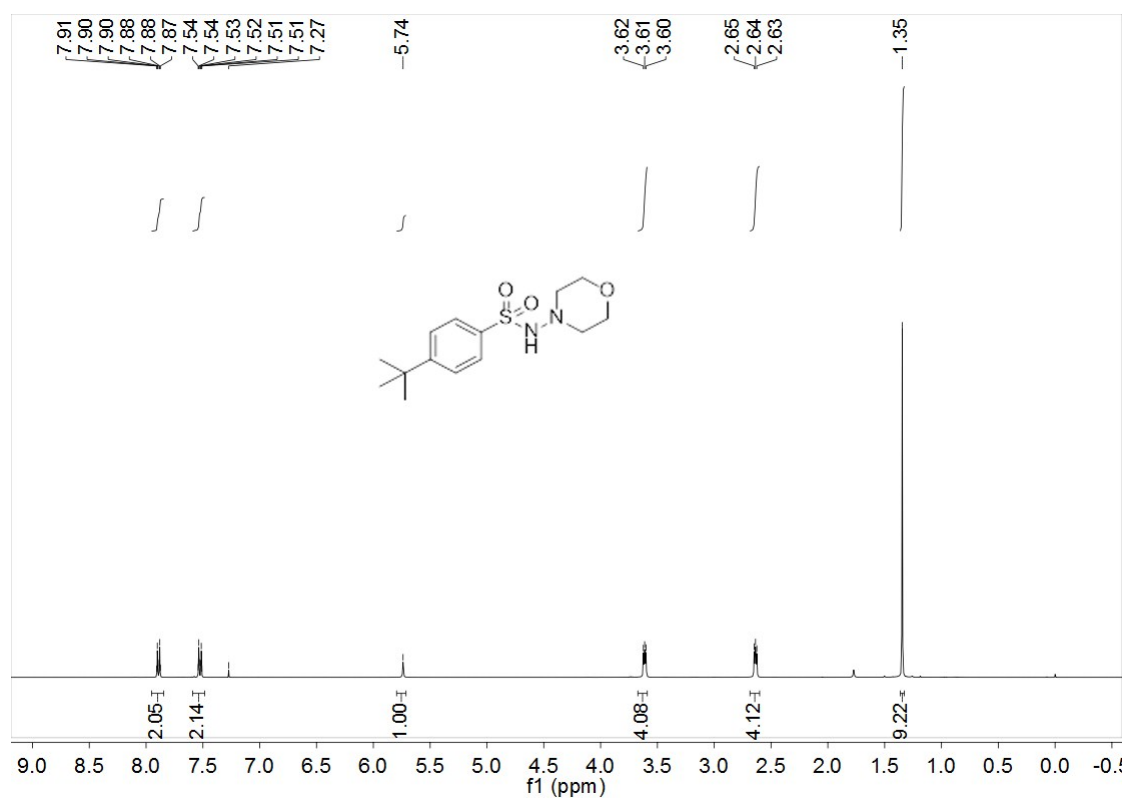
^1H and ^{13}C NMR for **3ca**



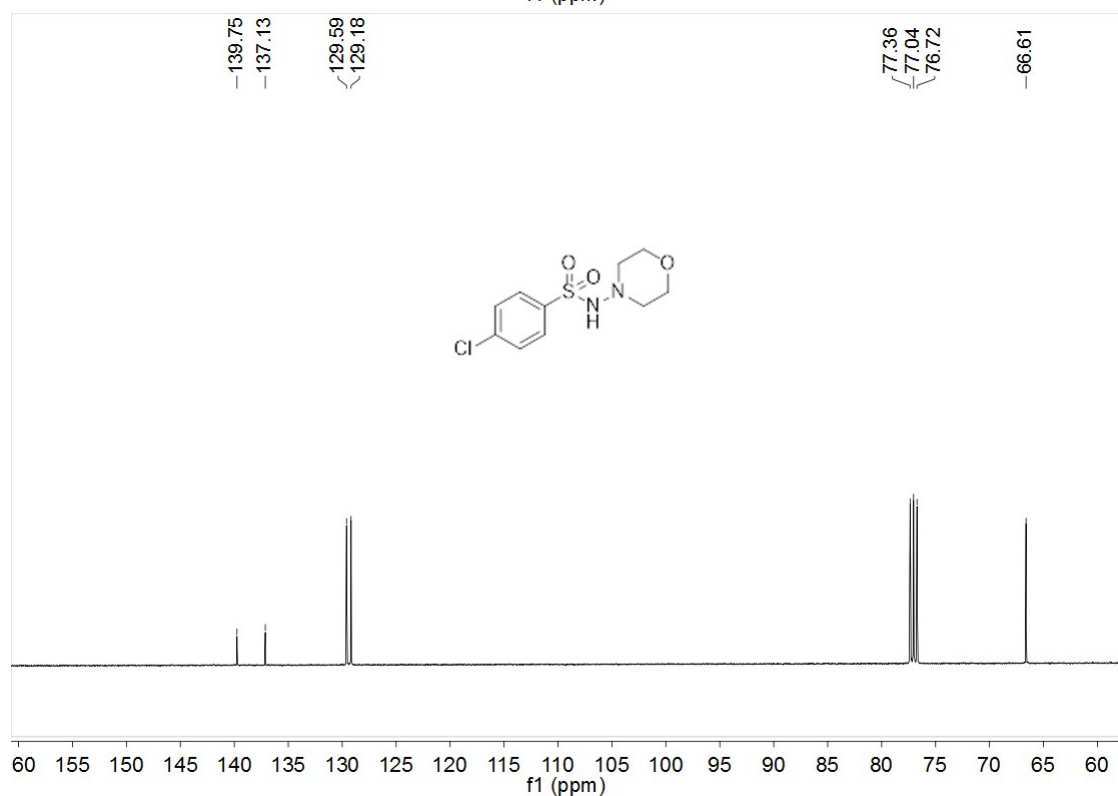
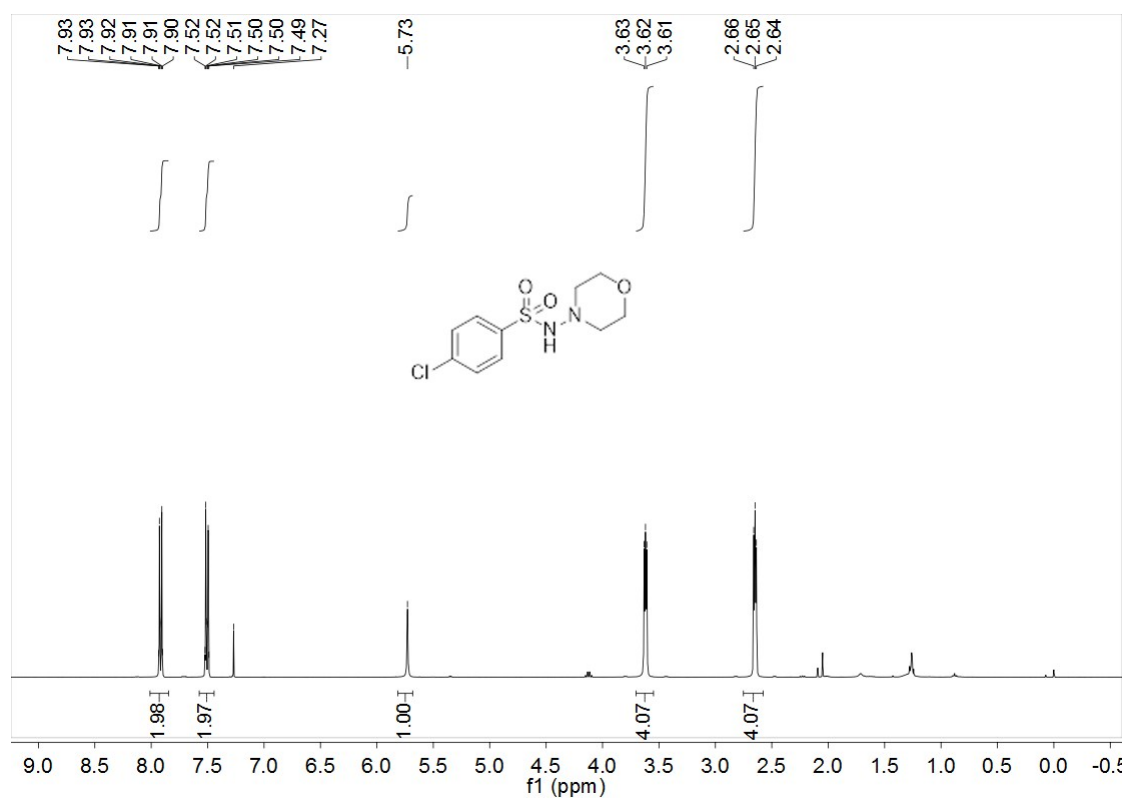
¹H and ¹³C NMR for **3da**



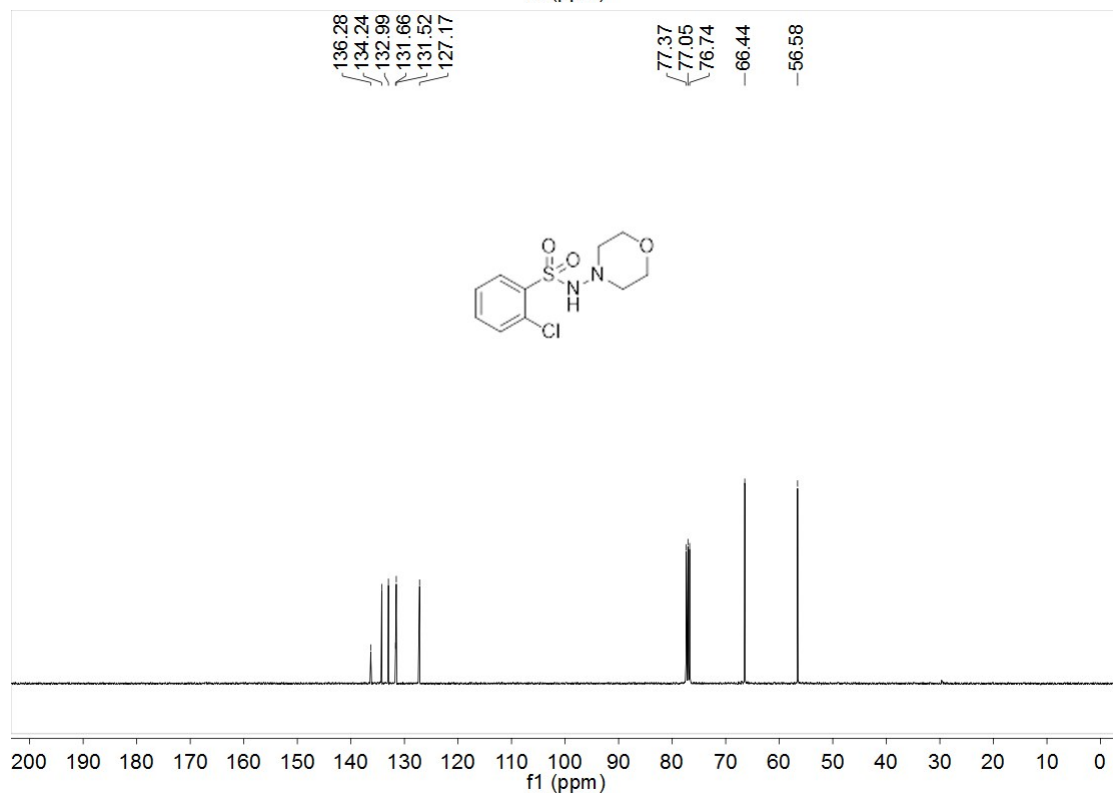
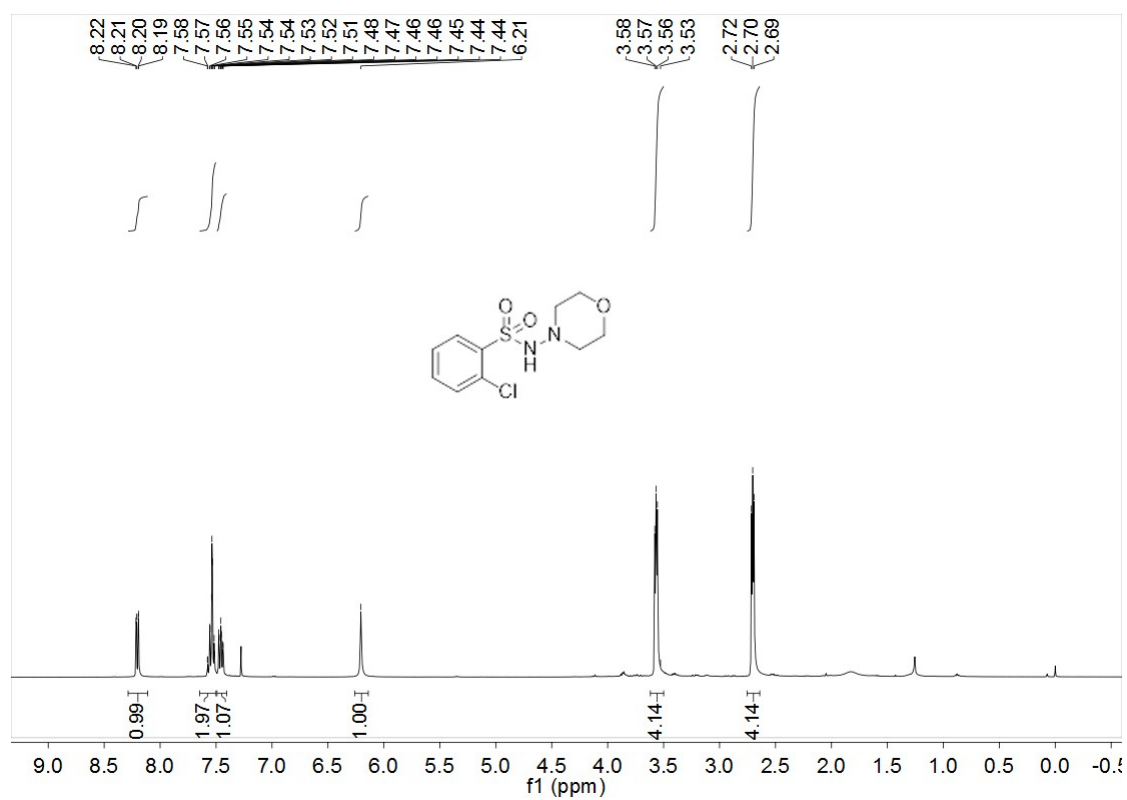
¹H and ¹³C NMR for **3ea**



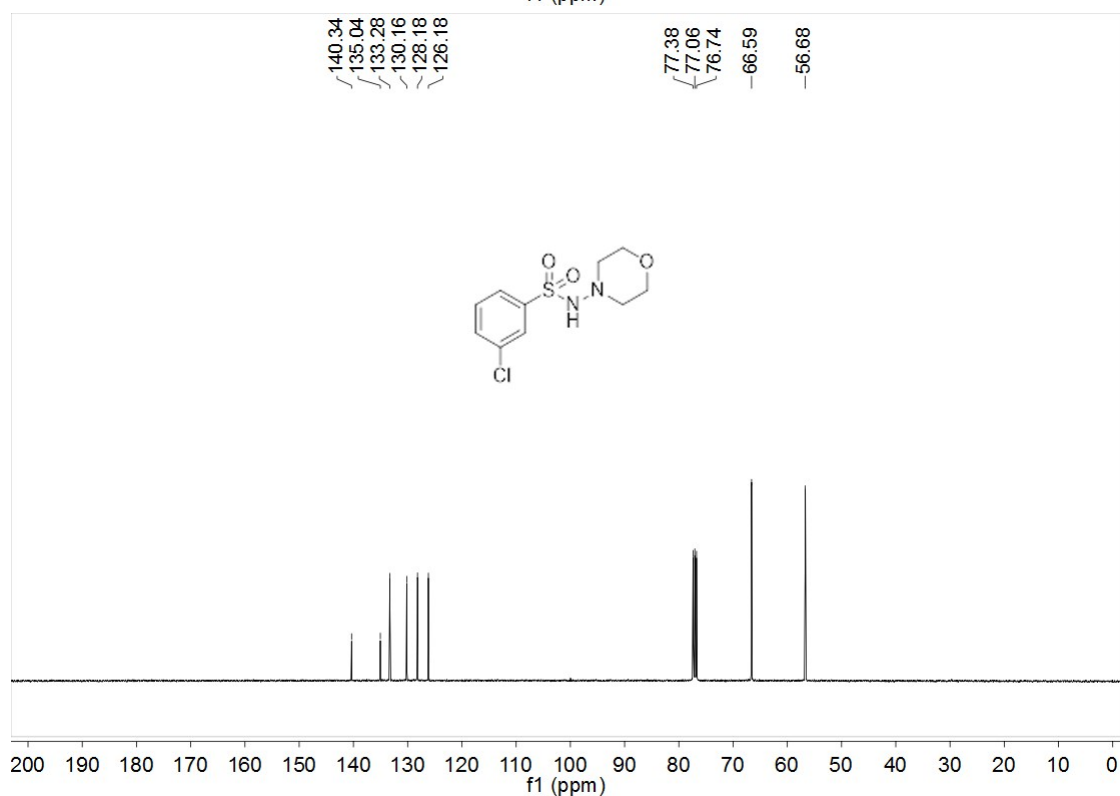
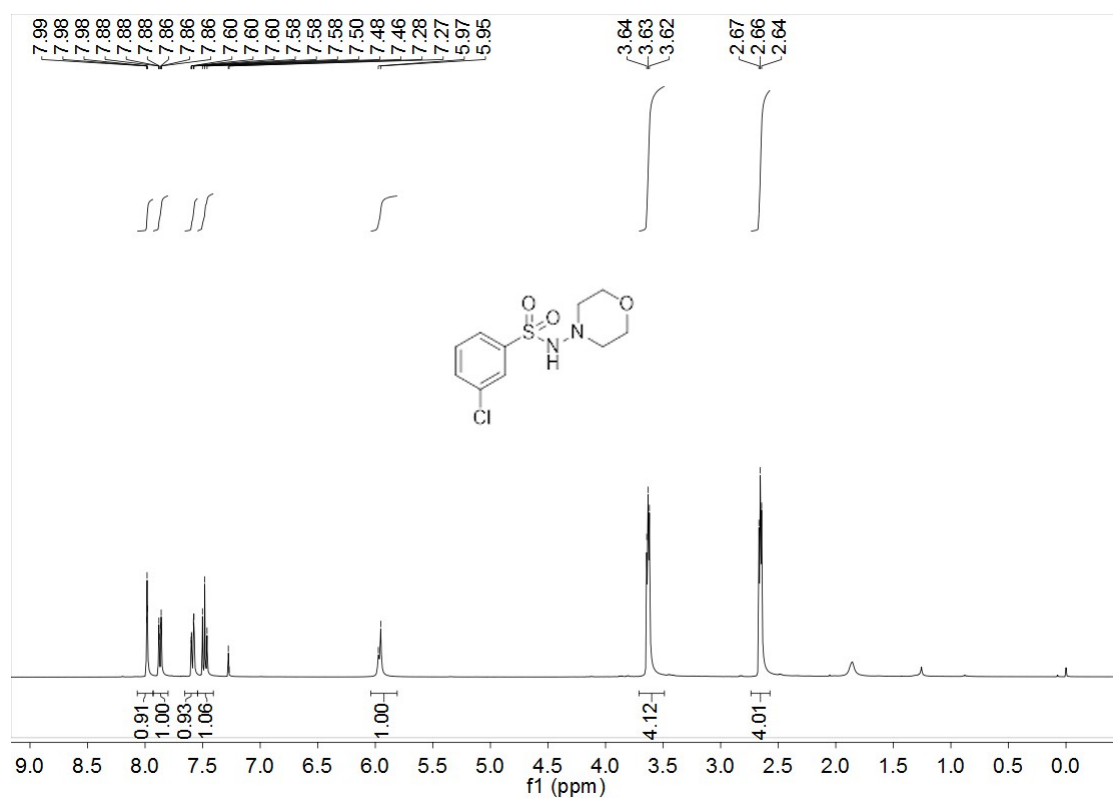
¹H and ¹³C NMR for **3fa**



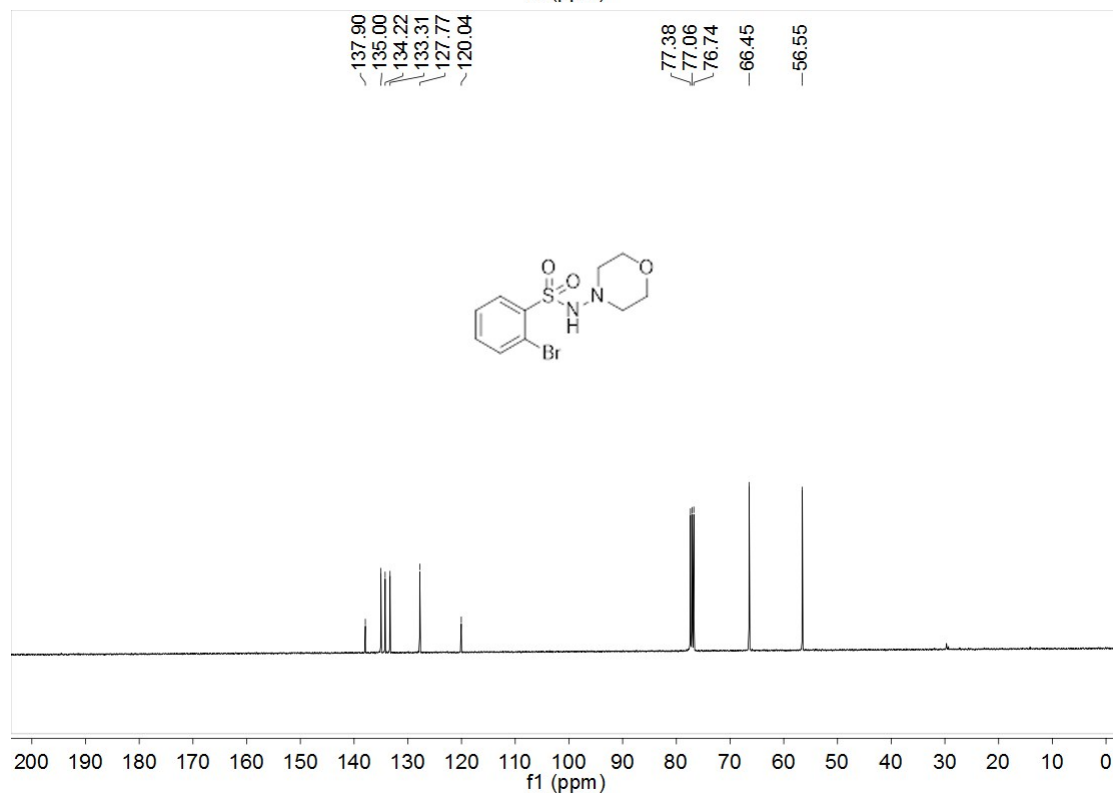
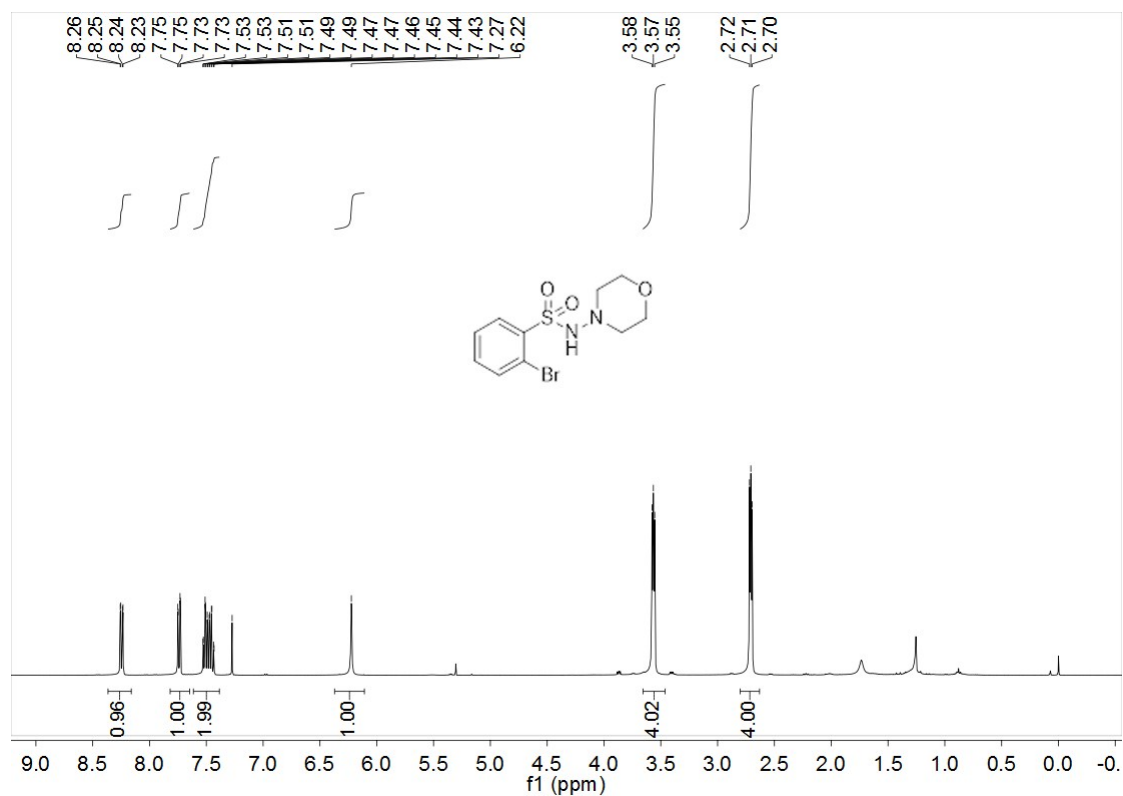
^1H and ^{13}C NMR for **3ga**



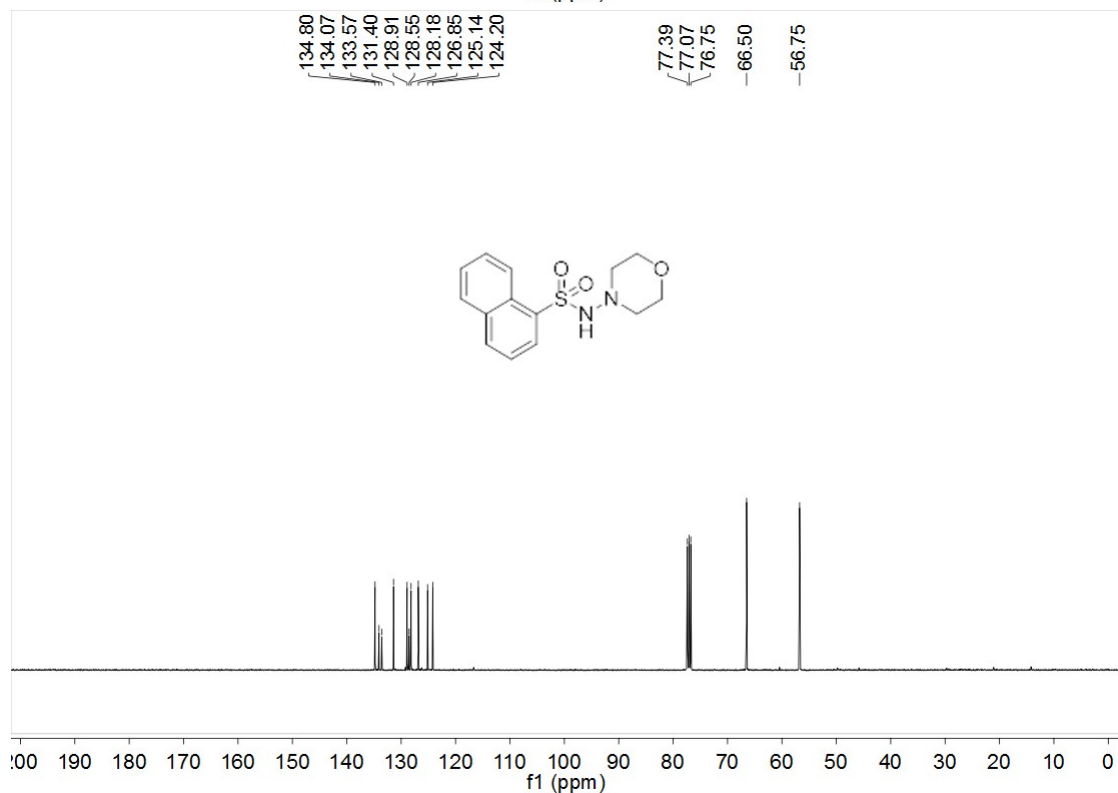
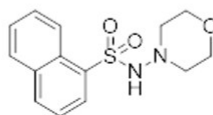
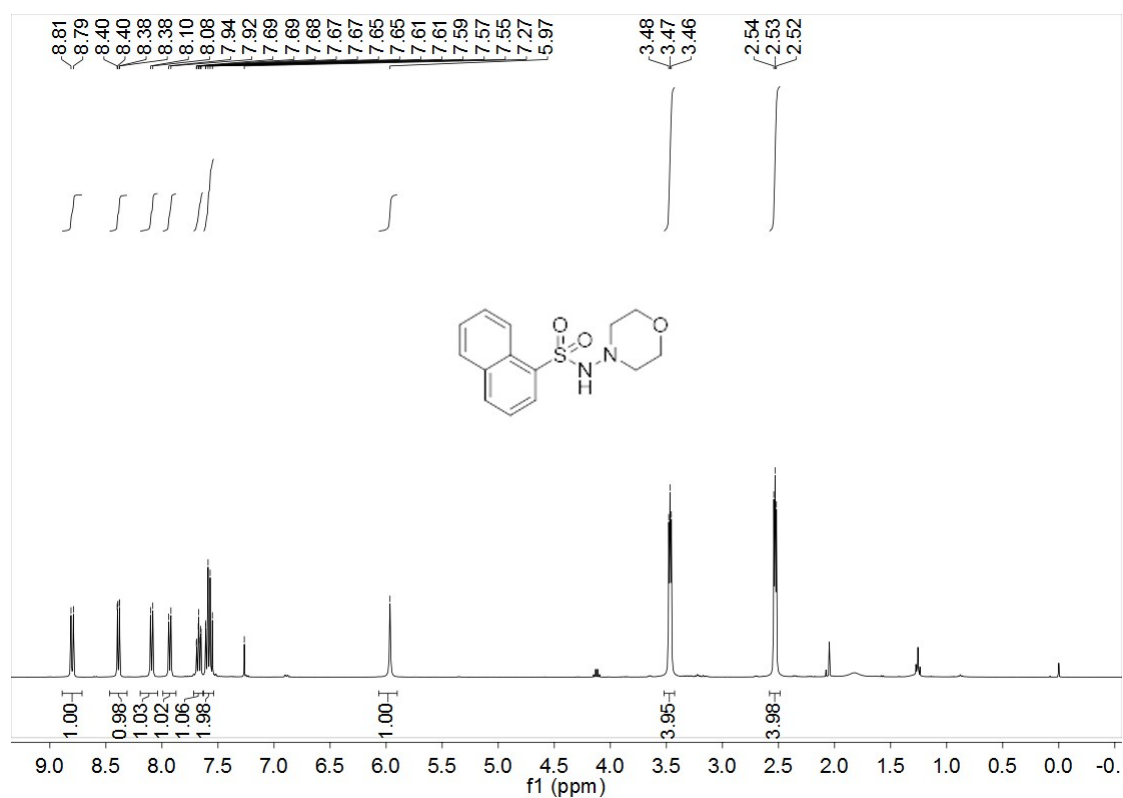
¹H and ¹³C NMR for **3ha**



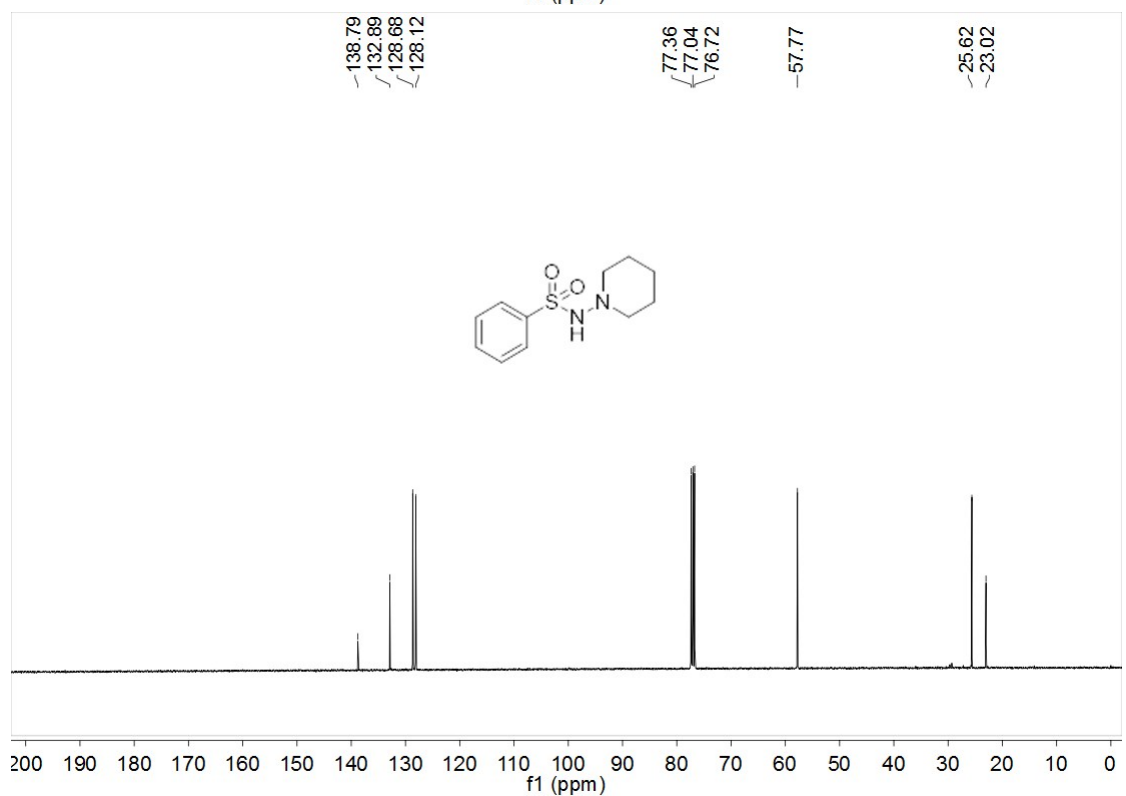
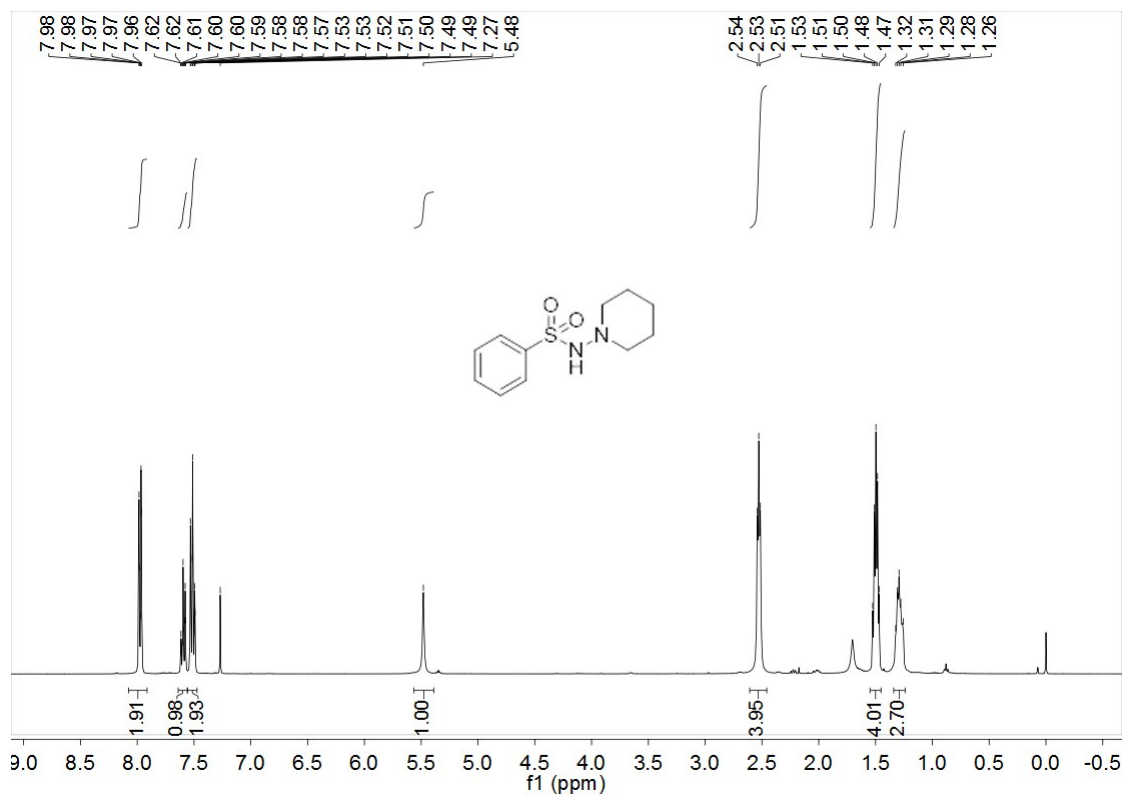
¹H and ¹³C NMR for **3ia**



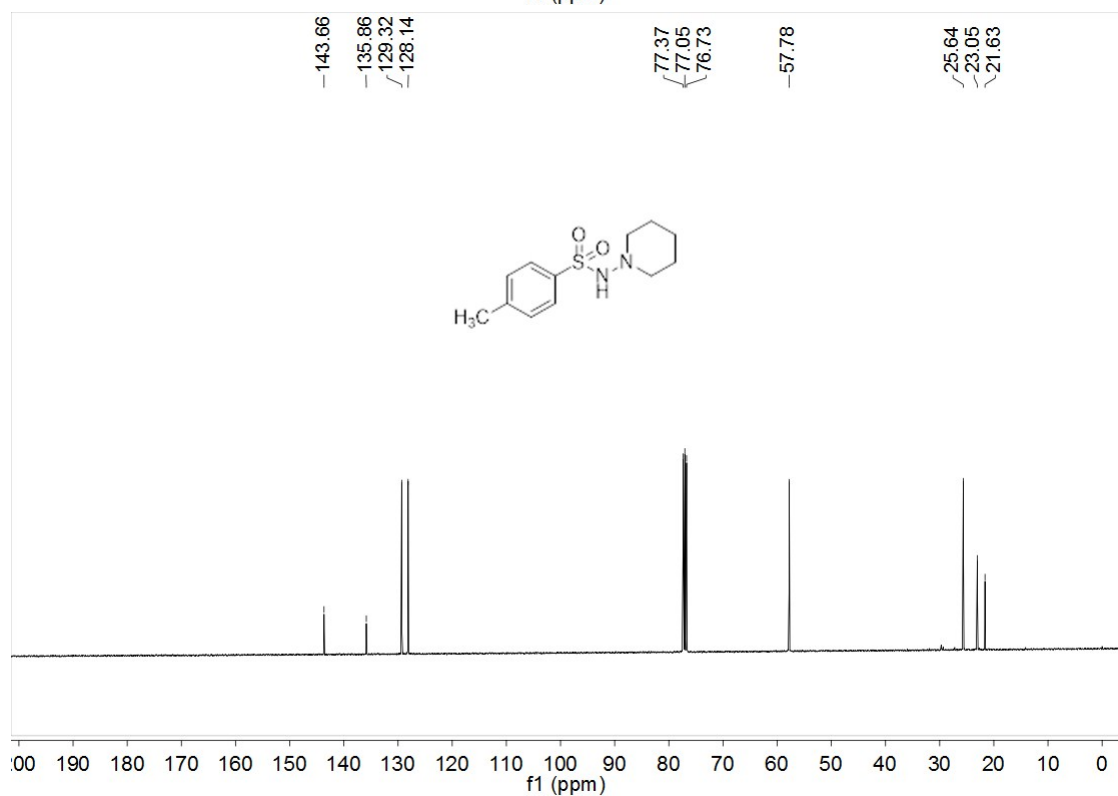
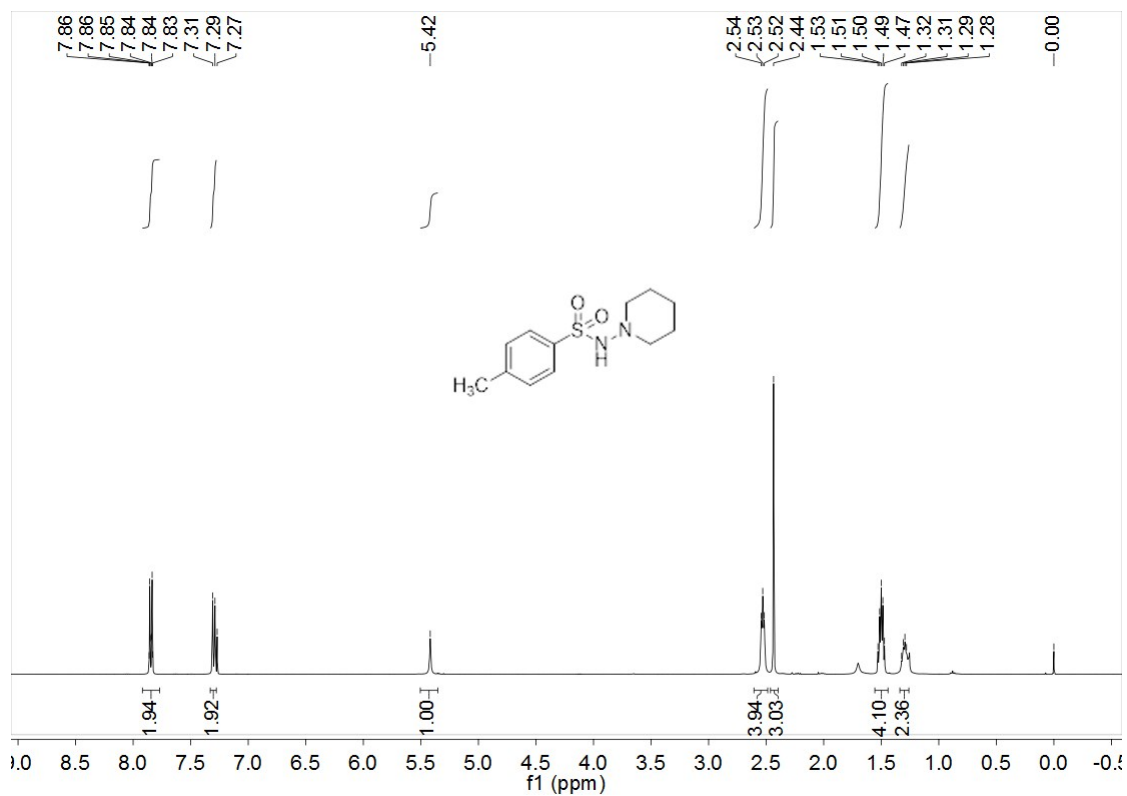
¹H and ¹³C NMR for **3ja**



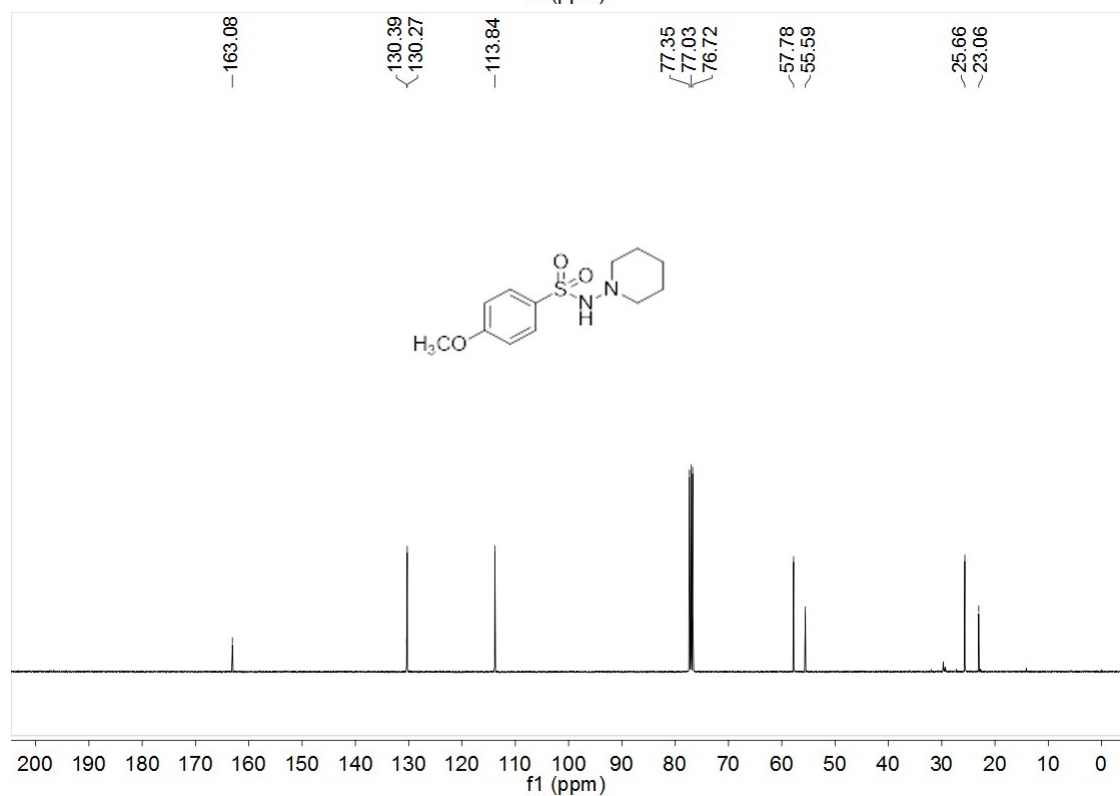
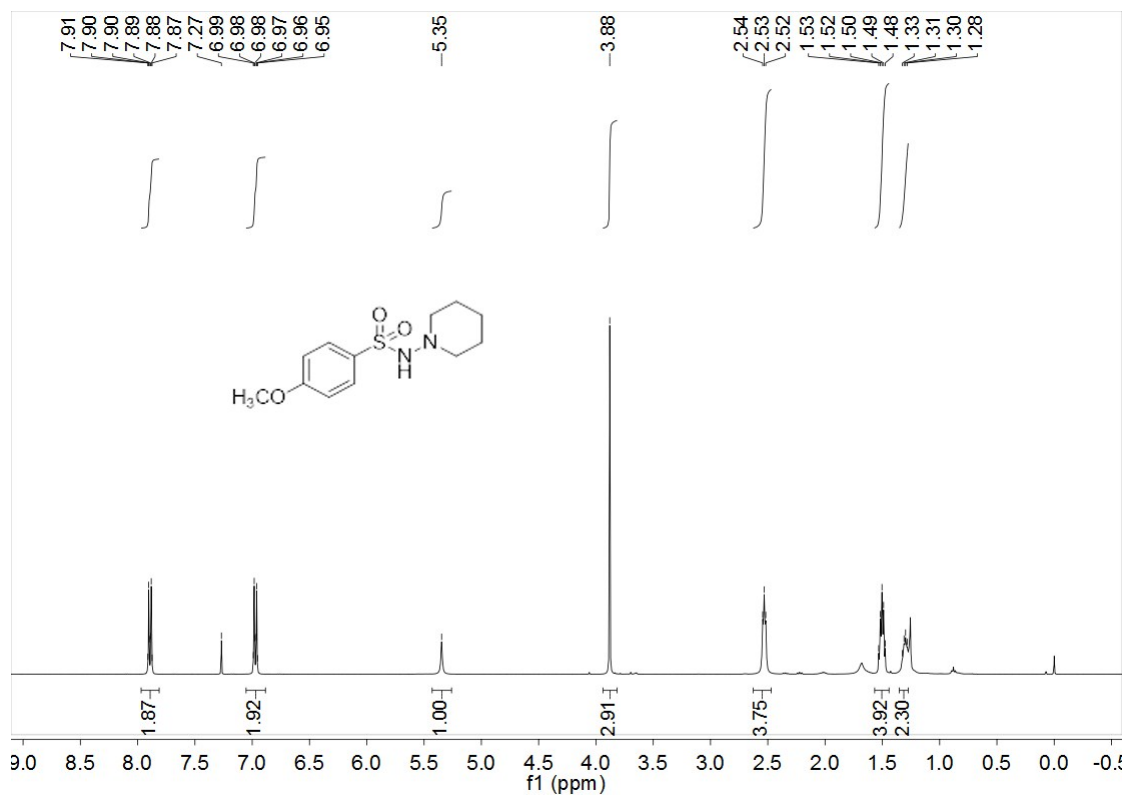
¹H and ¹³C NMR for **3ab**



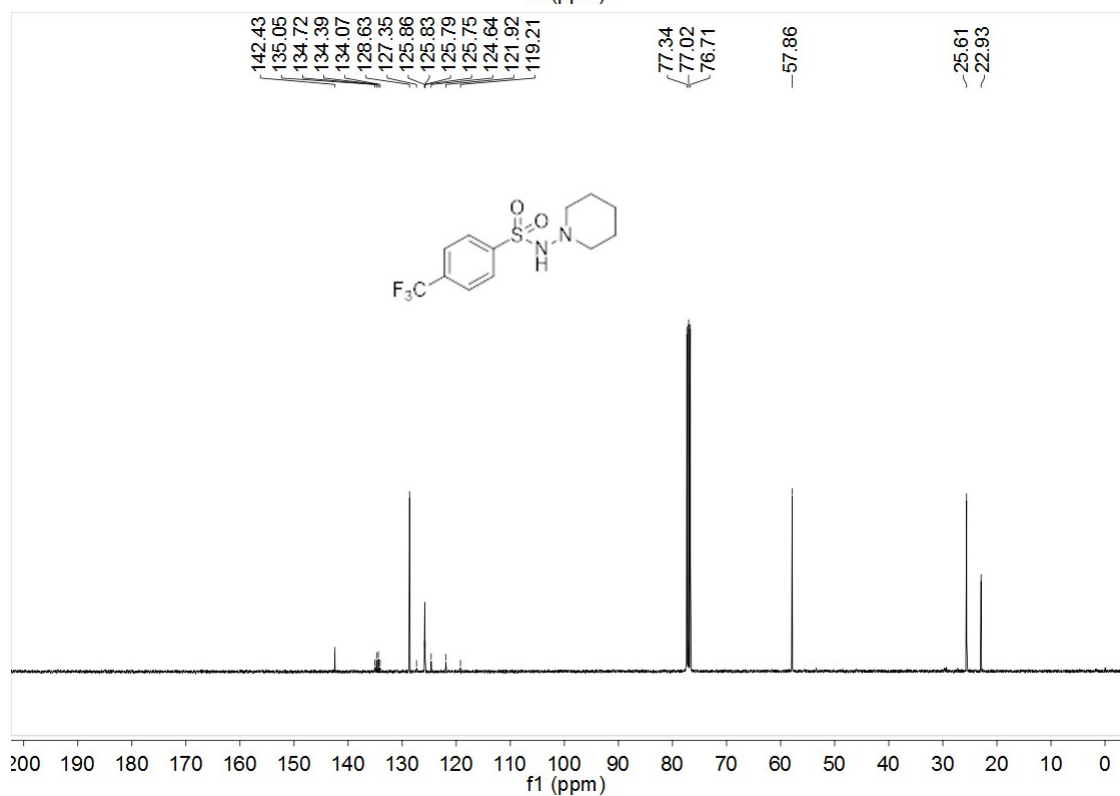
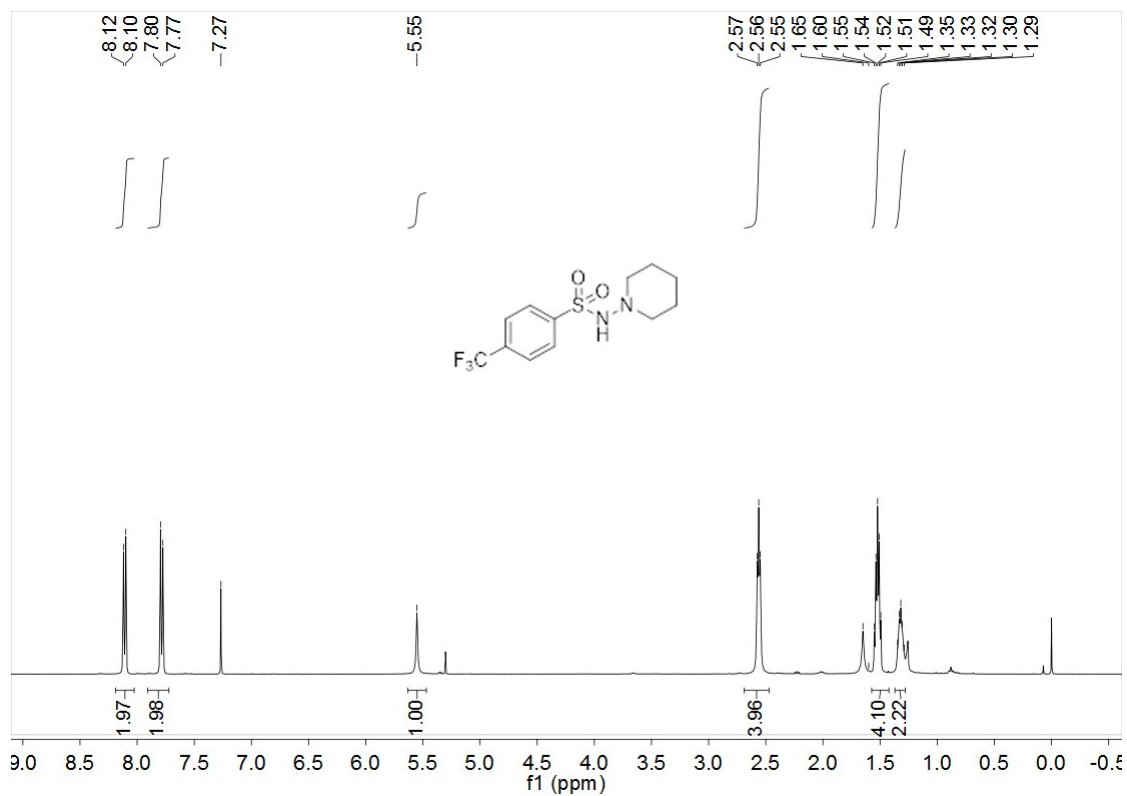
¹H and ¹³C NMR for **3ac**



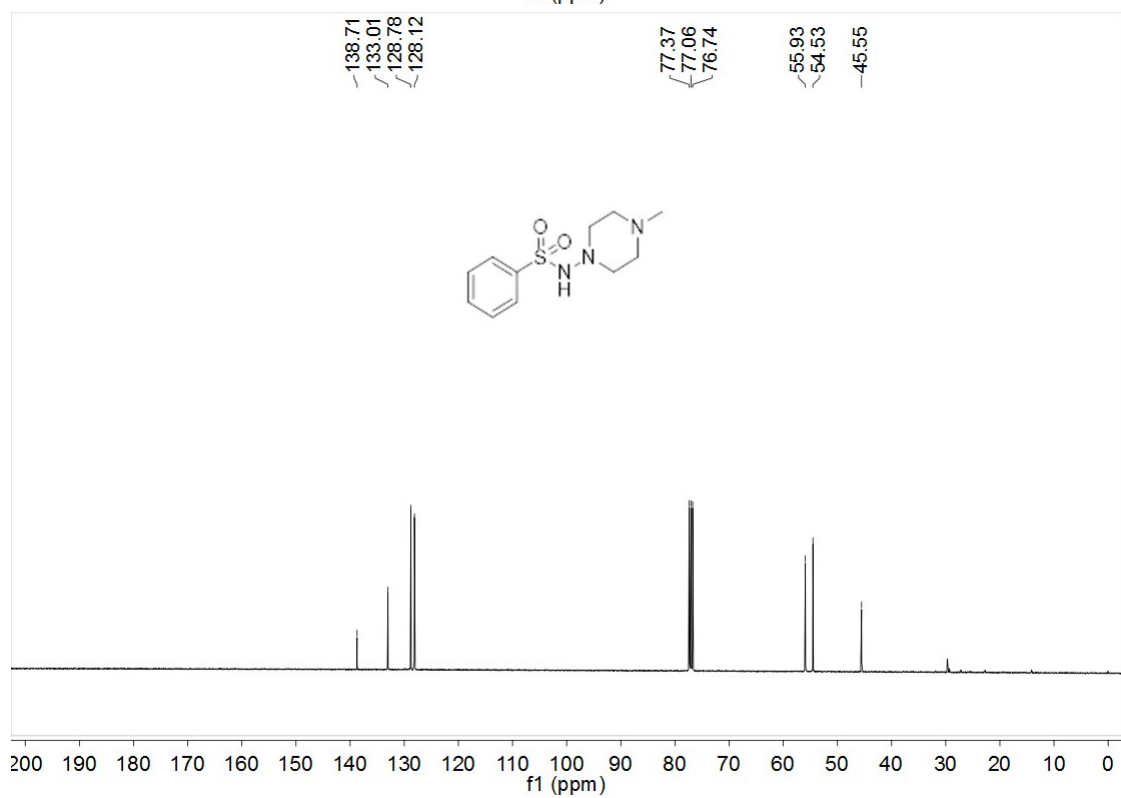
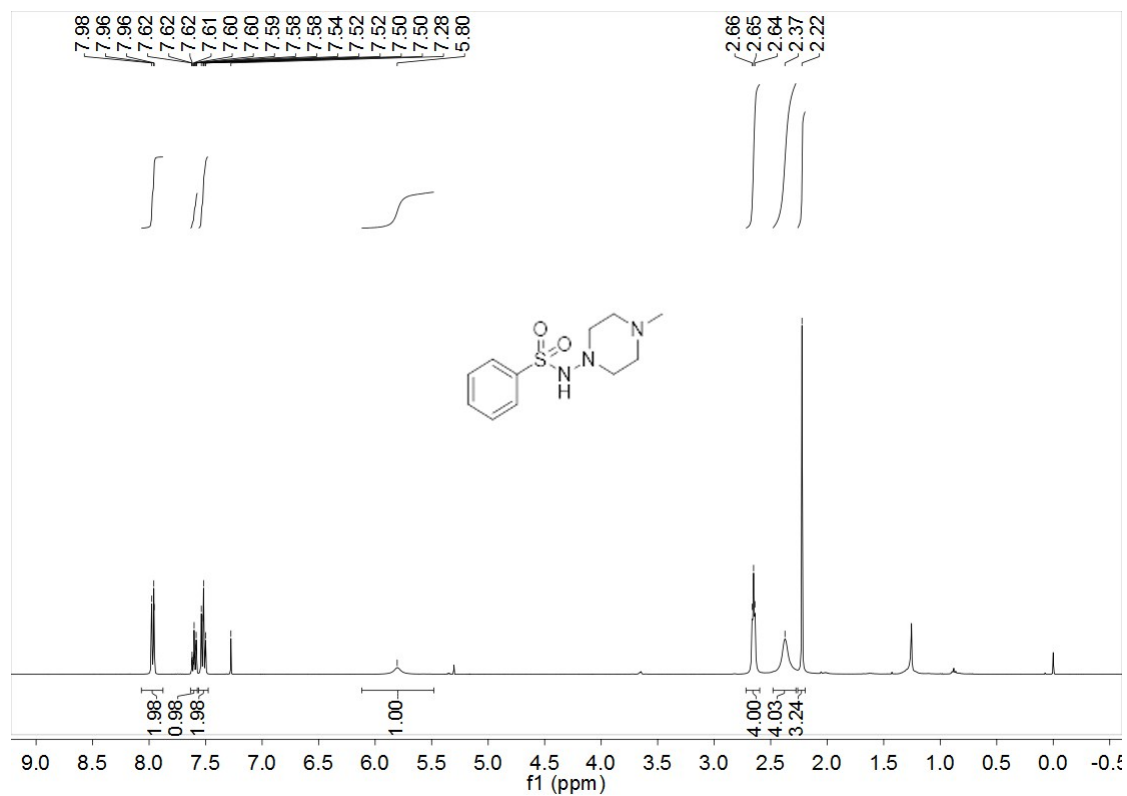
¹H and ¹³C NMR for **3ad**



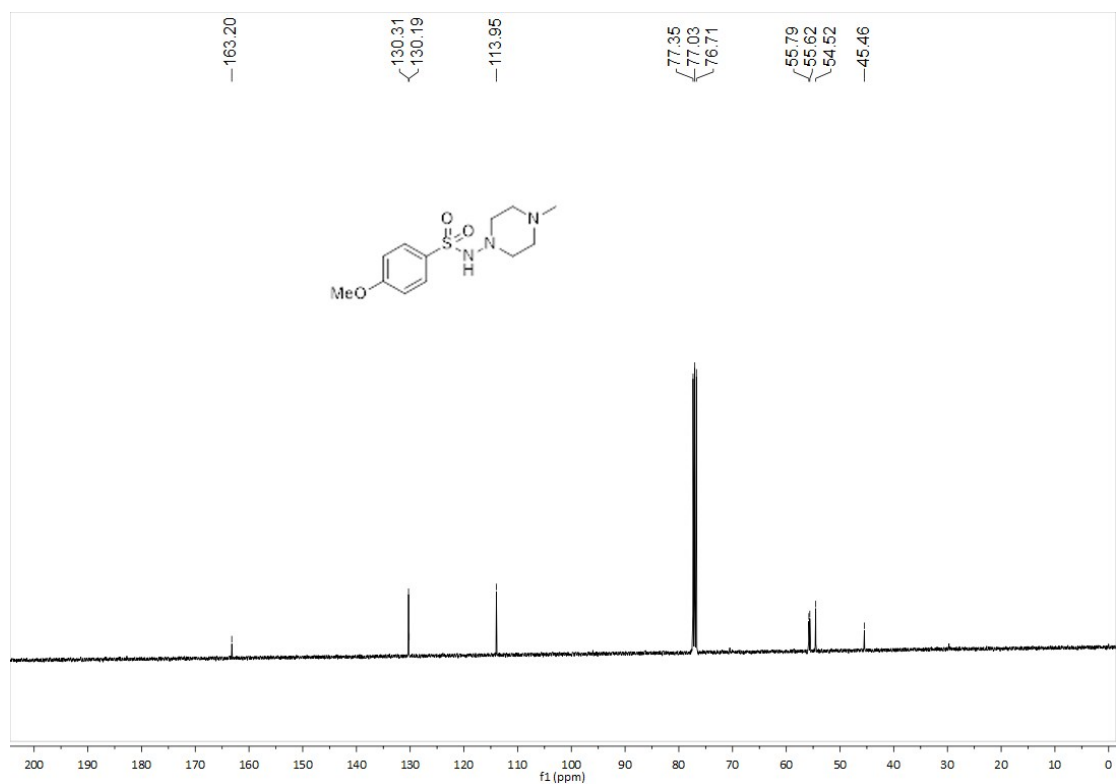
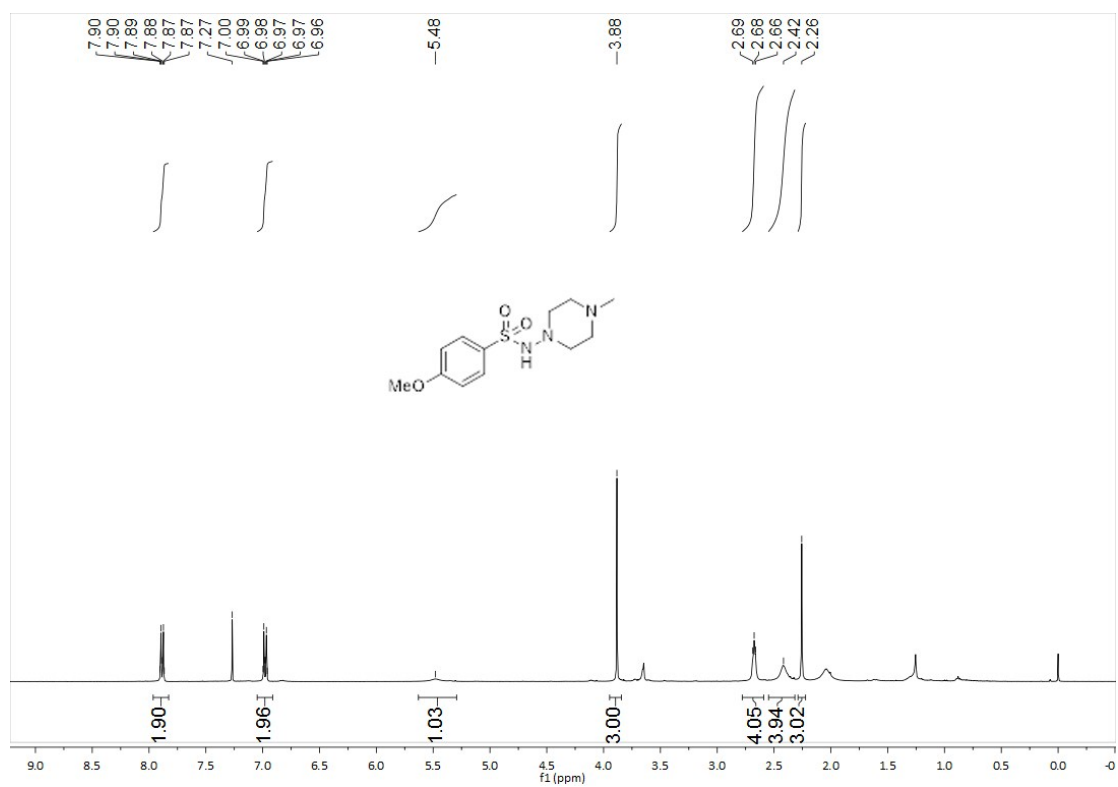
¹H and ¹³C NMR for **3ae**



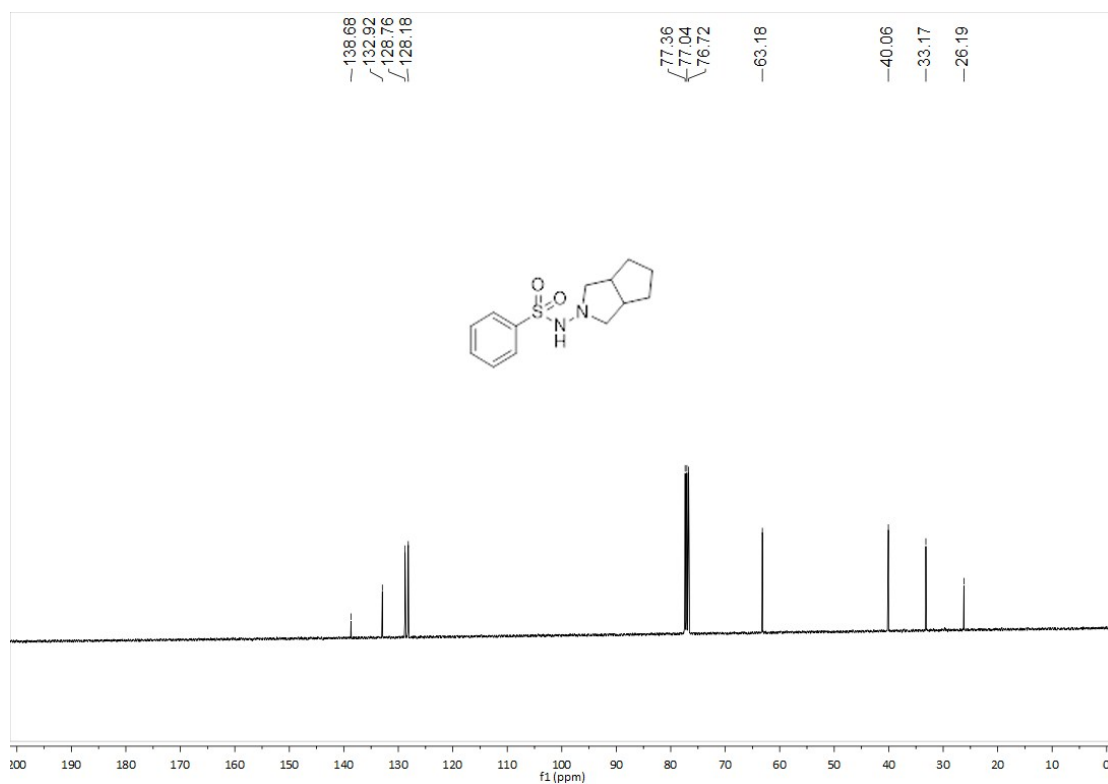
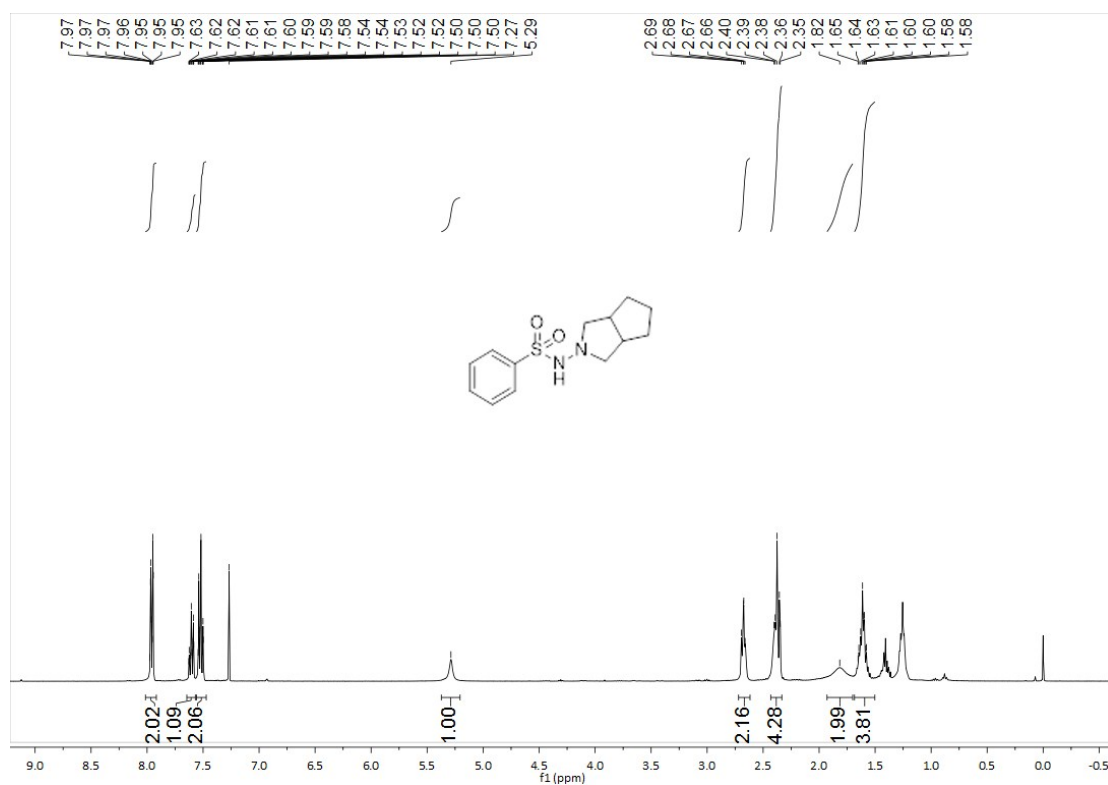
¹H and ¹³C NMR for **3af**



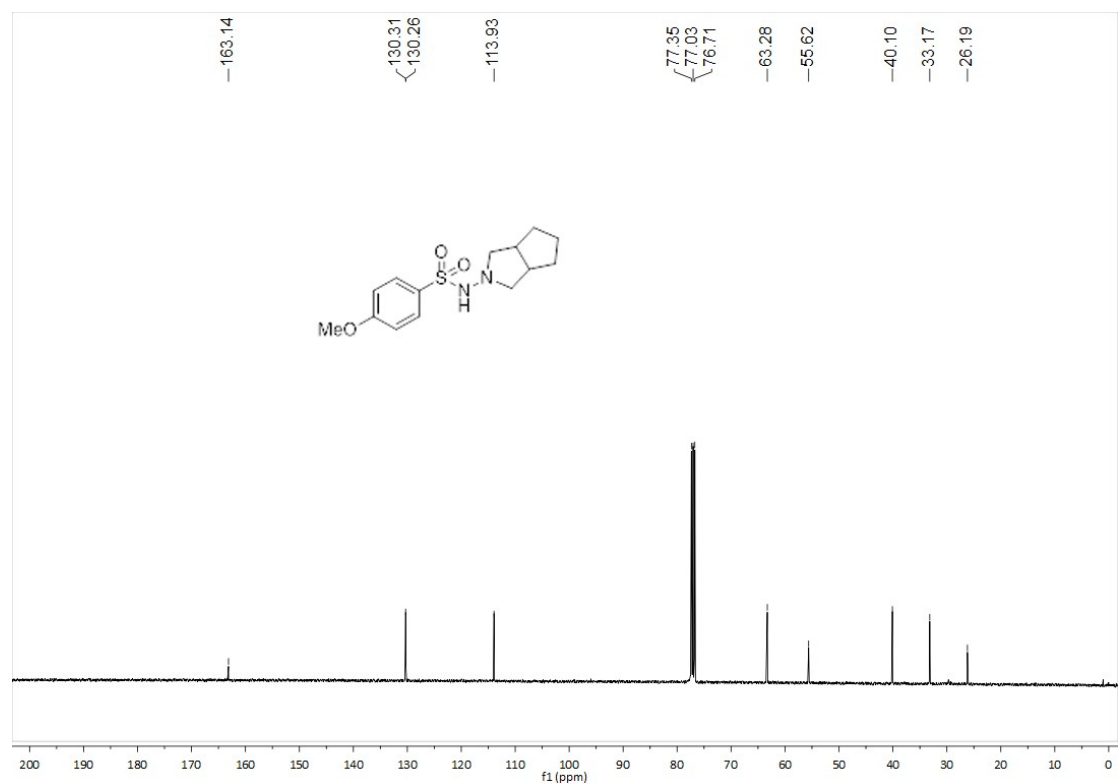
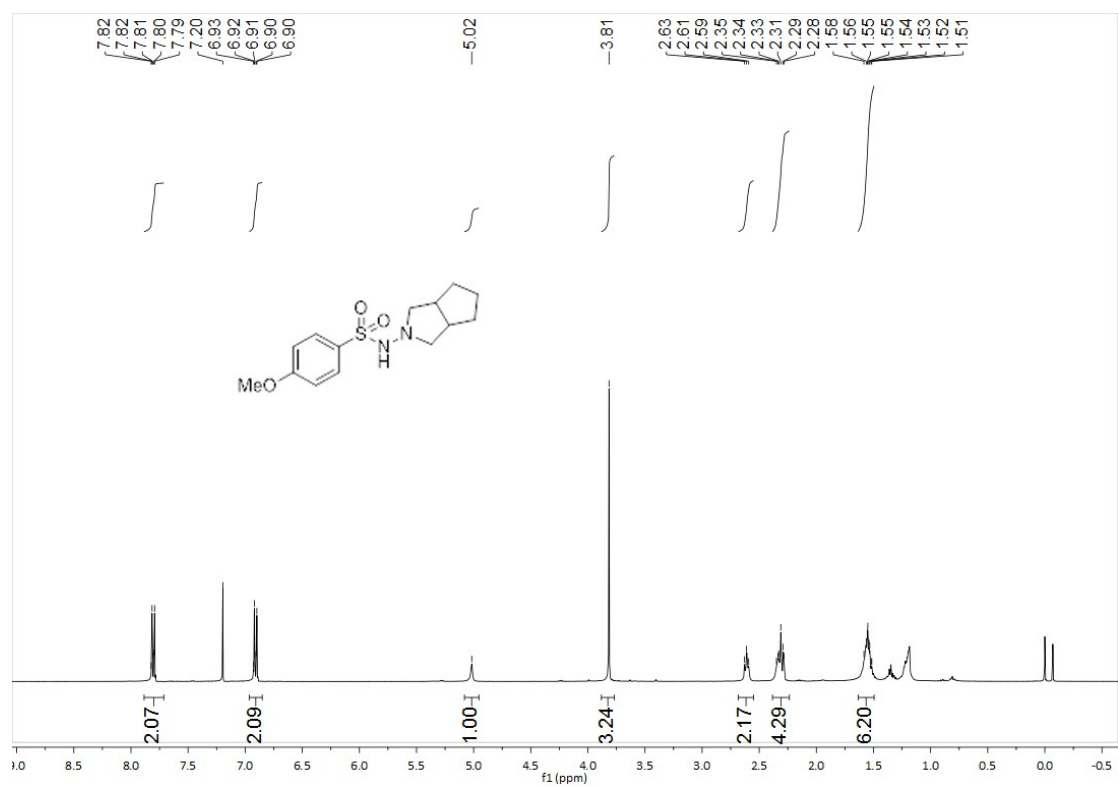
¹H and ¹³C NMR for **3ag**



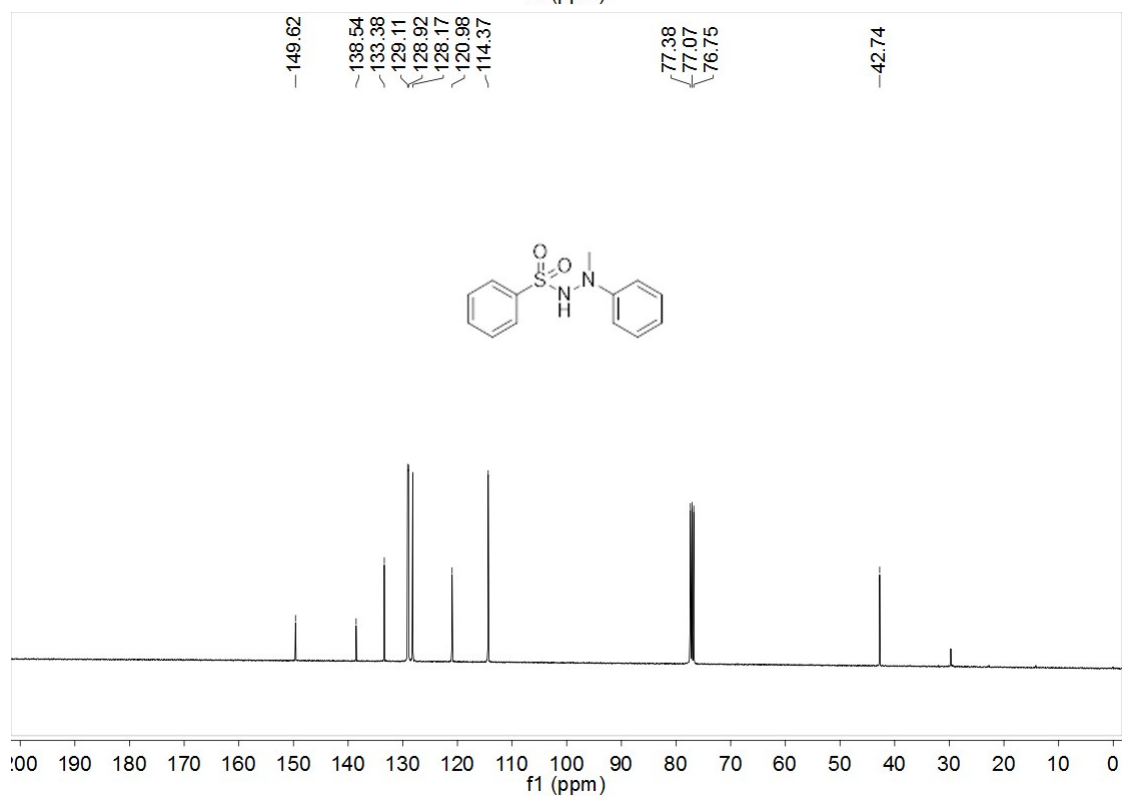
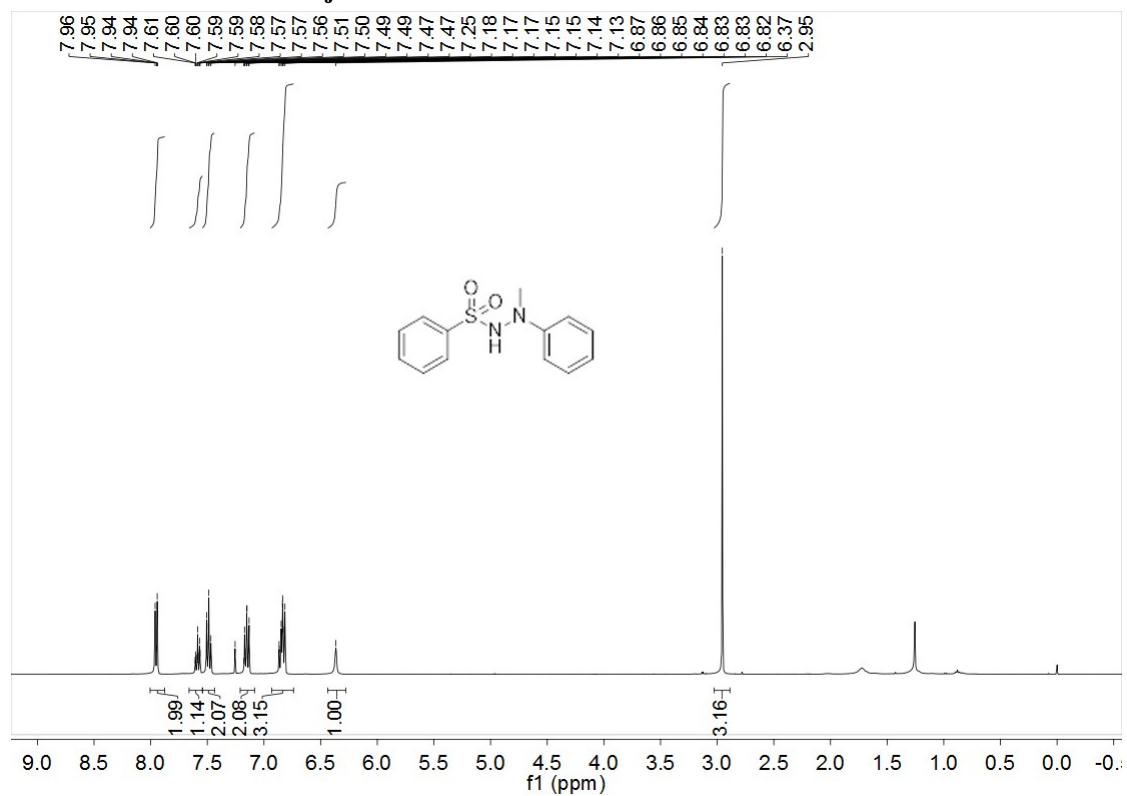
¹H and ¹³C NMR for 3ah



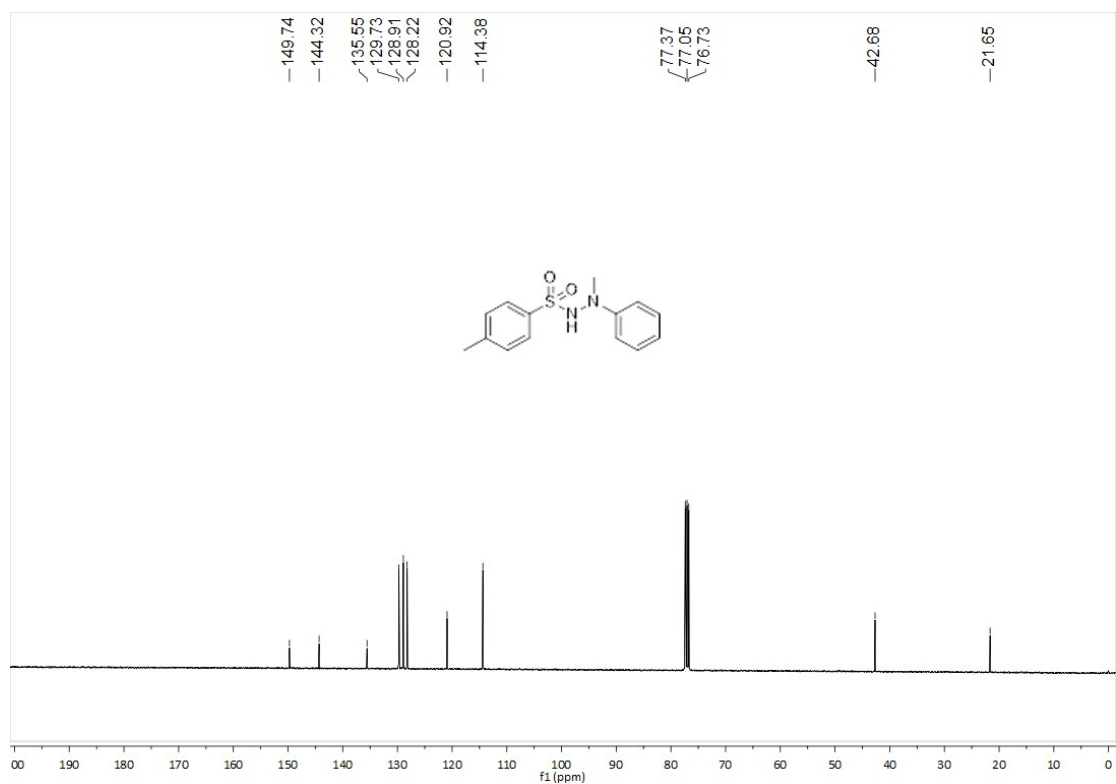
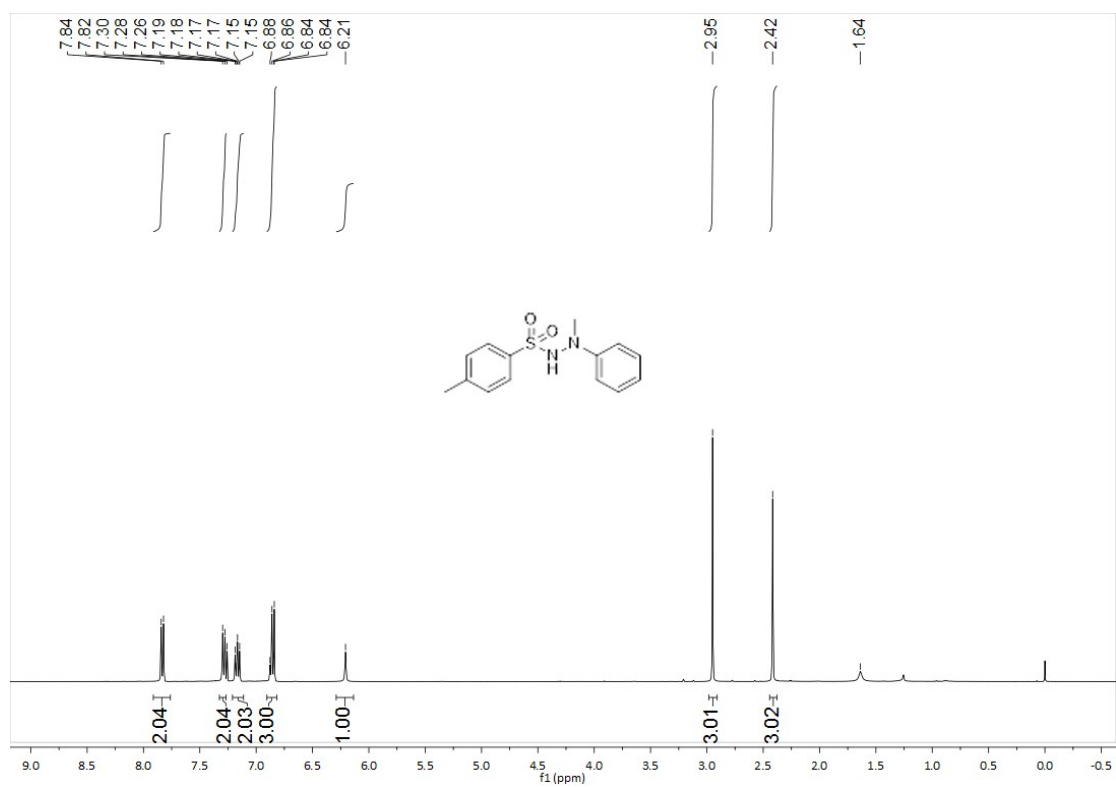
¹H and ¹³C NMR for 3ai



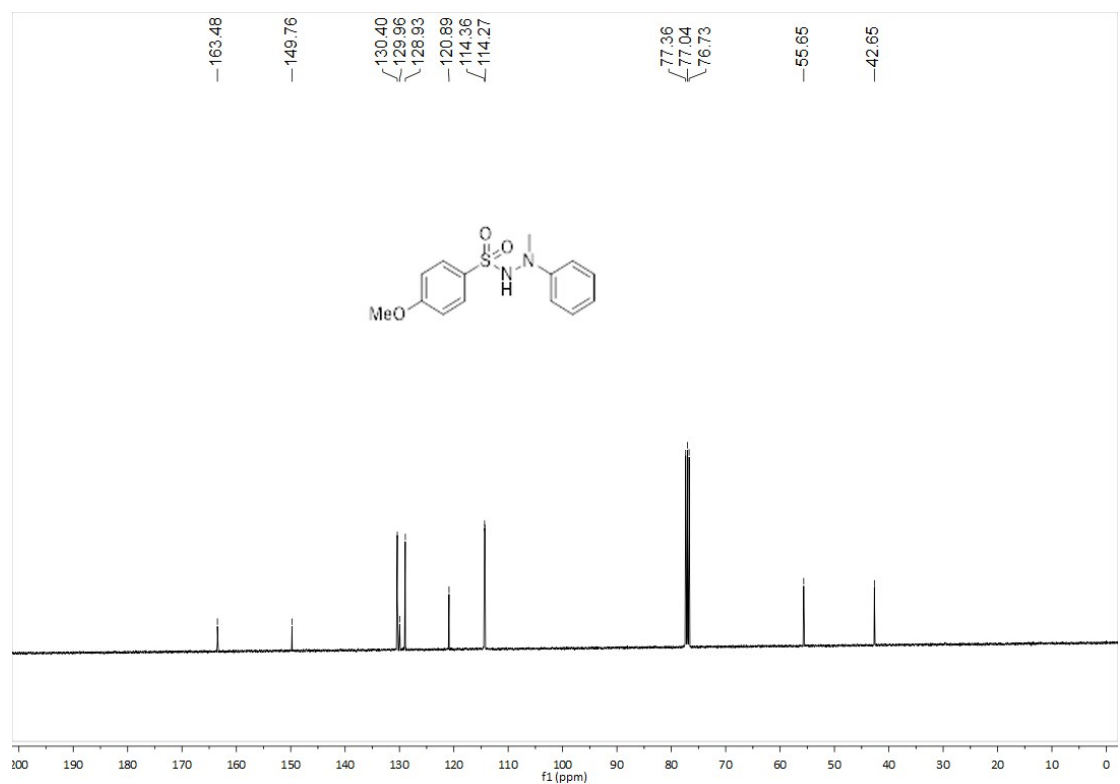
¹H and ¹³C NMR for 3aj



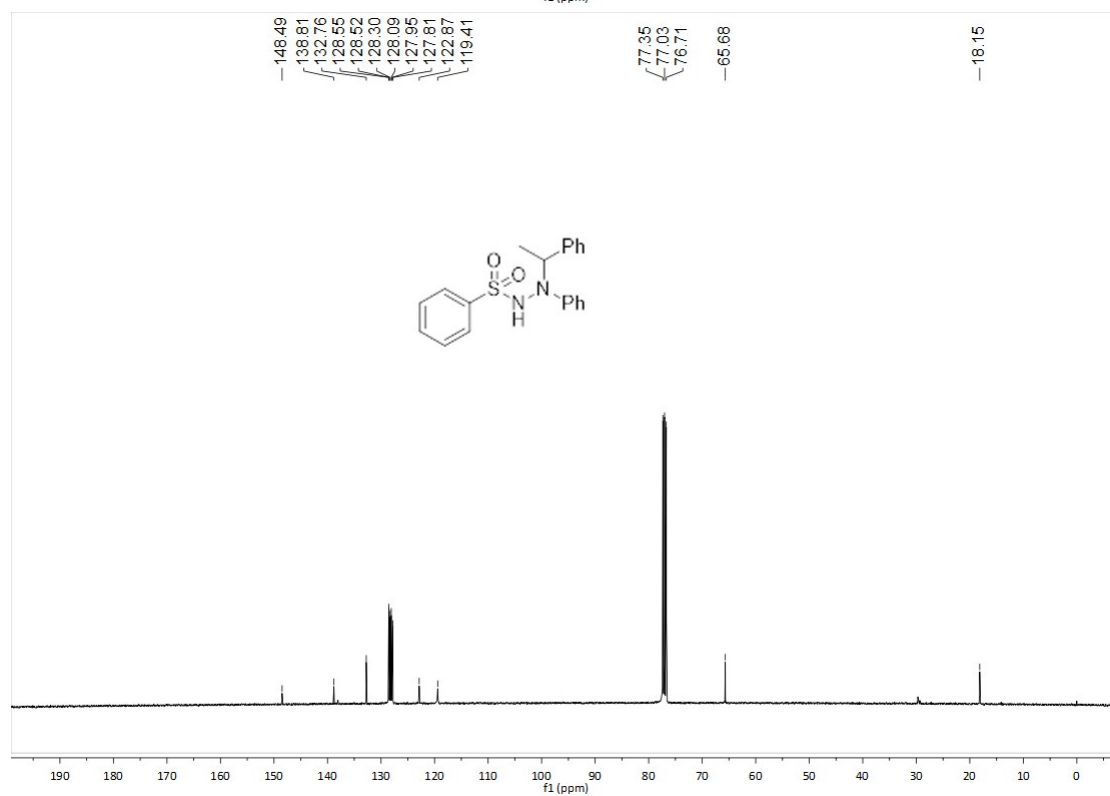
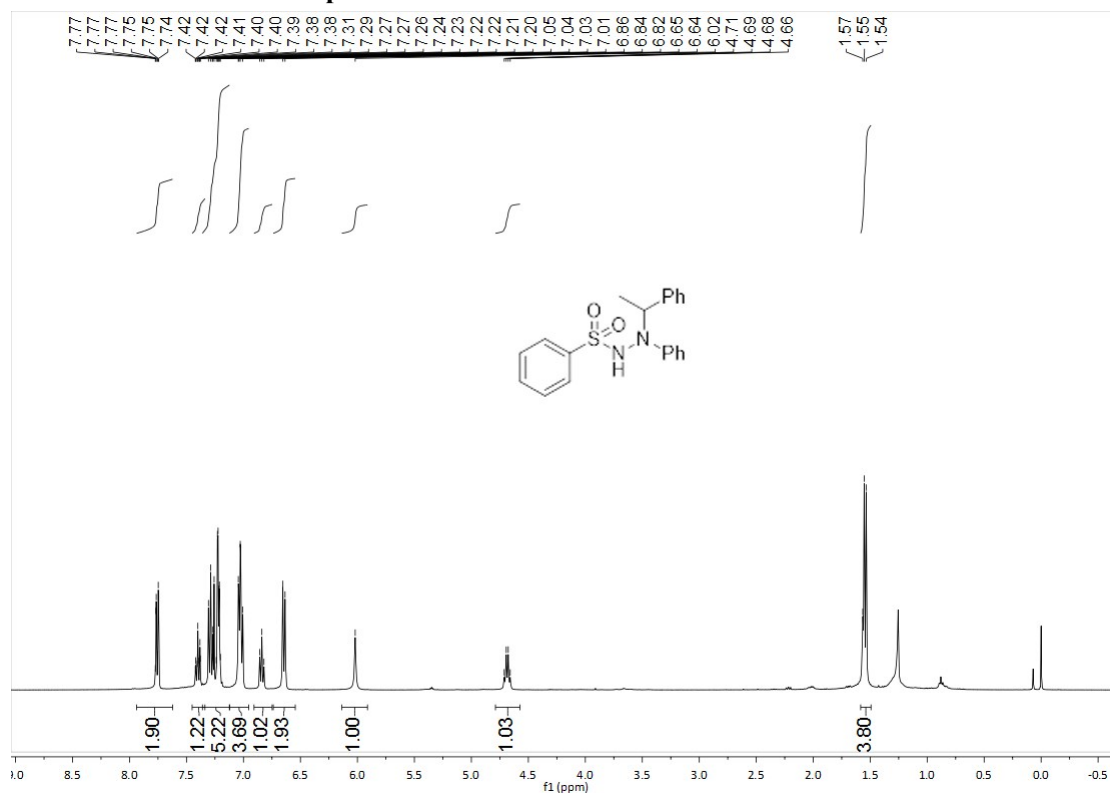
¹H and ¹³C NMR for **3ak**



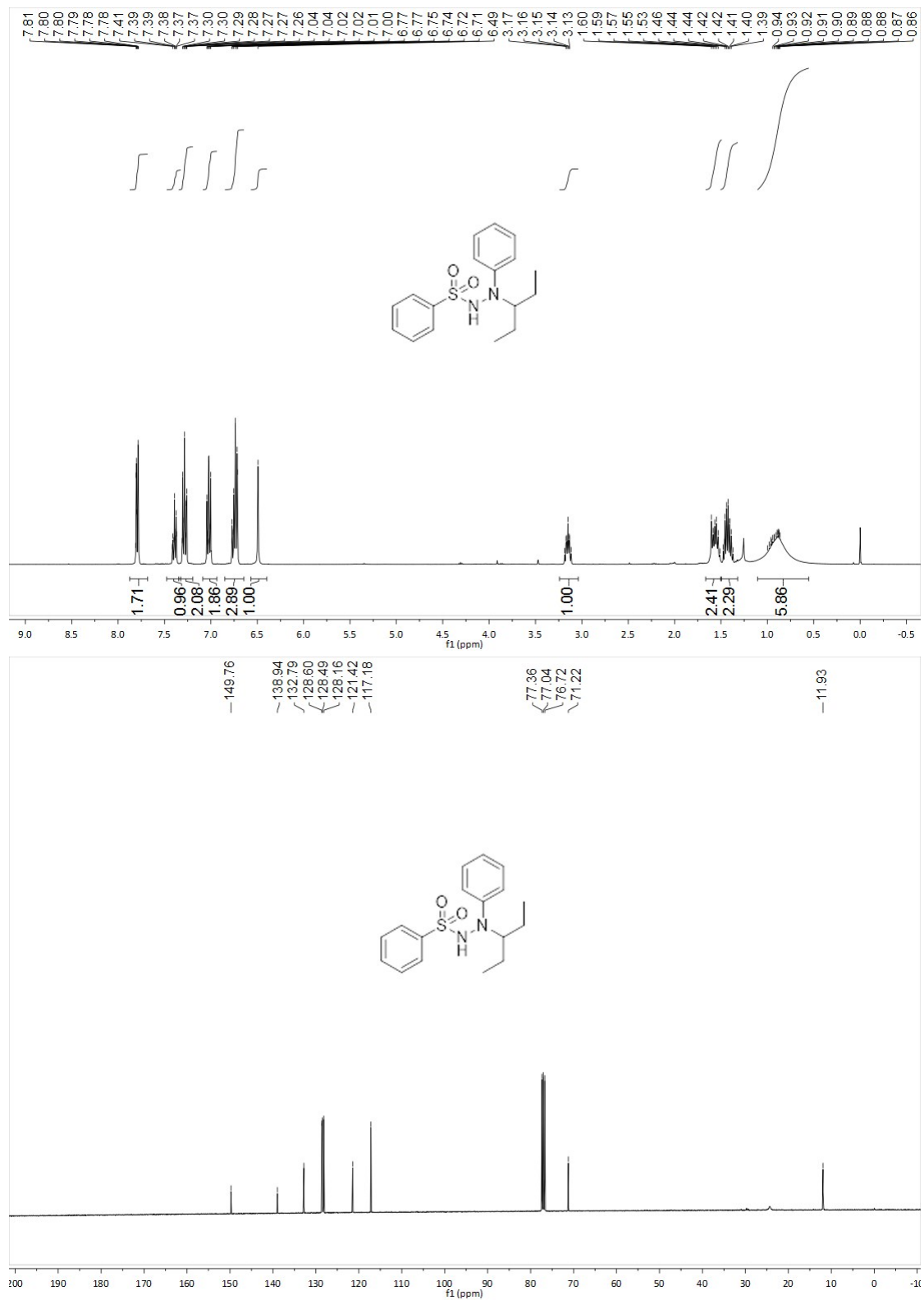
¹H and ¹³C NMR for **3a**



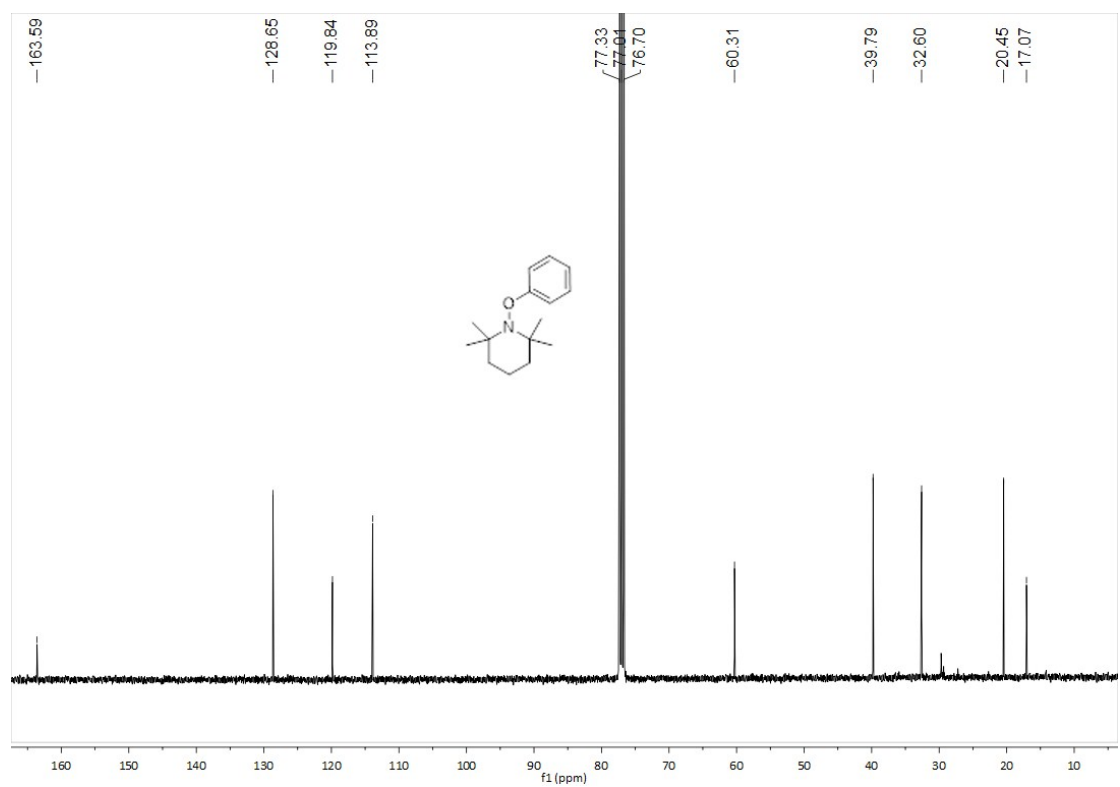
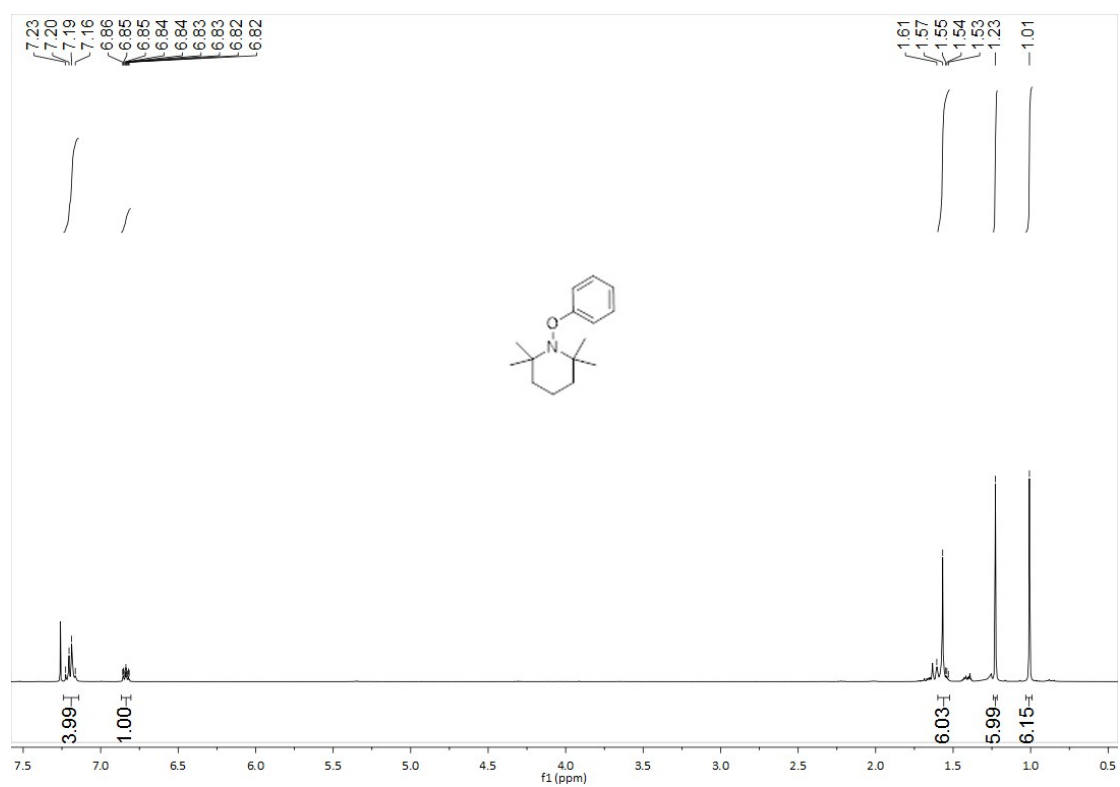
¹H and ¹³C NMR for **3ap**



¹H and ¹³C NMR for **3ar**



¹H and ¹³C NMR for 4



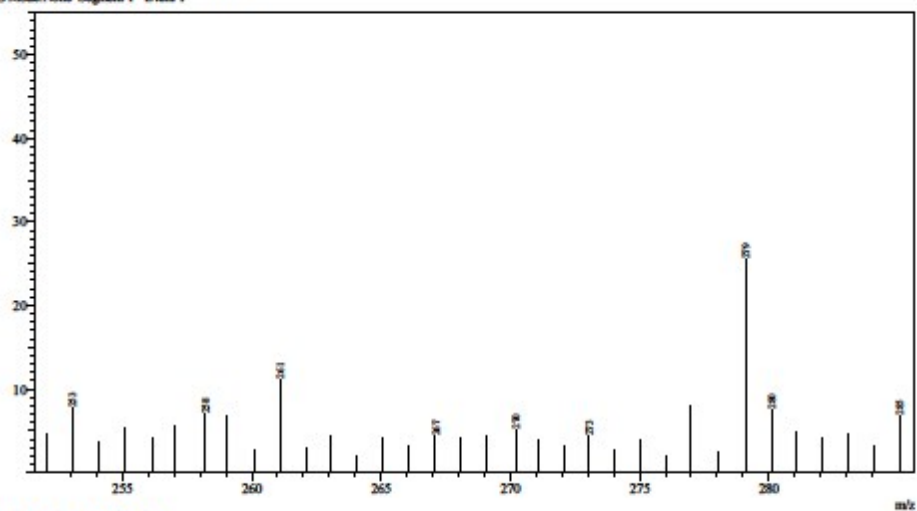
MS for **5** (calcd for C₁₃H₂₇N₃O₂⁺ [M+H]⁺ 258, found 258)

2016-9-5 21:36:34 1 / 1

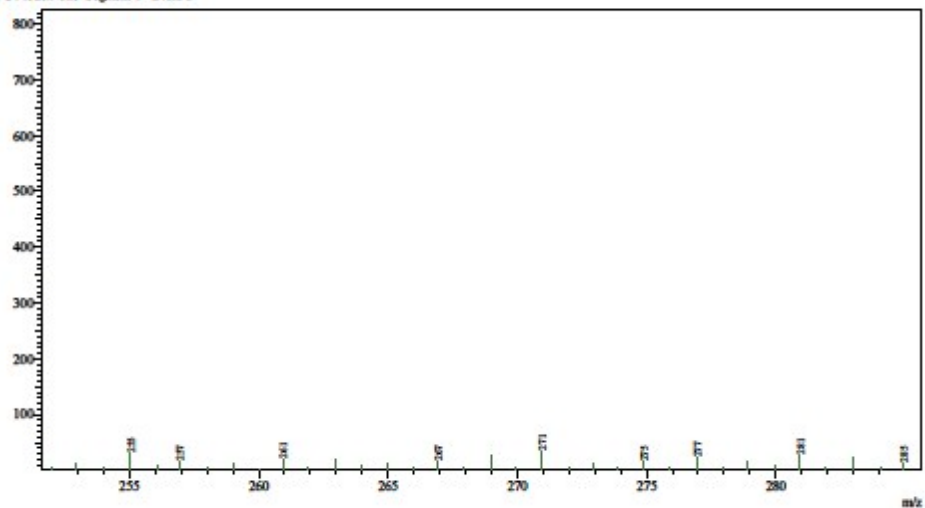
==== Shimadzu LabSolutions Data Report ====

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MassPeaks: 100
RawMode: Single 0.000(1) BasePeak: 203(157412)
BG Mode: None Segment 1 - Event 1



Line# 2 R. Time: 0.017(Scan# 2)
MassPeaks: 101
RawMode: Single 0.017(2) BasePeak: 293(10488)
BG Mode: None Segment 1 - Event 2



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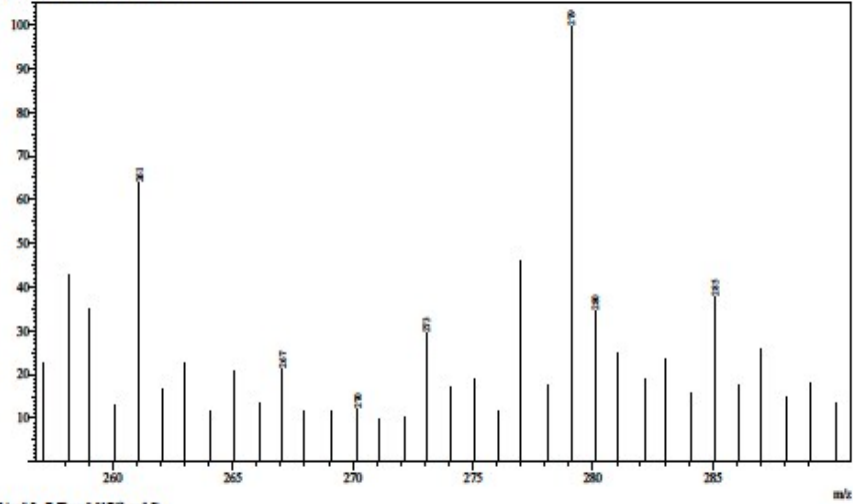
MS for **6** (calcd for $C_{15}H_{25}N_3ONa^+$ $[M+Na]^+$ 286, found 286)

2016-9-5 21:31:16 1 / 1

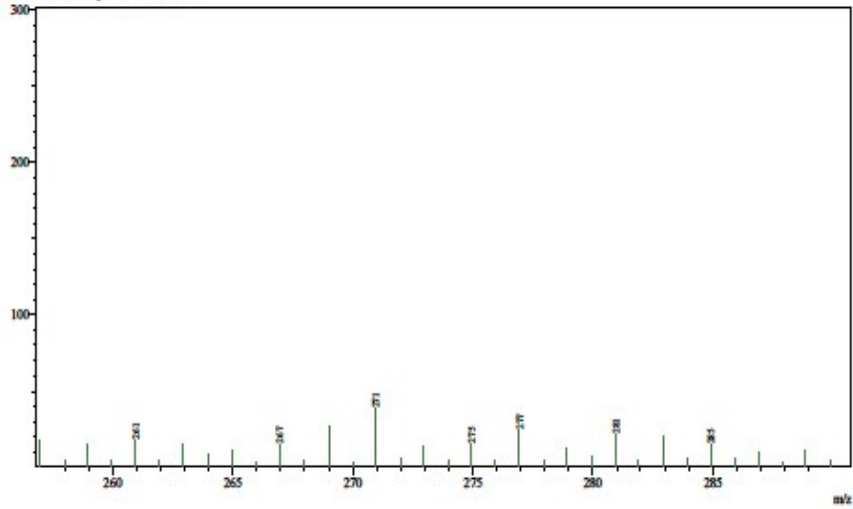
==== Shimadzu LabSolutions Data Report ====

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BG Mode: None Segment 1 - Event 2



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