(±)-Camphor-10-sulfonic acid catalyzed direct one-pot three-component Mannich type reaction of alkyl (hetero)aryl ketones under solvent-free conditions: Application to the synthesis of aminochromans

Kshama Kundu and Sandip K. Nayak*

Bio-Organic Division, Bhabha Atomic Research Centre, Mumbai – 400085, India

Tel: +91-22-25592410; Fax: +91-22-25505151; e-mail: sknayak@barc.gov.in

Supporting Information

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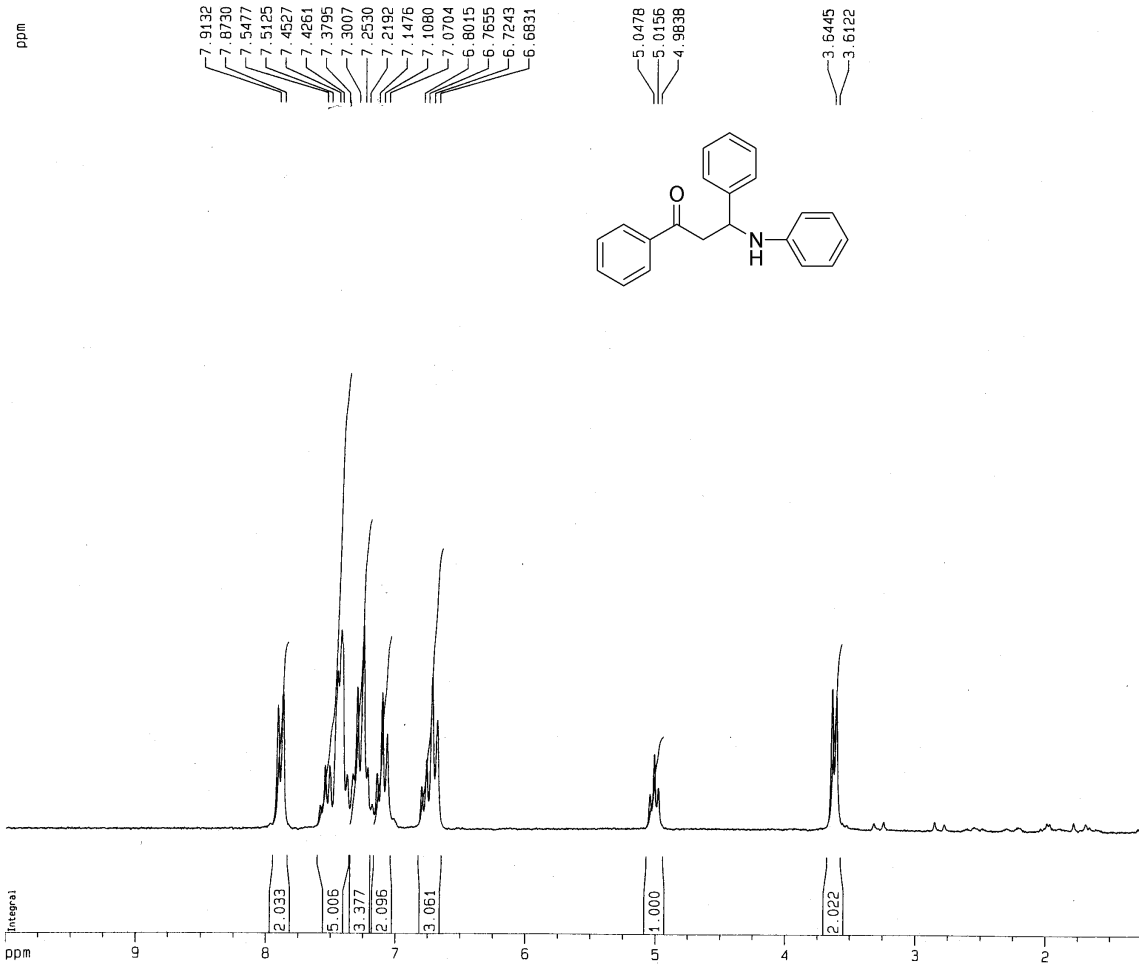
(I) ^{1}H and ^{13}C Spectra

(I) ^{1}H NMR and ^{13}C NMR Spectra

^{1}H NMR of 4a



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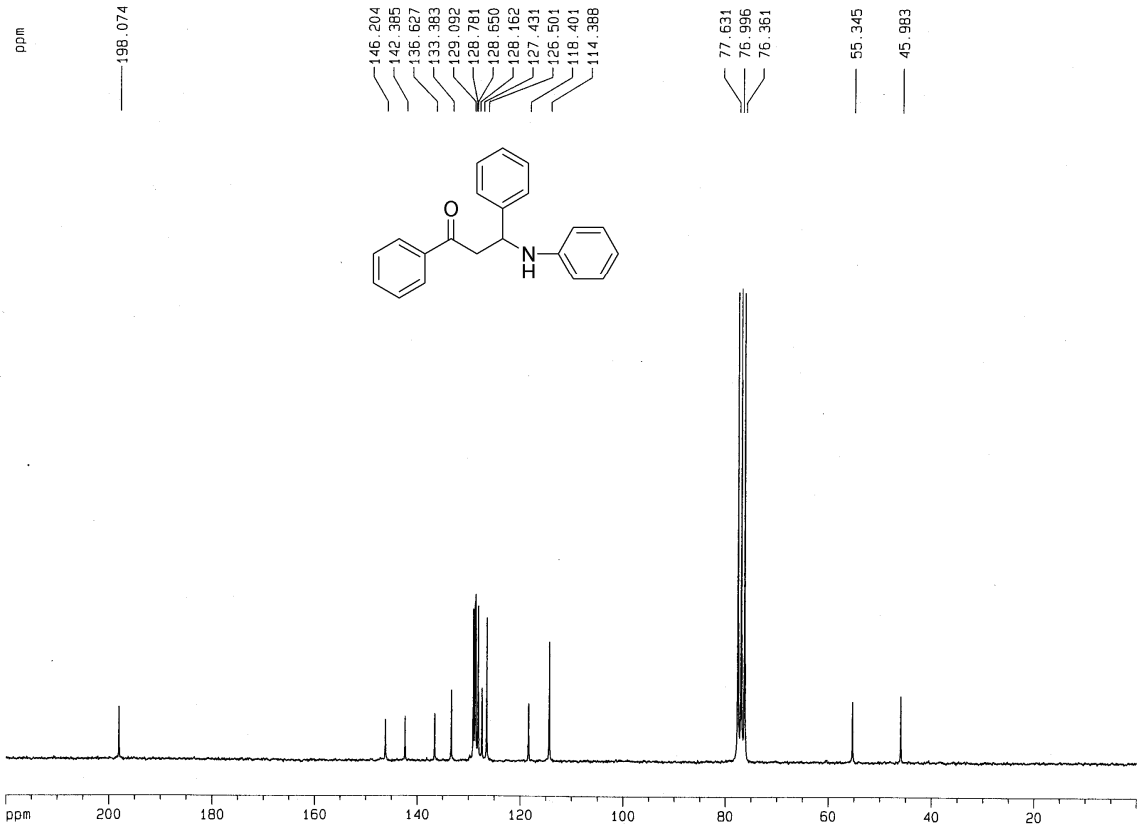
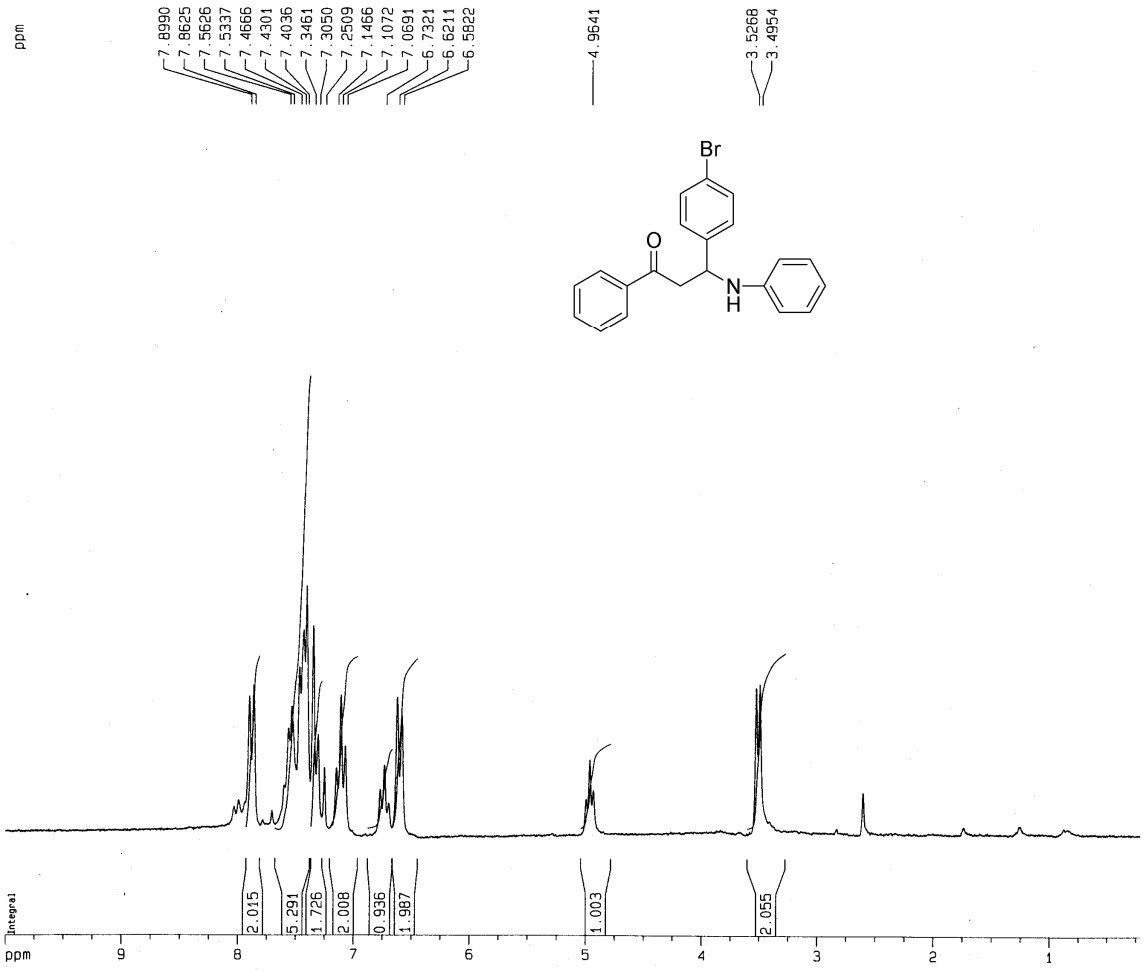


^{1}H NMR of 4b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7.1466
7.1072
7.0691
6.7321
6.6211
6.5822
4.9641
3.5268
3.4954
Br
O
N
H
Integral
2.015
5.291
1.726
2.008
0.936
1.987
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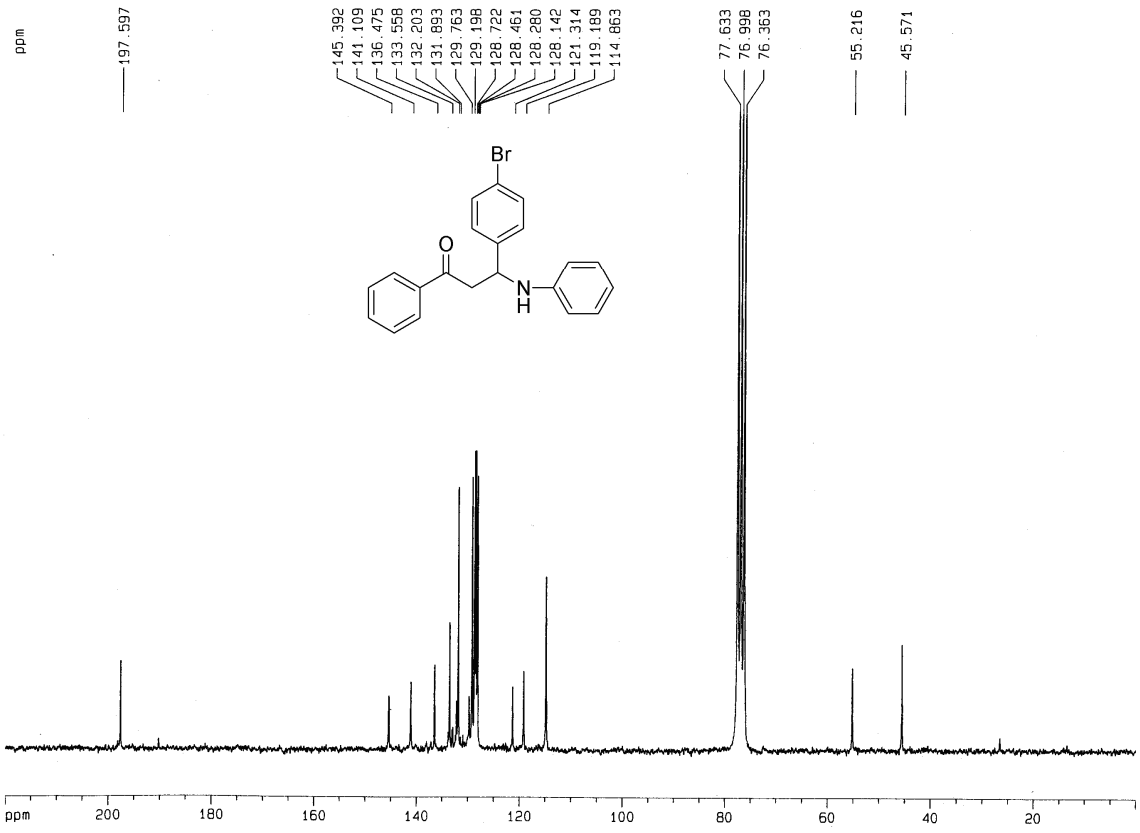
2.055
ppm
9
8
7
6
5
4
3
2
1

^{13}C NMR of 4b



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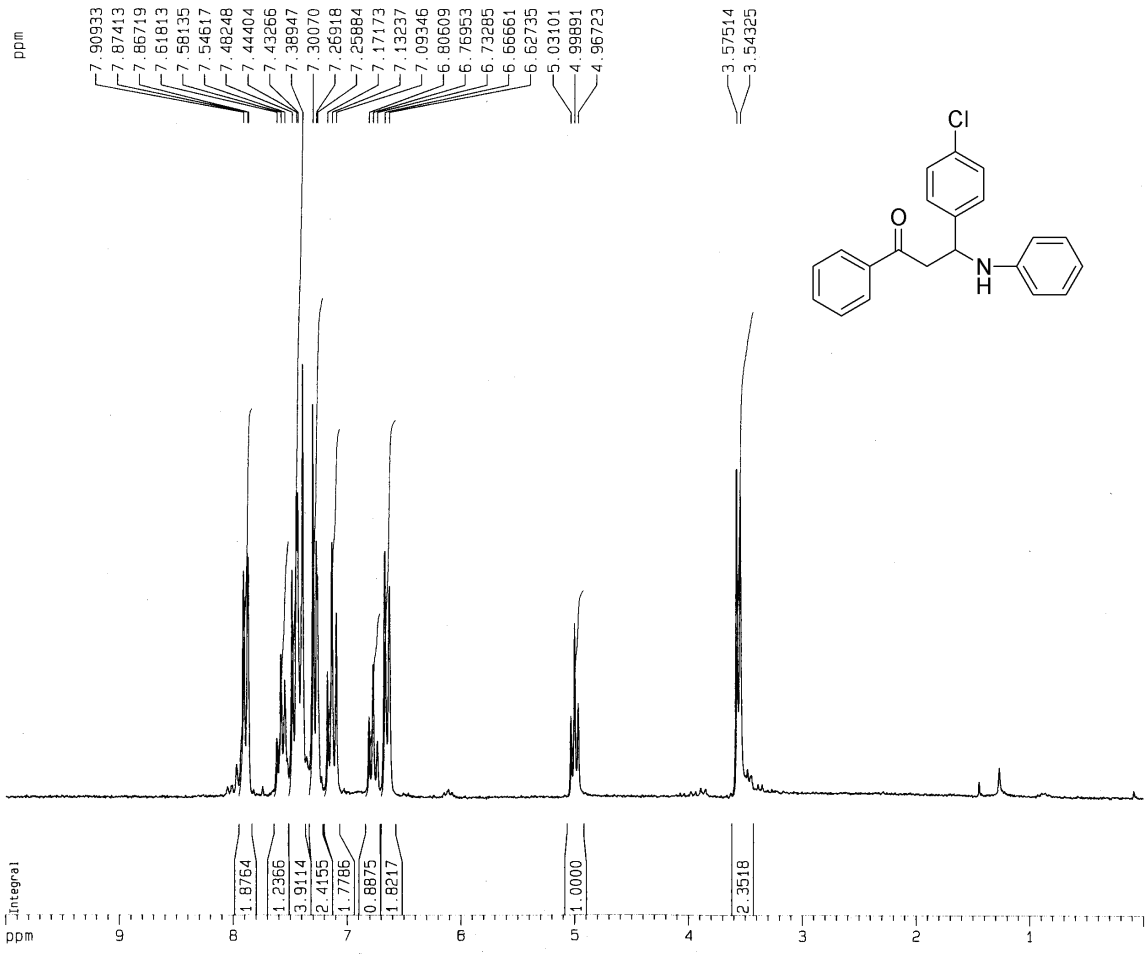
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7.30070
7.26918
7.25884
7.17173
7.13237
7.09346
6.80609
6.76953
6.73285
6.66661
6.62735
5.03101
4.99891
4.96723
3.57514
3.54325
Cl
O
N
H
Integral
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1.2366
3.9114
2.4155
1.7786
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8
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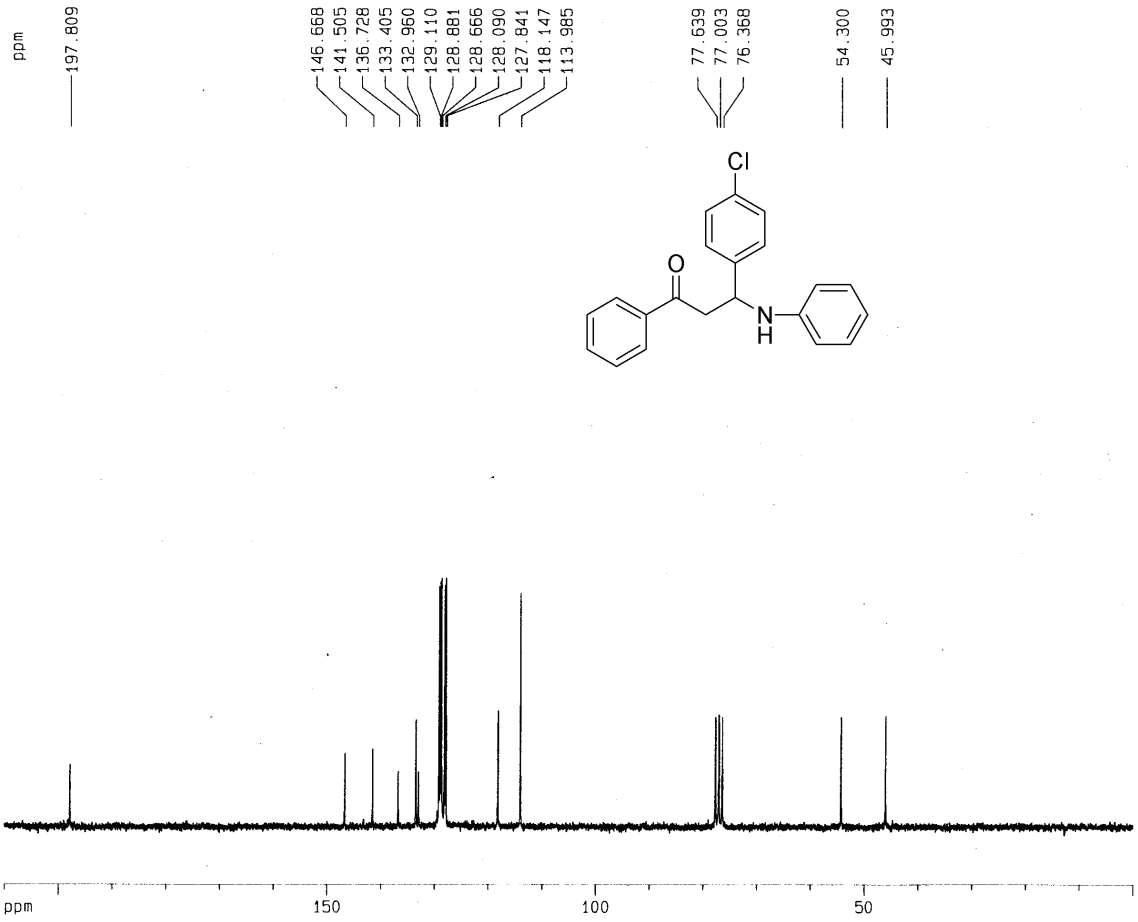
5
4
3
2
1

^{13}C NMR of 4c



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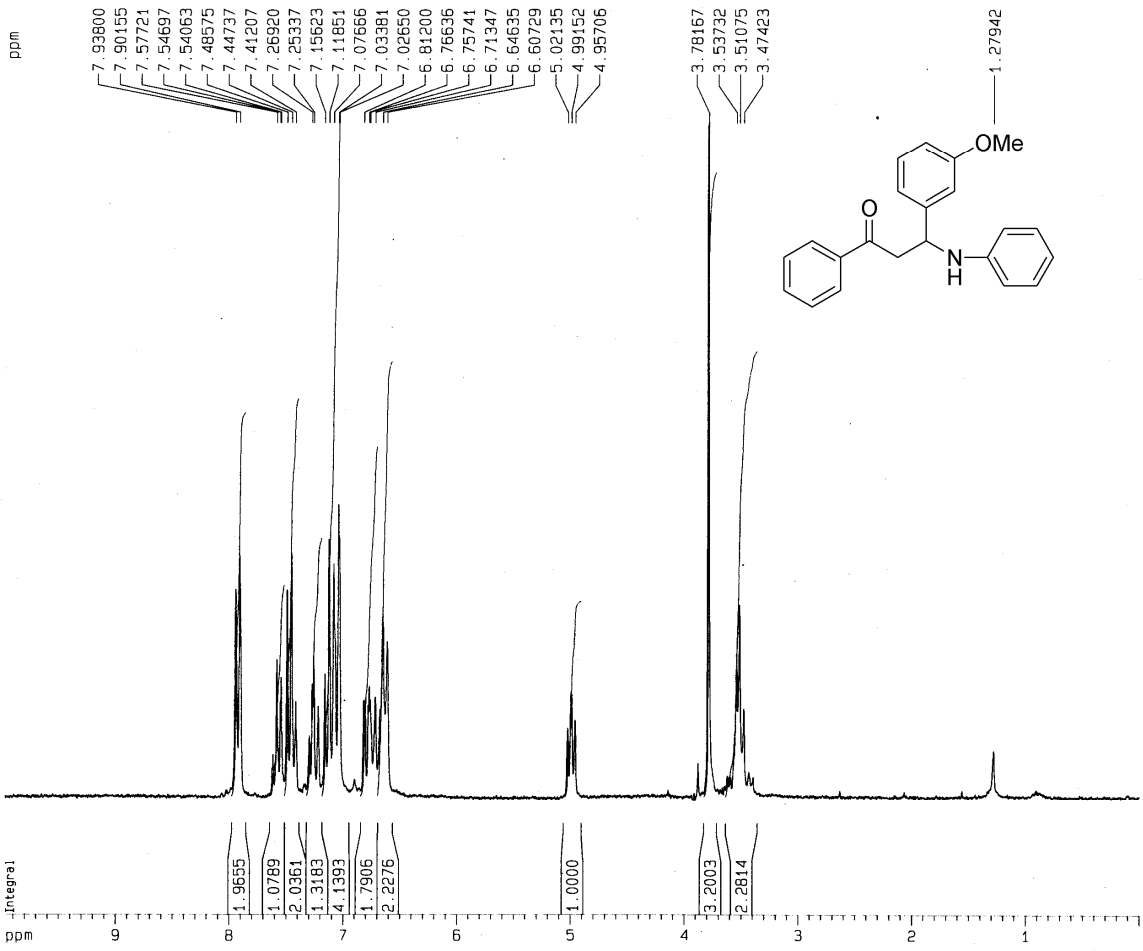
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7.15623
7.11851
7.07666
7.03381
7.02650
6.81200
6.76636
6.75741
6.71347
6.64635
6.60729
5.02135
4.99152
4.95706
3.78167
3.53732
3.51075
3.47423
1.27942
OMe
O
N
H
Integral
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1.0789
2.0361
1.3183
4.1393
1.7906
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5
4
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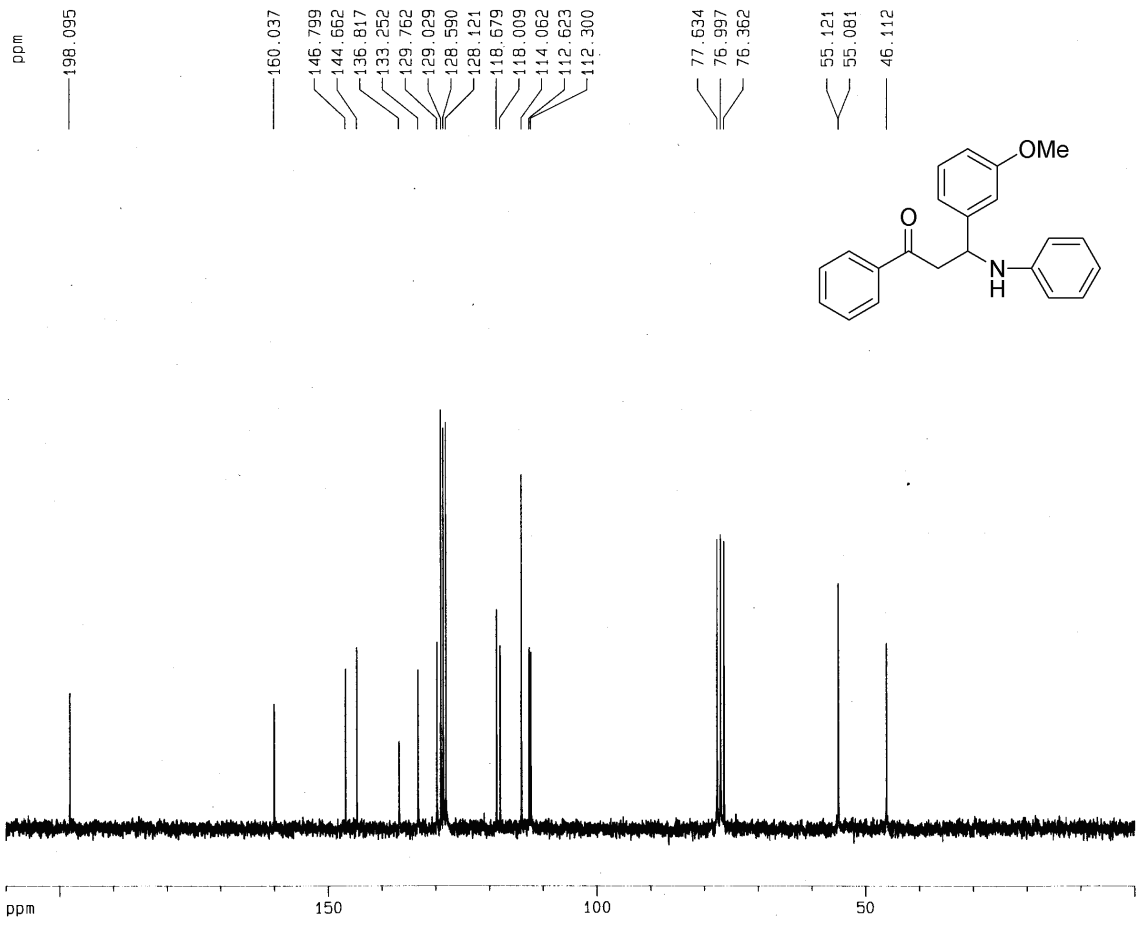
1

^{13}C NMR of 4d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114.062
112.623
112.300
77.634
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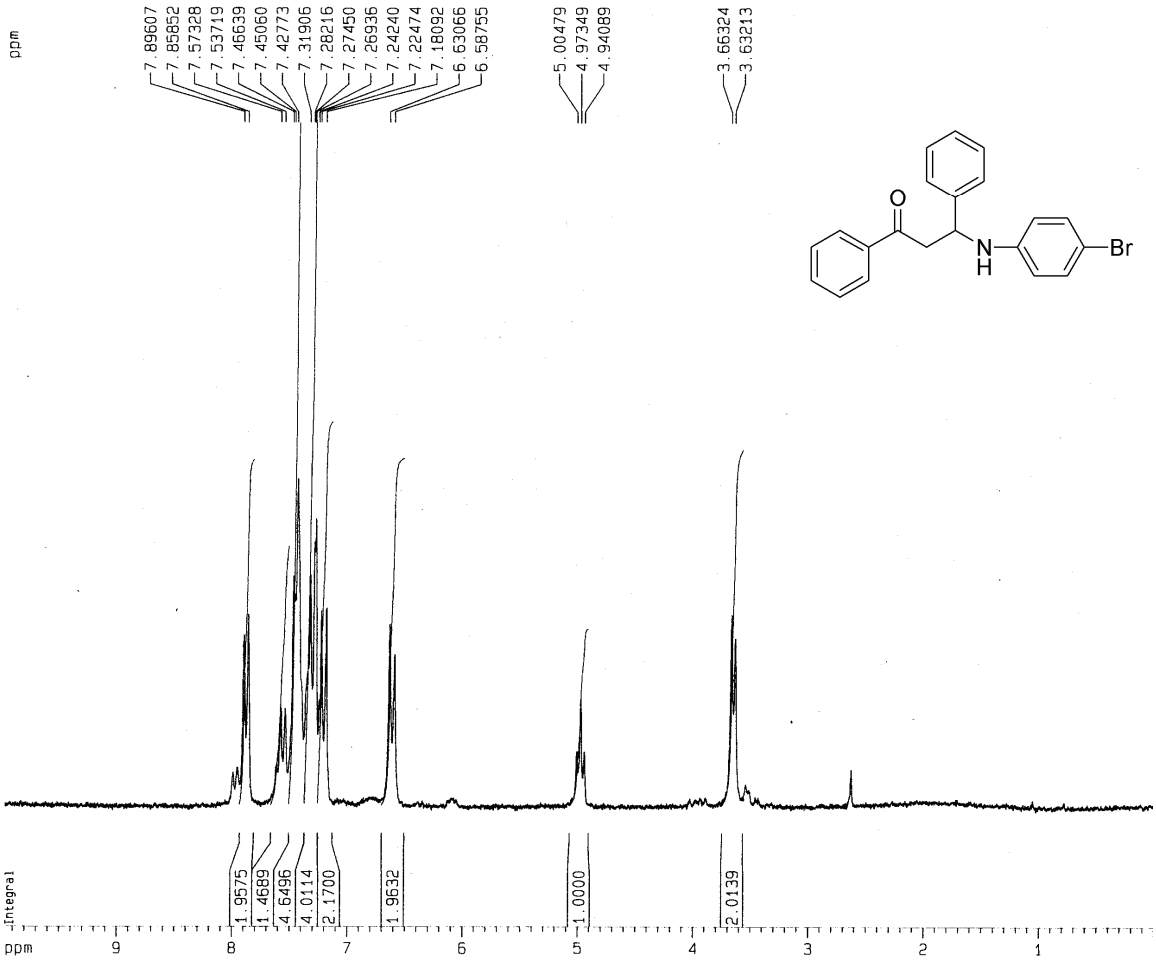
76.362
55.121
55.081
46.112
OMe
O
N
H
ppm
150
100
50

^{1}H NMR of 4e



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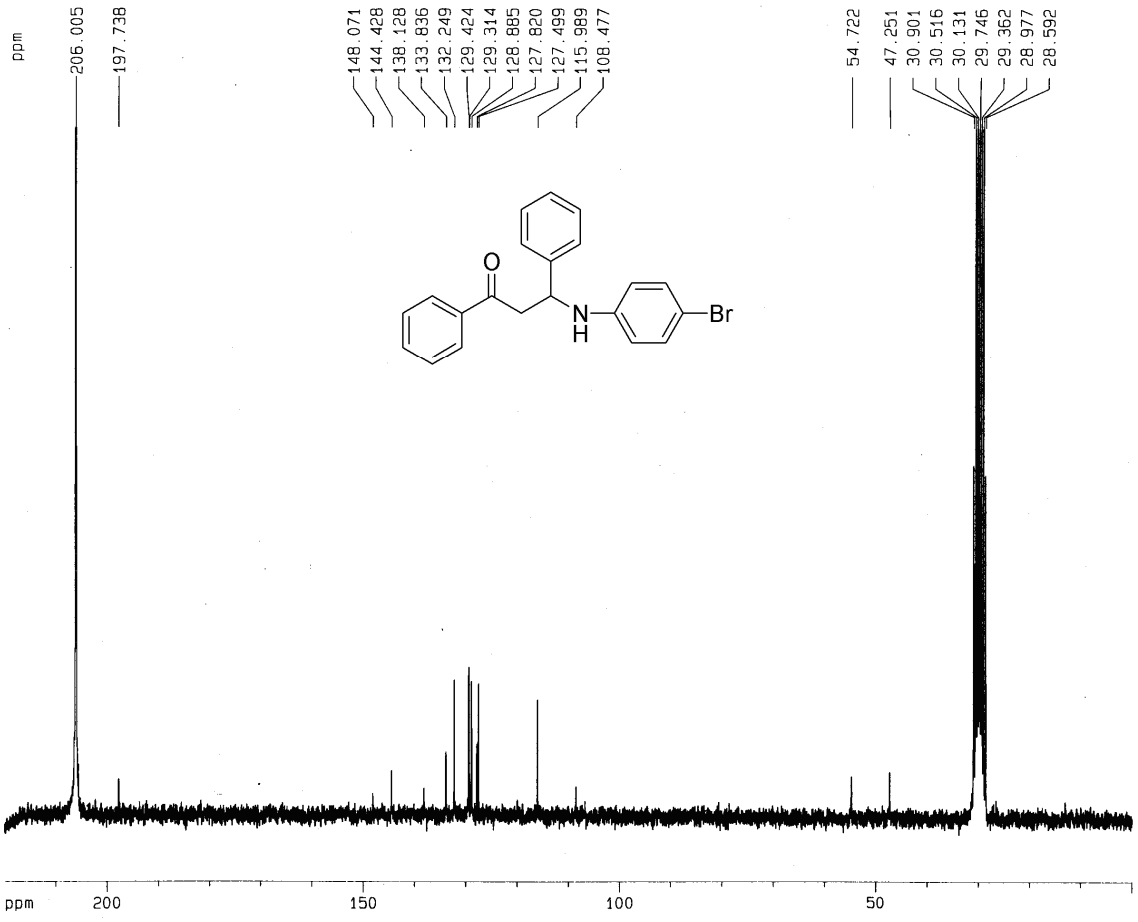
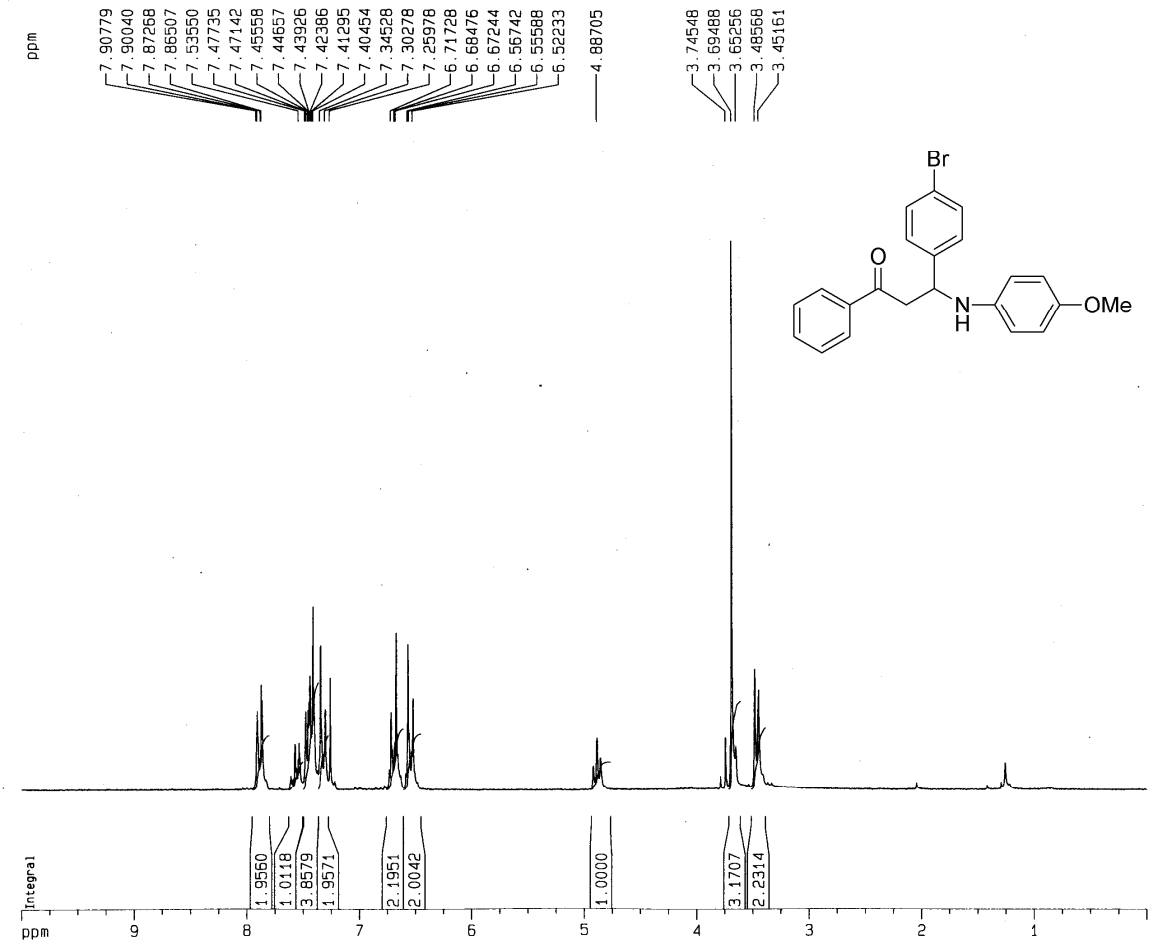


^{1}H NMR of 4f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7.42386
7.41295
7.40454
7.34528
7.30278
7.25978
6.71728
6.68476
6.67244
6.56742
6.55588
6.52233
4.88705
3.74548
3.69488
3.65256
3.48568
3.45161
Br
O
N
H
OMe
Integral
1.9560
1.0118
3.8579
1.9571
2.1951
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1.0000
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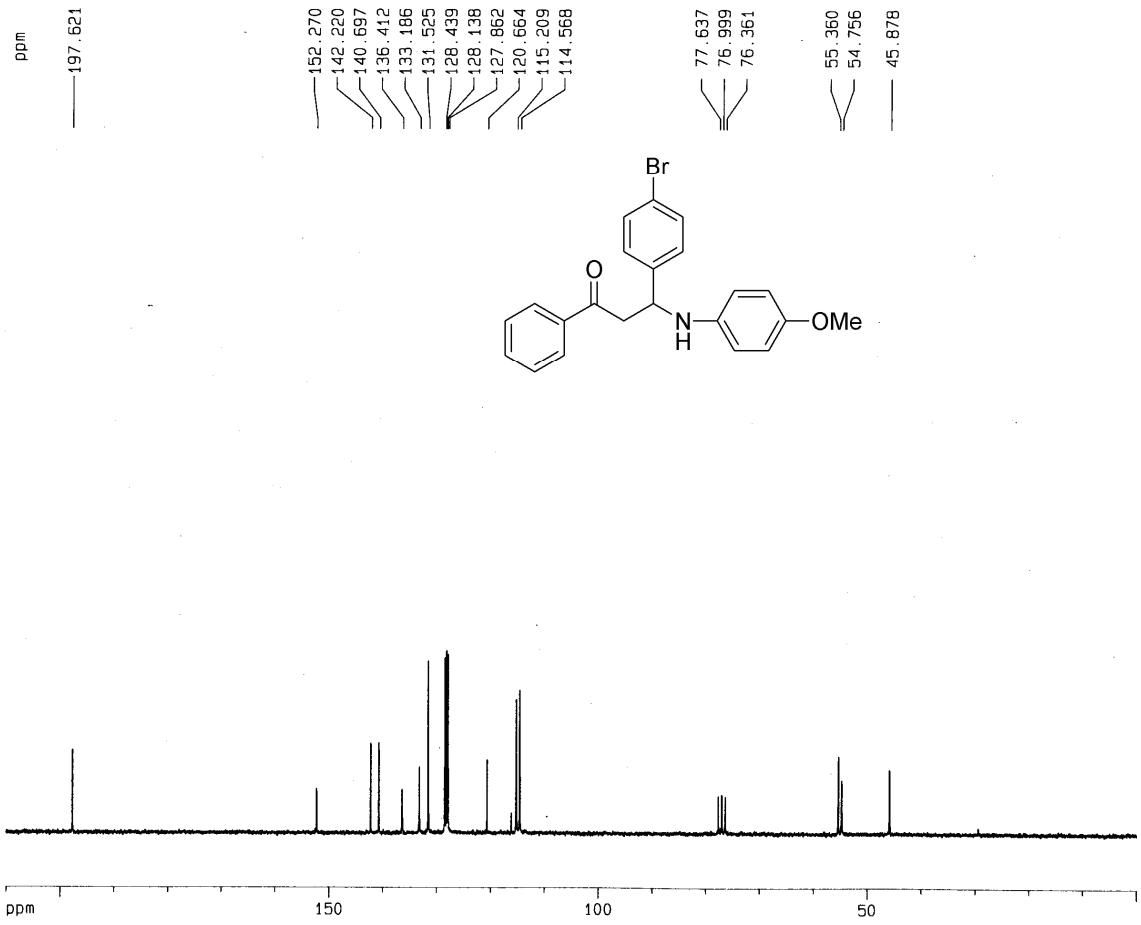
ppm
9
8
7
6
5
4
3
2
1

^{13}C NMR of 4f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114.568
77.637
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54.756
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Br
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N
H
OMe
ppm
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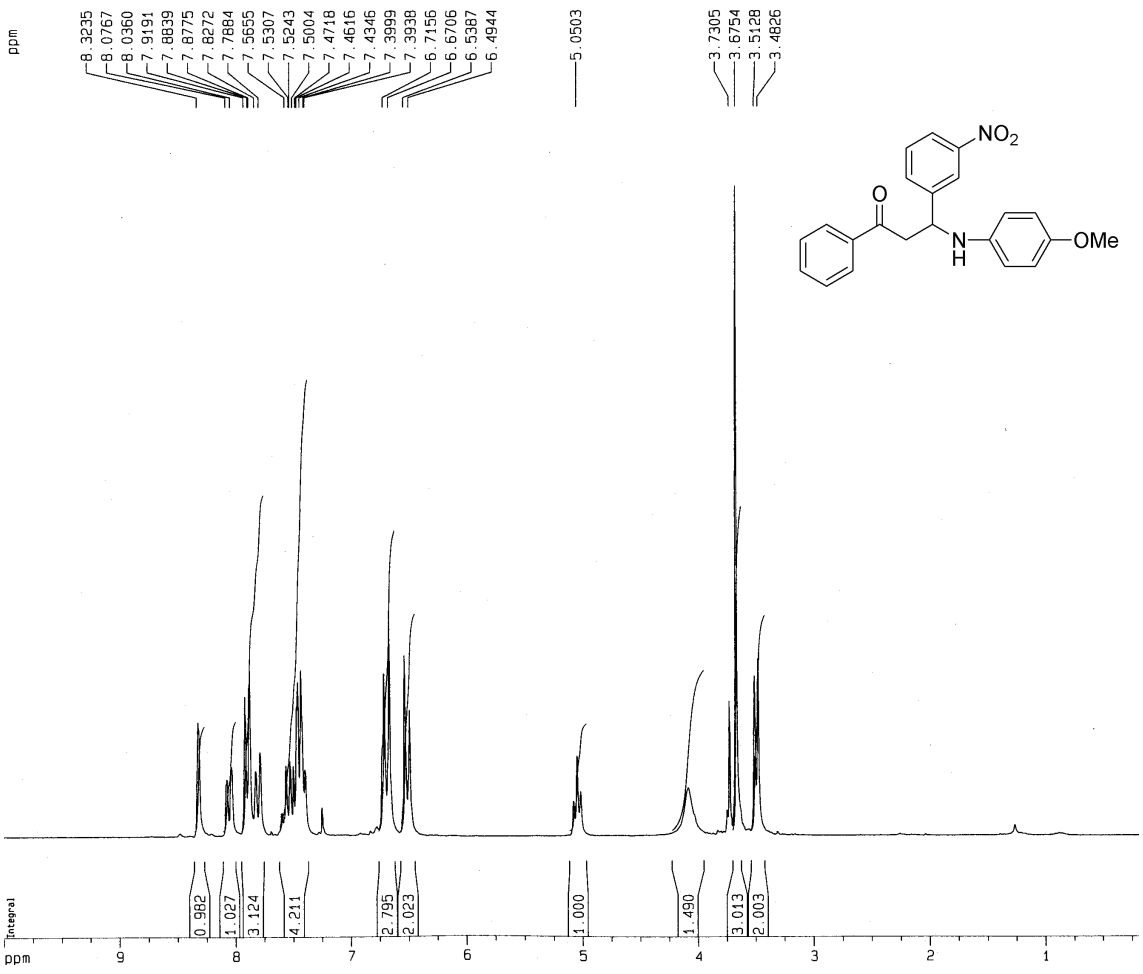
100
50

^{1}H NMR of 4g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7.5307
7.5243
7.5004
7.4718
7.4616
7.4346
7.3999
7.3938
7.7156
6.6706
6.5387
6.4944
5.0503
3.7305
3.6754
3.5128
3.4826
NO2
O
N
H
OMe
Integral
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4.211
2.795
2.023
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7
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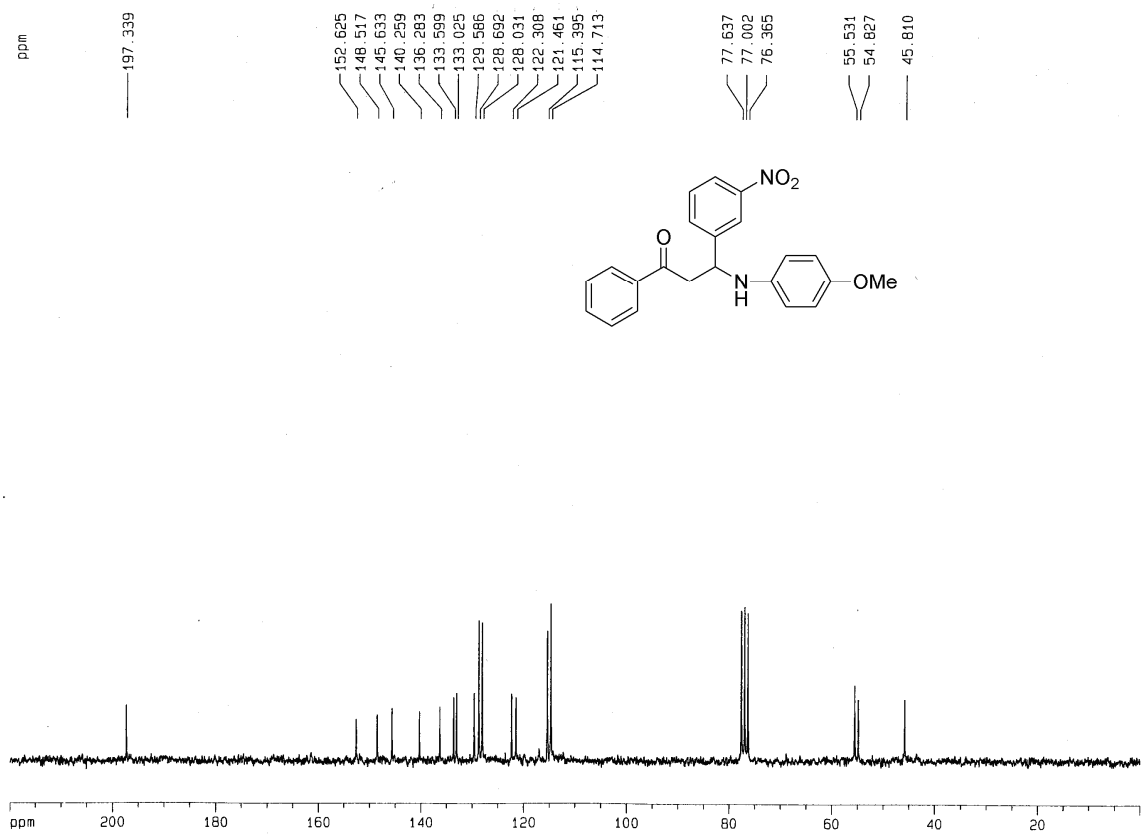
5
4
3
2
1

^{13}C NMR of 4g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121.461
115.395
114.713
77.637
77.002
76.365
55.531
54.827
45.810
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O
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H
OMe
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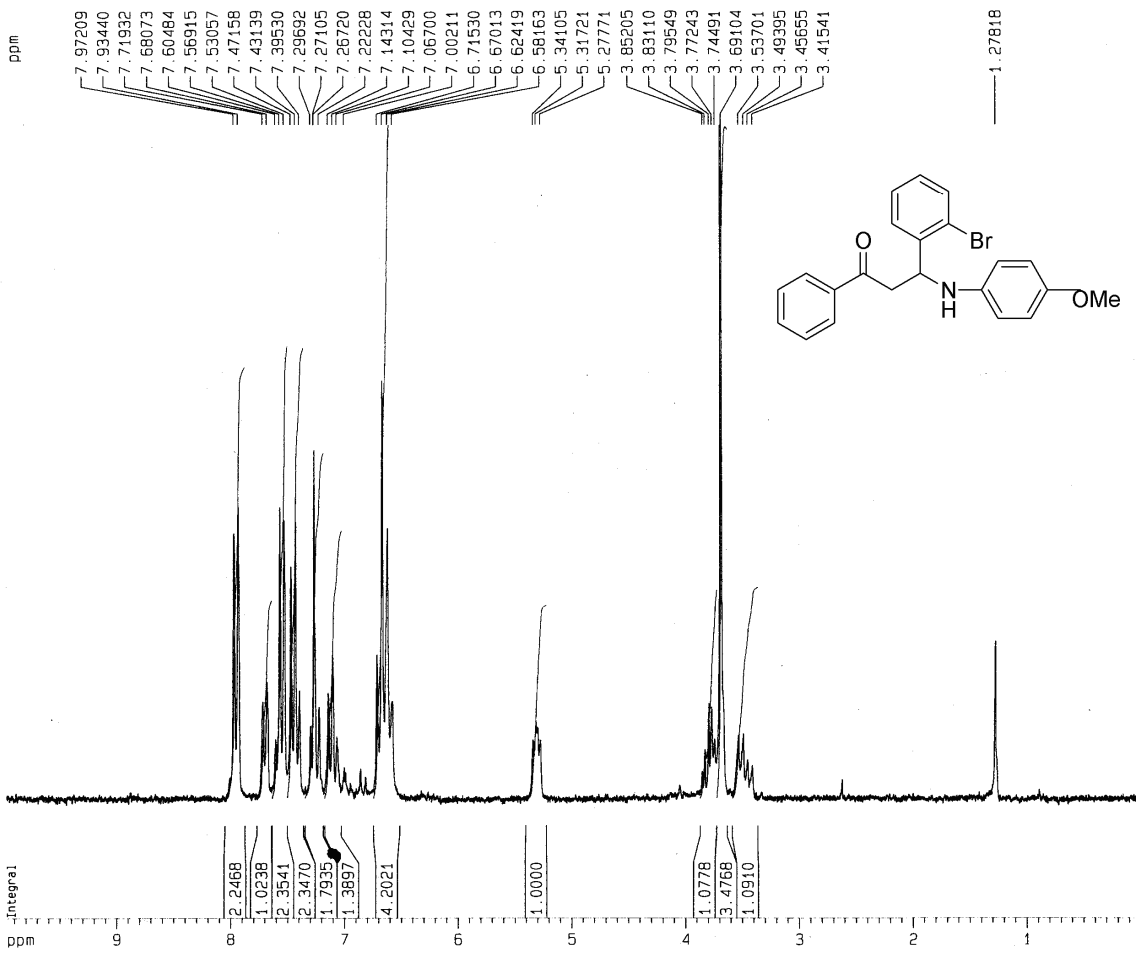
140
120
100
80
60
40
20

^{1}H NMR of 4h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7.29692
7.27105
7.26720
7.22228
7.14314
7.10429
7.06700
7.00211
6.71530
6.67013
6.62419
6.58163
5.34105
5.31721
5.27771
3.85205
3.83110
3.79549
3.77243
3.74491
3.69104
3.53701
3.49395
3.45655
3.41541
1.27818
O
Br
N
H
OMe
Integral
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ppm
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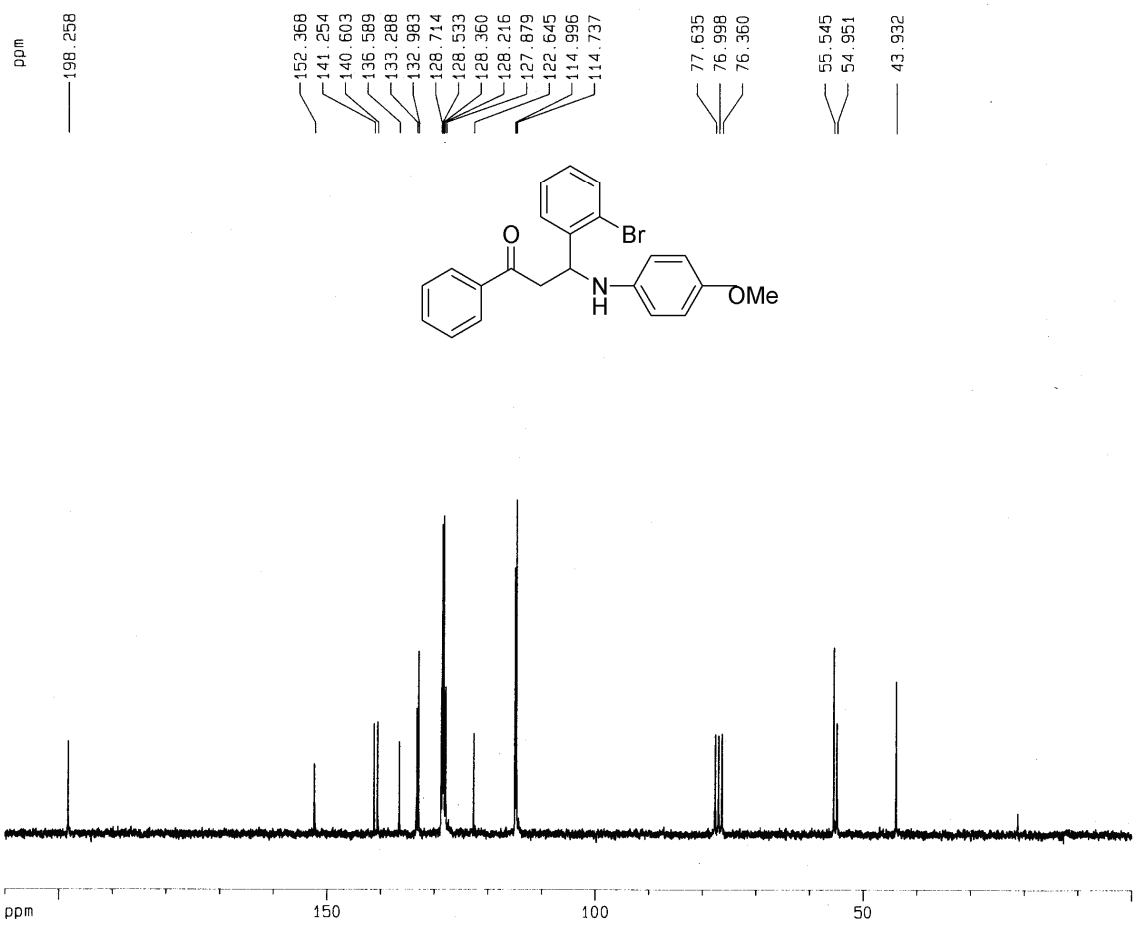
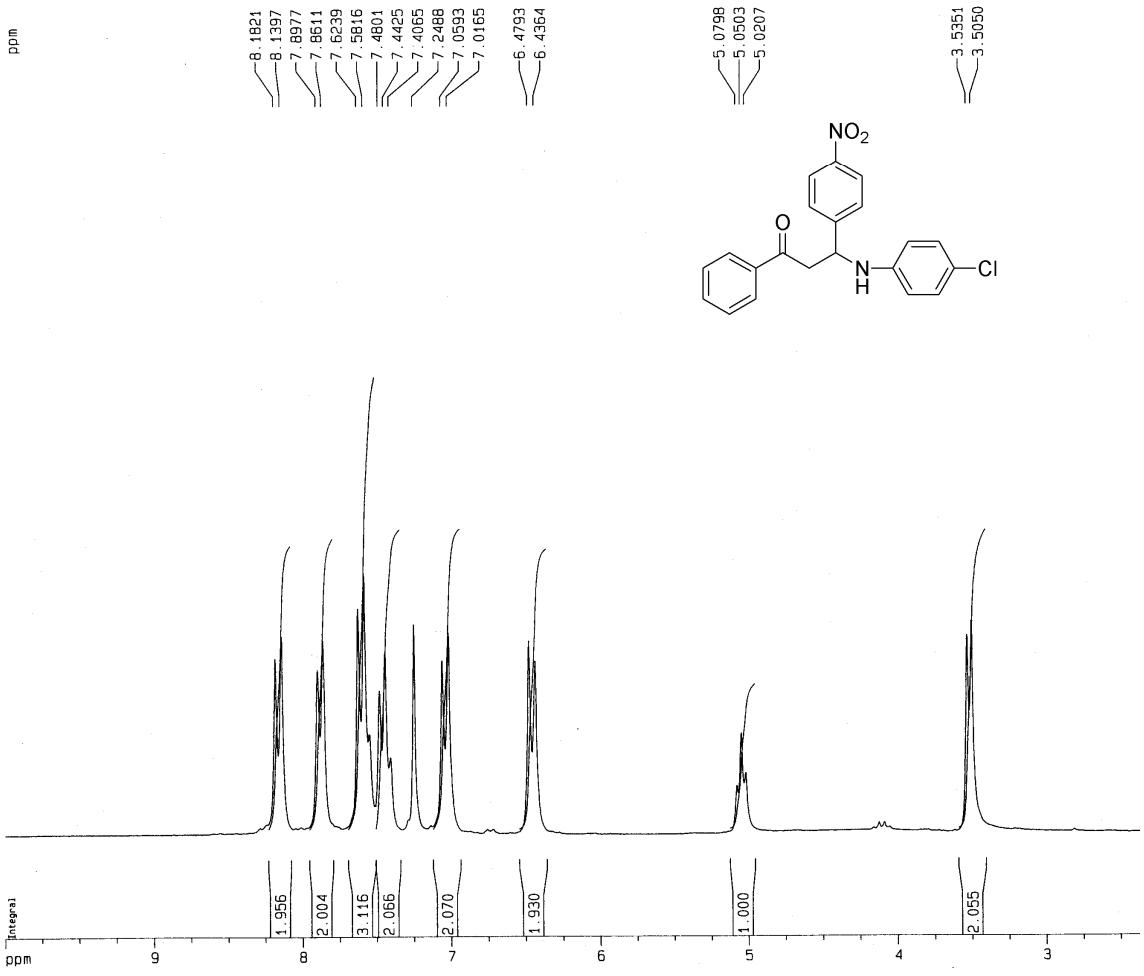
8
7
6
5
4
3
2
1

^{13}C NMR of 4h



^{1}H NMR of 4i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7.0165
6.4793
6.4364
5.0798
5.0503
5.0207
3.5351
3.5050
NO2
O
N
H
Cl
Integral
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2.004
3.116
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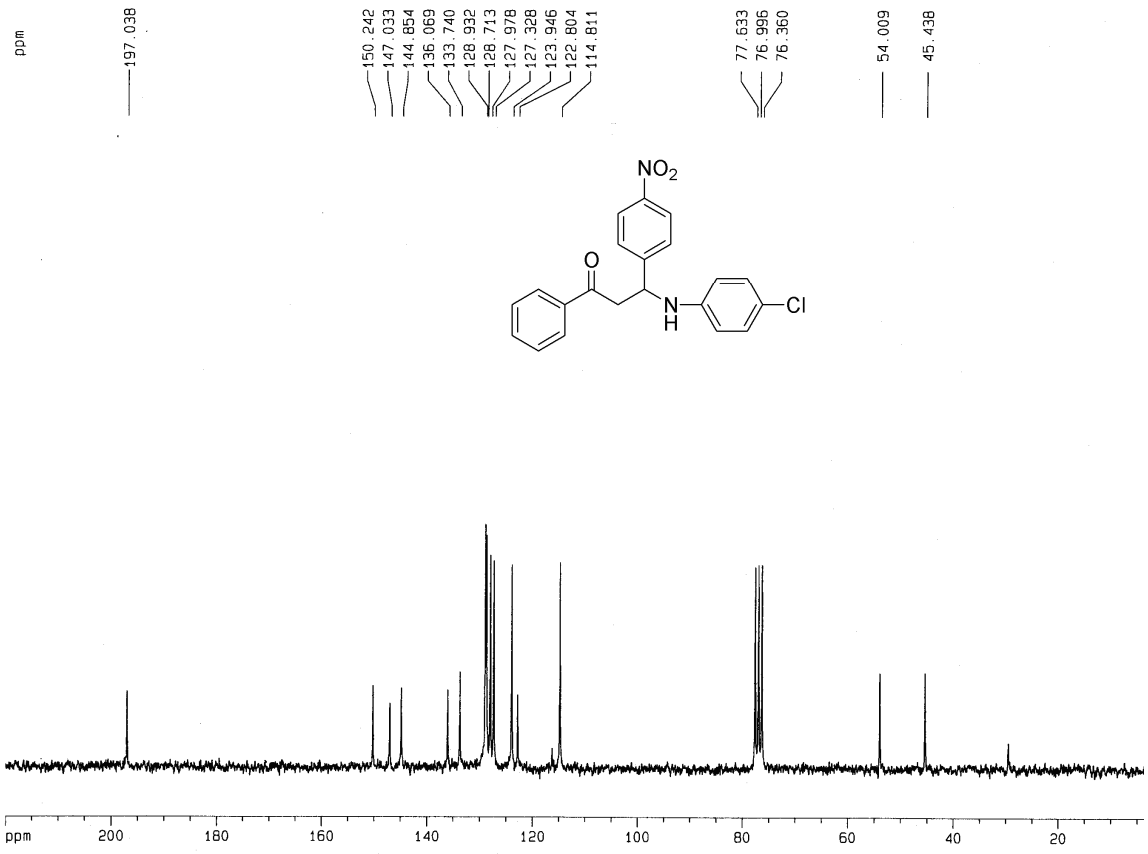
7
6
5
4
3

^{13}C NMR of 4i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76.360
54.009
45.438
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H
Cl
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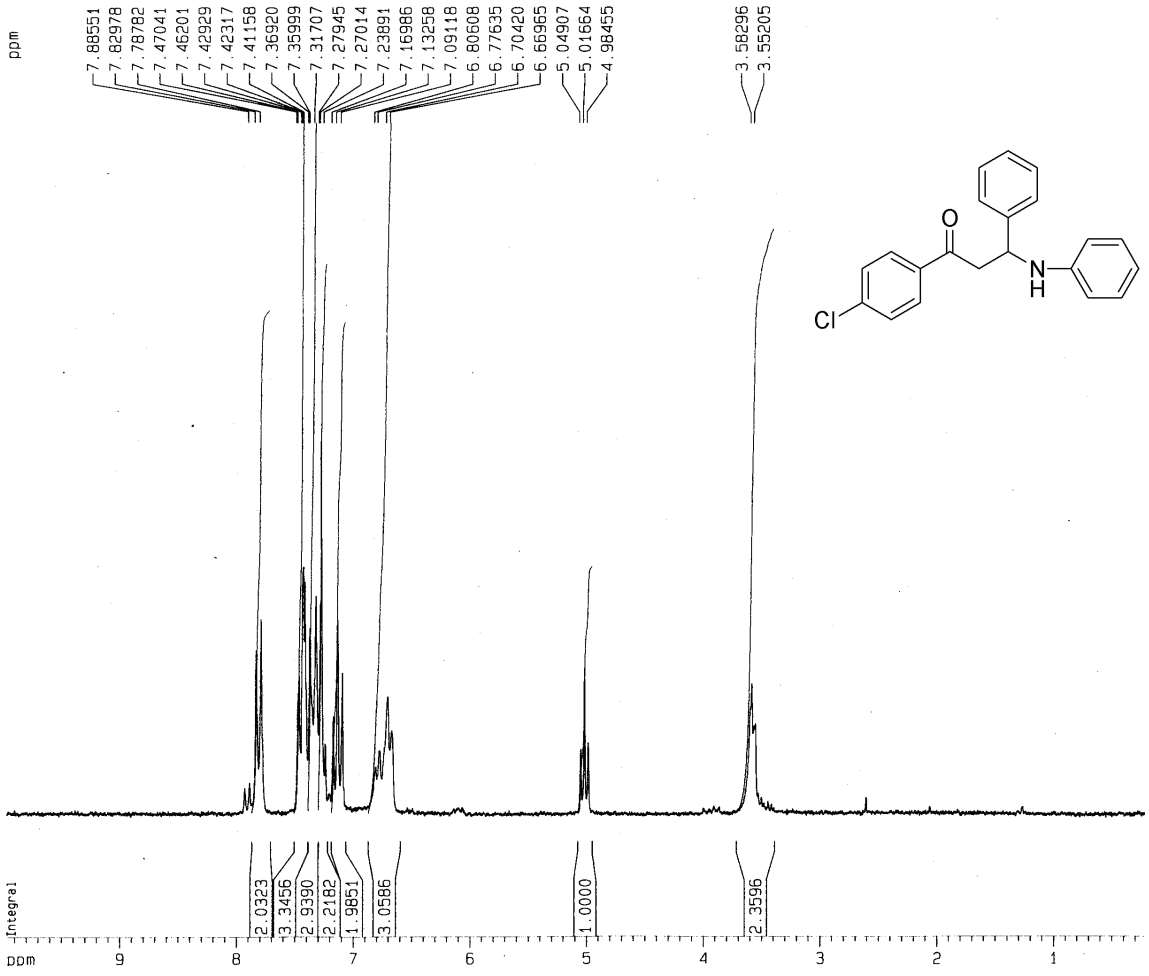
20

^{1}H NMR of 4j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7.31707
7.27945
7.27014
7.23891
7.16986
7.13258
7.09118
6.80608
6.77635
6.70420
6.66965
5.04907
5.01664
4.98455
3.58296
3.55205
O
N
H
Cl
Integral
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3.3456
2.9390
2.2182
1.9851
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4
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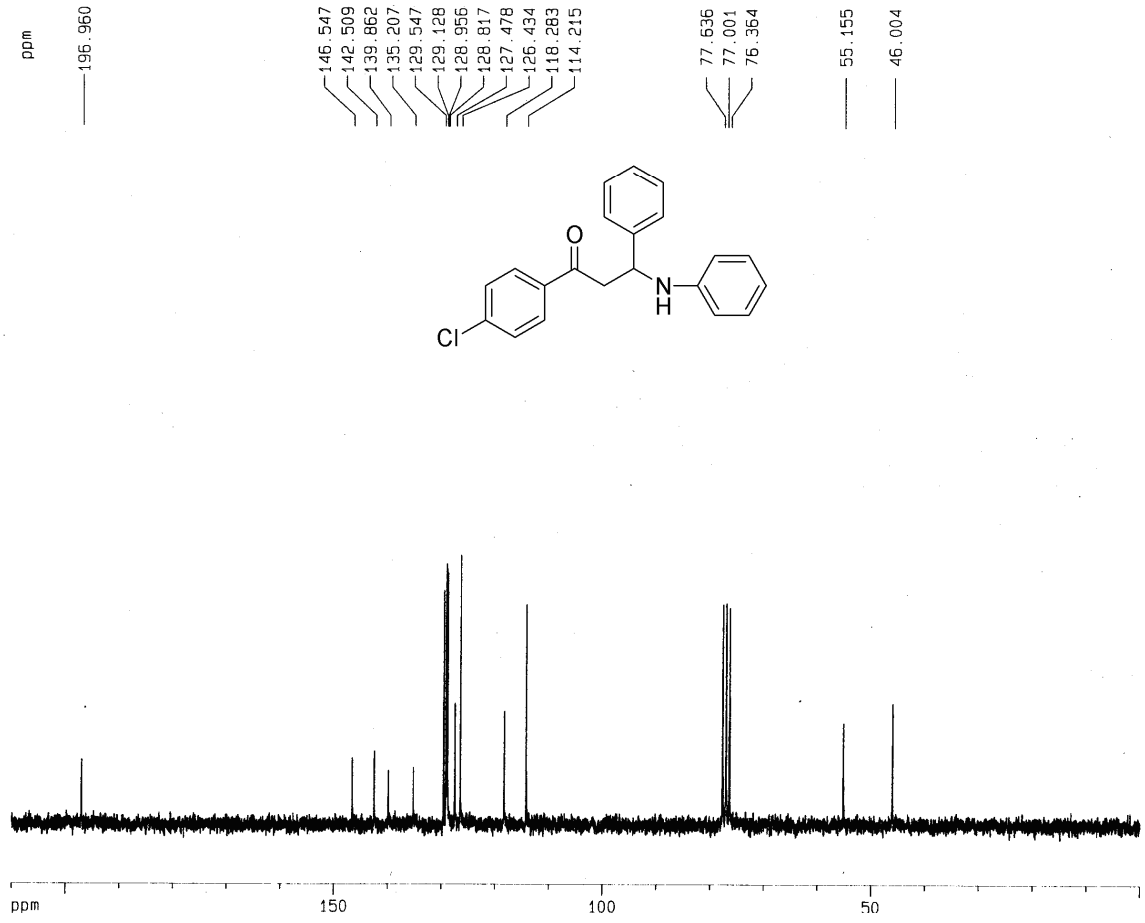
1

^{13}C NMR of 4j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118.283
114.215
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55.155
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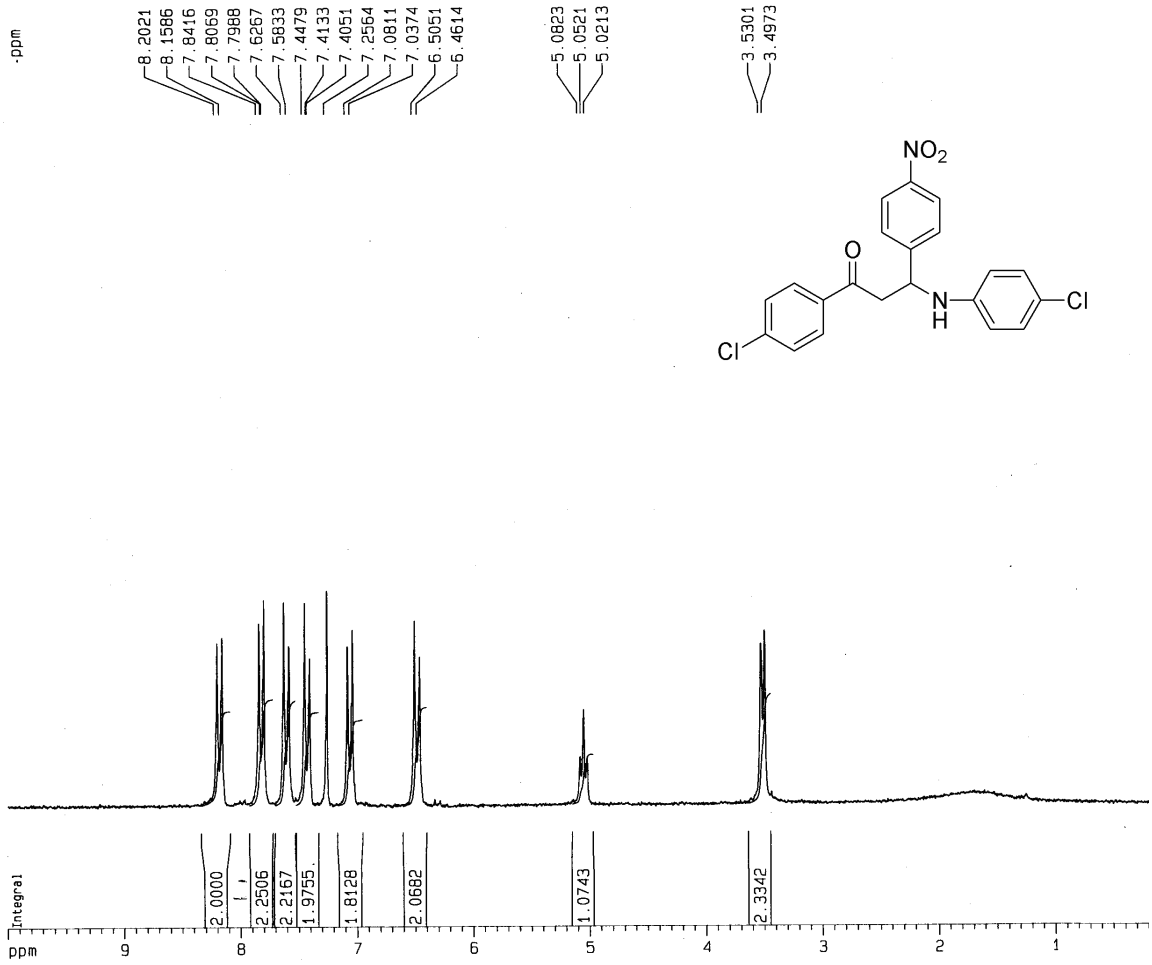
O
N
H
Cl
ppm
150
100
50

^{1}H NMR of 4k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7.2564
7.0811
7.0374
6.5051
6.4614
5.0823
5.0521
5.0213
3.5301
3.4973
NO2
O
N
H
Cl
Cl
Integral
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2.2506
2.2167
1.9755
1.8128
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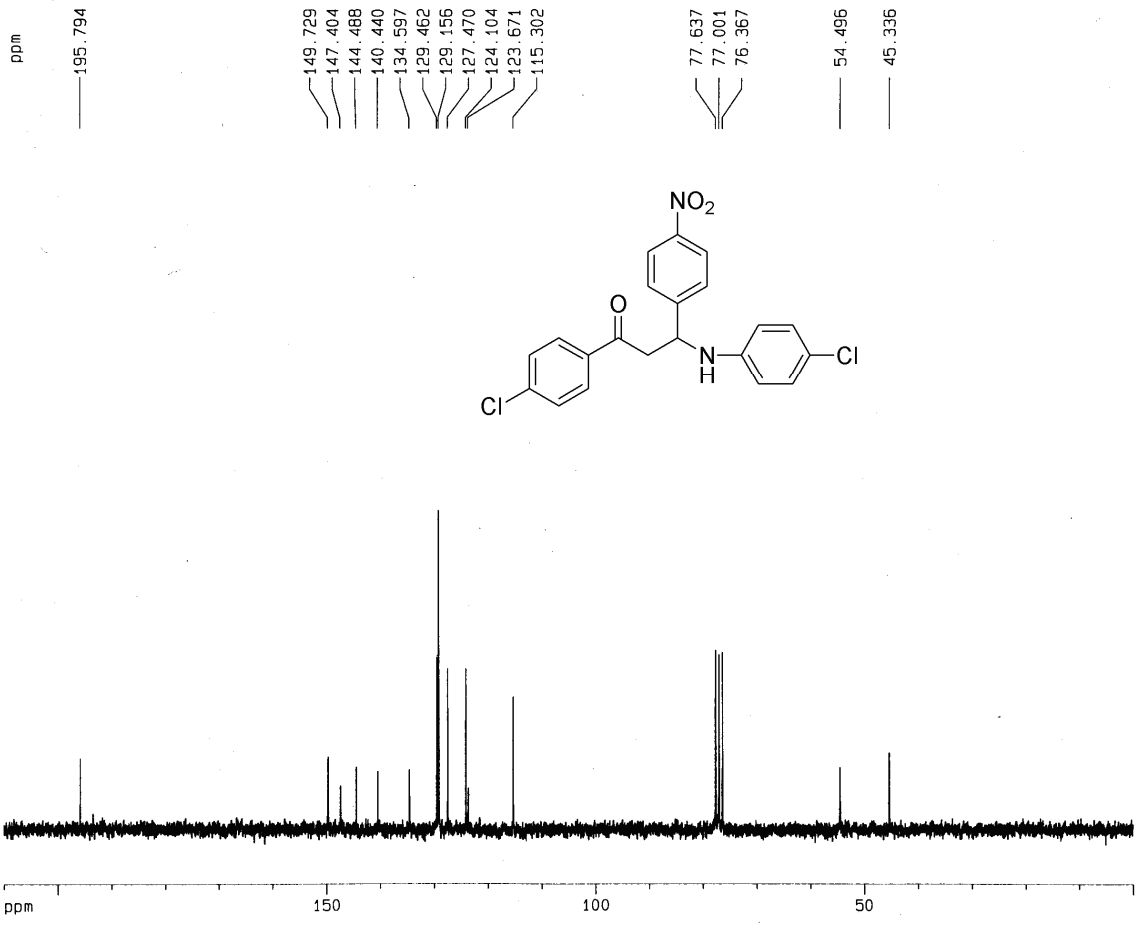
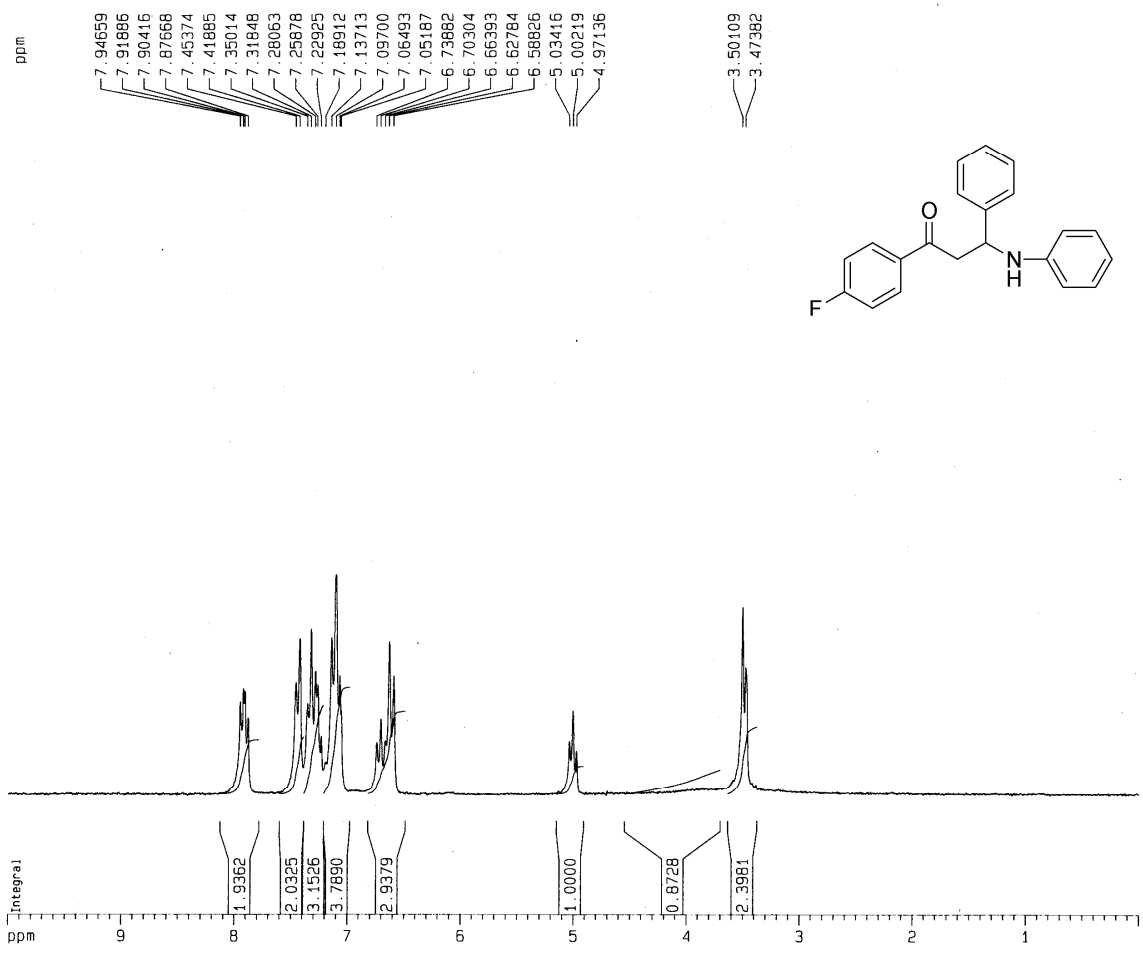
ppm
9
8
7
6
5
4
3
2
1

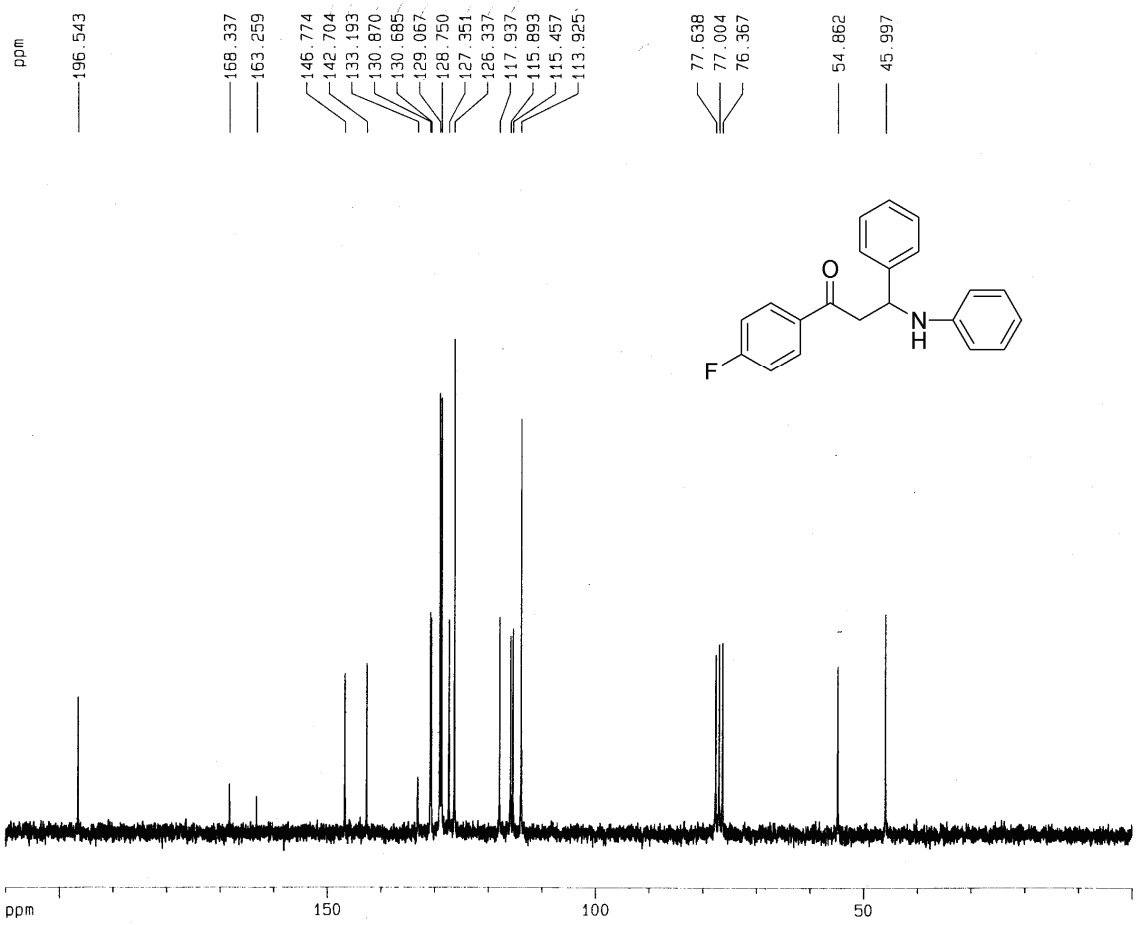
^{13}C NMR of 4k



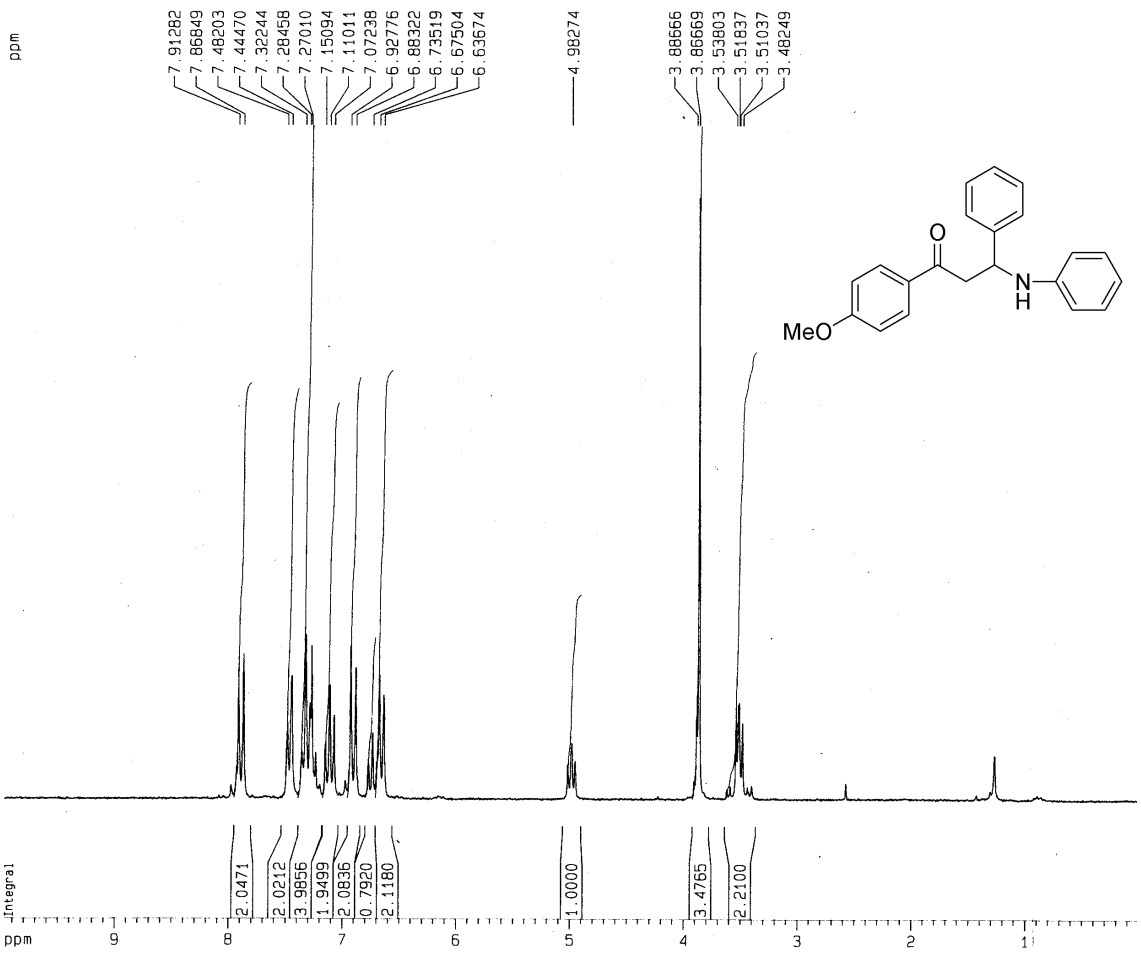
^{1}H NMR of 4l



^{13}C NMR of 4l



^{1}H NMR of 4m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6.88322
6.73519
6.67504
6.63674
4.98274
3.88666
3.86669
3.53803
3.51837
3.51037
3.48249
O
N
H
MeO
Integral
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3.9856
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2.0836
0.7920
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2.2100
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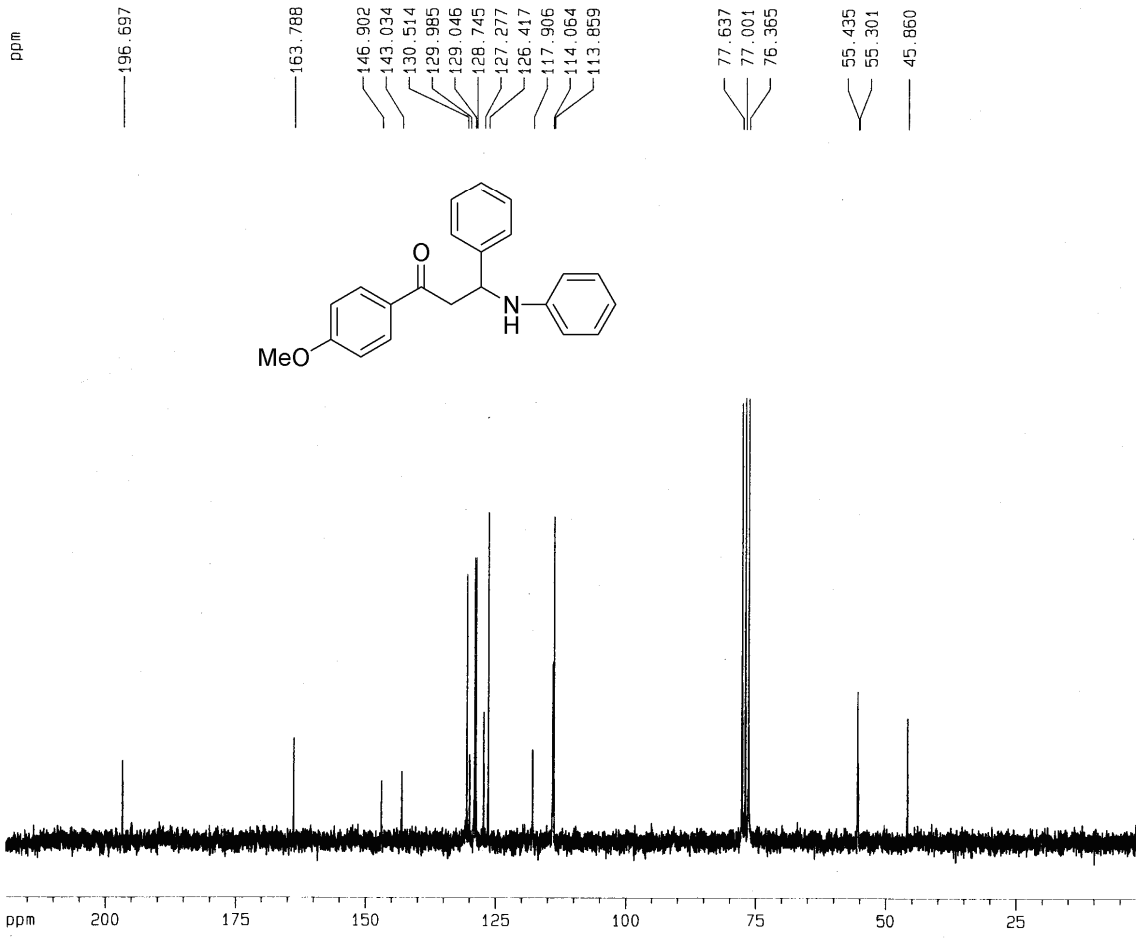
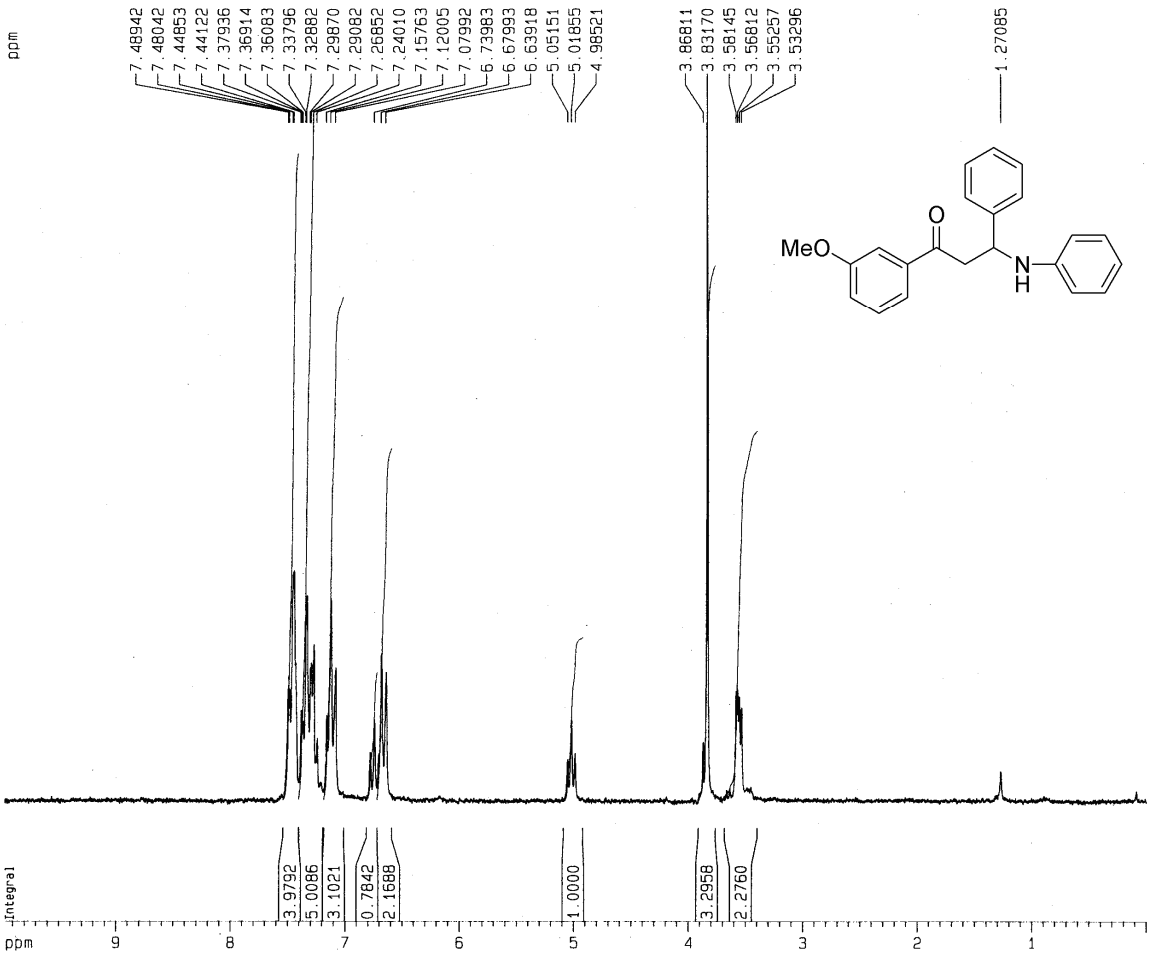
7
6
5
4
3
2
1

^{13}C NMR of 4m



^{1}H NMR of 4n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7.26852
7.24010
7.15763
7.12005
7.07992
6.73983
6.67993
6.63918
5.05151
5.01855
4.98521
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3.58145
3.56812
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3.53296
1.27085
MeO
O
N
H
Integral
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3.1021
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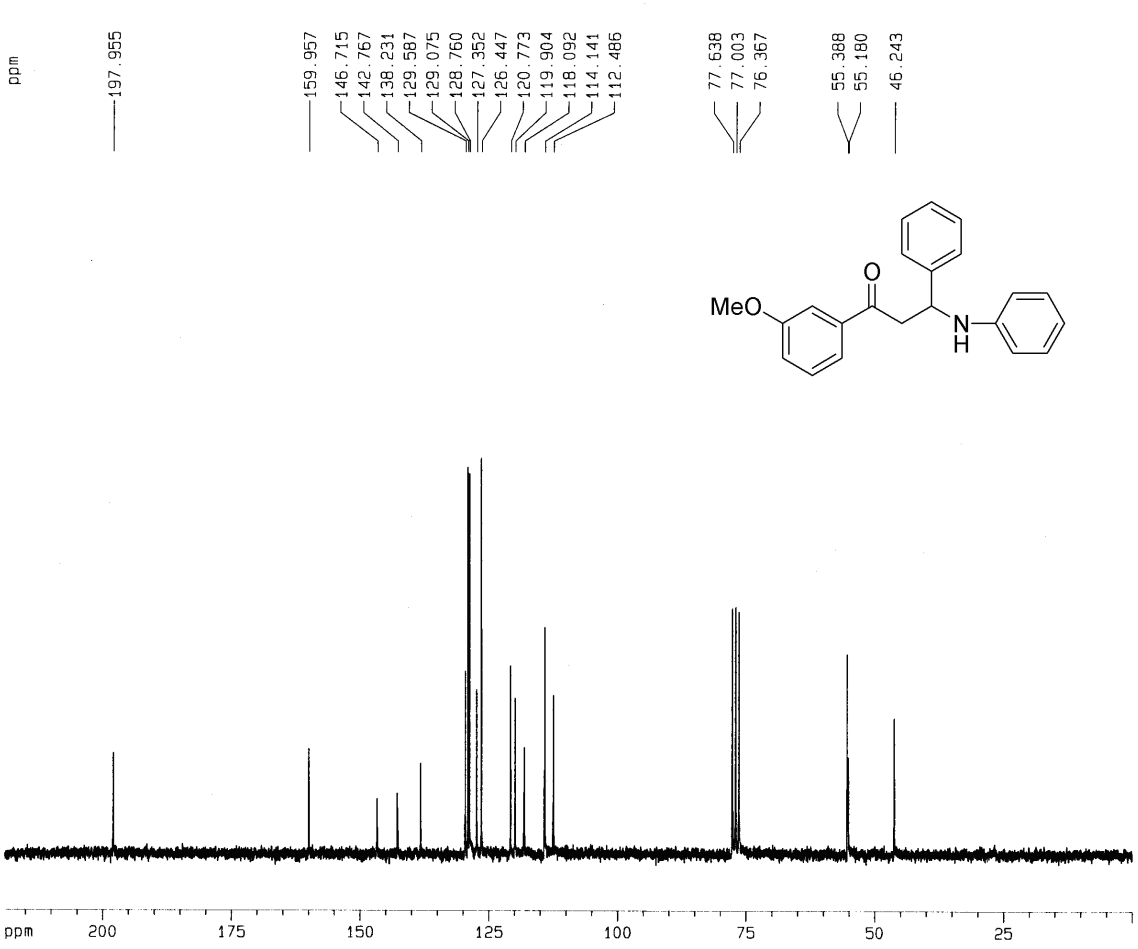
5
4
3
2
1

^{13}C NMR of 4n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146.715
142.767
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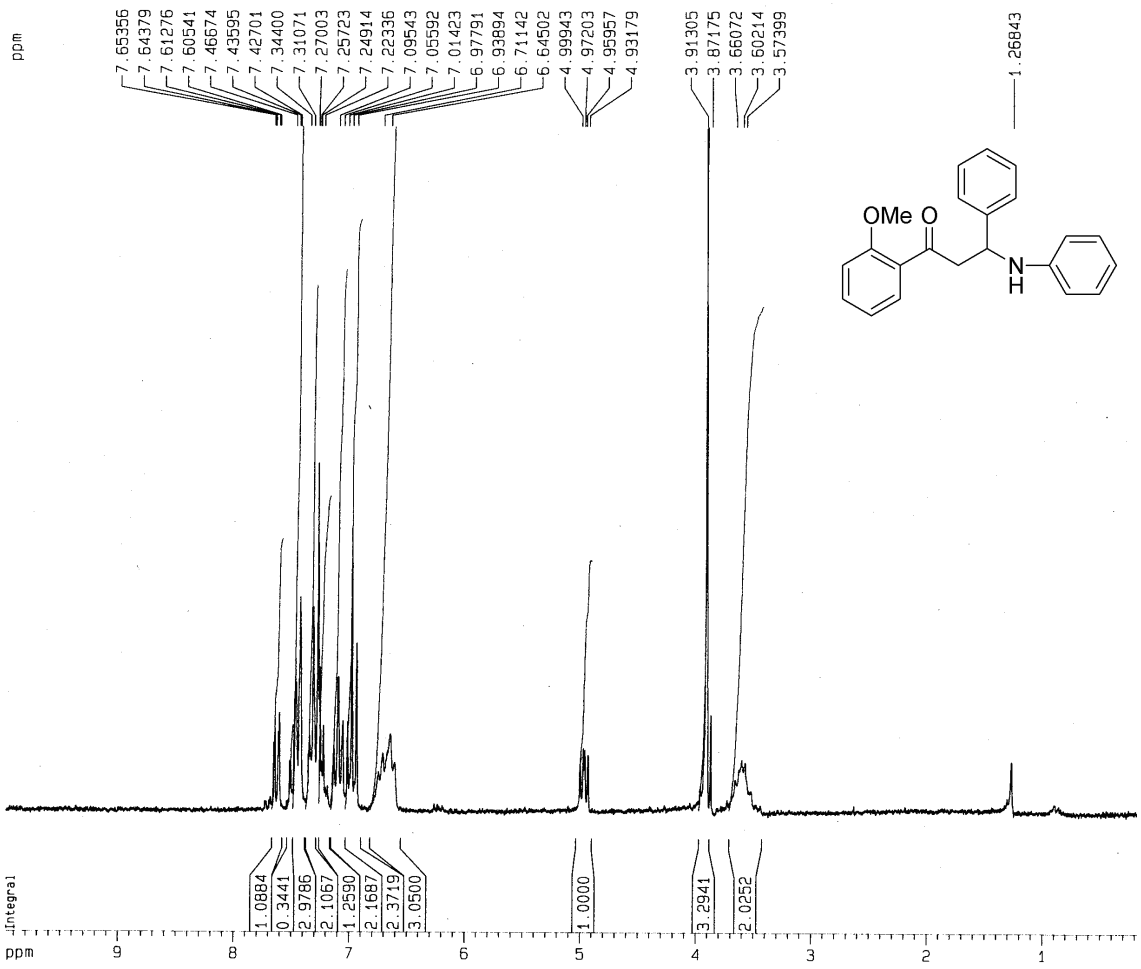
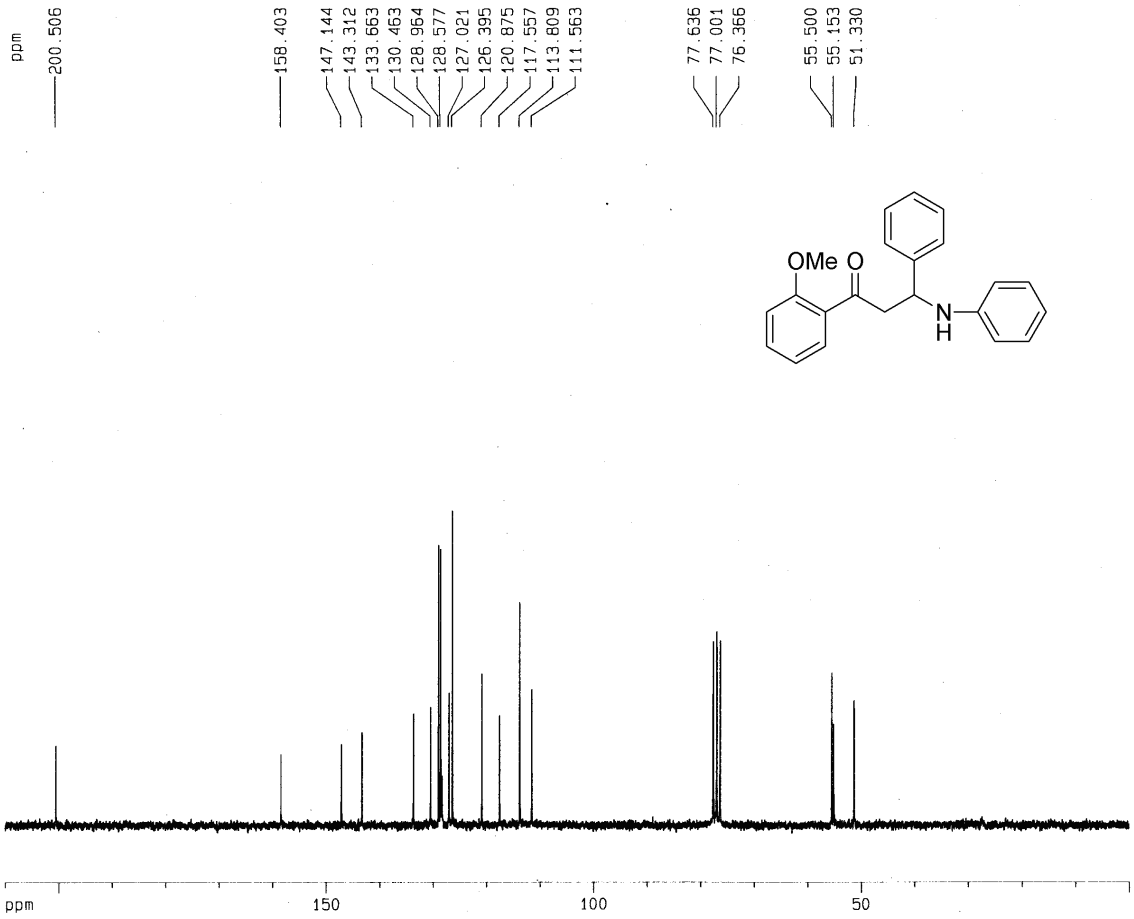
129.075
128.760
127.352
126.447
120.773
119.904
118.092
114.141
112.486
77.638
77.003
76.367
55.388
55.180
46.243
MeO
O
N
H
ppm
200
175
150
125
100
75
50
25

^{1}H NMR of 4o



^{13}C NMR of 4o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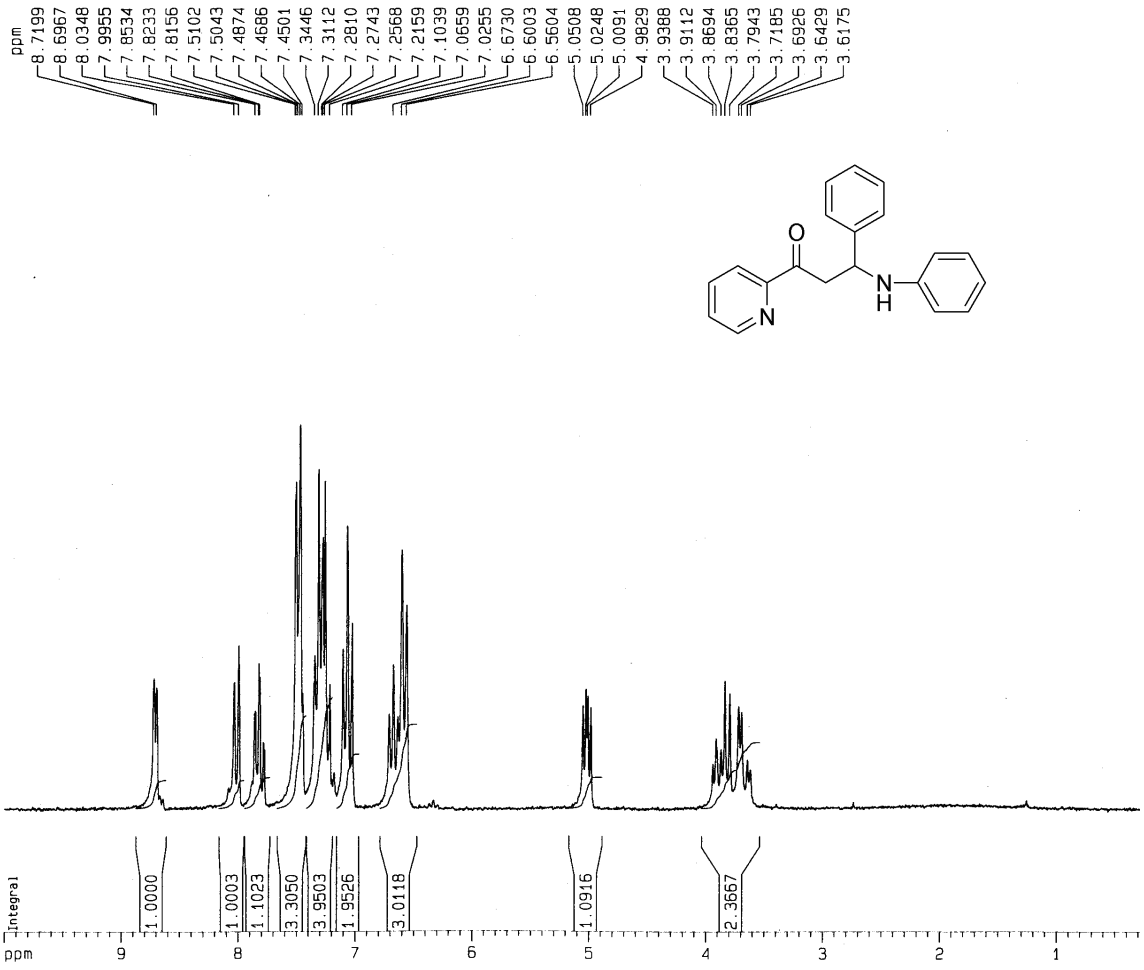
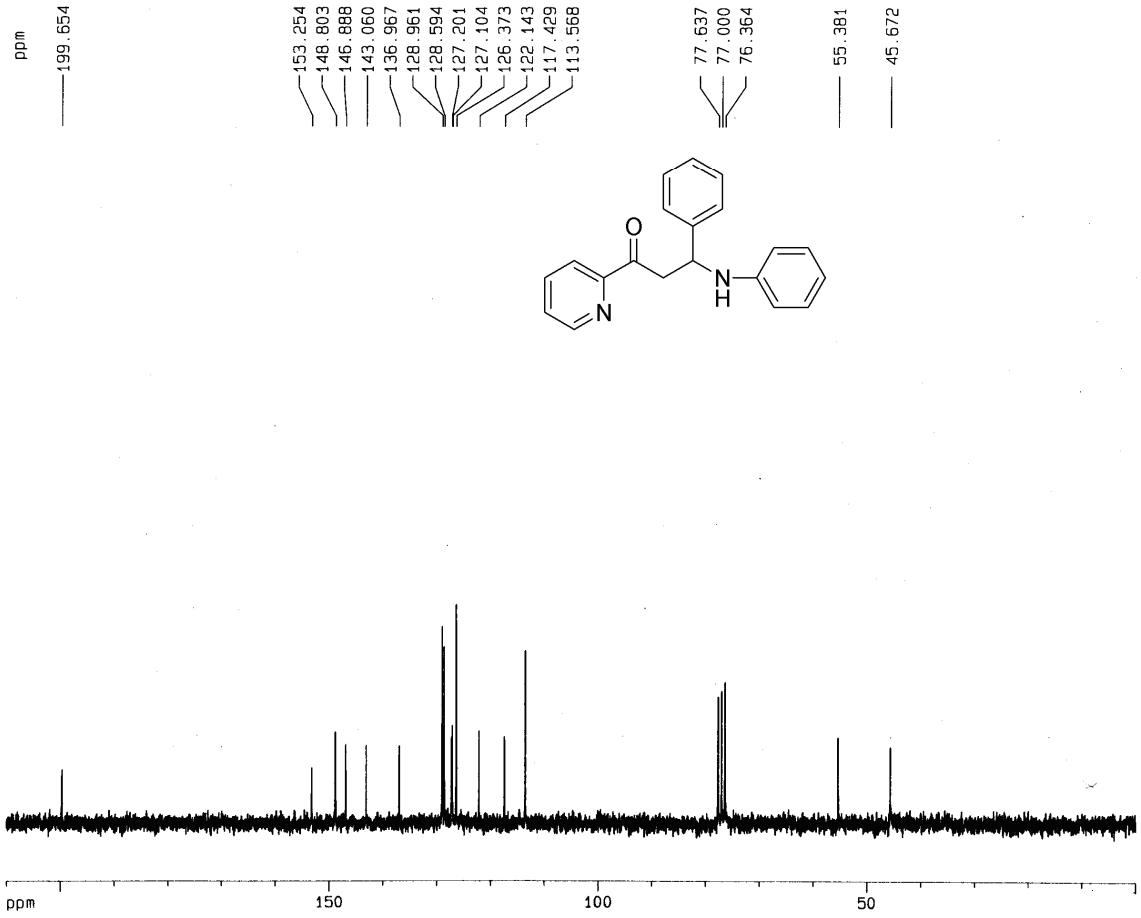
147.144
143.312
133.663
130.463
128.964
128.577
127.021
126.395
120.875
117.557
113.809
111.563
77.636
77.001
76.366
55.500
55.153
51.330
OMe O
N
H
ppm
150
100
50

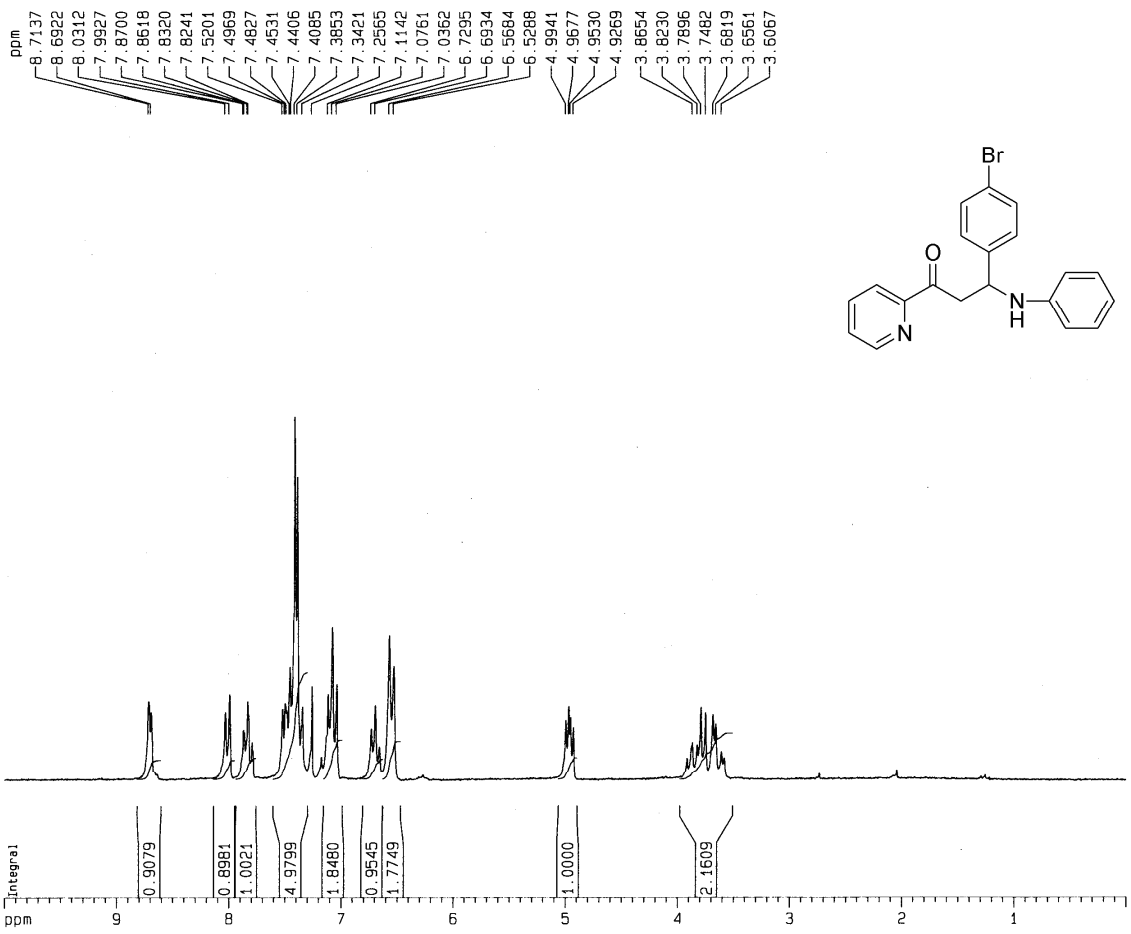
^{1}H NMR of 4p



^{13}C NMR of 4p



^{1}H NMR of 4q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7.8241
7.5201
7.4969
7.4827
7.4531
7.4406
7.4085
7.3853
7.3421
7.2565
7.1142
7.0761
7.0362
6.7295
6.6934
6.5684
6.5288
4.9941
4.9677
4.9530
4.9269
3.8654
3.8230
3.7896
3.7482
3.6819
3.6561
3.6067
Br
O
N
H
N
Integral
0.9079
0.8981
1.0021
4.9799
1.8480
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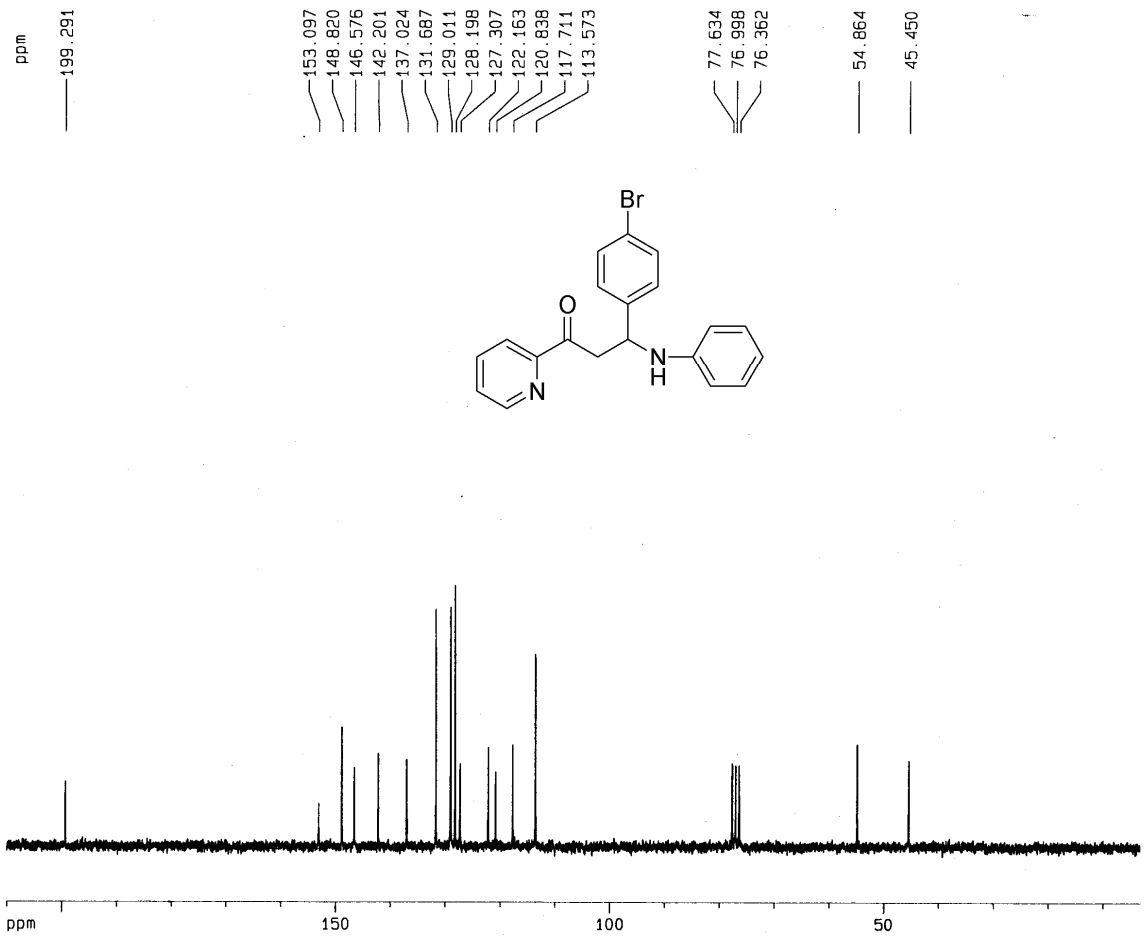
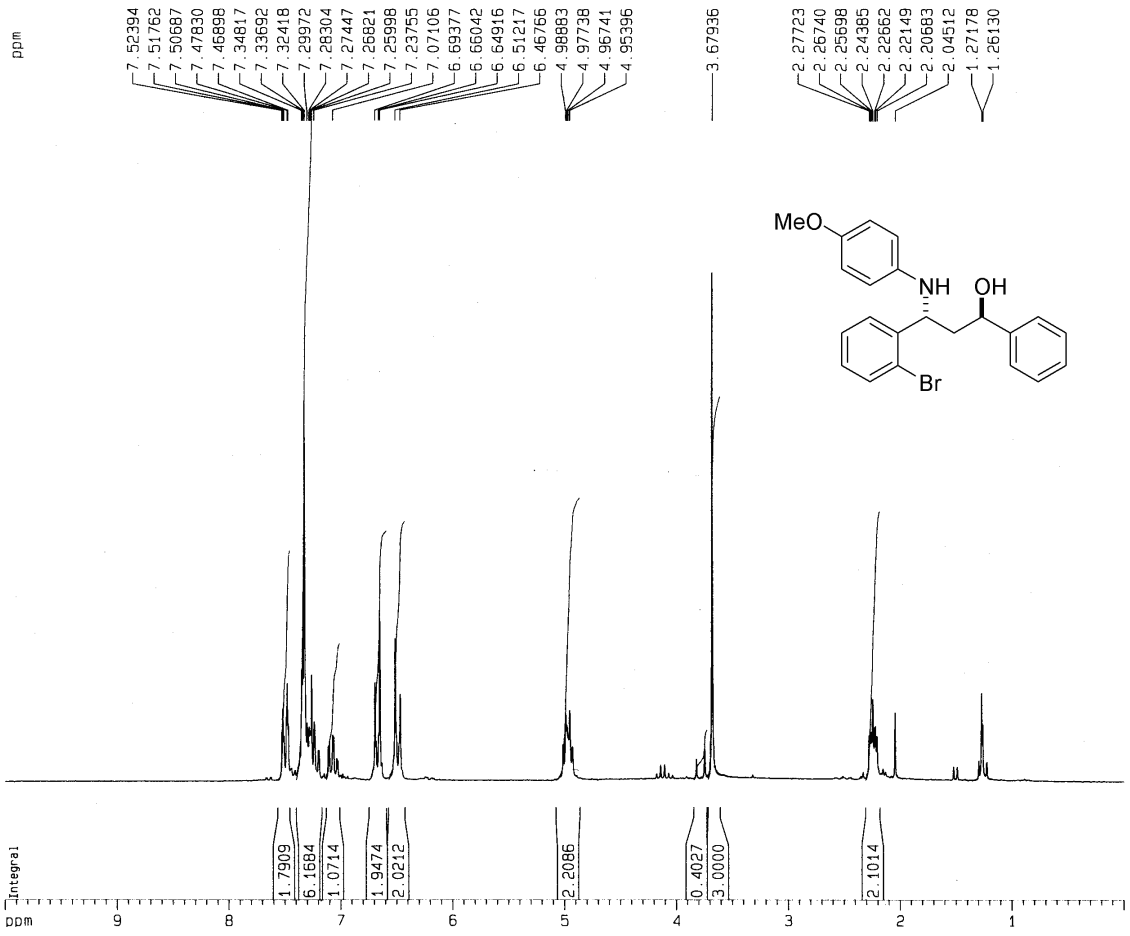
1.7749
1.0000
2.1609
ppm
9
8
7
6
5
4
3
2
1

^{13}C NMR of 4q



^{1}H NMR of 5a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7.45898
7.34817
7.33692
7.32418
7.29972
7.28304
7.27447
7.26821
7.25998
7.23755
7.07106
6.69377
6.66042
6.64916
6.51217
6.46766
4.98883
4.97738
4.96741
4.95396
3.67936
2.27723
2.26740
2.25698
2.24385
2.22662
2.22149
2.20683
2.04512
1.27178
1.26130
MeO
NH
OH
Br
Integral
1.7909
6.1684
1.0714
1.9474
2.0212
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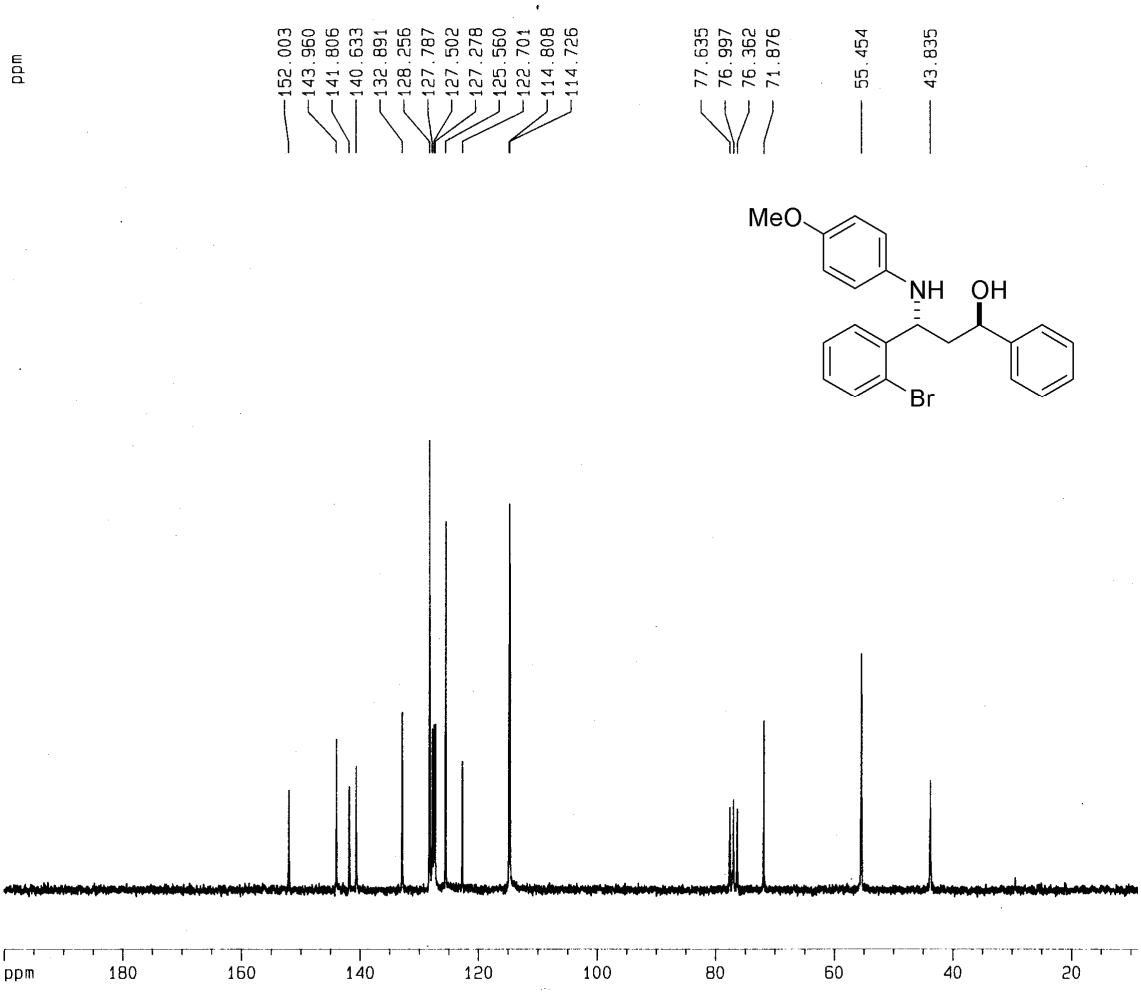
3.0000
2.1014
ppm
9
8
7
6
5
4
3
2
1

^{13}C NMR of 5a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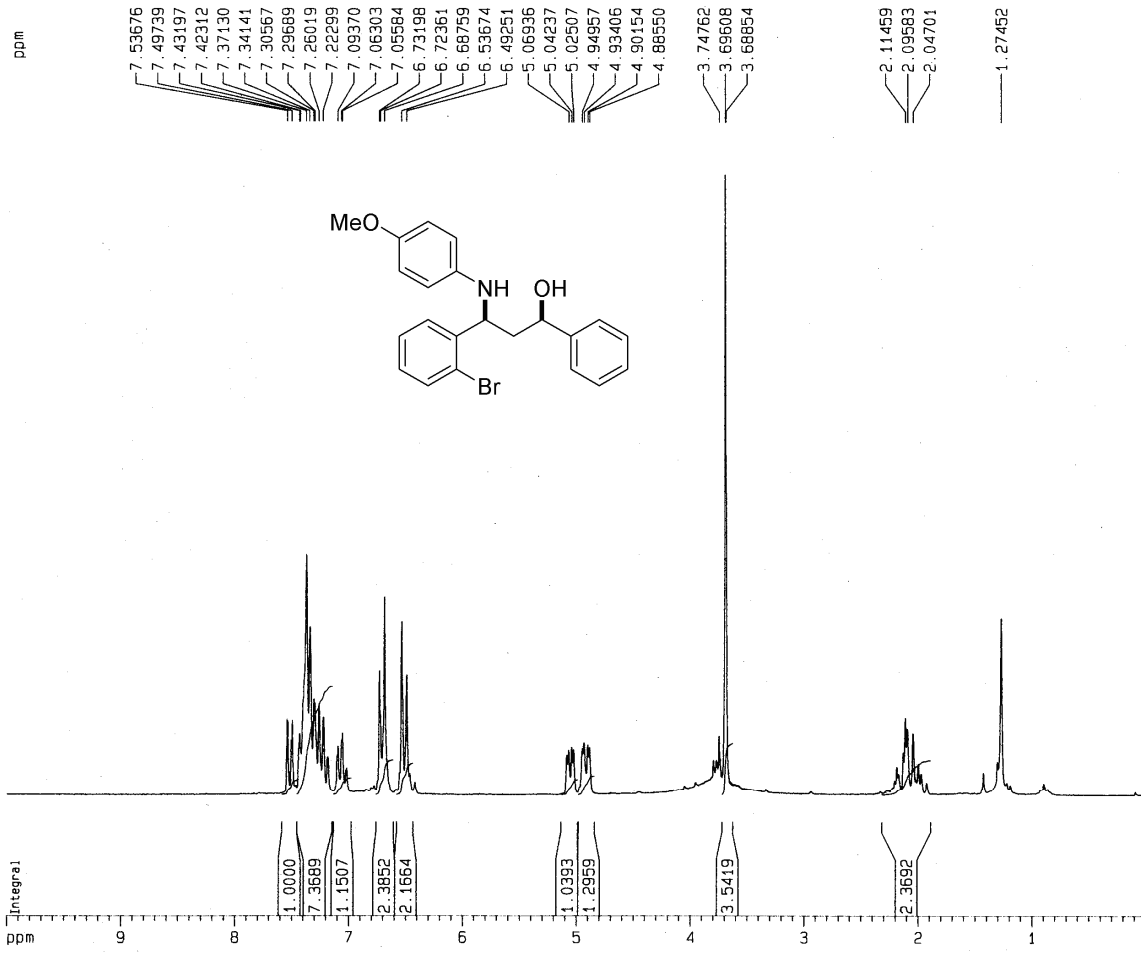
127.278
125.560
122.701
114.808
114.726
77.635
76.997
76.362
71.876
55.454
43.835
MeO
NH
OH
Br
ppm
180
160
140
120
100
80
60
40
20

^{1}H NMR of 5b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7.29689
7.26019
7.22299
7.09370
7.06303
7.05584
6.73198
6.72361
6.68759
6.53674
6.49251
5.06936
5.04237
5.02507
4.94957
4.93406
4.90154
4.88550
3.74762
3.69608
3.68854
2.11459
2.09583
2.04701
1.27452
MeO
NH
OH
Br
Integral
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7.3689
1.1507
2.3852
2.1664
1.0393
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2.3692
ppm
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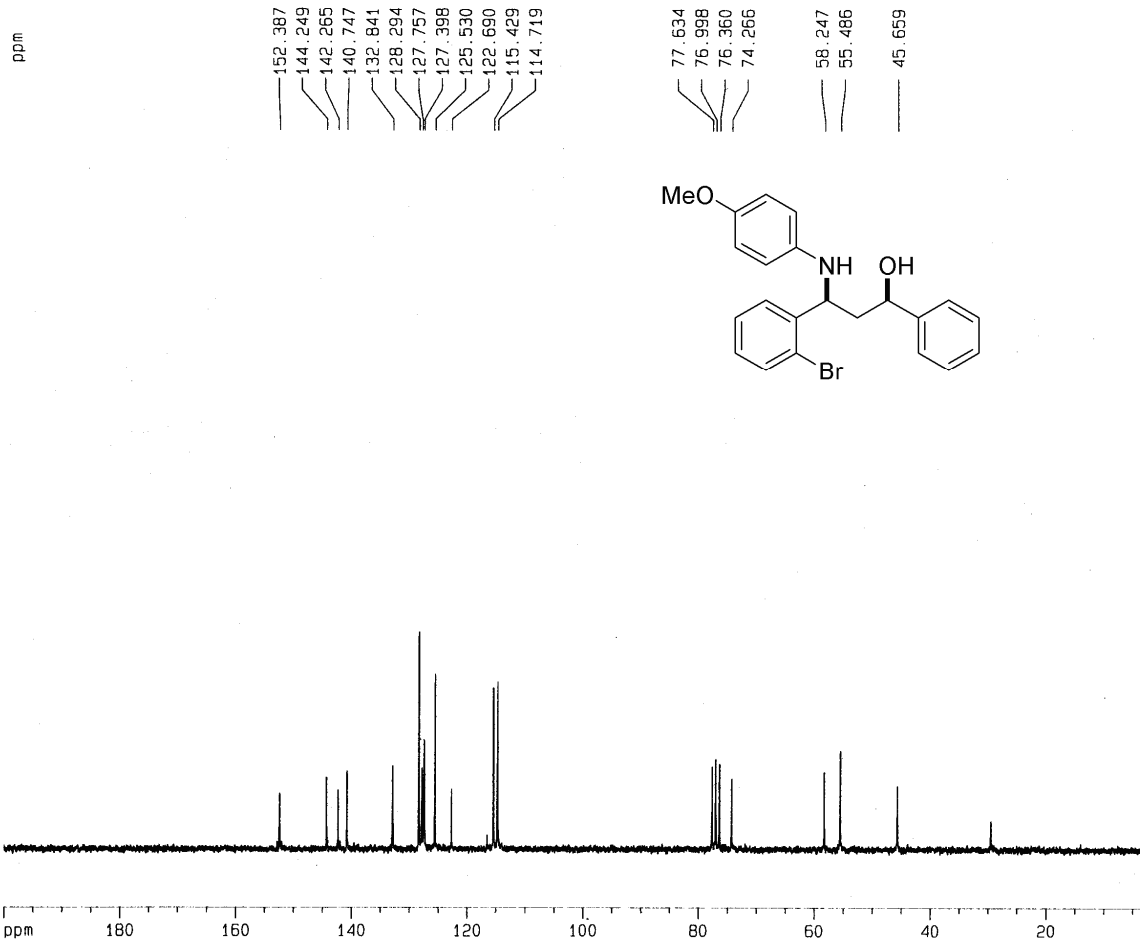
8
7
6
5
4
3
2
1

^{13}C NMR of 5b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142.265
140.747
132.841
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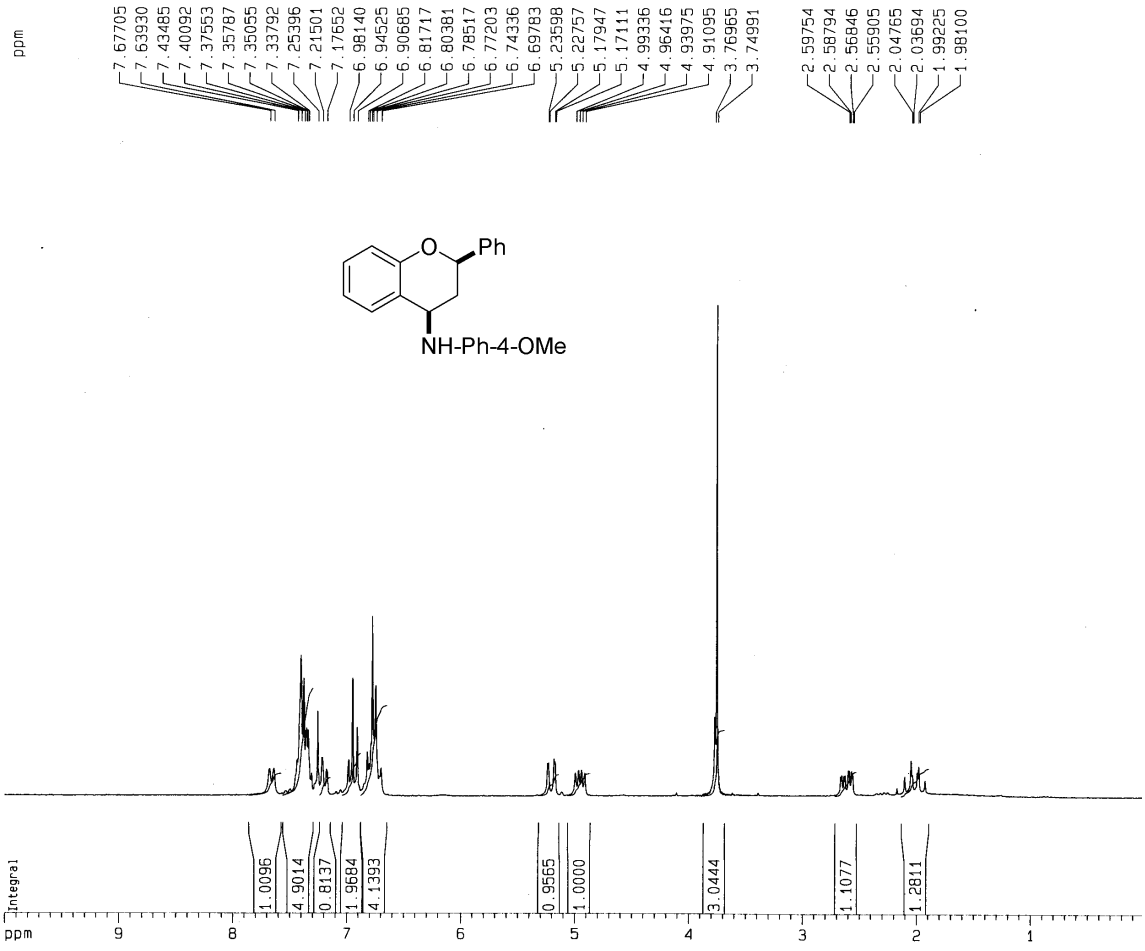
127.757
127.398
125.530
122.690
115.429
114.719
77.634
76.998
76.360
74.266
58.247
55.486
45.659
MeO
NH
OH
Br
ppm
180
160
140
120
100
80
60
40
20

^{1}H NMR of 6a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7.40092
7.37553
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7.25396
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7.17652
6.98140
6.94525
6.90685
6.81717
6.80381
6.78517
6.77203
6.74336
6.69783
5.23598
5.22757
5.17947
5.17111
4.99336
4.96416
4.93975
4.91095
3.76965
3.74991
2.59754
2.58794
2.56846
2.55905
2.04765
2.03694
1.99225
1.98100
O
Ph
NH-Ph-4-OMe
Integral
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4.9014
0.8137
1.9684
4.1393
0.9565
1.0000
3.0444
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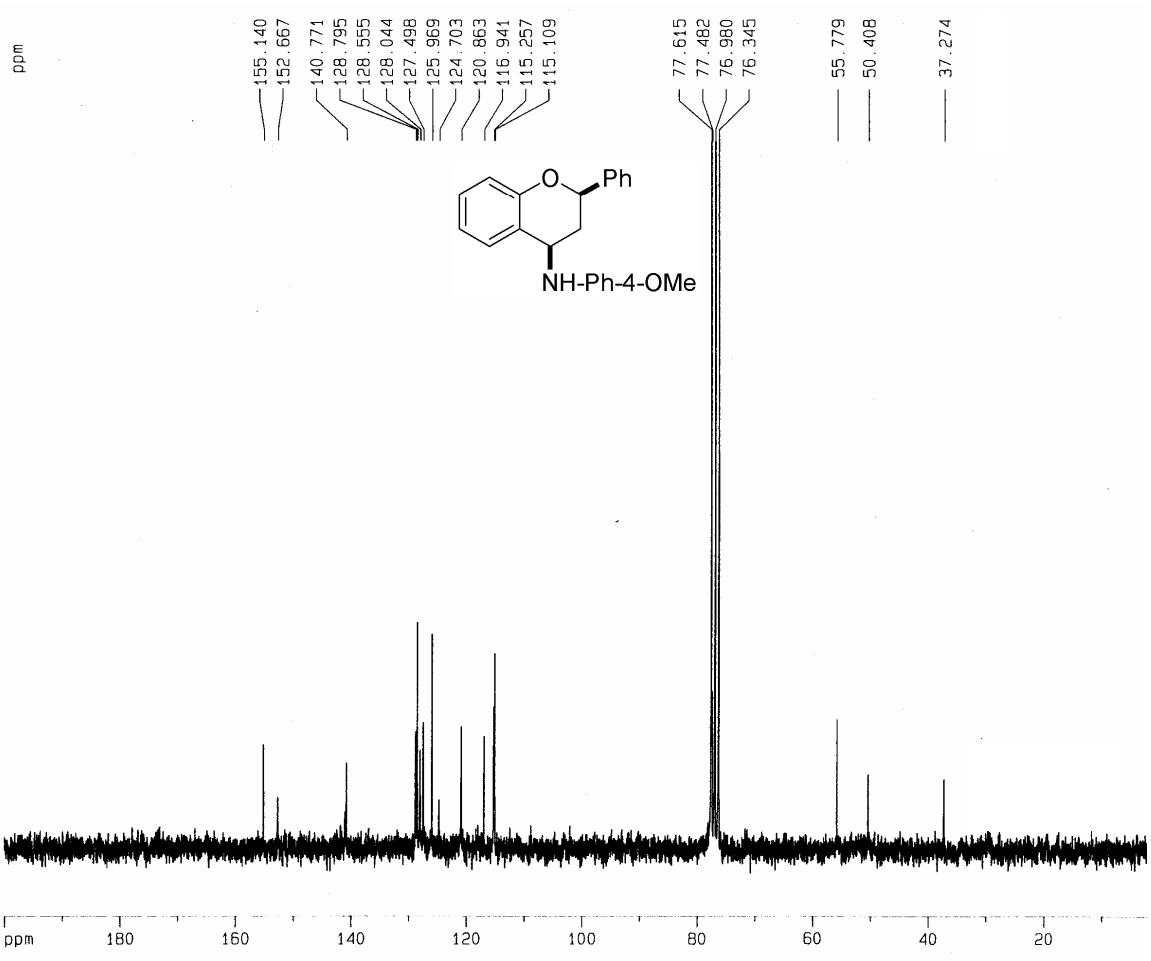
1.2811
ppm
9
8
7
6
5
4
3
2
1

^{13}C NMR of 6a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128.795
128.555
128.044
127.498
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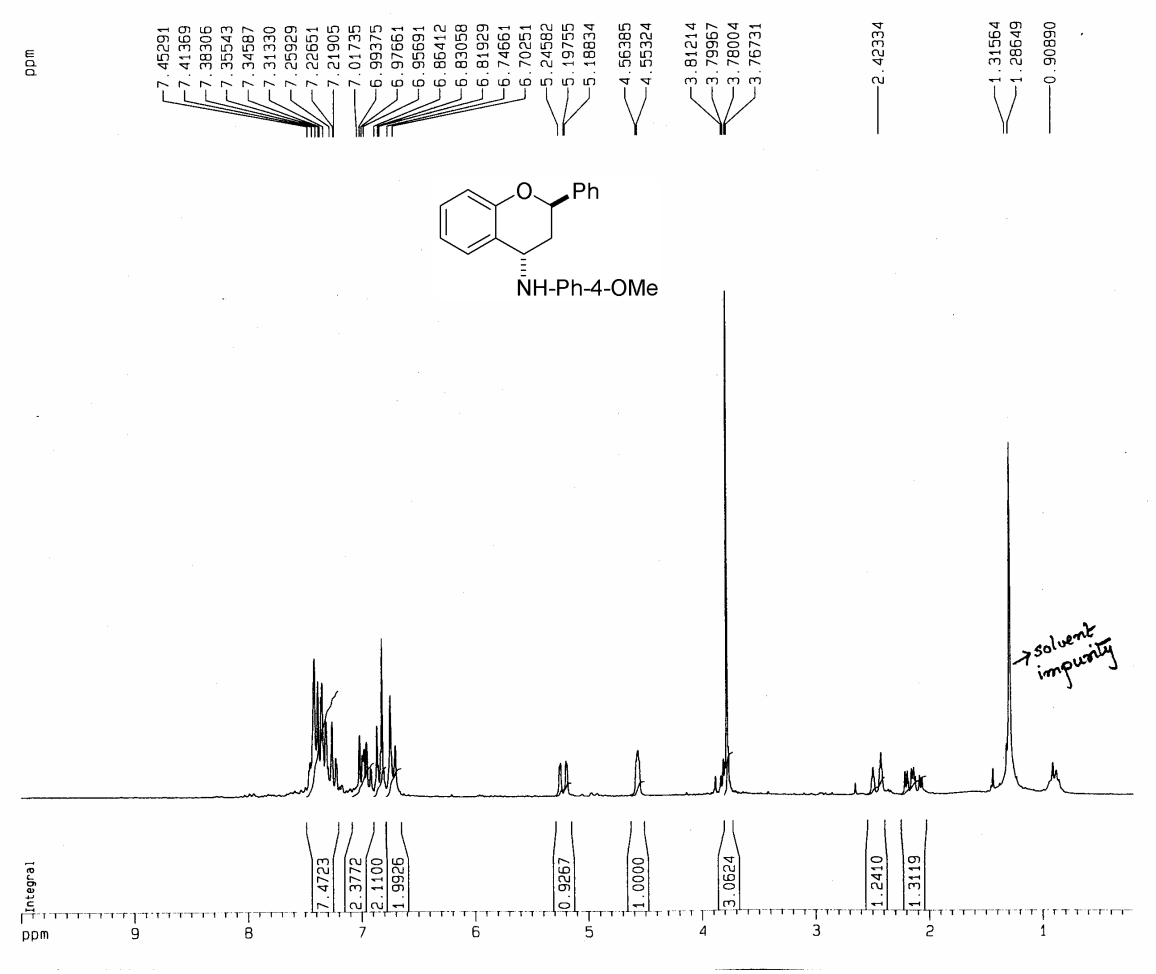
124.703
120.863
116.941
115.257
115.109
77.615
77.482
76.980
76.345
55.779
50.408
37.274
O
Ph
NH-Ph-4-OMe
ppm
180
160
140
120
100
80
60
40
20

^{1}H NMR of 6b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7.22651
7.21905
7.01735
6.99375
6.97661
6.95691
6.86412
6.83058
6.81929
6.74661
6.70251
5.24582
5.19755
5.18834
4.56385
4.55324
3.81214
3.79967
3.78004
3.76731
2.42334
1.31564
1.28649
0.90890
O
Ph
NH-Ph-4-OMe
solvent impurity
Integral
7.4723
2.3772
2.1100
1.9926
0.9267
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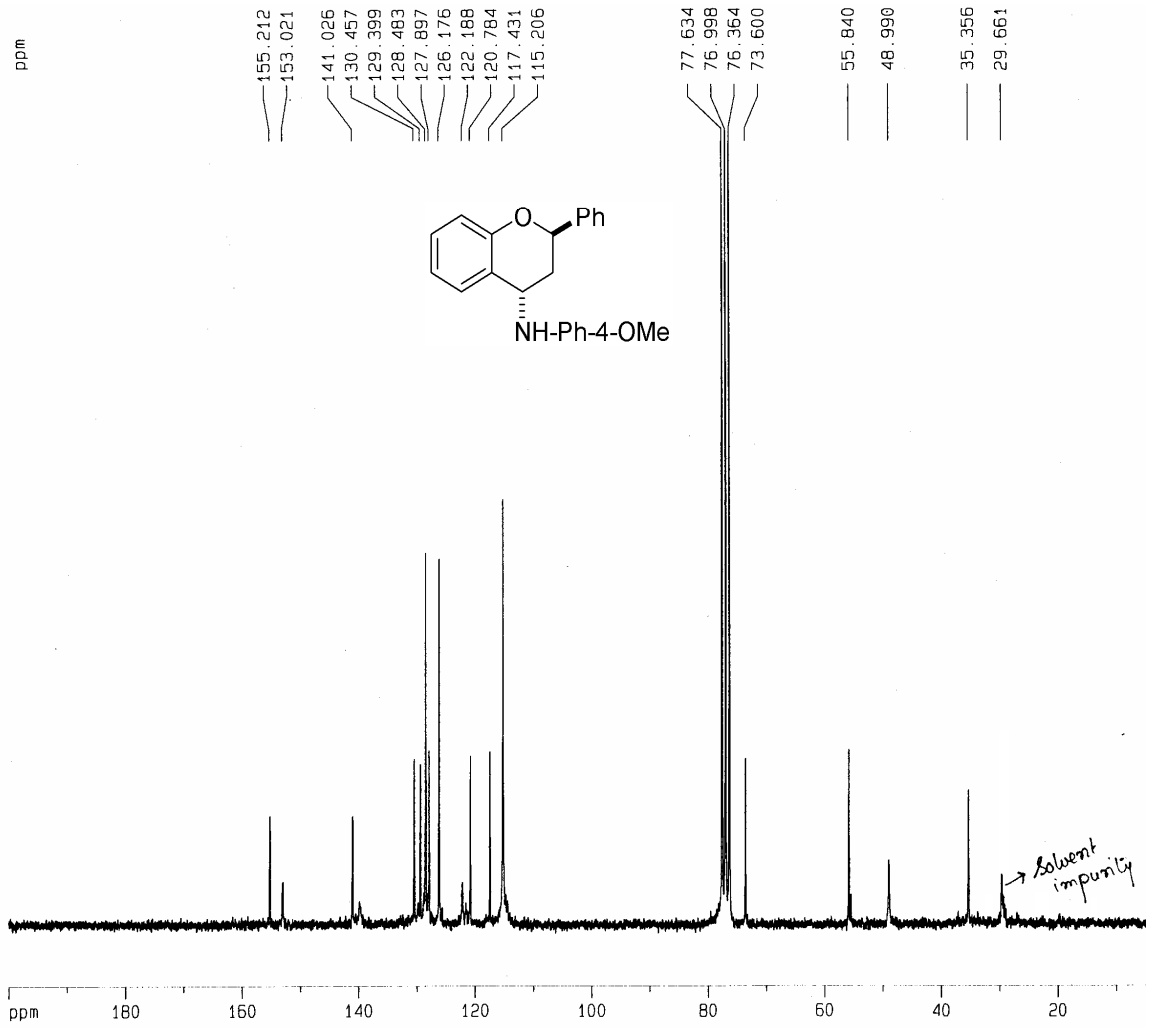
3.0624
1.2410
1.3119
ppm
9
8
7
6
5
4
3
2
1

^{13}C NMR of 6b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153.021
141.026
130.457
129.399
128.483
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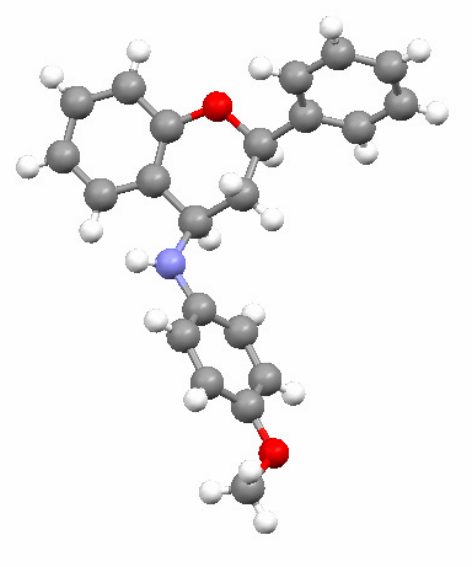
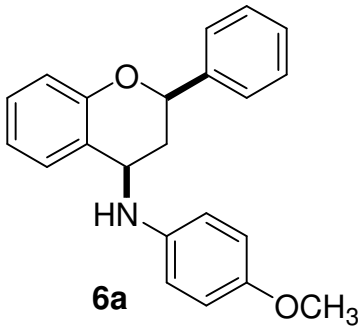
126.176
122.188
120.784
117.431
115.206
77.634
76.998
76.364
73.600
55.840
48.990
35.356
29.661
O
Ph
NH-Ph-4-OMe
Solvent impurity
ppm
180
160
140
120
100
80
60
40
20

(II) Crystal Structure of 6a





Crystal data and structure refinement for **6a**.

Identification code	barc025
Empirical formula	C22 H21 N O2
Formula weight	331.40
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Orthorhombic, P c a b
Unit cell dimensions	a = 8.5808(7) A alpha = 90 deg. b = 46.211(4) A beta = 90 deg. c = 8.7745(8) A gamma = 90 deg.
Volume	3479.3(5) A^3
Z, Calculated density	8, 1.265 Mg/m^3
Absorption coefficient	0.081 mm^-1
F(000)	1408

Crystal size	0.32 x 0.28 x 0.23 mm
Theta range for data collection	3.35 to 25.00 deg.
Limiting indices	-10<=h<=10, -54<=k<=54, -8<=l<=10
Reflections collected / unique	24296 / 3063 [R(int) = 0.0859]
Completeness to theta = 25.00	99.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9817 and 0.9747
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3063 / 0 / 227
Goodness-of-fit on F^2	0.862
Final R indices [I>2sigma(I)]	R1 = 0.0497, wR2 = 0.0941
R indices (all data)	R1 = 0.1316, wR2 = 0.1117
Largest diff. peak and hole	0.251 and -0.235 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for barc025.U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	91(2)	1742(1)	6505(2)	65(1)
O(2)	413(2)	187(1)	12771(2)	80(1)
N(1)	-428(2)	867(1)	7609(2)	59(1)
C(1)	-65(3)	1531(1)	5416(3)	54(1)
C(2)	-406(3)	1630(1)	3961(4)	68(1)
C(3)	-595(3)	1440(1)	2787(4)	73(1)
C(4)	-439(3)	1147(1)	3048(3)	70(1)
C(5)	-106(3)	1049(1)	4497(3)	60(1)
C(6)	81(2)	1238(1)	5711(3)	49(1)
C(7)	421(3)	1134(1)	7308(3)	51(1)
C(8)	50(3)	1371(1)	8452(3)	55(1)
C(9)	704(3)	1658(1)	7952(3)	56(1)
C(10)	350(3)	1899(1)	9034(3)	53(1)
C(11)	1342(3)	1967(1)	10206(3)	67(1)
C(12)	988(4)	2176(1)	11271(4)	80(1)
C(13)	-374(4)	2323(1)	11152(4)	90(1)
C(14)	-1381(4)	2264(1)	9976(4)	98(1)
C(15)	-1024(3)	2053(1)	8924(4)	78(1)
C(16)	-234(3)	713(1)	8967(3)	47(1)
C(17)	-1436(3)	542(1)	9491(3)	54(1)
C(18)	-1273(3)	365(1)	10744(3)	56(1)
C(19)	129(3)	356(1)	11512(3)	55(1)
C(20)	1316(3)	532(1)	11030(3)	58(1)
C(21)	1149(3)	707(1)	9774(3)	55(1)
C(22)	-638(3)	-46(1)	13041(3)	72(1)

Table 3. Bond lengths [A] and angles [deg] for barc025.

O(1)-C(1)	1.375(3)
O(1)-C(9)	1.428(3)
O(2)-C(19)	1.374(3)
O(2)-C(22)	1.424(3)
N(1)-C(16)	1.399(3)
N(1)-C(7)	1.456(3)
N(1)-H(1)	0.8600
C(1)-C(6)	1.385(3)
C(1)-C(2)	1.388(3)
C(2)-C(3)	1.365(4)
C(2)-H(2)	0.9300
C(3)-C(4)	1.380(4)
C(3)-H(3)	0.9300
C(4)-C(5)	1.380(3)
C(4)-H(4)	0.9300
C(5)-C(6)	1.386(3)
C(5)-H(5)	0.9300
C(6)-C(7)	1.509(3)
C(7)-C(8)	1.520(3)
C(7)-H(7)	0.9800
C(8)-C(9)	1.507(3)
C(8)-H(8A)	0.9700
C(8)-H(8B)	0.9700
C(9)-C(10)	1.492(3)
C(9)-H(9)	0.9800
C(10)-C(11)	1.372(3)
C(10)-C(15)	1.381(3)
C(11)-C(12)	1.378(4)
C(11)-H(11)	0.9300
C(12)-C(13)	1.356(4)
C(12)-H(12)	0.9300
C(13)-C(14)	1.373(4)
C(13)-H(13)	0.9300
C(14)-C(15)	1.376(4)
C(14)-H(14)	0.9300
C(15)-H(15)	0.9300
C(16)-C(17)	1.377(3)
C(16)-C(21)	1.382(3)
C(17)-C(18)	1.376(3)
C(17)-H(17)	0.9300
C(18)-C(19)	1.379(3)
C(18)-H(18)	0.9300

C(19)-C(20)	1.371(3)
C(20)-C(21)	1.374(3)
C(20)-H(20)	0.9300
C(21)-H(21)	0.9300
C(22)-H(22A)	0.9600
C(22)-H(22B)	0.9600
C(22)-H(22C)	0.9600

C(1)-O(1)-C(9)	117.4(2)
C(19)-O(2)-C(22)	116.8(2)
C(16)-N(1)-C(7)	121.8(2)
C(16)-N(1)-H(1)	119.1
C(7)-N(1)-H(1)	119.1
O(1)-C(1)-C(6)	123.9(2)
O(1)-C(1)-C(2)	115.1(3)
C(6)-C(1)-C(2)	121.1(3)
C(3)-C(2)-C(1)	120.3(3)
C(3)-C(2)-H(2)	119.8
C(1)-C(2)-H(2)	119.8
C(2)-C(3)-C(4)	119.7(3)
C(2)-C(3)-H(3)	120.1
C(4)-C(3)-H(3)	120.1
C(5)-C(4)-C(3)	119.7(3)
C(5)-C(4)-H(4)	120.2
C(3)-C(4)-H(4)	120.2
C(4)-C(5)-C(6)	121.7(3)
C(4)-C(5)-H(5)	119.1
C(6)-C(5)-H(5)	119.1
C(1)-C(6)-C(5)	117.4(2)
C(1)-C(6)-C(7)	120.1(2)
C(5)-C(6)-C(7)	122.4(2)
N(1)-C(7)-C(6)	109.9(2)
N(1)-C(7)-C(8)	112.7(2)
C(6)-C(7)-C(8)	110.1(2)
N(1)-C(7)-H(7)	108.0
C(6)-C(7)-H(7)	108.0
C(8)-C(7)-H(7)	108.0
C(9)-C(8)-C(7)	111.4(2)
C(9)-C(8)-H(8A)	109.3
C(7)-C(8)-H(8A)	109.3
C(9)-C(8)-H(8B)	109.3
C(7)-C(8)-H(8B)	109.3
H(8A)-C(8)-H(8B)	108.0
O(1)-C(9)-C(10)	106.7(2)
O(1)-C(9)-C(8)	111.1(2)
C(10)-C(9)-C(8)	113.3(2)

O(1)-C(9)-H(9)	108.5
C(10)-C(9)-H(9)	108.5
C(8)-C(9)-H(9)	108.5
C(11)-C(10)-C(15)	117.6(3)
C(11)-C(10)-C(9)	121.4(2)
C(15)-C(10)-C(9)	120.9(2)
C(10)-C(11)-C(12)	122.2(3)
C(10)-C(11)-H(11)	118.9
C(12)-C(11)-H(11)	118.9
C(13)-C(12)-C(11)	119.3(3)
C(13)-C(12)-H(12)	120.4
C(11)-C(12)-H(12)	120.4
C(12)-C(13)-C(14)	120.0(3)
C(12)-C(13)-H(13)	120.0
C(14)-C(13)-H(13)	120.0
C(13)-C(14)-C(15)	120.4(3)
C(13)-C(14)-H(14)	119.8
C(15)-C(14)-H(14)	119.8
C(14)-C(15)-C(10)	120.5(3)
C(14)-C(15)-H(15)	119.7
C(10)-C(15)-H(15)	119.7
C(17)-C(16)-C(21)	117.4(2)
C(17)-C(16)-N(1)	119.2(2)
C(21)-C(16)-N(1)	123.3(2)
C(18)-C(17)-C(16)	122.0(2)
C(18)-C(17)-H(17)	119.0
C(16)-C(17)-H(17)	119.0
C(17)-C(18)-C(19)	119.9(2)
C(17)-C(18)-H(18)	120.1
C(19)-C(18)-H(18)	120.1
C(20)-C(19)-O(2)	117.0(2)
C(20)-C(19)-C(18)	118.5(2)
O(2)-C(19)-C(18)	124.5(2)
C(19)-C(20)-C(21)	121.2(2)
C(19)-C(20)-H(20)	119.4
C(21)-C(20)-H(20)	119.4
C(20)-C(21)-C(16)	120.9(2)
C(20)-C(21)-H(21)	119.6
C(16)-C(21)-H(21)	119.6
O(2)-C(22)-H(22A)	109.5
O(2)-C(22)-H(22B)	109.5
H(22A)-C(22)-H(22B)	109.5
O(2)-C(22)-H(22C)	109.5
H(22A)-C(22)-H(22C)	109.5
H(22B)-C(22)-H(22C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for barc025.

The anisotropic displacement factor exponent takes the form:

-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	89(1)	53(1)	54(1)	11(1)	1(1)	2(1)
O(2)	90(1)	76(2)	74(2)	23(1)	-14(1)	-18(1)
N(1)	68(1)	50(2)	59(2)	6(1)	-15(1)	-10(1)
C(1)	49(2)	59(2)	54(2)	6(2)	10(2)	0(1)
C(2)	72(2)	72(2)	58(2)	15(2)	9(2)	4(2)
C(3)	69(2)	98(3)	53(2)	10(2)	10(2)	2(2)
C(4)	64(2)	93(3)	54(2)	-10(2)	6(2)	2(2)
C(5)	53(2)	62(2)	66(2)	-2(2)	9(2)	4(1)
C(6)	43(2)	52(2)	51(2)	3(2)	6(1)	6(1)
C(7)	46(1)	45(2)	62(2)	4(2)	1(1)	1(1)
C(8)	58(2)	54(2)	54(2)	15(2)	3(1)	2(1)
C(9)	56(2)	51(2)	60(2)	8(2)	1(1)	1(1)
C(10)	55(2)	44(2)	59(2)	6(1)	2(2)	-5(1)
C(11)	72(2)	61(2)	69(2)	7(2)	-6(2)	-1(2)
C(12)	99(3)	73(2)	67(2)	5(2)	-13(2)	-20(2)
C(13)	113(3)	71(2)	85(3)	-15(2)	9(2)	-11(2)
C(14)	88(2)	97(3)	110(3)	-31(2)	-2(2)	25(2)
C(15)	65(2)	85(2)	84(2)	-17(2)	-3(2)	9(2)
C(16)	48(2)	38(2)	54(2)	2(1)	0(1)	2(1)
C(17)	50(2)	56(2)	55(2)	-3(2)	-8(1)	-2(1)
C(18)	56(2)	57(2)	56(2)	1(2)	7(2)	-9(1)
C(19)	65(2)	45(2)	54(2)	9(2)	-6(2)	-2(2)
C(20)	55(2)	48(2)	72(2)	4(2)	-16(2)	-2(1)
C(21)	47(2)	45(2)	74(2)	6(2)	-2(2)	-5(1)
C(22)	87(2)	54(2)	76(2)	15(2)	8(2)	-6(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for barc025.

	x	y	z	U(eq)
H(1)	-1066	803	6934	71
H(2)	-505	1828	3786	81
H(3)	-829	1507	1815	88
H(4)	-558	1016	2251	84
H(5)	-3	851	4664	72
H(7)	1537	1092	7376	61
H(8A)	-1071	1387	8565	66
H(8B)	484	1320	9436	66
H(9)	1838	1640	7862	67
H(11)	2285	1869	10284	80
H(12)	1676	2216	12062	95
H(13)	-627	2465	11866	107
H(14)	-2309	2367	9891	118
H(15)	-1714	2014	8132	94
H(17)	-2386	546	8983	64
H(18)	-2106	253	11073	67
H(20)	2253	533	11564	70
H(21)	1976	822	9462	66
H(22A)	-1621	30	13379	109
H(22B)	-216	-171	13810	109
H(22C)	-786	-153	12116	109

Table 6. Torsion angles [deg] for barc025.

C(9)-O(1)-C(1)-C(6)	11.4(3)
C(9)-O(1)-C(1)-C(2)	-169.3(2)
O(1)-C(1)-C(2)-C(3)	-179.7(2)
C(6)-C(1)-C(2)-C(3)	-0.3(4)
C(1)-C(2)-C(3)-C(4)	-0.3(4)
C(2)-C(3)-C(4)-C(5)	0.5(4)
C(3)-C(4)-C(5)-C(6)	-0.1(4)
O(1)-C(1)-C(6)-C(5)	-180.0(2)
C(2)-C(1)-C(6)-C(5)	0.8(4)
O(1)-C(1)-C(6)-C(7)	0.5(3)
C(2)-C(1)-C(6)-C(7)	-178.8(2)
C(4)-C(5)-C(6)-C(1)	-0.6(4)
C(4)-C(5)-C(6)-C(7)	179.0(2)
C(16)-N(1)-C(7)-C(6)	175.2(2)
C(16)-N(1)-C(7)-C(8)	-61.6(3)
C(1)-C(6)-C(7)-N(1)	142.6(2)
C(5)-C(6)-C(7)-N(1)	-37.0(3)
C(1)-C(6)-C(7)-C(8)	17.8(3)
C(5)-C(6)-C(7)-C(8)	-161.7(2)
N(1)-C(7)-C(8)-C(9)	-169.5(2)
C(6)-C(7)-C(8)-C(9)	-46.3(3)
C(1)-O(1)-C(9)-C(10)	-164.66(19)
C(1)-O(1)-C(9)-C(8)	-40.7(3)
C(7)-C(8)-C(9)-O(1)	59.1(3)
C(7)-C(8)-C(9)-C(10)	179.3(2)
O(1)-C(9)-C(10)-C(11)	-146.6(2)
C(8)-C(9)-C(10)-C(11)	90.8(3)
O(1)-C(9)-C(10)-C(15)	35.9(3)
C(8)-C(9)-C(10)-C(15)	-86.7(3)
C(15)-C(10)-C(11)-C(12)	1.6(4)
C(9)-C(10)-C(11)-C(12)	-175.9(2)
C(10)-C(11)-C(12)-C(13)	-1.0(4)
C(11)-C(12)-C(13)-C(14)	-0.2(5)
C(12)-C(13)-C(14)-C(15)	0.7(5)
C(13)-C(14)-C(15)-C(10)	0.0(5)
C(11)-C(10)-C(15)-C(14)	-1.1(4)
C(9)-C(10)-C(15)-C(14)	176.4(3)
C(7)-N(1)-C(16)-C(17)	153.3(2)
C(7)-N(1)-C(16)-C(21)	-31.1(3)
C(21)-C(16)-C(17)-C(18)	-1.6(4)
N(1)-C(16)-C(17)-C(18)	174.3(2)
C(16)-C(17)-C(18)-C(19)	-0.1(4)

C(22)-O(2)-C(19)-C(20)	-164.5(2)
C(22)-O(2)-C(19)-C(18)	17.7(4)
C(17)-C(18)-C(19)-C(20)	2.3(4)
C(17)-C(18)-C(19)-O(2)	-179.9(2)
O(2)-C(19)-C(20)-C(21)	179.4(2)
C(18)-C(19)-C(20)-C(21)	-2.6(4)
C(19)-C(20)-C(21)-C(16)	0.9(4)
C(17)-C(16)-C(21)-C(20)	1.3(4)
N(1)-C(16)-C(21)-C(20)	-174.4(2)

Symmetry transformations used to generate equivalent atoms: