Electronic Supplementary Material (ESI) for RSC Advances.

A reaction of 1,2-diamines and aldehydes with silyl cyanide as cyanide pronucleophile to access 2-aminopyrazines and 2-aminoquinoxalines

Sankar K. Guchhait,*[,a] Garima Priyadarshani, [a,†] Nikhil M. Gulghane [a,†]

[a]*Department of Medicinal Chemistry, National Institute of Pharmaceutical Education and Research (NIPER), S. A. S. Nagar (Mohali) - 160062, Punjab, India, Fax: 91 (0)172 2214692; Tel: 91 (0)172 2214683*

Email: skguchhait@niper.ac.in

Contents

Table S1: Properties of Bases Evaluated in the Optimization Study (Table 1)

Base	pK_{HB}^{+}	Features	Unique features of base
DABCO	8.72[1]	Caged tertiary diamine	
DBU	11.6[1]	Sterically hindered, tertiary amidine-diamine	Amidine-diamine functionality acting as basic/nulcleophilic atom centers along with steric hindrance represents a particular type of functional motif and renders the compound as a practical reagent of choice for various reactions;[5-7] Compared to DABCO, it is relatively poor carbon-nucleophilic[8] and possesses higher carbon basicity.[9] These properties may favor the reagent for acting as a suitable silyl-targeting nucleophile and for promoting the present TMSCN-based reaction with dramatic rate-acceleration.
TMEDA	9.42[1]	Tertiary diamine	
DIPEA	10.75[2]	Tertiary amine	
Triethylamine	10.68[1]	Tertiary amine	
Piperazine	9.73[3]	Secondary amine	
Piperidine	11.22[4]	Secondary amine	

(1) L. Cecchi, F. De Sarlo and F. Machetti, *Eur. J. Org. Chem.*, 2006, 4852.

(2) D. D. Perrin, Dissociation constans of organic acids and bases; Butterworths: London, 1965. Supplement 1972.

(3) F. Khalili, A. Henni and A. L. East, *J. Chem. Eng. Data*, 2009, **54**, 2914.

(4) H. K. Jr. Hall, *J. Am. Chem. Soc.*, 1957, **79**, 5441.

(5) H. Oediger, F. Moeller and K. Eiter, *Synthesis*, 1972, 591.

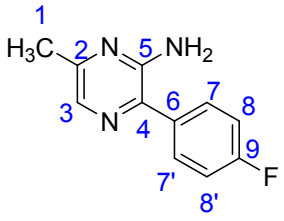
(6) W. C. Shieh, S. Dell and O. Repic, *Org. Lett.*, 2001, **3**, 4279.

(7) V. K. Aggarwal and A. Mereu, *Chem. Commun.*, 1999, 2311.

(8) M. Baidya and H. Mayr, *Chem. Commun.*, 2008, 1792.

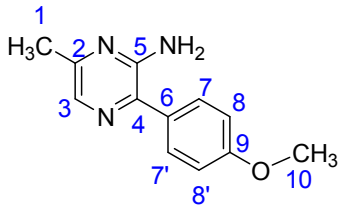
(9) J. Hine and R. D. Weimar Jr, *J. Am. Chem. Soc.*, 1965, **87**, 3387.

3-(4-Fluorophenyl)-6-methylpyrazin-2-amine (6h, 2D NMR, HMQC, HMBC): The C3 carbon has been assigned by HMQC correlation between C3-H (δ 7.75) and carbon at δ 132.13.



The methyl proton C1-H (δ 2.28) showed HMBC correlation with C2 (δ 150.21) and C3 (δ 132.1). The C3-H proton (δ 7.75) showed HMBC correlation with C2 (δ 150.21) and C4 (δ 135.78). The C7/C7'-H (δ 7.71-7.67) protons showed correlation with C8/C8' (δ 130.69-130.60, d), C6 (δ 134.50-134.47, d), C4 (δ 135.78) and C9 (δ 163.57-161.13, d). The C8/C8'-H (δ 7.30-7.25) protons showed correlation with C7/C7' (δ 115.97-115.76, d), C6 (δ 134.50-134.47, d), C4 (δ 135.78) and C9 (δ 163.57-161.13, d).

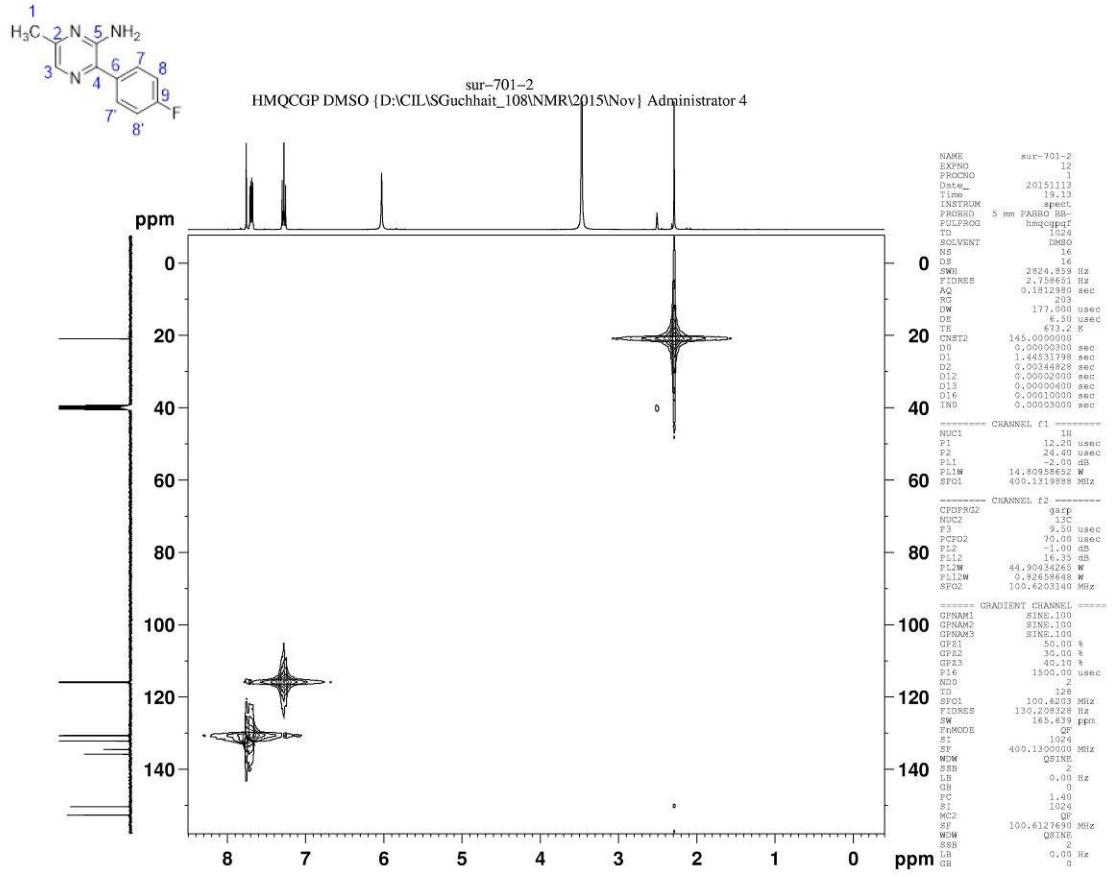
3-(4-Methoxyphenyl)-6-methylpyrazin-2-amine (6i, 2D NMR, HMQC, HMBC): The C3 carbon has been assigned by HMQC correlation between C3-H (δ 7.88) and carbon at δ 133.77.



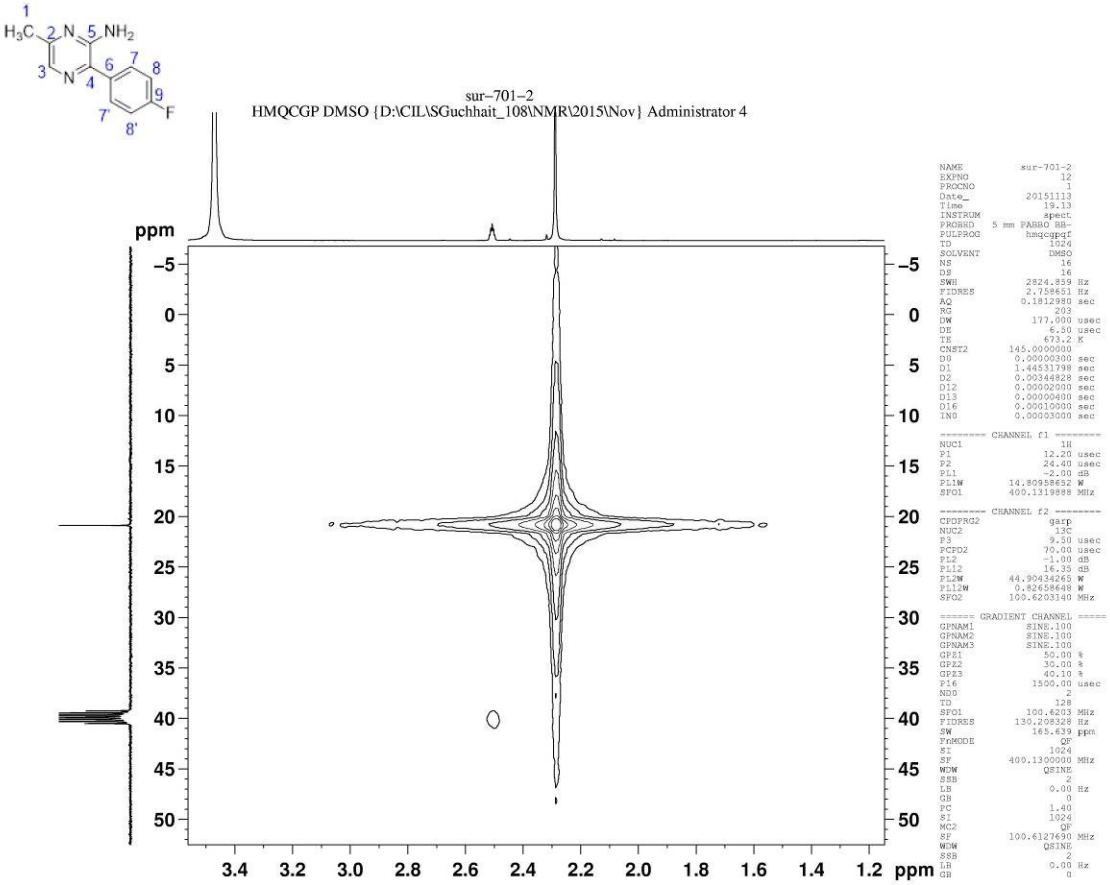
The methyl proton C1-H (δ 2.39) showed HMBC correlation with C2 (δ 149.48) and C3 (δ 133.77). The C3-H proton (δ 7.88) showed HMBC correlation with C2 (δ 149.48) and C4 (δ 137.63). The C7/C7'-H (δ 7.65-7.62) protons showed correlation with C8/C8' (δ 129.42), C6 (δ 129.76), C4 (δ 137.63) and C9 (δ 159.95). The C8/C8'-H (δ 7.01-6.98) protons showed correlation with C7/C7' (δ 114.38), C6 (δ 129.76), C4 (δ 137.63) and C9 (δ 159.95). The methoxy protons C10-H (δ 3.85) showed correlation with C9 (δ 159.95).

2D NMR of Compounds 6h and 6i (HMQC and HMBC)

6h: HMQC



6h: HMQC (Expansion 1)



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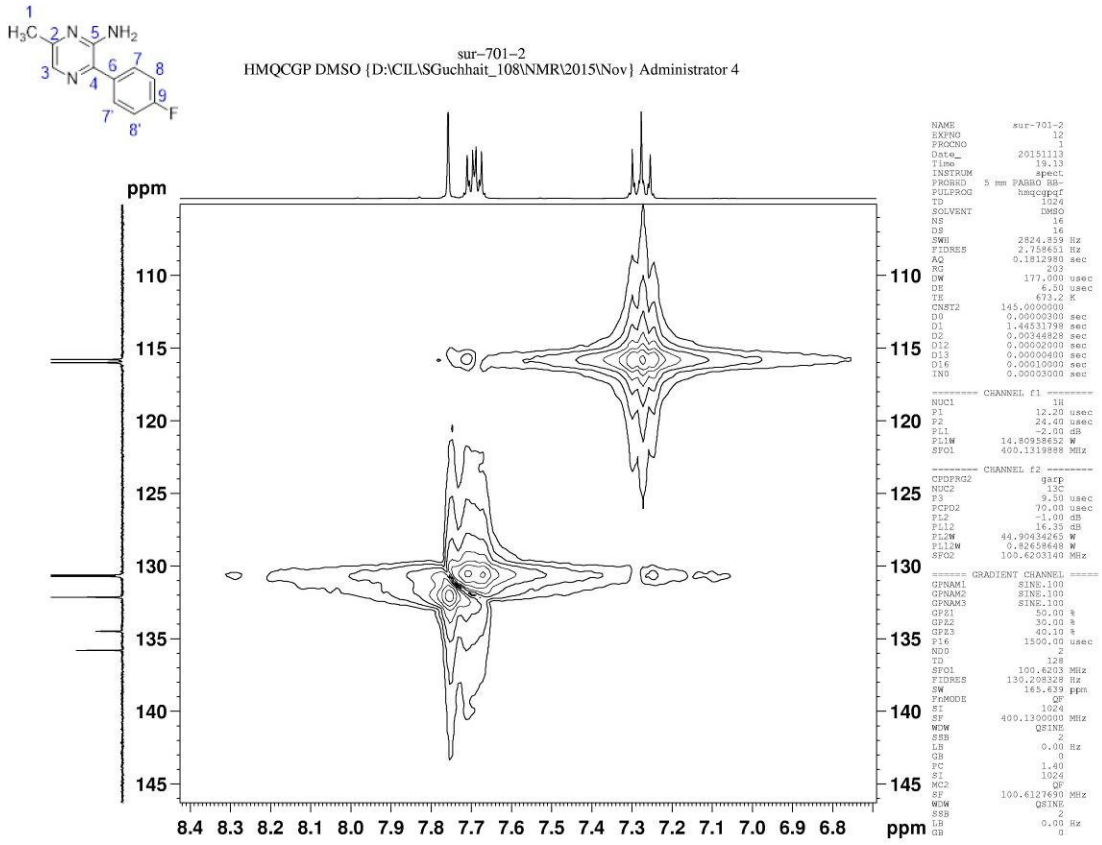

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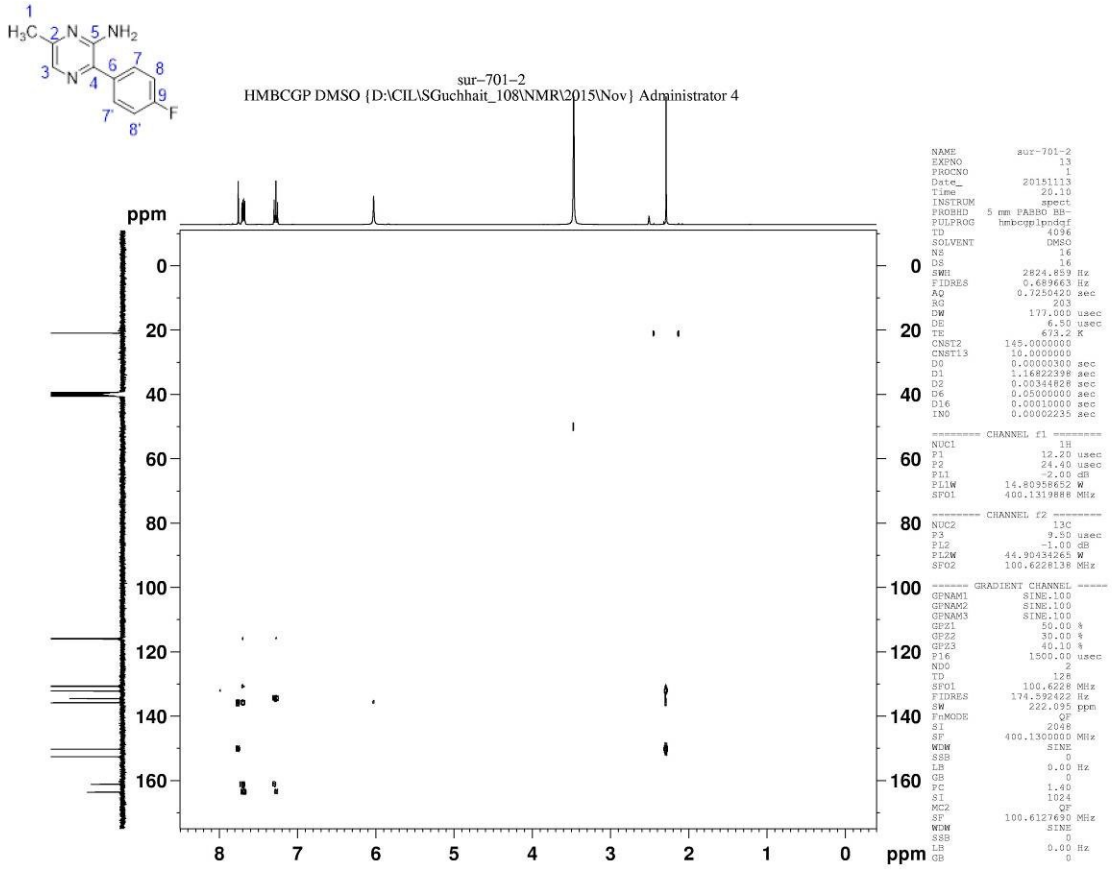

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CNST13 10.0000000
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SFO1 400.1319888 MHz

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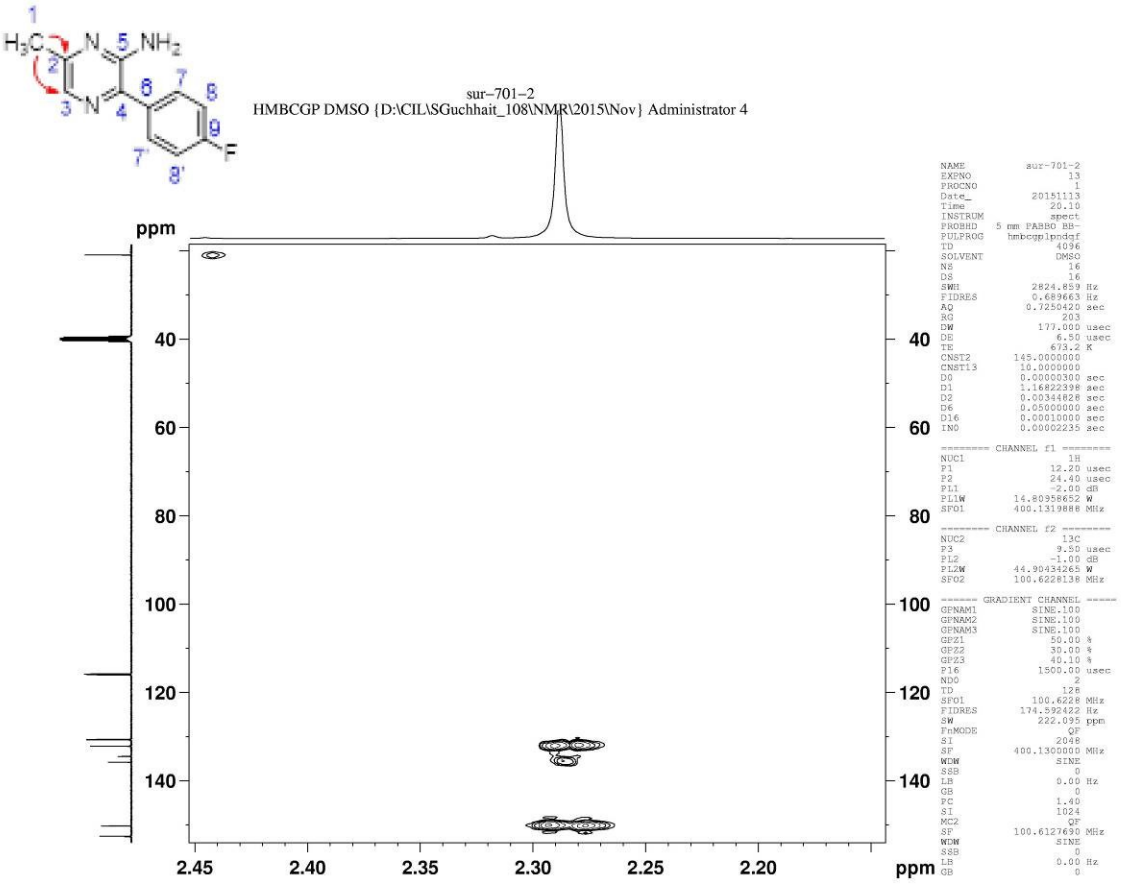

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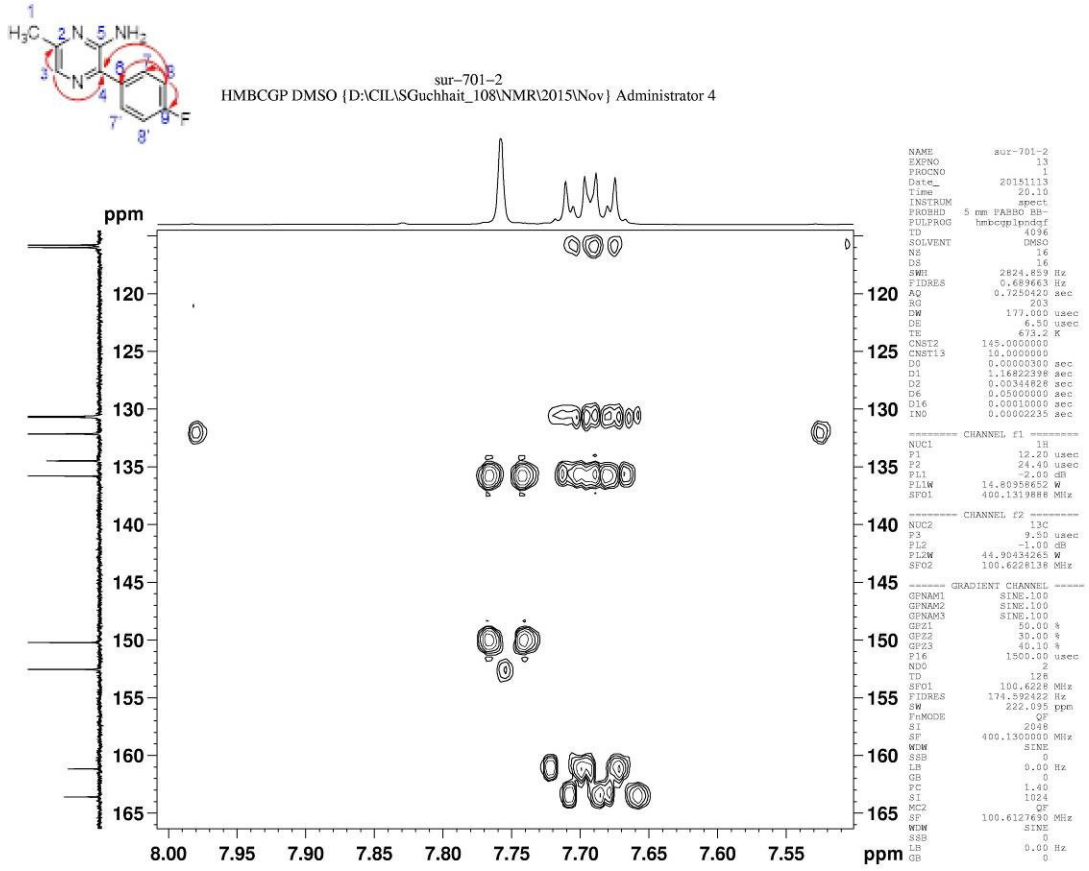

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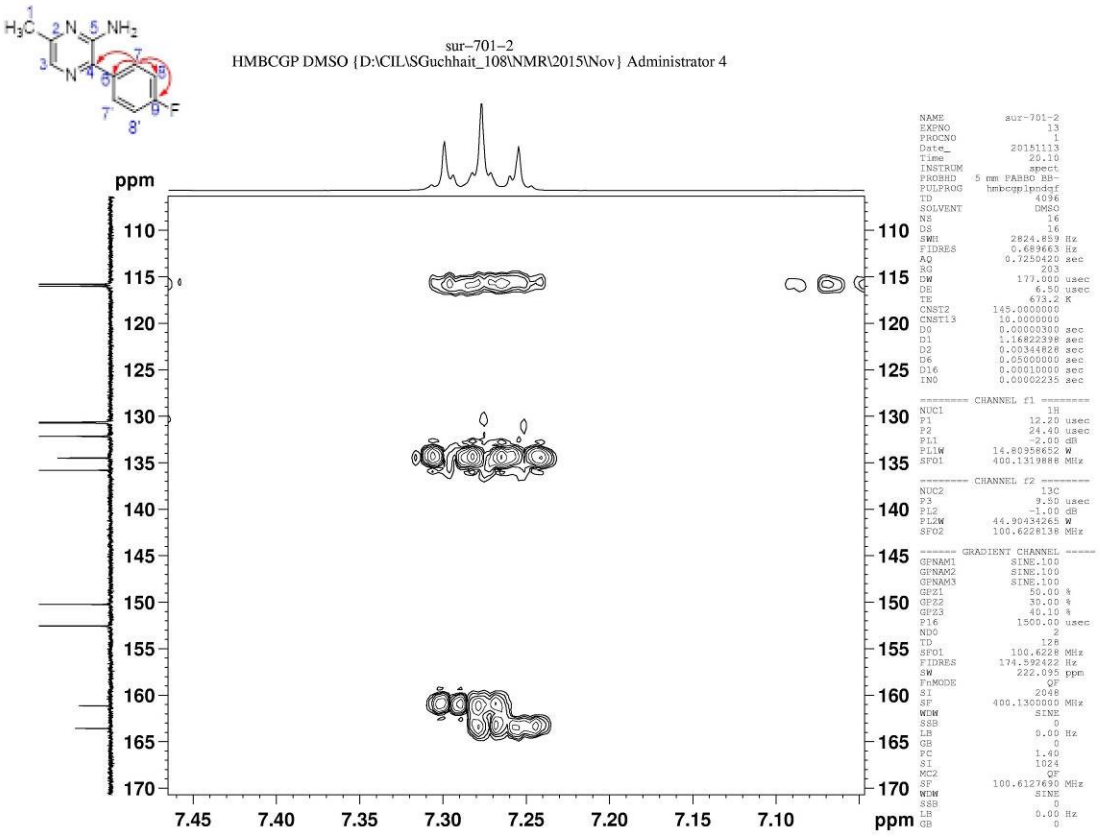

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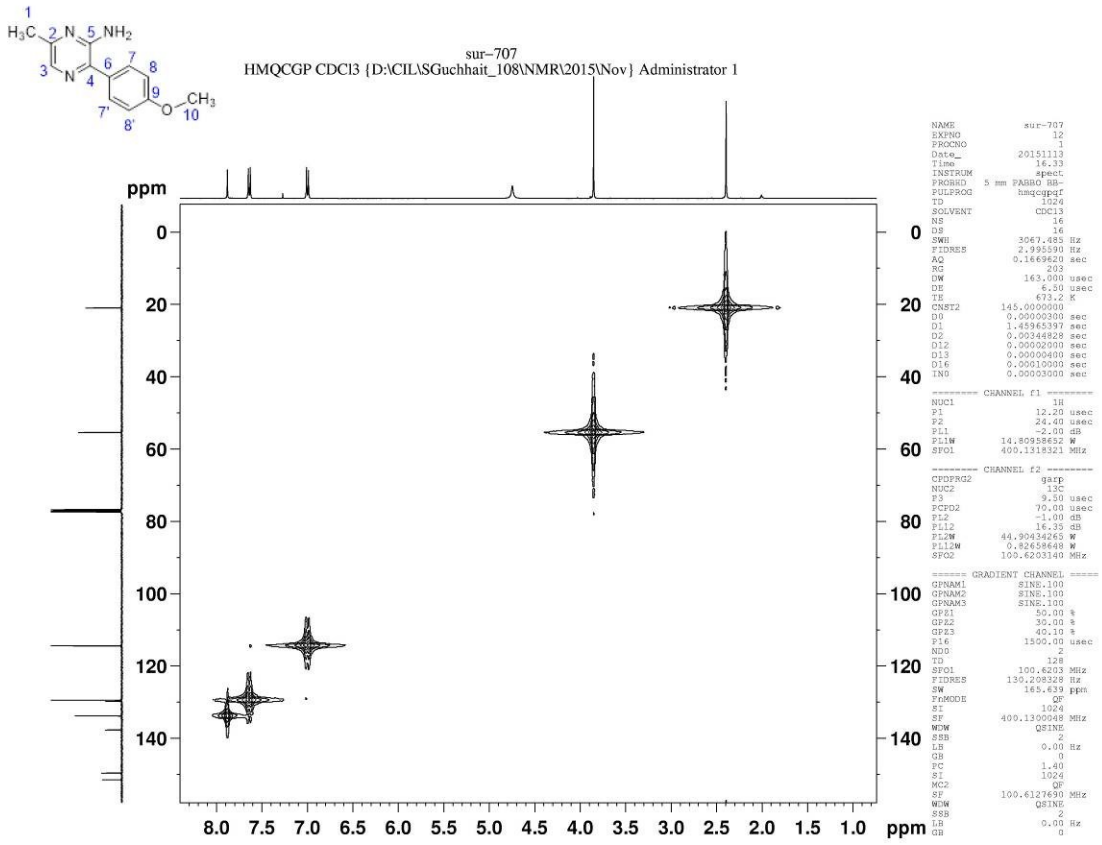
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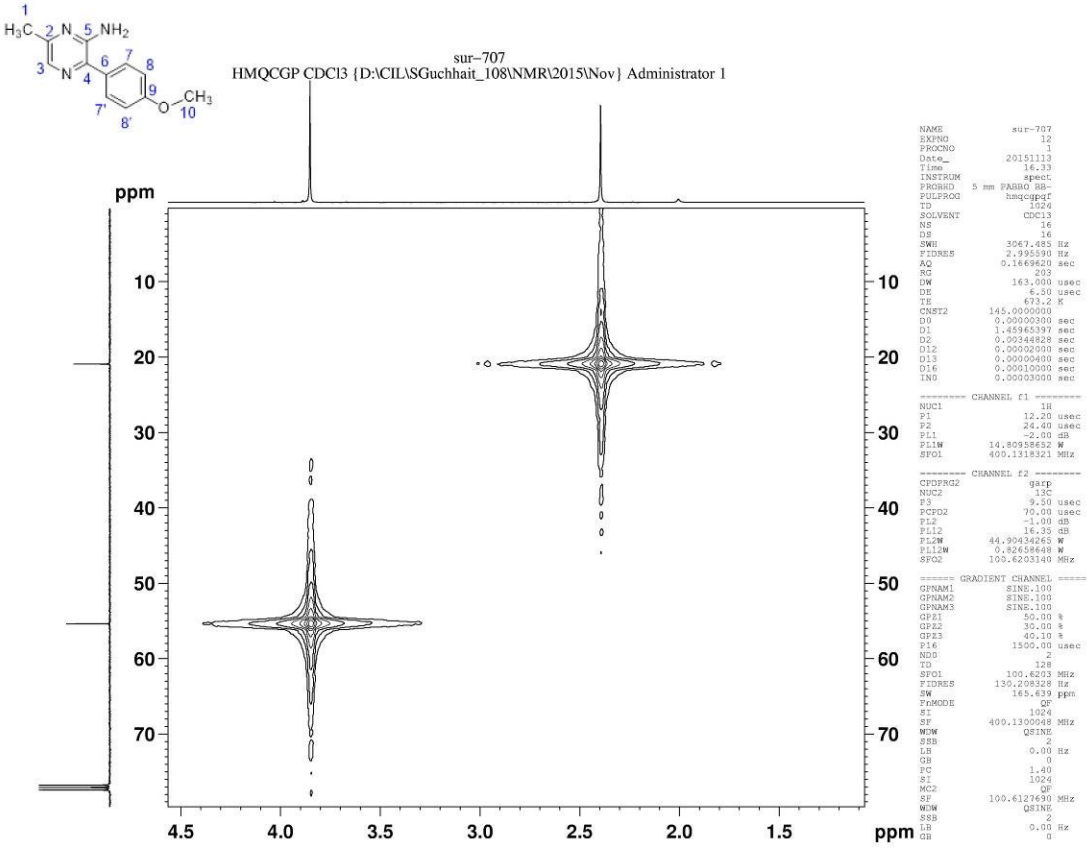

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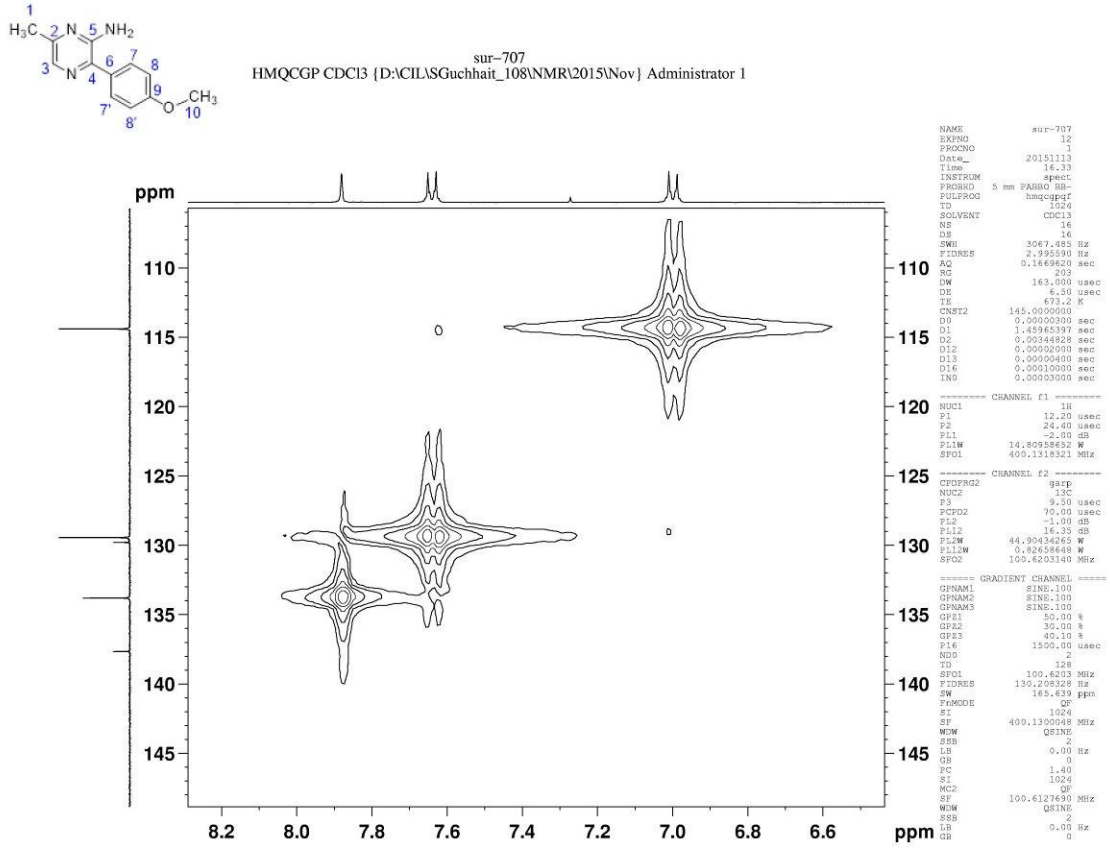

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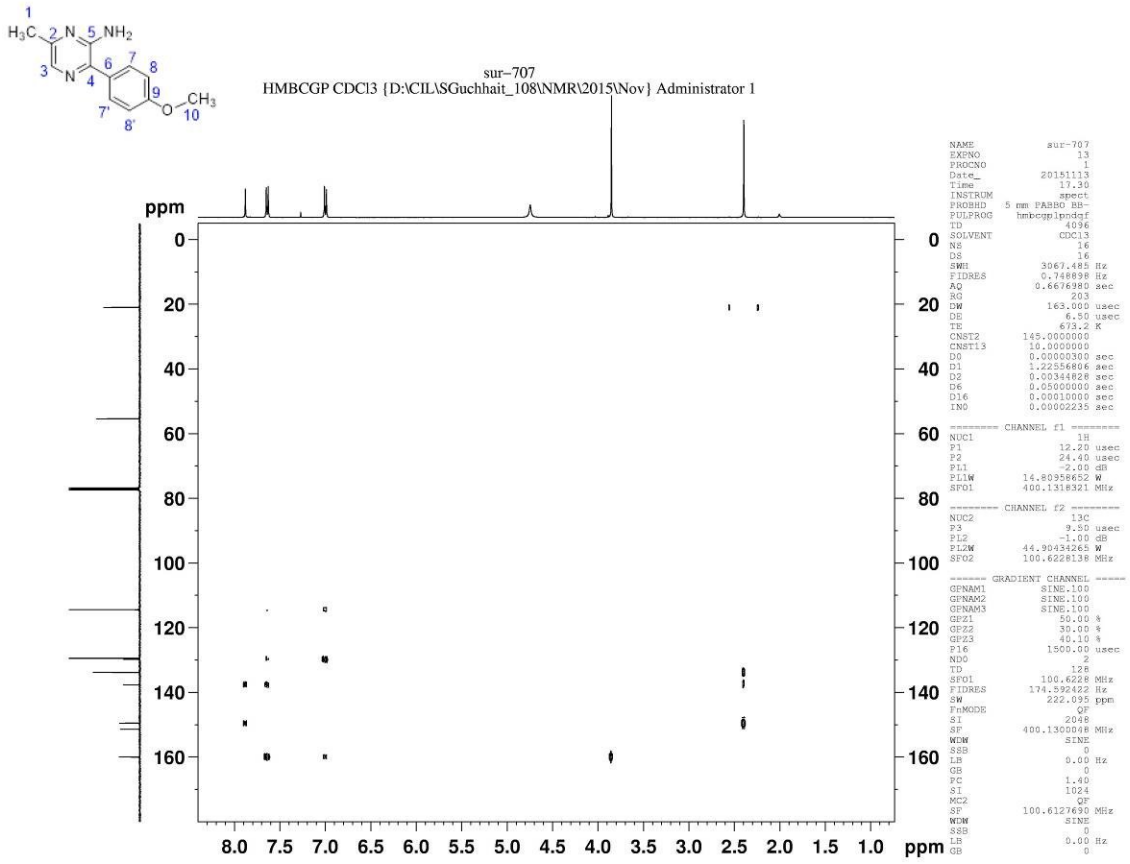
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Time 17.30
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DE 6.50 usec
TE 673.2 K
CNST2 145.0000000
CNST13 10.0000000
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D2 0.00344828 sec
D6 0.05000000 sec
D16 0.00010000 sec
IN0 0.00002235 sec
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P1 12.20 usec
P2 24.40 usec
PL1 -2.00 dB
PL1W 14.80958652 W
SFO1 400.1318321 MHz
======== CHANNEL f2 ========
NUC2 13C
P3 9.50 usec
PL2 -1.00 dB
PL2W 44.90434265 W
SFO2 100.6228138 MHz
====== GRADIENT CHANNEL =====
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GPNAM2 SINE.100
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GPZ3 40.10 %
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SF 400.1300048 MHz
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LB 0.00 Hz
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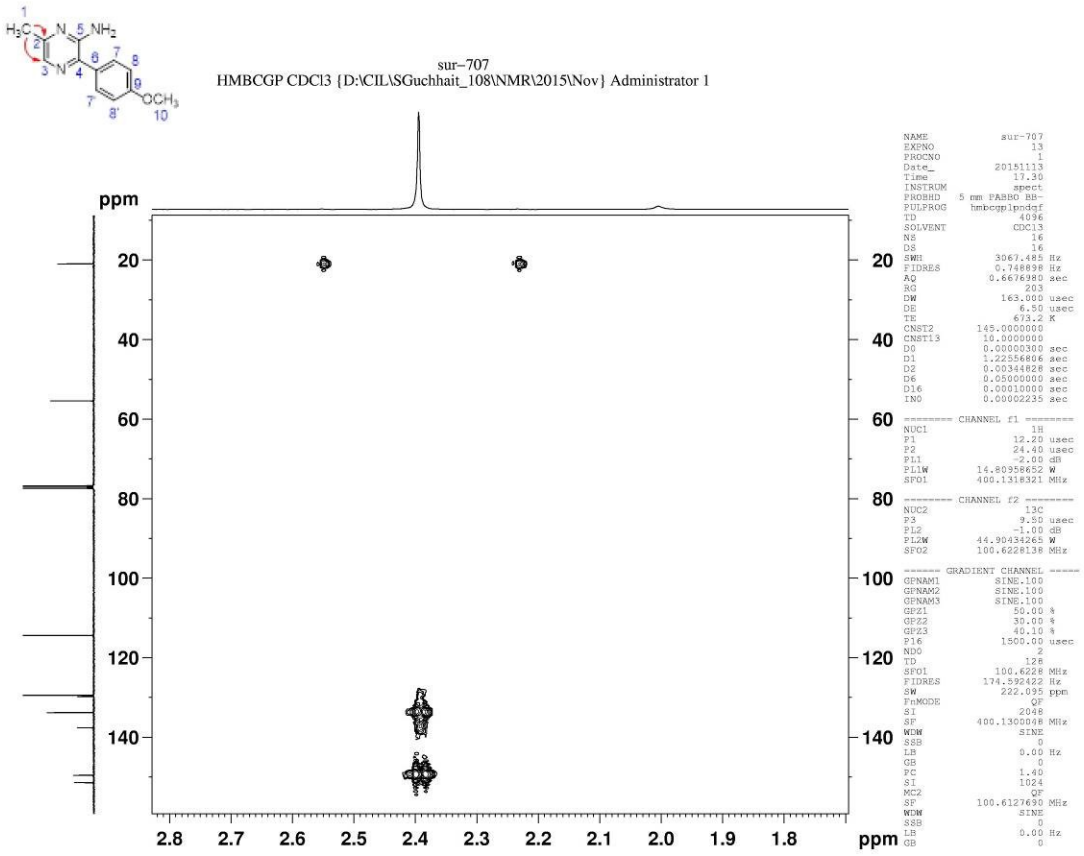
6i: HMBC (Expansion 1)



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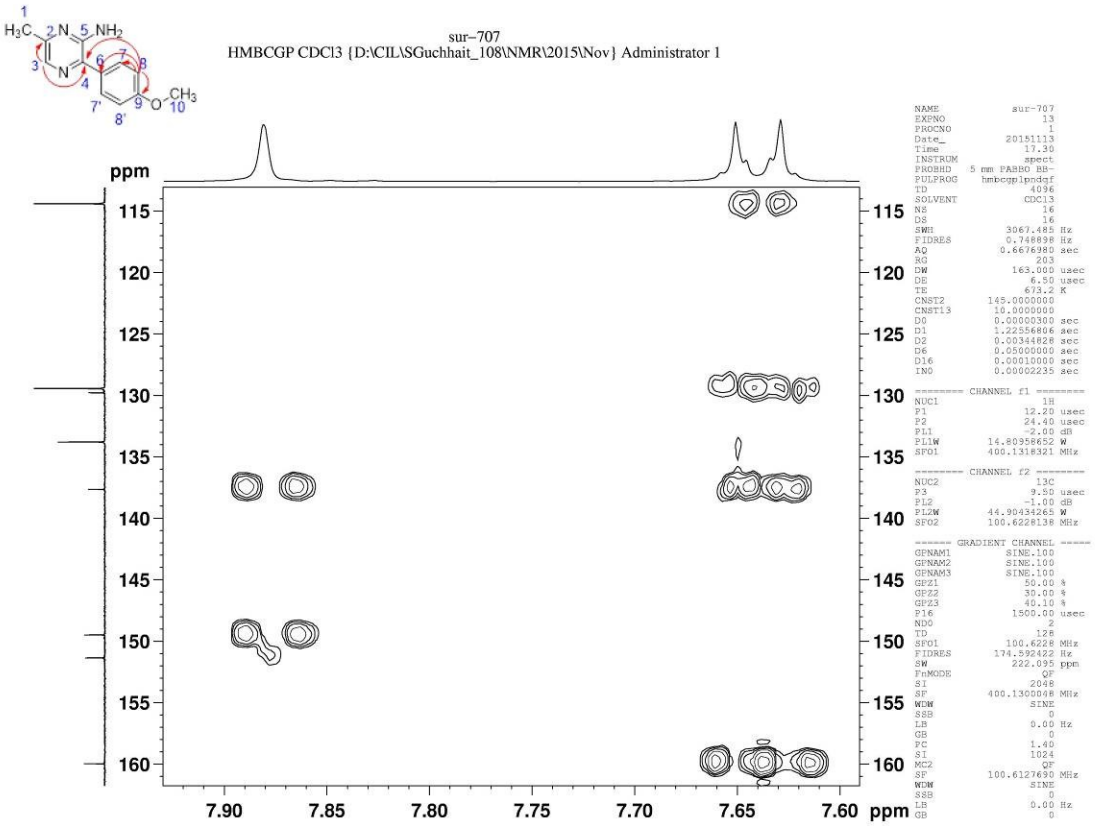

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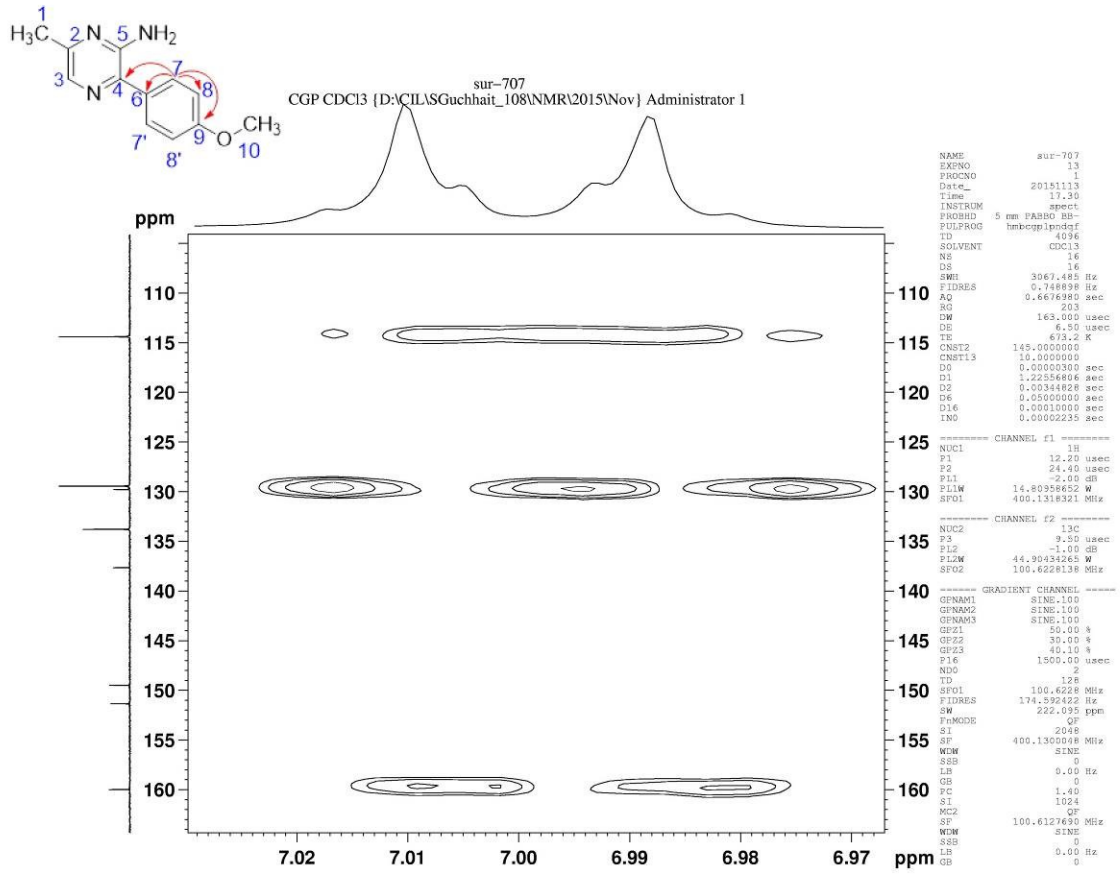

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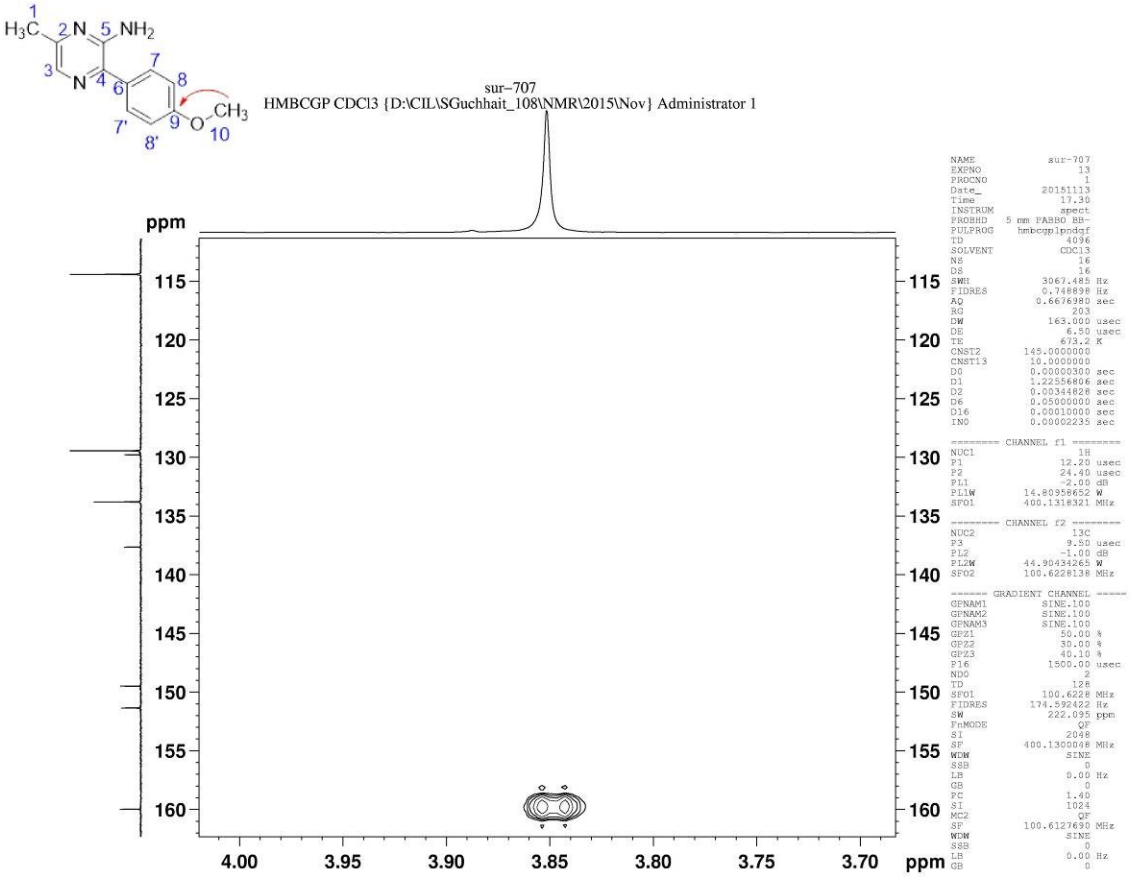
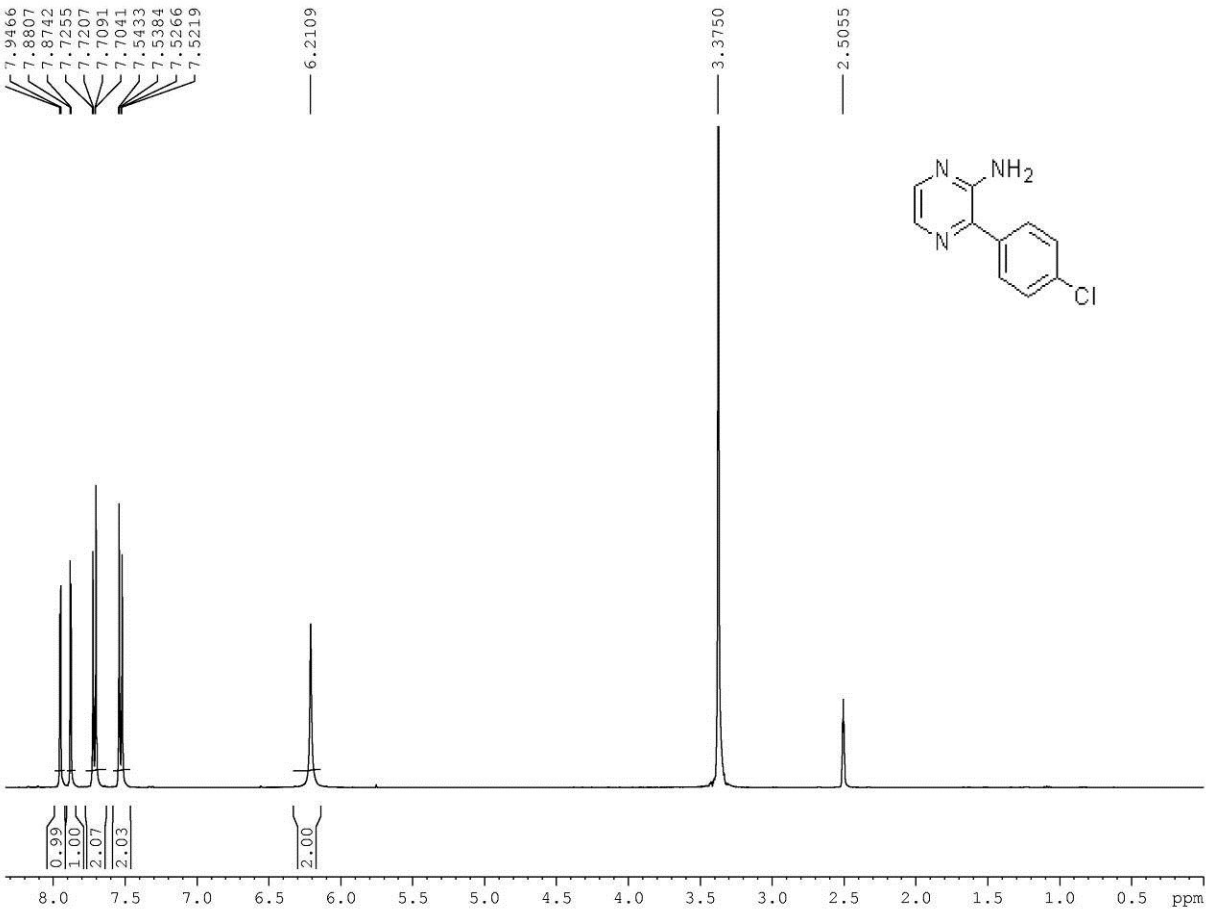


NMR Spectra of Compounds 6a-l, 4a-p

6a: ^{1}H NMR



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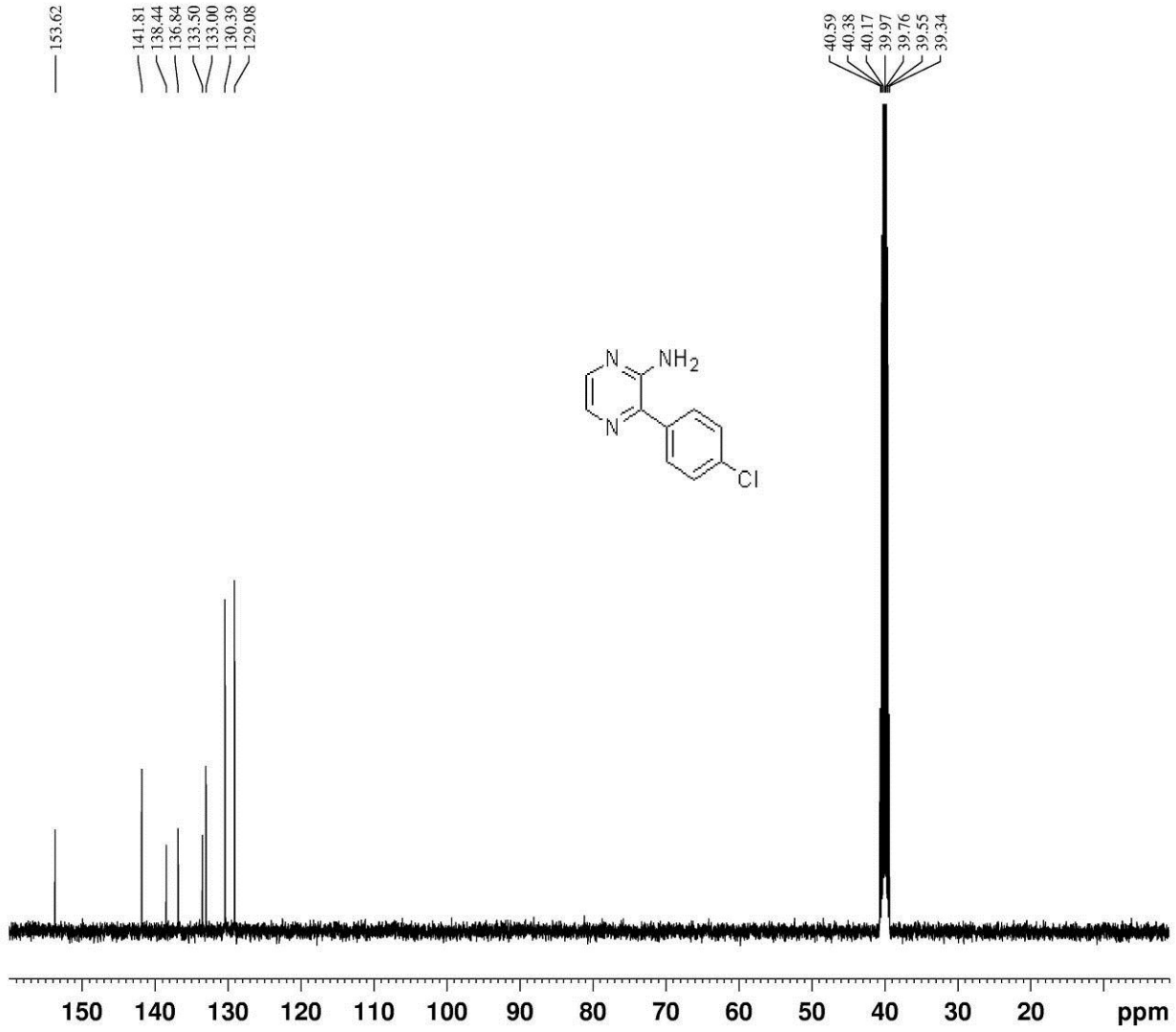
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39.55
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Cl
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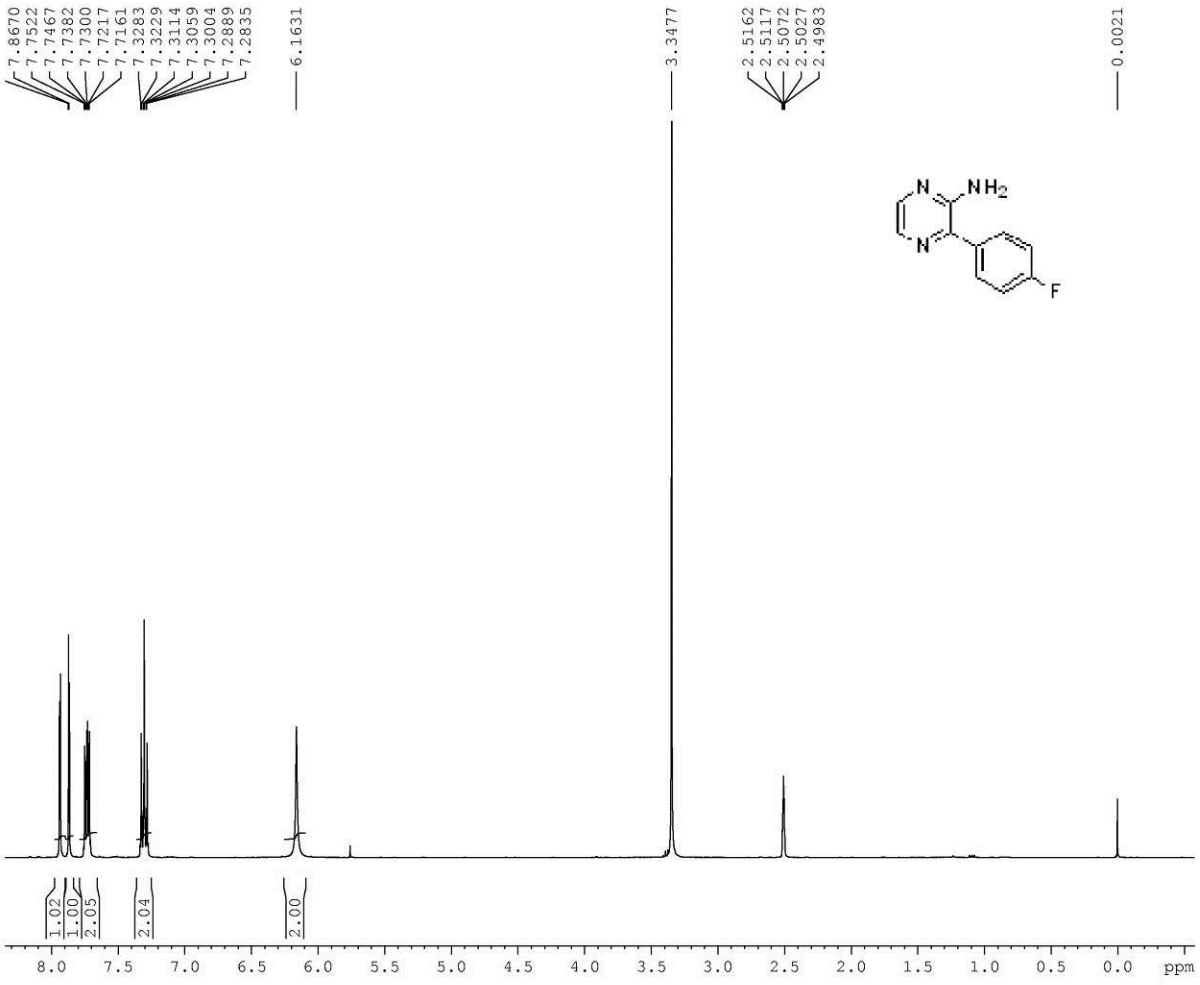
50
40
30
20
ppm

6b: ^{1}H NMR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7.3114
7.3059
7.3004
7.2889
7.2835
6.1631
3.3477
2.5162
2.5117
2.5072
2.5027
2.4983
0.0021
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NH2
N
F
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1.00
2.05
2.04
2.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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3.0
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0.5
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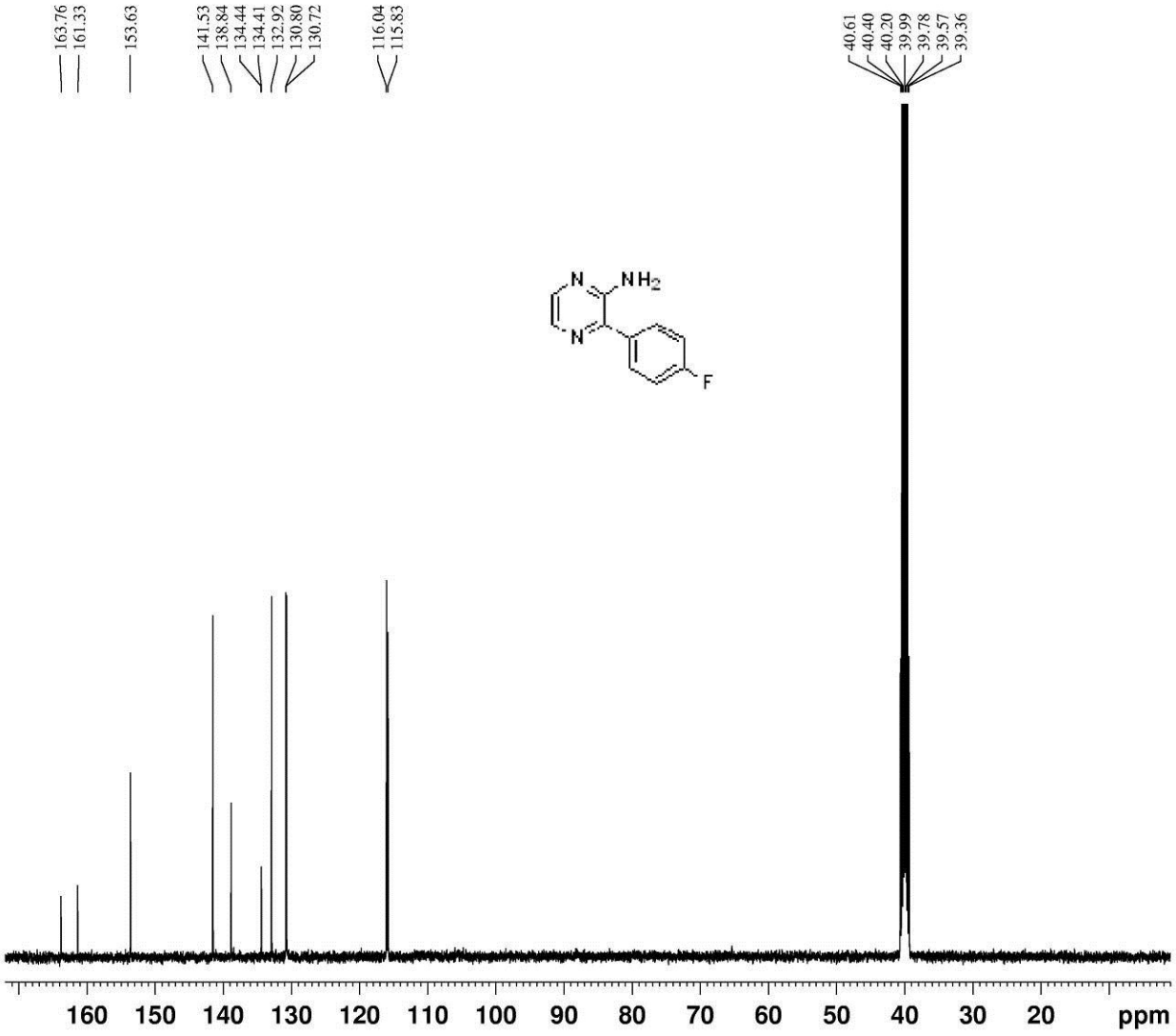
ppm

6b: ^{13}C NMR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138.84
134.44
134.41
132.92
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130.72
116.04
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40.61
40.40
40.20
39.99
39.78
39.57
39.36
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NH2
N
F
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130
120
110
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90
80
70
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50
40
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ppm

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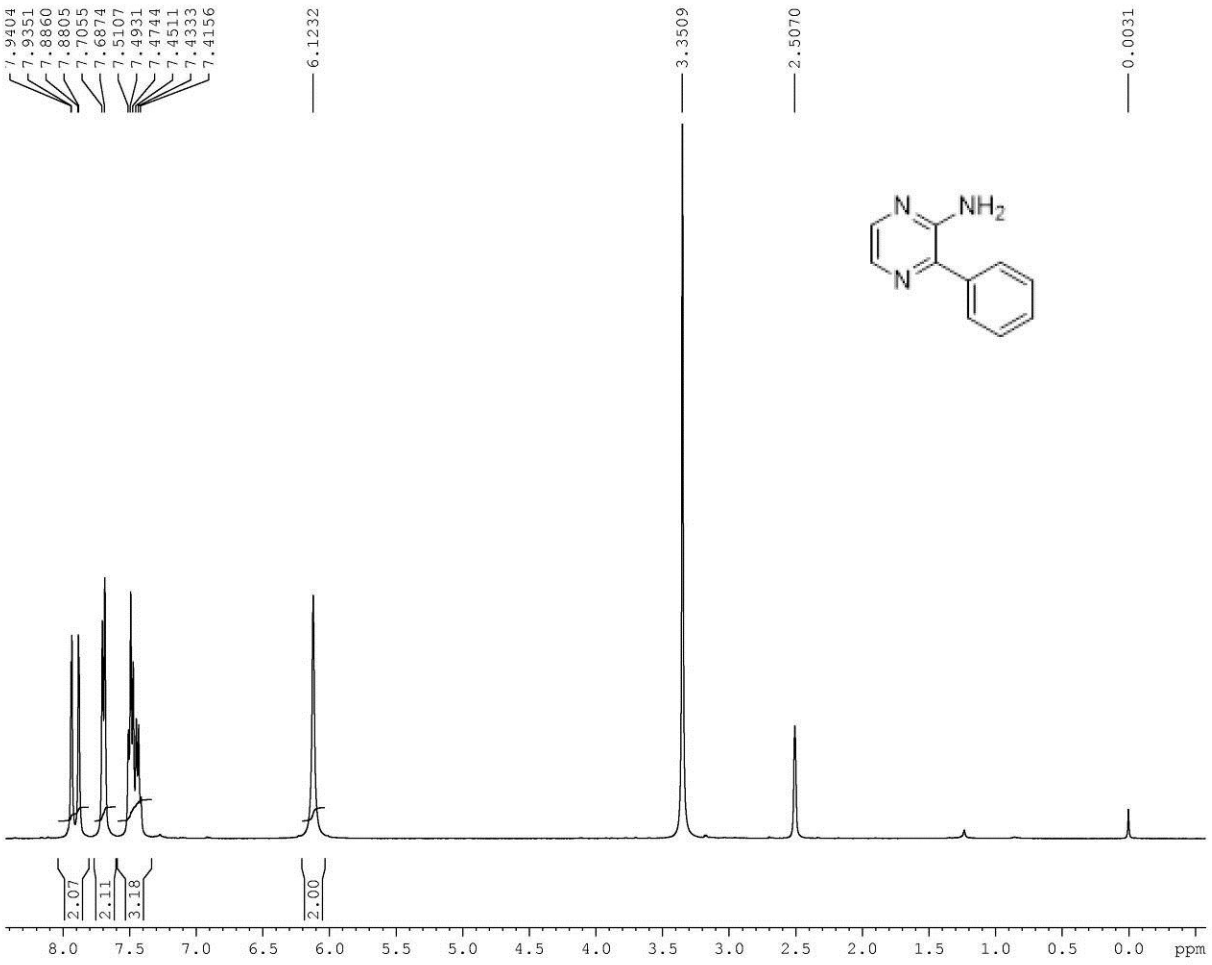
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7.4333
7.4156
6.1232
3.3509
2.5070
0.0031
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NH2
N
2.07
2.11
3.18
2.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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3.0
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ppm

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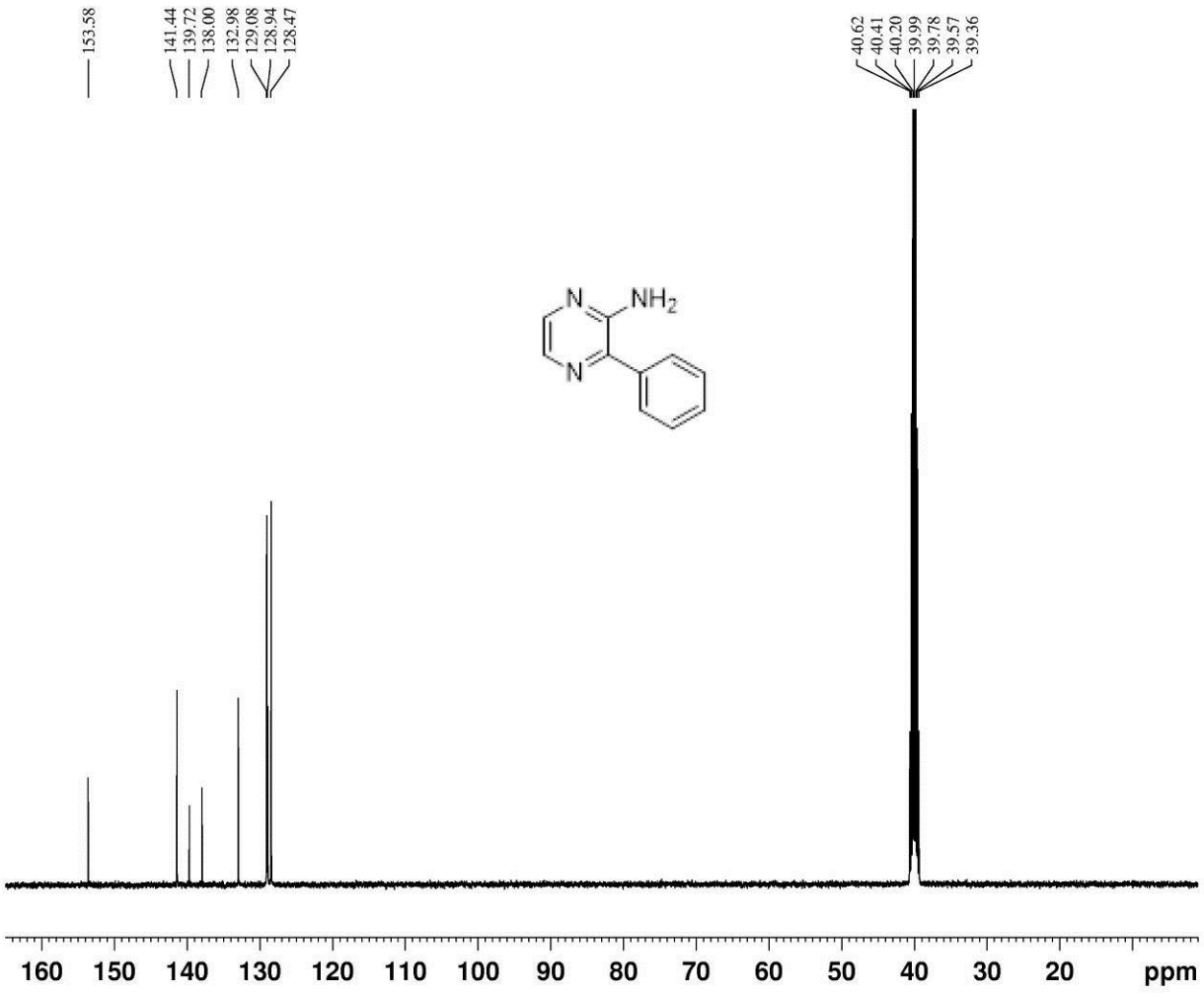
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39.57
39.36
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NH2
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50
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20
ppm

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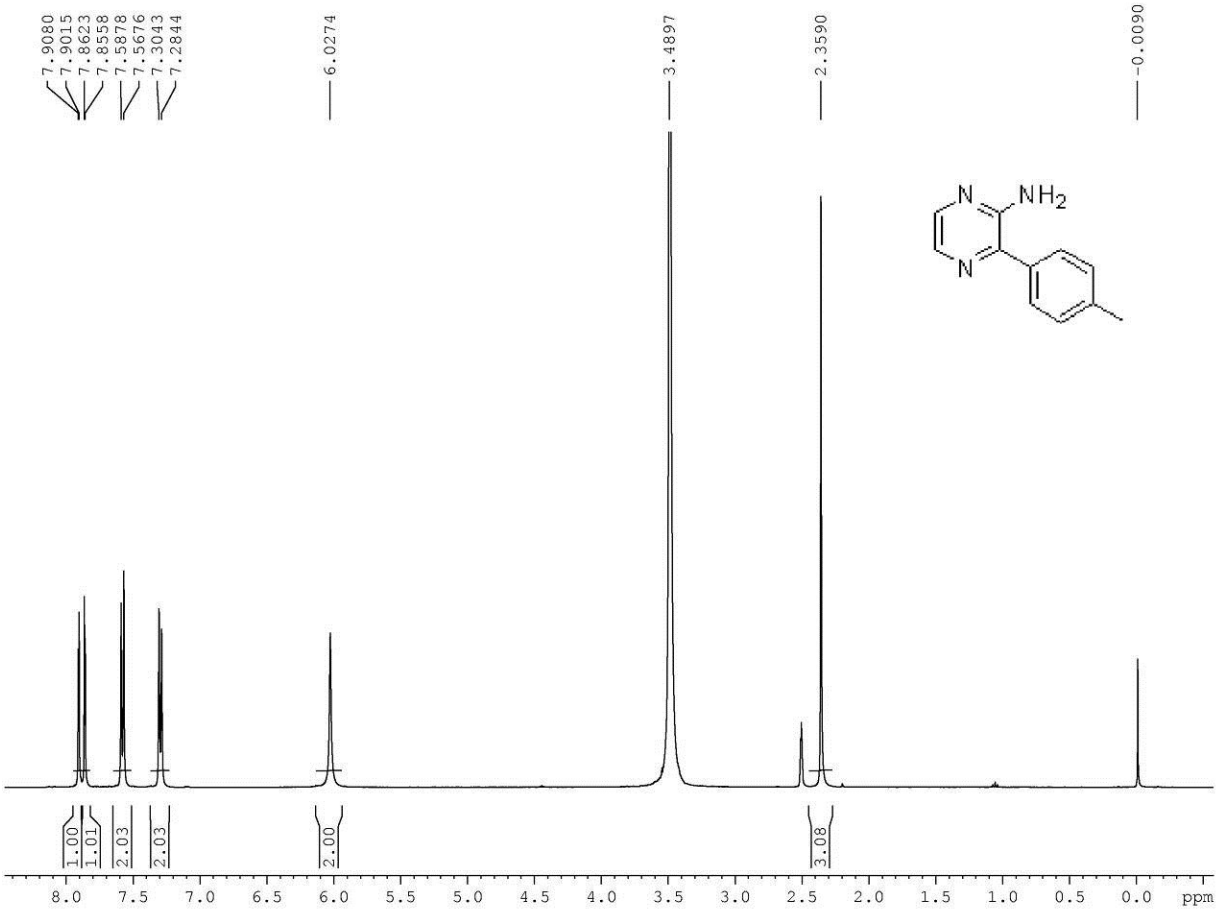
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NH2
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1.01
2.03
2.03
2.00
3.08
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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2.0
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ppm

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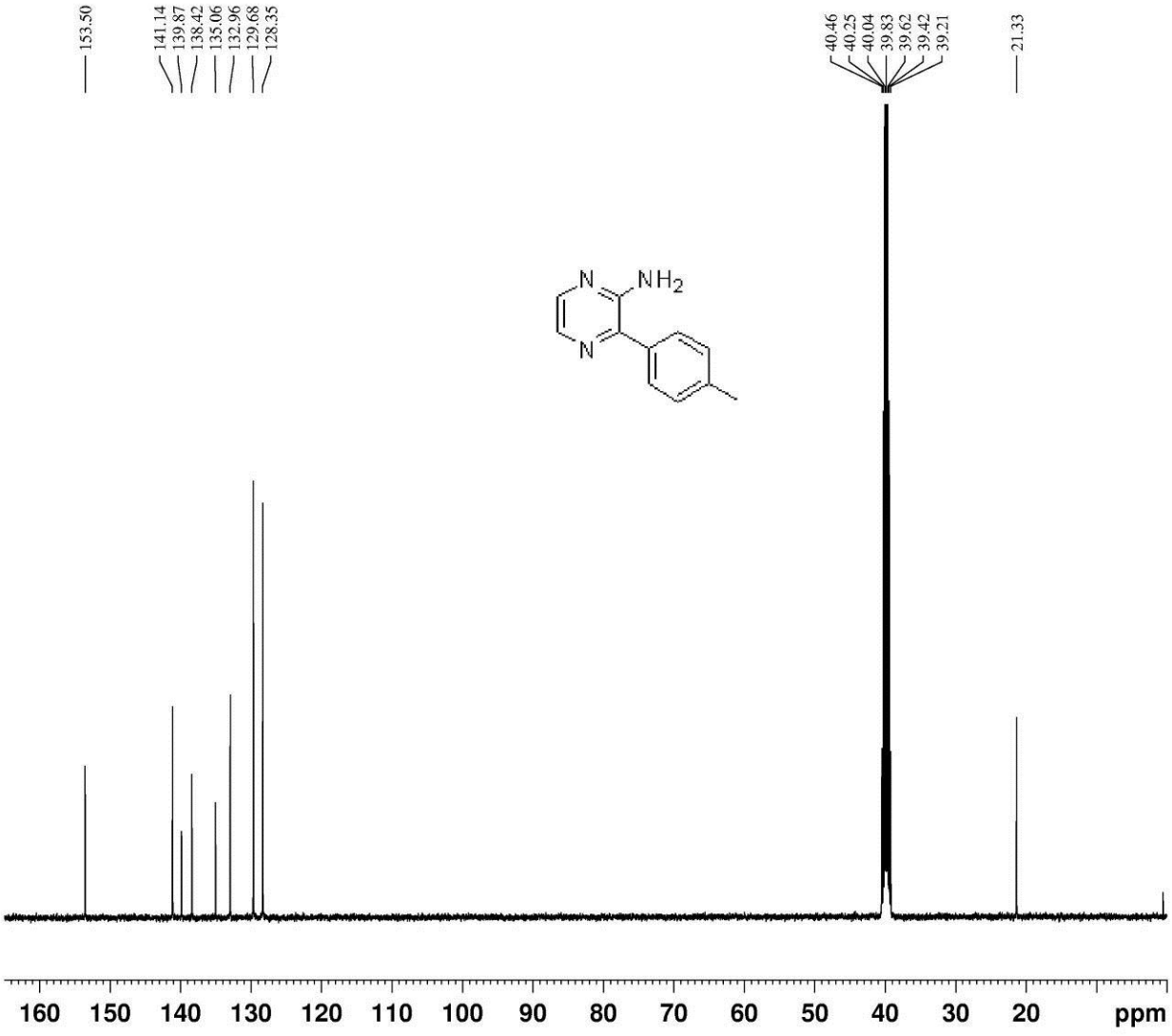
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128.35
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39.21
21.33
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NH2
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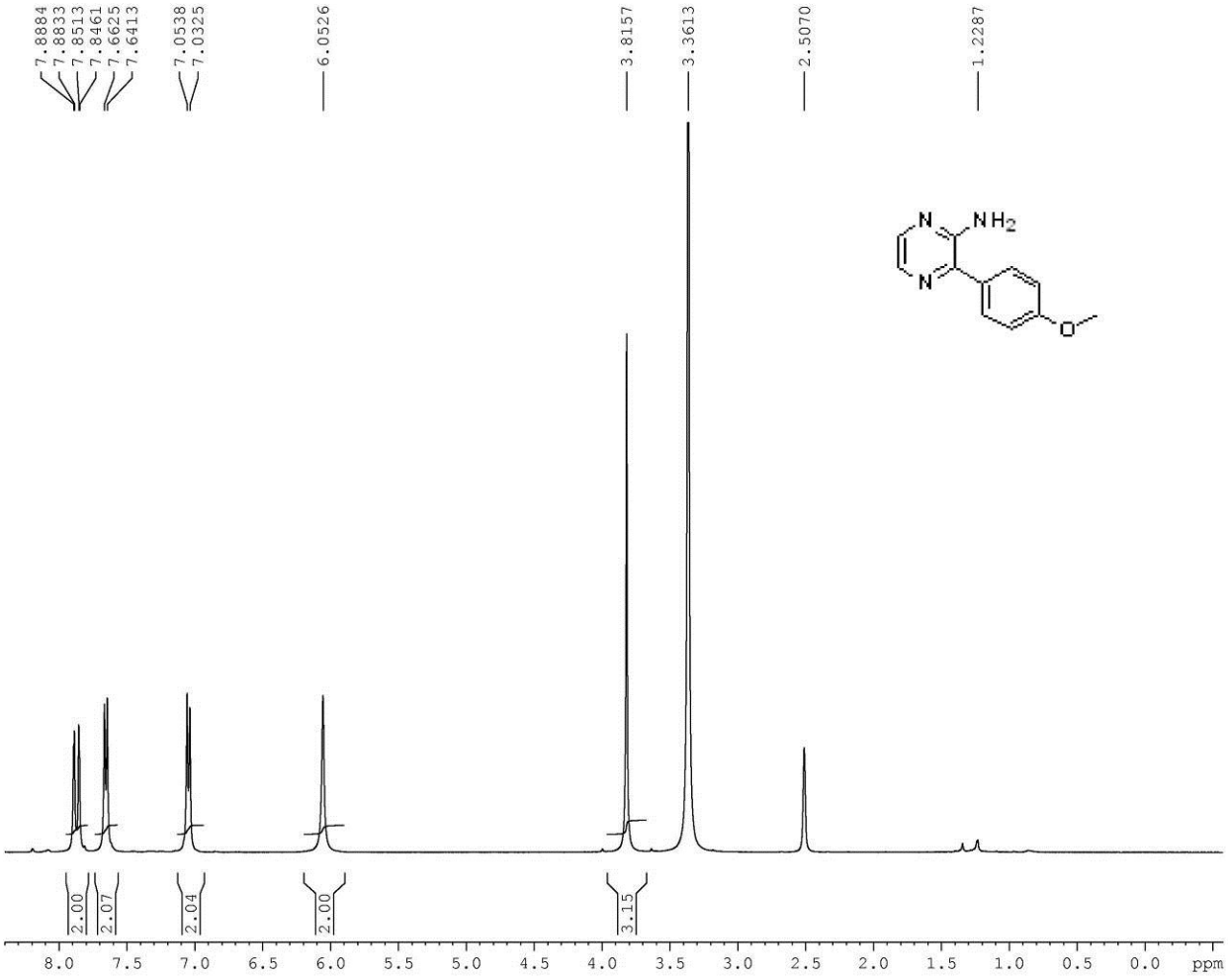
130
120
110
100
90
80
70
60
50
40
30
20
ppm

6e: ^{1}H NMR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3.3613
2.5070
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NH2
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O
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2.07
2.04
2.00
3.15
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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1.5
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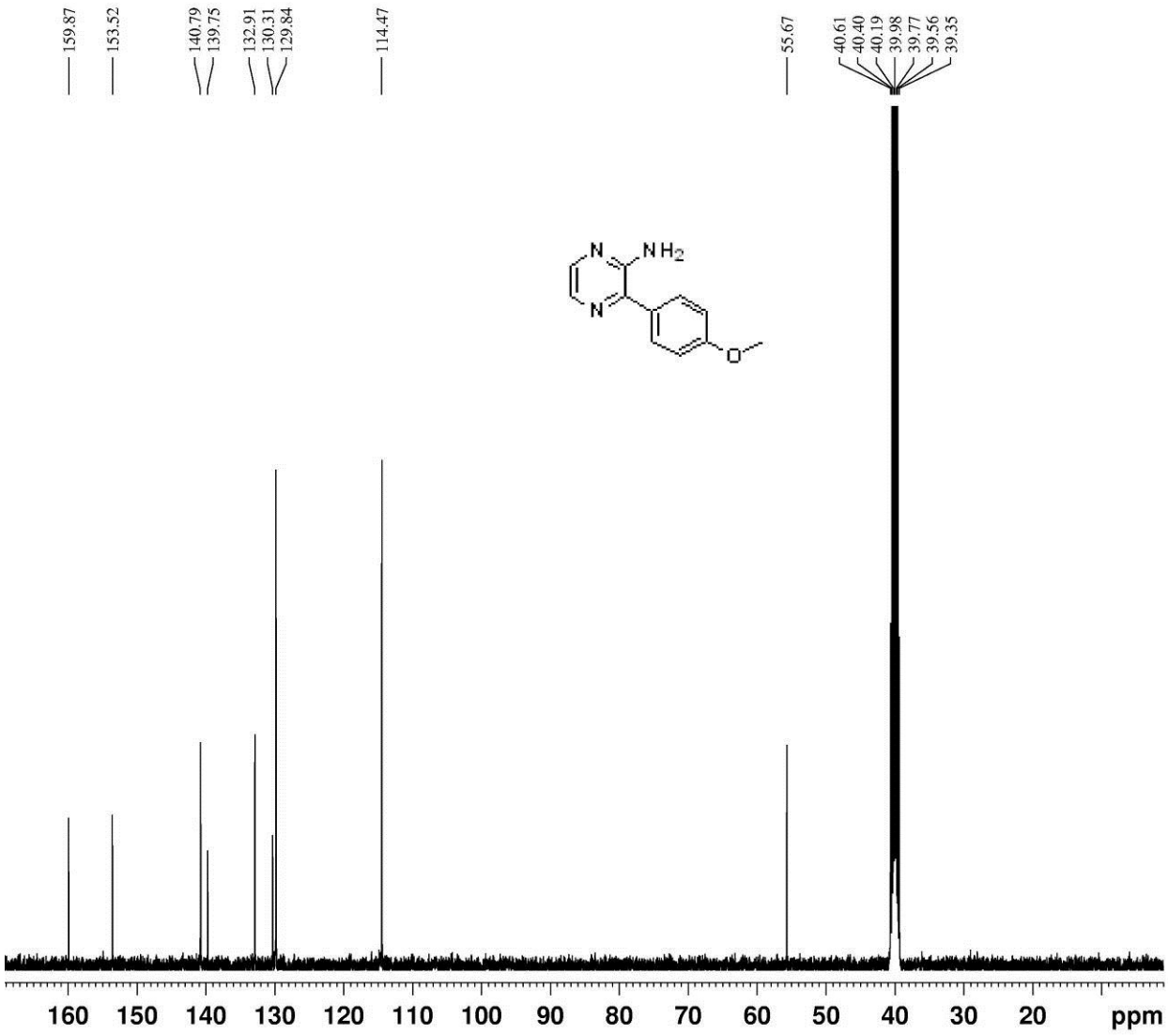
ppm

6e: ^{13}C NMR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39.35
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NH2
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O
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120
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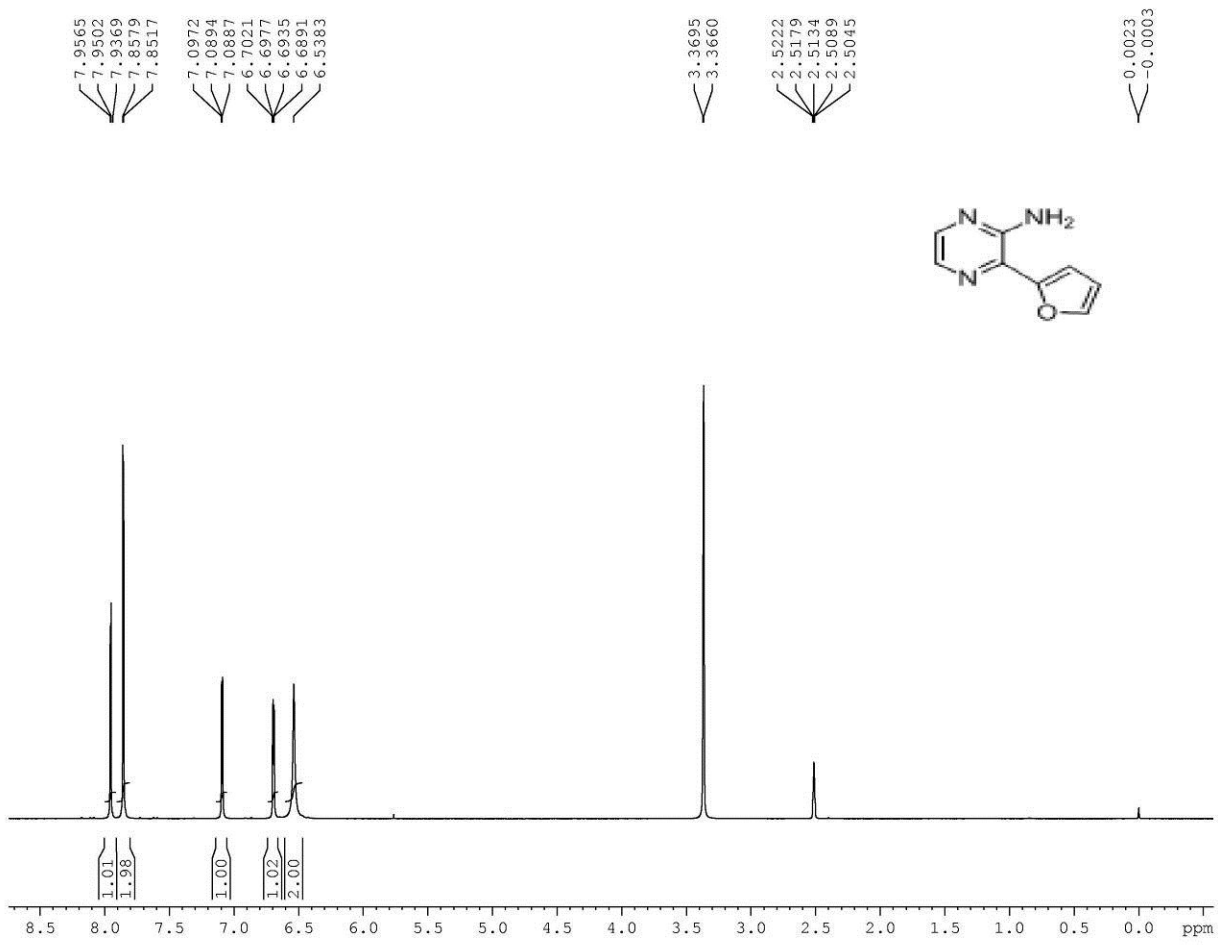
70
60
50
40
30
20
ppm

6f: ^{1}H NMR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6.6891
6.5383
3.3695
3.3660
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2.5179
2.5134
2.5089
2.5045
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NH2
N
O
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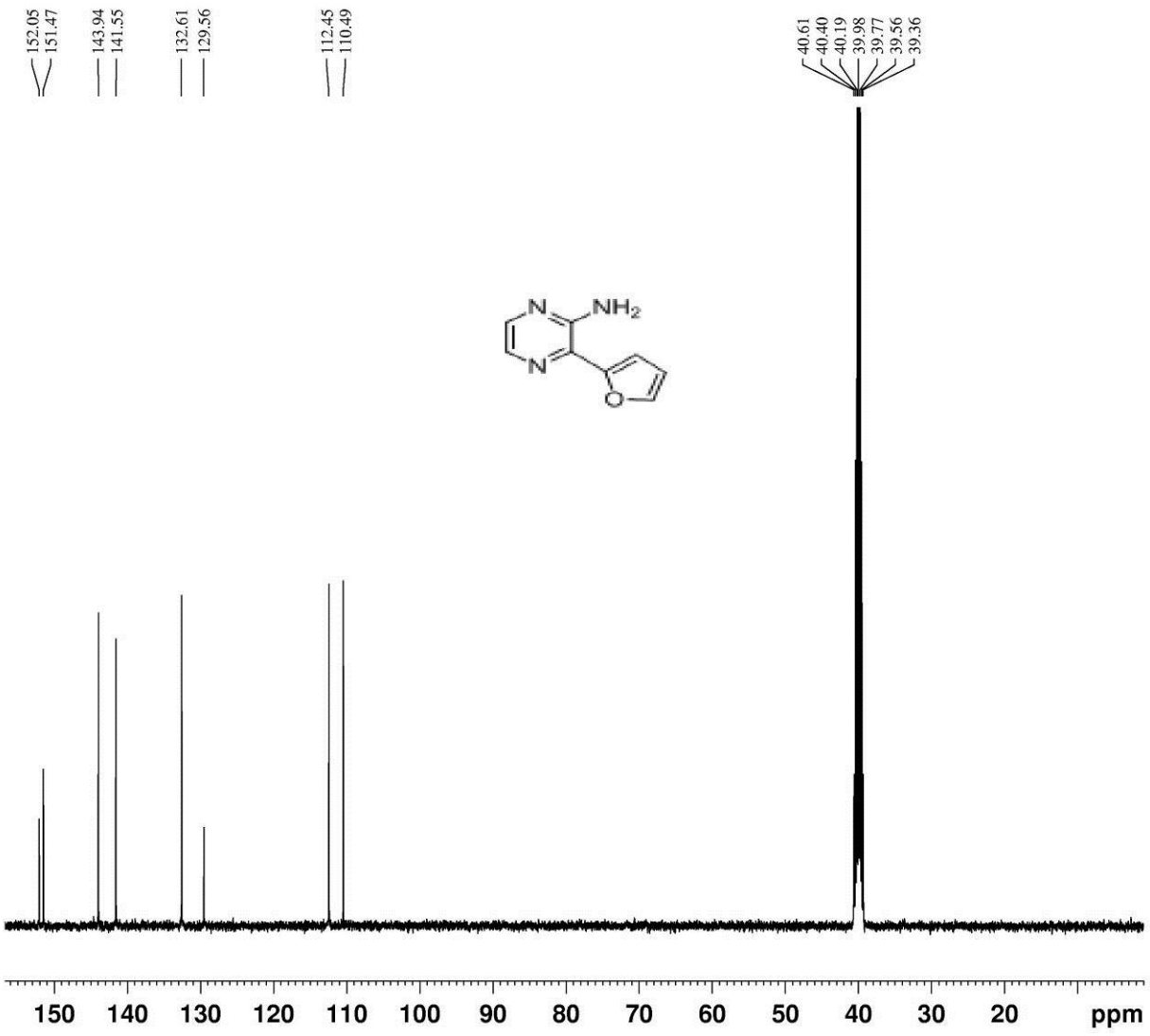
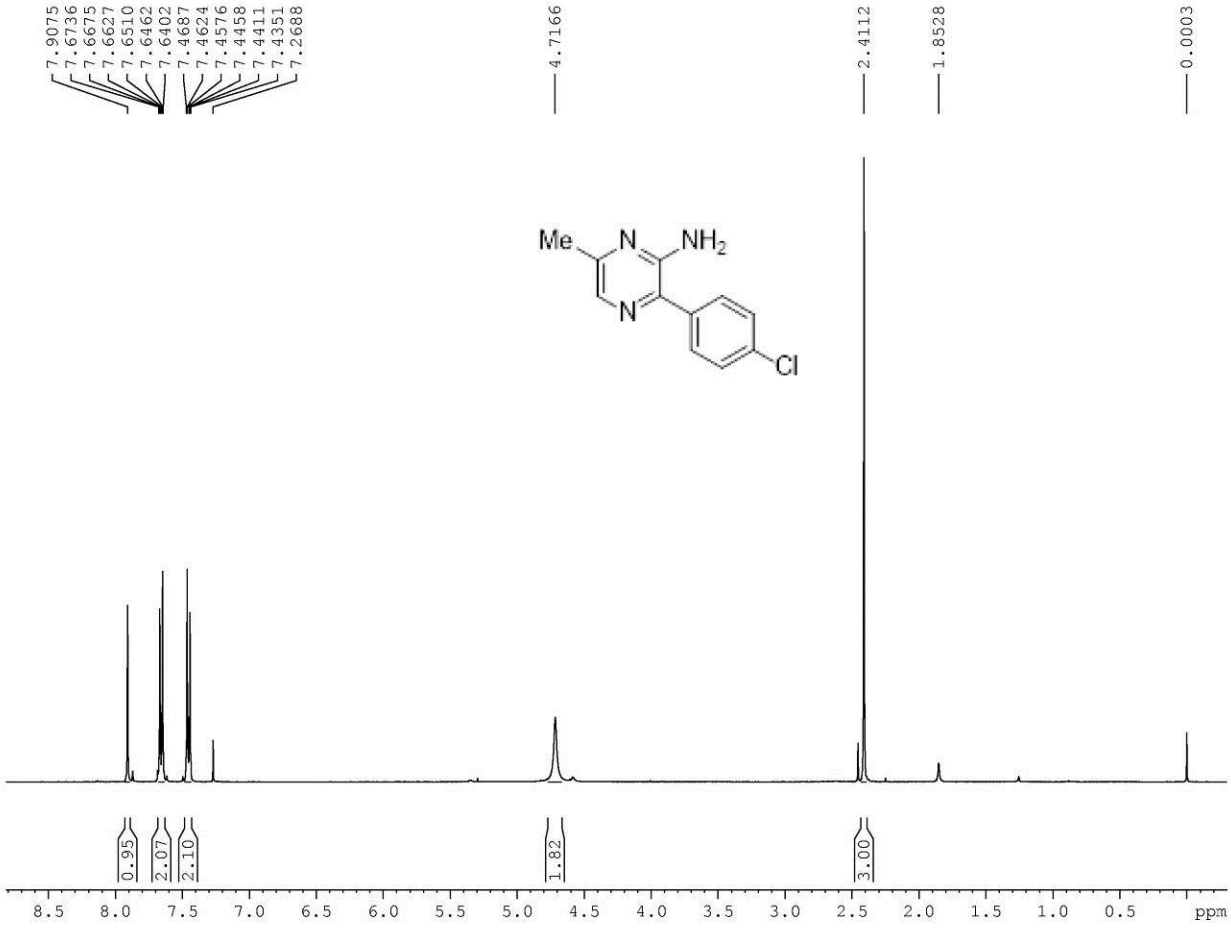
1.98
1.00
1.02
2.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

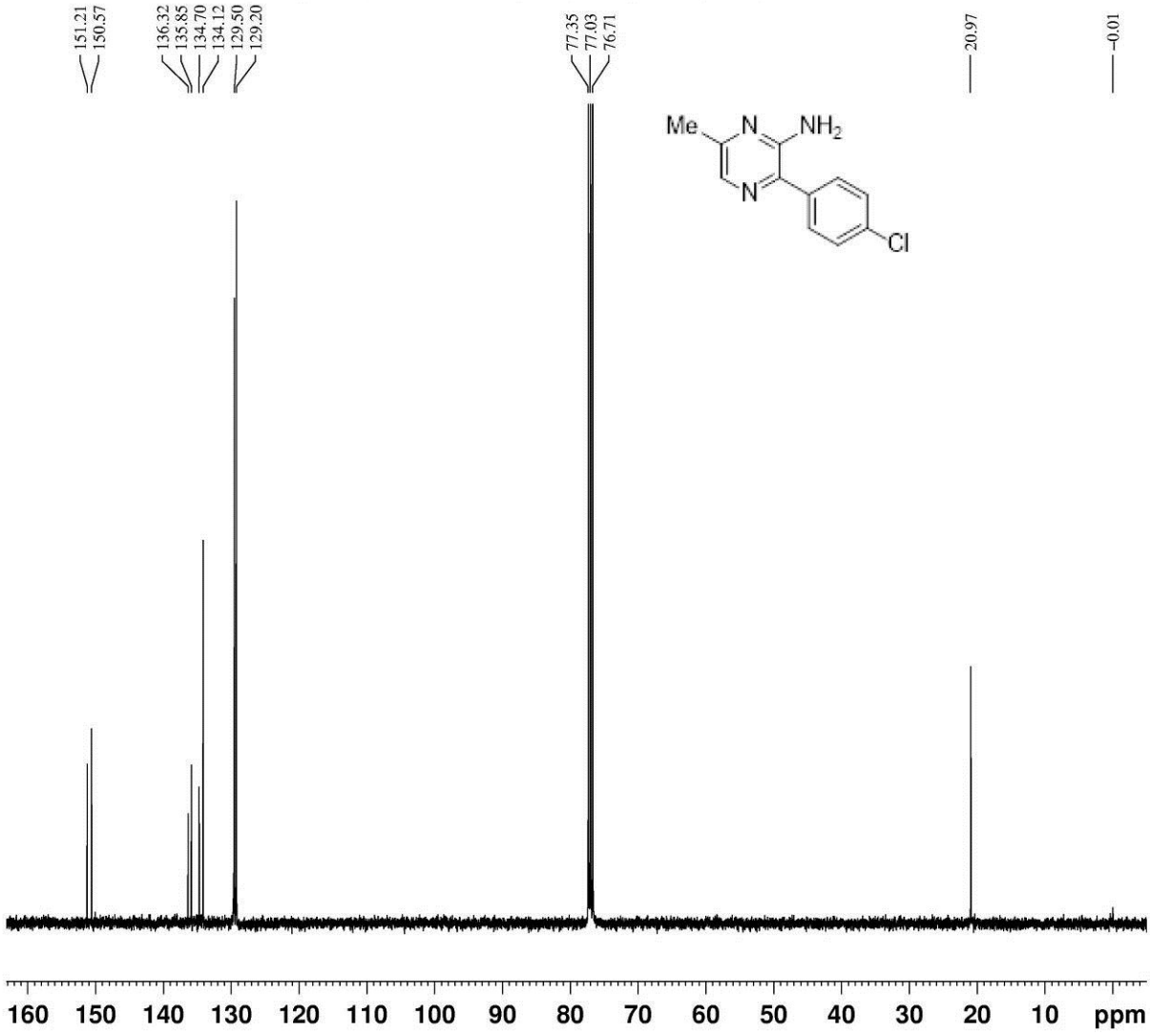
6f: ^{13}C NMR


152.05
151.47
143.94
141.55
132.61
129.56
112.45
110.49
40.61
40.40
40.19
39.98
39.77
39.56
39.36
N
NH2
N
O
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

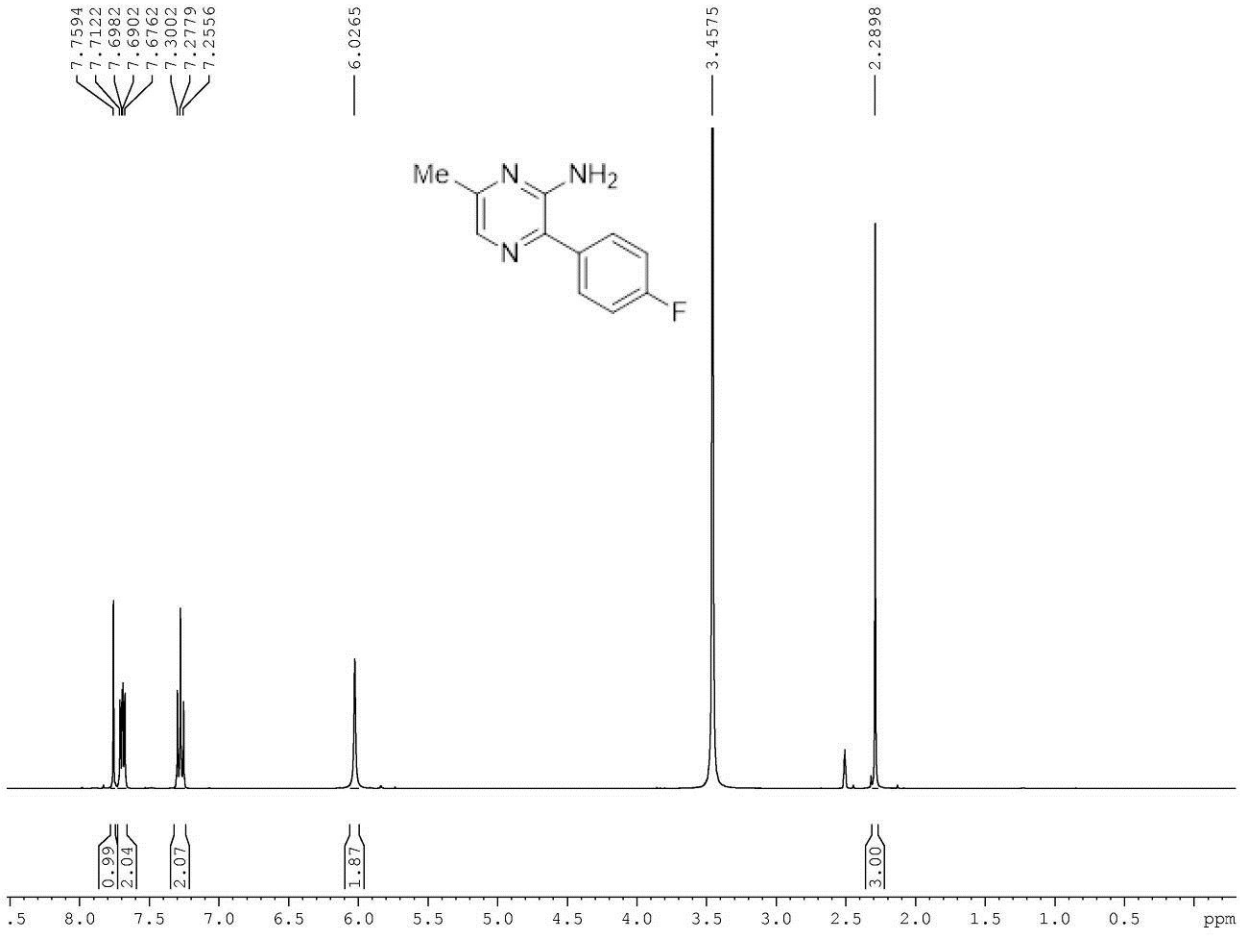
6g: ^{1}H NMR


7.9075
7.6736
7.6675
7.6627
7.6510
7.6462
7.6402
7.4687
7.4624
7.4576
7.4458
7.4411
7.4351
7.2688
4.7166
2.4112
1.8528
0.0003
Me
N
NH2
N
Cl
0.95
2.07
2.10
1.82
3.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

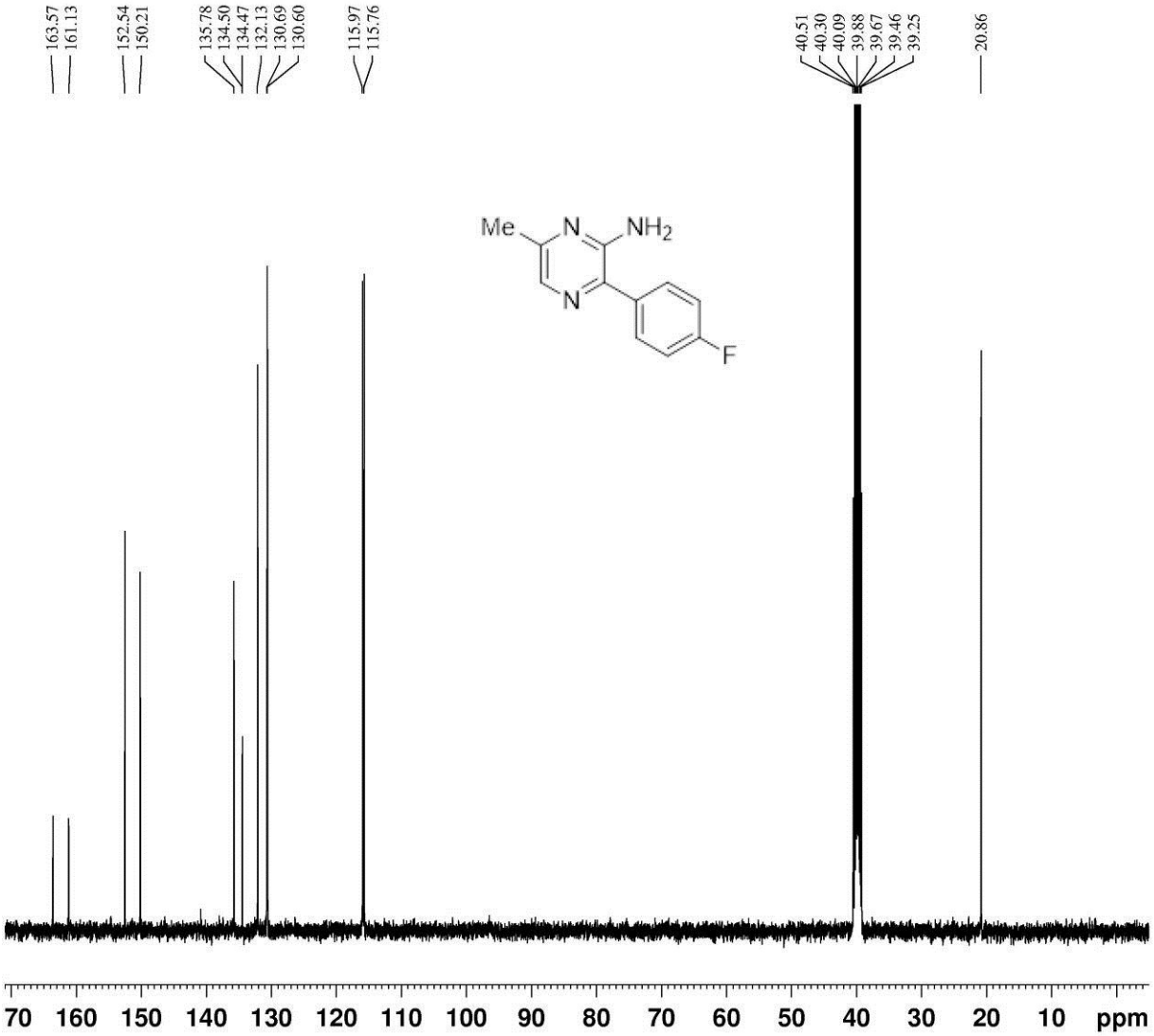
6g: ^{13}C NMR


151.21
150.57
136.32
135.85
134.70
134.12
129.50
129.20
77.35
77.03
76.71
20.97
-0.01
Me
N
NH2
N
Cl
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

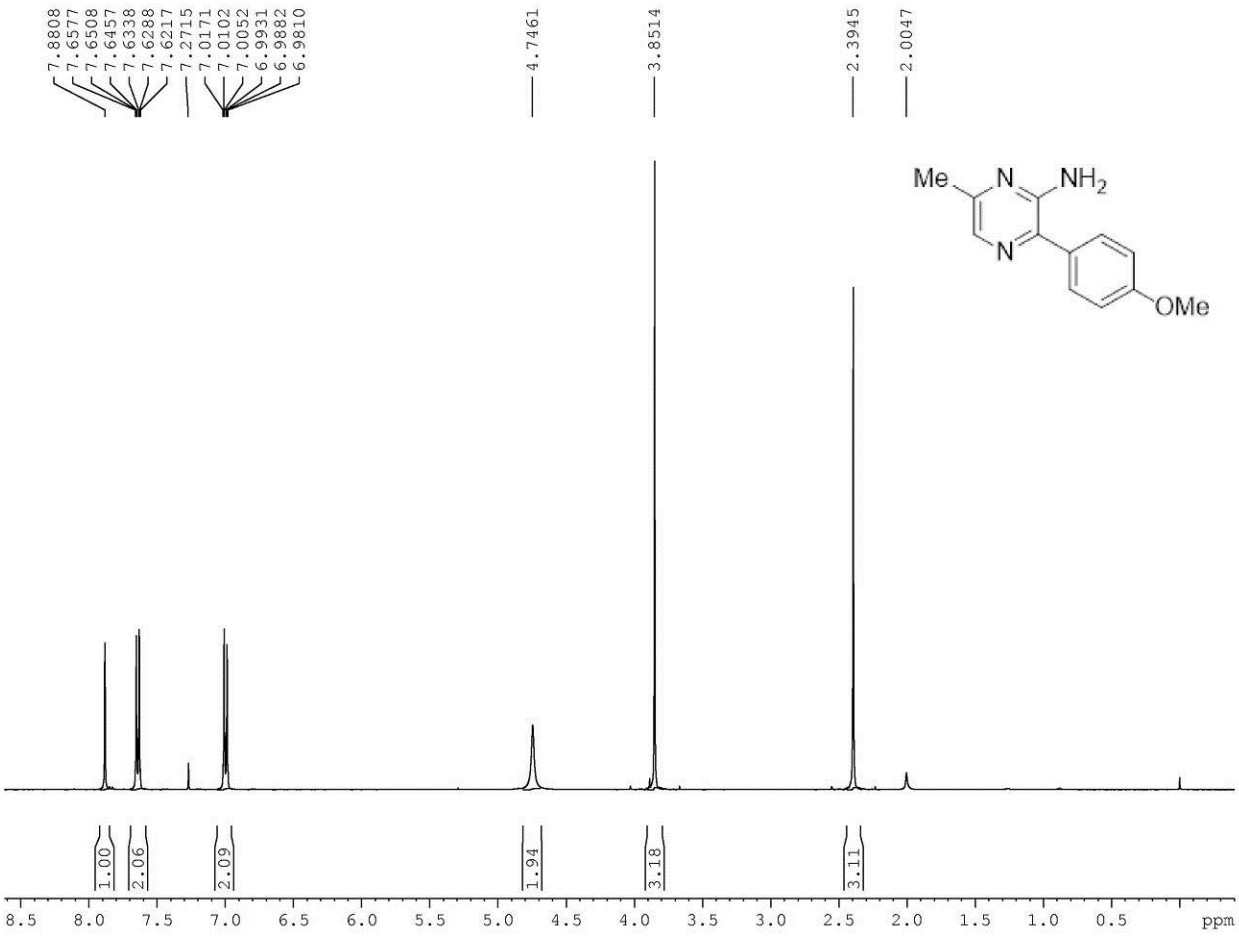
6h: ^{1}H NMR


7.7594
7.7122
7.6982
7.6902
7.6762
7.3002
7.2779
7.2556
6.0265
3.4575
2.2898
Me
N
NH2
N
F
0.99
2.04
2.07
1.87
3.00
.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

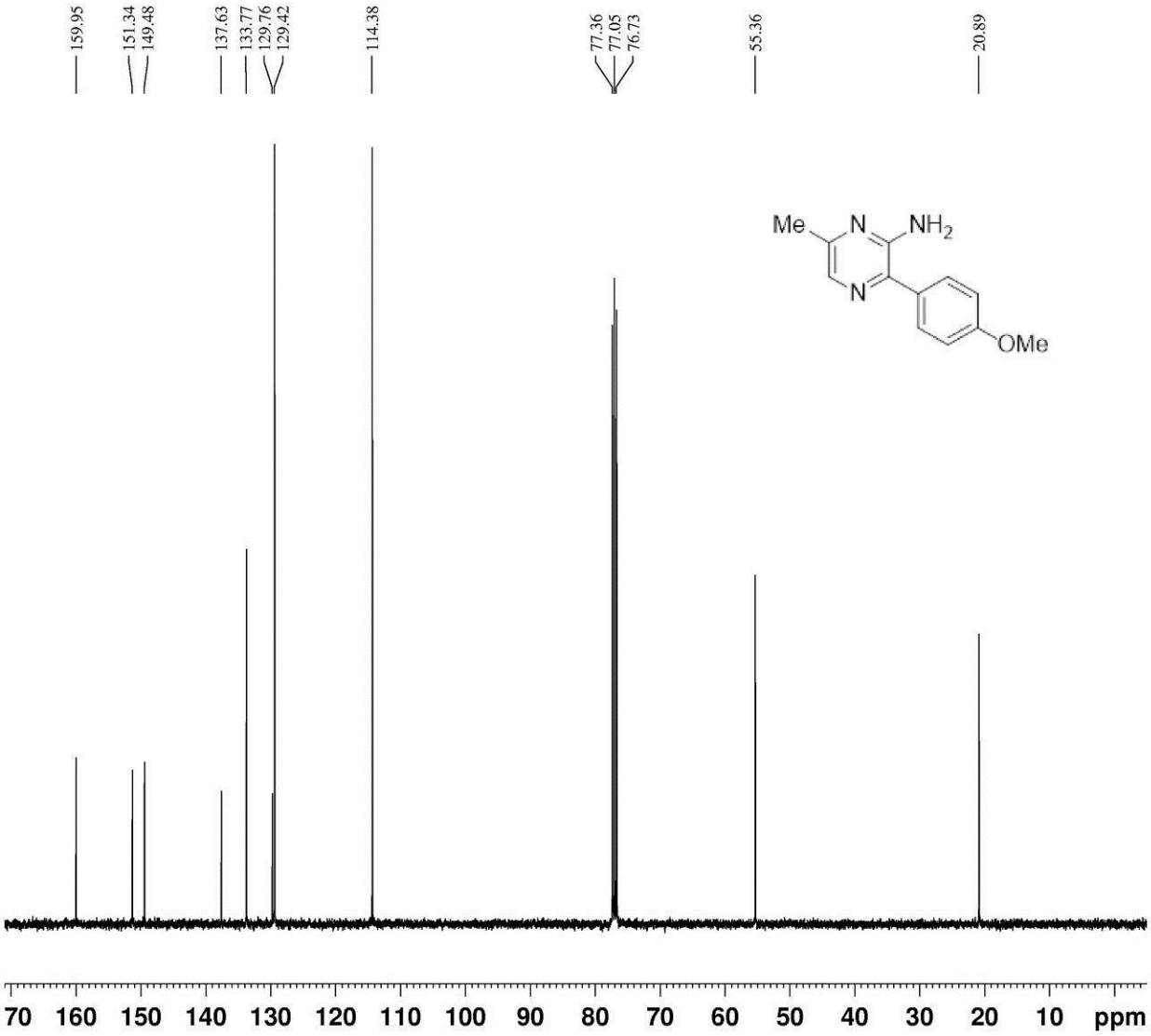
6h: ^{13}C NMR


163.57
161.13
152.54
150.21
135.78
134.50
134.47
132.13
130.69
130.60
115.97
115.76
40.51
40.30
40.09
39.88
39.67
39.46
39.25
20.86
Me
N
NH2
N
F
70 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

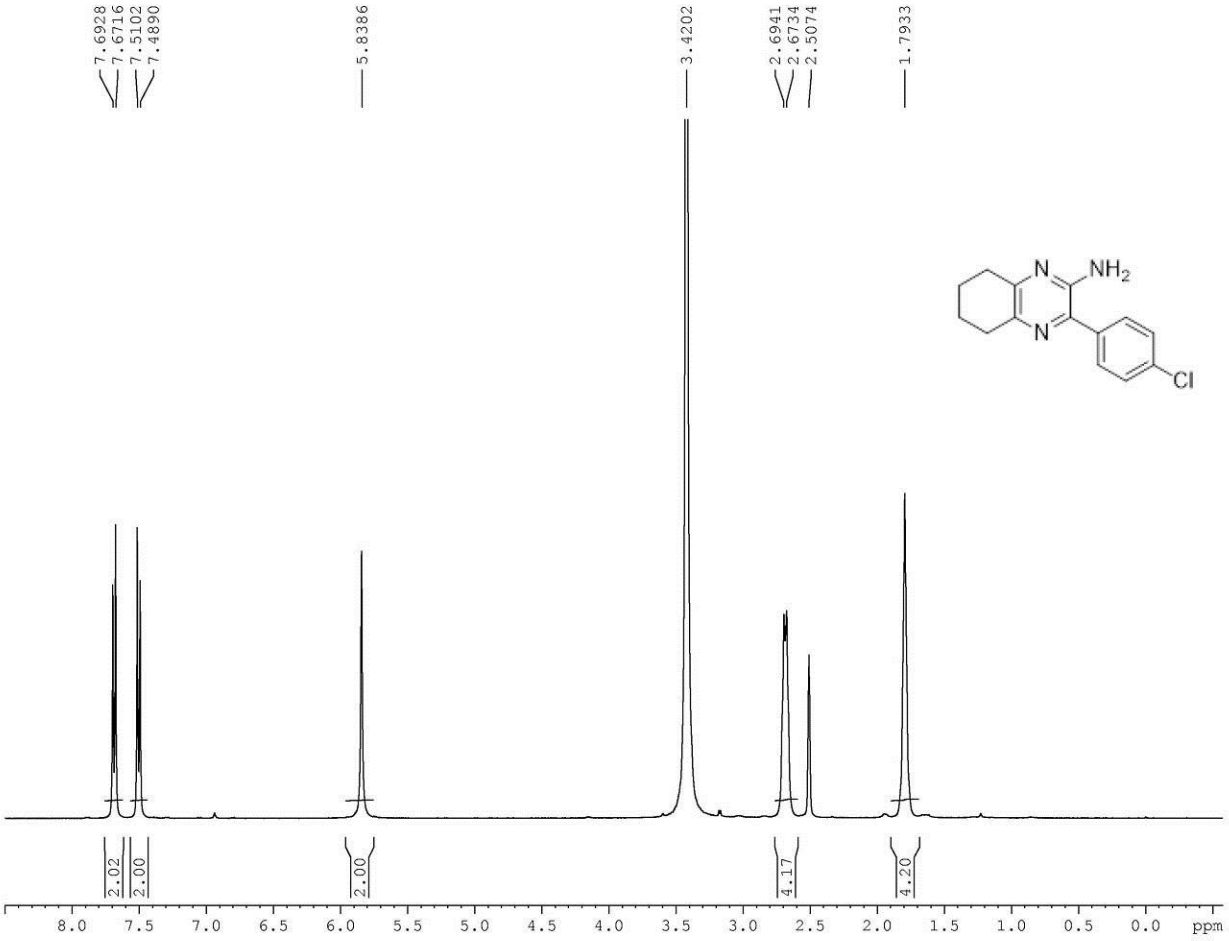
6i: ^{1}H NMR


7.8808
7.6577
7.6508
7.6457
7.6338
7.6288
7.6217
7.2715
7.0171
7.0102
7.0052
6.9931
6.9882
6.9810
4.7461
3.8514
2.3945
2.0047
Me
N
NH2
N
OMe
1.00
2.06
2.09
1.94
3.18
3.11
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

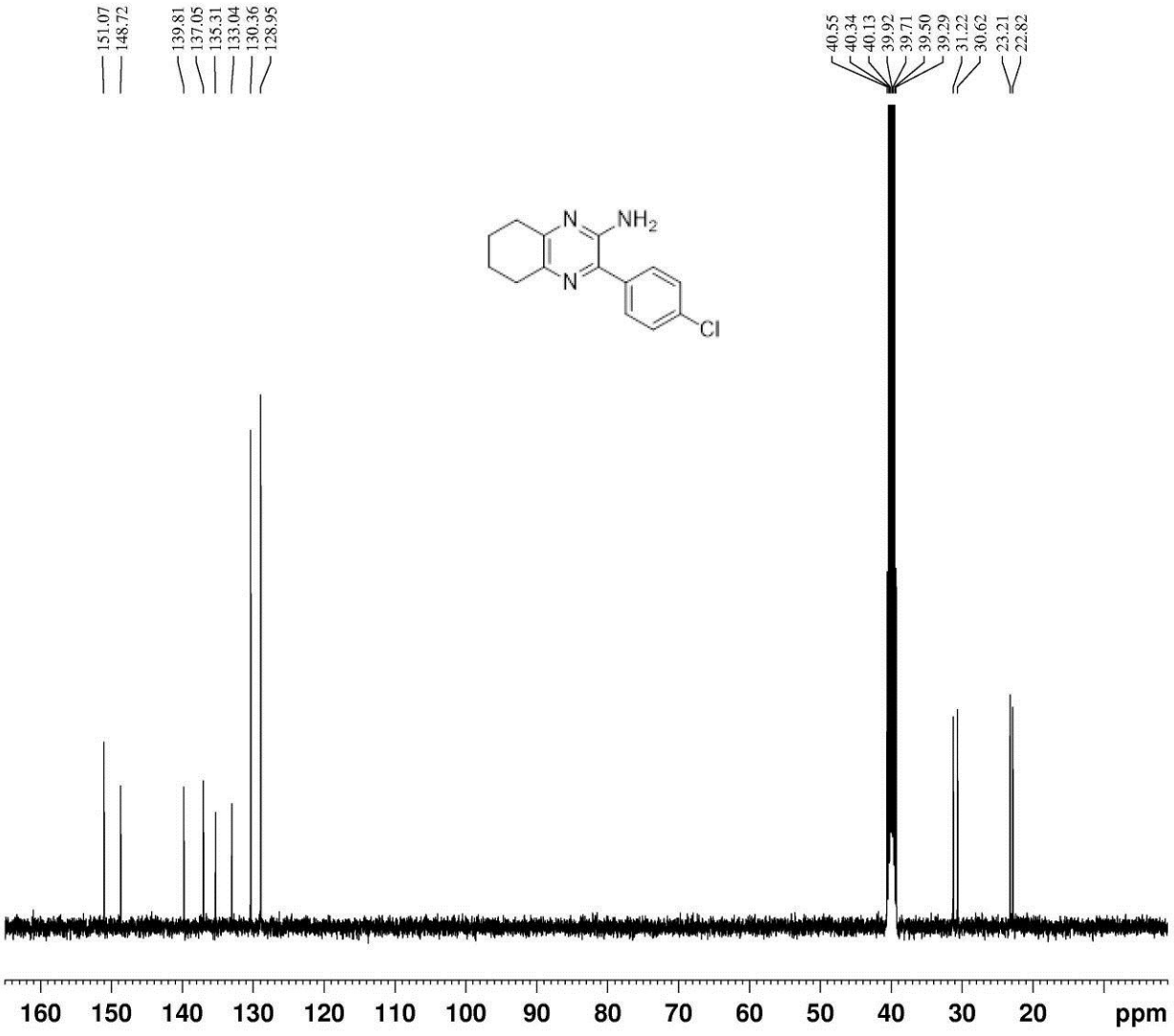
6i: ^{13}C NMR


159.95
151.34
149.48
137.63
133.77
129.76
129.42
114.38
77.36
77.05
76.73
55.36
20.89
Me
N
NH2
N
OMe
70 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

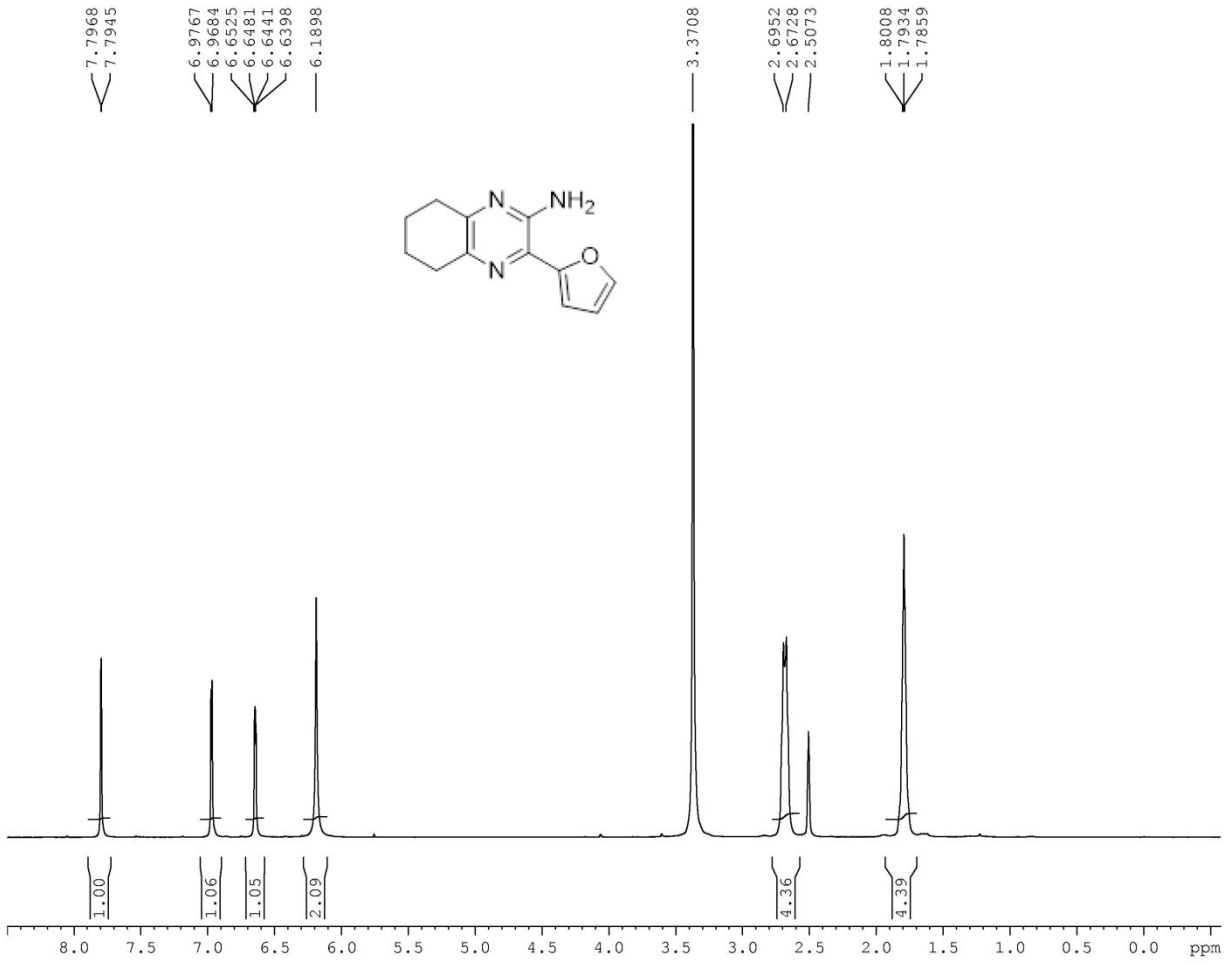
6j: ^{1}H NMR


7.6928
7.6716
7.5102
7.4890
5.8386
3.4202
2.6941
2.6734
2.5074
1.7933
N
NH2
N
Cl
2.02
2.00
2.00
4.17
4.20
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

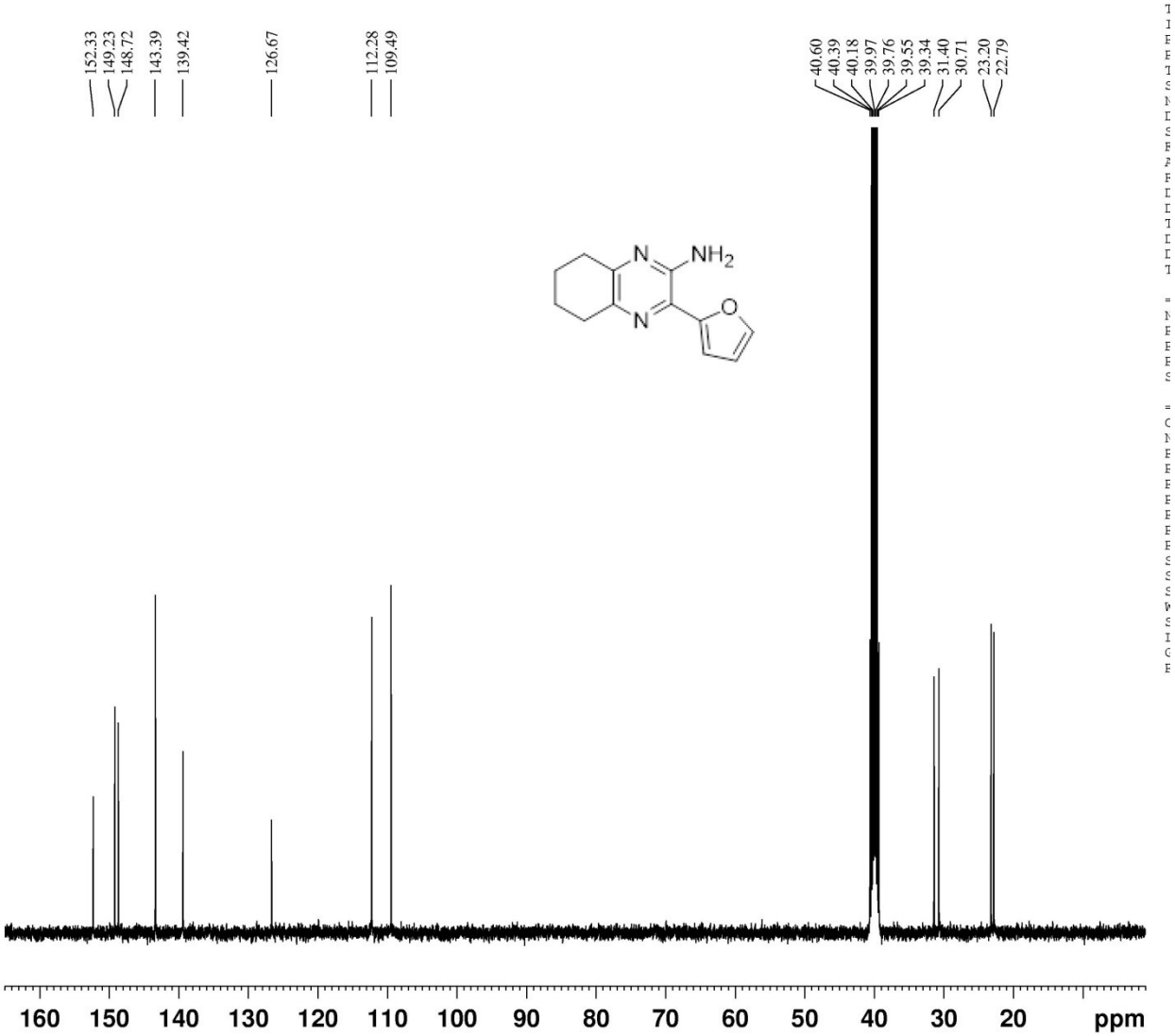
6j: ^{13}C NMR


151.07
148.72
139.81
137.05
135.31
133.04
130.36
128.95
40.55
40.34
40.13
39.92
39.71
39.50
39.29
31.22
30.62
23.21
22.82
N
NH2
N
Cl
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

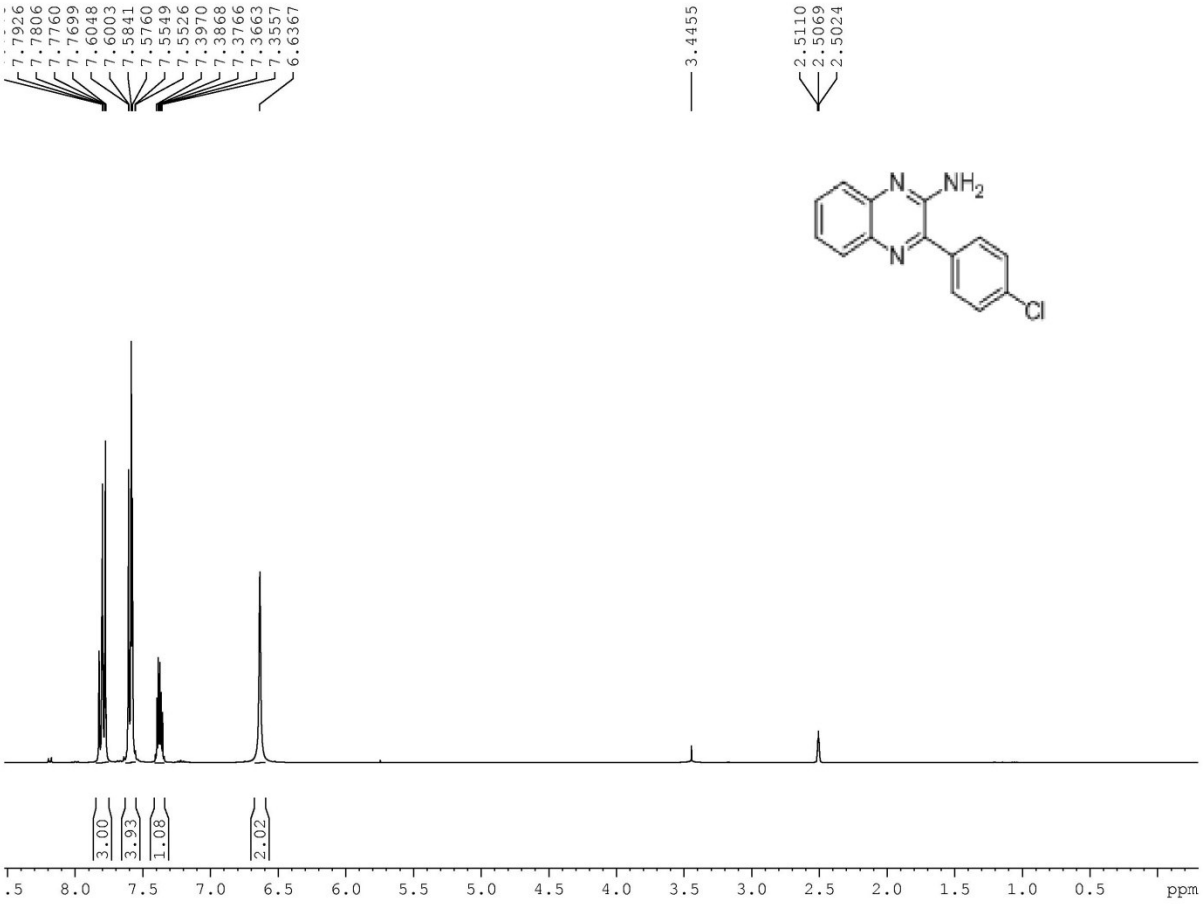
6k: ^{1}H NMR


7.7968
7.7945
6.9767
6.9684
6.6525
6.6481
6.6441
6.6398
6.1898
3.3708
2.6952
2.6728
2.5073
1.8008
1.7934
1.7859
NH2
N
N
O
1.00
1.06
1.05
2.09
4.36
4.39
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

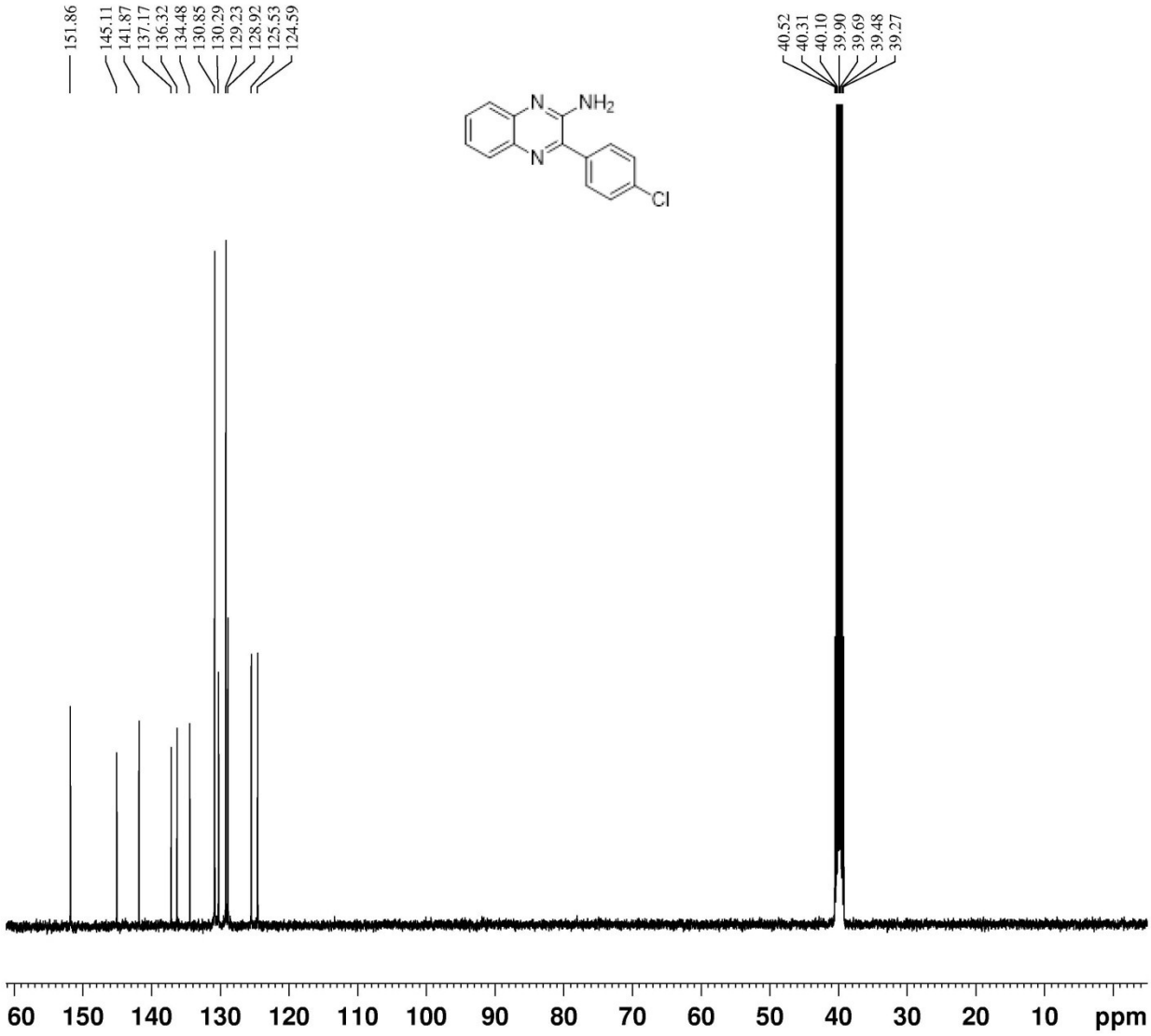
6k: ^{13}C NMR


152.33
149.23
148.72
143.39
139.42
126.67
112.28
109.49
40.60
40.39
40.18
39.97
39.76
39.55
39.34
31.40
30.71
23.20
22.79
N
NH2
N
O
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

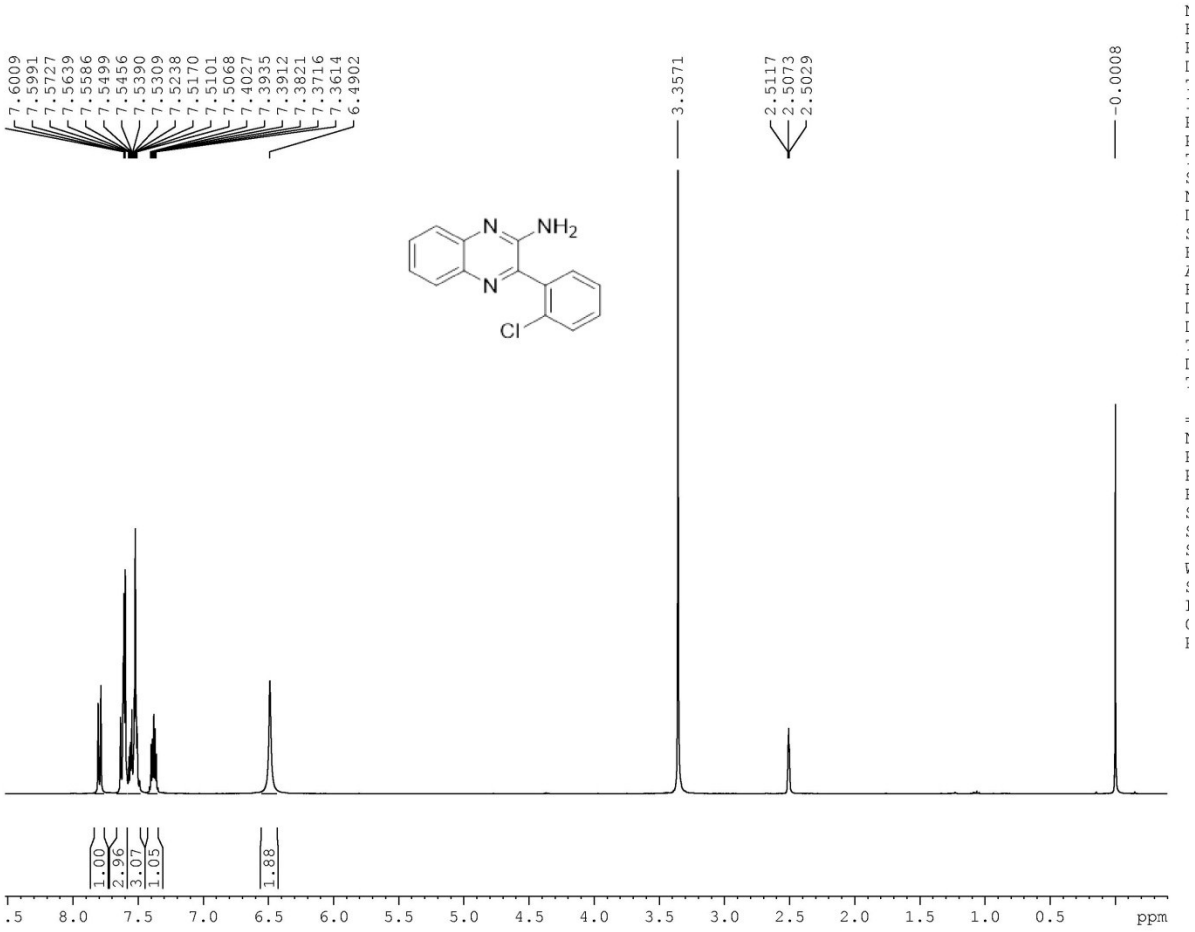
4a: ^{1}H NMR


7.7926
7.7806
7.7760
7.7699
7.6048
7.6003
7.5841
7.5760
7.5549
7.5526
7.3970
7.3868
7.3766
7.3663
7.3557
6.6367
3.4455
2.5110
2.5069
2.5024
N
NH2
N
Cl
3.00
3.93
1.08
2.02
.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

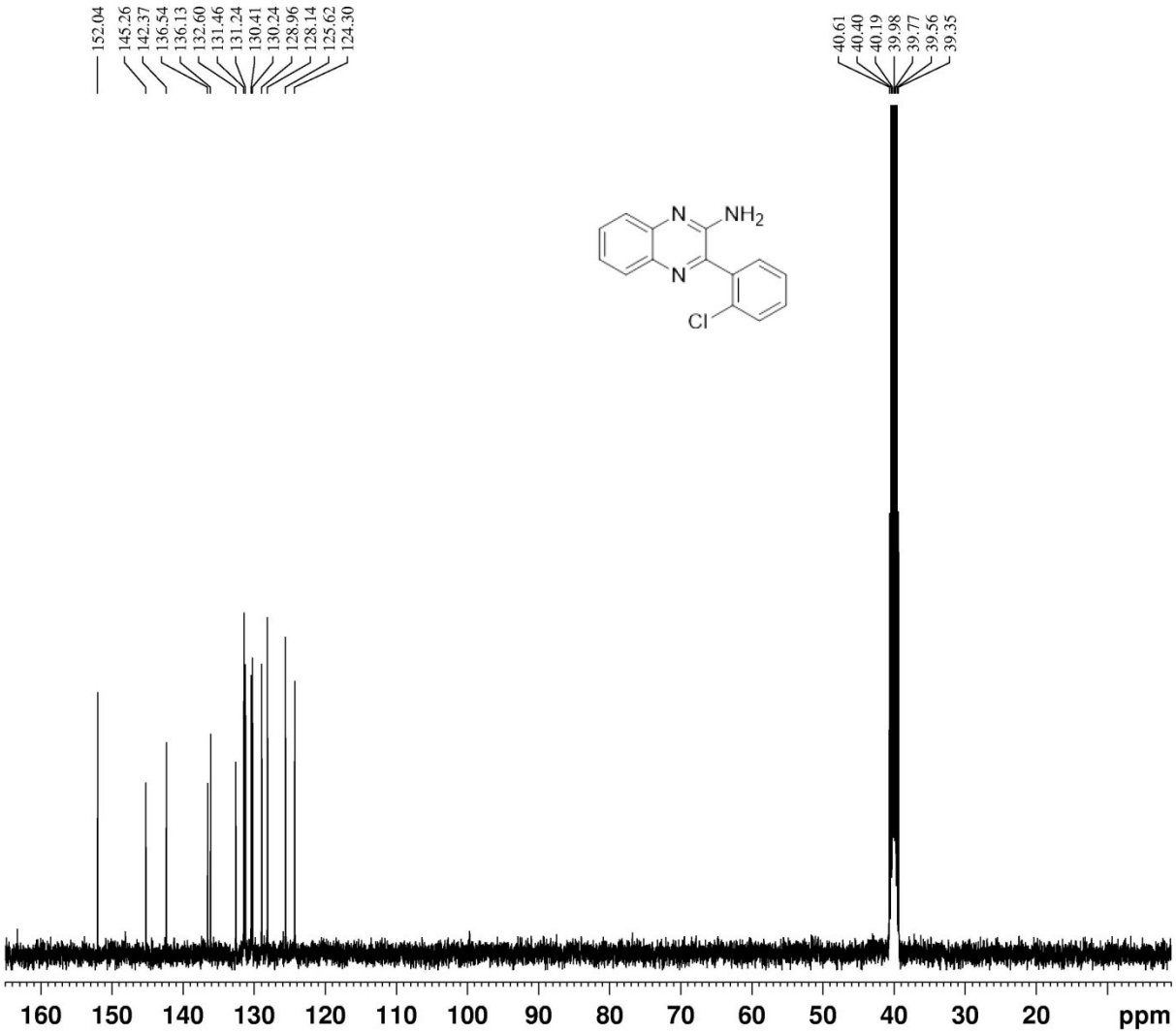
4a: ^{13}C NMR


151.86
145.11
141.87
137.17
136.32
134.48
130.85
130.29
129.23
128.92
125.53
124.59
40.52
40.31
40.10
39.90
39.69
39.48
39.27
N
NH2
N
Cl
60 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

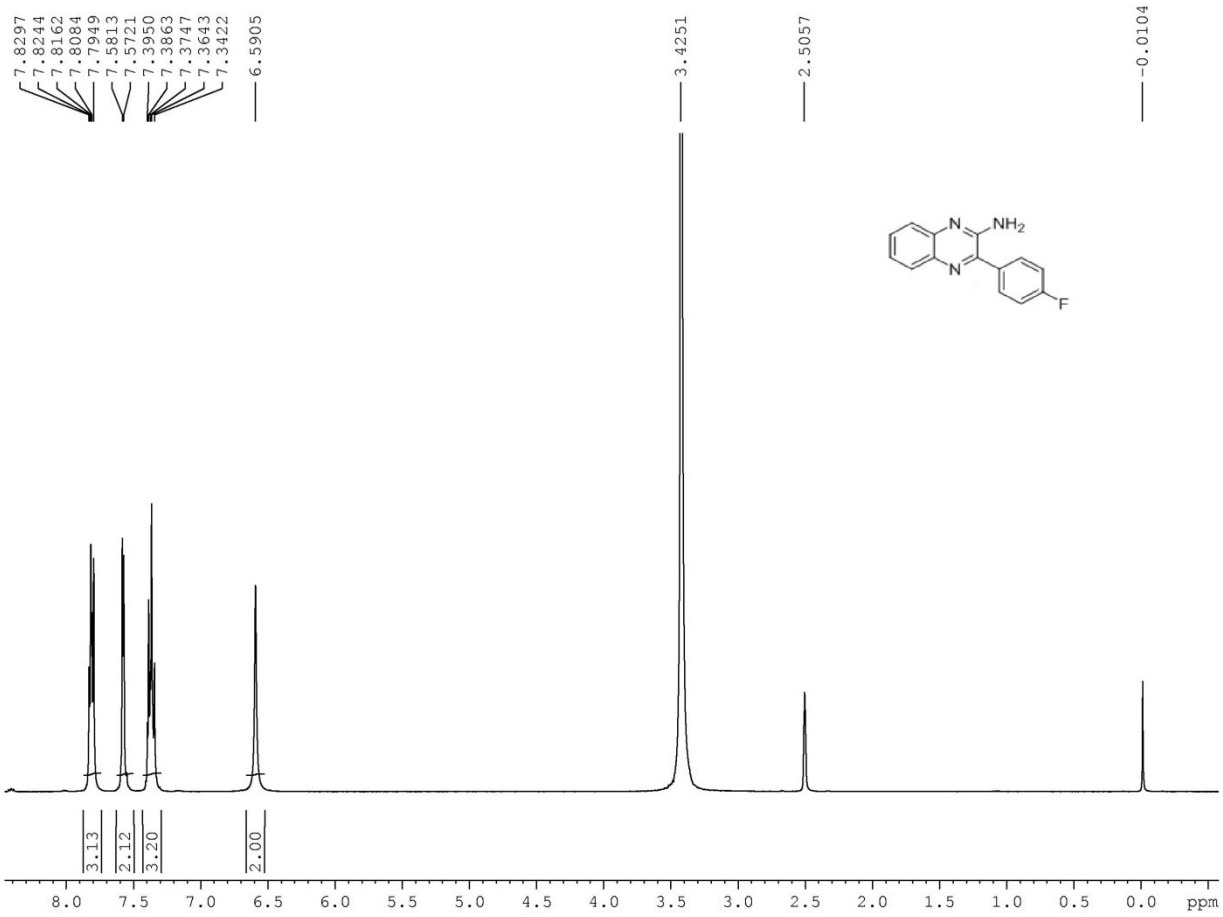
4b: ^{1}H NMR


7.6009
7.5991
7.5727
7.5639
7.5586
7.5499
7.5456
7.5390
7.5309
7.5238
7.5170
7.5101
7.5068
7.4027
7.3935
7.3912
7.3821
7.3716
7.3614
6.4902
3.3571
2.5117
2.5073
2.5029
0.0008
N
NH2
N
Cl
1.00
2.96
3.07
1.05
1.88
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

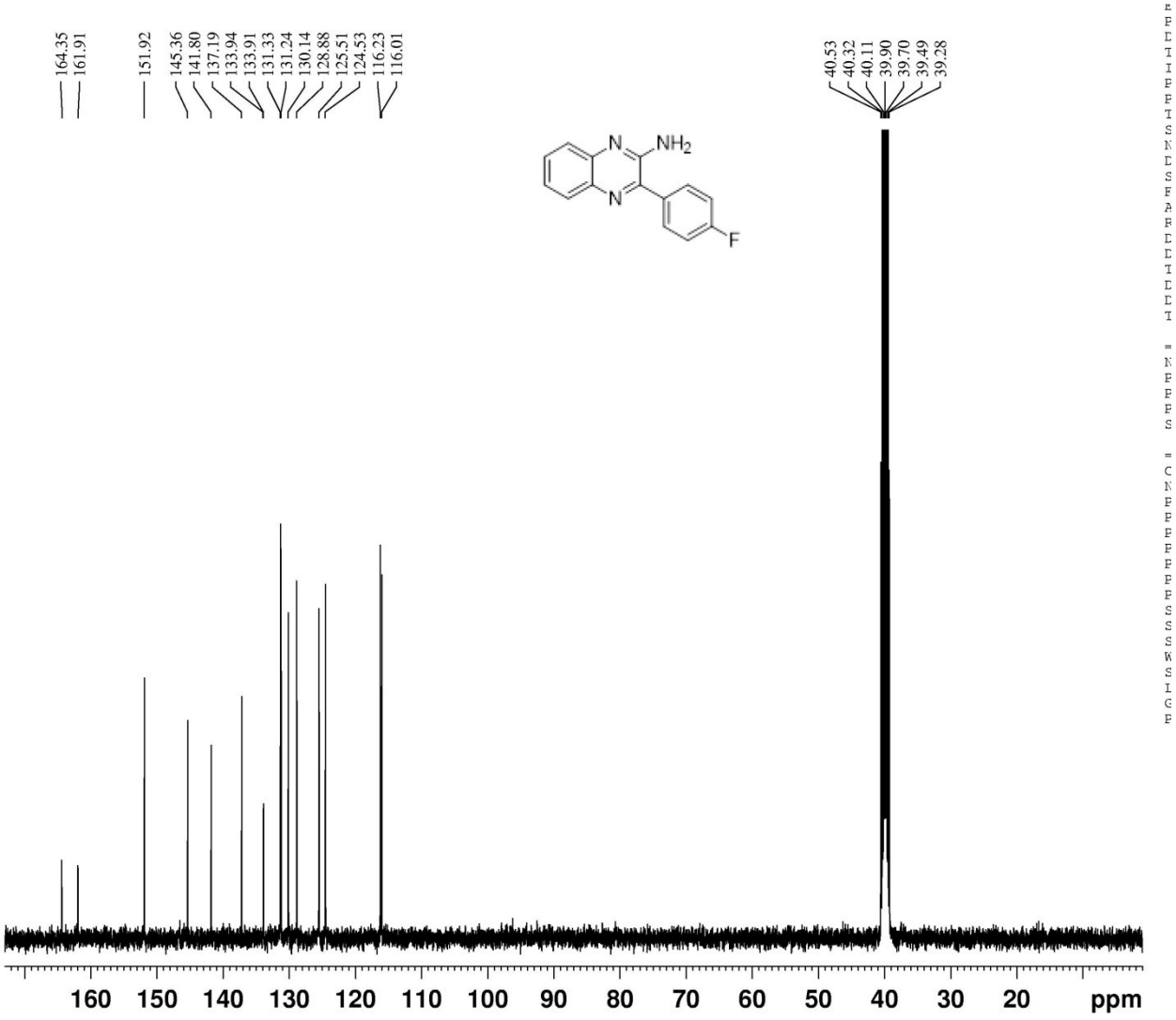
4b: ^{13}C NMR


152.04
145.26
142.37
136.54
136.13
132.60
131.46
131.24
130.41
130.24
128.96
128.14
125.62
124.30
40.61
40.40
40.19
39.98
39.77
39.56
39.35
N
NH2
N
Cl
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

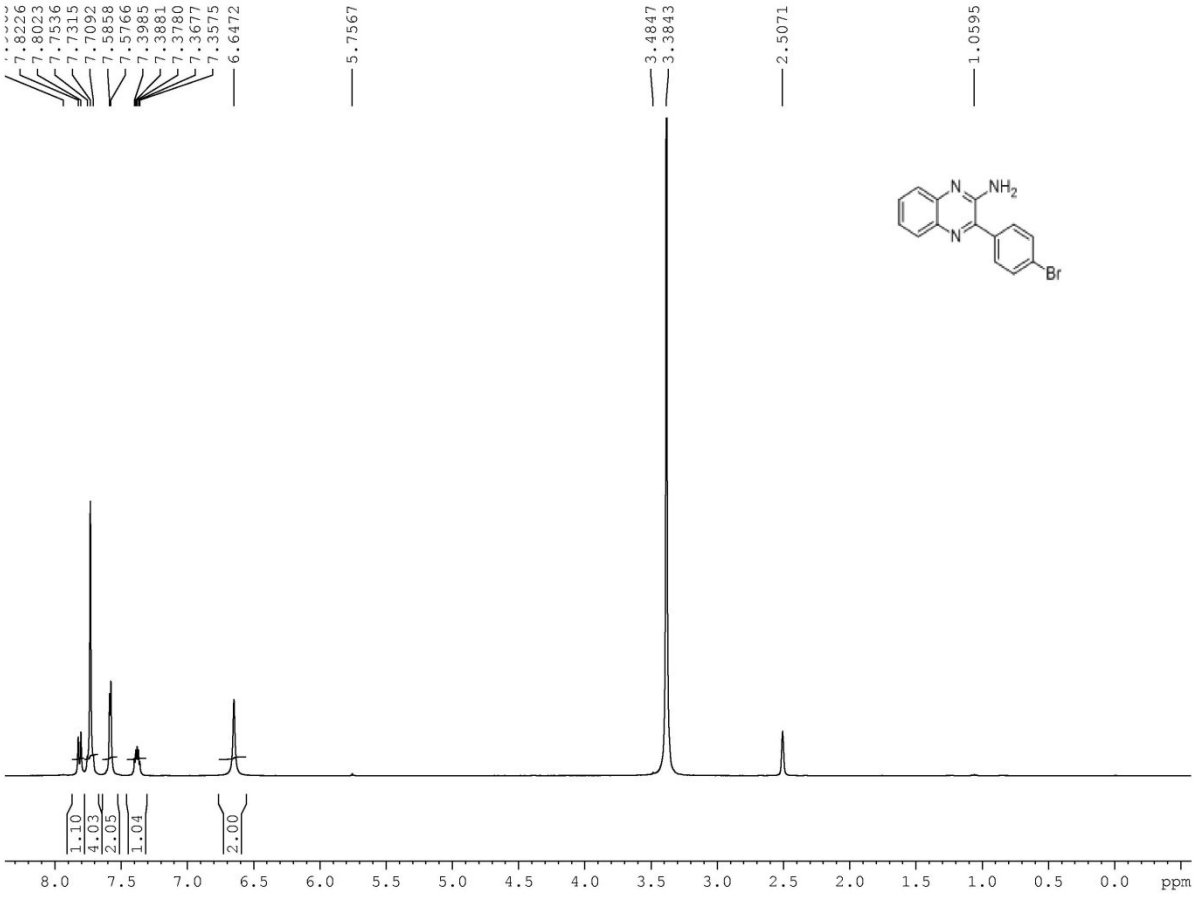
4c: ^{1}H NMR


7.8297
7.8244
7.8162
7.8084
7.7949
7.5813
7.5721
7.3950
7.3863
7.3747
7.3643
7.3422
6.5905
3.4251
2.5057
-0.0104
NH2
N
N
F
3.13
2.12
3.20
2.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

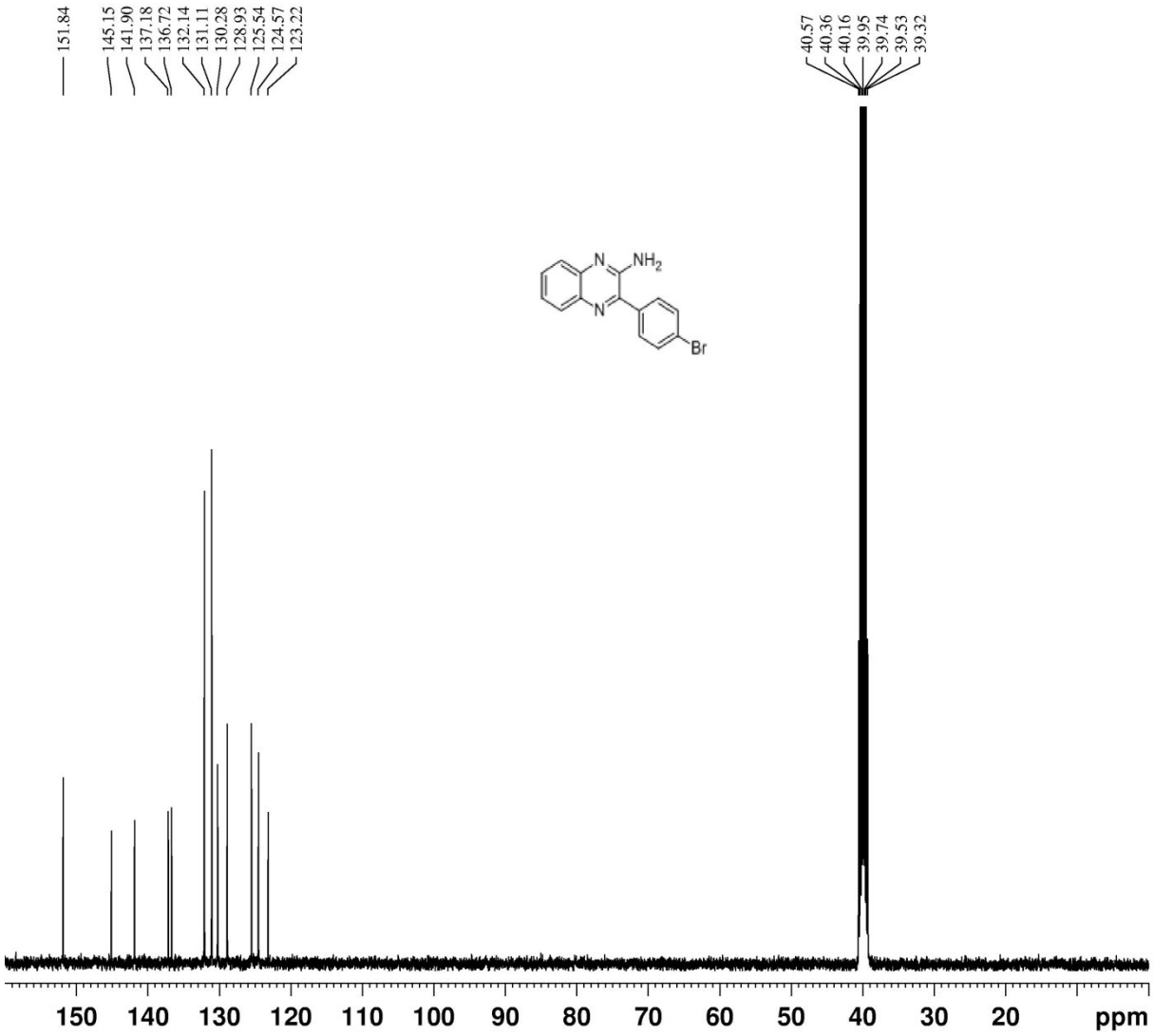
4c: ^{13}C NMR


164.35
161.91
151.92
145.36
141.80
137.19
133.94
133.91
131.33
131.24
130.14
128.88
125.51
124.53
116.23
116.01
40.53
40.32
40.11
39.90
39.70
39.49
39.28
NH2
N
N
F
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

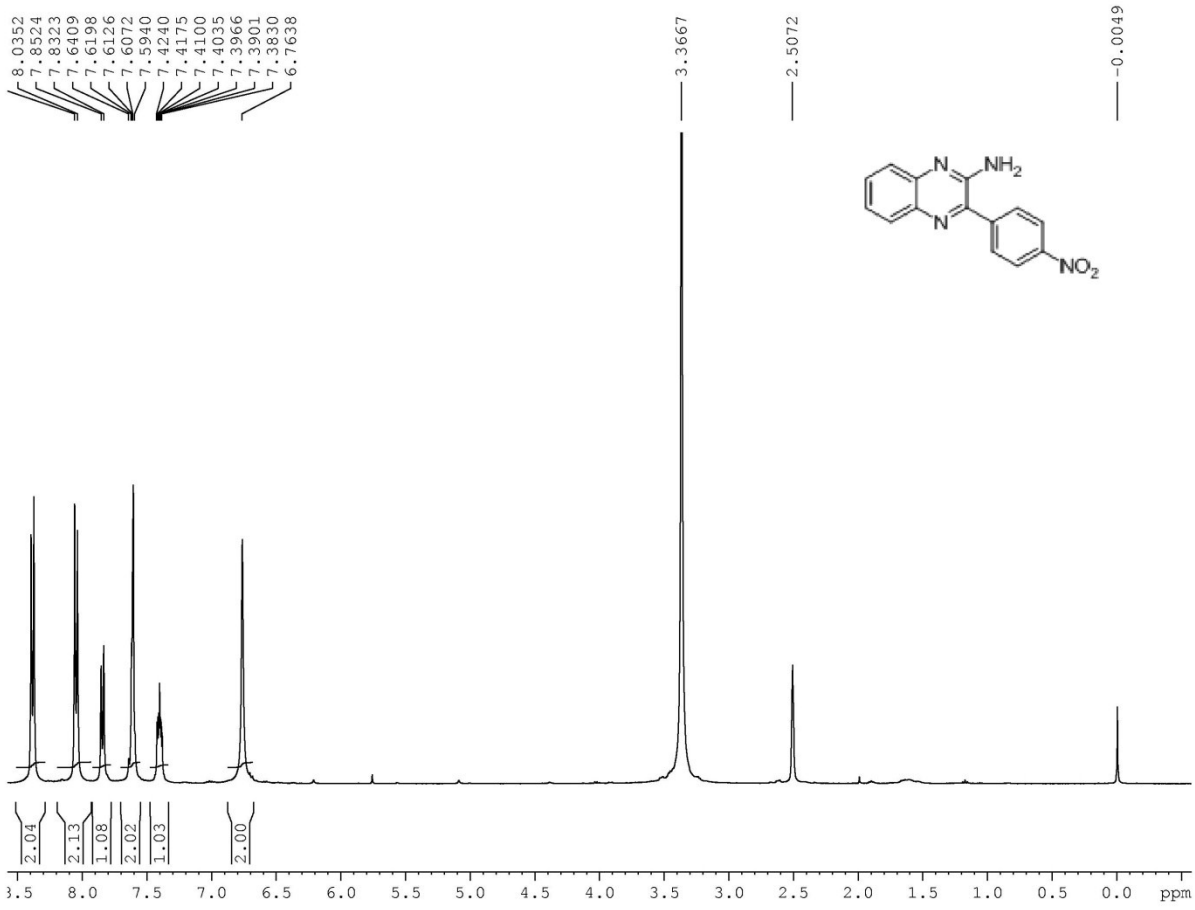
4d: ^{1}H NMR


7.8226
7.8023
7.7536
7.7315
7.7092
7.5858
7.5766
7.3985
7.3881
7.3780
7.3677
7.3575
6.6472
5.7567
3.4847
3.3843
2.5071
1.0595
NH2
Br
1.10
4.03
2.05
1.04
2.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

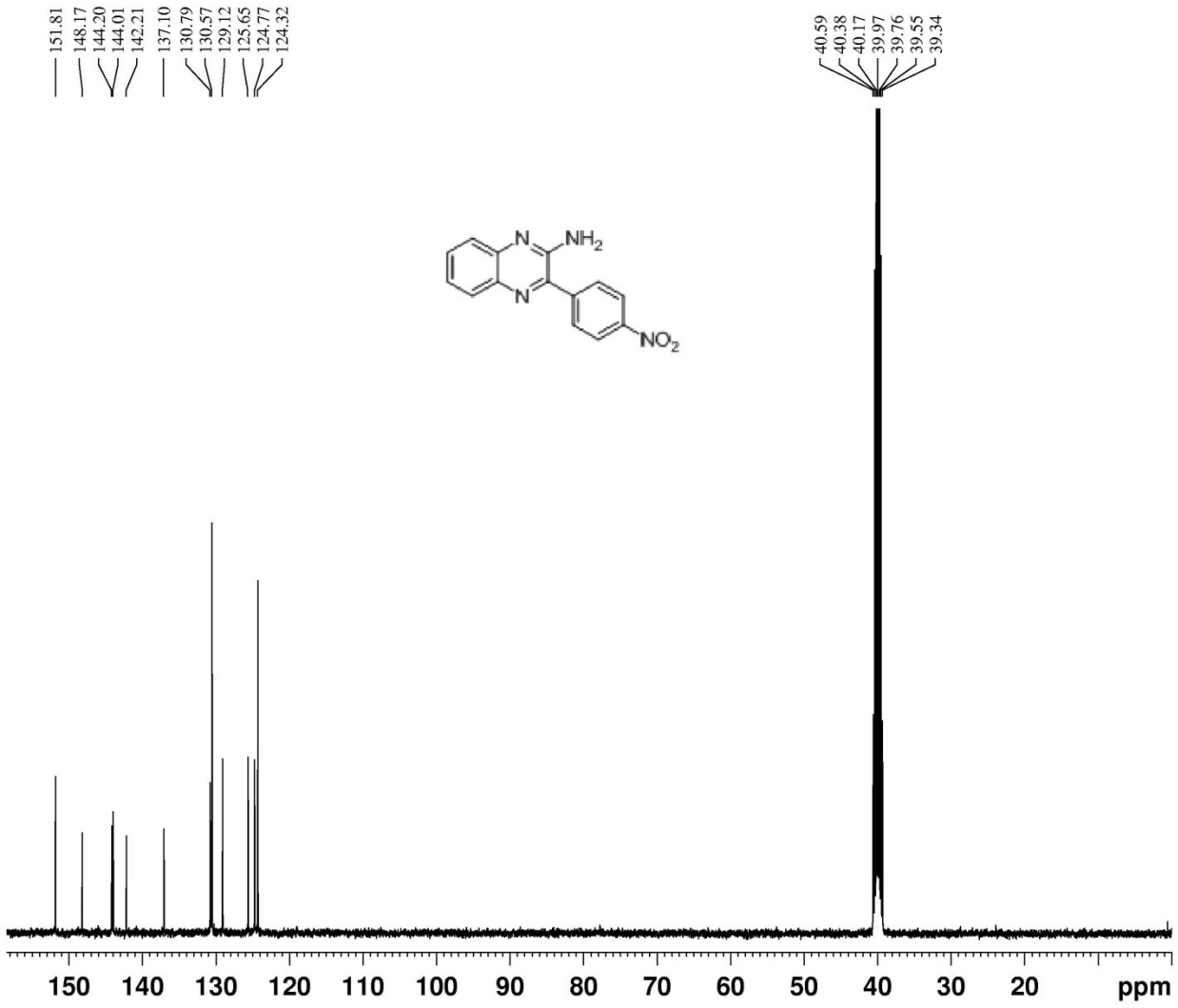
4d: ^{13}C NMR


151.84
145.15
141.90
137.18
136.72
132.14
131.11
130.28
128.93
125.54
124.57
123.22
40.57
40.36
40.16
39.95
39.74
39.53
39.32
N
NH2
N
Br
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

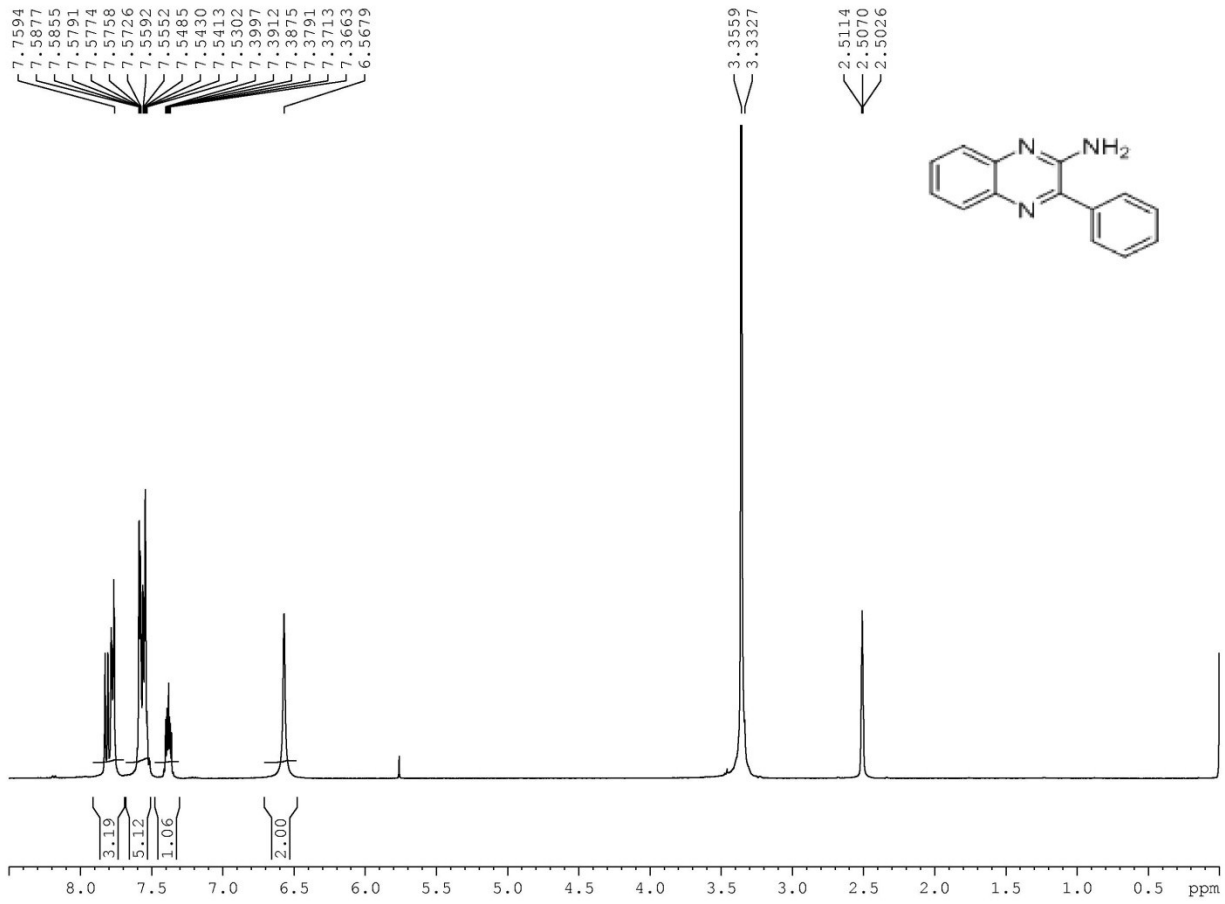
4e: ^{1}H NMR


8.0352
7.8524
7.8323
7.6409
7.6198
7.6126
7.6072
7.5940
7.4240
7.4175
7.4100
7.4035
7.3966
7.3901
7.3830
6.7638
3.3667
2.5072
-0.0049
NH2
N
N
NO2
2.04
2.13
1.08
2.02
1.03
2.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

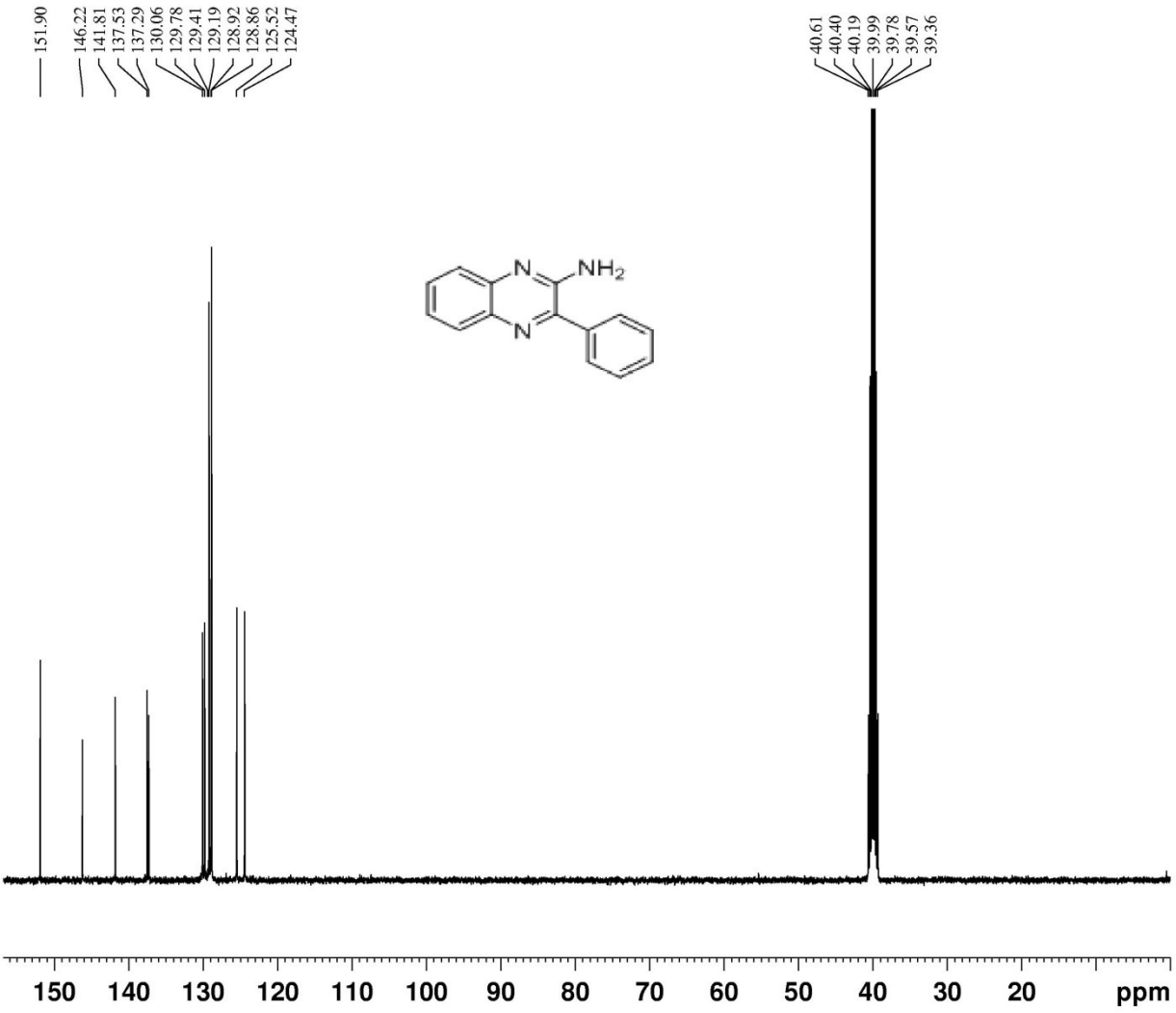
4e: ^{13}C NMR


151.81
148.17
144.20
144.01
142.21
137.10
130.79
130.57
129.12
125.65
124.77
124.32
40.59
40.38
40.17
39.97
39.76
39.55
39.34
N
NH2
N
NO2
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

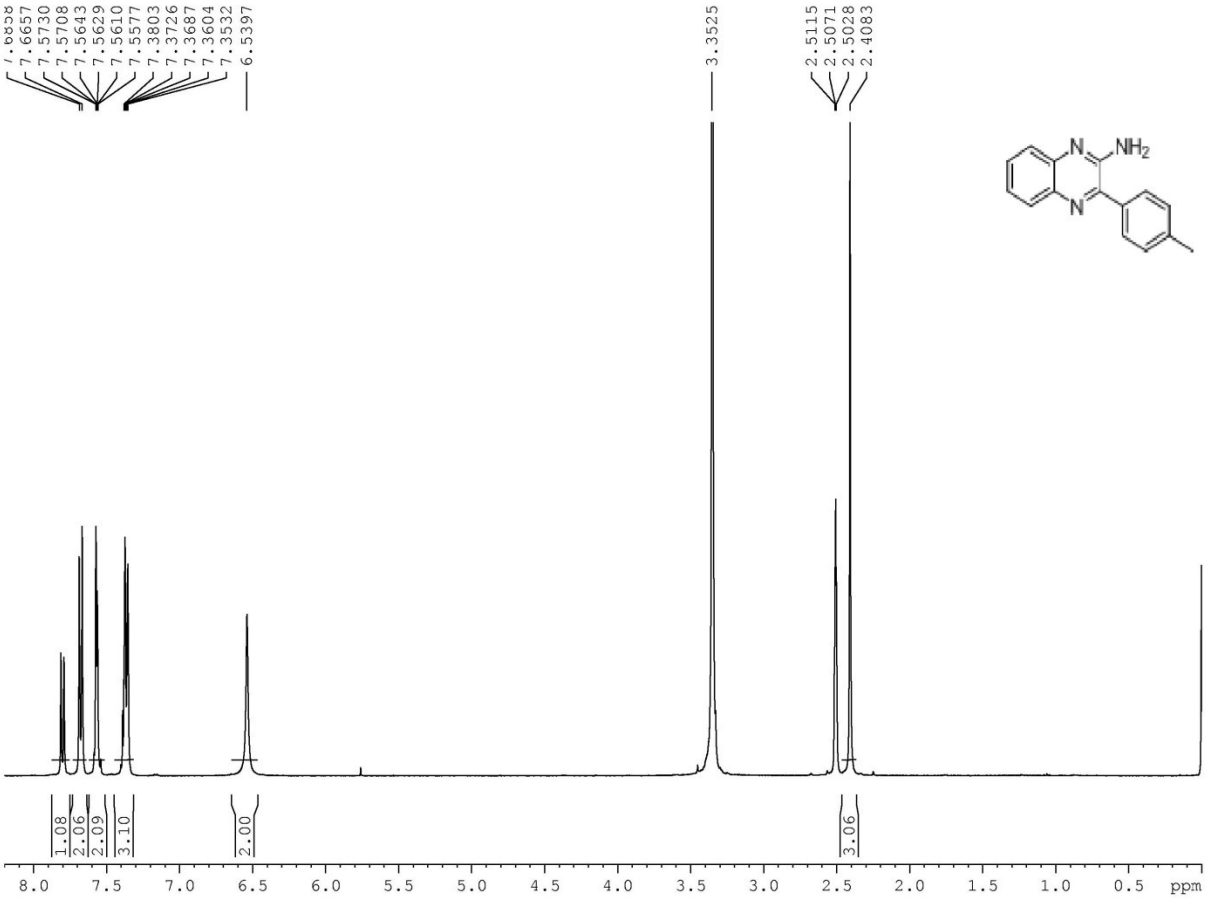
4f: ^{1}H NMR


7.7594
7.5877
7.5855
7.5791
7.5774
7.5758
7.5726
7.5592
7.5552
7.5485
7.5430
7.5413
7.5302
7.3997
7.3912
7.3875
7.3791
7.3713
7.3663
6.5679
3.3559
3.3327
2.5114
2.5070
2.5026
N
NH2
N
3.19
5.12
1.06
2.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

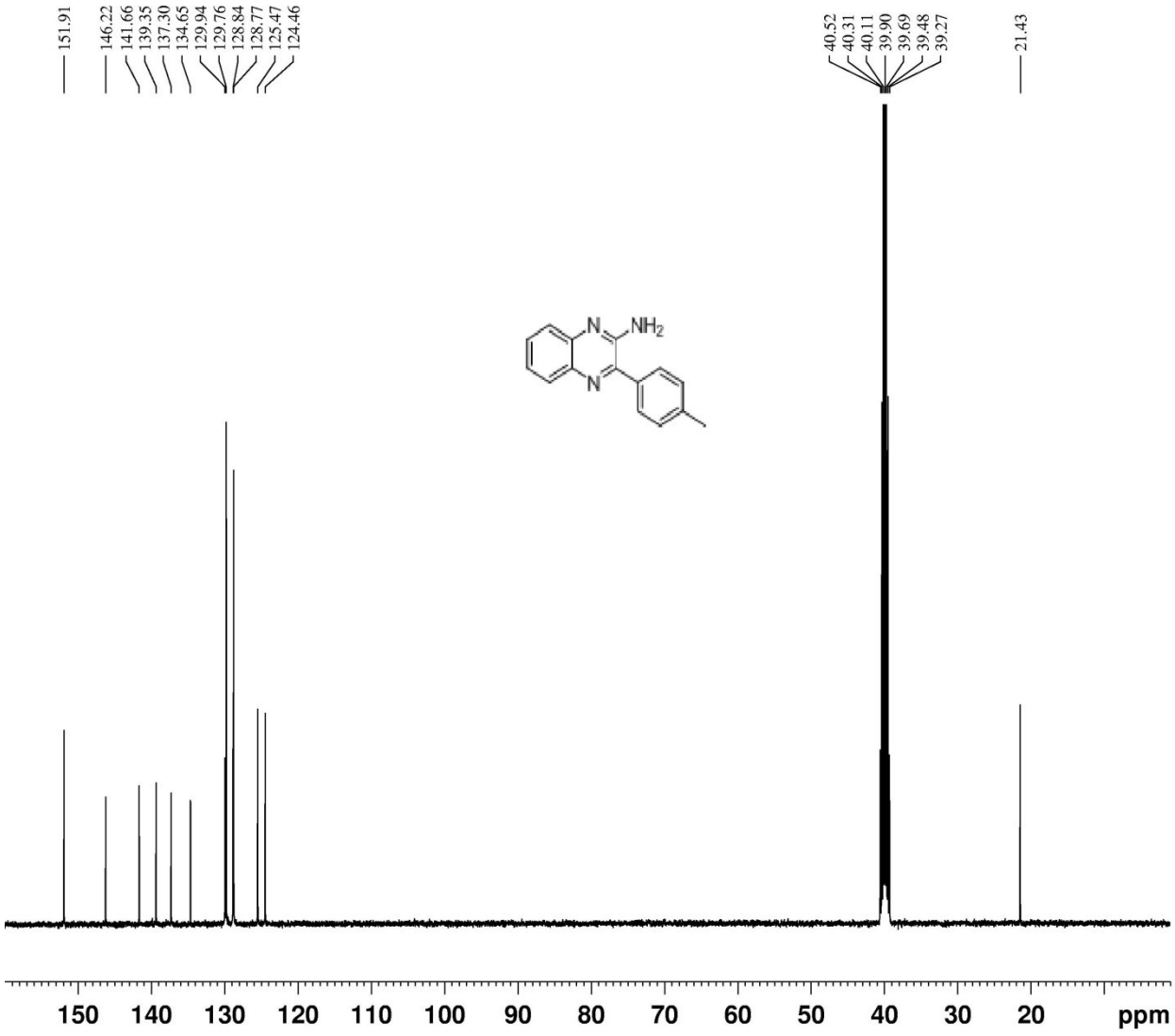
4f: ^{13}C NMR


151.90
146.22
141.81
137.53
137.29
130.06
129.78
129.41
129.19
128.92
128.86
125.52
124.47
40.61
40.40
40.19
39.99
39.78
39.57
39.36
N
NH2
N
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

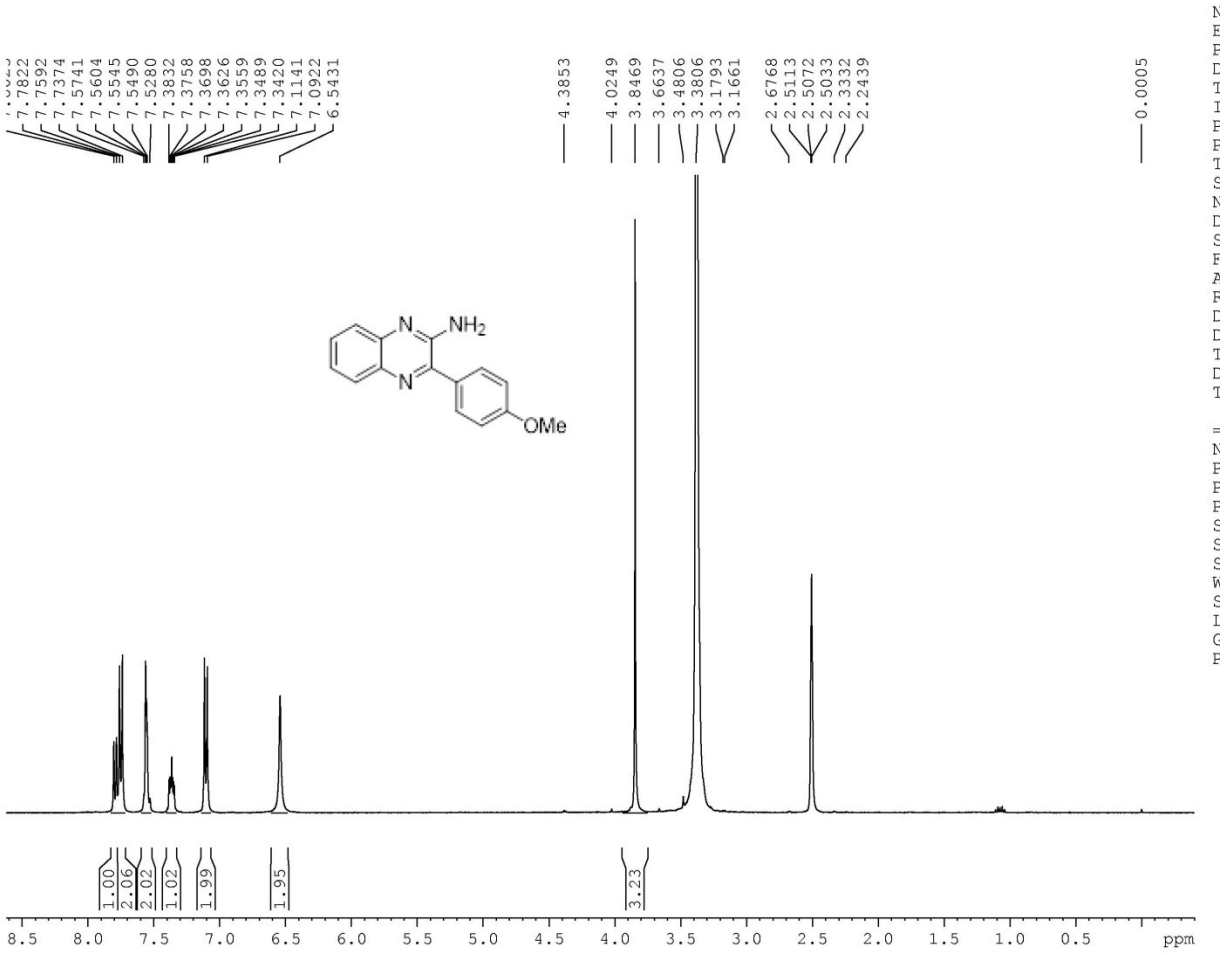
4g: ^{1}H NMR


7.6657
7.5730
7.5708
7.5643
7.5629
7.5610
7.5577
7.3803
7.3726
7.3687
7.3604
7.3532
6.5397
3.3525
2.5115
2.5071
2.5028
2.4083
NH2
1.08
2.06
2.09
3.10
2.00
3.06
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

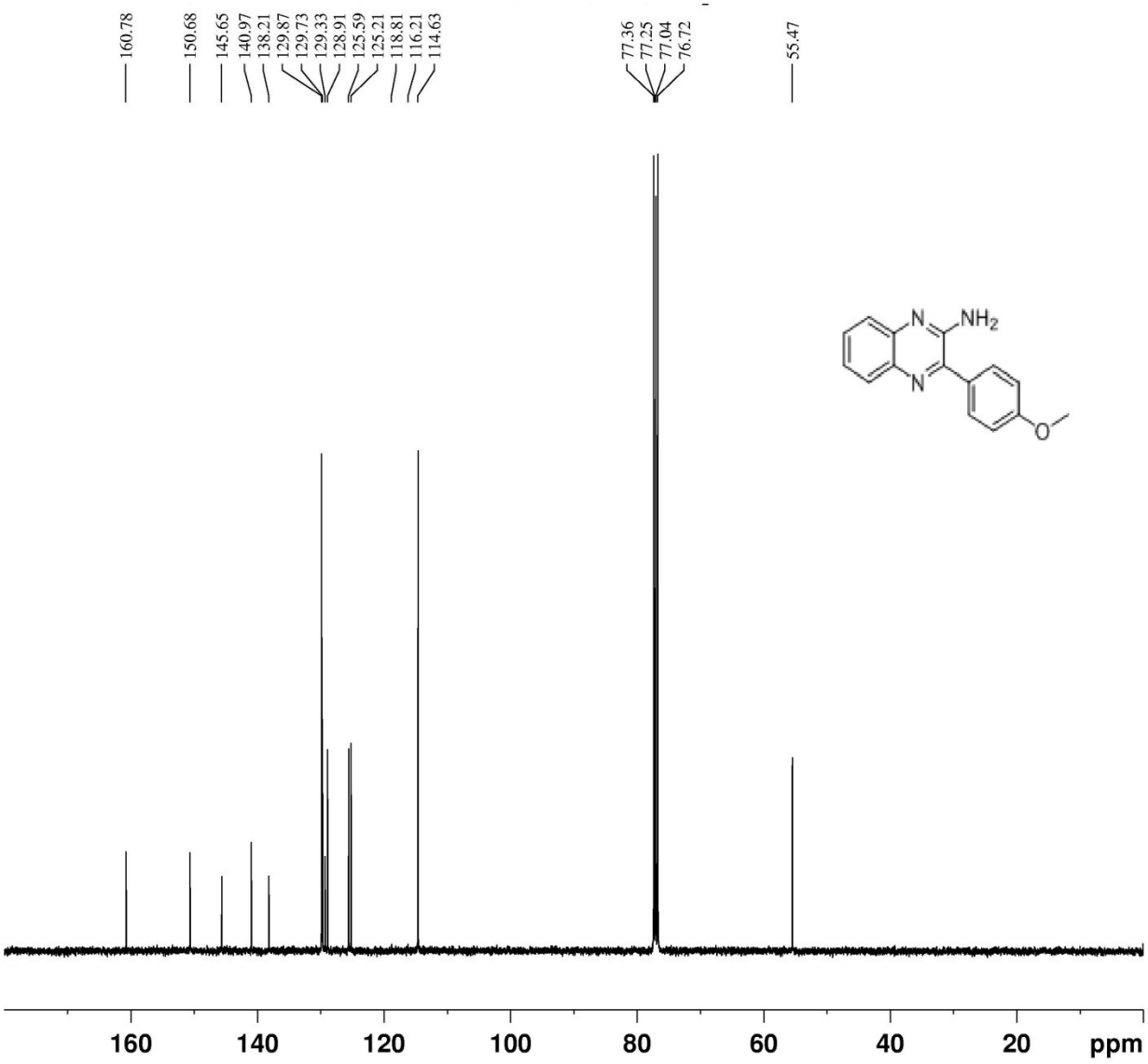
4g: ^{13}C NMR


151.91
146.22
141.66
139.35
137.30
134.65
129.94
129.76
128.84
128.77
125.47
124.46
40.52
40.31
40.11
39.90
39.69
39.48
39.27
21.43
N
NH2
N
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

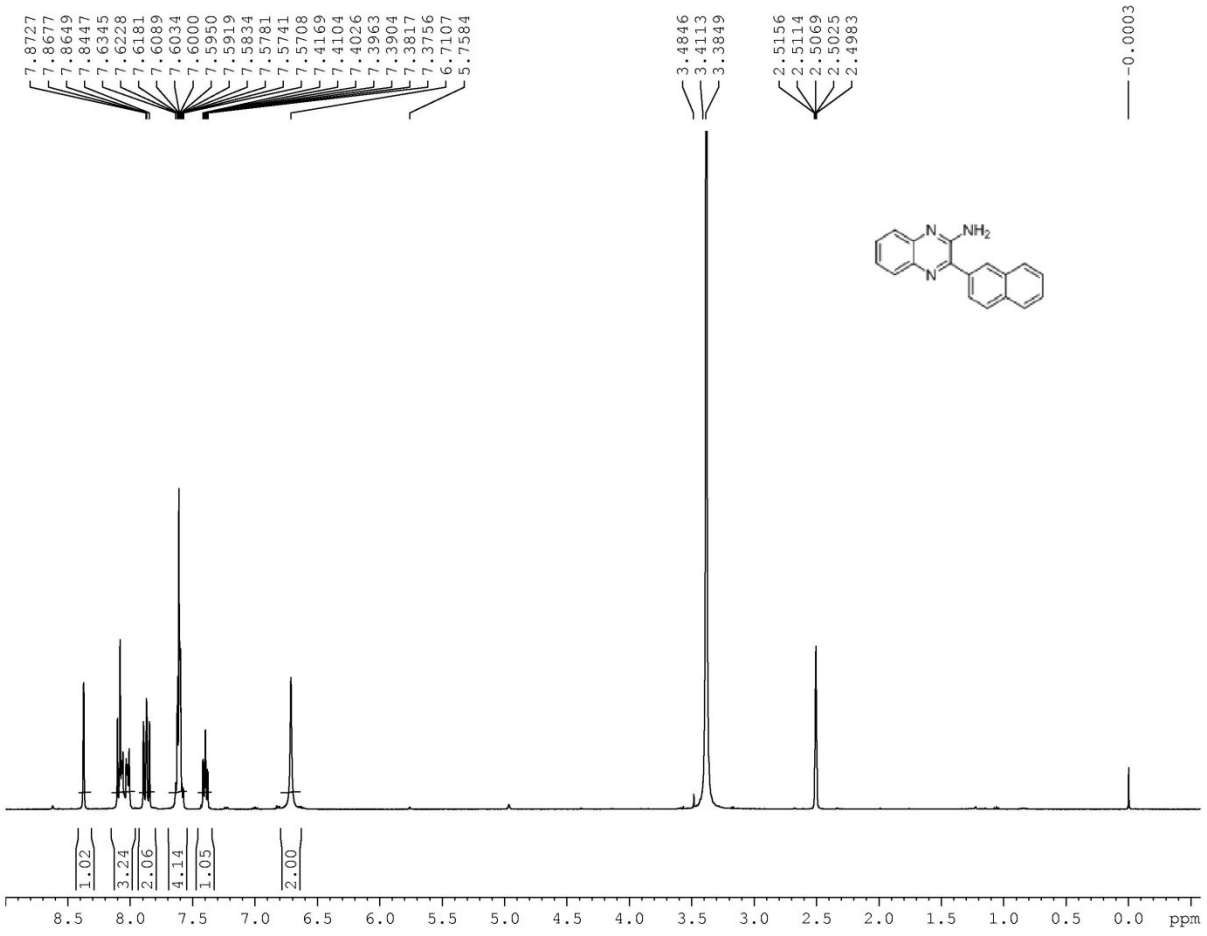
4h: ^{1}H NMR


7.7822
7.7592
7.7374
7.5741
7.5604
7.5545
7.5490
7.5280
7.3832
7.3758
7.3698
7.3626
7.3559
7.3489
7.3420
7.1141
7.0922
6.5431
4.3853
4.0249
3.8469
3.6637
3.4806
3.3806
3.1793
3.1661
2.6768
2.5113
2.5072
2.5033
2.3332
2.2439
0.0005
NH2
OMe
1.00
2.06
2.02
1.02
1.99
1.95
3.23
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

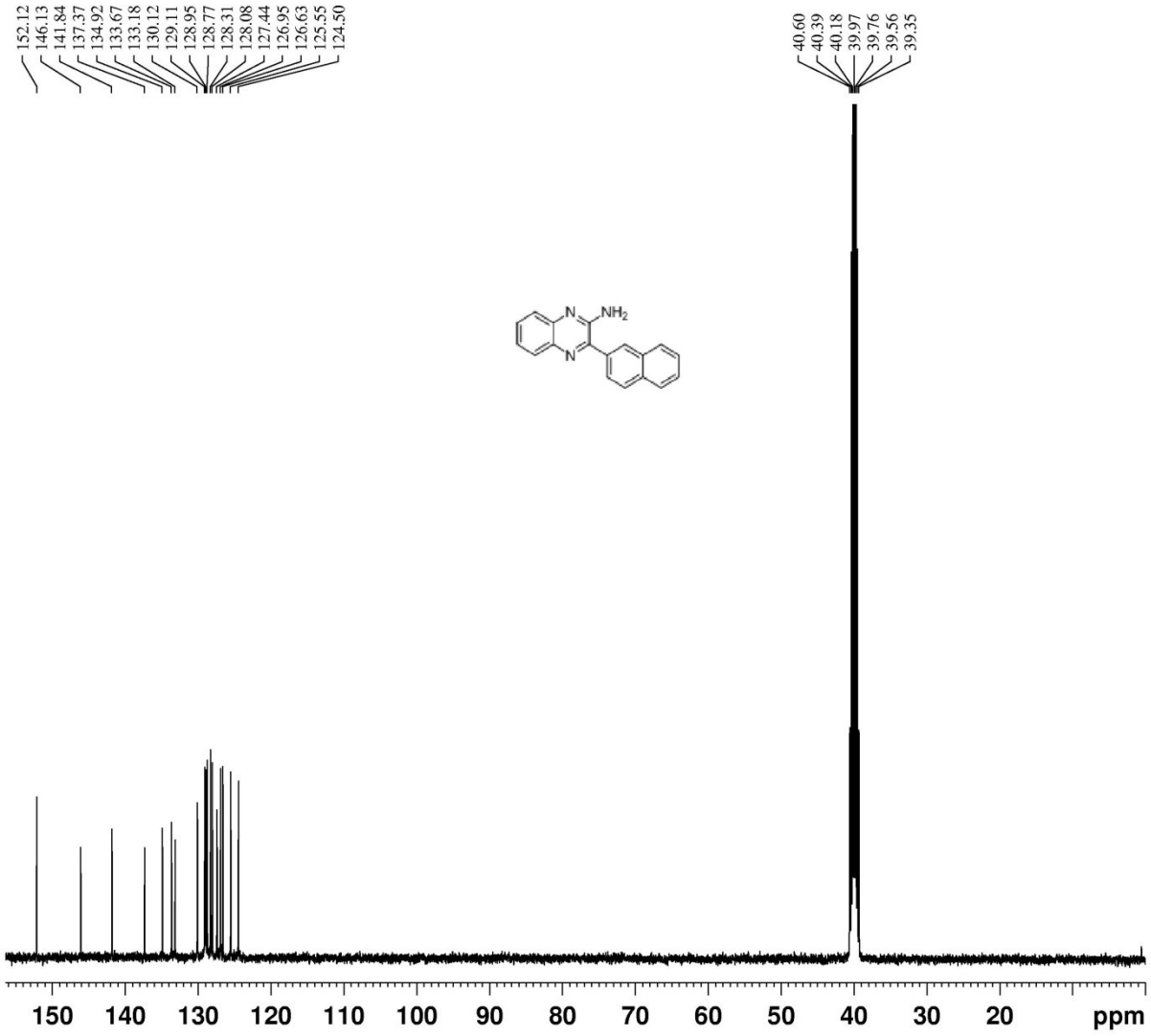
4h: ^{13}C NMR


160.78
150.68
145.65
140.97
138.21
129.87
129.73
129.33
128.91
125.59
125.21
118.81
116.21
114.63
77.36
77.25
77.04
76.72
55.47
N
NH2
N
O
160
140
120
100
80
60
40
20
ppm

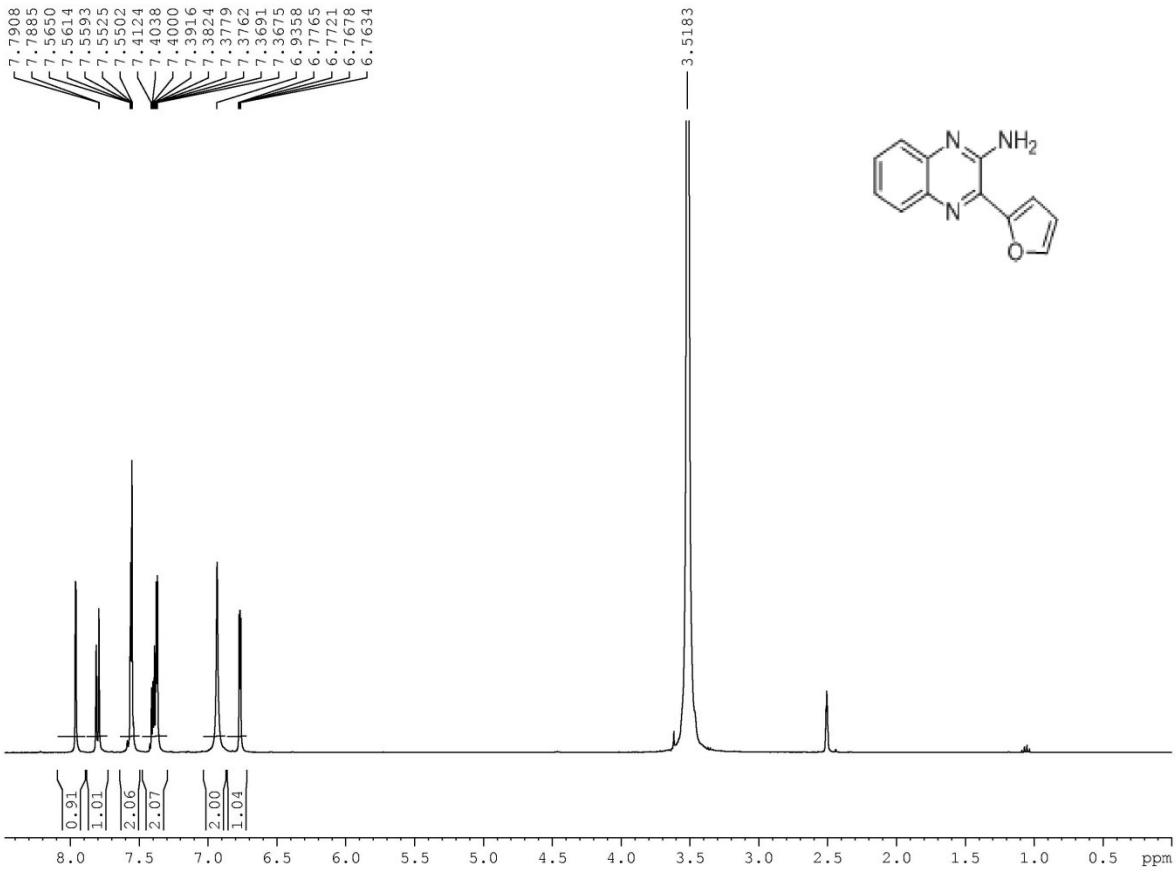
4i: ^{1}H NMR


7.8727
7.8677
7.8649
7.8447
7.6345
7.6228
7.6181
7.6089
7.6034
7.6000
7.5950
7.5919
7.5834
7.5781
7.5741
7.5708
7.4169
7.4104
7.4026
7.3963
7.3904
7.3817
7.3756
6.7107
5.7584
3.4846
3.4113
3.3849
2.5156
2.5114
2.5069
2.5025
2.4983
-0.0003
NH2
N
N
1.02
3.24
2.06
4.14
1.05
2.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

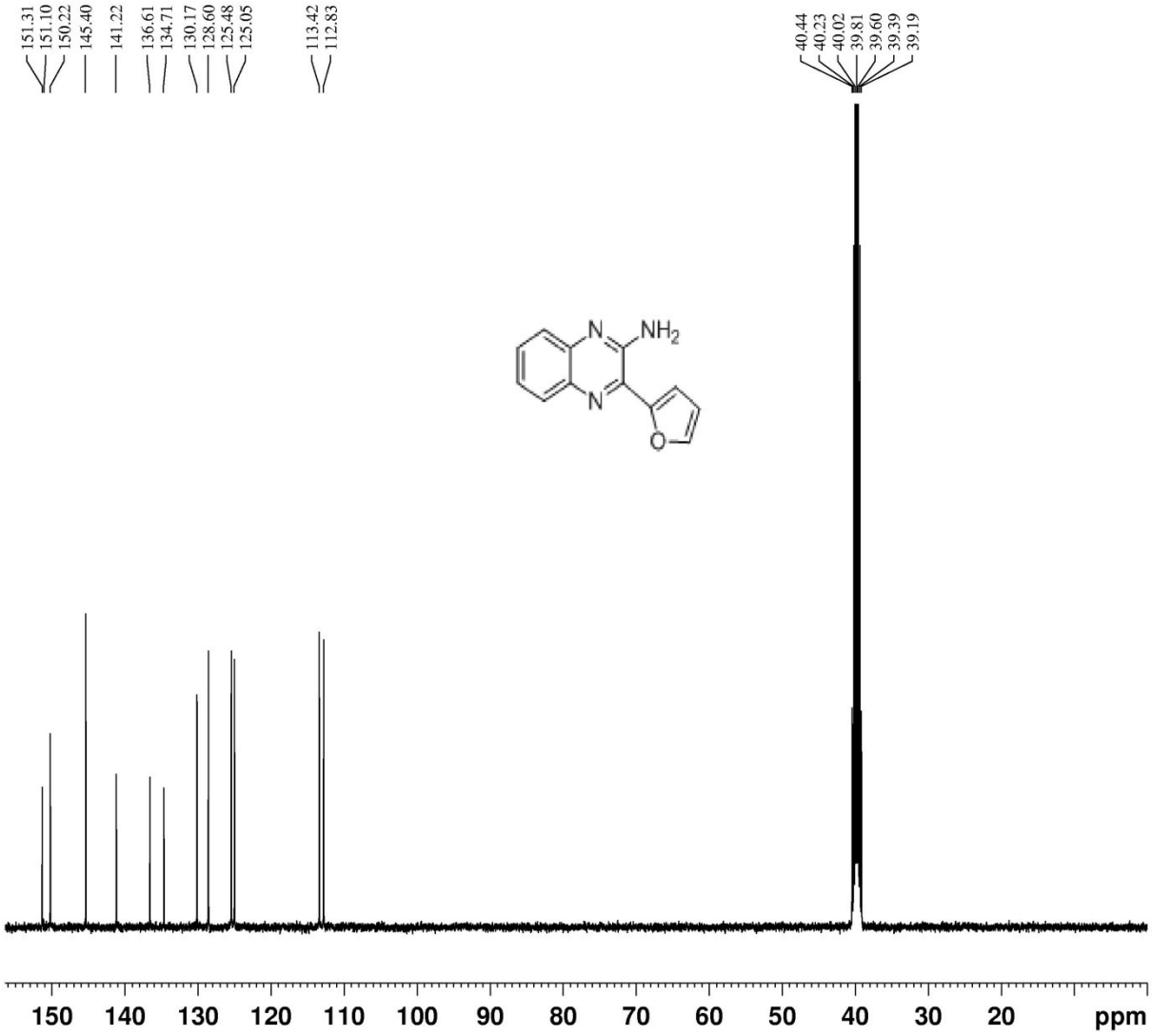
4i: ^{13}C NMR


152.12
146.13
141.84
137.37
134.92
133.67
133.18
130.12
129.11
128.95
128.77
128.31
128.08
127.44
126.95
126.63
125.55
124.50
40.60
40.39
40.18
39.97
39.76
39.56
39.35
N
NH2
N
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

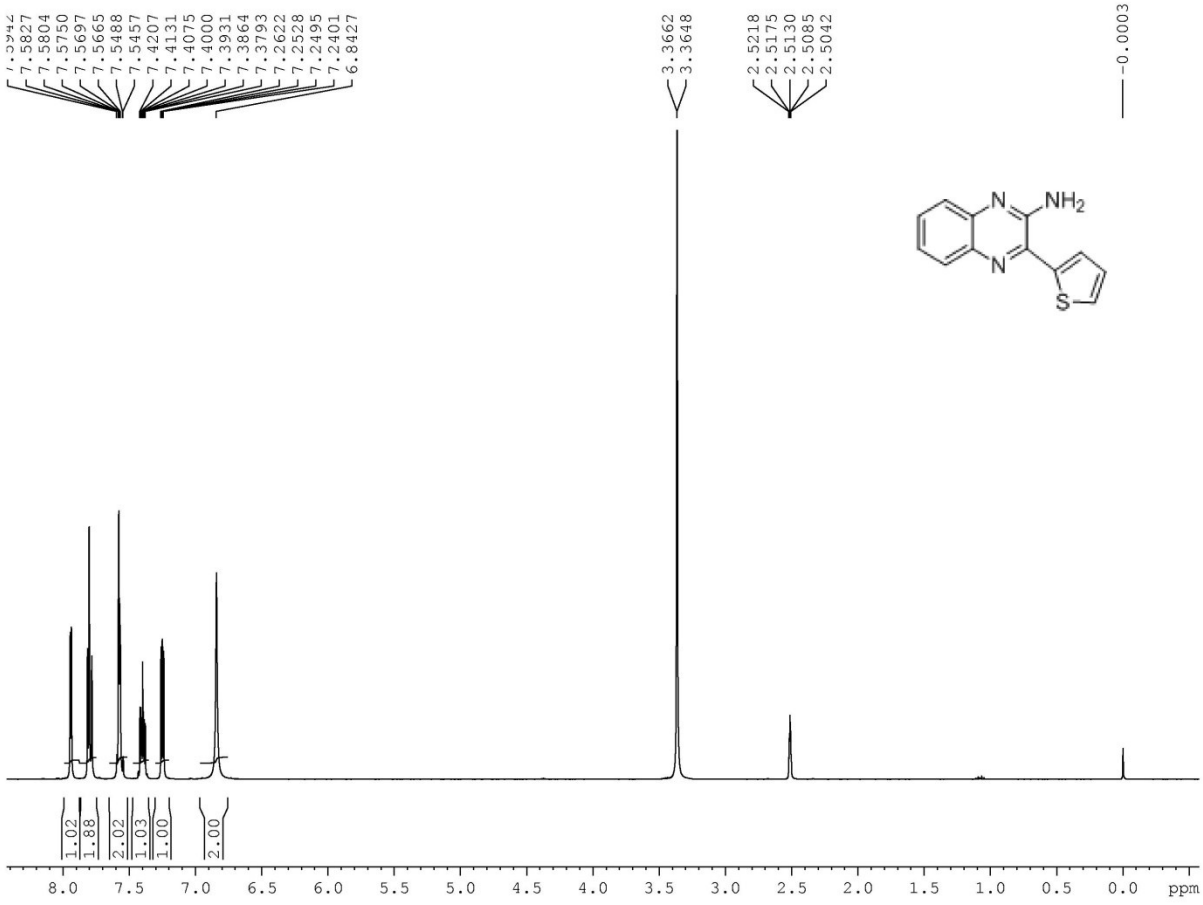
4j: ^{1}H NMR


7.7908
7.7885
7.5650
7.5614
7.5593
7.5525
7.5502
7.4124
7.4038
7.4000
7.3916
7.3824
7.3779
7.3762
7.3691
7.3675
6.9358
6.7765
6.7721
6.7678
6.7634
3.5183
NH2
N
N
O
0.91
1.01
2.06
2.07
2.00
1.04
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

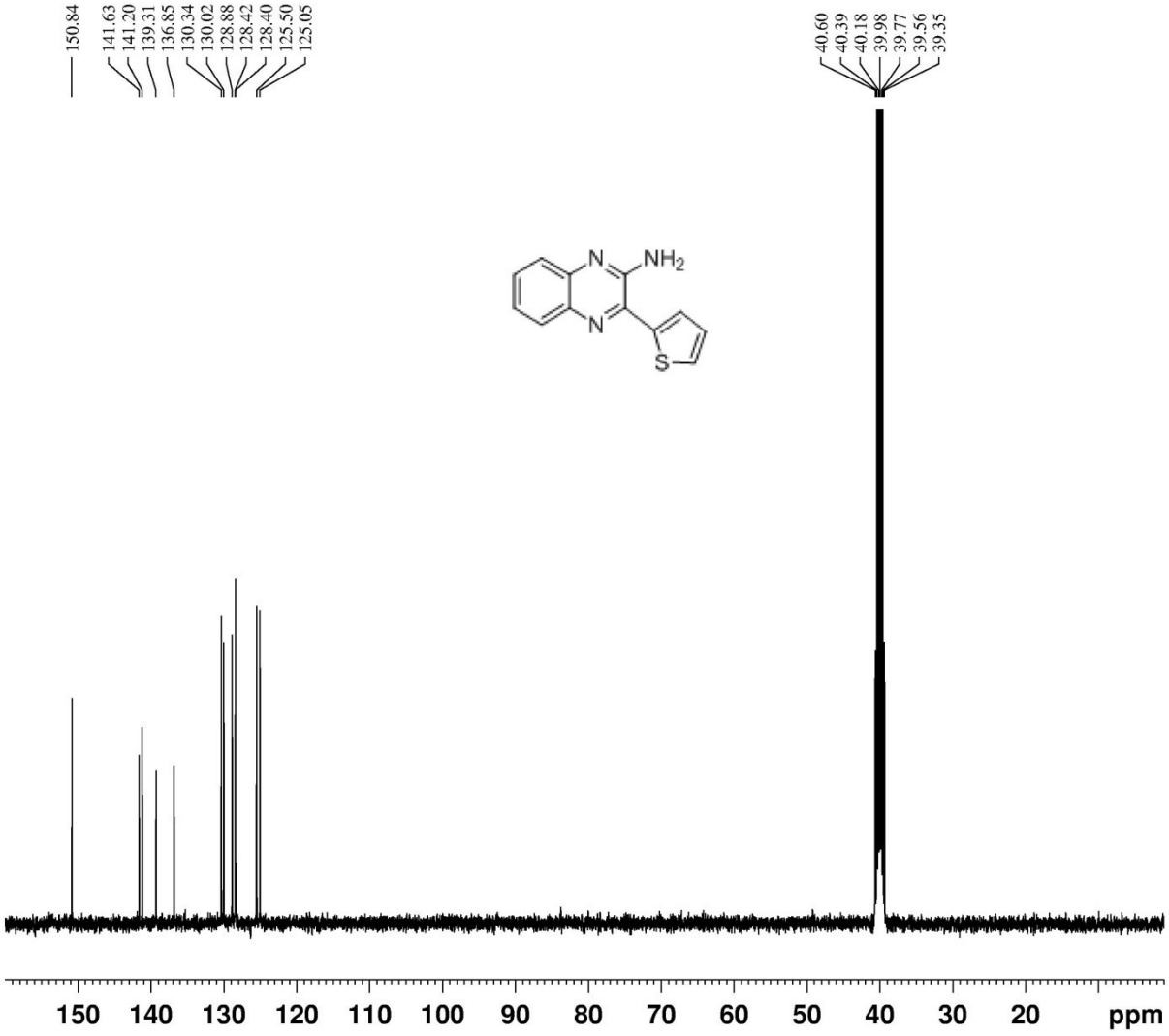
4j: ^{13}C NMR


151.31
151.10
150.22
145.40
141.22
136.61
134.71
130.17
128.60
125.48
125.05
113.42
112.83
40.44
40.23
40.02
39.81
39.60
39.39
39.19
N
NH2
N
O
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

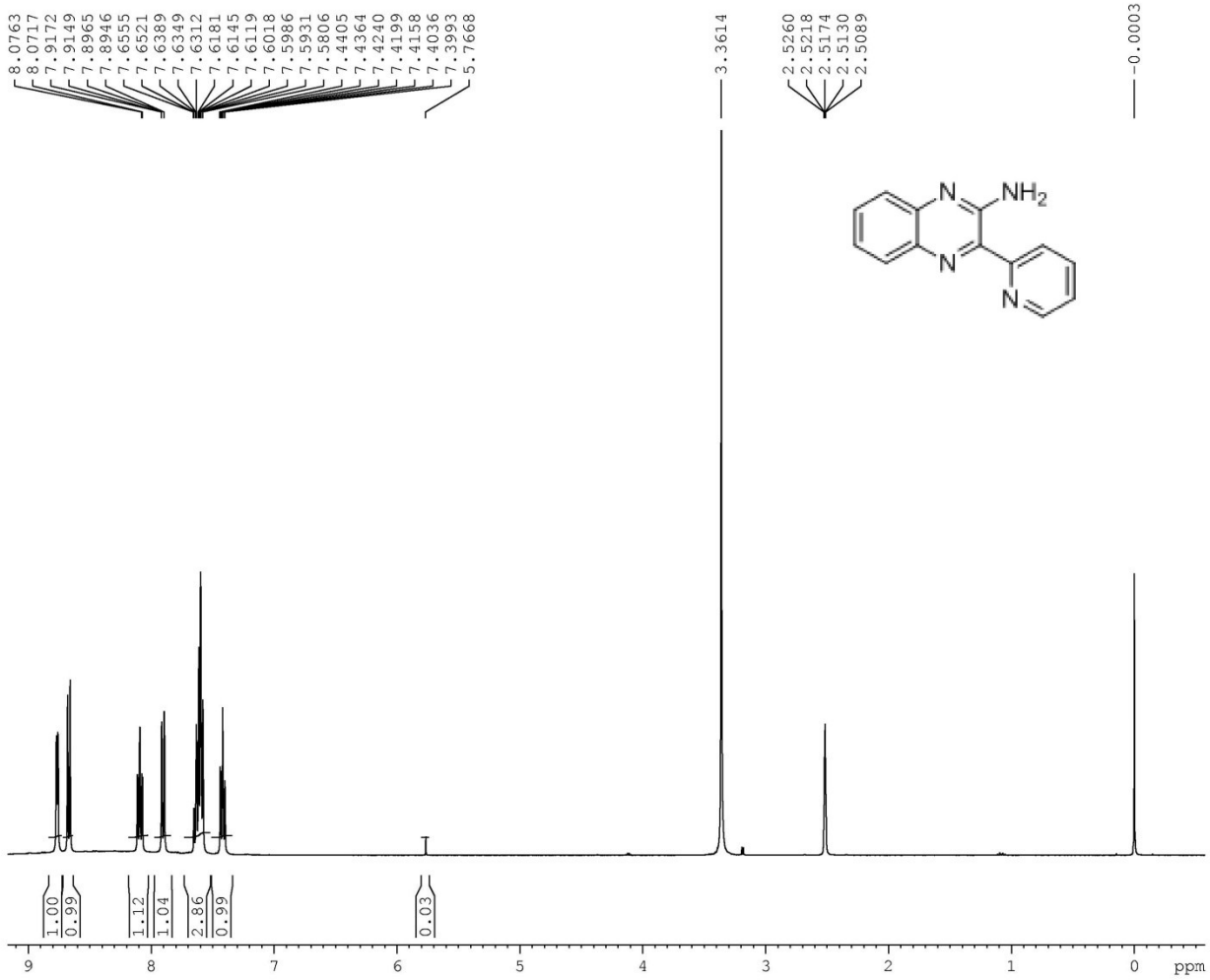
4k: ^{1}H NMR


7.5827
7.5804
7.5750
7.5697
7.5665
7.5488
7.5457
7.4207
7.4131
7.4075
7.4000
7.3931
7.3864
7.3793
7.2622
7.2528
7.2495
7.2401
6.8427
3.3662
3.3648
2.5218
2.5175
2.5130
2.5085
2.5042
-0.0003
NH2
N
N
S
1.02
1.88
2.02
1.03
1.00
2.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm

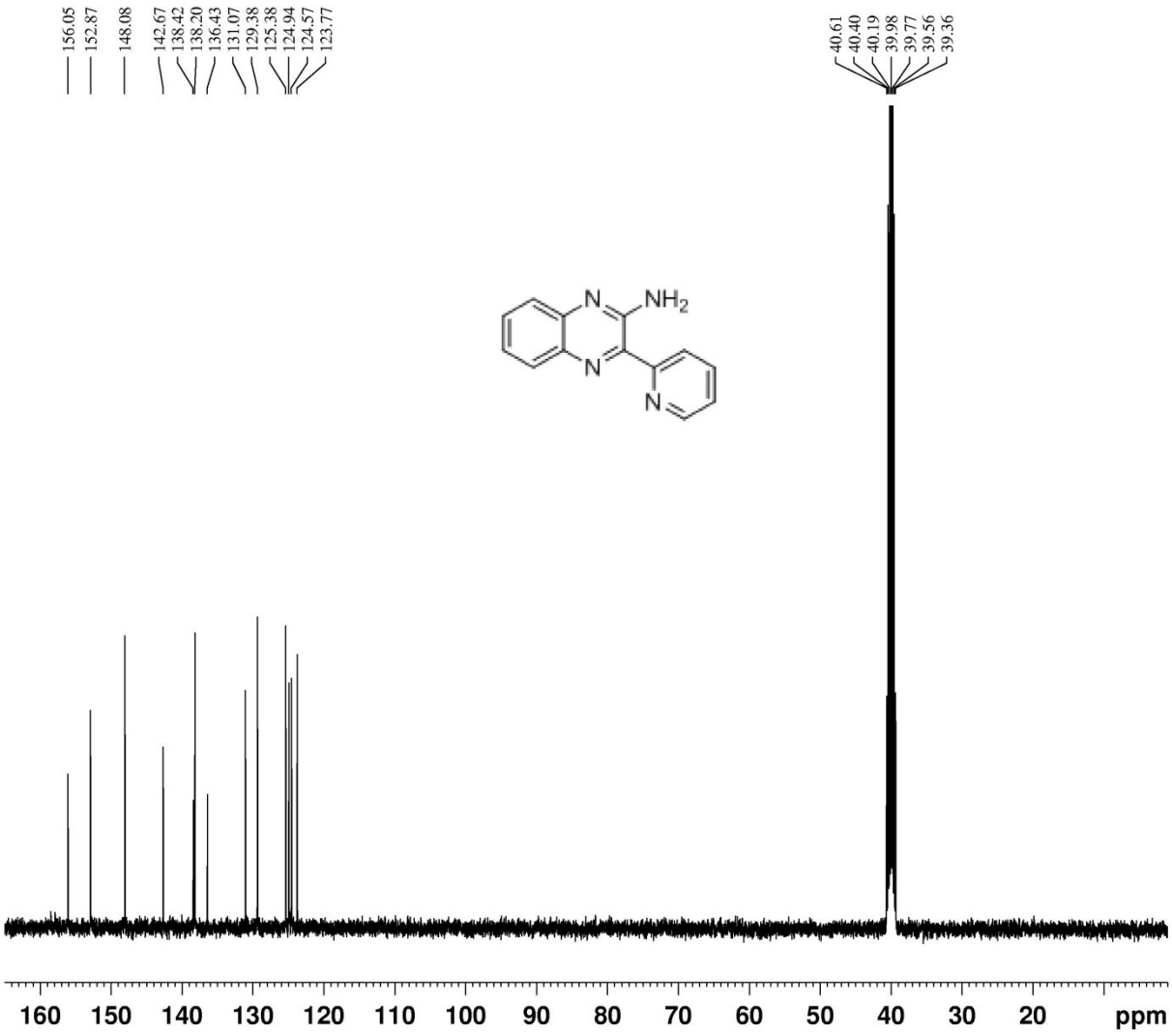
4k: ^{13}C NMR


150.84
141.63
141.20
139.31
136.85
130.34
130.02
128.88
128.42
128.40
125.50
125.05
40.60
40.39
40.18
39.98
39.77
39.56
39.35
N
NH2
N
S
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

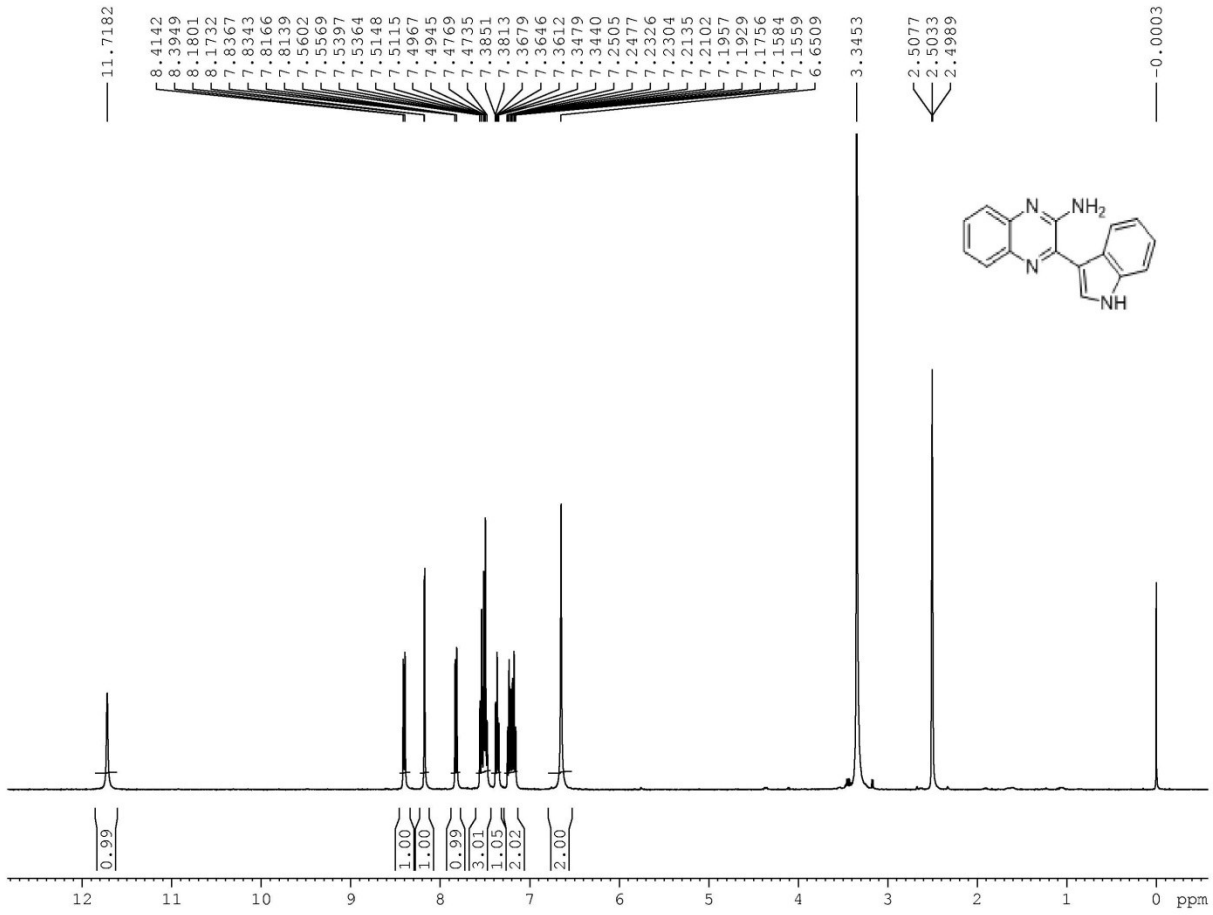
4l: ^{1}H NMR


8.0763
8.0717
7.9172
7.9149
7.8965
7.8946
7.6555
7.6521
7.6389
7.6349
7.6312
7.6181
7.6145
7.6119
7.6018
7.5986
7.5931
7.5806
7.4405
7.4364
7.4240
7.4199
7.4158
7.4036
7.3993
5.7668
3.3614
2.5260
2.5218
2.5174
2.5130
2.5089
-0.0003
N
NH2
N
N
1.00
0.99
1.12
1.04
2.86
0.99
0.03
9
8
7
6
5
4
3
2
1
0
ppm

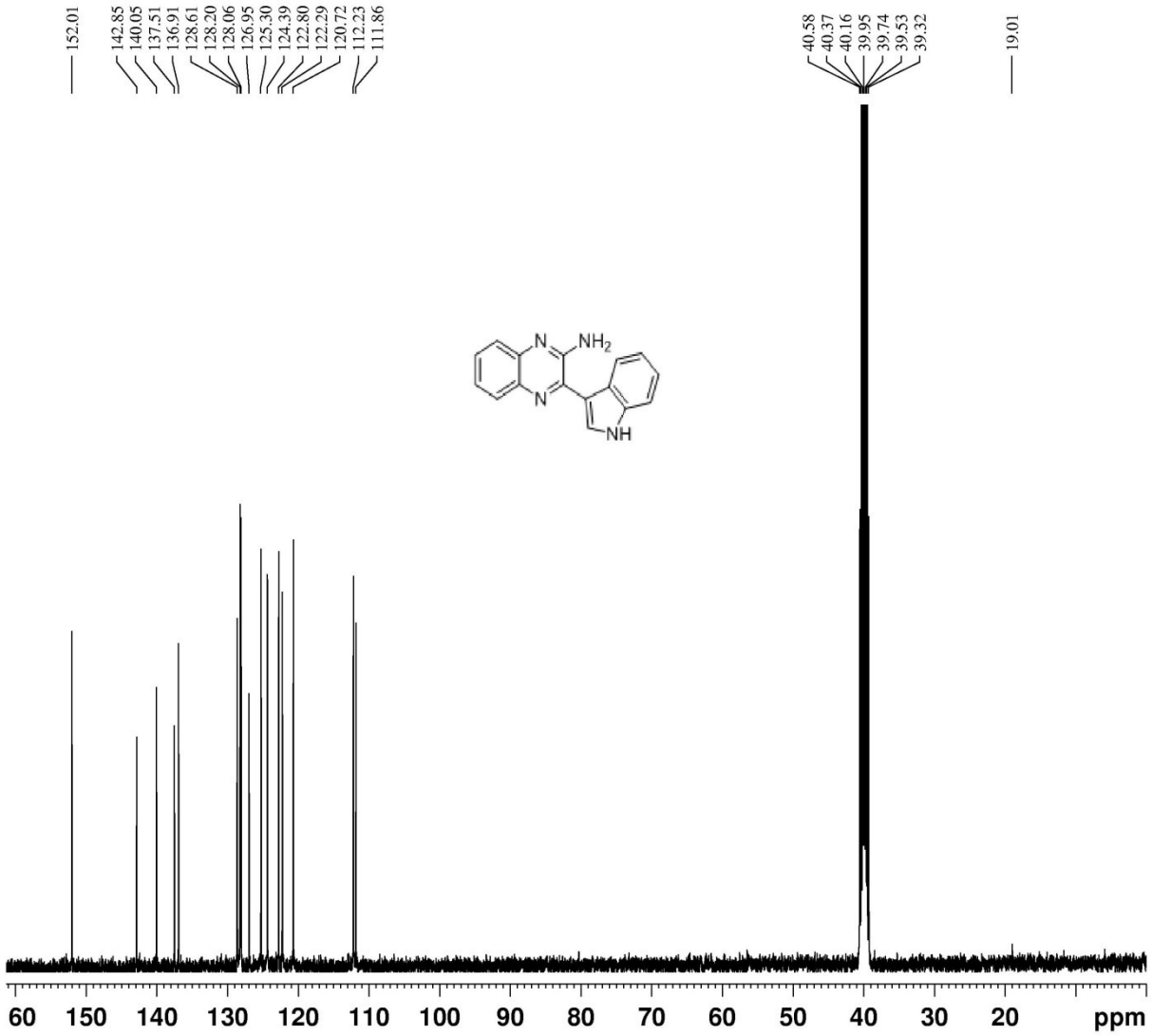
4l: ^{13}C NMR


156.05
152.87
148.08
142.67
138.42
138.20
136.43
131.07
129.38
125.38
124.94
124.57
123.77
40.61
40.40
40.19
39.98
39.77
39.56
39.36
N
NH2
N
N
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

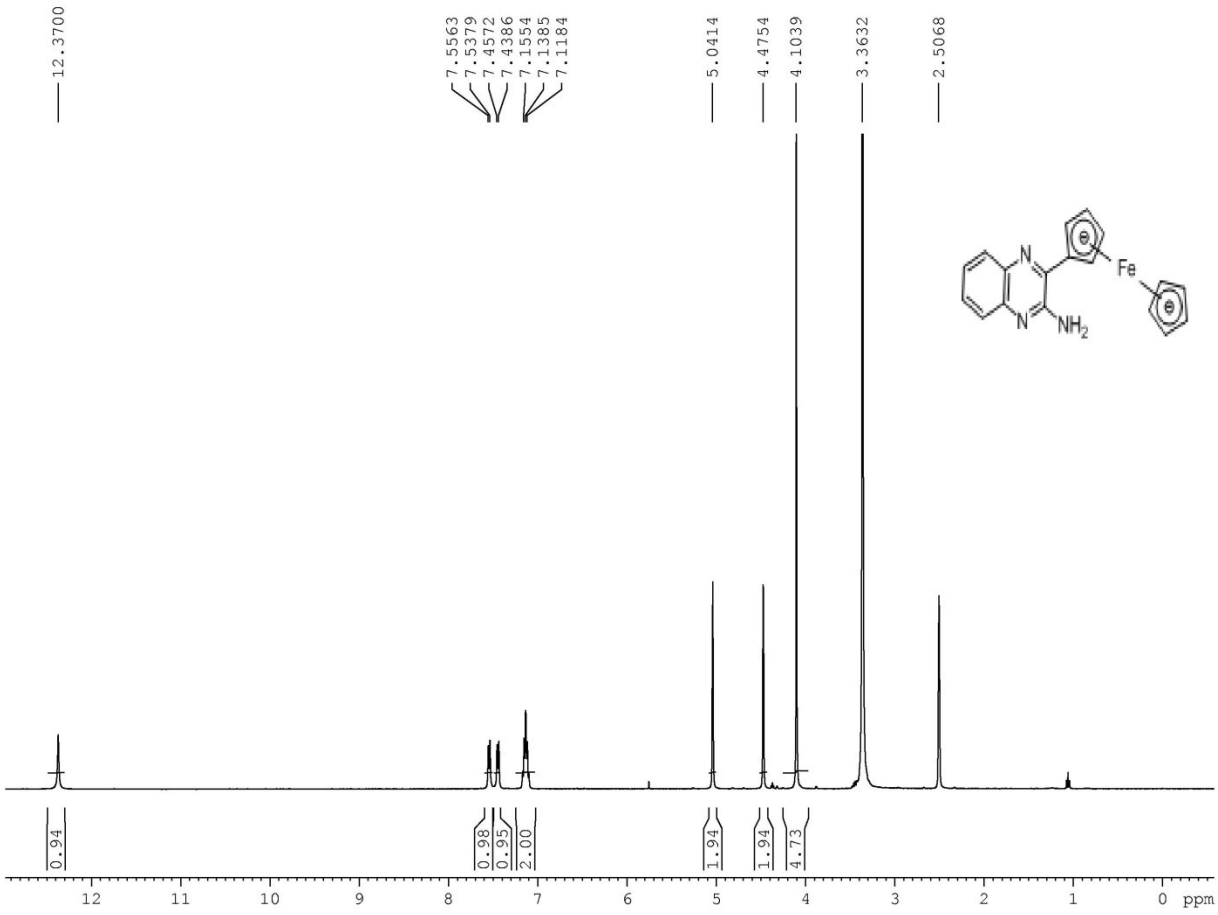
4m: ^{1}H NMR


11.7182
8.4142
8.3949
8.1801
8.1732
7.8367
7.8343
7.8166
7.8139
7.5602
7.5569
7.5397
7.5364
7.5148
7.5115
7.4967
7.4945
7.4769
7.4735
7.3851
7.3813
7.3679
7.3646
7.3612
7.3479
7.3440
7.2505
7.2477
7.2326
7.2304
7.2135
7.2102
7.1957
7.1929
7.1756
7.1584
7.1559
6.6509
3.3453
2.5077
2.5033
2.4989
-0.0003
NH2
N
N
NH
0.99
1.00
1.00
0.99
3.01
1.05
2.02
2.00
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm

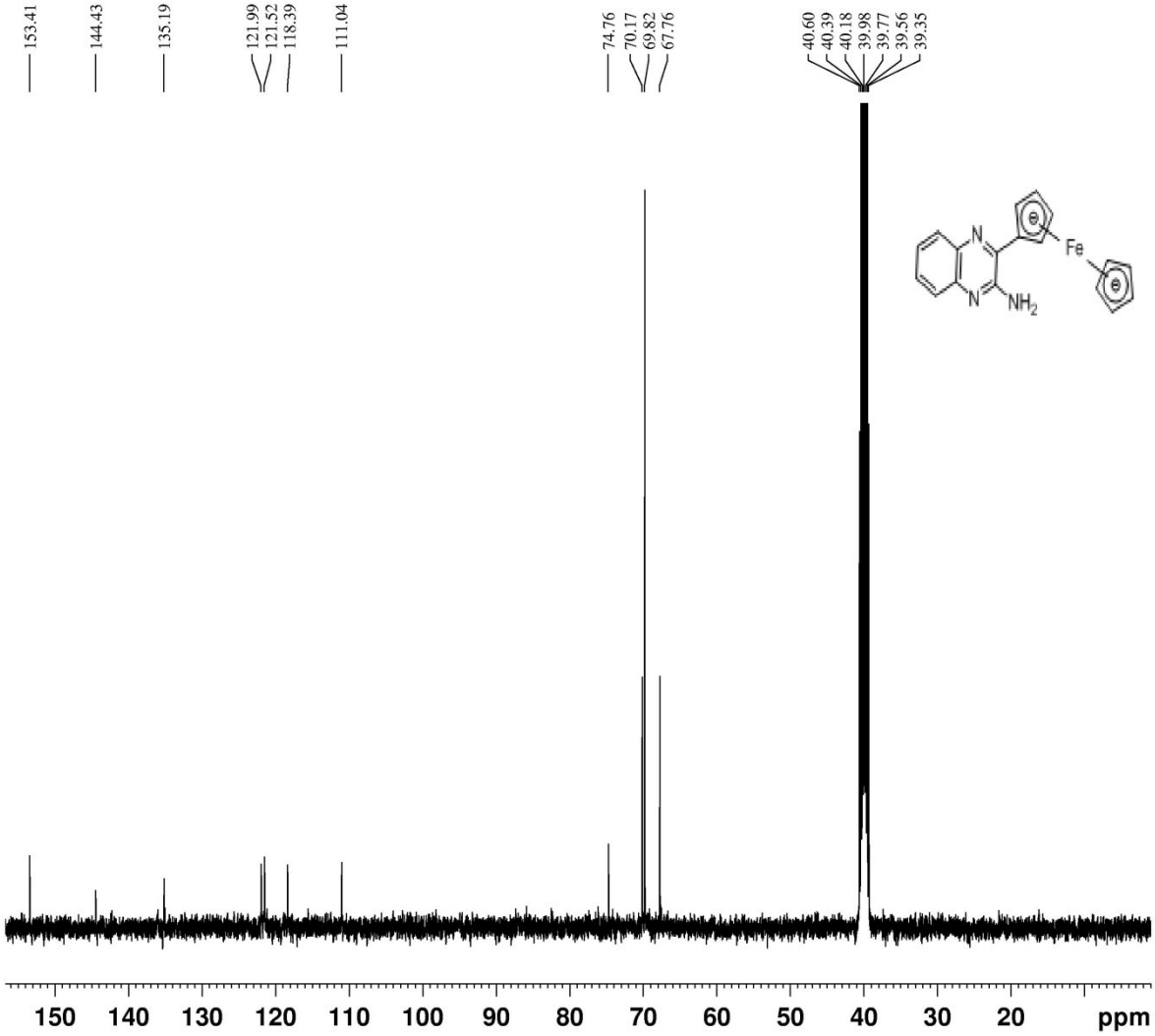
4m: ^{13}C NMR


152.01
142.85
140.05
137.51
136.91
128.61
128.20
128.06
126.95
125.30
124.39
122.80
122.29
120.72
112.23
111.86
40.58
40.37
40.16
39.95
39.74
39.53
39.32
19.01
NH2
N
N
NH
60
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

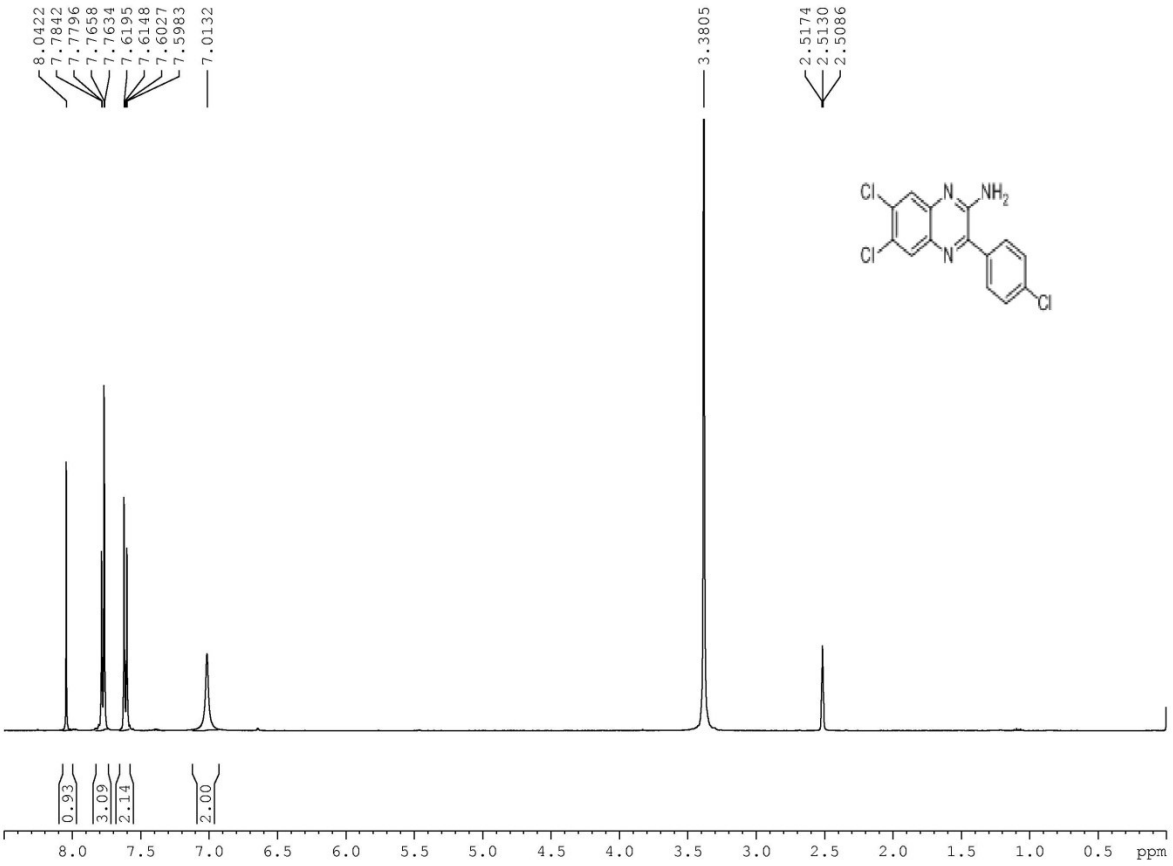
4n: ^{1}H NMR


12.3700
7.5563
7.5379
7.4572
7.4386
7.1554
7.1385
7.1184
5.0414
4.4754
4.1039
3.3632
2.5068
N
N
NH2
Fe
0.94
0.98
0.95
2.00
1.94
1.94
4.73
12
11
10
9
8
7
6
5
4
3
2
1
0 ppm

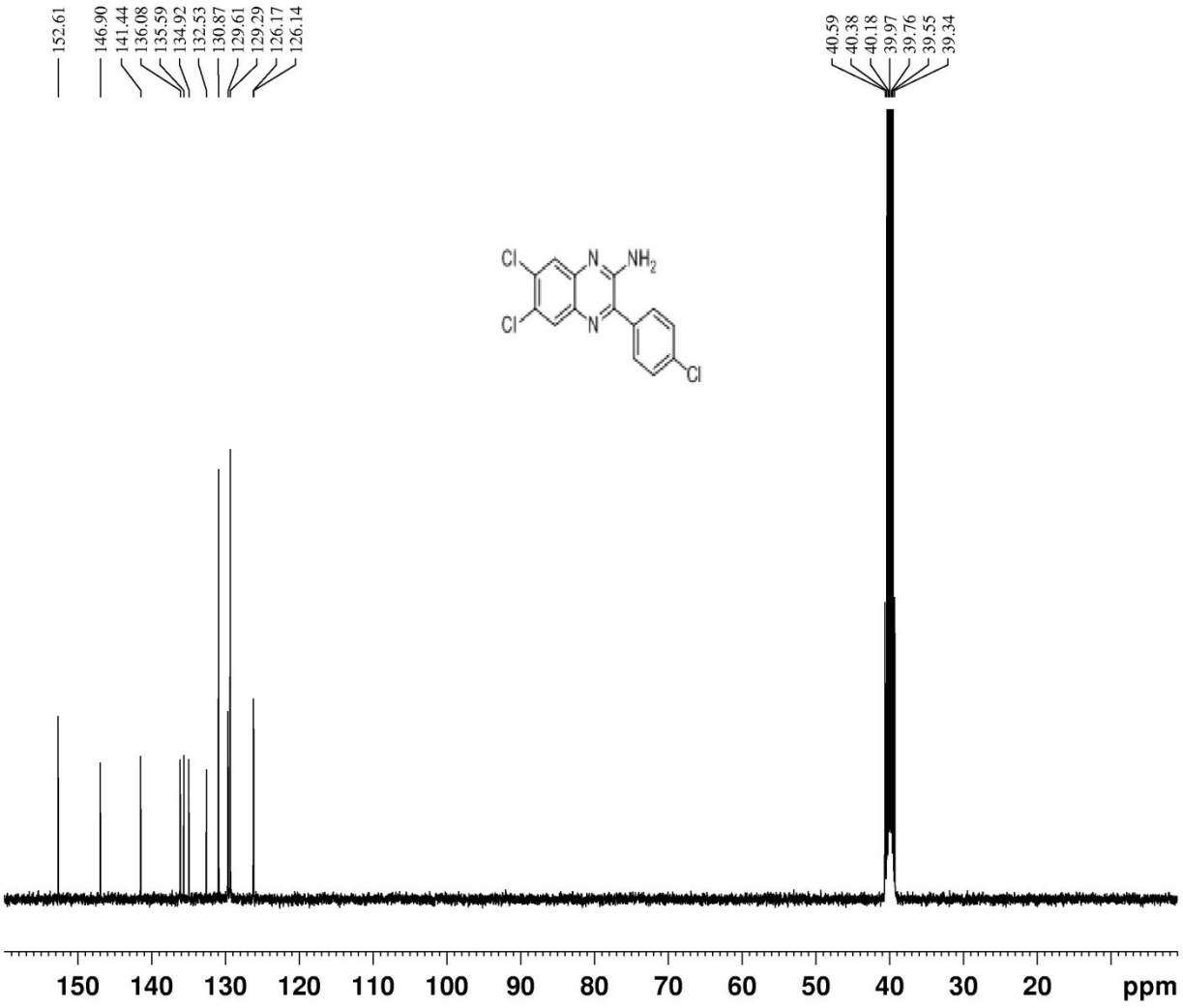
4n: ^{13}C NMR


153.41
144.43
135.19
121.99
121.52
118.39
111.04
74.76
70.17
69.82
67.76
40.60
40.39
40.18
39.98
39.77
39.56
39.35
N
N
NH2
Fe
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

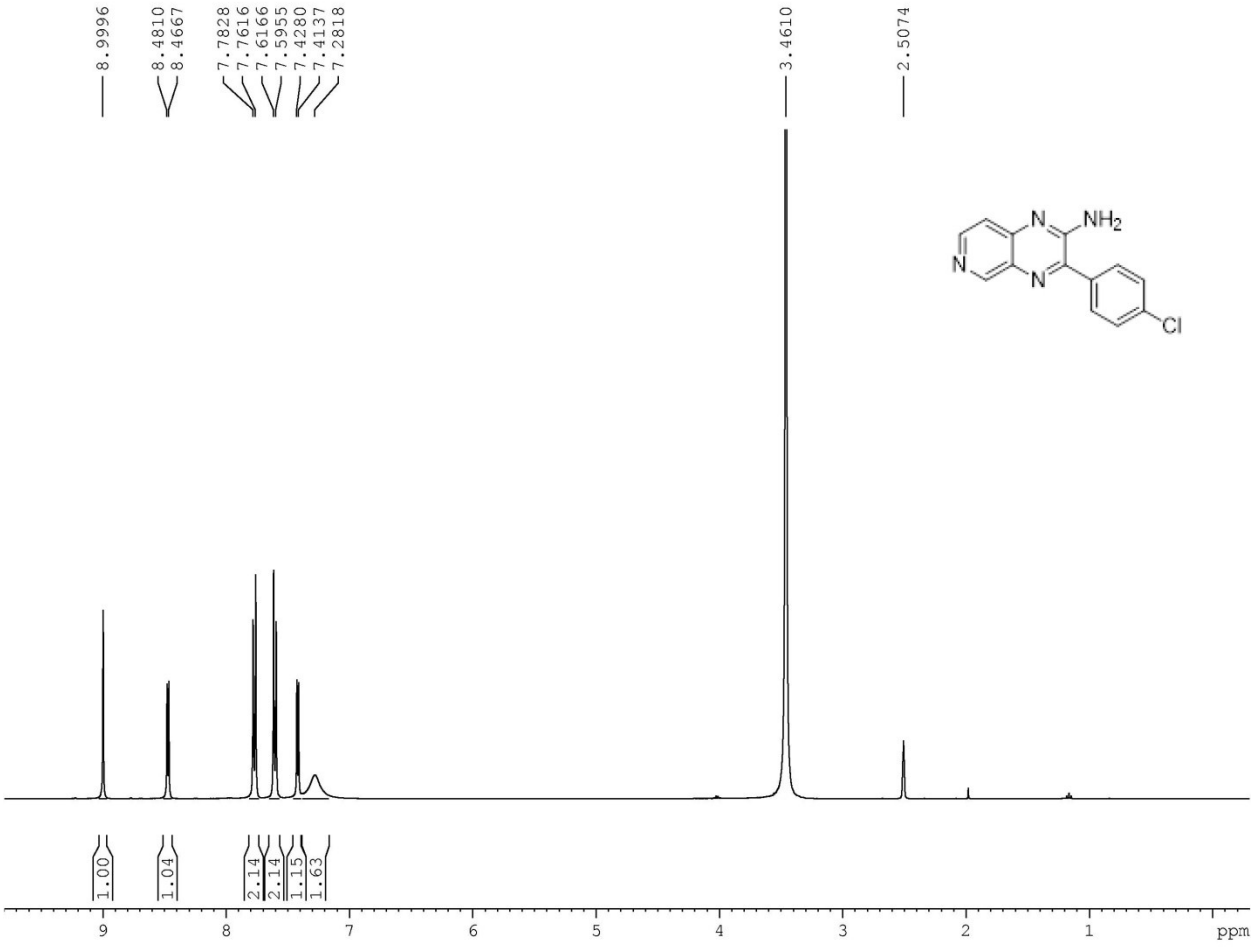
4o: ^{1}H NMR


8.0422
7.7842
7.7796
7.7658
7.7634
7.6195
7.6148
7.6027
7.5983
7.0132
3.3805
2.5174
2.5130
2.5086
Cl
Cl
N
NH2
N
Cl
0.93
3.09
2.14
2.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

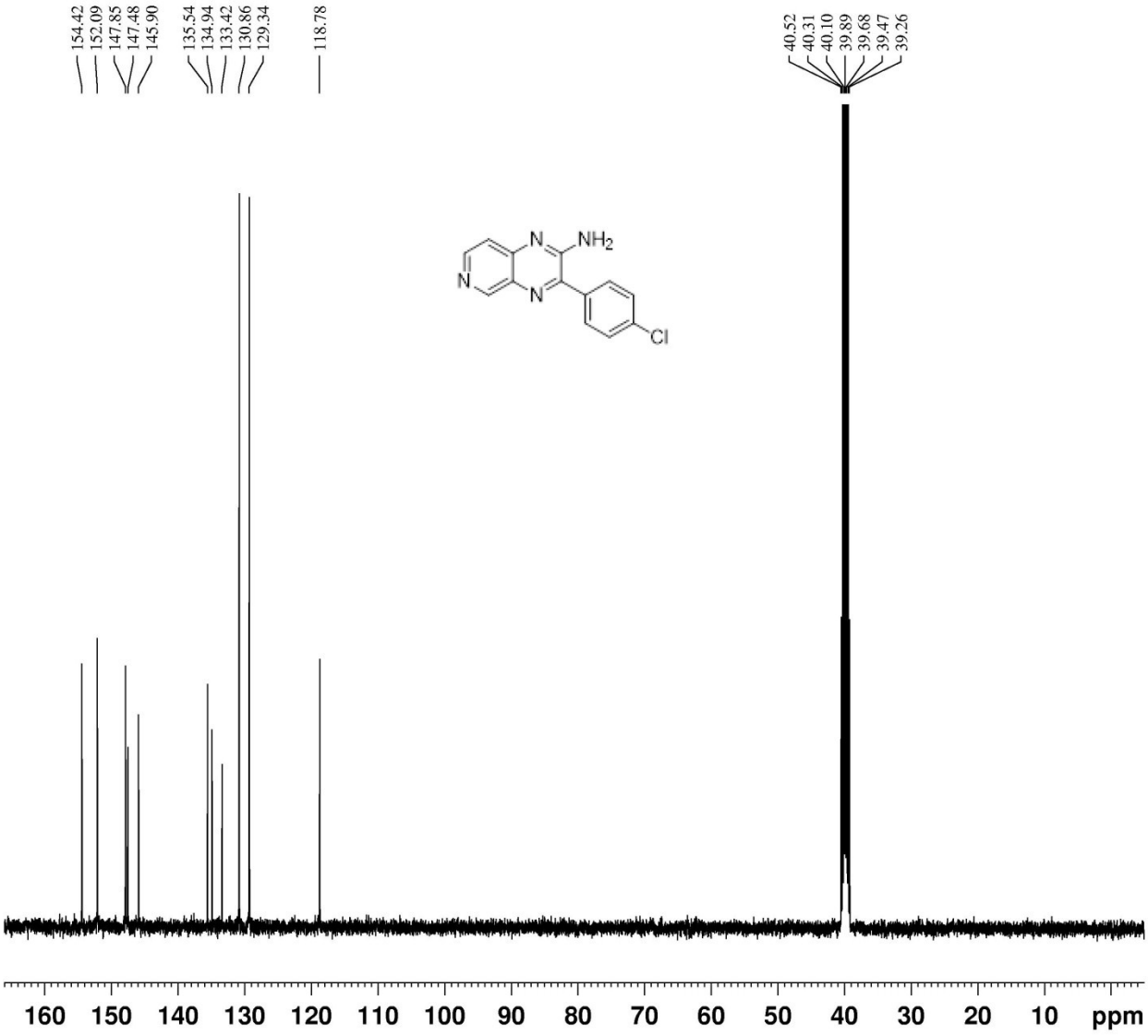
4o: ^{13}C NMR


152.61
146.90
141.44
136.08
135.59
134.92
132.53
130.87
129.61
129.29
126.17
126.14
40.59
40.38
40.18
39.97
39.76
39.55
39.34
Cl
Cl
N
NH2
N
Cl
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

4p: ^{1}H NMR


8.9996
8.4810
8.4667
7.7828
7.7616
7.6166
7.5955
7.4280
7.4137
7.2818
3.4610
2.5074
NH2
N
N
N
Cl
1.00
1.04
2.14
2.14
1.15
1.63
9
8
7
6
5
4
3
2
1
ppm

4p: ^{13}C NMR


154.42
152.09
147.85
147.48
145.90
135.54
134.94
133.42
130.86
129.34
118.78
40.52
40.31
40.10
39.89
39.68
39.47
39.26
NH2
N
N
N
Cl
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm