Supplementary Information

Metal-free yne-addition/aryl migration/decarboxylation cascade reaction of alkynoates with C_{sp3}-H centers

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Table of Contents

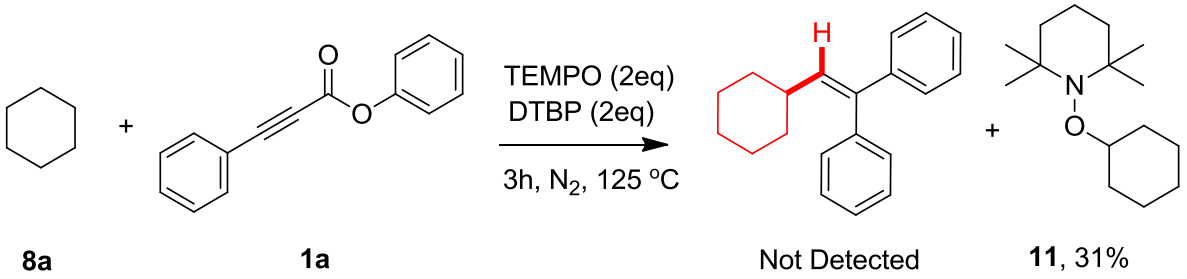
General Information:

All chemicals were obtained from commercial sources and were used as received unless otherwise noted. Alkynoate were prepared according to original or modified literature procedures. All reactions were carried out in flame-dried reaction vessels with Teflon screw caps under a nitrogen atmosphere by using standard Schlenk techniques. Anhydrous solvents were purified and dried following standard procedures. TLC analysis was performed on pre-coated, glass-backed silica gel plates and visualized with UV light. Column chromatography was performed on silica gel(200-300 mesh) using ethyl acetate (EA)/petroleum ether (PE).^{1}H-NMR and ^{13}C-NMR Spectra were obtained on a Bruker 300 MHz, 400MHz or 500 MHz NMR spectrometer in the deuterated solvents indicated Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as the internal standard. The following abbreviations were used to designate chemical shift multiplicities: s= singlet, d= doublet,t= triplet, q= quartet, h= heptet, m= multiplet,. All first-order splitting patterns were assigned on thebasis of the appearance of the multiplet. Splitting patterns that could not be easily interpreted are designated as multiplet (m) or broad (br).Melting points were measured on Beijing Tech X-4 apparatus without correction. IR spectra were recorded on a Nicolet 6700 FT-IR spectrometer. HRMS Masss pectra were obtained using electrospray ionization (ESI) or atmospheric pressure chemical ionization (APCI) mass spectrometer.

2. Mechanistic studies

2.1 Radical scavenger effect studies

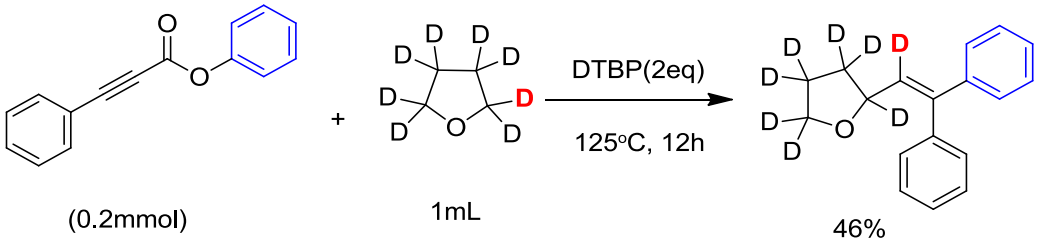
To gain insight into the pathway of the present reaction, we conducted some control experiments under the standard reaction conditions. Radical scavengers, TEMPO, were employed in the standard reaction, and no desired product was detected. This result suggested that a single electron transfer process (SET) was involved in the reaction.



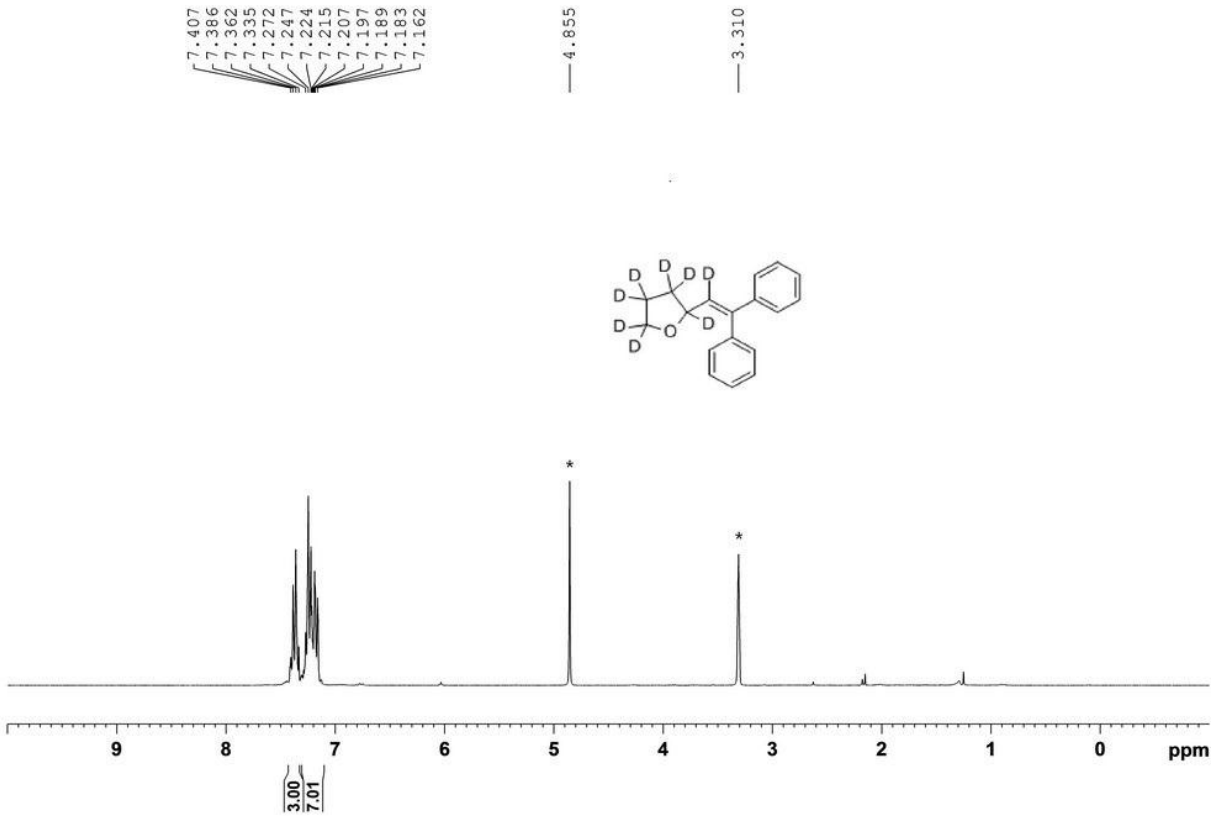
8a **1a** Not Detected **11**, 31%

[a]Reaction conditions: 1a (0.2 mmol), DTBP (0.4 mmol), TEMPO(0.5 mmol),4 mL of cyclohexane as solvent, 125 ℃, 12h.

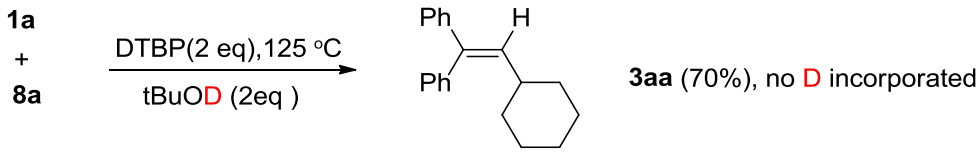
2.2 Deuterium-labeling experiment



Mixture of THF-D_8 (1 mL), 1a (0.2mmol) and DTBP (0.4 mmol) was sealed in a Teflon septum screw-capped tube underN_2. The mixture was stirred in an oil bath at 125 ^oC for 12 h. The corresponding reaction mixture was purified by flash column chromatography on a silica gel to give the desired product in 46% yield. The ^{1}NMR (in CD_3OD) was described in details as follows.

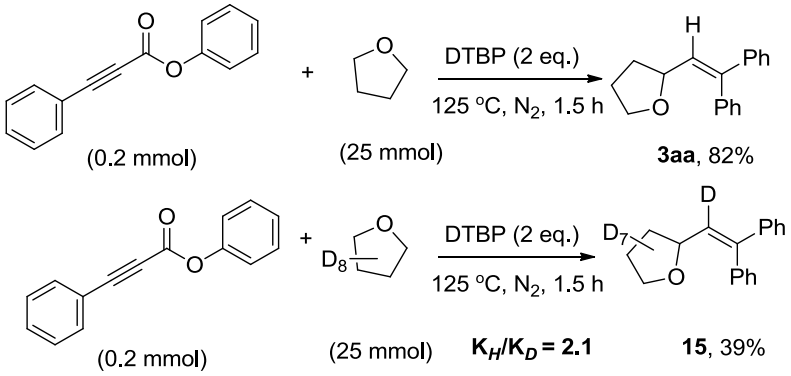


The addition of *t*-BuOD (2 eq) to the reaction mixture of **1a** with **8a**on the standard condition did not lead to any deuterium-containing products thus practically ruling out the possibility that the terminal hydrogen abstraction step of the tandem reaction might be involved by *t*-BuOH formed from homo-cleavage of DTBP.



2.3 Kinetic isotope effect studies

THF (2 mL, 25 mmol), **1a** (0.2 mmol) and DTBP (0.4 mmol) was sealed in a Teflon septum screw-capped tube under N_2. Same procedures was operated with THF-D_{12}. The mixture was stirred in an oil bath at 125 °C for 1.5 h. After completion of the reactions, the mixtures were detected by GC analysis with dibenzofuran as the internal standard. As a result, the desired coupling products were obtained in 82% and 39% yields, respectively, in which k_H/k_D = 2.1.



3. Copies of ^{1}H NMR and ^{13}C NMR

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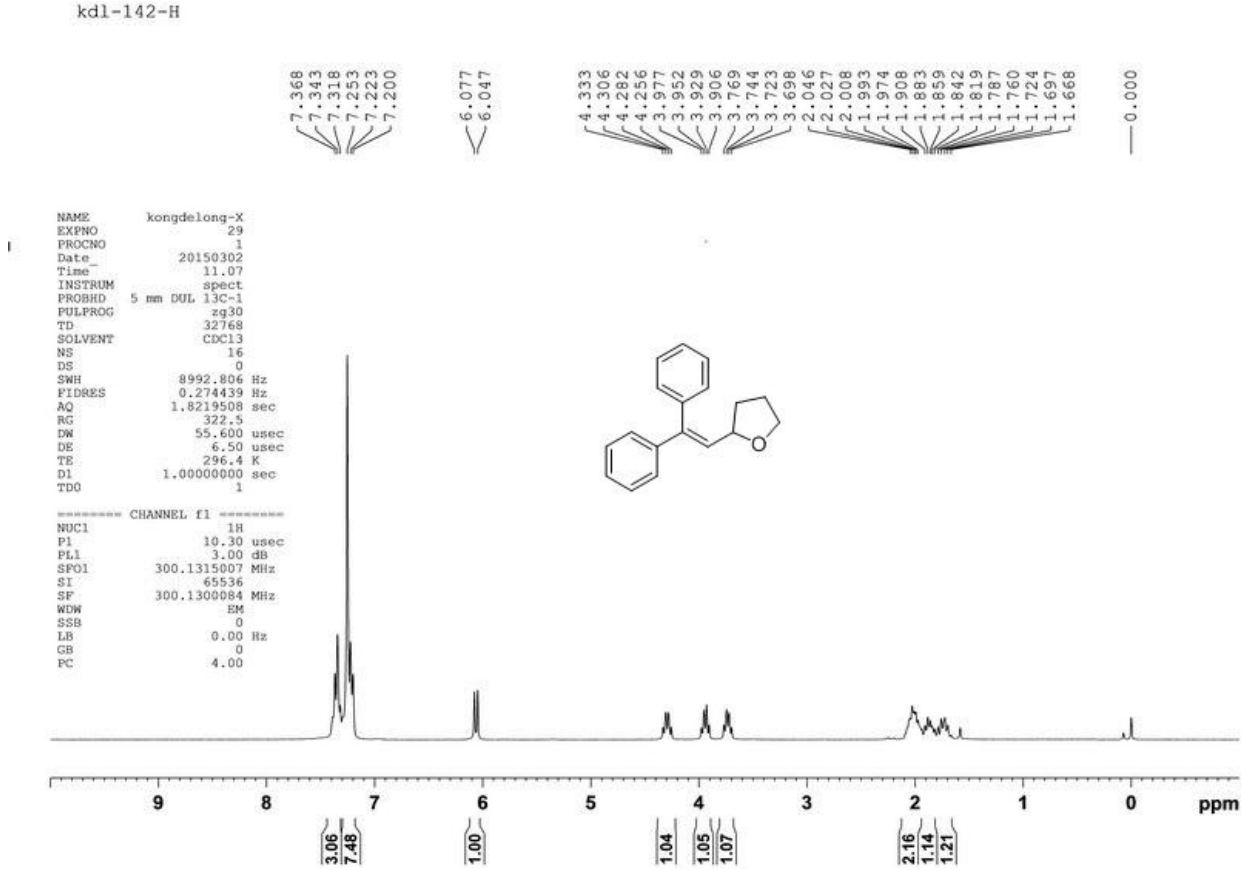

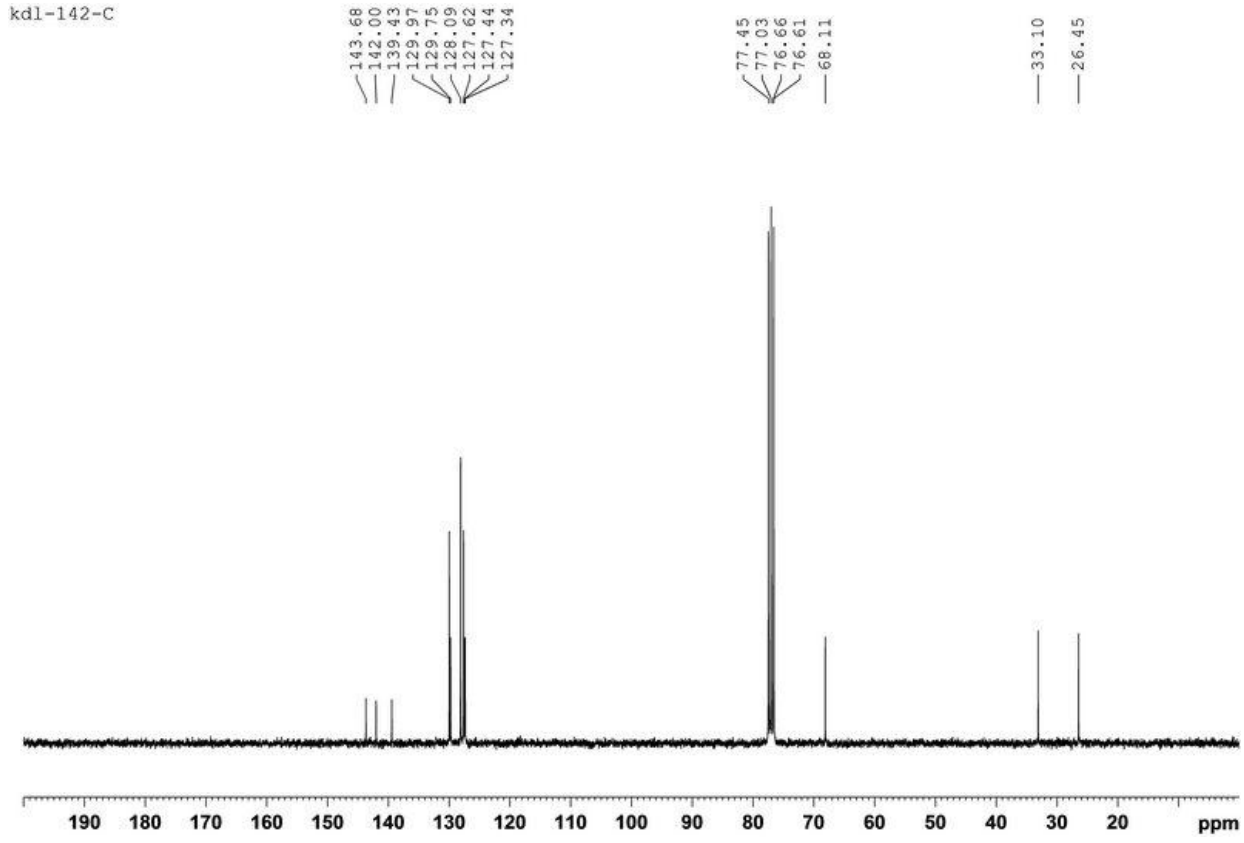

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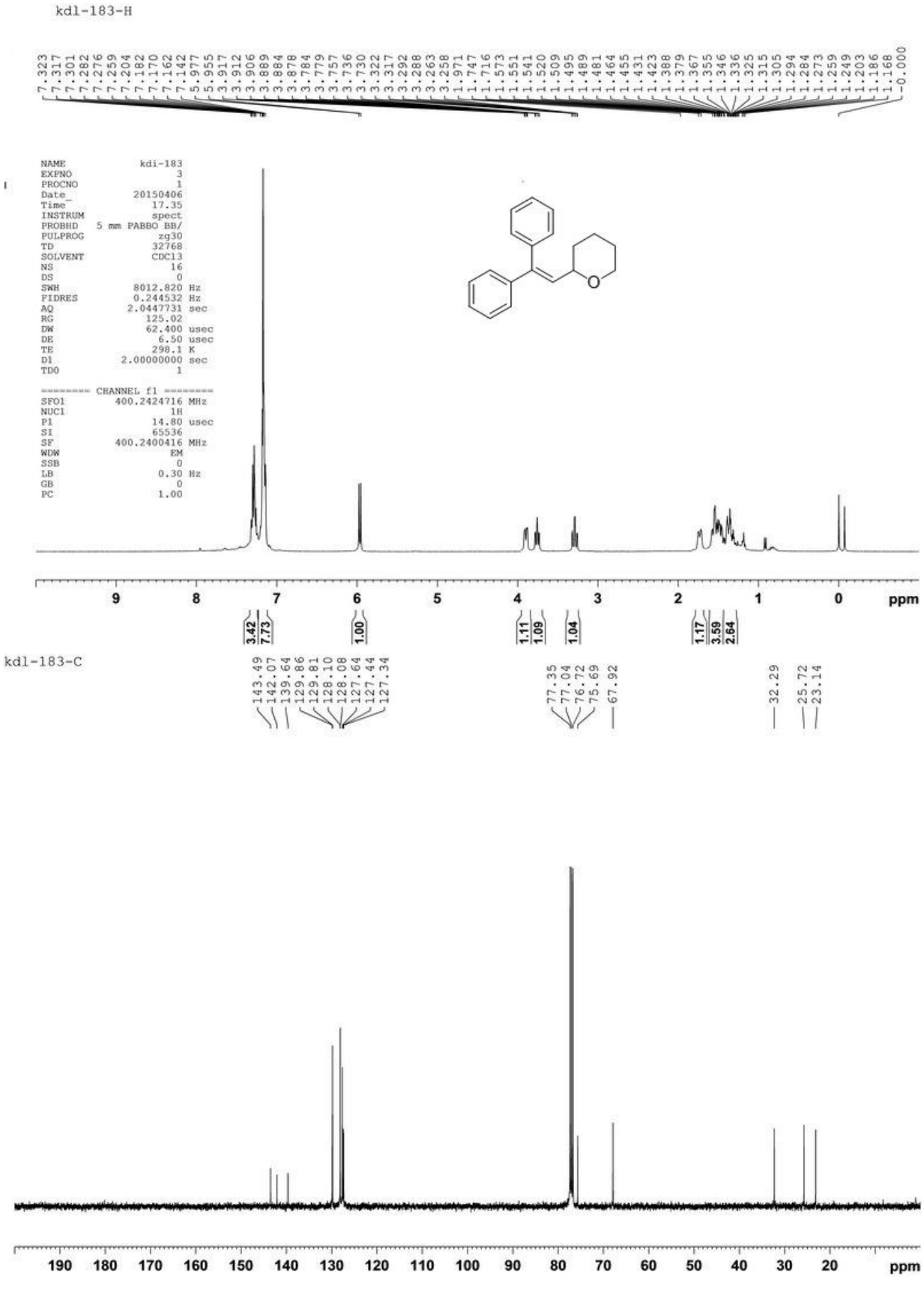
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3.912
3.906
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3.784
3.779
3.757
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3.322
3.317
3.292
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8
7
6
5
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127.34
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76.72
75.69
67.92
32.29
25.72
23.14
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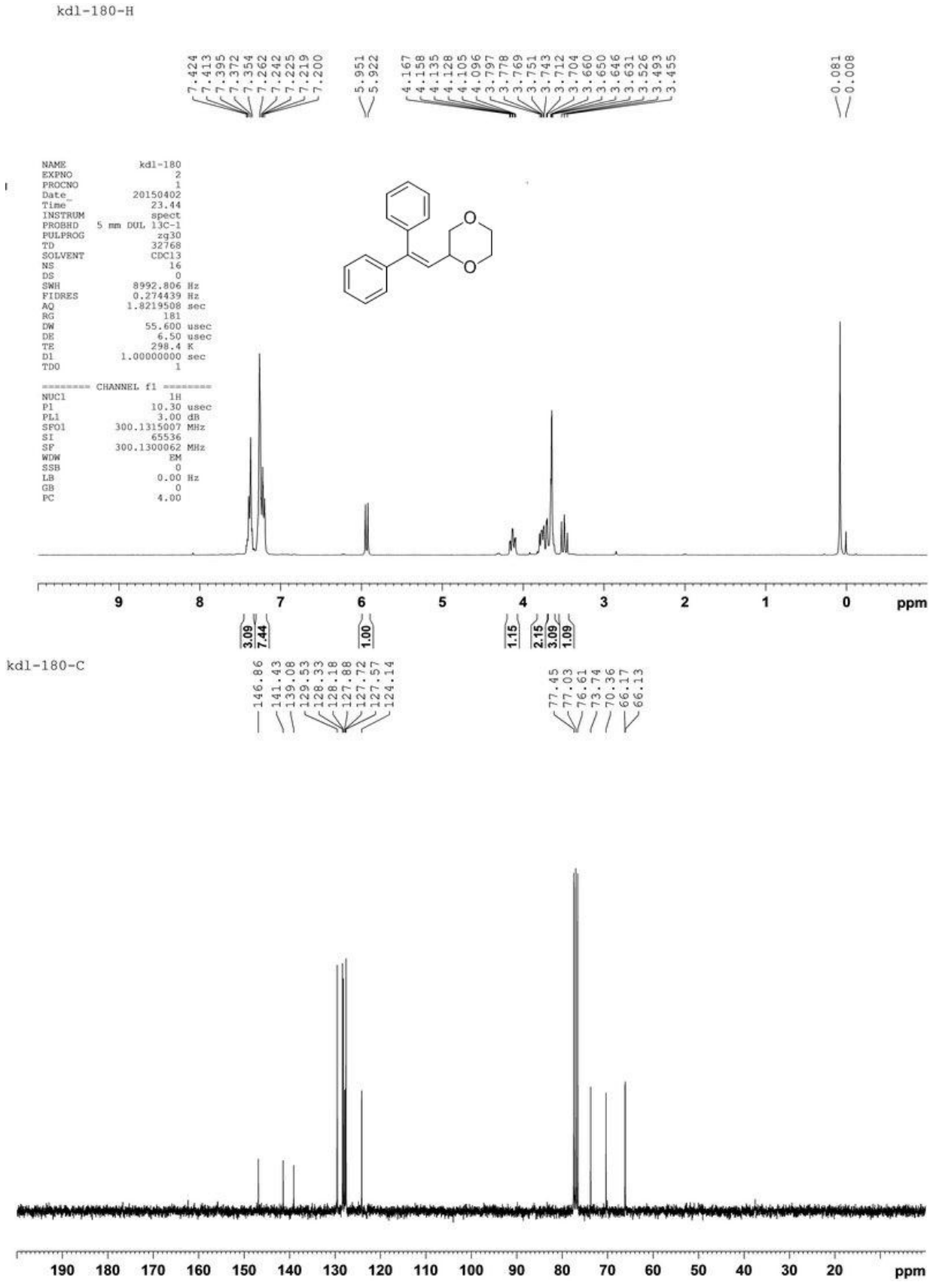
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9
8
7
6
5
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3
2
1
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ppm
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2.15
3.09
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66.13
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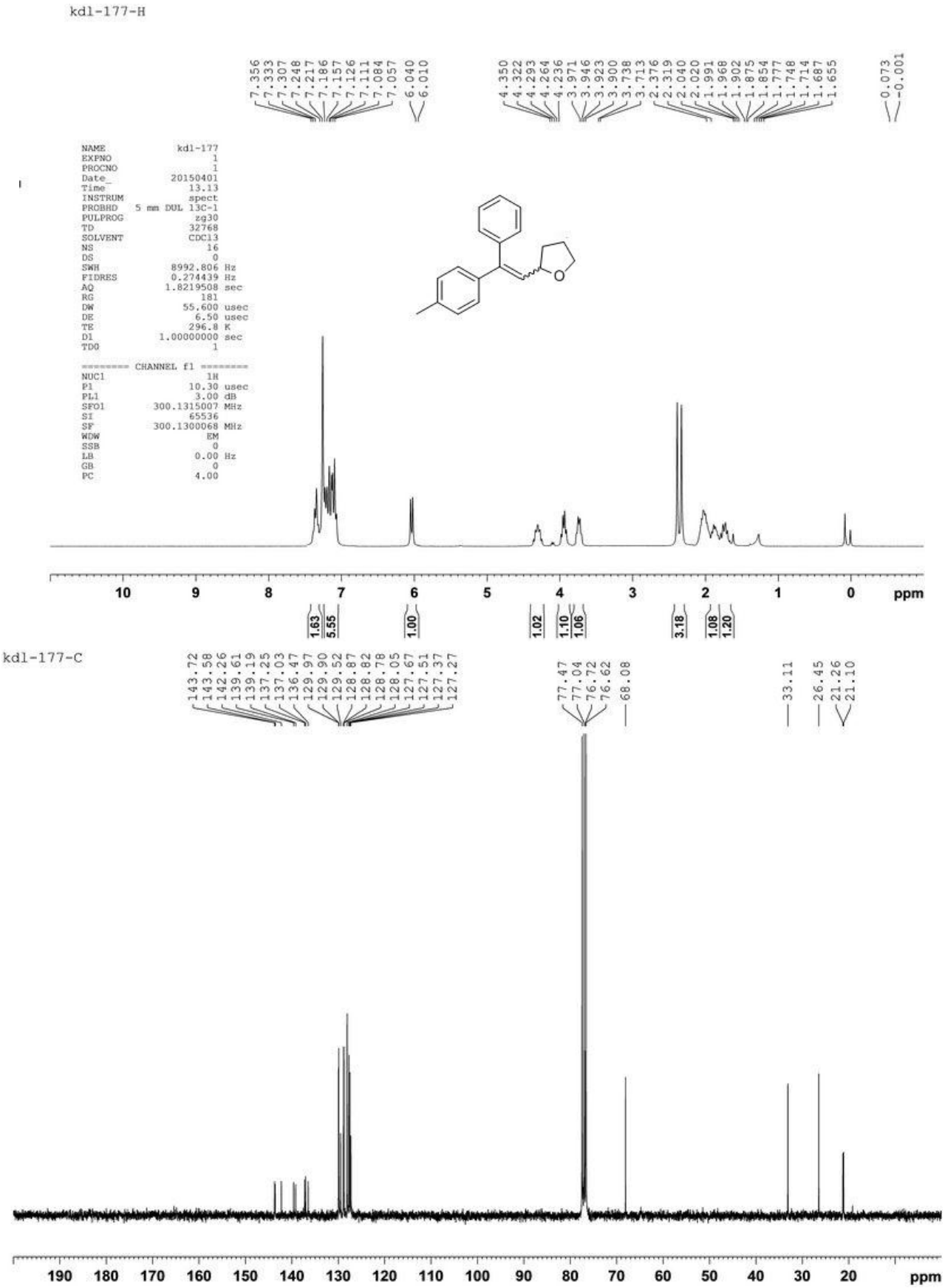
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3e

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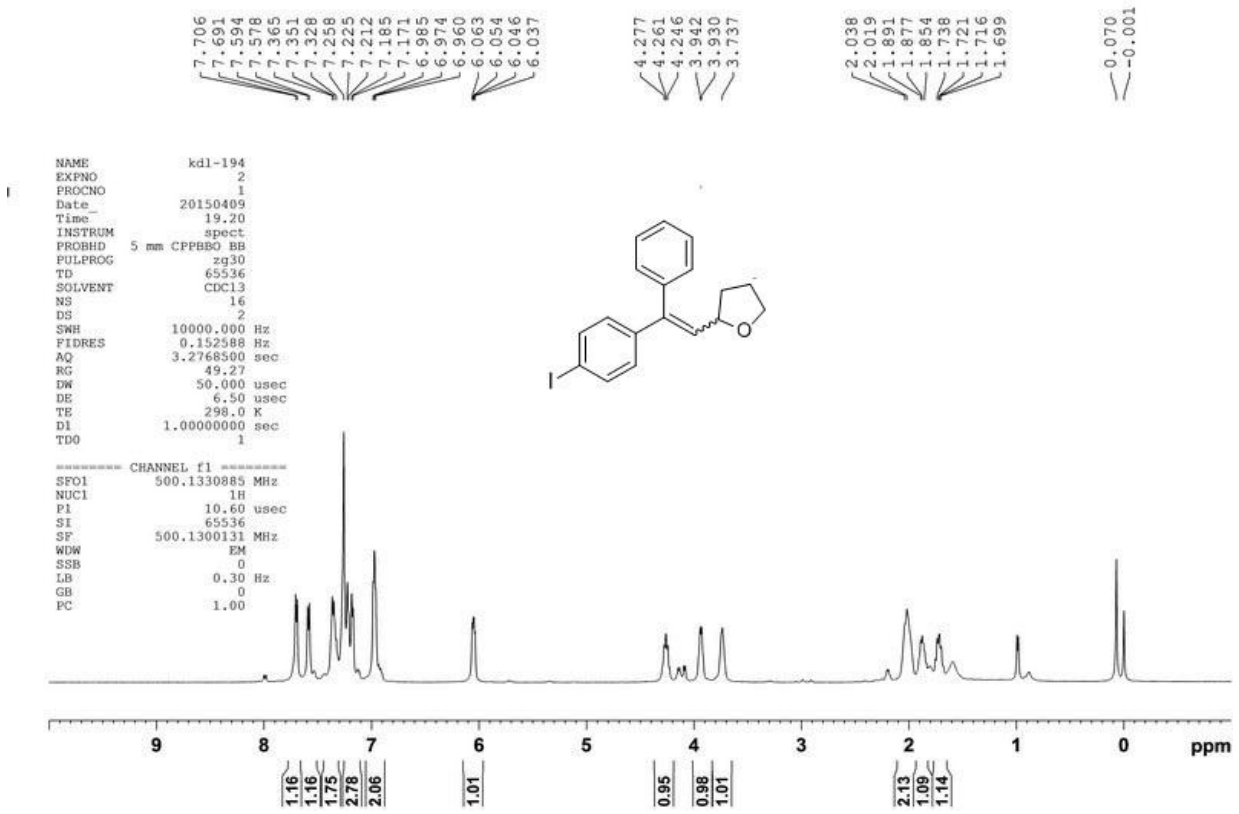

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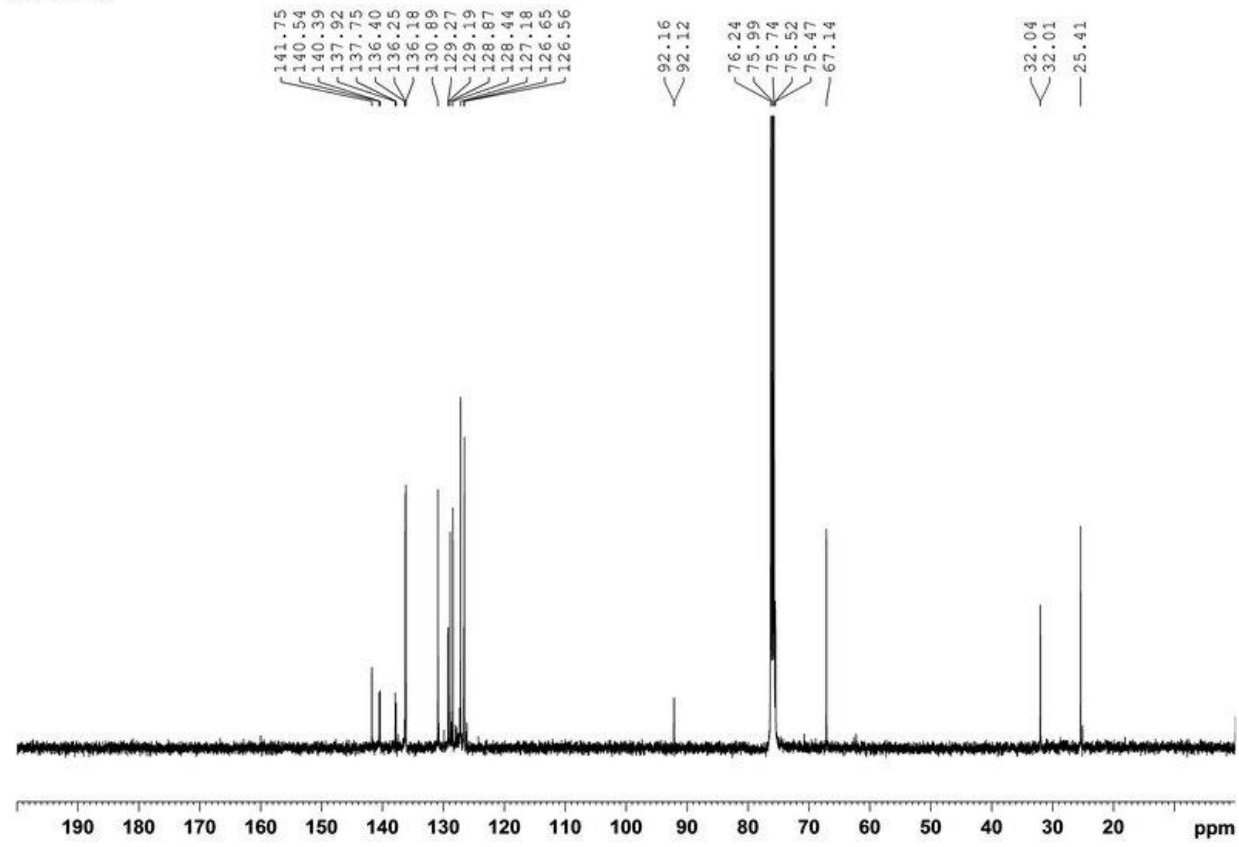

3f

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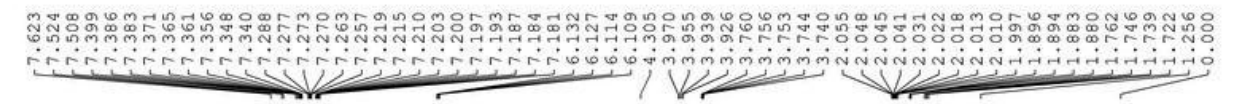

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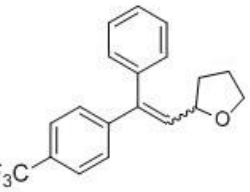

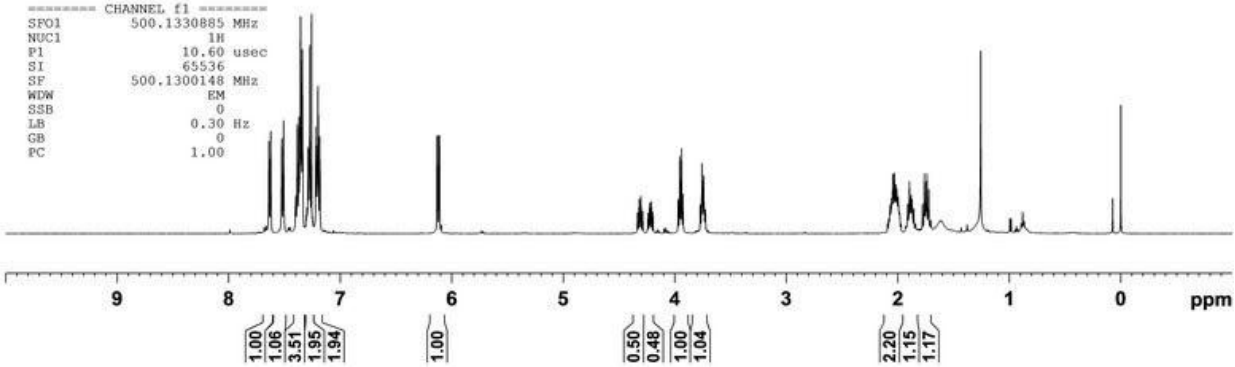

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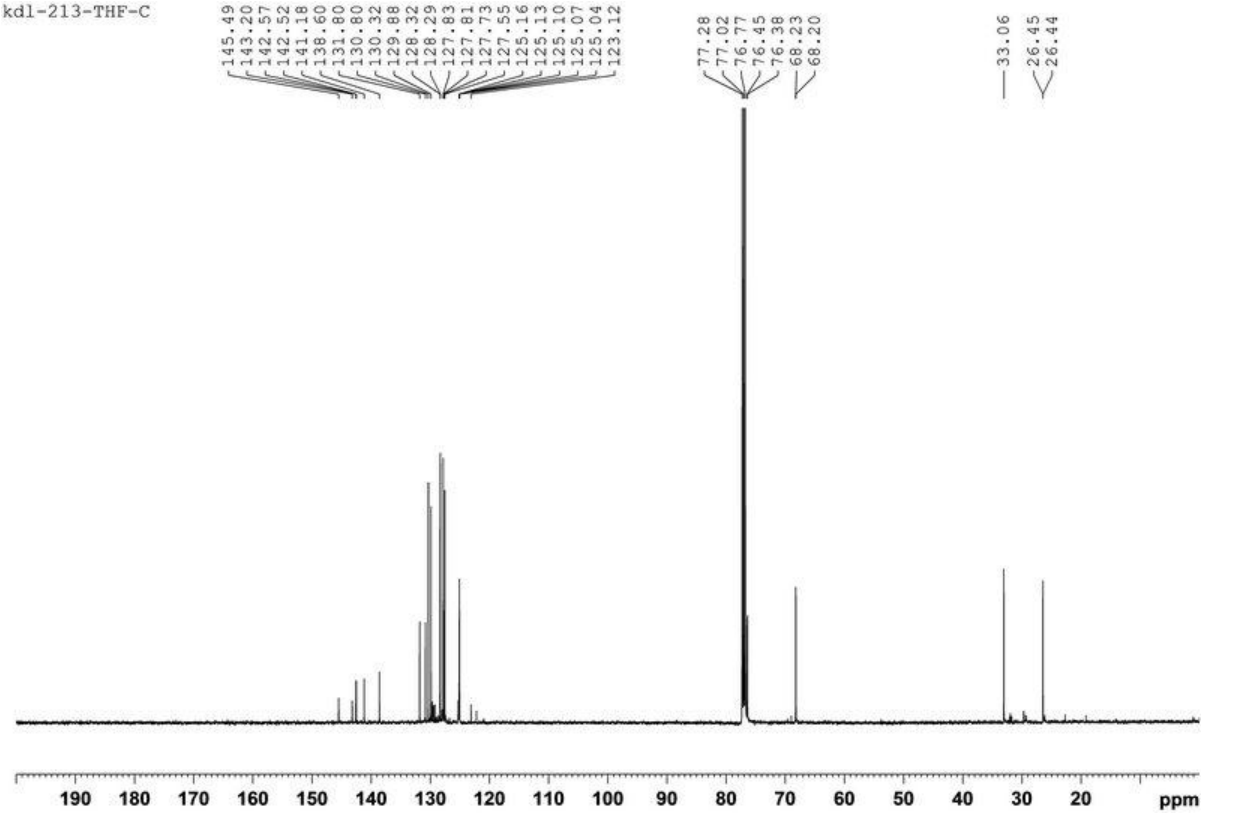

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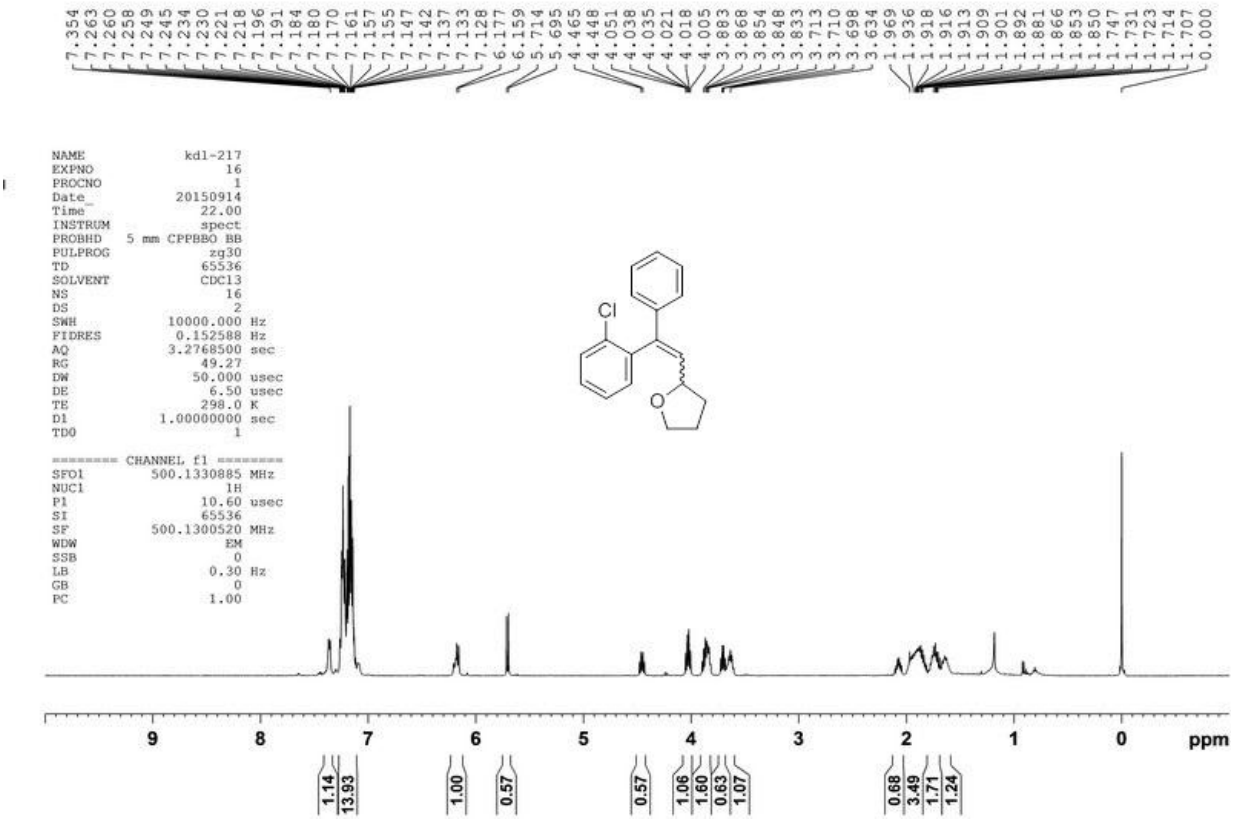

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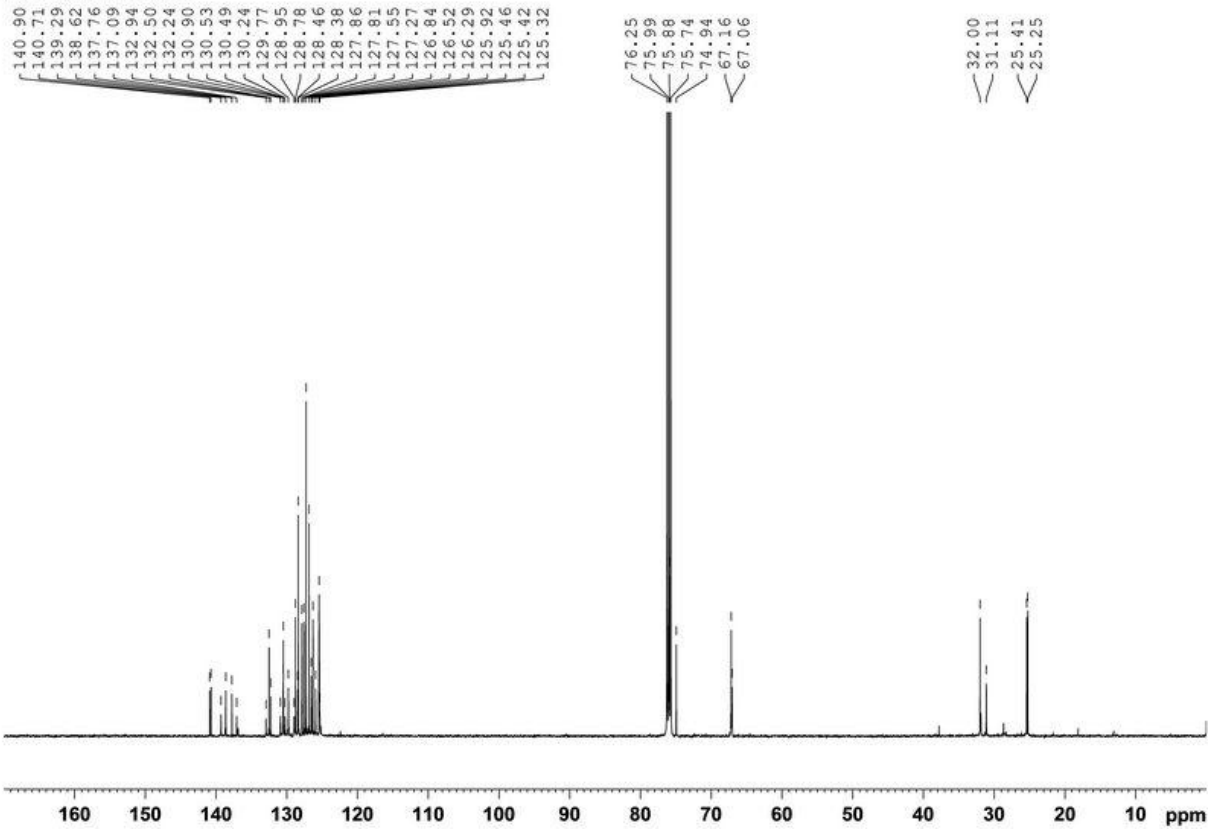

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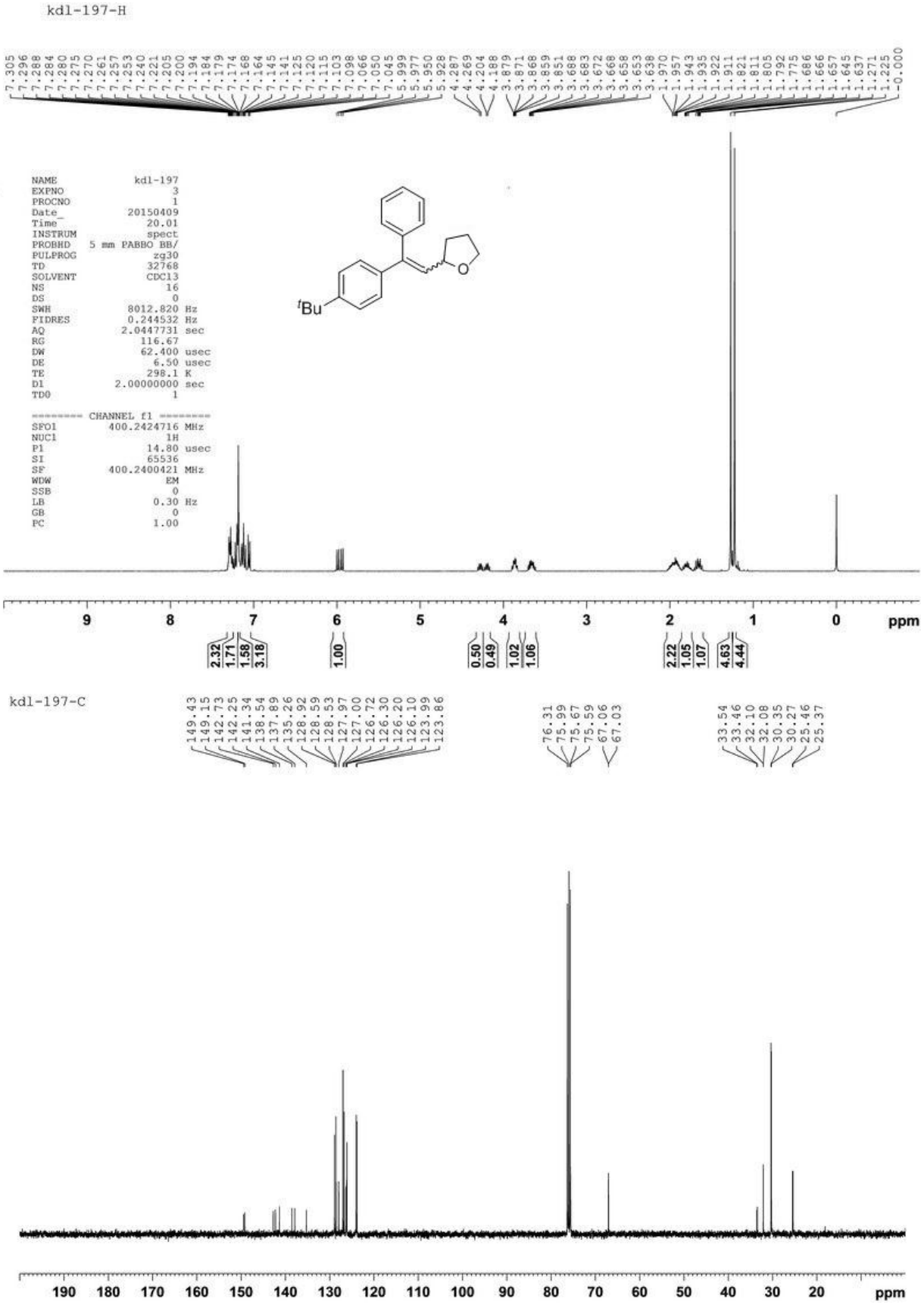
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5aa

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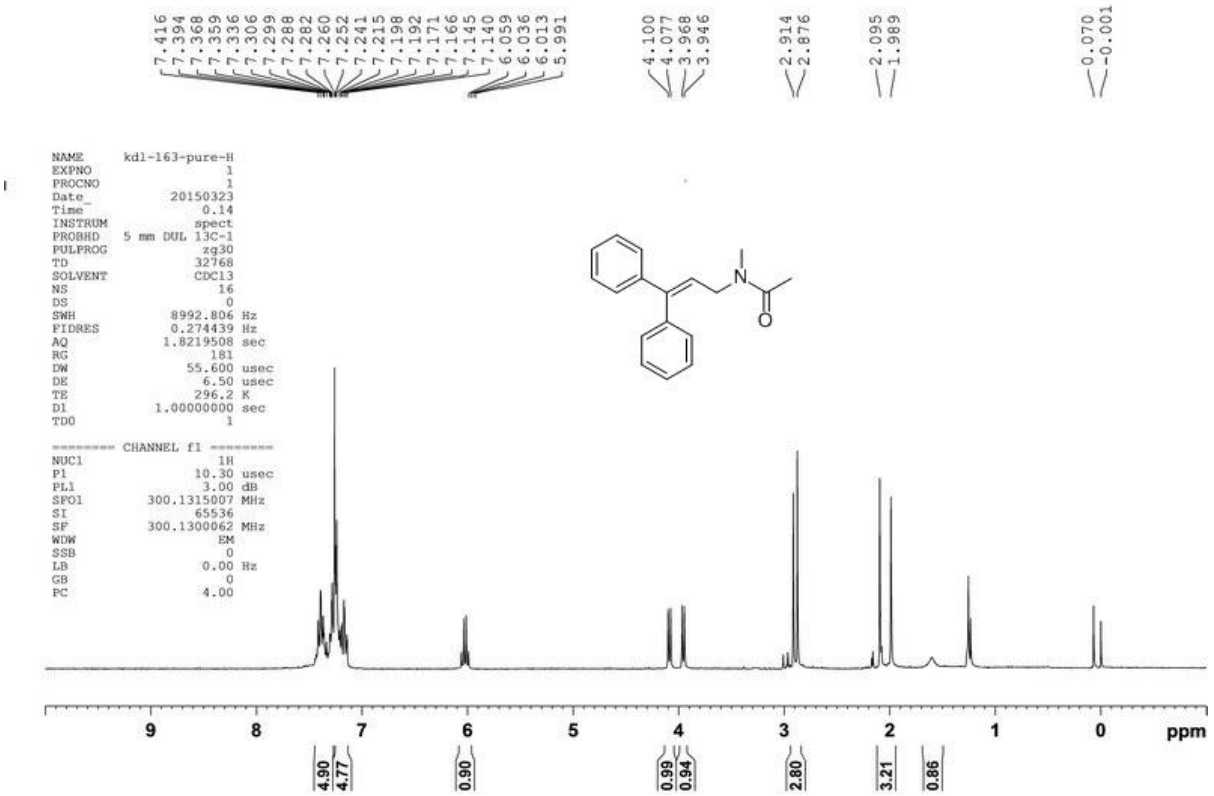

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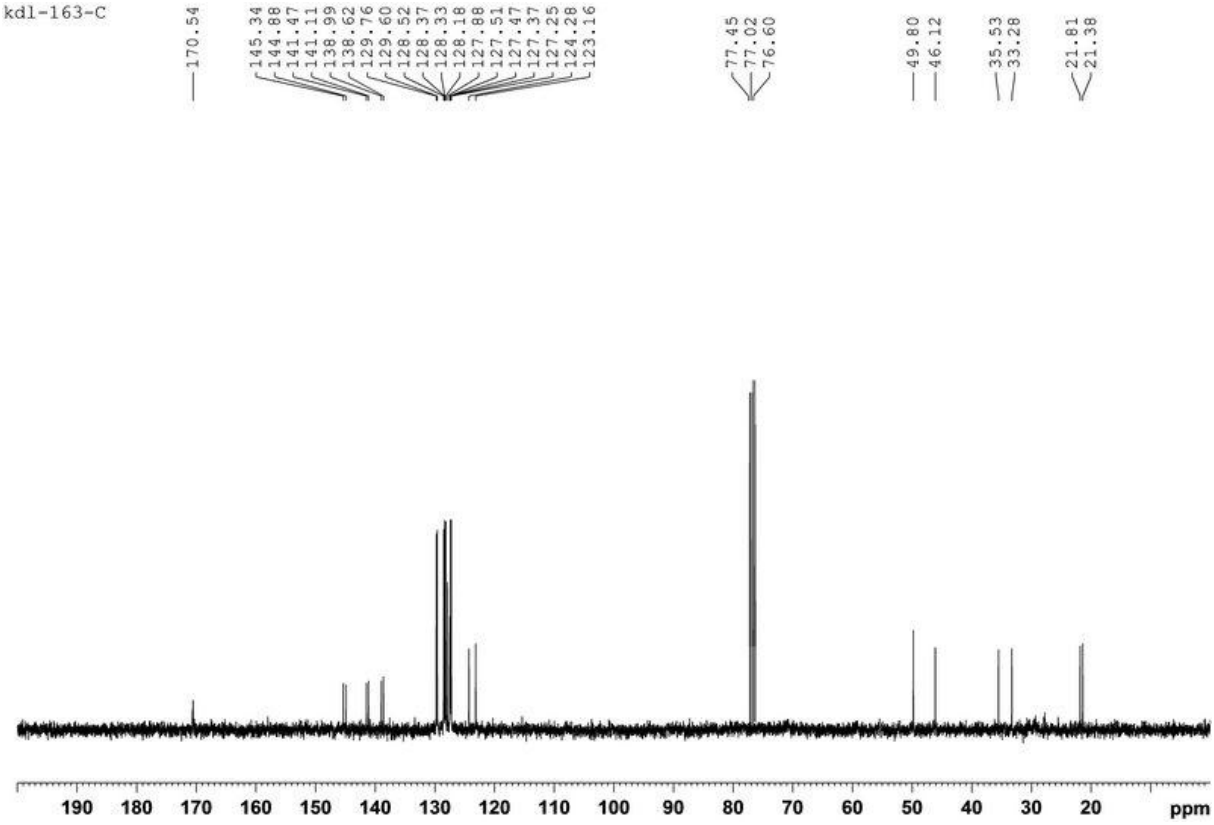

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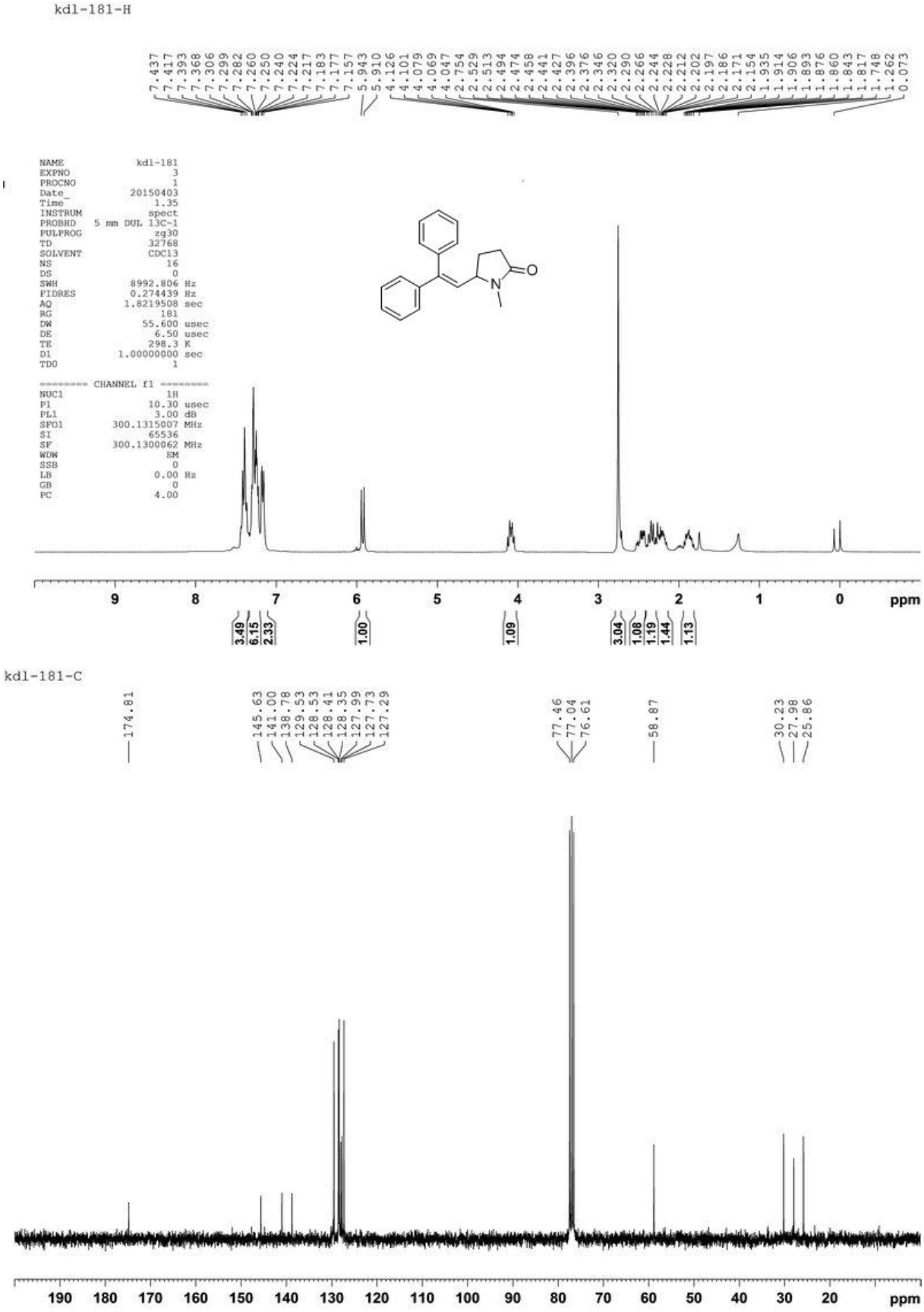
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D1 1.00000000 sec
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SF 300.1300062 MHz
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9
8
7
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5
4
3
2
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6.15
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1.44
1.13
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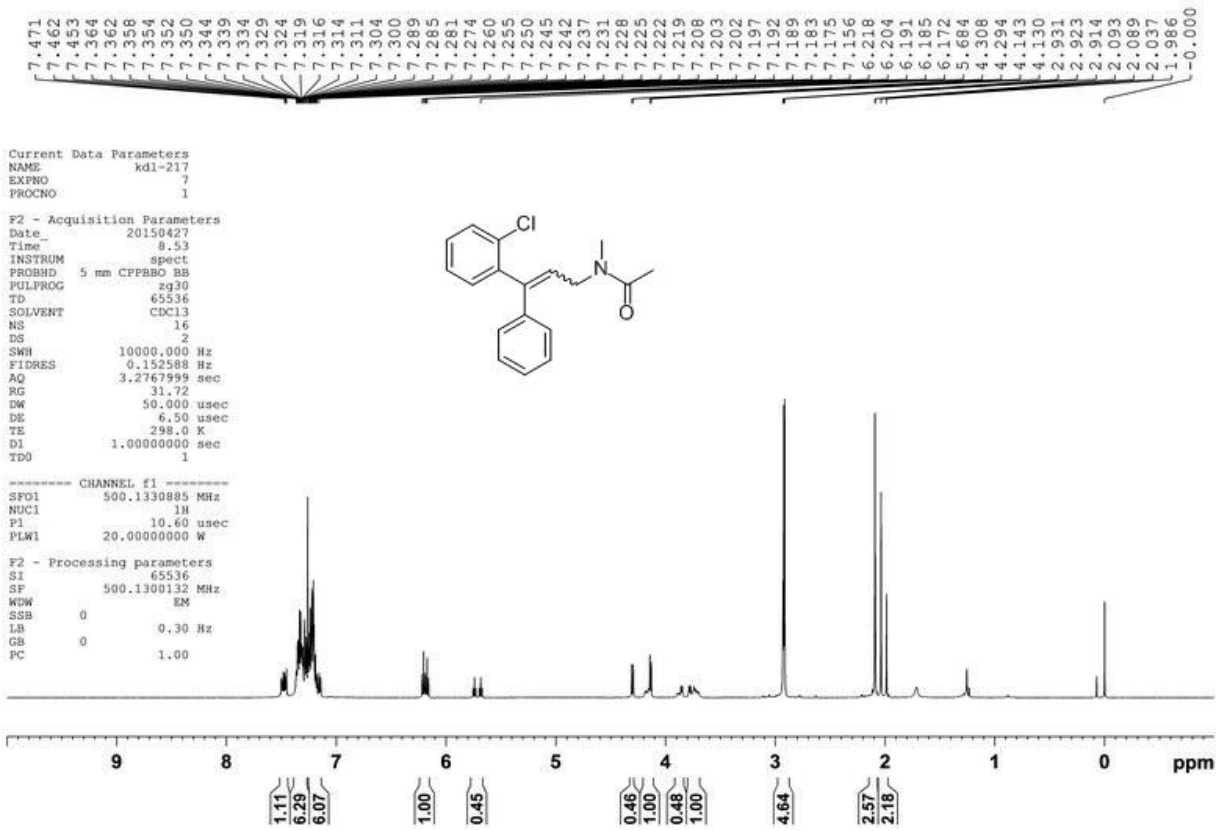

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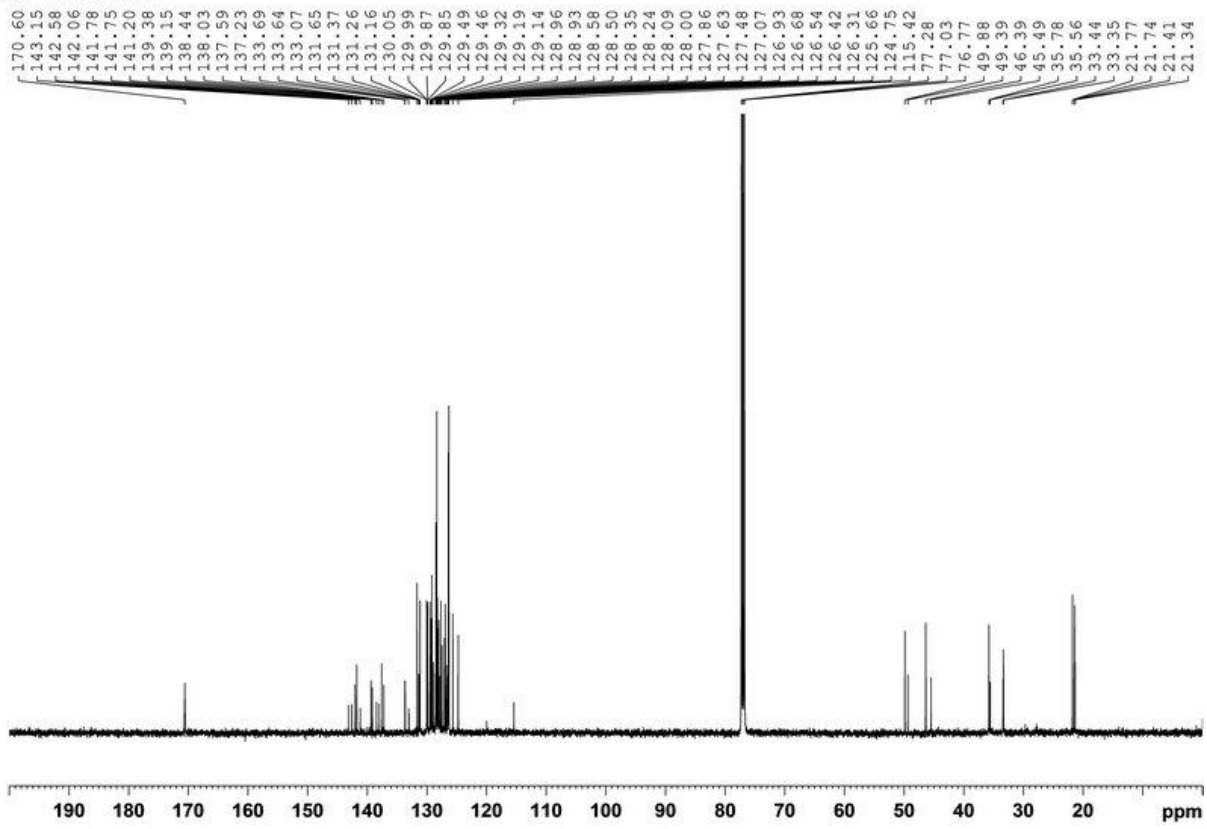

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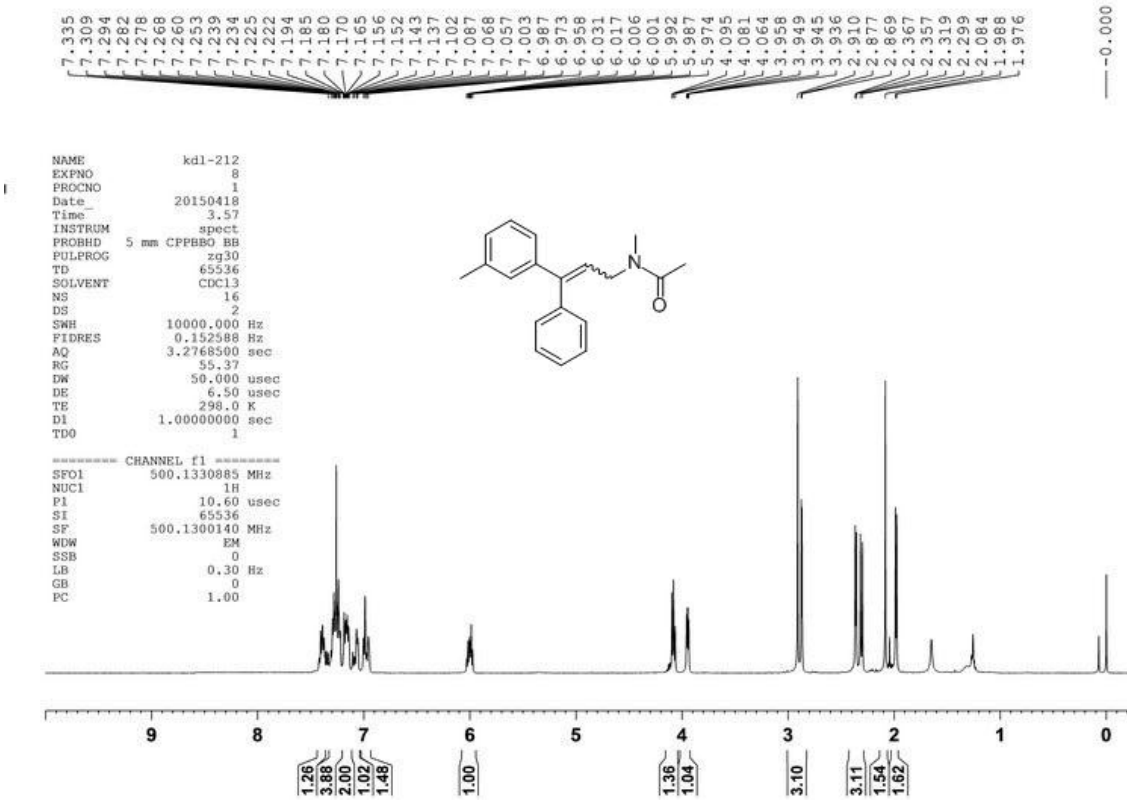

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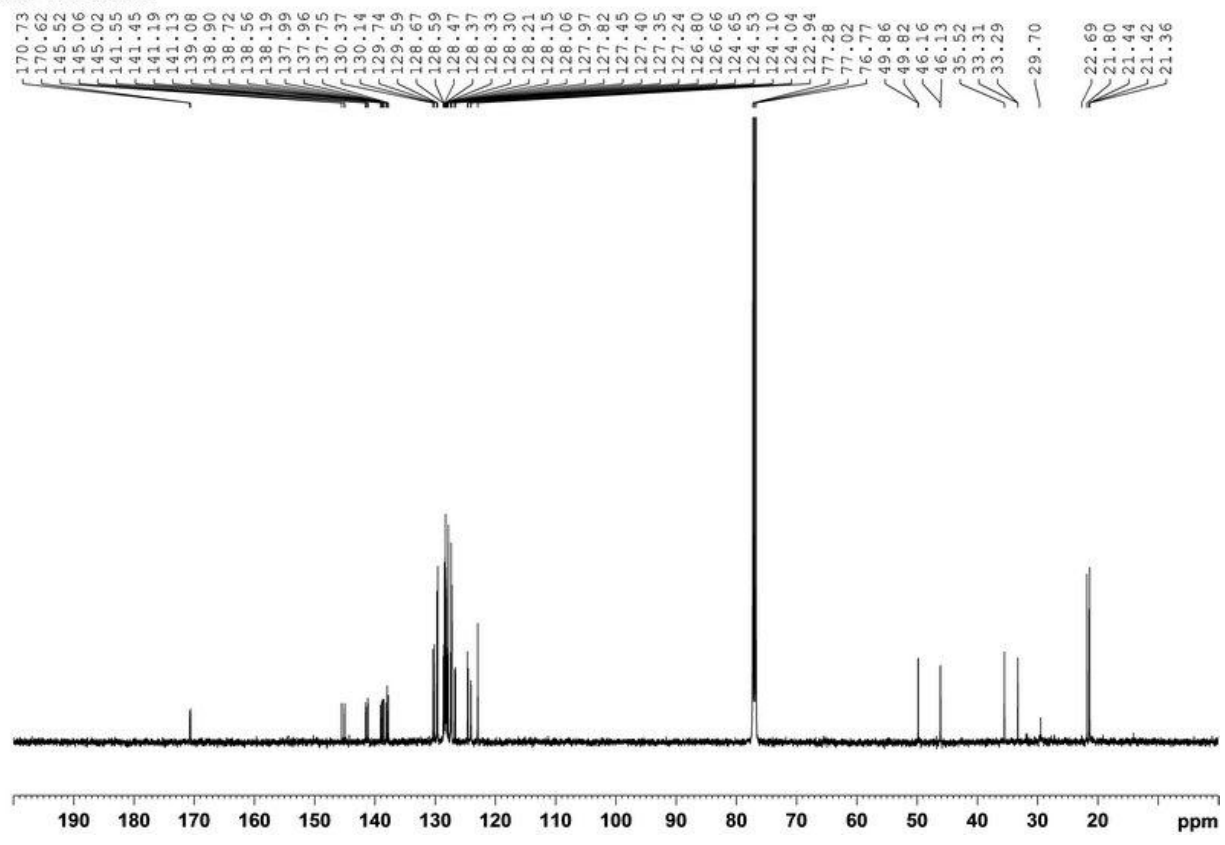

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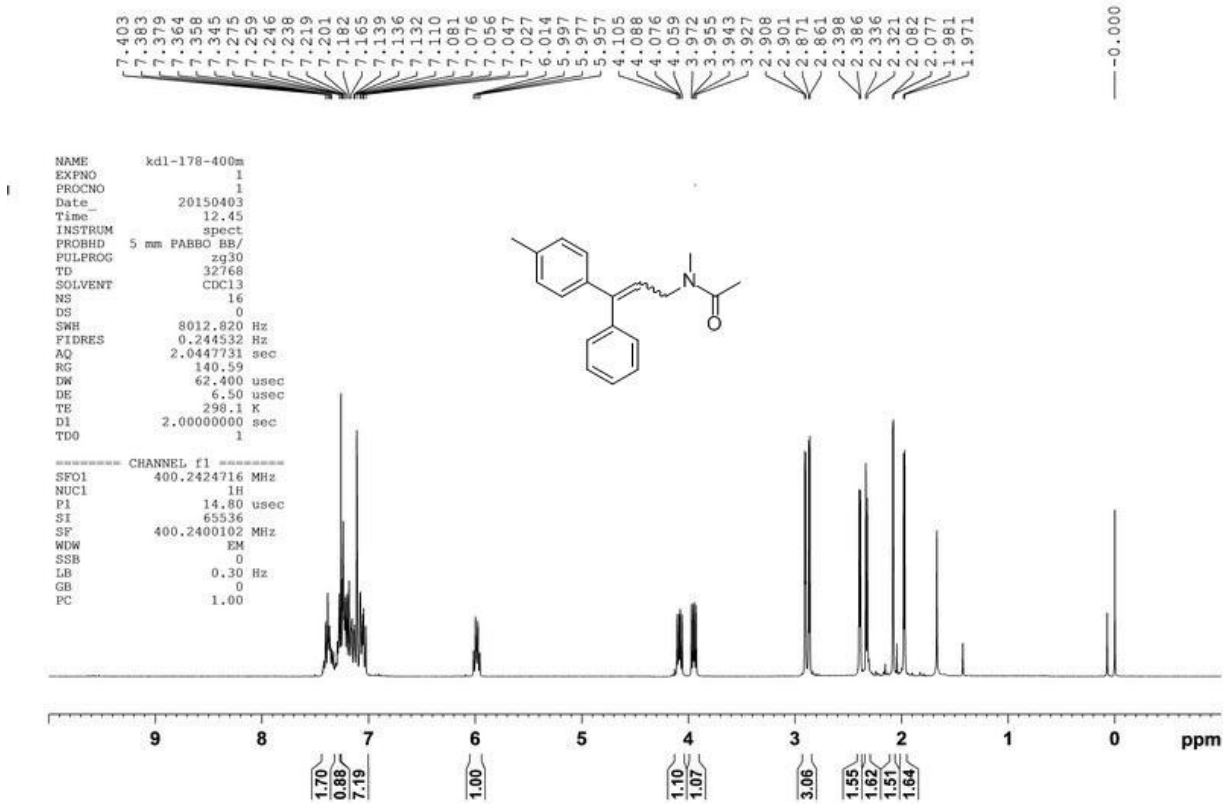
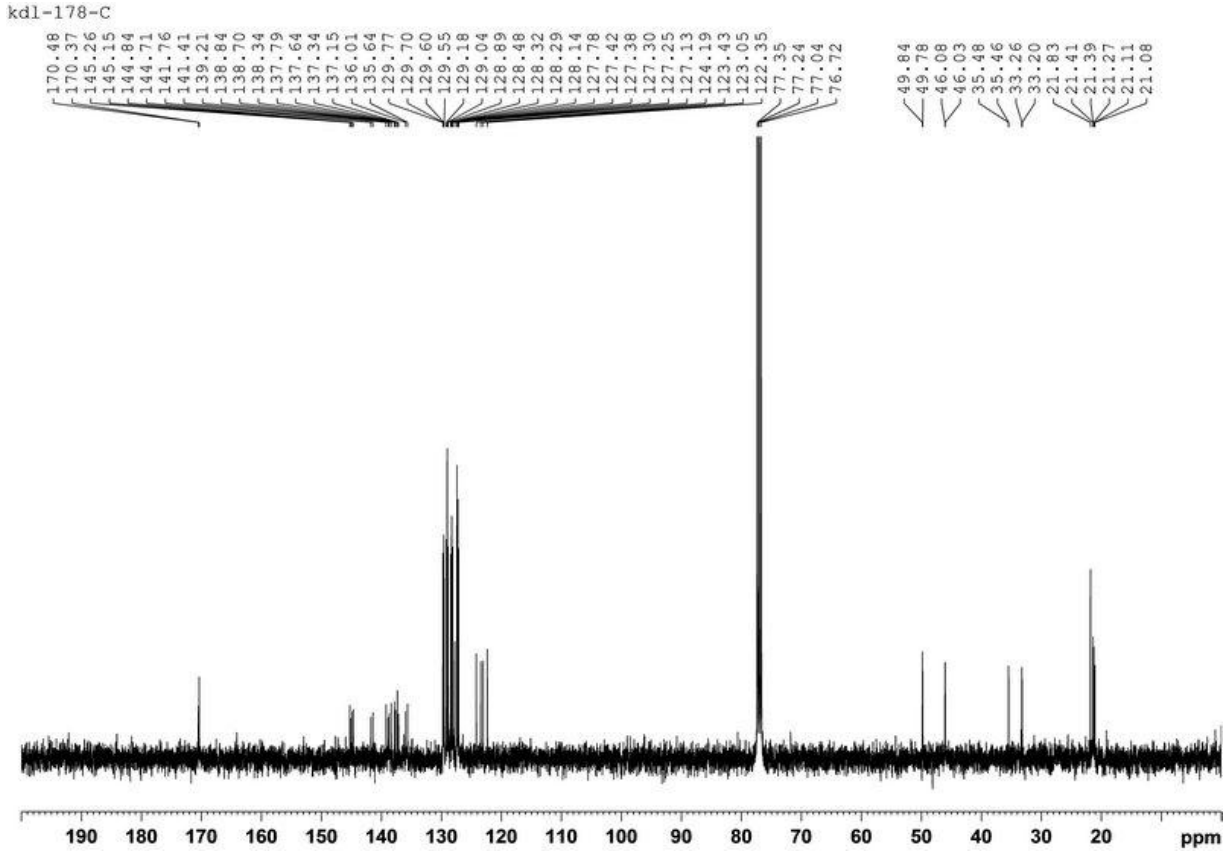
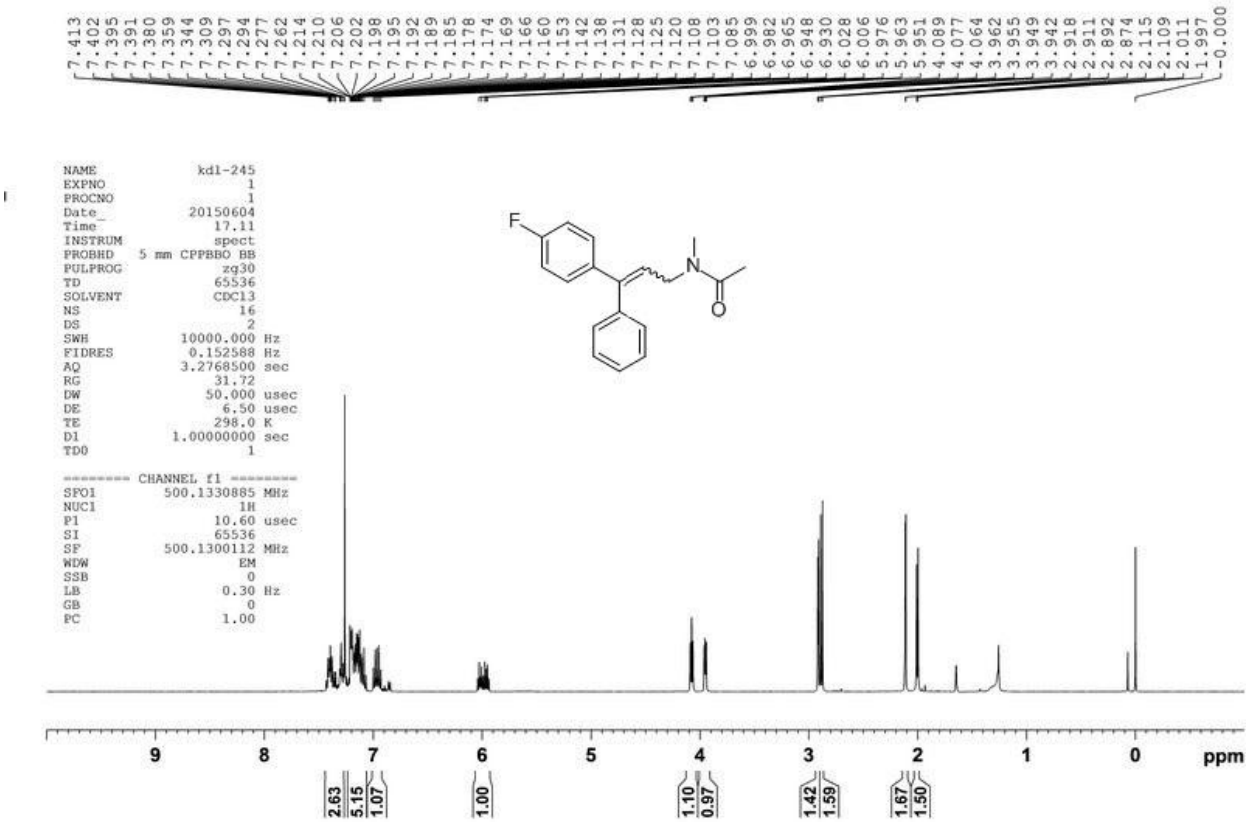


kdl-178-C

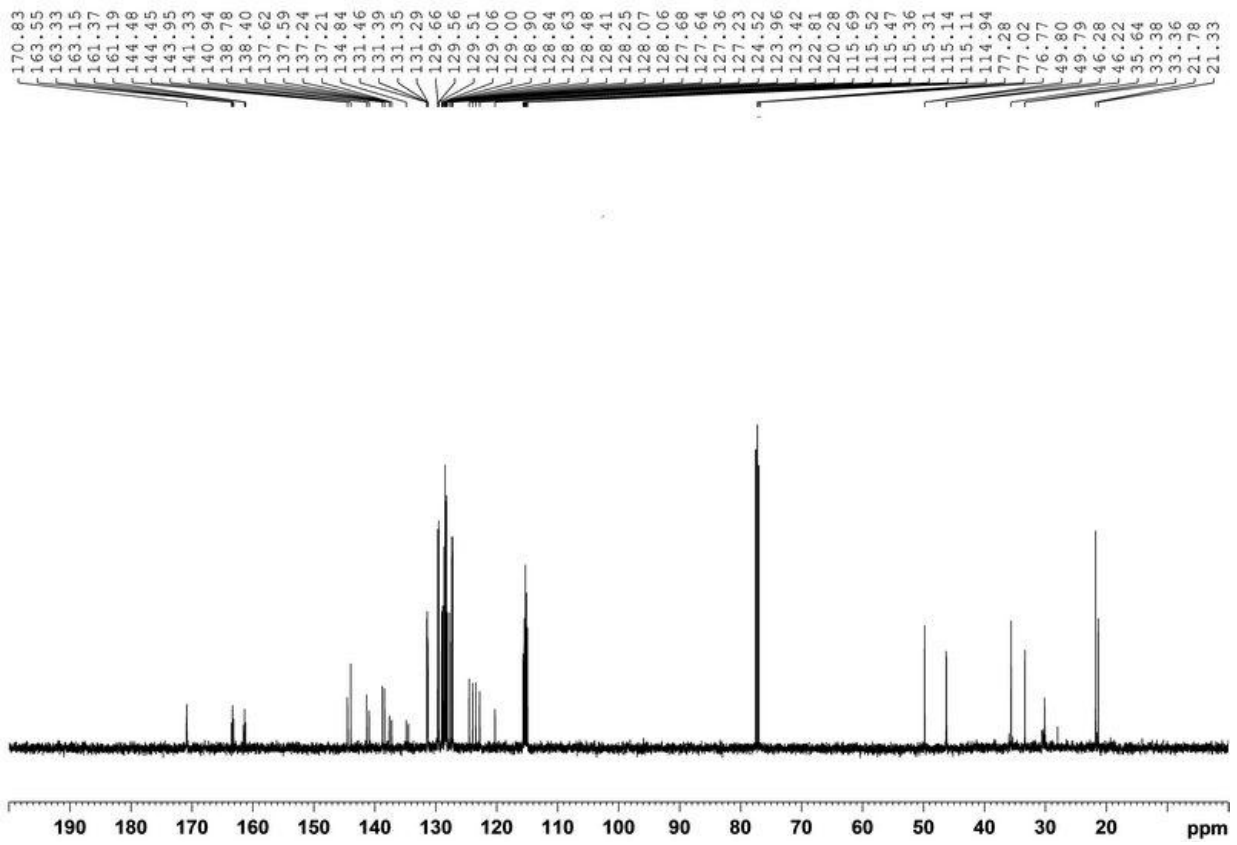


5f

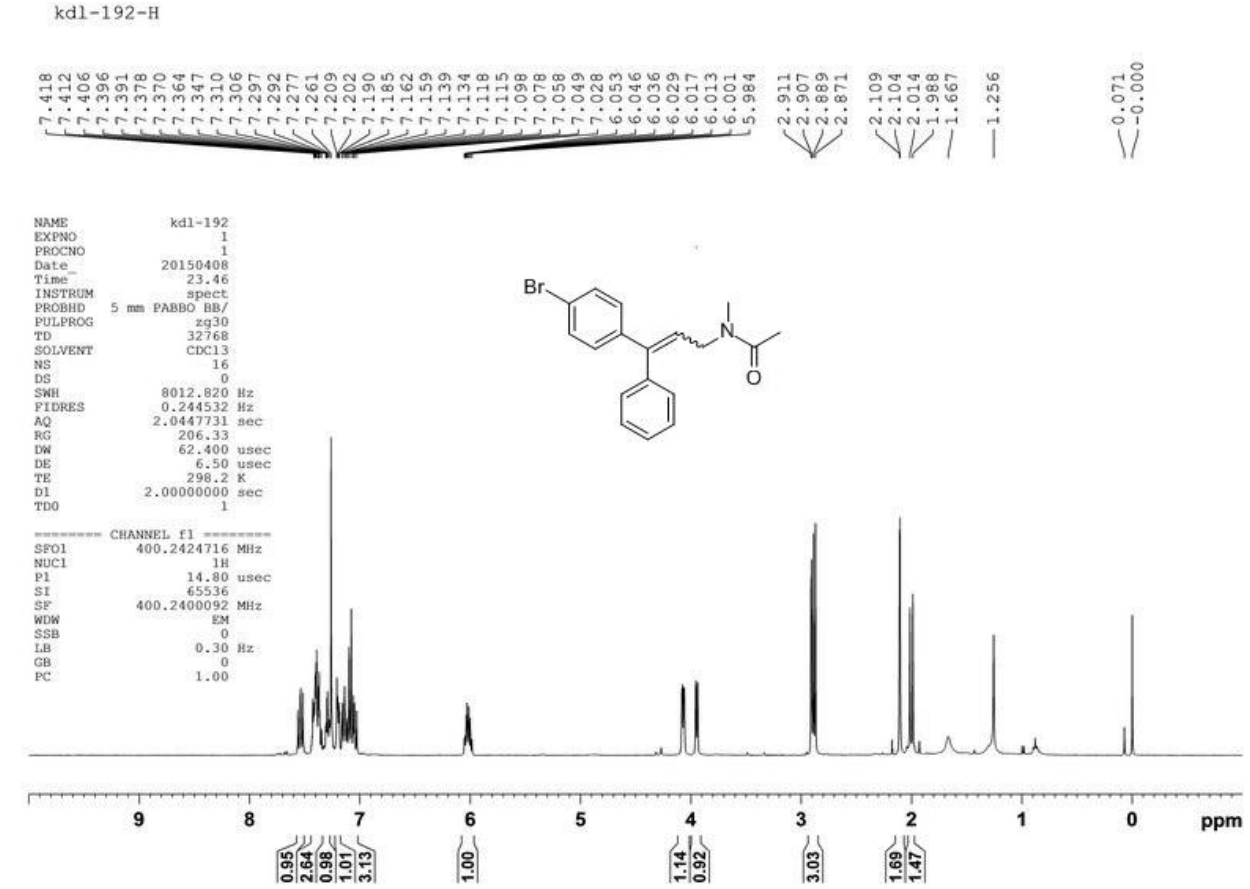
kdl-245-H

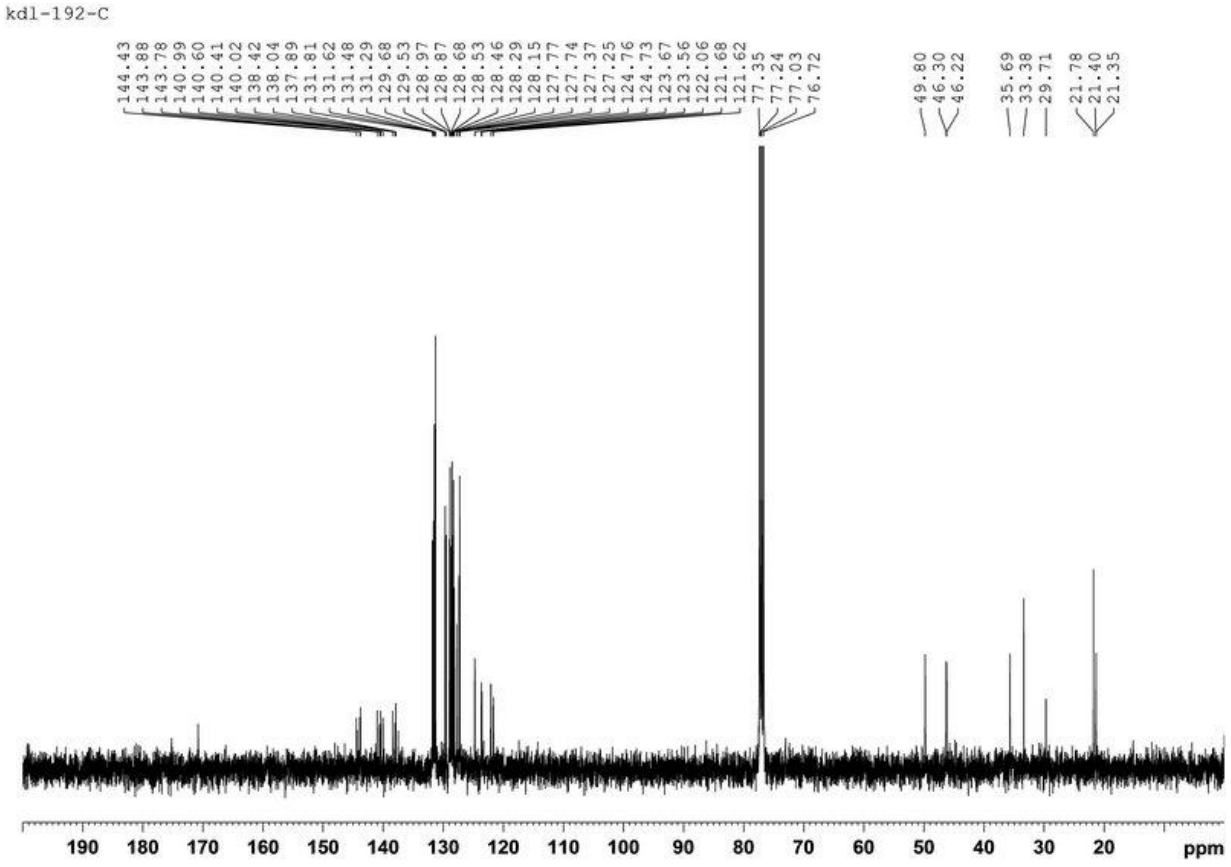


kdl-245-C

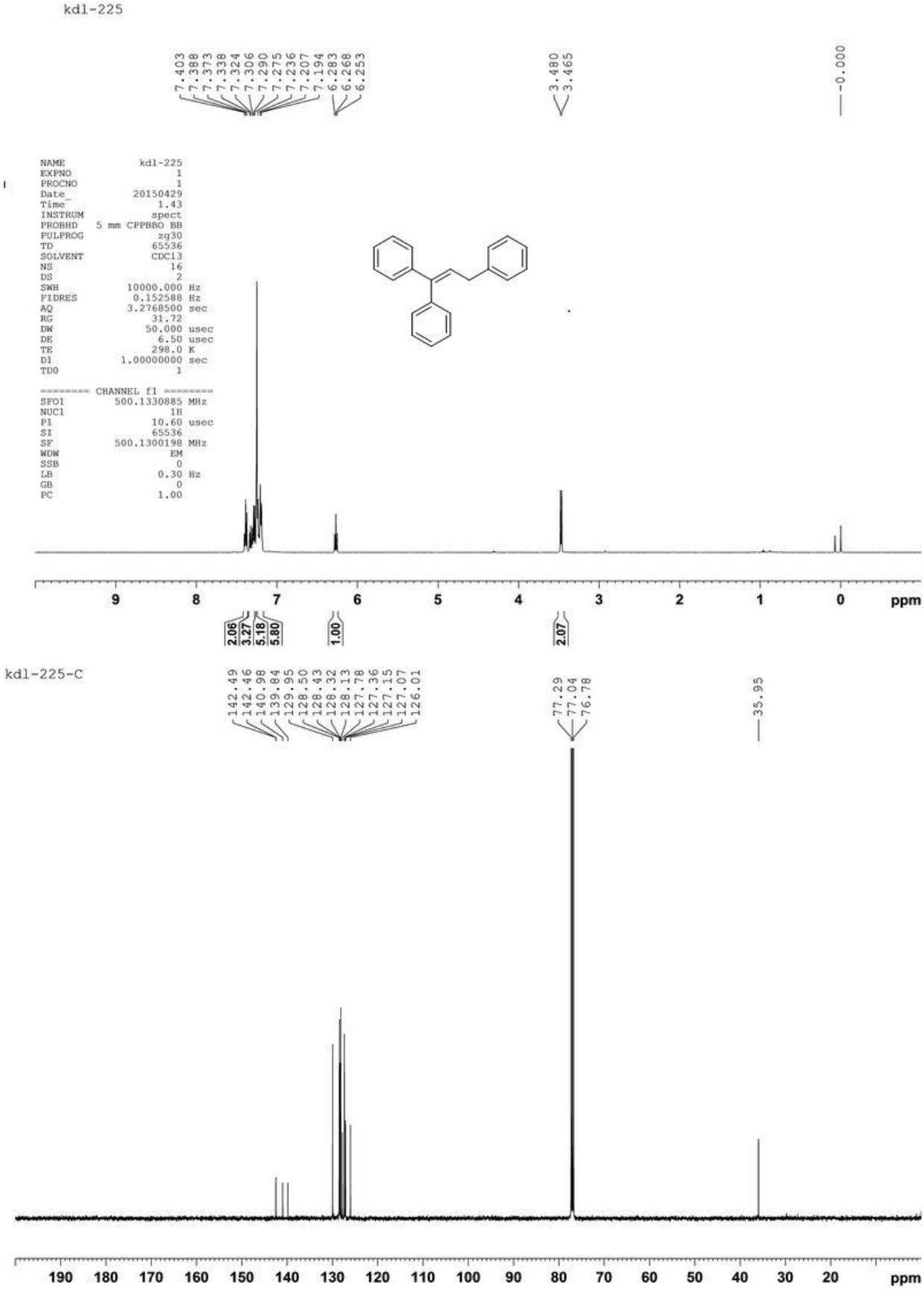


5g

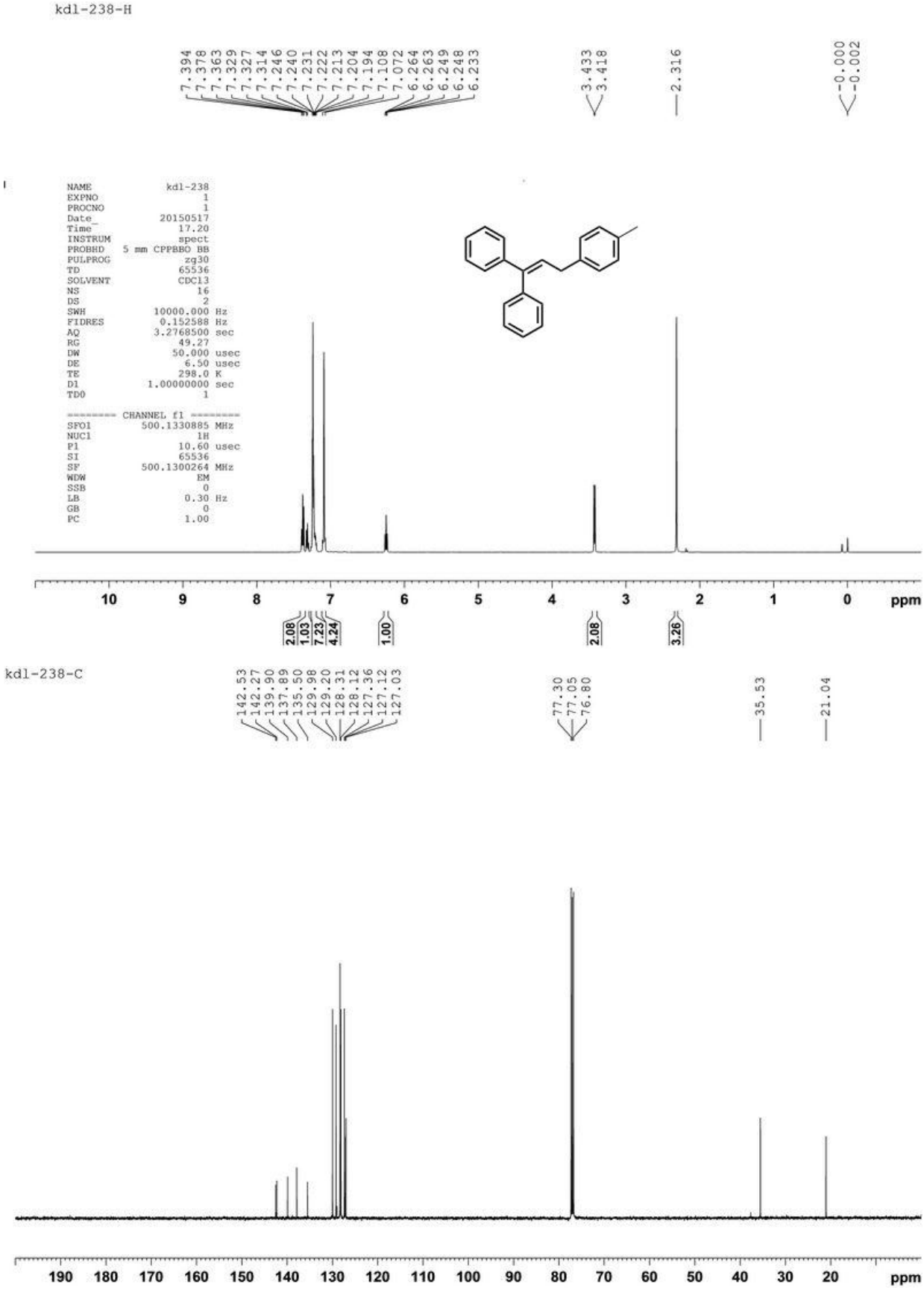

kdl-192-H
7.418
7.412
7.406
7.396
7.391
7.378
7.370
7.364
7.347
7.310
7.306
7.297
7.292
7.277
7.261
7.209
7.202
7.190
7.185
7.162
7.159
7.139
7.134
7.118
7.115
7.098
7.078
7.058
7.049
7.028
6.053
6.046
6.036
6.029
6.017
6.013
6.001
5.984
2.911
2.907
2.889
2.871
2.109
2.104
2.014
1.988
1.667
1.256
0.071
-0.000
NAME kdl-192
EXPNO 1
PROCNO 1
Date_ 20150408
Time_ 23.46
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 206.33
DW 62.400 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.2424716 MHz
NUC1 1H
P1 14.80 usec
SI 65536
SF 400.2400092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Br
N
O
9
8
7
6
5
4
3
2
1
0
ppm
0.95
2.64
0.98
1.01
3.13
1.00
1.14
0.92
3.03
1.69
1.47


kdl-192-C
144.43
143.88
143.78
140.99
140.60
140.41
140.02
138.42
138.04
137.89
131.81
131.62
131.48
131.29
129.68
129.53
128.97
128.87
128.68
128.53
128.46
128.29
128.15
127.77
127.74
127.37
127.25
124.76
124.73
123.67
123.56
122.06
121.68
121.62
77.35
77.24
77.03
76.72
49.80
46.30
46.22
35.69
33.38
29.71
21.78
21.40
21.35
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

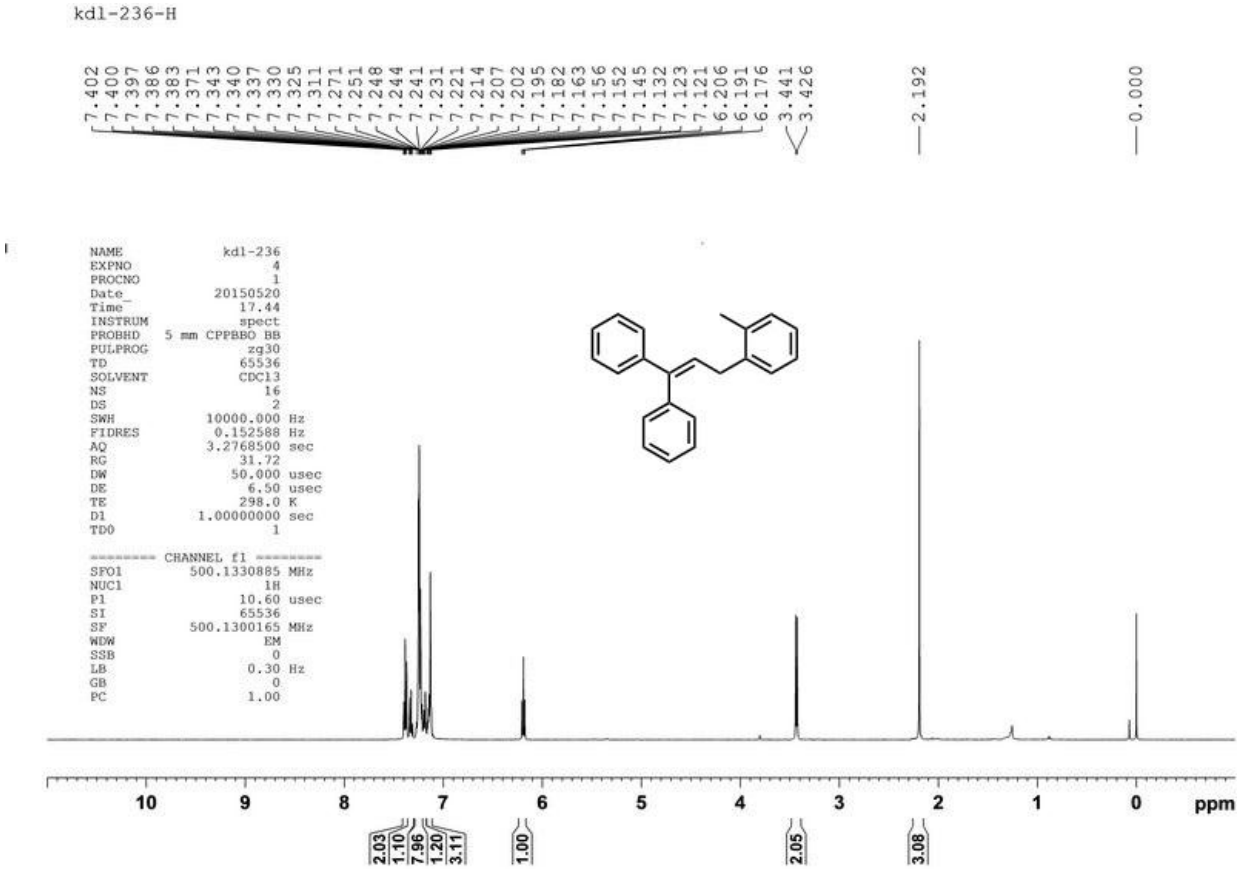
7aa

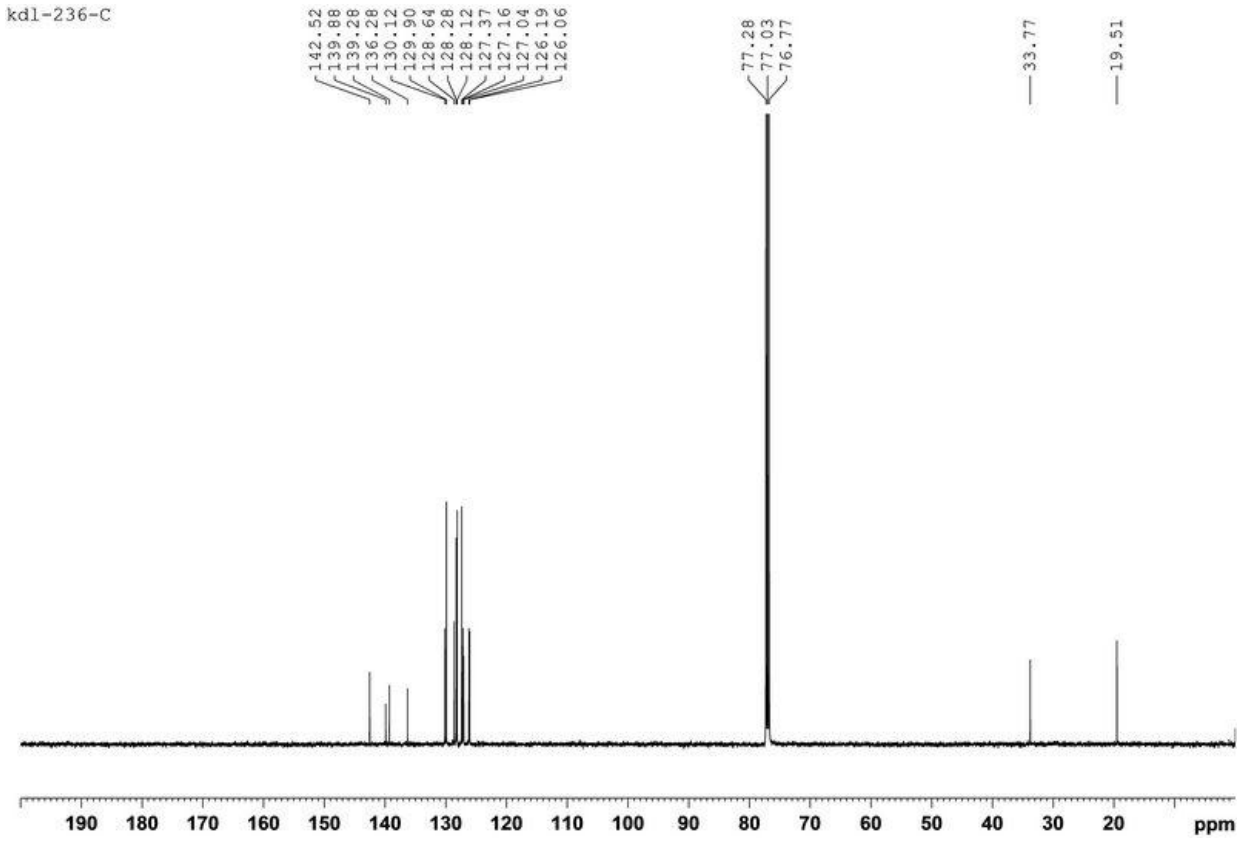

kdl-225
7.403
7.388
7.373
7.338
7.324
7.306
7.290
7.275
7.236
7.207
7.194
6.283
6.268
6.253
3.480
3.465
-0.000
NAME kdl-225
EXPNO 1
PROCNO 1
Date_ 20150429
Time 1.43
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 31.72
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 10.60 usec
SI 65536
SF 500.1300198 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
2.06
3.27
5.18
5.80
1.00
2.07
kdl-225-C
142.49
142.46
140.98
139.84
129.95
128.50
128.43
128.32
128.13
127.78
127.36
127.15
127.07
126.01
77.29
77.04
76.78
35.95
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

7ab

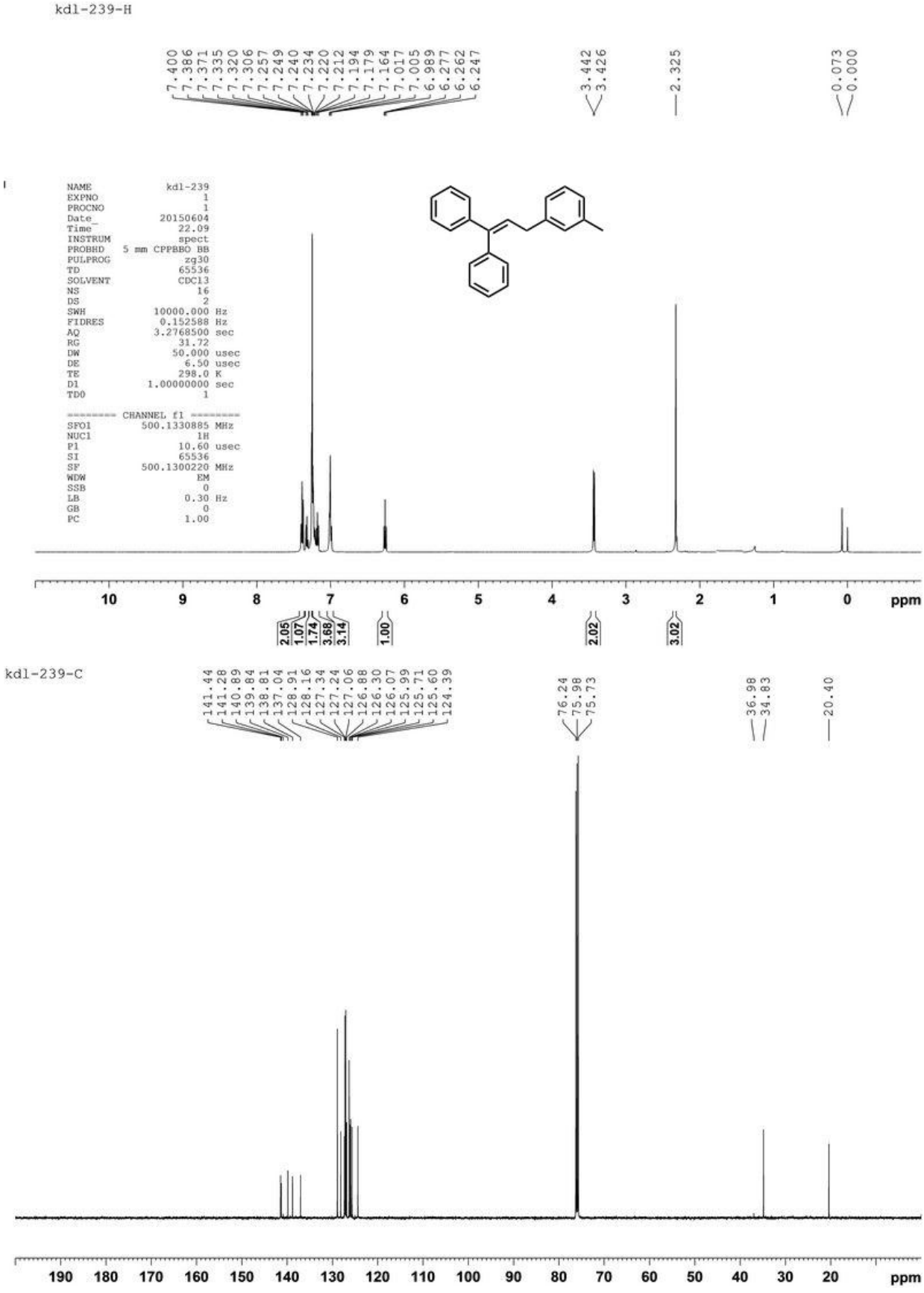

kdl-238-H
7.394
7.378
7.363
7.329
7.327
7.314
7.246
7.240
7.231
7.222
7.213
7.204
7.194
7.108
7.072
6.264
6.263
6.249
6.248
6.233
3.433
3.418
2.316
-0.000
-0.002
NAME kdl-238
EXPNO 1
PROCNO 1
Date_ 20150517
Time 17.20
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 49.27
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 10.60 usec
SI 65536
SF 500.1300264 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 0 ppm
2.08 1.03 7.23 4.24 1.00 2.08 3.26
kdl-238-C
142.53
142.27
139.90
137.89
135.50
129.98
129.20
128.31
128.12
127.36
127.12
127.03
77.30
77.05
76.80
35.53
21.04
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

7ac

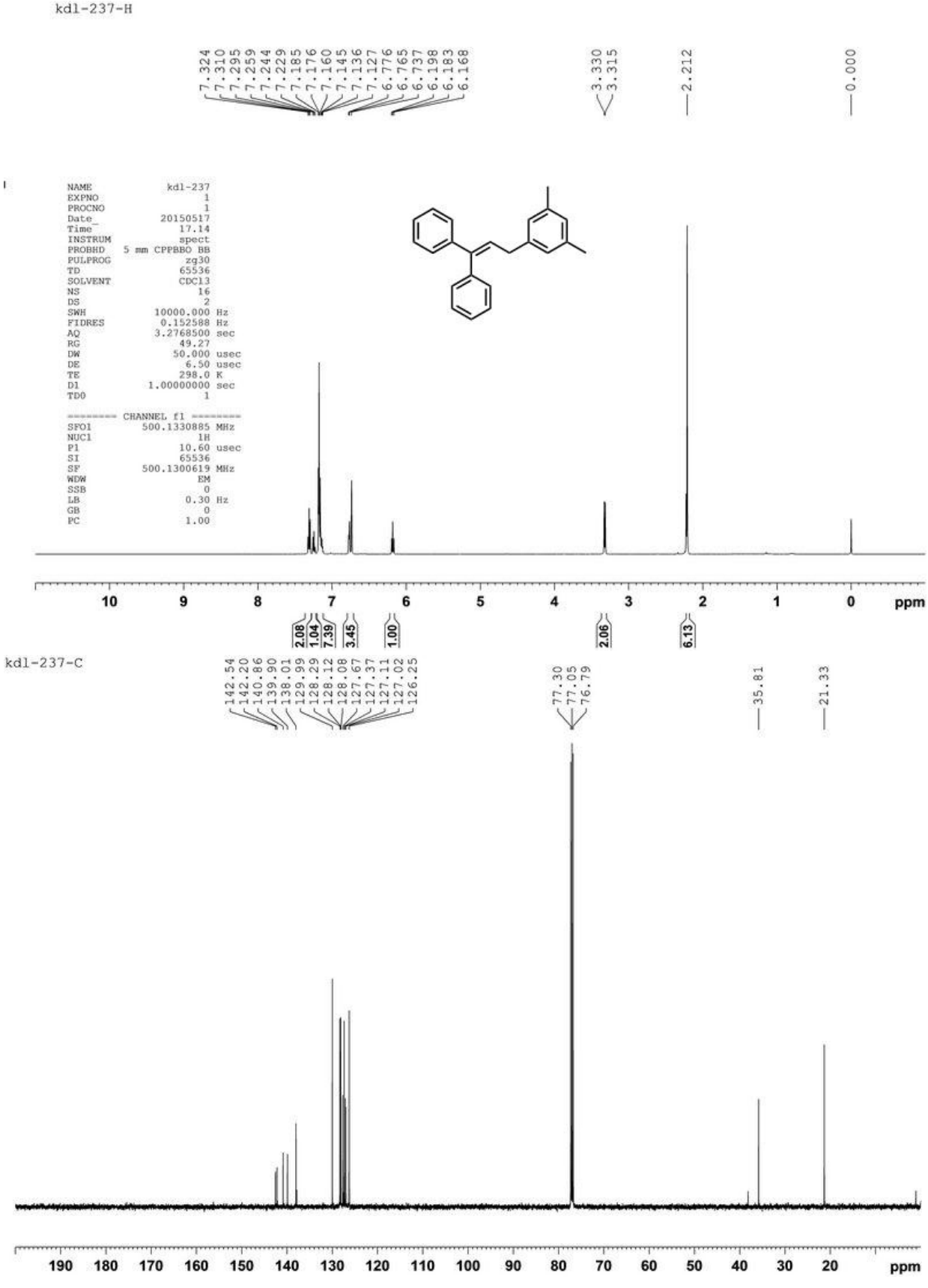

kdl-236-H
7.402
7.400
7.397
7.386
7.383
7.371
7.343
7.340
7.337
7.330
7.325
7.311
7.271
7.251
7.248
7.244
7.241
7.231
7.221
7.214
7.207
7.202
7.195
7.182
7.163
7.156
7.152
7.145
7.132
7.123
7.121
6.206
6.191
6.176
3.441
3.426
2.192
0.000
NAME kdl-236
EXPNO 4
PROCNO 1
Date_ 20150520
Time_ 17.44
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 31.72
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 10.60 usec
SI 65536
SF 500.1300165 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 0 ppm
2.03
1.10
7.96
1.20
3.11
1.00
2.05
3.08


kdl-236-C
142.52
139.88
139.28
136.28
130.12
129.90
128.64
128.28
128.12
127.37
127.16
127.04
126.19
126.06
77.28
77.03
76.77
33.77
19.51
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

7ad

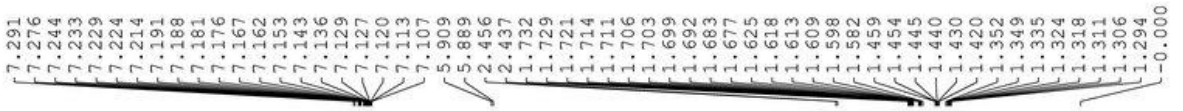

kdl-239-H
7.400
7.386
7.371
7.335
7.320
7.306
7.257
7.249
7.240
7.234
7.220
7.212
7.194
7.179
7.164
7.017
7.005
6.989
6.277
6.262
6.247
3.442
3.426
2.325
0.073
0.000
NAME kdl-239
EXPNO 1
PROCNO 1
Date_ 20150604
Time_ 22.09
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 31.72
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 10.60 usec
SI 65536
SF 500.1300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 0 ppm
2.05 1.07 1.74 3.68 3.14 1.00 2.02 3.02
kdl-239-C
141.44
141.28
140.89
139.84
138.81
137.04
128.91
128.16
127.34
127.24
127.06
126.88
126.30
126.07
125.99
125.71
125.60
124.39
76.24
75.98
75.73
36.98
34.83
20.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

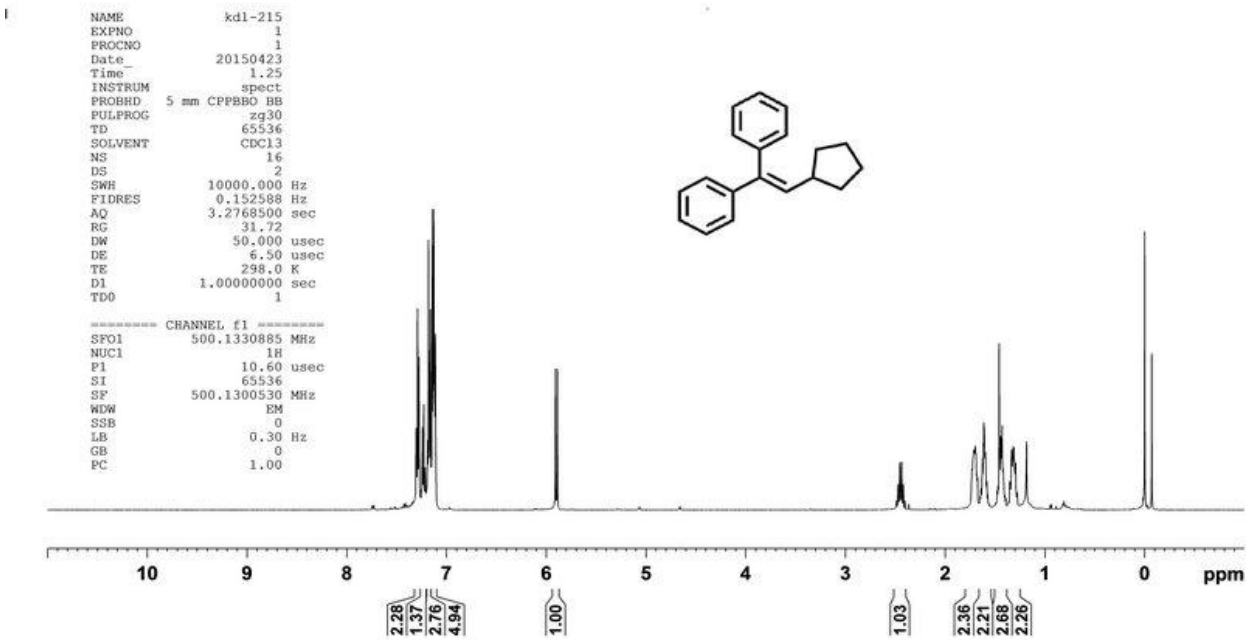
7ae


kdl-237-H
7.324
7.310
7.295
7.259
7.244
7.229
7.185
7.176
7.160
7.145
7.136
7.127
6.776
6.765
6.737
6.198
6.183
6.168
3.330
3.315
2.212
0.000
NAME kdl-237
EXPNO 1
PROCNO 1
Date_ 20150517
Time 17.14
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 49.27
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 10.60 usec
SI 65536
SF 500.1300619 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 0 ppm
2.08
1.04
7.39
3.45
1.00
2.06
6.13
kdl-237-C
142.54
142.20
140.86
139.90
138.01
129.99
128.29
128.12
128.08
127.67
127.37
127.11
127.02
126.25
77.30
77.05
76.79
35.81
21.33
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

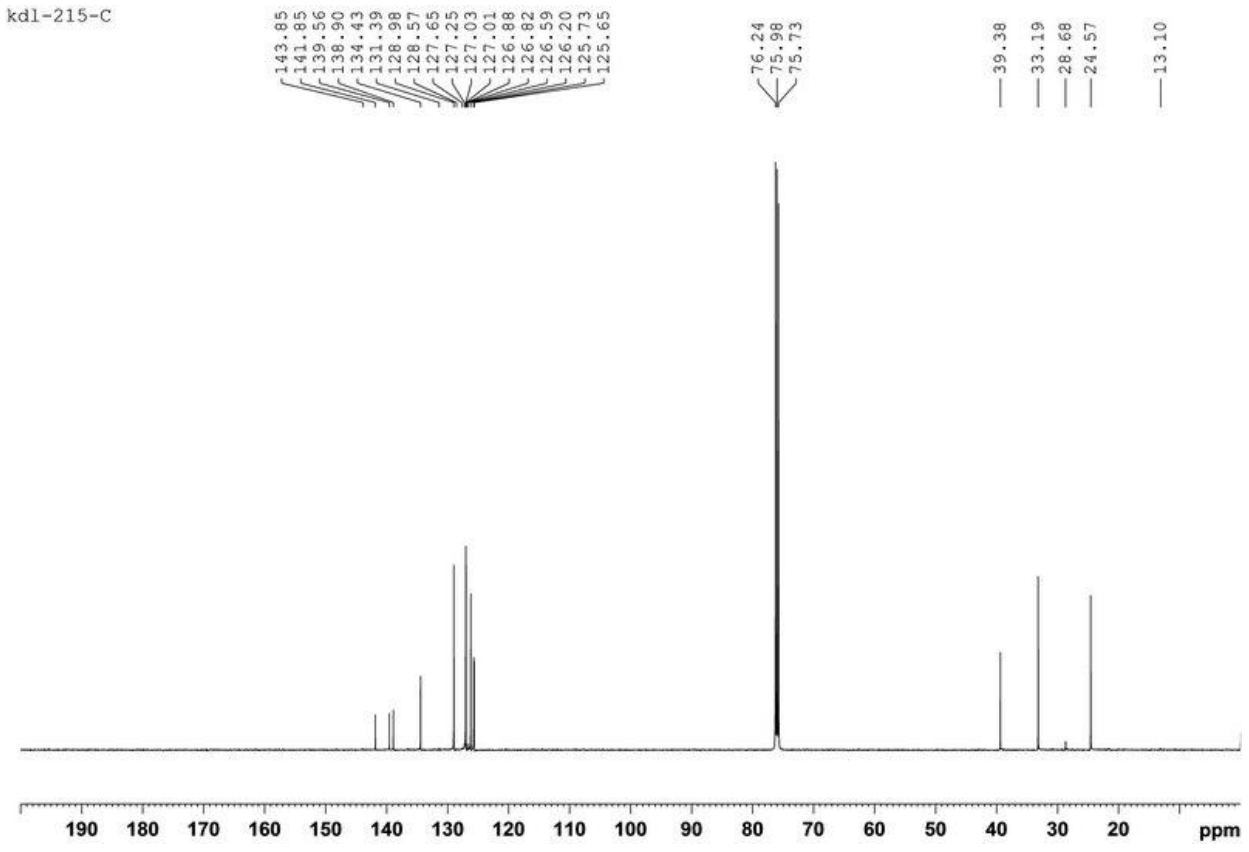
9aa

kdl-215-H

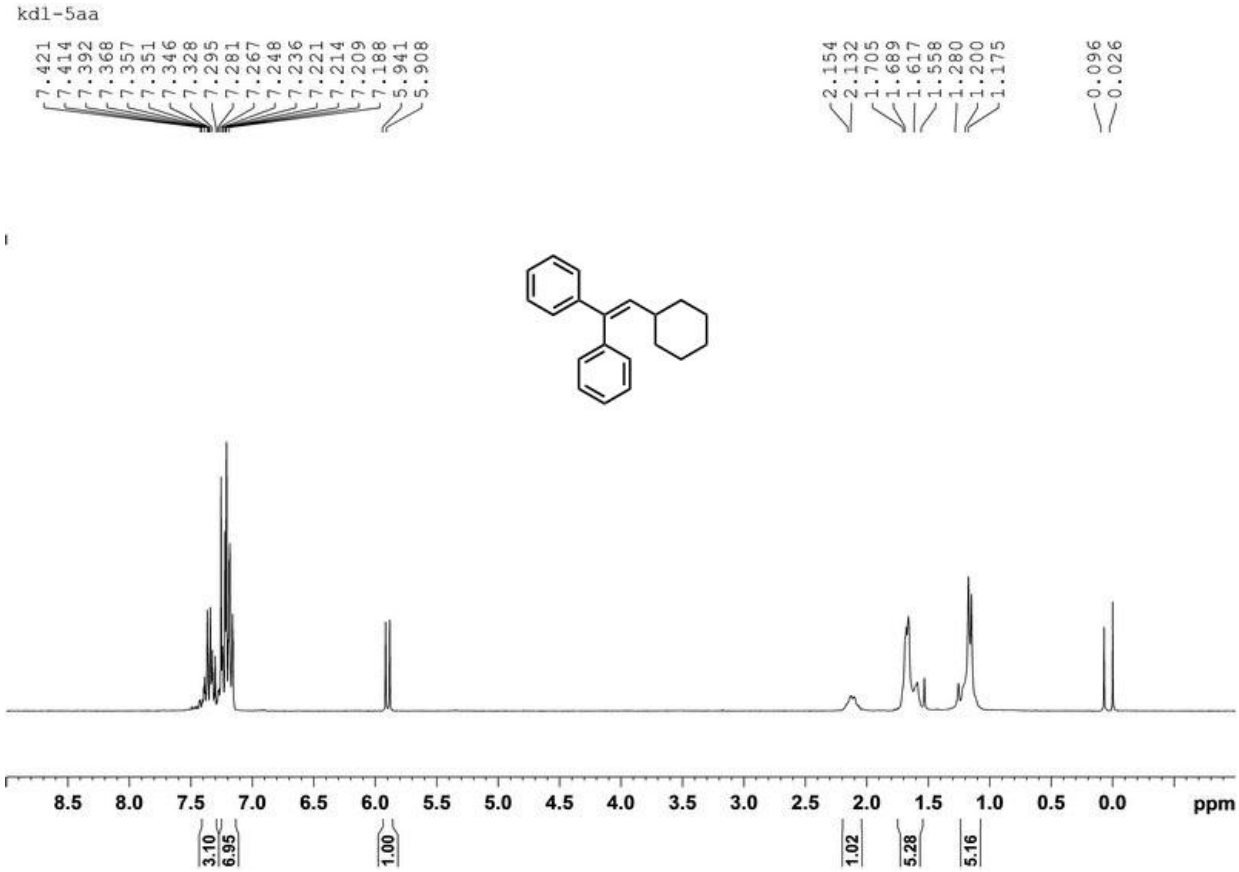


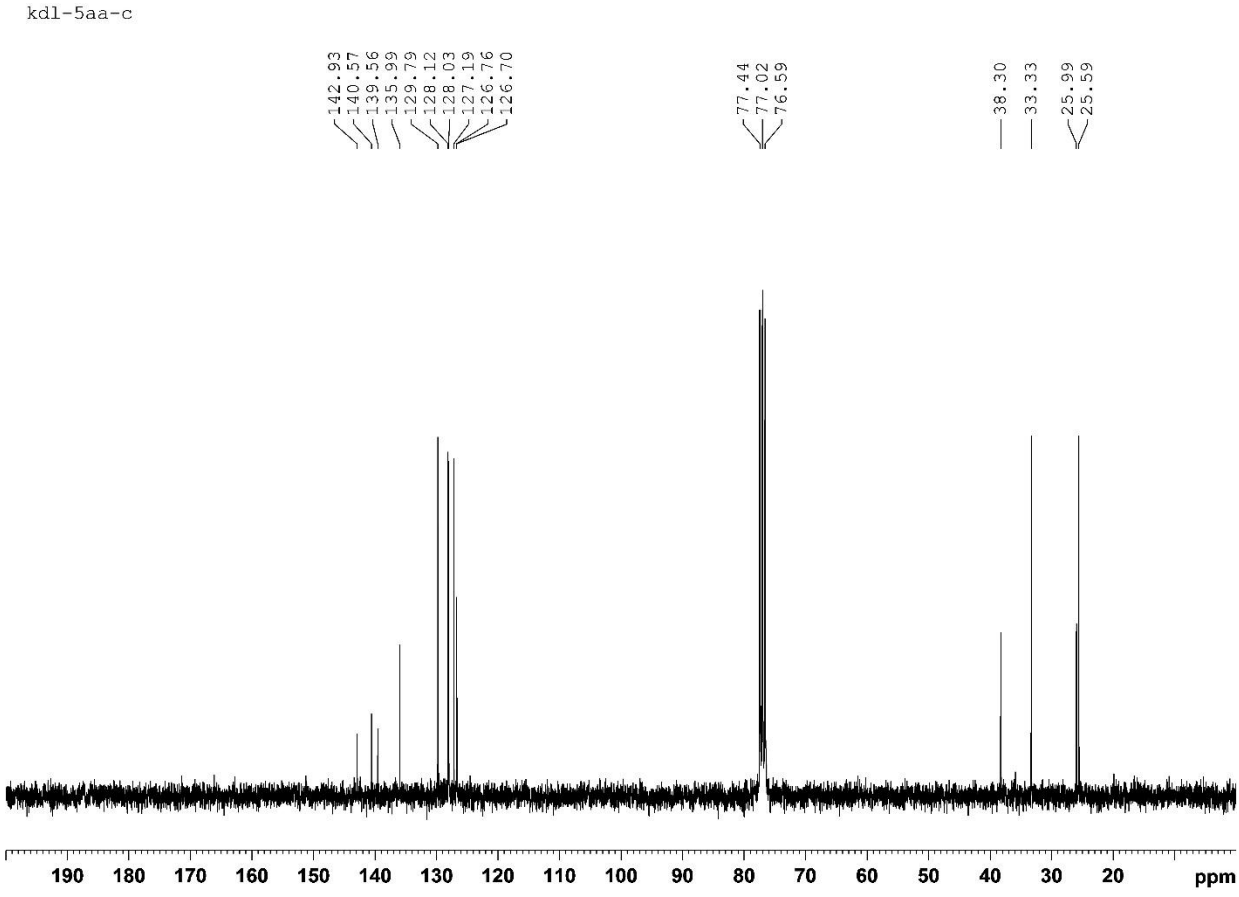


kdl-215-C

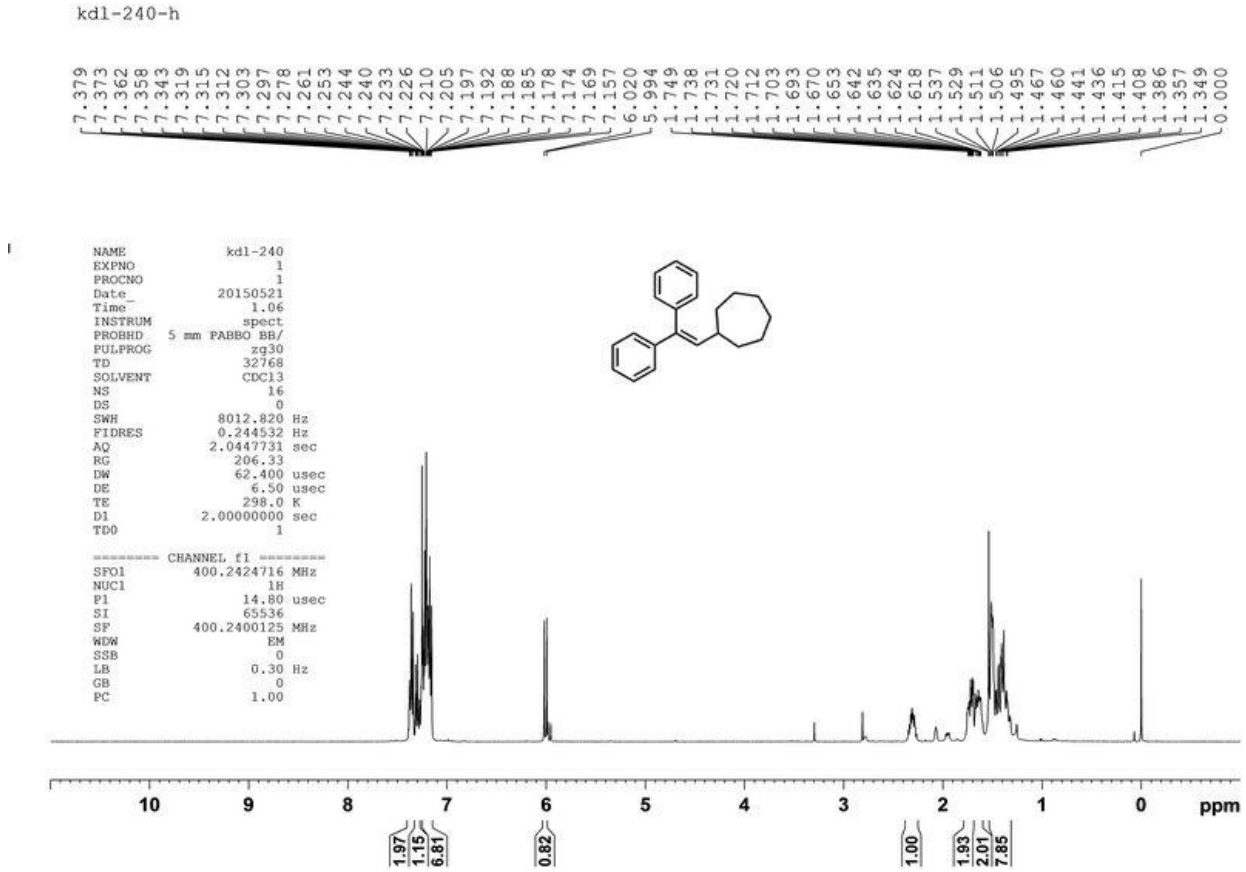


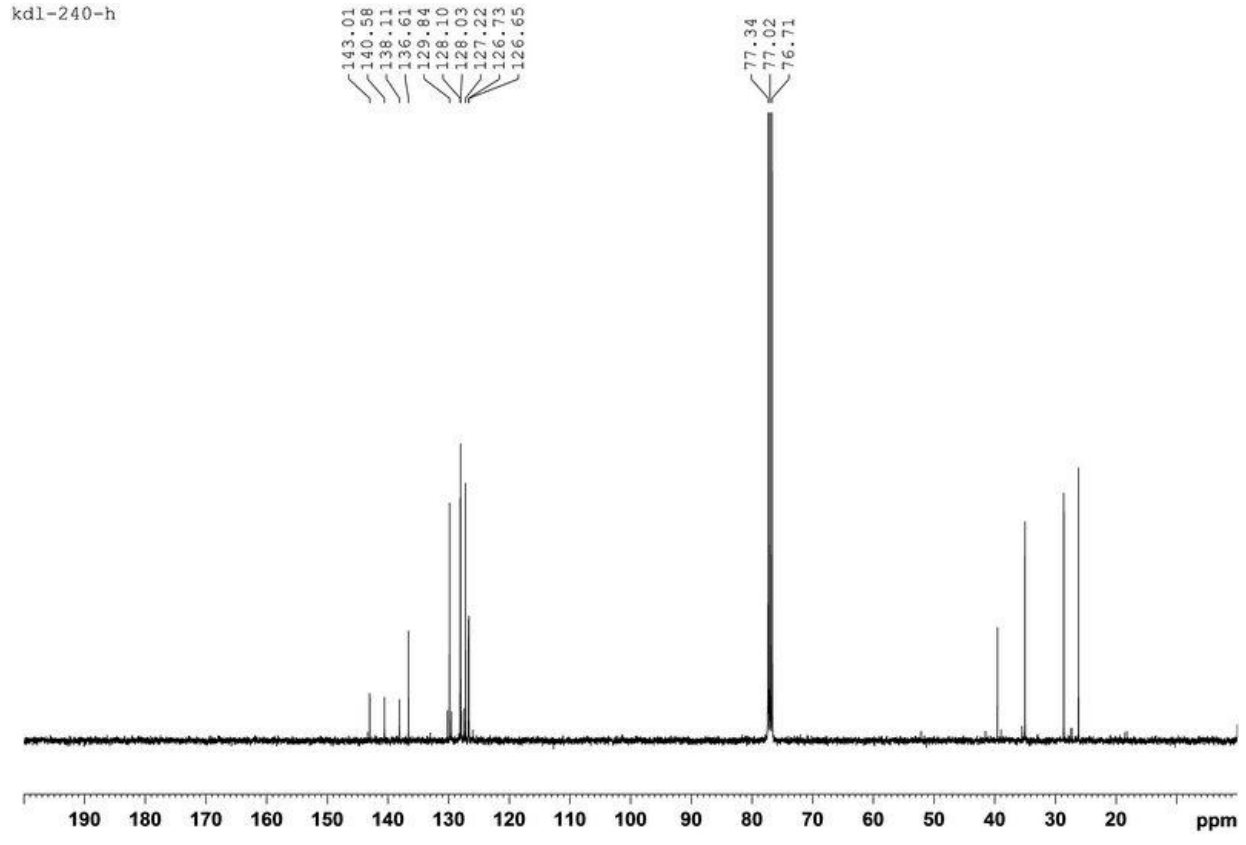
9ab


kdl-5aa
7.421
7.414
7.392
7.368
7.357
7.351
7.346
7.328
7.295
7.281
7.267
7.248
7.236
7.221
7.214
7.209
7.188
5.941
5.908
2.154
2.132
1.705
1.689
1.617
1.558
1.280
1.200
1.175
0.096
0.026
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
3.10
6.95
1.00
1.02
5.28
5.16


kdl-5aa-c
142.93
140.57
139.56
135.99
129.79
128.12
128.03
127.19
126.76
126.70
77.44
77.02
76.59
38.30
33.33
25.99
25.59
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

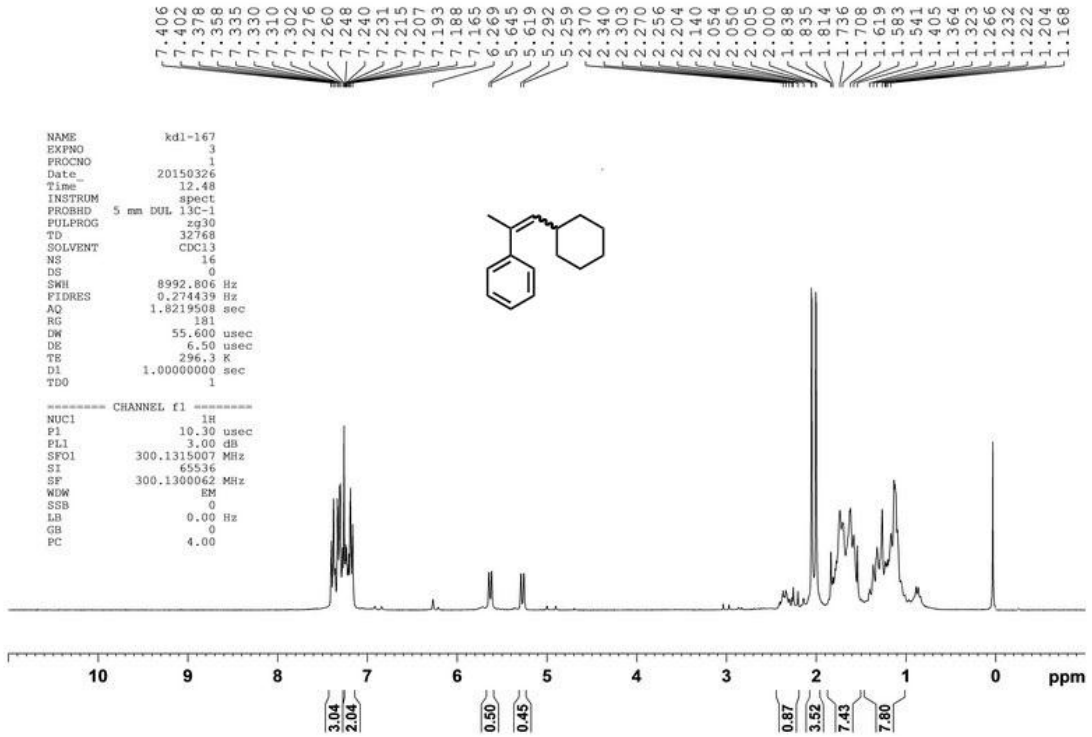
9ac


kdl-240-h
7.379
7.373
7.362
7.358
7.343
7.319
7.315
7.312
7.303
7.297
7.278
7.261
7.253
7.244
7.240
7.233
7.226
7.210
7.205
7.197
7.192
7.188
7.185
7.178
7.174
7.169
7.157
6.020
5.994
1.749
1.738
1.731
1.720
1.712
1.703
1.693
1.670
1.653
1.642
1.635
1.624
1.618
1.537
1.529
1.511
1.506
1.495
1.467
1.460
1.441
1.436
1.415
1.408
1.386
1.357
1.349
0.000
NAME kdl-240
EXPNO 1
PROCNO 1
Date_ 20150521
Time 1.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 206.33
DW 62.400 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.2424716 MHz
NUC1 1H
P1 14.80 usec
SI 65536
SF 400.2400125 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 0 ppm
1.97
1.15
6.81
0.82
1.00
1.93
2.01
7.85

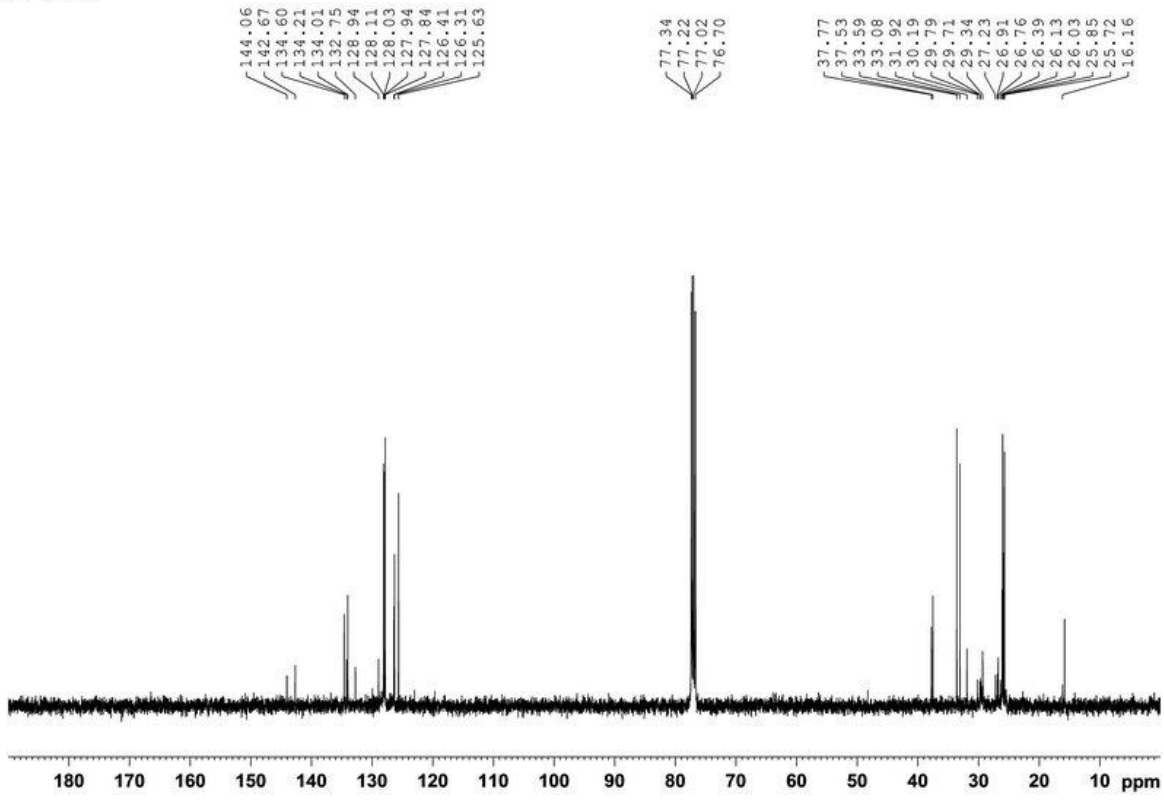

kdl-240-h
143.01
140.58
138.11
136.61
129.84
128.10
128.03
127.22
126.73
126.65
77.34
77.02
76.71
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

9bb

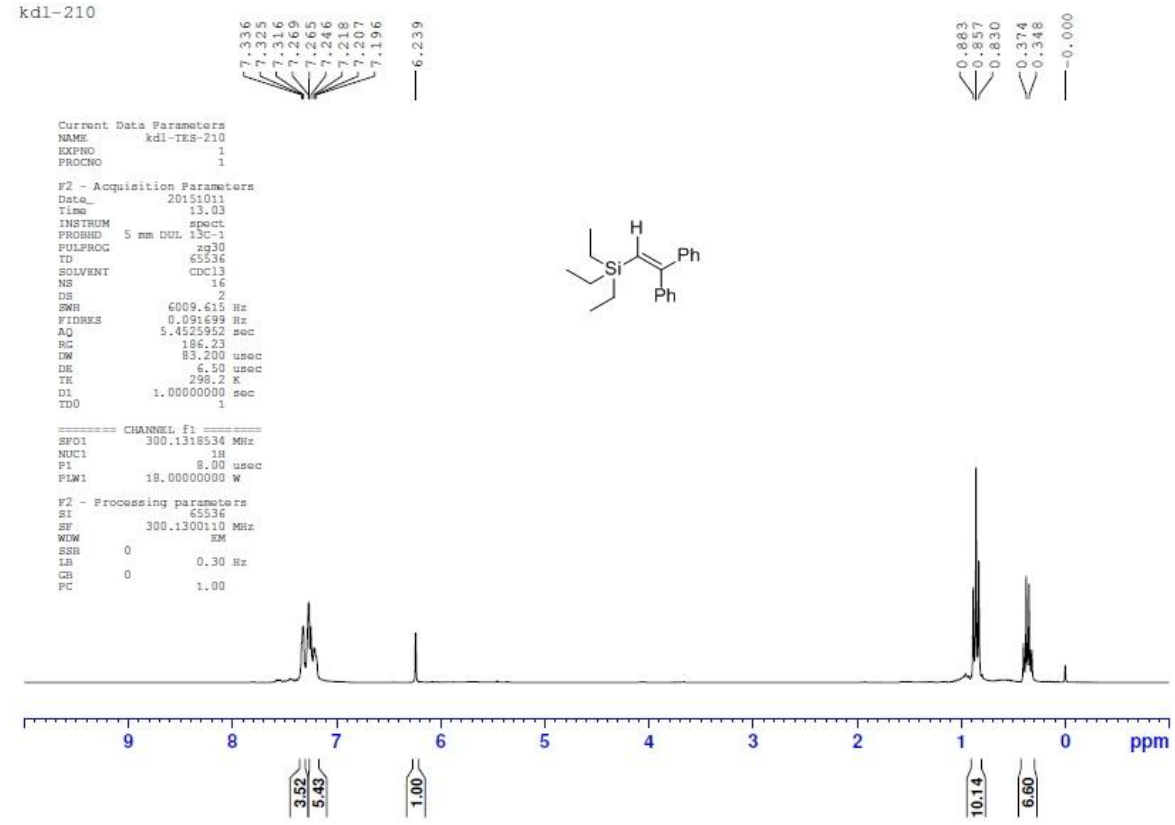
kdl-167- H

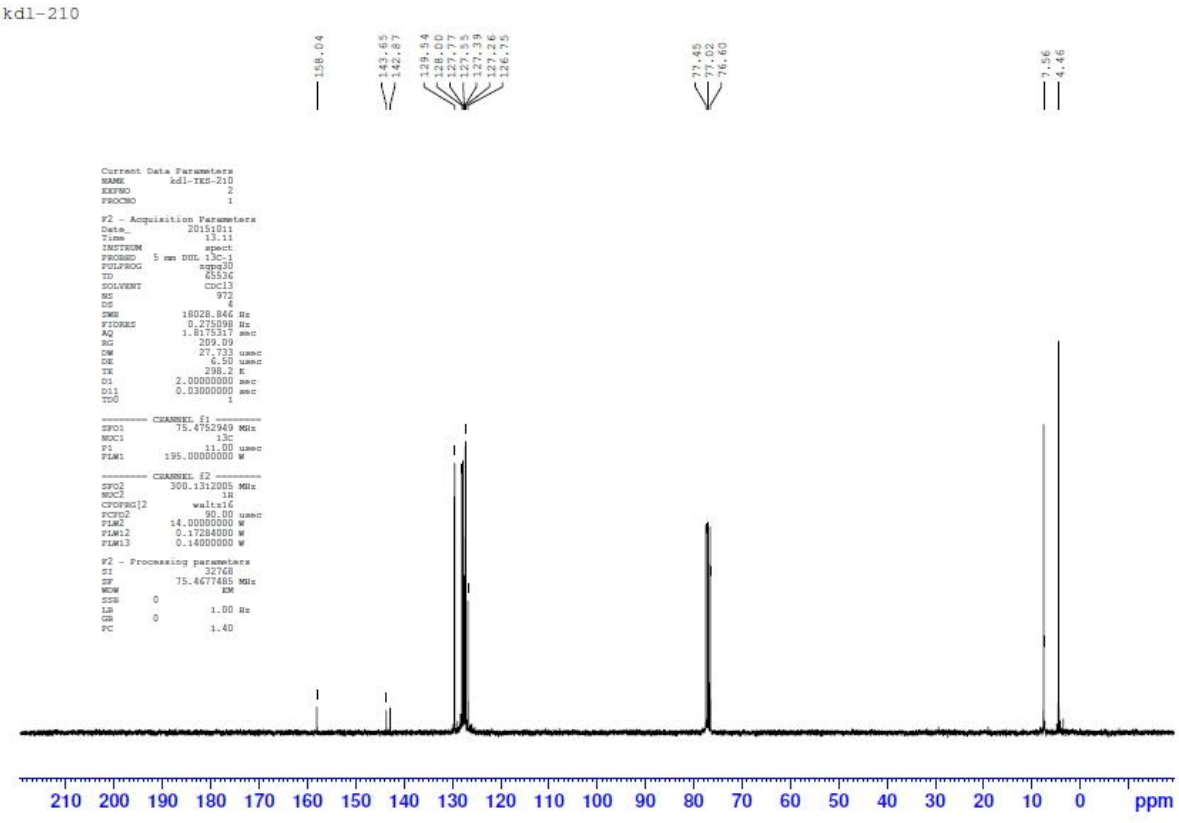


kdl-167-C



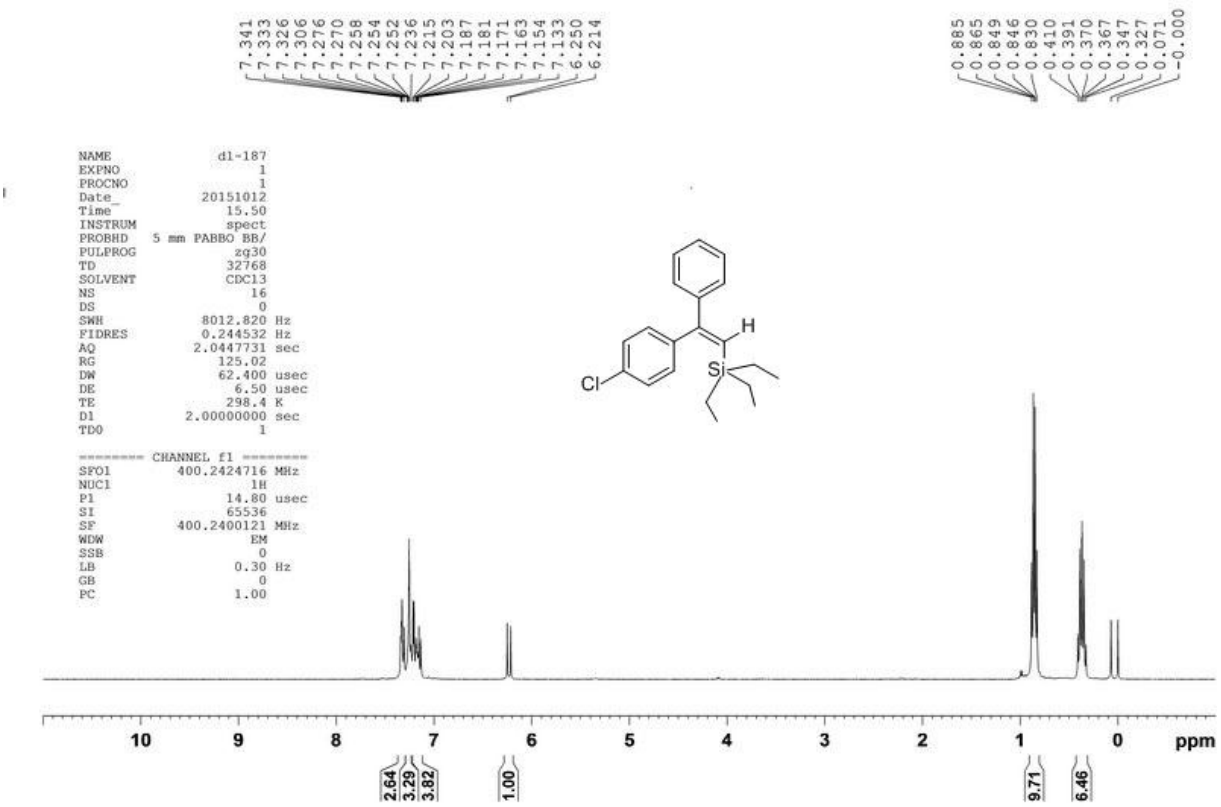
10a


kdl-210
7.336
7.325
7.316
7.269
7.265
7.246
7.218
7.207
7.196
6.239
0.883
0.857
0.830
0.374
0.348
-0.000
Current Data Parameters
NAME kdl-TES-210
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Data_ 20151011
Time 13.03
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6009.615 Hz
FIDRES 0.091699 Hz
AQ 5.4525952 sec
RG 186.23
DW 83.200 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 300.1318534 MHz
NUC1 1H
P1 8.00 usec
PLW1 18.00000000 W
F2 - Processing parameters
SI 65536
SF 300.1300110 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H
Si
Ph
Ph
9
8
7
6
5
4
3
2
1
0
ppm
3.52
5.43
1.00
10.14
6.60

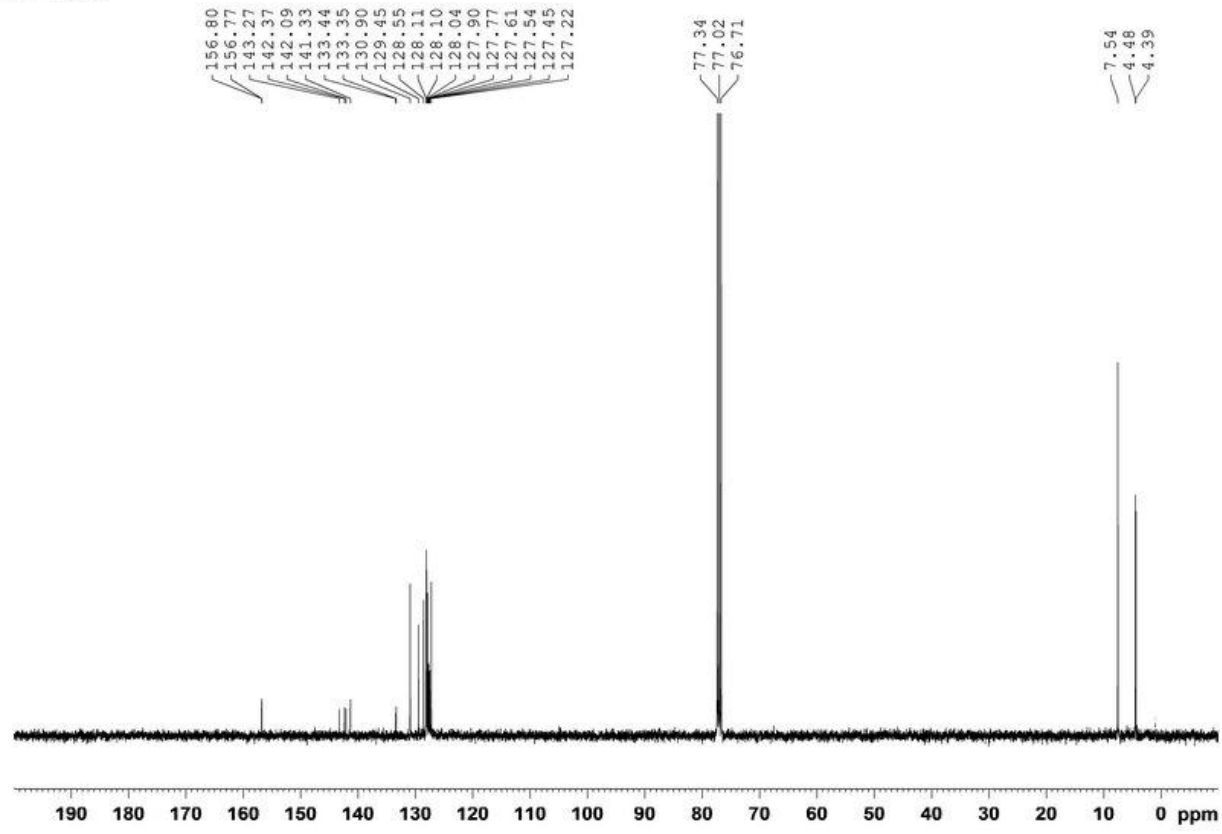

kdl-210
158.04
143.65
142.87
129.54
128.00
127.77
127.55
127.39
127.26
126.75
77.45
77.02
76.60
7.56
4.46
Current Data Parameters
NAME kdl-TES-210
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Data_ 20151011
Time 13.11
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 972
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175317 sec
RG 209.09
DW 27.733 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 75.4752949 MHz
NUC1 13c
P1 11.00 usec
PLW1 195.00000000 W
======== CHANNEL f2 ========
SFO2 300.1312005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 14.00000000 W
PLW12 0.17284000 W
PLW13 0.14000000 W
F2 - Processing parameters
SI 32768
SF 75.4677485 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

10b

kdl-187-h

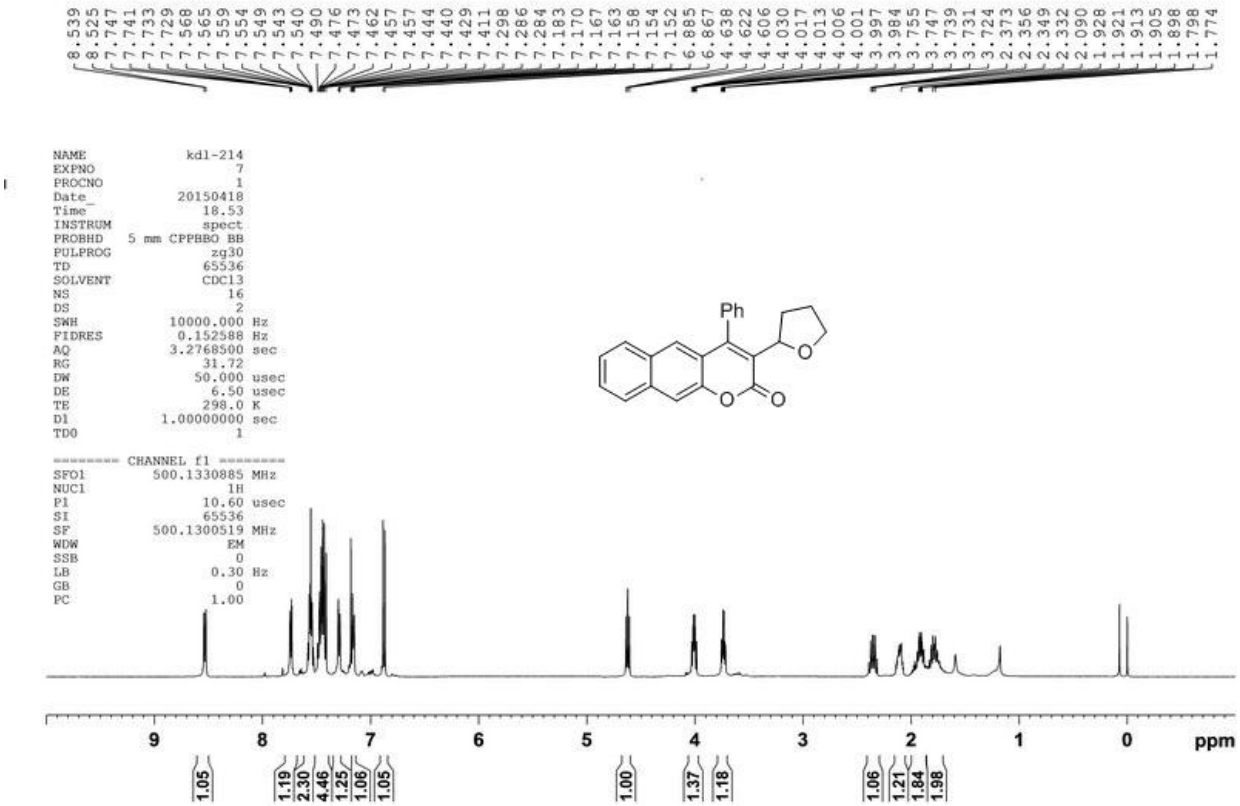


kdl-187-C

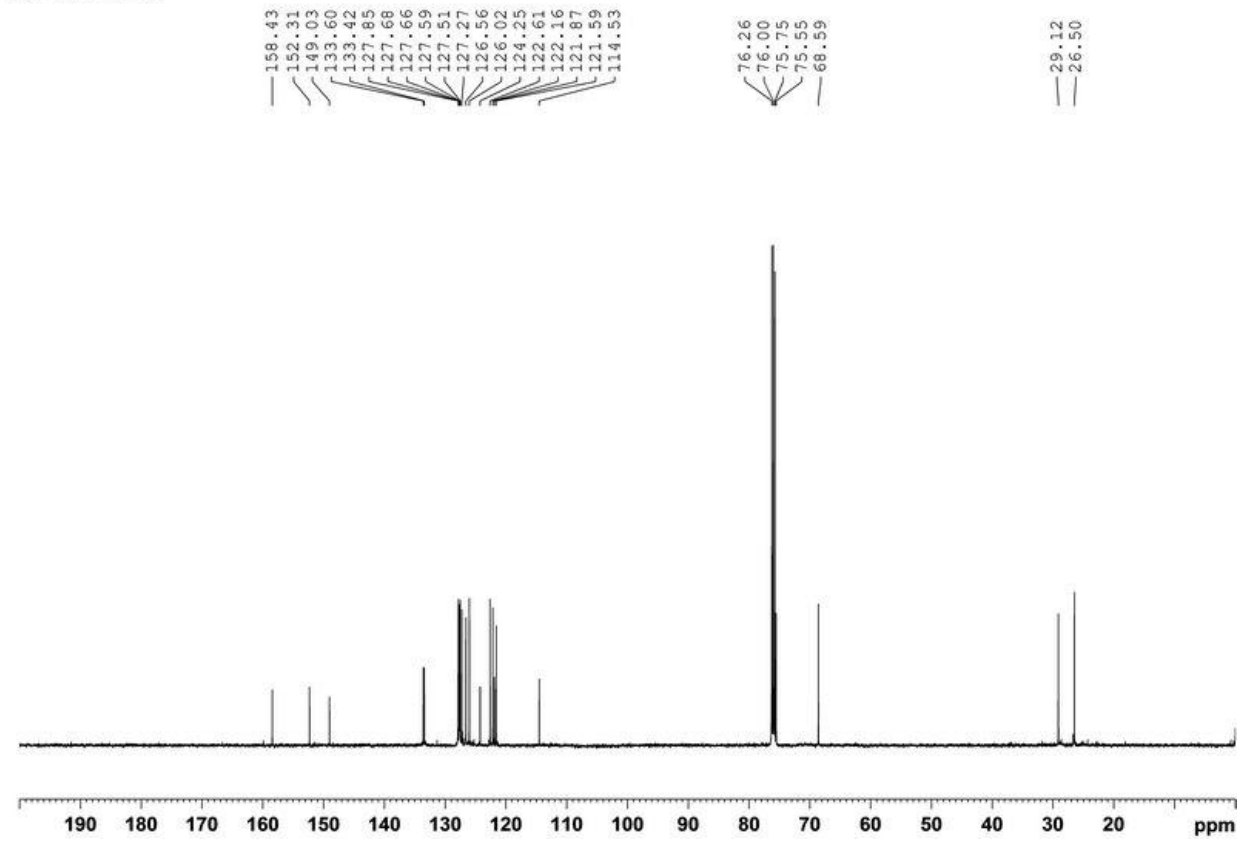


12b

kdl-214-THF-H

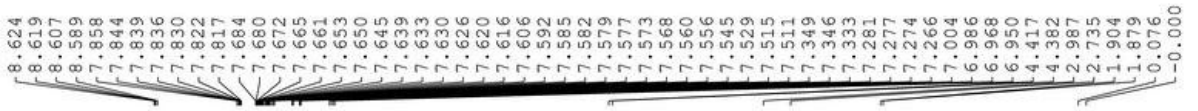


kdl-214-THF-C

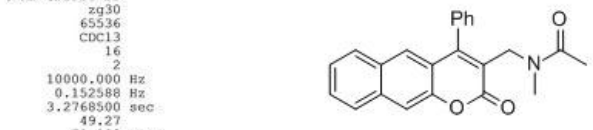


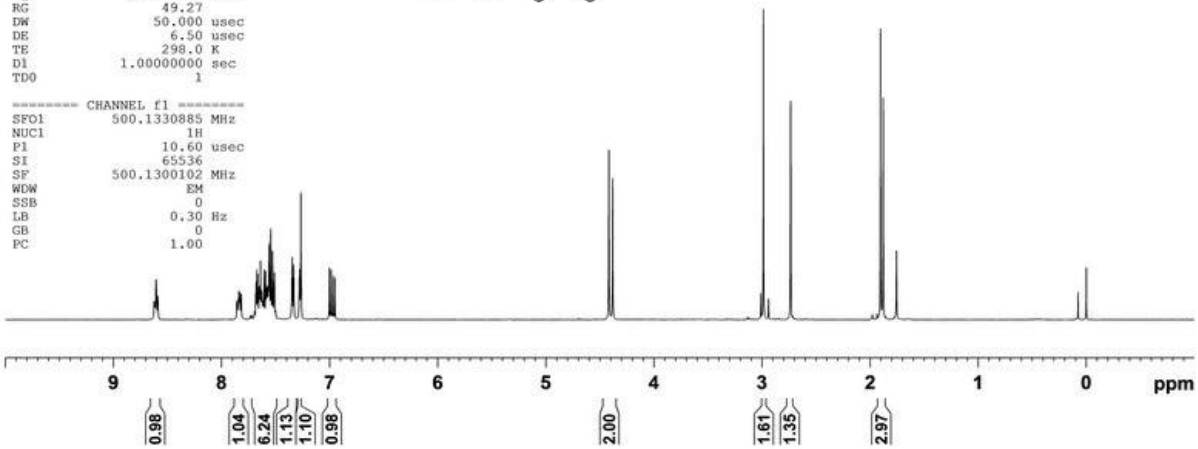
13b

kdl-214-DMA-H

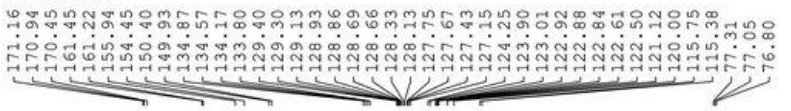


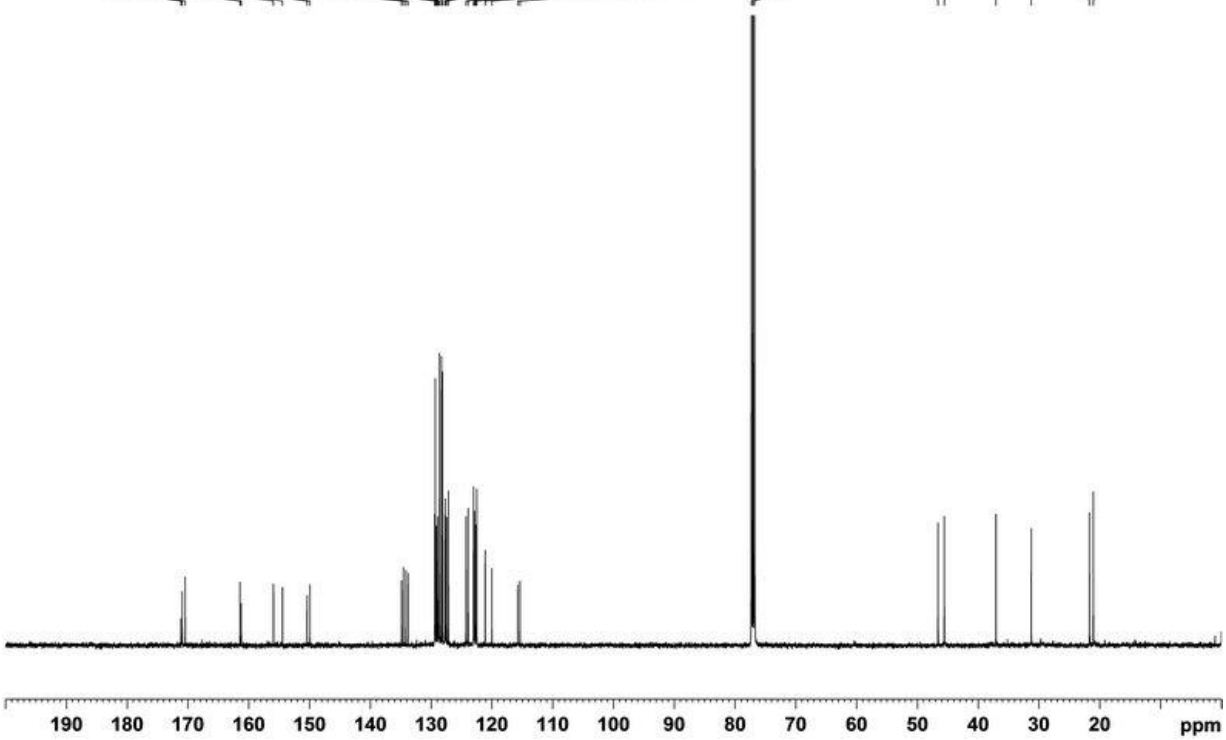
NAME	kdl-214
EXPNO	3
PROCNO	1
Date_	20150418
Time	14.08
INSTRUM	spect
PROBHD	5 mm CPPBBO BB
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	49.27
DW	50.000 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	500.1330885 MHz
NUC1	1H
P1	10.60 usec
SI	65536
SF	500.1300102 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00





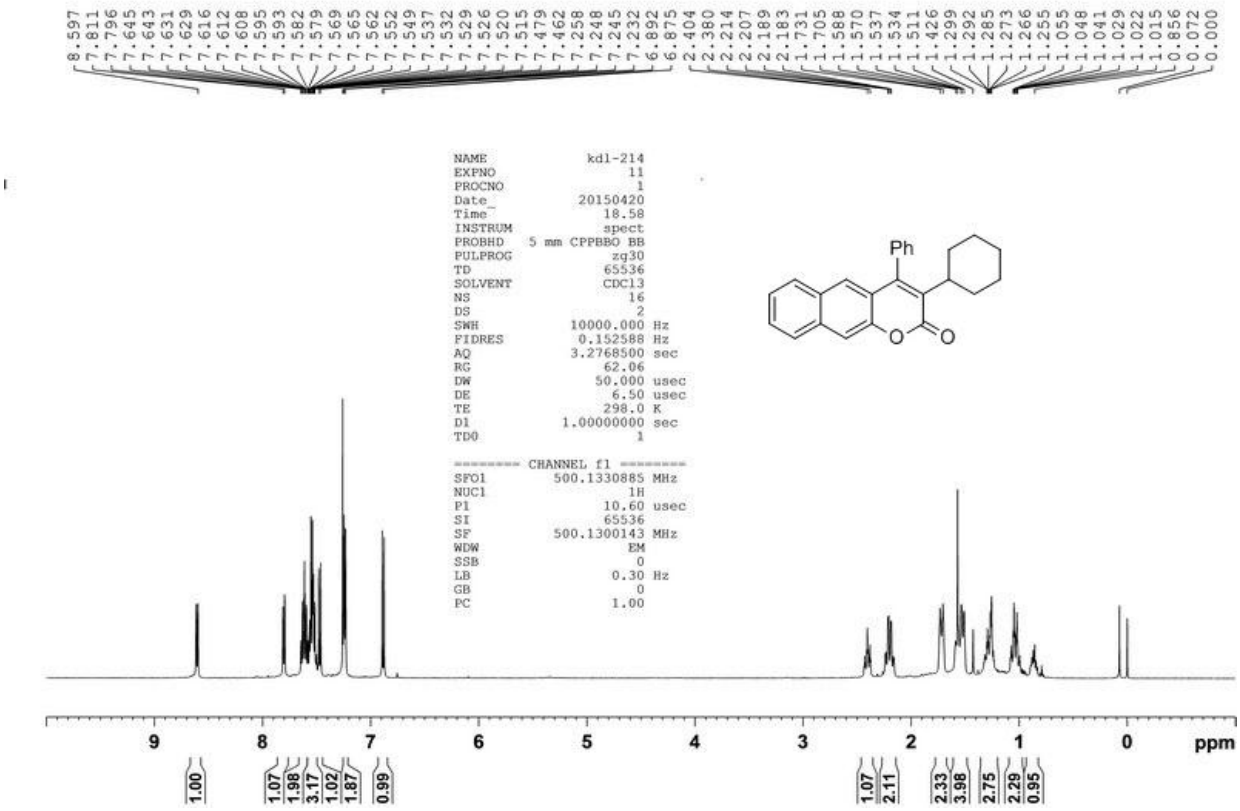
kdl-214-DMA-C



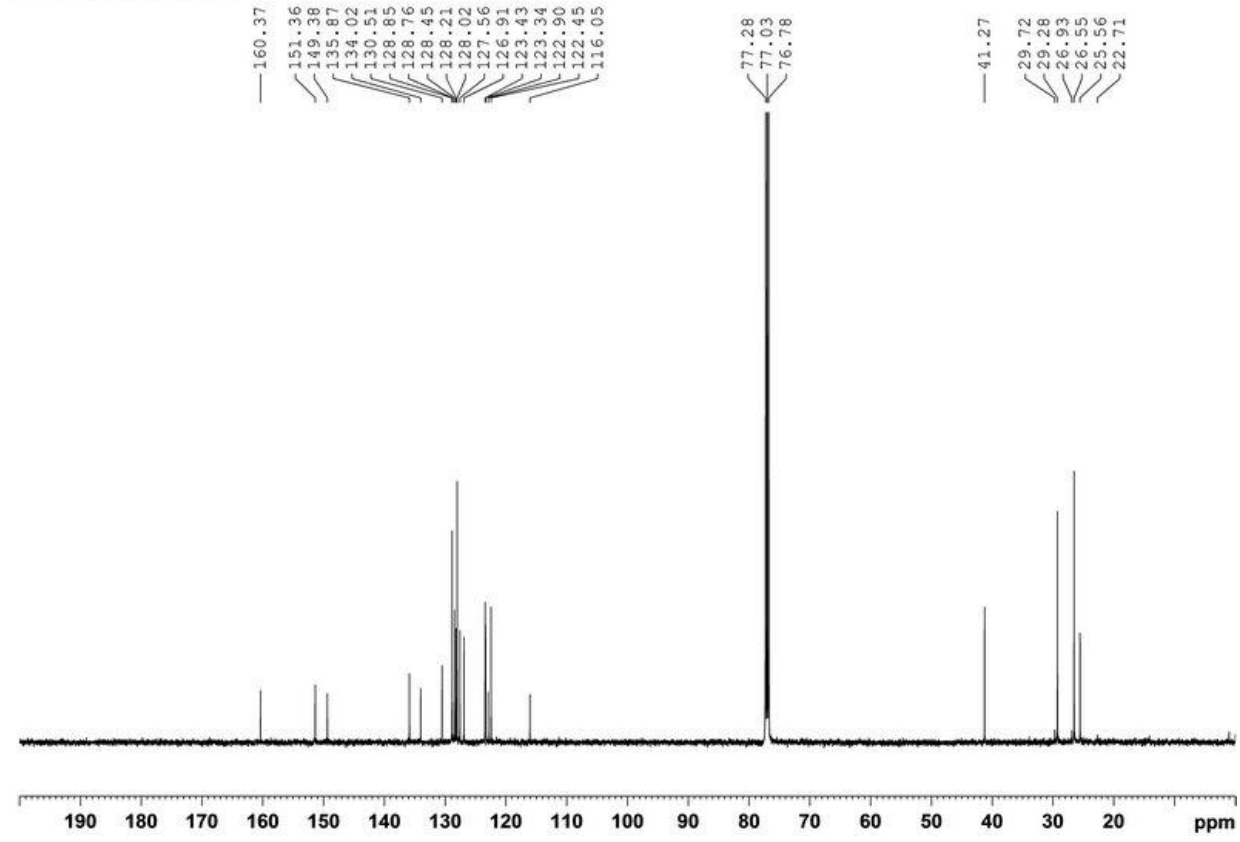


14b

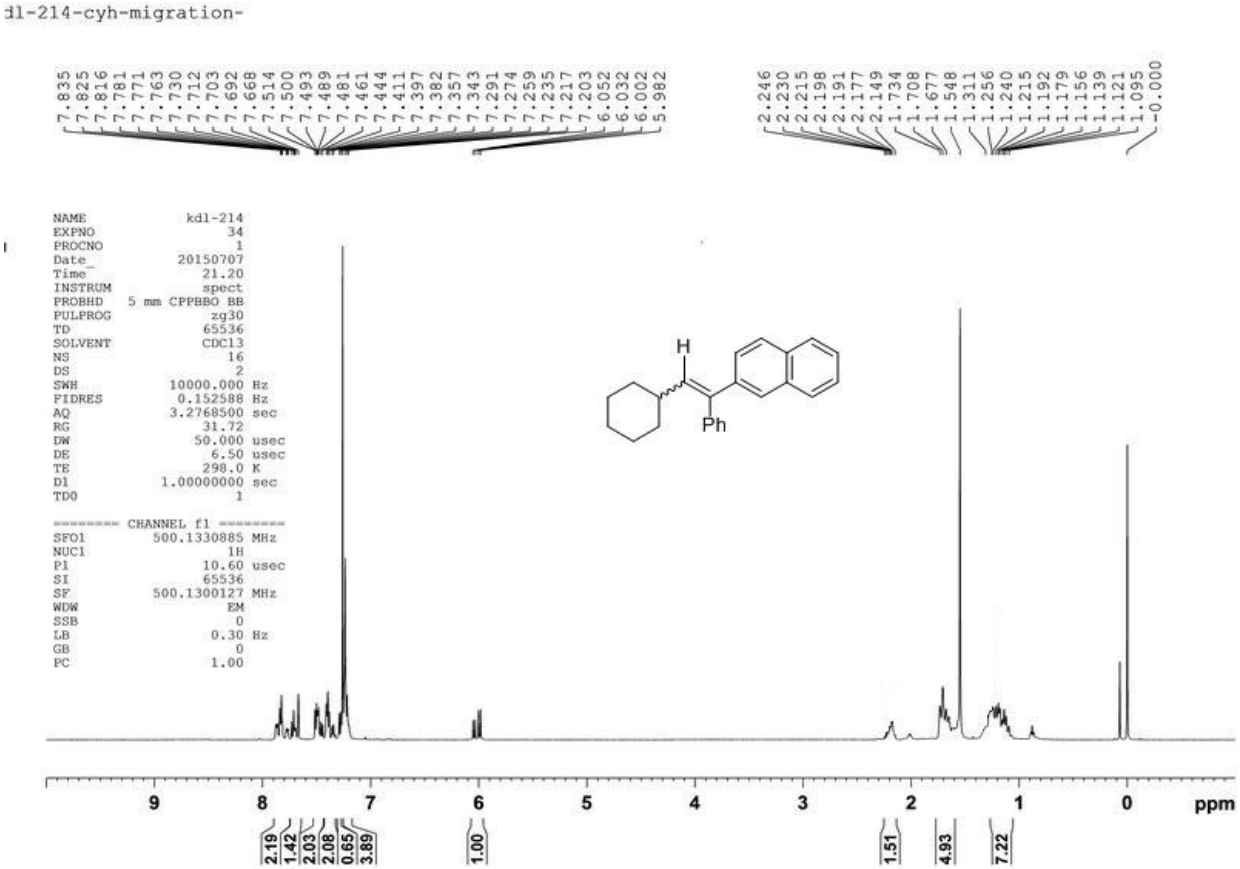
kdl-214-cyh-guanhuan

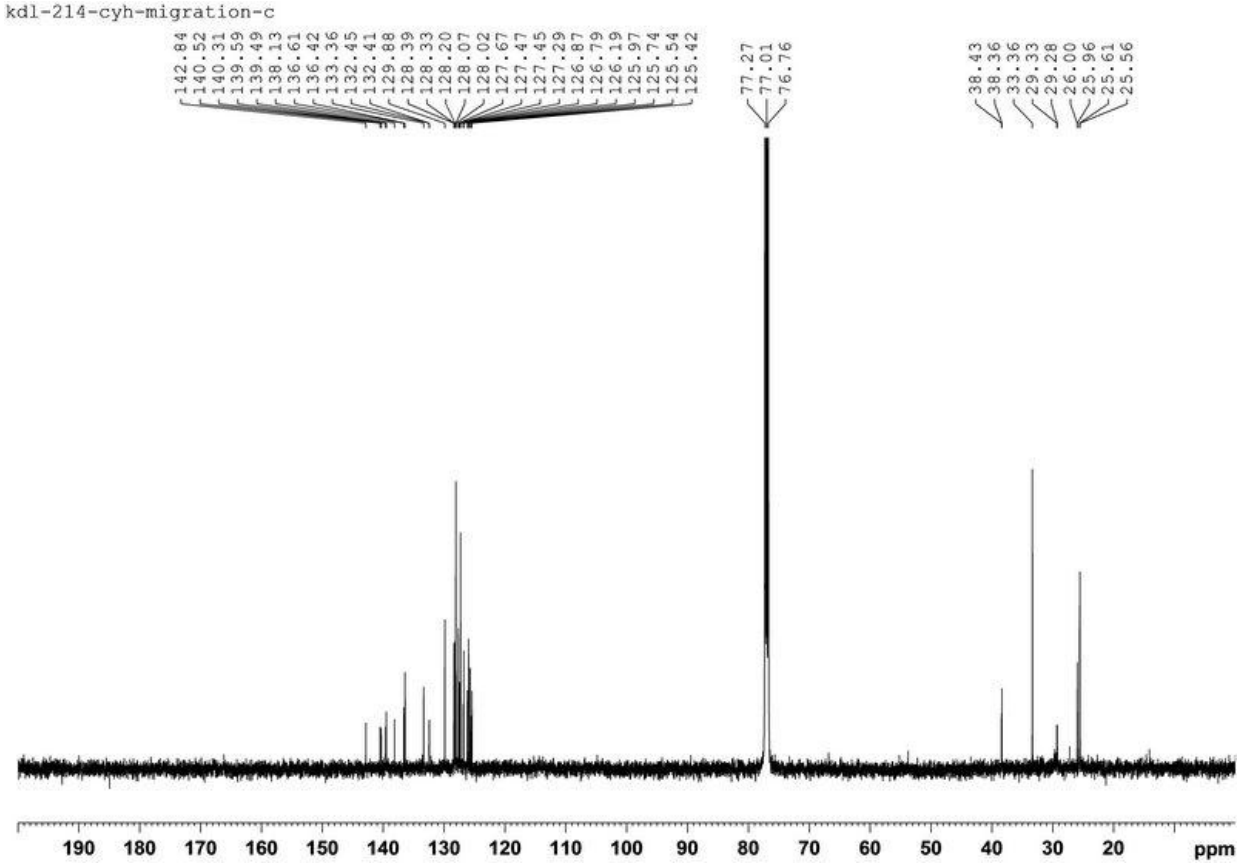


kdl-214-cyh-guanhuan-C



14a


kdl-214-cyh-migration-
7.835
7.825
7.816
7.781
7.771
7.763
7.730
7.712
7.703
7.692
7.668
7.514
7.500
7.493
7.489
7.481
7.461
7.444
7.411
7.397
7.382
7.357
7.343
7.291
7.274
7.259
7.235
7.217
7.203
6.052
6.032
6.022
5.982
2.246
2.230
2.215
2.198
2.191
2.177
2.149
1.734
1.708
1.677
1.548
1.311
1.256
1.240
1.215
1.192
1.179
1.156
1.139
1.121
1.095
-0.000
NAME kdl-214
EXPNO 34
PROCNO 1
Date_ 20150707
Time 21.20
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 31.72
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 10.60 usec
SI 65536
SF 500.1300127 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
H
Ph
9
8
7
6
5
4
3
2
1
0
ppm
2.19
1.42
2.03
2.08
0.65
3.89
1.00
1.51
4.93
7.22


kdl-214-cyh-migration-c
142.84
140.52
140.31
139.59
139.49
138.13
136.61
136.42
133.36
132.45
132.41
129.88
128.39
128.33
128.20
128.07
128.02
127.67
127.47
127.45
127.29
126.87
126.79
126.19
125.97
125.74
125.54
125.42
77.27
77.01
76.76
38.43
38.36
33.36
29.33
29.28
26.00
25.96
25.61
25.56
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm