

T3P Facilitated One Pot Multicomponent Reaction Comprising Unique Intramolecular Rearrangement

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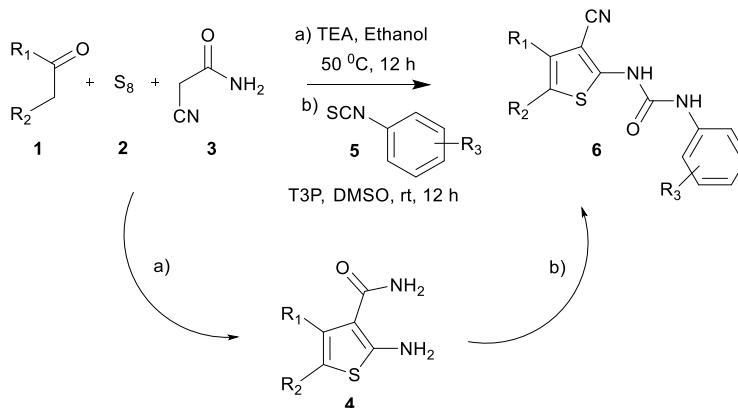
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1. General Experimental Details:

All the reagents, chemicals and solvents used were from Sigma Aldrich and Sd-fine chemicals and were used without additional purification. The instrumental techniques employed for the characterization of the newly synthesized compounds include ^1H and ^{13}C NMR and HRMS. ^1H and ^{13}C NMR spectra were recorded on Agilent 400 MHz spectrometer in DMSO-d_6 solution using tetramethylsilane (TMS) as internal standard. Chemical shifts were recorded in ppm relative to TMS. Column chromatography was performed on silica gel 60 (60–120 mesh) and thin layer chromatography was performed on TLC plates (Merck, silica gel 60 F254). The mobile phases employed for column chromatography and TLC were hexane and ethyl acetate in 9:1 ratio.

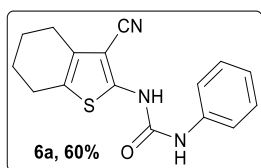
2. General Procedure for the synthesis of 1-(3-cyanothiophen-2-yl)-3-phenylurea derivatives

(6a-6t).



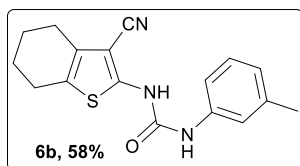
The mixture of methylene active-carbonyl compound (**1**) (1 mmol), S_8 (**2**) (1 mmol, 0.256 g) and 2-cyanoacetamide (**3**) were allowed to stir in ethanol solvent (5 ml) in a round bottomed flask at 50 °C for 12 h on an oil bath. After that, the ethanol solvent was evaporated under vacuum. The formed 2-aminothiophene-3-carboxamide (**4**) in the previous step was carried further without any purification and treated with 1 mmol each of phenyl isothiocyanate derivative (**5**) and T3P (0.704 g) in DMSO solvent and stirred for 12 h at room temperature. The contents of the flask was diluted with 30 ml of ethyl acetate and transferred to 50 ml of water taken in separating funnel, shook well and allowed separate the water and ethyl acetate layer. The ethyl acetate extract was separated and dried over anhydrous sodium sulphate then the solvent was removed under reduced pressure to afford corresponding 1-(3-cyanothiophen-2-yl)-3-phenylurea (**6**). The crude residue thus obtained was purified by column chromatography over silica gel using ethyl acetate in hexane as eluent to get pure 1-(3-cyanothiophen-2-yl)-3-phenylurea (**6**).

3. Characterization details of the synthesized Products



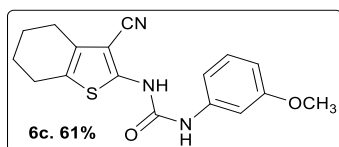
1-(3-cyano-4,5,6,7-tetrahydrobenzo[b]thiophen-2-yl)-3-phenylurea (**6a**)

Yield: 177 mg, 60 %; MP. >250 °C; White solid; ¹H-NMR (400 MHz, DMSO-d₆): δ ppm: 1.715 (s, 4H, -CH₂-), 2.440 (s, 2H, -CH₂-), 2.531 (s, 2H, -CH₂-), 6.987-7.023 (t, 1H, Ar, J=7.2 Hz), 7.268-7.307 (t, 2H, Ar, J=8.0 Hz), 7.409-7.429 (d, 2H, Ar, J=8.0 Hz), 9.175 (s, 1H, -NH-), 10.052 (s, 1H, -NH-); ¹³C- NMR (400 MHz, DMSO-d₆): δ ppm: 22.316, 23.269, 23.992, 24.067, 90.410, 115.288, 118.868, 123.344, 126.194, 129.570, 130.601, 139.115, 149.817, 151.412; HRMS: m/z calculated for [C₁₆H₁₅N₃OS+H⁺] = 298.1009: found = 298.1014.



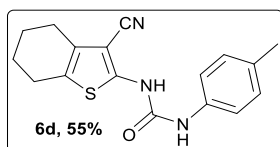
1-(3-cyano-4,5,6,7-tetrahydrobenzo[b]thiophen-2-yl)-3-(m-tolyl)urea (**6b**)

Yield: 181 mg, 58 %; MP. 235 °C; White solid; ¹H-NMR (400 MHz, DMSO-d₆): δ ppm: 1.720 (s, 4H, -CH₂-), 2.264 (s, 3H, -CH₃), 2.444 (s, 2H, -CH₂-), 2.535 (s, 2H, -CH₂-), 6.820-6.836 (d, 1H, Ar, J=6.4 Hz), 7.163-7.180 (d, 2H, Ar, J=6.8 Hz), 7.263 (s, 1H, Ar), 9.117 (s, 1H, -NH-), 10.055 (s, 1H, -NH-); ¹³C- NMR (400 MHz, DMSO-d₆): δ ppm: 21.612, 22.167, 23.120, 23.733, 23.908, 90.183, 115.168, 115.897, 119.186, 123.953, 126.016, 129.265, 130.452, 138.712, 138.878, 149.706, 151.234; HRMS: m/z calculated for [C₁₇H₁₇N₃OS+H⁺] = 312.1165: found = 312.1168.



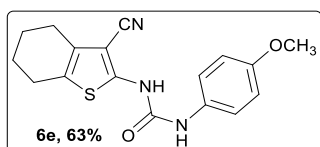
1-(3-cyano-4,5,6,7-tetrahydrobenzo[b]thiophen-2-yl)-3-(3-methoxyphenyl)urea (**6c**)

Yield: 200 mg, 61 %; MP. 230 °C; White solid; ¹H-NMR (400 MHz, DMSO-d₆): δ ppm: 1.740 (s, 4H, -CH₂-), 2.465 (s, 2H, -CH₂-), 2.558 (s, 2H, -CH₂-), 3.741 (s, 3H, -OCH₃), 6.603-6.628 (dd, 1H, Ar, J=8.0 Hz), 6.923-6.942 (dd, 1H, Ar, J=7.6 Hz), 7.128-7.137 (t, 1H, Ar), 7.193-7.233 (t, 1H, Ar, J=8.0 Hz), 9.213 (s, 1H, -NH-), 10.067 (s, 1H, -NH-); ¹³C- NMR (400 MHz, DMSO-d₆): δ ppm: 22.509, 23.452, 24.075, 24.260, 55.792, 90.691, 104.827, 109.030, 111.375, 115.461, 126.455, 130.600, 130.833, 140.504, 149.931, 151.556, 160.566; HRMS: m/z calculated for [C₁₇H₁₇N₃O₂S+H⁺] = 328.1114: found = 328.1119.



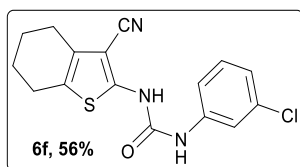
1-(3-cyano-4,5,6,7-tetrahydrobenzo[b]thiophen-2-yl)-3-(p-tolyl)urea (6d)

Yield: 171 mg, 55 %; MP. 240 °C; White solid; ¹H-NMR (400 MHz, DMSO-d₆): δ ppm: 1.743 (s, 4H, -CH₂-), 2.251 (s, 3H, -CH₃), 2.465 (s, 2H, -CH₂-), 2.556 (s, 2H, -CH₂-), 7.105-7.125 (d, 2H, Ar, J=8.0 Hz), 7.314-7.334 (d, 2H, Ar, J=8.0 Hz), 9.107 (s, 1H, -NH-), 10.038 (s, 1H, -NH-); ¹³C- NMR (400 MHz, DMSO-d₆): δ ppm: 21.137, 22.518, 23.472, 24.075, 24.260, 90.447, 115.510, 119.119, 125.297, 126.290, 130.152, 130.765, 132.487, 136.739, 150.136, 151.615; HRMS: m/z calculated for [C₁₇H₁₇N₃OS+H⁺] = 312.1165: found = 312.1169.



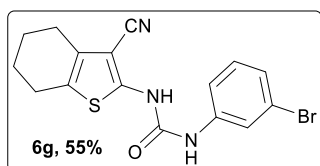
1-(3-cyano-4,5,6,7-tetrahydrobenzo[b]thiophen-2-yl)-3-(4-methoxyphenyl)urea (6e)

Yield: 206 mg, 63 %; MP. 220 °C; White solid; ¹H-NMR (400 MHz, DMSO-d₆): δ ppm: 1.718 (s, 4H, -CH₂-), 2.440 (s, 2H, -CH₂-), 2.529 (s, 2H, -CH₂-), 3.698 (s, 3H, -OCH₃), 6.858-6.880 (d, 2H, Ar, J=8.8 Hz), 7.314-7.336 (d, 2H, Ar, J=8.8 Hz), 8.995 (s, 1H, -NH-), 9.976 (s, 1H, -NH-); ¹³C- NMR (400 MHz, DMSO-d₆): δ ppm: 22.176, 23.120, 23.714, 23.908, 55.635, 89.911, 114.594, 115.226, 120.470, 125.831, 130.374, 131.931, 149.940, 151.360, 155.485; HRMS: m/z calculated for [C₁₇H₁₇N₃O₂S+H⁺] = 328.1114: found = 328.1120.



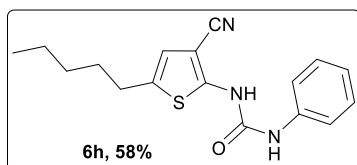
1-(3-chlorophenyl)-3-(3-cyano-4,5,6,7-tetrahydrobenzo[b]thiophen-2-yl)urea (6f)

Yield: 185 mg, 56 %; MP. 212 °C; White solid; ¹H-NMR (400 MHz, DMSO-d₆): δ ppm: 1.726 (s, 4H, -CH₂-), 2.449 (s, 2H, -CH₂-), 2.545 (s, 2H, -CH₂-), 7.196 (s, 1H, Ar), 7.264 (s, 2H, Ar), 7.799 (s, 1H, Ar), 9.349 (s, 1H, -NH-), 10.115 (s, 1H, -NH-); ¹³C- NMR (400 MHz, DMSO-d₆): δ ppm: 22.080, 23.034, 23.676, 23.841, 90.758, 114.955, 117.591, 120.880, 122.193, 125.686, 126.299, 130.521, 131.251, 140.562, 149.182, 151.147; HRMS: m/z calculated for [C₁₆H₁₄ClN₃OS+H⁺] = 332.0619: found = 332.0623.



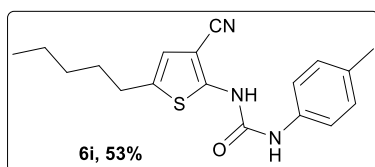
1-(3-bromophenyl)-3-(3-cyano-4,5,6,7-tetrahydrobenzo[b]thiophen-2-yl)urea (6g)

Yield: 207 mg, 55 %; MP. >250 °C; White solid; ¹H-NMR (400 MHz, DMSO-d₆): δ ppm: 1.726 (s, 4H, -CH₂-), 2.451 (s, 2H, -CH₂-), 2.546 (s, 2H, -CH₂-), 7.187-7.265 (q, 3H, Ar), 7.802 (s, 1H, Ar), 9.336 (s, 1H, -NH-), 10.132 (s, 1H, -NH-); ¹³C- NMR (400 MHz, DMSO-d₆): δ ppm: 22.158, 23.101, 23.743, 23.919, 90.807, 115.042, 117.688, 120.957, 122.271, 125.793, 126.376, 130.609, 131.348, 140.620, 149.269, 151.215; HRMS: m/z calculated for [C₁₆H₁₄BrN₃OS+H⁺] = 376.0114: found = 376.0119.



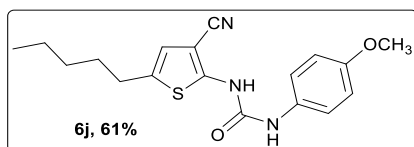
1-(3-cyano-5-pentylthiophen-2-yl)-3-phenylurea (6h)

Yield: 181 mg, 58 %; MP. 210 °C; White solid; ¹H-NMR (400 MHz, DMSO-d₆): δ ppm: 0.882 (s, 3H, -CH₃), 1.246 (s, 4H, -CH₂-), 1.530 (s, 2H, -CH₂-), 2.611 (s, 2H, -CH₂-), 6.721 (s, 1H, thiophenyl), 7.000 (s, 1H, Ar), 7.283 (s, 2H, Ar), 7.420 (s, 2H, Ar), 9.462 (s, 1H, -NH-), 10.192 (s, 1H, -NH-); ¹³C- NMR (400 MHz, DMSO-d₆): δ ppm: 14.599, 22.538, 29.309, 31.002, 31.284, 89.475, 115.889, 119.042, 120.822, 123.507, 129.734, 136.058, 139.346, 150.564, 151.751; HRMS: m/z calculated for [C₁₇H₁₉N₃OS+H⁺] = 314.1322: found = 314.1325.



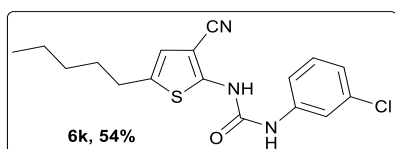
1-(3-cyano-5-pentylthiophen-2-yl)-3-(p-tolyl)urea (6i)

Yield: 174 mg, 53 %; MP. 201 °C; White solid; ¹H-NMR (400 MHz, DMSO-d₆): δ ppm: 0.834 (s, 3H, -CH₃), 1.258 (s, 4H, -CH₂-), 1.539 (s, 2H, -CH₂-), 2.228 (s, 3H, -CH₃), 2.600-2.634 (t, 2H, -CH₂-. J=6.8Hz), 6.732 (s, 1H, thiophenyl), 7.084-7.102 (d, 2H, Ar, J=7.2 Hz), 7.300-7.319 (d, 2H, Ar, J=7.6 Hz), 9.111 (s, 1H, -NH-), 10.032 (s, 1H, -NH-); ¹³C- NMR (400 MHz, DMSO-d₆): δ ppm: 14.286, 20.785, 22.225, 28.987, 30.689, 30.981, 88.879, 115.674, 118.787, 120.392, 129.800, 132.165, 135.609, 136.387, 150.446, 151.292; HRMS: m/z calculated for [C₁₈H₂₁N₃OS+H⁺] = 328.1478: found = 328.1476.



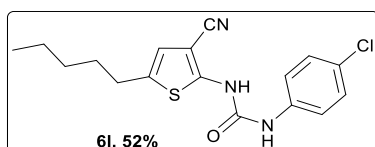
1-(3-cyano-5-pentylthiophen-2-yl)-3-(4-methoxyphenyl)urea (**6j**)

Yield: 210 mg, 61 %; MP. 195 °C; White solid; $^1\text{H-NMR}$ (400 MHz, DMSO-d_6): δ ppm: 0.827-0.859 (t, 3H, - CH_3 , $J=6.8$ Hz), 1.266-1.274 (d, 4H, - CH_2 -, $J=3.2$ Hz), 1.531-1.565 (t, 2H, - CH_2 -, $J=6.4$ Hz), 2.608-2.645 (t, 2H, - CH_2 -, $J=7.2$ Hz), 3.705 (s, 3H, - OCH_3), 6.748 (s, 1H, thiophenyl), 6.868-6.890 (d, 2H, Ar, $J=8.8$ Hz), 7.322-7.344 (d, 2H, Ar, $J=8.8$ Hz), 9.031 (s, 1H, -NH-), 10.004 (s, 1H, -NH-); $^{13}\text{C-NMR}$ (400 MHz, DMSO-d_6): δ ppm: 14.267, 22.225, 28.987, 30.689, 30.981, 55.635, 88.743, 114.594, 115.703, 120.334, 120.499, 131.941, 135.531, 150.572, 151.399, 155.534; HRMS: m/z calculated for $[\text{C}_{18}\text{H}_{21}\text{N}_3\text{O}_2\text{S}+\text{H}^+]$ = 344.1427: found = 344.1431.



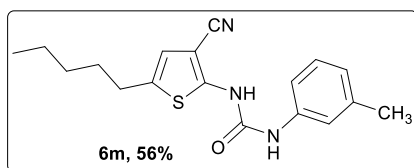
1-(3-chlorophenyl)-3-(3-cyano-5-pentylthiophen-2-yl)urea (**6k**)

Yield: 188 mg, 54 %; MP. 201 °C; White solid; $^1\text{H-NMR}$ (400 MHz, DMSO-d_6): δ ppm: 0.832-0.867 (t, 3H, - CH_3 , $J=7.2$ Hz), 1.263-1.281 (t, 4H, - CH_2 -, $J=4.0$ Hz), 1.541-1.577 (t, 2H, - CH_2 -, $J=7.2$ Hz), 2.626-2.663 (t, 2H, - CH_2 -, $J=7.2$ Hz), 6.779 (s, 1H, thiophenyl), 7.066-7.089 (q, 1H, Ar), 7.233-7.256 (t, 1H, Ar, $J=8.4$ Hz), 7.307-7.348 (t, 1H, Ar, $J=8.0$ Hz), 7.676-7.686 (t, 1H, Ar, $J=2.0$ Hz), 9.397 (s, 1H, -NH-), 10.162 (s, 1H, -NH-); $^{13}\text{C-NMR}$ (400 MHz, DMSO-d_6): δ ppm: 14.219, 22.148, 28.920, 30.603, 30.895, 89.571, 115.480, 117.260, 118.107, 120.520, 122.845, 130.988, 133.751, 135.989, 140.416, 149.853, 151.225; HRMS: m/z calculated for $[\text{C}_{17}\text{H}_{18}\text{ClN}_3\text{OS}+\text{H}^+]$ = 348.0932: found = 348.0937.



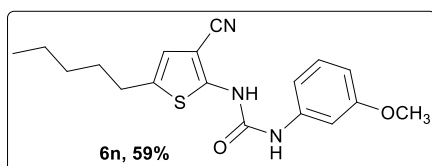
1-(4-chlorophenyl)-3-(3-cyano-5-pentylthiophen-2-yl)urea (**6l**)

Yield: 181 mg, 52 %; MP. 230 °C; White solid; $^1\text{H-NMR}$ (400 MHz, DMSO-d_6): δ ppm: 0.832-0.866 (t, 3H, - CH_3 , $J=6.8$ Hz), 1.272-1.281 (t, 4H, - CH_2 -, $J=3.6$ Hz), 1.540-1.593 (q, 2H, - CH_2 -), 2.622-2.659 (t, 2H, - CH_2 -, $J=7.2$ Hz), 6.771 (s, 1H, thiophenyl), 7.339-7.361 (d, 2H, Ar, $J=8.8$ Hz), 7.454-7.476 (d, 2H, Ar, $J=8.8$ Hz), 9.324 (s, 1H, -NH-), 10.104 (s, 1H, -NH-); $^{13}\text{C-NMR}$ (400 MHz, DMSO-d_6): δ ppm: 14.216, 22.138, 28.910, 30.603, 30.895, 89.367, 115.509, 120.277, 120.481, 126.766, 129.218, 135.872, 137.886, 149.999, 151.215; HRMS: m/z calculated for $[\text{C}_{17}\text{H}_{18}\text{ClN}_3\text{OS}+\text{H}^+]$ = 348.0932: found = 348.0935.



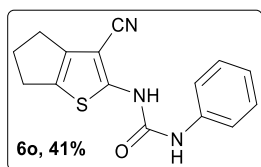
1-(3-cyano-5-pentylthiophen-2-yl)-3-(m-tolyl)urea (**6m**)

Yield: 183 mg, 56 %; MP. 178 °C; White solid; ¹H-NMR (400 MHz, DMSO-d₆): δ ppm: 0.839 (s, 3H, -CH₃), 1.264 (s, 4H, -CH₂-), 1.545 (s, 2H, -CH₂-), 2.269 (s, 3H, -CH₃), 2.628 (s, 2H, -CH₂-), 6.748 (s, 1H, thiophenyl), 6.840 (s, 1H, Ar), 7.185 (s, 2H, Ar), 7.278 (s, 1H, Ar), 9.146 (s, 1H, -NH-), 10.068 (s, 1H, -NH-); ¹³C- NMR (400 MHz, DMSO-d₆): δ ppm: 14.313, 21.620, 22.240, 28.983, 30.692, 30.967, 89.009, 115.696, 115.942, 120.466, 124.028, 129.317, 135.764, 138.773, 138.854, 150.350, 151.309; HRMS: m/z calculated for [C₁₈H₂₁N₃OS+H⁺] = 328.1478: found = 328.1481 .



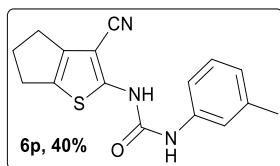
1-(3-cyano-5-pentylthiophen-2-yl)-3-(3-methoxyphenyl)urea (**6n**)

Yield: 202 mg, 59 %; MP. 210 °C; White solid; ¹H-NMR (400 MHz, DMSO-d₆): δ ppm: 0.820-0.851 (t, 3H, -CH₃, J=6.8 Hz), 1.258-1.265 (d, 4H, -CH₂-, J=2.8 Hz), 1.528-1.561 (t, 2H, -CH₂-), 2.610-2.646 (t, 2H, -CH₂-, J=7.2 Hz), 3.722 (s, 3H, -OCH₃), 6.592-6.612 (d, 1H, Ar, J=8.0 Hz), 6.752 (s, 1H, thiophenyl), 6.897-6.917 (d, 1H, Ar, J=8.8 Hz), 7.126 (s, 1H, Ar), 7.179-7.219 (t, 1H, Ar, J = 8.0 Hz), 9.224 (s, 1H, -NH-), 10.072 (s, 1H, -NH-); ¹³C- NMR (400 MHz, DMSO-d₆): δ ppm: 14.312, 22.237, 28.987, 30.708, 30.973, 55.462, 89.154, 104.444, 108.769, 111.042, 115.648, 120.489, 130.305, 135.841, 140.136, 150.224, 151.268, 160.227; HRMS: m/z calculated for [C₁₈H₂₁N₃O₂S+H⁺] = 344.1427: found = 344.1428.



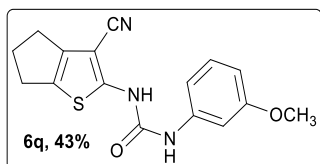
1-(3-cyano-5,6-dihydro-4H-cyclopenta[b]thiophen-2-yl)-3-phenylurea (**6o**)

Yield: 117 mg, 41 %; MP. 208 °C; White solid; ¹H-NMR (400 MHz, DMSO-d₆): δ ppm: 2.323 (s, 2H, -CH₂-), 2.674 (s, 2H, -CH₂-), 2.779 (s, 2H, -CH₂-), 7.021 (s, 1H, Ar), 7.302 (s, 2H, Ar), 7.425-7.443 (d, 2H, Ar, J=7.2 Hz), 9.192 (s, 1H, -NH-), 10.066 (s, 1H, -NH-); ¹³C- NMR (400 MHz, DMSO-d₆): δ ppm: 27.879, 27.986, 29.338, 85.962, 115.354, 118.603, 118.691, 123.156, 129.364, 132.195, 138.781, 138.888, 140.474, 151.108, 151.196, 154.611, 154.747; HRMS: m/z calculated for [C₁₅H₁₃N₃OS+H⁺] = 284.0852: found = 284.0857.



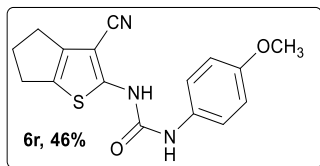
1-(3-cyano-5,6-dihydro-4H-cyclopenta[b]thiophen-2-yl)-3-(m-tolyl)urea (6p)

Yield: 120 mg, 40 %; MP. 218 °C; White solid; ¹H-NMR (400 MHz, DMSO-d₆): δ ppm: 2.267 (s, 3H, -CH₃), 2.300-2.316 (d, 2H, -CH₂-, J=6.8 Hz), 2.667 (s, 2H, -CH₂-), 2.772 (s, 2H, -CH₂-), 6.822-6.835 (d, 1H, Ar, J=5.2 Hz), 7.181 (s, 2H, Ar), 7.273 (s, 2H, Ar), 9.120 (s, 1H, -NH-), 10.049 (s, 1H, -NH-); ¹³C- NMR (400 MHz, DMSO-d₆): δ ppm: 21.612, 27.946, 28.043, 29.405, 85.970, 115.450, 115.917, 119.205, 123.953, 129.255, 132.203, 138.712, 138.878, 140.522, 151.204, 154.853; HRMS: m/z calculated for [C₁₆H₁₅N₃OS+H⁺] = 298.1009; found = 298.1012.



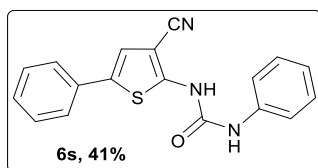
1-(3-cyano-5,6-dihydro-4H-cyclopenta[b]thiophen-2-yl)-3-(3-methoxyphenyl)urea (6q)

Yield: 135 mg, 43 %; MP. 232 °C; White solid; ¹H-NMR (400 MHz, DMSO-d₆): δ ppm: 2.339 (s, 2H, -CH₂-), 2.687 (s, 2H, -CH₂-), 2.790 (s, 2H, -CH₂-), 3.739 (s, 3H, -OCH₃), 6.612 (s, 1H, Ar), 6.936 (s, 1H, Ar), 7.126-7.209 (d, 2H, Ar), 9.220 (s, 1H, -NH-), 10.074 (s, 1H, -NH-); ¹³C- NMR (400 MHz, DMSO-d₆): δ ppm: 27.888, 27.986, 29.348, 55.402, 86.088, 104.457, 108.611, 110.995, 115.344, 130.181, 132.234, 140.085, 140.494, 151.128, 154.669, 160.166; HRMS: m/z calculated for [C₁₆H₁₅N₃O₂S+H⁺] = 314.0958; found = 314.0953.



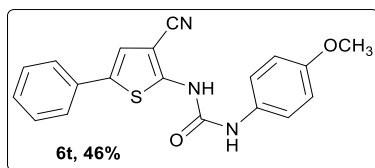
1-(3-cyano-5,6-dihydro-4H-cyclopenta[b]thiophen-2-yl)-3-(4-methoxyphenyl)urea (6r)

Yield: 145 mg, 46 %; MP. 246 °C; White solid; ¹H-NMR (400 MHz, DMSO-d₆): δ ppm: 2.295 (s, 2H, -CH₂-), 2.646 (s, 2H, -CH₂-), 2.746 (s, 2H, -CH₂-), 3.675 (s, 3H, -OCH₃), 6.839-6.857 (d, 2H, Ar, J = 7.2 Hz), 7.295-7.314 (d, 2H, Ar, J = 7.6 Hz), 8.982 (s, 1H, -NH-), 9.989 (s, 1H, -NH-); ¹³C- NMR (400 MHz, DMSO-d₆): δ ppm: 27.914, 28.031, 29.383, 55.632, 85.715, 114.581, 115.486, 120.526, 131.880, 132.036, 151.338, 155.074, 155.502; HRMS: m/z calculated for [C₁₆H₁₅N₃O₂S+H⁺] = 314.0958; found = 314.0953.



1-(3-cyano-5-phenylthiophen-2-yl)-3-phenylurea (6s)

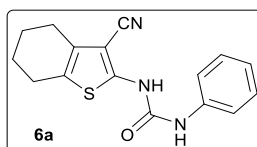
Yield: 131 mg, 41 %; MP. >250 °C; White solid; $^1\text{H-NMR}$ (400 MHz, DMSO- d_6): δ ppm : 7.026-7.062 (t, 1H, Ar, $J=7.2$ Hz), 7.265-7.411 (q, 5H, Ar), 7.466-7.513 (t, 3H, Ar, $J=7.6$ Hz), 7.587-7.606 (d, 2H, Ar, $J=7.6$ Hz), 9.334 (s, 1H, -NH-), 10.344 (s, 1H, -NH-); $^{13}\text{C-NMR}$ (400 MHz, DMSO- d_6): δ ppm: 90.797, 115.354, 118.778, 120.442, 123.322, 125.258, 127.972, 129.402, 129.539, 133.158, 138.772, 151.283, 151.575; HRMS: m/z calculated for $[\text{C}_{18}\text{H}_{13}\text{N}_3\text{OS}+\text{H}^+]$ = 320.0852: found = 320.0850.



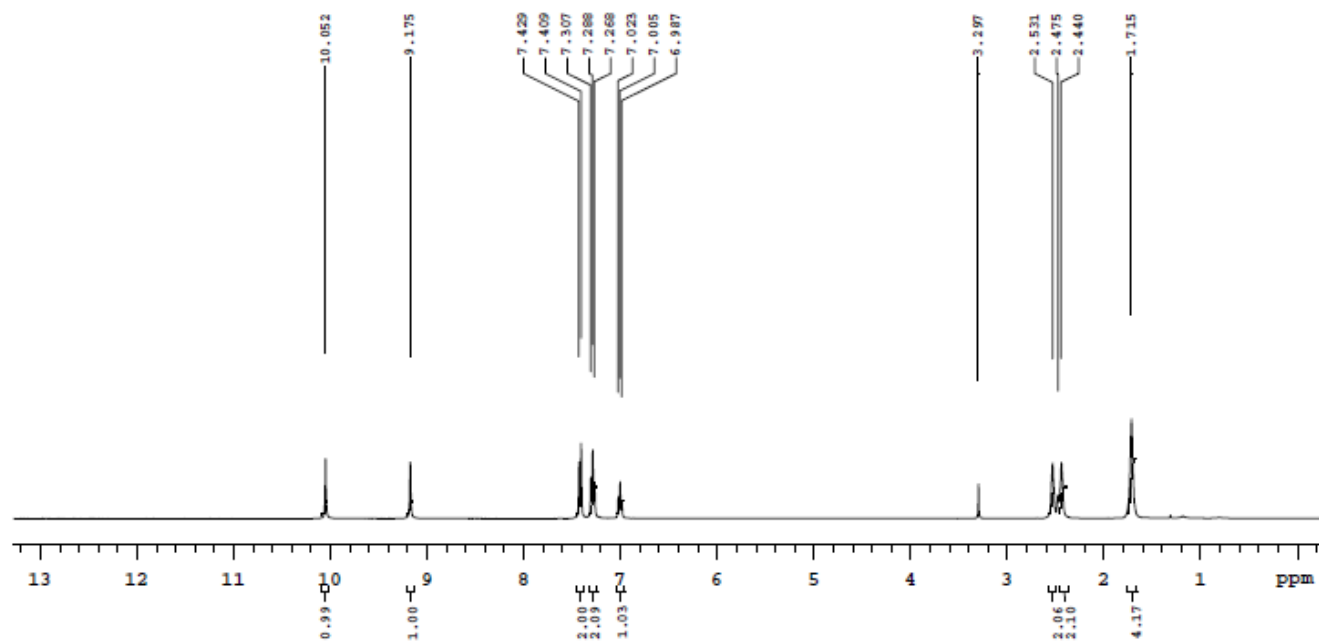
1-(3-cyano-5-phenylthiophen-2-yl)-3-(4-methoxyphenyl)urea (6t)

Yield: 161 mg, 46 %; MP. 238 °C; White solid; $^1\text{H-NMR}$ (400 MHz, DMSO- d_6): δ ppm : 7.026-7.062 (t, 1H, Ar, $J=7.2$ Hz), 7.265-7.411 (q, 5H, Ar), 7.466-7.513 (t, 3H, Ar, $J=7.6$ Hz), 7.587-7.606 (d, 2H, Ar, $J=7.6$ Hz), 9.334 (s, 1H, -NH-), 10.344 (s, 1H, -NH-); $^{13}\text{C-NMR}$ (400 MHz, DMSO- d_6): δ ppm: 90.797, 115.354, 118.778, 120.442, 123.322, 125.258, 127.972, 129.402, 129.539, 133.158, 138.772, 151.283, 151.575; HRMS: m/z calculated for $[\text{C}_{18}\text{H}_{13}\text{N}_3\text{OS}+\text{H}^+]$ = 350.0958: found = 350.0955.

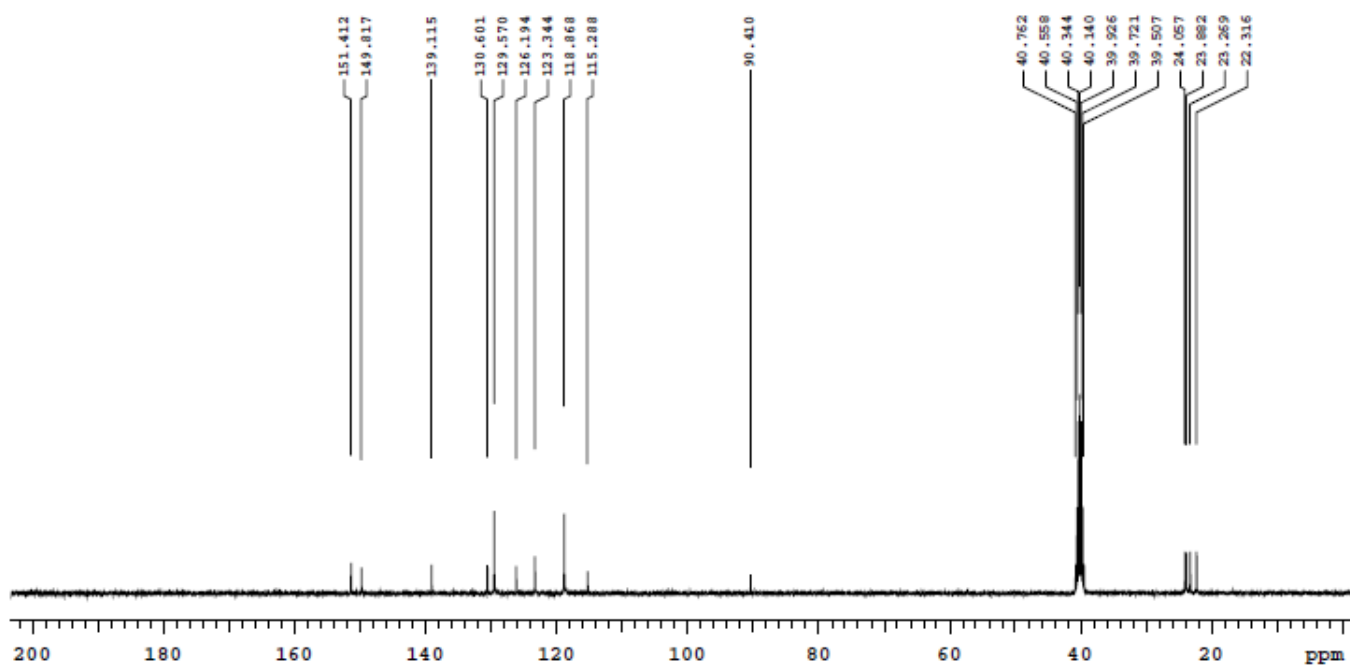
4. $^1\text{H-NMR}$, $^{13}\text{C-NMR}$ Spectra

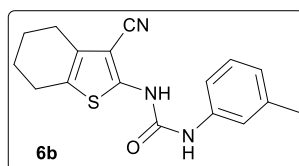


$^1\text{H-NMR}$ spectra of 6a:

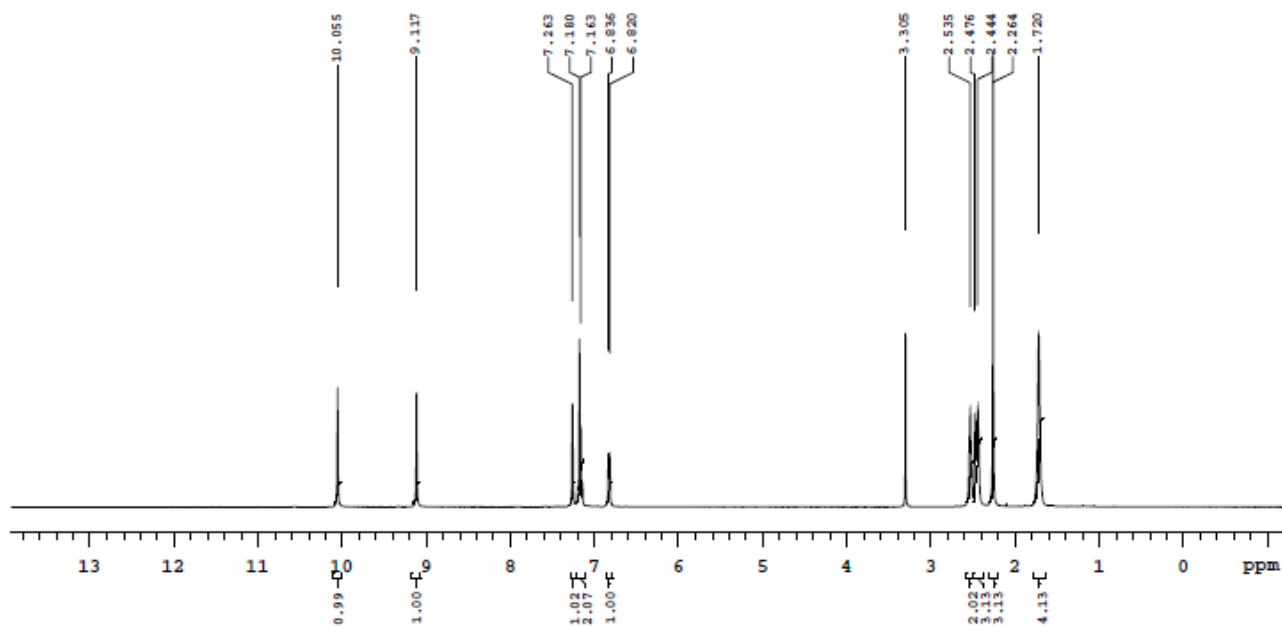


$^{13}\text{C-NMR}$ of spectra 6a:

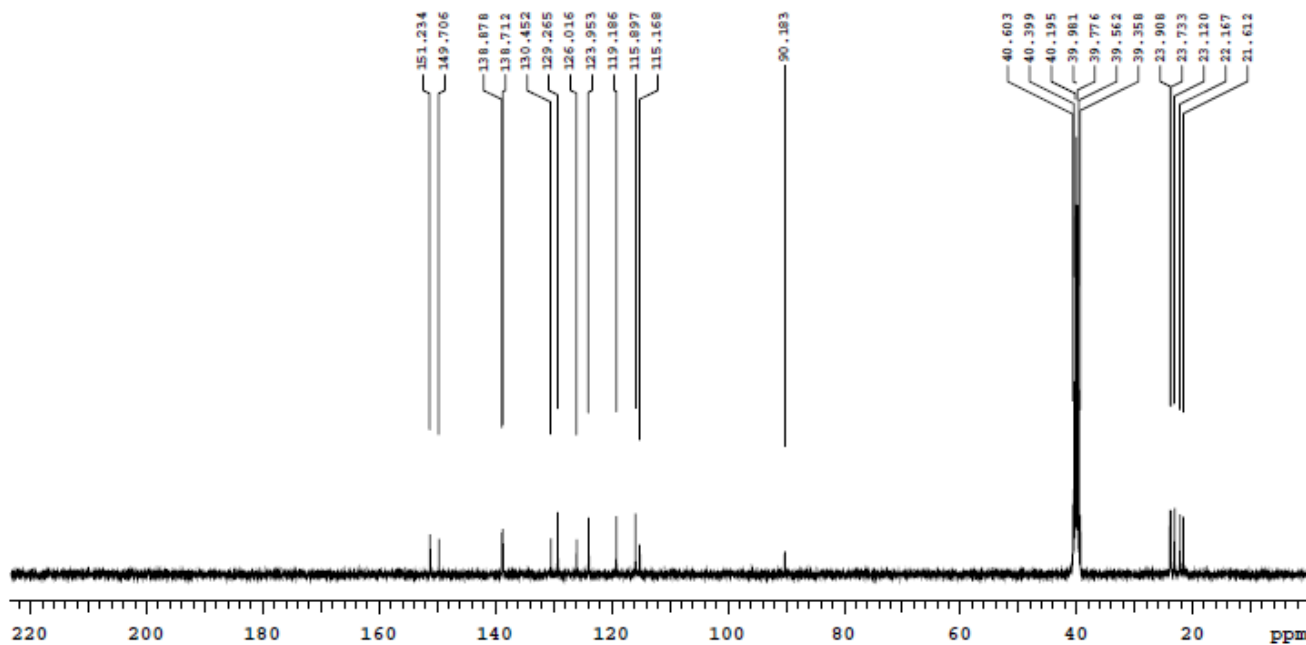


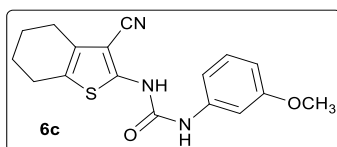


$^1\text{H-NMR}$ spectra of **6b**:

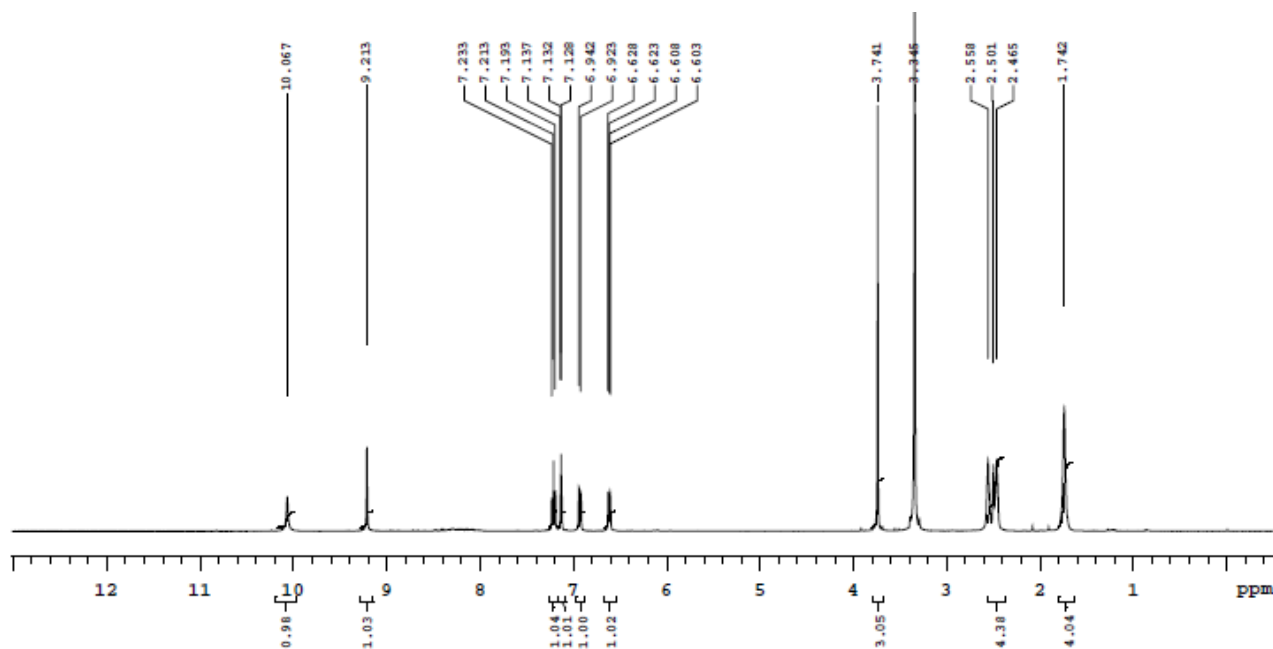


$^{13}\text{C-NMR}$ of spectra **6b**:

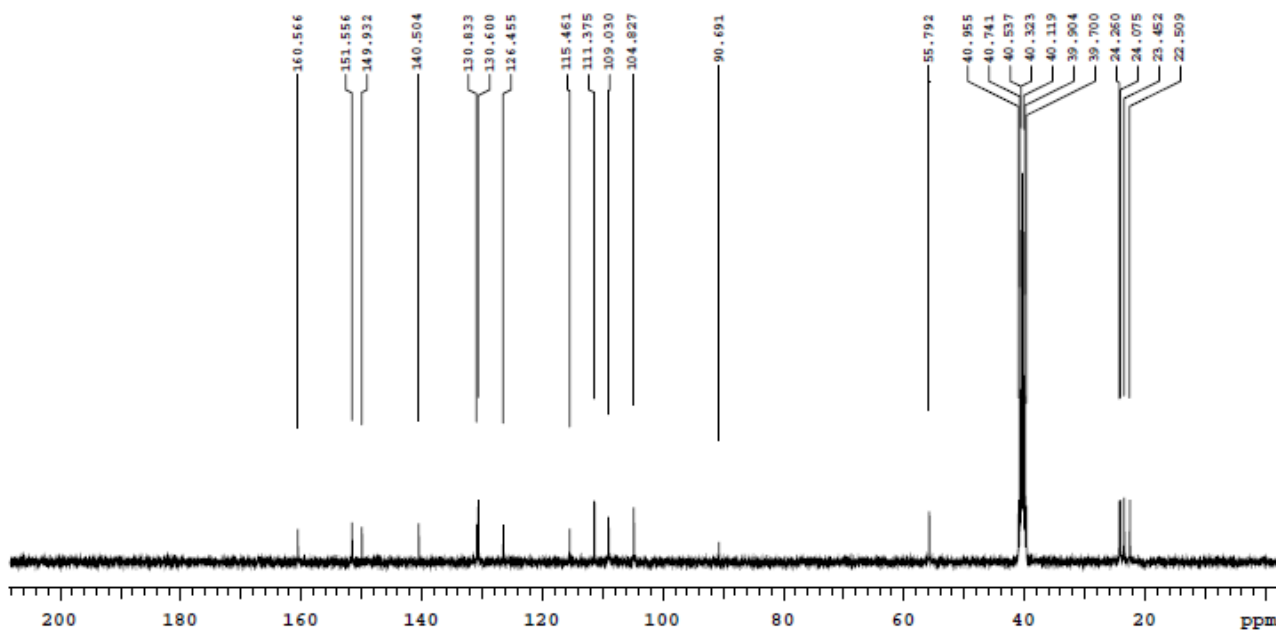


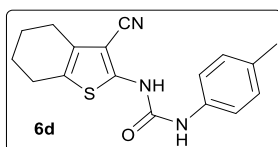


¹H-NMR spectra of 6c:

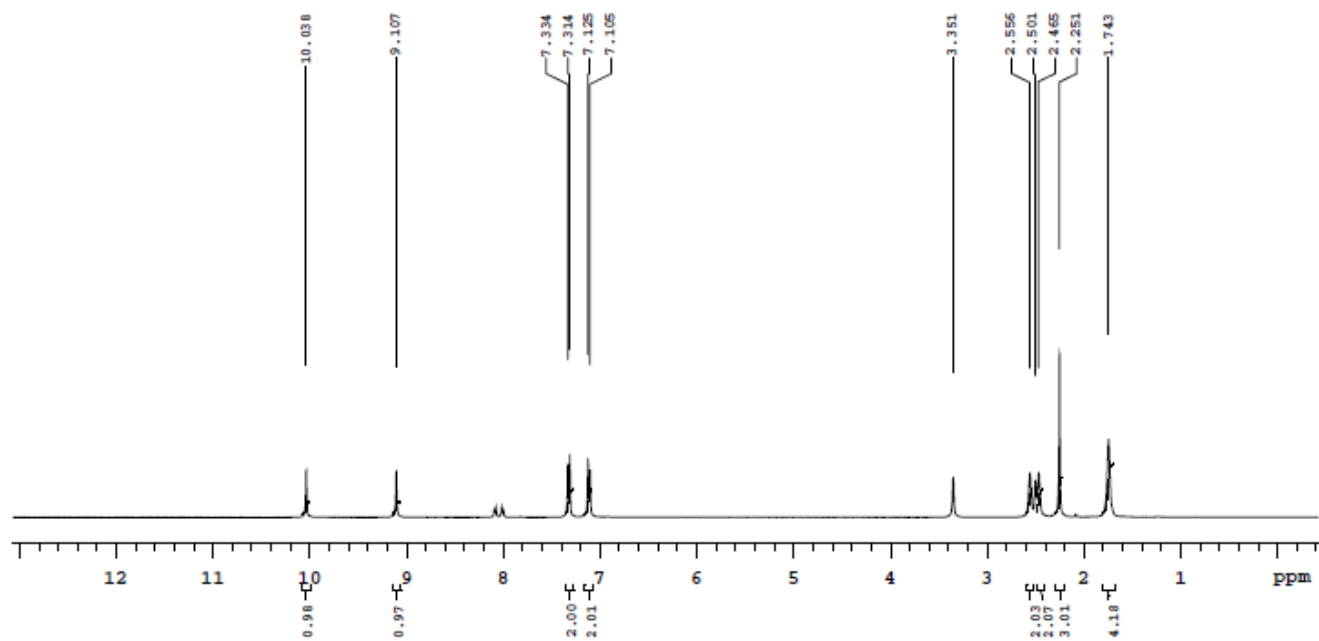


¹³C-NMR spectra of 6c:

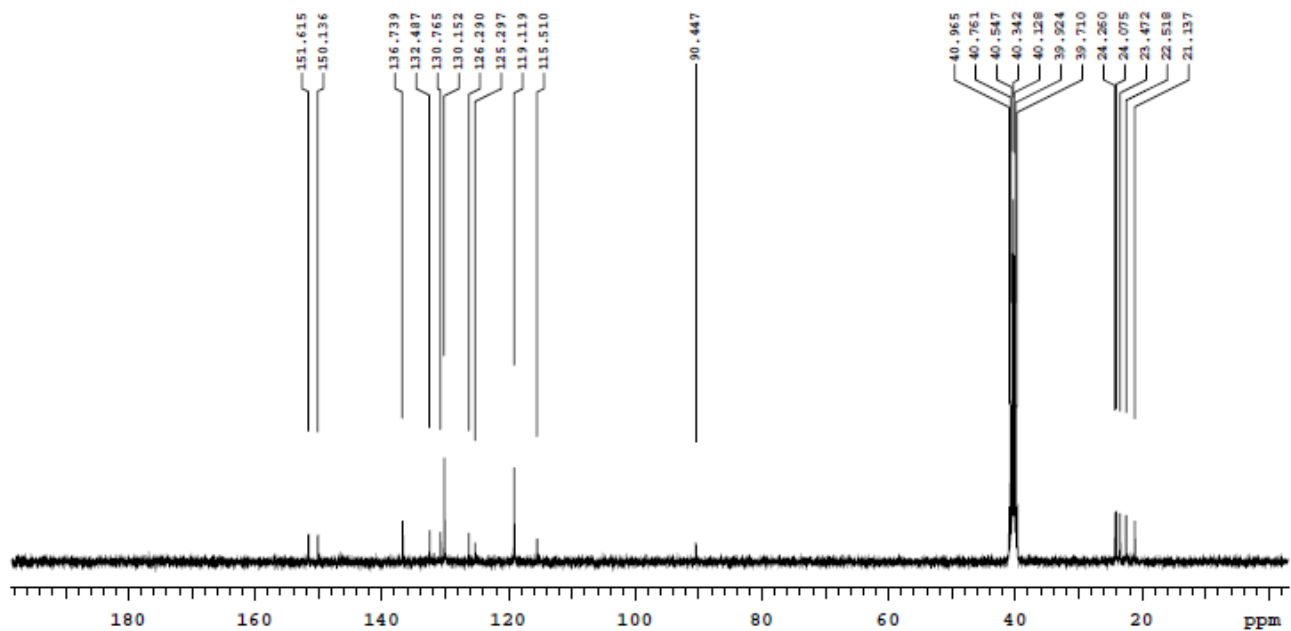


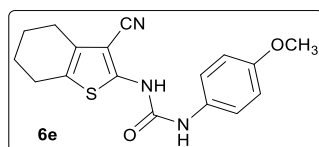


$^1\text{H-NMR}$ spectra of 6d:

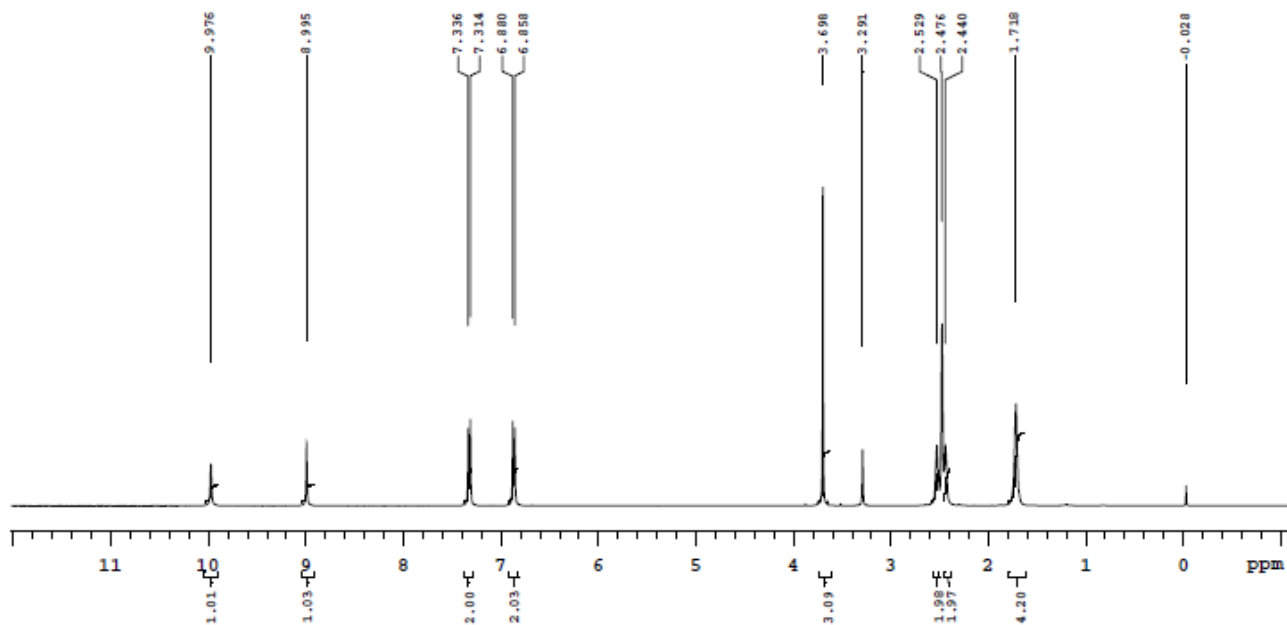


$^{13}\text{C-NMR}$ spectra of 6d:

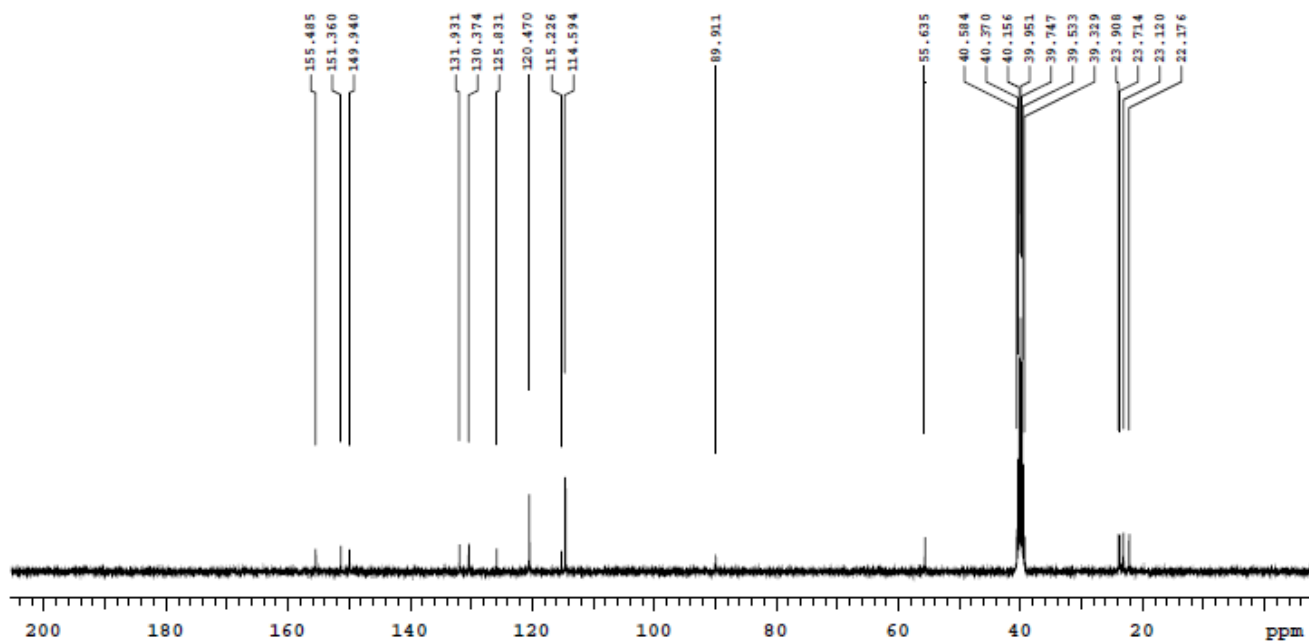


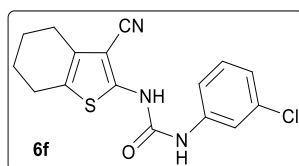


$^1\text{H-NMR}$ spectra of **6e**:

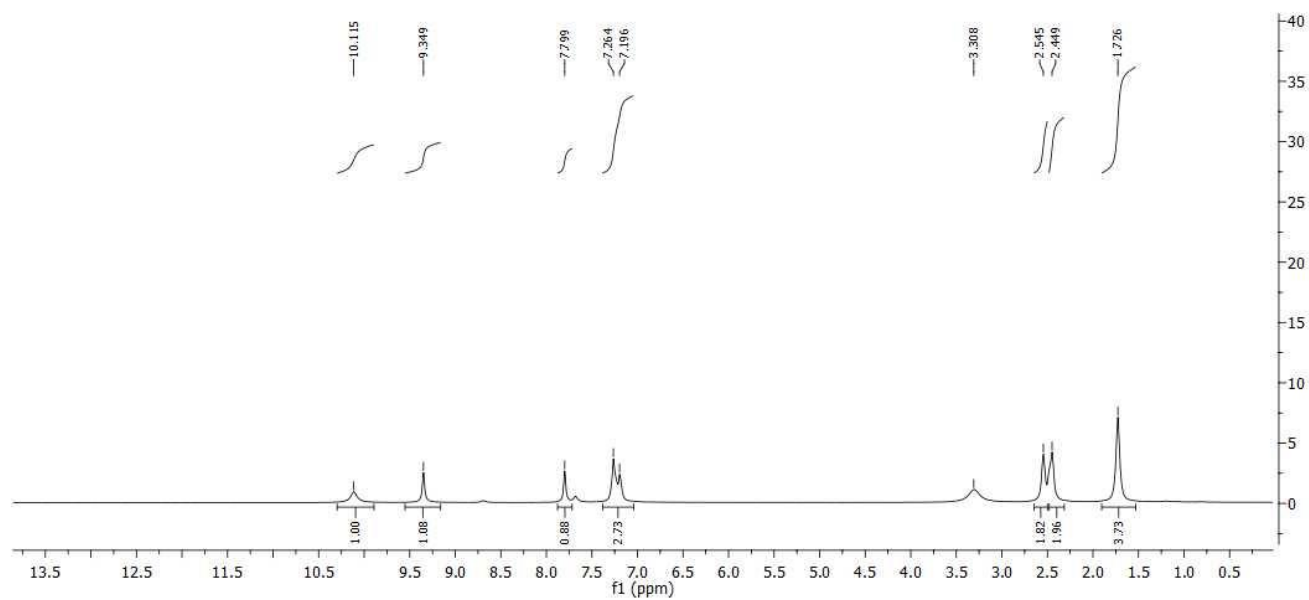


$^{13}\text{C-NMR}$ spectra of **6e**:

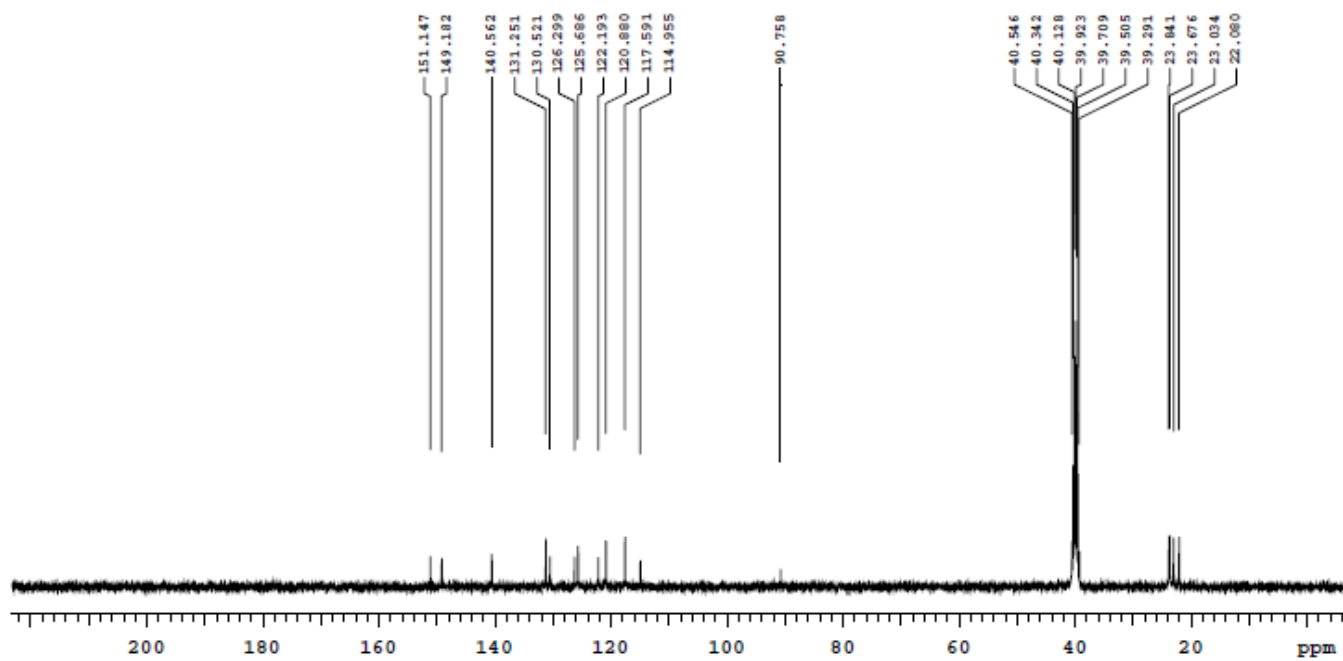


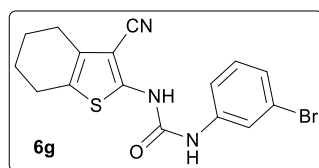


$^1\text{H-NMR}$ spectra of 6f:

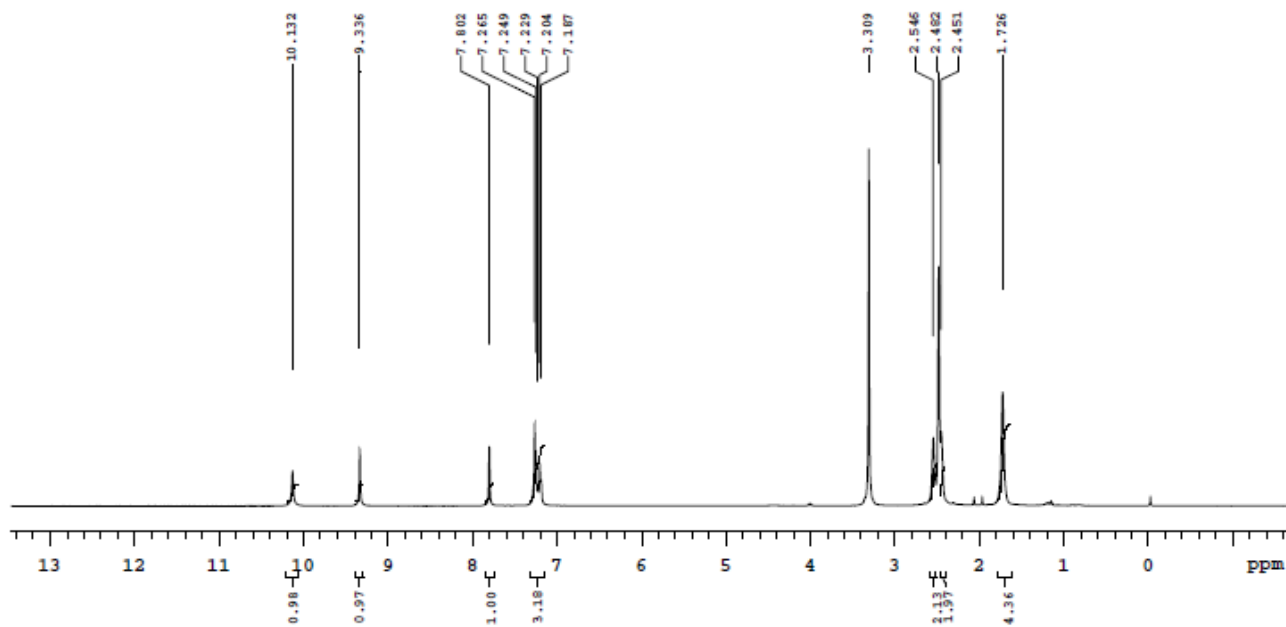


$^{13}\text{C-NMR}$ spectra of 6f:

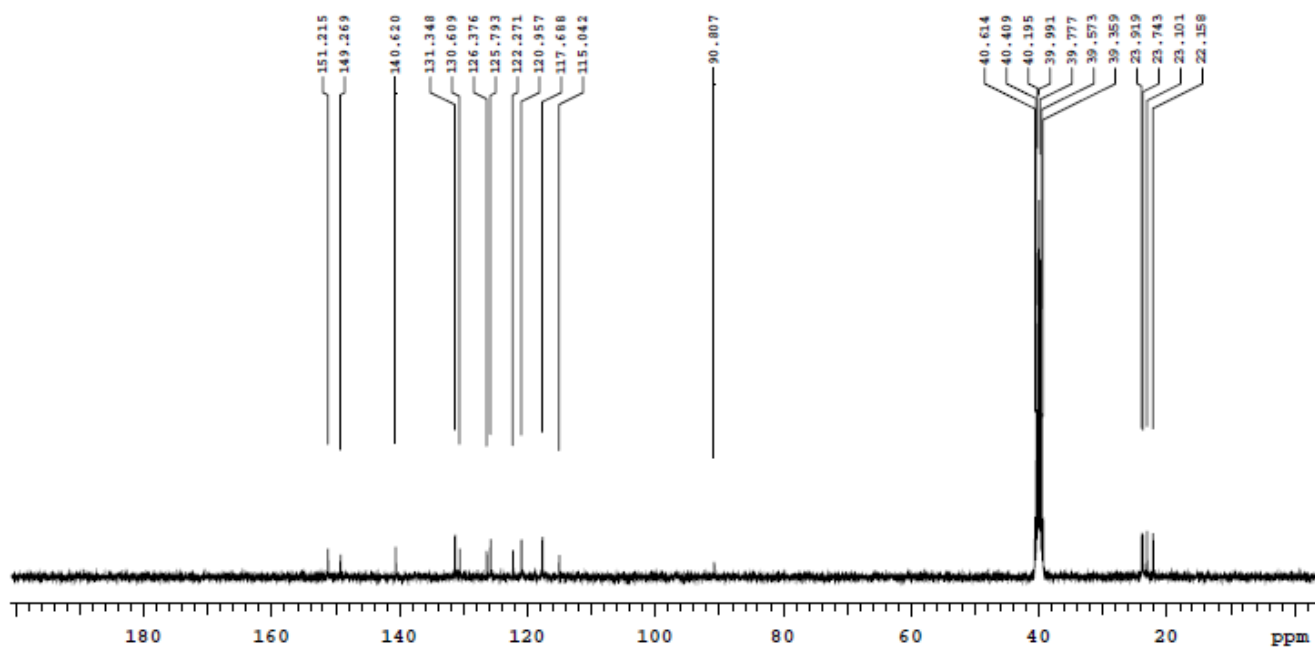


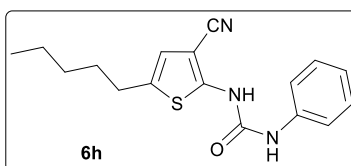


$^1\text{H-NMR}$ spectra of **6g**:

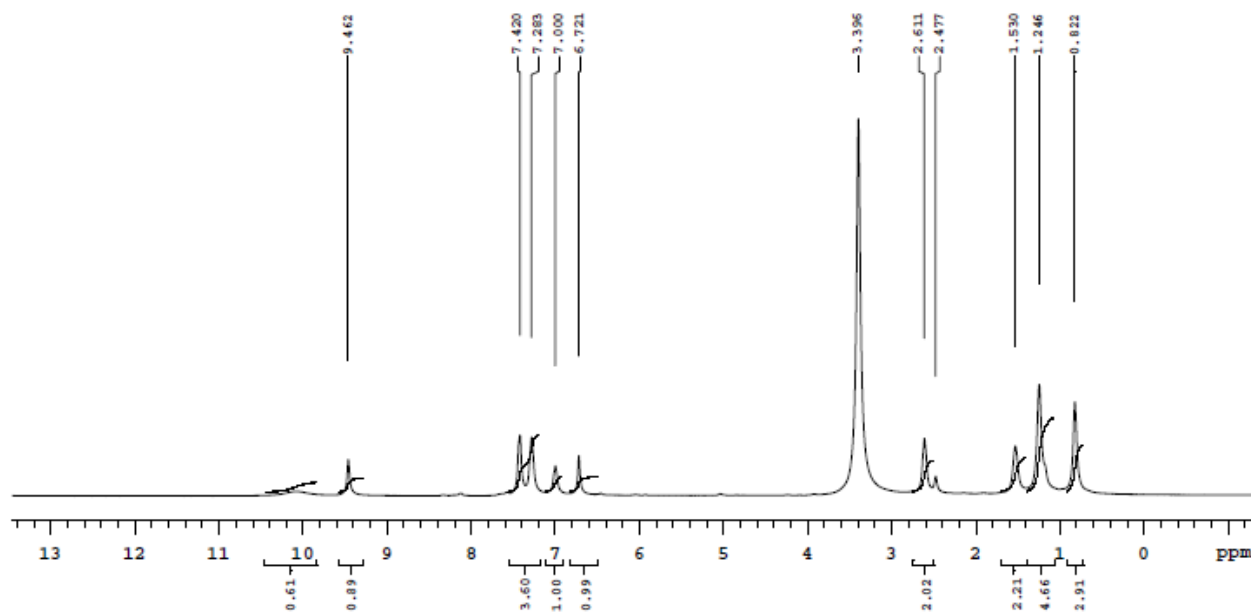


$^{13}\text{C-NMR}$ spectra of **6g**:

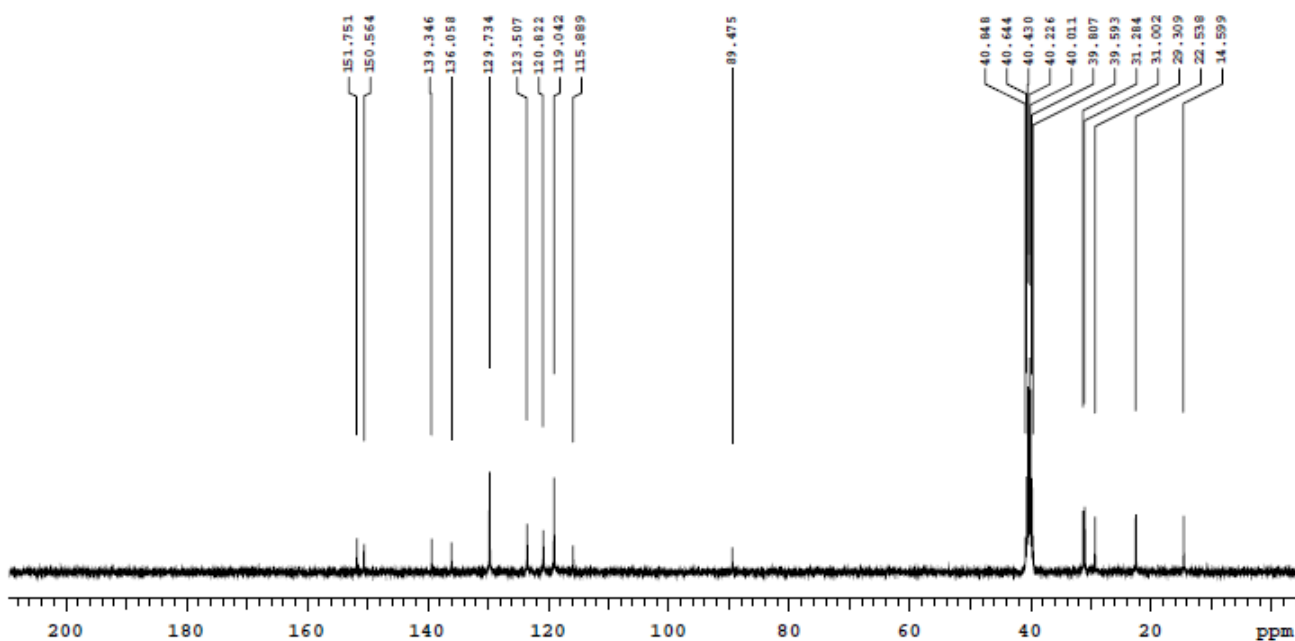


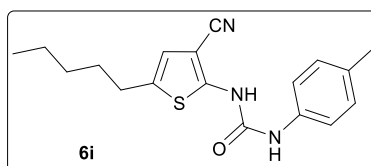


$^1\text{H-NMR}$ spectra of 6h:

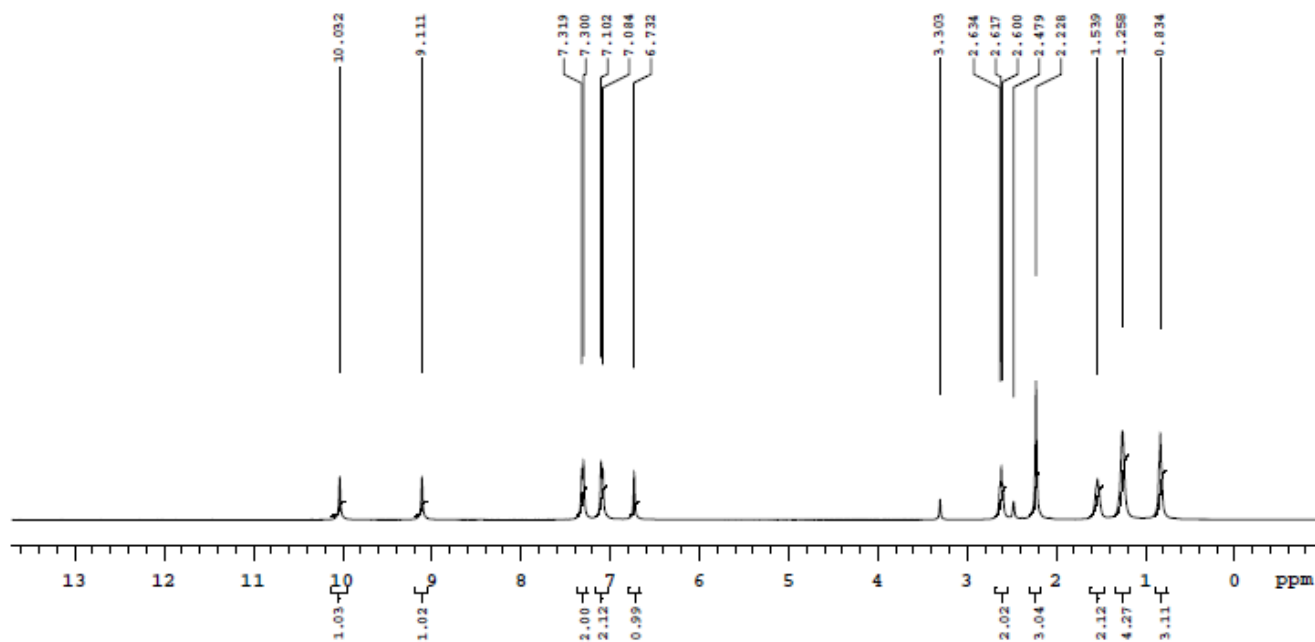


$^{13}\text{C-NMR}$ spectra of 6h:

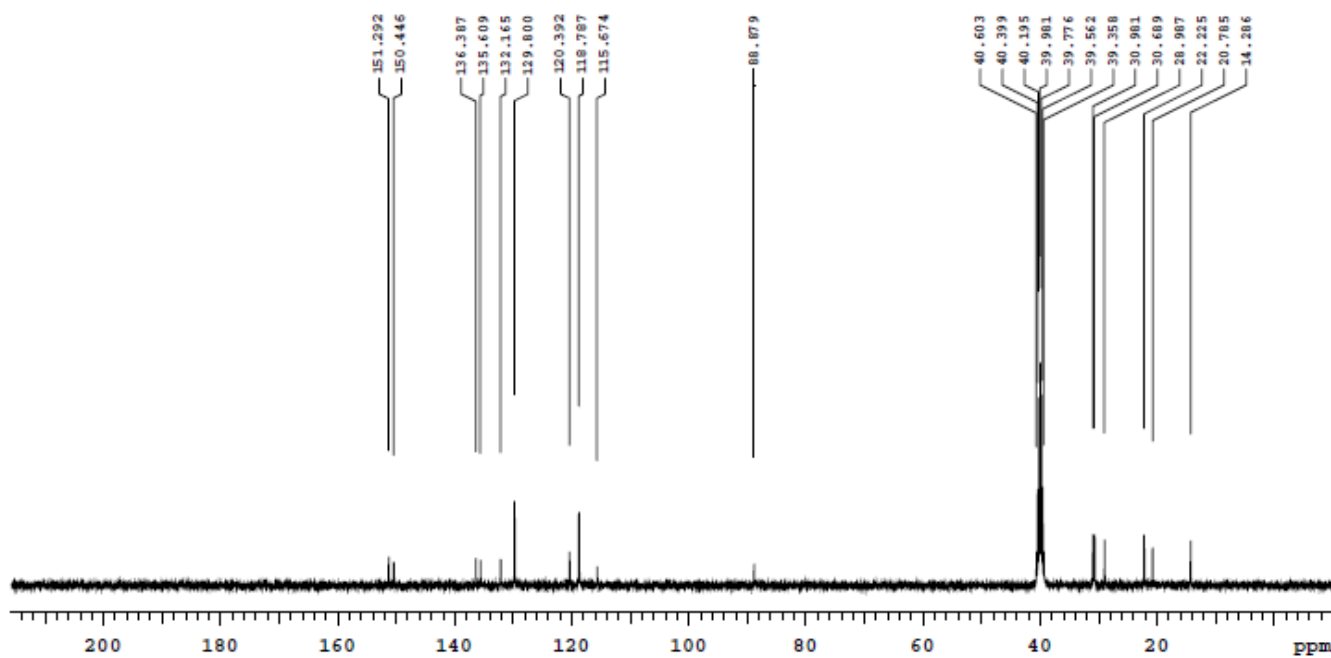


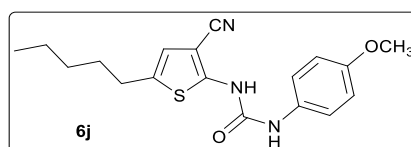


$^1\text{H-NMR}$ spectra of **6i**:

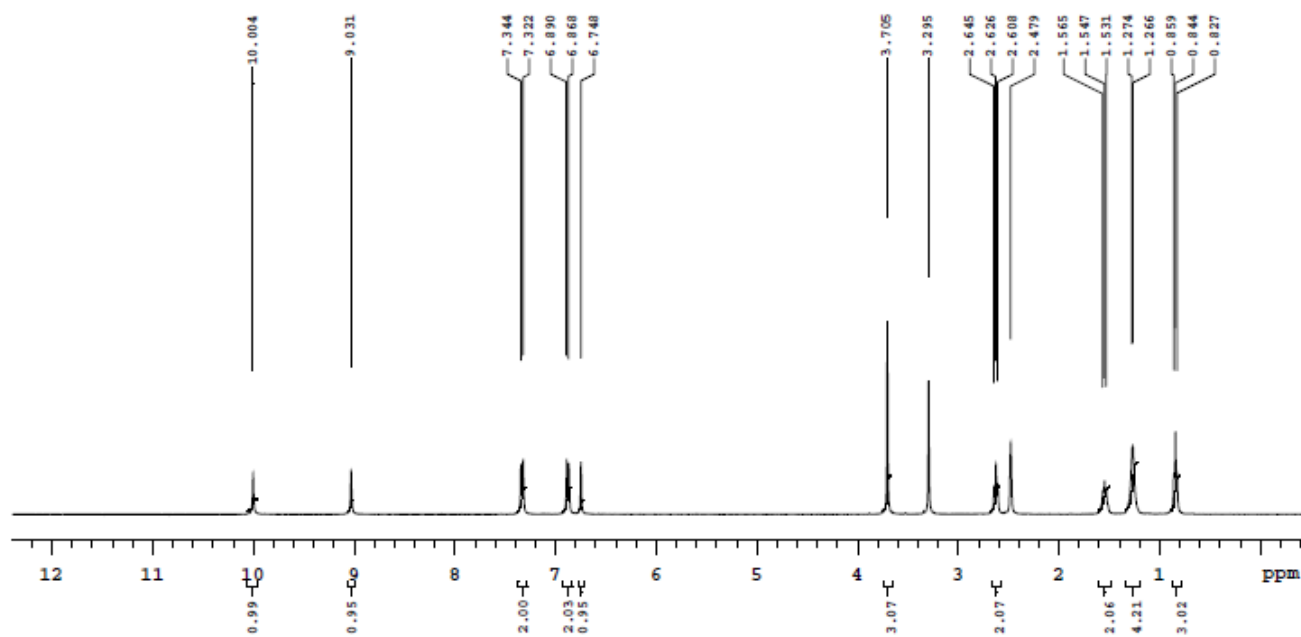


$^{13}\text{C-NMR}$ spectra of **6i**:

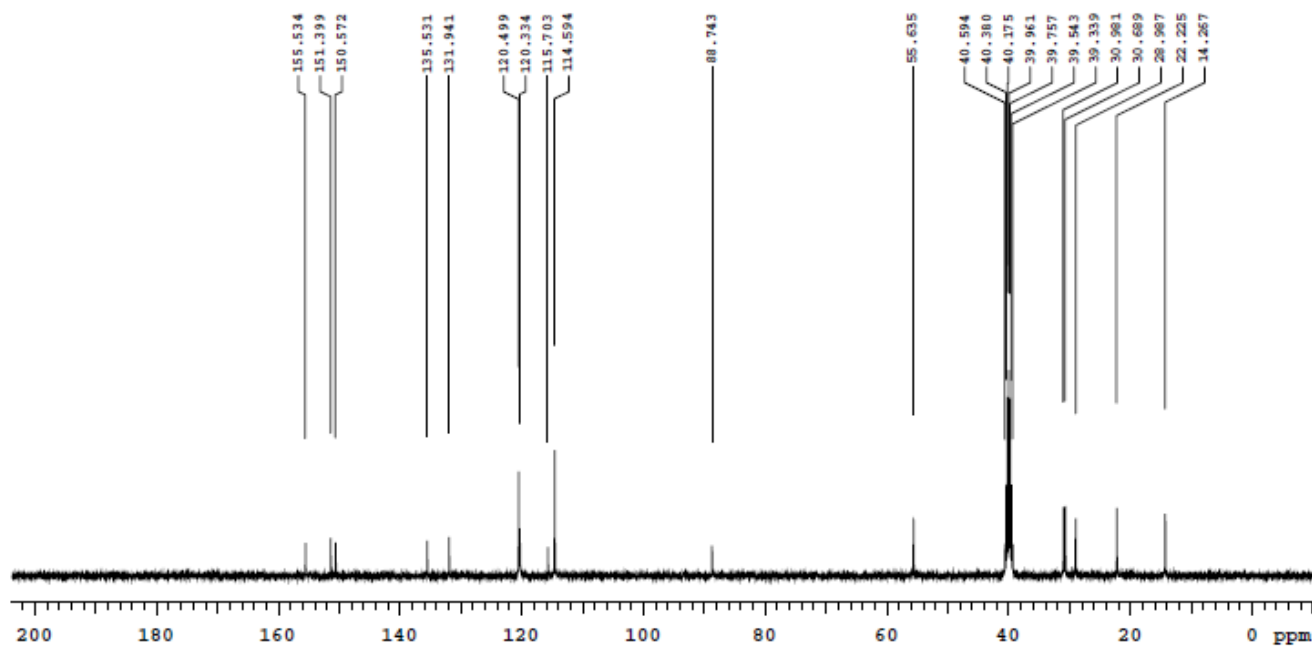


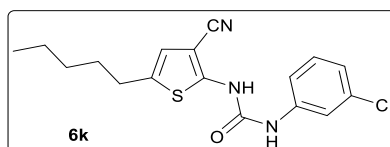


¹H-NMR spectra of 6j:

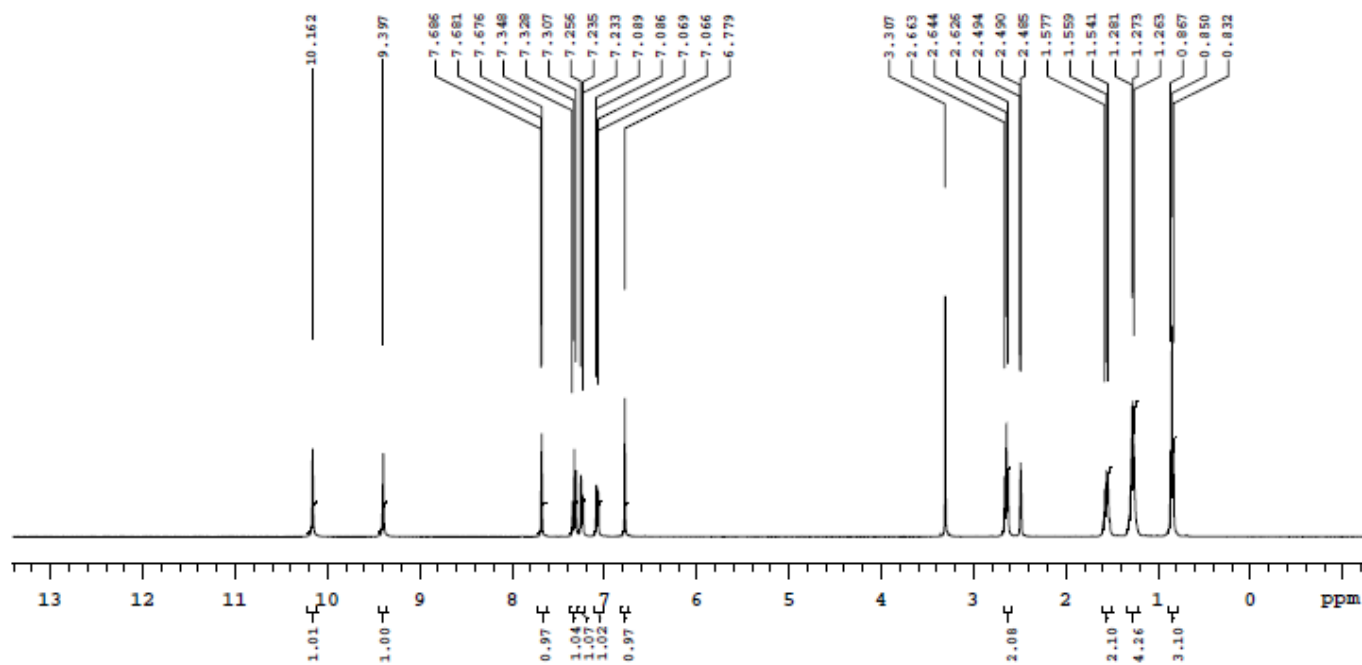


¹³C-NMR spectra of 6j:

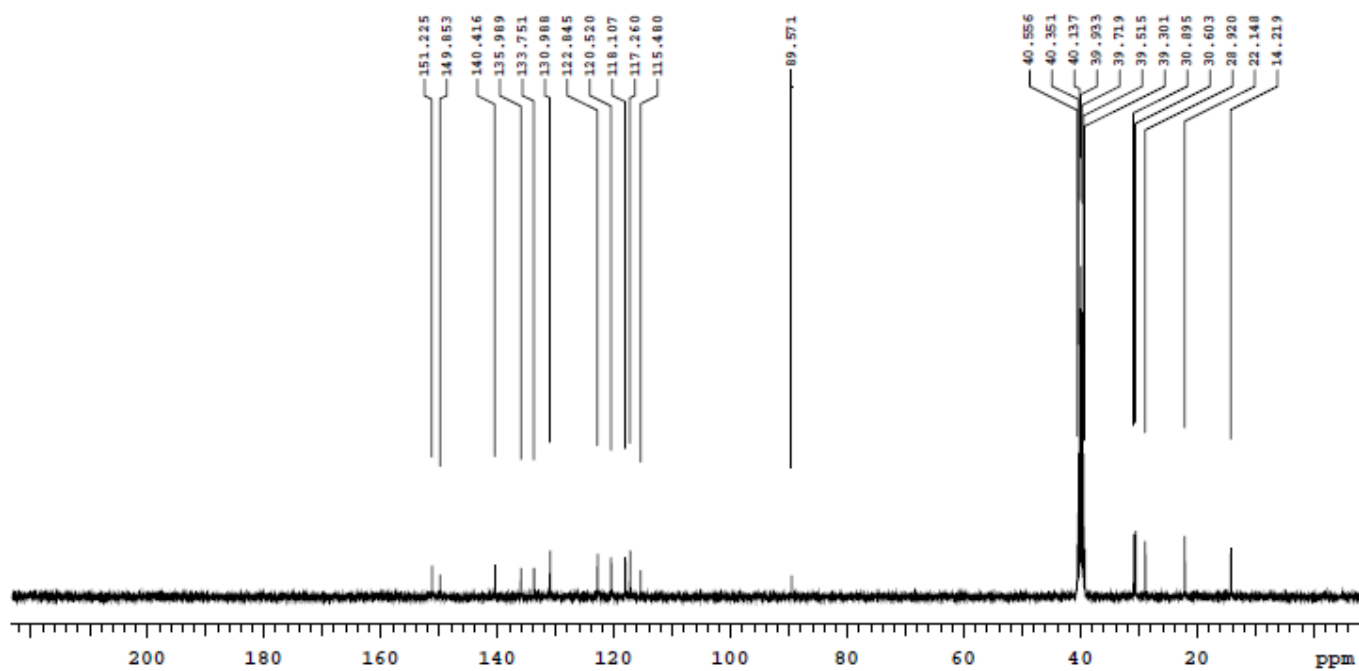


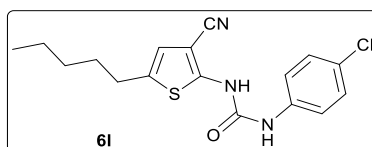


¹H-NMR spectra of 6k:

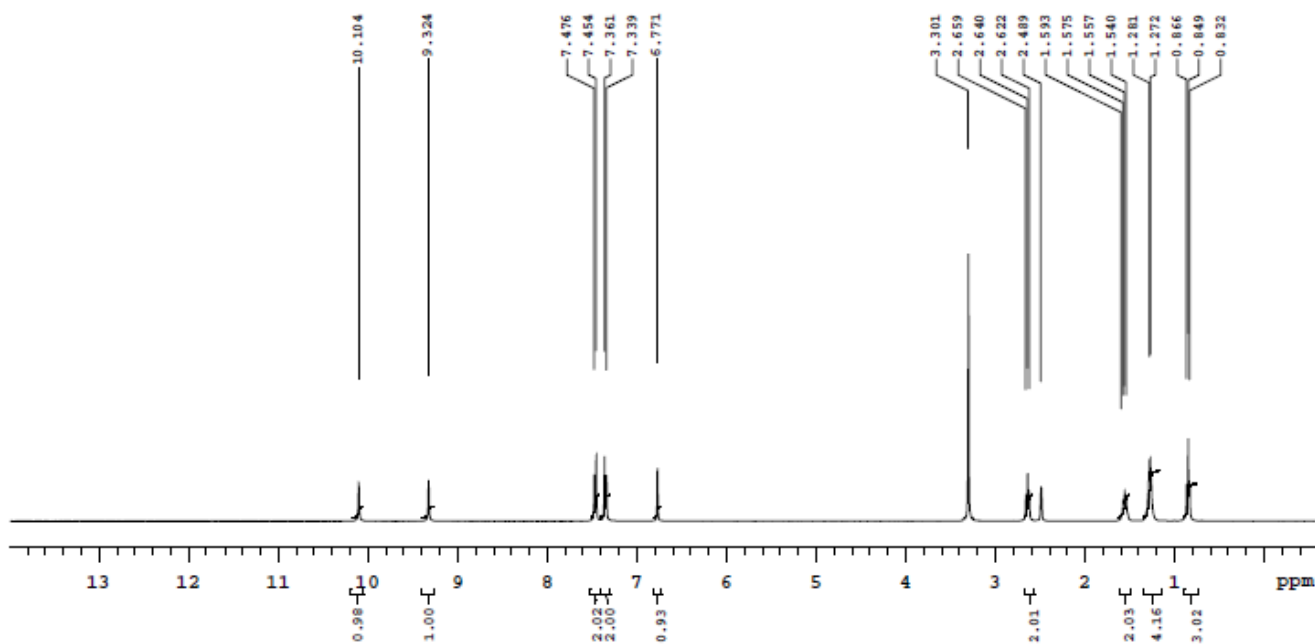


¹³C-NMR spectra of 6k:

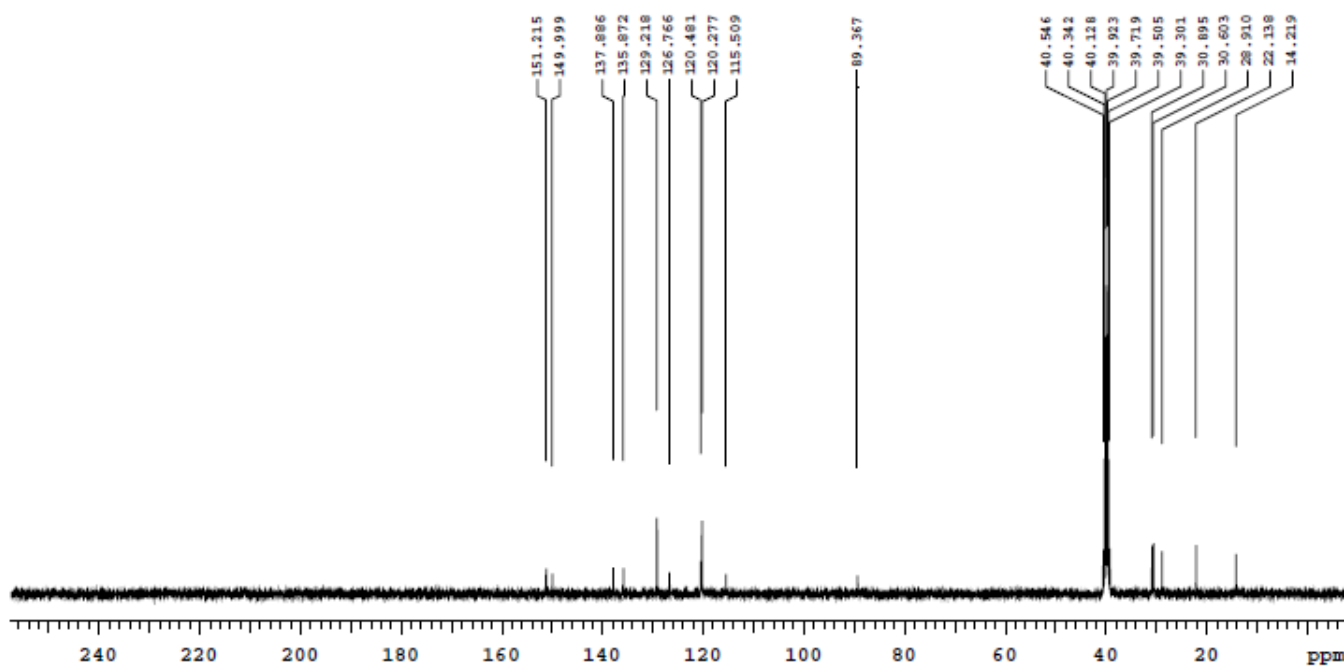


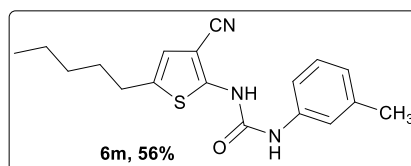


¹H-NMR spectra of 6l:

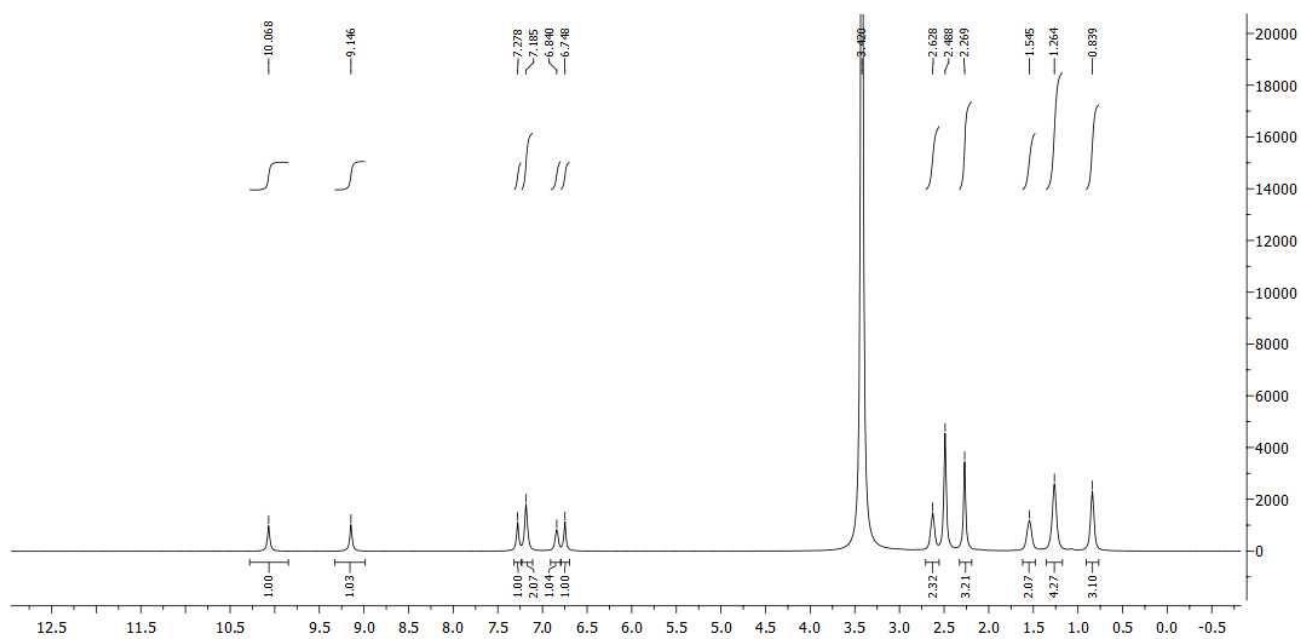


¹³C-NMR spectra of 6l:

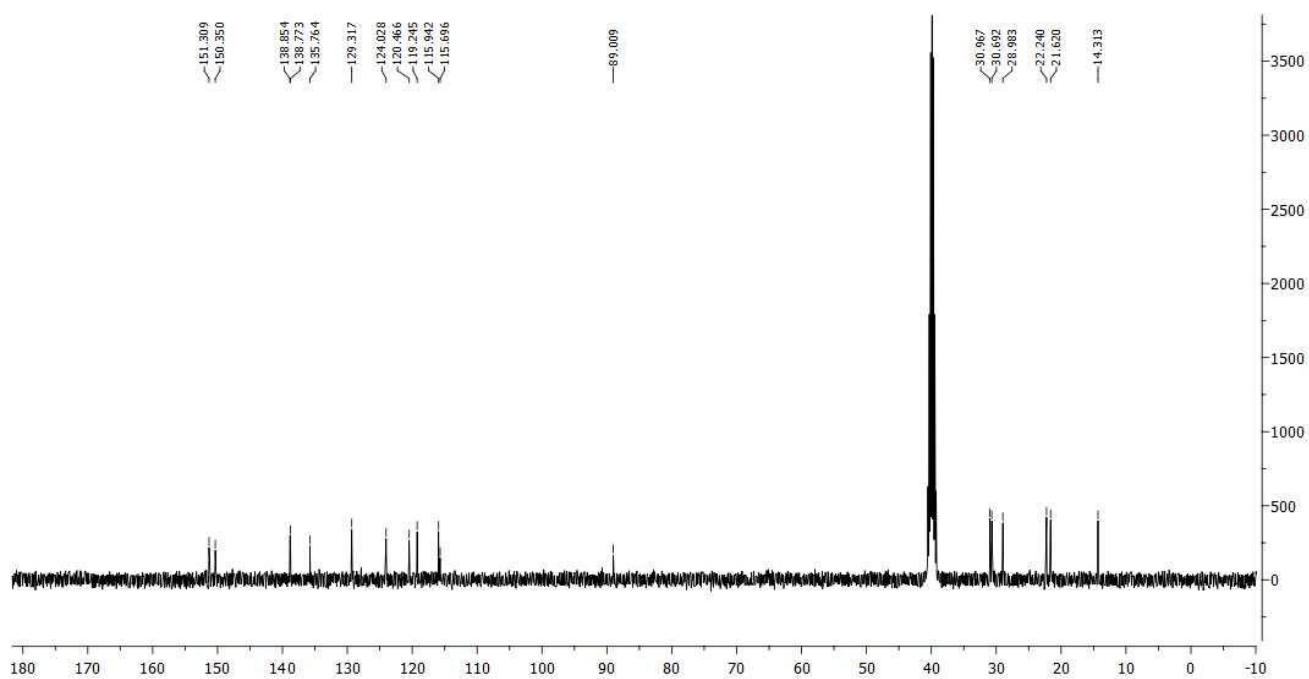


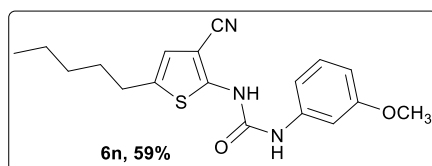


$^1\text{H-NMR}$ spectra of 6m:

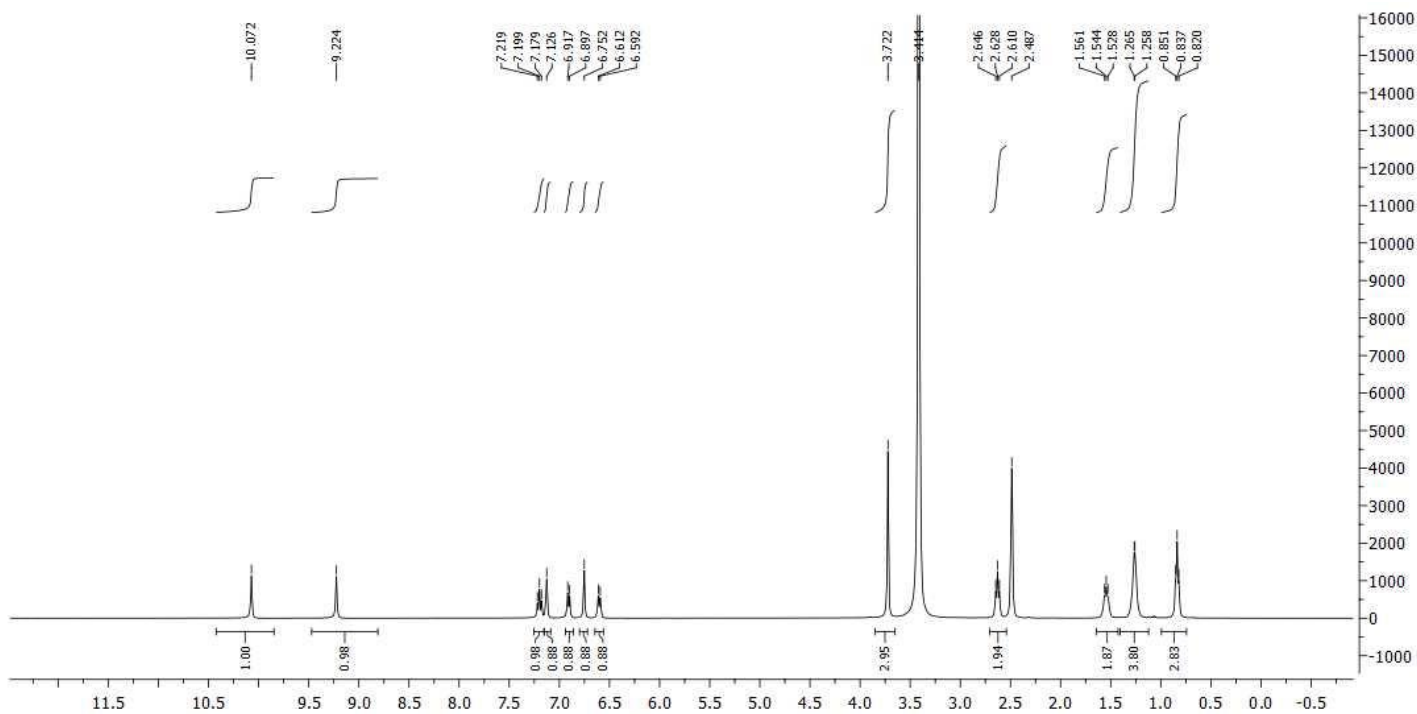


$^{13}\text{C-NMR}$ spectra of 6m:

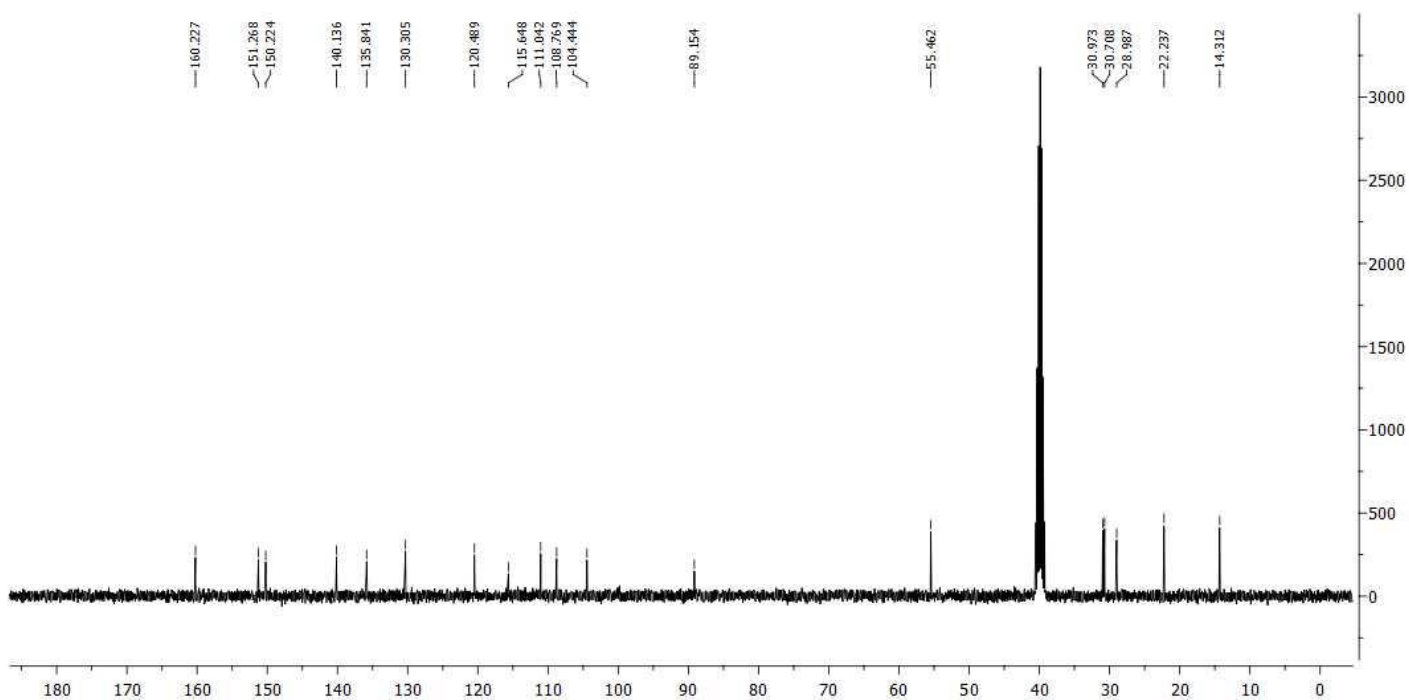


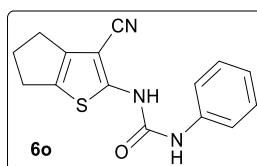


$^1\text{H-NMR}$ spectra of 6n:

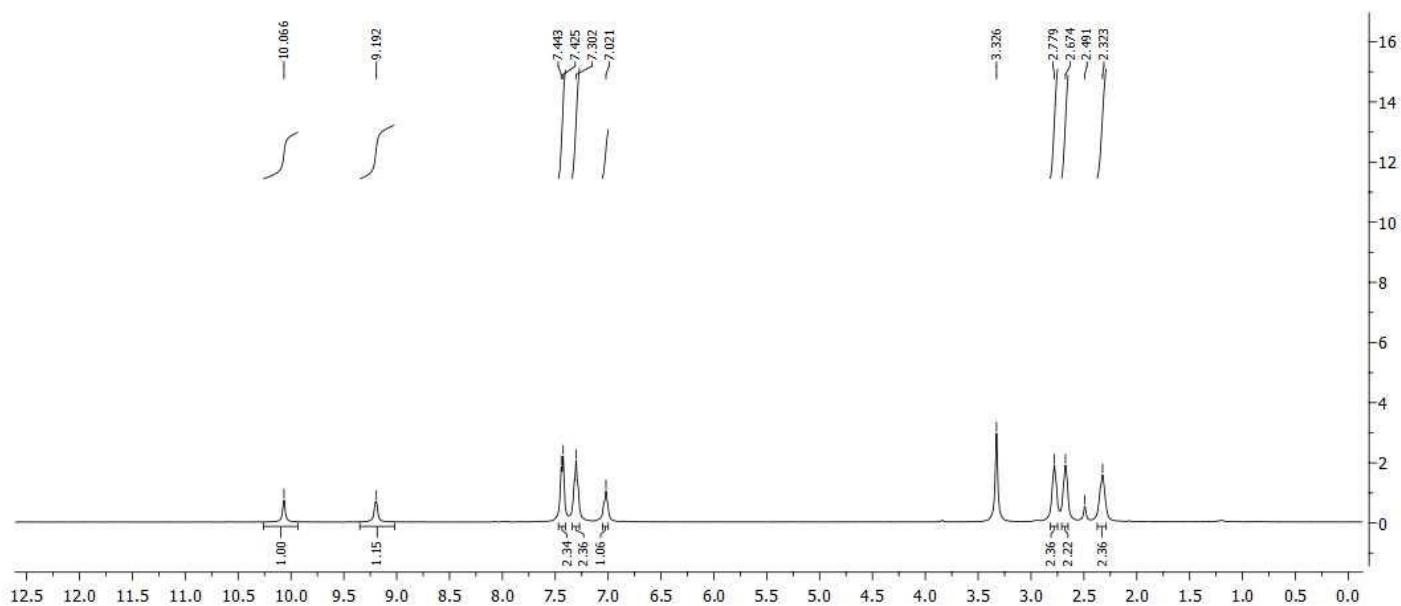


$^{13}\text{C-NMR}$ spectra of 6n:

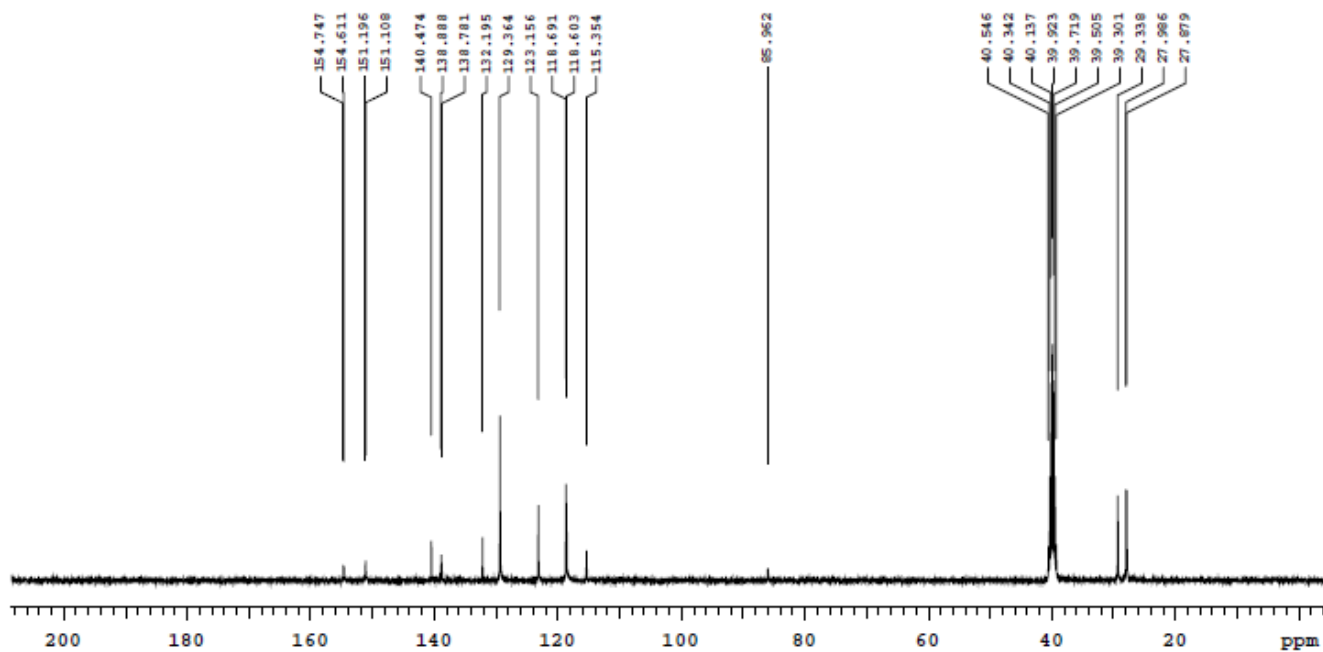


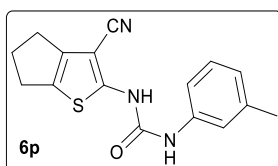


$^1\text{H-NMR}$ spectra of **6o**:

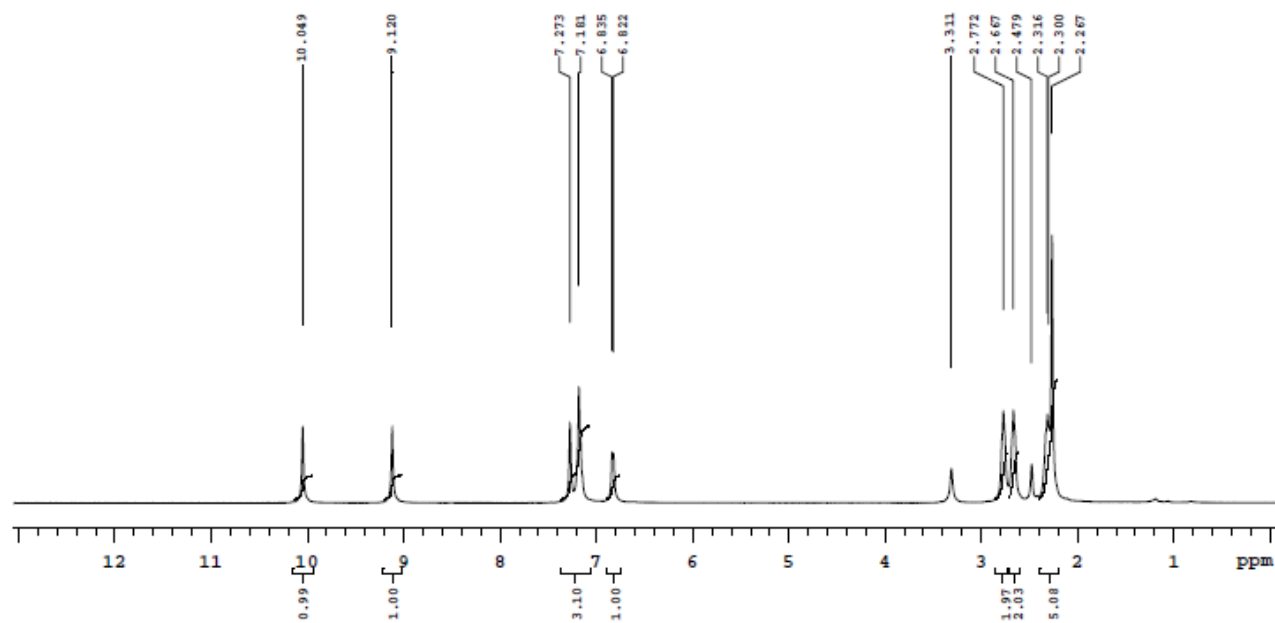


$^{13}\text{C-NMR}$ spectra of **6o**:

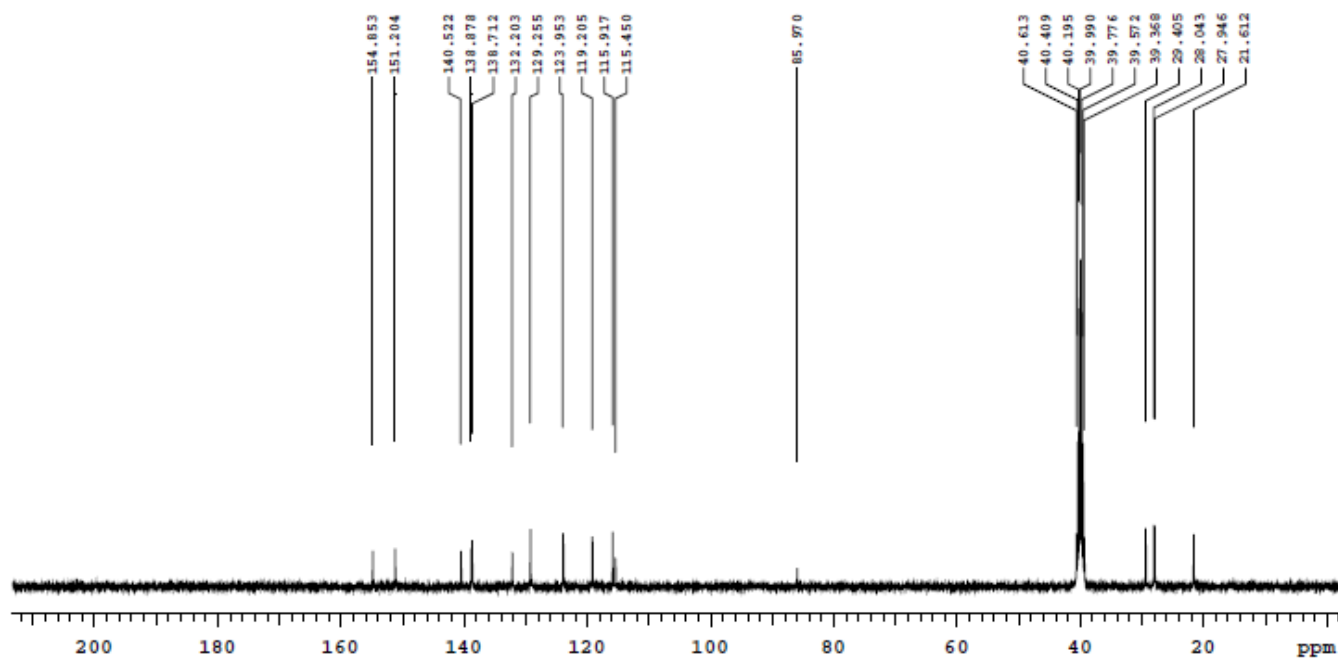


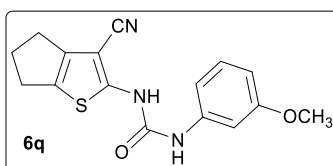


$^1\text{H-NMR}$ spectra of **6p**:

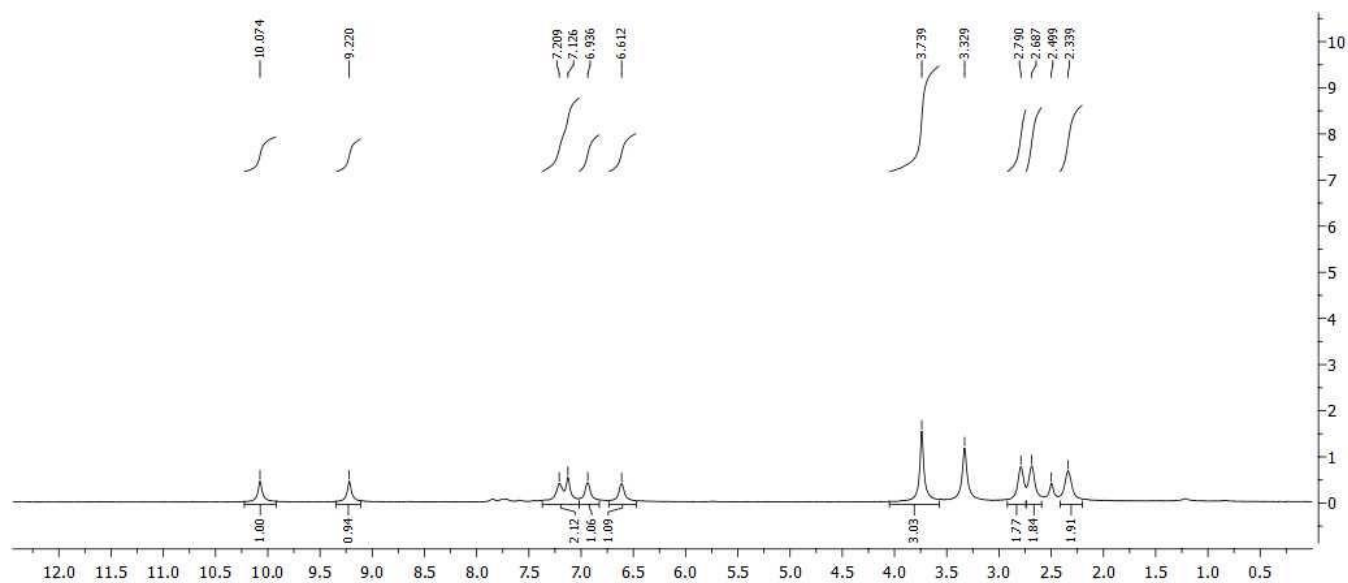


$^{13}\text{C-NMR}$ spectra of **6p**:

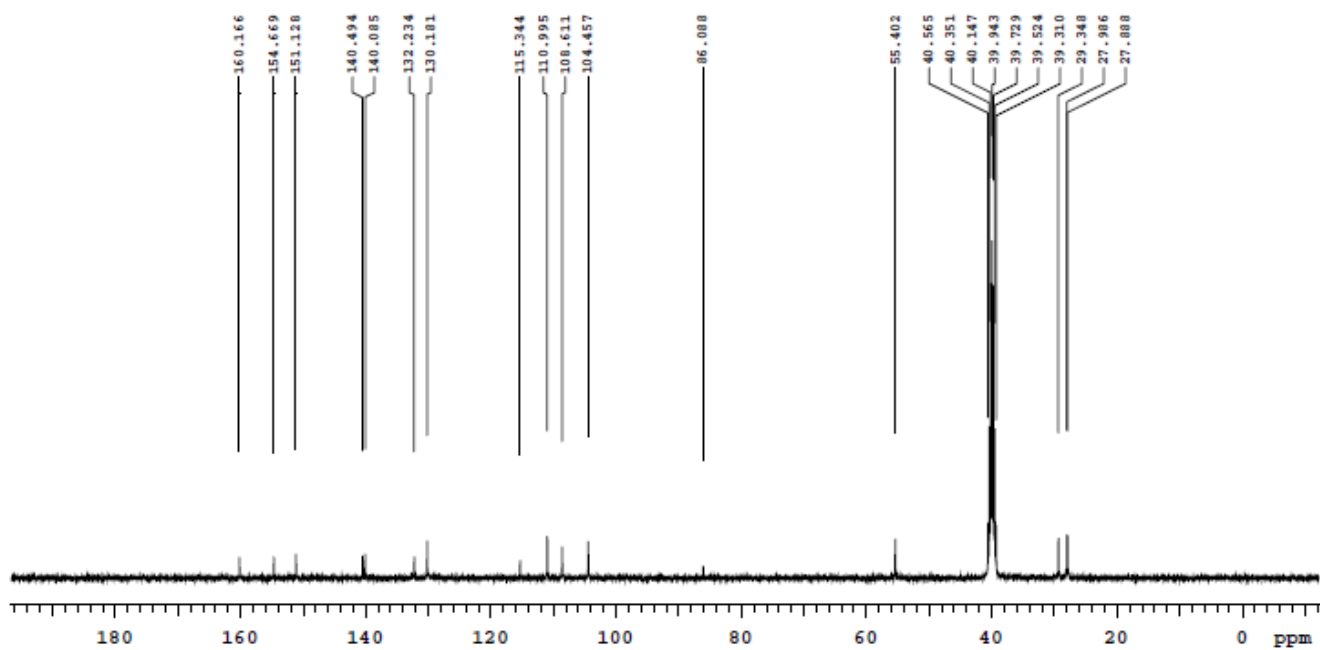


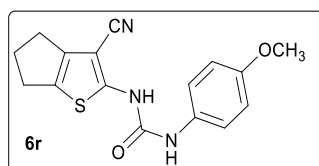


¹H-NMR spectra of 6q:

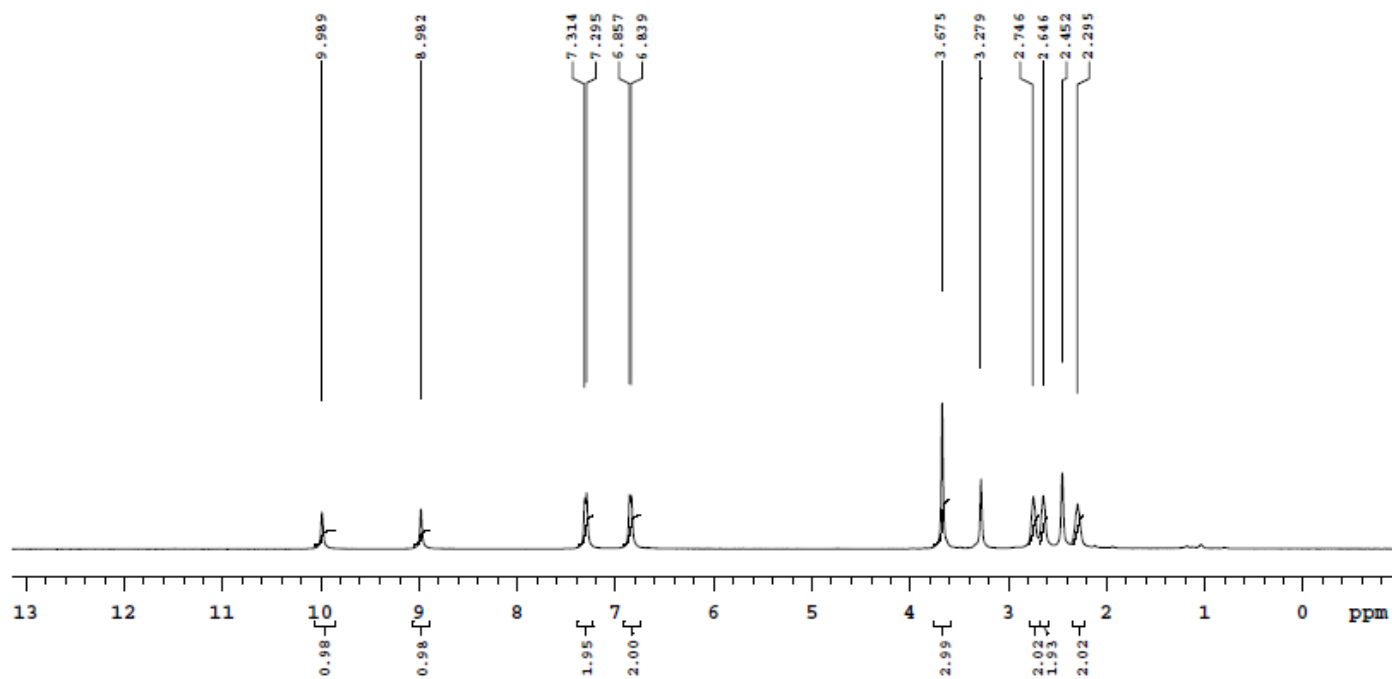


¹³C-NMR spectra of 6q:

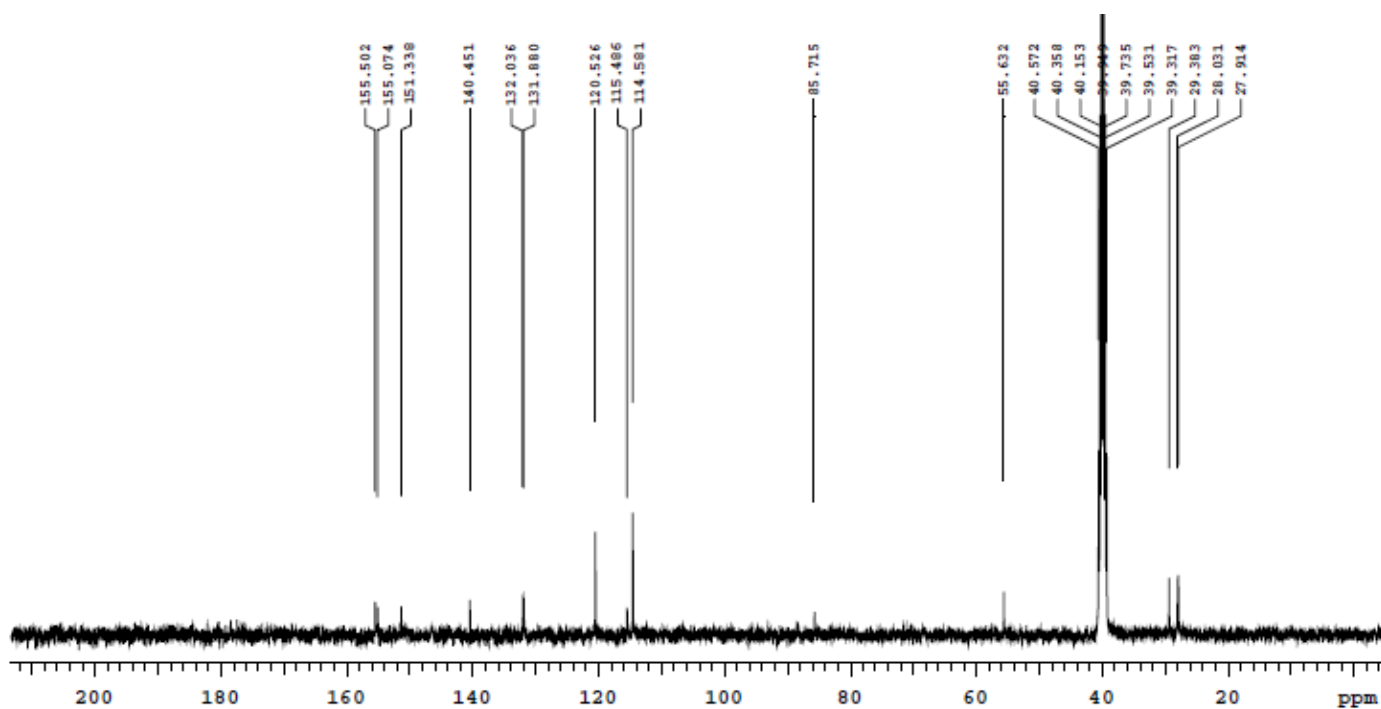


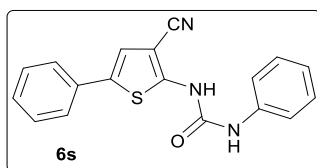


$^1\text{H-NMR}$ spectra of **6r**:

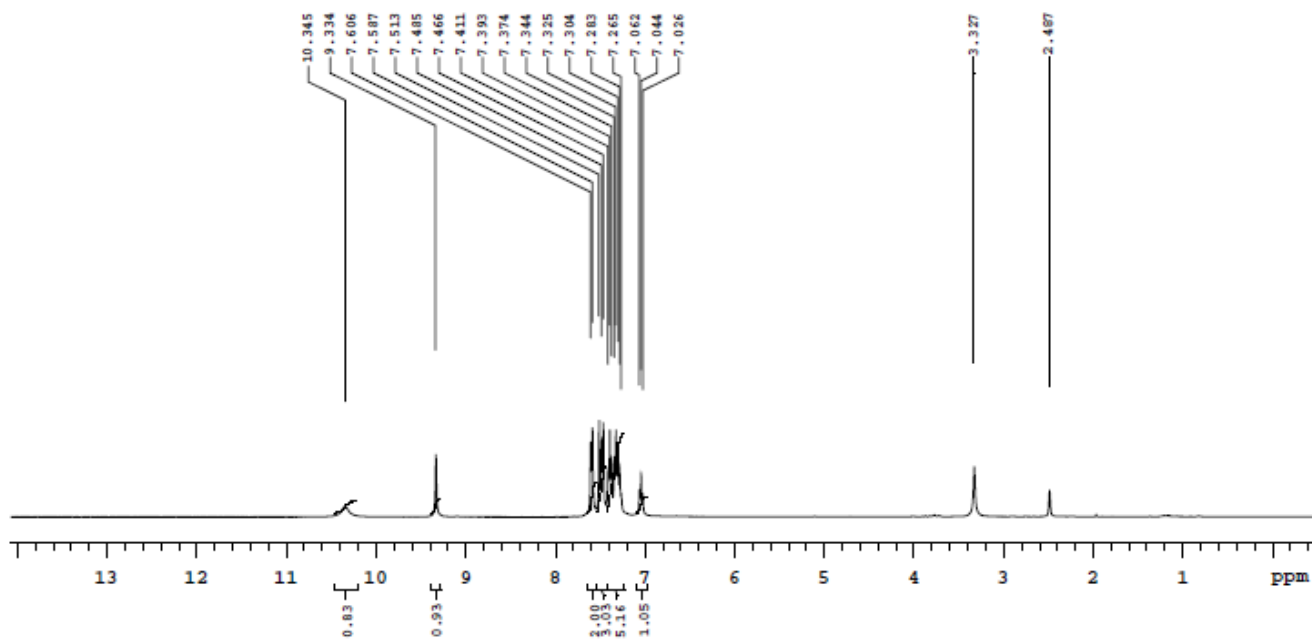


$^{13}\text{C-NMR}$ spectra of **6r**:

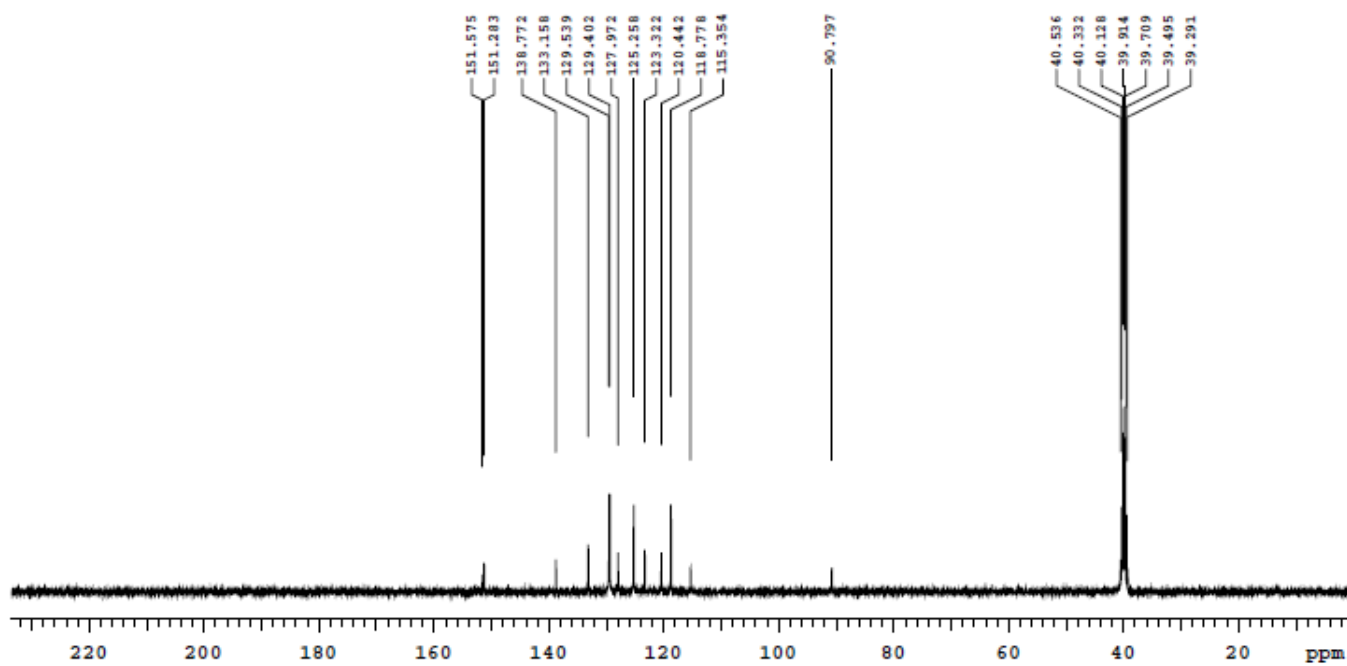


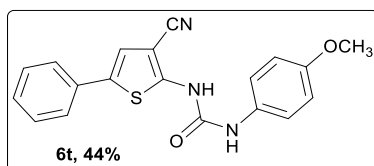


$^1\text{H-NMR}$ spectra of 6s:

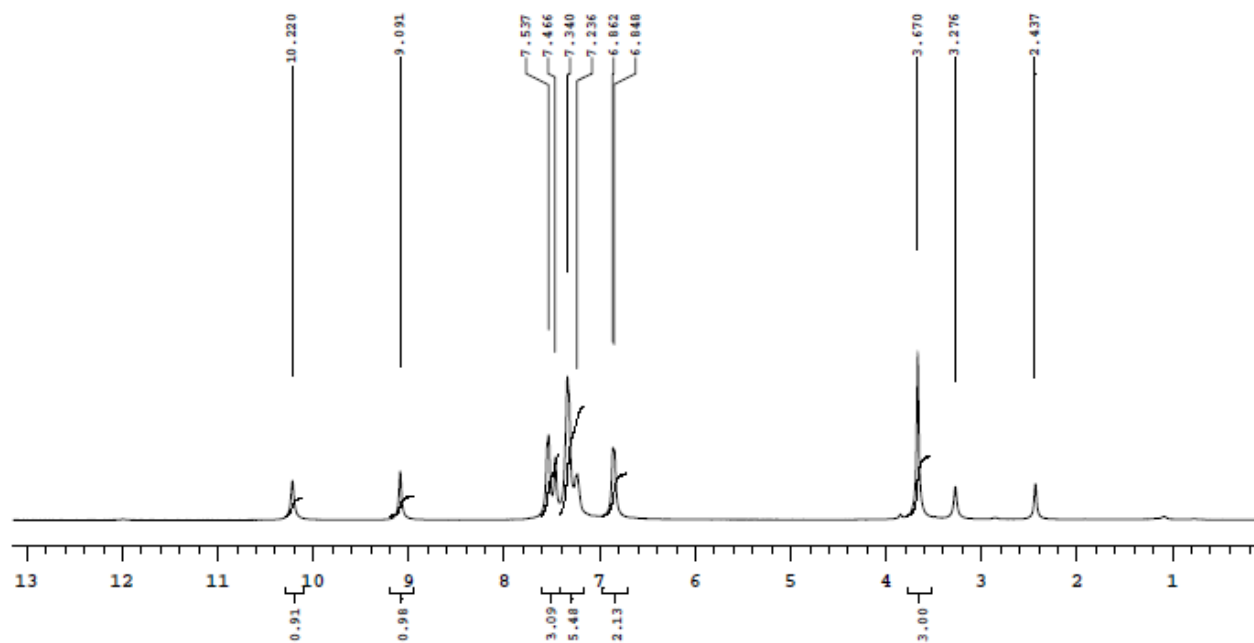


$^{13}\text{C-NMR}$ spectra of 6s:





$^1\text{H-NMR}$ spectra of 6t:



$^{13}\text{C-NMR}$ spectra of 6t:

