SUPPORTING INFORMATION FOR

*n*Bu$_4$NI-catalyzed C3-formylation of indoles with N-methylaniline

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1. General experimental methods

^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker AVANCE AV400 (400MHz and 100MHz). Signal positions were recorded in ppm with the abbreviations s, d, t, m and b denoting singlet, doublet, triplet, multiplet and broadened respectively. All NMR chemical shifts were referenced to residual solvent peaks or to $Si(CH3)_4$ as an internal standard, spectra recorded in $CDCl_3$ were referenced to residual $CHCl_3$ at 7.26 ppm for ^{1}H or 77.0 ppm for ^{13}C, spectra recorded in CD_3OD were referenced to residual CD_2HOD at 3.31 ppm for ^{1}H or 49.15 ppm for ^{13}C, spectra recorded in $(CD_3)_2SO$ were referenced to residual $(CD_2H)SO(CD_3)$at 2.50 ppm for ^{1}H or 39.52 ppm for ^{13}C. All coupling constants J were quoted in Hz. Data were reported as follows: chemical shift, multiplicity, coupling constant and integration. Reactions were monitored by thin-layer chromatography (TLC) on 0.25mm silica gel glass plates coated with 60 F_{254}. Column chromatography was performed on silica gel (200-300 mesh) using a mixture of petroleum ether (60-90°C)/ethyl acetate as eluant. Reactions were carried out under a nitrogen atmosphere. Commercially available reagents were used as received without purification.

2. General procedure for the Bu_4NI-catalyzed formylation of indoles with N-methylaniline

To a mixture of indole (0.5 mmol, 1 equiv), PivOH (255.3 mg, 2.5 mmol, 5 equiv), and *n*BuNI (18.5 mg, 0.05 mmol, 0.1 equiv) in a 50 mL Schlenk tube were added DMSO (1 mL) under N_2. Then N-methylaniline (107 μL, 1.0 mmol, 2 equiv) and TBPB (373 μL, 2.0 mmol, 4 equiv) were added separately. The mixture was stirred at 80°C for 8h, quenched with saturated $NaHCO_3$ solution (10 mL). The mixture was extracted by ethyl acetate for 3 times. The combined organic phase was washed with brine and dried with anhydrous Na_2SO_4, filtrated, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography to give the formylation products.

3. Optimization of reaction conditions[a]

entry[a]	**1a** (mmol)	**2a** (mmol)	catalyst (mmol)	oxidant (mmol)	Yield (%)
1	0.5	1	*n*Bu_4NCl (0.05)	TBPB (2)	37[b]
2	0.5	1	*n*Bu_4NBr (0.05)	TBPB (2)	29[b]
3	0.5	1	*n*Bu_4NI (0.05)	TBHP(2)[c]	0
4	0.5	1	*n*Bu_4NI (0.05)	IBX (2)	trace
5	0.5	1	*n*Bu_4NI (0.05)	NBS (2)	trace
6	0.5	1	*n*Bu_4NI (0.05)	NIS (2)	0
7	0.5	1	*n*Bu_4NI (0.05)	$PhI(OAc)_2$ (2)	trace
8	0.5	1	*n*Bu_4NI (0.05)	Oxone (2)	0
9	0.5	1	*n*Bu_4NI (0.05)	$K_2S_2O_8$ (2)	0
10	0.5	1	*n*Bu_4NI (0.05)	*m*-CPBA (2)	0
11	0.5	1	*n*Bu_4NI (0.05)	*p*-BQ (2)	0
12	0.5	1	*n*Bu_4NI(0.05)	DDQ (2)	0

[a]general reaction conditions: to a mixture of indole (0.5mmol), catalyst (0.05mmol), PivOH (2.5mmol), oxidant (2mmol) in a schlank tube was added DMSO (1mL) under N_2. N-methylaniline (1.0mmol) was added, then the mixture was heated at 80°C for 8h. [b]GC yield. [c]solution of TBHP in decane (5.0-6.0M) was used.

4. Preparation of 3-formylindole with compound 5

To a mixture of compound **5** (0.5 mmol, 1 equiv), PivOH (255.3 mg, 2.5 mmol, 5 equiv), and *n*BuNI (18.5 mg, 0.05 mmol, 0.1 equiv) in a 50 mL Schlenk tube were added DMSO (1 mL) under N_2. Then TBPB (373 μL, 2.0 mmol, 4 equiv) were added separately. The mixture was stirred at 80°C for 8h, quenched with saturated $NaHCO_3$ solution (10 mL). The mixture was extracted by ethyl acetate for 3 times. The combined organic phase was washed with brine and dried with anhydrous Na_2SO_4, filtrated, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography to give the formylation product **3a** in 70% yield.

5. The characterization of the products

3a[1]

^{1}H-NMR (DMSO-d_6, 400MHz): 12.1 (b, 1H), 9.94(s, 1H), 8.29(s, 1H), 8.10(d, 7.2Hz, 1H), 7.51(d, 8.0Hz, 1H), 7.28-7.20(m, 2H)
^{13}C-NMR (DMSO-d_6, 100MHz): 185.00, 138.53, 137.07, 124.14, 123.49, 122.16, 120.85, 118.18, 112.45

3b[1]

^{1}H-NMR (DMSO-d_6, 400MHz): 12.2(b, 1H), 9.92(s, 1H), 8.35(d, 3.2Hz, 1H), 7.76(dd, 2.4Hz, 9.6Hz, 1H), 7.53(dd, 4.8Hz, 8.8Hz, 1H), 7.11(td, 2.8Hz, 9.2Hz, 1H)
^{13}C-NMR (DMSO-d_6, 100MHz): 185.04, 158.74 (239.6Hz), 139.63, 133.61, 124.73 (7Hz), 118.14, 113.77 (9.5Hz), 111.61 (25.8Hz), 105.70 (24.3Hz)

3c[1]

^{1}H-NMR (DMSO-d_6, 400MHz): 12.3(b, 1H), 9.93(s, 1H), 8.36(d, 3.2Hz, 1H), 8.06(d, 2.2Hz, 1H), 7.54(d, 8.4Hz, 1H), 7.28(dd, 2.2Hz, 8.6Hz, 1H)
^{13}C-NMR (DMSO-d_6, 100MHz): 185.10, 139.39, 135.48, 126.77, 125.27, 123.47, 119.87, 117.55, 114.11

3d[2]

^{1}H-NMR (CD_3OD, 400MHz): 9.86(s, 1H), 8.05(s, 1H), 7.99(d, 8.0Hz, 1H), 7.16(t, 7.6Hz, 1H), 7.09(d, 7.2Hz, 1H), 2.90(q, 7.6Hz, 2H), 1.31(t, 7.6Hz, 3H)
^{13}C-NMR (CD_3OD, 100MHz): 187.57, 139.56, 137.63, 129.50, 125.84, 124.13, 124.00, 120.57, 120.20, 25.21, 15.10
^{1}H-NMR (DMSO-d_6, 400MHz): 12.1(b, 1H), 9.93(s, 1H), 8.27(d, 3.2Hz, 1H), 7.93(d, 7.6Hz, 1H), 7.15(t, 7.6Hz, 1H), 7.08(d, 7.2Hz, 1H), 2.89(t, 7.4Hz, 2H), 1.26(t, 7.4Hz, 3H)
^{13}C-NMR (DMSO-d_6, 100MHz): 184.92, 138.12, 135.74, 128.04, 124.08, 122.42,

122.25, 118.56, 118.43, 23.52, 14.55

3e[3]

^{1}H-NMR (DMSO-d_6, 400MHz): 12.2(b, 1H), 9.93(s, 1H), 8.32(d, 3.2Hz, 1H), 8.02(d, 8.4Hz, 1H), 7.71(d, 1.6Hz, 1H), 7.36(dd, 1.6Hz, 8.4Hz, 1H)
^{13}C-NMR (DMSO-d_6, 100MHz): 185.15, 139.18, 137.91, 125.05, 123.14, 122.46, 117.97, 115.91, 115.15

3f[1]

^{1}H-NMR (DMSO-d_6, 400MHz): 12.3(b, 1H), 9.91(s, 1H), 8.43(d, 1.6Hz, 1H), 8.30(d, 3.2Hz, 1H), 7.54(dd, 1.6Hz, 8.6Hz, 1H), 7.37(d, 8.4Hz, 1H)
^{13}C-NMR (DMSO-d_6, 100MHz): 185.15, 138.89, 136.15, 131.53, 129.12, 126.60, 117.14, 114.92, 86.58

3g[1]

^{1}H-NMR (CD_3OD, 400MHz): 10.0(s, 1H), 8.09(d, 7.2Hz, 1H), 7.34(d, 7.2Hz, 1H), 7.18(t, 4.4Hz, 2H), 2.70(s, 3H)
^{13}C-NMR (CD_3OD, 100MHz): 186.55, 151.03, 137.34, 127.38, 124.33, 123.57, 121.68, 115.60, 112.33, 11.80

3h[4]

^{1}H-NMR (DMSO-d_6, 400MHz): 12.3(b, 1H), 9.93(s, 1H), 8.19(d, 3.2Hz, 1H), 7.67(d, 7.6Hz, 1H), 7.57(d, 7.2Hz, 2H), 7.42(t, 7.4Hz, 2H), 7.35(d, 7.2Hz, 1H), 7.12(t, 8.0Hz, 1H), 6.93(d, 8.0Hz, 1H), 5.29(s, 2H)
^{13}C-NMR (DMSO-d_6, 100MHz): 185.12, 145.26, 137.81, 137.03, 128.43, 127.90, 127.68, 127.18, 125.83, 122.95, 118.68, 113.57, 105.54, 69.38

3i[1]

^{1}H-NMR (DMSO-d_6, 400MHz): 12.6(b, 1H), 10.00(s, 1H), 8.55(d, 3.2Hz, 1H), 8.24(d, 8.0Hz, 1H), 8.04(s, 1H), 7.59(dd, 1.4Hz, 8.2Hz, 1H)
^{13}C-NMR (DMSO-d_6, 100MHz): 185.44, 141.30, 135.95, 127.38, 125.08, 121.74, 119.81, 118.04, 117.43, 105.13

3j[1]

^{1}H-NMR (DMSO-d_6, 400MHz): 12.0(b, 1H), 9.89(s, 1H), 8.21(d, 3.2Hz, 1H), 7.58(d, 2.8Hz, 1H), 7.40(d, 8.8Hz, 1H), 6.88(dd, 2.4Hz, 8.8Hz, 1H), 3.78(s, 3H)
^{13}C-NMR (DMSO-d_6, 100MHz): 184.78, 155.59, 138.38, 131.76, 124.86, 118.00, 113.26, 113.15, 102.45, 55.23

3k[1]

^{1}H-NMR (DMSO-d_6, 400MHz): 12.4(b, 1H), 9.98(s, 1H), 8.50(d, 3.2Hz, 1H), 8.18(d, 8.4Hz, 1H), 8.14(s, 1H), 7.83(d, 8.4Hz, 1H), 3.87(s, 3H)
^{13}C-NMR (DMSO-d_6, 100MHz): 185.25, 166.64, 140.94, 136.47, 127.76, 124.53, 122.77, 120.68, 118.04, 114.18, 52.06

3l[1]

^{1}H-NMR (CD_3OD, 400MHz):9.86(s, 1H), 8.06(s, 1H), 7.98(d, 7.6Hz, 1H), 7.13(t, 7.4Hz, 1H), 7.06(d, 7.2Hz, 1H), 2.51(s, 3H)
^{13}C-NMR (CD_3OD, 100MHz): 187.63, 139.57, 138.43, 125.71, 125.60, 123.99, 123.05, 120.57, 120.13, 16.90
^{1}H-NMR (DMSO-d_6, 400MHz): 12.2(b, 1H), 9.95(s, 1H), 8.29(d, 3.2Hz, 1H), 7.93(d, 7.6Hz, 1H), 7.13(t, 7.6Hz, 1H), 7.06(d, 7.2Hz, 1H), 2.50(s, 3H)
^{13}C-NMR (DMSO-d_6, 100MHz): 185.01, 138.17, 136.55, 124.01, 123.92, 122.32, 121.75, 118.56, 118.38, 16.67

3m[1]

^{1}H-NMR (DMSO-d_6, 400MHz): 12.3(b, 1H), 9.93(s, 1H), 8.34(d, 3.2Hz, 1H), 8.22(d, 1.6Hz, 1H), 7.49(d, 8.4Hz, 1H), 7.39(dd, 2.0Hz, 8.8Hz, 1H)
^{13}C-NMR (DMSO-d_6, 100MHz): 185.13, 139.23, 135.74, 126.04, 125.89, 122.91, 117.43, 114.80, 114.55

3n[5]

^{1}H-NMR ($CDCl_3$, 400MHz): 9.99(s, 1H), 8.33(d, 6.4Hz, 1H), 7.66(s, 1H), 7.37(s, 3H), 3.86(s, 3H)
^{13}C-NMR ($CDCl_3$, 100MHz): 184.48, 139.35, 137.91, 125.28, 124.04, 122.95, 122.02, 118.05, 109.90, 33.68

3o[6]

^{1}H-NMR (DMSO-d_6, 400MHz): 9.94(s, 1H), 8.48(s, 1H), 8.12(d, 6.8Hz, 1H), 7.59(d, 8.0Hz, 1H), 7.34-7.26(m, 7H), 5.55(s, 2H)
^{13}C-NMR (DMSO-d_6, 100MHz): 184.68, 140.97, 136.94, 136.75, 128.71, 127.77, 127.33, 124.78, 123.60, 122.54, 121.06, 117.38, 111.39, 49.78

3p[7]

^{1}H-NMR (DMSO-d_6, 400MHz): 10.0(s, 1H), 8.61(s, 1H), 8.24-8.20(m, 1H), 7.70-7.63(m, 4H), 7.56-7.54(m, 2H), 7.37-7.34(m, 2H)
^{13}C-NMR (DMSO-d_6, 100MHz): 185.29, 140.57, 137.61, 136.74, 130.04, 128.15, 124.97, 124.70, 124.49, 123.23, 121.39, 118.80, 111.32

3q[8]

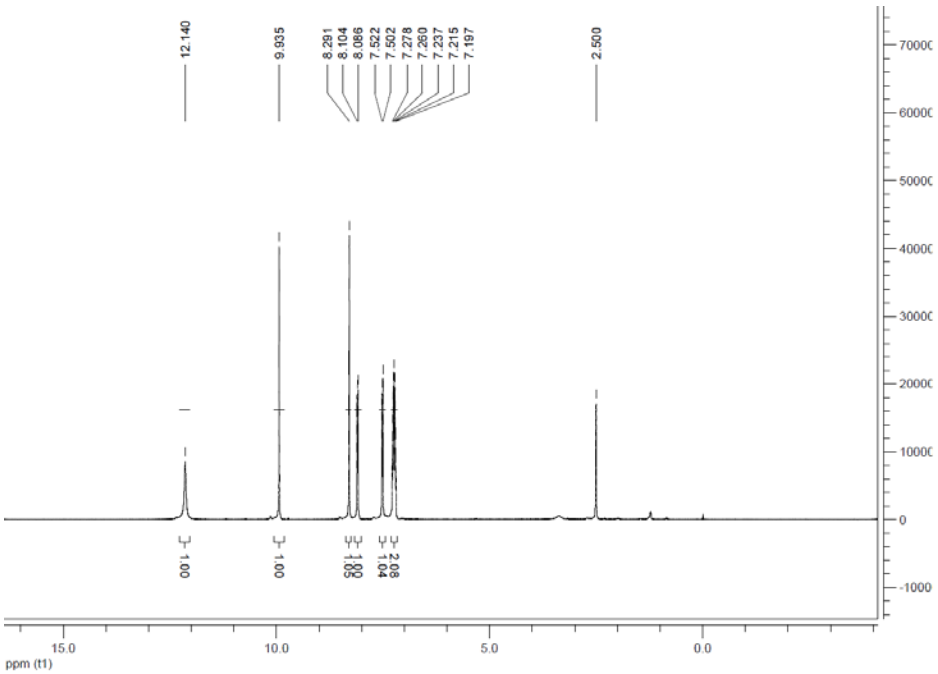
^{1}H-NMR ($CDCl_3$, 400MHz): 10.1(s, 1H), 8.34-8.32(m, 2H), 7.95(s, 1H), 7.80-7.78(m, 2H), 7.70(t, 7.4Hz, 1H), 7.60(t, 7.6Hz, 2H), 7.50-7.45(m, 2H)
^{13}C-NMR ($CDCl_3$, 100MHz): 185.69, 168.50, 137.59, 136.81, 132.99, 129.44, 129.01, 126.59, 126.24, 125.58, 122.18, 122.04, 116.08

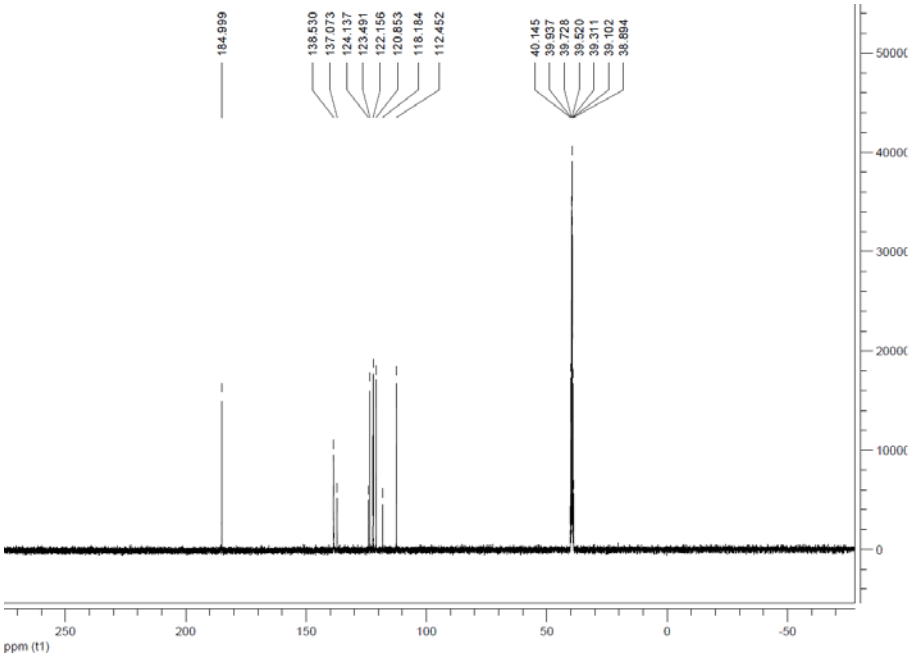
4[9]

^{1}H-NMR ($CDCl_3$, 400MHz): 7.06 (d, 8.4Hz, 2H), 6.71(d, 5.6Hz, 2H), 3.81(s, 2H), 2.91(s, 12H)
^{13}C-NMR ($CDCl_3$, 100MHz): 129.41, 113.12, 40.97, 39.88

6. ^{1}H and ^{13}C NMR charts of the products

3a

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39.311
39.102
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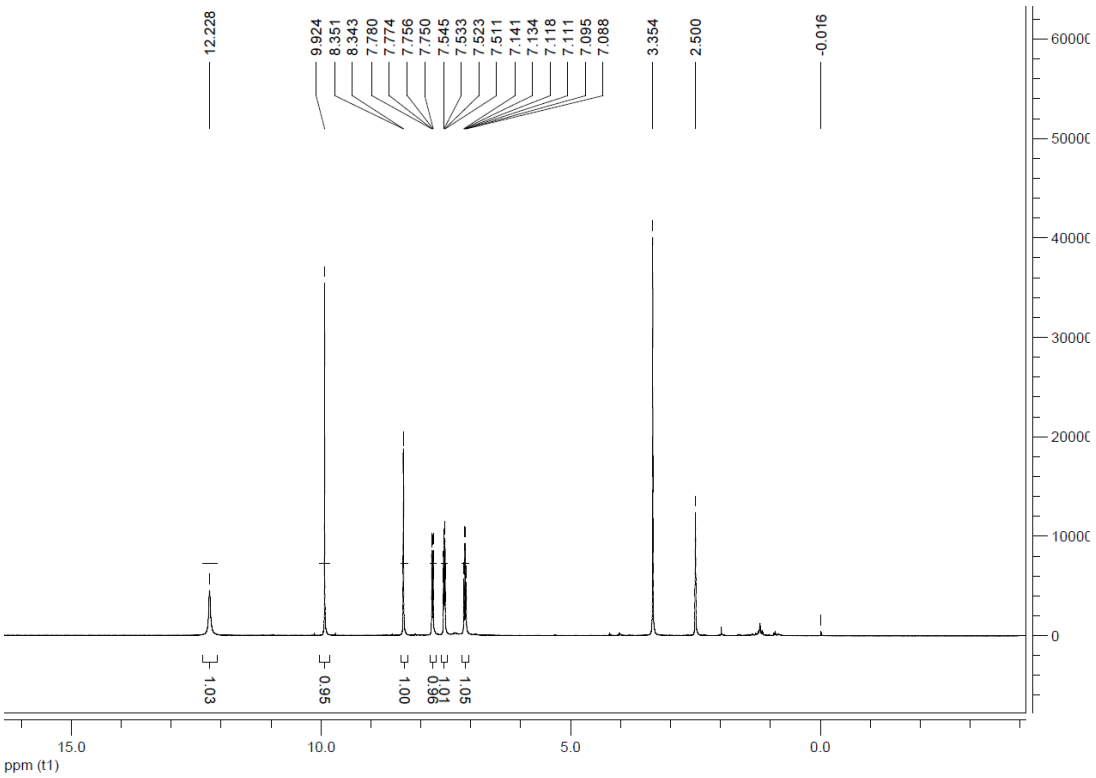
3b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7.134
7.118
7.111
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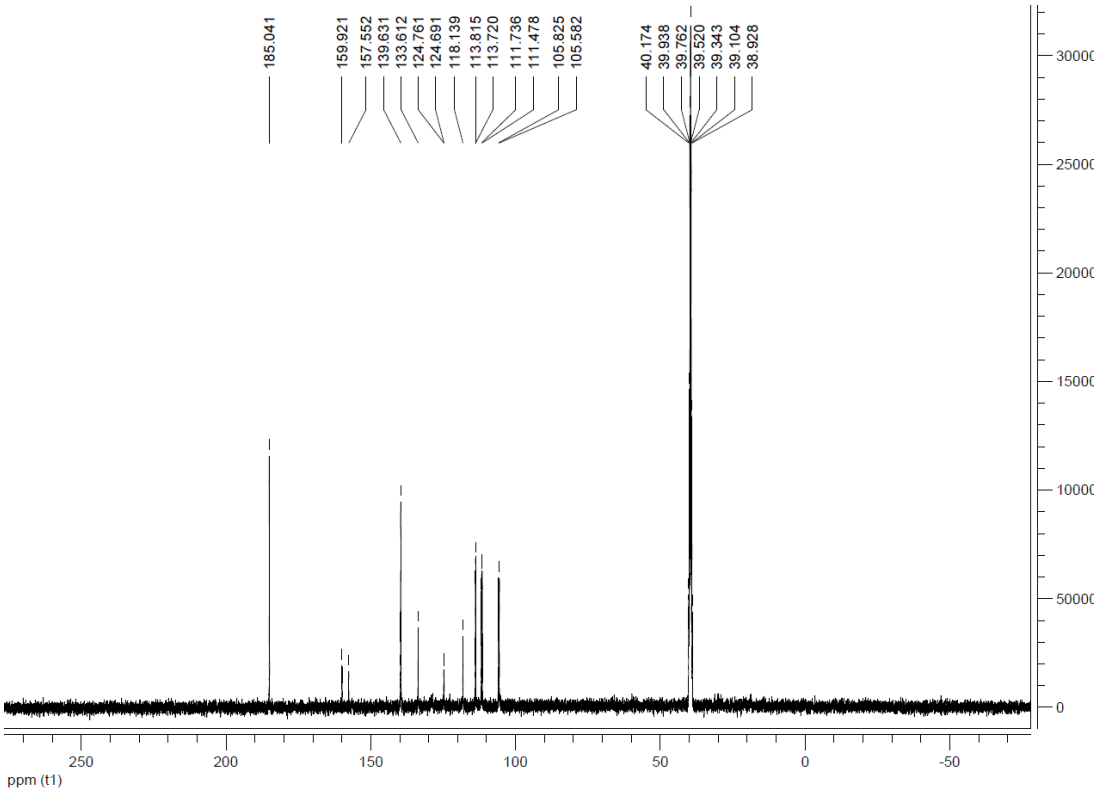
ppm (t1)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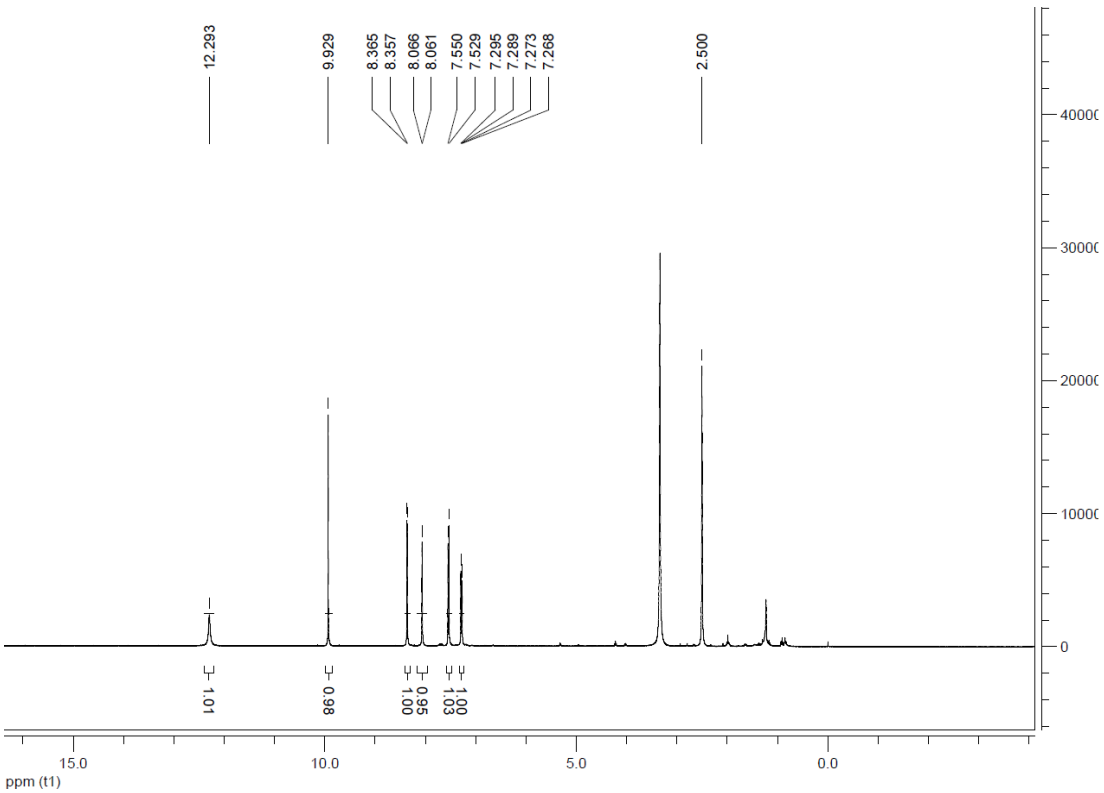
ppm (t1)

3c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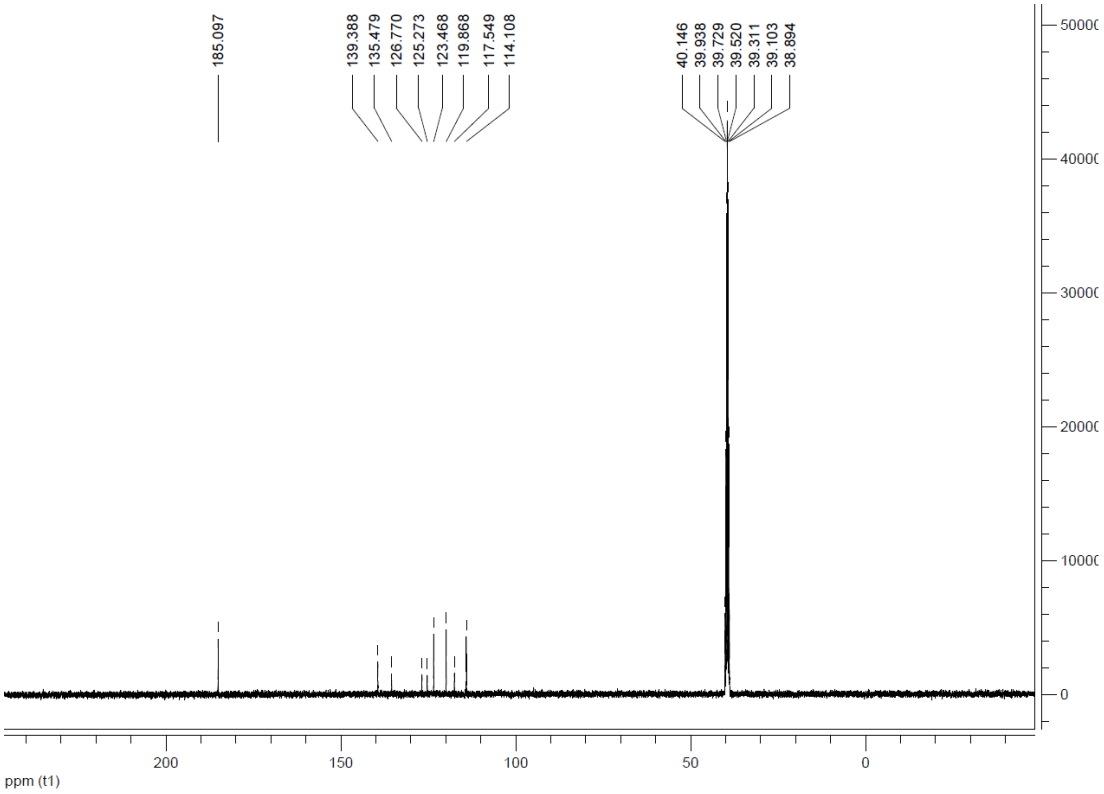
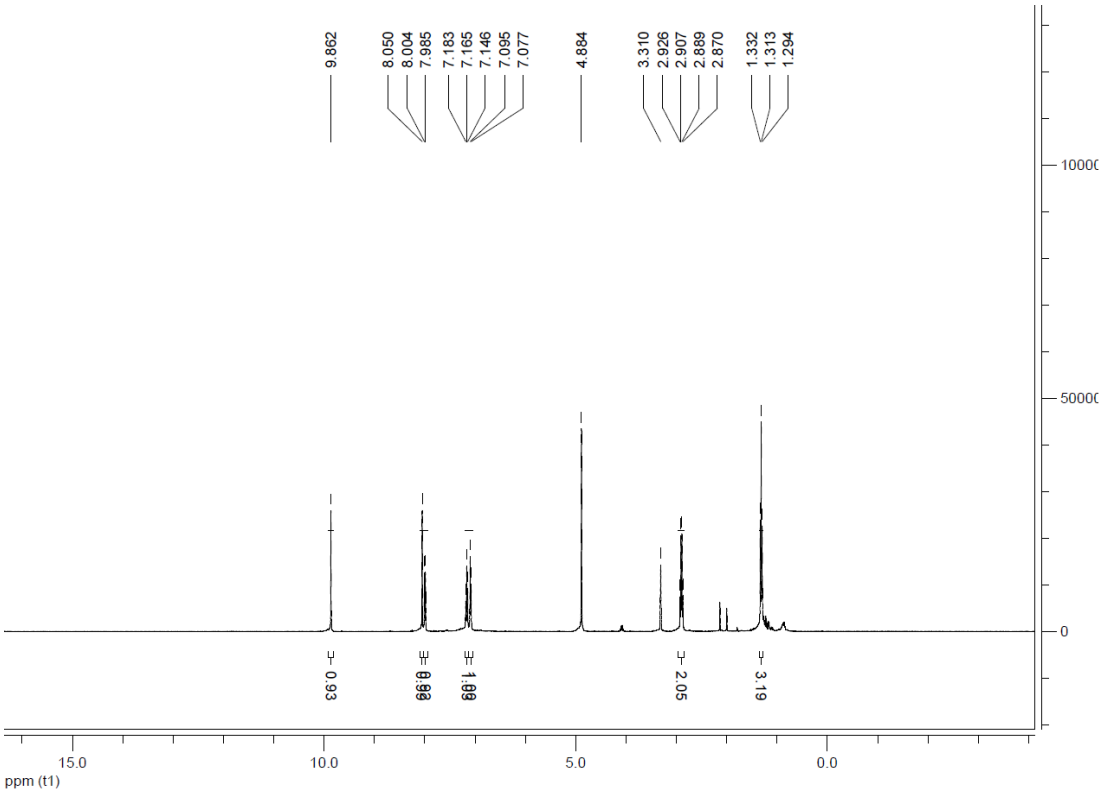
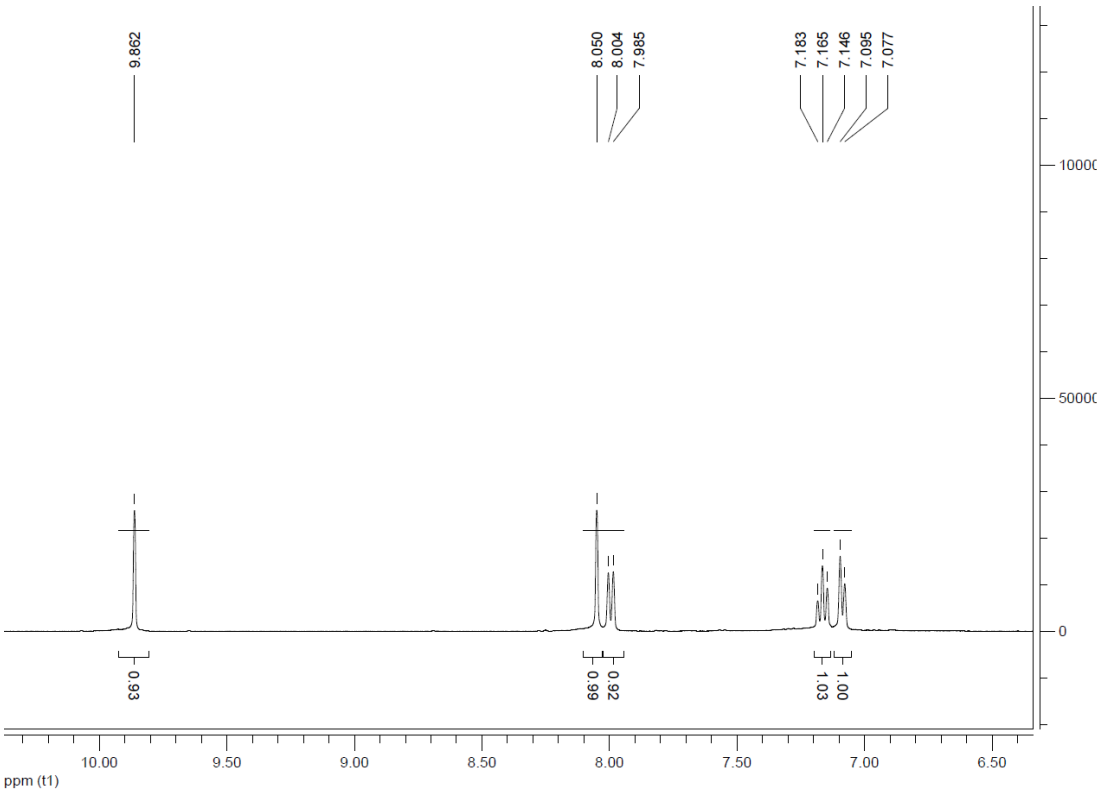
1.00
0.95
1.03
1.00
ppm (t1)



3d in methanol-d_4

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8.004
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7.165
7.146
7.095
7.077
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0.99
0.92
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9.00
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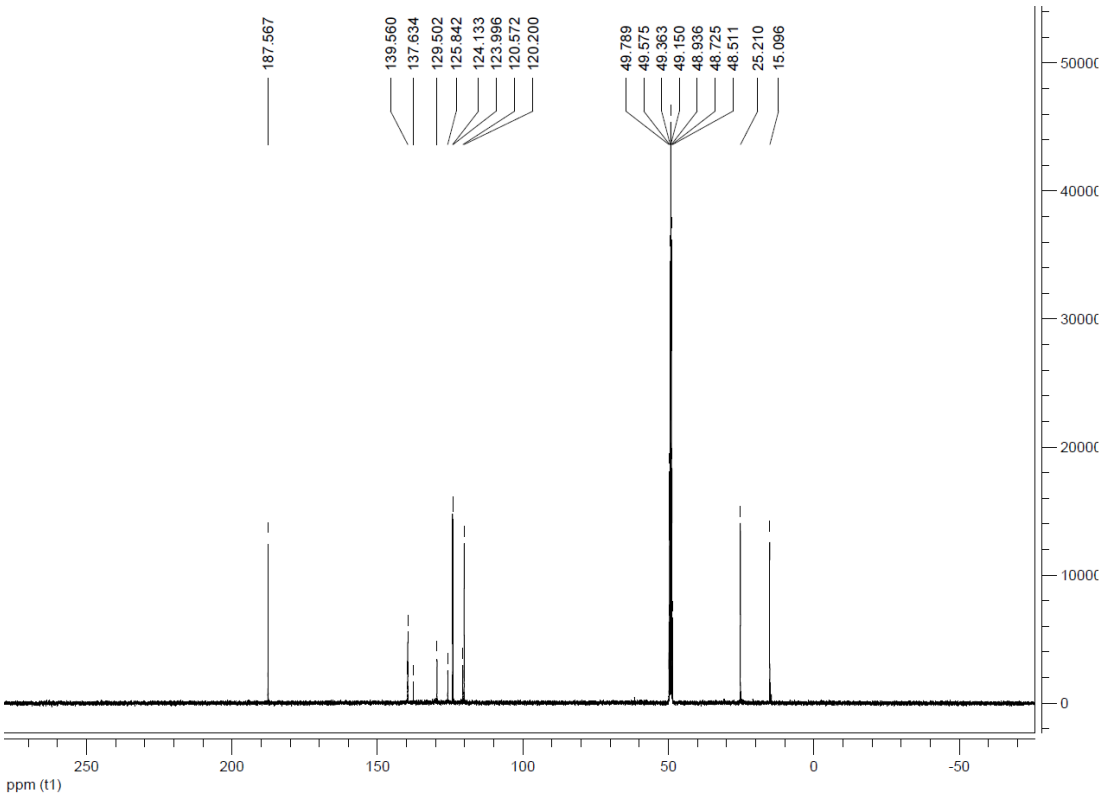
7.50
7.00
6.50
ppm (t1)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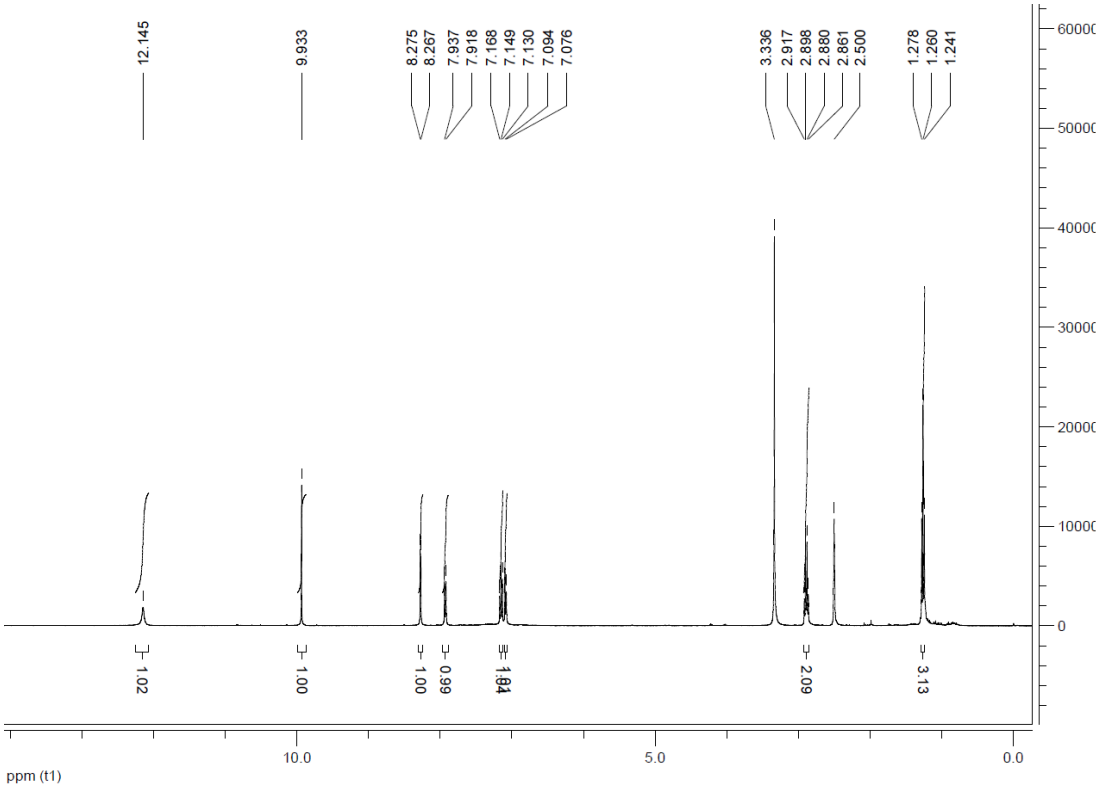
125.842
124.133
123.996
120.572
120.200
49.789
49.575
49.363
49.150
48.936
48.725
48.511
25.210
15.096
250
200
150
100
50
0
-50
ppm (t1)

3d in DMSO-d_6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7.130
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7.076
3.336
2.917
2.898
2.880
2.861
2.500
1.278
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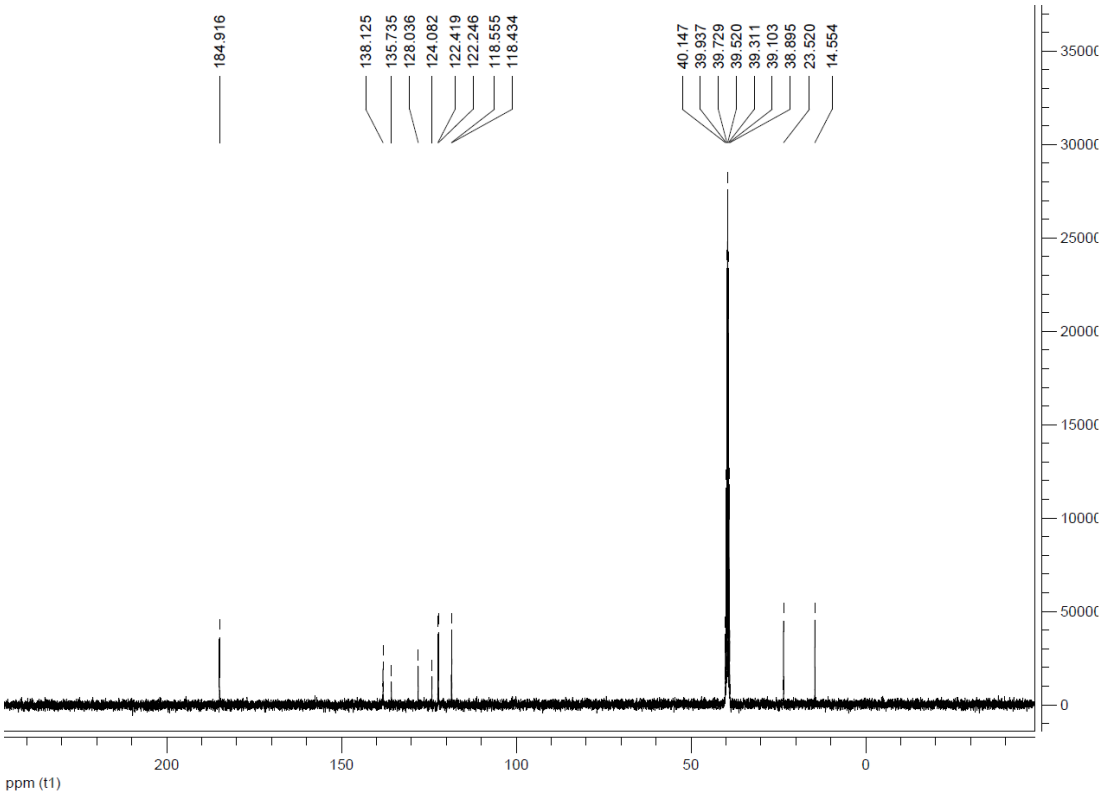
1.241
1.02
1.00
1.00
0.99
1.04
2.09
3.13
ppm (t1)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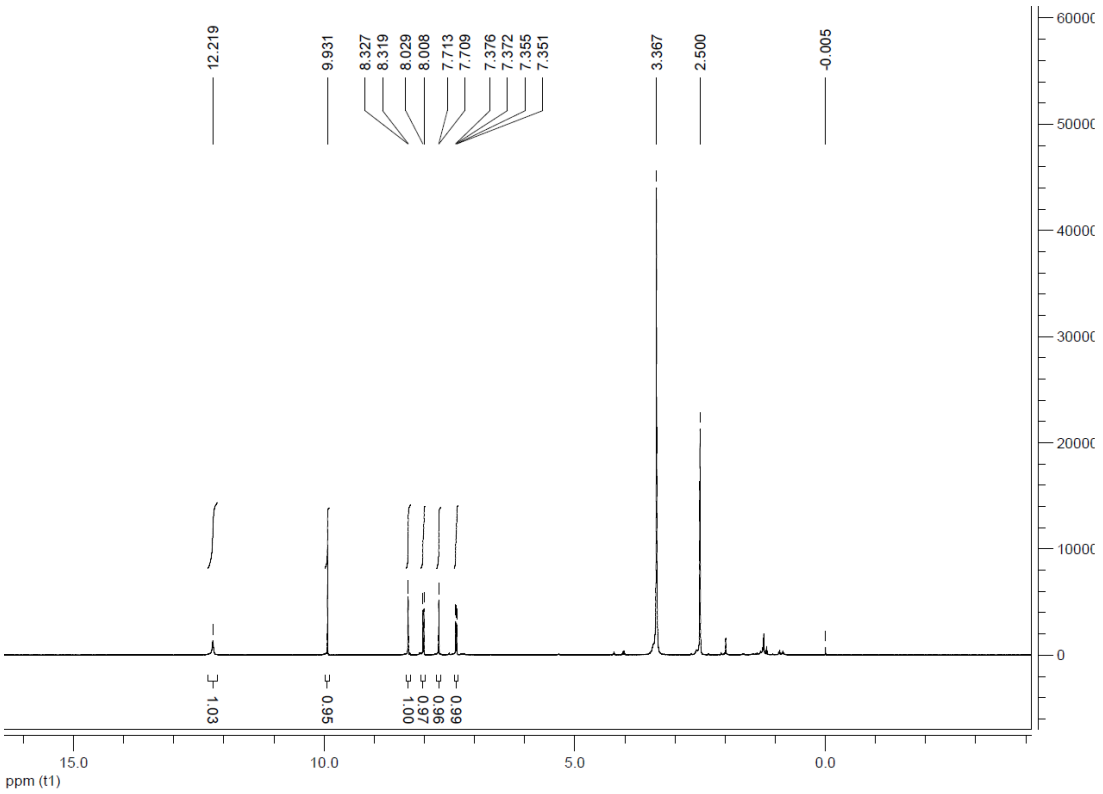
23.520
14.554
ppm (t1)

3e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8.008
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7.372
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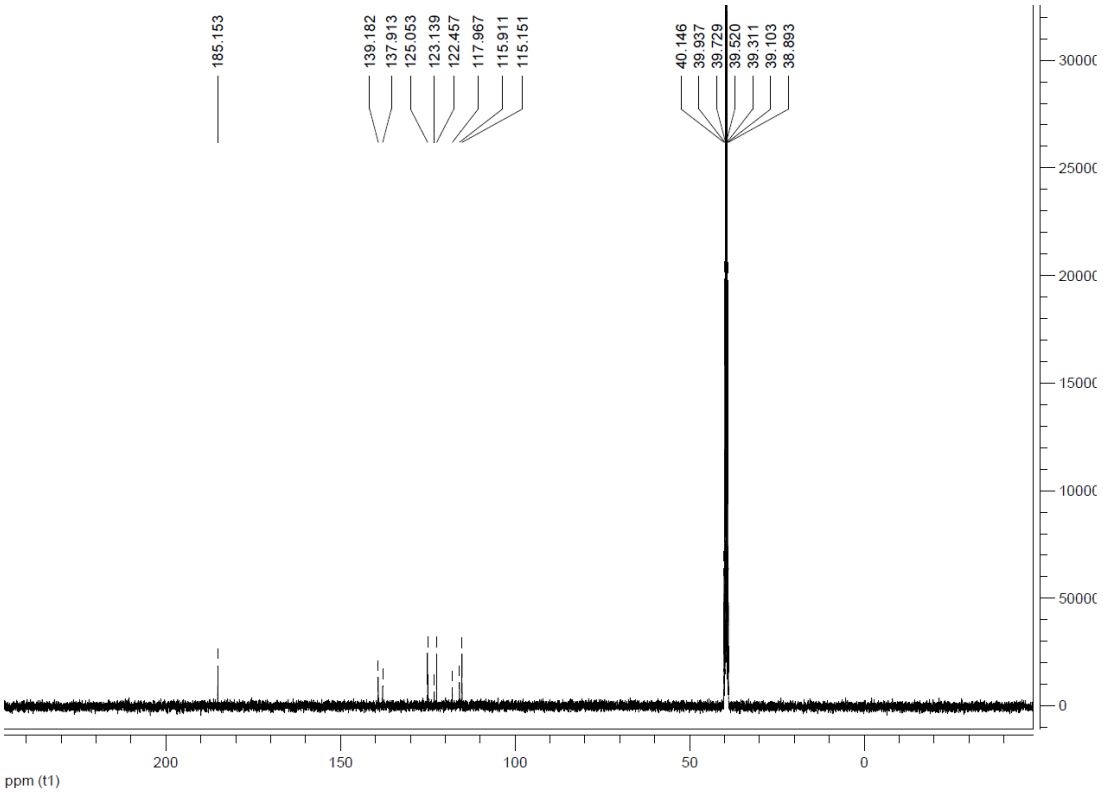
2.500
-0.005
1.03
0.96
1.00
0.97
0.96
0.99
ppm (t1)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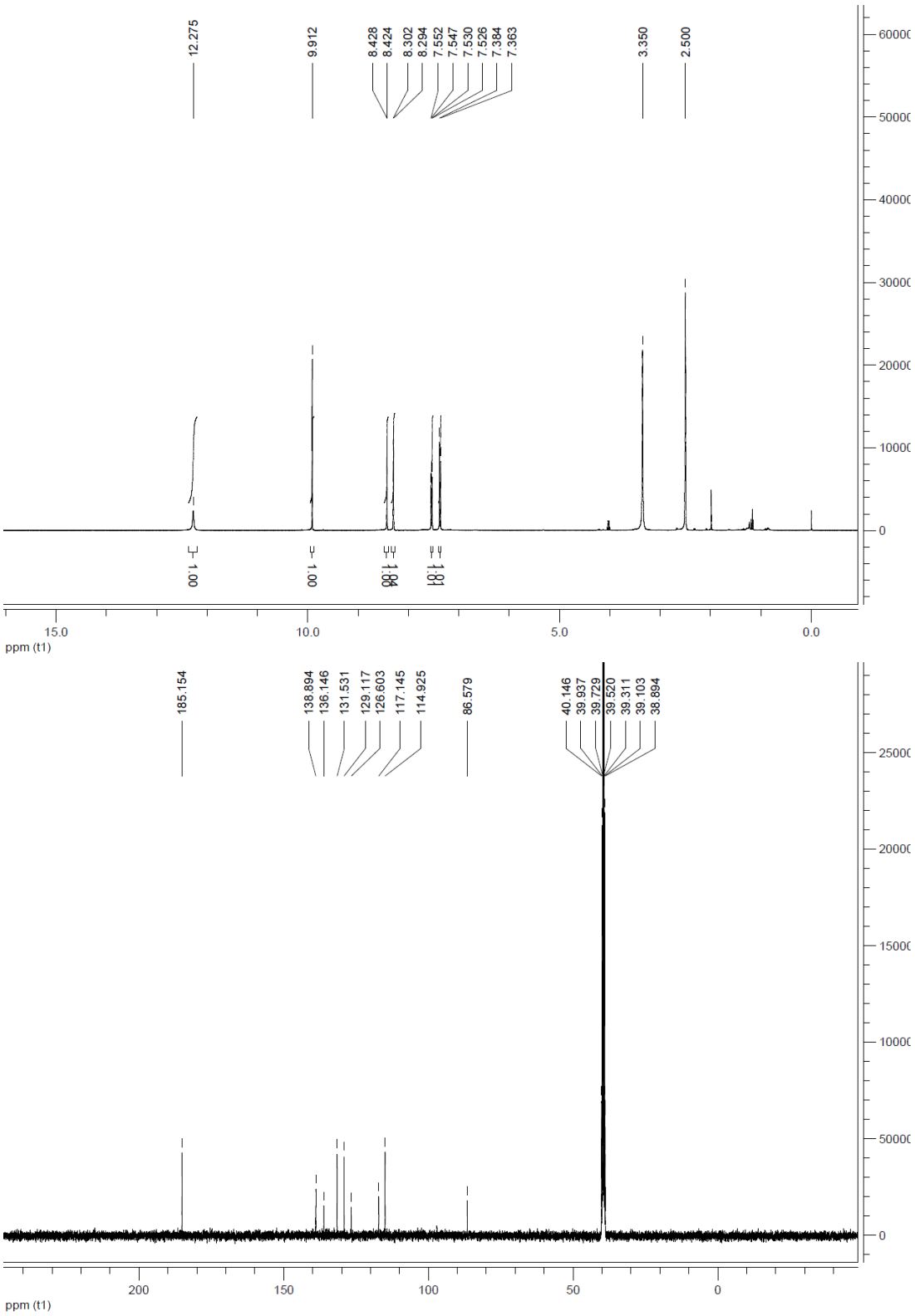
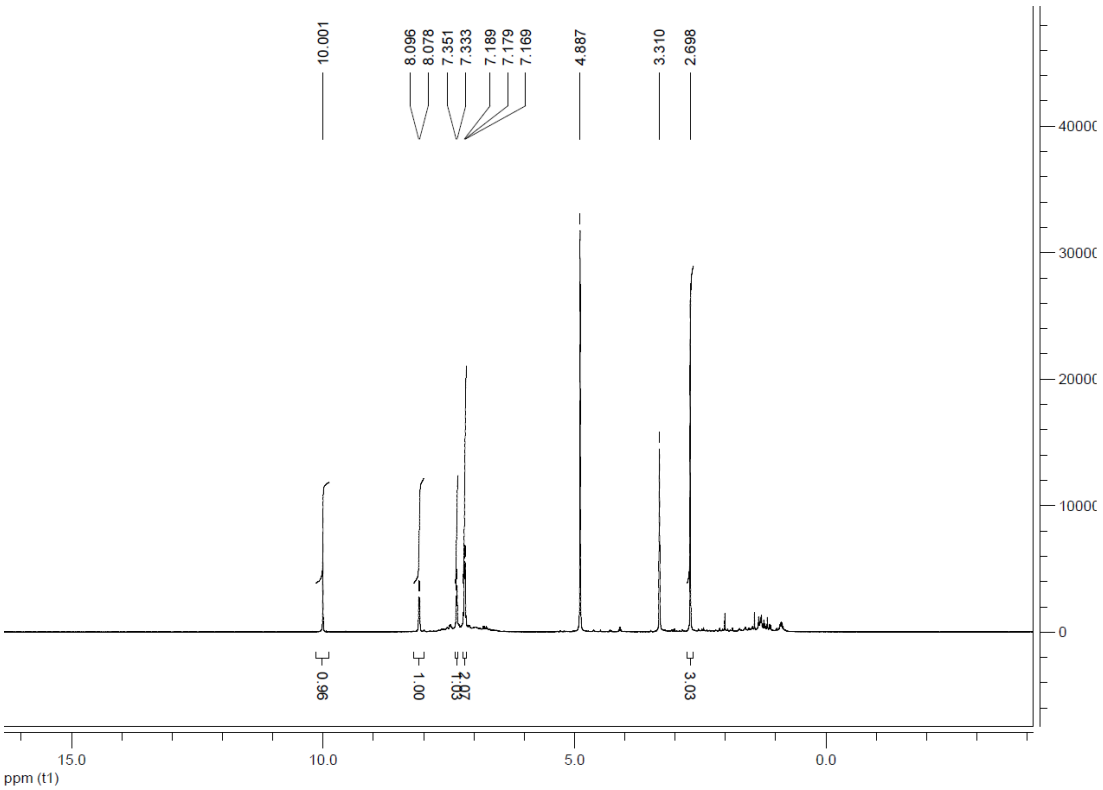
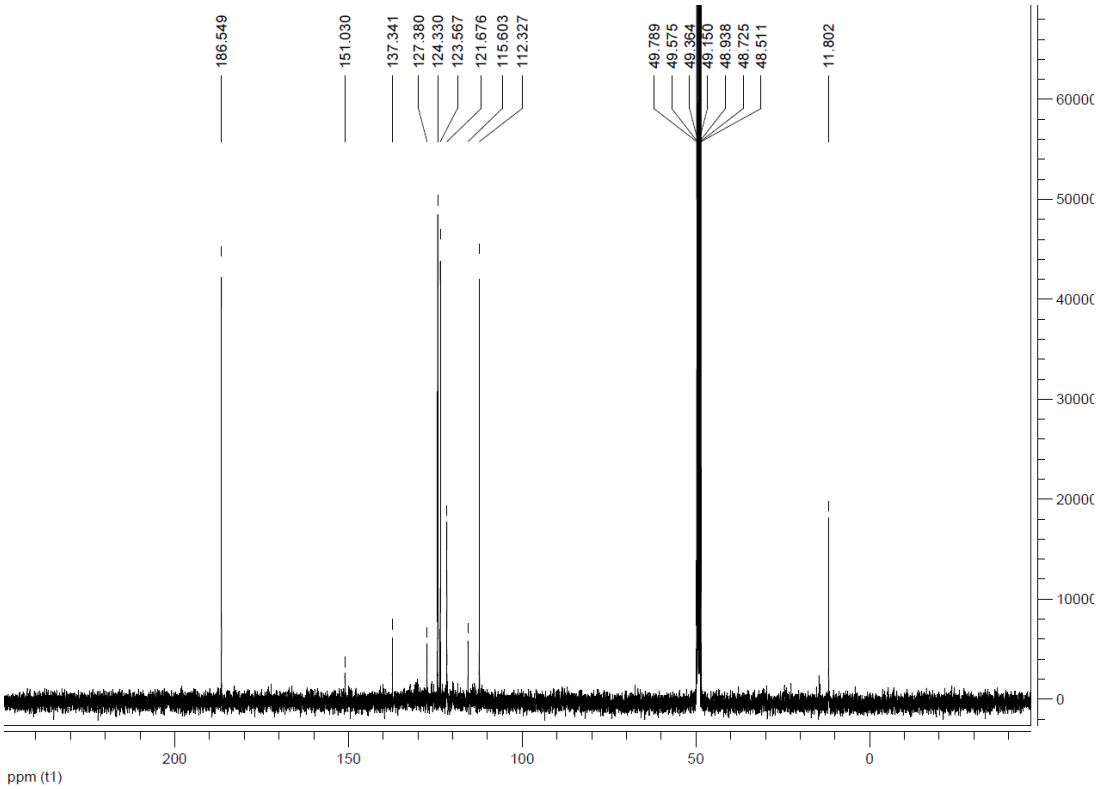
39.103
38.893
ppm (t1)

3f



3g in methanol-d_4


10.001
8.096
8.078
7.351
7.333
7.189
7.179
7.169
4.887
3.310
2.698
0.96
1.00
2.02
2.03
3.03
ppm (t1)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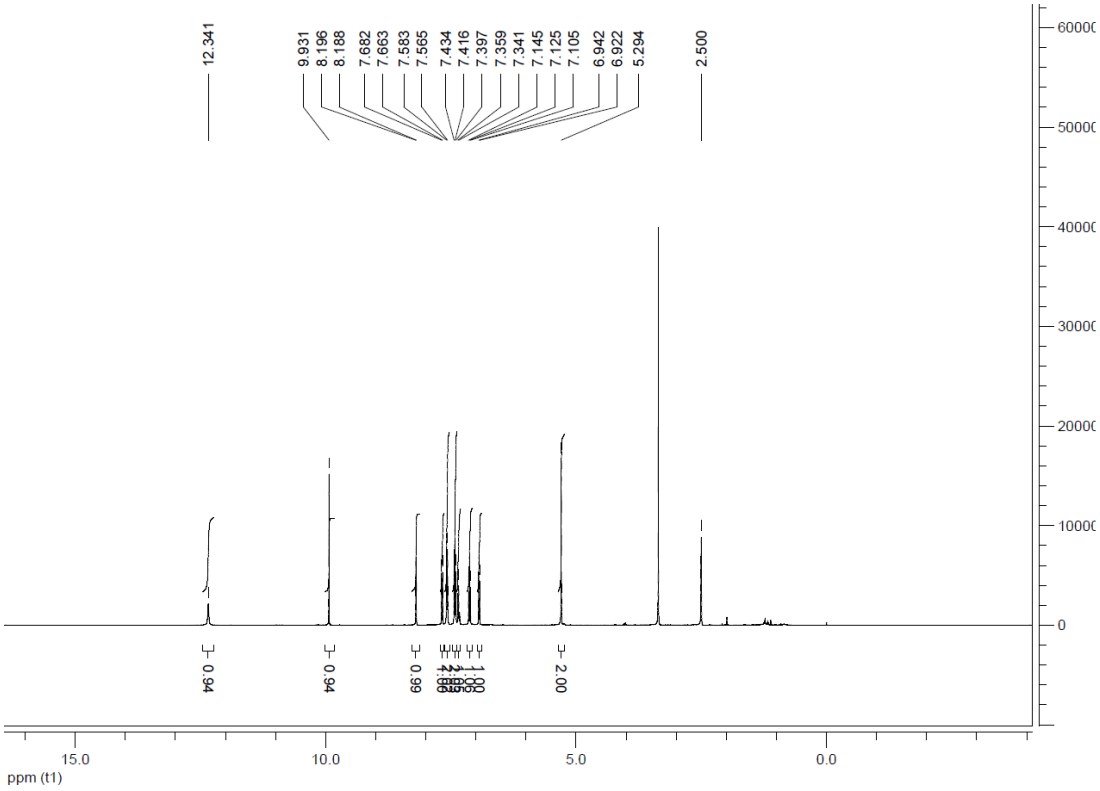
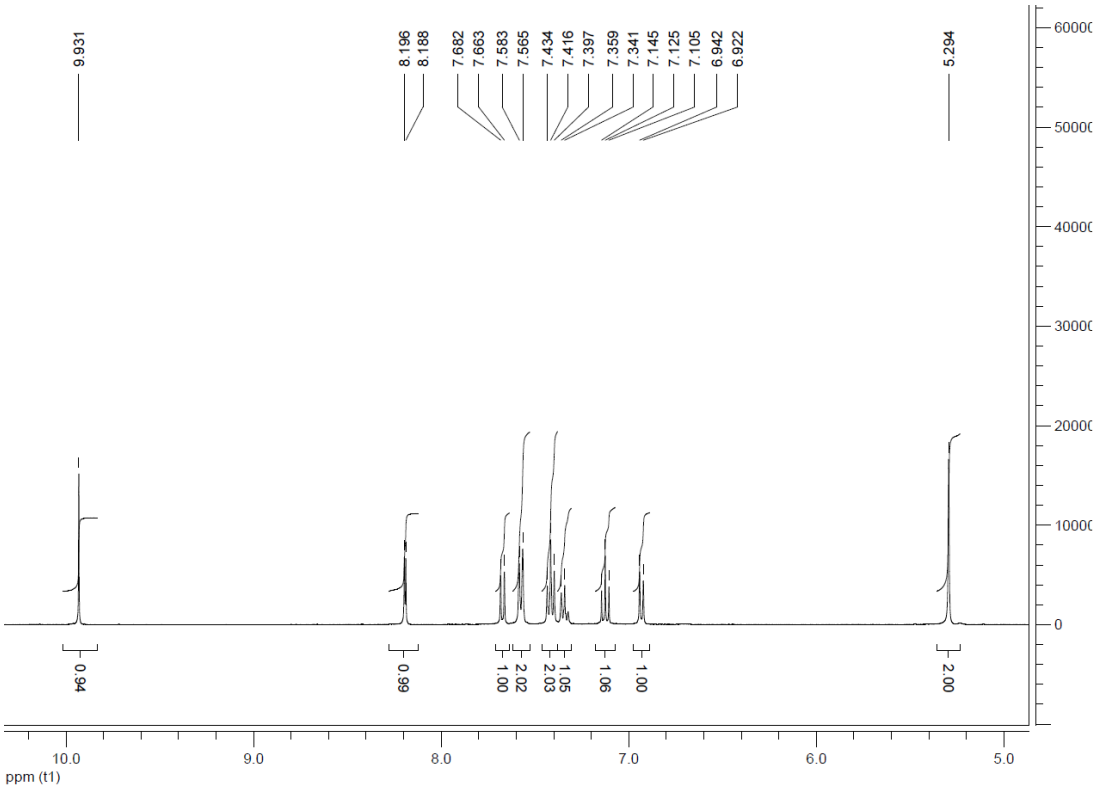
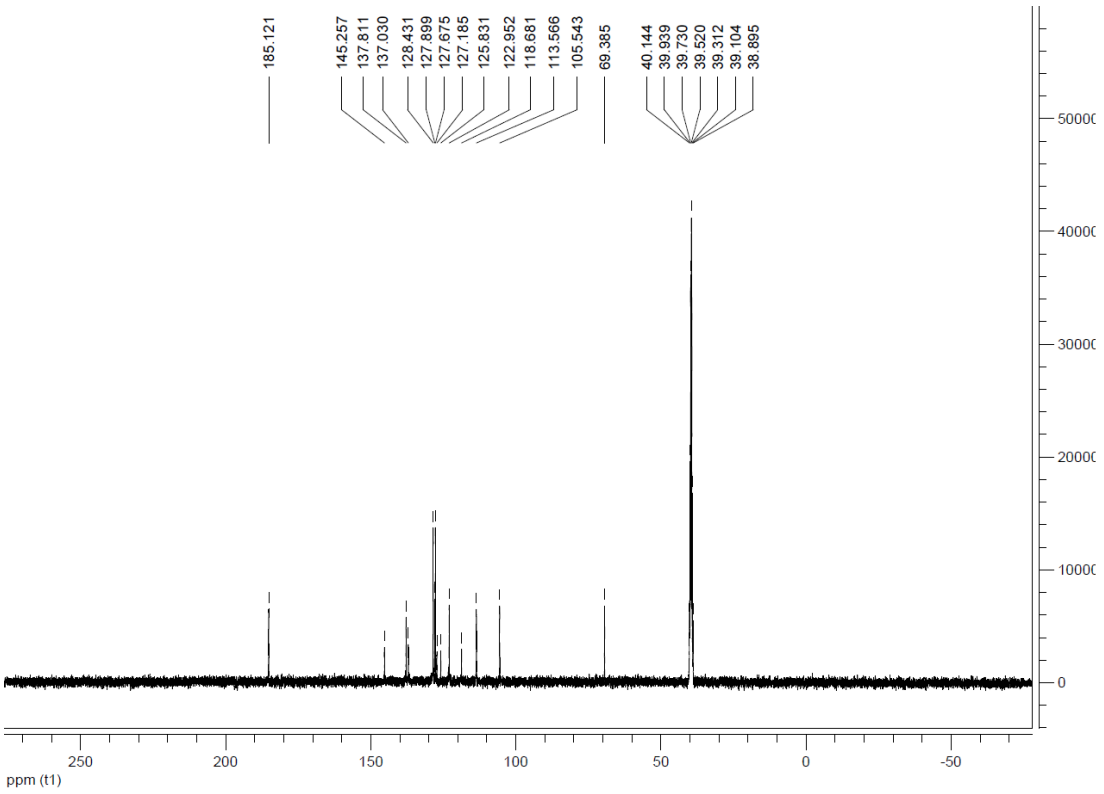
127.380
124.330
123.567
121.676
115.603
112.327
49.789
49.575
49.364
49.150
48.938
48.725
48.511
11.802
ppm (t1)

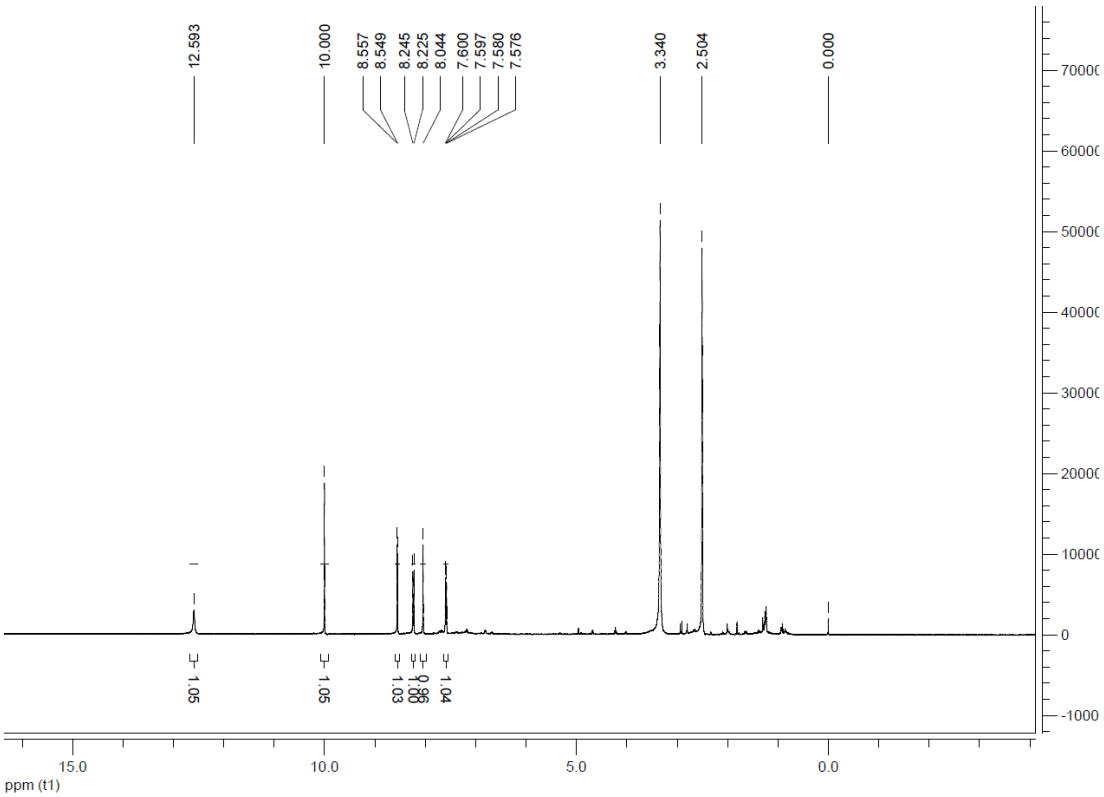
3h

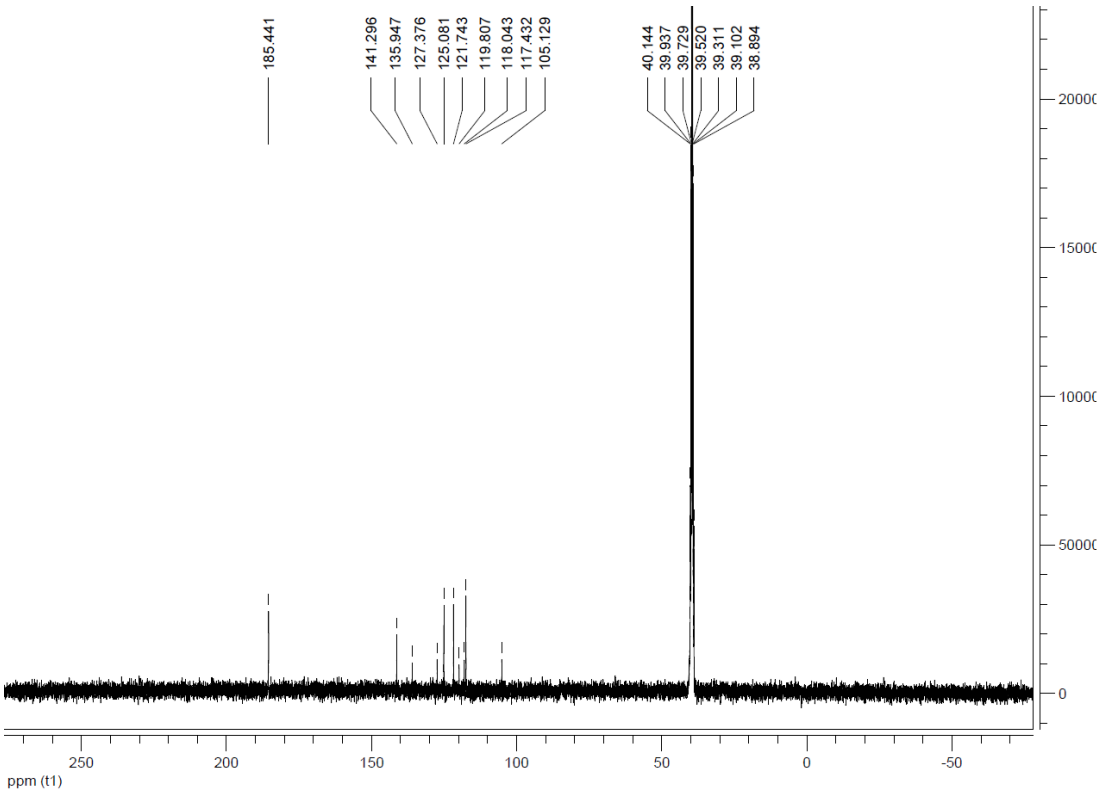

12.341
9.931
8.196
8.188
7.682
7.663
7.583
7.565
7.434
7.416
7.397
7.359
7.341
7.145
7.125
7.105
6.942
6.922
5.294
2.500
0.94
0.94
0.99
1.00
2.00
ppm (t1)


9.931
8.196
8.188
7.682
7.663
7.583
7.565
7.434
7.416
7.397
7.359
7.341
7.145
7.125
7.105
6.942
6.922
5.294
0.94
0.99
1.00
2.02
2.03
1.05
1.06
1.00
2.00
ppm (t1)

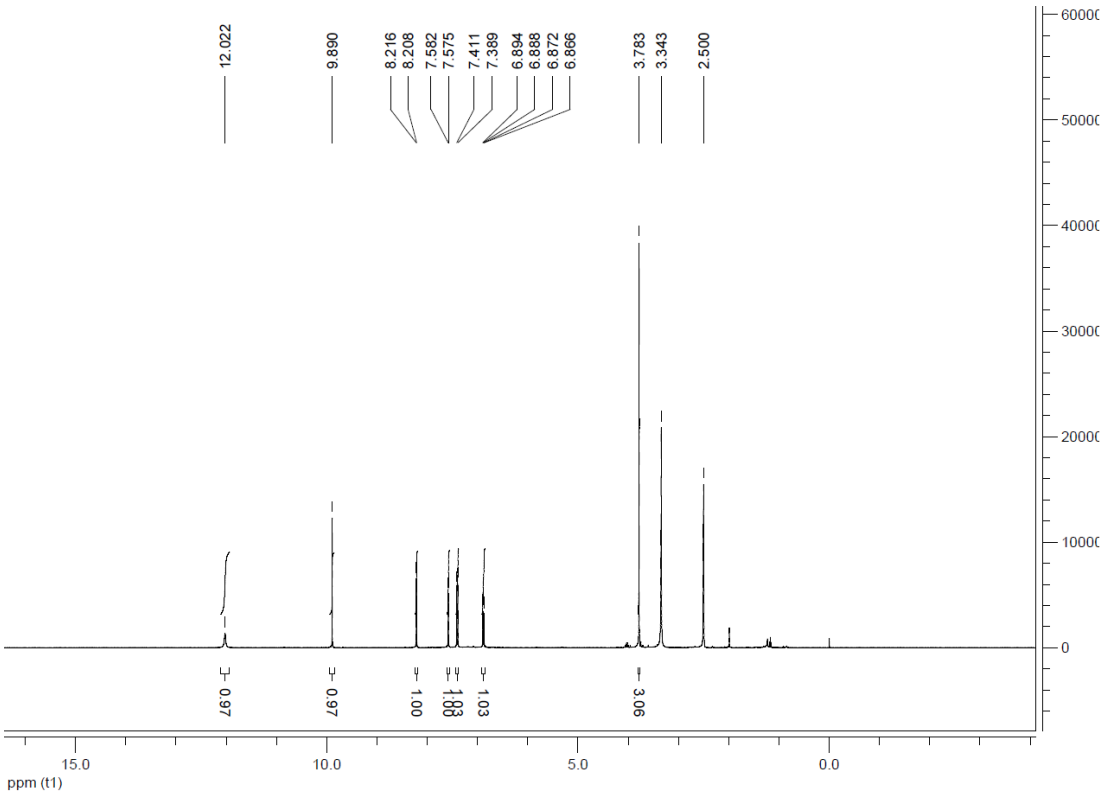

185.121
145.257
137.811
137.030
128.431
127.899
127.675
127.185
125.831
122.952
118.681
113.566
105.543
69.385
40.144
39.939
39.730
39.520
39.312
39.104
38.895
ppm (t1)

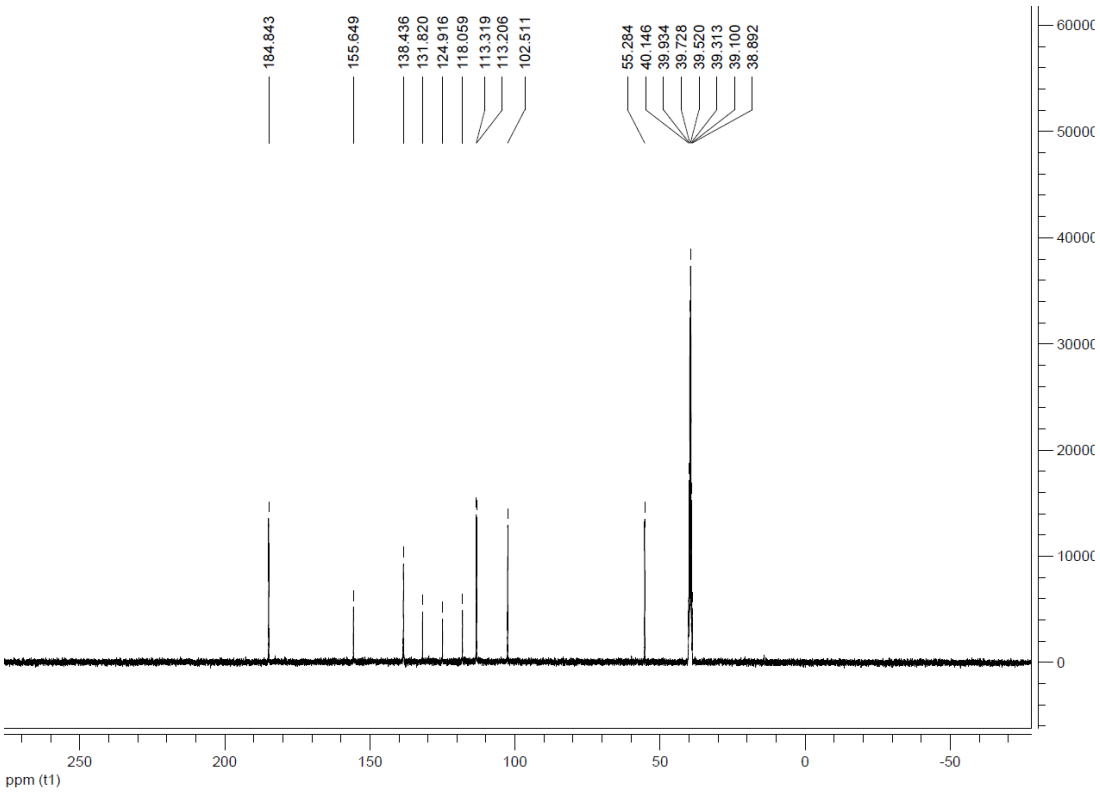
3i


12.593
10.000
8.557
8.549
8.245
8.225
8.044
7.600
7.597
7.580
7.576
3.340
2.504
0.000
1.05
1.00
1.03
0.98
0.98
1.04
ppm (t1)

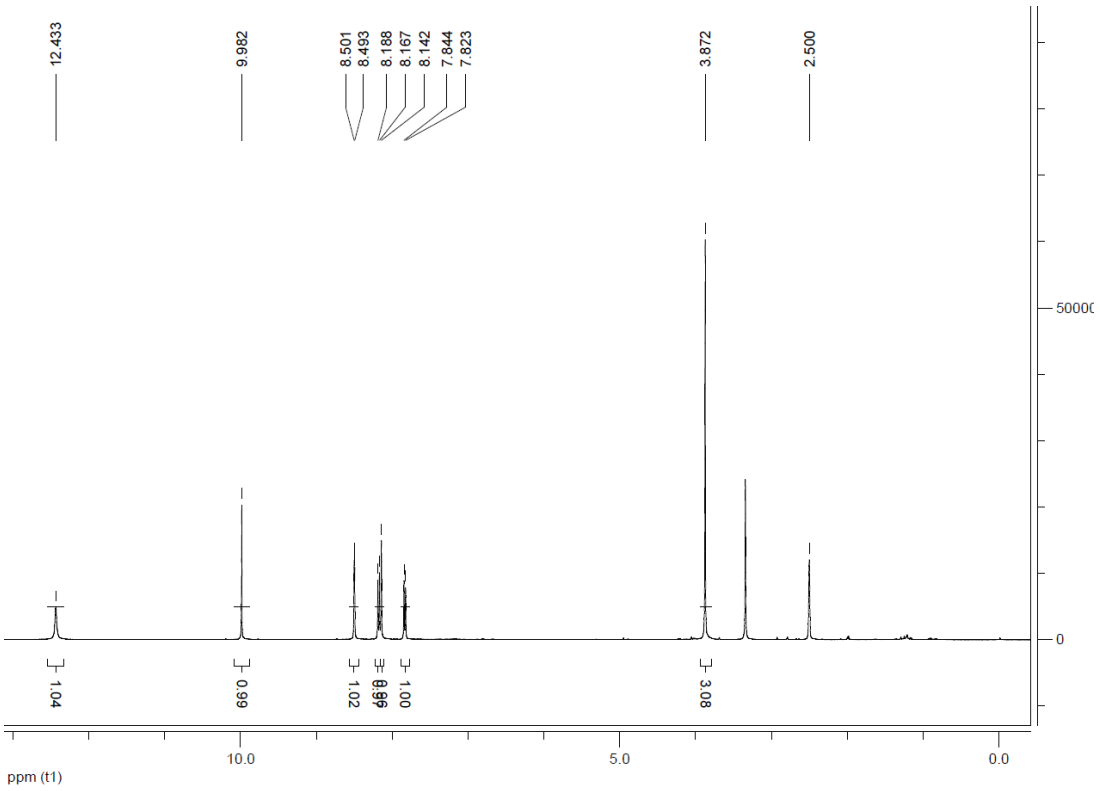

185.441
141.296
135.947
127.376
125.081
121.743
119.807
118.043
117.432
105.129
40.144
39.937
39.729
39.520
39.311
39.102
38.894
250
200
150
100
50
0
-50
ppm (t1)

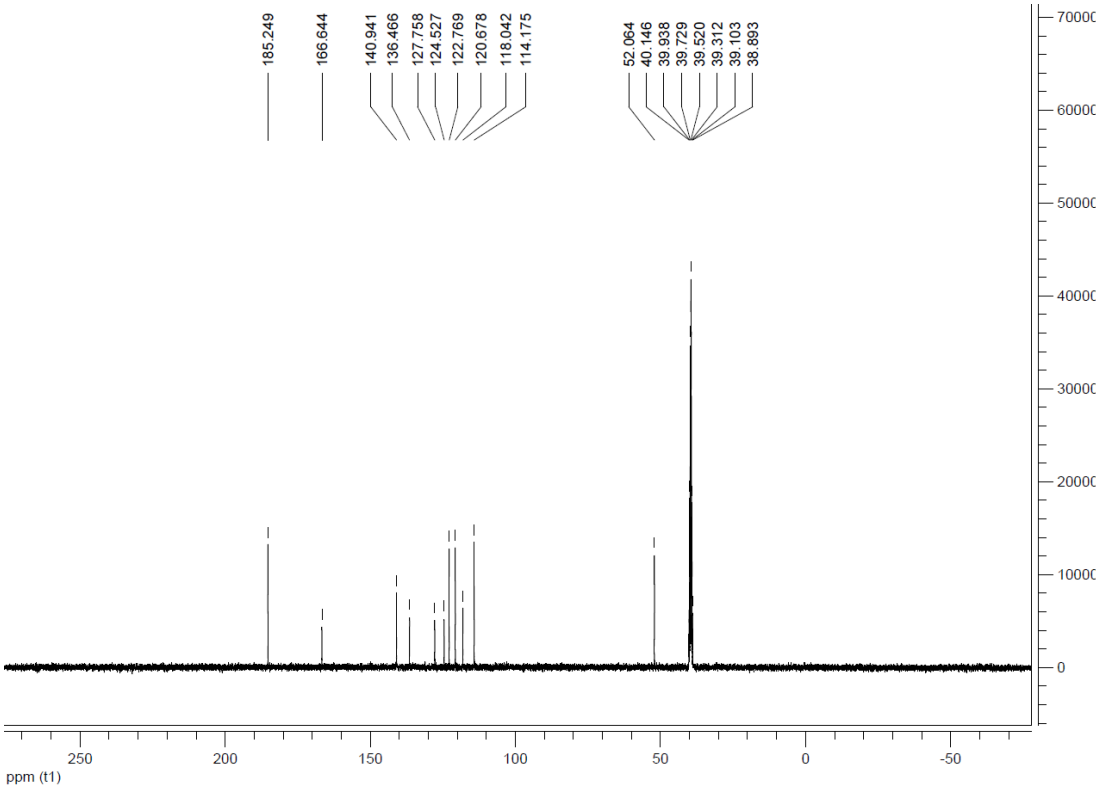
3j


12.022
9.890
8.216
8.208
7.582
7.575
7.411
7.389
6.894
6.888
6.872
6.866
3.783
3.343
2.500
0.97
0.97
1.00
1.03
3.06
15.0
10.0
5.0
0.0
ppm (t1)

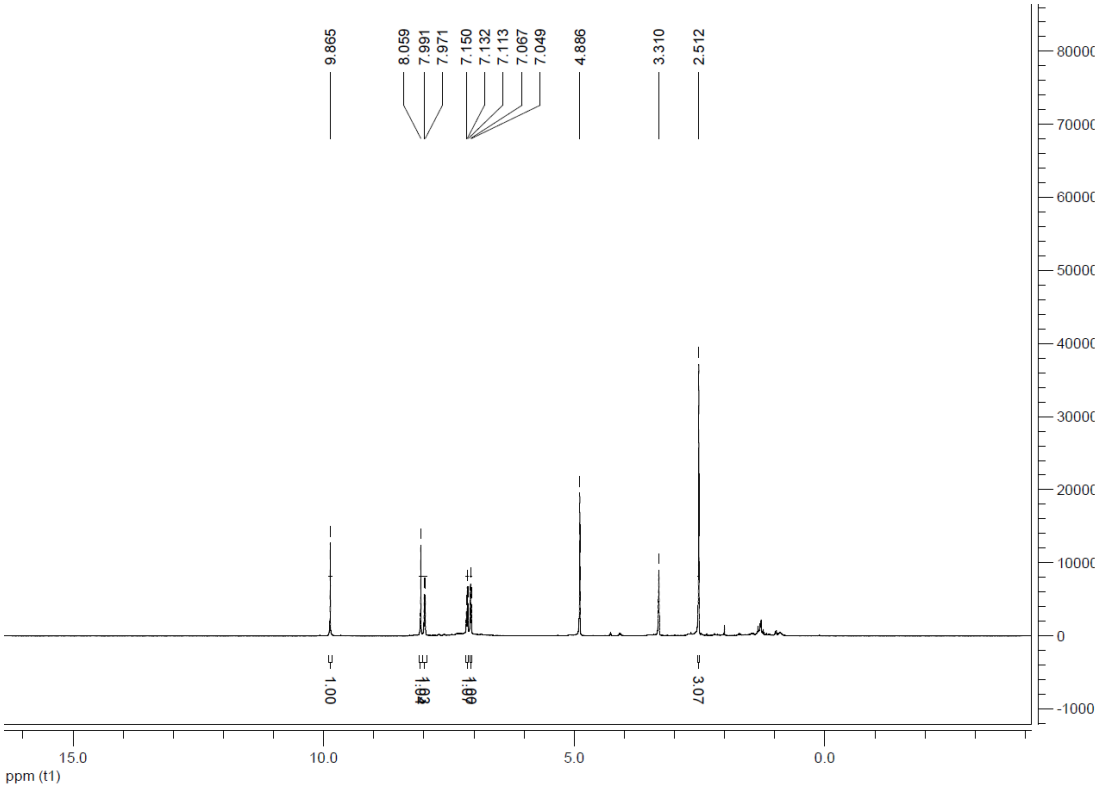

184.843
155.649
138.436
131.820
124.916
118.059
113.319
113.206
102.511
55.284
40.146
39.934
39.728
39.520
39.313
39.100
38.892
60000
50000
40000
30000
20000
10000
0
250
200
150
100
50
0
-50
ppm (t1)

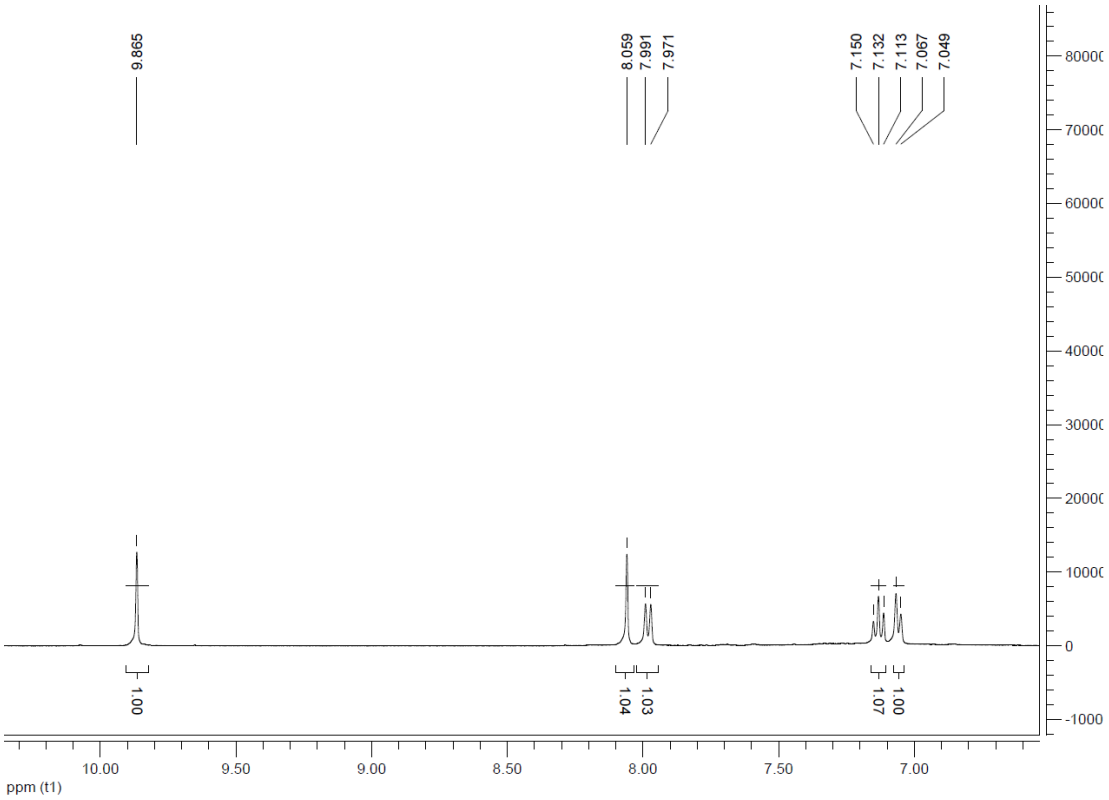
3k

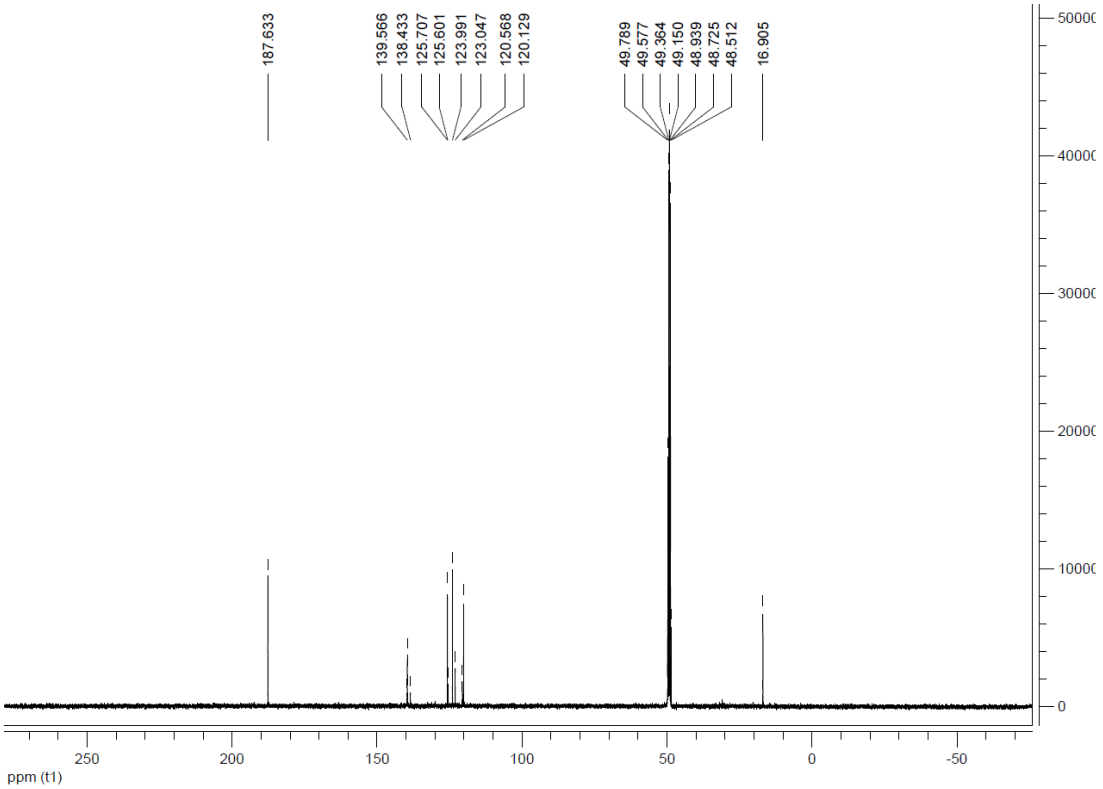

12.433
9.982
8.501
8.493
8.188
8.167
8.142
7.844
7.823
3.872
2.500
50000
0
1.04
0.99
1.02
1.00
3.08
10.0
5.0
0.0
ppm (t1)



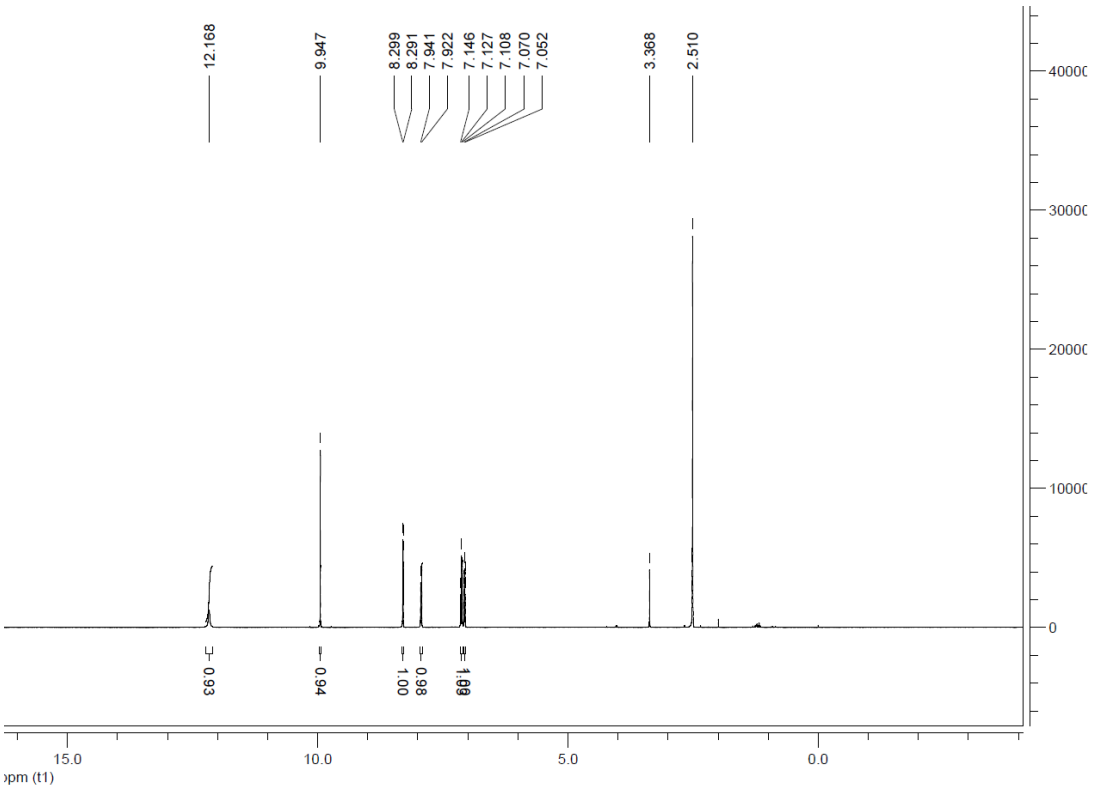
3l in methanol-d_4

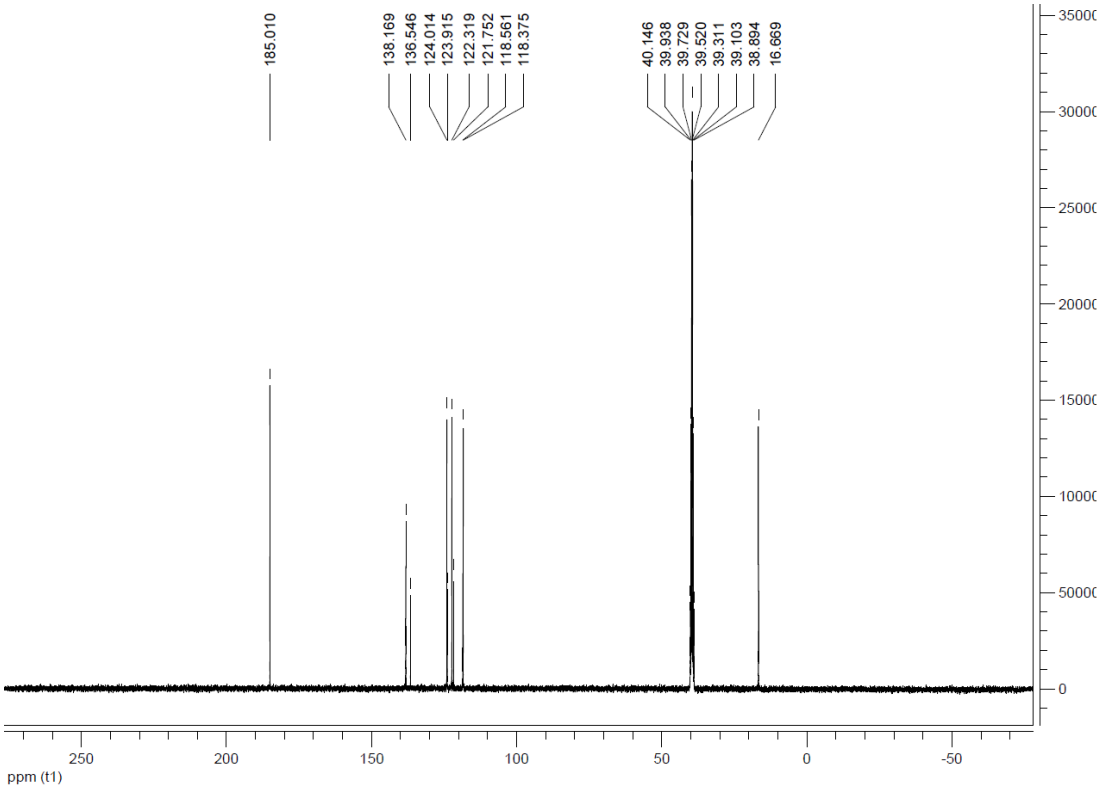



9.865
8.059
7.991
7.971
7.150
7.132
7.113
7.067
7.049
1.00
1.04
1.03
1.07
1.00
10.00
9.50
9.00
8.50
8.00
7.50
7.00
ppm (t1)

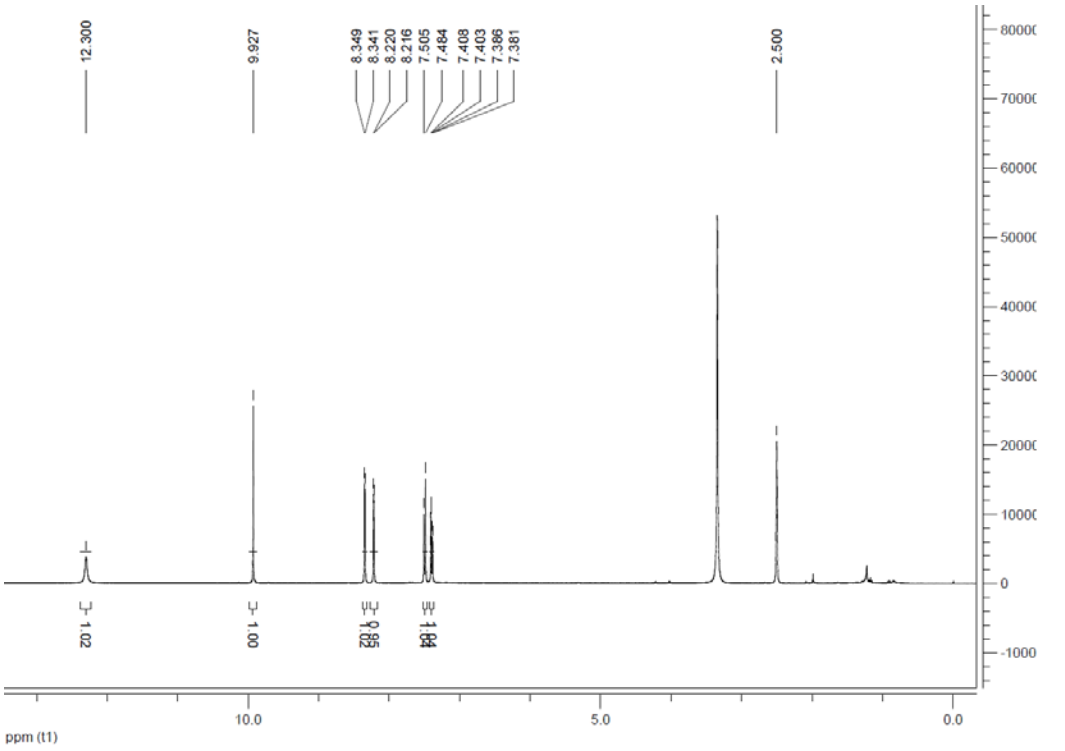

187.633
139.566
138.433
125.707
125.601
123.991
123.047
120.568
120.129
49.789
49.577
49.364
49.150
48.939
48.725
48.512
16.905
250
200
150
100
50
0
-50
ppm (t1)

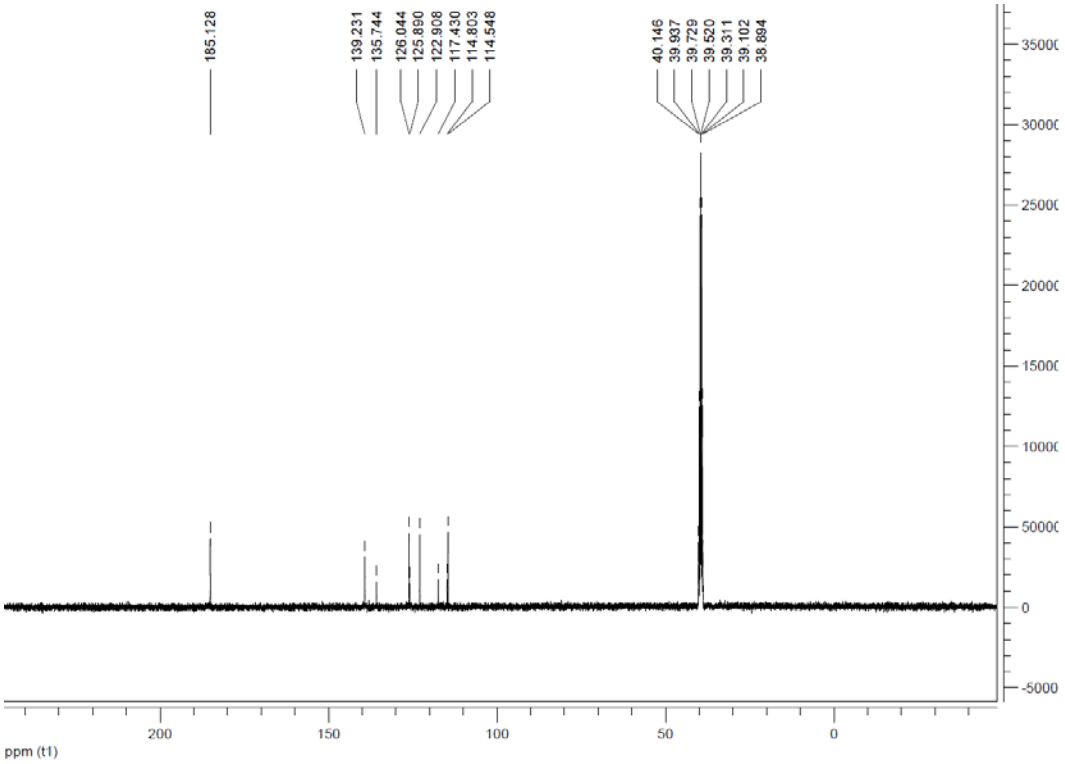
3l in DMSO-d_6


12.168
9.947
8.299
8.291
7.941
7.922
7.146
7.127
7.108
7.070
7.052
3.368
2.510
0.93
0.94
1.00
0.98
1.00
1.00
15.0
10.0
5.0
0.0
ppm (t1)

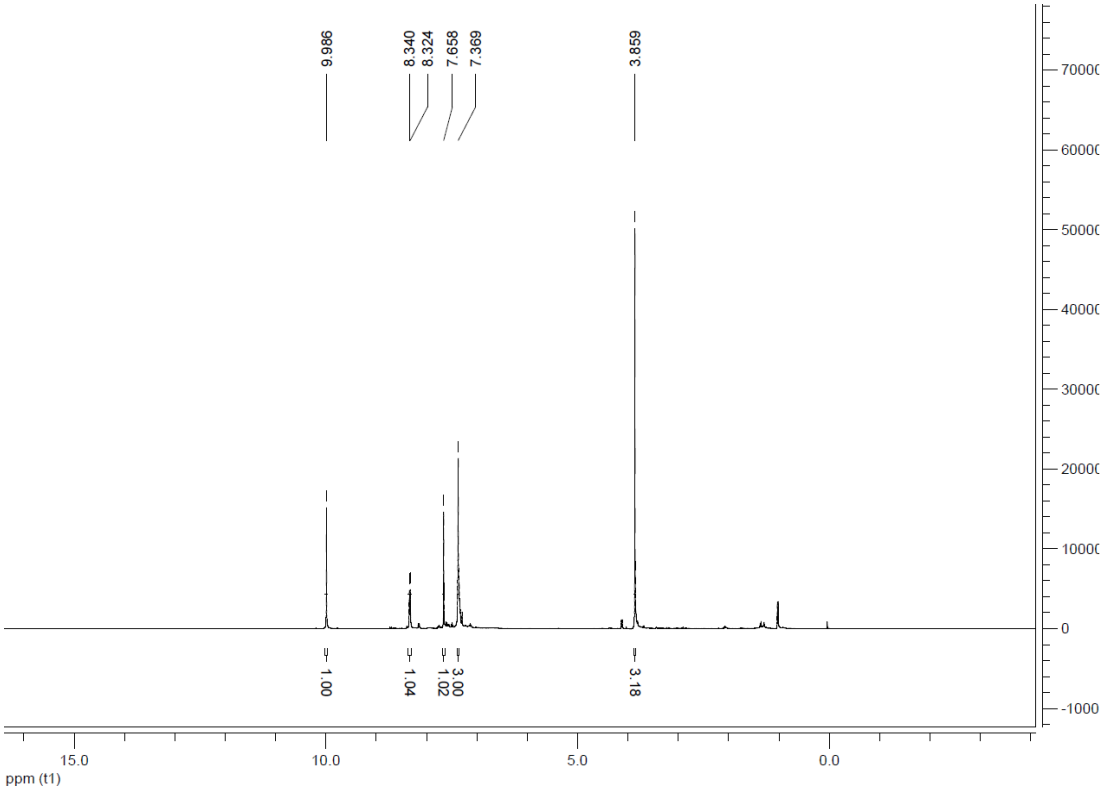

185.010
138.169
136.546
124.014
123.915
122.319
121.752
118.561
118.375
40.146
39.938
39.729
39.520
39.311
39.103
38.894
16.669
250
200
150
100
50
0
-50
ppm (t1)

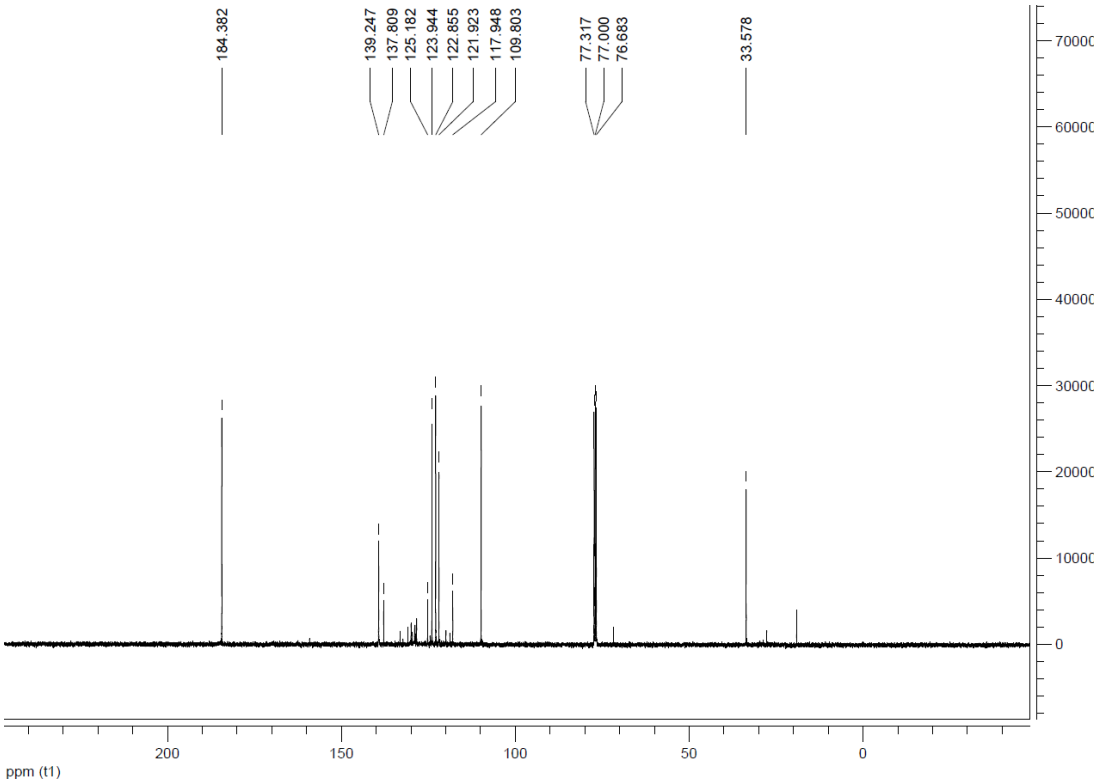
3m


12.300
9.927
8.349
8.341
8.220
8.216
7.505
7.484
7.408
7.403
7.386
7.381
2.500
1.02
1.00
2.02
1.84
10.0
5.0
0.0
ppm (t1)

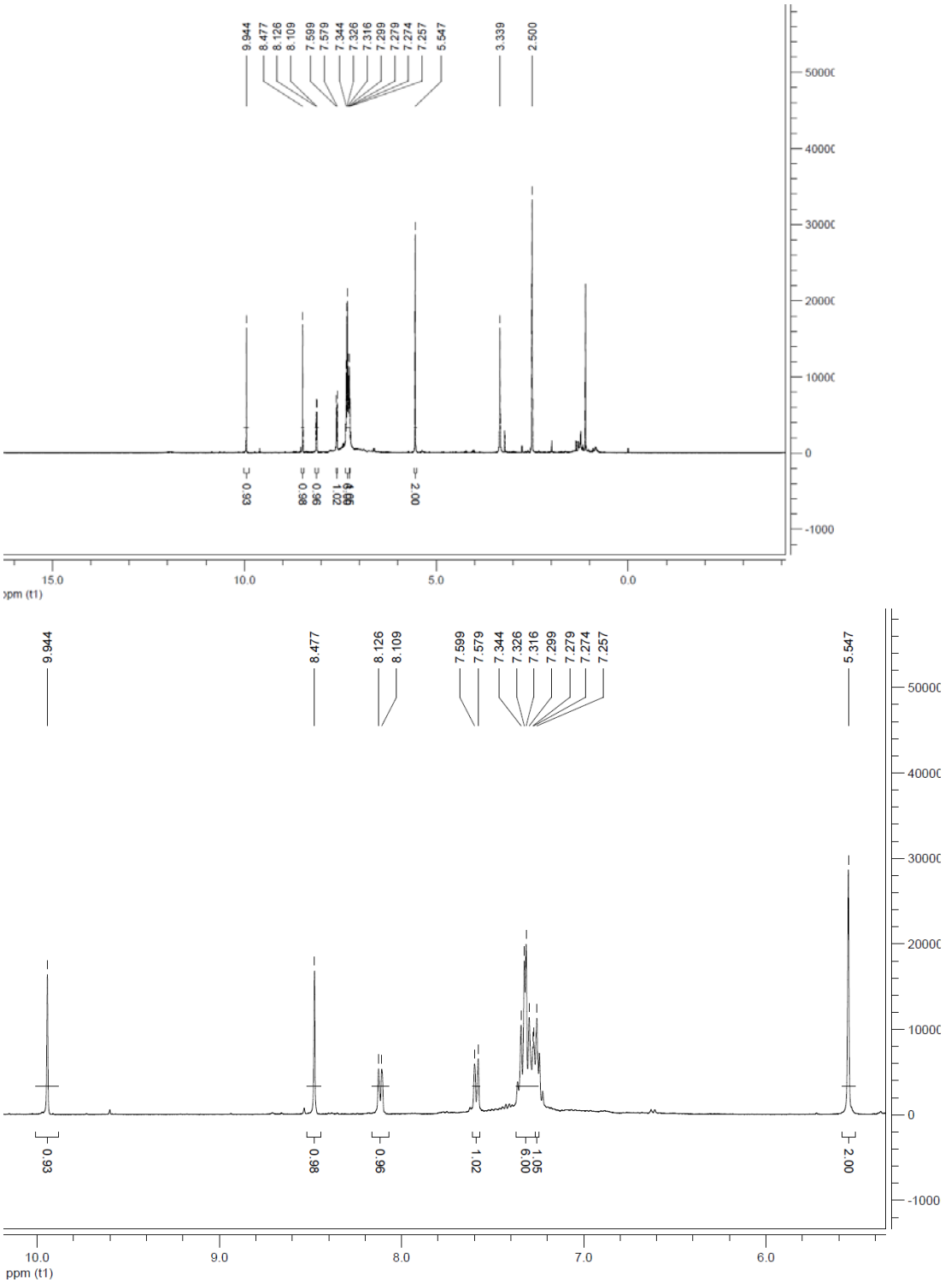

185.128
139.231
135.744
126.044
125.890
122.908
117.430
114.803
114.548
40.146
39.937
39.729
39.520
39.311
39.102
38.894
200
150
100
50
0
ppm (t1)

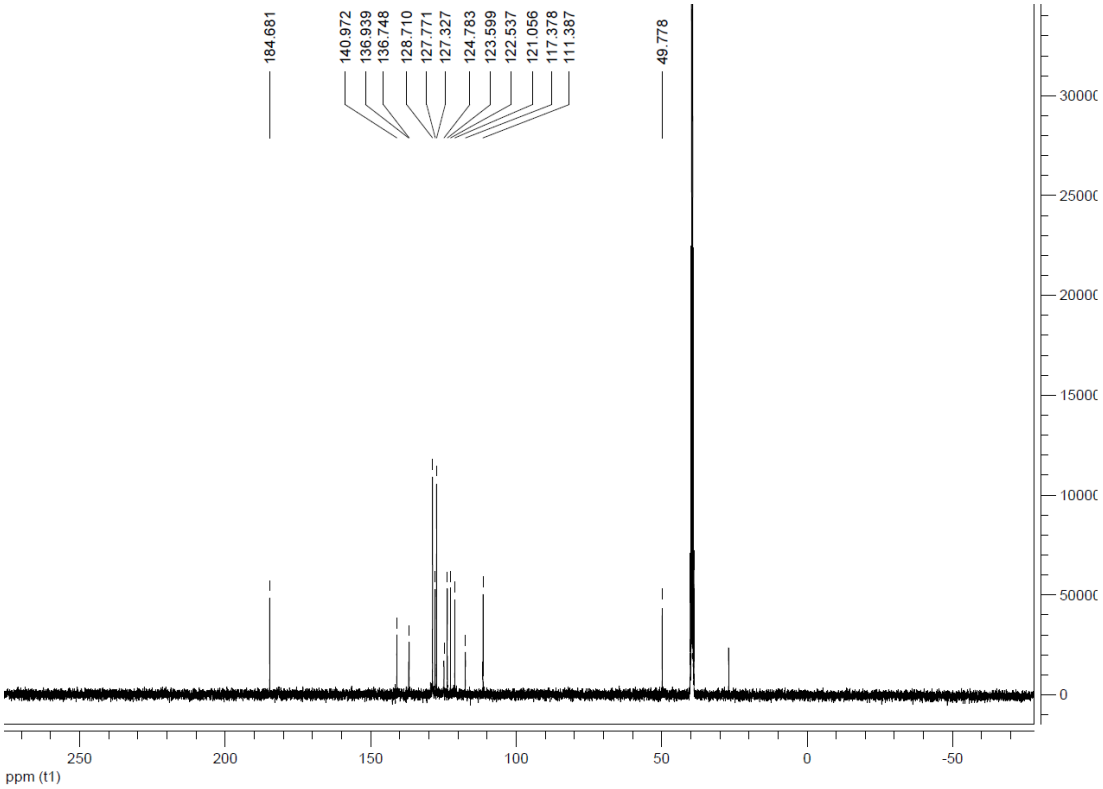
3n


9.986
8.340
8.324
7.658
7.369
3.859
1.00
1.04
1.20
3.00
3.18
ppm (t1)

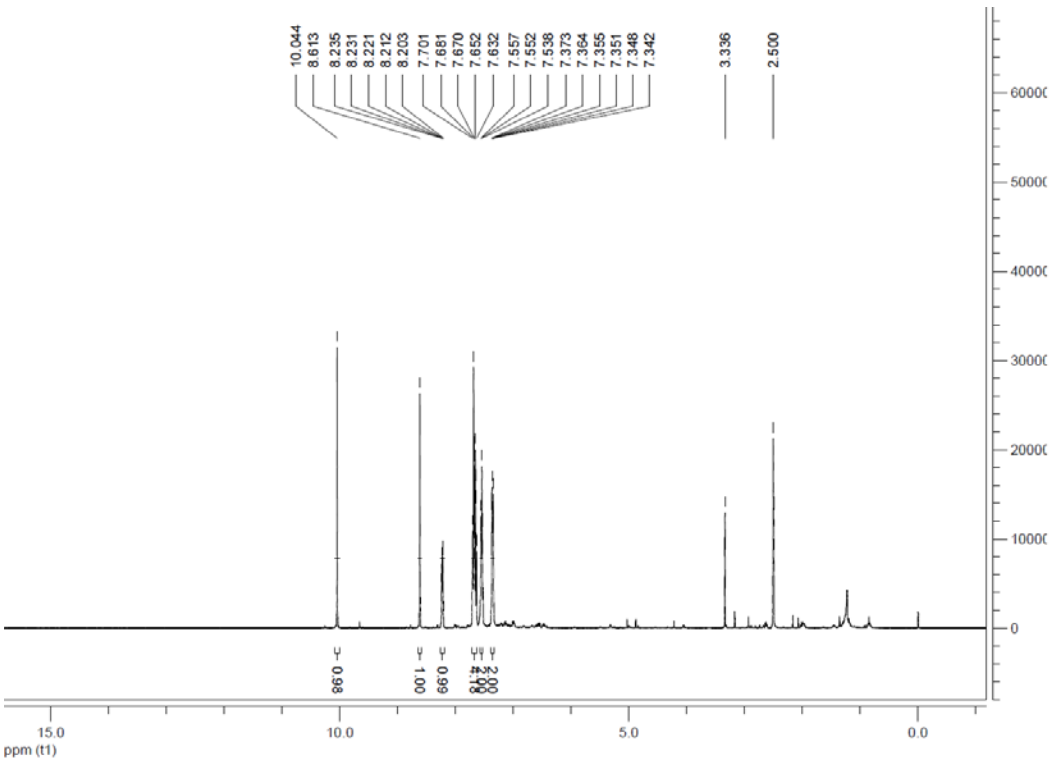

184.382
139.247
137.809
125.182
123.944
122.855
121.923
117.948
109.803
77.317
77.000
76.683
33.578
ppm (t1)

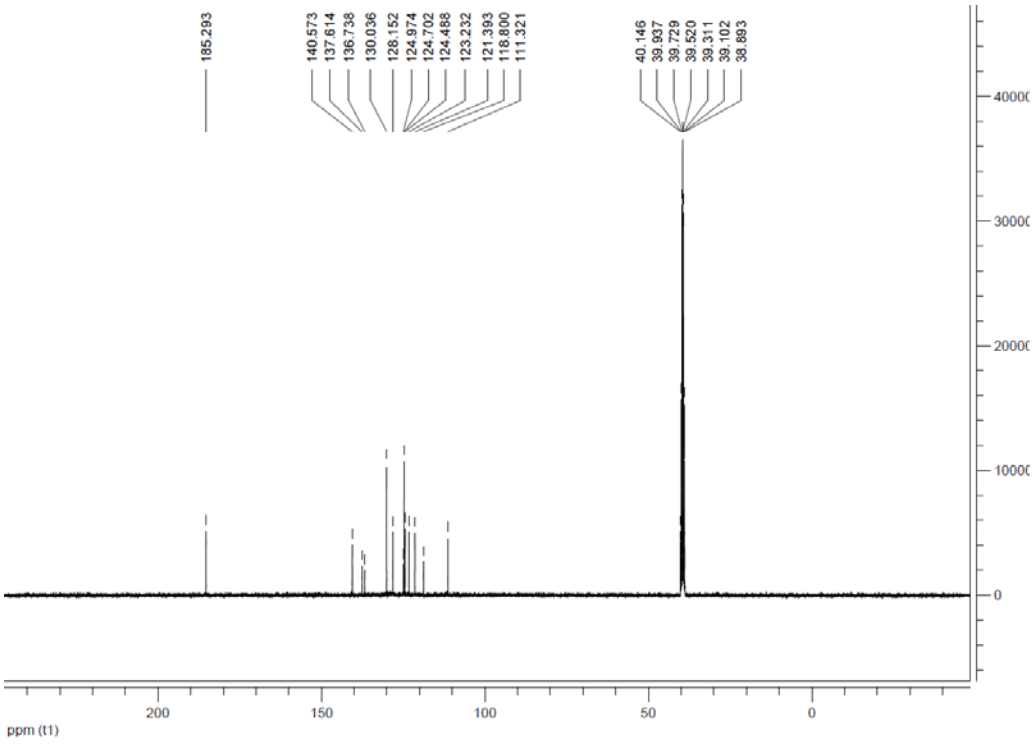
3o


9.944
8.477
8.126
8.109
7.599
7.579
7.344
7.326
7.316
7.299
7.279
7.274
7.257
5.547
3.339
2.500
0.93
0.98
0.96
1.02
6.00
1.05
2.00
ppm (t1)

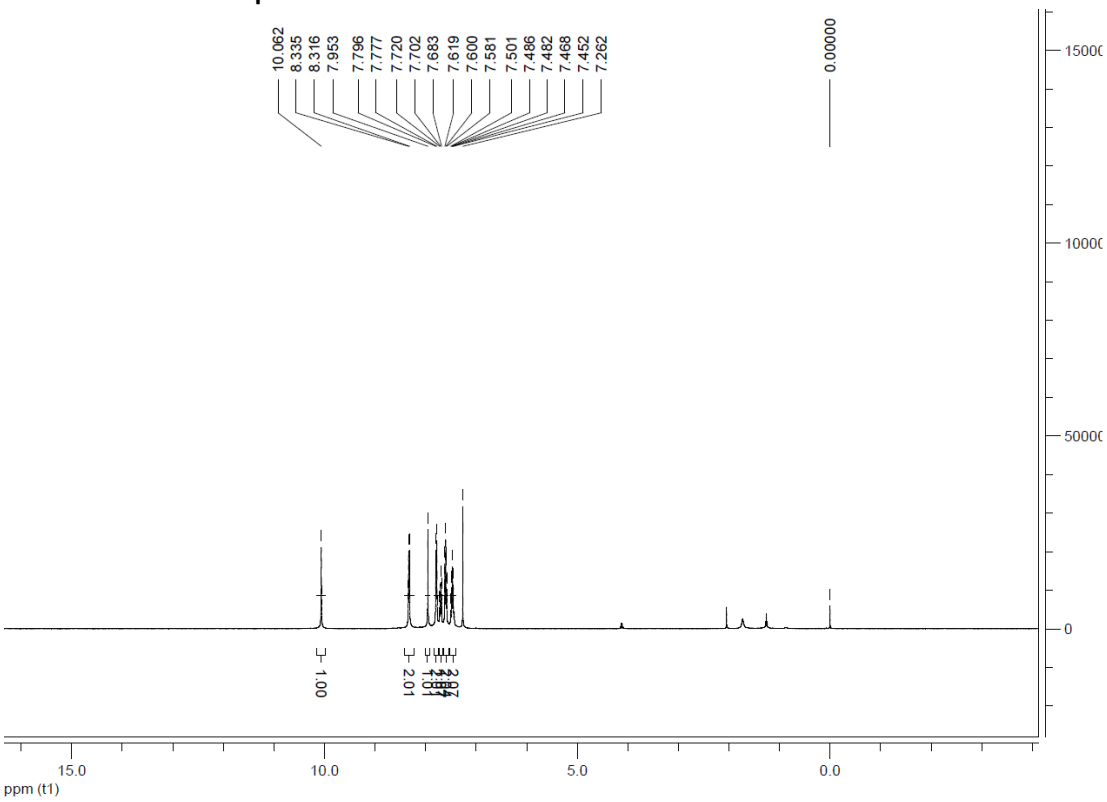

184.681
140.972
136.939
136.748
128.710
127.771
127.327
124.783
123.599
122.537
121.056
117.378
111.387
49.778
ppm (t1)

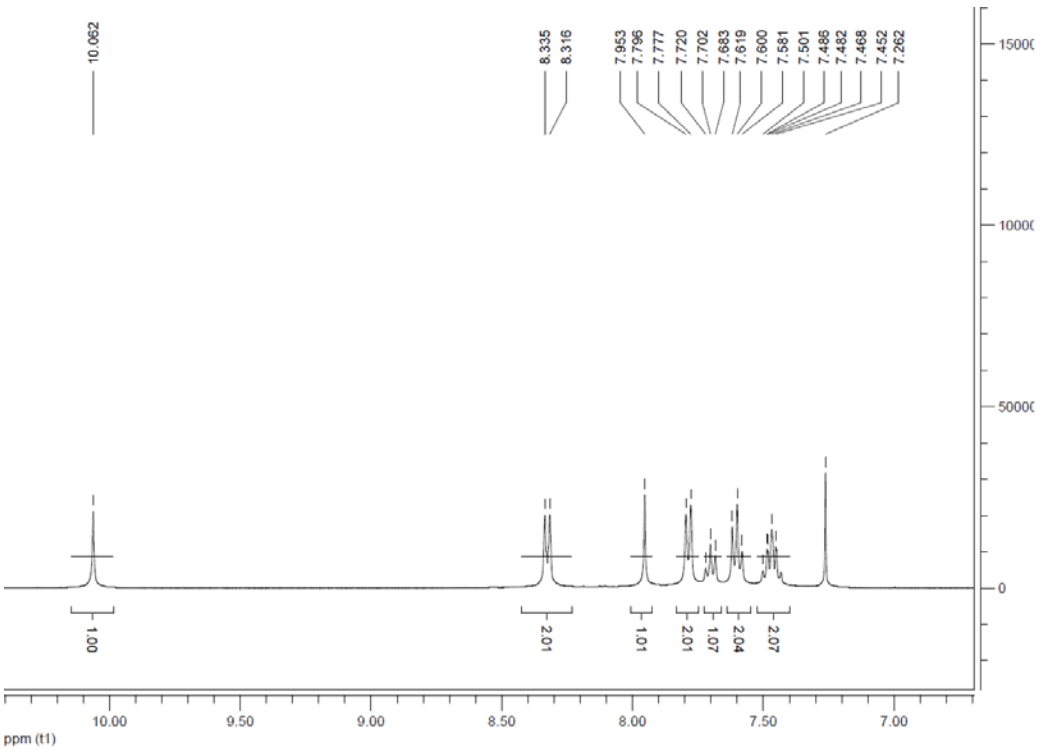
3p

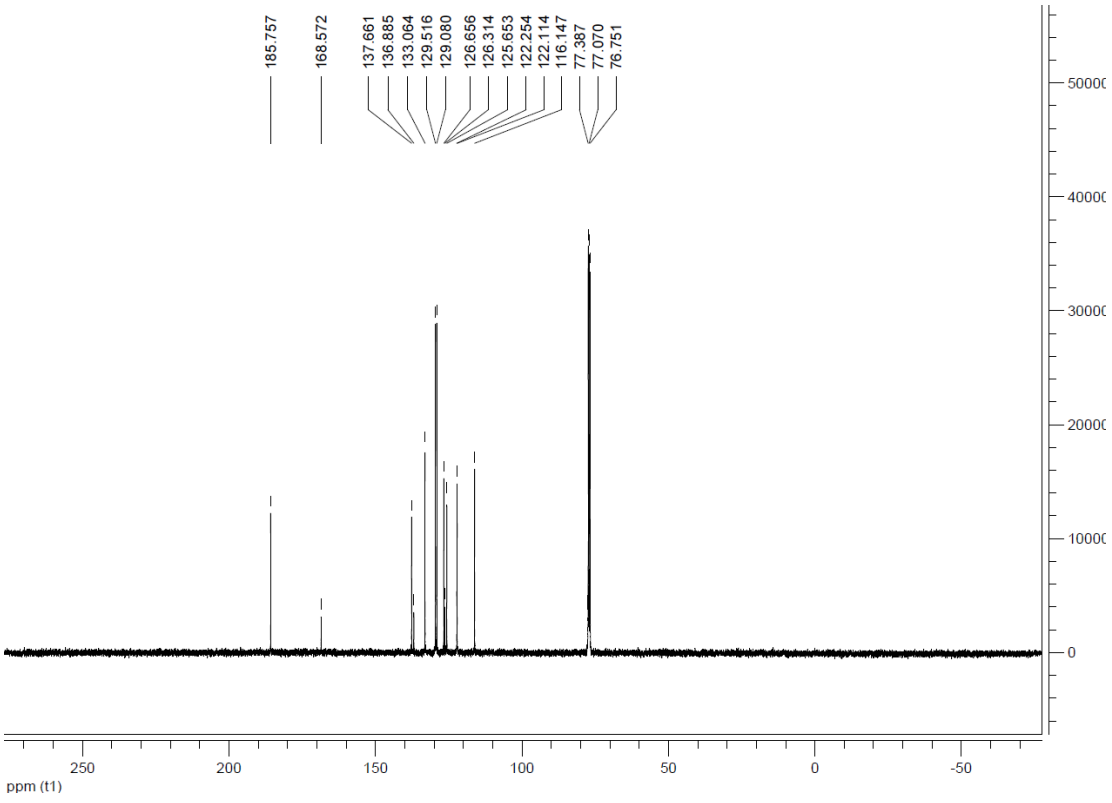

10.044
8.613
8.235
8.231
8.221
8.212
8.203
7.701
7.681
7.670
7.652
7.632
7.557
7.552
7.538
7.373
7.364
7.355
7.351
7.348
7.342
3.336
2.500
0.98
1.00
0.99
2.08
2.00
ppm (t1)


185.293
140.573
137.614
136.738
130.036
128.152
124.974
124.702
124.488
123.232
121.393
118.800
111.321
40.146
39.937
39.729
39.520
39.311
39.102
38.893
4000
3000
2000
1000
0
200
150
100
50
0
ppm (t1)

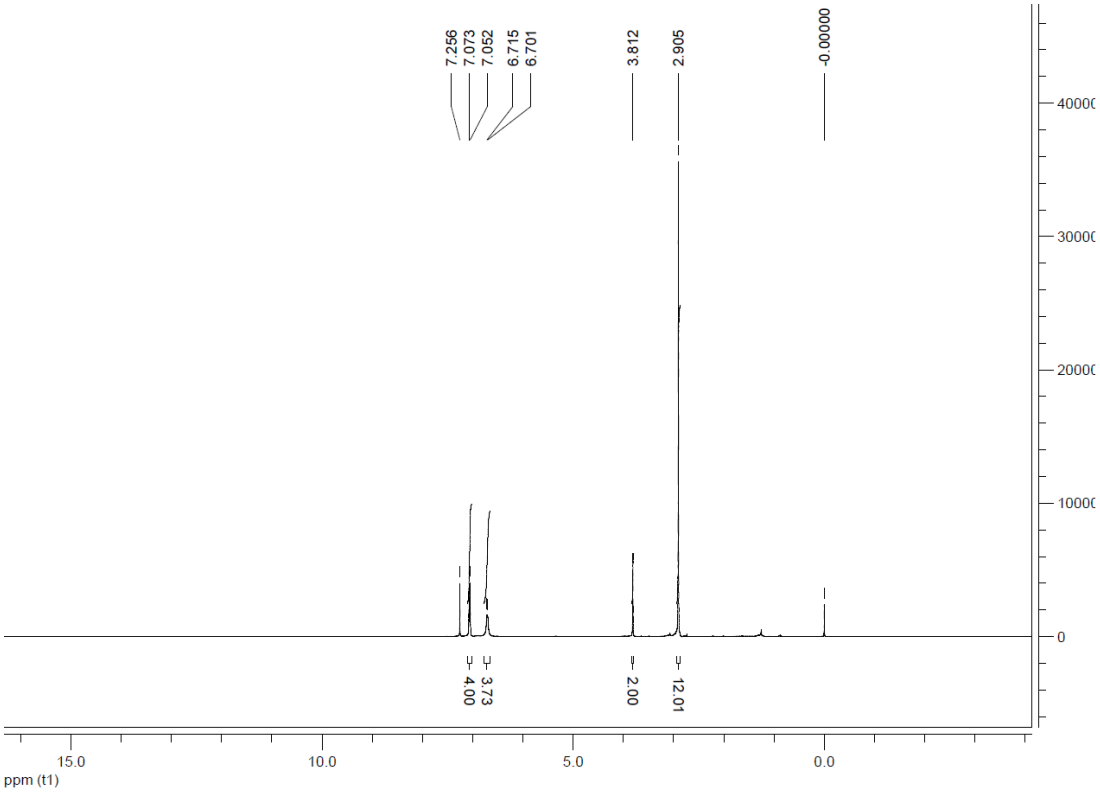
3q

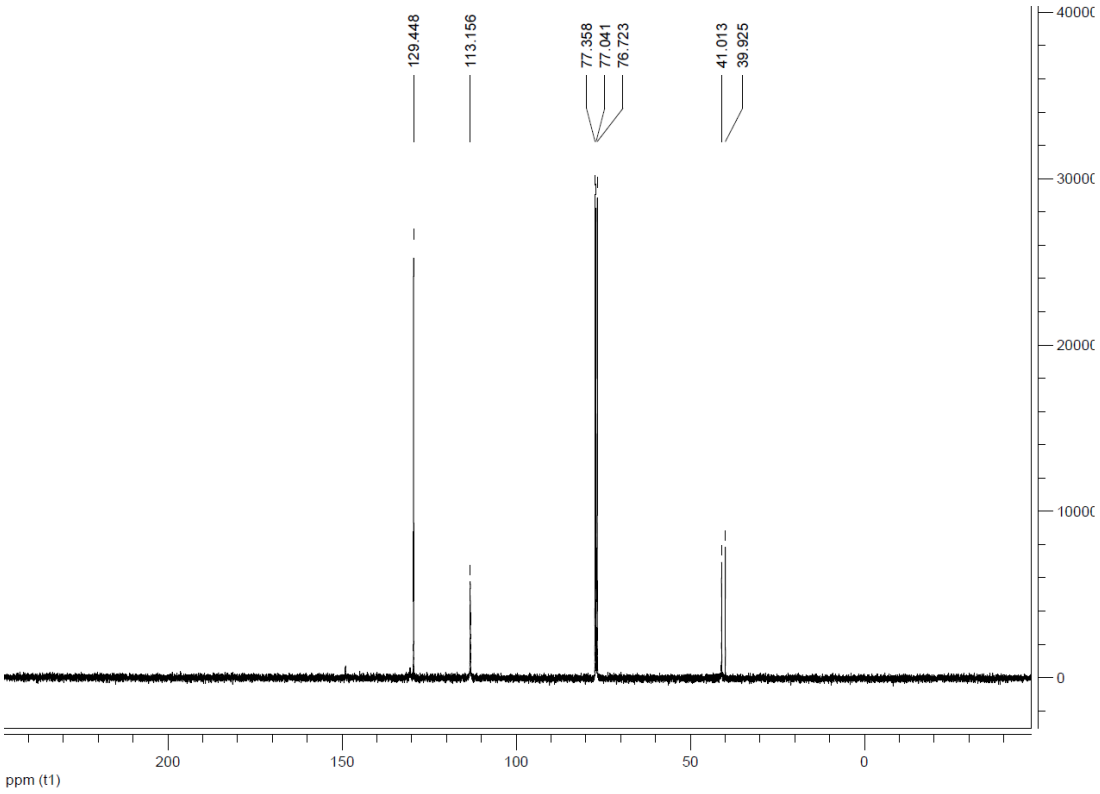

10.062
8.335
8.316
7.953
7.796
7.777
7.720
7.702
7.683
7.619
7.600
7.581
7.501
7.486
7.482
7.468
7.452
7.262
0.00000
15000
10000
5000
0
1.00
2.01
15.0
10.0
5.0
0.0
ppm (t1)


10.062
8.335
8.316
7.953
7.796
7.777
7.720
7.702
7.683
7.619
7.600
7.581
7.501
7.486
7.482
7.468
7.452
7.262
1.00
2.01
1.01
2.01
1.07
2.04
2.07
ppm (t1)


185.757
168.572
137.661
136.885
133.064
129.516
129.080
126.656
126.314
125.653
122.254
122.114
116.147
77.387
77.070
76.751
ppm (t1)

4


7.256
7.073
7.052
6.715
6.701
3.812
2.905
-0.00000
4.00
3.73
2.00
12.01
15.0
10.0
5.0
0.0
ppm (t1)


129.448
113.156
77.358
77.041
76.723
41.013
39.925
200
150
100
50
0
ppm (t1)

7. References

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