

Supplementary materials/data

Ionic liquid catalyzed reusable protocol for one-pot synthesis of 2,3-dihydroquinazolin-4(1*H*)-one under mild conditions

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Supplementary Information

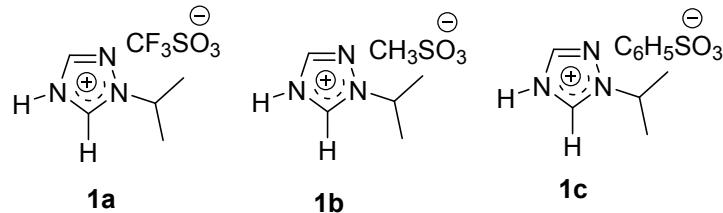
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1. General

Solvents were freshly distilled prior to use and glassware was dried in oven at 120 °C for 5 h. All the reagents were purchased from Sigma Aldrich. ¹H NMR spectra were recorded at 400 MHz and ¹³C NMR spectra at 100 MHz on a Bruker AVANCE FT NMR instrument using CDCl₃ and DMSO-d₆ as standard solvents. Electrospray ionization mass spectrometry (ESI-MS) spectra were recorded on a Waters Q-TOF Premier mass spectrometer. LC/MS data were performed on a Agilent MSD mass spectrometer. Method info: A: 0.05 % HCOOH in H₂O, B: 0.04 % HCOOH in ACN; Flow rate: 2.0 ml/min; Column: Chromolith RP-18e, 100-3mm, +ve mode. Elemental analyses of the compounds were obtained from thermoquest CE instruments CHNS-O, EA/110 model. IR spectra were recorded as KBr pellets on a Bruker Vector 22FT IR spectrometer operating at 400-4000 cm⁻¹. Melting points were measured in DALAL melting point apparatus, India and corrected.

2. General procedure for the preparation of new triazolium based Bronsted acidic ionic liquids (1a-c)



1-isopropyl-1,2,4-triazolium triflate (1a)

To a solution of 1- isopropyl-1,2,4-triazoles (10 mmole) in toluene (10 ml) was added drop wise trifluromethanesulfonic acid (10 mmole). This reaction mixture was then heated to 80 °C for 12 h. After completion of the reaction, flask was cooled to room temperature (25 °C) and excess of toluene was removed under reduced pressure. The resulting residue was thoroughly washed with hexane (20 mL x 2) and further dried over vacuum to afford pure catalyst **1a**.

Note: Similarly we have prepared 1-isopropyl-1,2,4-triazolum methanesulfonate (**1b**) and 1-butyl-1,2,4-triazolium phenylsulfonate (**1c**) ionic liquids.

Large scale preparation of 1-isopropyl-1,2,4-triazolium triflate (1a)

As a large scale experiment, 1-isopropyl-1,2,4-triazole (11.1 g, 100 mmole) and toluene (100 mL) were stirred in a 500 mL round bottom flask under nitrogen atmosphere. To this reaction mixture trifluromethanesulfonic acid (15.0 g, 100 mmole) was added slowly at 20-25 °C. Then the resulting mixture was heated at 80 °C for 12 h under nitrogen. The reaction mixture was then cooled to room temperature. The layers were separated, and the toluene layer was decanted from the reaction mixture. Remaining viscous liquid was washed with hexane (2 x 100 mL) and dried over vacuum at 45 °C for 2 h. The desired product was isolated as colorless oil (24.78 g, yield = 94%).

3.1 General Procedure for the preparation of 2,3-dihydroquinazolinone from Aldehyde:

Catalyst **1a** (10 mol %) was added to a solution of anthranilamide (3.0 mmole) and the corresponding aldehyde (3.0 mmole) in water (1 mL). The reaction mixture was mixed in a mortar and pestle at room temperature for designated time. The progress of the reaction was monitored by TLC. After completion of the reaction, the reaction mixture was quenched with

mixture of water:ethanol (5:0.5 mL). The corresponding solid product was filtered, and washed with *n*-hexane (5 mL x 2), which afforded pure 2,3-dihydroquinazolinone.

3.2 General Procedure for the preparation of 2,3-dihydroquinazolinone from ketones:

Catalyst **1a** (10 mol%) was added to a solution of anthranilamide (3.0 mmole) and the corresponding ketone (3.0 mmole) in ethanol (1 mL). The reaction mixture was stirred in 5 mL glass vial at room temperature. The progress of the reaction was monitored by TLC. After completion of the reaction, the reaction mixture was quenched with mixture of water:ethanol (5:0.5 mL). The solid was filtered, and washed with *n*-hexane (5 mL x 2), which afforded pure 2,3-dihydroquinazolinone.

4. General procedure for the reusability of new triazolium based Brønsted acidic ionic liquids.

The catalyst was separated from the reaction mixture by simple filtration technique and the filtrate was concentrated to remove excess of water and small amount of ethanol. Then the crude residue was thoroughly washed with mixture of hexane: ethyl acetate (4:1) 5 mL and dried at 45 °C over vacuum for 1 h. This residue was subjected directly to catalyst reusable study.

5. Characterization Data of the Products

1-Isopropyl 1, 2, 4-triazolium trifluoromethanesulfonate (Figure 1, 1a)

Colorless liquid, yield 94%, Elemental Analysis: calcd. for C, 27.48; H, 4.23; N, 16.02, Found : C, 27.24, H, 4.11, N, 16.01; ¹H NMR (CDCl₃, 400 MHz) δ 12.9 (s, 1H, NH), 9.6 (s, 1H, 5-CH), 8.6 (s, 1H, 3-CH), 4.9 (m, 1H, CH(CH₃)₂), 1.6 (d, J = 8Hz, 6H, CH(CH₃)₂), ¹³C NMR (CDCl₃,

100 MHz) δ 143.1(C-5), 139.5(C-3), 124.8, 121.6, 118.5 115.3, (CF₃SO₃), 55.7 CH(CH₃)₂), 21.4 CH(CH₃)₂) ES-MS *m/z* 112.1580, ¹⁹F NMR -78.9378;

1-Isopropyl 1, 2, 4-triazolium methanesulfonate (Figure 1, 1b)

Colorless liquid, yield 91%, Elemental Analysis: calcd. For, C, 34.60; H, 6.78; N, 20.18: Found, C, 34.54, H, 6.56, N, 20.10; ¹H NMR (CDCl₃, 300 MHz) δ: 12.4 (s, 1H, NH), 10.1 (s, 1H, 5-CH), 8.6 (s, 1H, 3-CH), 4.9 (m, 1H, CH (CH₃)₂), 2.8 (s, 3H, CH₃SO₃), 1.6 (d, 6H, CH (CH₃)₂). ¹³C NMR (CDCl₃, 75 MHz) δ :143.3 (C-5), 140.3 (C-3), 55.4 (CH(CH₃)₂), 39.6 (CH₃SO₃), 21.8 (CH(CH₃)₂).

1-Isopropyl 1, 2, 4-triazolium phenylsulfonate (Figure 1, 1c)

Colorless solid, yield 89%, Elemental Analysis: calcd. For, C, 48.87; H, 5.97; N, 15.54; Found : C, 48.64, H, 5.85, N, 15.34; ¹H NMR (CDCl₃, 300 MHz) δ :12.8 (bs, 1H, NH), 10.1 (s, 1H, 5-CH), 8.6 (s, 1H, 3-CH), 7.8 (m, 2H, CH), 7.3 (m, 3H, CH) 4.8 (m, 1H, CH (CH₃)₂), 1.5 (d, *J* = 6.6 Hz, 6H, CH (CH₃)₂). ¹³C NMR (CDCl₃, 75 MHz) δ: 143.0 (C-5), 140.2 (C-3), 130.3, 128.3, 125.8 (C₆H₅SO₃), 55.5 (CH(CH₃)₂), 21.4 (CH(CH₃)₂).

2-phenyl-2, 3-dihydroquinazolin-4(1H)-one (Table 2, entry 1), White solid,

yield 96%, m. p. 217-219 °C (Lit. ¹: 218-220 °C); ¹H NMR (400MHz, DMSO-d₆): δ 8.2 (s, 1H), 7.6 (m, 1H), 7.5 (m, 2H), 7.3 (m, 3H), 7.2 (m, 1H), 7.1 (s, 1H), 6.7 (m, 2H), 5.7 (s, 1H); ¹³C NMR (100MHz, DMSO-d₆): δ 163.5, 147.8, 141.6, 133.2, 128.4, 128.3, 127.3, 126.8, 117.0, 114.9, 114.3, 66.5. IR (KBr) : 3305, 3180, 3059, 1665, 1615, 1535, 1450 cm⁻¹ LC/MS [M+H]⁺ (m/z) 235

2-(3, 5-dimethoxyphenyl)-2,3-dihydroquinazolin-4(1H)-one (Table 2, entry 4)

White solid, yield 95 %, m. p. 139-140 °C; ¹H NMR (400MHz, DMSO-d₆): δ 8.2 (s, 1H), 7.5 (d, *J* = 12 Hz, 1H), 7.2 (m, 1H), 7.1 (s, 1H), 6.7 (d, *J* = 8Hz, 1H), 6.6 (m, 3H), 6.4 (m, 1H), 5.6 (s, 1H), 3.7 (s, 6H); ¹³ C NMR (100MHz, DMSO-d₆): δ 163.5, 160.3, 147.7, 144.1, 133.2, 127.3,

117.1, 114.9, 114.3, 104.9, 99.7, 66.1, 55.2. IR (KBr): 3302, 3186, 3061, 2936, 2806, 1908, 1655, 1613, 1510, 1440, 1388, 1300, 1250, 1152, 1029, cm⁻¹; LC/MS [M+H]⁺ (m/z)285

4-(4-oxo-1,2,3,4-tetrahydroquinazolin-2-yl)benzoic acid (Table 2, entry 12) White solid, yield 91%, m. p. 271-273 °C; ¹H NMR (400MHz, DMSO-d₆): δ 12.9 (s, 1H), 8.3 (s, 1H), 7.9 (d, J = 8 Hz, 2H), 7.6 (m, 3H), 7.2 (m, 2H), 6.7 (m, 2H), 5.8 (s, 1H). ¹³C NMR (100MHz, DMSO-d₆): δ 167.0, 163.4, 147.5, 146.4, 133.4, 130.8, 129.3, 127.3, 127.0, 117.2, 114.9, 114.4, 66.0. IR (KBr): 3300, 3179, 3127, 3063, 2934, 2865, 2677, 2551, 1698, 1656, 1610, 1584, 1509, 1431, 1385, 1291, 1155, 1015, cm⁻¹ LC/MS [M+H]⁺ (m/z)269

2-(naphthalen-2-yl)-2, 3-dihydroquinazolin-4(1H)-one (Table 2, entry 13)

Colorless solid, yield 92%, m. p. 167-169 °C; ¹H NMR (400MHz, DMSO-d₆): δ 8.5 (m, 1H), 8.2 (s, 1H), 7.9 (t, J = 8Hz, 2H), 7.7 (m, 2H), 7.5 (m, 3H), 7.2 (m, 1H), 7.1 (s, 1H), 6.7 (m, 1H), 6.5 (s, 1H); ¹³C NMR (100MHz, DMSO-d₆): δ 164.0, 148.4, 135.1, 133.7, 133.2, 130.5, 129.3, 128.5, 127.5, 126.0, 125.8, 125.1, 124.5, 117.2, 114.9, 114.5, 65.9. IR (KBr): 3381, 3306, 3227, 3053, 2921, 1935, 1655, 1610, 1503, 1424, 1373, 1299, 1152 cm⁻¹; LC/MS [M+H]⁺ (m/z) 275

2-methyl-2-phenyl-2, 3-dihydroquinazolin-4(1H)-one (Table 3, entry 1)

Colorless solid, yield 93%, m. p. 225-226 °C (Lit.²: 224-225 °C); ¹H NMR (400 MHz, DMSO-d₆): δ 8.7 (s, 1H), 7.6 (s, 1H), 7.5 (m, 3H), 7.2 (m, 2H), 7.1 (m, 2H), 6.7 (m, 1H), 6.5 (m, 1H), 1.6 (s, 3H); ¹³C NMR (100MHz, DMSO-d₆): δ: 163.7, 147.6, 147.1, 133.2, 127.9, 127.2, 126.9, 125.1, 116.7, 115.0, 114.2, 70.1, 30.7. IR (KBr): 3400, 3045, 1667, 1618, 1510, 1599, 14420, 1395, 1330, 1275, 1220, 1191, 1153, 1120, 1082, 1030 cm⁻¹; LC/MS [M+H]⁺ (m/z) 239

1'H-spiro [cyclohexane-1,2'-quinazolin]-4'(3'H)-one (Table 3, entry 8)

White solid, yield 91%, m. p. 223-225 °C (Lit.²: 224-225 °C); ¹H NMR (400MHz, DMSO-d₆): δ 7.9 (s, 1H), 7.5 (m, 1H), 7.2 (m, 1H), 6.8 (s, 1H), 6.6 (m, 2H), 1.7 (m, 2H), 1.6 (m, 6H), 1.4 (m, 1H), 1.2 (m, 1H); ¹³C NMR (100MHz, DMSO-d₆): δ 163.1, 146.7, 133.0, 127.0, 116.4, 114.5, 114.4, 67.7, 37.1, 24.6, 20.8. IR (KBr): 3280, 3184, 3030, 2980, 2932, 2858, 1660, 1620, 1520, 1484, 1420, 1390, 1276, 1185, 1150, cm⁻¹; LC/MS [M+H]⁺ (m/z) 217

1'H-spiro[cyclopentane-1,2'-quinazolin]-4'(3'H)-one (Table 3, entry 9)

White solid, yield 90%, m. p. 258-260 °C (Lit.³ : 257-260 °C); ¹H NMR (400MHz, DMSO-d₆): δ 8.0 (s, 1H), 7.5 (d, J = 8 Hz, 1H), 7.2 (t, J = 12Hz, 1H), 6.7 (s, 1H), 6.7 (m, 1H), 6.6 (m, 1H); ¹³C NMR (100MHz, DMSO-d₆) δ : 163.9, 148.0, 133.4, 127.7, 117.0, 115.0, 114.8, 77.5, 22.4. IR (KBr): 3283, 3186, 3035, 2982, 2929, 2940, 2855, 1666, 1620, 1519, 1483, 1431, 1390, 1325, 1270, 1177, 1150 cm⁻¹; LC/MS [M+H]⁺ (m/z) 203

2, 2-dimethyl-2,3-dihydroquinazolin-4(1H)-one: (Table 3, entry 10)

colorless solid, yield 89%, m.p.183-184 °C (Lit.²: 182-183 °C); ¹H NMR (400MHz, DMSO-d₆) : δ 7.9 (s, 1H), 7.6 (d, J = 8Hz 1H), 7.2 (t, J = 8 Hz, 1H), 6.6 (m, 3H), 1.3 (s, 6H); ¹³C NMR (100MHz, DMSO-d₆): 163.5, 147.5, 133.6, 127.6, 116.9, 114.7, 114.3, 67.3, 29.4. IR (KBr): 3330, 3261, 3033, 2991, 2969, 1655, 1620, 1515, 1484, 1388, 1361, 1335, 1278, 1197, 1180, 1140, cm⁻¹;

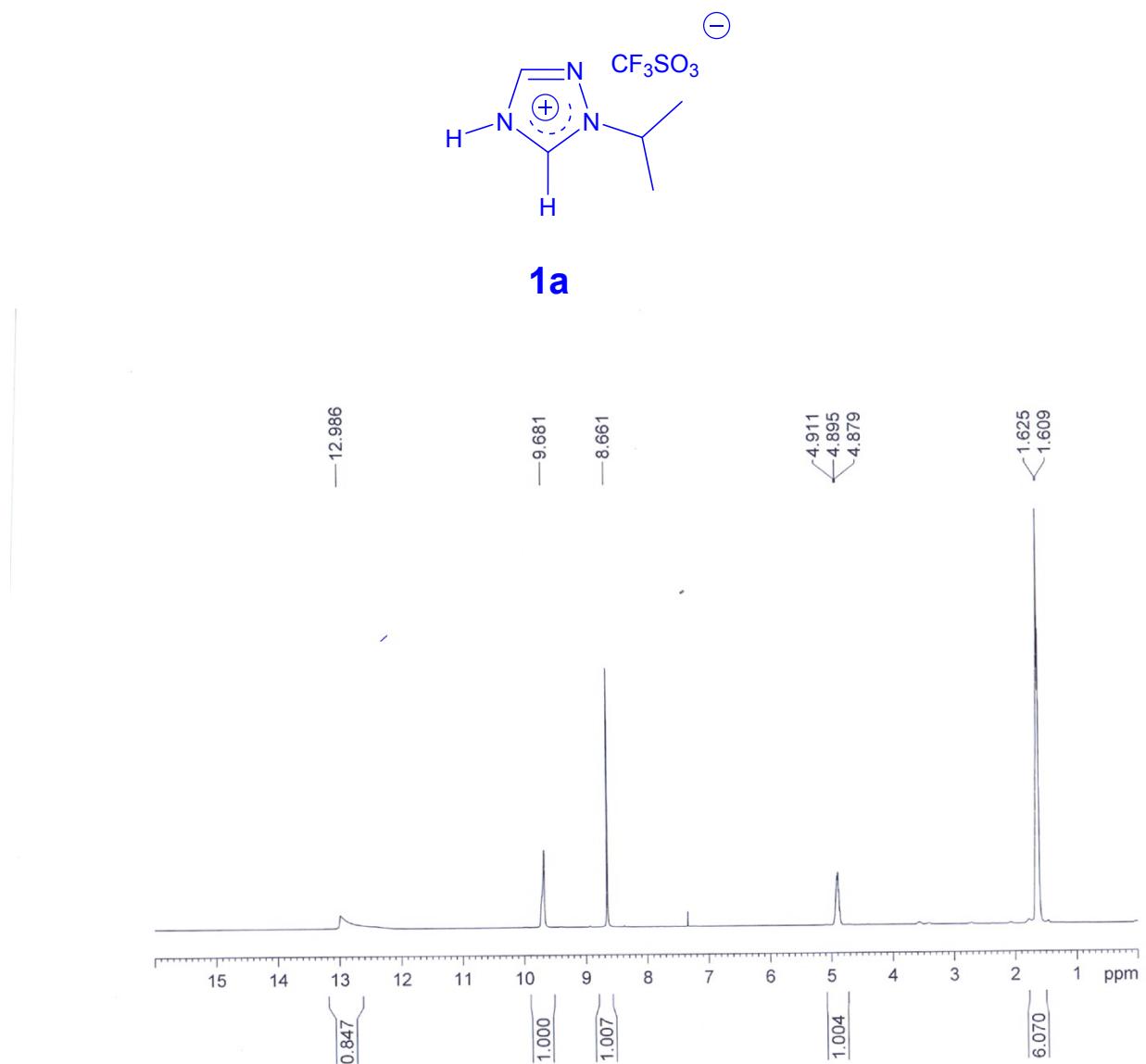
2-isobutyl-2-methyl-2,3-dihydroquinazolin-4(1H)-one: (Table 3, entry 11)

White solid, yield 94%, m. p. 172-174 °C (Lit.²: 171-173 °C);; ¹H NMR (400MHz, DMSO-d₆) δ 7.8 (s, 1H), 7.5 (d, J = 8Hz 1H), 7.2 (m, 1H), 6.6 (m, 3H) 1.9 (m, 1H), 1.8 (m, 1H), 1.5 (m, 3H), 0.9 (m, 6H); ¹³C NMR (100MHz, DMSO-d₆): δ 163.5, 147.5, 133.6, 127.5, 116.4, 114.4, 113.8, 69.8, 49.8, 29.1, 24.8, 24.6, 23.8. IR (KBr) : 3324, 3170, 2950, 2865, 1657, 1612, 1580, 1511, 1484, 1432, 1390, 1362, 1320, 1285, 1189, 1150, 1060, 825, 745 cm⁻¹;

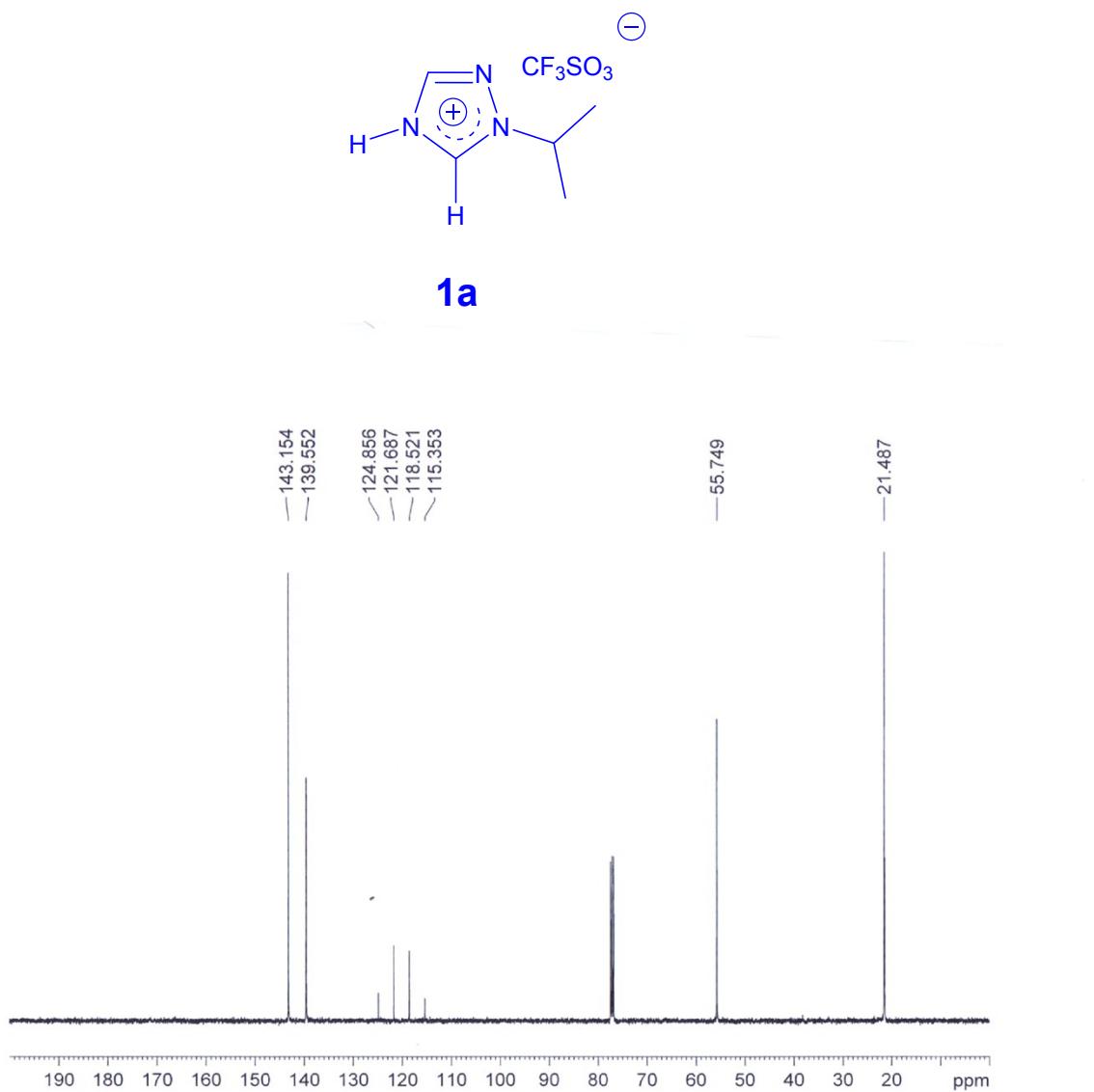
2, 2-diethyl-2,3-dihydroquinazolin-4(1H)-one (Table 3, entry 12)

White solid ,yield 93%, m. p. 189-190 °C (Lit.⁴: 190-191 °C); ¹H NMR (400MHz, DMSO-d₆): δ 7.7 (s, 1H), 7.5 (m, 1H), 7.1 (m, 1H), 6.6 (d, J = 8Hz, 1H), 6.6 (m, 1H), 6.4 (s, 1H), 1.6 (m, 4H), 0.9 (m, 6H); ¹³C NMR (100MHz, DMSO-d₆) δ: 163.8, 148.1, 133.5, 127.4, 115.9, 114.0, 113.4, 72.4, 33.2, 8.3. IR (KBr): 3320, 3167, 2870, 1665, 1600, 1570, 1521, 1479, 1428, 1385, 1323, 1285, 1178, 1090, 1050, 840, 739 cm⁻¹; LC/MS [M+H]⁺ (m/z) 205.

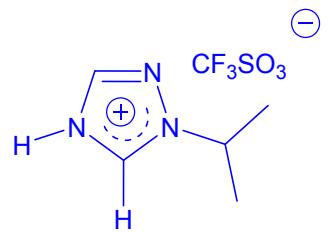
¹H NMR spectrum of 1-isopropyl-1,2,4-triazolium trifluoromethanesulfonate (1a)



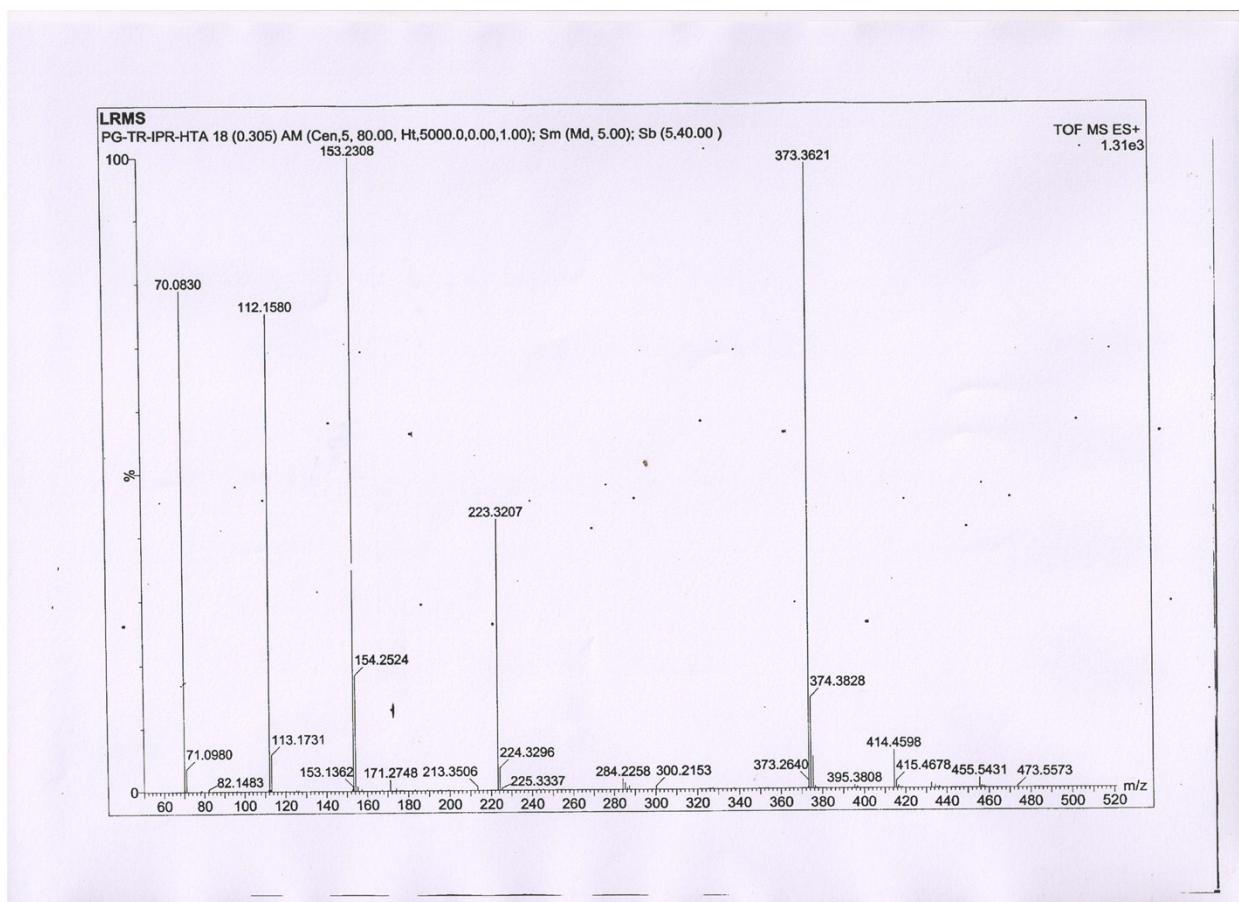
^{13}C NMR spectrum of 1-isopropyl-1,2,4-triazolium trifluoromethanesulfonate (1a)



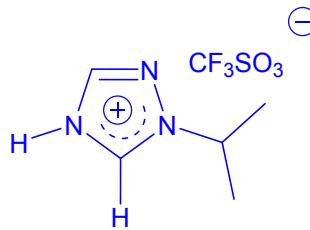
Mass-Spectrum of 1-isopropyl-1,2,4-triazolium trifluoromethanesulfonate (1a)



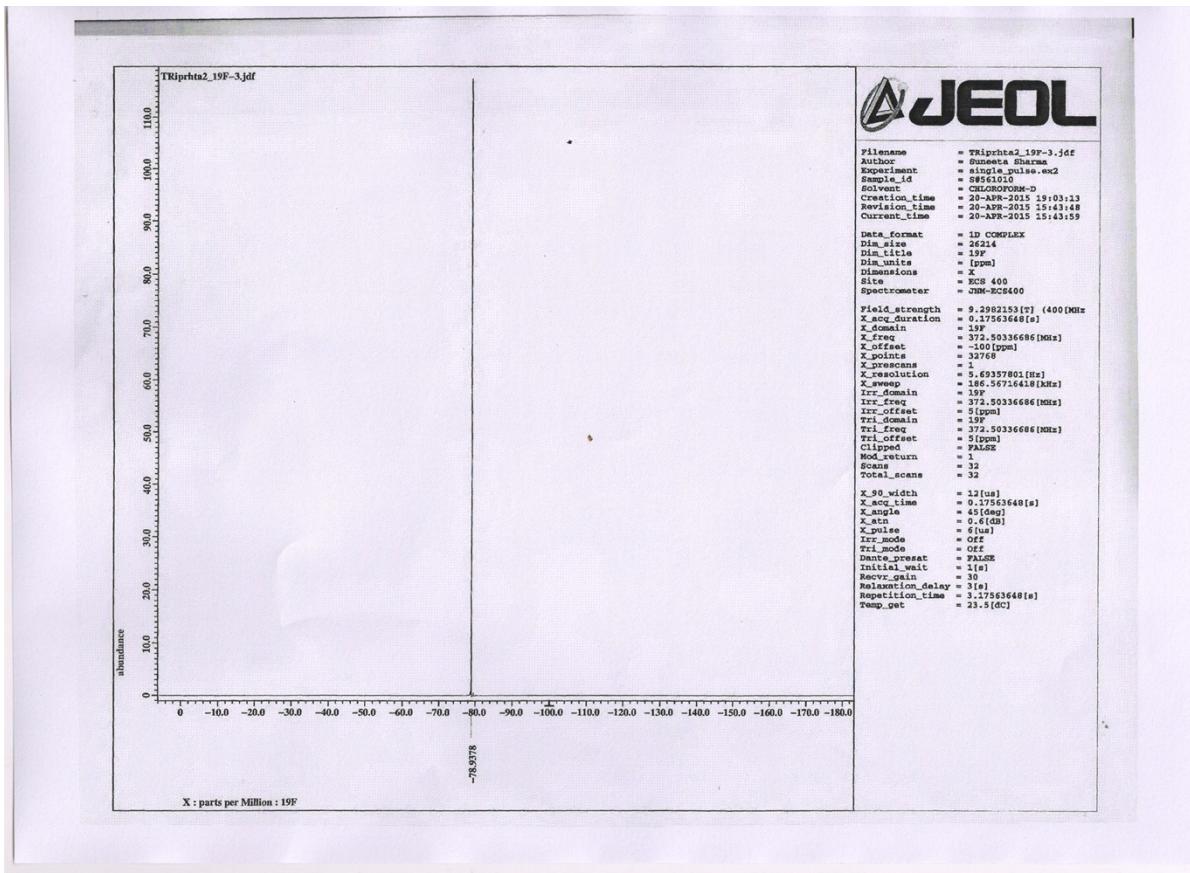
1a



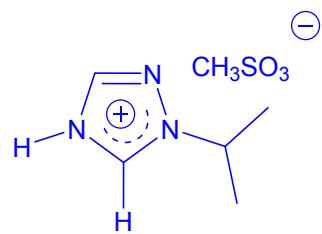
¹⁹F NMR Spectrum of 1-isopropyl-1,2,4-triazolium trifluoromethanesulfonate (1a)



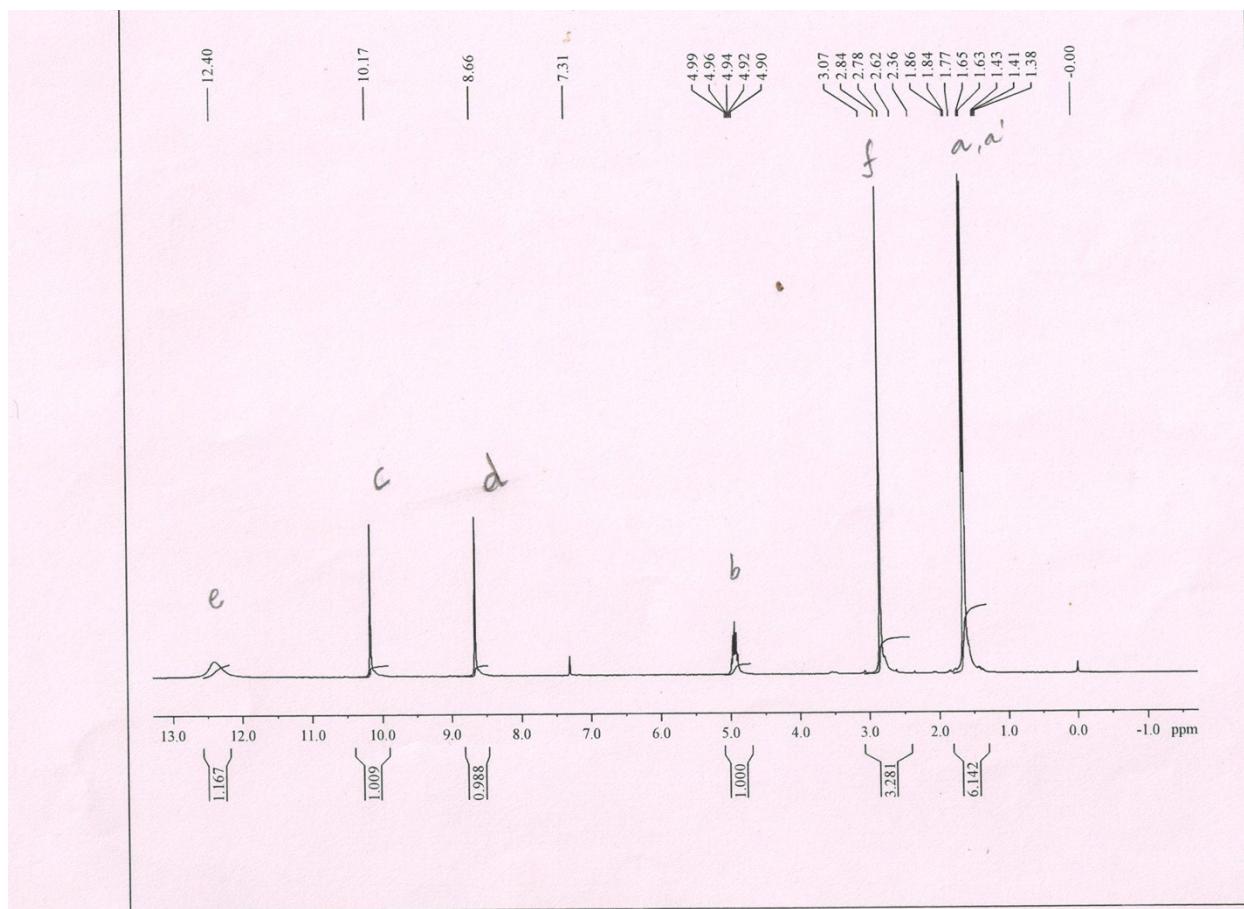
1a



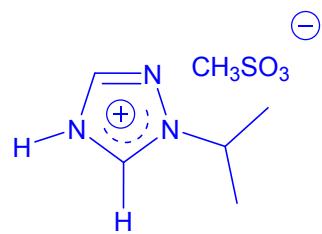
¹H NMR spectrum of 1-isopropyl-1,2,4-triazolium methanesulfonate (1b)



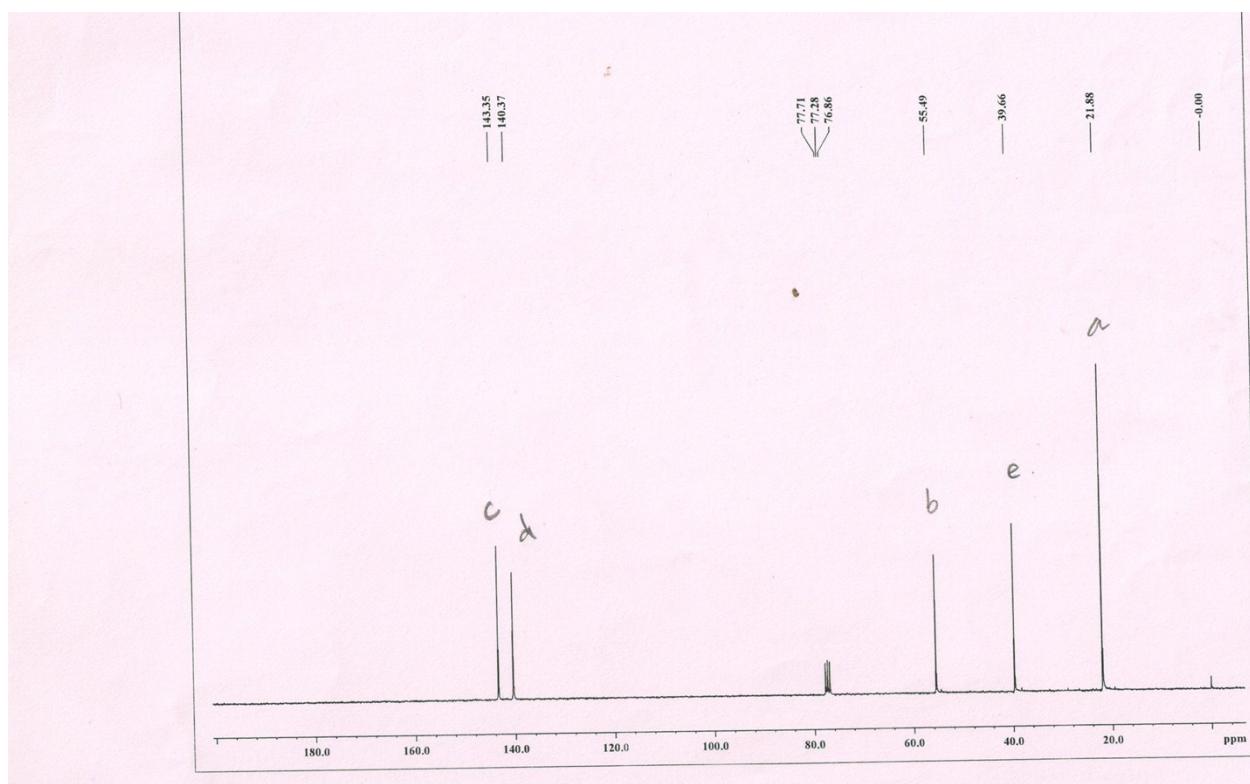
1b



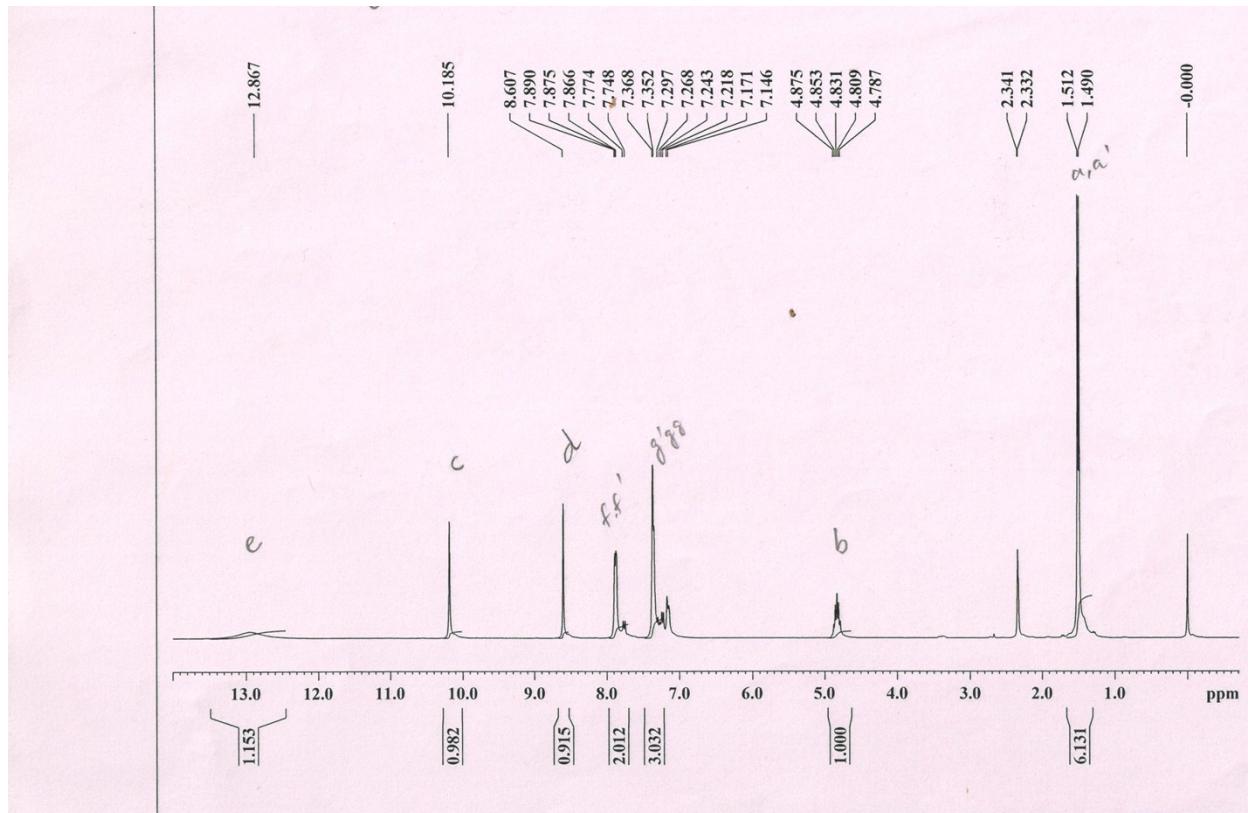
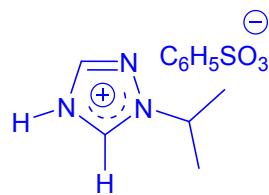
^{13}C NMR spectrum of 1-isopropyl-1,2,4-triazolium methanesulfonate (1b)



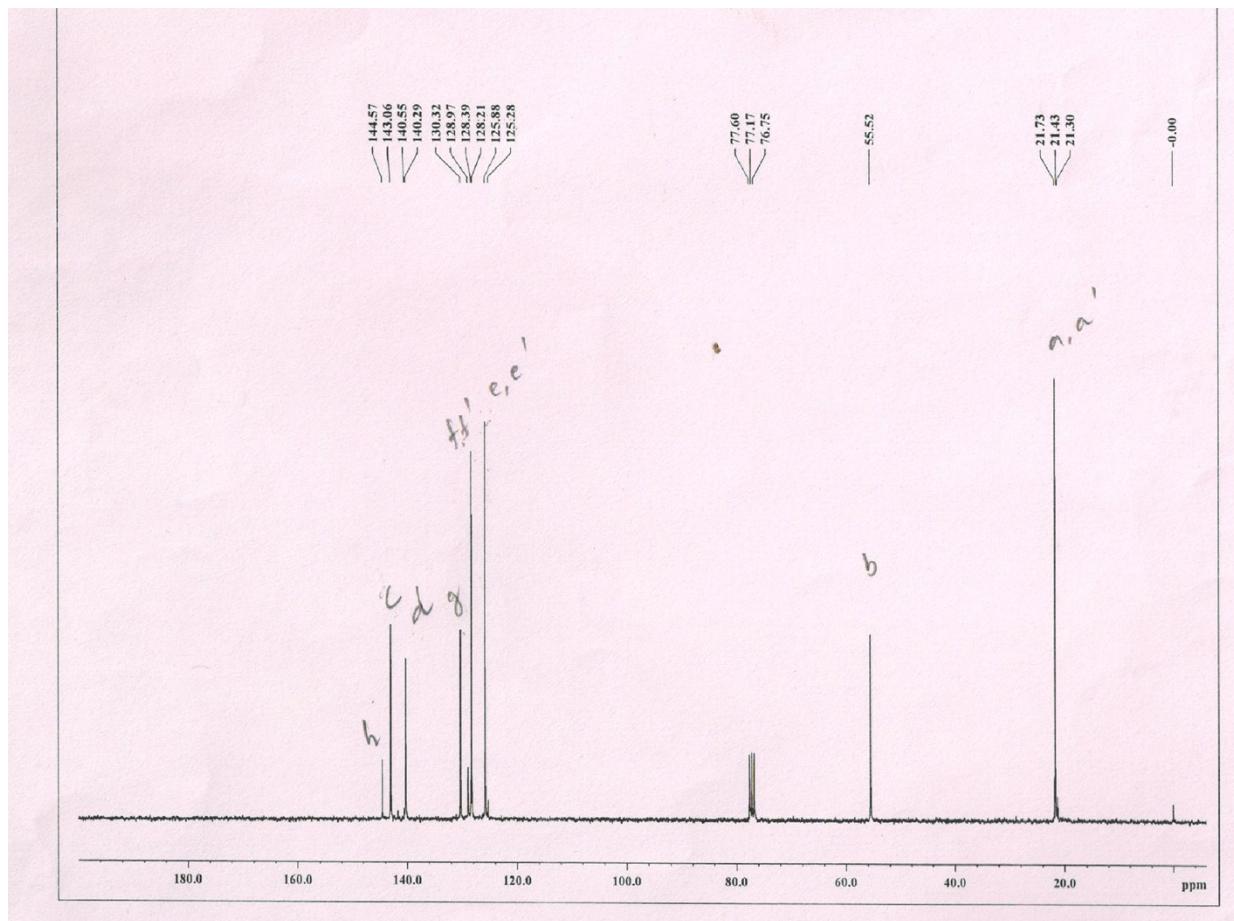
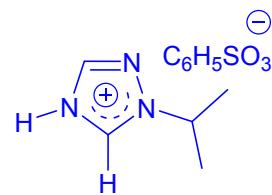
1b



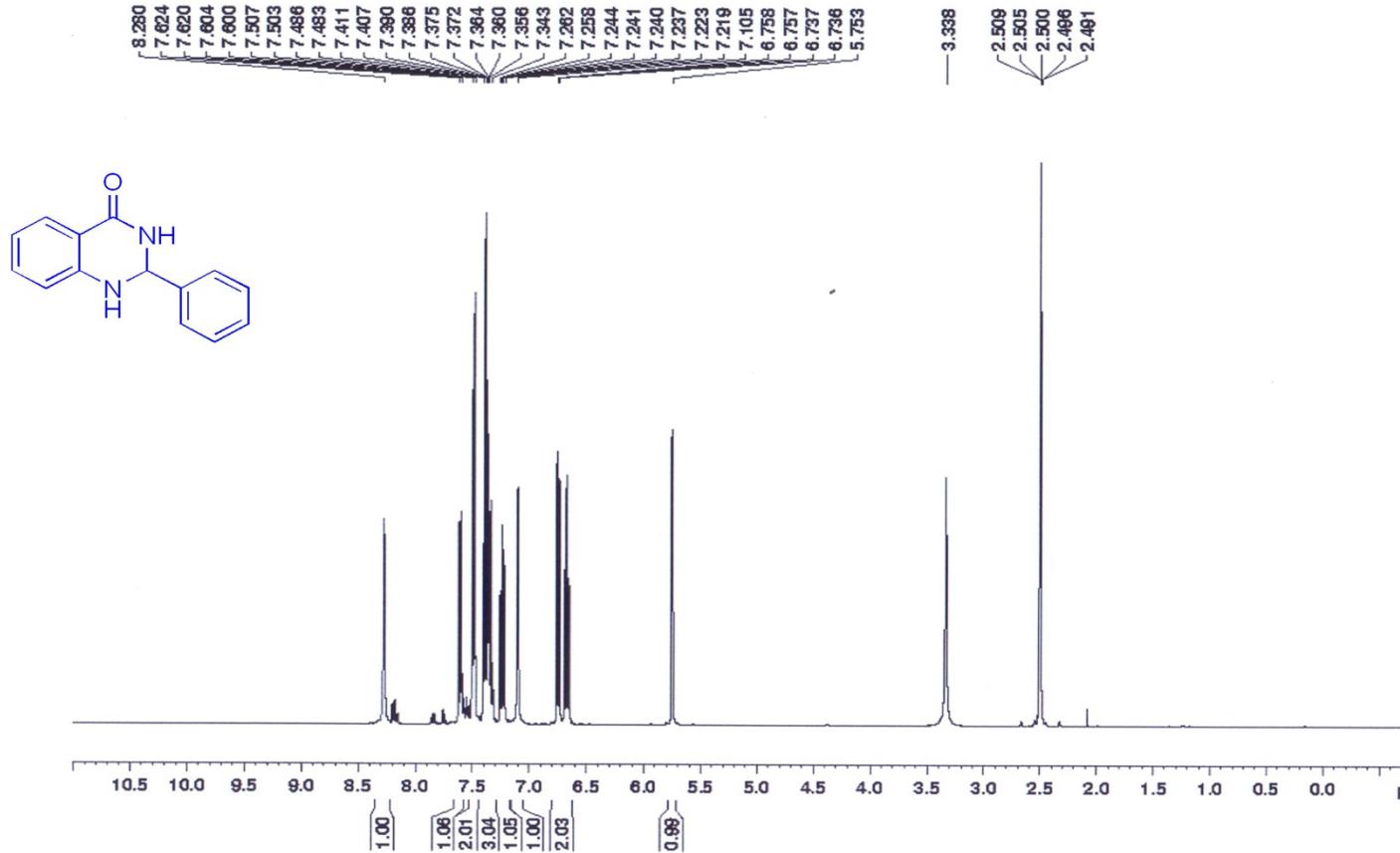
^1H NMR spectrum of 1-isopropyl-1,2,4-triazolium phenylsulfonate (1c)



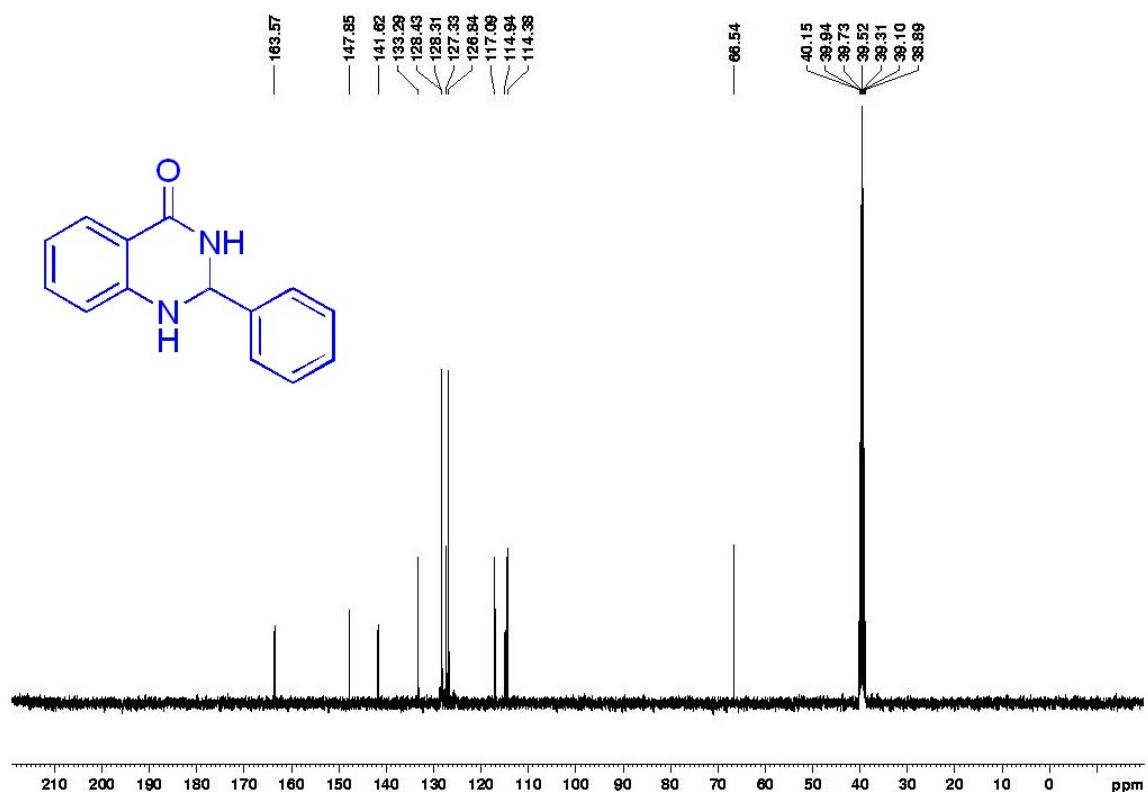
¹³C NMR spectrum of 1-isopropyl-1,2,4-triazolium phenylsulfonate (1c)



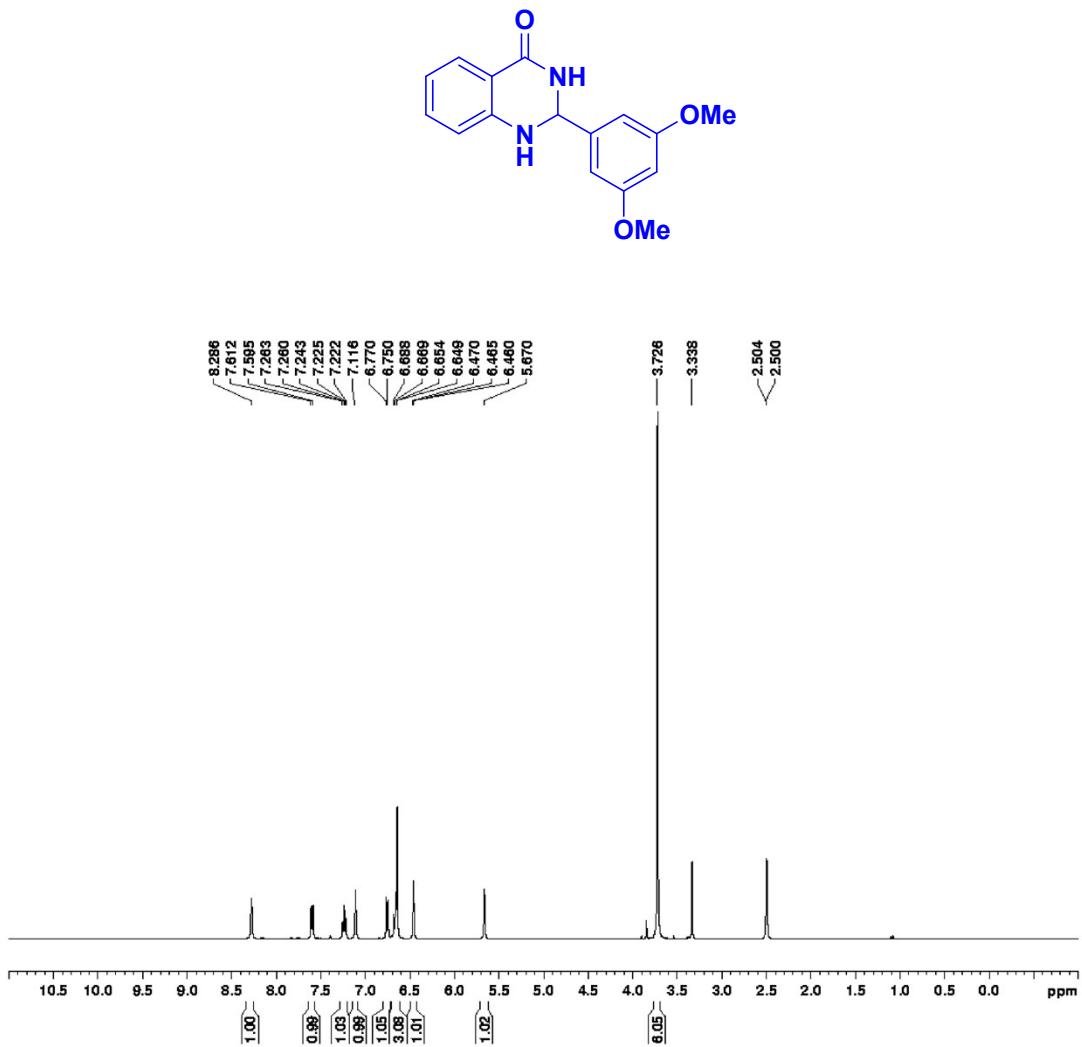
¹H NMR spectrum of 2-phenyl-2, 3-dihydroquinazolin-4(1H)-one (Table 2, entry 1)



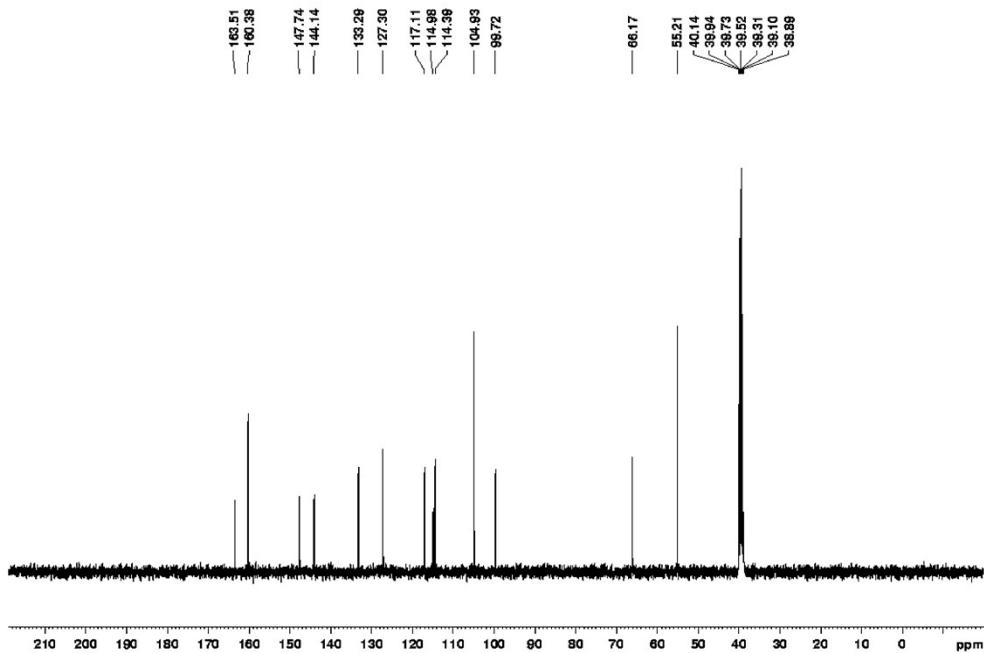
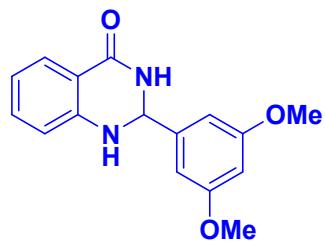
¹³C NMR spectrum of 2-phenyl-2,3-dihydroquinazolin-4(1H)-one (Table 2, entry 1)



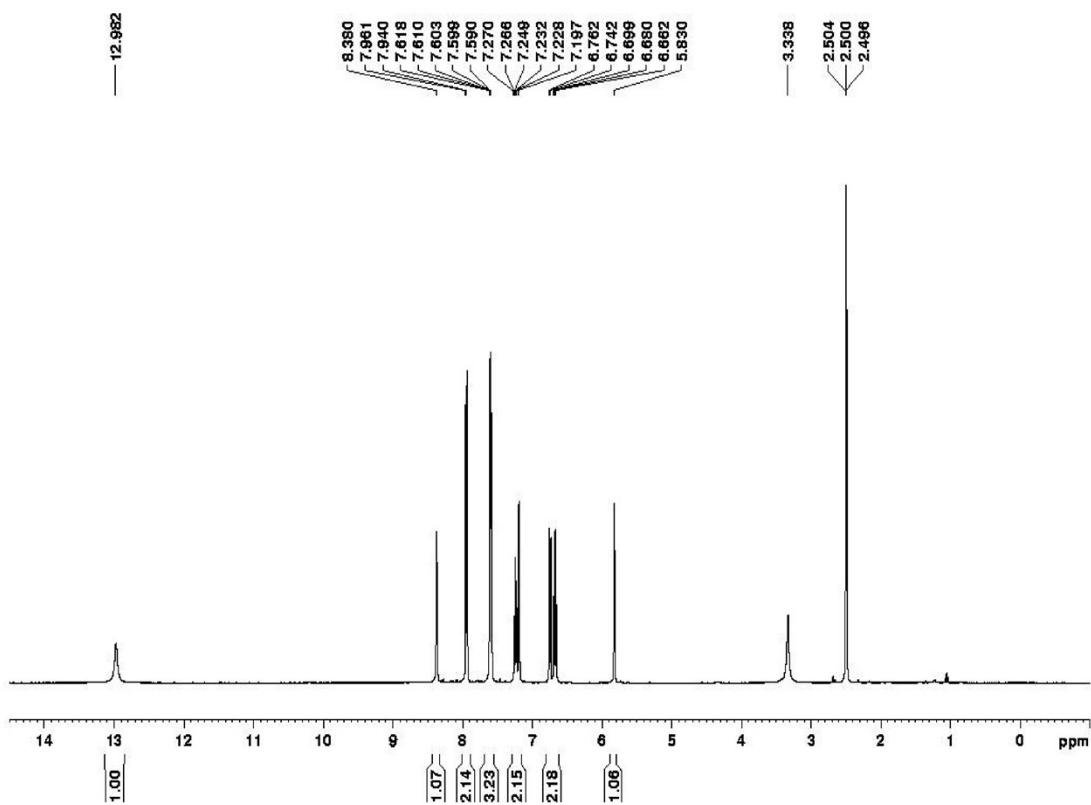
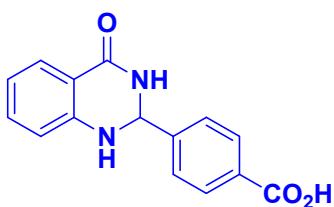
¹H NMR spectrum of 2-(3, 5-dimethoxyphenyl)-2,3-dihydroquinazolin-4(1H)-one (Table 2, entry 4)



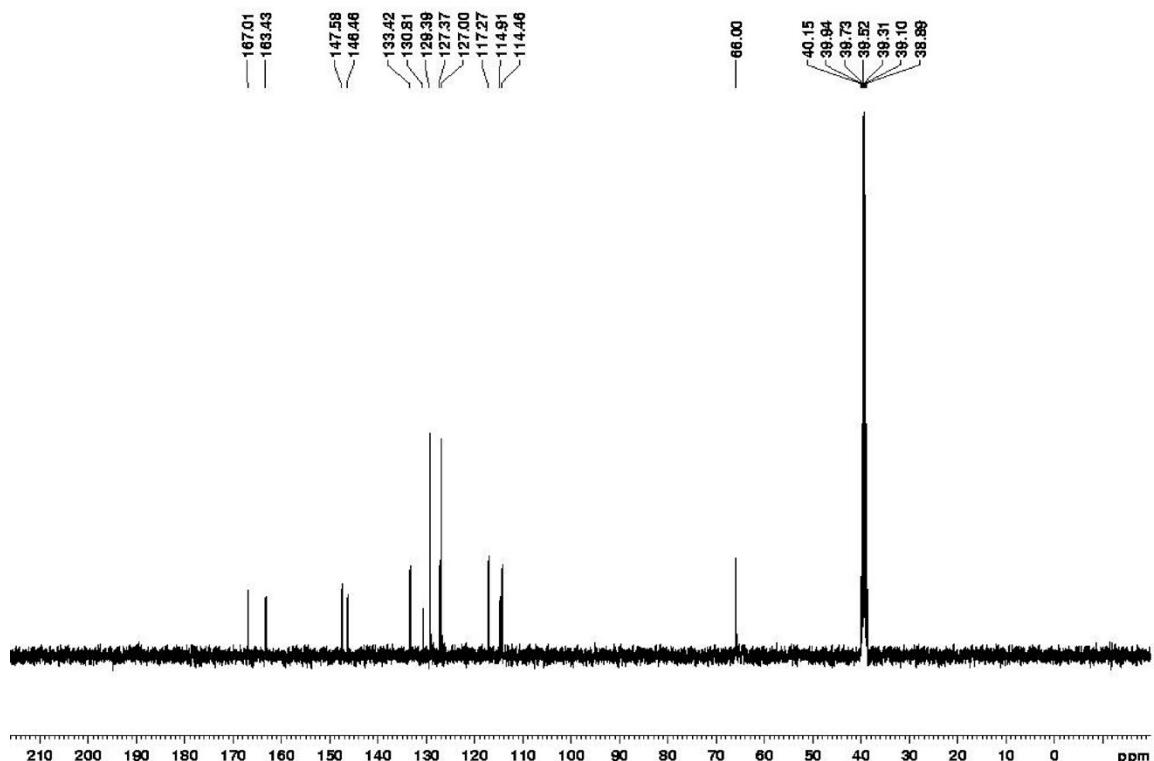
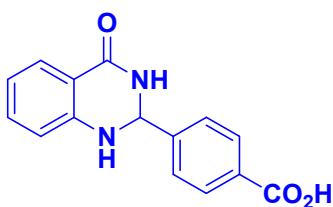
¹³C NMR spectrum of 2-(3, 5-dimethoxyphenyl)-2,3-dihydroquinazolin-4(1H)-one (Table 2, entry 4)



¹H NMR spectrum of 4-(4-oxo-1,2,3,4-tetrahydroquinazolin-2-yl)benzoic acid (Table 2, entry 12)

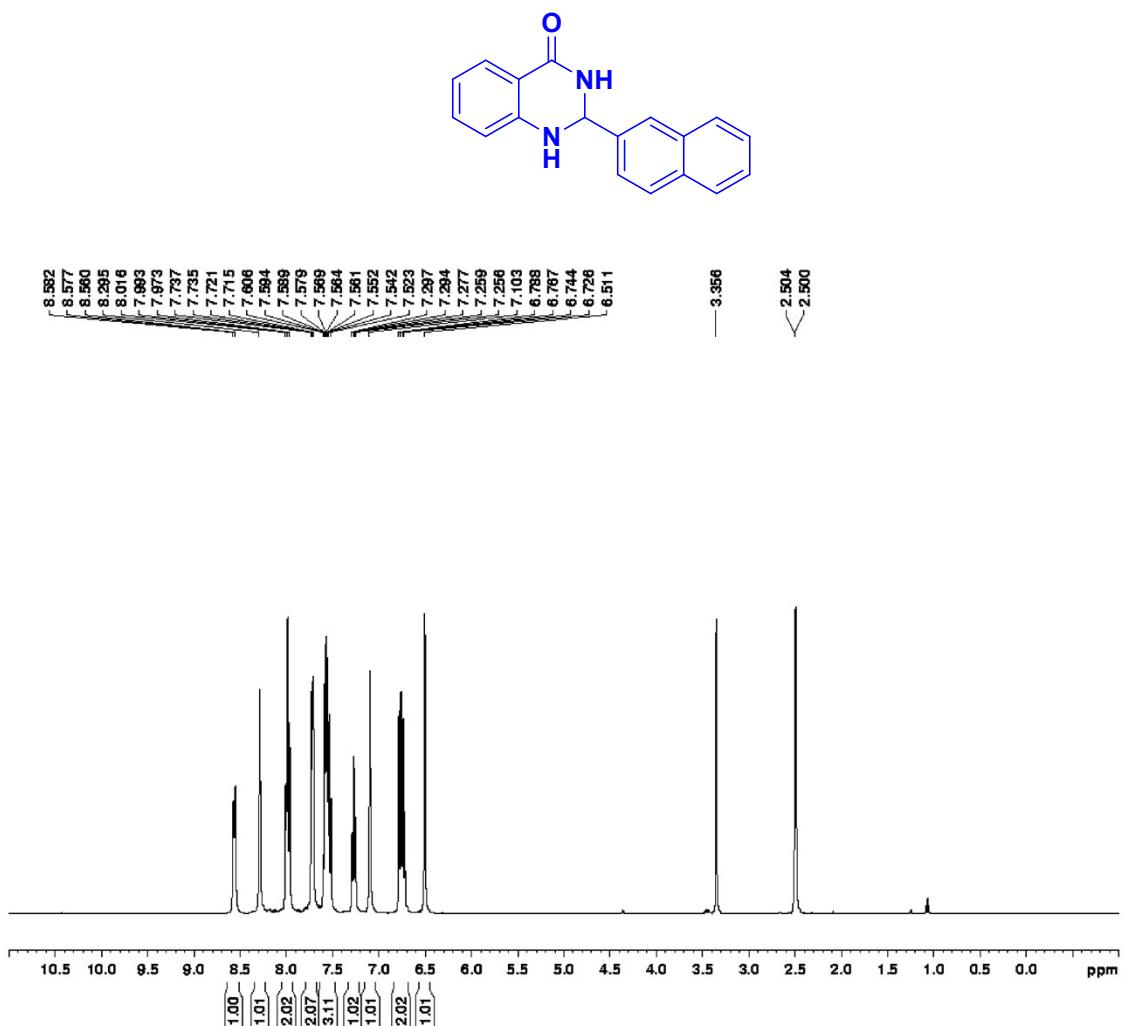


¹³C NMR spectrum of 4-(4-oxo-1,2,3,4-tetrahydroquinazolin-2-yl)benzoic acid (Table 2, entry 12)



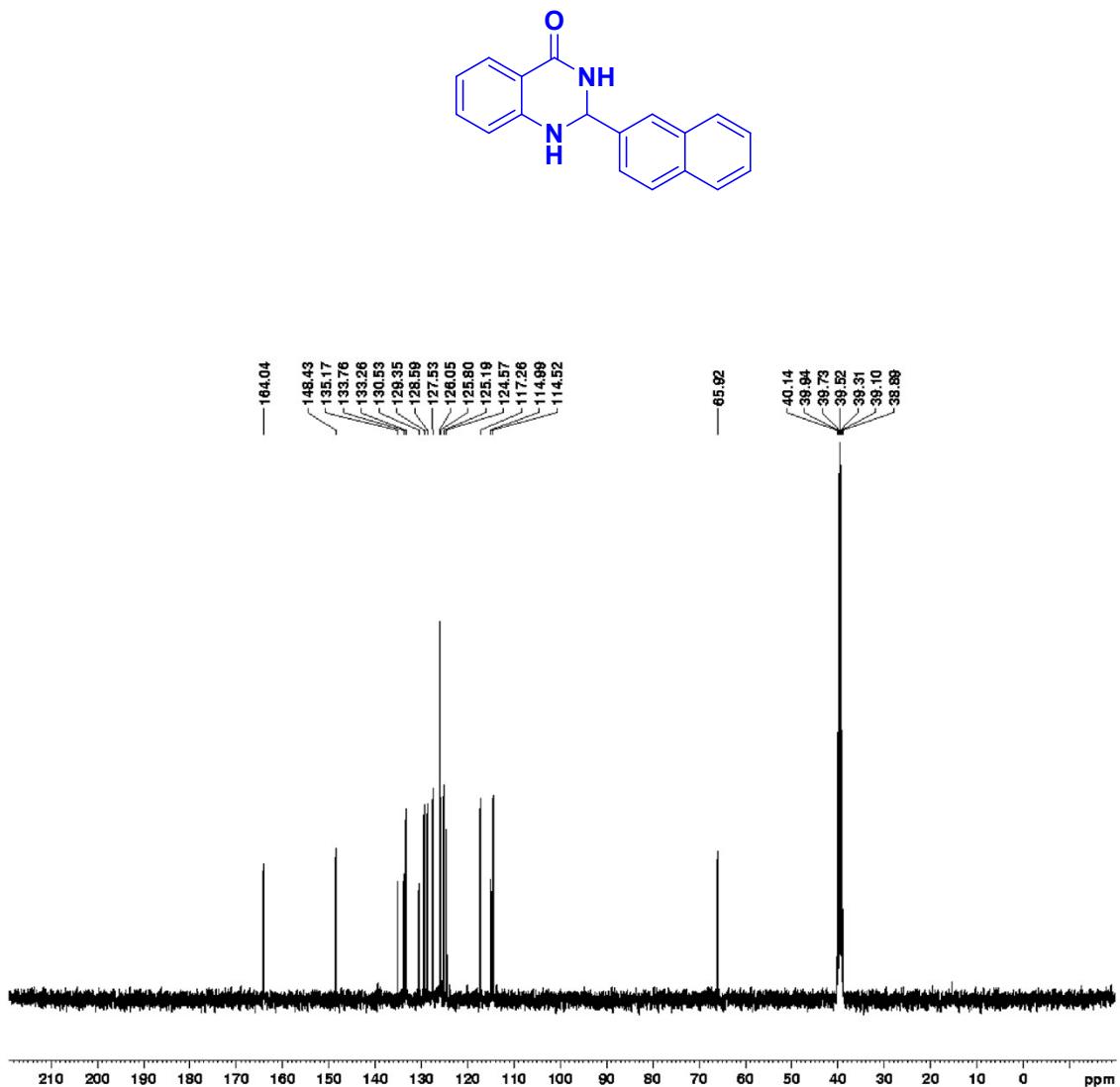
¹H NMR spectrum of 2-(naphthalen-2-yl)-2,3-dihydroquinazolin-4(1H)-one

(Table 2, entry 13)

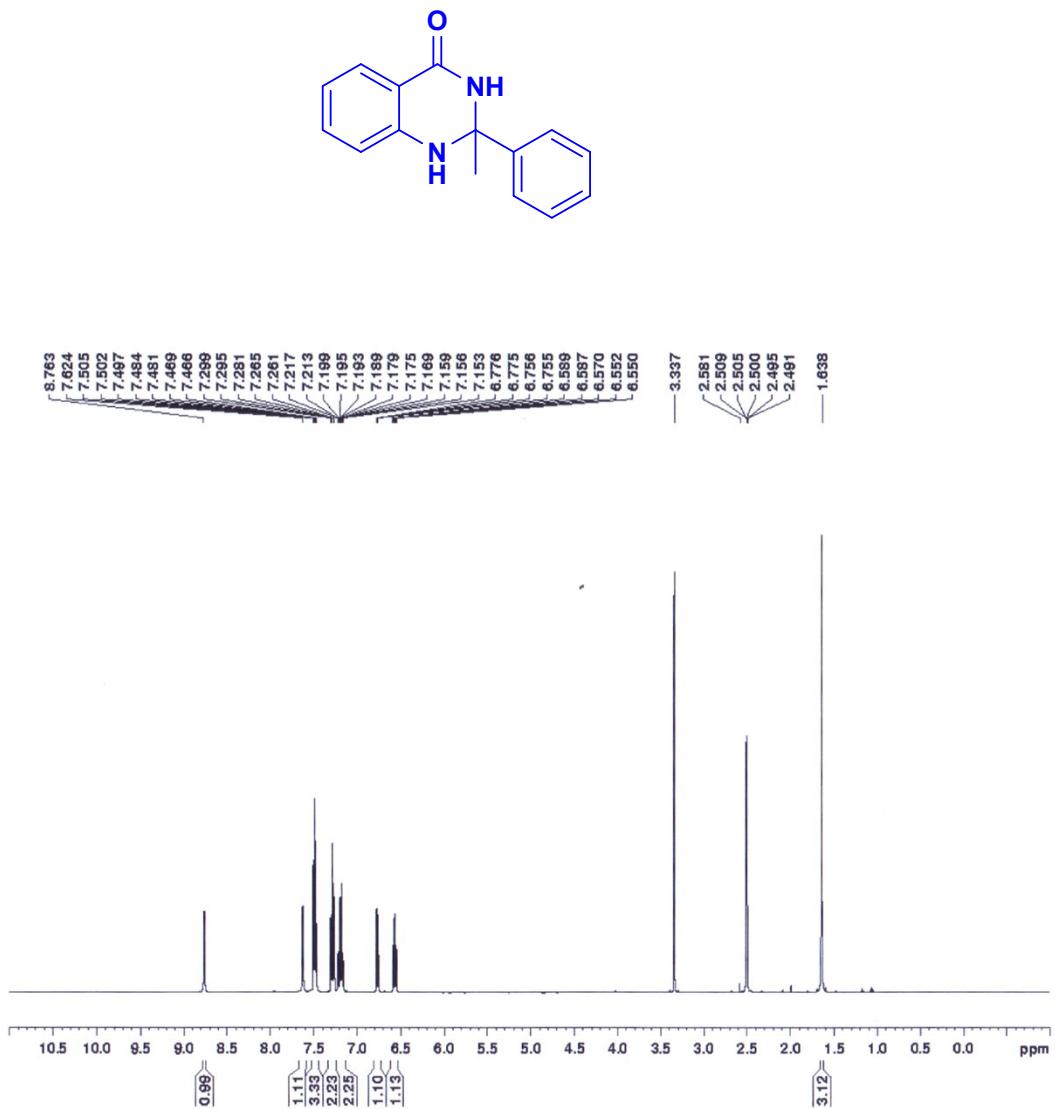


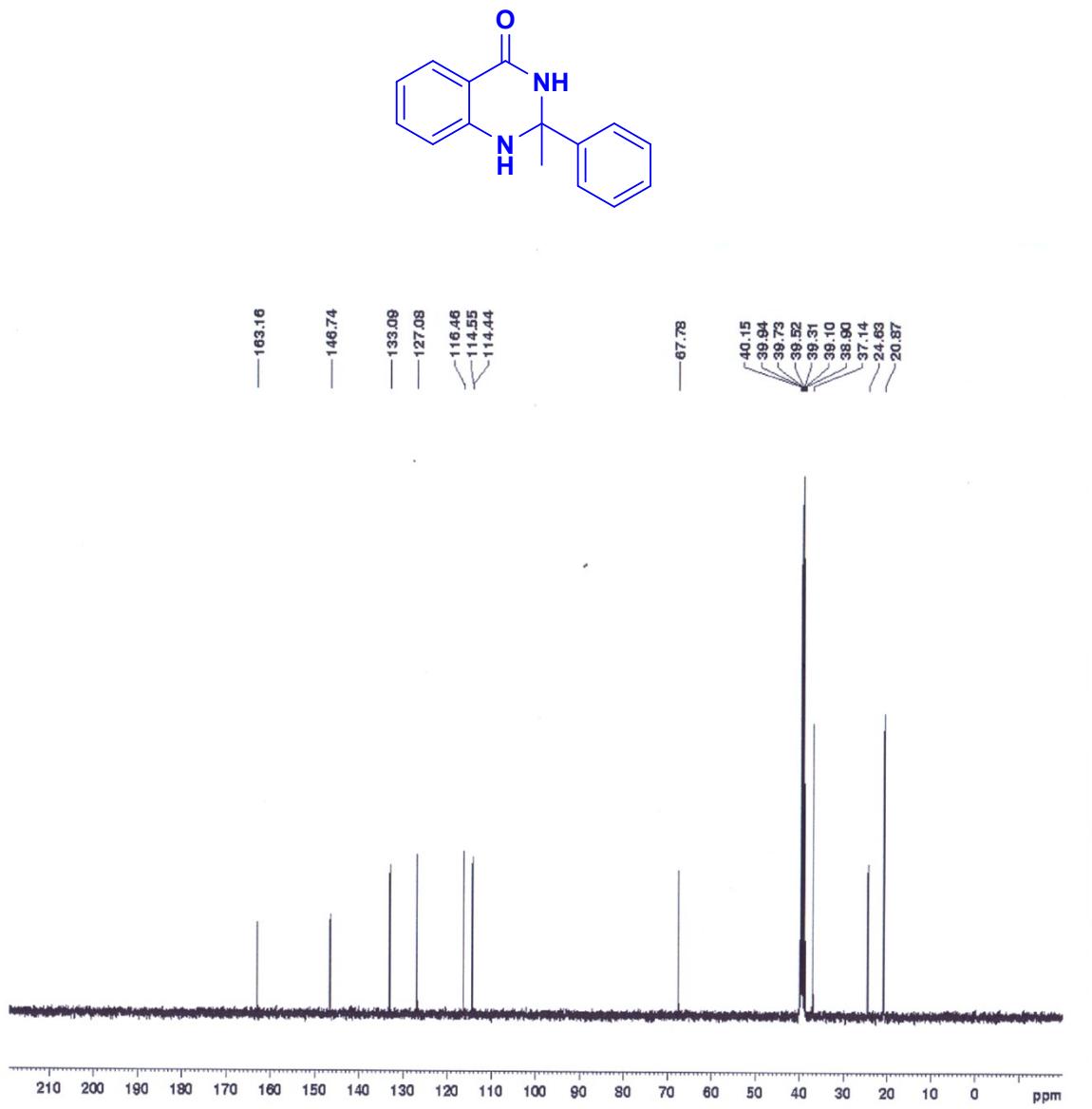
¹³C NMR spectrum of 2-(naphthalen-2-yl)-2,3-dihydroquinazolin-4(1H)-one

(Table 2, entry 13)

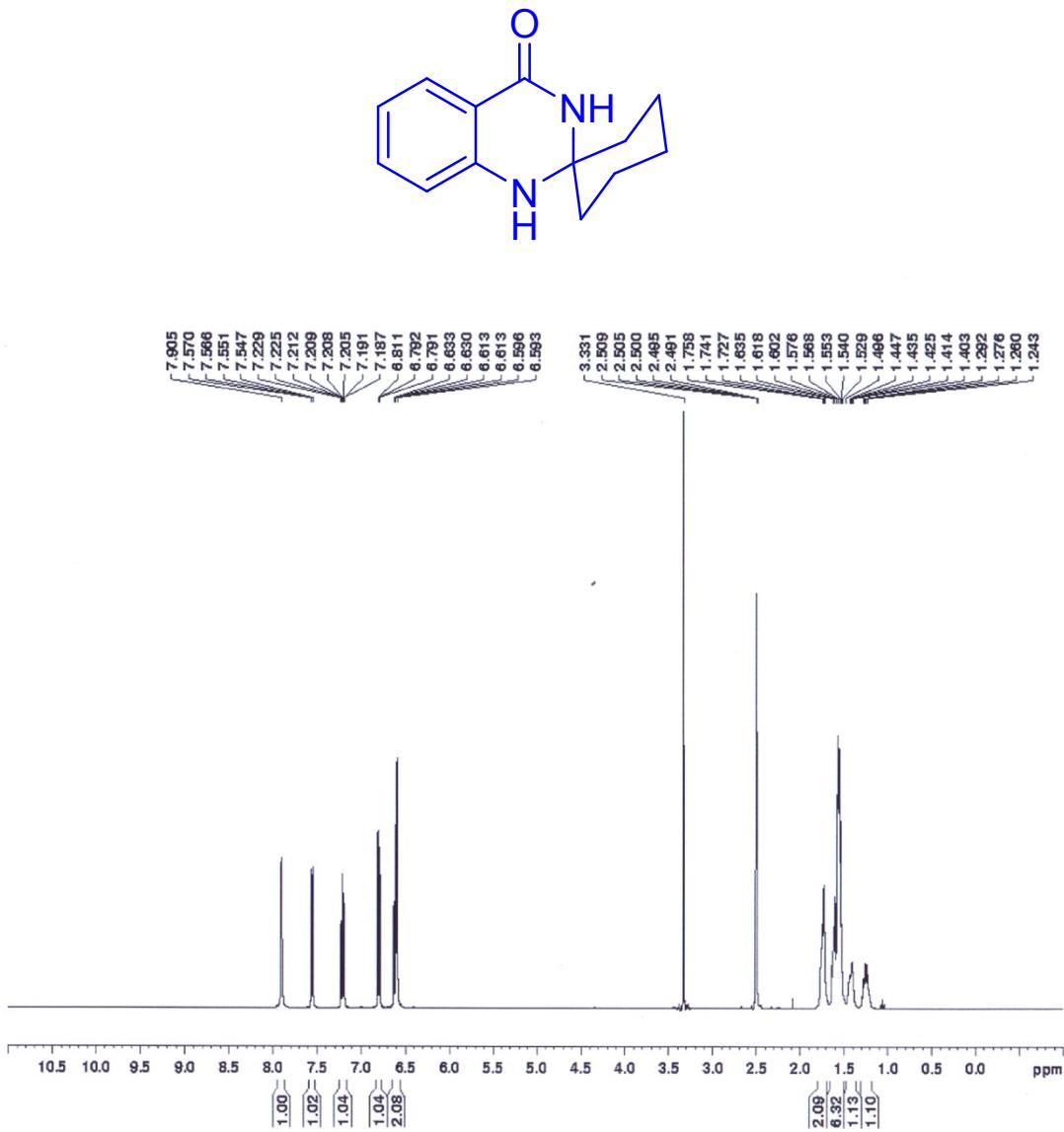


^1H NMR spectrum 2-methyl-2-phenyl-2,3-dihydroquinazolin-4(1H)-one (Table 3, entry 1)



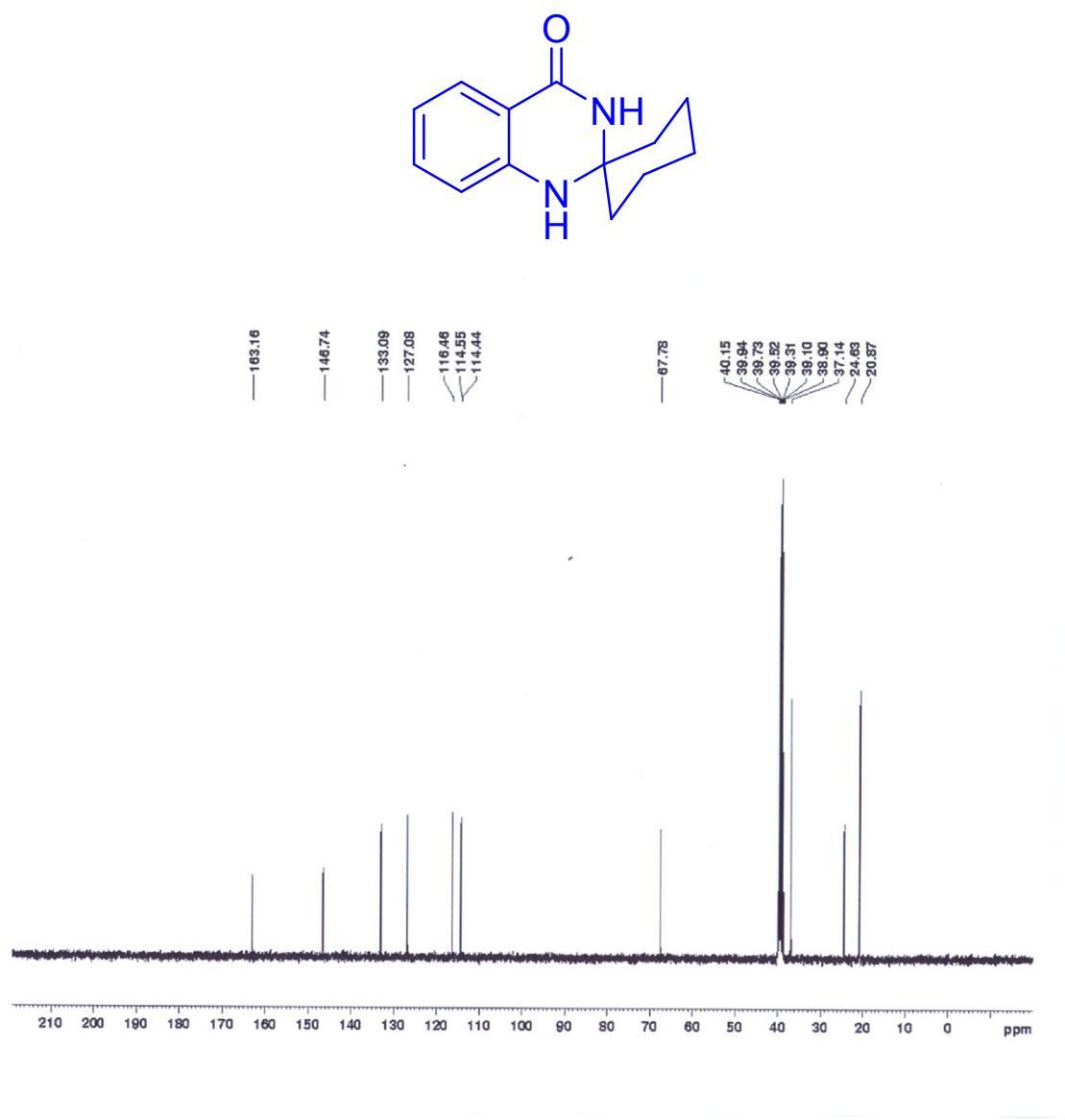


¹H NMR spectrum of 1'H-spiro[cyclohexane-1,2'-quinazolin]-4'(3'H)-one (Table 3, entry 8)



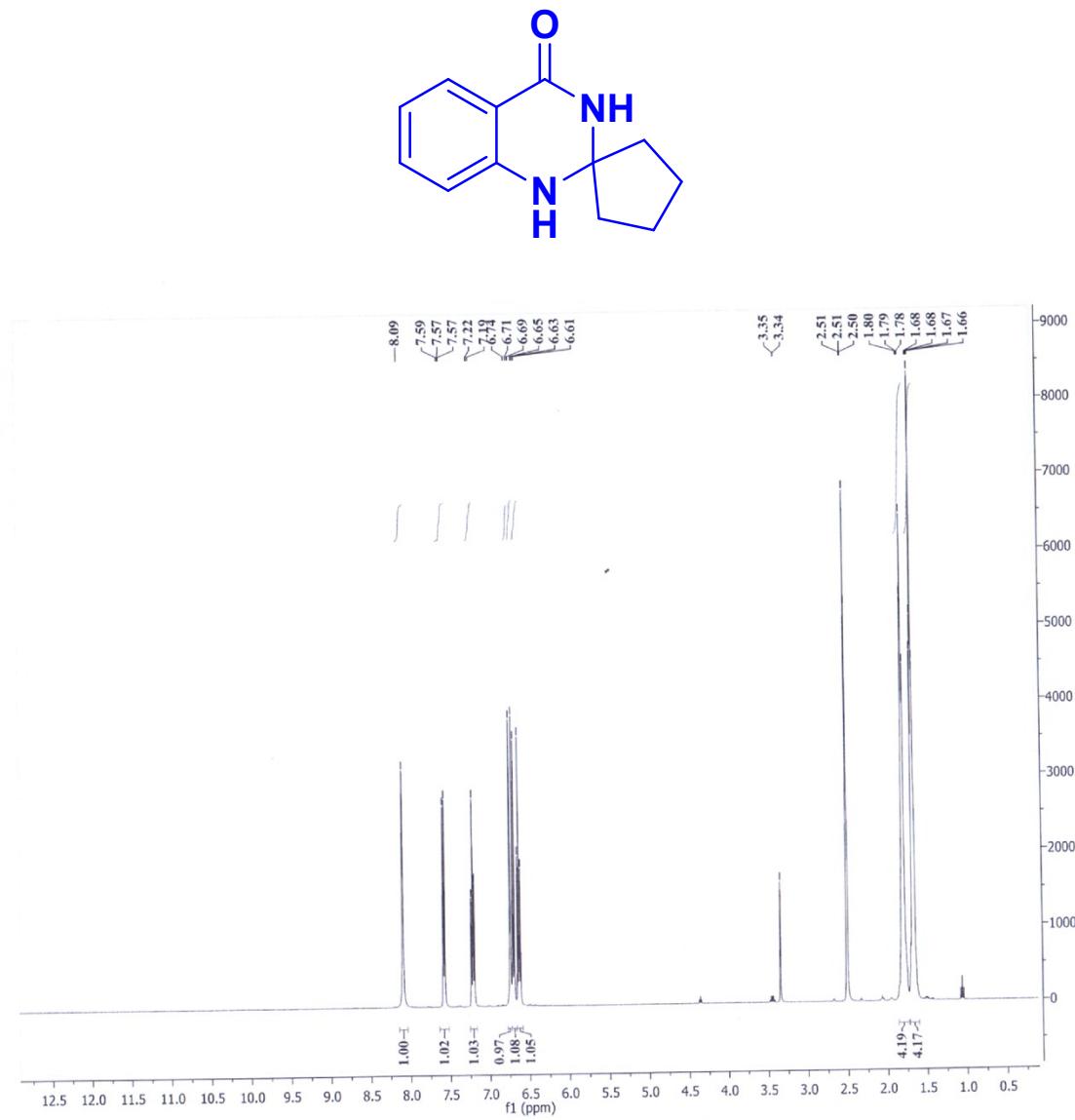
¹³C NMR spectrum of 1'H-spiro[cyclohexane-1,2'-quinazolin]-4'(3'H)-one

(Table 3, entry 8)



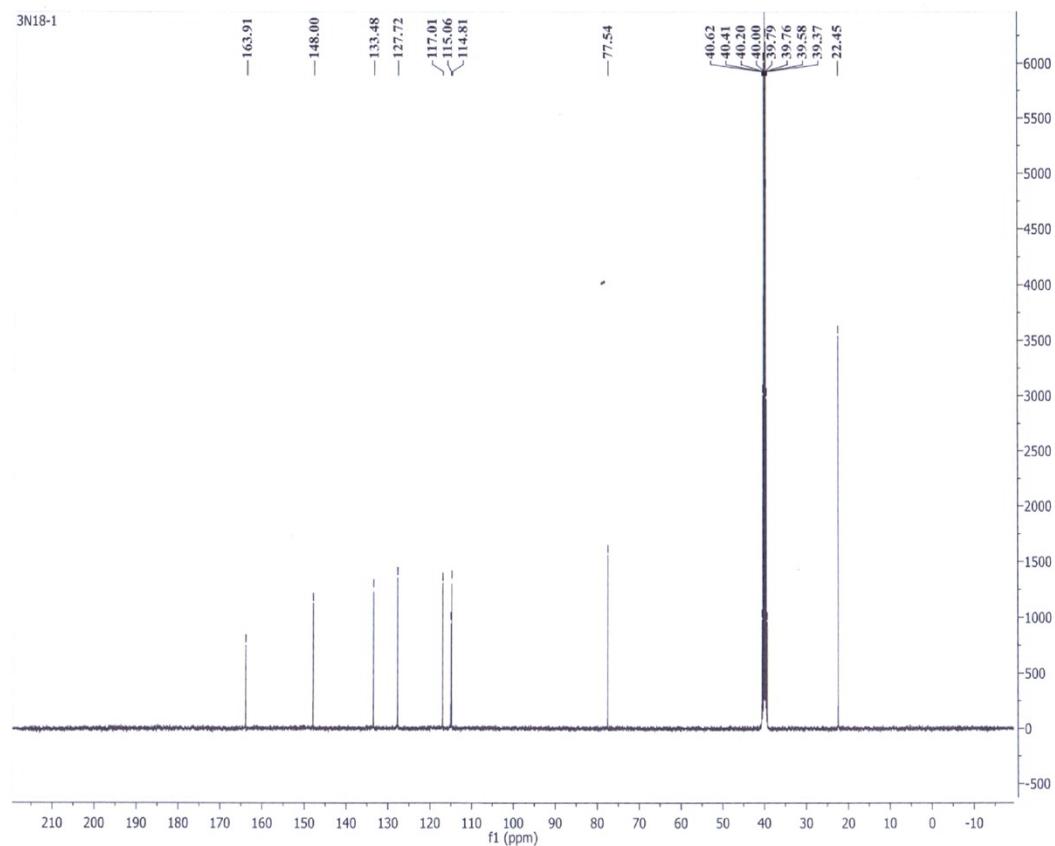
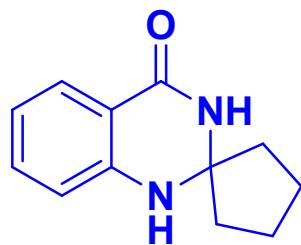
¹H NMR spectrum of 1'H-spiro[cyclopentane-1,2'-quinazolin]-4'(3'H)-one

(Table 3, entry 9)



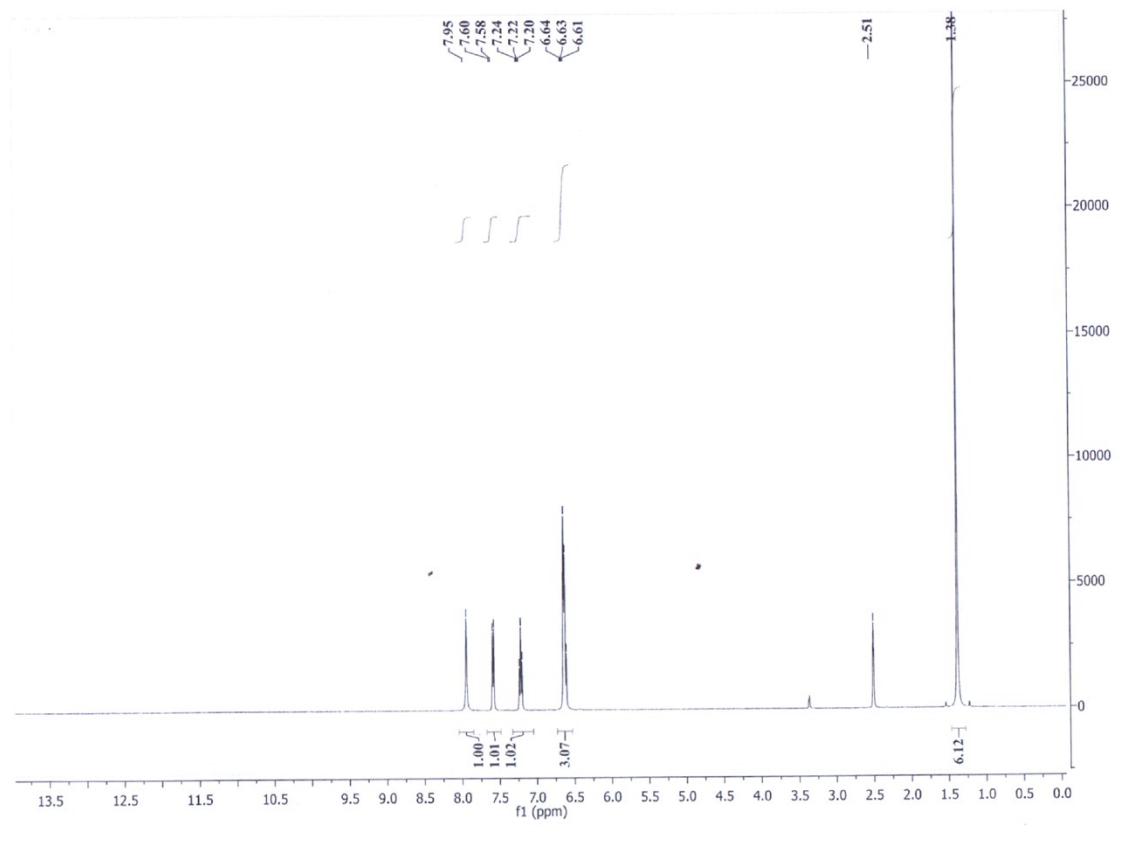
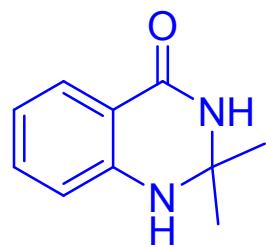
¹³C NMR spectrum of 1'H-spiro[cyclopentane-1,2'-quinazolin]-4'(3'H)-one

(Table 3, entry 9)



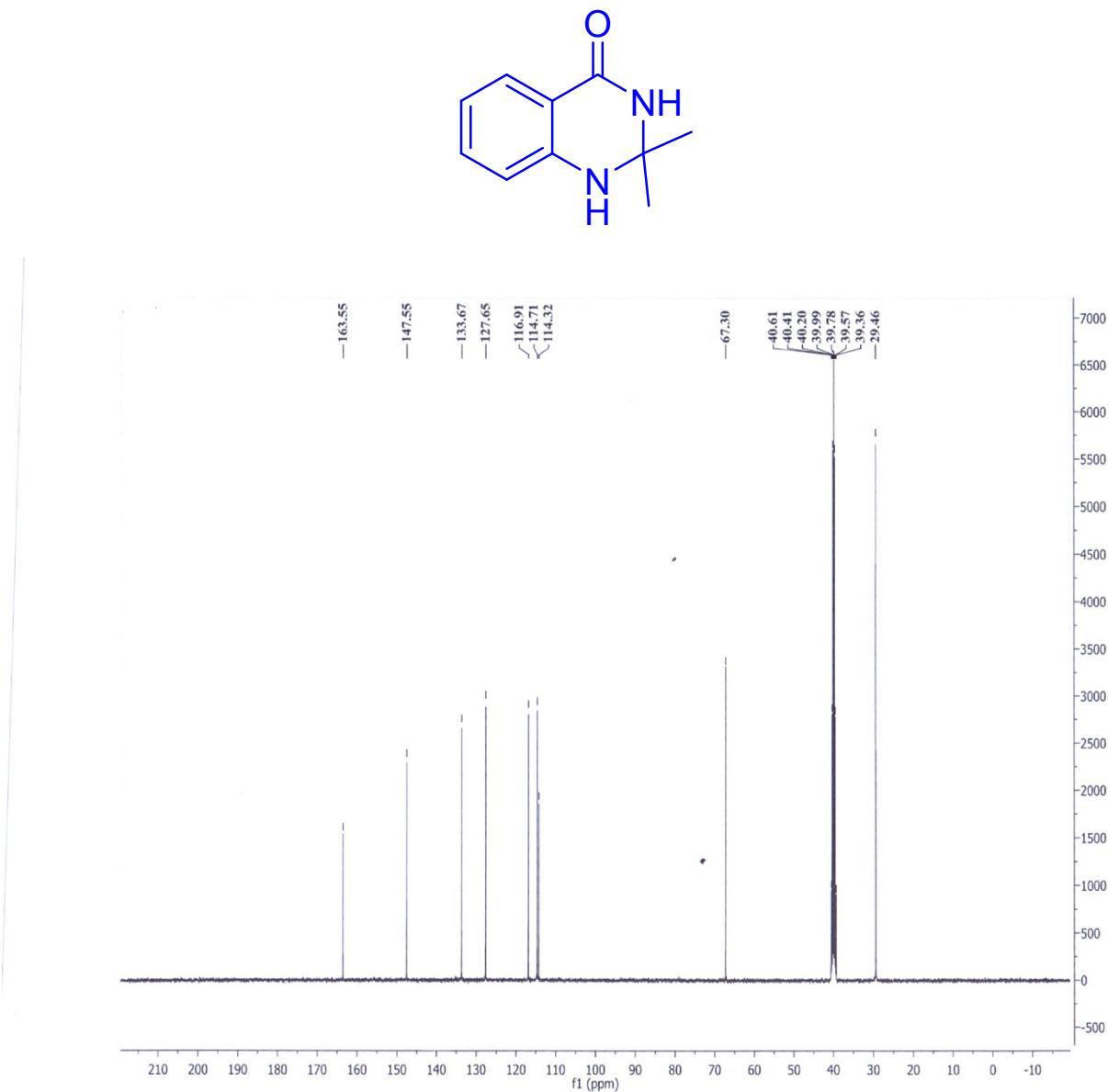
¹H NMR spectrum of 2,2-dimethyl-2,3-dihydroquinazolin-4(1H)-one:

(Table 3, entry 10)



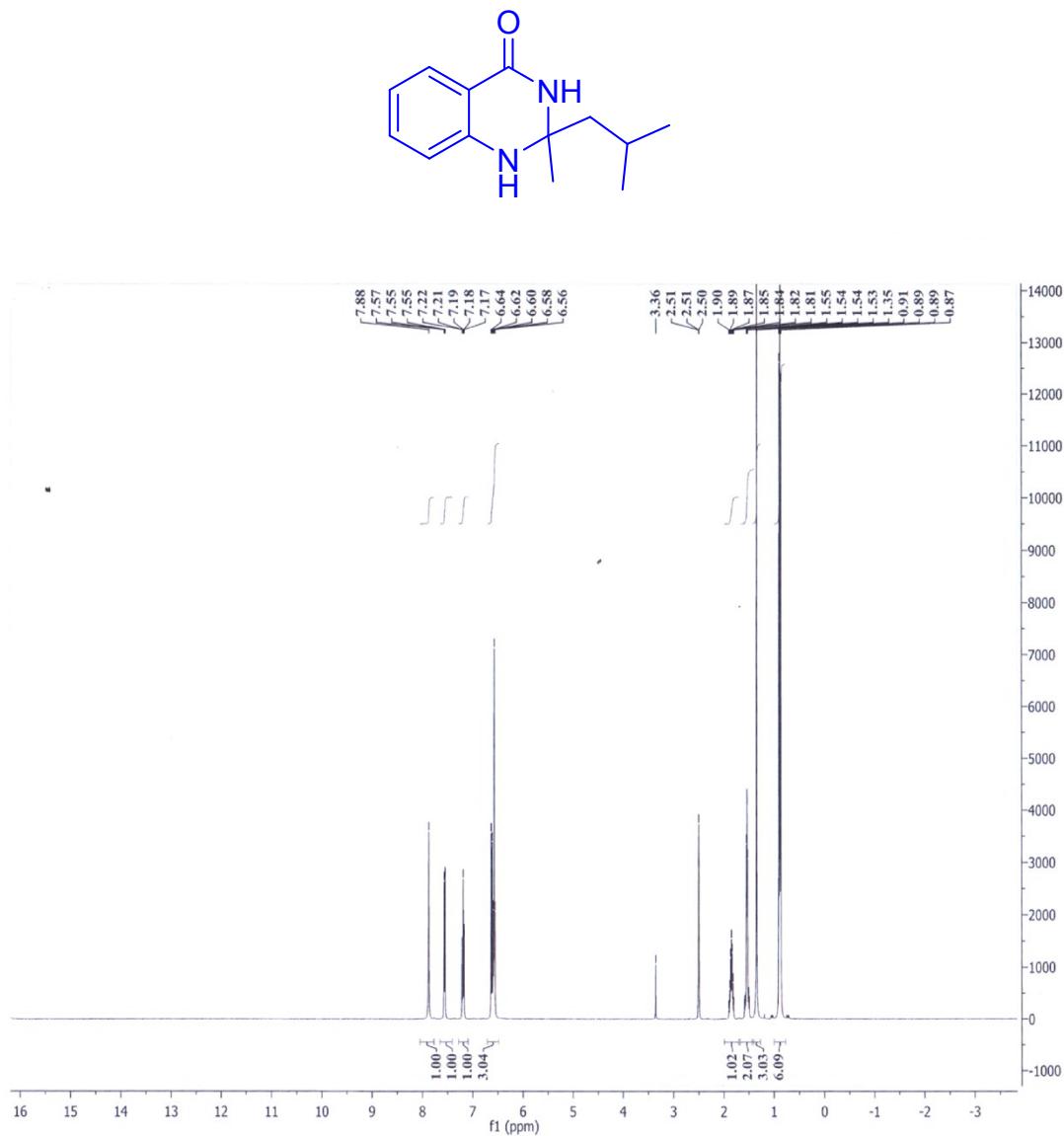
¹³C NMR spectrum 2,2-dimethyl-2,3-dihydroquinazolin-4(1H)-one:

(Table 3, entry 10)



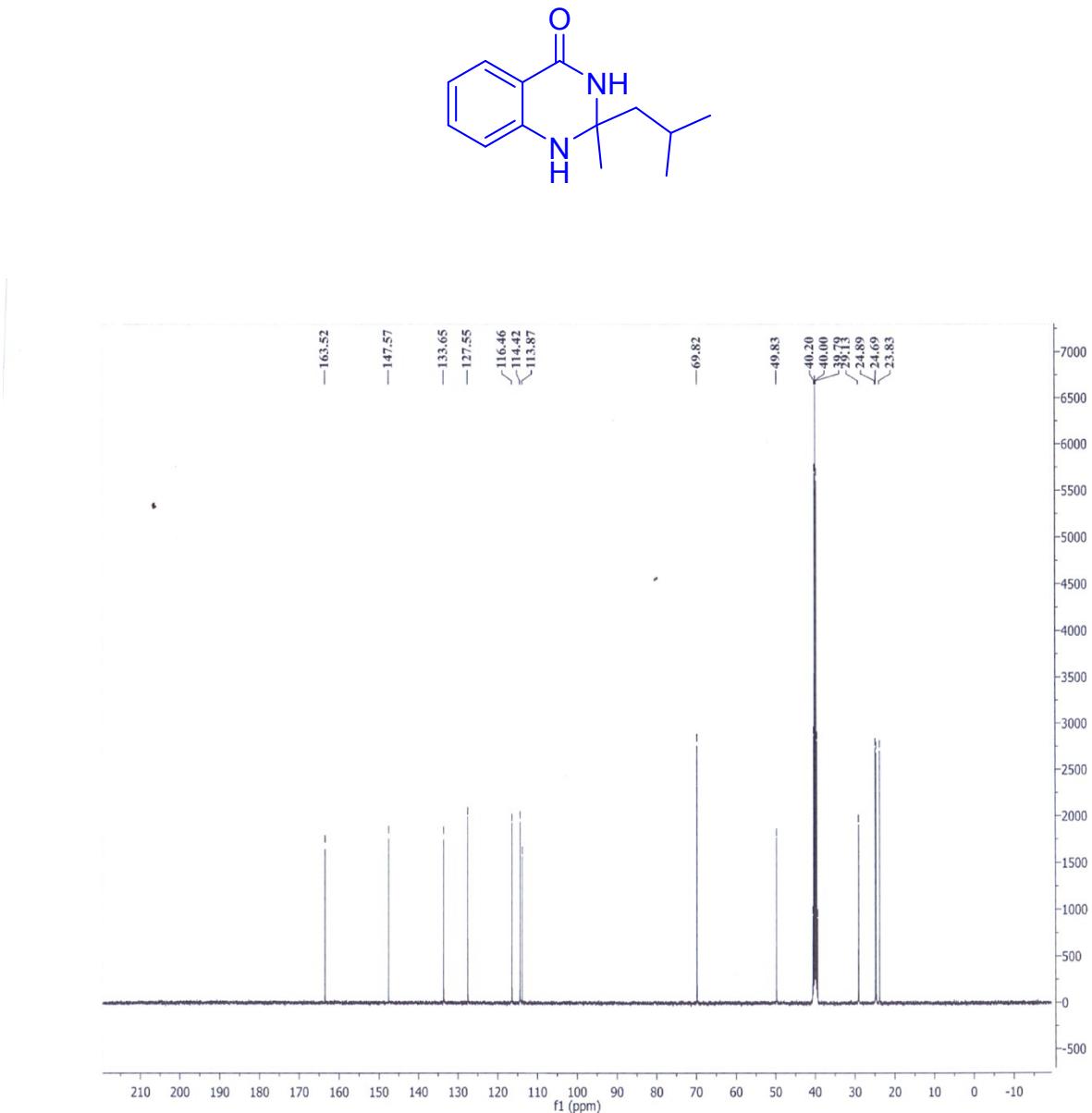
¹H NMR spectrum of 2-isobutyl-2-methyl-2,3-dihydroquinazolin-4(1H)-one:

(Table 3, entry 11)



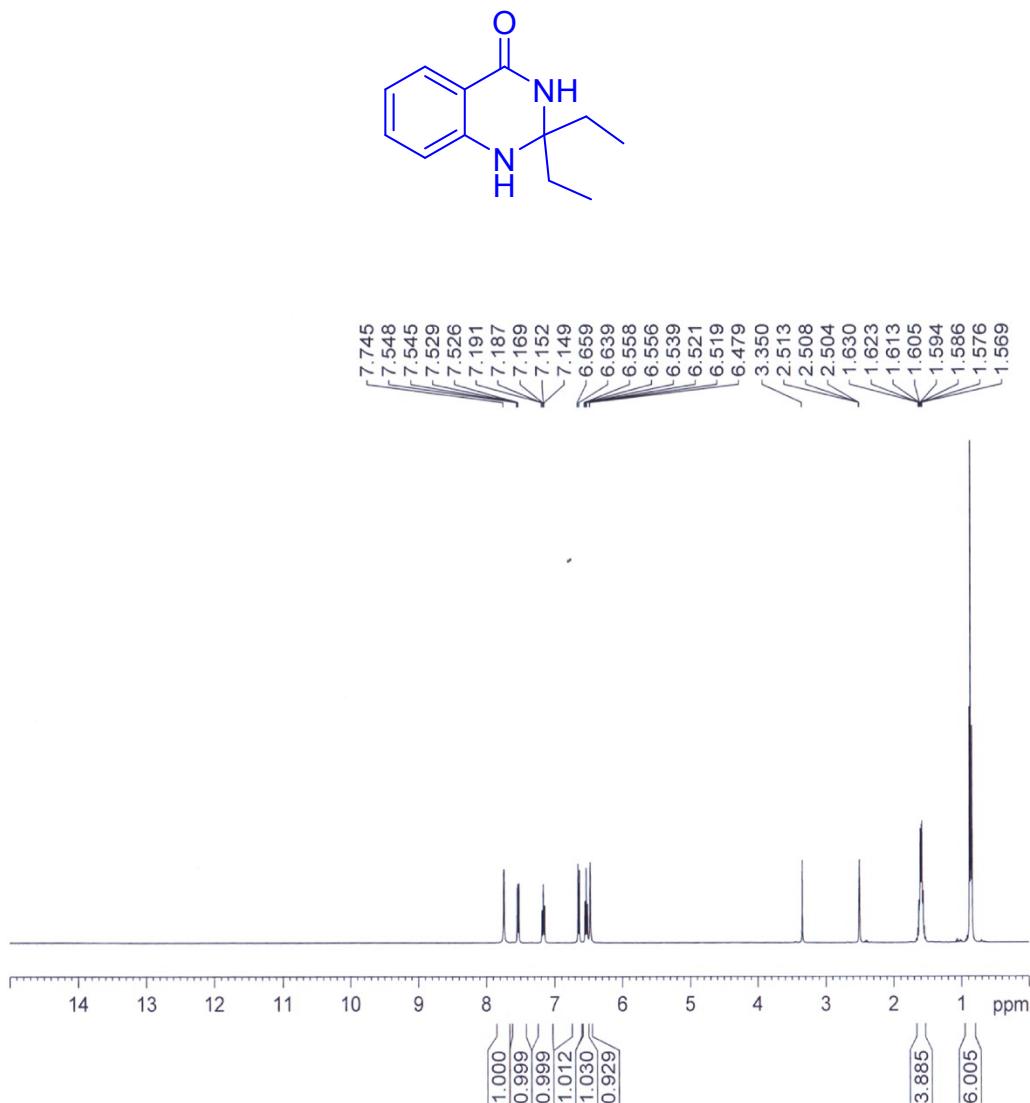
^{13}C NMR spectrum of 2-isobutyl-2-methyl-2,3-dihydroquinazolin-4(1H)-one:

(Table 3, entry 11)



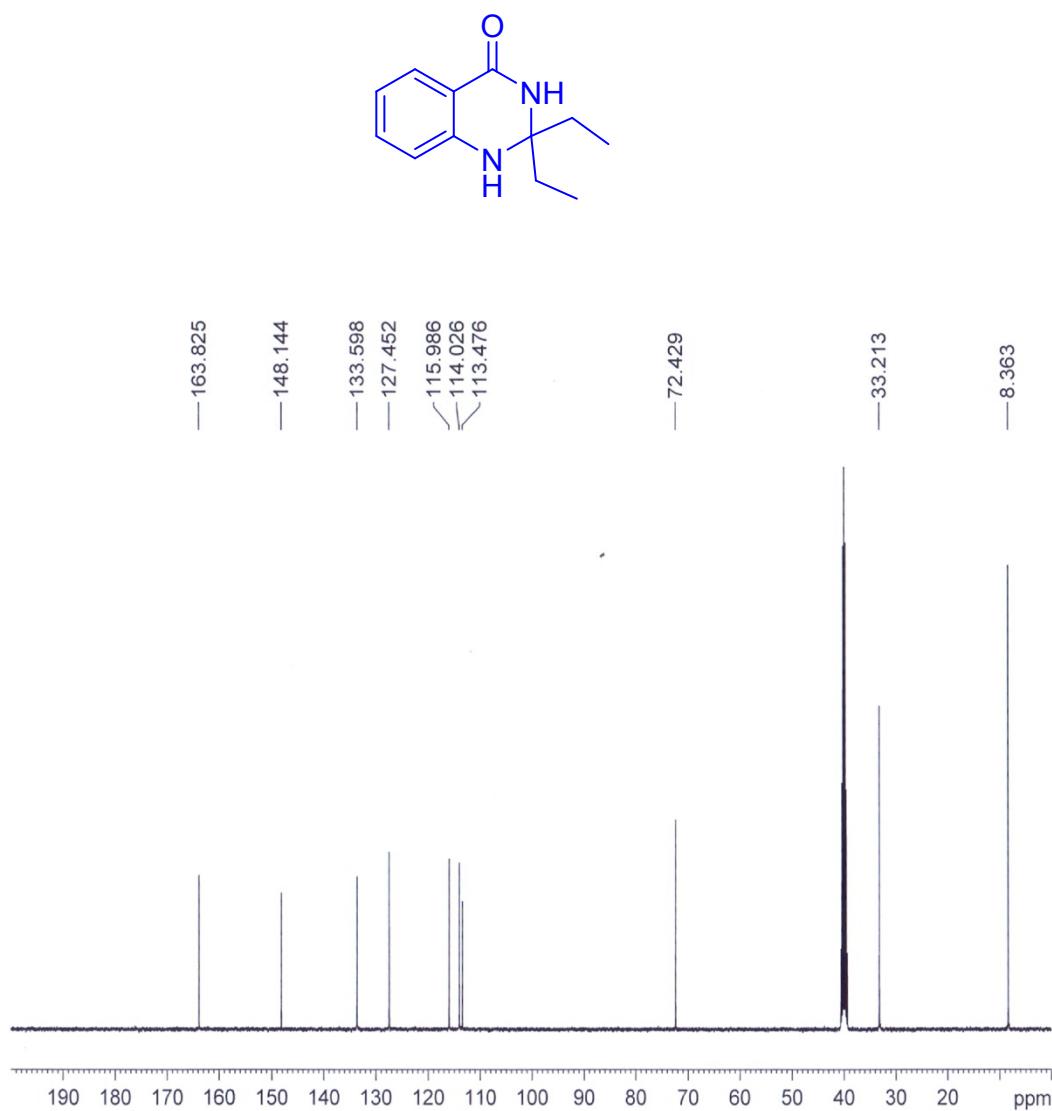
¹H NMR spectrum of 2, 2-diethyl-2,3-dihydroquinazolin-4(1H)-one

(Table 3, entry 12)

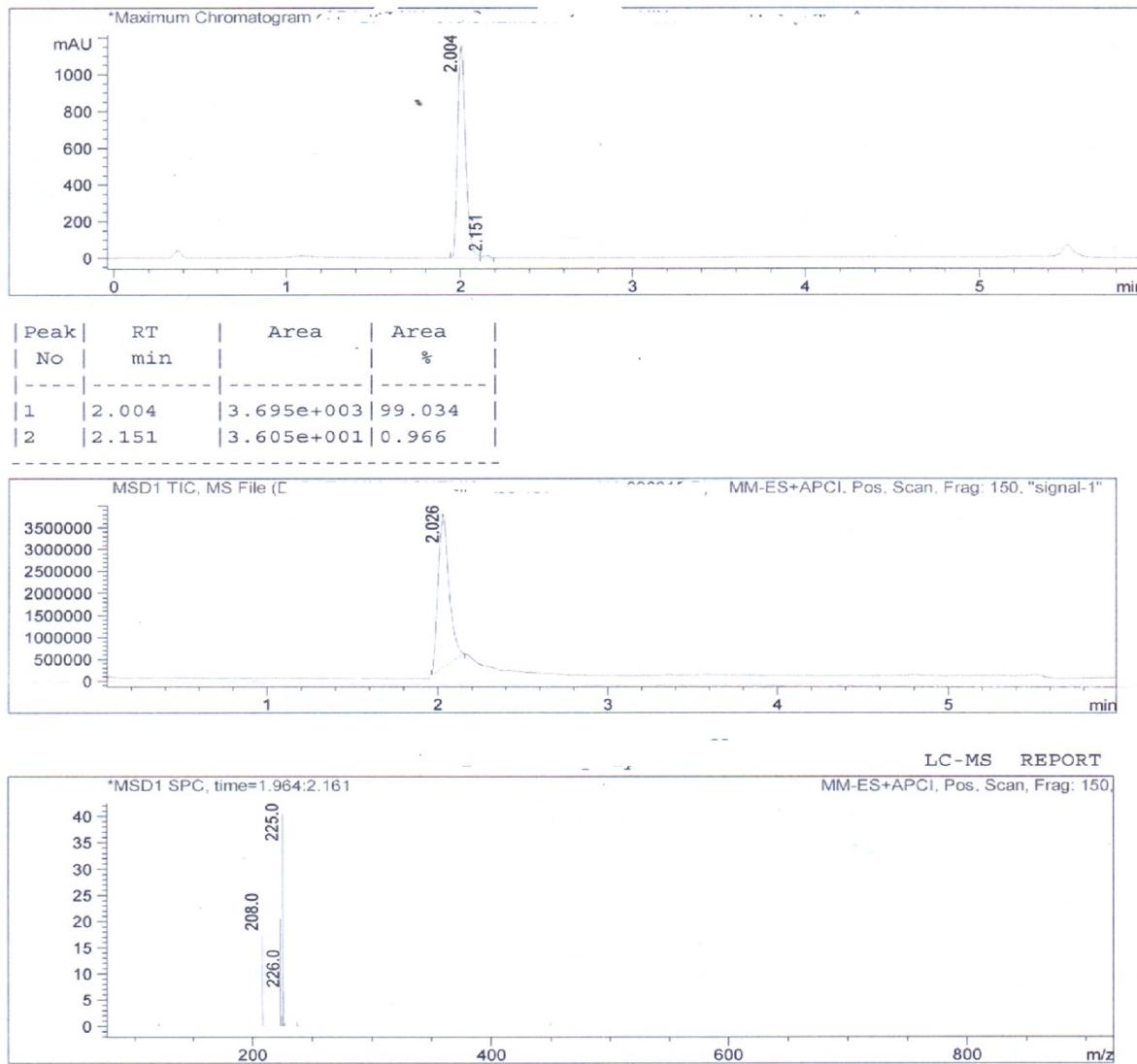
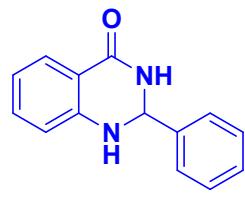


¹³C NMR spectrum of 2, 2-diethyl-2,3-dihydroquinazolin-4(1H)-one

(Table 3, entry 12)

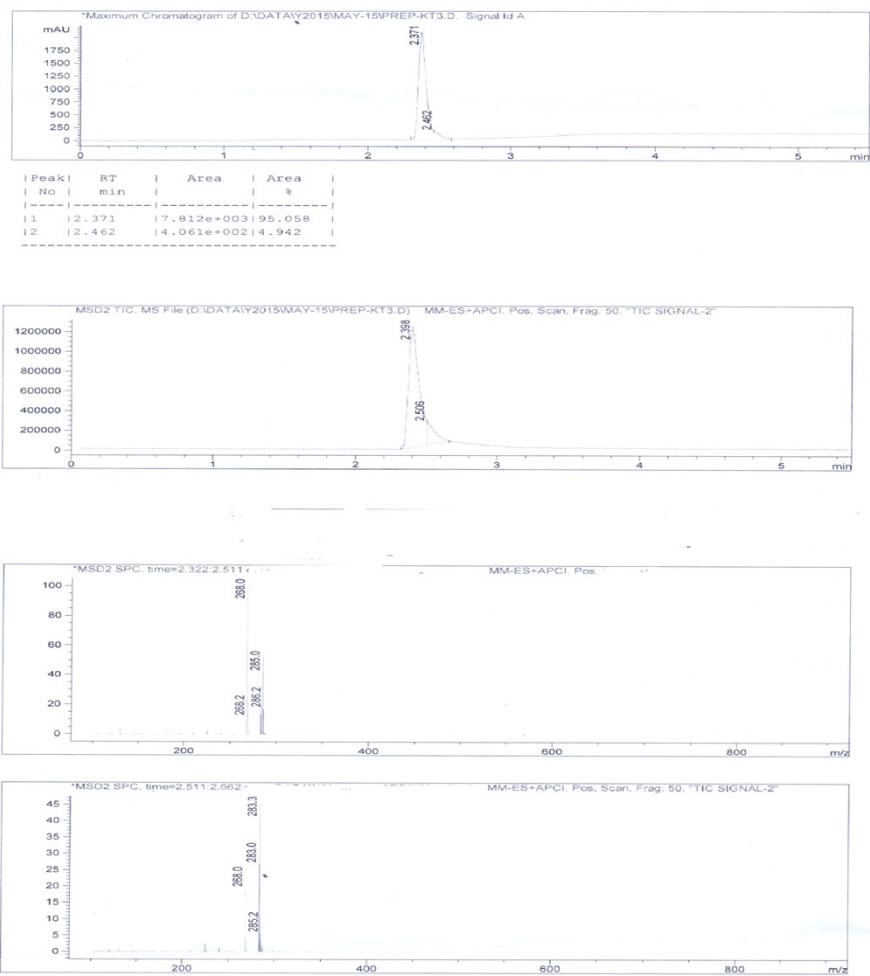
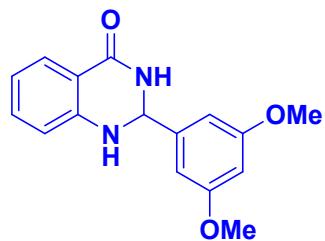


LC-Mass spectrum of 2-phenyl-2,3-dihydroquinazolin-4(1H)-one (Table 2, entry 1)

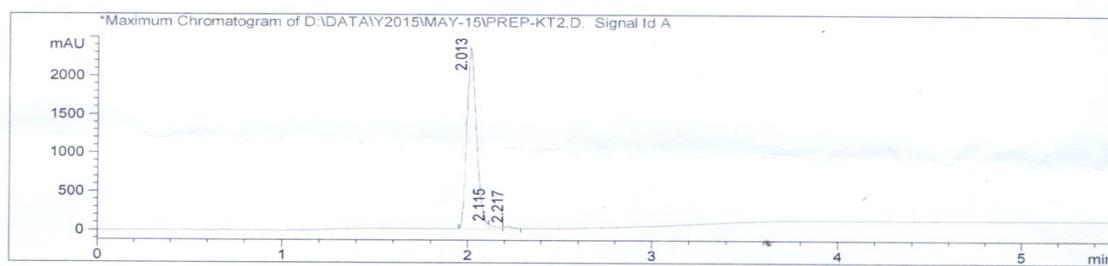
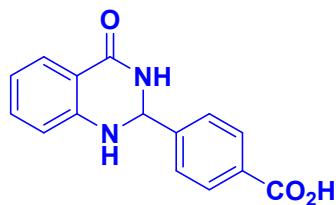


LC-Mass spectrum of 2-(3, 5-dimethoxyphenyl)-2,3-dihydroquinazolin-4(1H)-one

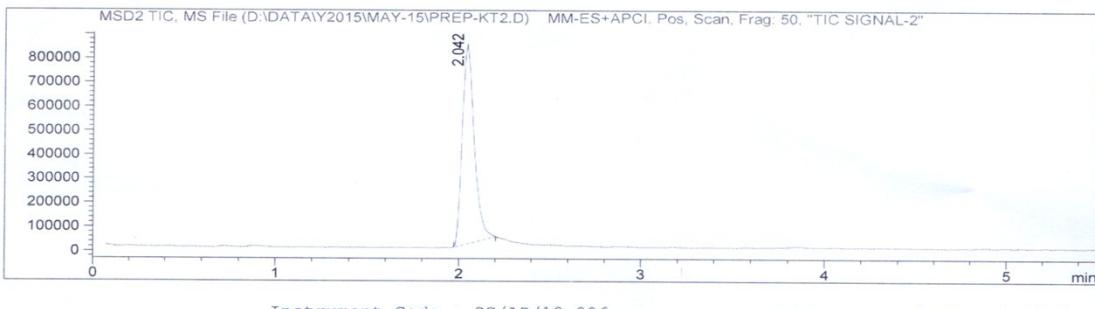
(Table 2, entry 4)



**LC-Mass spectrum of 4-(4-oxo-1,2,3,4-tetrahydroquinazolin-2-yl)benzoic acid
(Table 2, entry 12)**

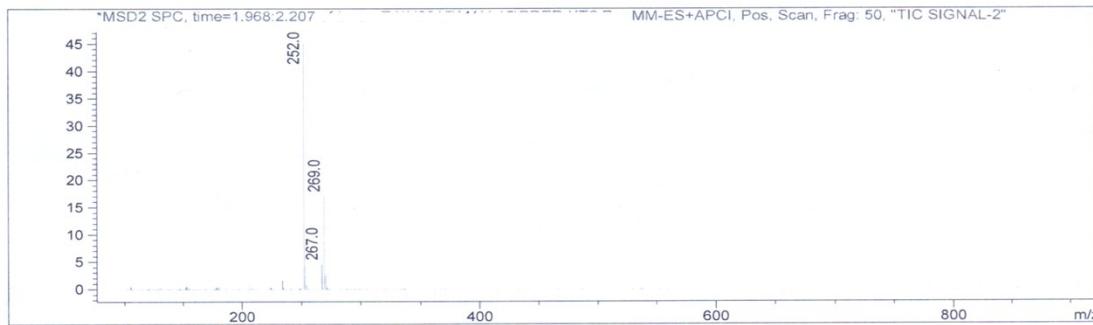


Peak	RT	Area	Area %
No	min		
1	2.013	8.609e+003	97.437
2	2.115	1.287e+002	1.456
3	2.217	9.778e+001	1.107

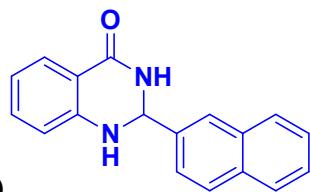


Instrument Code : SC/AD/10-006

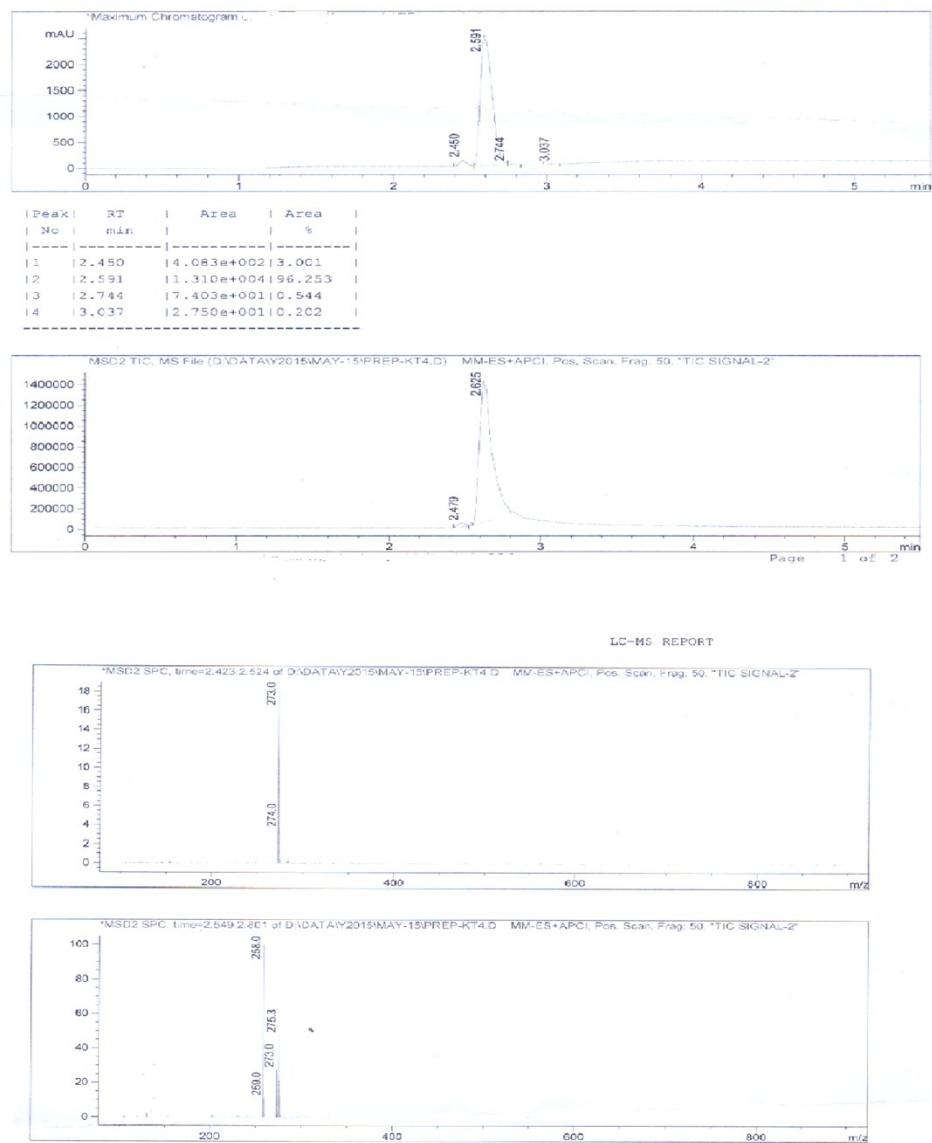
Page 1 of 2



LC-Mass spectrum of 2-(naphthalen-2-yl)-2, 3-dihydroquinazolin-4(1H)-one

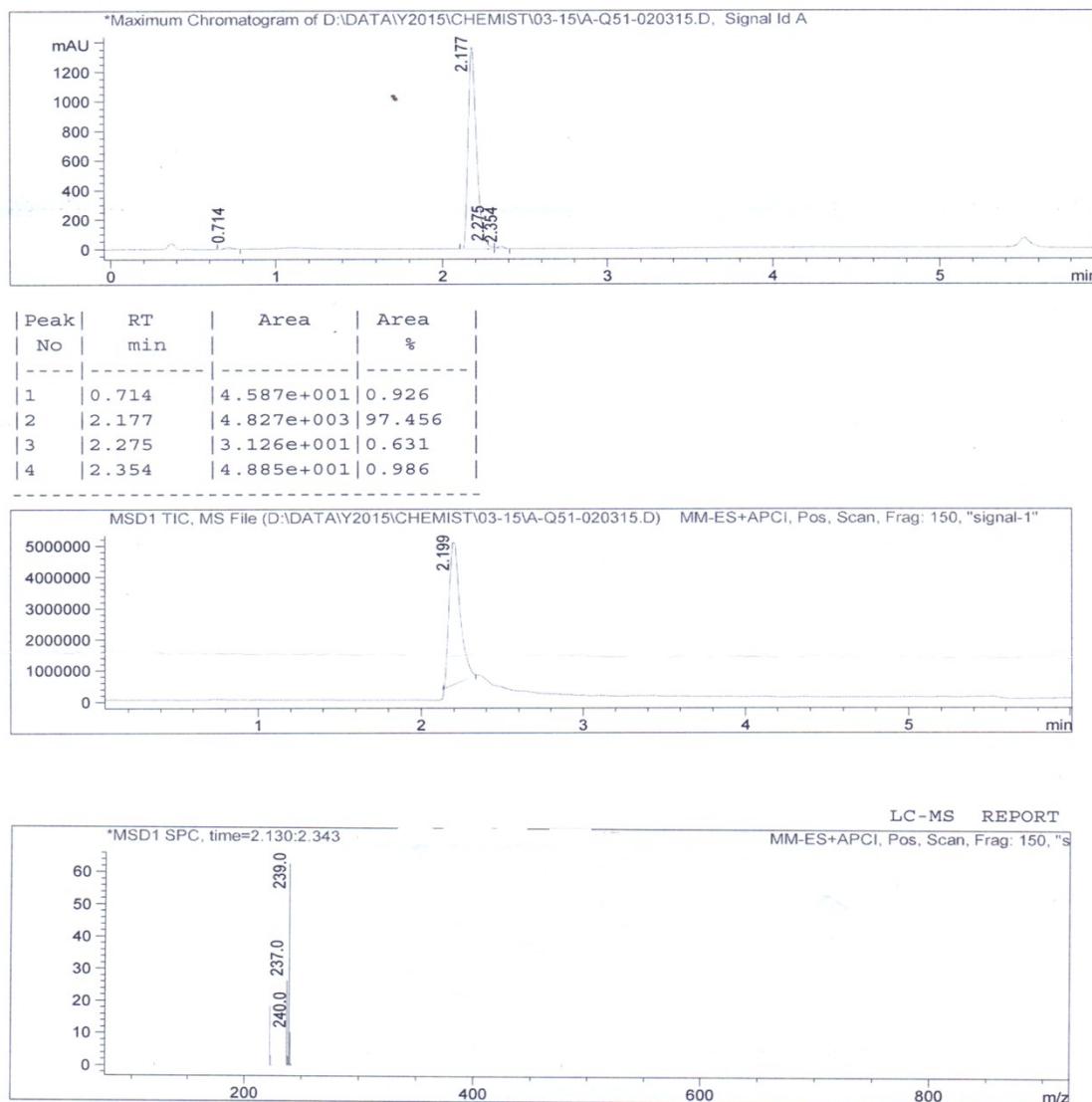
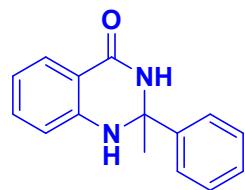


(Table 2, entry 13)



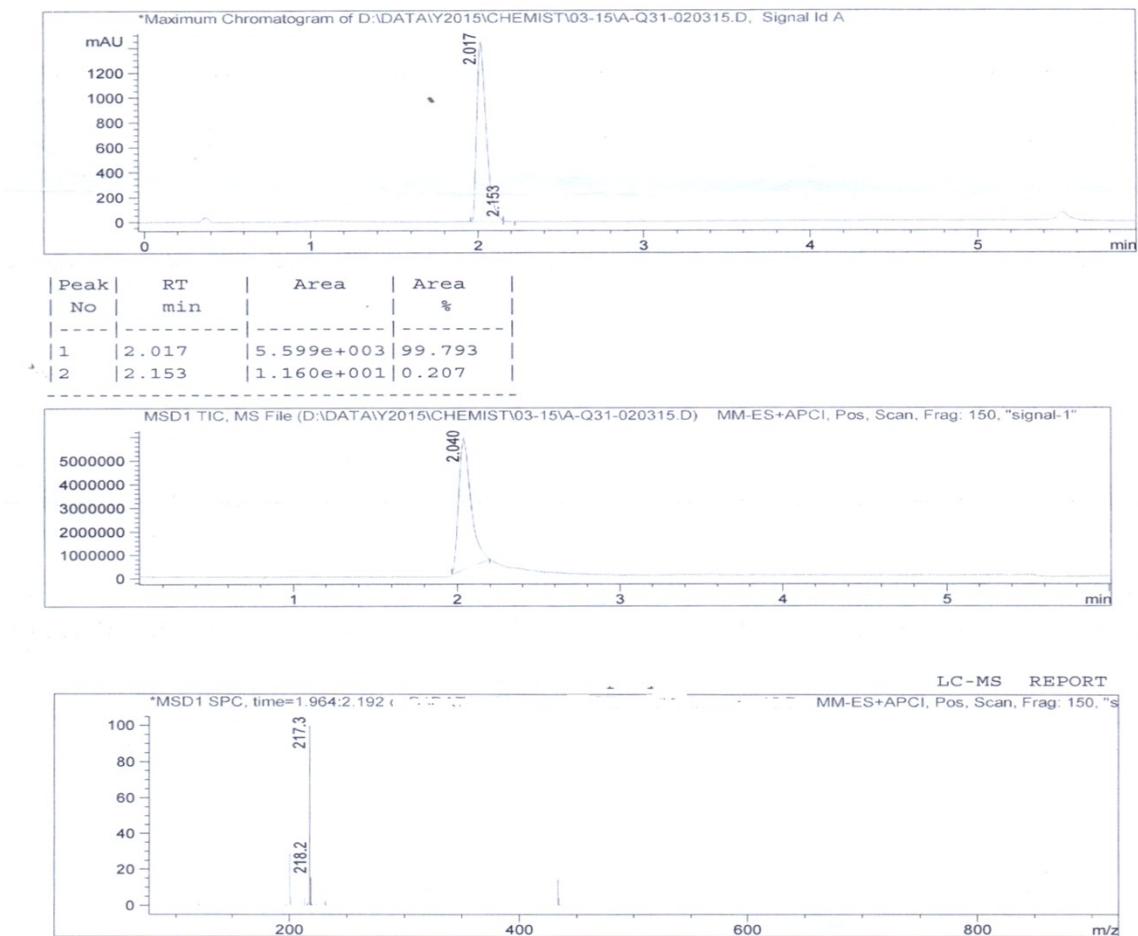
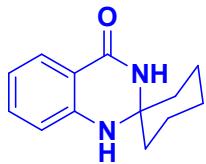
LC-Mass spectrum of 2-methyl-2-phenyl-2, 3-dihydroquinazolin-4(1H)-one

(Table 3, entry 1)



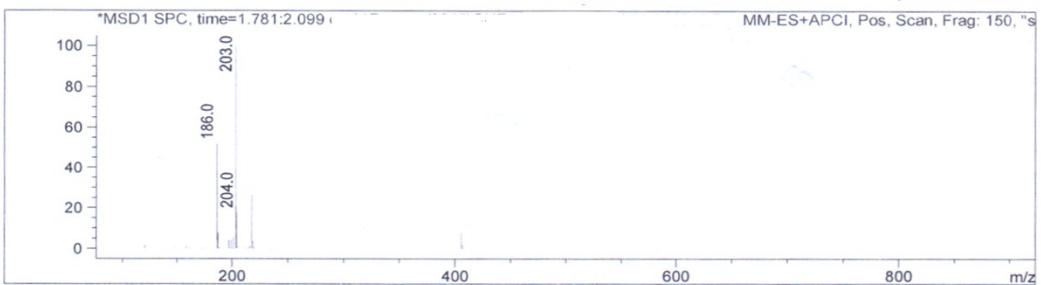
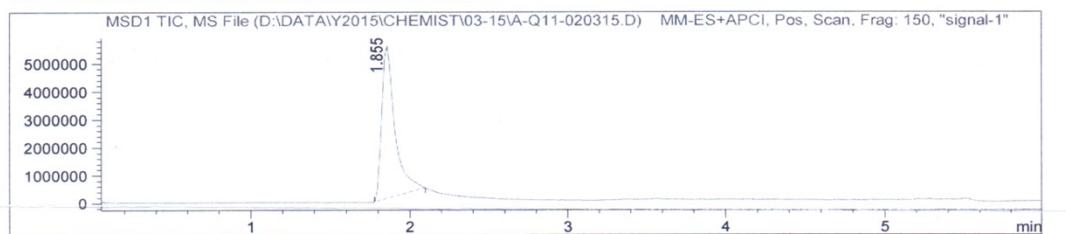
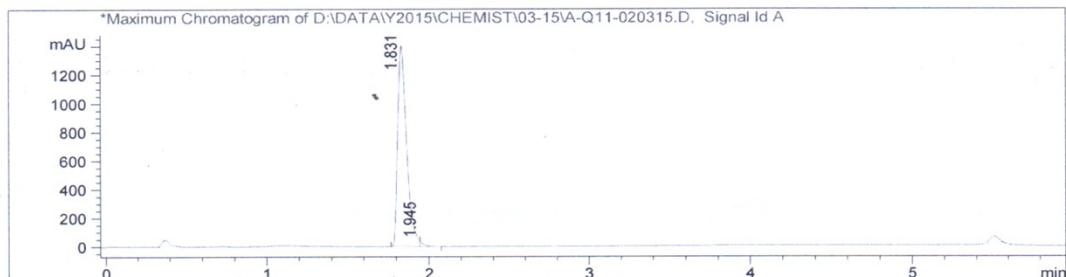
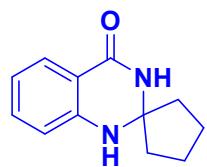
LC-Mass spectrum of 1'H-spiro[cyclohexane-1,2'-quinazolin]-4'(3'H)-one

(Table 3, entry 8)



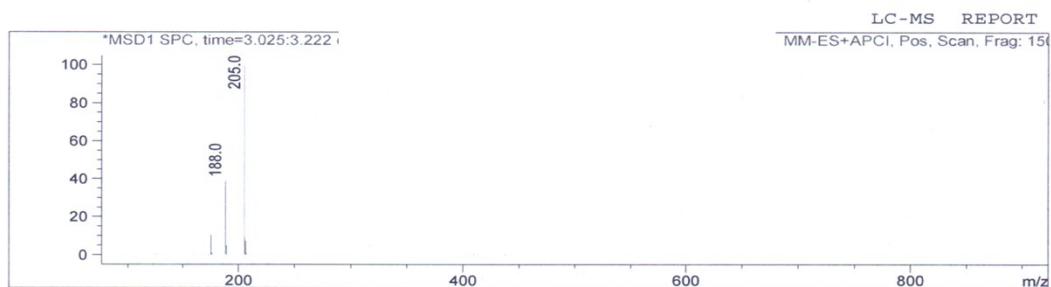
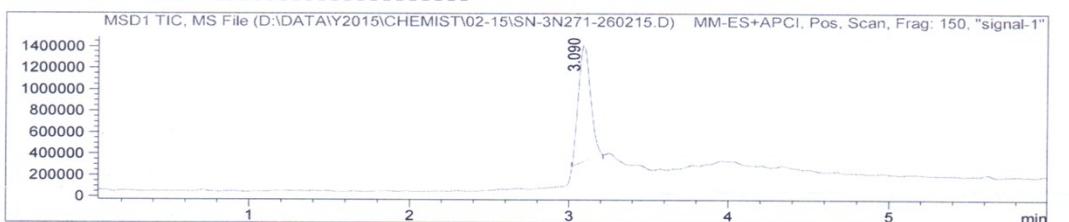
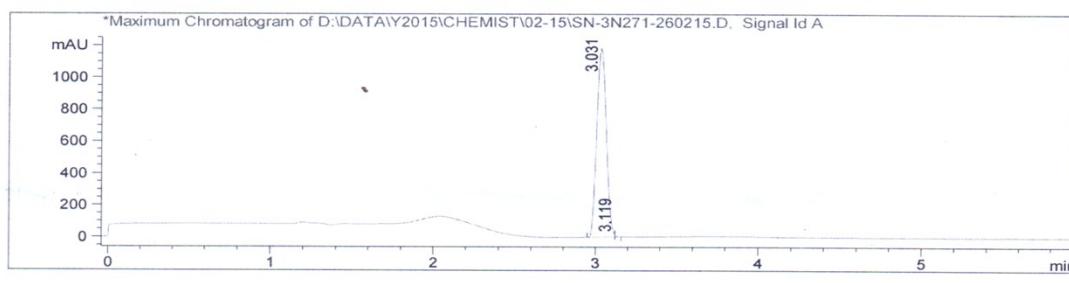
LC-Mass spectrum of 1'H-spiro[cyclopentane-1,2'-quinazolin]-4'(3'H)-one

(Table 3, entry 9)



LC-Mass spectrum of 2,2-diethyl-2,3-dihydroquinazolin-4(1H)-one

(Table 3, entry 12)



- 1) M. Sharma, S. Pandey, K. Chauhan, D. Sharma, B. Kumar, P. M. Chauhan J. Org .Chem. 77 (2012) 929
- 2) X.-S. Wang, K. Yang, J. Zhou, S.-J. Tu, J. Comb. Chem. 12 (2010) 417
- 3) A. Shaabani, A. Maleki, H. Mofakham, Synth. Commun 38 (2008) 3751
- 4) Shi, Daqing; Journal of Heterocyclic Chemistry 42 (2005) 173