

Synthesis of 2,3-dihydroquinazolinones and quinazolin-4(3H)-one catalyzed by Graphene Oxide nanosheets in aqueous medium: “on-water” synthesis accompanied by carbocatalysis and selective C-C bond cleavage

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Materials and Methods

¹H-NMR and ¹³C-NMR spectral analysis were carried out on Bruker-Advance Digital 300 MHz and 75 MHz instruments where tetramethylsilane (TMS) was used as internal standard. Infrared spectra were recorded in KBr pallets in reflection mode on a Perkin Elmer RX-1 FTIR spectrophotometer. Suitable single crystal of compound **3h, 5a and 5j** was mounted on a Bruker-AXS SMART APEX II diffractometer equipped with a graphite monochromator. All the reactions were monitored by thin layer chromatography carried out on Merck aluminum-blocked silica gel plates coated with silica gel G under UV light and also by exposure to iodine vapor for detection. Melting points were recorded on a Köfler Block apparatus. Synthetic grade chemicals from Sigma-Aldrich, Spectrochem and E-Merck were used for carrying out the organic reactions.

Preparation of GO

Natural graphite powder is used for the synthesis of GO nanosheets. Graphite powder (1000 mg) and NaNO₃ (1000 mg) were added to 35ml of concentrated H₂SO₄ (98%) under vigorous stirring in a 250 ml conical flask placed in an ice bath. The whole mass was converted to black slurry (it takes 2 min), then KMnO₄ (5000 mg) was added slowly to the slurry maintaining the reaction temperature between 15 °C and 20 °C. After 3 h, the entire system was taken out of the ice bath and diluted with 100 ml water and then further stirred for 3 h at ambient temperature. 200 ml hot water was added to the above reaction mixture followed by 30% H₂O₂ until the excess permanganate and manganese dioxide had been reduced to colourless soluble manganese sulfate. The resultant yellow precipitate was washed with distilled water several times and then was subjected to centrifuge to get the pure graphene oxide powder. After repeated centrifugation, salts and ions results from the oxidation process can be removed from GO suspensions. The GO nanosheets sample was collected and dried at 60 °C for 24 h. The GO nanosheets were characterized using XRD, FTIR, FESEM and TEM images.

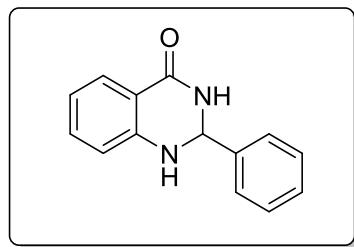
General Procedure for the synthesis of 2, 3-dihydroquinazolinones:

Anthraniilamide (1 mmol) was added to aldehyde/ketone derivative (1 mmol) in aqueous medium (3 ml water) followed by GO nanosheets (25 mg) at room temperature. The mixture was then stirred for the required period of time (indicated by TLC). After completion of each reaction, product and the catalysts separated from reaction system by simple filtration. The crude product was then dissolved in EtOH (3 ml) and this allows separation of minute quantity of the catalyst through filtration and crystallization of the crude product from EtOH furnished pure compound. All compounds were well characterized by ¹H, ¹³C NMR and FT-IR analysis.

General Procedure for the synthesis of quinazolin-4(3*H*)-one:

- a) Anthanilamide (1 mmol) was added to aldehyde/ketone derivative (1 mmol) in aqueous medium (3 ml water) followed by GO nanosheets (25 mg) and oxone (307 mg) at room temperature. The mixture was then stirred for the required period of time (indicated by TLC). After completion of each reaction, crude product was separated from reaction system by simple filtration. The crude product was then dissolved in EtOH (3 ml) and filtered. Finally crystallization of the crude product from EtOH offered the pure compound. All compounds were well characterized by ¹H, ¹³C NMR and FT-IR analysis.
- b) Anthanilamide (1 mmol) was added to 1,3-diketones derivative (1 mmol) in aqueous medium (3 ml water) followed by GO nanosheets (25 mg) at 60 °C. The mixture was then stirred for the required period of time (indicated by TLC). After completion of each reaction, the crude product mixture was extracted with ethyl acetate (2x3ml) and finally purified by column chromatography (eluent- ethyl acetate/petroleum ether: 1:4). All compounds were well characterized by ¹H, ¹³C NMR and FT-IR analysis.

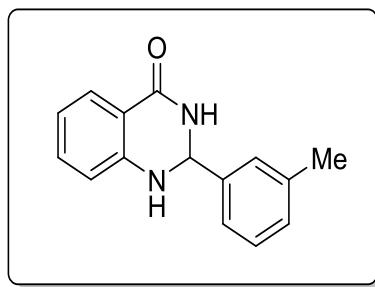
2-phenyl-2,3-dihydroquinazolin-4(1H)-one (3a)



Yield: 94%, (0.211 g); M.p. 224-226 °C; Characteristics: White crystalline solid;

¹H NMR (300 MHz, CDCl₃): δ 5.79 (s, 1H), 5.87 (s, 1H), 6.6 (d, 1H, J=7.8 Hz), 6.88 (t, 1H, J= 7.5 Hz), 7.23-7.34 (m, 1H), 7.42-7.58 (m, 3H), 7.5-7.6 (m, 2H), 7.9 (dd, 1H, J= 7.8 Hz, J= 1.5 Hz) ; ¹³C NMR (75 MHz, CDCl₃): δ 89.0, 124.7, 127.6, 127.7, 128.6, 128.7, 129.1, 131.2, 131.3, 133.4, 133.5, 139.3, 144.5, 160.1 ; IR (KBr) cm⁻¹: 1615, 1659, 2925, 3061, 3186, 3302.

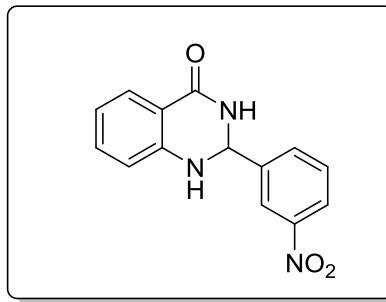
2-(*m*-tolyl)-2,3-dihydroquinazolin-4(1H)-one (3b)



Yield: 92%, (0.219 g); M.p. 185-187 °C; Characteristics: White crystalline solid;

¹H NMR (300 MHz, DMSO-d₆): δ 2.32 (s, 3H), 5.72 (s, 1H), 6.65-6.76 (m, 2H), 7.08 (s, 1H), 7.18-7.32 (m, 5H), 7.6 (d, 1H, J= 6.9 Hz), 8.26 (s, 1H); ¹³C NMR (75 MHz, DMSO-d₆): δ 21.5, 67.00, 114.8, 117.5, 124.4, 127.7, 127.9, 128.6, 129.5, 133.7, 137.8, 141.9, 148.3, 164.0; IR (KBr) cm⁻¹: 1610, 1655, 2920, 3063, 3185, 3305.

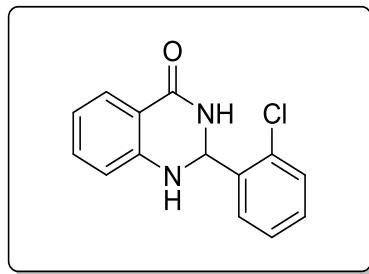
2-(3-nitrophenyl)-2,3-dihydroquinazolin-4(1*H*)-one (3c)



Yield: 96%, (0.258 g); M.p. 192-194 °C; Characteristics: Yellow crystalline solid;

¹H NMR (300 MHz, CDCl₃): δ 5.39 (s, 1H), 5.82 (s, 1H), 6.65 (t, 2H, , J=8.1 Hz), 7.13-7.18 (m, 1H), 7.34-7.44 (m, 1H), 7.62-7.69 (m, 2H), 8.1 (d, 2H, J=6.9 Hz); ¹³C NMR (75 MHz, DMSO-D₆): δ 82.6, 114.9, 117.9, 127.6, 127.8, 128.8, 130.3, 130.9, 132.4, 133.9, 141.0, 147.8, 163.9; HRMS (ESI-TOF) m/z Calculated for [C₁₄H₁₁N₃O₃+Na]⁺: 292.0693, found: 292.0695; IR (KBr) cm⁻¹: 1345, 1461, 1520, 1608, 1647, 2855, 2922, 3032, 3174, 3278.

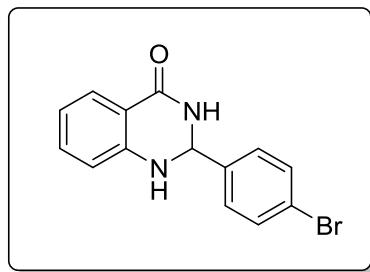
2-(2-chlorophenyl)-2,3-dihydroquinazolin-4(1*H*)-one (3d)



Yield: 95%, (0.236 g); M.p. 200-202 °C; Characteristics: White crystalline solid;

¹H NMR (300 MHz, CDCl₃): δ 6.13 (s, 1H), 6.36 (s, 1H), 6.67 (d, 1H, J= 8.1 Hz), 6.89 (t, 1H, J= 7.5Hz), 7.31-7.44 (m, 4H), 7.73-7.77 (m, 1H), 7.93 (d, 1H, J= 7.5 Hz) ; ¹³C NMR (75 MHz, CDCl₃): δ 66.2, 114.9, 115.4, 117.7, 127.8, 128.8, 129.2, 133.4, 133.9, 141.1, 148.1, 163.9; IR (KBr) cm⁻¹: 1618, 1650, 2922, 3051, 3176, 3307.

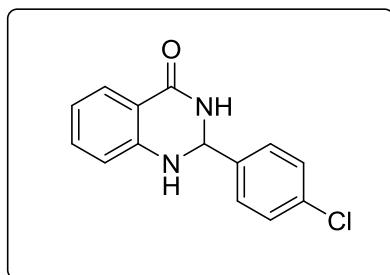
2-(4-bromophenyl)-2,3-dihydroquinazolin-4(1*H*)-one (3e)



Yield: 95%, (0.287 g); M.p. 203-205 °C; Characteristics: White crystalline solid;

¹H NMR (300 MHz, CDCl₃): δ 5.74 (s, 1H), 6.02 (s, 1H), 6.55-6.71 (m, 2H), 7.17-7.44 (m, 6H), 7.74 (s, 1H); ¹³C NMR (75 MHz, CDCl₃): δ 66.3, 114.9, 115.5, 117.8, 127.8, 128.8, 129.2, 133.4, 133.9, 141.2, 148.1, 163.9; IR (KBr) cm⁻¹: 16012, 1662, 2932, 3062, 3180, 3310.

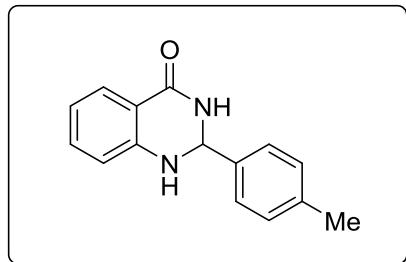
2-(4-chlorophenyl)-2,3-dihydroquinazolin-4(1*H*)-one (3f)



Yield: 95%, (0.245 g); M.p. 197-198 °C; Characteristics: White crystalline solid;

¹H NMR (300 MHz, CDCl₃): δ 5.82 (s, 1H), 5.90(s, 1H), 6.69 (d, 1H, *J*= 8.1 Hz), 6.92 (t, 1H, *J*= 7.7 Hz) 7.33-7.44(m, 2H), 7.43 (d, 2H, *J*= 8.4 Hz), 7.55 (d, 2H, *J*= 8.4 Hz), 7.93-7.96 (m, 1H); ¹³C NMR (75 MHz, CDCl₃): δ 66.2, 114.9, 115.4, 117.7, 127.8, 128.8, 129.2, 133.4, 133.9, 141.1, 148.1, 163.9 ; IR (KBr) cm⁻¹: 1607, 1658, 2927, 3060, 3188, 3306.

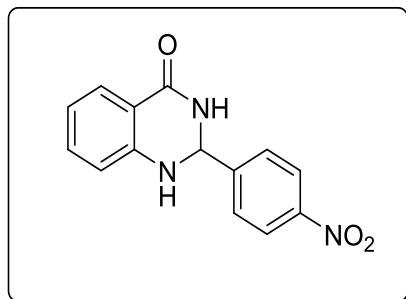
2-(*p*-tolyl)-2,3-dihydroquinazolin-4(1*H*)-one (3g)



Yield: 92%, (0.219 g); M.p. 232-233 °C; Characteristics: White crystalline solid;

¹H NMR (300 MHz, CDCl₃): δ 2.39 (s, 3H), 5.78 (s, 1H), 5.87 (s, 1H), 6.67 (d, 1H, J= 8.1Hz), 6.90 (t, 1H, J= 7.5Hz), 7.26-7.42 (m, 5H), 7.95 (d, 1H, J= 7.8Hz) ; ¹³C NMR (75 MHz, CDCl₃): δ 21.4, 67.10, 114.8, 117.5, 124.4, 127.7, 127.9, 128.6, 129.5, 133.7, 137.8, 141.9, 148.3, 154.1, 164.1; IR (KBr) cm⁻¹: 1512, 1607, 1660, 1908, 2855, 2924, 3060, 3189, 3312.

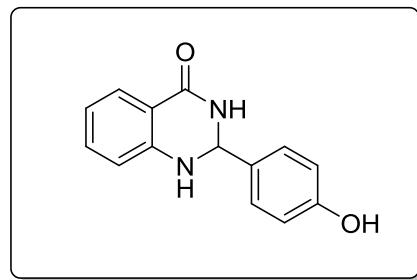
2-(4-nitrophenyl)-2,3-dihydroquinazolin-4(1*H*)-one (3h)



Yield: 97%, (0.261 g); M.p. 200-202 °C; Characteristics: Yellow crystalline solid;

¹H NMR (300 MHz, CDCl₃): δ 6.05 (s, 1H), 6.09 (s, 1H) 6.71 (d, 1H, J= 7.8 Hz), 6.94 (t, 1H, J= 7.5Hz), 7.37 (t, 1H, J= 7.8Hz), 7.79 (d, 2H, J= 8.1 Hz), 7.94 (d, 1H, J= 7.8 Hz), 8.30 (d, 2H, J= 7.8 Hz) ; ¹³C NMR (75 MHz, DMSO-D₆): δ 79.9, 114.9, 117.9, 127.6, 127.8, 128.8, 130.3, 130.9, 132.4, 133.9, 141.0, 147.7, 163.9; IR (KBr) cm⁻¹: 1525, 1615, 1643, 2856, 2923, 3030, 3179, 3288.

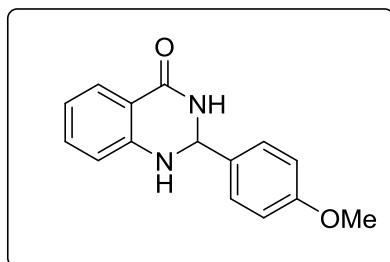
2-(4-hydroxyphenyl)-2,3-dihydroquinazolin-4(1*H*)-one (3i)



Yield: 88%, (0.211g); M.p. 210-212 °C; Characteristics: White crystalline solid;

¹H NMR (300 MHz, CDCl₃): δ 4.43 (s, 1H), 5.78 (s, 1H), 5.86 (s, 1H), 6.67(d, 1H, *J*=8.1 Hz), 6.90 (t, 1H, *J*=7.5 Hz), 7.26-7.42 (m, 5H), 7.95 (d, 1H, *J*=7.8 Hz), 8.33 (s, 1H) ; ¹³C NMR (75 MHz, DMSO-d₆): δ 66.8, 114.0, 114.8, 115.5, 117.5, 127.7, 128.6, 133.7, 133.9, 148.4 159.8, 164.1; IR (KBr) cm⁻¹: 1622, 1655, 2930, 3062, 3179, 3302, 3342.

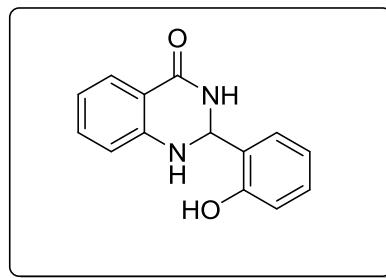
2-(4-methoxyphenyl)-2,3-dihydroquinazolin-4(1*H*)-one (3j)



Yield: 89%, (0.226 g); M.p. 177-178 °C; Characteristics: White crystalline solid;

¹H NMR (300 MHz, DMSO-D₆): δ 3.77 (s, 3H), 5.72 (s, 1H), 6.65-6.76 (m, 2H), 7.17-7.32 (m, 6H), 7.61 (d, 1H, *J*=6.9 Hz) 8.26 (s, 1H) ; ¹³C NMR (75 MHz, DMSO-D₆): δ 55.6, 66.7, 114.0, 114.8, 115.4, 117.5, 127.7, 128.6, 133.6, 133.9, 148.4 159.8, 164.1; IR (KBr) cm⁻¹: 1473, 1599, 1676, 2923, 3183, 3315, 3448.

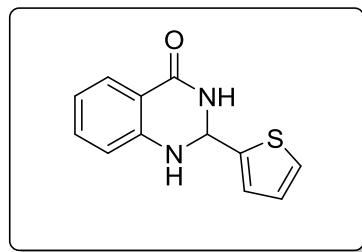
2-(2-hydroxyphenyl)-2,3-dihydroquinazolin-4(1H)-one (3k)



Yield: 89%, (0.214 g); M.p. 220-221 °C; Characteristics: White crystalline solid;

¹H NMR (300 MHz, CDCl₃): δ 4.61 (s, 1H), 5.92 (s, 1H), 5.98 (s, 1H), 6.54-6.72 (m, 4H), 6.77 (d, 1H, J=7.8 Hz), 7.02 (d, 1H, J=7.5 Hz), 7.11-7.33 (m, 2H), 8.91 (s, 1H); ¹³C NMR (75 MHz, DMSO-D₆): δ 69.9, 128.4, 128.5, 128.6, 129.0, 132.1, 132.7, 133.9, 135.8, 140.5, 156.7, 166.6 ; IR (KBr) cm⁻¹: 1618, 1663, 2932, 3056, 3176, 3306, 3340.

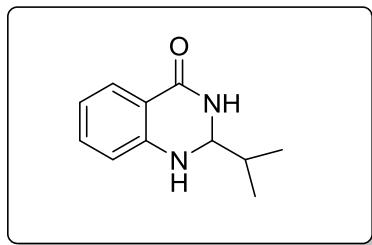
2-(thiophen-2-yl)-2,3-dihydroquinazolin-4(1H)-one (3l)



Yield: 90%, (0.207 g); M.p. 191-193 °C; Characteristics: White crystalline solid;

¹H NMR (300 MHz, CDCl₃): δ 5.95 (s, 1H), 6.21 (s, 1H), 6.71 (d, 1H, J=7.5 Hz), 6.93 (t, 1H, J=8.1 Hz), 7.02-7.05 (m, 1H), 7.23-7.42 (m, 3H), 7.95 (d, 1H, J=5.7) ; ¹³C NMR (75 MHz, CDCl₃): δ 62.1, 112.9, 113.9, 121.0, 126.5, 126.7, 127.6, 134.9, 143.4, 145.4, 146.2, 160.0; IR (KBr) cm⁻¹: 1376, 1457, 1651, 1763, 2853, 2923, 3448.

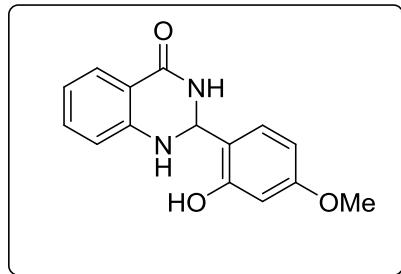
2-isopropyl-2,3-dihydroquinazolin-4(1H)-one (3m)



Yield: 88%, (0.167 g); M.p. 180-182 °C; Characteristics: White crystalline solid;

¹H NMR (300 MHz, CDCl₃): δ 0.93 (d, 6H, J=6.9 Hz), 1.89 (m, 1H), 4.59 (d, 1H, J=6.9 Hz), 6.59-6.69 (m, 4H), 7.18 (s, 1H), 7.74 (s, 1H); ¹³C NMR (75 MHz, DMSO-D₆): δ 16.9, 17.3, 33.2, 69.6, 114.5, 115.1, 116.9, 127.6, 133.4, 148.9, 164.3; IR (KBr) cm⁻¹: 1372, 1457, 1656, 1763, 2853, 2923, 3206, 3448.

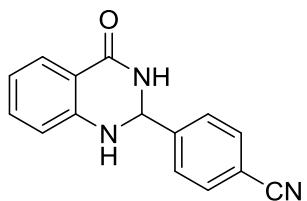
2-(2-hydroxy-4-methoxyphenyl)-2,3-dihydroquinazolin-4(1H)-one (3n)



Yield: 87%, (0.235 g); M.p. 227-228 °C; Characteristics: White crystalline solid;

¹H NMR (300 MHz, DMSO-D₆): δ 3.72 (s, 3H), 4.92 (s, 1H), 5.42 (s, 1H), 5.94 (s, 1H), 6.58-6.70 (m, 3H), 7.03-7.16 (m, 2H), 7.19 (t, 1H, J=7.2 Hz), 7.53 (d, 1H, J=7.5 Hz); ¹³C NMR (75 MHz, DMSO-D₆): δ 55.7, 66.8, 114.0, 114.8, 115.4, 117.5, 127.7, 128.6, 133.6, 133.9, 148.4, 159.8, 164.2; IR (KBr) cm⁻¹: 1473, 1592, 1676, 2937, 3182, 3312, 3320, 3445.

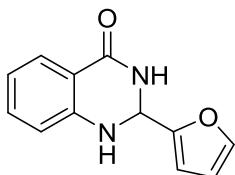
4-(4-oxo-1,2,3,4-tetrahydroquinazolin-2-yl)benzonitrile (3o)



Yield: 92%, (0.229 g); M.p. 178-180 °C; Characteristics: White crystalline solid;

^1H NMR (300 MHz, CDCl_3): 5.78 (s, 1H), 5.86 (s, 1H), 6.67 (d, 1H, $J=8.1$ Hz), 6.90 (t, 1H, $J=7.5$ Hz), 7.26-7.42 (m, 6H), 8.49 (s, 1H) ; ^{13}C NMR (75 MHz, DMS)-d₆): δ 76.9, 114.9, 118.1, 127.6, 127.8, 128.8, 130.3, 130.9, 132.4, 133.9, 141.1, 147.7, 1653.0; IR (KBr) cm⁻¹: 1475, 1595, 1676, 2208, 2923, 3180, 3316, 3445.

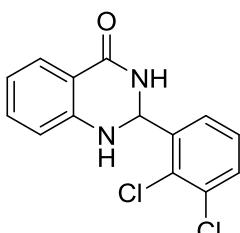
2-(furan-2-yl)-2,3-dihydroquinazolin-4(1H)-one (3p)



Yield: 90%, (0.193 g); M.p. 166-167 °C; Characteristics: White crystalline solid;

^1H NMR (300 MHz, CDCl_3): δ 5.76 (s, 1H), 6.72 (s, 1H), 7.25-7.49 (m, 3H), 7.61 (s, 1H), 7.73 (d, 2H, $J=6.9$ Hz), 8.24 (d, 2H, $J=6.9$ Hz) ; ^{13}C NMR (75 MHz, CDCl_3): δ 60.0, 112.9, 113.9, 121.0, 126.5, 126.7, 127.6, 134.9, 143.4, 145.4, 146.2, 149.2; IR (KBr) cm⁻¹: 1673, 2928, 3174, 3309, 3446.

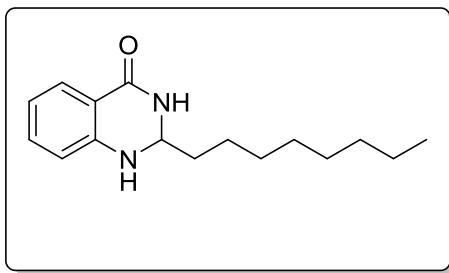
2-(2,3-dichlorophenyl)-2,3-dihydroquinazolin-4(1H)-one (3q)



Yield: 94%, (0.274g); M.p. 182-184 °C; Characteristics: White crystalline solid;

¹H NMR (300 MHz, CDCl₃): δ 6.16 (s, 1H), 6.62-6.66 (m, 2H), 7.11-7.17 (m, 2H), 7.35 (dd, 1H, J=8.1 Hz, J=1.5 Hz), 7.44 (s, 1H), 7.49-7.53 (m, 2H), 7.68 (dd, 1H, J=7.9 Hz, J=1.5 Hz) ; ¹³C NMR (75 MHz, DMSO-D₆): δ 64.7, 114.9, 117.9, 127.6, 127.8, 128.8, 130.3, 130.9, 132.4, 133.9, 141.1, 147.7, 163.8 ; IR (KBr) cm⁻¹: 1618, 1656, 2922, 3178, 3311, 3439.

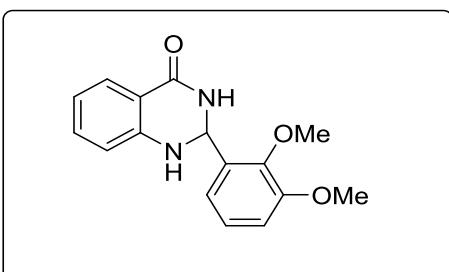
2-octyl-2,3-dihydroquinazolin-4(1H)-one (3r)



Yield: 85%, (0.221g); M.p. 238-240 °C; Characteristics: White crystalline solid;

¹H NMR (300 MHz, CDCl₃): δ 0.92 (t, 3H, J=6.9 Hz), 1.10-1.38 (m, 14H), 6.00 (s, 1H), 6.73 (t, 1H, J=7.5 Hz), 7.19-7.24 (m, 3H), 8.49 (s, 1H); ¹³C NMR (75 MHz, CDCl₃): δ 16.9, 17.3, 23.9, 26.2, 29.6, 33.2, 69.9, 114.5, 115.1, 116.9, 127.6, 133.4, 148.9, 164.3; IR (KBr) cm⁻¹: 1617, 2928, 3174, 3309, 3443.

2-(2,3-dimethoxyphenyl)-2,3-dihydroquinazolin-4(1H)-one (3s)

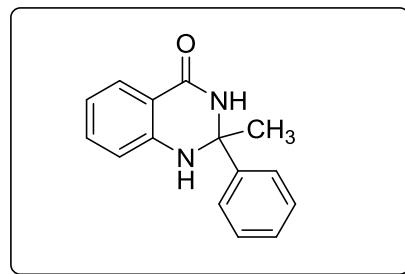


Yield: 89%, (0.253 g); M.p. 211-213 °C; Characteristics: White crystalline solid;

¹H NMR (300 MHz, DMSO-D₆): δ 3.75 (s, 6H), 5.94 (s, 1H), 6.58-6-70 (m, 3H), 6.98 (s, 1H), 7.16 (t, 2H, J=7.5 Hz), 7.53 (d, 2H, J=7.5 Hz), 7.9 (s, 1H) ; ¹³C NMR (75 MHz, DMSO-D₆): δ 56.2, 61.1, 61.6,

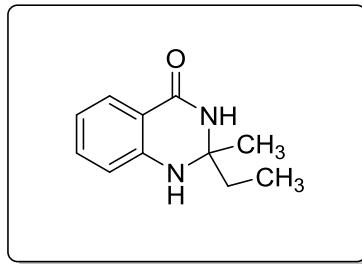
113.4, 114.8, 115.1, 117.4, 119.5, 124.3, 127.7, 133.6, 134.9, 146.5, 148.4, 152.7, 164.1; IR (KBr) cm^{-1} : 1622, 2924, 3170, 3319, 3438.

2-methyl-2-phenyl-2,3-dihydroquinazolin-4(1*H*)-one (5a)



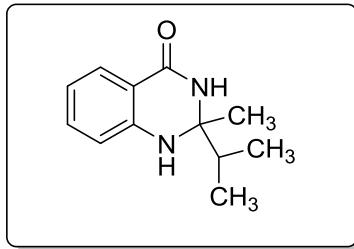
Yield: 86%, (0.205 g); M.p. 227-228 $^{\circ}\text{C}$; Characteristics: White crystalline solid;
 ^1H NMR (300 MHz, DMSO- D_6): δ 1.63 (s, 3H), 6.55 (t, 1H, $J=7.5$ Hz), 6.76 (d, 1H, $J=8.1$ Hz), 7.19 (d, 1H, $J=7.2$ Hz), 7.28 (t, 2H, $J=7.2$ Hz), 7.46-7.50 (m, 3H), 7.66 (s, 1H), 8.79 (s, 1H); ^{13}C NMR (75 MHz, DMSO- D_6): δ 31.1, 70.5, 114.7, 115.4, 117.2, 125.6, 127.4, 127.6, 128.3, 133.7, 147.6, 148.1, 164.2; IR (KBr) cm^{-1} : 1622, 1477, 1658, 2933, 3060, 3181, 3310.

2-ethyl-2-methyl-2,3-dihydroquinazolin-4(1*H*)-one (5b)



Yield: 85%, (0.162 g); M.p. 180-182 $^{\circ}\text{C}$; Characteristics: White crystalline solid;
 ^1H NMR (300 MHz, CDCl_3): δ 0.97 (t, 3H, $J=6.5$ Hz), 1.42 (s, 3H), 1.73 (q, 2H, $J=7.5$ Hz), 6.37 (s, 1H), 6.54 (d, 1H, $J=8.1$ Hz), 6.73 (t, 1H, $J=7.5$ Hz), 7.19-7.24 (m, 2H), 7.79 (d, 1H, $J=6.6$ Hz); ^{13}C NMR (75 MHz, CDCl_3): δ 8.1, 27.5, 34.8, 70.1, 114.4, 118.5, 128.3, 133.9, 160.6; IR (KBr) cm^{-1} : 1612, 1489, 1653, 2925, 3186, 3300.

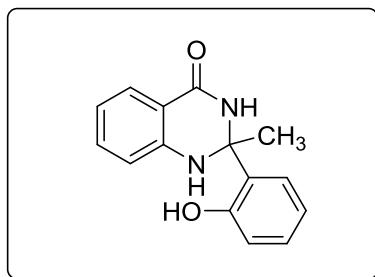
2-isopropyl-2-methyl-2,3-dihydroquinazolin-4(1*H*)-one (5c)



Yield: 89%, (0.182 g); M.p. 189-190 °C; Characteristics: White crystalline solid;

¹H NMR (300 MHz, CDCl₃): δ 0.81 (d, 6H, *J*=6.6 Hz), 1.29 (s, 3H), 1.74-1.78 (m, 1H), 6.47-6.55 (m, 2H), 7.08-7.16 (m, 1H), 7.47 (t, 1H, *J*=6.8 Hz), 7.78 (s, 1H), 7.83 (s, 1H); ¹³C NMR (75 MHz, CDCl₃): δ 18.9, 27.5, 34.8, 70.1, 114.5, 118.5, 128.3, 133.9, 143.8, 160.6; IR (KBr) cm⁻¹: 1610, 1485, 1513, 1658, 2931, 3186, 3301.

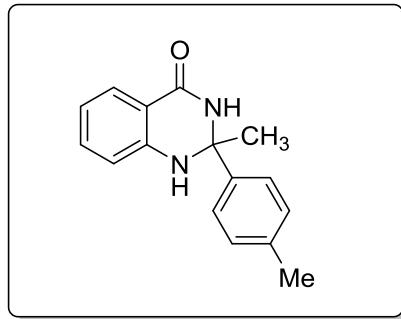
2-(2-hydroxyphenyl)-2-methyl-2,3-dihydroquinazolin-4(1H)-one (5d)



Yield: 84%, (0.213 g); M.p. 255-257 °C; Characteristics: White crystalline solid;

¹H NMR (300 MHz, CDCl₃): δ 1.35 (s, 3H), 6.42-6.66 (m, 4H), 7.09 (t, 1H, *J*=7.7 Hz), 7.20 (t, 1H, *J*=7.5 Hz), 7.49-7.57 (m, 2H), 7.68 (s, 1H), 7.90 (s, 1H); ¹³C NMR (75 MHz, CDCl₃): δ 24.1, 70.5, 114.7, 115.4, 117.2, 125.6, 127.4, 127.6, 128.3, 133.7, 147.6, 148.1, 164.3; IR (KBr) cm⁻¹: 1616, 1482, 1518, 1656, 2929, 3060, 3187, 3310, 3361.

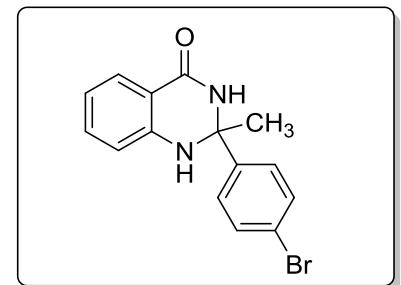
2-methyl-2-(p-tolyl)-2,3-dihydroquinazolin-4(1H)-one (5e)



Yield: 83%, (0.209 g); M.p. 228-230; Characteristics: White crystalline solid;

¹H NMR (300 MHz, CDCl₃): δ 1.78 (s, 3H), 2.38 (s, 3H), 6.53-6.73 (m, 2H), 6.89 (s, 1H), 7.03 (d, 1H, J=7.5 Hz), 7.16-7.24 (m, 2H), 7.34 (d, 2H, J=8.4 Hz), 7.75-7.82 (m, 1H); ¹³C NMR (75 MHz, CDCl₃): δ 21.4, 29.5, 70.6, 114.5, 114.6, 115.2, 118.5, 118.8, 125.0, 128.2, 129.0, 133.8, 137.7, 141.9, 145.6, 164.5; IR (KBr) cm⁻¹: 1612, 1482, 1508, 1658, 2929, 3062, 3178, 3303.

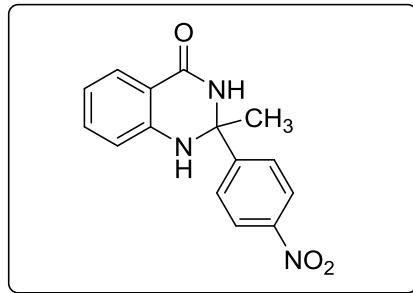
2-(4-bromophenyl)-2-methyl-2,3-dihydroquinazolin-4(1H)-one (5f)



Yield: 85%, (0.269 g); M.p. 238-240 °C; Characteristics: White crystalline solid;

¹H NMR (300 MHz, CDCl₃): δ 1.69 (s, 3H), 5.82 (s, 1H), 6.68 (d, 1H, J=8.1 Hz), 6.92 (t, 1H, J=7.7 Hz), 7.33-7.44 (m, 1H), 7.43 (d, 2H, J=8.4 Hz), 7.55 (d, 2H, J=8.4 Hz), 7.94 (d, 1H, J=7.8 Hz); ¹³C NMR (75 MHz, CDCl₃): δ 27.9, 84.8, 114.9, 117.9, 127.6, 127.8, 128.8, 130.3, 130.9, 132.4, 133.9, 141.1, 147.7, 163.9; IR (KBr) cm⁻¹: 1614, 1488, 1510, 1655, 2931, 3066, 3180, 3302.

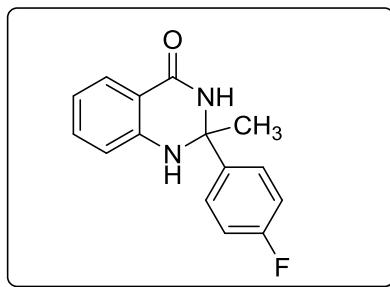
2-methyl-2-(4-nitrophenyl)-2,3-dihydroquinazolin-4(1H)-one (5g)



Yield: 89%, (0.252 g); M.p. 239-241 °C; Characteristics: Yellow crystalline solid;

¹H NMR (300 MHz, CDCl₃): δ 1.72 (s, 3H), 5.88 (s, 1H), 6.67 (d, 1H, J=8.1 Hz), 6.90 (t, 1H, J=7.5 Hz), 7.33-7.42 (m, 5H), 7.95 (d, 1H, J=7.8 Hz); ¹³C NMR (75 MHz, DMSO-d₆): δ 25.7, 82.7, , 114.9, 117.9, 127.6, 127.8, 128.8, 130.3, 130.9, 132.4, 133.9, 141.1, 147.7, 163.8; IR (KBr) cm⁻¹: 1615, 1479, 1508, 1658, 2926, 3066, 3182, 3306.

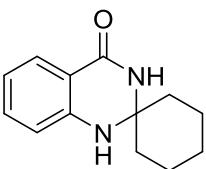
2-(4-fluorophenyl)-2-methyl-2,3-dihydroquinazolin-4(1H)-one (5h)



Yield: 87%, (0.223 g); M.p. 233-236 °C; Characteristics: White crystalline solid;

¹H NMR (300 MHz, CDCl₃): δ 1.72 (s, 3H), 6.39 (s, 1H), 6.60 (d, 1H, J=7.8 Hz), 6.76 (t, 1H, J=7.5 Hz), 6.94 (t, 2H, J=8.6 Hz), 7.07 (s, 1H), 7.23 (t, 2H, J=8.1 Hz), 7.44-7.48 (m, 2H), 7.93; ¹³C NMR (75 MHz, CDCl₃): δ 28.8, 70.6, , 114.9, 117.9, 127.6, 127.8, 128.8, 130.3, 130.9, 132.4, 133.9, 141.1, 147.7, 163.8; IR (KBr) cm⁻¹: 1610, 1482, 1508, 1658, 2929, 3066, 3183, 3300.

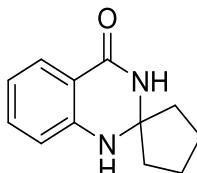
1'H-spiro[cyclohexane-1,2'-quinazolin]-4'(3'H)-one (5i)



Yield: 95%, (0.205 g); M.p. 224-226 °C; Characteristics: White crystalline solid;

¹H NMR (300 MHz, DMSO-D₆): δ 1.26-1.74 (m, 10H), 6.64 (s, 1H), 6.82 (d, 1H, *J*=8.1 Hz), 7.23 (m, 2H), 7.57 (d, 1H, *J*=7.5 Hz), 7.94 (s, 1H) ; ¹³C NMR (75 MHz, DMSO-D₆): δ 21.3, 25.1, 68.2, 114.9, 116.9, 127.5, 133.5, 147.2, 163.6 ; IR (KBr) cm⁻¹: 1618, 1658, 2927, 3183, 3311.

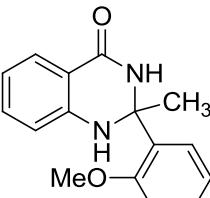
1'H-spiro[cyclopentane-1,2'-quinazolin]-4'(3'H)-one (5j)



Yield: 95%, (0.192 g); M.p. 210-212 °C; Characteristics: White crystalline solid;

¹H NMR (300 MHz, DMSO-D₆): 1.67-1.79 (m, 8H), 6.61-6.74 (m, 2H), 7.19-7.25 (m, 1H), 7.75 (d, 1H, *J*=7.8 Hz), 7.94 (s, 1H), 8.09 (s, 1H) ; ¹³C NMR (75 MHz, DMSO-D₆): δ 22.4, 29.4, 67.2, 77.5, 115.0, 116.8, 127.6, 133.4, 147.9, 163.8; IR (KBr) cm⁻¹: 1616, 1655, 2933, 3178, 3310.

2-(2-methoxyphenyl)-2-methyl-2,3-dihydroquinazolin-4(1H)-one (5k)

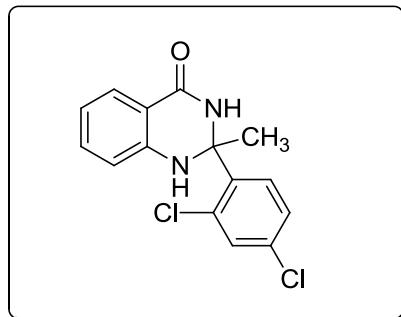


Yield: 87%, (0.192233 g); M.p. 220-2212 °C; Characteristics: White crystalline solid;

¹H NMR (300 MHz, DMSO-D₆): 1.69 (s, 3H), 3.87 (s, 3H), 5.69 (s, 1H), 6.65-6.73 (m, 3H), 7.16-7.24

(m, 2H), 7.35 (d, 2H, $J=7.8$ Hz), 7.59 (d, 1H, $J=7.5$ Hz), 8.20 (s, 1H) ; ^{13}C NMR (75 MHz, DMSO-D₆): δ 28.9, 56.9, 73.1, 114.9, 116.5, 121.6, 123.5, 131.7, 140.4, 147.8, 161.5, 162.9 ; IR (KBr) cm⁻¹: 1618, 1657, 2933, 3175, 3312.

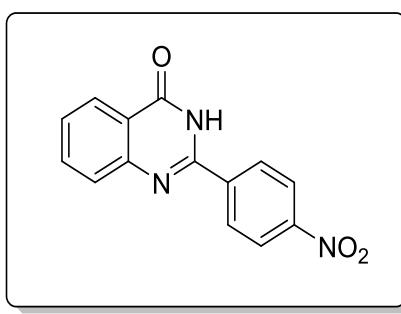
2-(2,4-dichlorophenyl)-2-methyl-2,3-dihydroquinazolin-4(1H)-one (5l)



Yield: 89%, (0.269 g); M.p. 215-2172 °C; Characteristics: White crystalline solid;

^1H NMR (300 MHz, DMSO-D₆): 1.67 (s, 3H), 5.88 (s, 1H), 7.20 (d, 2H, $J=7.2$ Hz), 7.44 (d, 1H, $J=7.5$ Hz), 7.57-7.62 (m, 2H), 7.87 (dd, 2H, $J=7.9$ Hz, $J=1.4$ Hz), 9.69 (s, 1H) ; ^{13}C NMR (75 MHz, DMSO-D₆): δ 21.1, 66.8, 114.8, 115.4, 117.5, 127.1, 127.8, 129.2, 133.7, 138.1, 139.1, 148.2, 164.1; IR (KBr) cm⁻¹: 1615, 1659, 2930, 31878, 3309.

2-(4-nitrophenyl)quinazolin-4(3H)-one (6a)

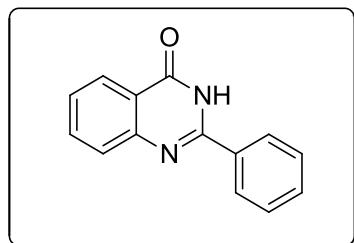


Yield: 97%, (0.259 g); M.p. 280-282 °C; Characteristics: White solid;

^1H NMR (300 MHz, CDCl₃): δ 7.53 (d, 1H, $J=6.6$ Hz), 7.73-7.85 (m, 4H), 8.09-8.14 (m, 3H), 12.61 (s, 1H); ^{13}C NMR (75 MHz, DMSO-D₆): δ 114.8, 114.9, 117.8, 127.7, 127.8, 129.0, 129.9, 130.6, 132.1, 133.7, 138.2, 147.9, 163.9; HRMS (ESI-TOF) m/z Calculated for [C₁₄H₉N₃O₃+Na]⁺: 290.0536, found:

290.0536; IR (KBr) cm^{-1} : 1663, 3178.

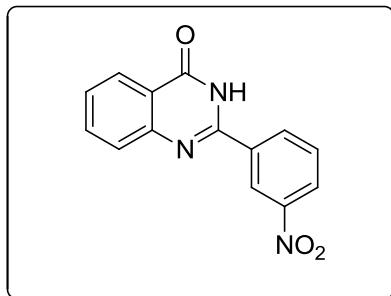
2-phenylquinazolin-4(3H)-one (6b)



Yield: 94%, (0.208 g); M.p. 122-123 $^{\circ}\text{C}$; Characteristics: White solid;

^1H NMR (300 MHz, CDCl_3): δ 7.52 (t, 2H, $J= 7.1$ Hz), 7.71-7.86 (m, 5H), 8.09-8.15 (m, 2H), 12.59 (s, 1H); ^{13}C NMR (75 MHz, DMSO-D_6): δ 117.7, 127.8, 128.8, 129.2, 133.4, 133.9, 141.1, 148.1, 152.0, 158.1, 163.9; IR (KBr) cm^{-1} : 1661, 3175.

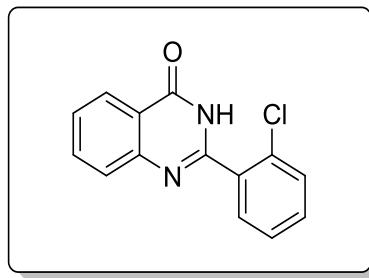
2-(3-nitrophenyl)quinazolin-4(3H)-one (6c)



Yield: 96%, (0.256 g); M.p. 292-294 $^{\circ}\text{C}$; Characteristics: White solid;

^1H NMR (300 MHz, DMSO-D_6): δ 6.65-6.73 (m, 2H), 7.22 (t ,1H, $J=7.7$ Hz),7.34-7.38 (m, 2H), 7.43-7.47 (m, 1H), 7.61 (dd, 2H, $J=6.0$ Hz , $J=2.7$ Hz) ; ^{13}C NMR (75 MHz, DMSO-D_6): 114.8, 114.9, 117.8, 127.7, 127.8, 129.1, 129.8, 130.6, 132.1, 133.7, 138.2, 147.9, 163.9; IR (KBr) cm^{-1} : 1659, 3180.

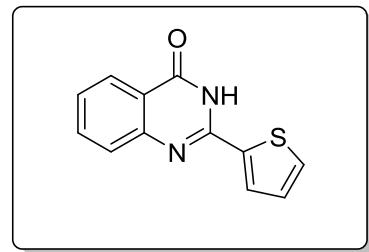
2-(2-chlorophenyl)-2,3-dihydroquinazolin-4(1H)-one (6d)



Yield: 95%, (0.243 g); M.p. 295-296 °C; Characteristics: White solid;

¹H NMR (300 MHz, DMSO-D₆): 6.13-6.77 (m, 2H), 7.25 (t, 1H, *J*= 7.7 Hz), 7.38-7.41 (m, 2H), 7.48-7.51 (m, 1H), 7.65 (d, 2H, *J*=6.6 Hz); ¹³C NMR (75 MHz, DMSO-D₆): δ 117.9, 127.8, 127.9, 129.2, 130.1, 130.7, 133.9, 148.1, 157.1, 164.1; HRMS (ESI-TOF) m/z Calculated for [C₁₄H₉ClN₂O+K]⁺: 295.0035, found: 295.0035. IR (KBr) cm⁻¹: 1653, 3138.

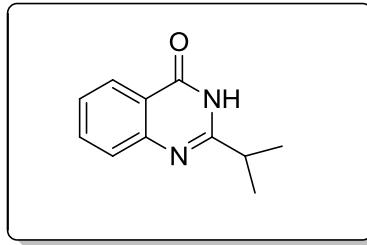
2-(thiophen-2-yl)-2,3-dihydroquinazolin-4(1H)-one (6e)



Yield: 90%, (0.205 g); M.p. 244-246 °C; Characteristics: White solid;

¹H NMR (300 MHz, DMSO-D₆): δ 6.64-6.73 (m, 2H), 6.92-6.94 (m, 1H), 7.07-7.08 (m, 1H), 7.20-7.24 (m, 1H), 7.40 (d, 1H, *J*=5.1), 7.57 (d, 1H, *J*=7.5); ¹³C NMR (75 MHz, DMSO-D₆): δ 117.6, 125.7, 125.9, 126.6, 127.4, 133.5, 146.6, 147.2, 163.2; HRMS (ESI-TOF) m/z Calculated for [C₁₂H₉N₂OS+K]⁺: 266.9989, found: 266.9986; IR (KBr) cm⁻¹: 1637, 3166.

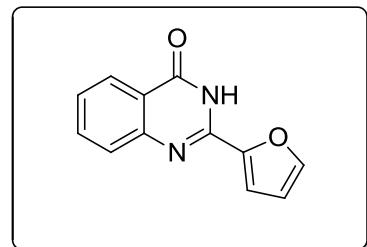
2-isopropylquinazolin-4(3H)-one (6f)



Yield: 88%, (0.165 g); M.p. 180-182 °C; Characteristics: White solid;

¹H NMR (300 MHz, DMSO-D₆): δ 0.83 (d, 6H, *J*=6.6Hz), 1.77-1.79 (m, 1H), 6.53 (t, 1H, *J*=7.4Hz), 6.67 (d, 1H, *J*=8.1Hz), 7.12 (t, 1H, *J*=7.5Hz), 7.48 (d, 1H, *J*=7.5Hz), 9.07 (s, 1H) ; ¹³C NMR (75 MHz, DMSO-D₆): δ 30.7, 32.8, 114.2, 114.8, 116.6, 127.3, 133.1, 148.6, 163.9. IR (KBr) cm⁻¹: 1250, 1369, 1457, 1651, 3160.

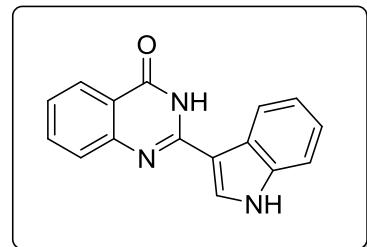
2-(furan-2-yl)quinazolin-4(3*H*)-one (6g)



Yield: 90%, (0.190 g); M.p. 210-212 °C; Characteristics: White solid;

¹H NMR (300 MHz, DMSO-D₆): δ 6.64-6.73 (m, 2H), 6.92-6.94 (m, 1H), 7.07-7.08 (m, 1H), 7.20-7.24 (m, 1H), 7.40 (d, 1H, *J*=5.1), 7.57 (d, 1H, *J*=7.5); ¹³C NMR (75 MHz, DMSO-D₆): δ 117.6, 125.7, 125.9, 126.6, 127.4, 133.5, 146.6, 147.2, 163.2; IR (KBr) cm⁻¹: 1370, 1457, 1652, 3155.

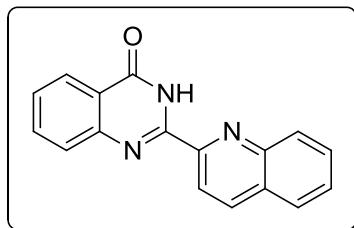
2-(1*H*-indol-3-yl)quinazolin-4(3*H*)-one (6h)



Yield: 95%, (0.247 g); M.p. 302-304 °C; Characteristics: Yellow solid;

¹H NMR (300 MHz, DMSO-D₆): δ 6.82-7.04 (m, 4H), 7.29-7.41 (m, 5H), 9.75 (s, 1H), 10.69 (s, 1H); ¹³C NMR (75 MHz, DMSO-D₆): δ 111.8, 118.3, 118.7, 119.6, 121.0, 123.6, 127.2, 136.9, 149.6, 162.9; IR (KBr) cm⁻¹: 1376, 1457, 1651, 3160.

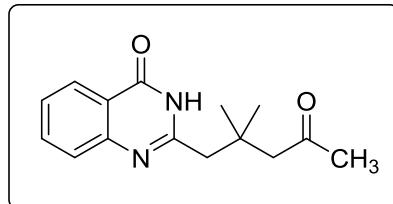
2-(quinolin-2-yl)quinazolin-4(3*H*)-one (6i)



Yield: 95%, (0.259 g); M.p. 188-190 °C; Characteristics: White solid;

¹H NMR (300 MHz, DMSO-D₆): δ 7.62-7.89 (m, 5H), 8.12 (d, 1H, *J*=7.5), 8.25 (t, 2H, *J*=7.6), 8.54-8.65 (m, 2H), 12.05 (s, 1H); ¹³C NMR (75 MHz, DMSO-D₆): δ 115.2, 119.6, 122.5, 122.6, 124.1, 127.5, 130.6, 140.2, 145.2, 152.5, 168.1; IR (KBr) cm⁻¹: 1675, 3140.

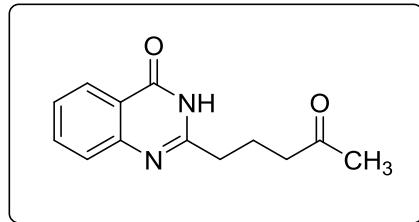
2-(2,2-dimethyl-4-oxopentyl)quinazolin-4(3*H*)-one (7a)



Yield: 69%, (0.178 g); M.p. 160-162 °C; Characteristics: White solid

¹H NMR (300 MHz, DMSO-D₆): δ 1.00 (s, 6H), 2.31 (s, 2H), 2.52 (s, 2H), 2.71 (s, 3H), 7.50 (t, 1H, *J*=7.1), 7.58 (d, 1H, *J*=7.8), 7.77 (d, 1H, *J*=7.5), 8.08 (d, 1H, *J*=7.8), 12.22 (s, 1H); ¹³C NMR (75 MHz, DMSO-D₆): δ 26.2, 30.1, 33.2, 43.9, 53.6, 121.1, 127.1, 133.5, 142.6, 148.9, 153.9, 164.2; IR (KBr) cm⁻¹: 1659, 3150.

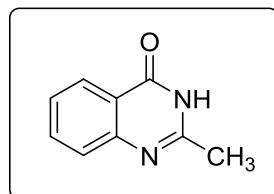
2-(4-oxopentyl)quinazolin-4(3*H*)-one (7b)



Yield: 85%, (0.195 g); M.p. 144-145 °C; Characteristics: White solid

¹H NMR (300 MHz, DMSO-D₆): δ 1.49 (t, 2H, *J*=5.1), 1.65 (m, 2H), 1.98 (s, 3H), 2.63 (t, 2H, *J*=5.1) 7.46-7.54 (m, 1H), 7.69 (d, 1H, *J*=7.8), 7.79 (t, 1H, *J*=7.7), 8.29 (d, 1H, *J*=7.8), 11.74 (s, 1H); ¹³C NMR (75 MHz, DMSO-D₆): δ 22.1, 30.1, 33.2, 43.4, 120.6, 127.1, 133.4, 142.6, 148.9, 153.9, 164.3; IR (KBr) cm⁻¹: 1659, 3133.

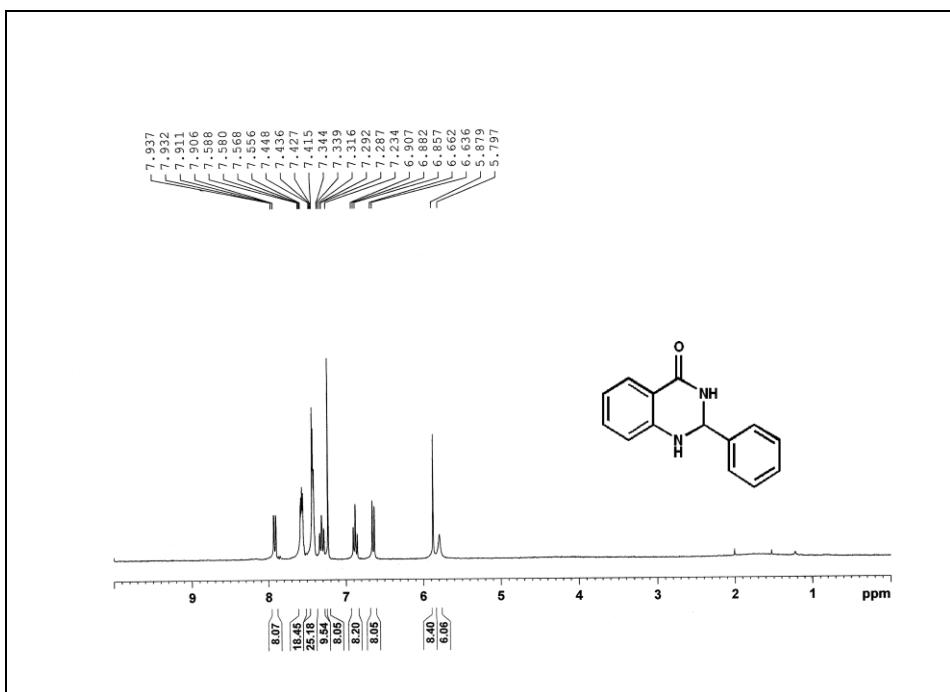
2-methylquinazolin-4(3H)-one (8a)/ (8b)/ (8c)



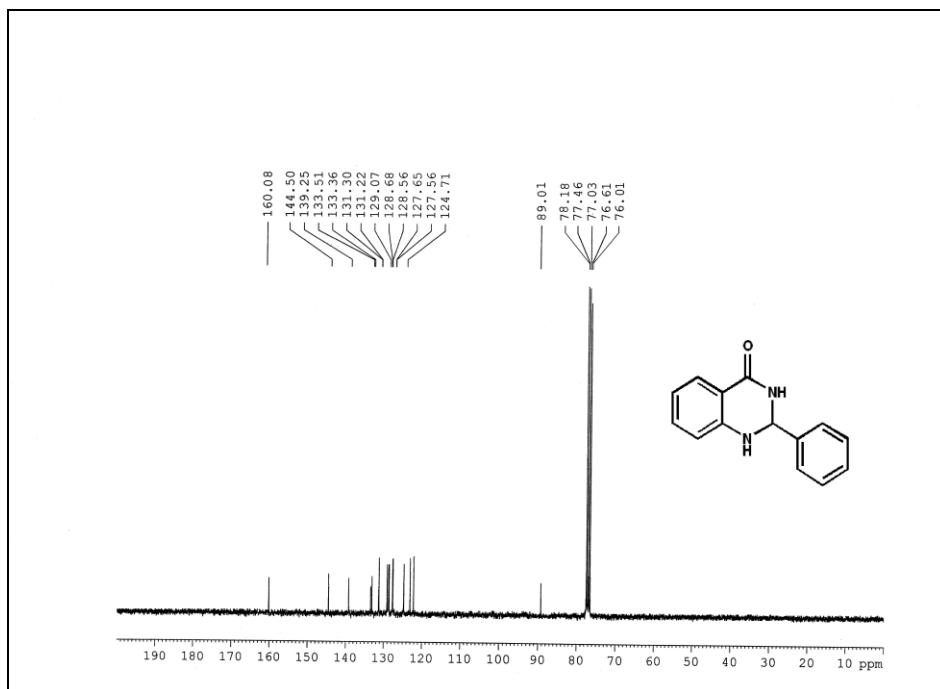
Yield: 98%, (0.156 g); M.p. 175-176 °C; Characteristics: White solid

¹H NMR (300 MHz, CDCl₃): δ 2.61 (s, 3H), 7.33-7.54 (m, 1H), 7.69 (d, 1H, *J*=7.8), 7.78 (t, 1H, *J*=7.7), 8.29 (d, 1H, *J*=7.8), 12.03 (s, 1H); ¹³C NMR (75 MHz, DMSO-D₆): δ 22.4, 120.5, 127.6, 133.4, 138.5, 147.9, 154.9, 163.8; IR (KBr) cm⁻¹: 1665, 3137.

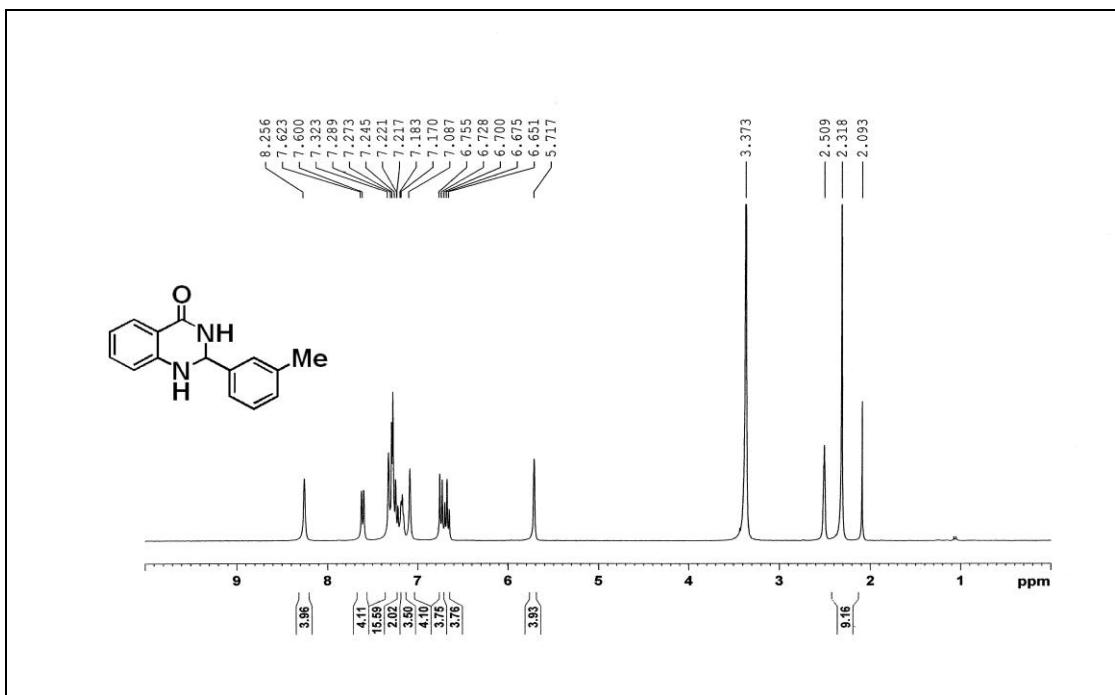
¹H NMR, ¹³C NMR Spectra of the Compounds (3a-3i):



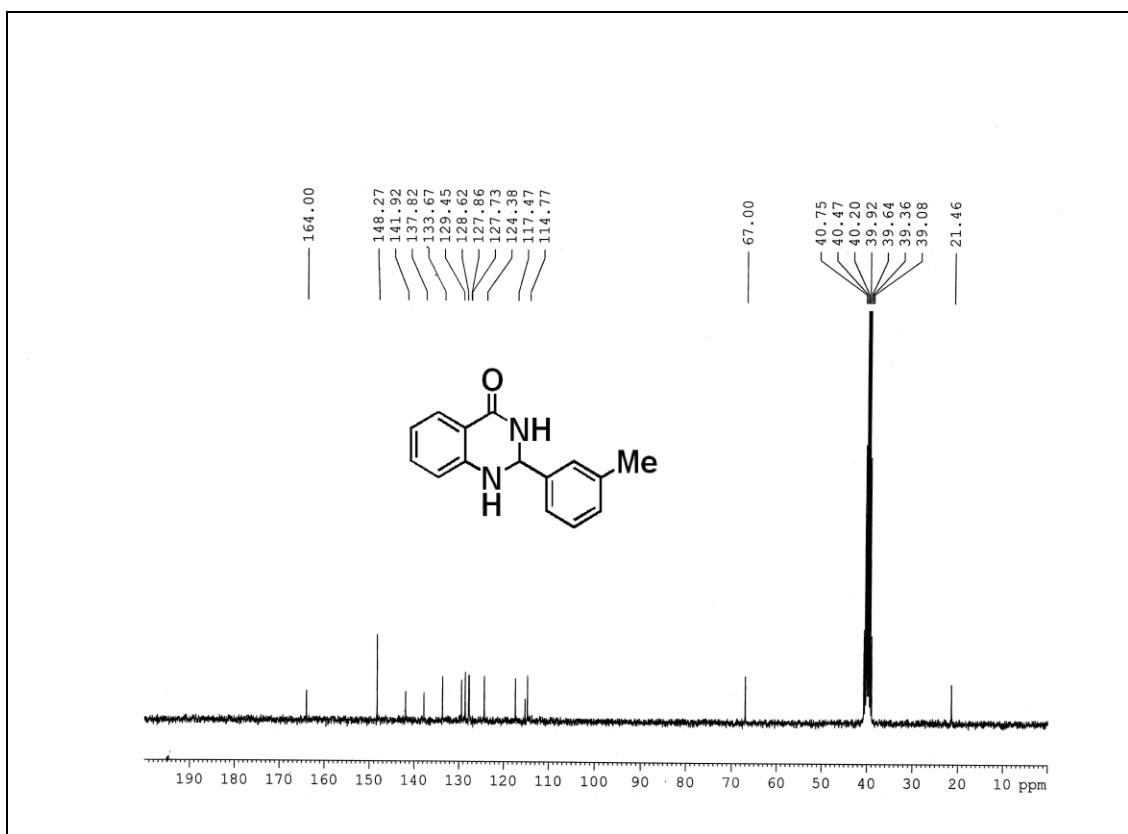
¹H NMR of Compound 3a



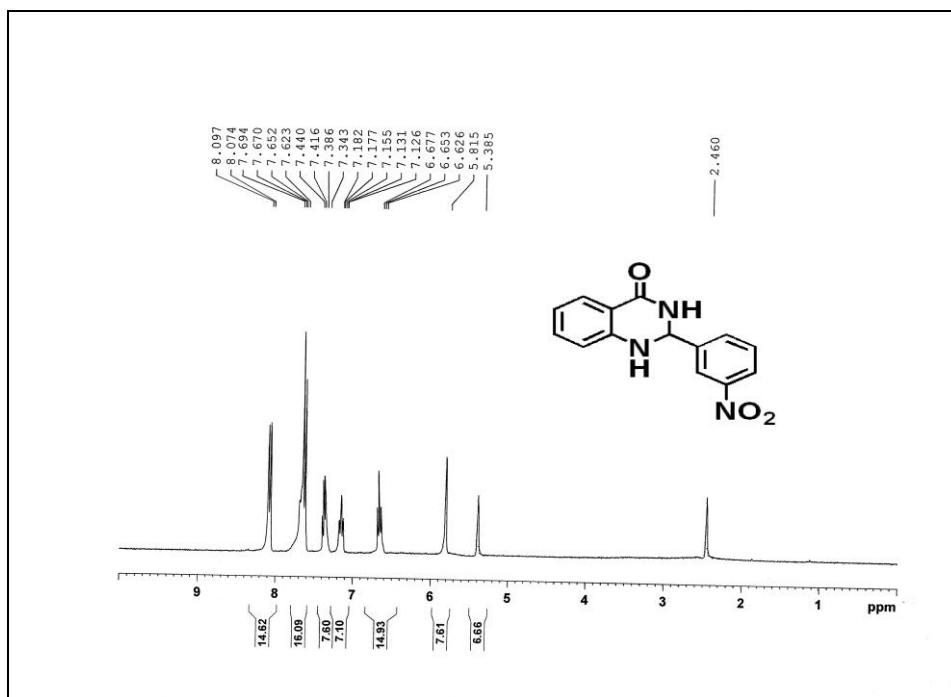
¹³C NMR of Compound 3a



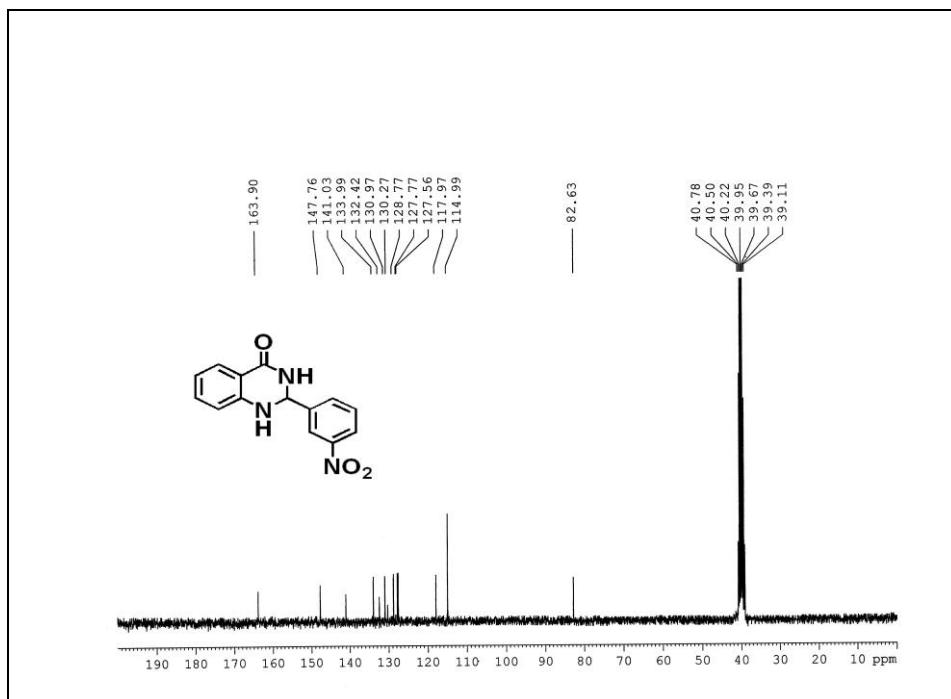
¹H NMR of Compound 3b



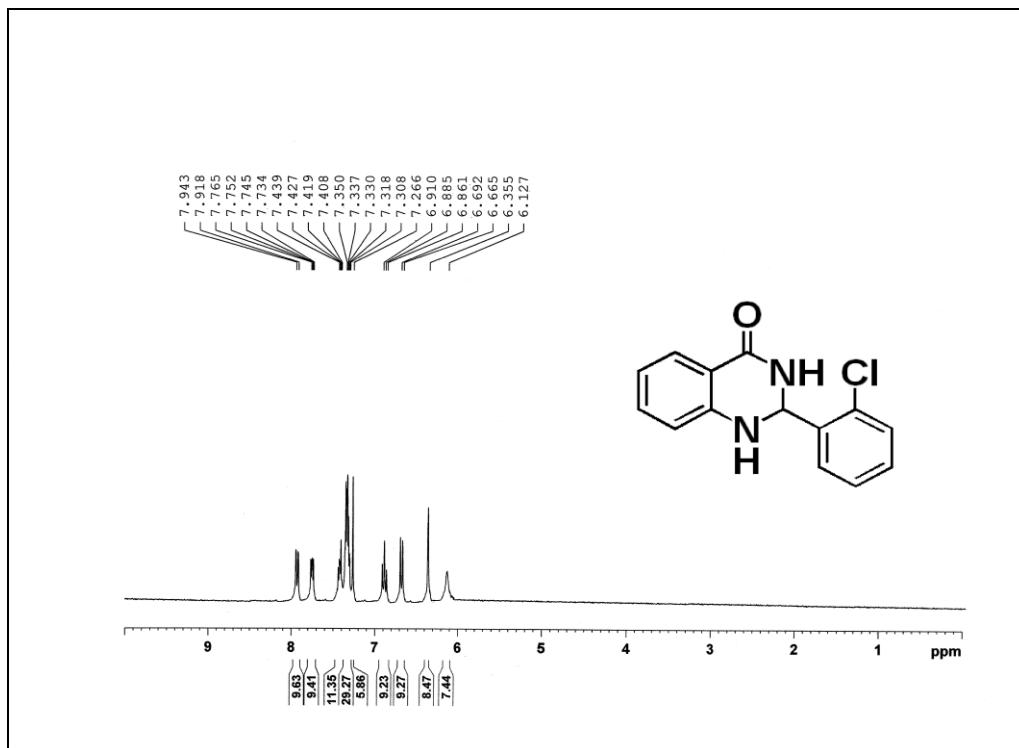
¹³C NMR of Compound 3b



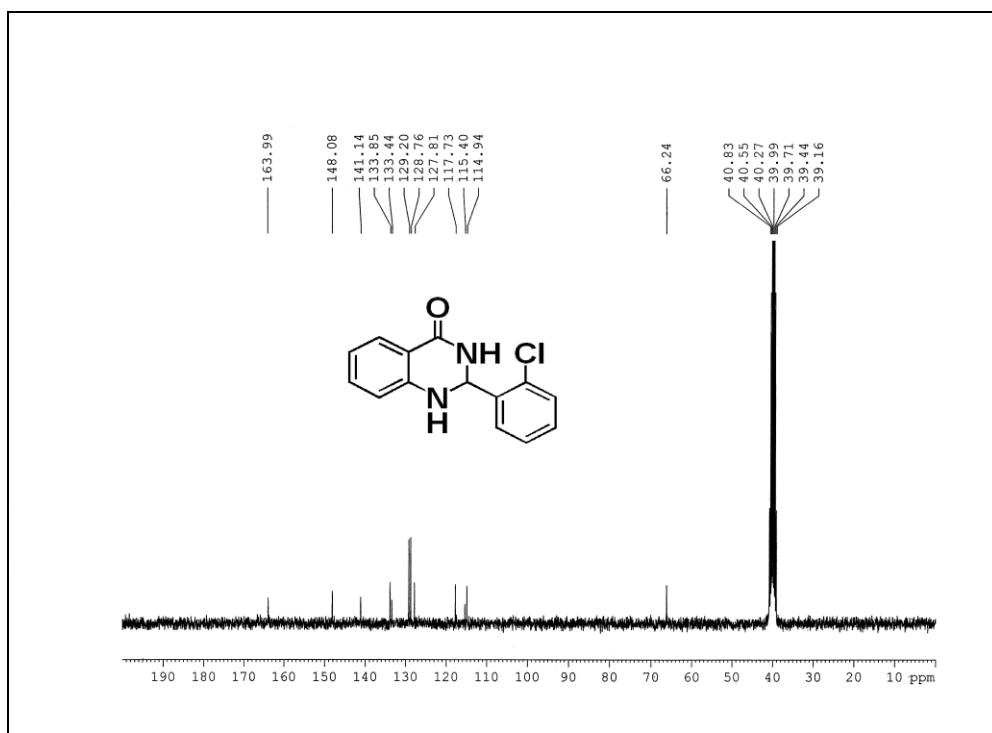
¹H NMR of Compound 3c



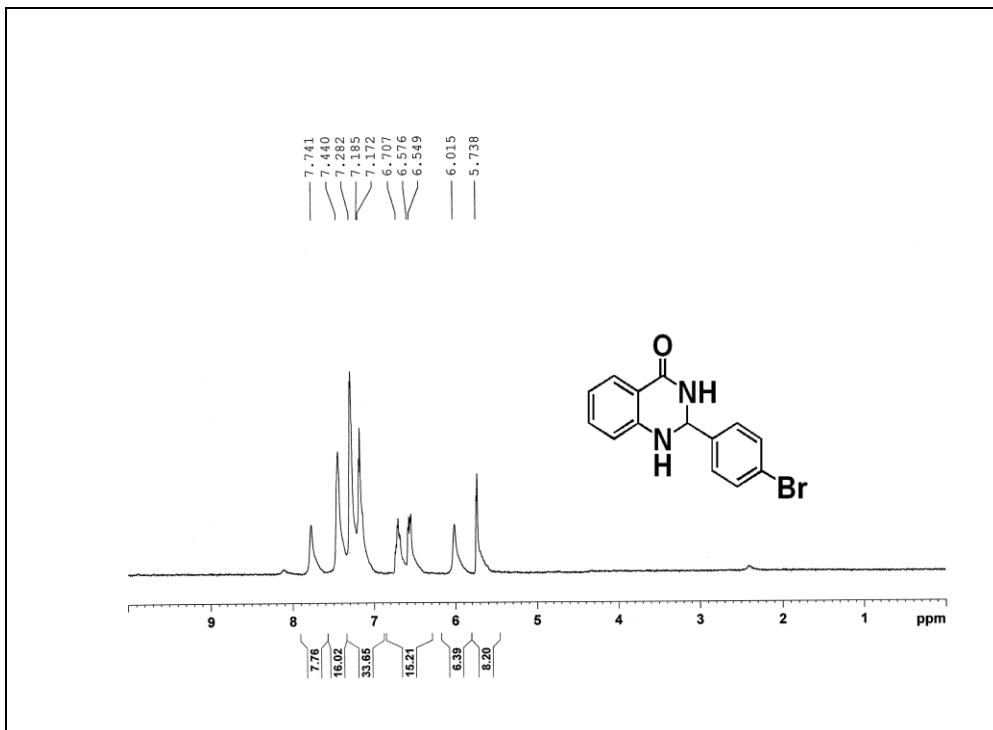
¹³C NMR of Compound 3c



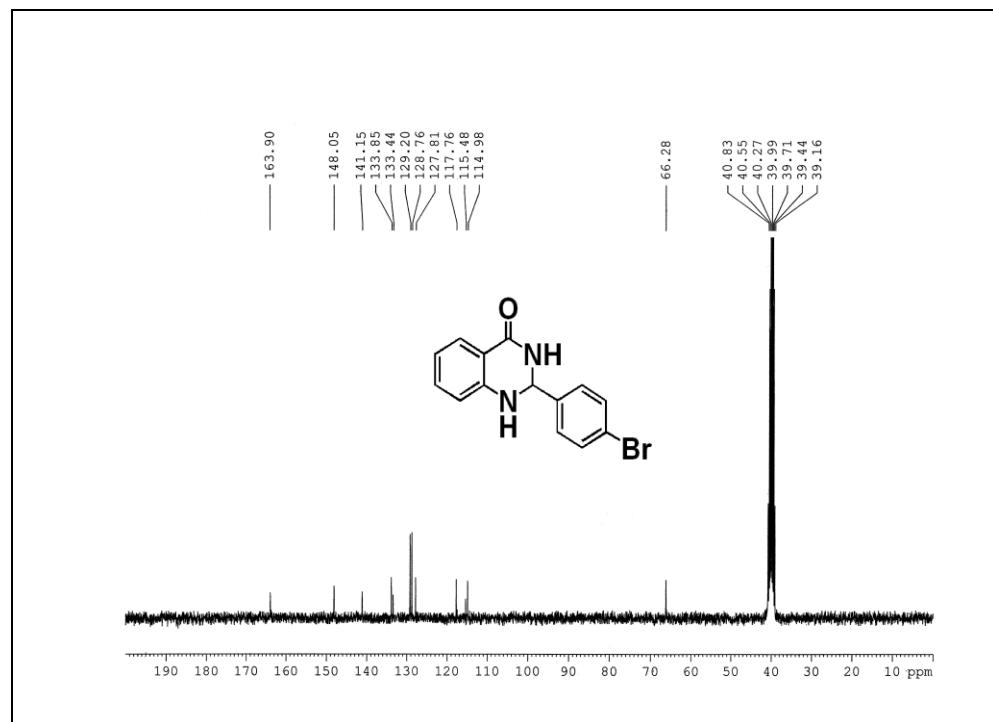
¹H NMR of Compound 3d



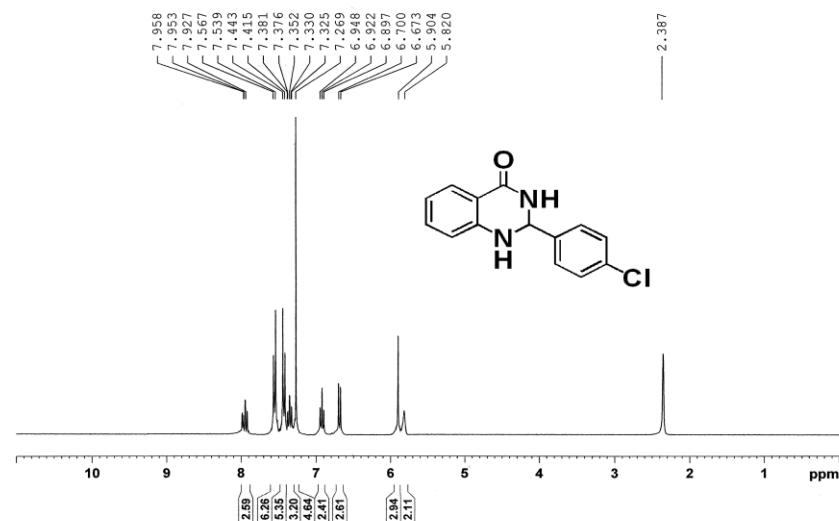
¹³C NMR of Compound 3d



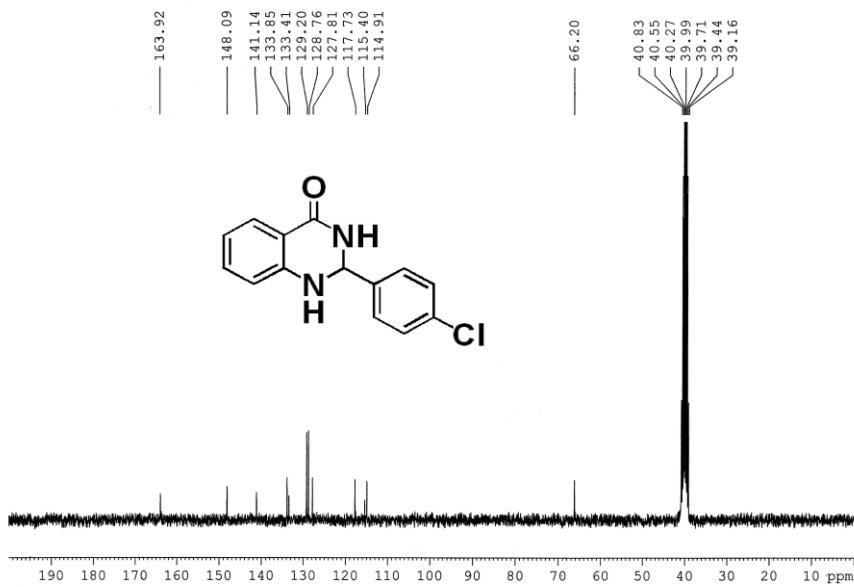
¹H NMR of Compound 3e



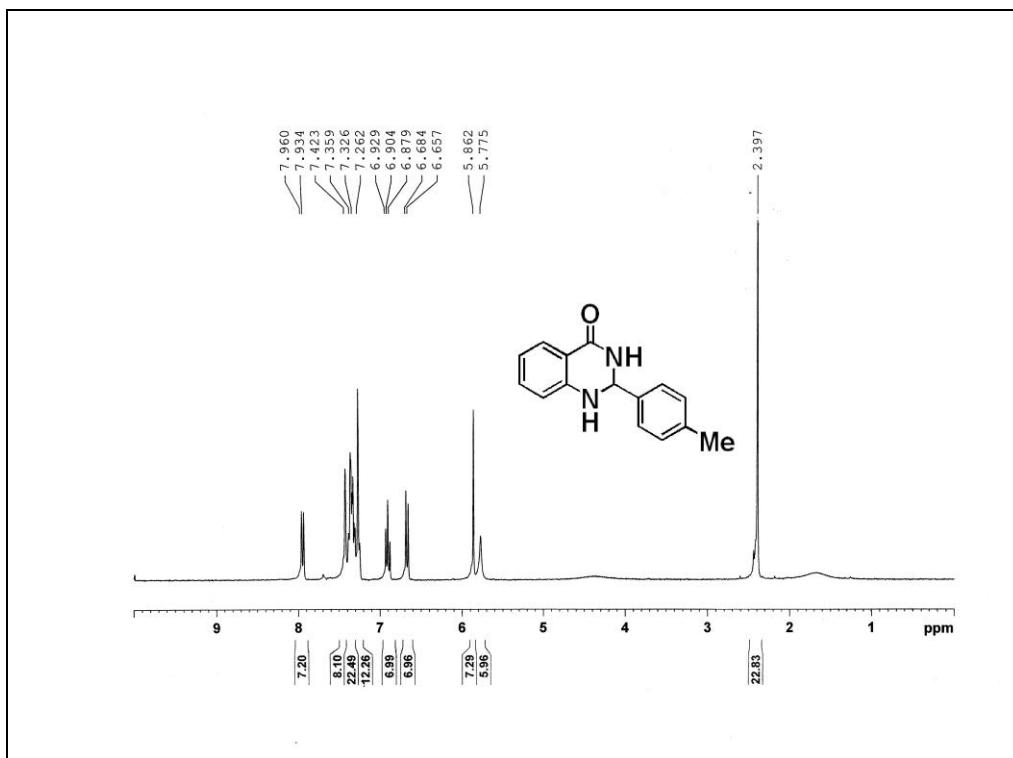
¹³C NMR of Compound 3e



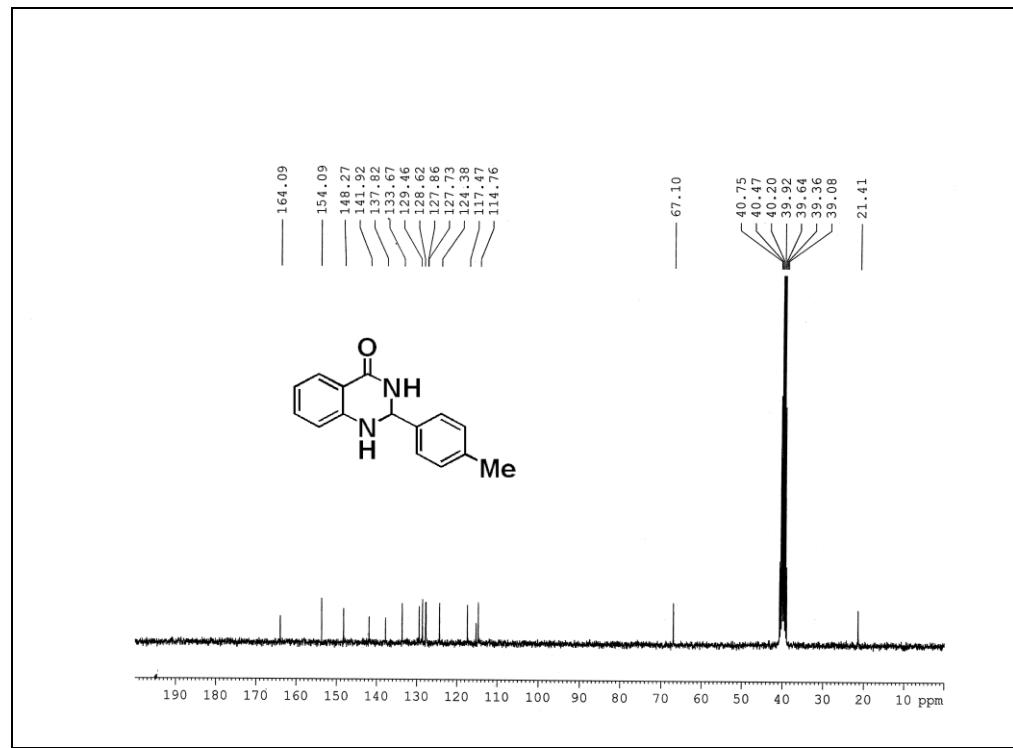
¹H NMR of Compound 3f



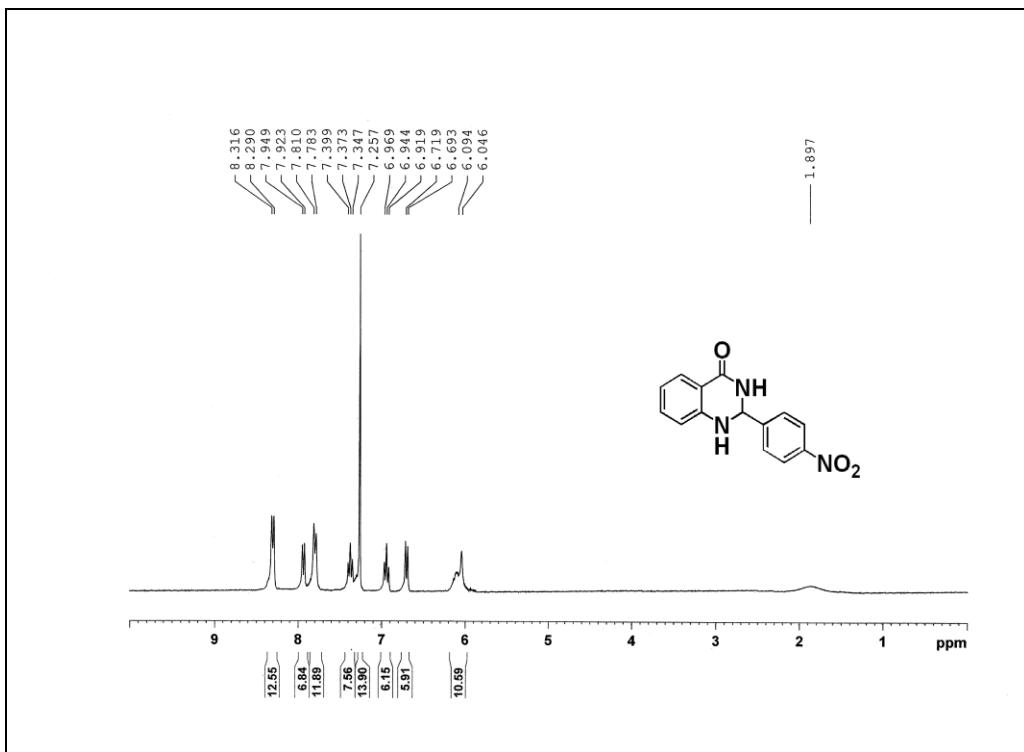
¹³C NMR of Compound 3f



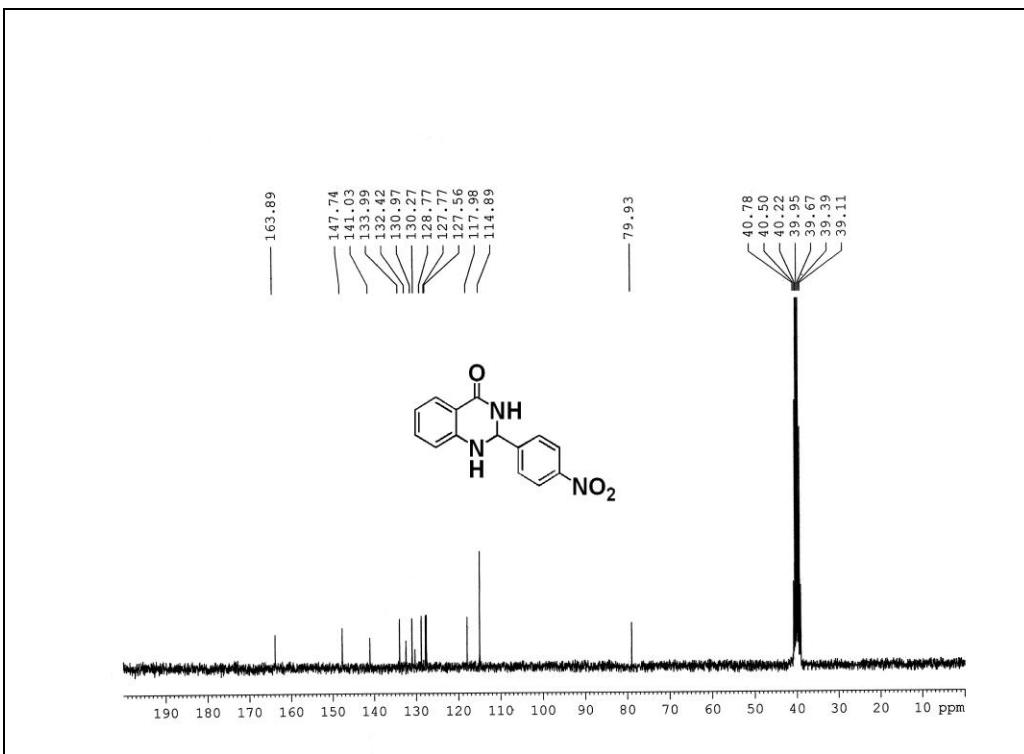
¹H NMR of Compound 3g



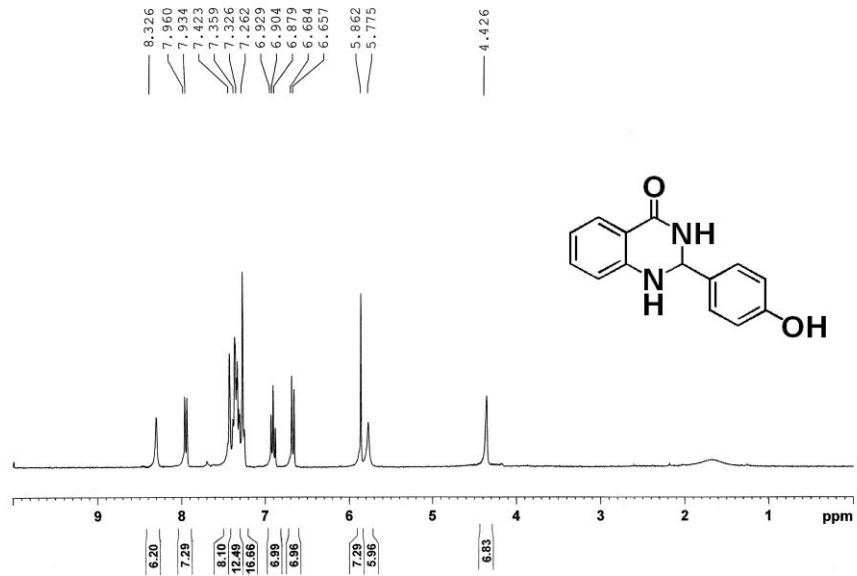
¹³C NMR of Compound 3g



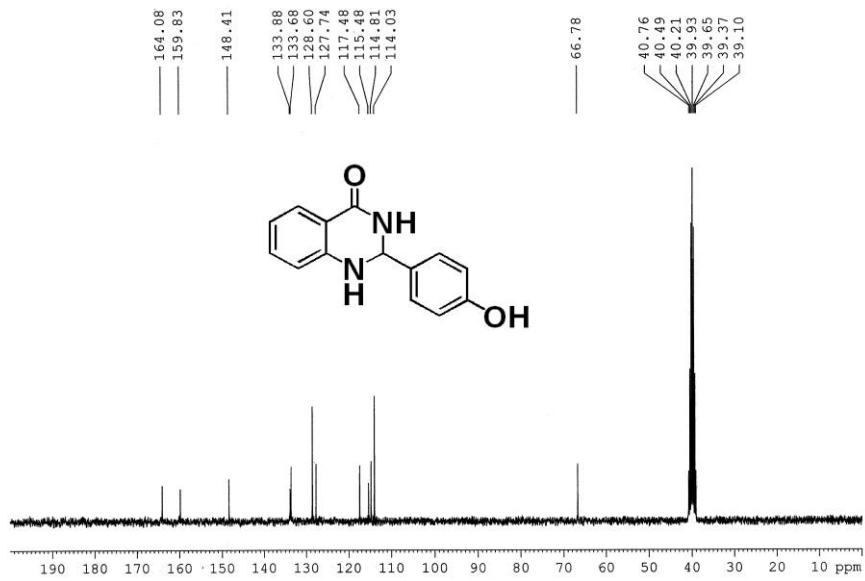
¹H NMR of Compound 3h



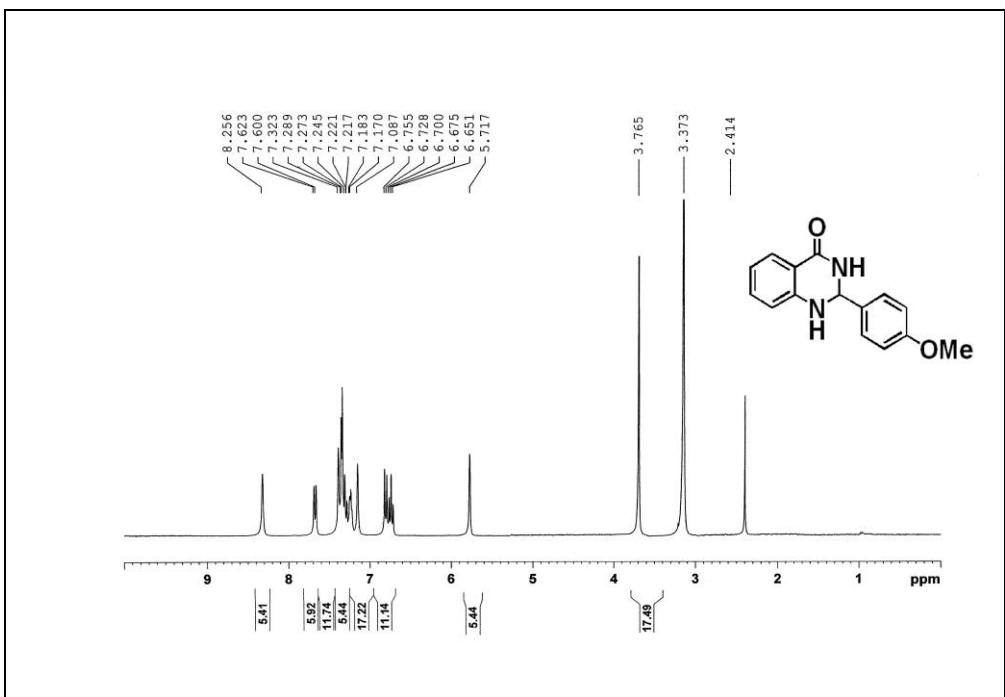
¹³C NMR of Compound 3h



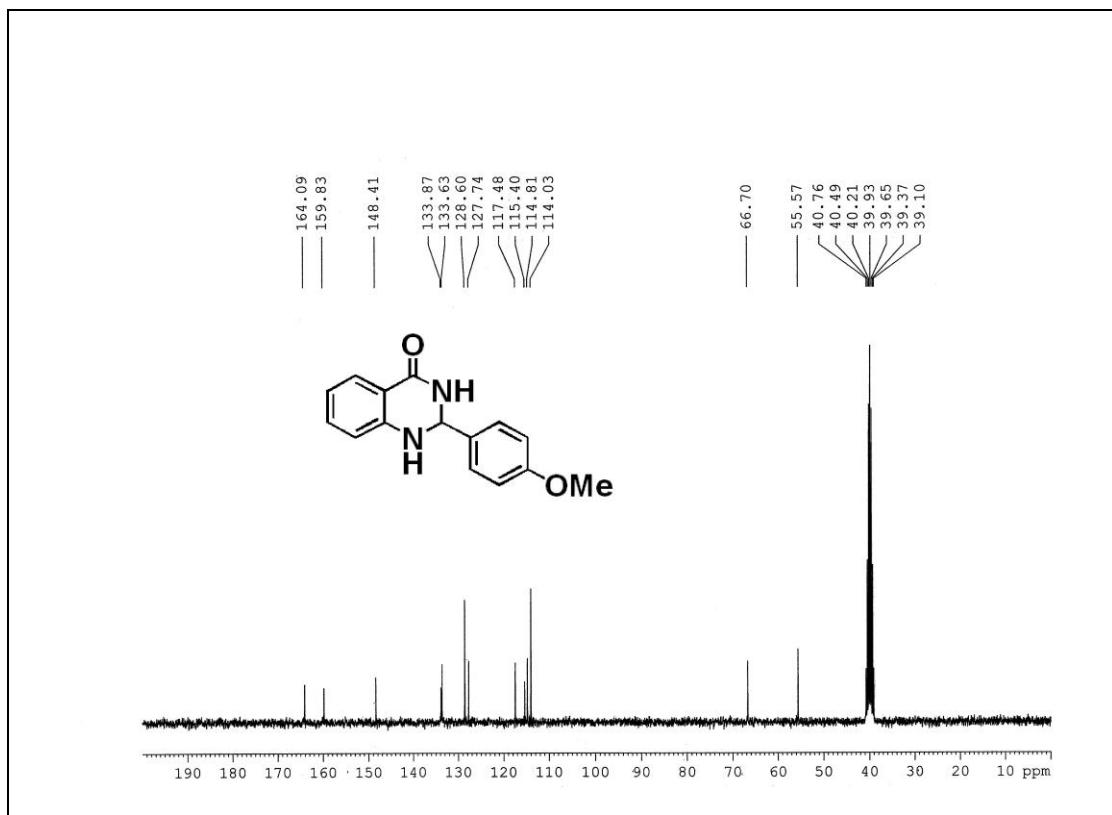
¹H NMR of Compound 3i



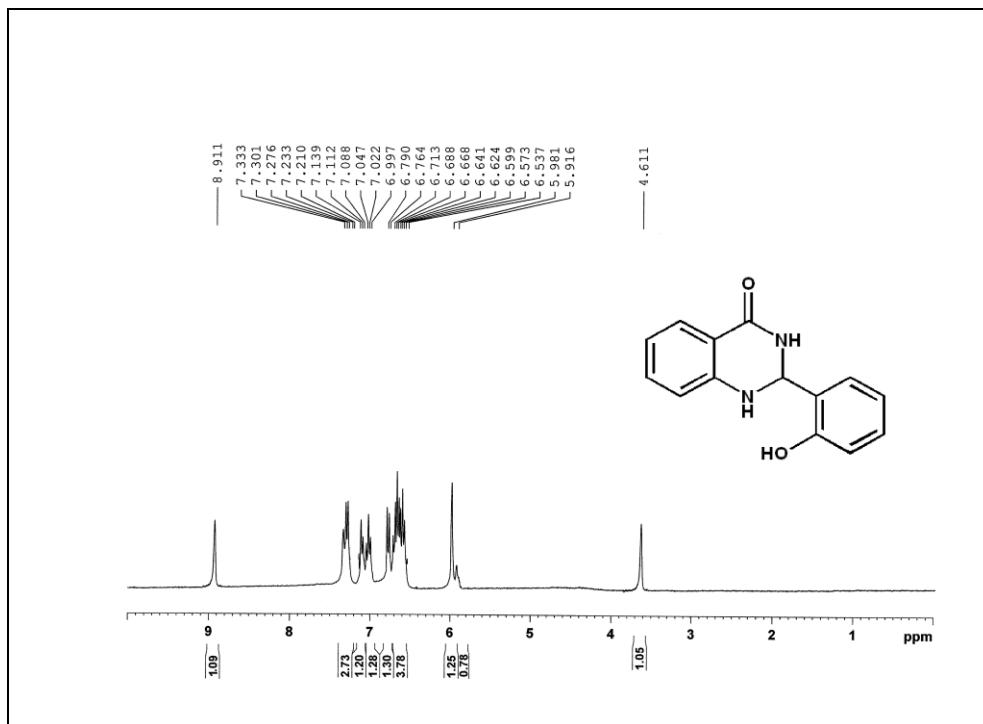
¹³C NMR of Compound 3i



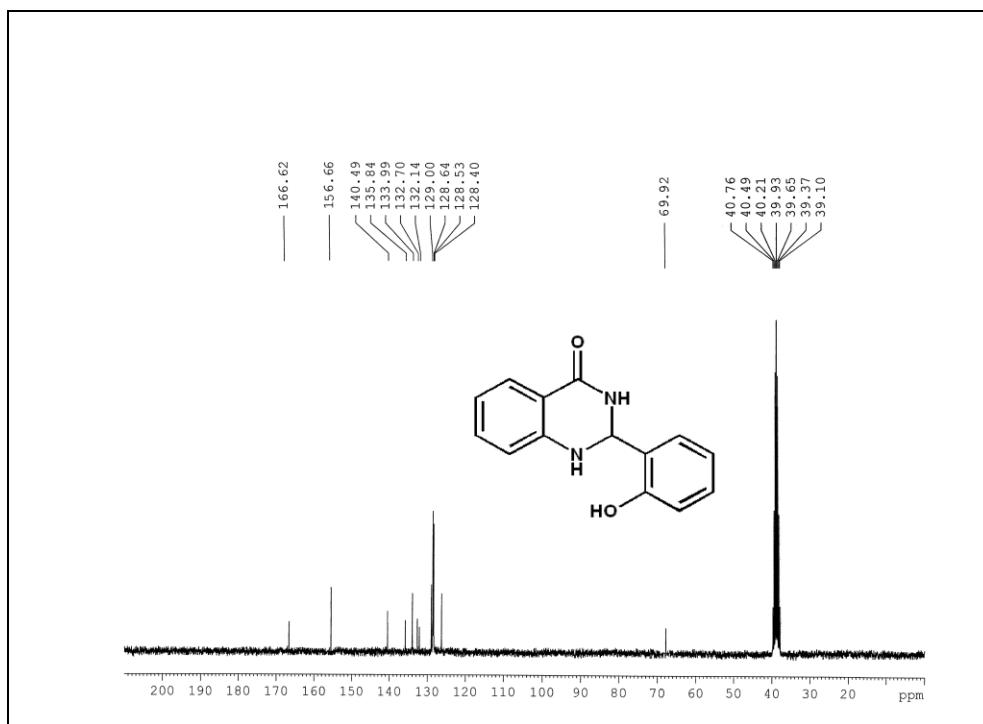
¹H NMR of Compound 3j



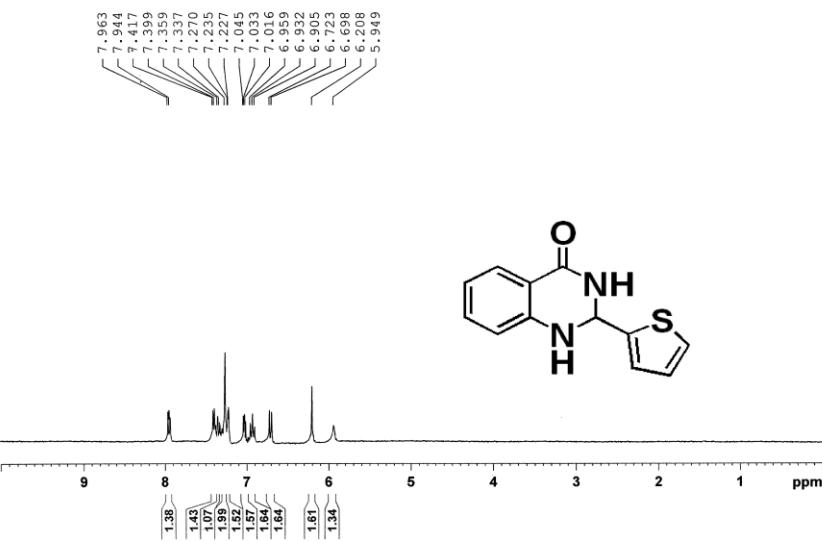
¹³C NMR of Compound 3j



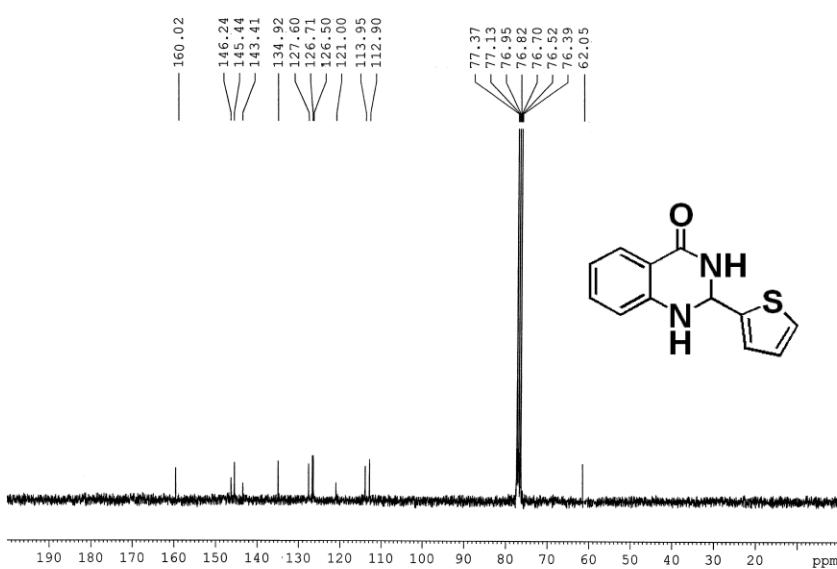
¹H NMR of Compound 3k



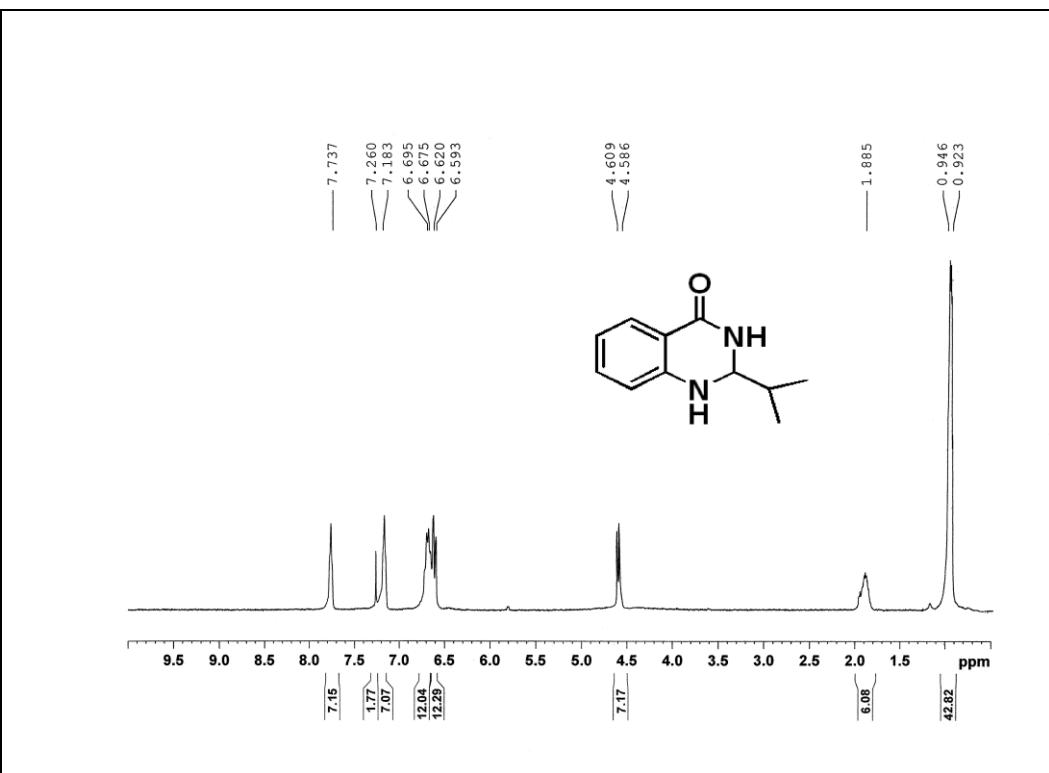
¹³C NMR of Compound 3k



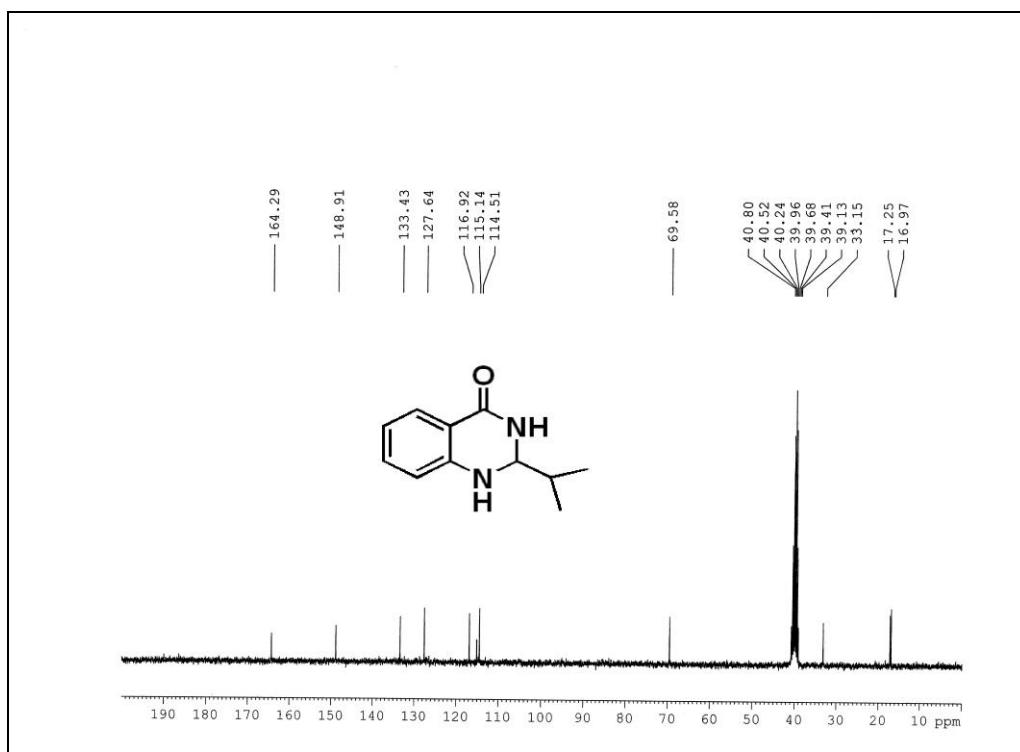
¹H NMR of Compound 31



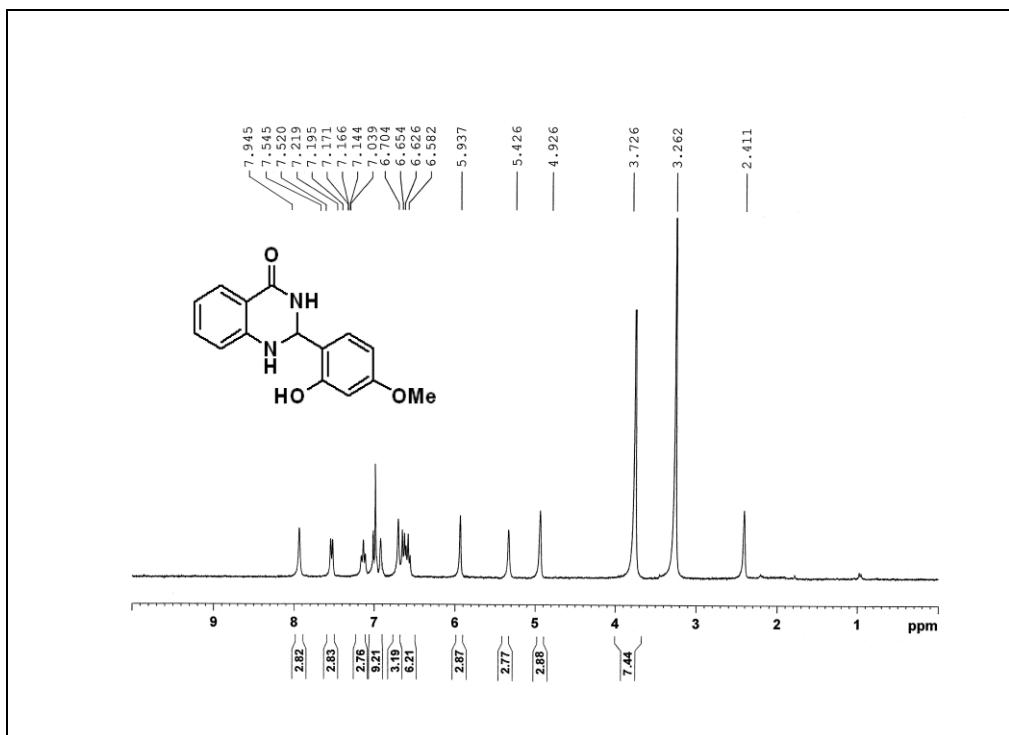
¹³C NMR of Compound 31



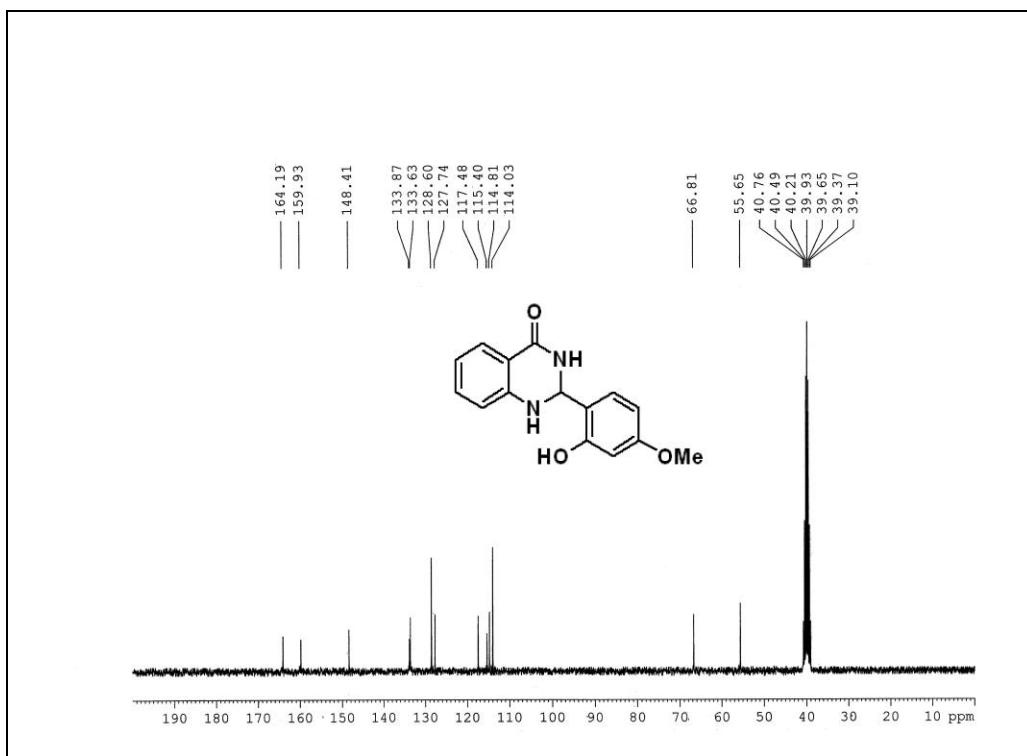
¹H NMR of Compound 3m



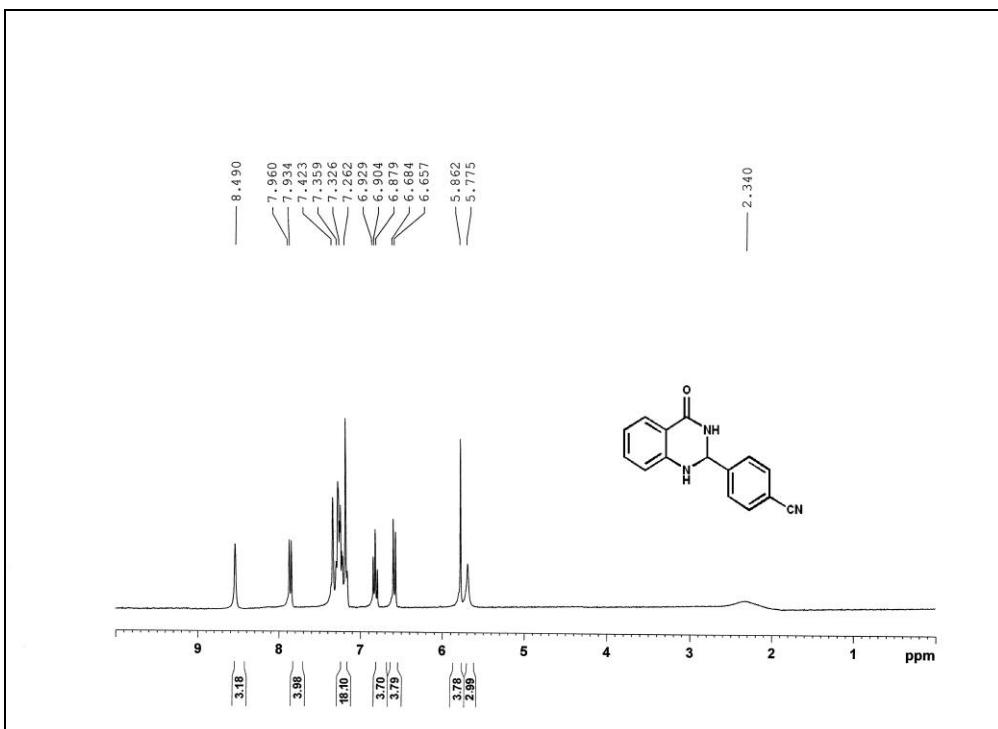
¹³C NMR of Compound 3m



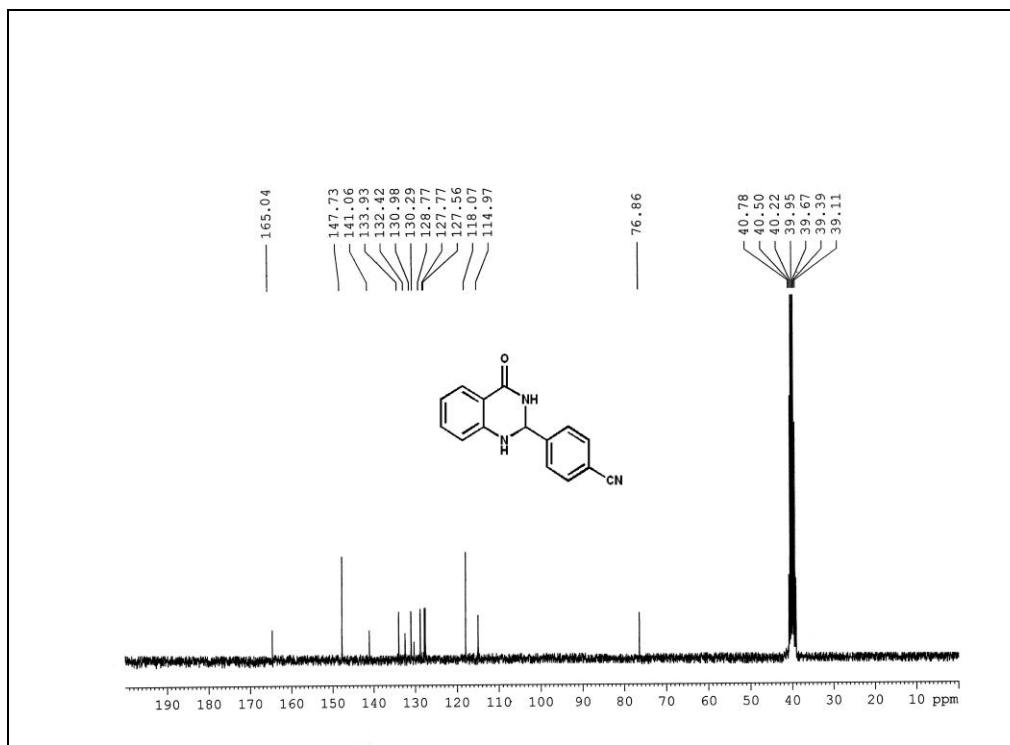
¹H NMR of Compound 3n



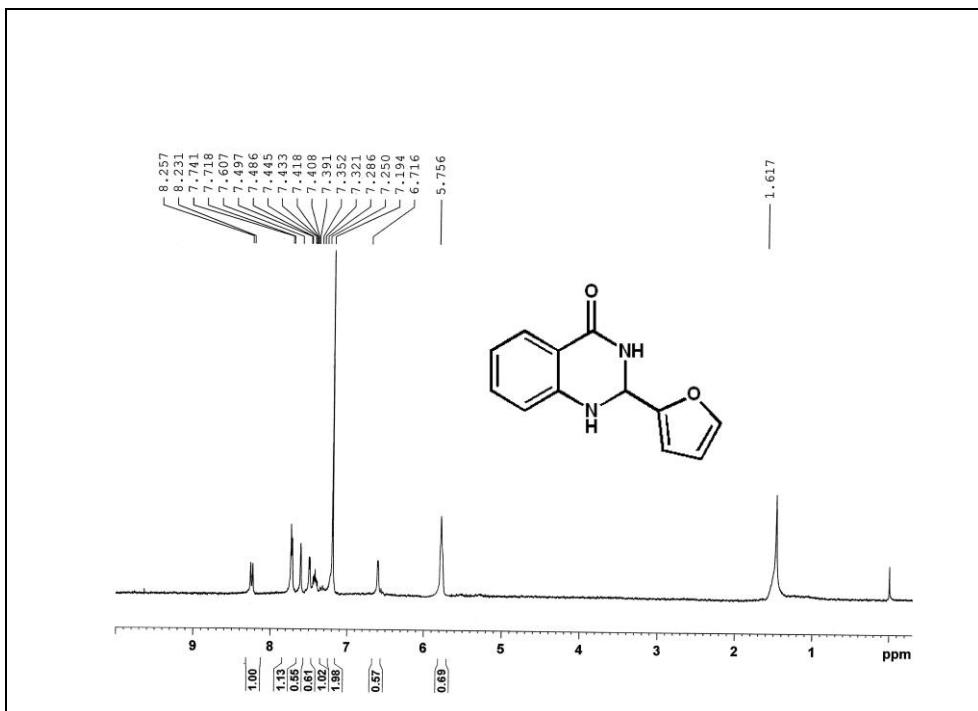
¹³C NMR of Compound 3n



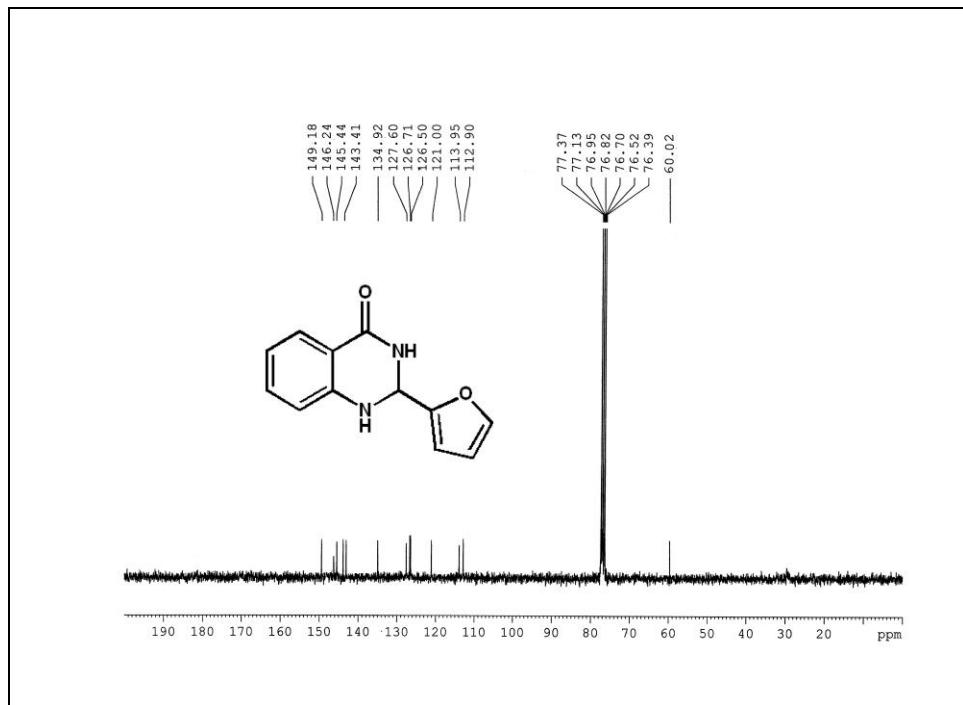
¹H NMR of Compound 3o



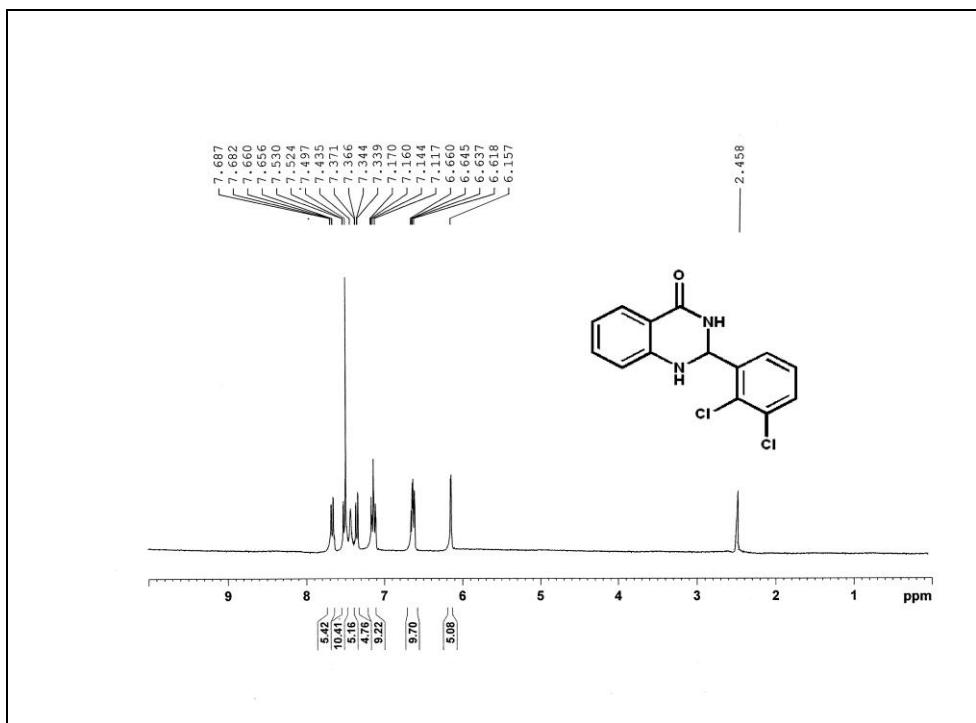
¹³C NMR of Compound 3o



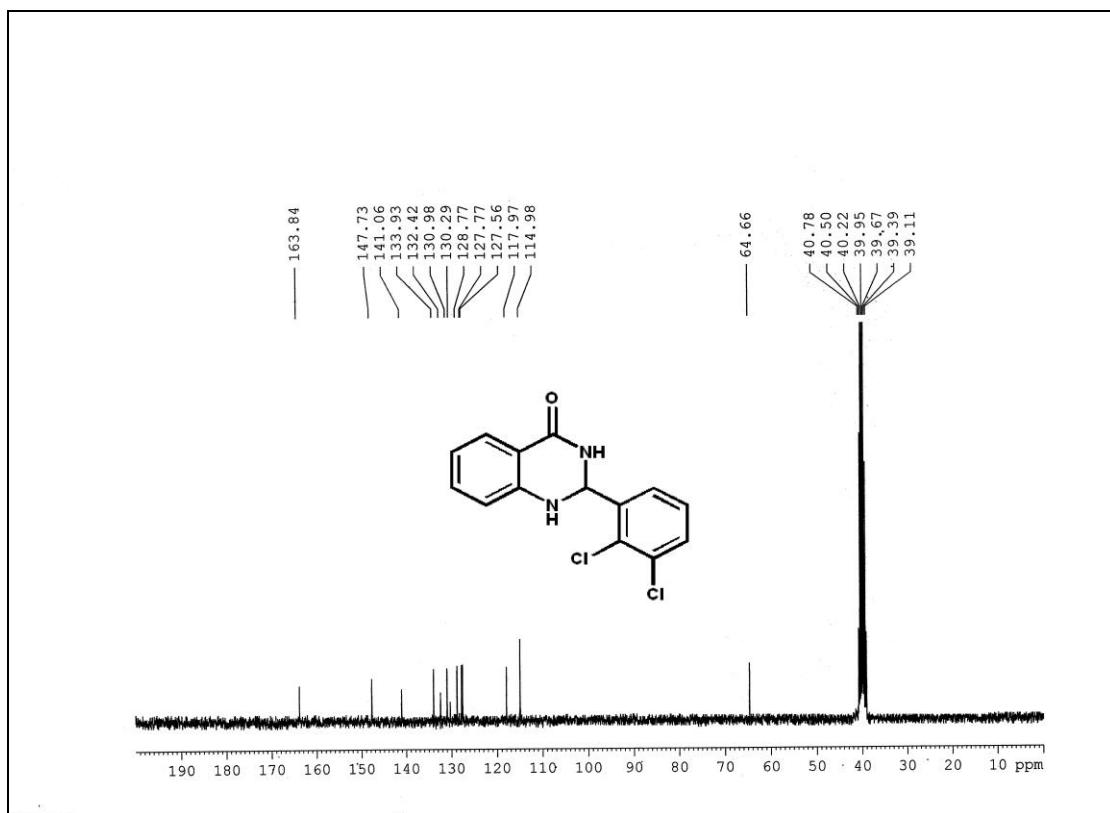
¹H NMR of Compound 3p



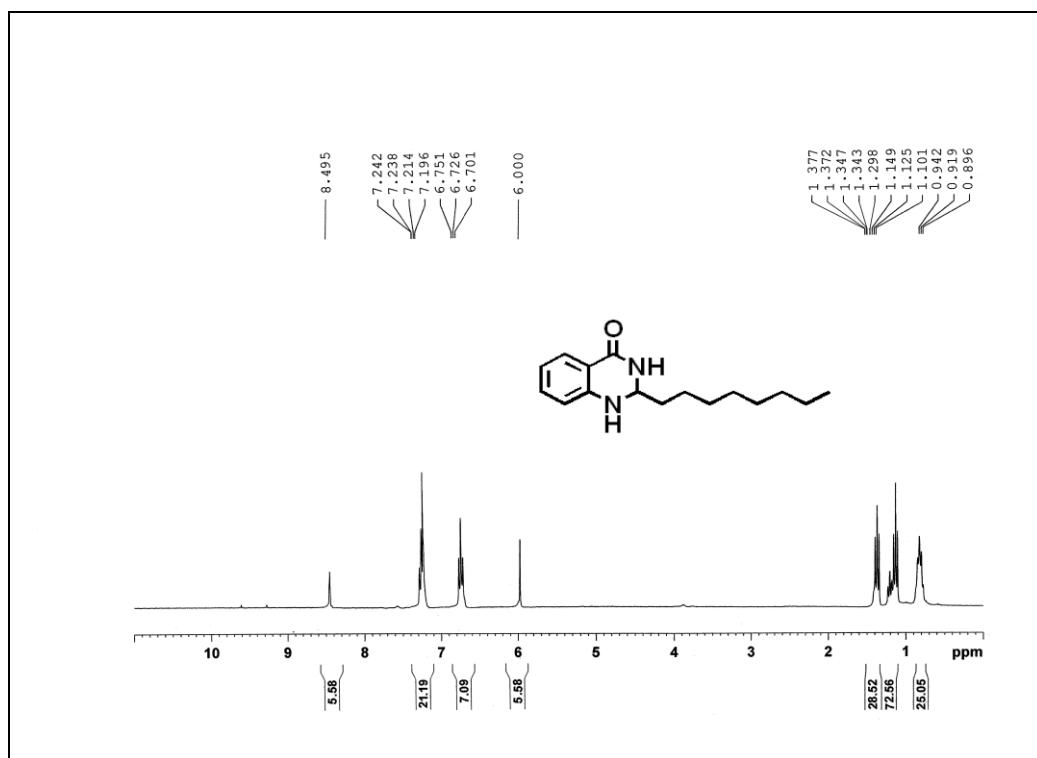
¹³C NMR of Compound 3p



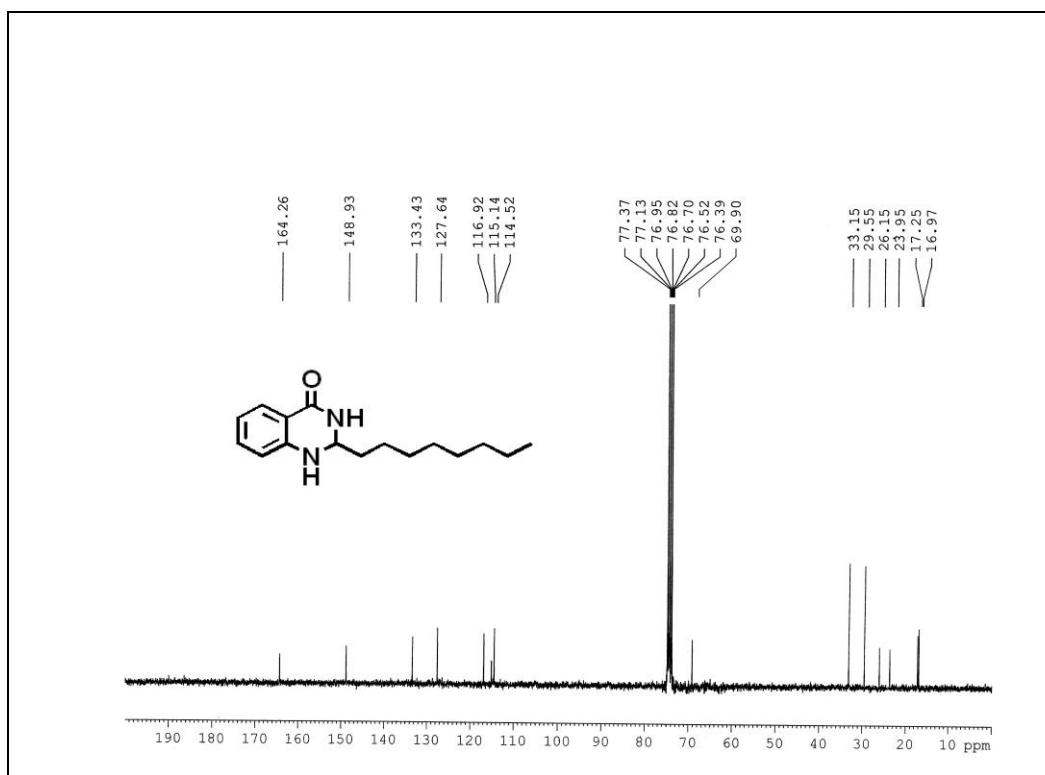
¹H NMR of Compound 3q



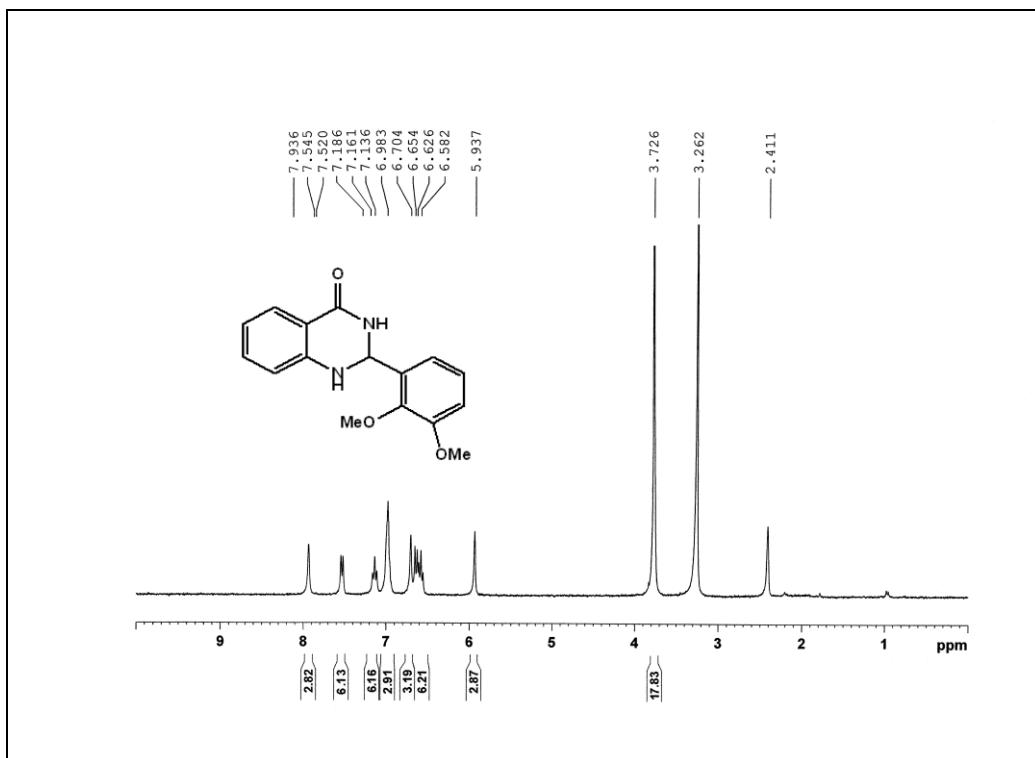
¹³C NMR of Compound 3q



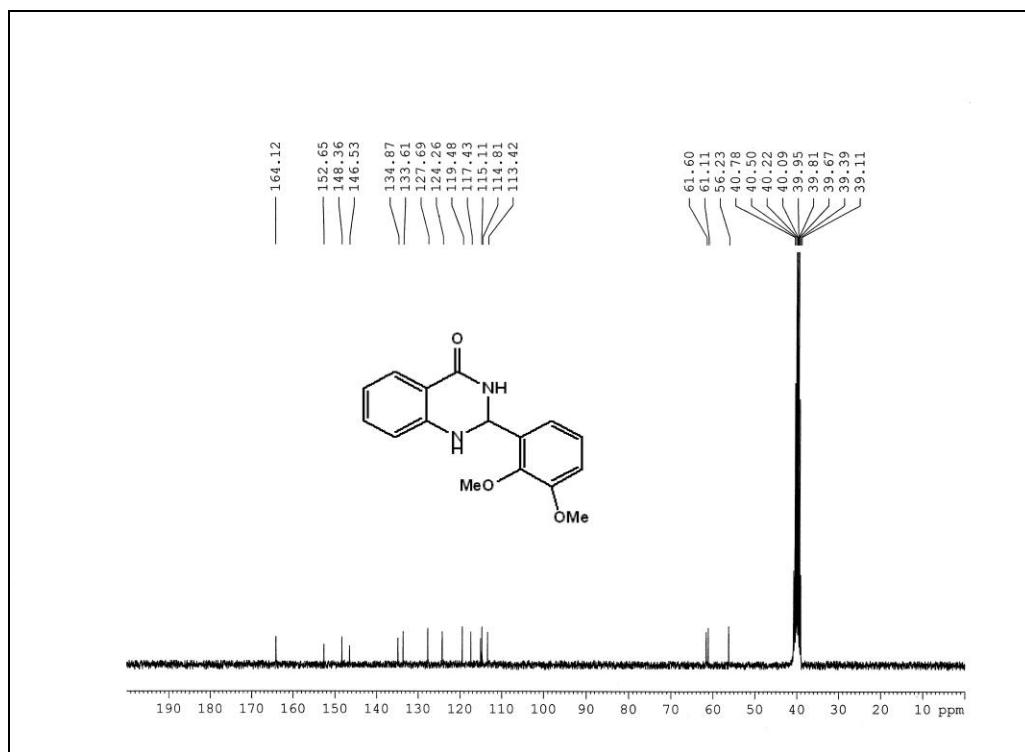
¹H NMR of Compound 3r



¹³C NMR of Compound 3r

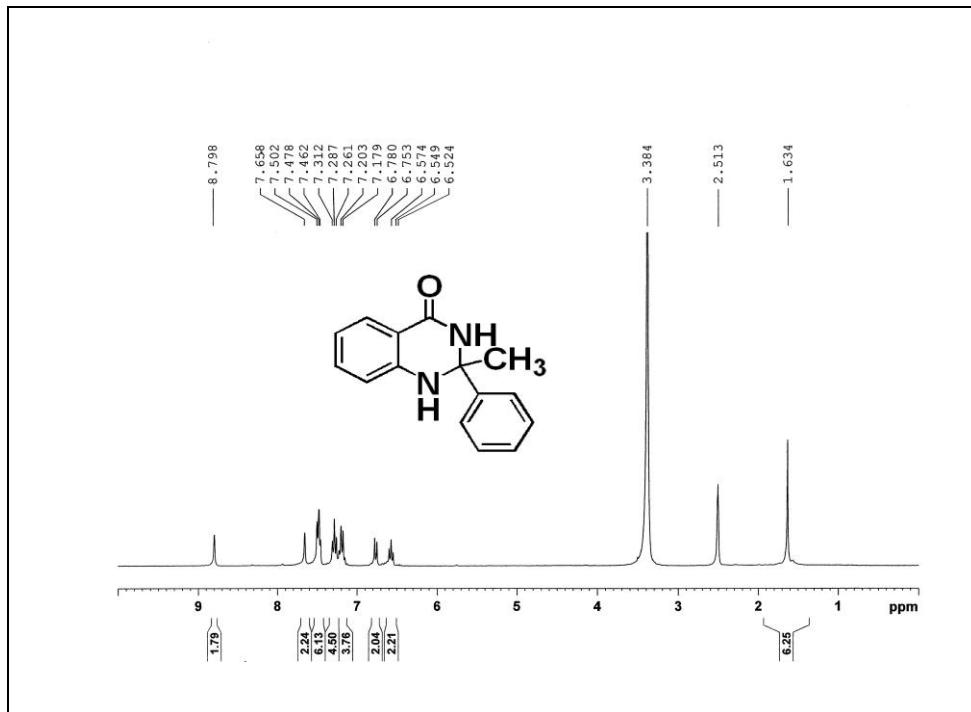


¹H NMR of Compound 3r

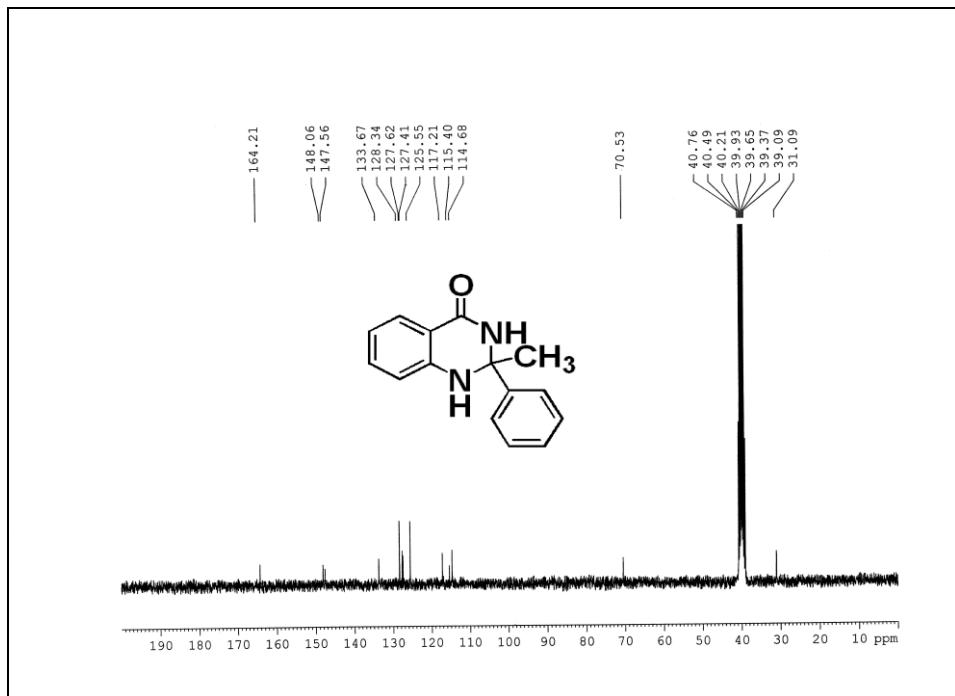


¹³C NMR of Compound 3s

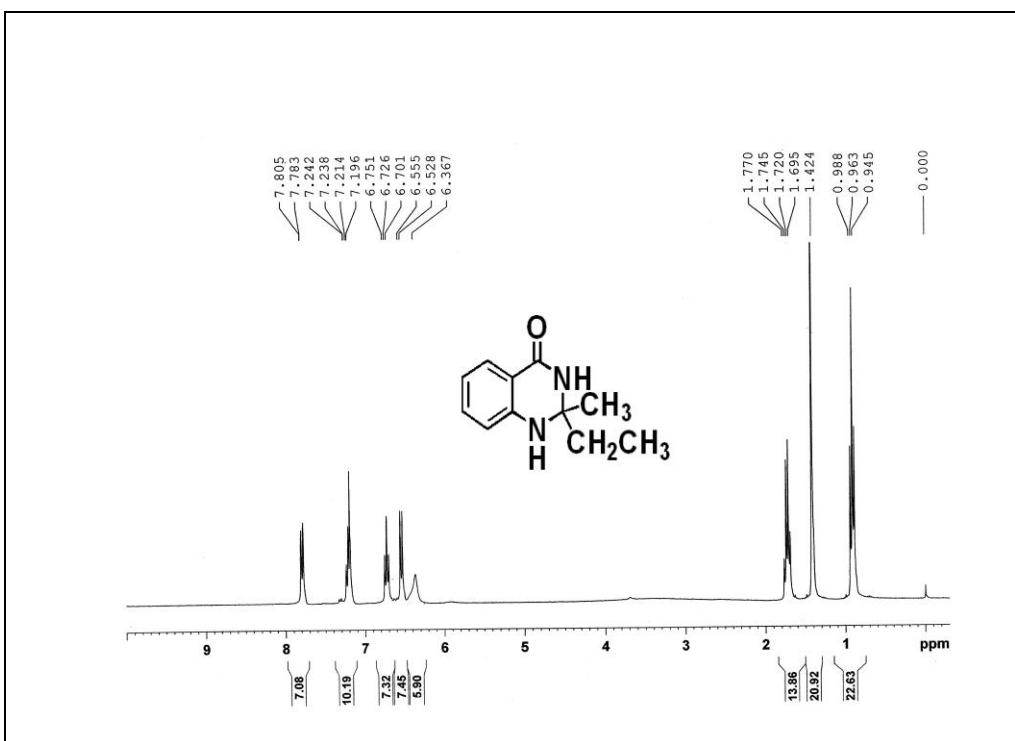
¹H NMR, ¹³C NMR Spectra of the Compounds (5a-5l):



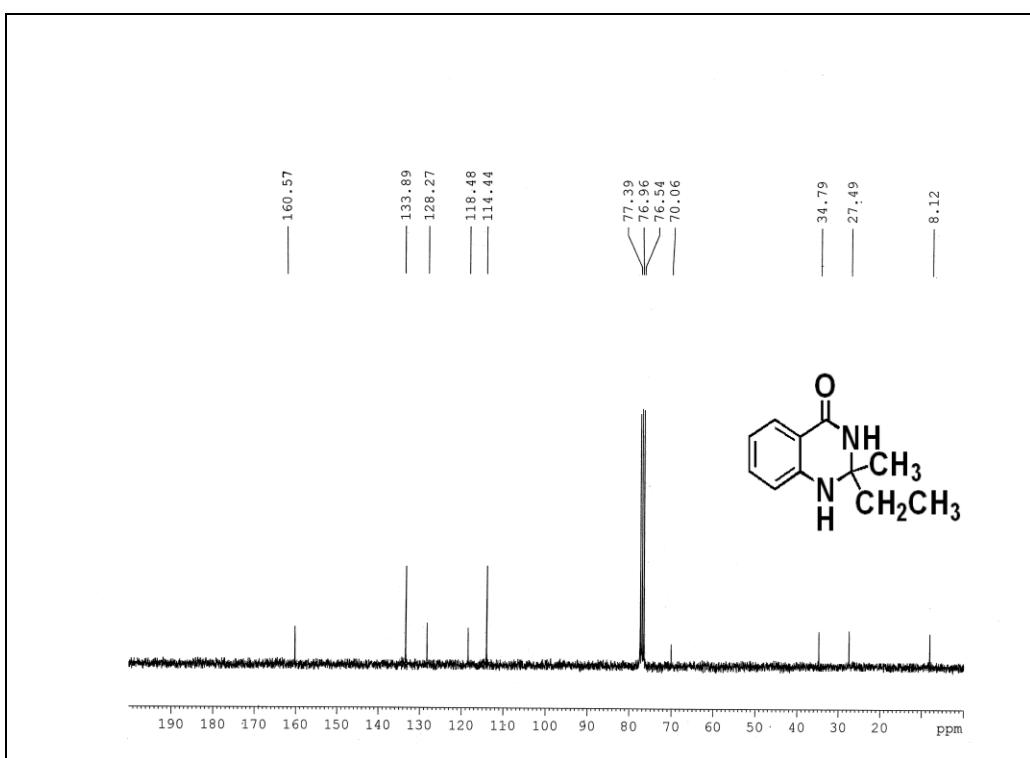
¹H NMR of Compound 5a



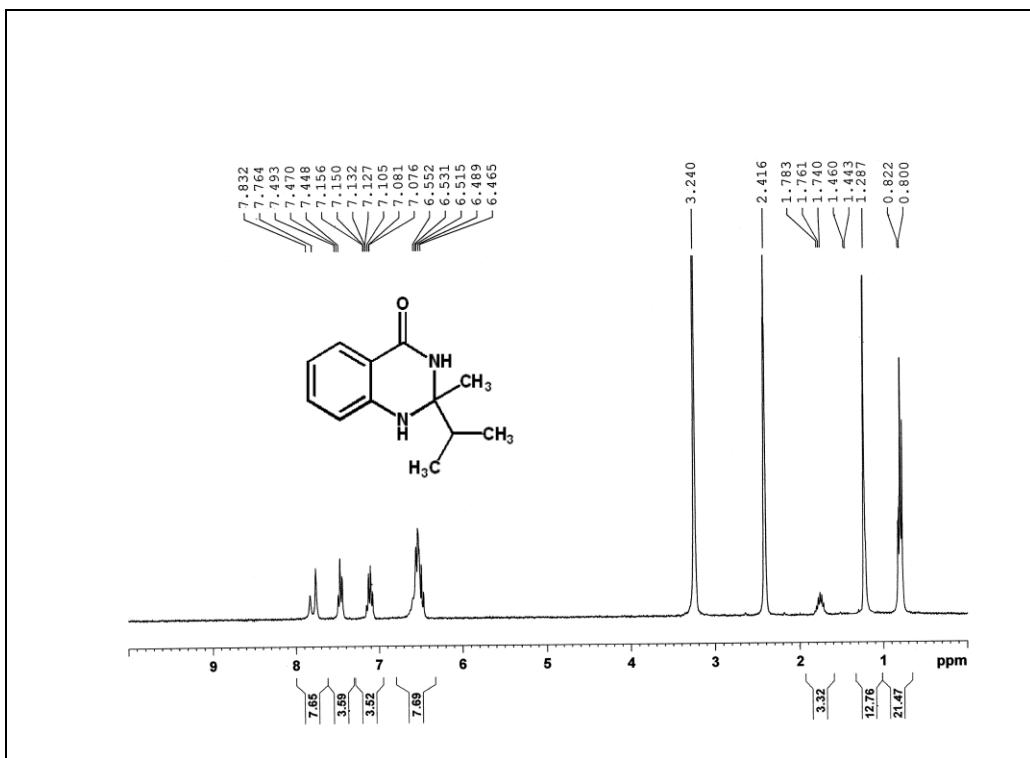
¹³C NMR of Compound 5a



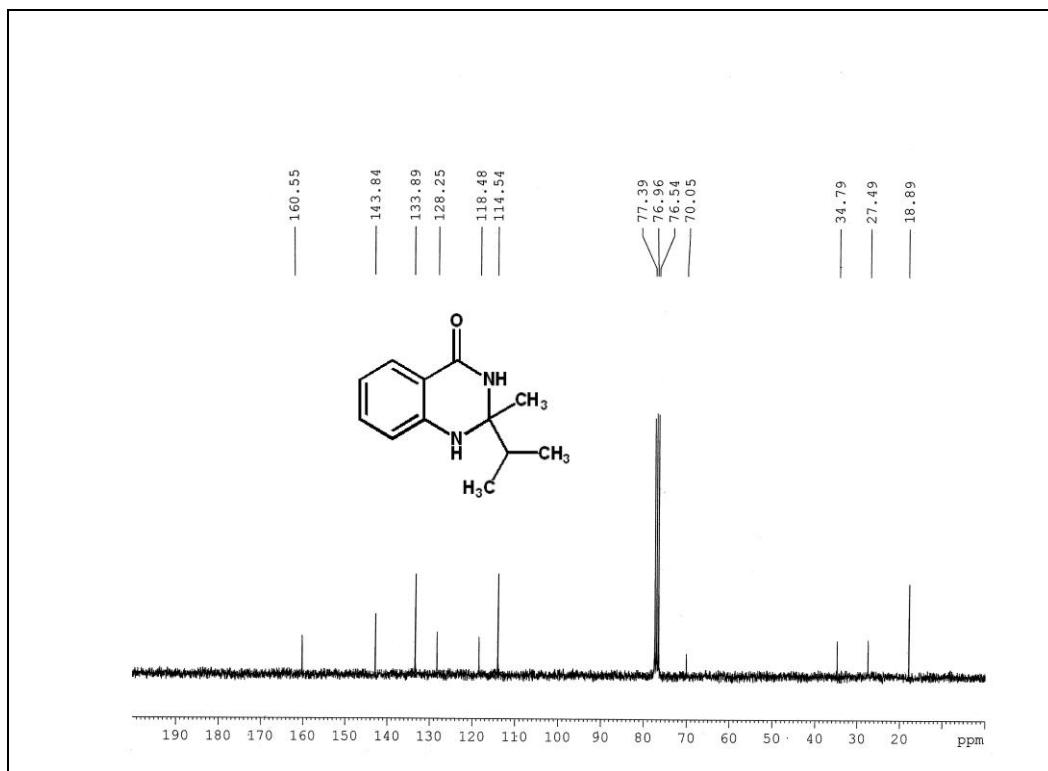
¹H NMR of Compound 5b



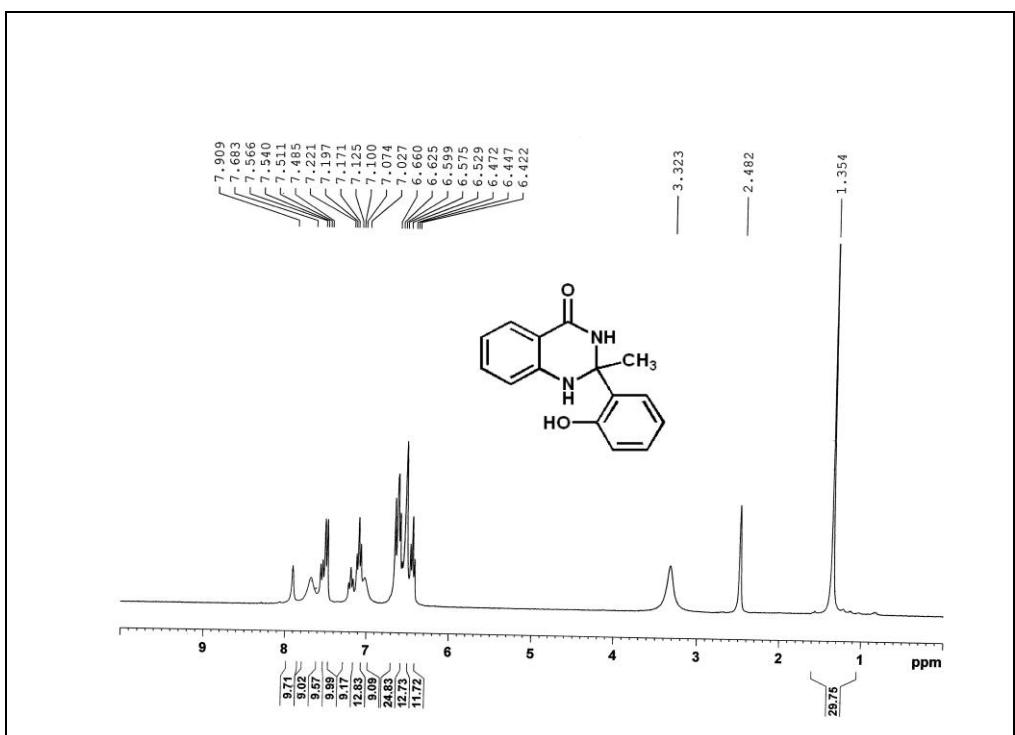
¹³C NMR of Compound 5b



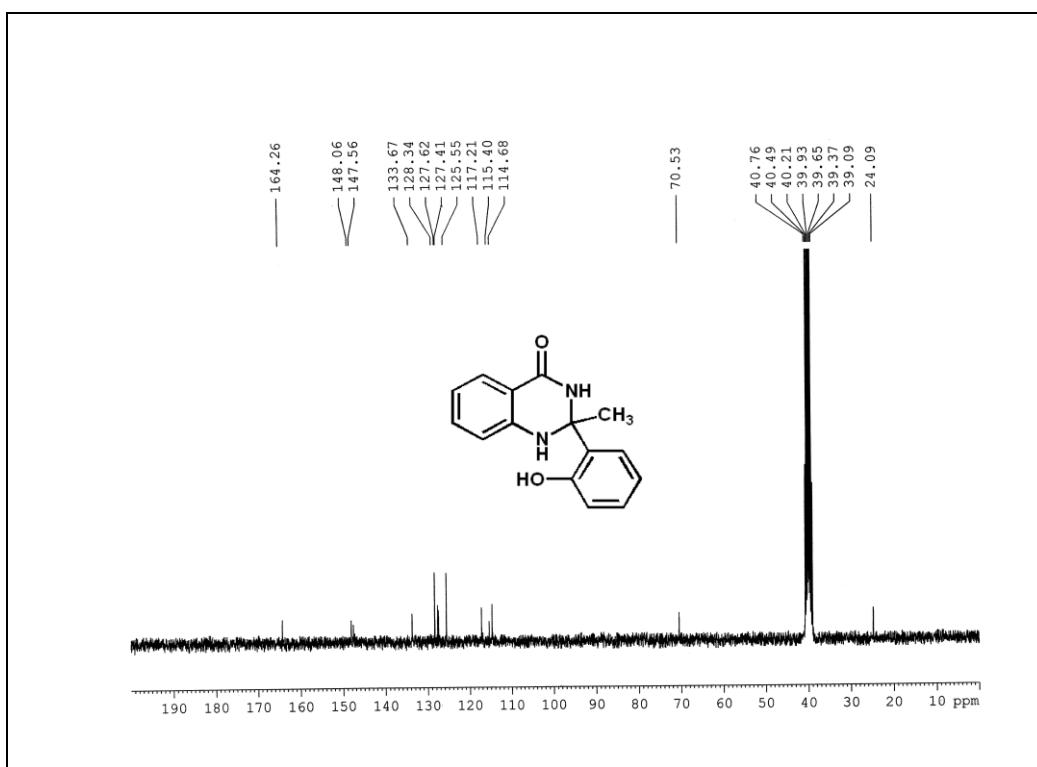
¹H NMR of Compound 5c



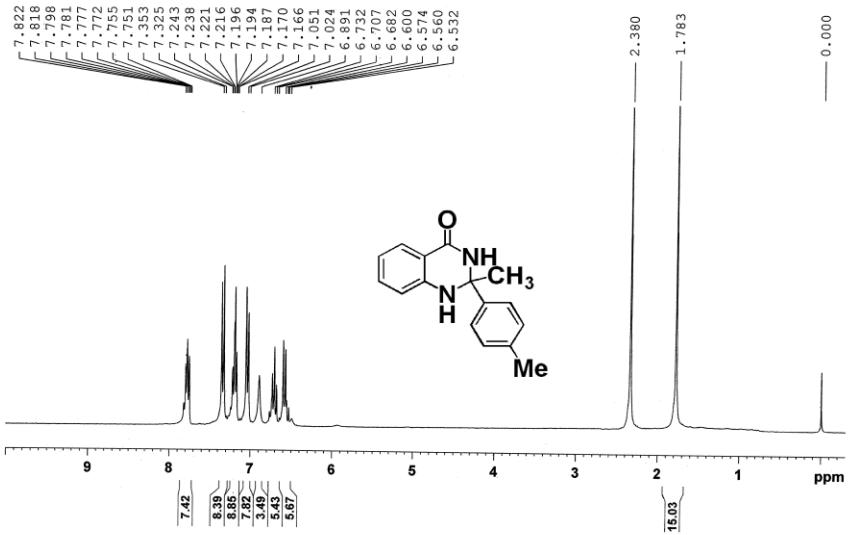
¹³C NMR of Compound 5c



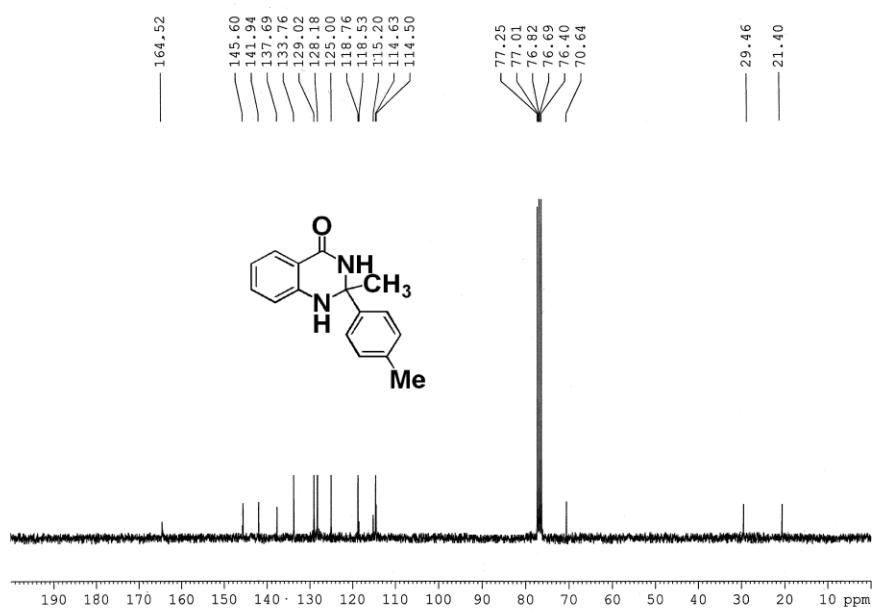
¹H NMR of Compound 5d



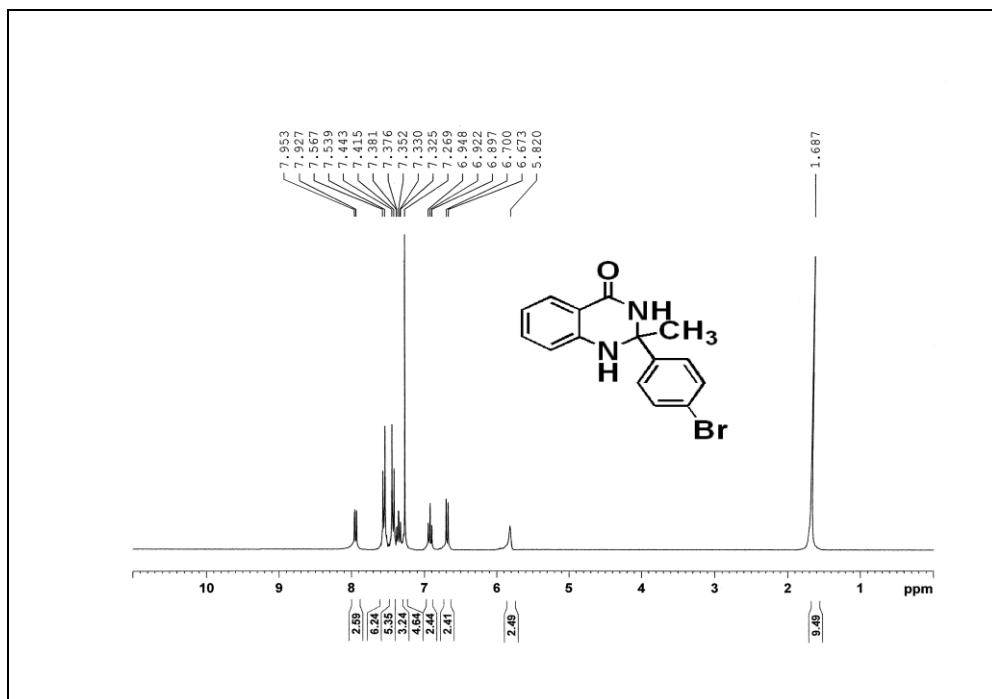
¹³C NMR of Compound 5d



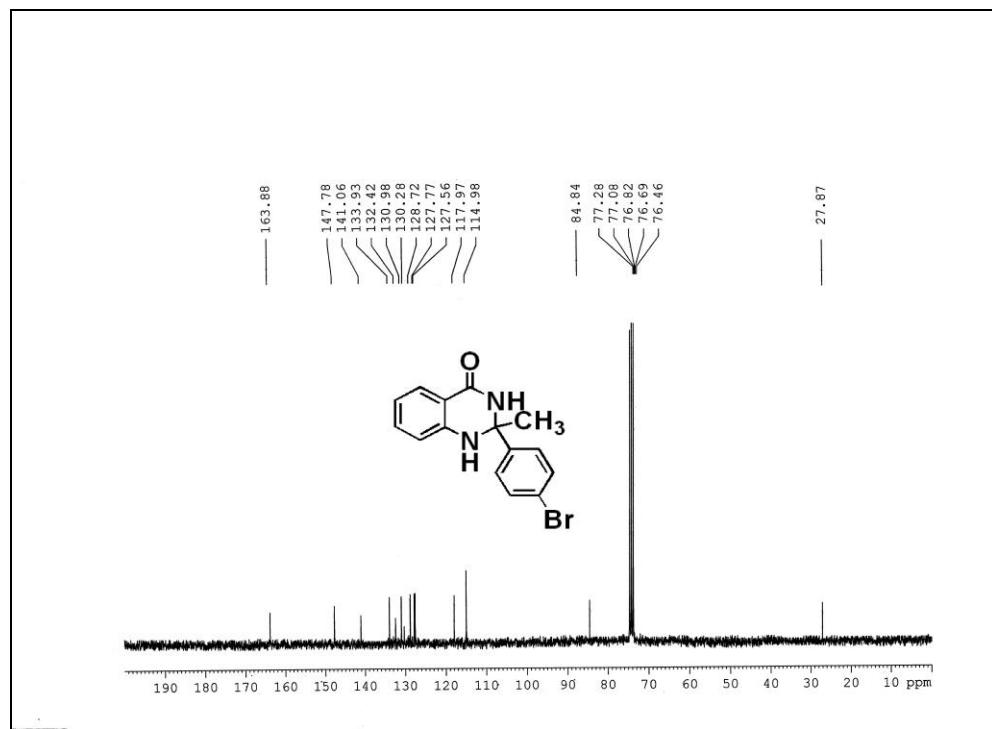
¹H NMR of Compound 5e



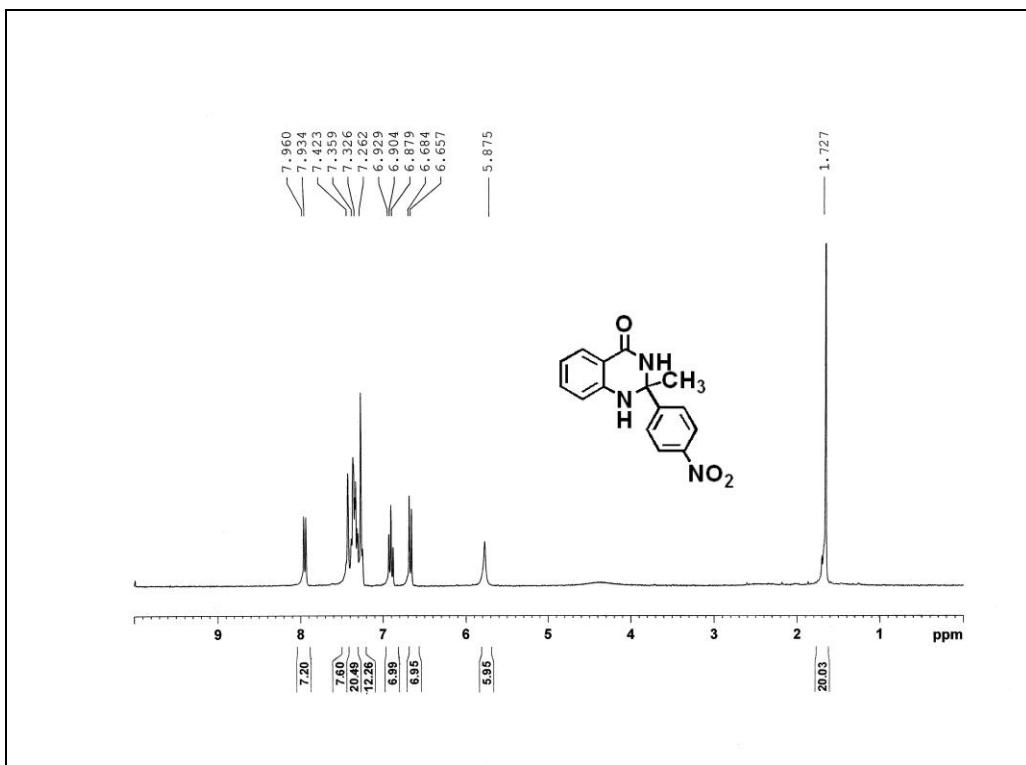
¹³C NMR of Compound 5e



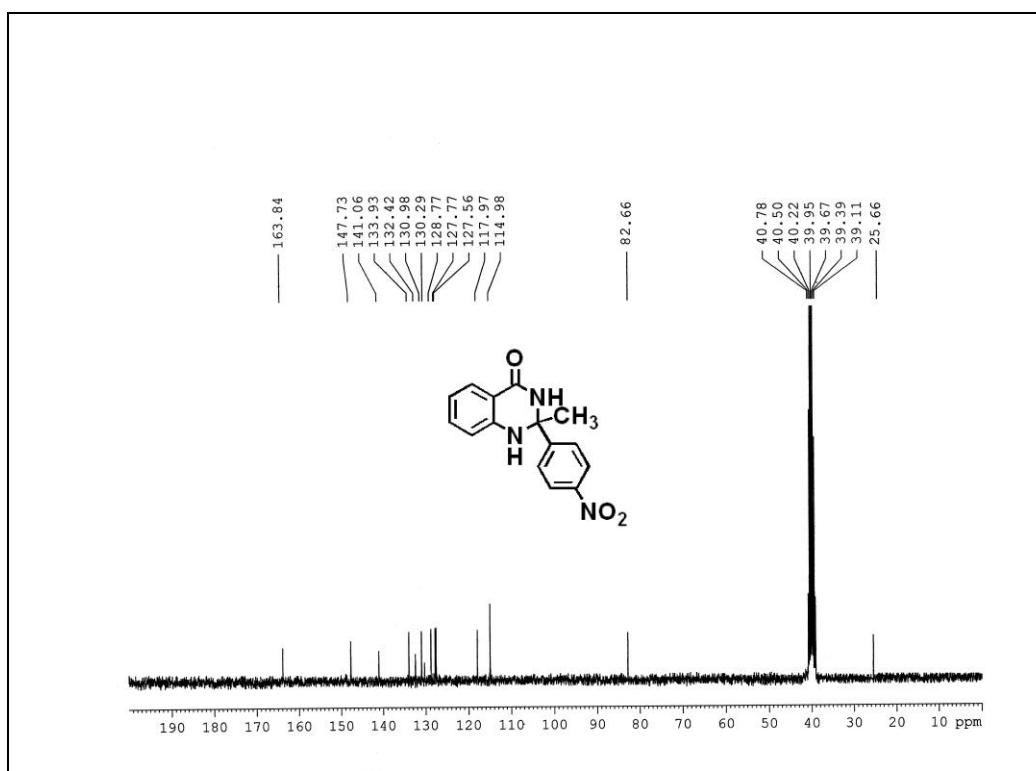
¹H NMR of Compound 5f



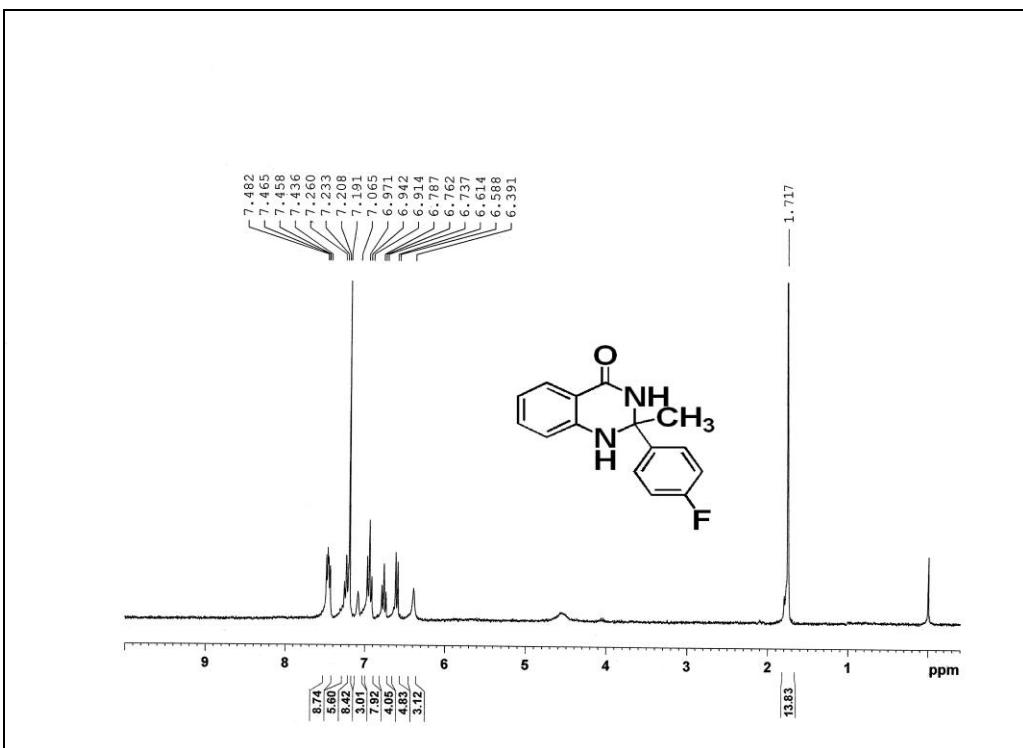
¹³C NMR of Compound 5f



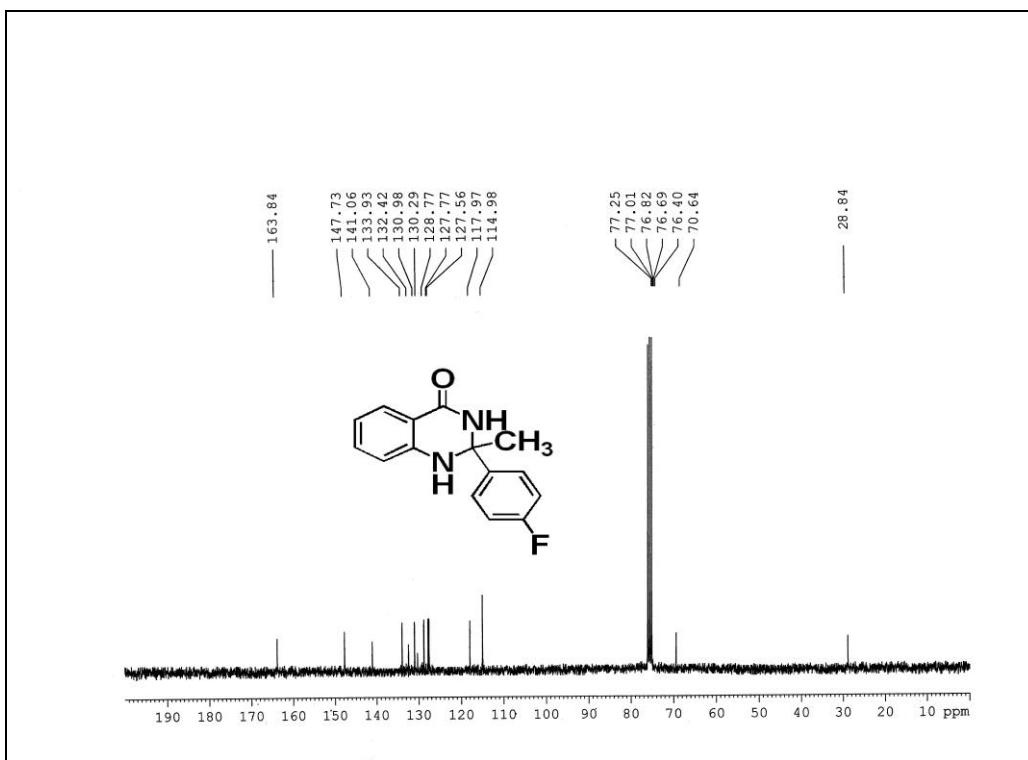
¹H NMR of Compound 5g



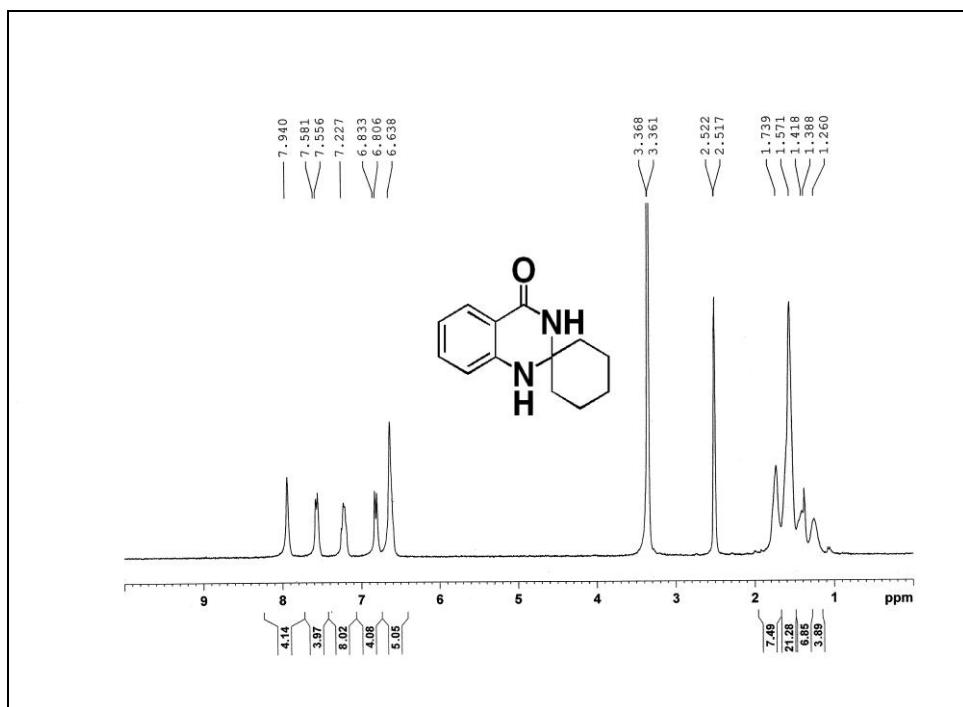
¹³C NMR of Compound 5g



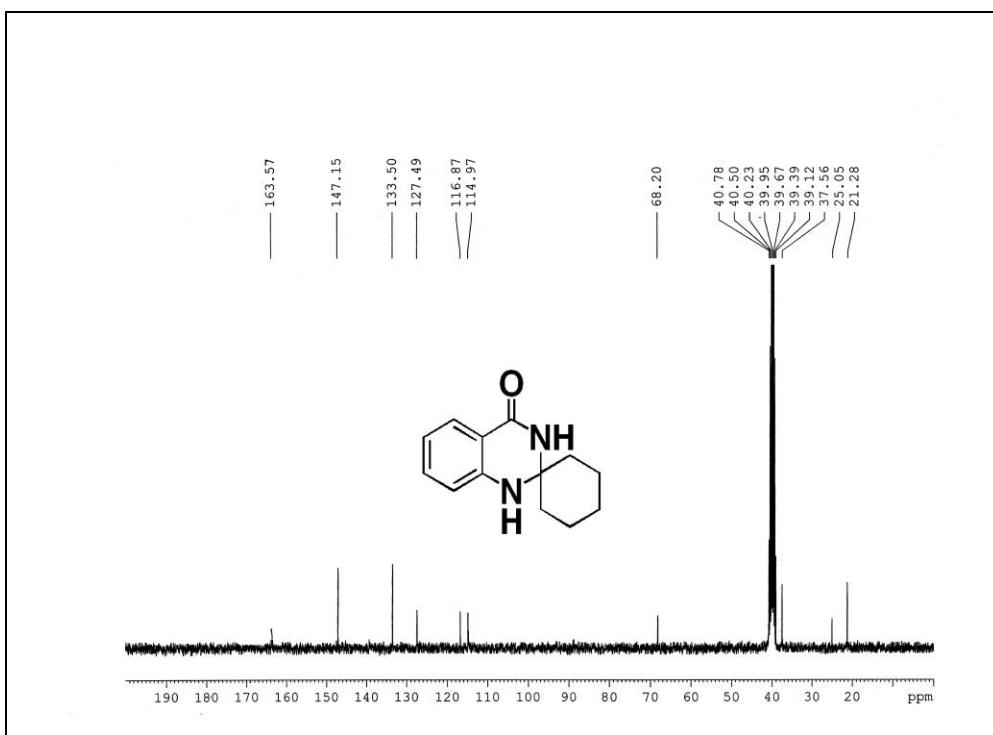
¹H NMR of Compound 5h



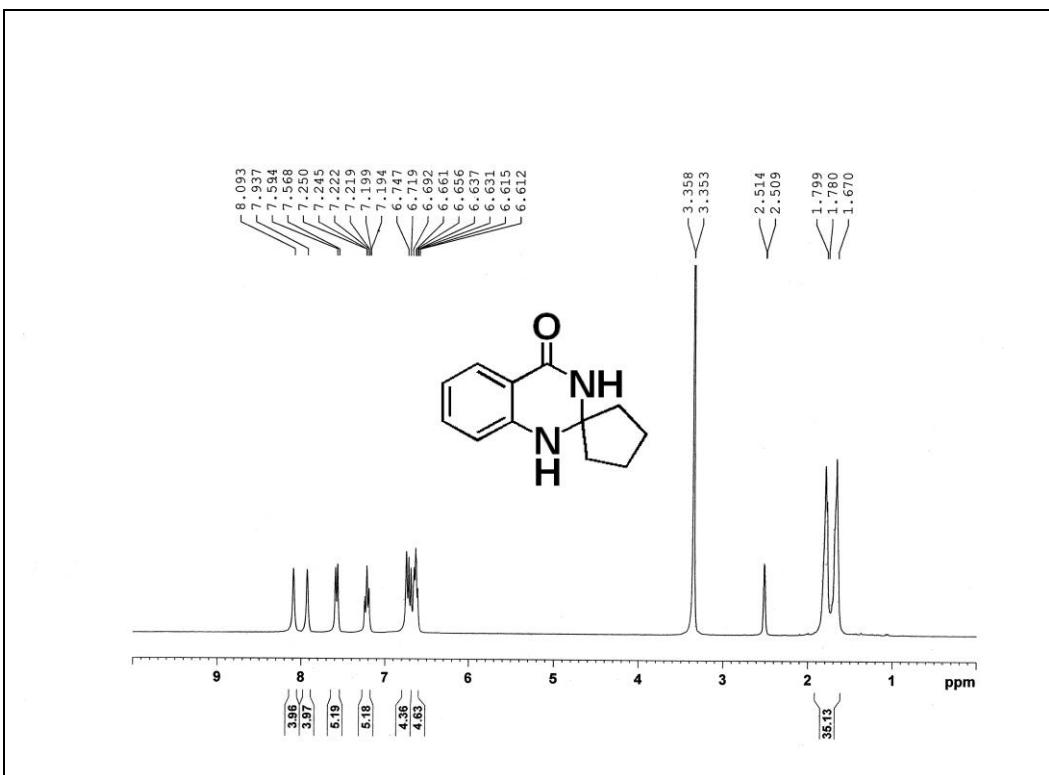
¹³H CMR of Compound 5h



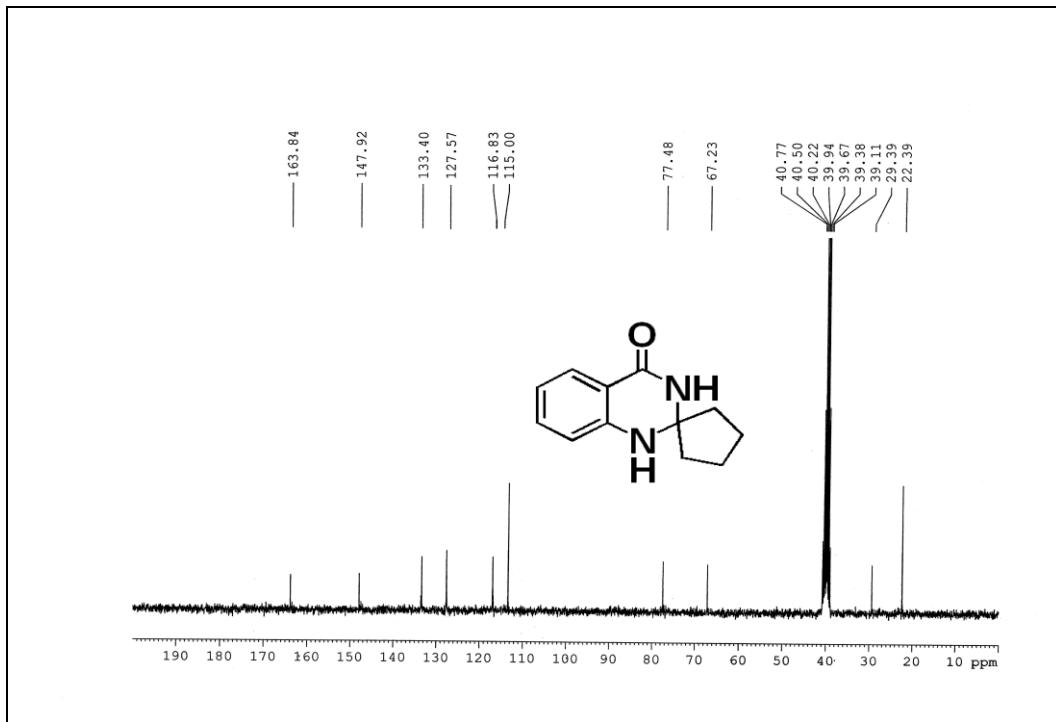
¹H NMR of Compound 5i



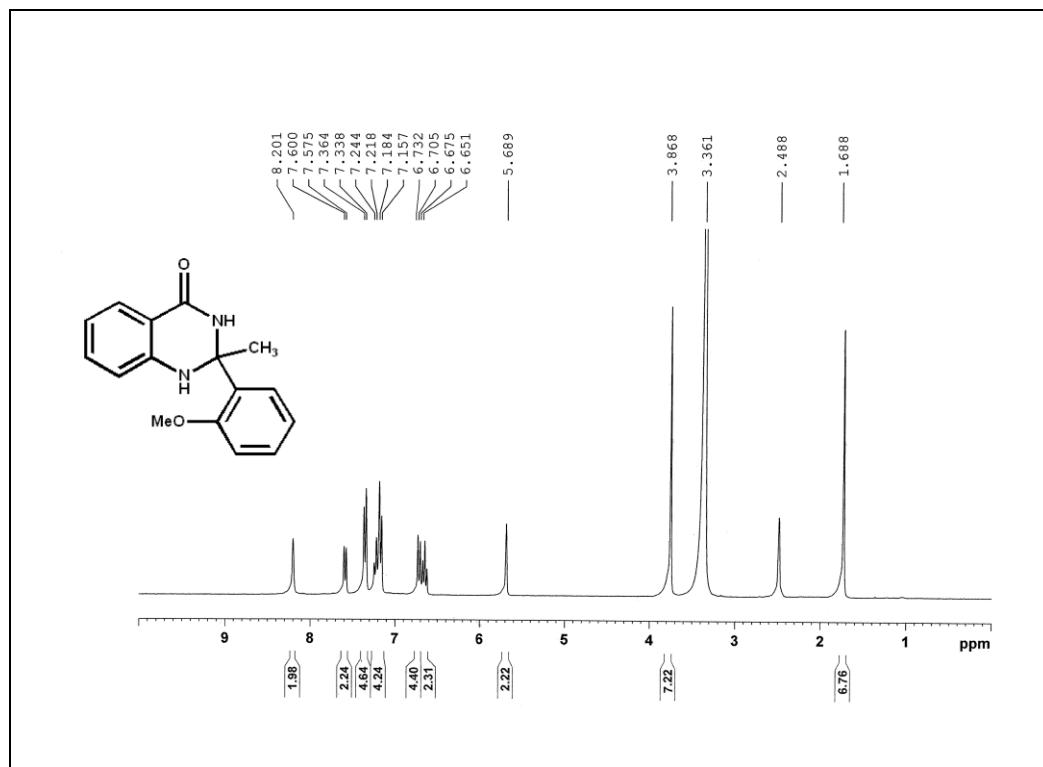
¹³C NMR of Compound 5i



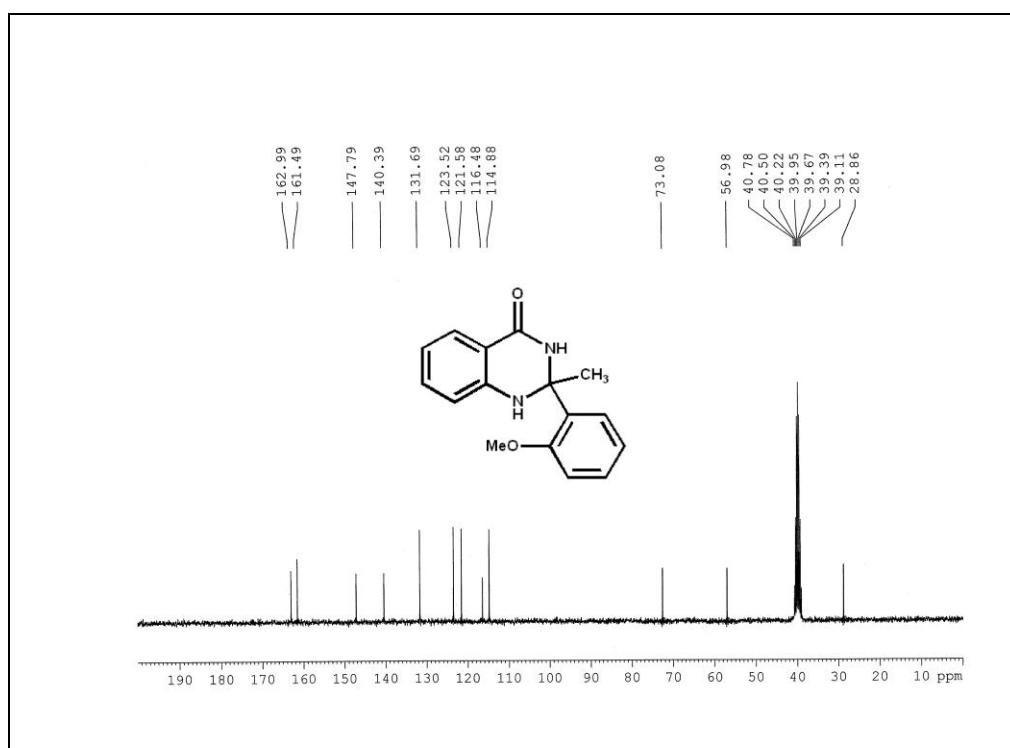
¹H NMR of Compound 5j



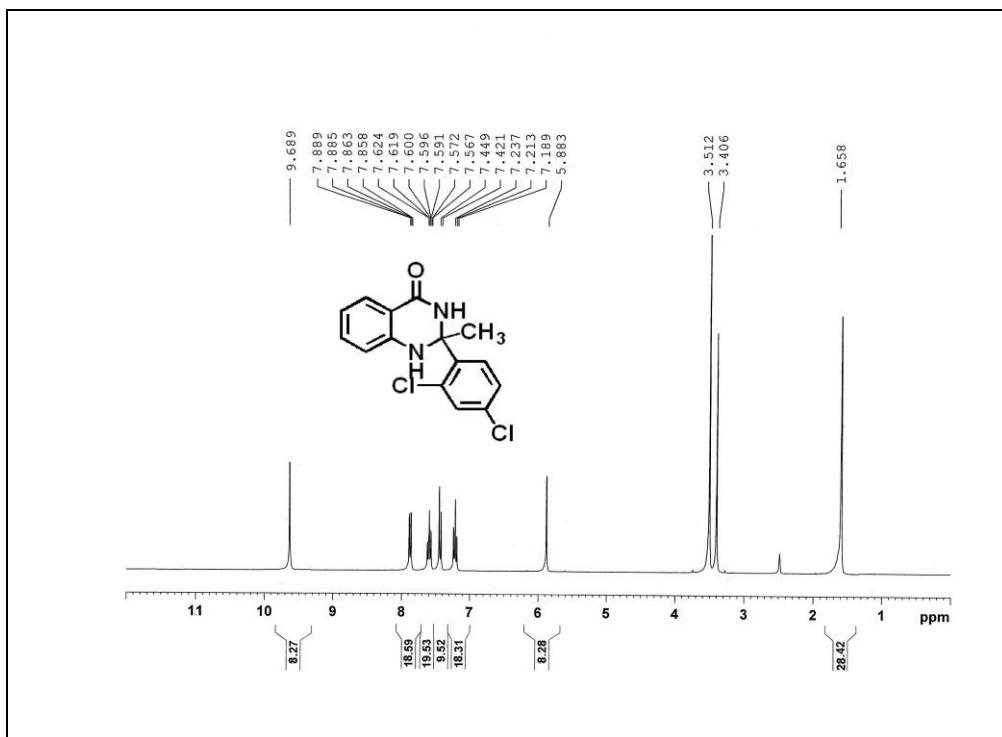
¹³C NMR of Compound 5j



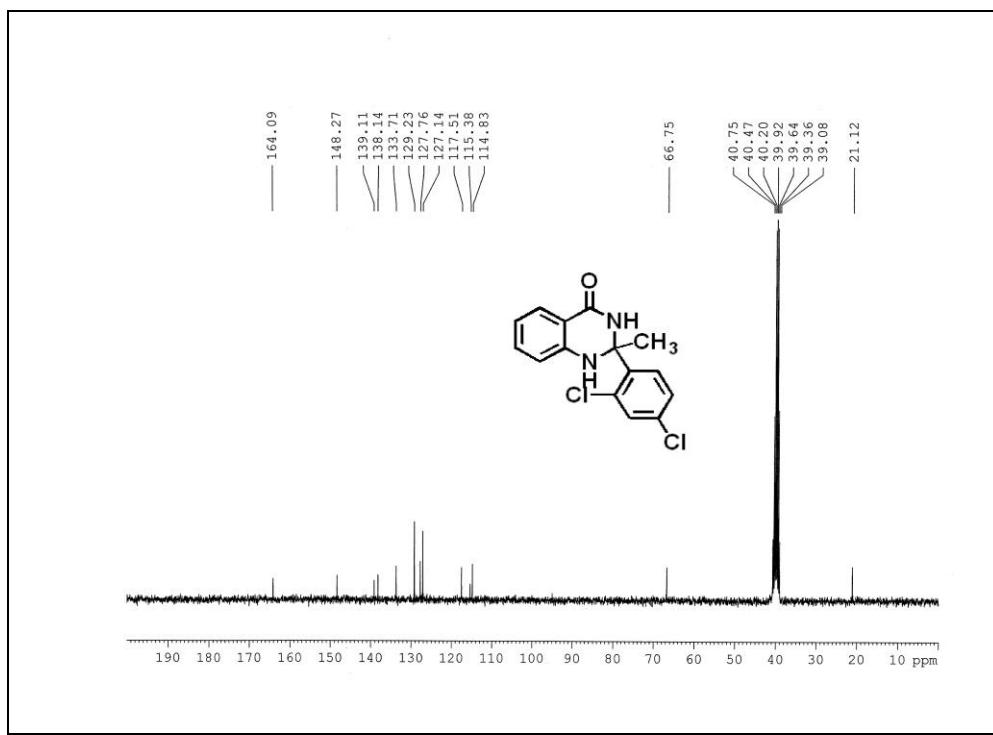
¹H NMR of Compound 5k



¹³C NMR of Compound 5k

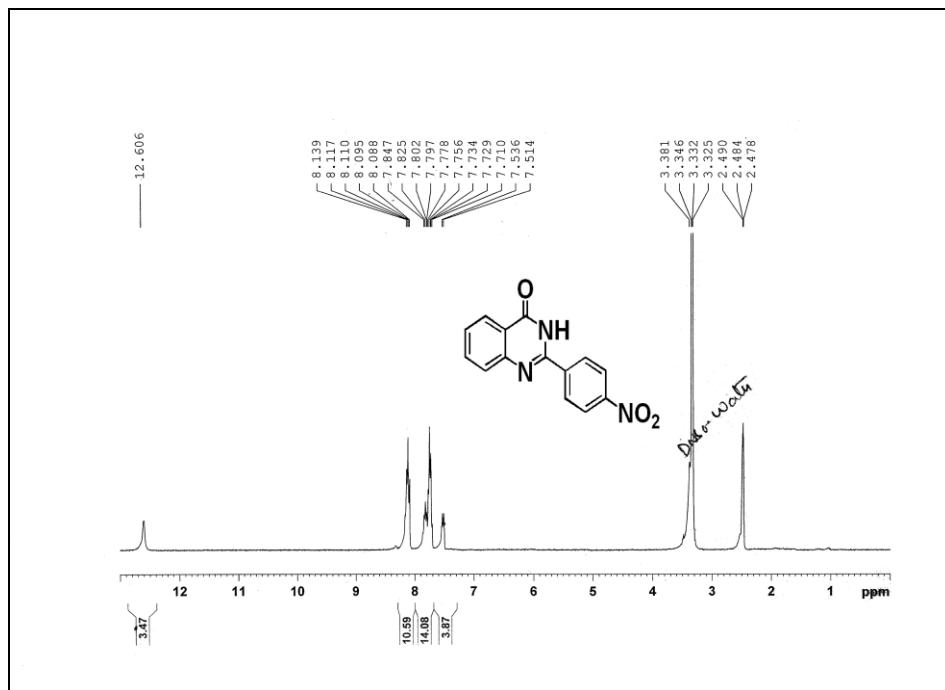


¹H NMR of Compound 51

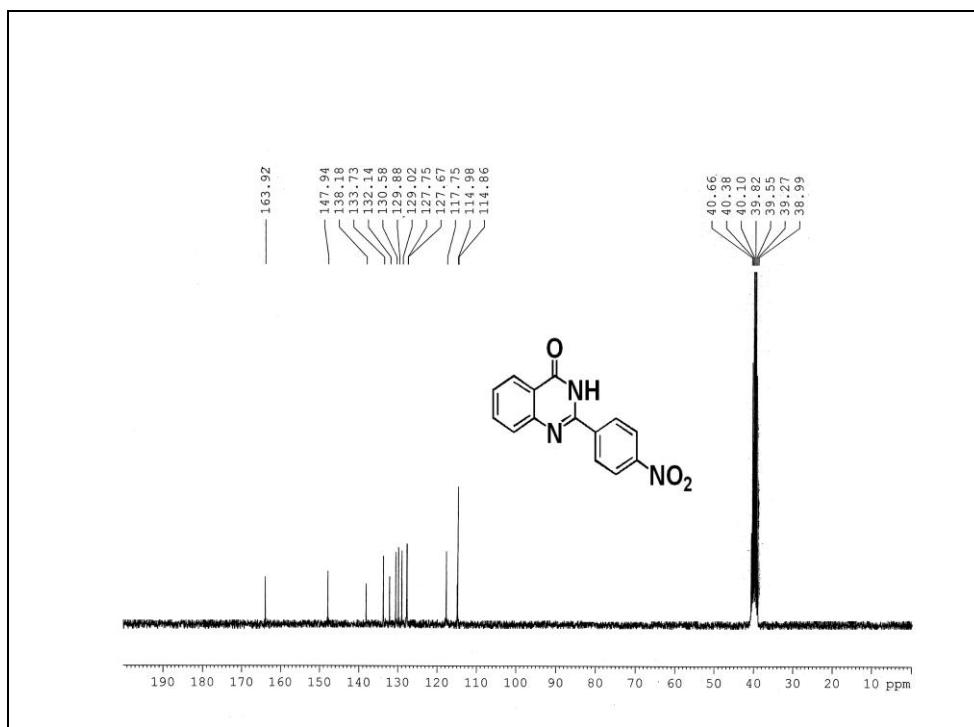


¹³C NMR of Compound 51

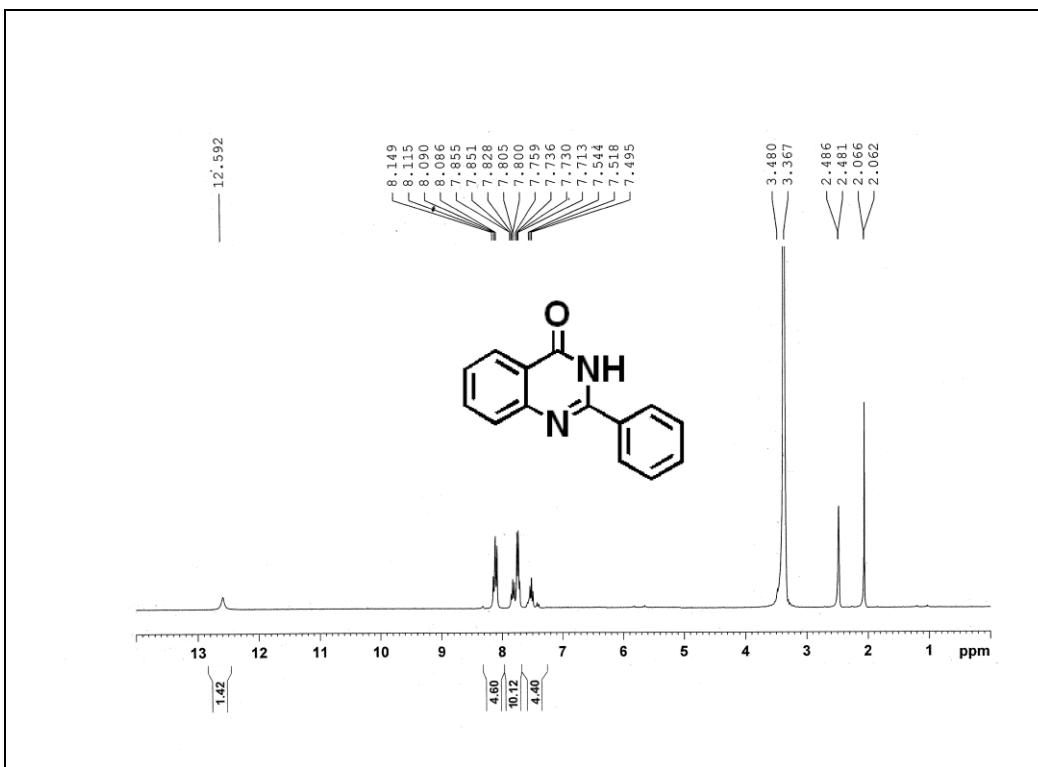
¹H NMR, ¹³C NMR Spectra of the Compounds (6a-6i)):



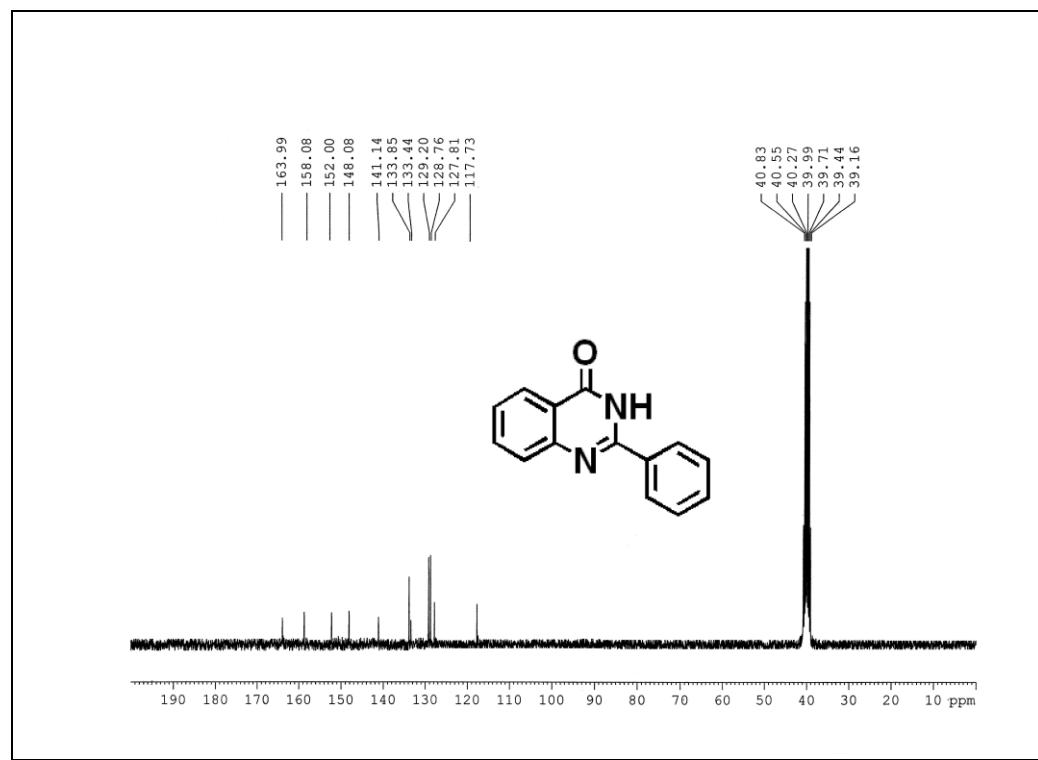
¹H NMR of Compound 6a



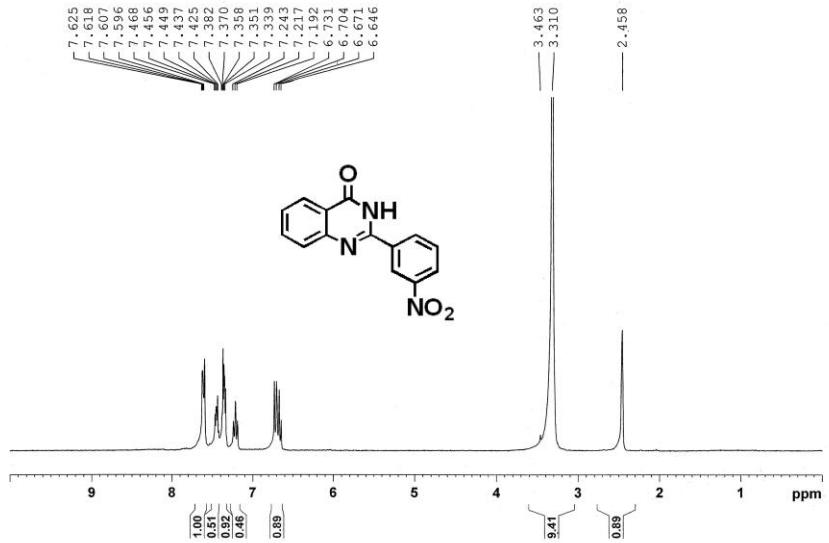
¹³C NMR of Compound 6a



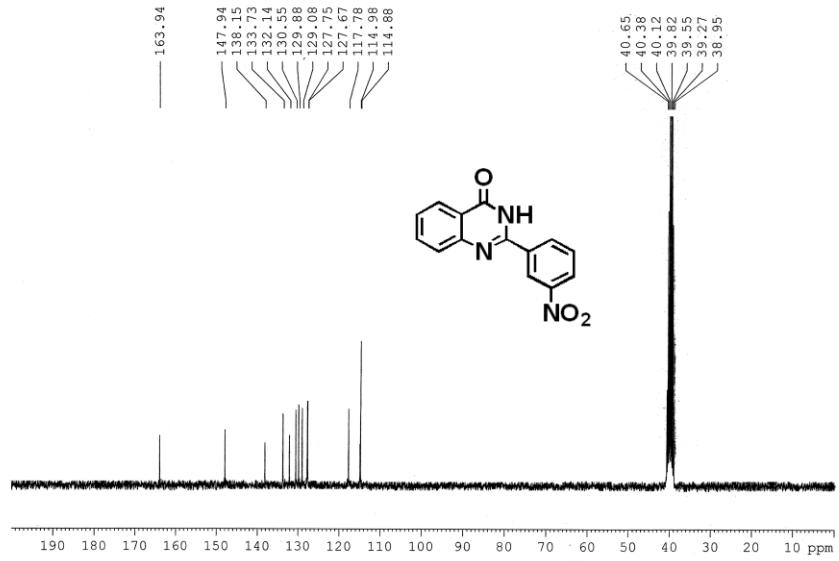
¹H NMR of Compound 6b



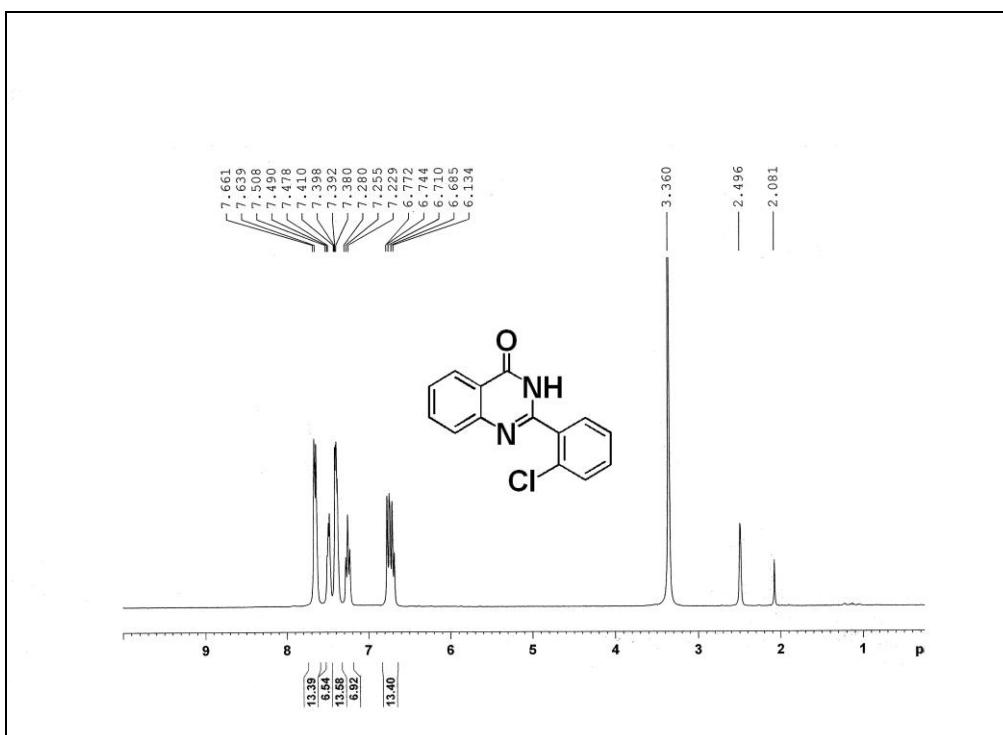
¹³C NMR of Compound 6b



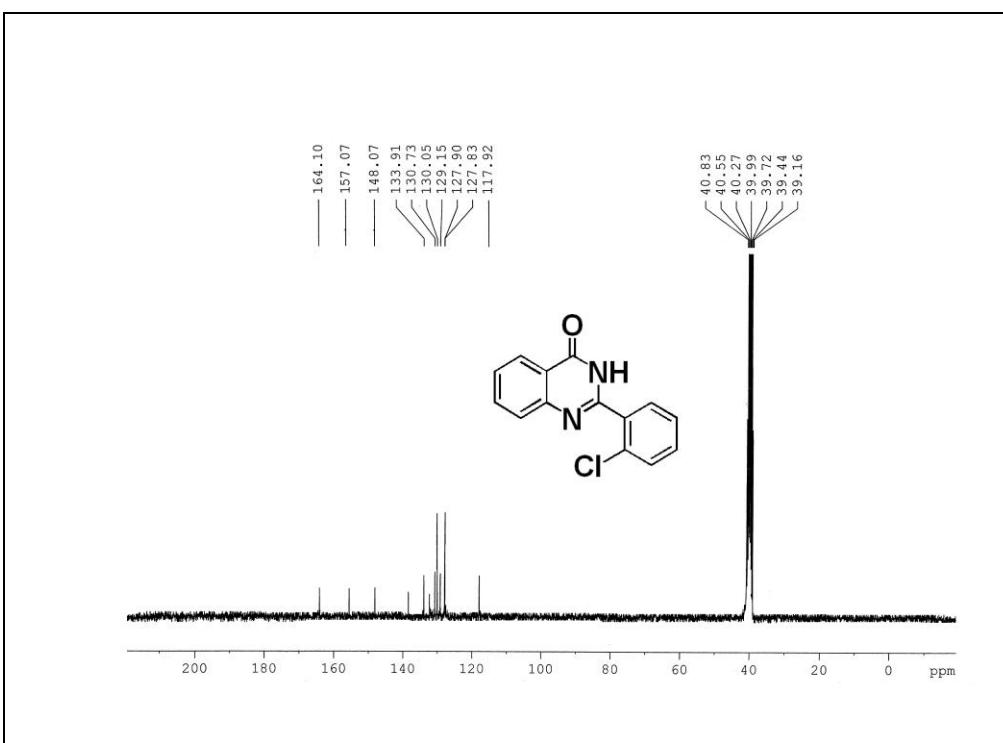
¹H NMR of Compound 6c



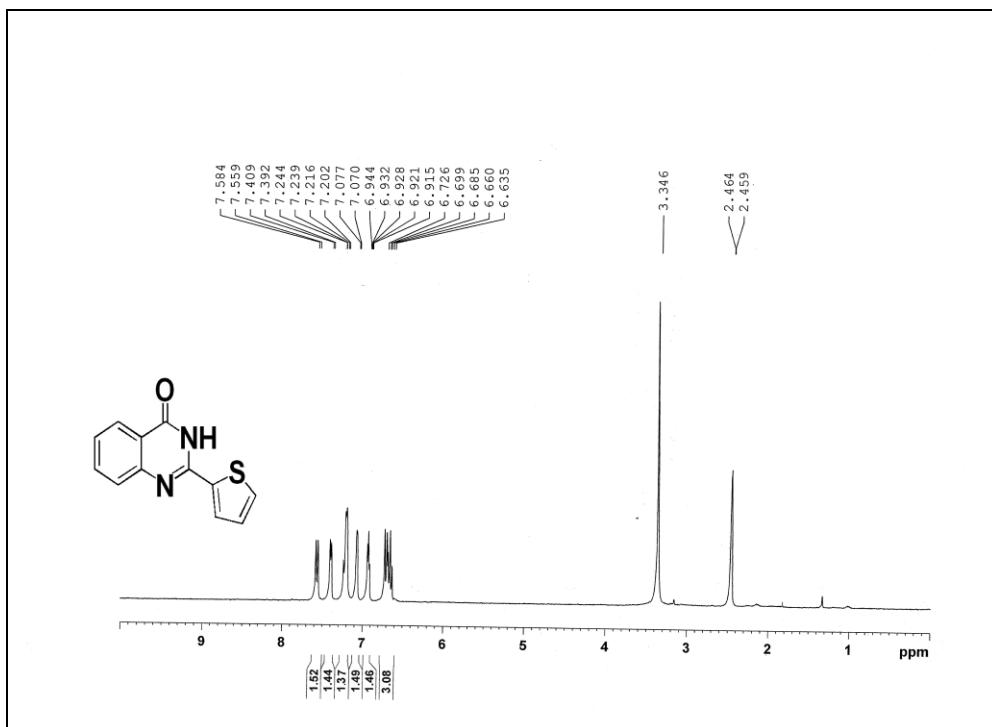
¹³C NMR of Compound 6c



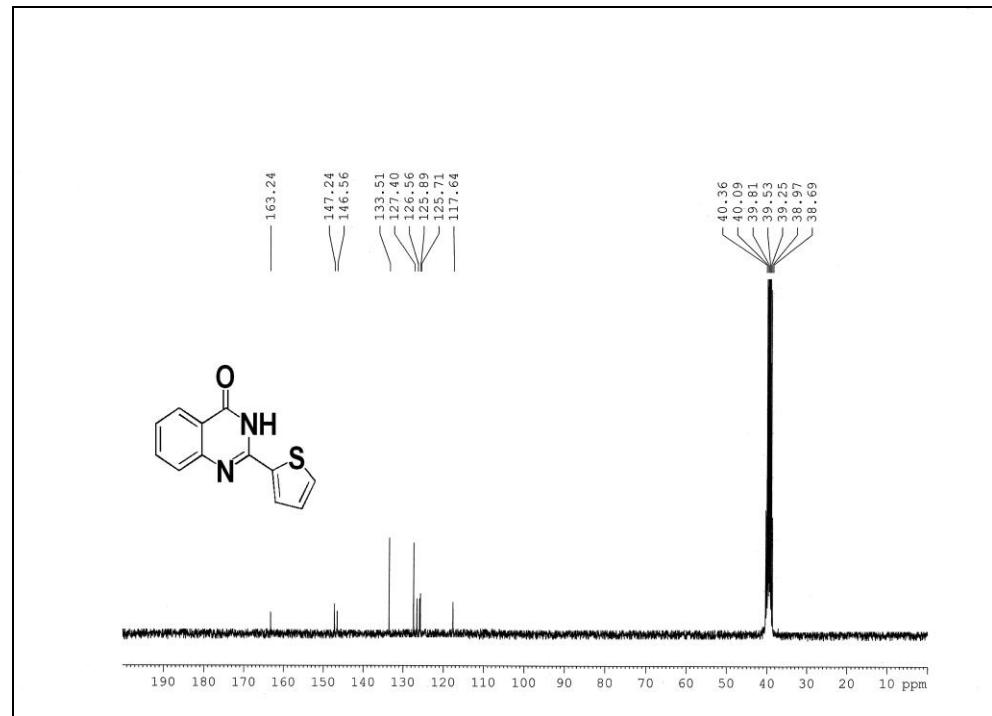
¹H NMR of Compound 6d



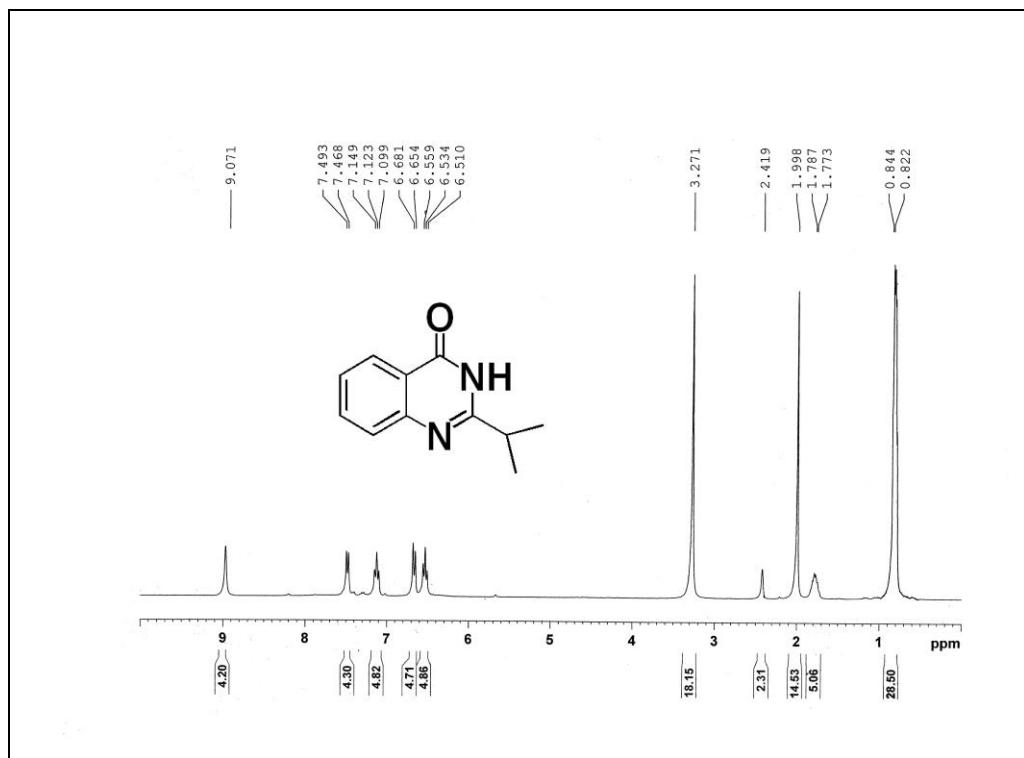
¹³C NMR of Compound 6d



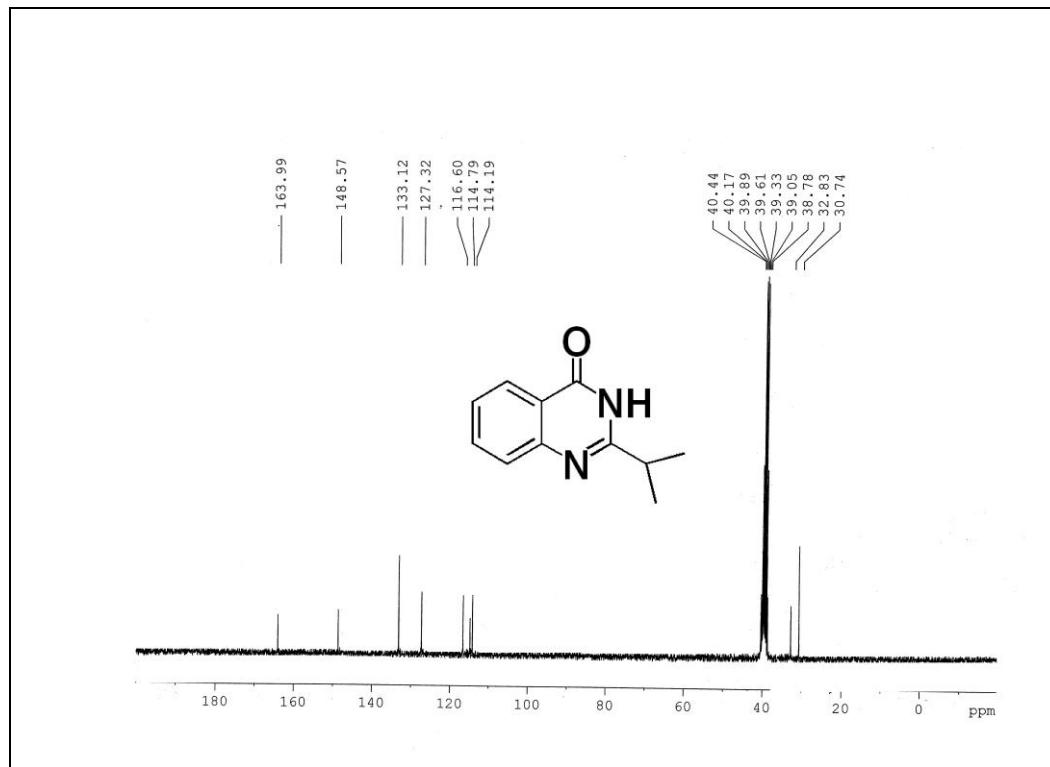
¹H NMR of Compound 6e



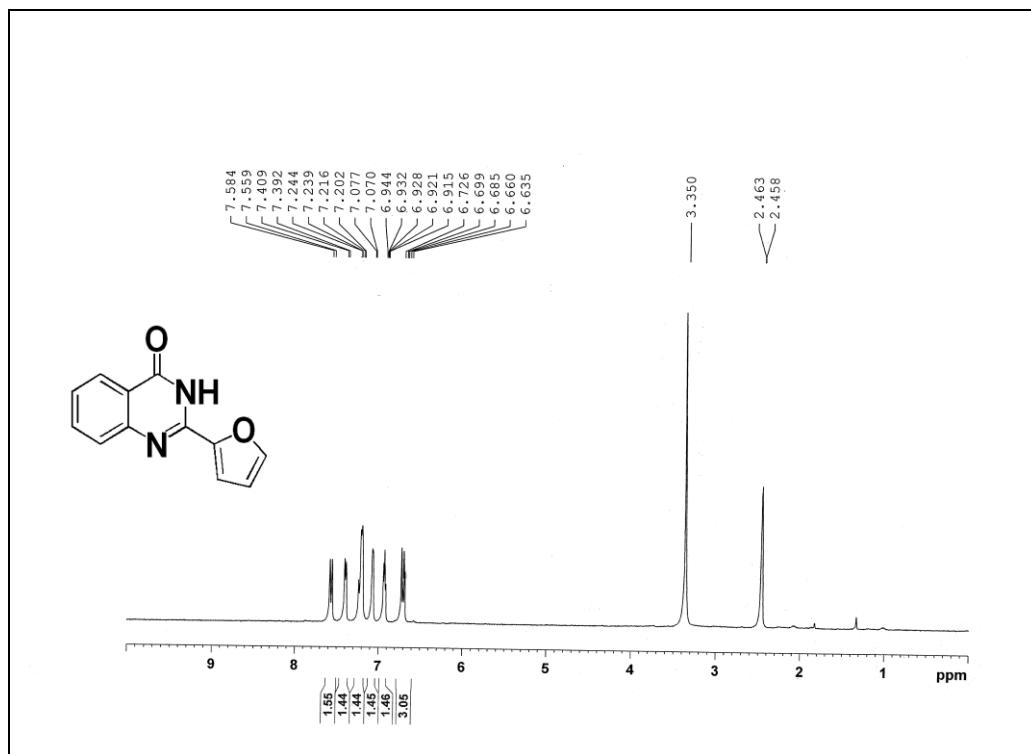
¹³C NMR of Compound 6e



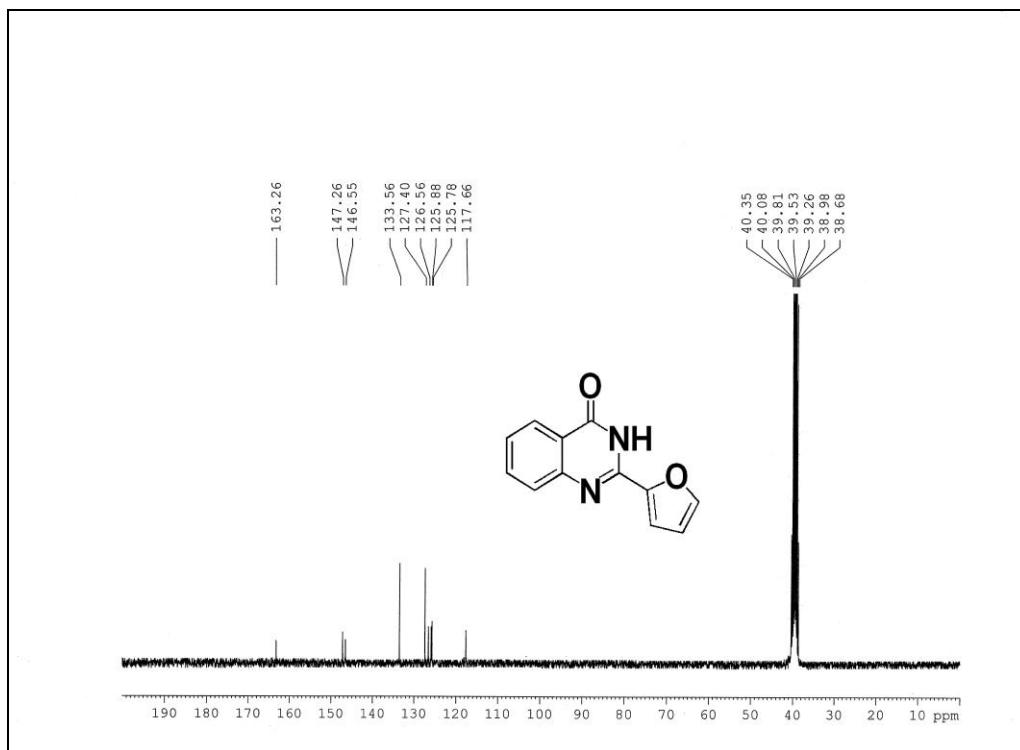
¹H NMR of Compound 6f



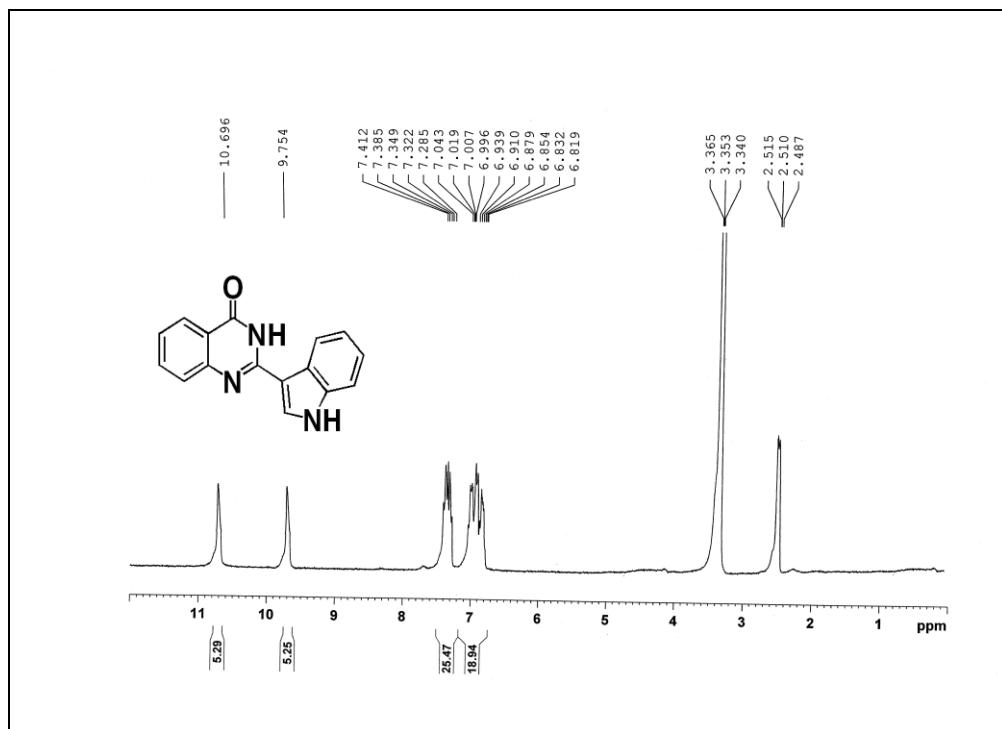
¹³C NMR of Compound 6f



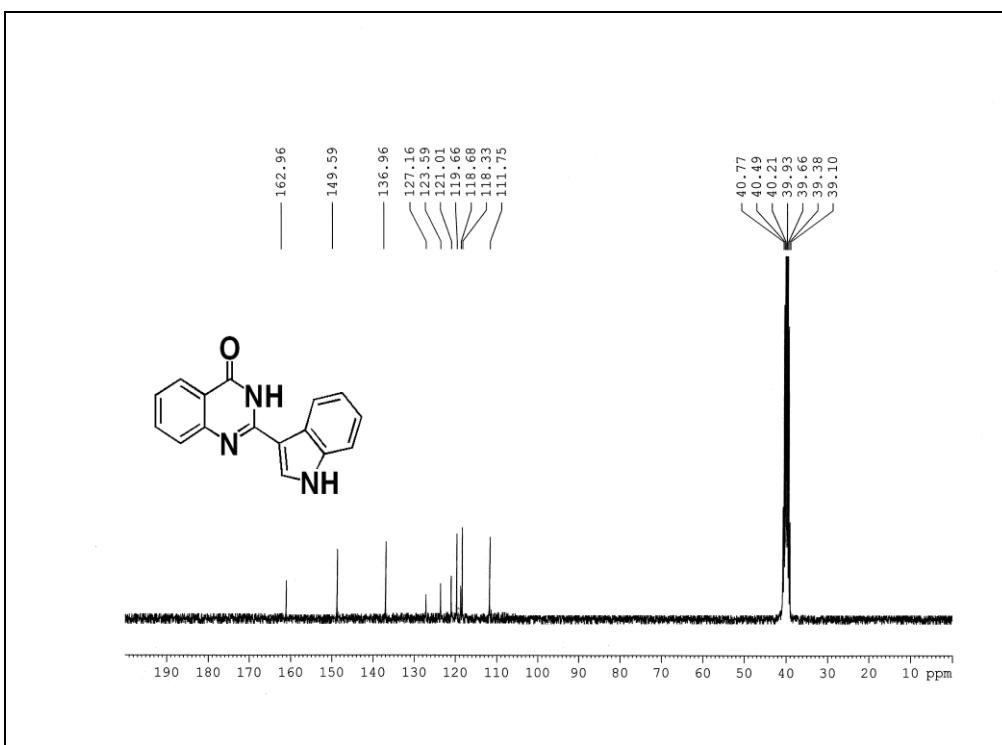
¹H NMR of Compound 6g



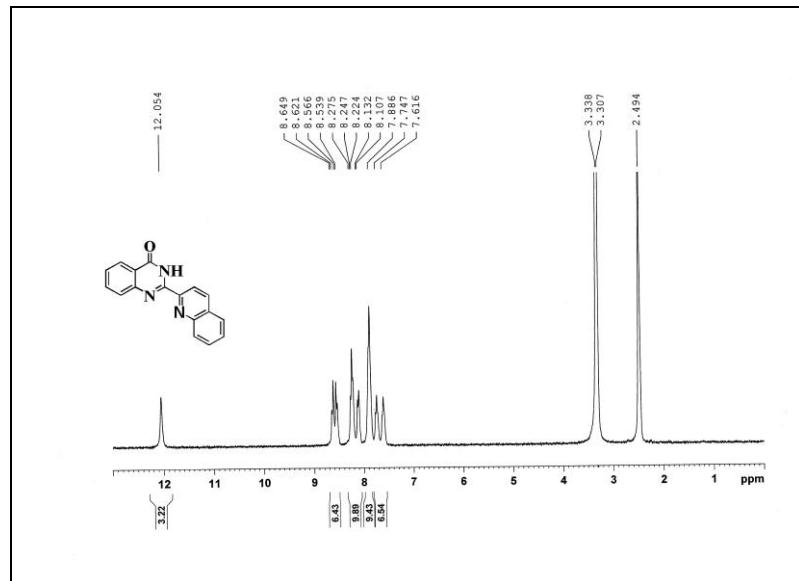
¹³C NMR of Compound 6g



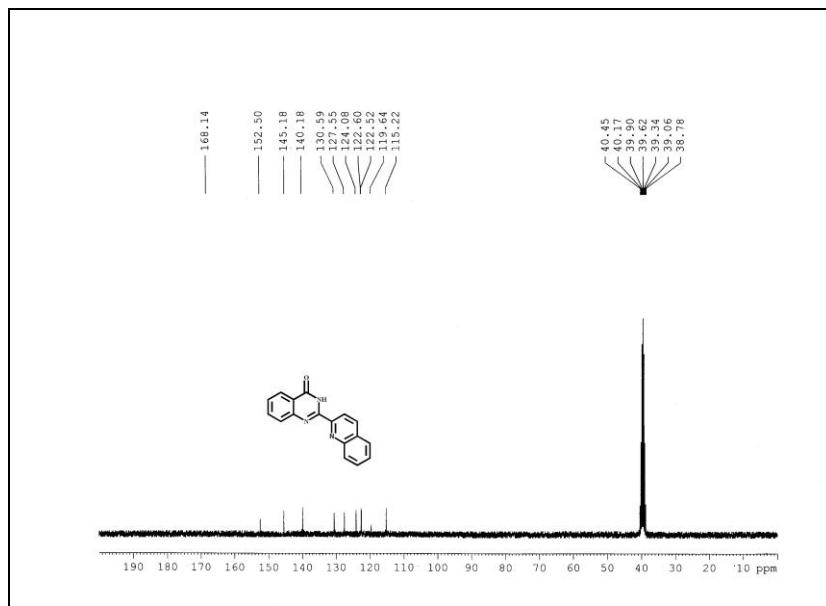
¹H NMR of Compound 6h



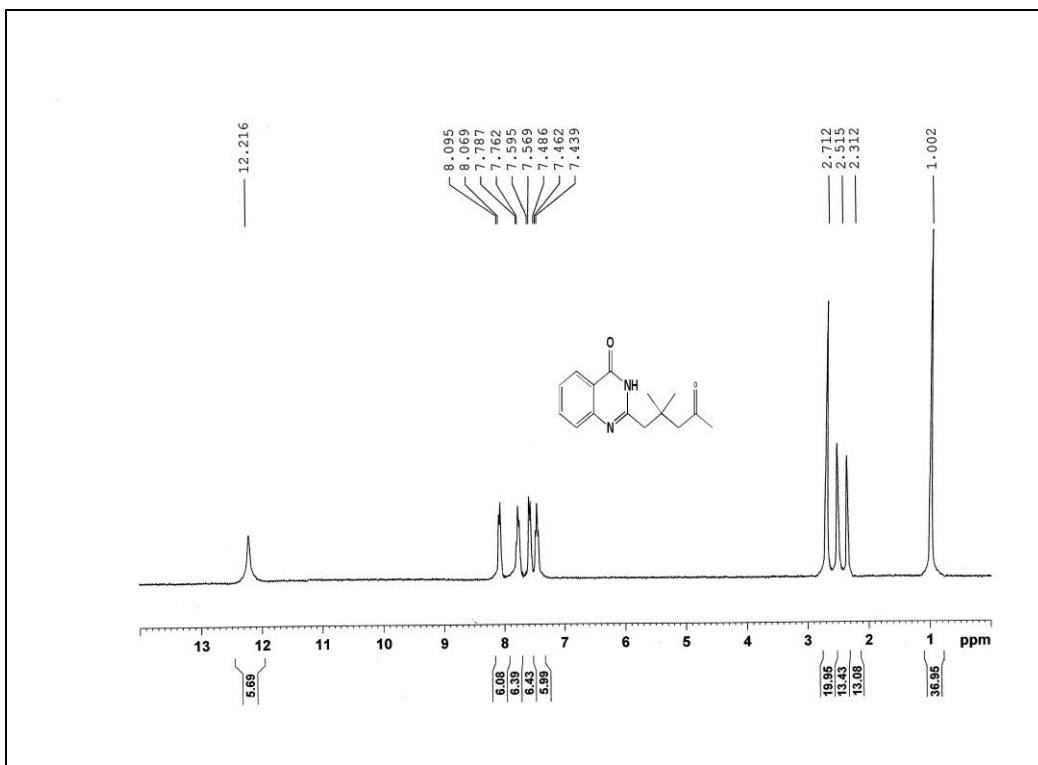
¹³C NMR of Compound 6h



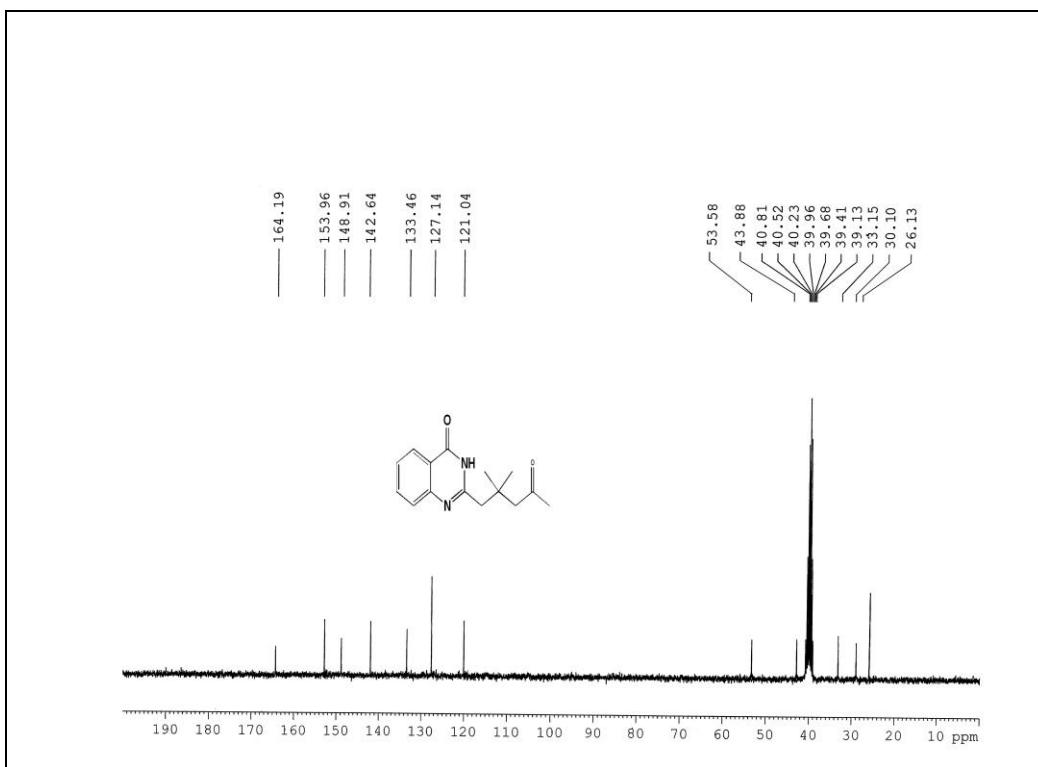
¹H NMR of Compound 6i



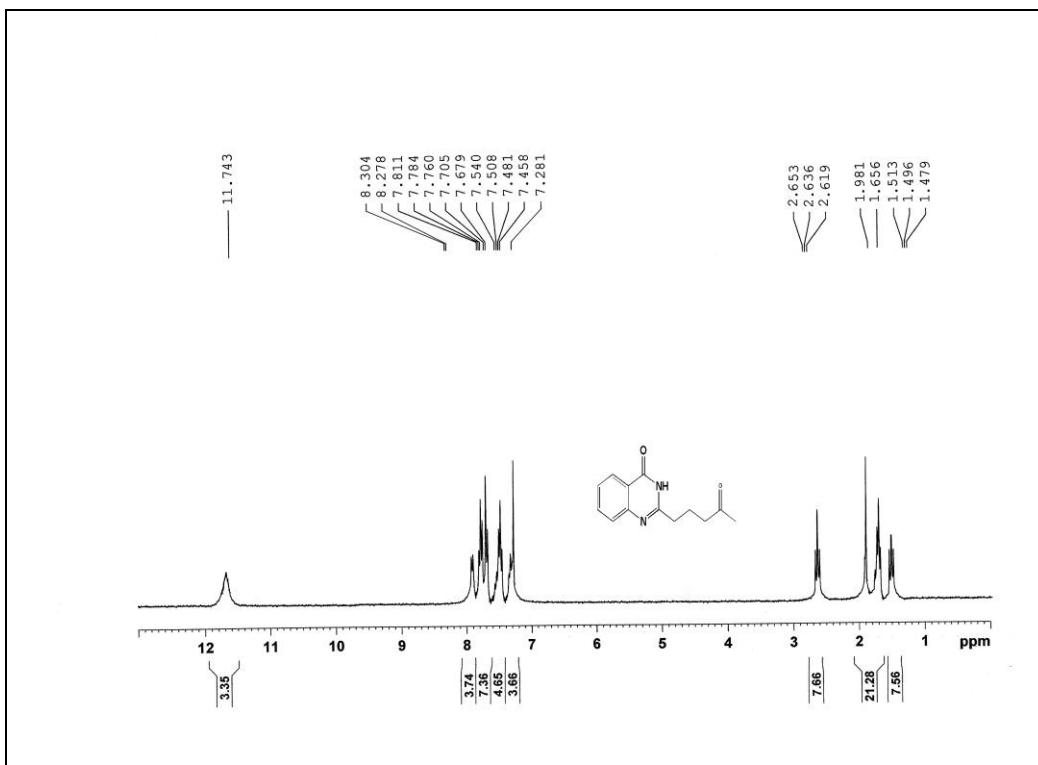
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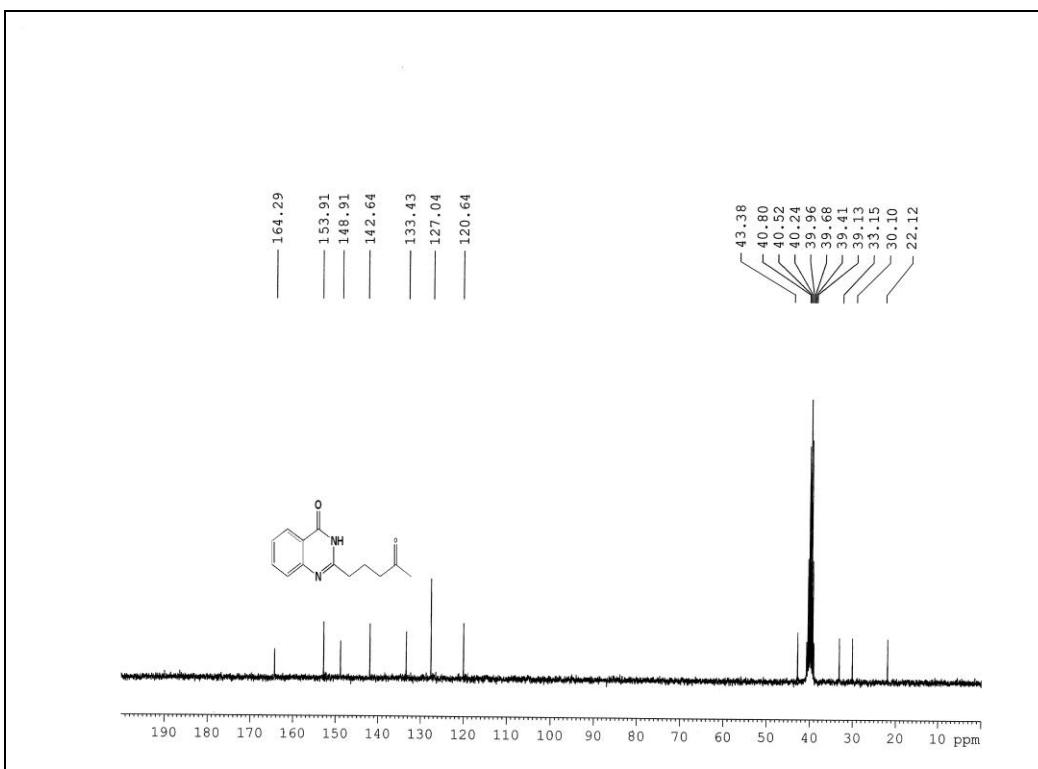
¹H NMR of Compound 7a



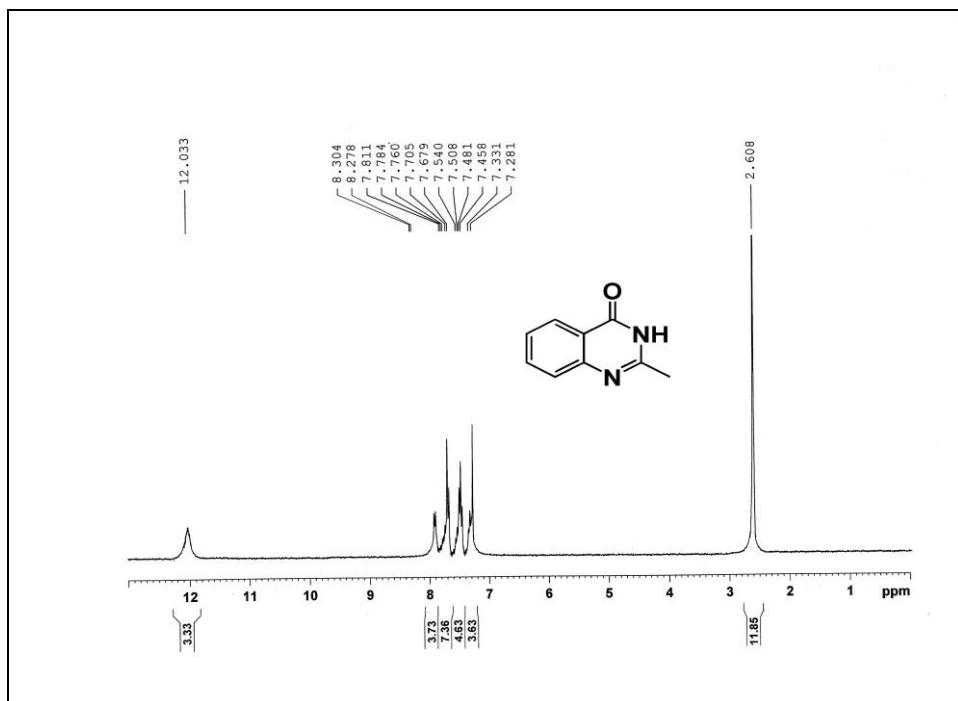
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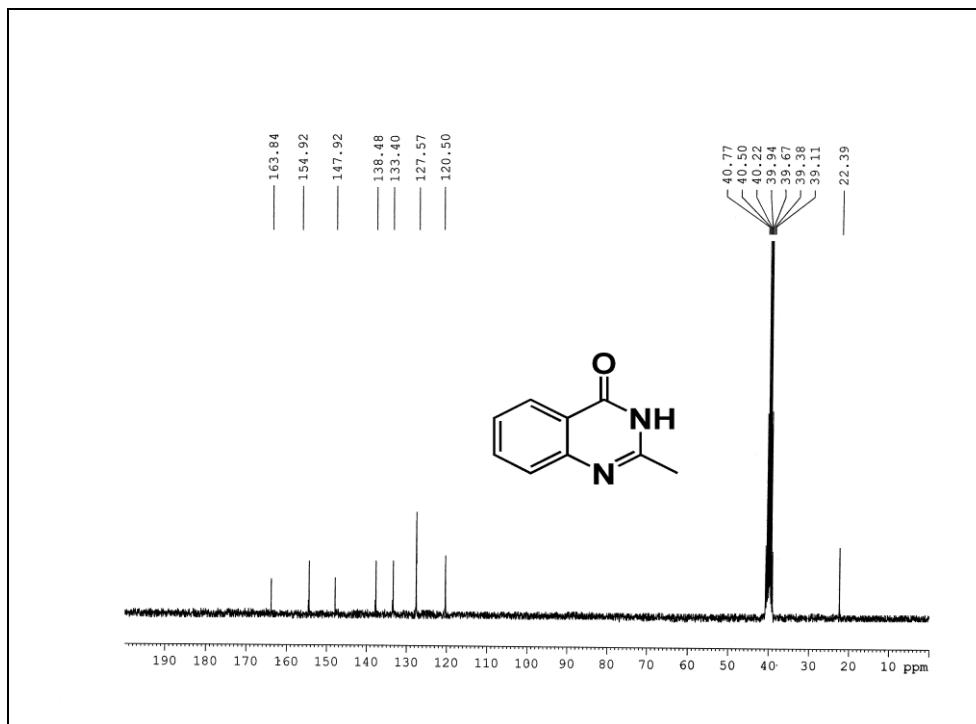
¹H NMR of Compound 7b



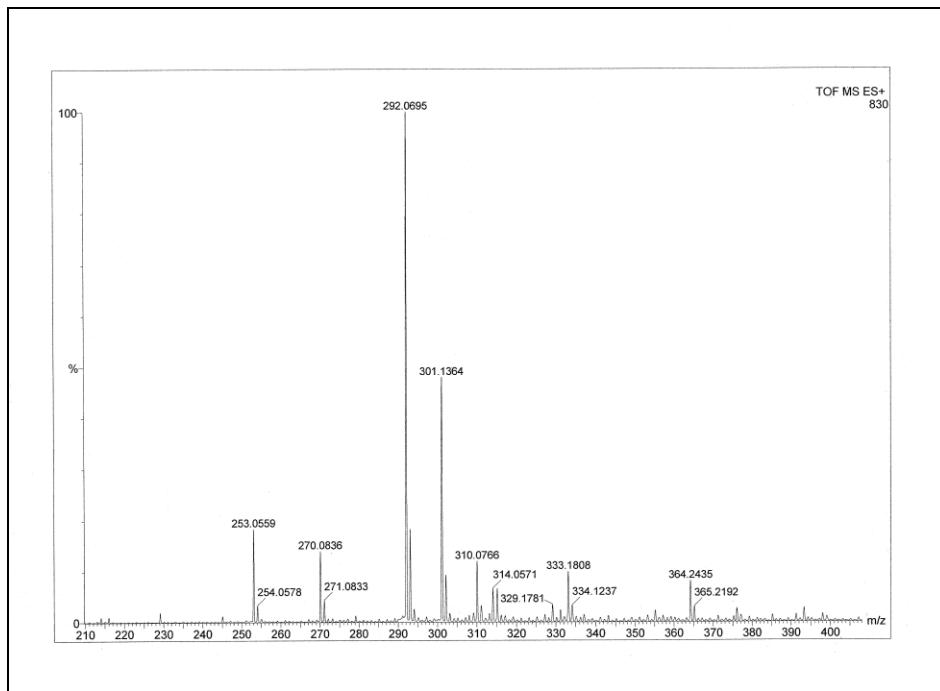
¹³C NMR of Compound 7b



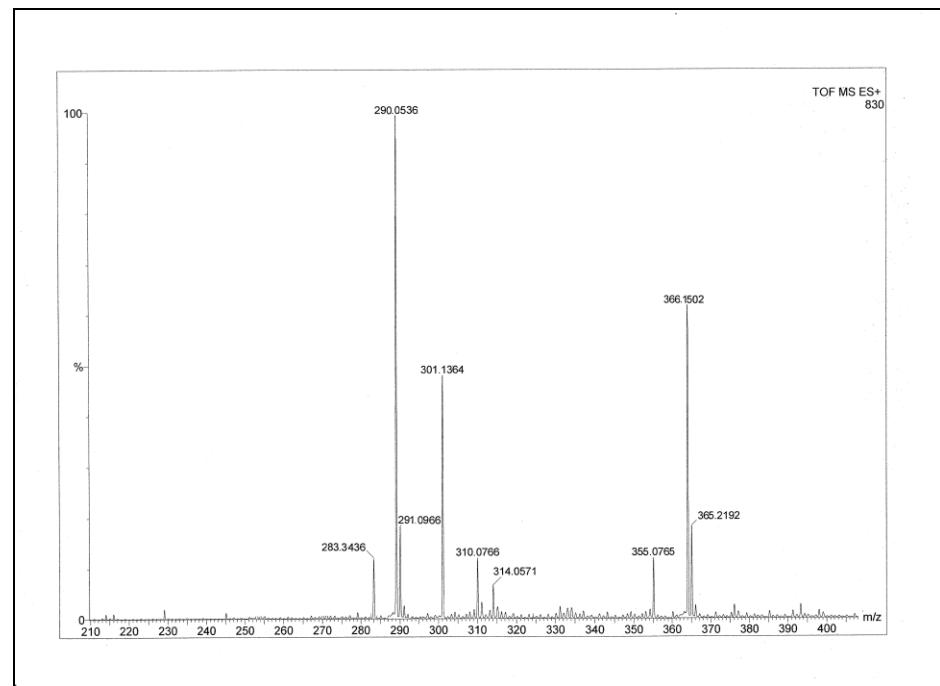
¹H NMR of Compound 8a/8b/8c



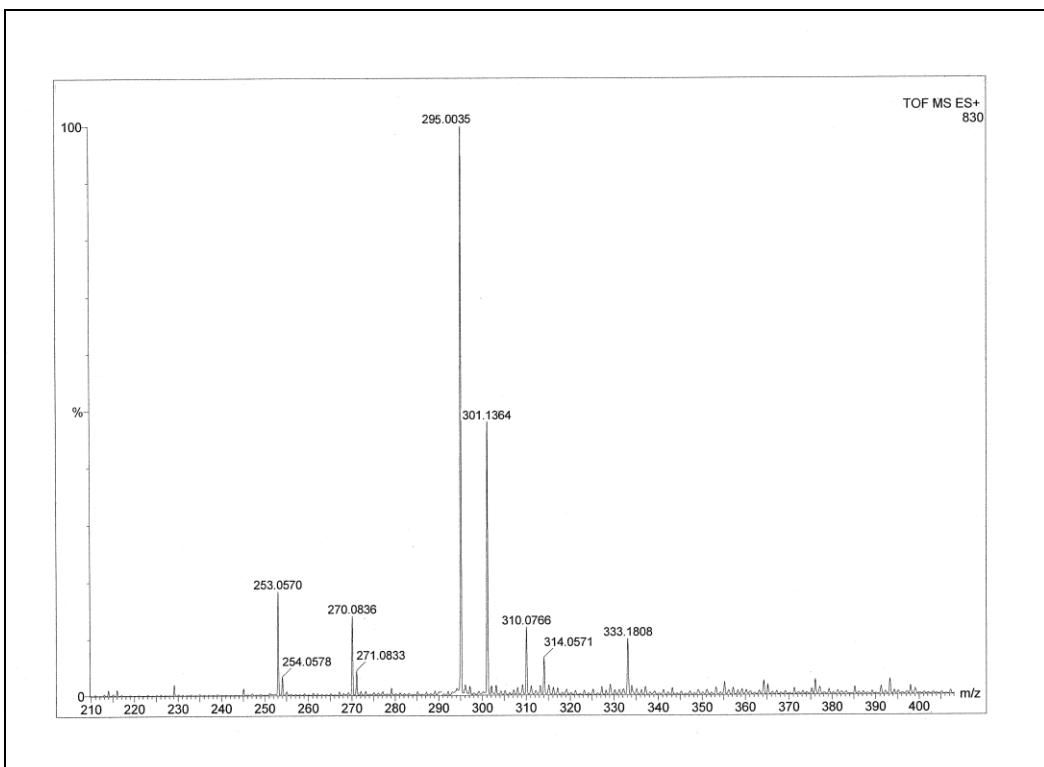
¹³C NMR of Compound 8a/8b/8c



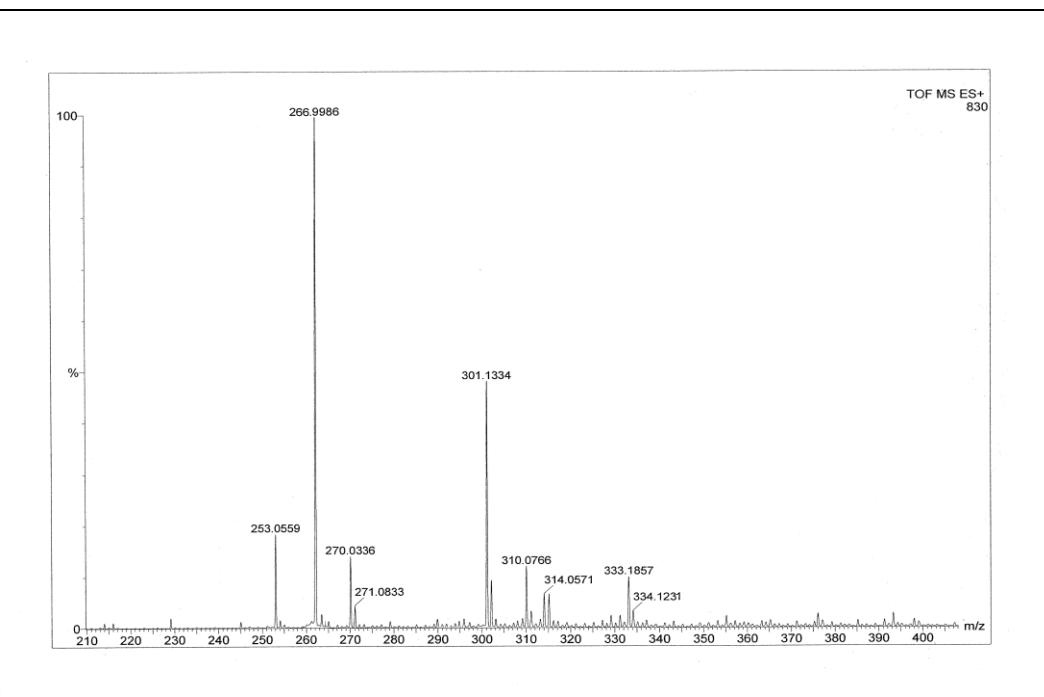
HRMS of Compound 3c



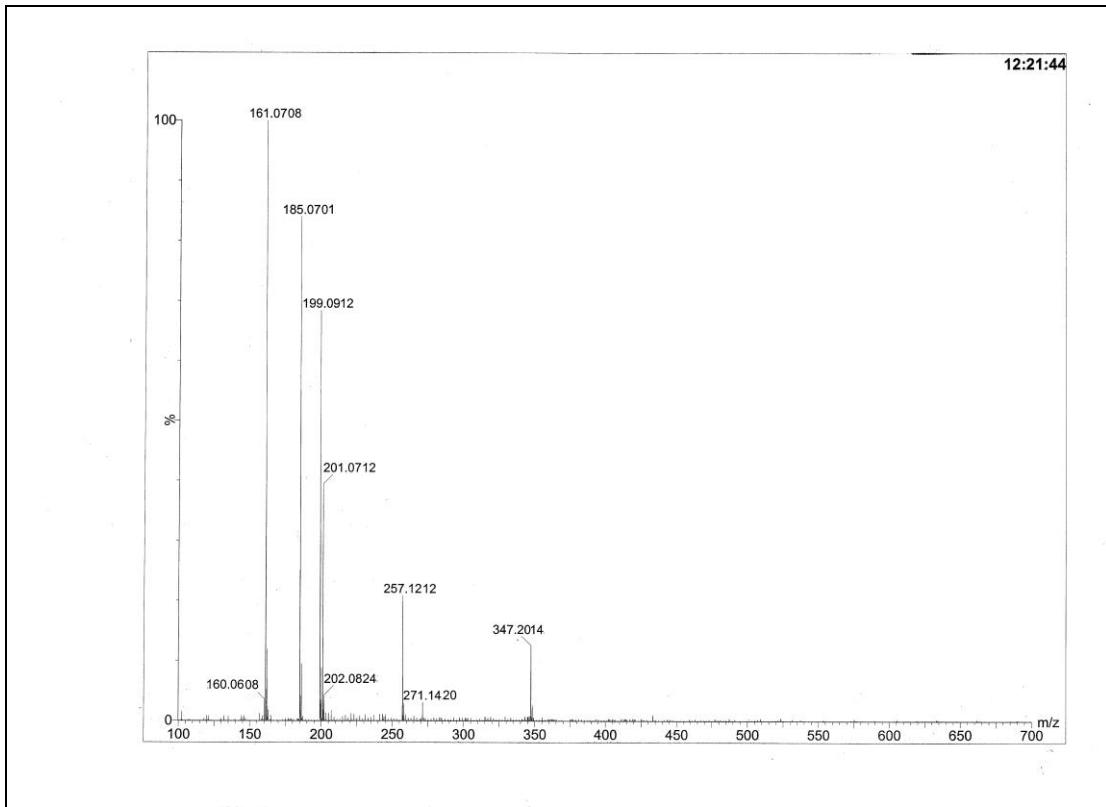
HRMS of Compound 6a



HRMS of Compound 6d



HRMS of Compound 6e



HRMS of compound 8a