

Supporting Information

β -Cyclodextrin: A supramolecular catalyst for metal-free approach towards the synthesis of 2-amino-4,6-diphenylnicotinonitriles and 2,3-dihydroquinazolin-4(1*H*)-one

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I. General Information

Melting point of the solid compounds was determined in concentrated H₂SO₄ bath. ¹H NMR, ¹³C NMR and ¹⁹F NMR were recorded using 300 MHz, 400 MHz and 75 MHz, 100 MHz and 376 MHz respectively on Bruker AV 300 NMR spectrometer and Bruker AV 400 NMR spectrometer using TMS as internal standard. IR spectra were recorded on KBr disc in the range 4000-400 cm⁻¹ on Shimadzu FT-IR 8300 Spectrometer. Splitting patterns of protons were described as s (singlet), d (doublet), t (triplet), br (broad) and m (multiplet).

General procedure for the synthesis of 2-amino-4,6-disubstituted nicotinonitriles

To a mixture of aromatic aldehydes (1 mmol), acetophenone (1 mmol), malononitrile (1 mmol) and ammonium acetate (1 mmol) in a round-bottom flask, β-Cyclodextrin (10 mol%) and water were added. The resulting mixture was stirred at 90 °C for 2h in open air. The reaction progress was monitored using TLC with a mixture of ethyl acetate and n-hexane as the eluent system. After completion of the reaction, the mixture was quenched to room temperature and was extracted with ethyl acetate twice (2×20 ml). Combined extracts were washed with distilled water, dried over anhydrous Na₂SO₄ and concentrated. The crude product was purified by passing through a column packed with silica gel. The products obtained were known compounds and identified by melting point, FT-IR and ¹H, ¹³C NMR spectroscopy.

General procedure for the synthesis of 2,3-dihydroquinazolin-4(1H)-ones

Aldehydes (1 mmol), isatoic anhydride (1 mmol) and ammonium acetate (1 mmol) were added in a round-bottom flask and then β-Cyclodextrin (10 mol%) was added to the mixture. The resulting mixture was stirred at 90 °C for 1h in open air. The reaction progress was monitored using TLC with a mixture of ethyl acetate and n-hexane as the eluent system. After completion of the reaction, the mixture was quenched to room temperature and was extracted with ethyl acetate twice (2×20 ml). Combined extracts were washed with distilled water, dried over anhydrous Na₂SO₄ and concentrated. The residue obtained was recrystallized using a mixture of petroleum ether and ethyl acetate and then filtered to afford the desired pure solid products. All the products were separated without column chromatography. The products obtained were known compounds and were identified by melting point, FT-IR and ¹H, ¹³C NMR spectroscopy.

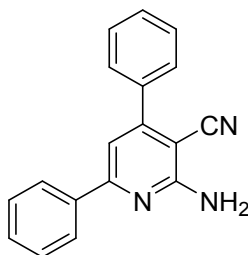
IV. Characterization data of synthesized 2-amino-4,6-diphenylnicotinonitriles and 2,3-dihydroquinazolin-4(1H)-one Derivatives

1.2-Amino-4,6-diphenylpyridine-3-carbonitrile¹

Pale yellow solid; M.P.: 210 °C;

¹H NMR (300 MHz, DMSO-*d*₆) δ (ppm): 5.48 (s, 2H), 6.98 (d, *J* = 7.8 Hz, 2H), 7.20 (s, 1H), 7.40-7.47 (m, 3H), 7.68-7.82 (m, 3H), 8.14-8.26 (m, 2H);

¹³C NMR (75 MHz, DMSO-*d*₆) δ (ppm): 88.4, 110.0, 114.2, 117.8, 127.8, 129.4, 129.9, 130.3, 130.5, 138.1, 155.6, 158.8, 160.7, 161.9.

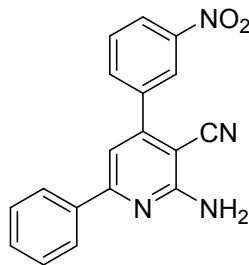


2.2-Amino-4-(3-nitrophenyl)-6-phenylnicotinonitrile²

Dark brown solid; M.P.: 251-253 °C;

¹H NMR (300 MHz, DMSO-*d*₆) δ (ppm): 6.28 (s, 2H), 7.35 (s, 1H), 7.45-8.20 (m, 7H), 8.15 (d, *J* = 7.2 Hz, 1H), 8.42 (s, 1H);

¹³C NMR (75 MHz, DMSO-*d*₆) δ (ppm): 85.8, 110.6, 116.9, 120.4, 128.6, 129.3, 130.6, 133.4, 136.7, 138.5, 148.0, 156.5, 158.9, 162.8.

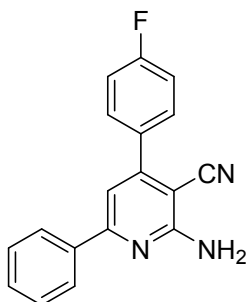


3.2-Amino-4-(4-fluorophenyl)-6-phenylnicotinonitrile³

Yellow solid; M.P.: 148-150 °C;

¹H NMR (300 MHz, DMSO-*d*₆) δ (ppm): 6.88 (s, 2H), 7.19 (s, 1H), 7.41 (t, *J* = 7.8 Hz, 2H), 7.46-7.52 (m, 3H), 7.69-7.72 (m, 2H), 8.16-8.20 (m, 2H);

¹³C NMR (75 MHz, DMSO-*d*₆) δ (ppm): 88.9, 109.2, 115.6, 117.9, 127.2, 128.6, 130.1, 130.9, 133.3, 138.7, 154.8, 163.8.

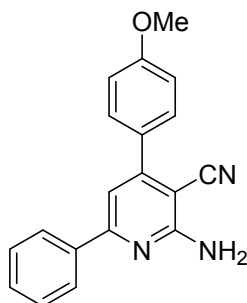


4.2-Amino-4-(4-methoxyphenyl)-6-phenylnicotinonitrile¹

Yellow solid; M.P.: 192-195 °C;

¹H NMR (300 MHz, DMSO-*d*₆) δ (ppm): 3.64 (s, 3H), 7.06 (d, *J* = 7.8 Hz, 2H), 7.20 (s, 1H), 7.42-7.68 (m, 3H), 7.65 (d, *J* = 7.8 Hz, 2H), 7.88 (d, *J* = 7.2 Hz, 2H);

¹³C NMR (75 MHz, DMSO-*d*₆) δ (ppm): 55.6, 86.9, 109.5, 114.6, 117.8, 127.7, 129.1, 129.5, 130.3, 130.5, 138.1, 154.9, 158.9, 160.8, 161.4.

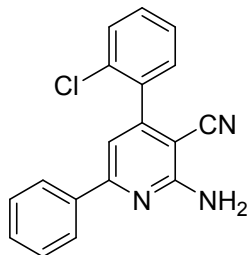


5.2-Amino-4-(2-chlorophenyl)-6-phenylnicotinonitrile²

White solid; M.P.: 194-197 °C;

¹H NMR (300 MHz, DMSO-*d*₆) δ (ppm): 6.12 (s, 2H), 7.18 (s, 1H), 7.45-7.58 (m, 5H), 7.66 (d, *J* = 7.2 Hz, 2H), 8.21-8.34 (m, 2H);

¹³C NMR (75 MHz, DMSO-*d*₆) δ (ppm): 88.4, 109.7, 116.1, 125.2, 127.5, 128.7, 129.7, 130.2, 130.9, 131.2, 136.6, 137.9, 153.0, 158.5, 160.1.

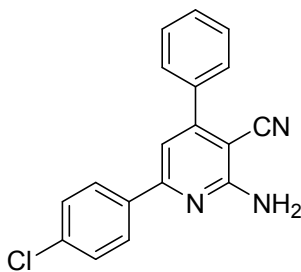


6.2-Amino-4-phenyl-6-(4-chlorophenyl)nicotinonitrile³

Yellow solid;

¹H NMR (300 MHz, DMSO-*d*₆) δ (ppm): 5.68(s, 2H), 7.16 (d, *J* = 8.4 Hz, 2H), 7.21 (s, 1H), 7.56-7.59 (m, 3H), 7.69 (d, *J* = 8.4 Hz, 3H), 8.24-8.29 (m, 2H);

¹³C NMR (75 MHz, DMSO-*d*₆) δ (ppm): 86.9, 111.5, 115.6, 117.8, 127.3, 128.6, 129.5, 130.3, 131.6, 138.1, 154.9, 158.9, 160.8, 161.4.

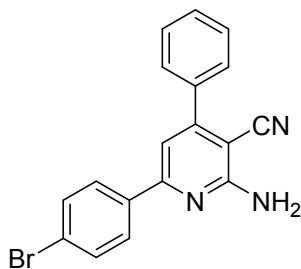


7. 2-Amino-4-phenyl-6-(4-bromophenyl)nicotinonitrile³

Yellow solid;

¹H NMR (300 MHz, DMSO-*d*₆) δ (ppm): 6.98 (s, 2H), 7.16 (s, 1H), 7.28 (d, *J* = 7.8 Hz, 2H), 7.60-7.65 (m, 3H), 7.79 (d, *J* = 7.8 Hz, 2H), 8.12-8.20 (m, 2H);

¹³C NMR (75 MHz, DMSO-*d*₆) δ (ppm): 87.2, 108.9, 113.9, 117.8, 127.3, 129.1, 129.5, 130.3, 130.8, 138.0, 153.9, 159.4, 161.0, 162.1.

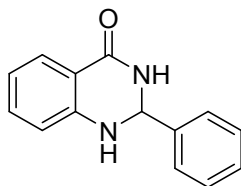


8. 2,3-Dihydro-2-phenylquinazolin-4(1H)-one⁴

White solid; M.P.: 224-226 °C;

¹H NMR (400 MHz, DMSO-*d*₆) δ (ppm): 5.73 (s, 1H), 6.65 (t, *J* = 7.6 Hz, 1H), 6.72 (d, *J* = 8 Hz, 1H), 7.09 (s, 1H), 7.20-7.24 (m, 1H), 7.30-7.39 (m, 3H), 7.47 (d, *J* = 8 Hz, 2H), 7.59 (d, *J* = 8 Hz, 1H), 8.27 (s, 1H);

¹³C NMR (100 MHz, DMSO-*d*₆) δ (ppm): 67.1, 114.9, 115.5, 117.6, 127.4, 127.9, 128.8, 129.0, 133.8, 142.1, 148.4, 164.1.



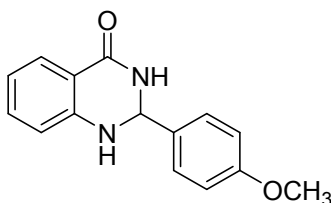
9. 2,3-Dihydro-2-(4-methoxyphenyl)quinazolin-4(1H)-one⁴

White solid; M.P.: 178-180 °C;

¹H NMR (400 MHz, DMSO-*d*₆) δ (ppm): 3.72 (s, 3H), 5.68 (s, 1H), 6.67 (t, *J* = 7.6 Hz, 1H), 6.71 (d, *J* = 8 Hz, 1H), 6.92 (d, *J* = 8.8 Hz, 2H), 6.98 (s, 1H), 7.21 (t, *J* = 8.4 Hz, 1H), 7.39 (d, *J* = 8.4 Hz, 2H), 7.58 (d, *J* = 8 Hz, 1H), 8.16 (s, 1H);

¹³C NMR (100 MHz, DMSO-*d*₆) δ (ppm): 55.4, 66.8, 114.1, 114.9, 115.5, 117.6, 127.8, 128.7, 133.7, 134.0, 148.5, 159.9, 164.2;

IR (KBr) cm⁻¹: 757, 1251, 1505, 1654, 2363, 2922, 3295, 3424.

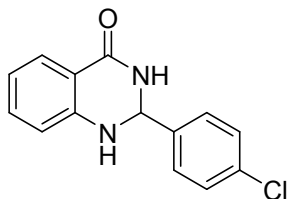


10. 2-(4-Chlorophenyl)-2,3-dihydroquinazolin-4(1H)-one⁴

White solid; M.P.: 201-203 °C;

¹H NMR (400 MHz, DMSO-*d*₆) δ (ppm): 5.90 (s, 1H), 6.67 (t, *J* = 7.6 Hz, 1H), 6.76 (d, *J* = 8 Hz, 1H), 7.24 (t, *J* = 7.6 Hz, 1H), 7.31 (s, 1H), 7.60 (d, *J* = 7.6 Hz, 1H), 7.73 (d, *J* = 8.8 Hz, 2H), 8.23 (d, *J* = 8.8 Hz, 2H), 8.51 (s, 1H);

¹³C NMR (100 MHz, DMSO-*d*₆) δ (ppm): 65.8, 115.4, 118.0, 124.1, 126.4, 127.9, 128.5, 129.8, 134.1, 147.7, 149.8, 163.8.



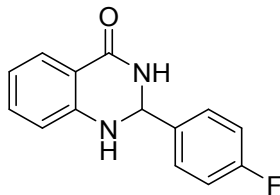
11. 2-(4-Fluorophenyl)-2,3-dihydroquinazolin-4(1H)-one⁵

White solid; M.P.: 202-204 °C;

¹H NMR (400 MHz, DMSO-*d*₆) δ (ppm): 5.73 (s, 1H), 6.67 (t, *J* = 7.6 Hz, 1H), 6.74 (d, *J* = 8 Hz, 1H), 7.09 (s, 1H), 7.18-7.25 (m, 2H), 7.51-7.54 (m, 2H), 7.61 (d, *J* = 7.6 Hz, 1H), 8.29 (s, 1H);

^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ (ppm): 66.5, 115.0, 115.6 (d, $J = 21.5$ Hz, 1C), 117.8, 127.9, 129.6 (d, $J = 8.3$ Hz, 1C), 133.9, 138.3 (d, $J = 2.2$ Hz, 1C), 148.3, 161.4, 163.8, 164.1;

^{19}F NMR (376 MHz, $\text{DMSO-}d_6$) δ (ppm): -113.76 (s, 1F).

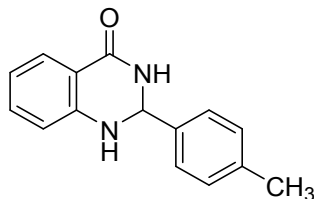


12. 2,3-Dihydro-2-*p*-tolylquinazolin-4(1*H*)-one⁴

White crystalline solid; M.P.: 232-233 °C;

^1H NMR (300 MHz, $\text{DMSO-}d_6$) δ (ppm): 2.39 (s, 3H), 5.78 (s, 1H), 5.87 (s, 1H), 6.67 (d, $J = 8.1$ Hz, 1H), 6.90 (t, $J = 7.5$ Hz, 1H), 7.26-7.42 (m, 5H), 7.95 (d, $J = 7.8$ Hz, 1H);

^{13}C NMR (75 MHz, $\text{DMSO-}d_6$) δ (ppm): 21.4, 67.1, 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.

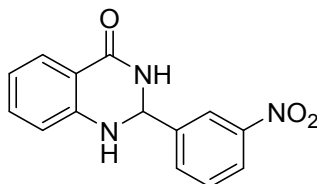


13. 2,3-Dihydro-2-(3-nitrophenyl)quinazolin-4(1*H*)-one⁴

Yellow solid; M.P.: 192-194 °C;

^1H NMR (300 MHz, $\text{DMSO-}d_6$) δ (ppm): 5.99 (s, 1H), 6.70 (d, $J = 6.6$ Hz, 1H), 6.82 (d, $J = 7.5$ Hz, 1H), 7.28 (d, $J = 5.4$ Hz, 2H), 7.66 (t, $J = 6.6$ Hz, 2H), 7.96 (d, $J = 6.6$ Hz, 1H), 8.19 (s, 1H), 8.40 (s, 1H), 8.59 (s, 1H);

^{13}C NMR (75 MHz, $\text{DMSO-}d_6$) δ (ppm): 65.7, 115.1, 115.3, 118.0, 122.0, 123.7, 127.9, 130.4, 133.8, 134.1, 144.6, 147.6, 148.1, 164.0.

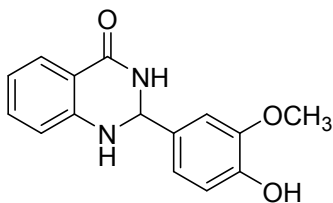


14. 2,3-Dihydro-2-(4-hydroxy-3-methoxyphenyl)quinazolin-4(1H)-one⁵

White solid; M.P.: 188-190 °C;

¹H NMR (300 MHz, DMSO-*d*₆) δ (ppm): 4.00 (s, 3H), 4.29 (s, 1H), 6.20 (s, 1H), 7.21-7.32 (m, 4H), 7.63 (s, 1H), 7.78 (d, *J* = 6.9 Hz, 1H), 8.16 (d, *J* = 7.2 Hz, 1H), 8.67 (s, 1H), 9.64 (s, 1H);

¹³C NMR (75 MHz, DMSO-*d*₆) δ (ppm): 56.5, 67.8, 112.0, 115.4, 115.9, 115.9, 118.1, 120.6, 128.3, 132.9, 143.2, 147.8, 148.4, 149.1, 164.8.

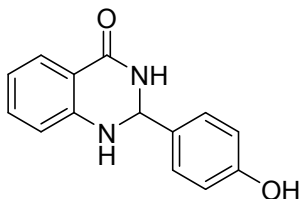


15. 2,3-dihydro-2-(4-hydroxyphenyl)quinazolin-4(1H)-one⁴

White crystalline solid; M.P.: 210-212 °C;

¹H NMR (300 MHz, CDCl₃) δ (ppm): 4.43 (s, 1H), 5.78 (s, 1H), 5.86 (s, 1H), 6.67 (d, *J* = 8.1 Hz, 1H), 6.90 (t, *J* = 7.5 Hz, 1H), 7.26-7.42 (m, 5H), 7.95 (d, *J* = 7.8 Hz, 1H), 8.33 (s, 1H);

¹³C NMR (75 MHz, DMSO-*d*₆) δ (ppm): 66.8, 114.0, 114.8, 115.5, 117.5, 127.7, 128.6, 133.7, 133.9, 148.4 159.8, 164.1.

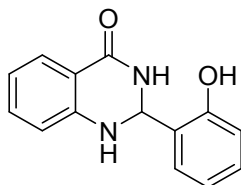


16. 2,3-Dihydro-2-(2-hydroxyphenyl)quinazolin-4(1H)-one⁴

White solid; M.P.: 220-221 °C;

¹H NMR (300 MHz, CDCl₃) δ (ppm): 4.61 (s, 1H), 5.92 (s, 1H), 5.98 (s, 1H), 6.54-6.72 (m, 4H), 6.77 (d, *J* = 7.8 Hz, 1H), 7.02 (d, *J* = 7.5 Hz, 1H), 7.11-7.33 (m, 2H), 8.91 (s, 1H);

¹³C NMR (75 MHz, DMSO-*d*₆) δ (ppm): 69.9, 128.4, 128.5, 128.6, 129.0, 132.1, 132.7, 133.9, 135.8, 140.5, 156.7, 166.6.

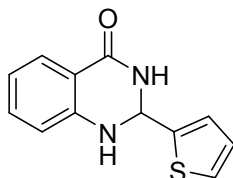


17. 2,3-Dihydro-2-(thiophen-2-yl)quinazolin-4(1H)-one⁴

White solid; M.P.: 213-215 °C;

¹H NMR (300 MHz, DMSO-*d*₆) δ (ppm): 6.01 (s, 1H), 6.69 (t, *J* = 7.6 Hz, 1H), 6.75 (d, *J* = 8 Hz, 1H), 6.96 (dd, *J* = 5.2 Hz & 3.6 Hz, 1H), 7.11 (s, 1H), 7.22-7.29 (m, 2H), 7.43 (dd, *J* = 4.8 Hz & 0.8 Hz, 1H), 7.61 (d, *J* = 7.6 Hz, 1H), 8.44 (s, 1H);

¹³C NMR (75 MHz, DMSO-*d*₆) δ (ppm): 63.1, 115.2, 115.6, 118.0, 126.2, 126.4, 127.0, 127.8, 133.9, 146.9, 147.7, 163.6.

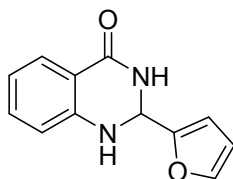


18. 2-(Furan-2-yl)-2,3-dihydroquinazolin-4(1H)-one⁵

White crystalline solid; M.P.: 166-167 °C;

¹H NMR (300 MHz, DMSO-*d*₆) δ (ppm): 5.76 (s, 1H), 6.72 (s, 1H), 7.25-7.49 (m, 3H), 7.61 (s, 1H), 7.73 (d, *J* = 6.9 Hz, 2H), 8.24 (d, *J* = 6.9 Hz, 2H);

¹³C NMR (75 MHz, DMSO-*d*₆) δ (ppm): 60.0, 112.9, 113.9, 121.0, 126.5, 126.7, 127.6, 134.9, 143.4, 145.4, 146.2, 149.2.

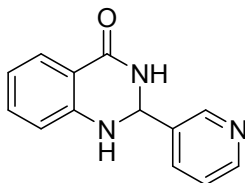


19. 2,3-Dihydro-2-(pyridin-3-yl)quinazolin-4(1H)-one⁵

Pale yellow solid; M.P.: 219-221 °C;

¹H NMR (400 MHz, DMSO-*d*₆) δ (ppm): 5.85 (s, 1H), 7.69 (t, *J* = 7.6 Hz, 1H), 6.75 (d, *J* = 8 Hz, 1H), 7.17 (s, 1H), 7.25 (t, *J* = 7.2 Hz, 1H), 7.40 (dd, *J* = 7.6 Hz & 4.8 Hz, 1H), 7.62 (d, *J* = 7.2 Hz, 1H), 7.88 (d, *J* = 8 Hz, 1H), 8.39 (s, 1H), 8.53 (d, *J* = 4 Hz, 1H), 8.66 (s, 1H);

¹³C NMR (100 MHz, DMSO-*d*₆) δ (ppm): 65.2, 115.1, 115.5, 118.0, 124.1, 127.9, 134.0, 135.2, 137.3, 148.2, 148.9, 150.2, 164.1.



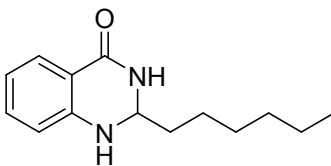
20. 2-Hexyl-2,3-dihydroquinazolin-4(1H)-one⁵

White solid; M.P.: 156-158 °C;

¹H NMR (300 MHz, DMSO-*d*₆) δ (ppm): 0.85 (d, 3H), 1.26-1.60 (m, 10H), 1.60 (q, 2H), 6.54 (s, 1H), 6.69 (q, *J* = 8.4 Hz, 2H), 7.57 (d, *J* = 7.5 Hz, 1H), 7.22 (t, 1H), 7.85(s, 1H);

¹³C NMR (75 MHz, DMSO-*d*₆) δ (ppm): 14.3, 22.4, 23.6, 29.0, 31.6, 35.5, 64.9, 114.8, 115.4, 117.3, 127.8, 133.4, 148.9, 164.3;

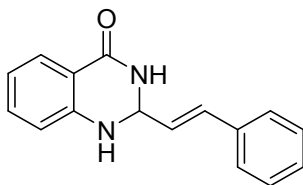
IR (KBr) cm⁻¹: 751, 1502, 1642, 2361, 2856, 2926, 3210, 3324.



21. 2,3-Dihydro-2-styrylquinazolin-4(1H)-one⁵

White solid; M.P.: 224-226 °C;

¹³C NMR (75 MHz, DMSO-*d*₆) δ (ppm): 66.2, 115.0, 115.3, 117.6, 118.1, 127.1, 127.8, 128.5, 128.8, 129.1, 132.0, 133.6, 136.2, 148.2, 163.8.

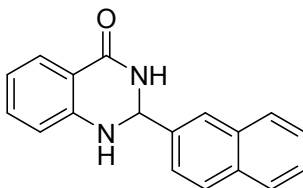


22. 2,3-Dihydro-2-(naphthalen-3-yl)quinazolin-4(1H)-one⁵

White solid; M.P.: 225-227 °C;

¹H NMR (300 MHz, DMSO-*d*₆) δ (ppm): 5.95 (s, 1H), 6.61 (s, 1H), 6.85 (t, *J* = 7.5 Hz, 1H), 6.78 (d, *J* = 8.1 Hz, 1H), 7.26 (q, *J* = 7.5 Hz, 2H), 7.53 (q, *J* = 6.3 Hz, 2H), 7.68 (q, *J* = 8.4 Hz, 2H), 7.91-7.97 (m, 4H), 8.38 (s, 1H);

¹³C NMR (75 MHz, DMSO-*d*₆) δ (ppm): 67.3, 114.9, 115.4, 117.6, 125.2, 126.3, 126.8, 127.8, 128.0, 128.4, 128.5, 132.9, 133.4, 133.8, 139.3, 148.3, 164.1.

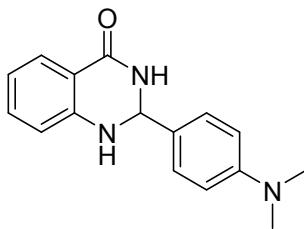


23. 2-(4-(Dimethylamino)phenyl)-2,3-dihydroquinazolin-4(1H)-one

White solid; M.P.: 224-227 °C;

^1H NMR (300 MHz, $\text{DMSO-}d_6$) δ (ppm): 2.86 (s, 6H), 5.62 (s, 1H), 6.64 (t, $J = 7.6$ Hz, 1H), 6.78 (t, $J = 7.6$ Hz, 3H), 6.90 (s, 1H), 7.21 (t, $J = 7.5$ Hz, 1H), 7.28 (d, $J = 8.8$ Hz, 2H), 7.59 (d, $J = 7.6$ Hz, 1H), 8.06 (s, 1H);

^{13}C NMR (75 MHz, $\text{DMSO-}d_6$) δ (ppm): 67.2, 112.4, 114.9, 115.6, 117.4, 127.8, 128.2, 129.1, 133.6, 148.7, 151.2, 164.3.

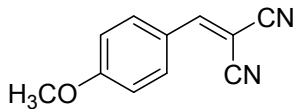


24. 2-(4-Methoxybenzylidene)malononitrile

Yellow solid; M.P.: 110-112 °C;

^1H NMR (400 MHz, CDCl_3) δ (ppm): 3.9 (s, 3H), 7.00 (d, $J = 8.8$ Hz, 2H), 7.64 (s, 1H), 7.90 (d, $J = 8.8$ Hz, 2H);

^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 55.9, 78.5, 113.4, 114.5, 115.2, 124.1, 133.5, 159.0, 164.9.

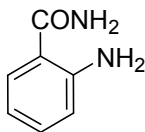


25. 2-Aminobenzamide

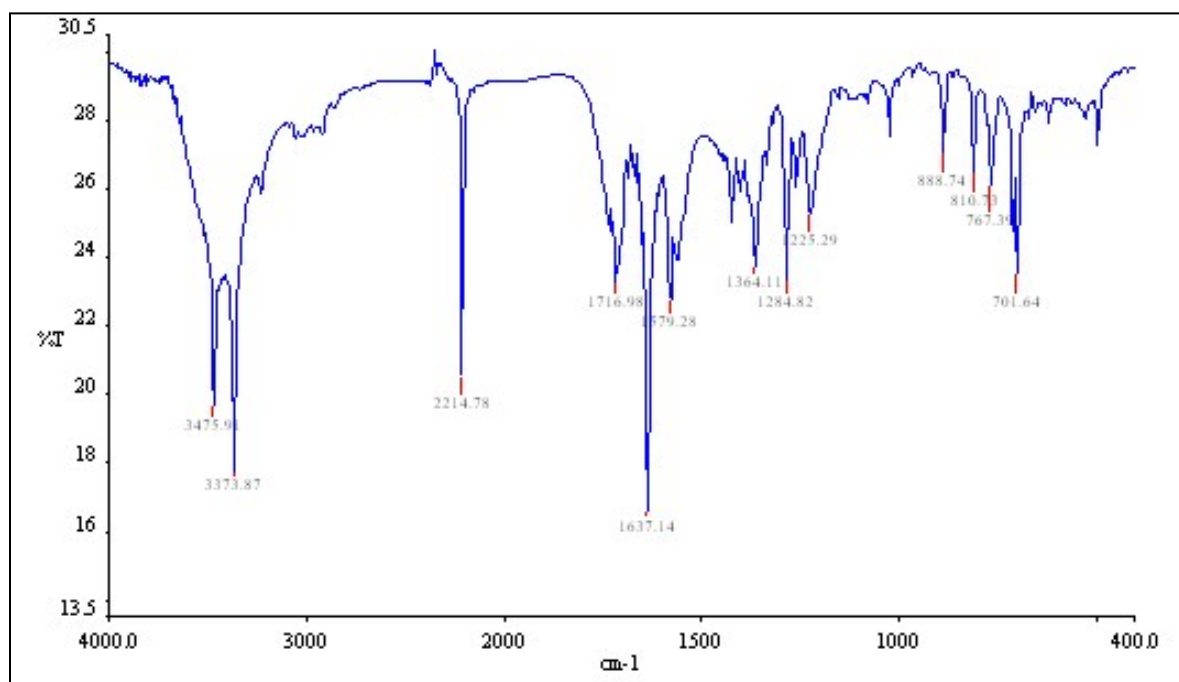
Pale white; M.P.: 108-110 °C;

^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ (ppm): 7.74 (s, br, 1H), 7.54-7.52 (m, 1H), 7.14-7.11 (m, 1H), 7.07 (brs, 1H), 6.68 (d, $J = 8.1$ Hz, 1H), 6.55 (brs, 2H), 6.48 (t, $J = 7.5$ Hz, 1H);

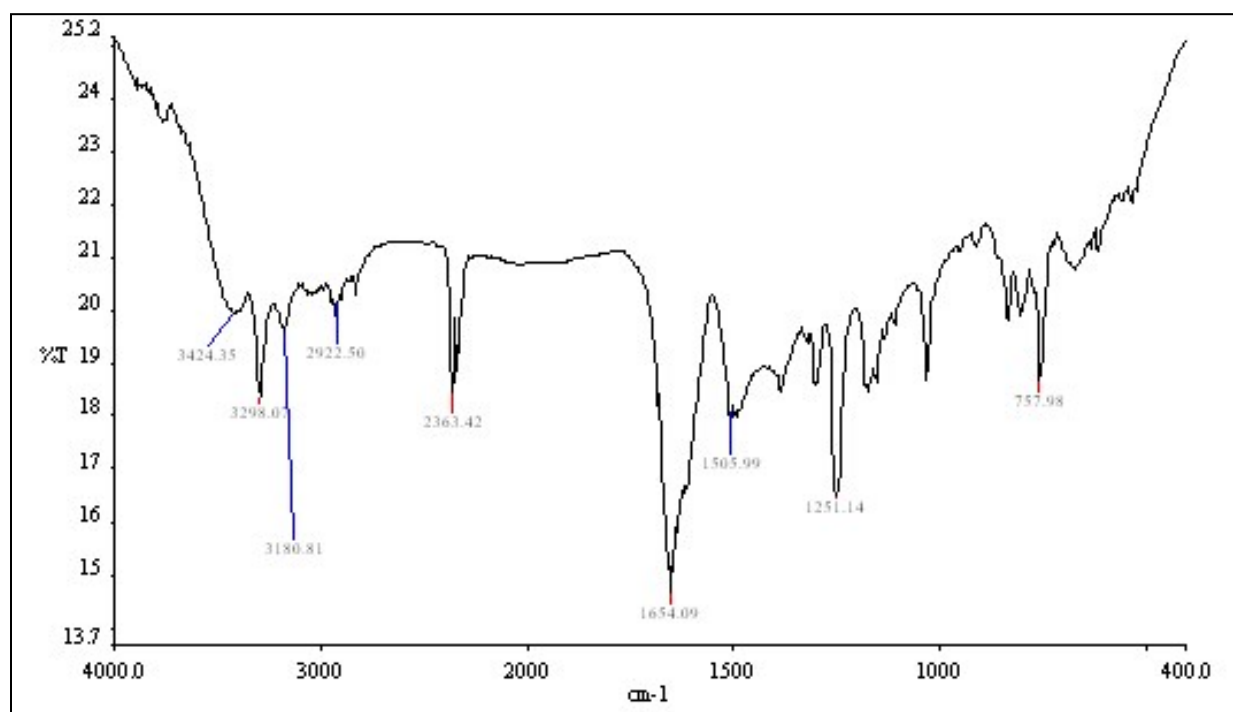
^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ (ppm): 114.2, 115.0, 117.0, 129.3, 132.5, 150.7, 171.9.



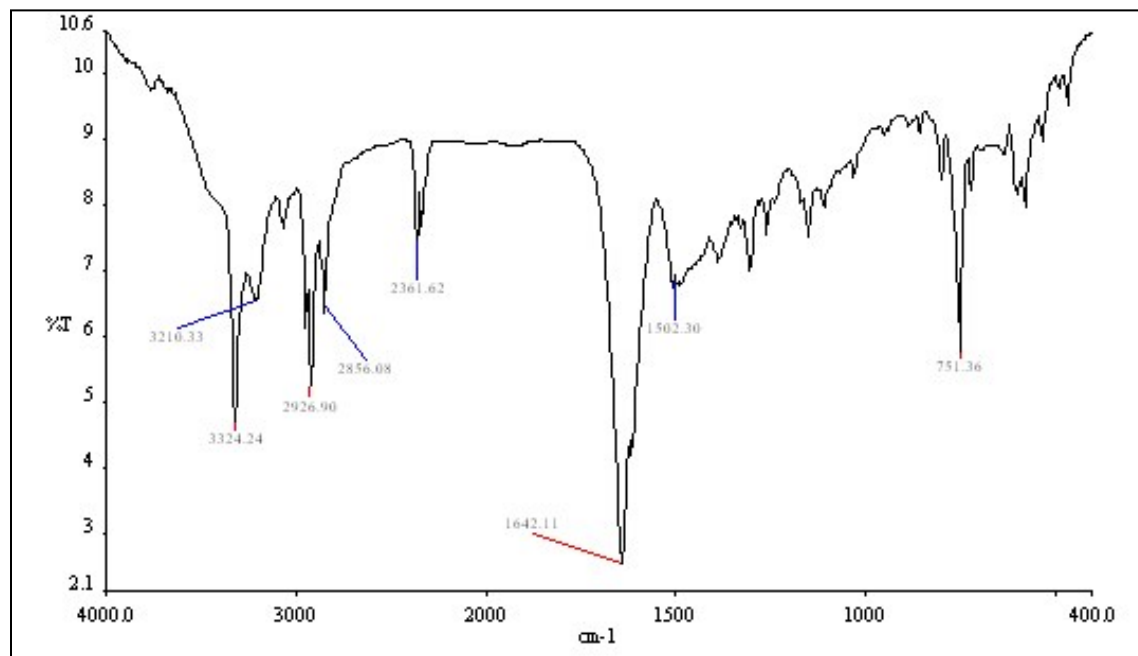
V. FT-IR spectra of 2-amino-4,6-disubstituted nicotinitriles



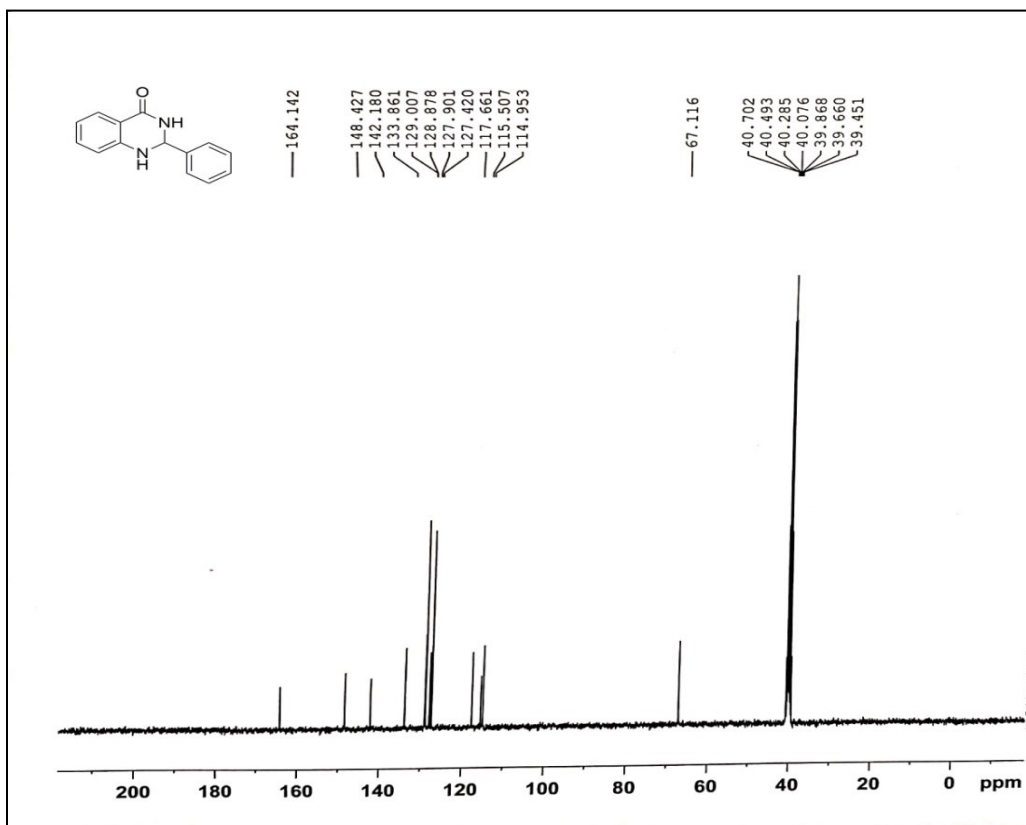
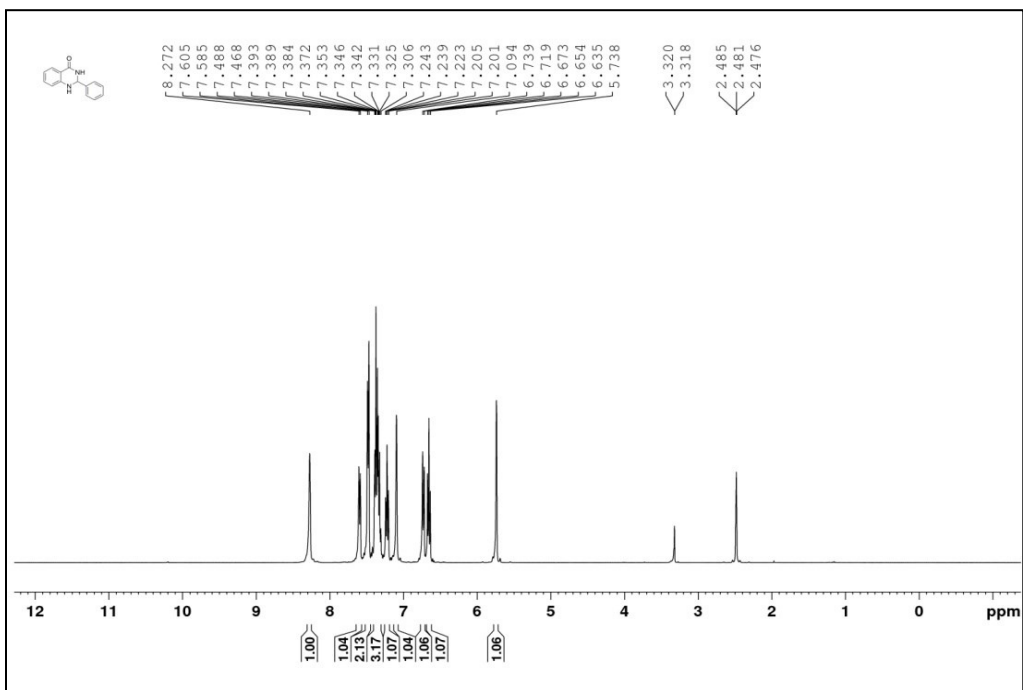
VI. FT-IR spectra of 2,3-dihydro-2-(4-methoxyphenyl)quinazolin-4(1H)-one

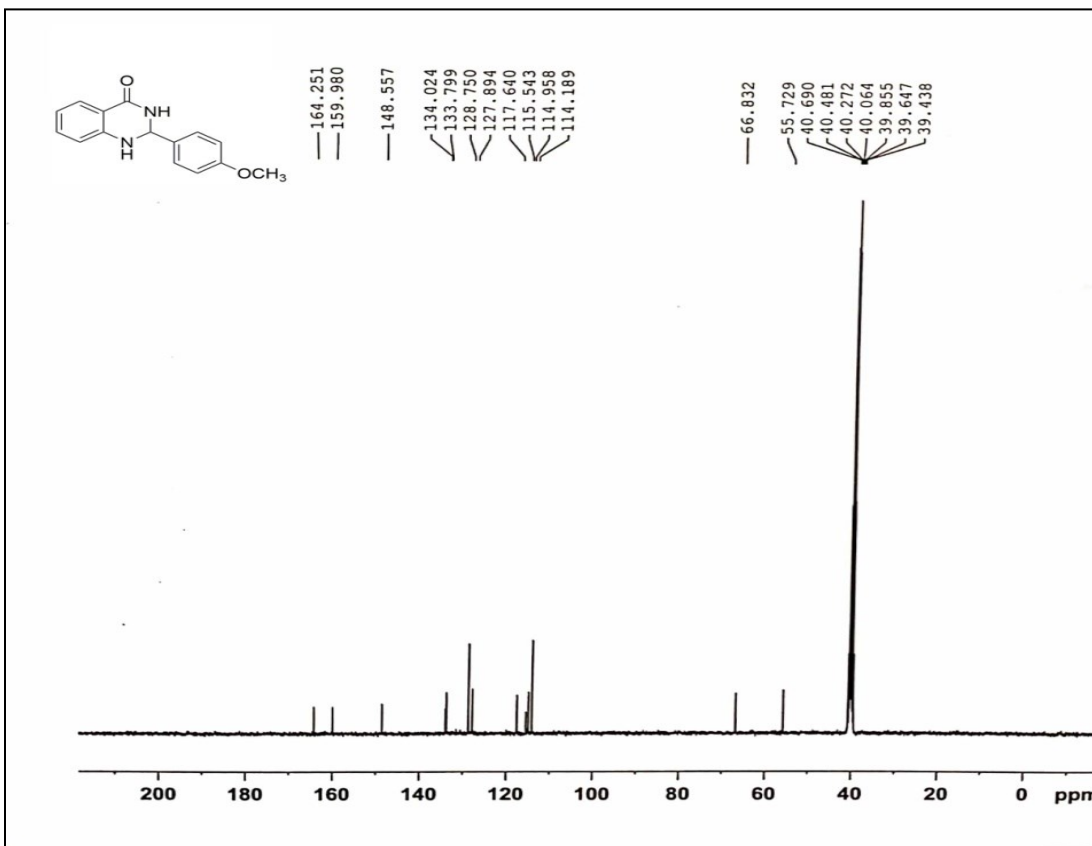
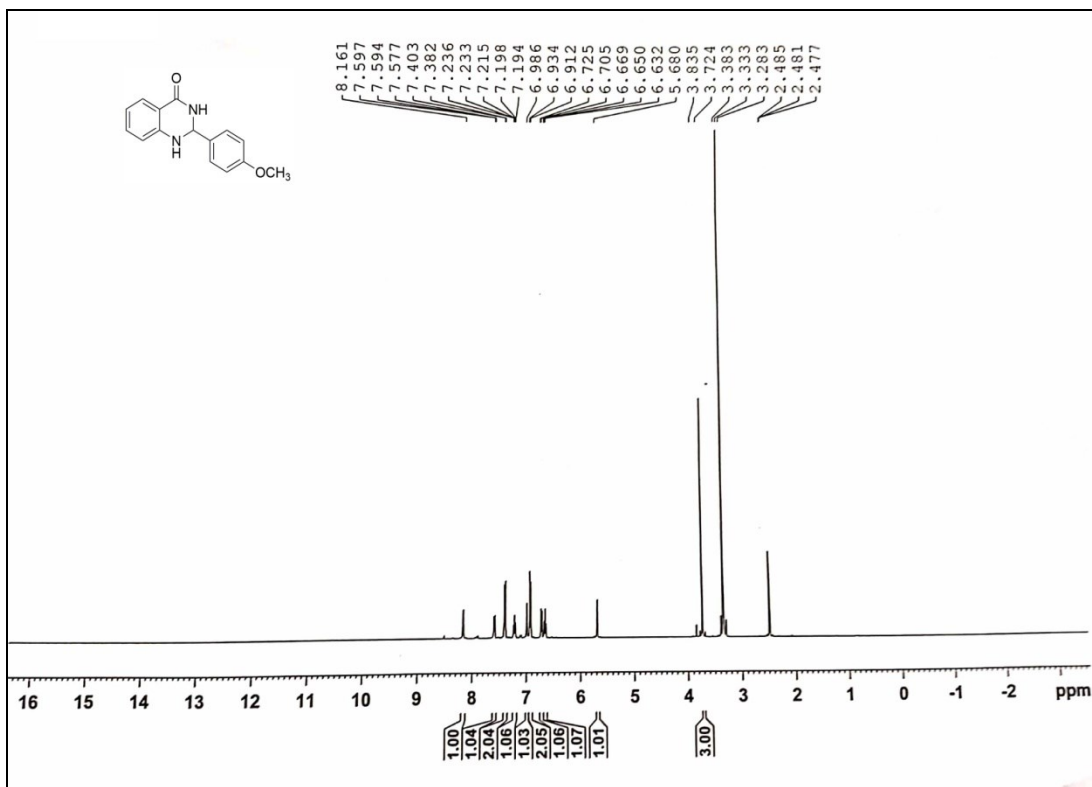


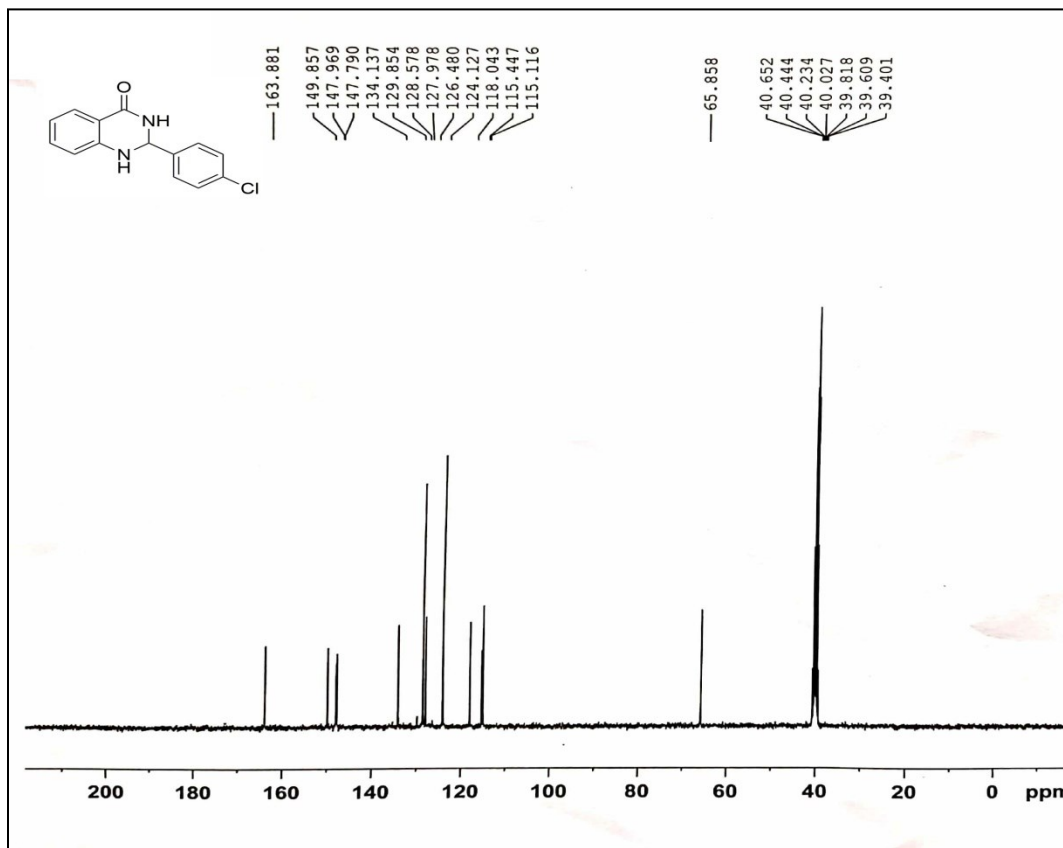
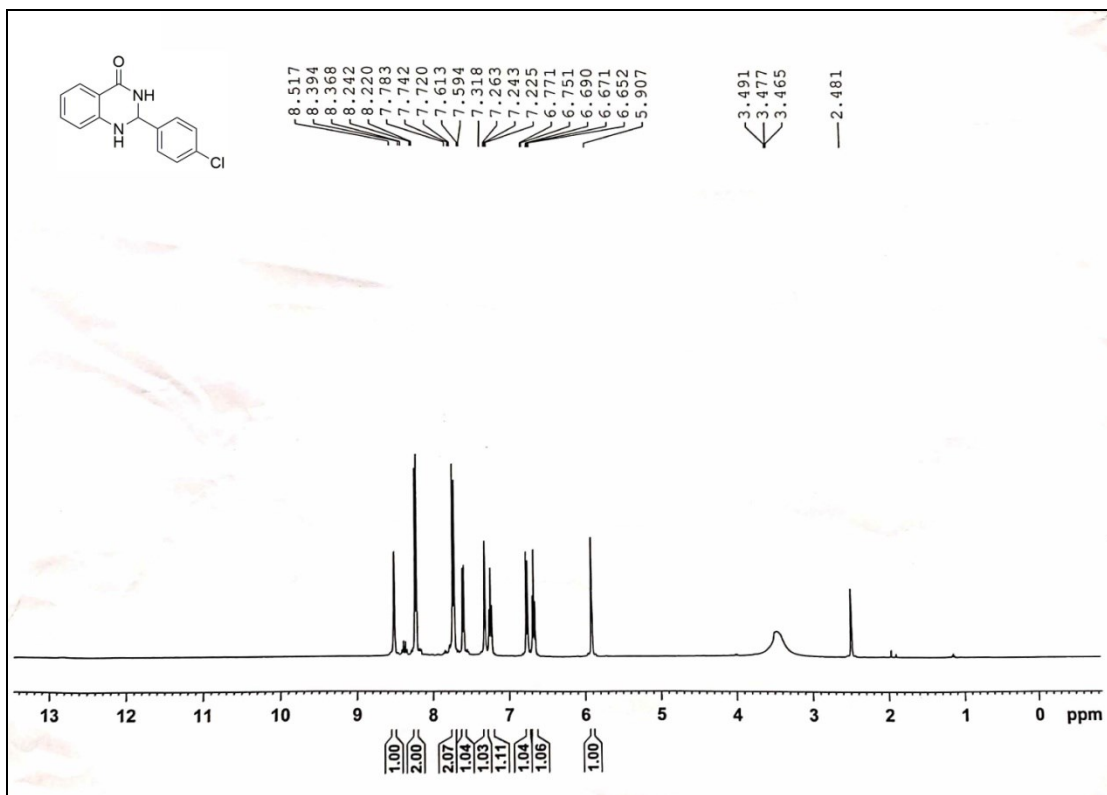
VII. FT-IR spectra of 2-hexyl-2,3-dihydroquinazolin-4(1H)-one

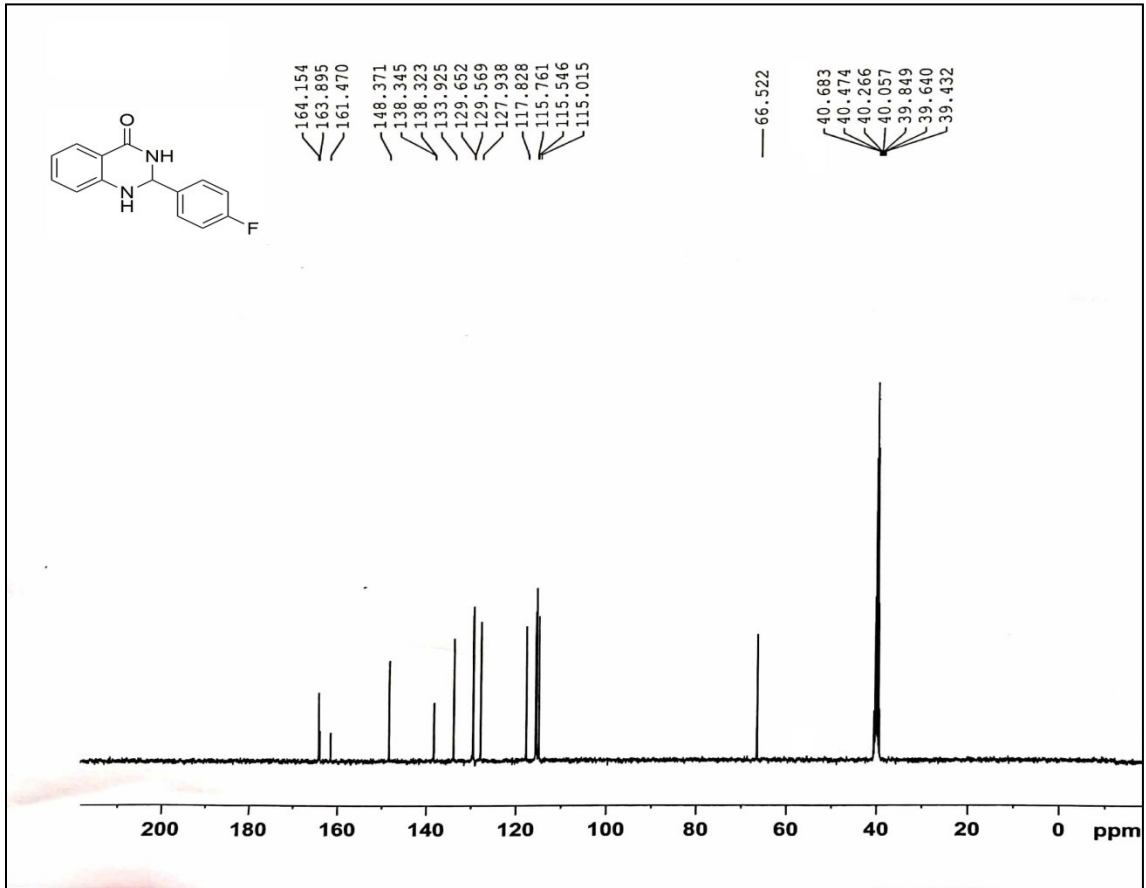
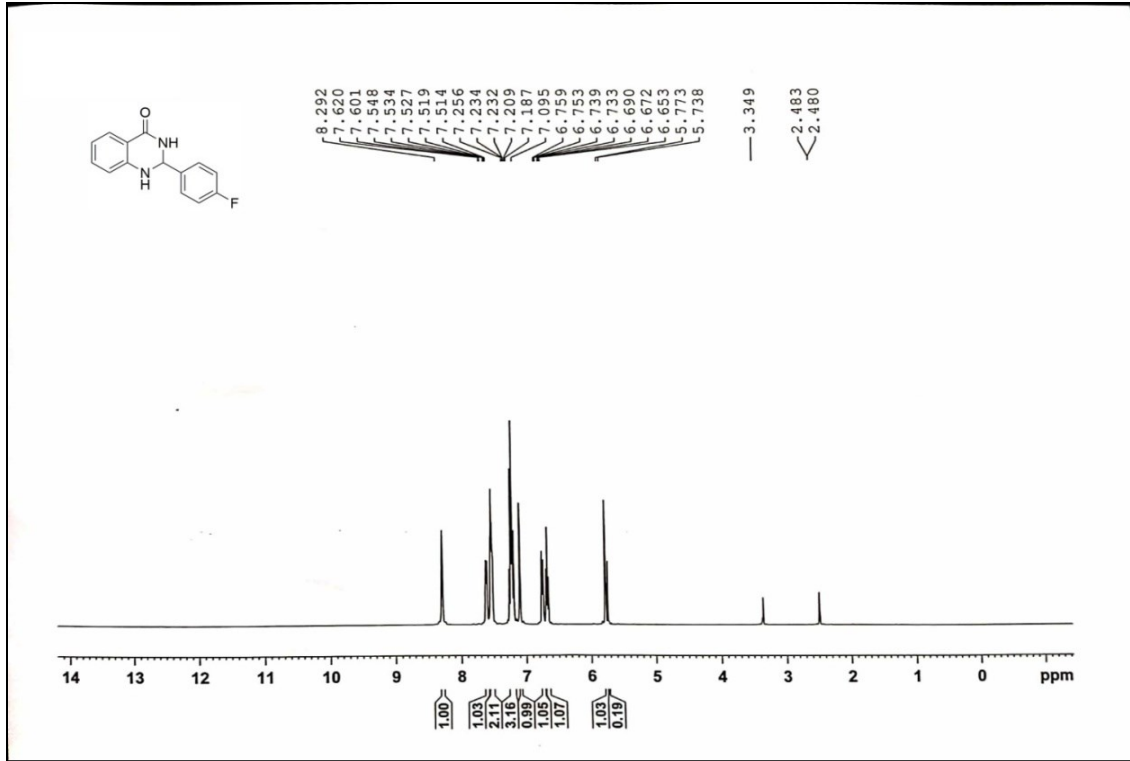


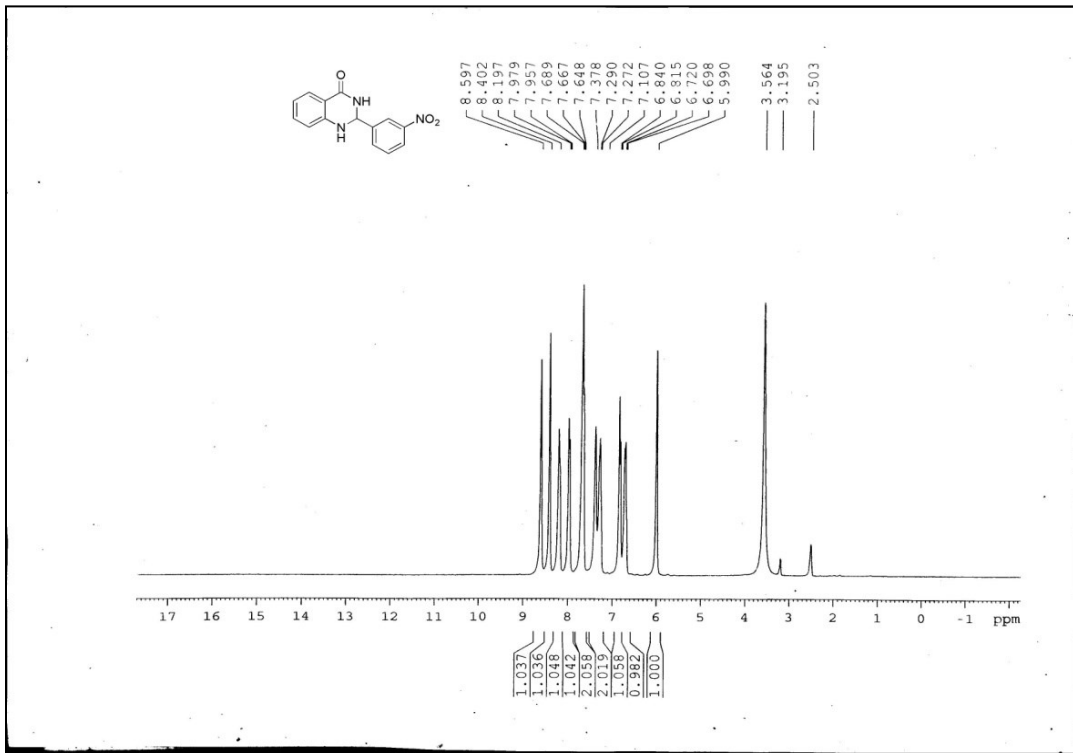
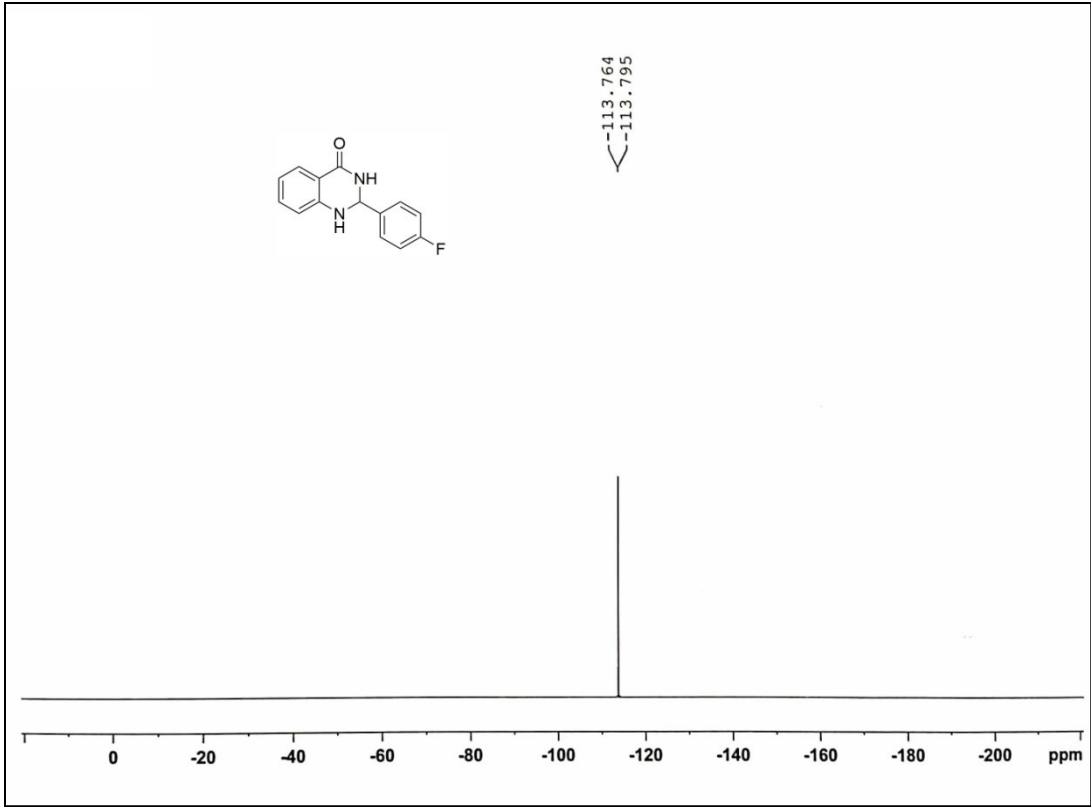
VIII. Scanned copies of ^{13}C , ^1H and ^{19}F NMR spectra of derivatives

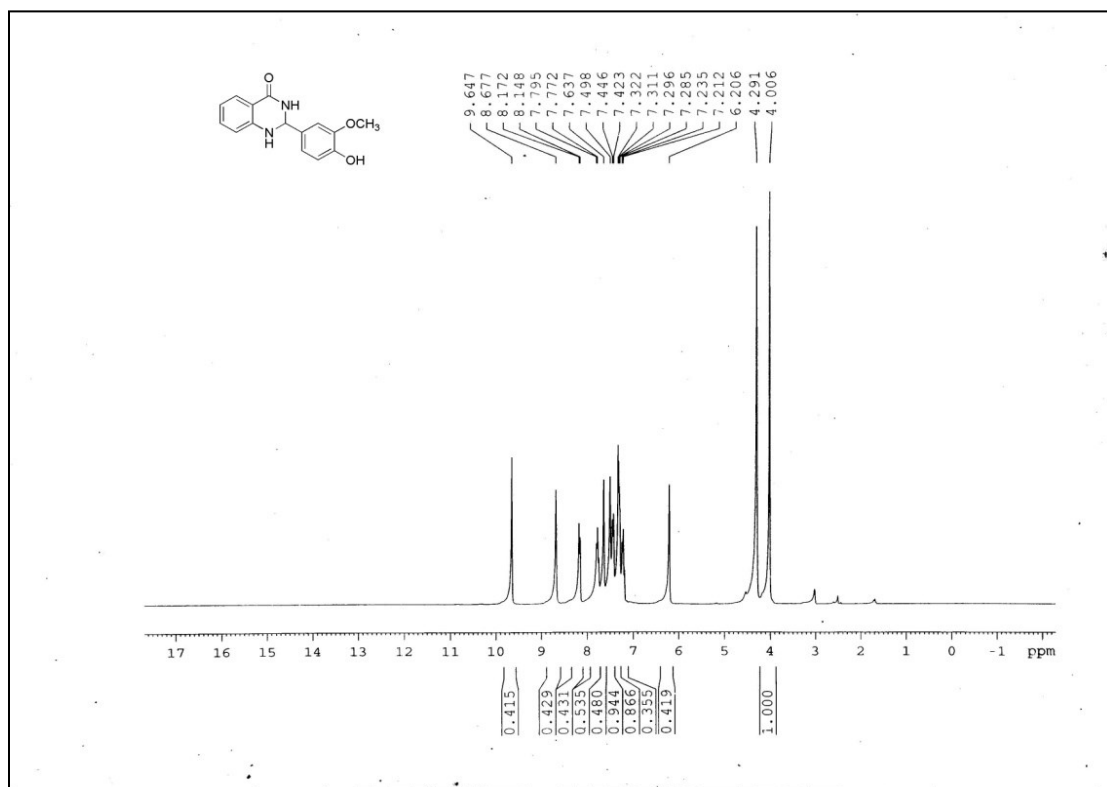
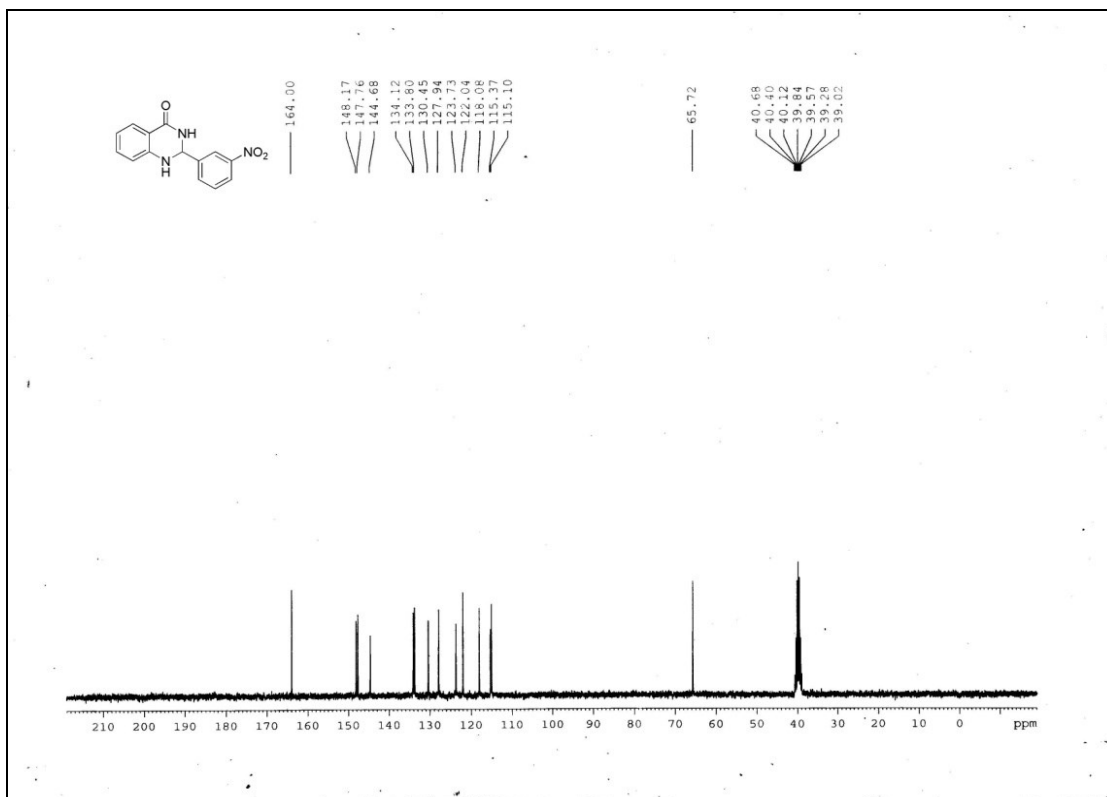


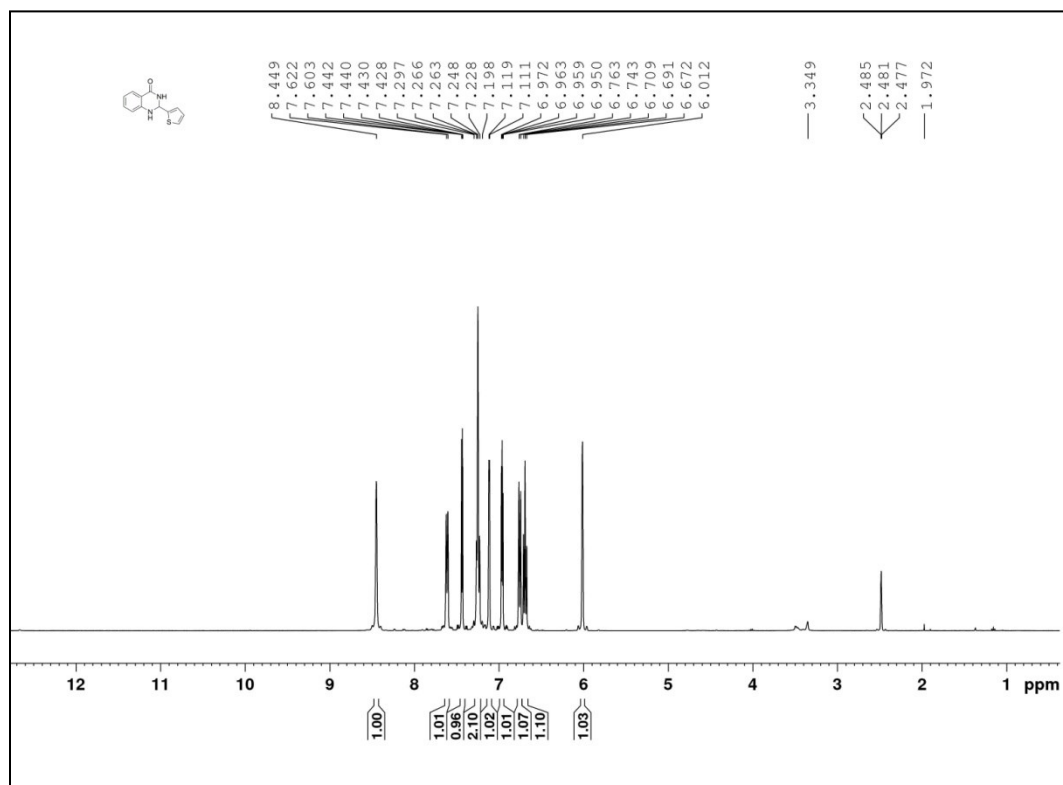
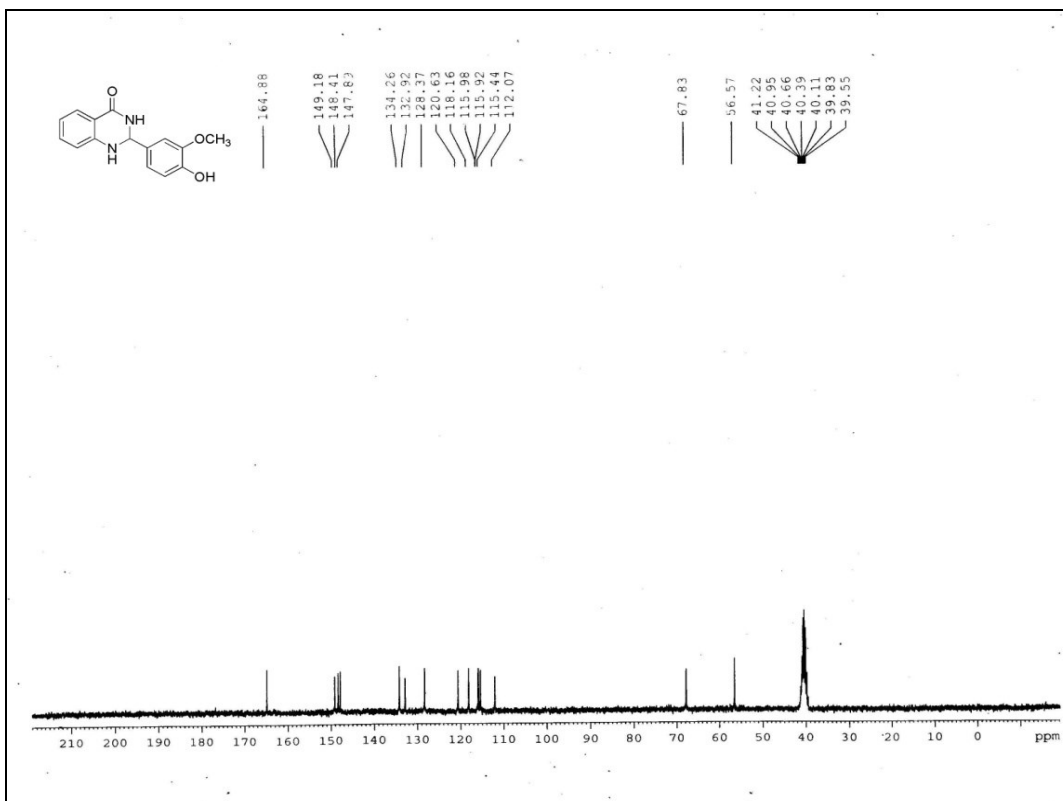


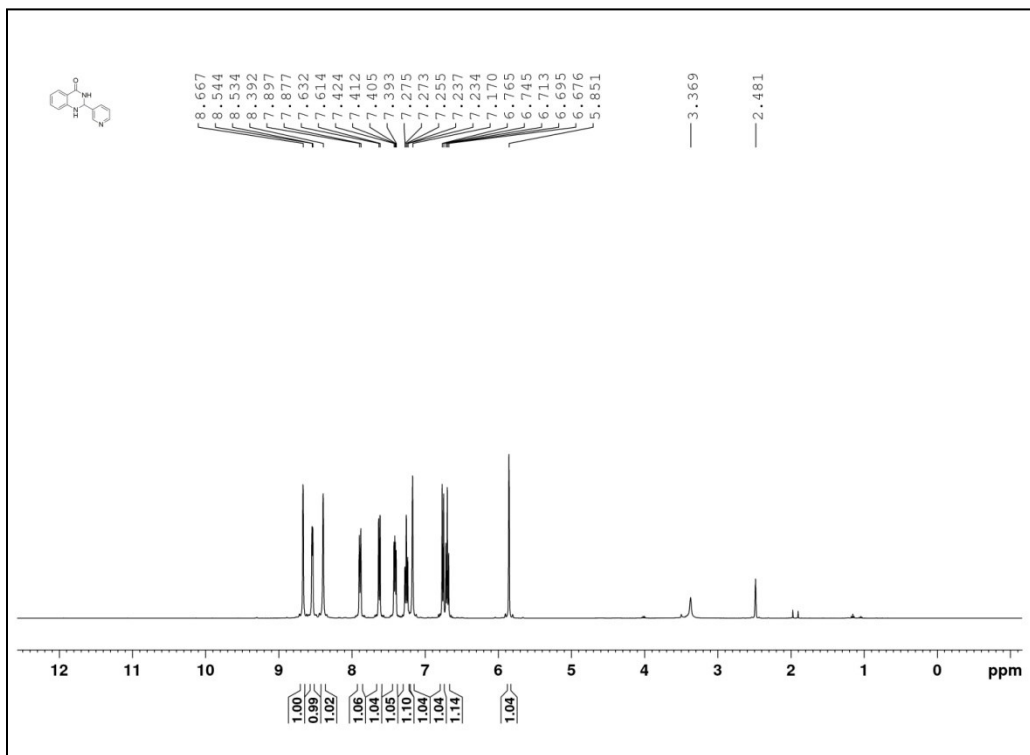
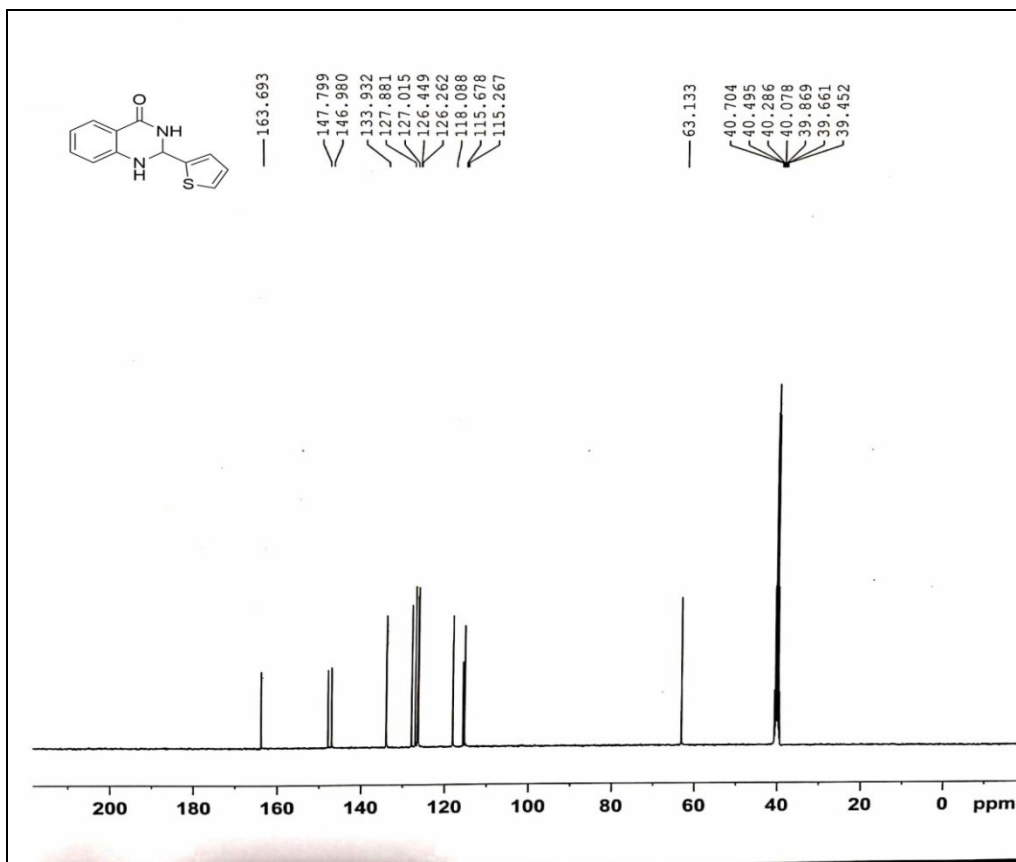


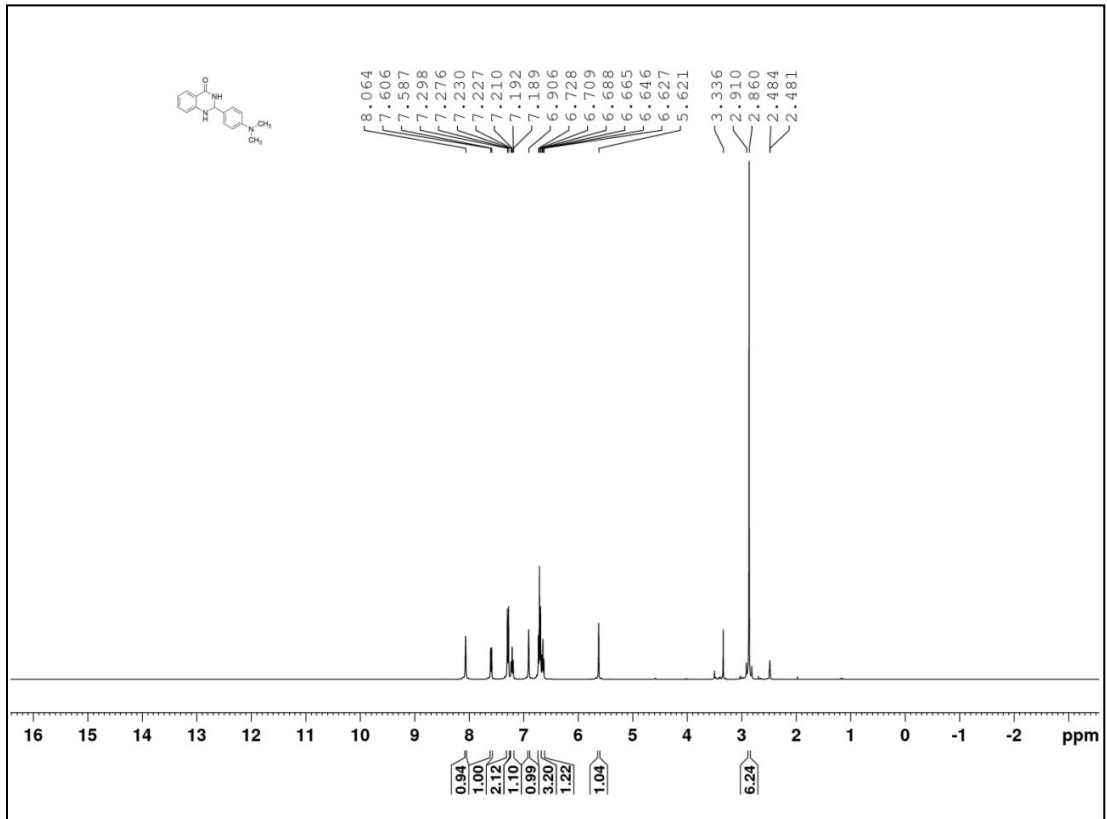
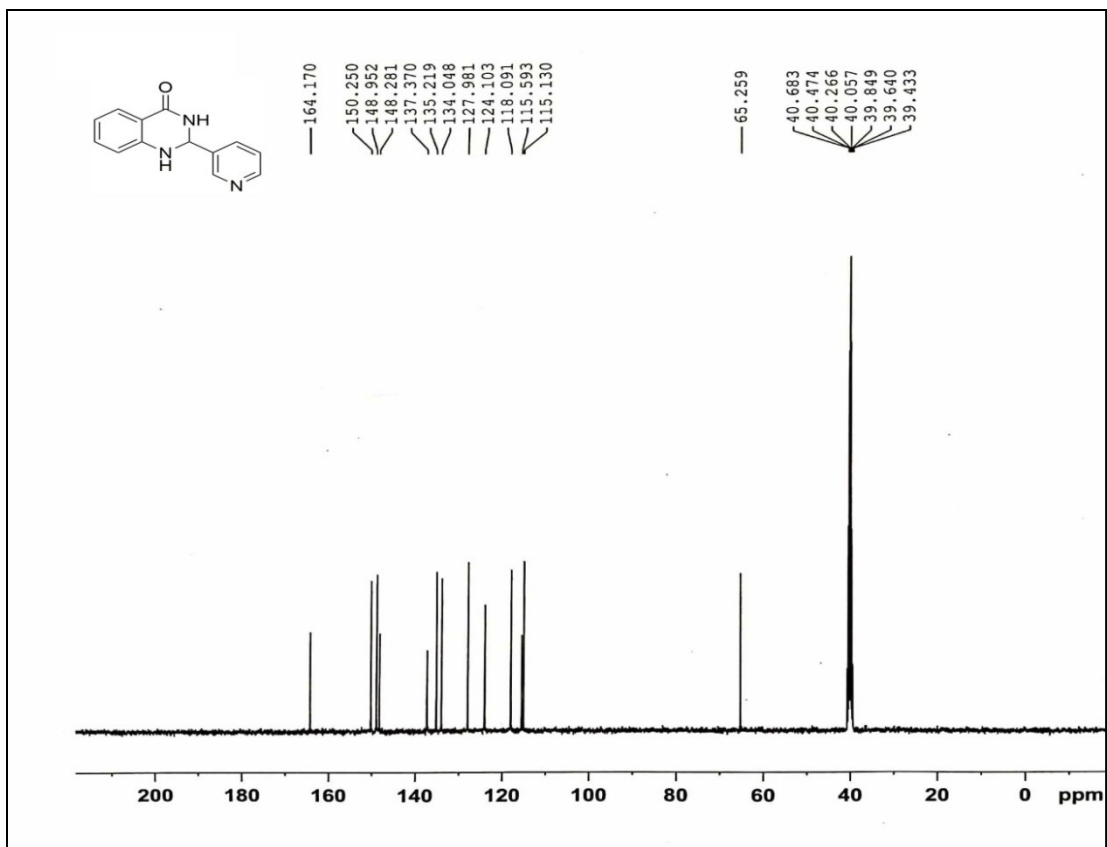


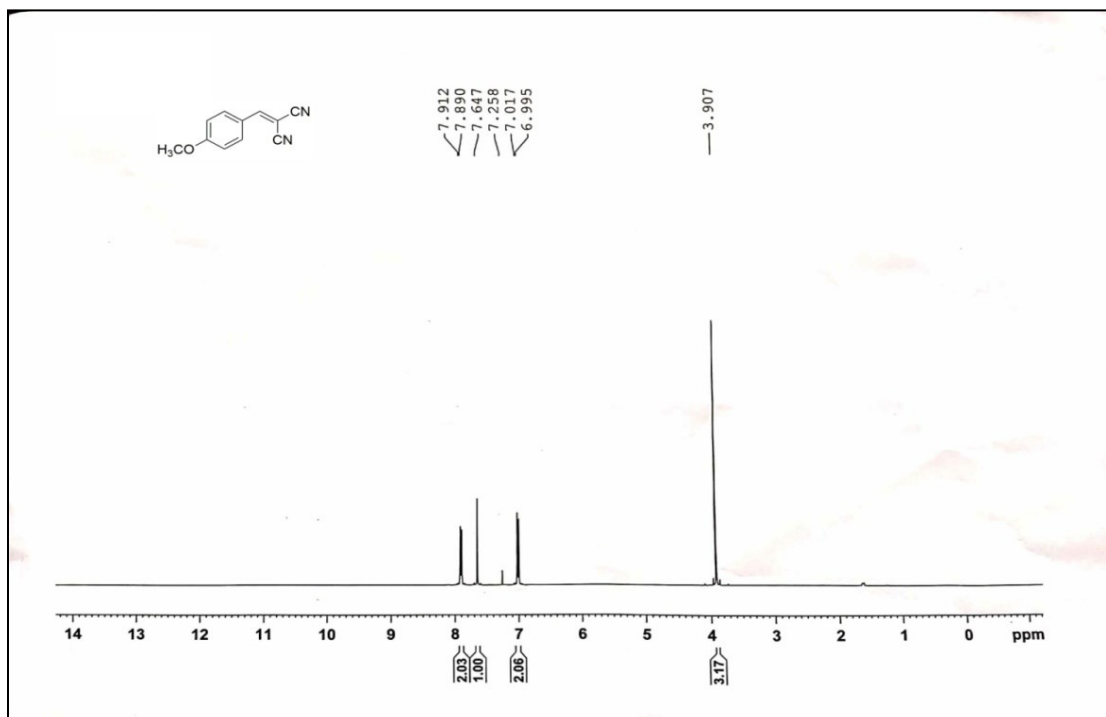
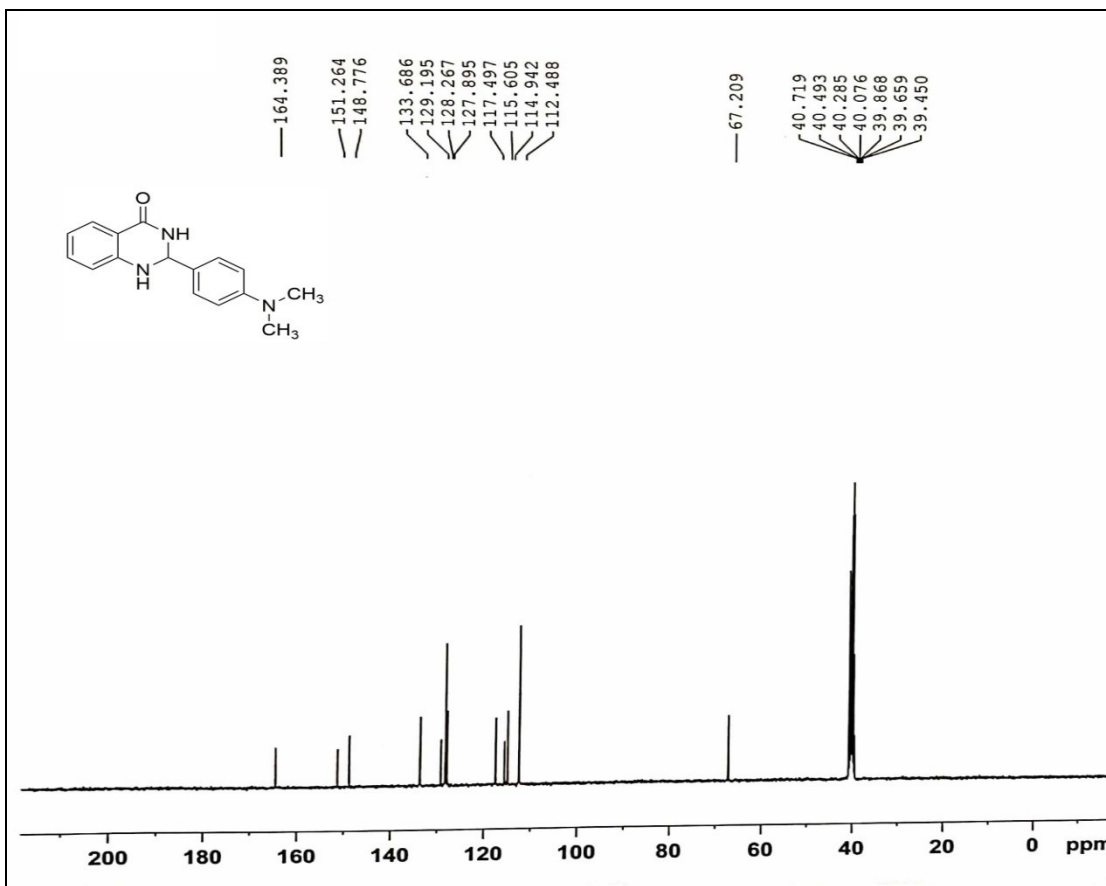


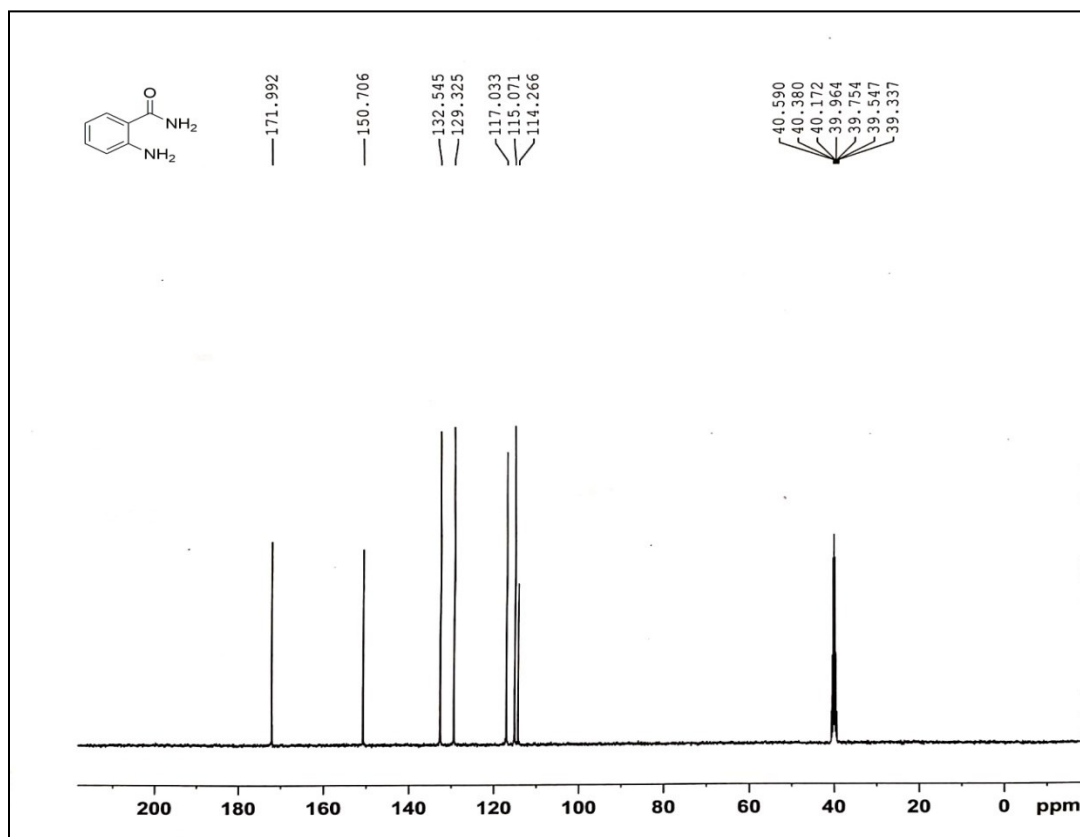
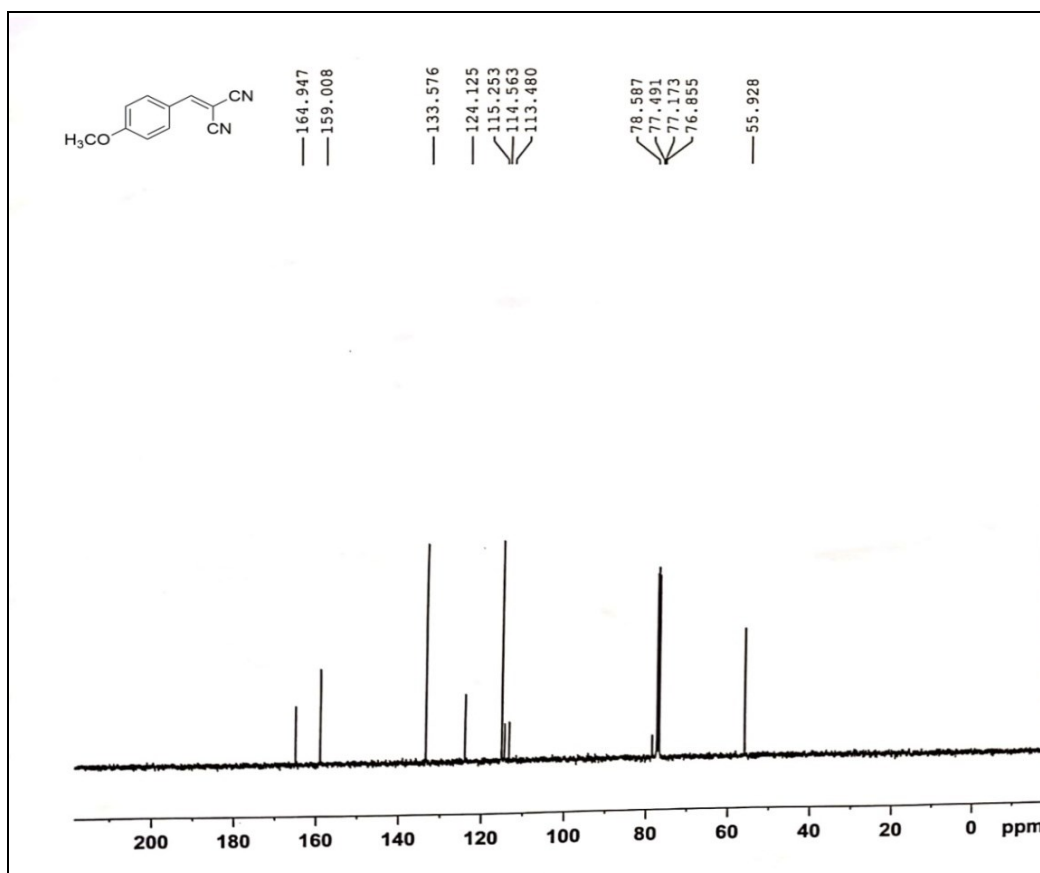












IX. References

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