Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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A Domino Synthesis of Benzoquinolinamide in the Presence of Iodine

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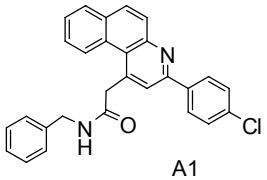
Contents

General information:

All of the reagents were obtained from commercial suppliers and used without further purification. All melting points were uncorrected. The purity was analyzed qualitatively by high performance liquid chromatography (HPLC) on Waters 600E chromatograph, 2487 detector. Mass spectra were taken on an Finnigan TSQ Quantum－LC/MS/MS instrument in the electrospray ionization (positive) mode. ^{1}H NMR, ^{13}C NMR, DEPT90, COSY, HSQC and HMBC spectra were recorded on a AVANCE 500 and 300 Bruker spectrometer operating at 500 MHz and 125 or 75 MHz in DMSO-*d*6, respectively, and chemical shifts were reported in ppm. Elemental analyses were performed on a Yanagimoto MT3CHN recorder.

Synthetic procedure and analytic data for Table 2:

General procedure for the preparation of compounds A1-A4, A6-A9; general procedure exemplified for A1:



A1

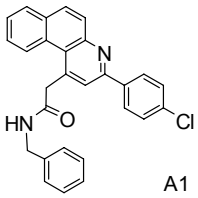
A solution of benzyl amine (2 mmol) and diketene (2 mmol) was stirred in 5 ml of MeCN at room temperature, 2 hours later, naphthalene-2-amine (2 mmol) and 4-chlorobenzaldehyde (2 mmol) were added, the temperature was increased up to 82 ℃, and then iodine (0.4 mmol) was added to the refluxing mixture, yellow precipitates were observed after 3 minutes, the reaction was terminated and cooled to room temperature after the mixture was solidified in 5 minutes. 2 ml of MeCN was added, the precipitates were isolated by filtration, and the solid was washed with a solution of sodium thiosulfate followed by water and EtOH. The yellow product N-Benzyl-2-(3-(4-chlorophenyl)benzo[*f*]quinolin-1-yl)acetamide A1 was obtained after drying in 72% (0.63g), the purity of the corresponding product was high up to 98% based on the analysis of HPLC.

The procedure for the synthesis of A1 under the protection of N_2:

A solution of benzyl amine (2 mmol) and diketene (2 mmol) was stirred in 5 ml of MeCN at room temperature for 2 hours, under dry N_2, the temperature was increased, and naphthalene-2-amine (2 mmol), 4-chlorobenzaldehyde (2 mmol) and iodine (0.4 mmol) were added, after the reaction mixture was solidified, the temperature was then decreased to room temperature while the dry N_2 was continual. The operation was the same as above, and the product A1 was obtained in 72%.

Characterization Data for compounds A1-A4, A6-A9:

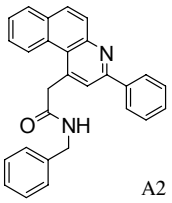
N-Benzyl-2-(3-(4-chlorophenyl)benzo[*f*]quinolin-1-yl)acetamide (A1):



A1

Yellow powder (yield 72%), 98% pure based on the analysis of HPLC. Mp 248-250℃; ^{1}H NMR (500MHz, DMSO-*d*6) δ 4.35-4.36 (d, CH_2, *J*=6.00Hz, 2H), 4.60 (s, CH_2, 2H), 7.24-7.30 (m, aromatic, 5H), 7.62-7.74 (m, aromatic, 4H), 7.99-8.01 (d, aromatic, *J*=4.00Hz, 1H), 8.09-8.14 (m, aromatic, 2H), 8.24 (s, CH, 1H), 8.32-8.34 (m, aromatic, 2H), 8.69-8.71 (d, aromatic, *J*=5.60Hz, 1H), 8.82 (s, NH, 1H); ^{13}C NMR (125MHz, DMSO-*d*6) δ 42.93, 44.92, 124.69, 125.43, 126.02, 126.35, 126.74, 127.31, 127.5, 127.79,128.03, 128.13, 128.74, 129.18, 129.29, 129.61, 129.90, 129.93, 130.15, 132.81, 133.79, 134.76, 136.02, 139.75, 146.78, 147.60, 151.86, 169.07; MS (ESI^+) *m*/*z* 437(M + H); Anal. Calcd for $C_{28}H_{21}ClN_2O$: C, 76.97; H, 4.84; N, 6.41. Found: C, 76.91; H, 4.87; N, 6.50.

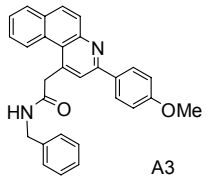
N-Benzyl-2-(3-phenylbenzo[*f*]quinolin-1-yl)acetamide A2:



A2

Light yellow floc (yield 67%), 99% pure based on the analysis of HPLC. Mp 208-210℃; ^{1}H NMR (500MHz, DMSO-*d*6) δ 4.36-4.39 (m, CH_2, 2H), 4.55 (s, CH_2, 2H), 7.23-7.31 (m, aromatic, 5H), 7.55-7.58 (m, aromatic, 1H), 7.61-7.67 (m, aromatic, 3H), 7.73-7.76 (m, aromatic, 1H), 8.04-8.06 (d, aromatic, *J*=9.00Hz, 1H), 8.12-8.14 (m, aromatic, *J*=6.00Hz, 1H), 8.18-8.20 (d, aromatic, *J*=9.00Hz, 1H), 8.27 (s, aromatic, 2H), 8.29 (s, CH, 1H), 8.85-8.86 (d, aromatic, *J*=5.50Hz, 2H), 8.87 (s, NH, 1H); ^{13}C NMR (75MHz, DMSO-*d*6) δ 42.40, 44.41, 124.26, 124.75, 126.79, 126.96, 126.27, 127.49, 127.53, 127.72, 128.21, 128.79, 129.03, 129.36, 130.55, 132.24, 133.22, 139.24, 146.10, 147.70, 152.53, 168.59; MS (ESI^+) *m*/*z* 403(M + H); Anal. Calcd for $C_{28}H_{22}N_2O$: C, 83.56; H, 5.51; N, 6.96. Found: C, 83.60; H, 5.54; N, 6.99.

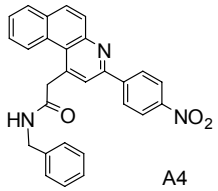
N-Benzyl-2-(3-(4-methoxyphenyl)benzo[*f*]quinolin-1-yl)acetamide A3:



A3

Light yellow powder (yield 61%), 99% pure based on the analysis of HPLC. Mp 244-246℃; ^{1}H NMR (500MHz, DMSO-*d*6) δ 3.92 (s, CH_3, 3H), 4.36-4.37 (d, CH_2, *J*=4.50Hz, 2H), 4.71 (s, CH_2, 2H), 7.26-7.42 (m, aromatic, 7H), 7.73-7.85 (m, aromatic, 2H), 8.19-8.23 (m, aromatic, 4H), 8.41-8.43 (d, aromatic, *J*=7.50Hz, 1H), 8.48 (s, CH, 1H), 8.72-8.73 (d, aromatic, *J*=6.00Hz, 1H), 8.95 (s, NH, 1H); ^{13}C NMR (125MHz, DMSO-*d*6) δ 42.95, 45.14, 56.22, 115.38, 124.93, 125.08, 125.67, 127.25, 127.36, 127.80, 128.64, 128.77, 128.82, 130.30, 130.88, 132.56, 135.70, 139.63, 143.31, 151.10, 162.84, 168.58; MS (ESI^+) *m*/*z* 433(M + H); Anal. Calcd for $C_{29}H_{24}N_2O_2$: C, 80.53; H, 5.59; N, 6.48. Found: C, 80.51; H, 5.47; N, 6.46.

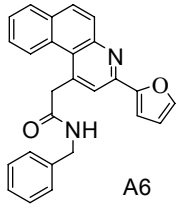
N-Benzyl-2-(3-(4-nitrophenyl)benzo[*f*]quinolin-1-yl)acetamide A4:



A4

Brown yellow powder (yield 50%), 96% pure based on the analysis of HPLC. Mp 239-241℃; ^{1}H NMR (500MHz, DMSO-*d*6) δ 4.35-4.37 (d, CH_2, *J*=5.50 Hz, 2H), 4.55 (s, CH_2, 2H), 7.24-7.31 (m, aromatic, 5H), 7.63-7.78 (m, aromatic, 2H), 8.03-8.20 (m, aromatic, 3H), 8.38 (s, CH, 1H), 8.41-8.42 (d, aromatic, *J*=9.00 Hz, 2H), 8.51-8.53 (s, CH, *J*=8.00Hz, 2H), 8.70-8.71 (d, aromatic, *J*=7.50 Hz, 1H), 8.84-8.87 (m, NH, 1H); ^{13}C NMR (125MHz, DMSO-*d*6) δ 42.89, 44.77, 124.38, 124.61, 125.67, 127.28, 127.43, 127.72, 127.77, 127.82, 128.55, 128.74, 129.26, 129.70, 132.25, 133.11, 139.86, 144.42, 144.58, 148.48, 149.59, 152.18, 169.48; MS (ESI^+) *m*/*z* 448(M + H); Anal. Calcd for $C_{28}H_{21}N_3O_3$: C, 75.15; H, 4.73; N, 9.39. Found: C, 75.09; H, 4.62; N, 9.41.

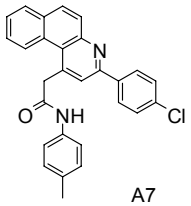
N-Benzyl-2-(3-(furan-2-yl)benzo[*f*]quinolin-1-yl)acetamide A6:



A6

Brown yellow (yield 52%), 97% pure based on the analysis of HPLC. Mp 198-200℃; ^{1}H NMR (500MHz, DMSO-*d*6) δ 4.29-4.35 (d, CH_2, 2H), 4.53 (s, CH_2, 2H), 6.82 (s, CH, 1H), 7.26-7.39 (m, aromatic, 5H), 7.47-7.58 (m, aromatic, 1H), 7.60-7.86 (m, aromatic, 2H), 8.01-8.06 (m, aromatic, 2H), 8.12-8.25 (m, aromatic, 3H), 8.66-8.68 (d, aromatic, *J*=8.00 Hz, 1H), 8.85 (s, NH, 1H); ^{13}C NMR (125MHz, DMSO-*d*6) δ 42.89, 44.80, 113.47, 122.22, 124.81, 127.30, 127.39, 127.72, 127.77, 128.73, 129.60, 129.74, 132.70, 133.11, 139.78, 146.30, 169.24; MS (ESI^+) *m*/*z* 393(M + H); Anal. Calcd for $C_{26}H_{20}N_2O_2$: C, 79.57; H, 5.14; N, 7.14. Found: C, 79.51; H, 5.08; N, 7.19.

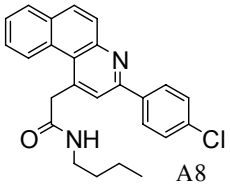
2-(3-(4-Chlorophenyl)benzo[*f*]quinolin-1-yl)-N-*p*-tolylacetamide A7:



A7

Yellow powder (yield 67%), 98% pure based on the analysis of HPLC. Mp 262-265℃; ^{1}H NMR (500MHz, DMSO-*d*6) δ 2.24 (s, CH_3, 3H), 4.77 (s, CH_2, 2H), 7.11-7.12 (d, aromatic, *J*=7.50 Hz, 2H), 7.47-7.48 (d, aromatic, *J*=7.50 Hz, 2H), 7.69-7.71 (d, aromatic, *J*=8.00 Hz, 1H), 7.71-7.75 (m, aromatic, 1H), 8.08-8.10 (d, aromatic, *J*=9.00 Hz, 1H), 7.60-7.86 (m, aromatic, 1H), 8.01-8.06 (m, aromatic, 1H), 8.10-8.13 (m, aromatic, 1H), 8.24-8.26 (d, aromatic, *J*=9.00 Hz, 1H), 8.29-8.30 (d, aromatic, J=8.00 Hz, 2H), 8.43 (s, CH, 1H), 8.76-8.77 (d, aromatic, 1H), 10.49 (s, NH, 1H); ^{13}C NMR (125MHz, DMSO-*d*6) δ 20.90, 45.64, 119.72, 120.02, 120.30, 124.57, 125.40, 126.51, 127.30, 127.96, 128.06, 128.84, 129.39, 129.60, 129.64, 129.84, 129.90, 132.84, 133.04, 133.52, 135.11, 135.89, 136.84, 146.98, 147.16, 152.11, 167.85; MS (ESI^+) *m*/*z* 437(M + H); Anal. Calcd for $C_{28}H_{21}ClN_2O$: C, 76.97; H, 4.84; N, 6.41. Found: C, 76.98; H, 4.79; N, 6.47.

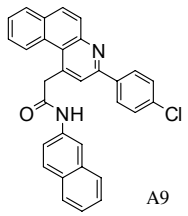
N-Butyl-2-(3-(4-chlorophenyl)benzo[*f*]quinolin-1-yl)acetamide A8:



A8

Light yellow powder (yield 64%), 99% pure based on the analysis of HPLC. Mp 218-215℃; ^{1}H NMR (500MHz, DMSO-*d*6) δ 0.84-0.86 (m, CH_3, 3H), 1.25-1.32 (m, CH_2, 2H), 1.38-1.44 (m, CH_2, 2H), 3.11-3.15 (m, CH_2, 2H), 4.42 (s, CH_2, 2H), 7.61-7.74 (m, aromatic, 4H), 8.00-8.01 (d, aromatic, *J*=9.00 Hz, 1H), 8.09-8.11 (d, aromatic, *J*=7.50 Hz, 1H), 8.13-8.15 (d, aromatic, *J*=9.00 Hz, 1H), 8.23 (s, CH, 1H), 8.29-8.31 (m, NH, 1H), 8.34-8.36 (d, aromatic, *J*=8.50 Hz, 2H), 8.67-8.70 (d, aromatic, *J*=8.50 Hz, 1H); ^{13}C NMR (125MHz, DMSO-*d*6) δ 14.10, 19.99, 31.65, 38.89, 44.80, 123.59, 125.06, 127.22, 127.56, 128.71, 129.00, 129.20, 129.44, 129.55, 129.80, 132.03, 132.83, 135.03, 137.00, 144.76, 149.19, 153.11, 169.32; MS (ESI^+) *m*/*z* 437(M + H); Anal. Calcd for $C_{28}H_{21}ClN_2O$: C, 76.97; H, 4.84; N, 6.41. Found: C, 76.98; H, 4.79; N, 6.47.

2-(3-(4-Chlorophenyl)benzo[f]quinolin-1-yl)-N-(naphthalen-2-yl)acetamide A9:

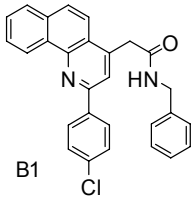


A9

Brown yellow powder (yield 66%), 95% pure based on the analysis of HPLC. Mp 260-263℃; ^{1}H NMR (500MHz, DMSO-*d*6) δ 4.81 (s, CH_2, 2H), 7.40-7.45 (m, aromatic, 2H), 7.64-7.66 (d, aromatic, *J*=8.50 Hz, 1H), 7.70-7.78 (m, aromatic, 5H), 7.84-7.85 (d, aromatic, *J*=7.00 Hz, 1H), 7.89-7.90 (d, aromatic, *J*=8.50 Hz, 1H), 8.09-8.15 (m, aromatic, 2H), 8.24-8.26 (m, aromatic, 2H), 8.34-8.35 (d, aromatic, *J*=7.00 Hz, 2H), 8.43 (s, CH, 1H), 8.79-8.81 (d, aromatic, 1H), 10.80 (s, NH, 1H); ^{13}C NMR (125MHz, DMSO-*d*6) δ 45.73, 116.03, 120.51, 124.37, 126.19, 125.33, 126.93, 127.31, 127.75, 127.93, 128.96, 129.60, 129.70, 129.87, 130.35, 132.89, 133.84, 135.67, 137.01, 152.48, 168.48; MS (ESI^+) *m*/*z* 473(M + H); Anal. Calcd for $C_{31}H_{21}ClN_2O$: C, 78.72; H, 4.48; N, 5.92. Found: C, 78.75; H, 4.51; N, 5.98.

Synthetic procedure and analytic data for Scheme 2:

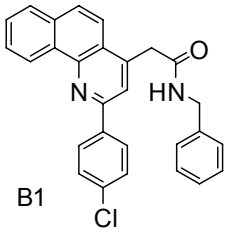
General procedure for the preparation of compounds B1-B2; general procedure exemplified for B1:



A solution of benzyl amine (2 mmol) and diketene (2 mmol) was stirred in 5 ml of MeCN at room temperature, after 2 hours, naphthalene-1-amine (2 mmol) and 4-chlorobenzaldehyde (2 mmol) were added, the temperature was increased up to 82 ℃, and then iodine (0.4 mmol) was added to the refluxing mixture, yellow precipitate was observed after 15 minutes, the reaction was terminated and cooled to room temperature after the mixture was solidified in 45 minutes. 2 ml of MeCN was added, the precipitates were isolated by filtration, and the solid was washed with a solution of sodium thiosulfate followed by water and EtOH. The yellow product N-benzyl-2-(2-(4-chlorophenyl)benzo[*h*]quinolin-4-yl)acetamide B1 was obtained after drying in 36% (0.31g), the purity of the corresponding product was high up to 97% based on the analysis of HPLC.

Characterization Data for compounds B1, B2:

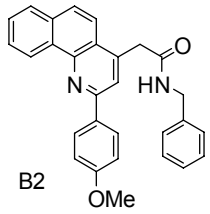
N-Benzyl-2-(2-(4-chlorophenyl)benzo[*h*]quinolin-4-yl)acetamide B1：



Light yellow powder (yield 36%), 91% pure based on the analysis of HPLC. Mp 248-250℃; ^{1}H NMR (500MHz, DMSO-*d*6) δ 4.18 (s, CH_2, 2H), 4.32 (s, CH_2, 2H), 7.27-7.44 (m, aromatic, 5H), 7.67-7.68 (d, aromatic, *J*=7.5Hz, 2H), 7.79 (s, aromatic, *J*=4.00Hz, 2H), 7.97-7.98 (d, aromatic, *J*=7.5Hz, 1H), 8.06-8.15 (m, aromatic, 2H), 8.25 (s, CH, 1H), 8.41-8.43 (d, aromatic, *J*=7.5Hz, 2H), 8.82 (s, NH, 1H), 8.39 (m,

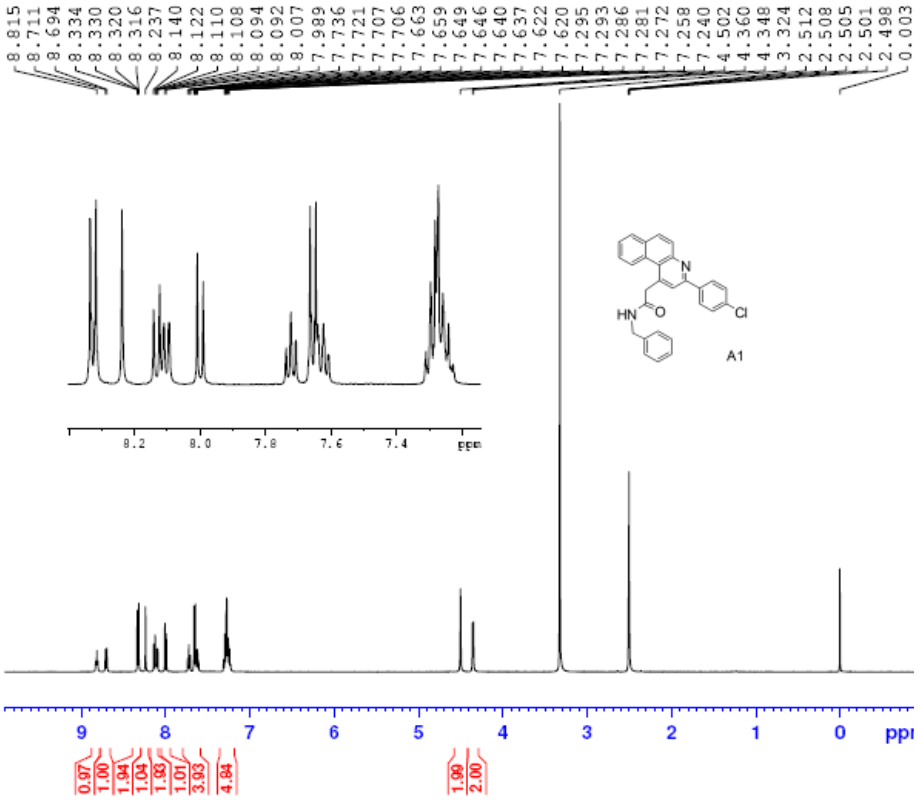
aromatic, 1H); ^{13}C NMR (125MHz, DMSO-*d*6) δ 31.16, 42.85, 121.03, 122.25, 124.84, 124.97, 127.32, 127.64, 127.74, 127.80, 128.39, 128.76, 128.91, 129.18, 129.25, 129.46, 131.59, 133.68, 134.87, 137.98, 139.80, 144.30, 145.79, 153.22, 169.42; MS (ESI$^+$) *m*/*z* 437(M + H); Anal. Calcd for $C_{28}H_{21}ClN_2O$: C, 76.97; H, 4.84; N, 6.41. Found: C, 76.86; H, 4.91; N, 6.43.

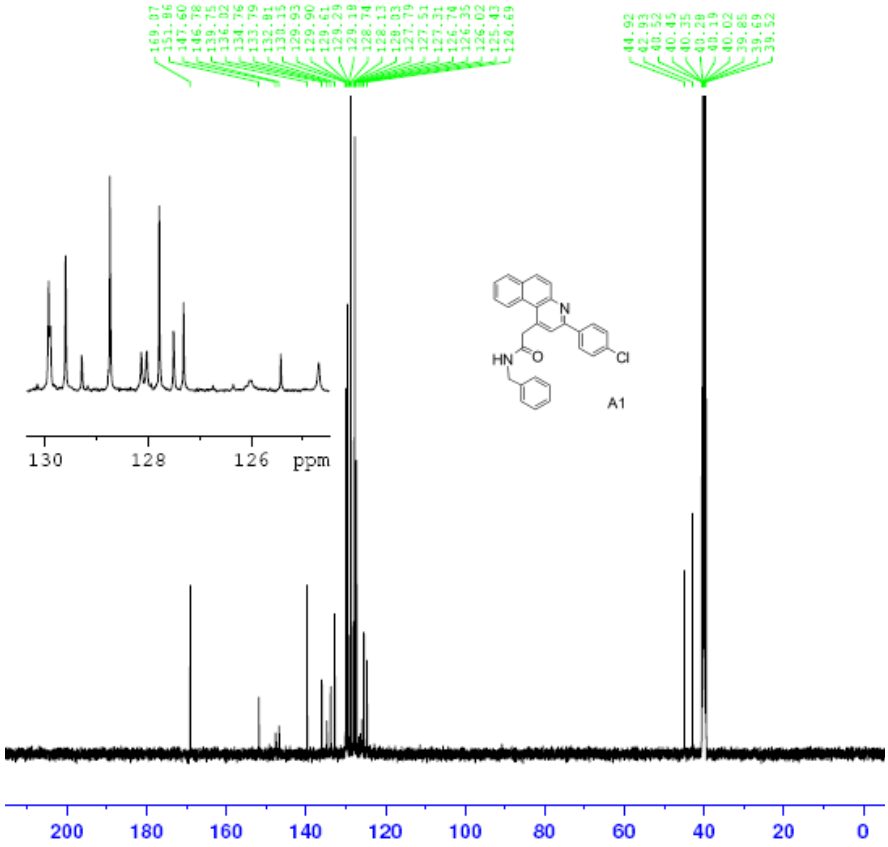
N-Benzyl-2-(2-(4-methoxyphenyl)benzo[*h*]quinolin-4-yl)acetamide B2:

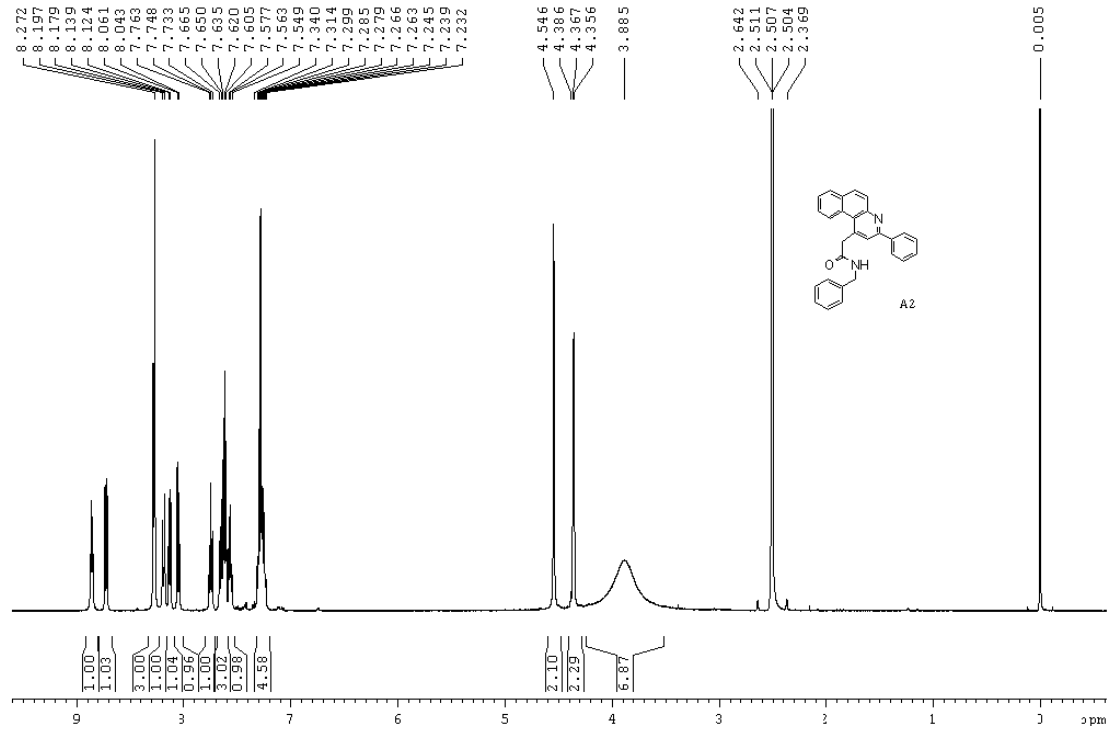


Grey powder (yield 45%), 98% pure based on the analysis of HPLC. Mp 249-251℃; ^{1}H NMR (500MHz, DMSO-*d*6) δ 3.88 (s, CH_3, 3H), 4.16 (s, CH_2, 2H), 4.33-4.34 (d, CH_2, *J*=5.50 Hz, 2H), 7.15-7.17 (d, aromatic, *J*=9.0 Hz, 2H), 7.24-7.30 (m, aromatic, 5H), 7.76-7.79 (m, aromatic, 2H), 7.91-7.93 (d, aromatic, *J*=9.0 Hz, 1H), 8.03-8.08 (m, aromatic, 2H), 8.17 (s, CH, 1H), 8.34-8.36 (d, aromatic, *J*=9.0 Hz, 2H), 8.80 (s, NH, 1H), 9.38-9.39 (d, aromatic, *J*=8.0 Hz, 1H); ^{13}C NMR (125MHz, DMSO-*d*6) δ 42.86, 55.79, 114.81, 120.50, 122.30, 124.32, 124.81, 127.04, 128.29, 128.67, 128.74, 128.89, 131.68, 131.72, 133.69, 139.84, 143.85, 145.79, 154.35, 161.06, 169.50; MS (ESI$^+$) *m*/*z* 433(M + H); Anal. Calcd for $C_{29}H_{24}N_2O_2$: C, 80.53; H, 5.59; N, 6.48. Found: C, 80.51; H, 5.56; N, 6.52.

Copies of ^{1}H, ^{13}C NMR for compounds A1-A4, A6-A9, B1, B2:



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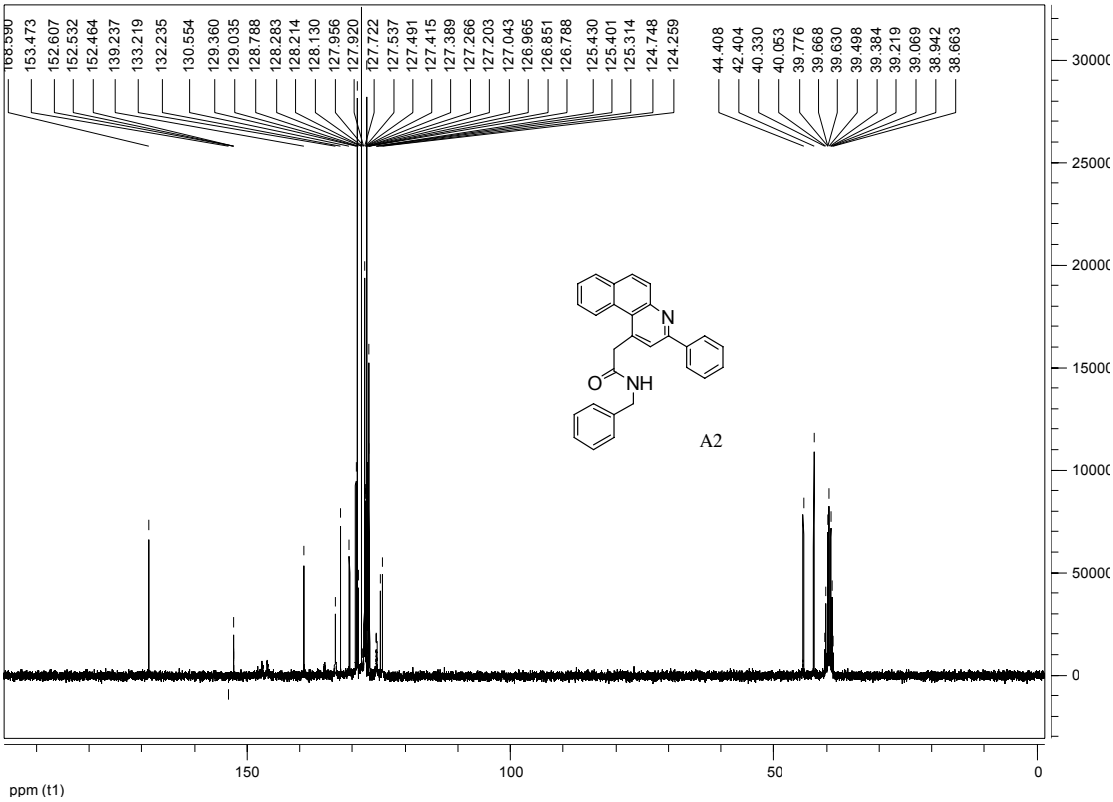
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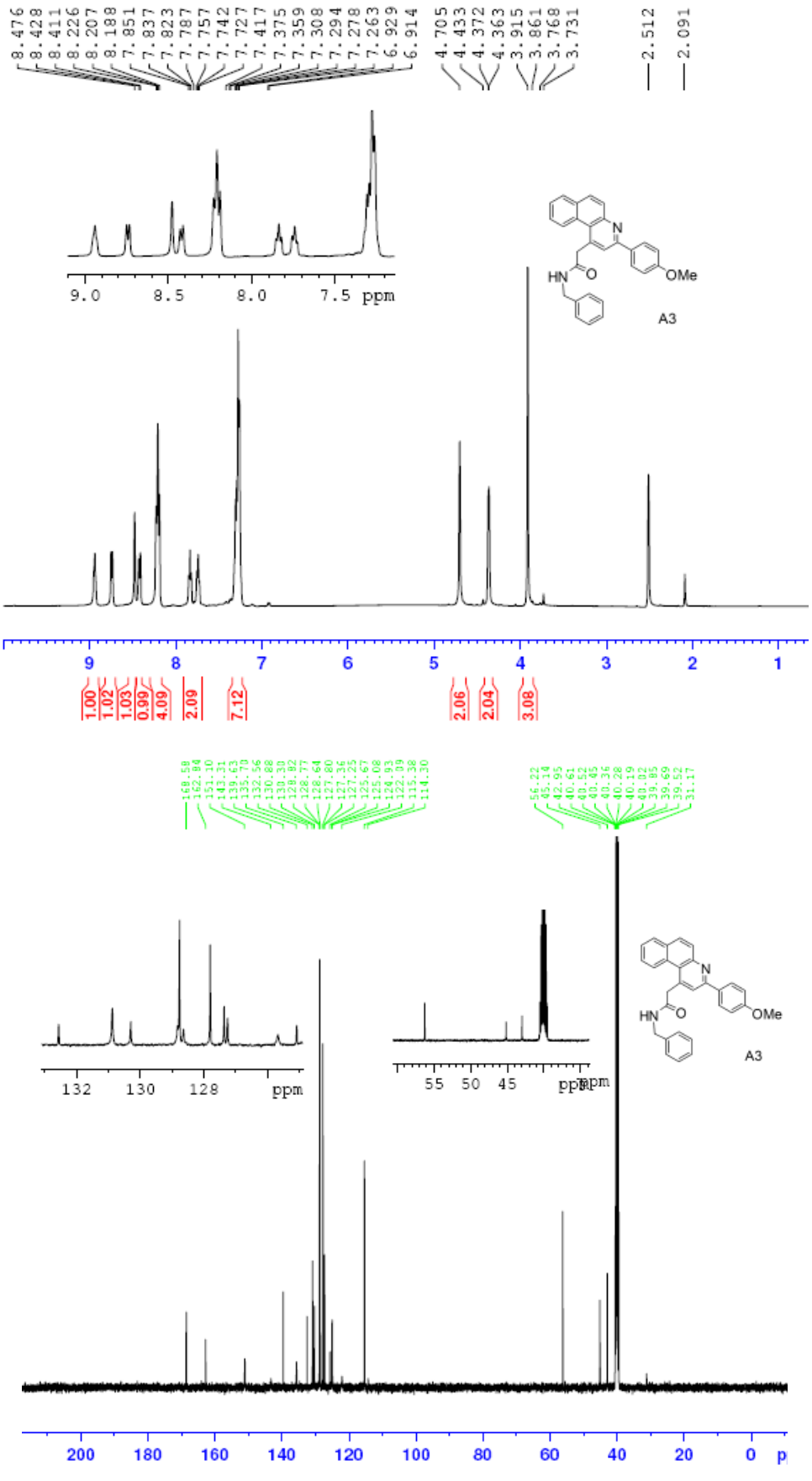
39.668
39.630
39.498
39.384
39.219
39.069
38.942
38.663
A2
ppm (t1)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8.411
8.226
8.207
8.188
7.851
7.837
7.823
7.787
7.757
7.742
7.727
7.417
7.375
7.359
7.308
7.294
7.278
7.263
6.929
6.914
4.705
4.433
4.372
4.363
3.915
3.861
3.768
3.731
2.512
2.091
9.0
8.5
8.0
7.5 ppm
HN
O
N
OMe
A3
9
8
7
6
5
4
3
2
1
1.00
1.02
1.03
0.99
4.09
2.09
7.12
2.06
2.04
3.08
169.58
162.84
151.10
143.31
139.63
135.70
132.56
130.88
130.30
128.82
128.77
128.64
127.80
127.36
127.25
125.67
125.08
124.93
122.09
115.38
114.30
56.22
45.14
42.95
40.61
40.52
40.45
40.36
40.28
40.19
40.02
39.85
39.69
39.52
31.17
132
130
128
ppm
55
50
45
ppm
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O
N
OMe
A3
200
180
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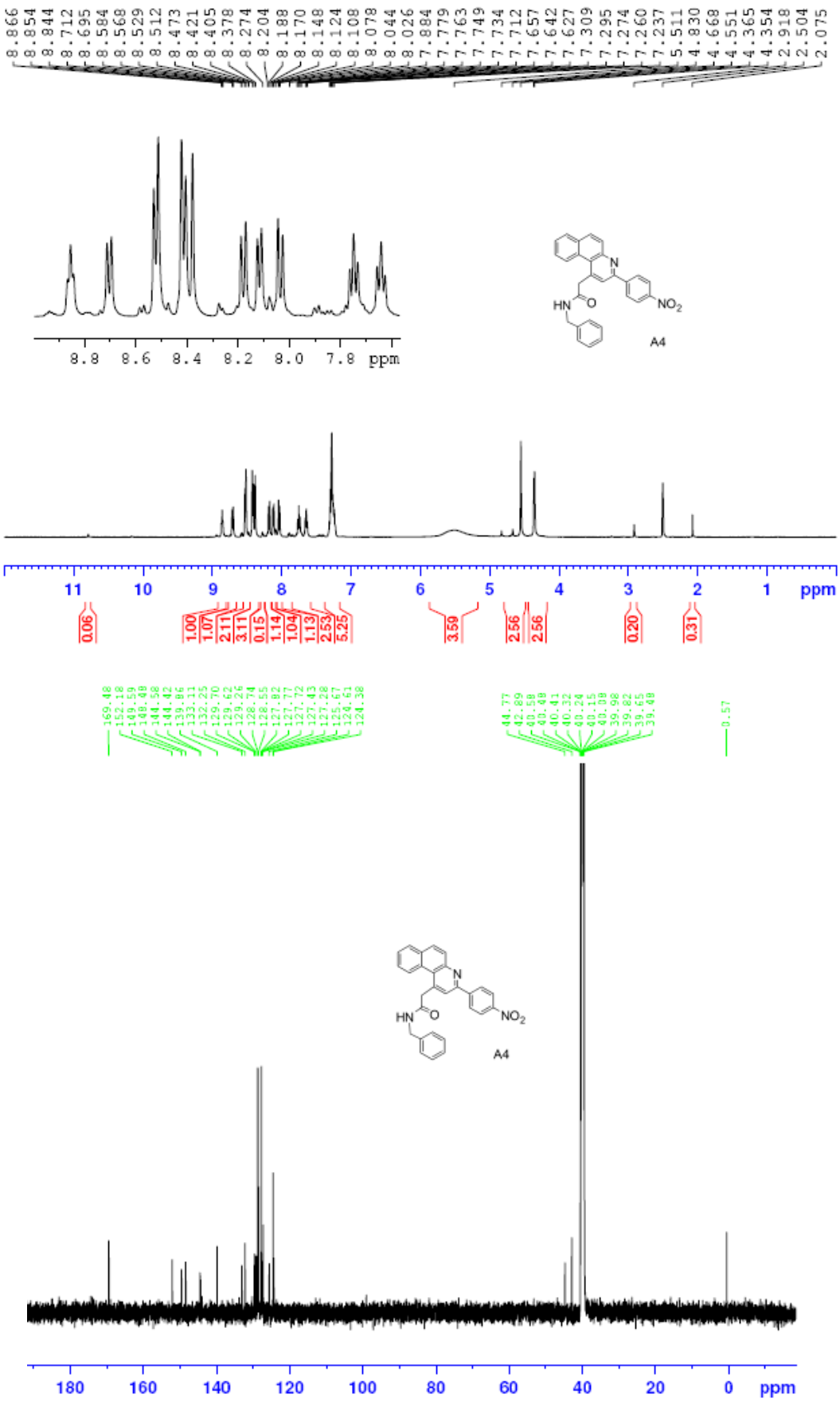
140
120
100
80
60
40
20
0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8.844
8.712
8.695
8.584
8.568
8.529
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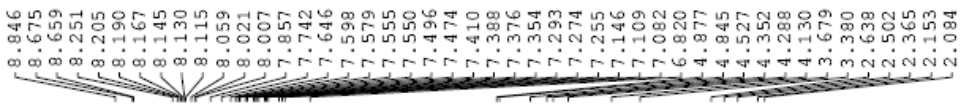
8.473
8.421
8.405
8.378
8.274
8.204
8.188
8.170
8.148
8.124
8.108
8.078
8.044
8.026
7.884
7.779
7.763
7.749
7.734
7.712
7.657
7.642
7.627
7.309
7.295
7.274
7.260
7.237
5.511
4.830
4.668
4.551
4.365
4.354
2.918
2.504
2.075
8.8 8.6 8.4 8.2 8.0 7.8 ppm
HN
O
N
NO2
A4
11 10 9 8 7 6 5 4 3 2 1 ppm
0.06
1.00
1.07
2.11
3.11
0.15
1.14
1.04
1.13
2.53
5.25
3.50
2.56
2.56
0.20
0.31
169.48
152.18
149.59
149.49
144.58
144.42
139.86
133.11
132.25
129.70
129.62
129.26
128.74
128.55
127.82
127.77
127.72
127.43
127.28
125.67
124.61
124.38
44.77
42.89
40.58
40.49
40.41
40.32
40.24
40.15
40.09
39.98
39.82
39.65
39.48
0.57
HN
O
N
NO2
A4
180 160 140 120 100 80 60 40 20 0 ppm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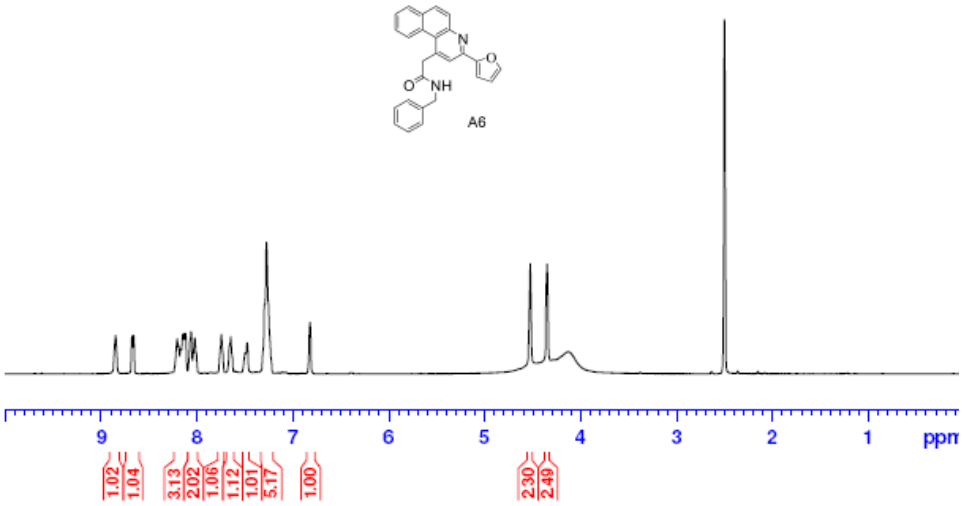
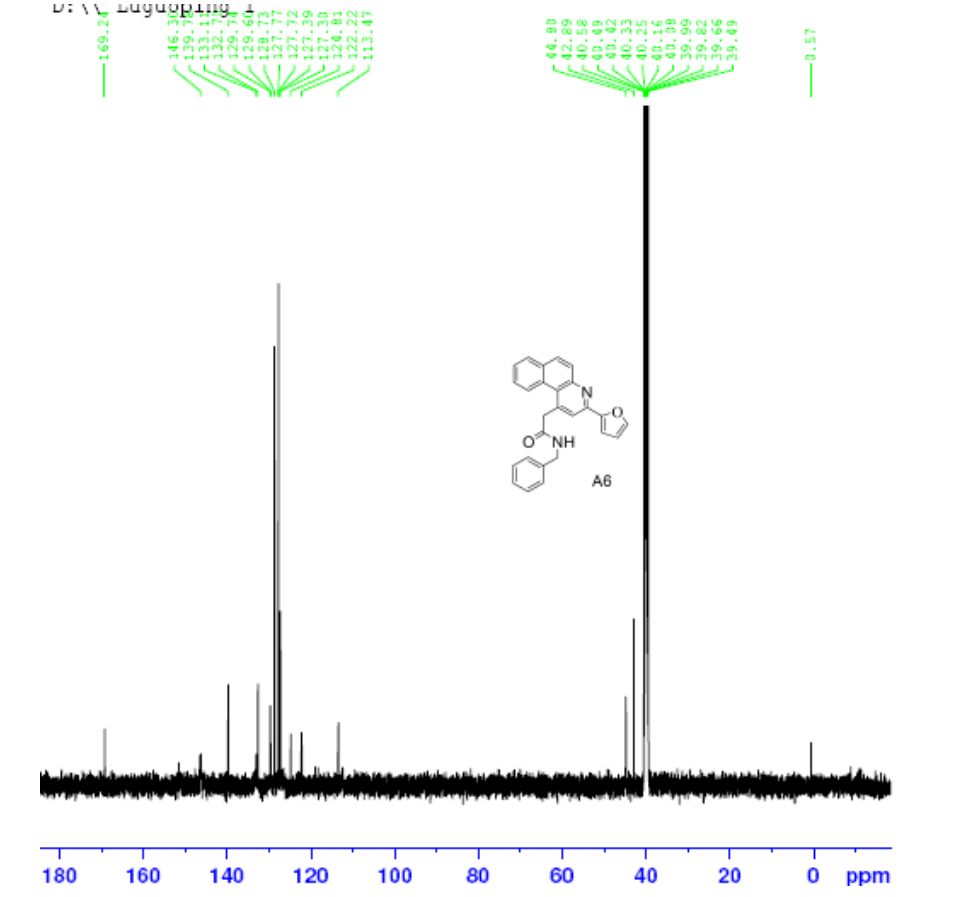
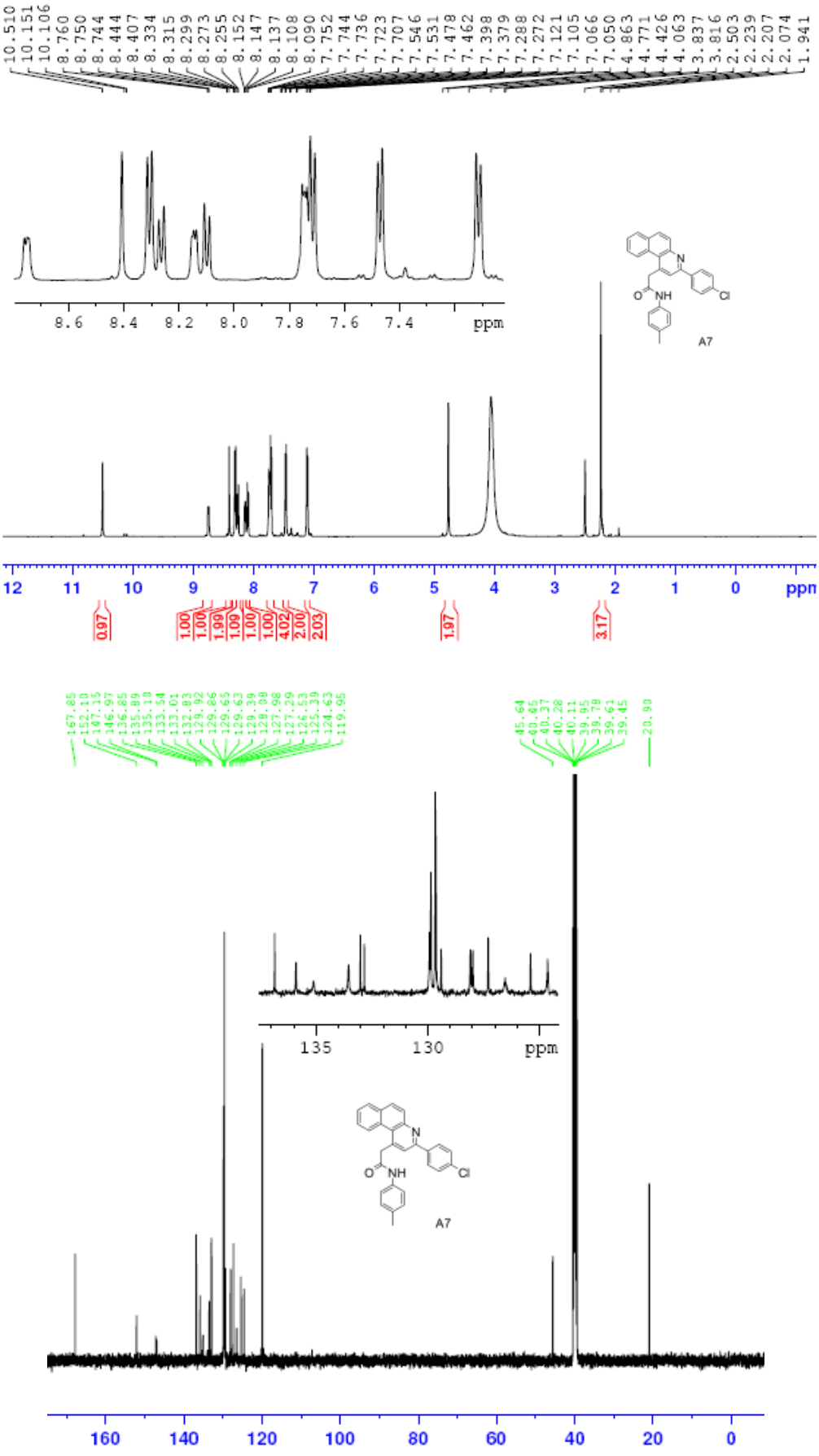
8.675
8.659
8.251
8.205
8.190
8.167
8.145
8.130
8.115
8.059
8.021
8.007
7.857
7.742
7.646
7.598
7.579
7.555
7.550
7.496
7.474
7.410
7.388
7.376
7.354
7.293
7.274
7.255
7.146
7.109
7.082
6.820
4.877
4.845
4.527
4.352
4.288
4.130
3.679
3.380
2.638
2.502
2.365
2.153
2.084


N
O
NH
O
A6
9
8
7
6
5
4
3
2
1
ppm
1.02
1.04
3.13
2.02
1.06
1.12
1.01
5.17
1.00
2.30
2.49


169.24
146.30
139.17
133.11
133.11
132.74
129.74
129.60
128.73
127.77
127.72
127.39
127.30
124.81
122.22
113.47
44.80
42.89
40.58
40.49
40.42
40.33
40.25
40.16
40.08
39.99
39.82
39.66
39.49
0.57
N
O
NH
O
A6
180
160
140
120
100
80
60
40
20
0
ppm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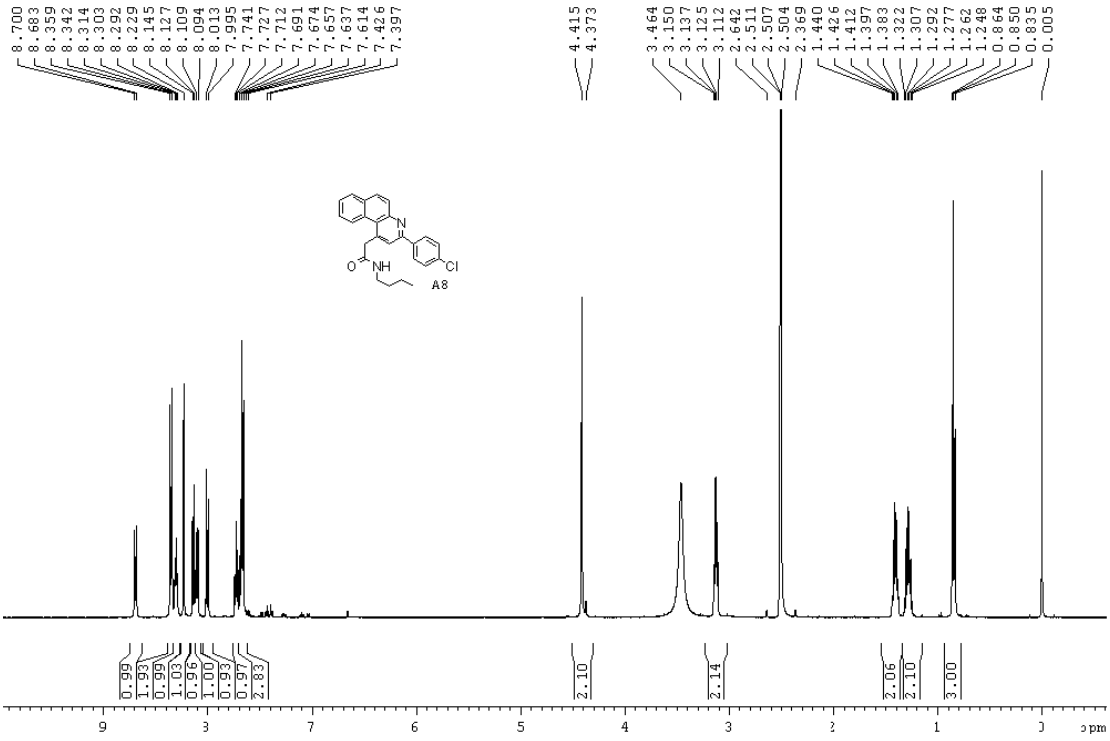
10.151
10.106
8.760
8.750
8.744
8.444
8.407
8.334
8.315
8.299
8.273
8.255
8.152
8.147
8.137
8.108
8.090
7.752
7.744
7.736
7.723
7.707
7.546
7.531
7.478
7.462
7.398
7.379
7.288
7.272
7.121
7.105
7.066
7.050
4.863
4.771
4.426
4.063
3.837
3.816
2.503
2.239
2.207
2.074
1.941
8.6
8.4
8.2
8.0
7.8
7.6
7.4
ppm
N
O
NH
Cl
A7
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.97
1.00
1.00
1.99
1.00
1.00
1.00
4.02
2.00
2.03
1.97
3.17
167.85
152.10
147.15
146.97
136.85
135.89
135.10
133.54
133.01
132.83
129.92
129.86
129.65
129.63
129.39
128.88
127.98
127.29
126.53
125.39
124.63
119.95
45.64
40.45
40.37
40.28
40.11
39.95
39.78
39.61
39.45
20.90
135
130
ppm
N
O
NH
Cl
A7
160
140
120
100
80
60
40
20
0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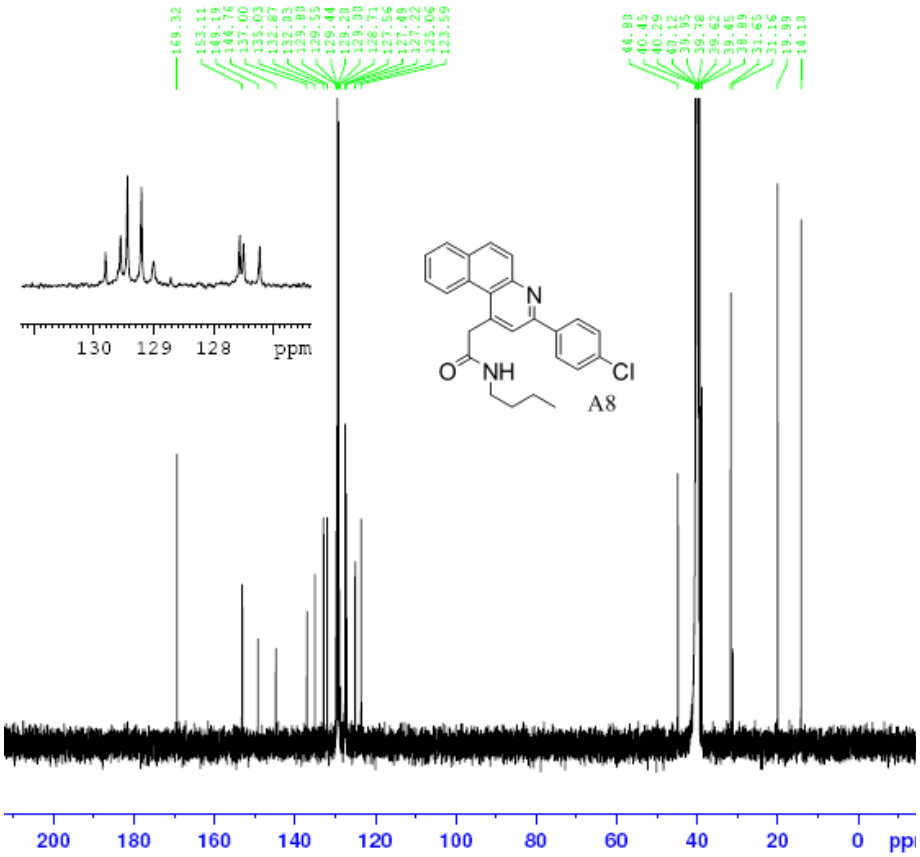
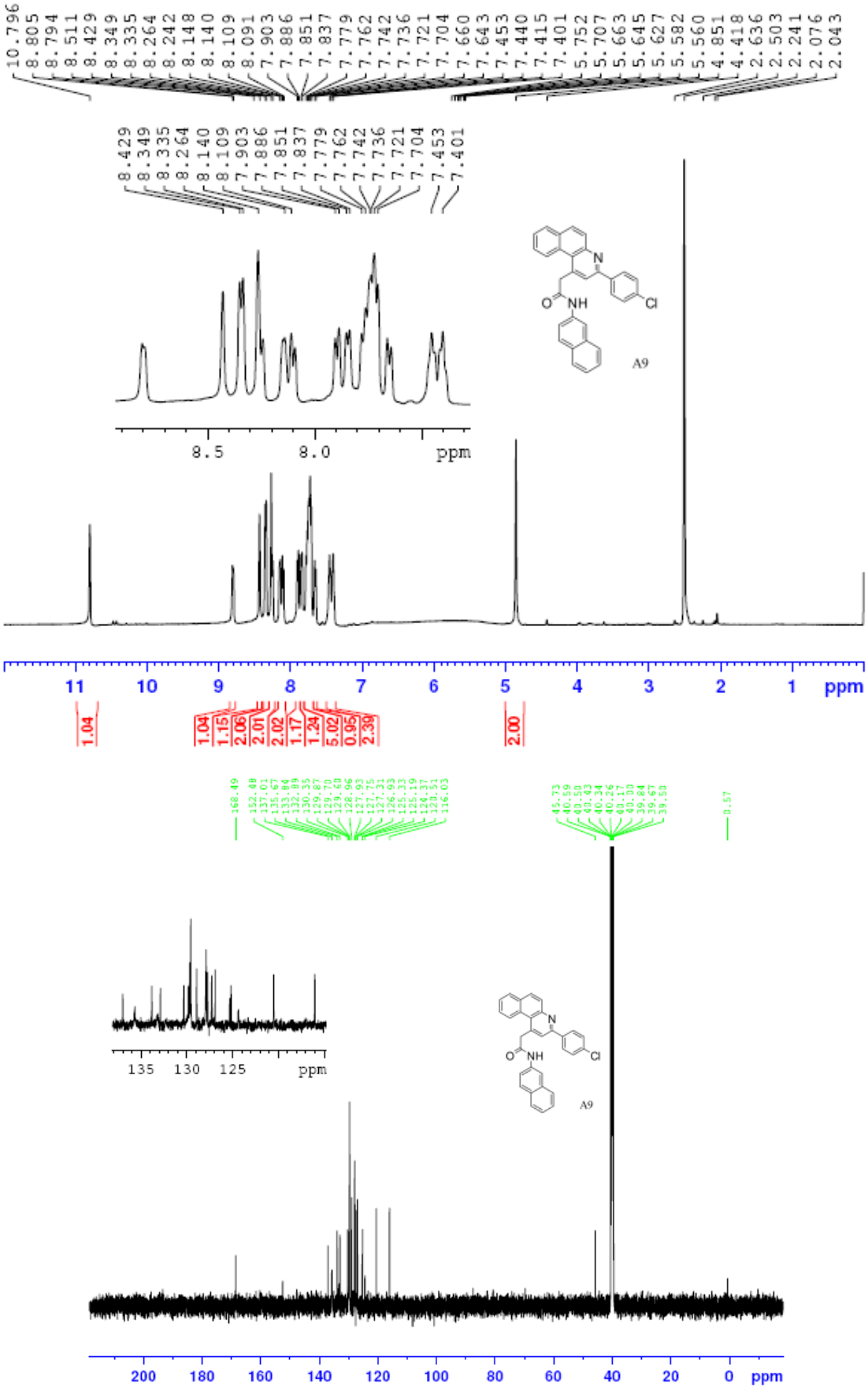
8.683
8.359
8.342
8.314
8.303
8.292
8.229
8.145
8.127
8.109
8.094
8.013
7.995
7.741
7.727
7.712
7.691
7.674
7.657
7.637
7.614
7.426
7.397
4.415
4.373
3.464
3.150
3.137
3.125
3.112
2.642
2.511
2.507
2.504
2.369
1.440
1.426
1.412
1.397
1.383
1.322
1.307
1.292
1.277
1.262
1.248
0.864
0.850
0.835
0.005
A8
0.99
1.93
0.99
1.03
0.96
1.00
0.93
0.97
2.83
2.10
2.14
2.06
2.10
3.00
ppm


169.32
153.11
149.19
144.76
137.00
135.03
132.87
132.03
129.80
129.55
129.44
129.20
129.00
128.71
127.56
127.49
127.22
125.06
123.59
44.80
40.45
40.29
40.12
39.95
39.78
39.62
39.45
39.29
31.65
31.16
19.99
14.10
130
129
128
ppm
A8
200
180
160
140
120
100
80
60
40
20
0
ppm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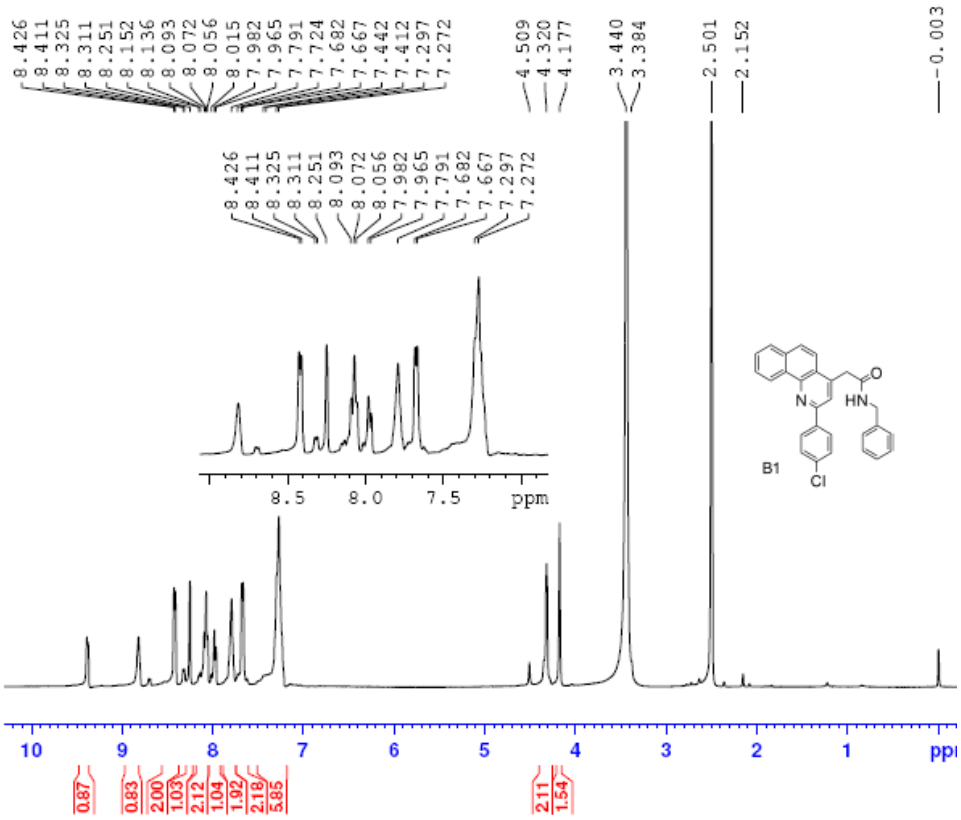
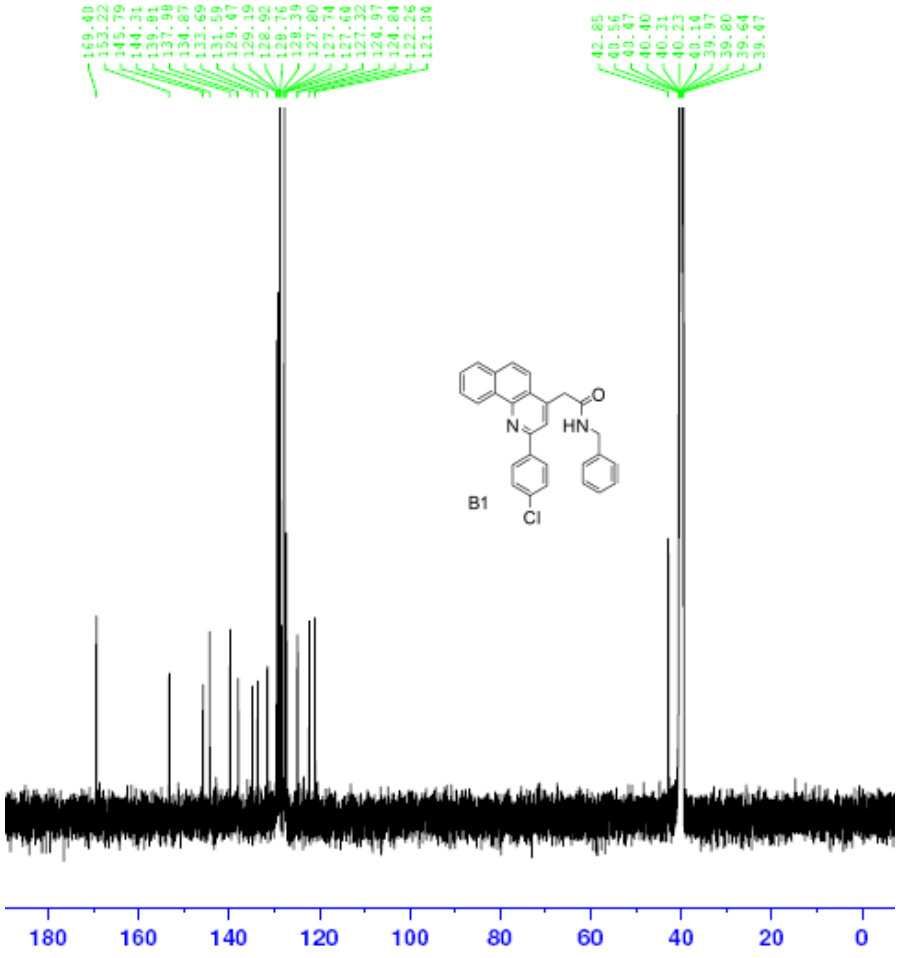
8.794
8.511
8.429
8.349
8.335
8.264
8.242
8.148
8.140
8.109
8.091
7.903
7.886
7.851
7.837
7.779
7.762
7.742
7.736
7.721
7.704
7.660
7.643
7.453
7.440
7.415
7.401
5.752
5.707
5.663
5.645
5.627
5.582
5.560
4.851
4.418
2.636
2.503
2.241
2.076
2.043
8.5
8.0
ppm
A9
11
10
9
8
7
6
5
4
3
2
1
ppm
1.04
1.04
1.15
2.06
2.01
2.02
1.17
1.24
5.02
0.95
2.39
2.00
135
130
125
ppm
A9
200
180
160
140
120
100
80
60
40
20
0
ppm


8.426
8.411
8.325
8.311
8.251
8.152
8.136
8.093
8.072
8.056
8.015
7.982
7.965
7.791
7.724
7.682
7.667
7.442
7.412
7.297
7.272
4.509
4.320
4.177
3.440
3.384
2.501
2.152
-0.003
8.5
8.0
7.5
ppm
B1
Cl
10
9
8
7
6
5
4
3
2
1
0.87
0.83
2.00
1.03
2.12
1.04
1.92
2.18
5.85
2.11
1.54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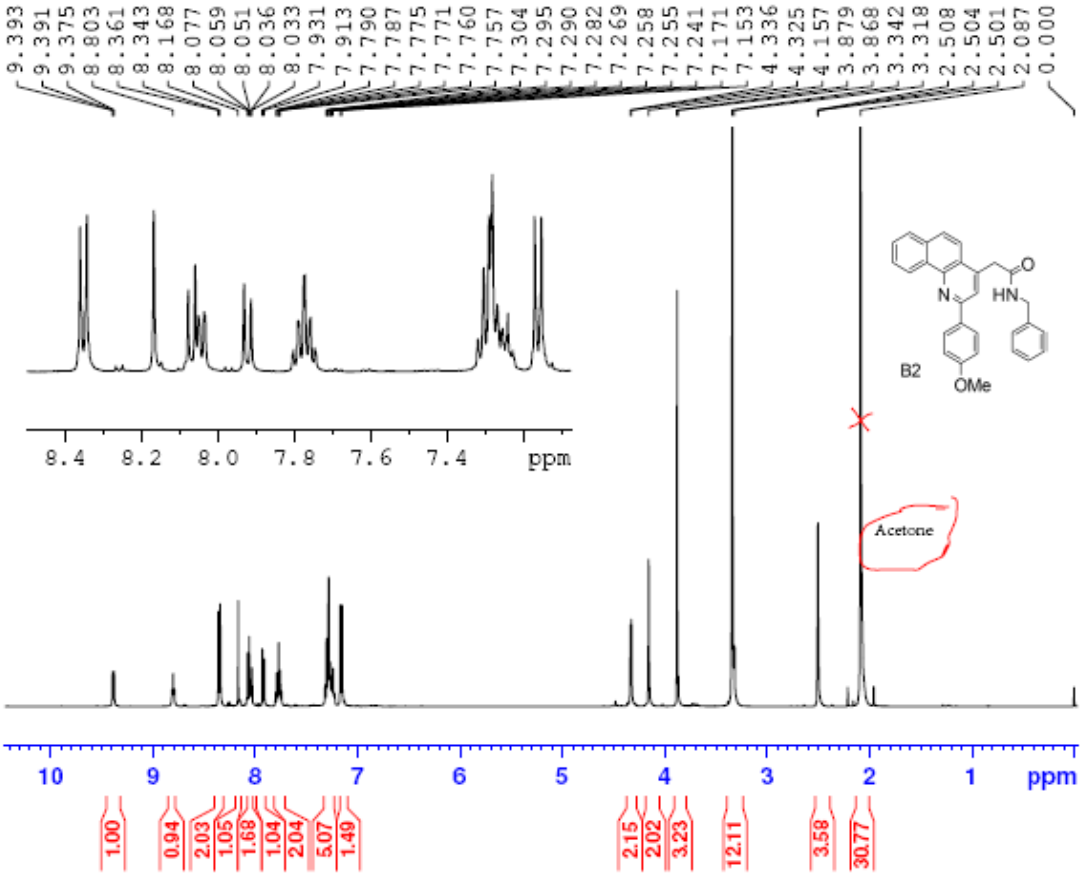
145.79
144.31
139.81
137.98
134.87
131.69
131.59
129.47
129.19
128.92
128.76
128.39
127.80
127.74
127.64
127.32
124.97
124.84
122.26
121.04
42.85
40.56
40.47
40.40
40.31
40.23
40.14
39.97
39.80
39.64
39.47
B1
Cl
180
160
140
120
100
80
60
40
20
0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9.391
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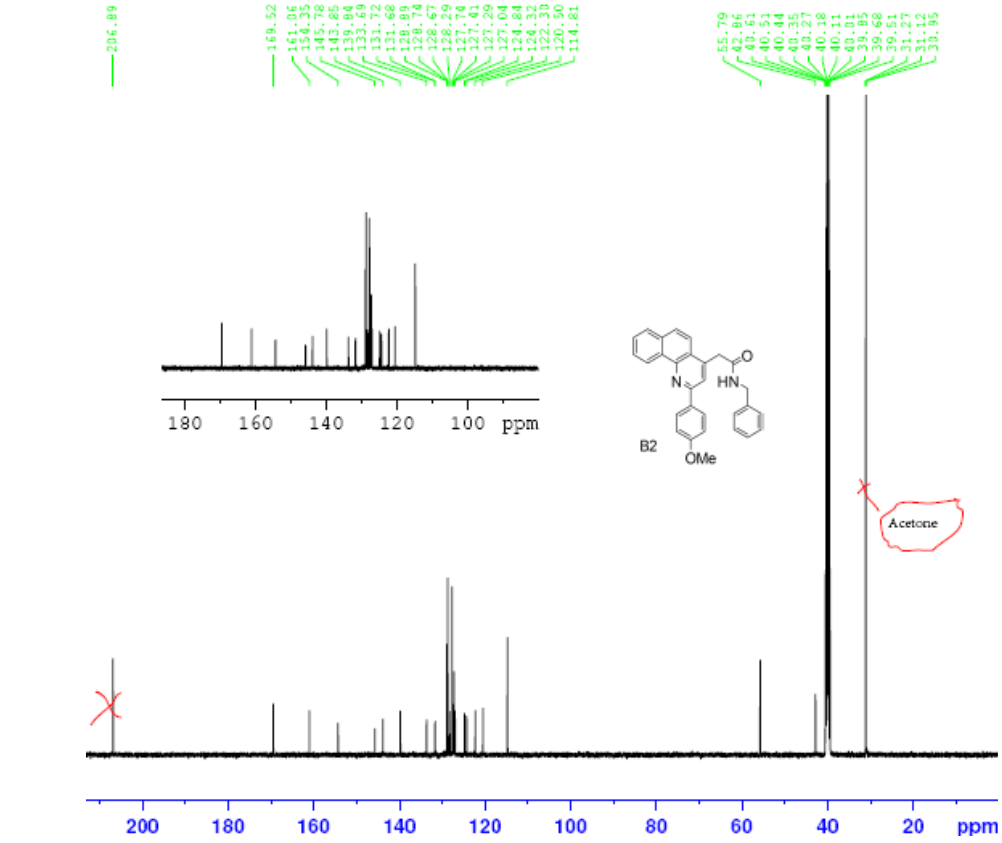
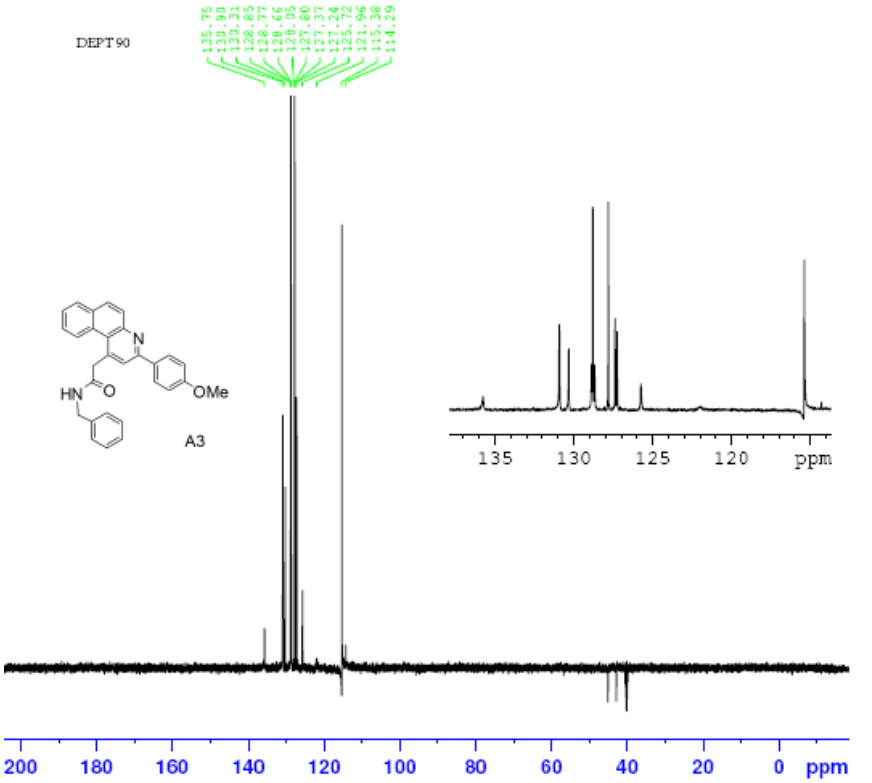
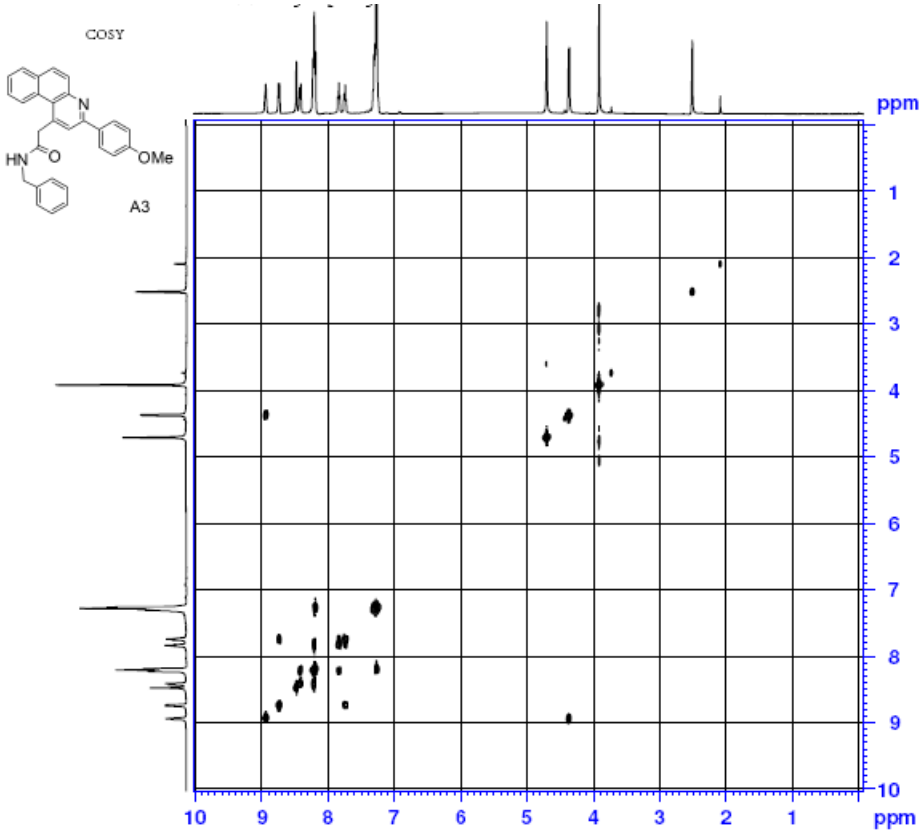
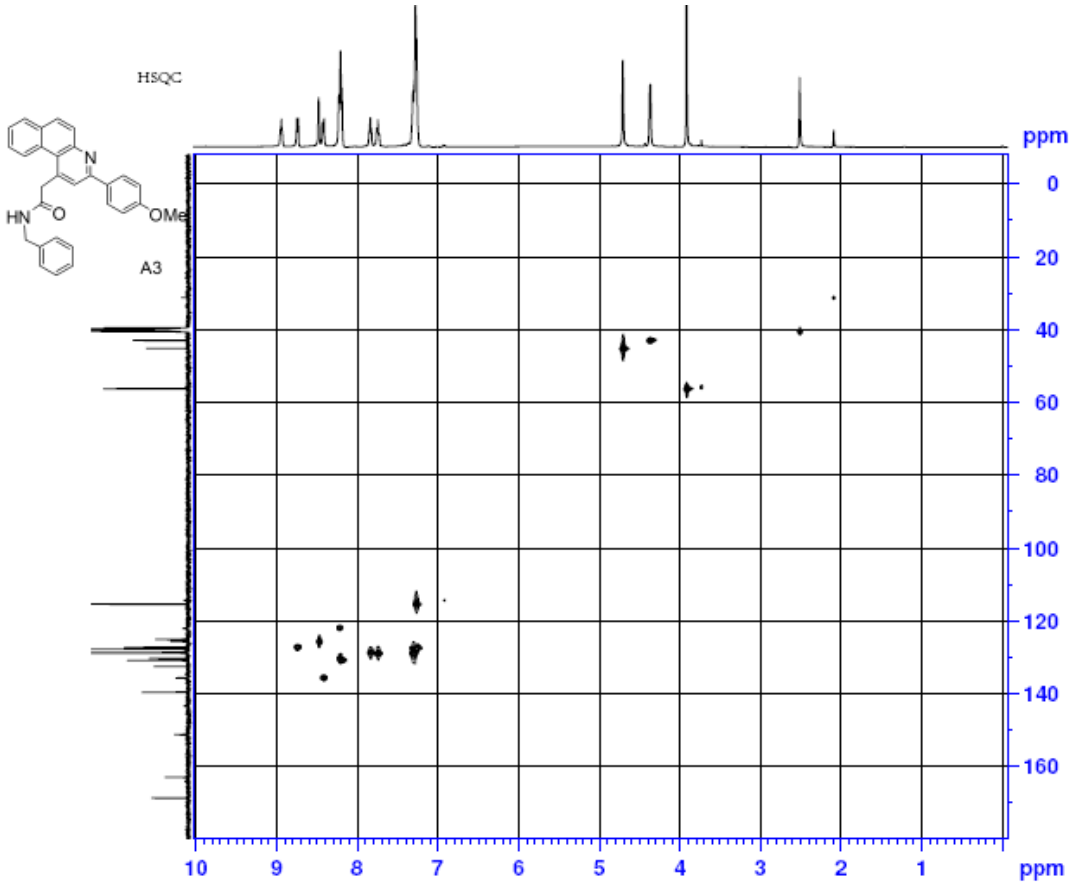
8.168
8.077
8.059
8.051
8.036
8.033
7.931
7.913
7.790
7.787
7.775
7.771
7.760
7.757
7.304
7.295
7.290
7.282
7.269
7.258
7.255
7.241
7.171
7.153
4.336
4.325
4.157
3.879
3.868
3.342
3.318
2.508
2.504
2.501
2.087
0.000
8.4
8.2
8.0
7.8
7.6
7.4
ppm
O
HN
N
B2
OMe
Acetone
10
9
8
7
6
5
4
3
2
1
ppm
1.00
0.94
2.03
1.05
1.68
1.04
2.04
5.07
1.49
2.15
2.02
3.23
12.11
3.58
30.77

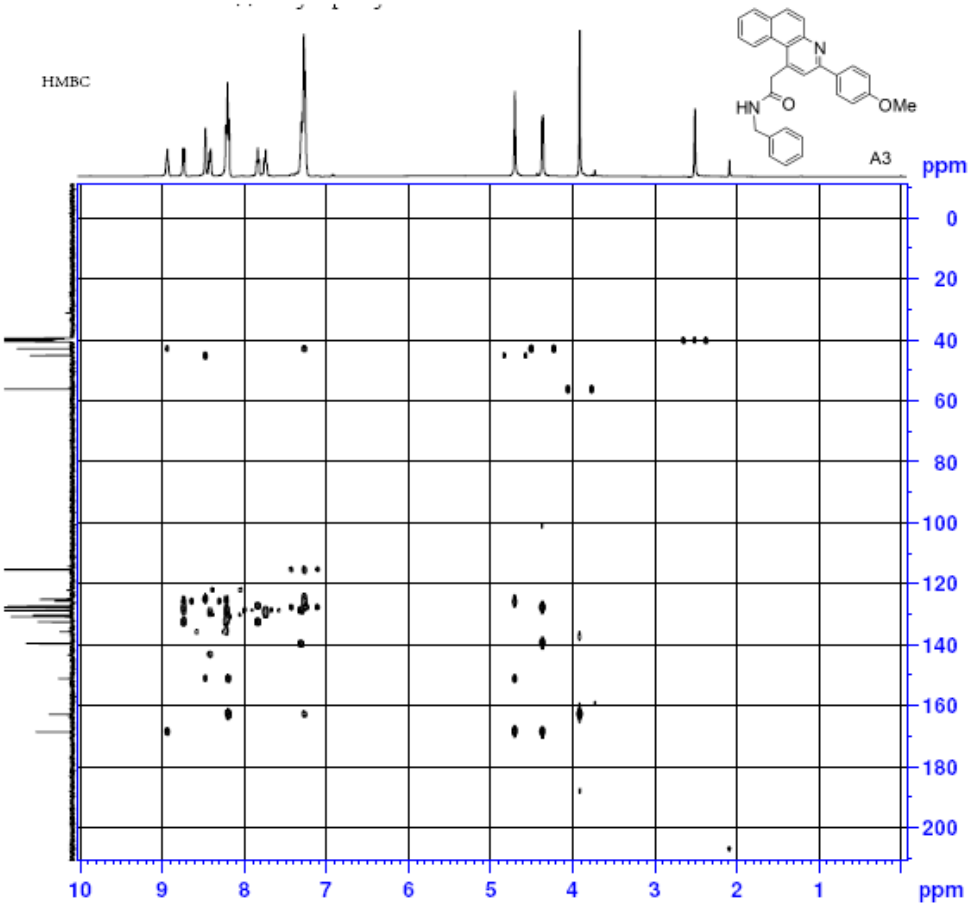

206.89
169.52
161.06
154.35
145.78
143.85
139.84
133.69
131.72
131.68
128.89
128.74
128.67
128.29
127.74
127.41
127.29
127.04
124.84
124.32
123.30
120.30
114.81
55.79
42.86
40.61
40.51
40.44
40.35
40.27
40.18
40.11
40.01
39.85
39.68
39.51
31.27
31.12
30.95
180
160
140
120
100
ppm
O
HN
N
B2
OMe
Acetone
200
180
160
140
120
100
80
60
40
20
ppm

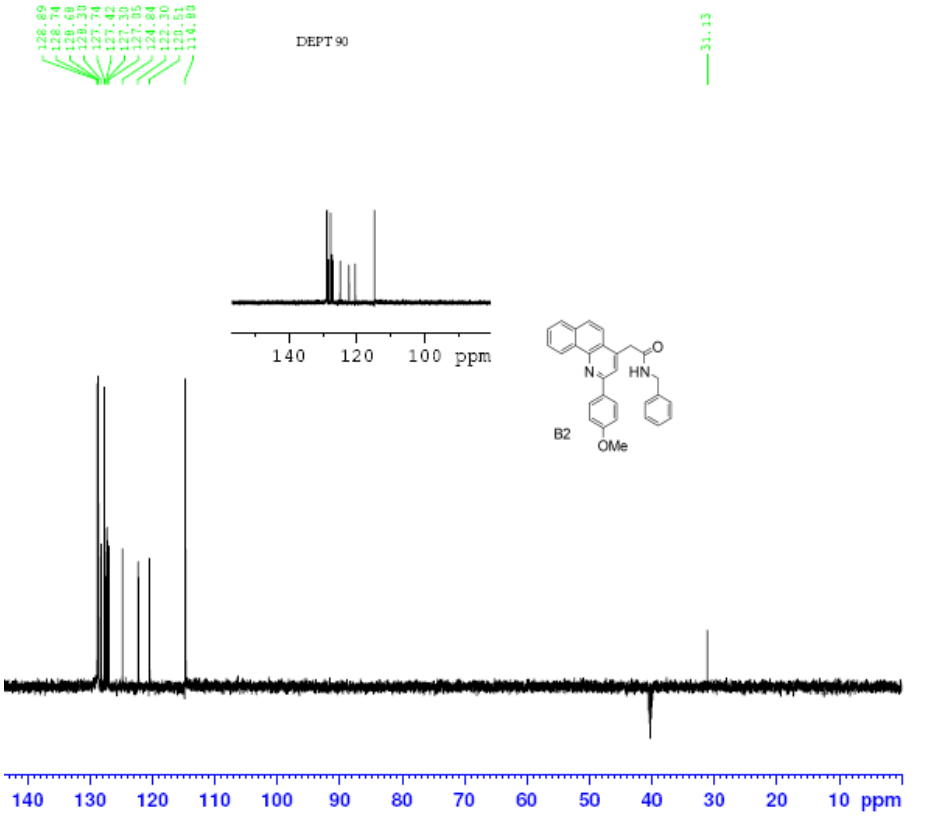
Copies of 2D NMR for A3, B2:

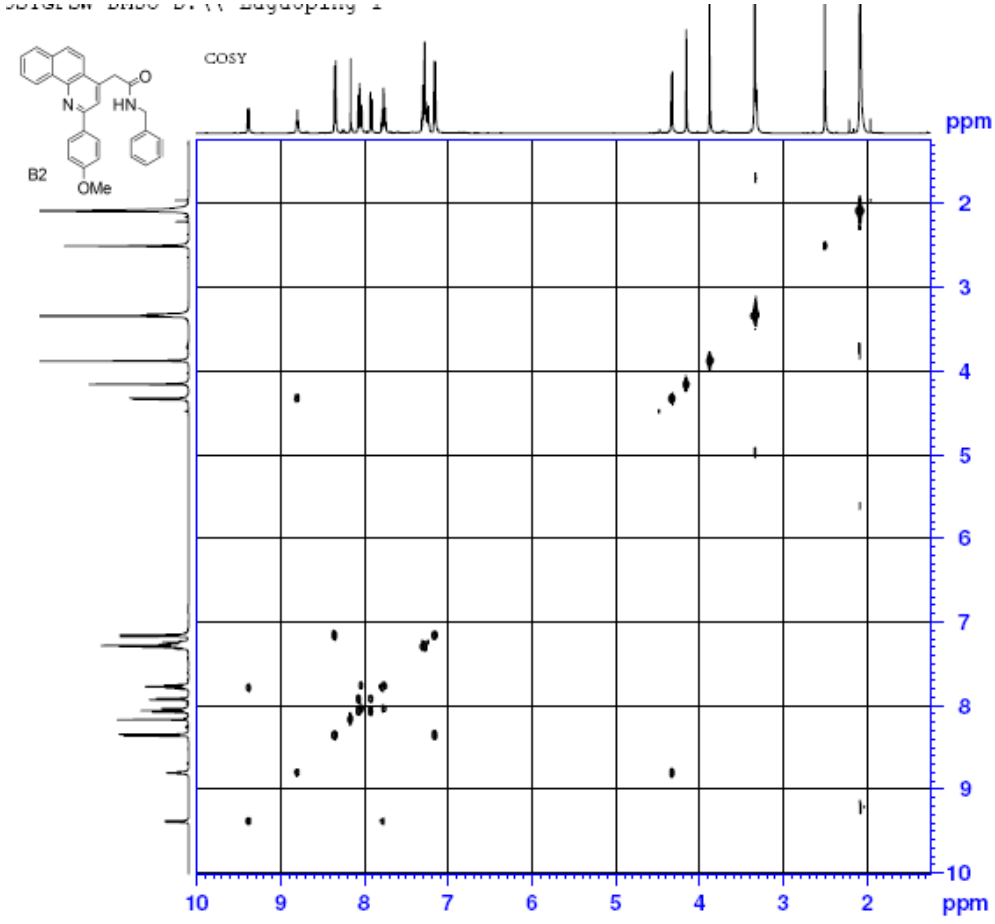


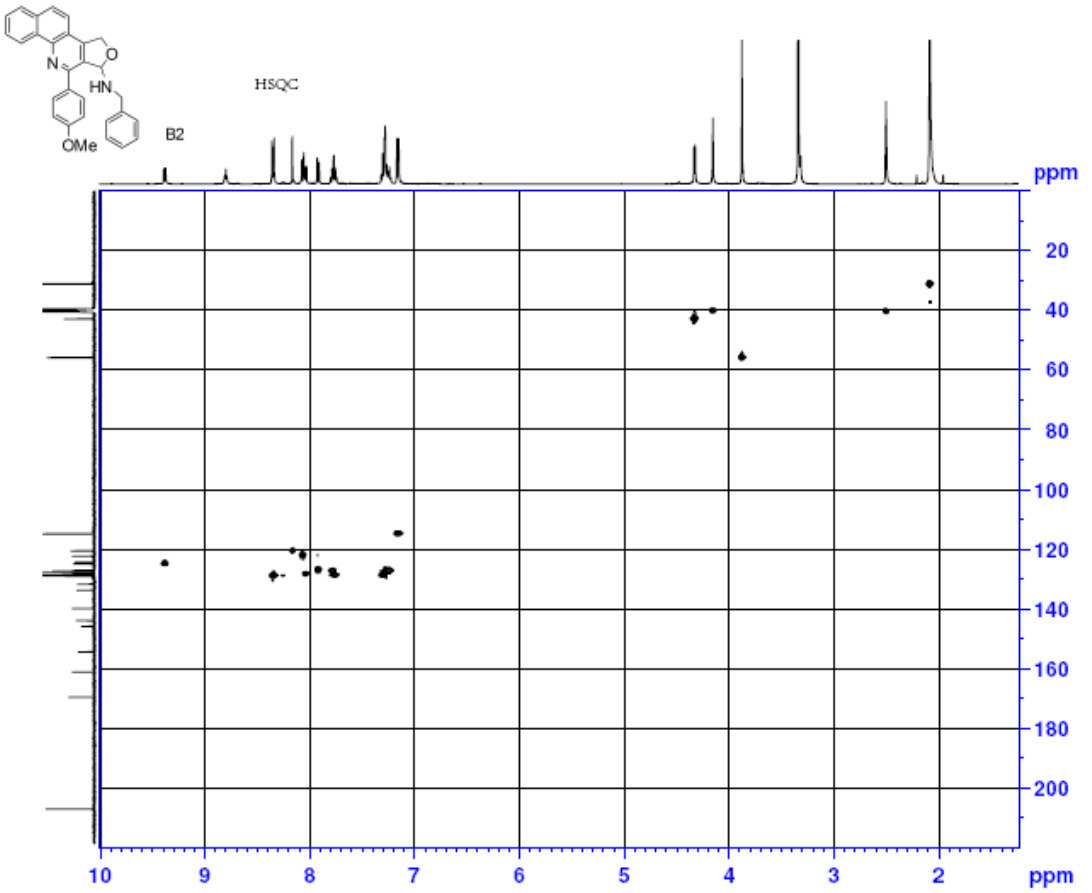


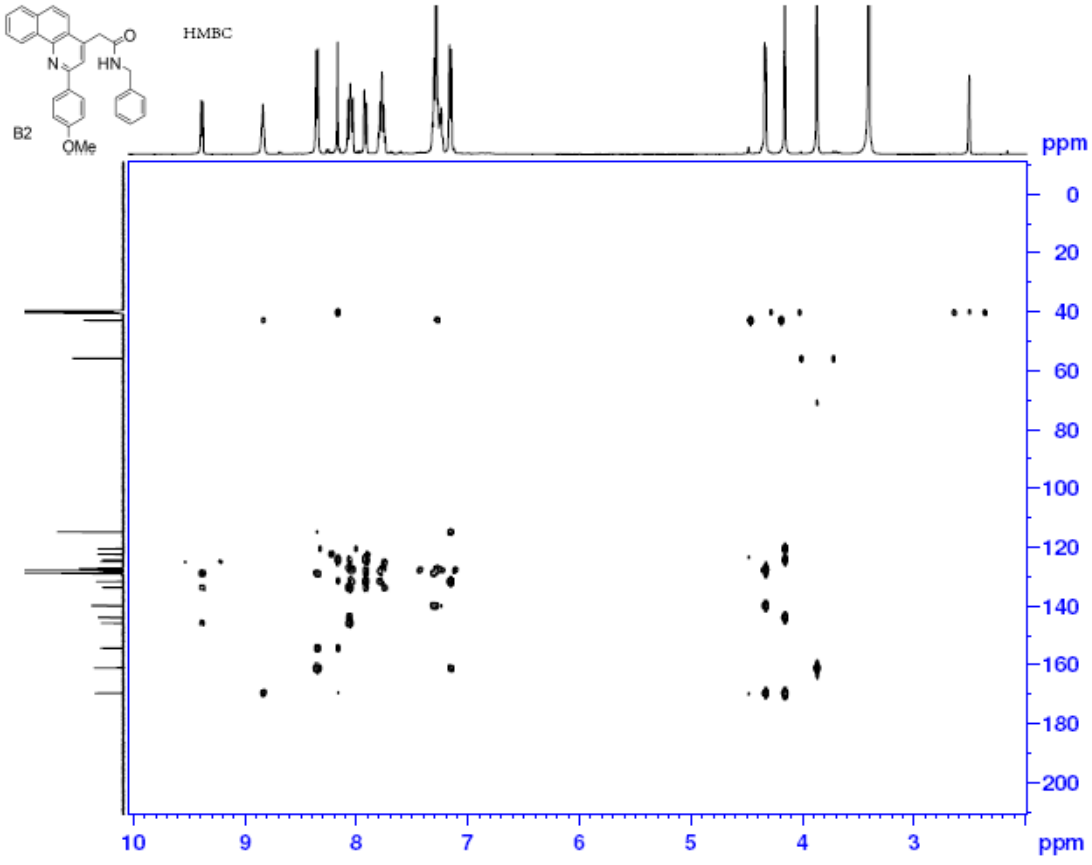

HSQC
HN
O
N
OMe
A3
ppm
0
20
40
60
80
100
120
140
160
10
9
8
7
6
5
4
3
2
1
ppm


HMBC
HN
O
N
OMe
A3
ppm
0
20
40
60
80
100
120
140
160
180
200
10
9
8
7
6
5
4
3
2
1
ppm


128.89
128.74
128.68
128.30
127.74
127.42
127.30
127.05
124.84
122.30
120.51
114.80
DEPT 90
31.13
140
120
100 ppm
O
HN
N
B2
OMe
140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


COSY
O
HN
N
B2
OMe
ppm
2
3
4
5
6
7
8
9
10
10 9 8 7 6 5 4 3 2 ppm


HSQC
B2
OMe
HN
N
O
ppm
20
40
60
80
100
120
140
160
180
200
10
9
8
7
6
5
4
3
2
ppm


HMBC
B2
OMe
HN
N
O
ppm
0
20
40
60
80
100
120
140
160
180
200
10
9
8
7
6
5
4
3
ppm