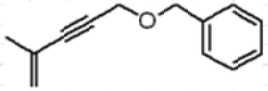
Coinage Metals-Catalyzed Cascade Reactions of Aryl Alkynylaziridines: Silver(I)-Single vs Gold(I)-Double Cyclizations

Nicolas Kern, Aurélien Blanc*, Solène Miaskiewicz, Michelle Robinette, Jean-Marc Weibel and Patrick Pale*

Laboratoire de Synthèse et Réactivité Organiques, Institut de Chimie, CNRS et Université de Strasbourg, 4 rue Blaise Pascal, 67070 Strasbourg France

Supporting Information

Ca



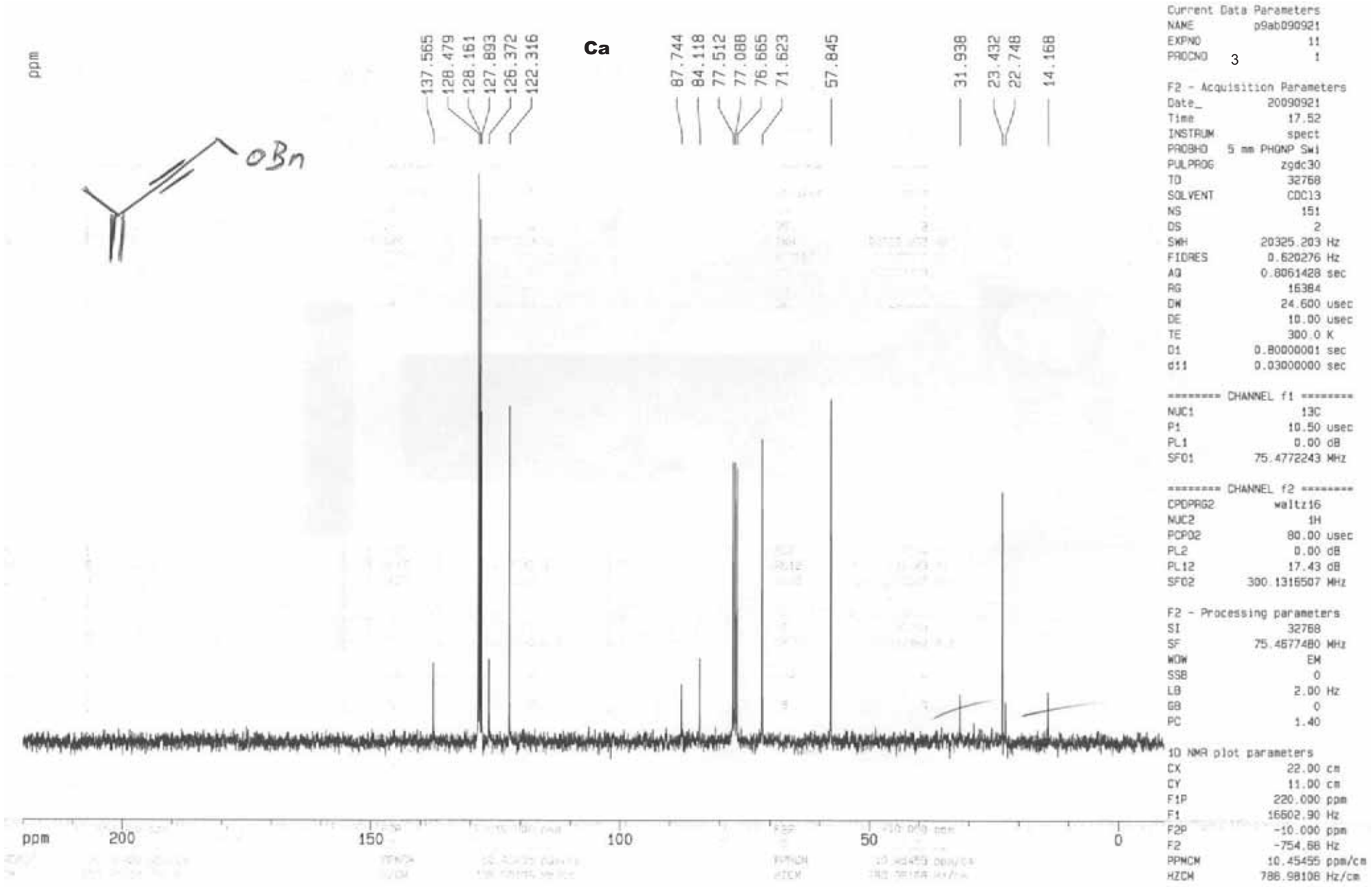
7,411
7,350
7,331
7,304
7,200
7,276
7,270
7,258

5,329
5,259
5,253
5,248
5,242
4,833
4,794
4,552
4,512
4,507
4,237
4,215
4,193

1,937
1,933
1,829
1,815
1,807
1,794
1,784
1,780
1,762
1,758
1,736
1,713
1,528
1,511
1,506
1,488
1,461
1,450
1,431
1,366
1,352
1,305
1,244
0,923
0,900
0,877

5,00
0,86
0,87
0,96
1,03
0,91
2,63
2,00
2,04
6,18
3,17

ppm



Current Data Parameters

NAME	p9ab090921
EXPNO	11
PROCNO	1

F2 - Acquisition Parameters

Date_	20090921
Time	17.52
INSTRUM	spect
PROBHD	5 mm PHONP Swi
PULPROG	zgdc30
TD	32768
SOLVENT	CDCl3
NS	151
DS	2
SWH	20325.203 Hz
FIDRES	0.620276 Hz
AQ	0.8061428 sec
RG	16384
DW	24.600 usec
DE	10.00 usec
TE	300.0 K
D1	0.80000001 sec
d11	0.03000000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	10.50 usec
PL1	0.00 dB
SFO1	75.4772243 MHz

======== CHANNEL f2 ========

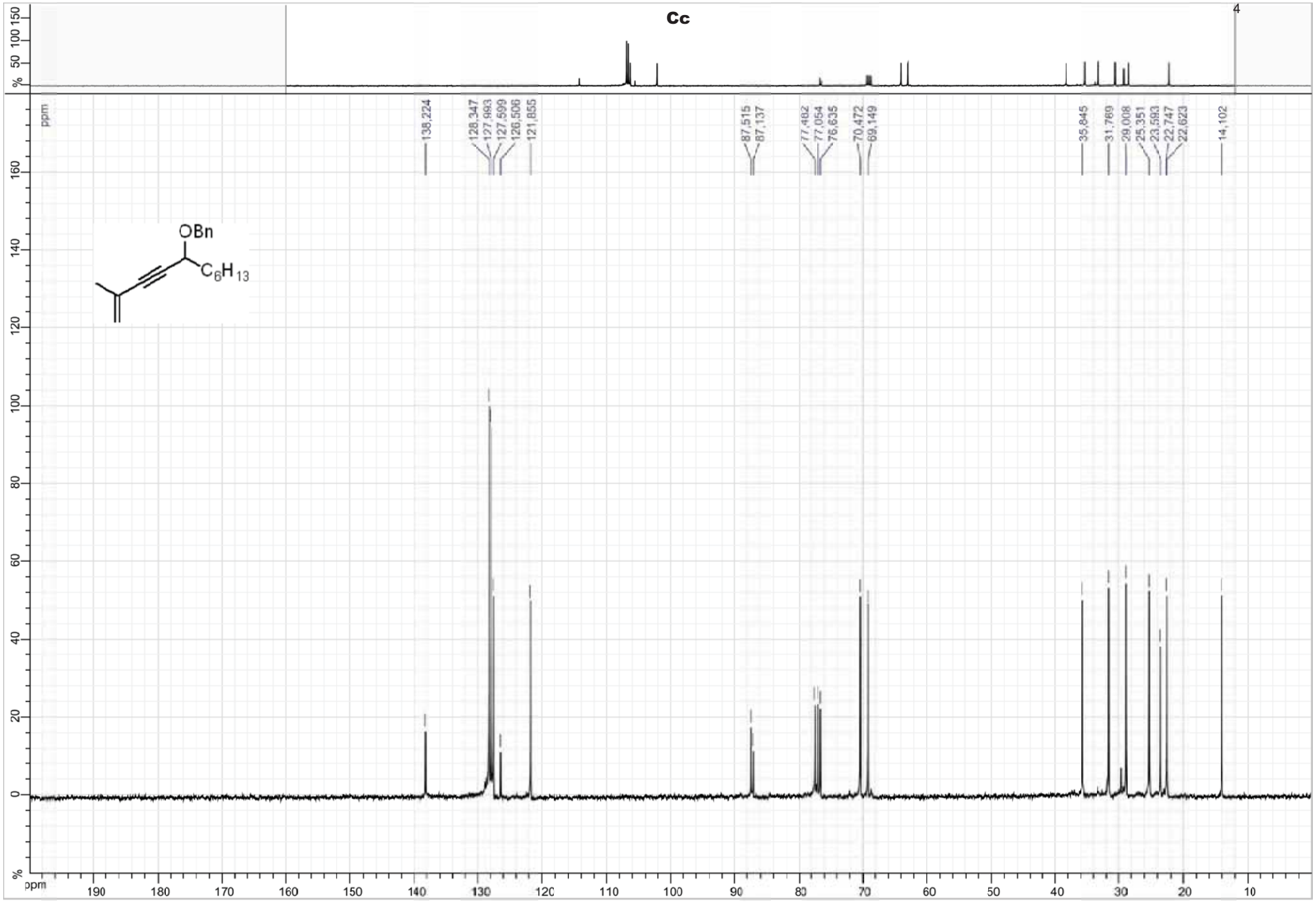
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	17.43 dB
SFO2	300.1316507 MHz

F2 - Processing parameters

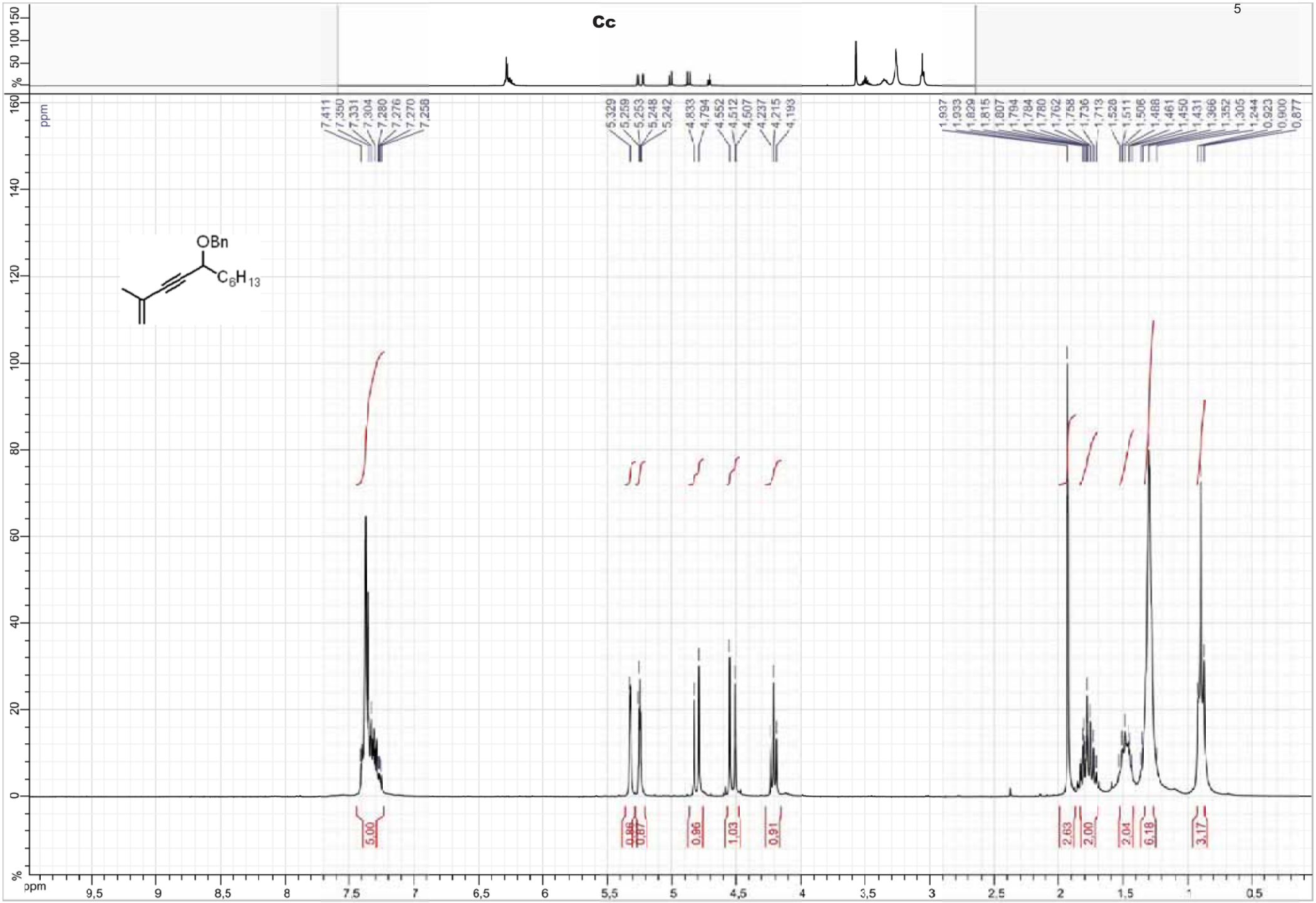
SI	32768
SF	75.4677480 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.40

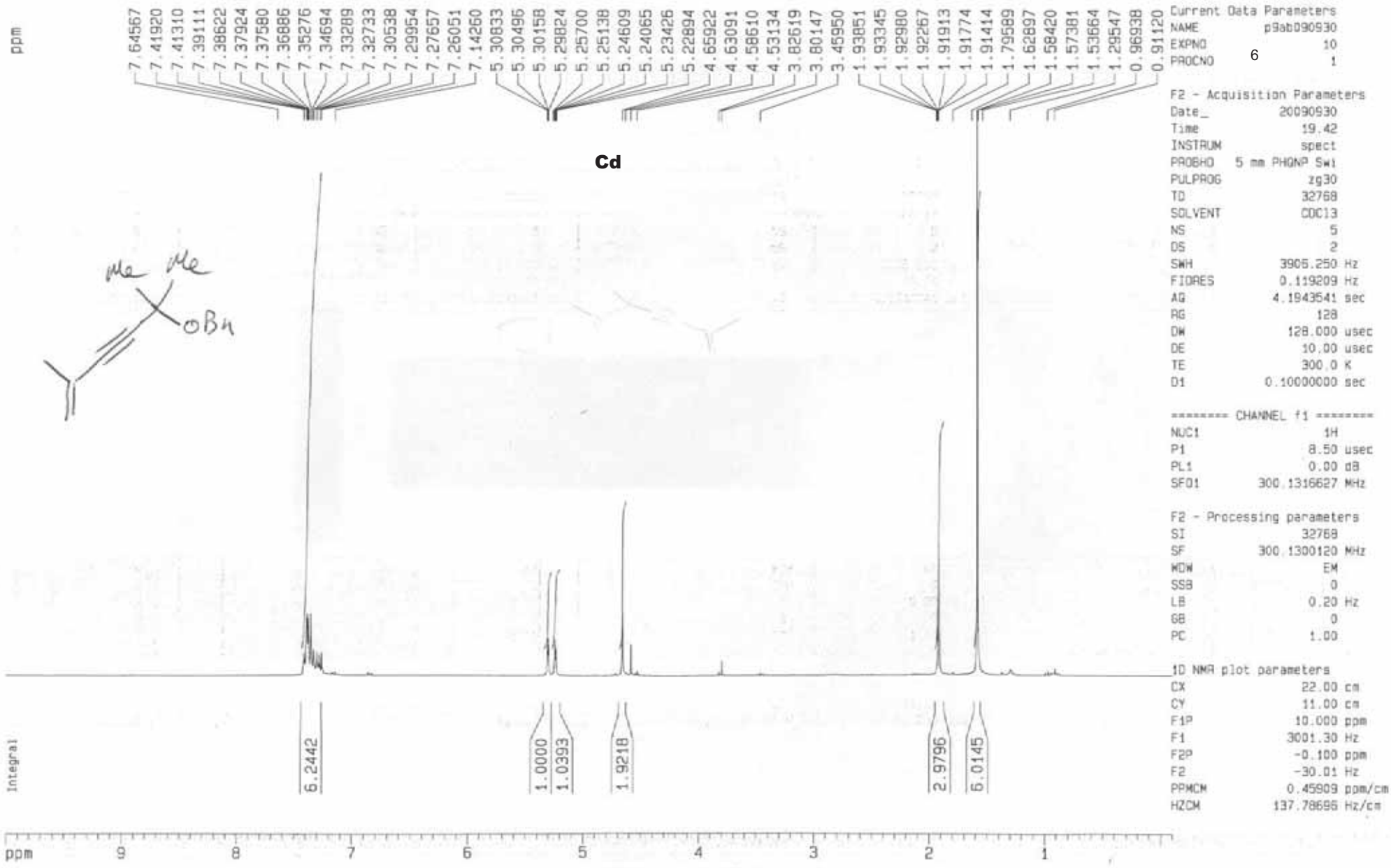
1D NMR plot parameters

CX	22.00 cm
CY	11.00 cm
F1P	220.000 ppm
F1	16602.90 Hz
F2P	-10.000 ppm
F2	-754.66 Hz
PPMCM	10.45455 ppm/cm
HZCM	786.98108 Hz/cm


Cc
4
ppm
138,224
128,347
127,993
127,599
126,506
121,855
87,515
87,137
77,482
77,054
76,635
70,472
69,149
35,845
31,769
29,008
25,351
23,593
22,747
22,623
14,102
OBn
C6H13

Cc


OBn
C_6H_{13}
ppm
7,411
7,350
7,331
7,304
7,280
7,276
7,270
7,258
5,329
5,259
5,253
5,248
5,242
4,833
4,794
4,552
4,512
4,507
4,237
4,215
4,193
1,937
1,933
1,829
1,815
1,807
1,794
1,784
1,780
1,762
1,758
1,736
1,713
1,528
1,511
1,506
1,488
1,461
1,450
1,431
1,366
1,352
1,305
1,244
0,923
0,900
0,877
5,00
0,86
0,87
0,96
1,03
0,91
2,63
2,00
2,04
6,18
3,17
ppm
9,5
9
8,5
8
7,5
7
6,5
6
5,5
5
4,5
4
3,5
3
2,5
2
1,5
1
0,5



Current Data Parameters
NAME p9ab090930
EXPNO 10
PROCNO 1

6

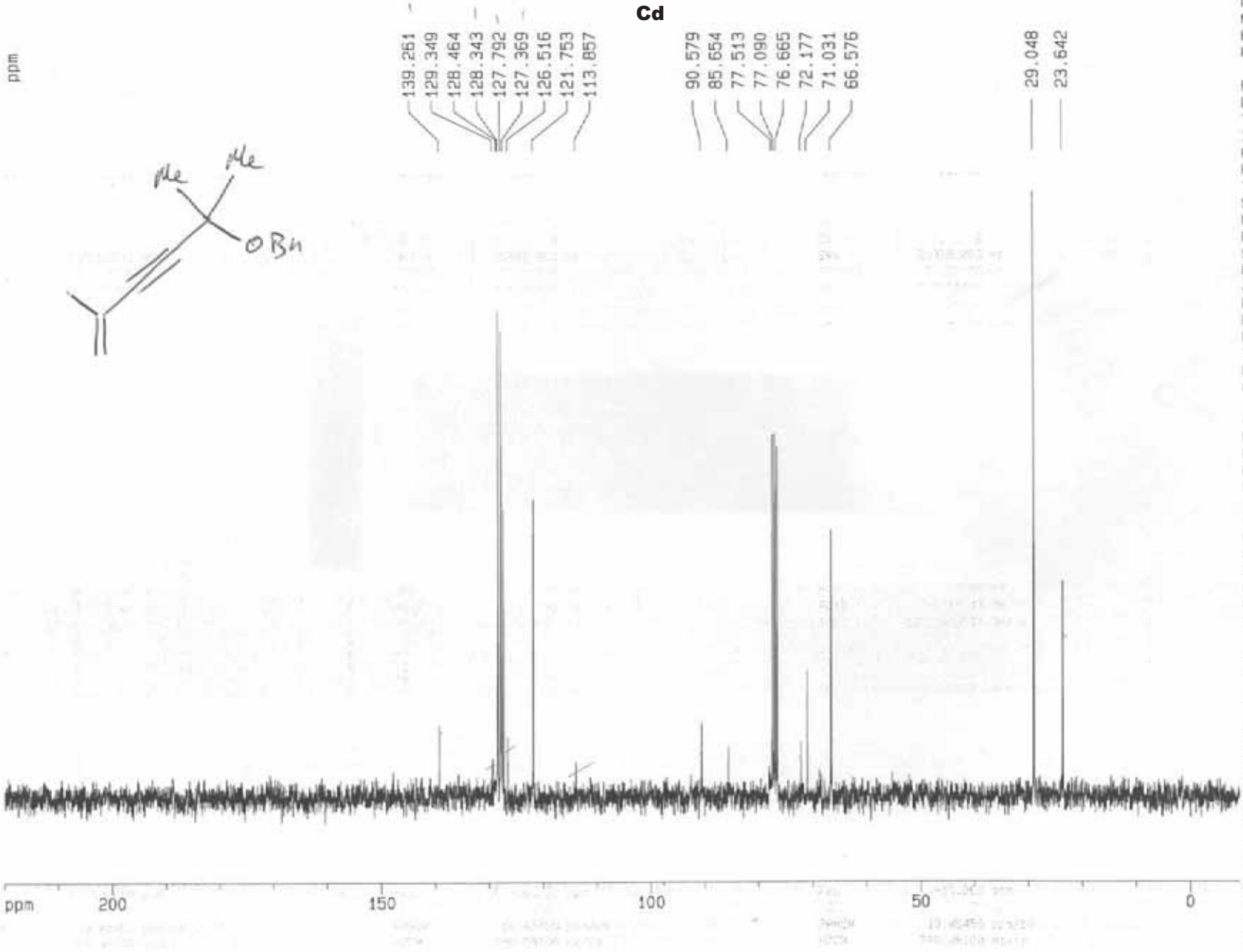
F2 - Acquisition Parameters
Date_ 20090930
Time 19.42
INSTRUM spect
PROBHD 5 mm PHONP Swi
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 5
DS 2
SWH 3905.250 Hz
FIDRES 0.119209 Hz
AQ 4.1943541 sec
RG 128
DW 128.000 usec
DE 10.00 usec
TE 300.0 K
D1 0.10000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 8.50 usec
PL1 0.00 dB
SFO1 300.1316627 MHz

F2 - Processing parameters
SI 32768
SF 300.1300120 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
CY 11.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.100 ppm
F2 -30.01 Hz
PPMCM 0.45909 ppm/cm
HZCM 137.78696 Hz/cm

Cd



Current Data Parameters

NAME	p9ab090930
EXPNO	11
PROCNO	1

F2 - Acquisition Parameters

Date_	20090930
Time	19.44
INSTRUM	spect
PROBHD	5 mm PHONP Swi
PULPROG	zgdc30
TD	32768
SOLVENT	CDCl3
NS	75
DS	2
SWH	20325.203 Hz
FIDRES	0.620276 Hz
AQ	0.8051428 sec
RG	16384
DW	24.600 usec
DE	10.00 usec
TE	300.0 K
D1	0.80000001 sec
d11	0.03000000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	10.50 usec
PL1	0.00 dB
SFO1	75.4772243 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	17.43 dB
SFO2	300.1316507 MHz

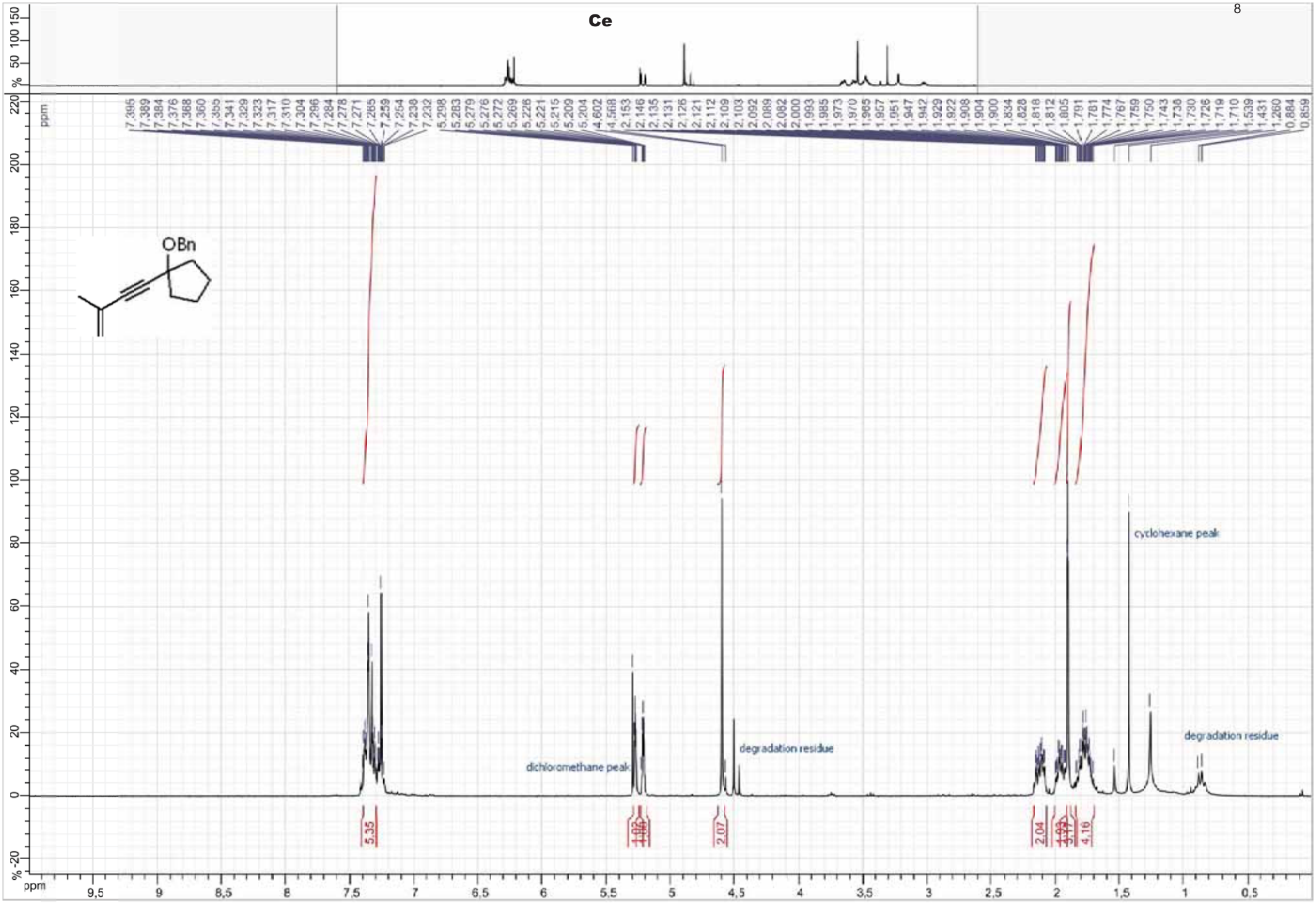
F2 - Processing parameters

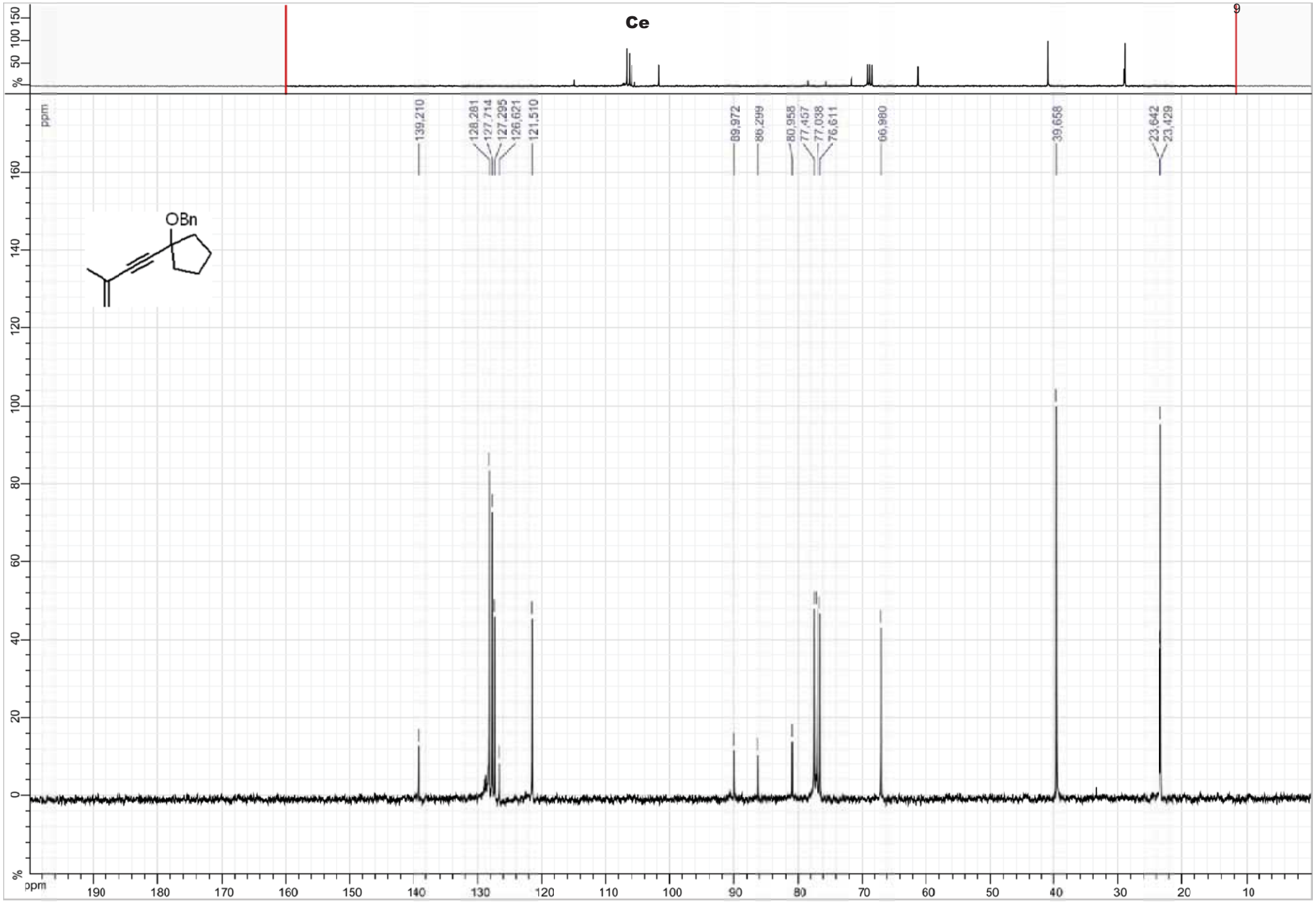
SI	32768
SF	75.4677480 MHz
WDW	EM
SSB	0
LB	2.00 Hz
GB	0
PC	1.40

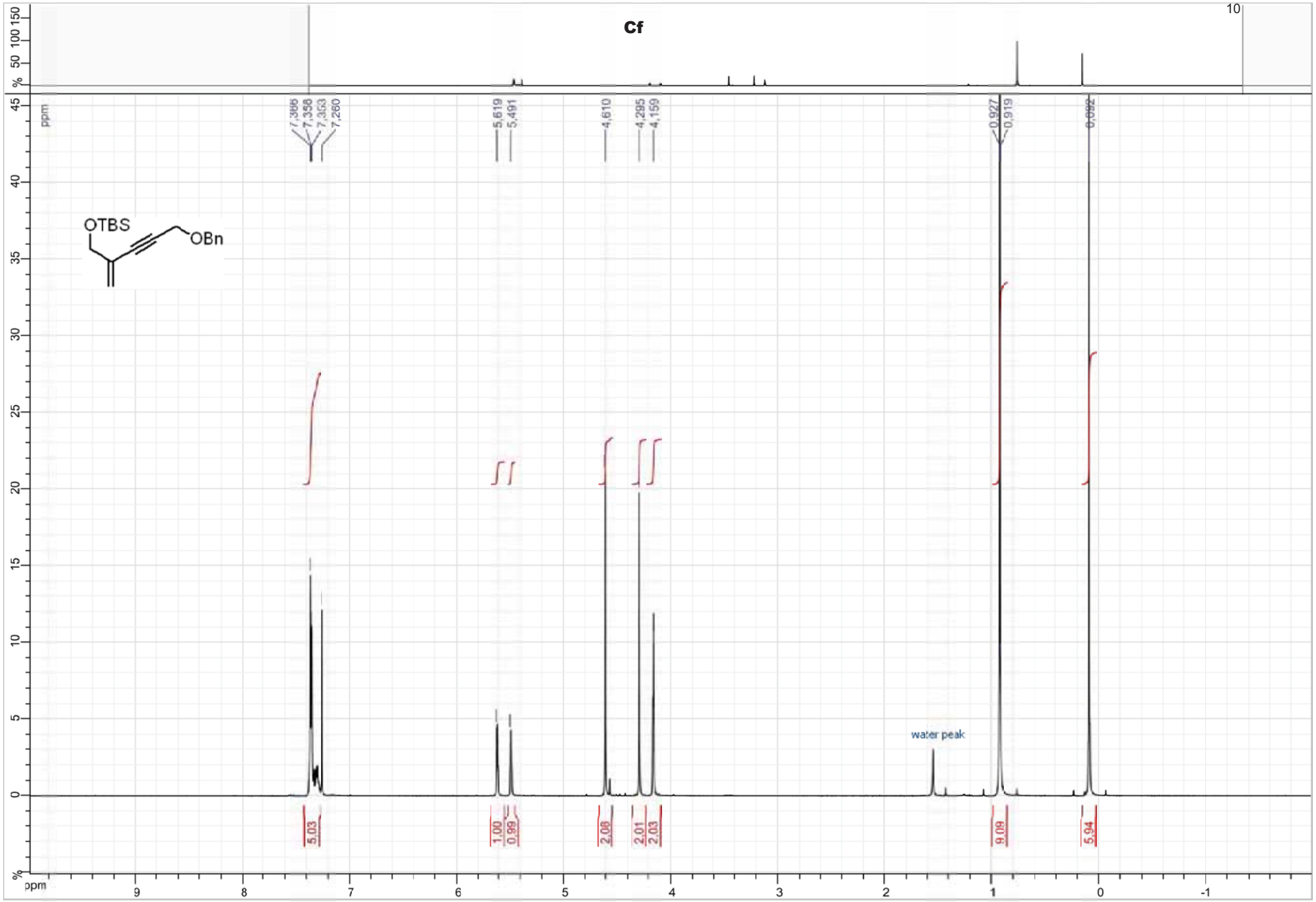
1D NMR plot parameters

CX	22.00 cm
CY	11.00 cm
F1P	220.000 ppm
F1	16602.90 Hz
F2P	-10.000 ppm
F2	-754.68 Hz
PPMCM	10.45455 ppm/cm
HZCM	786.98108 Hz/cm

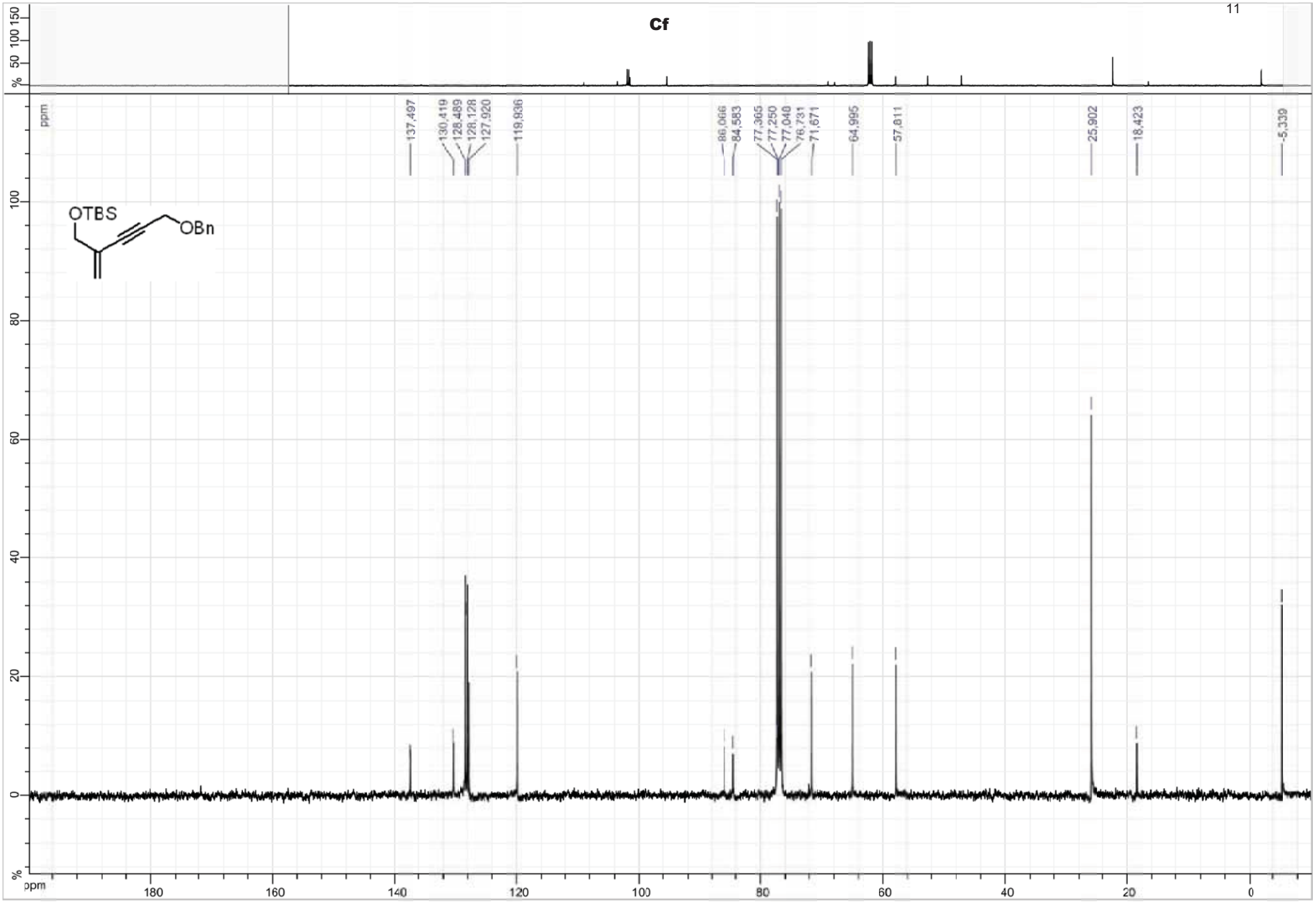
Ce


OBn
dichloromethane peak
degradation residue
cyclohexane peak
degradation residue
5,35
2,07
2,04
4,16
ppm


Ce
9
ppm
139,210
128,281
127,714
127,295
126,621
121,510
89,972
86,299
80,958
77,457
77,038
76,611
66,980
39,658
23,642
23,429
OBn

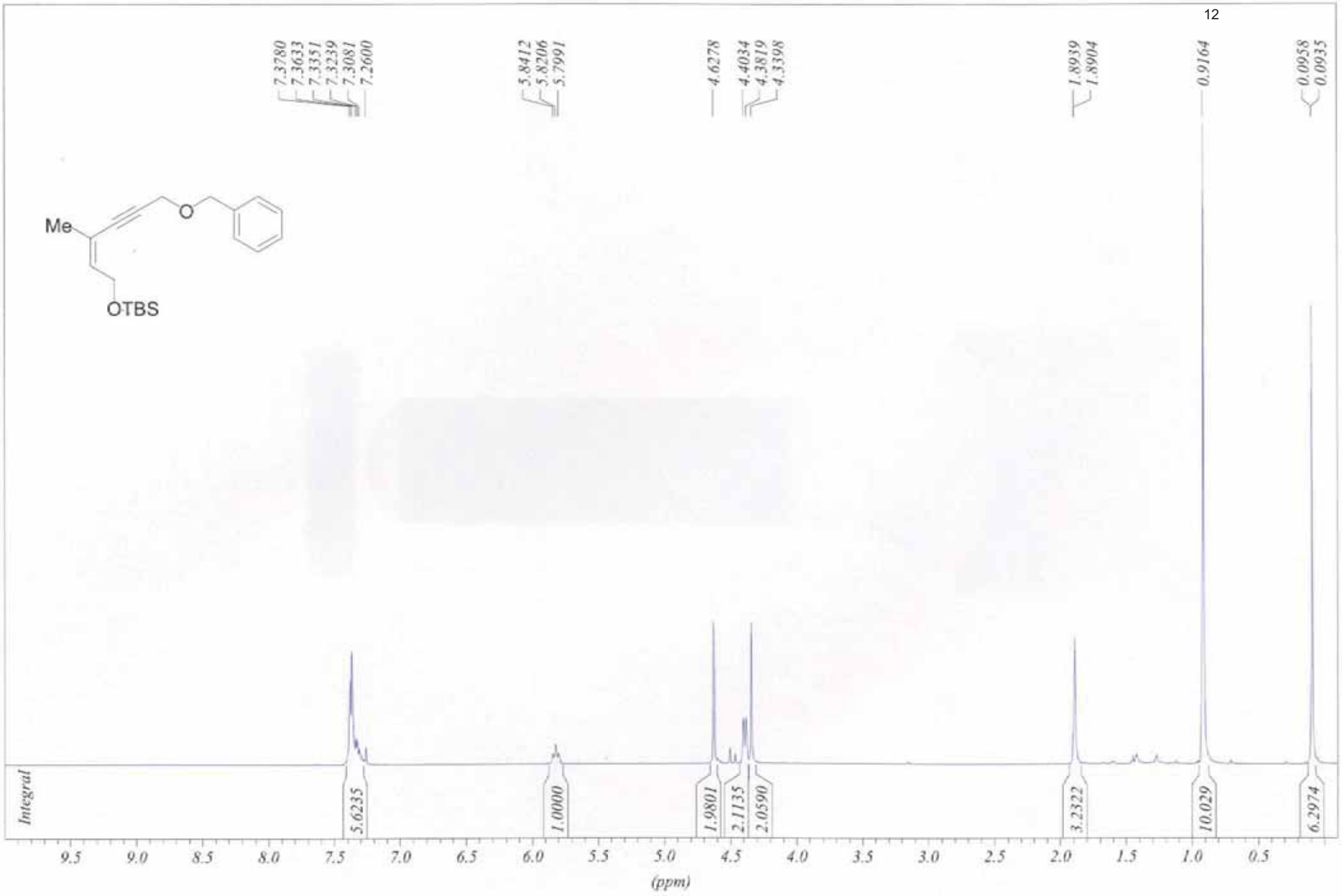

Cf
10
ppm
7,366
7,358
7,353
7,260
5,619
5,491
4,610
4,295
4,159
0,927
0,919
0,092
OTBS
OBn
water peak
5,03
1,00
0,99
2,08
2,01
2,03
9,09
5,94

Cf

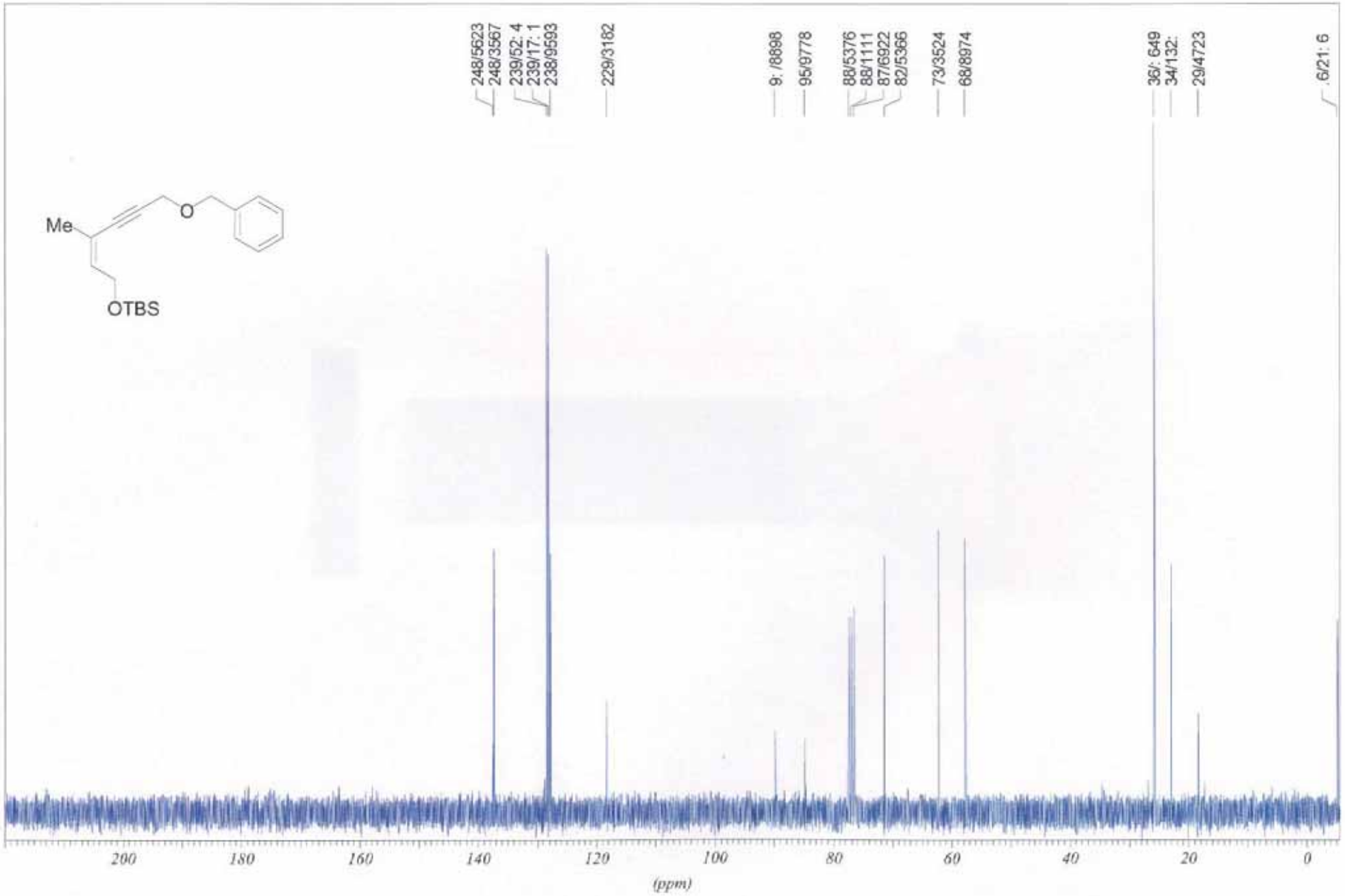

137,497
130,419
128,489
128,128
127,920
119,936
88,066
84,583
77,365
77,250
77,040
76,731
71,671
64,995
57,611
25,902
18,423
-5,339
OTBS
OBn
ppm
180
160
140
120
100
80
60
40
20
0

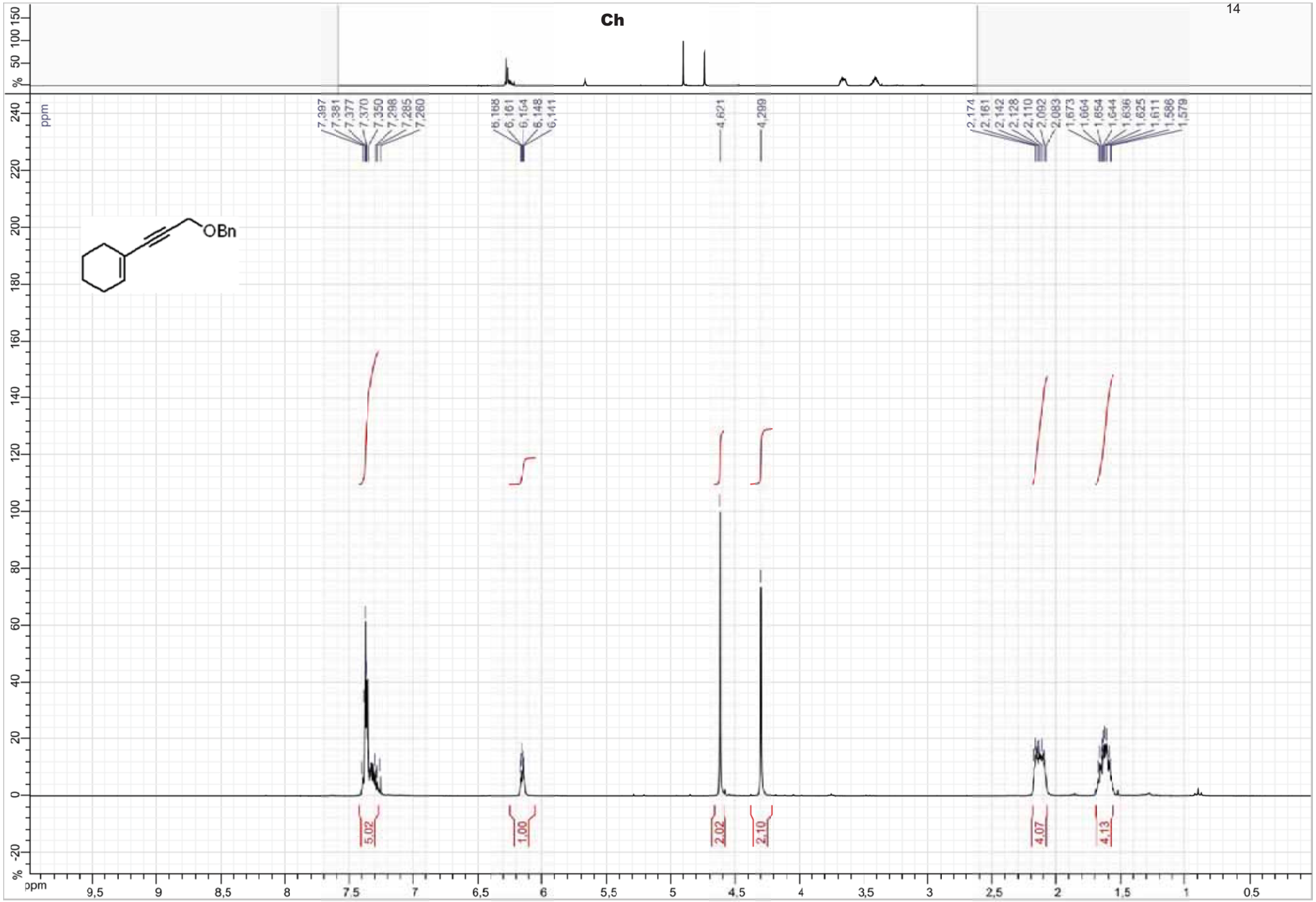
Proton CDCL3 300 MHz

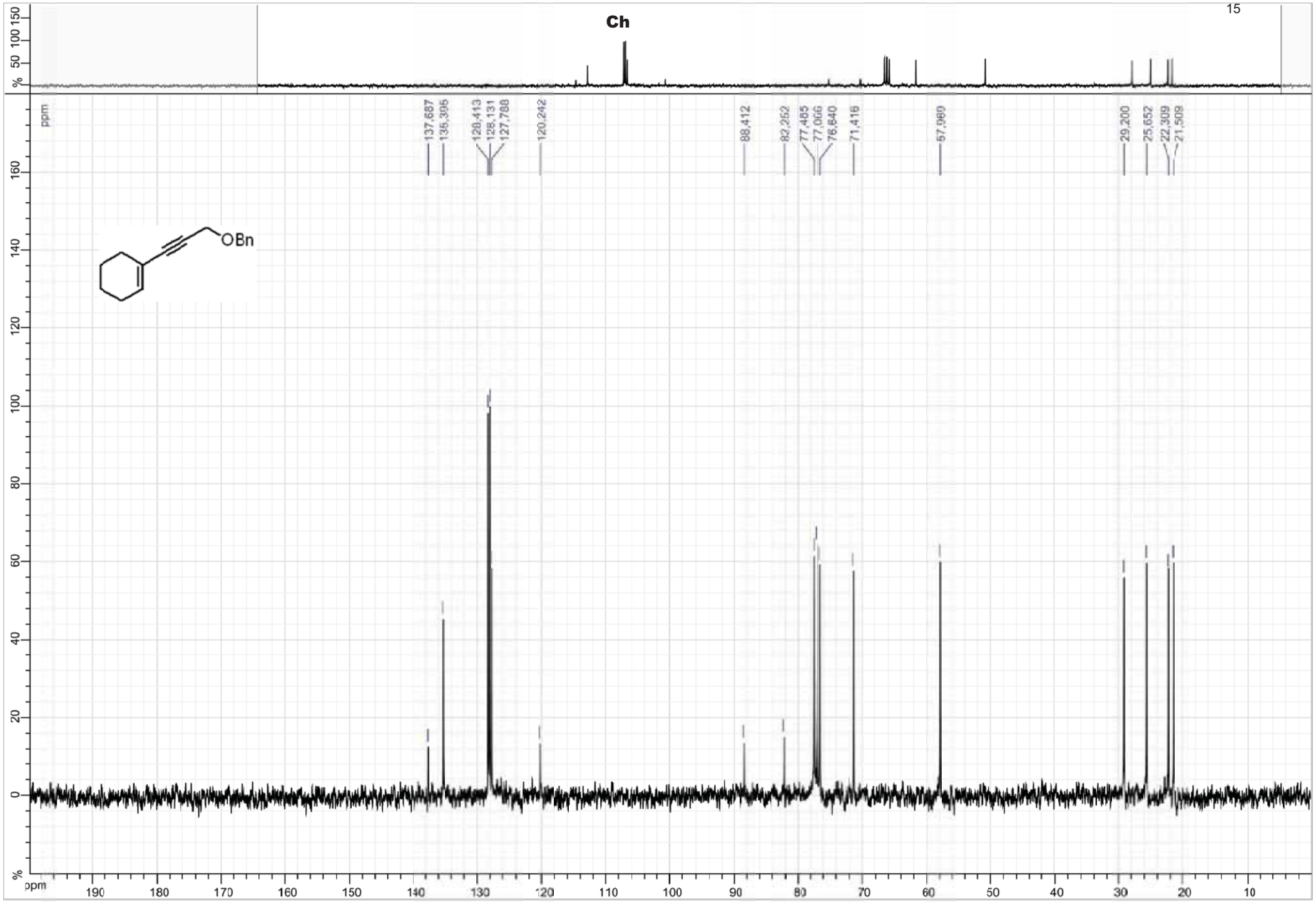
Cg



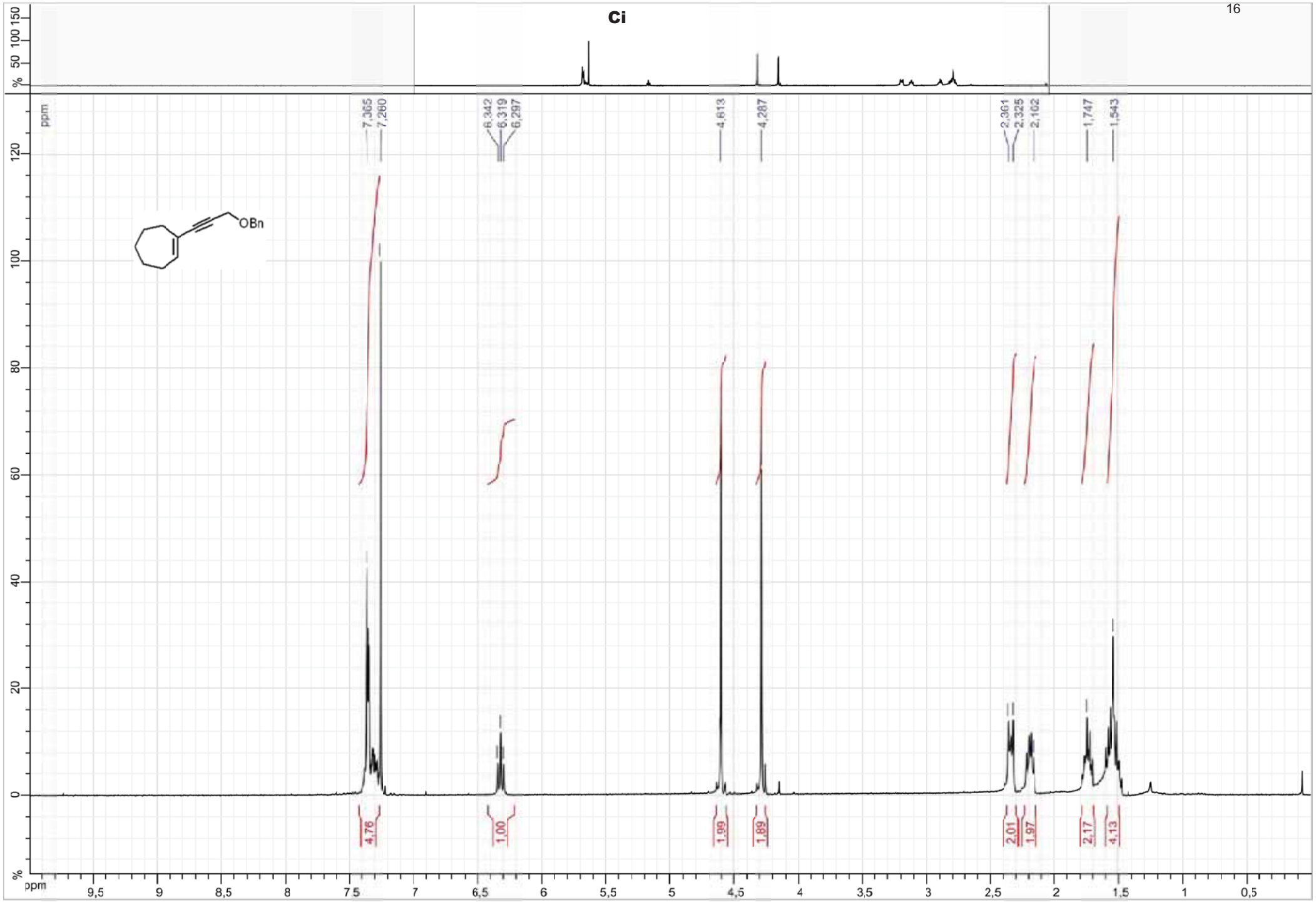
Cg


248/5623
248/3567
239/52: 4
239/17: 1
238/9593
229/3182
9: /8898
95/9778
88/5376
88/1111
87/6922
82/5366
73/3524
68/8974
35/: 649
34/132:
29/4723
.6/21: 6
Me
O
OTBS
200
180
160
140
120
100
80
60
40
20
0
(ppm)

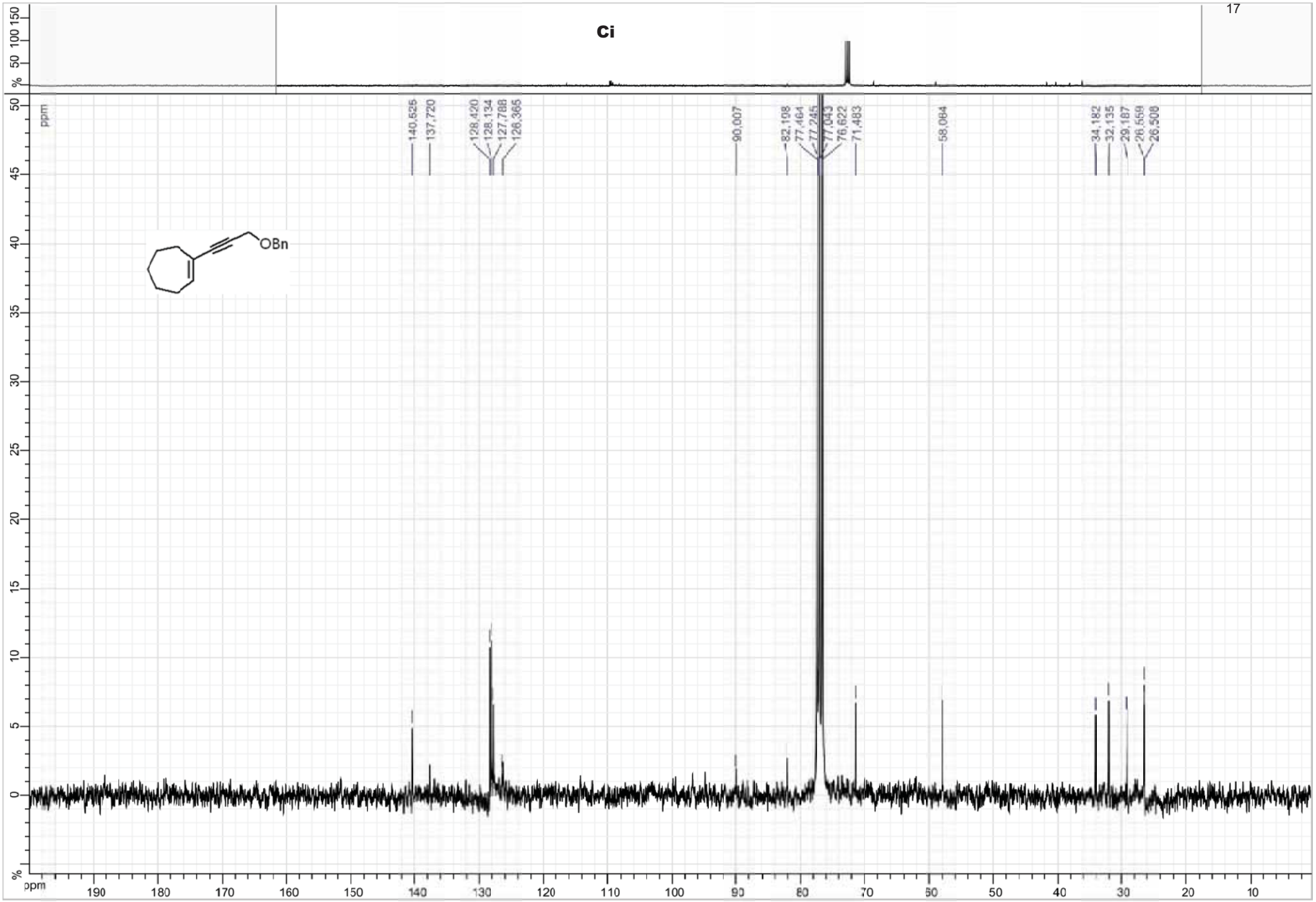

Ch
ppm
7,397
7,381
7,377
7,370
7,350
7,298
7,285
7,260
6,168
6,161
6,154
6,148
6,141
4,621
4,299
2,174
2,161
2,142
2,128
2,110
2,092
2,083
1,673
1,664
1,654
1,644
1,636
1,625
1,611
1,586
1,579
OBn
5,02
1,00
2,02
2,10
4,07
4,13


Ch
ppm
137,687
135,395
128,413
128,131
127,788
120,242
88,412
82,252
77,465
77,066
76,840
71,416
57,969
29,200
25,652
22,309
21,509
OBn

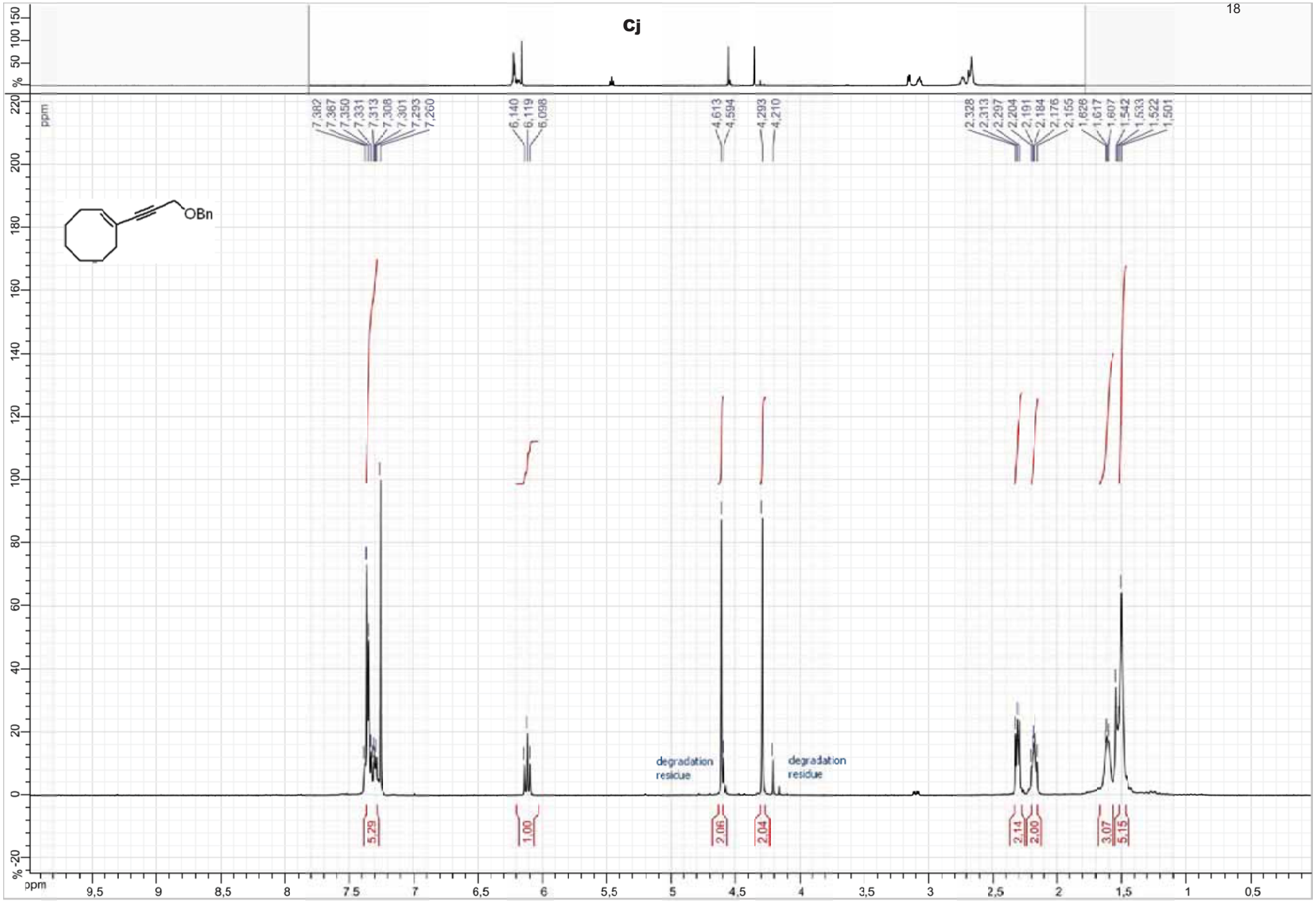
Ci

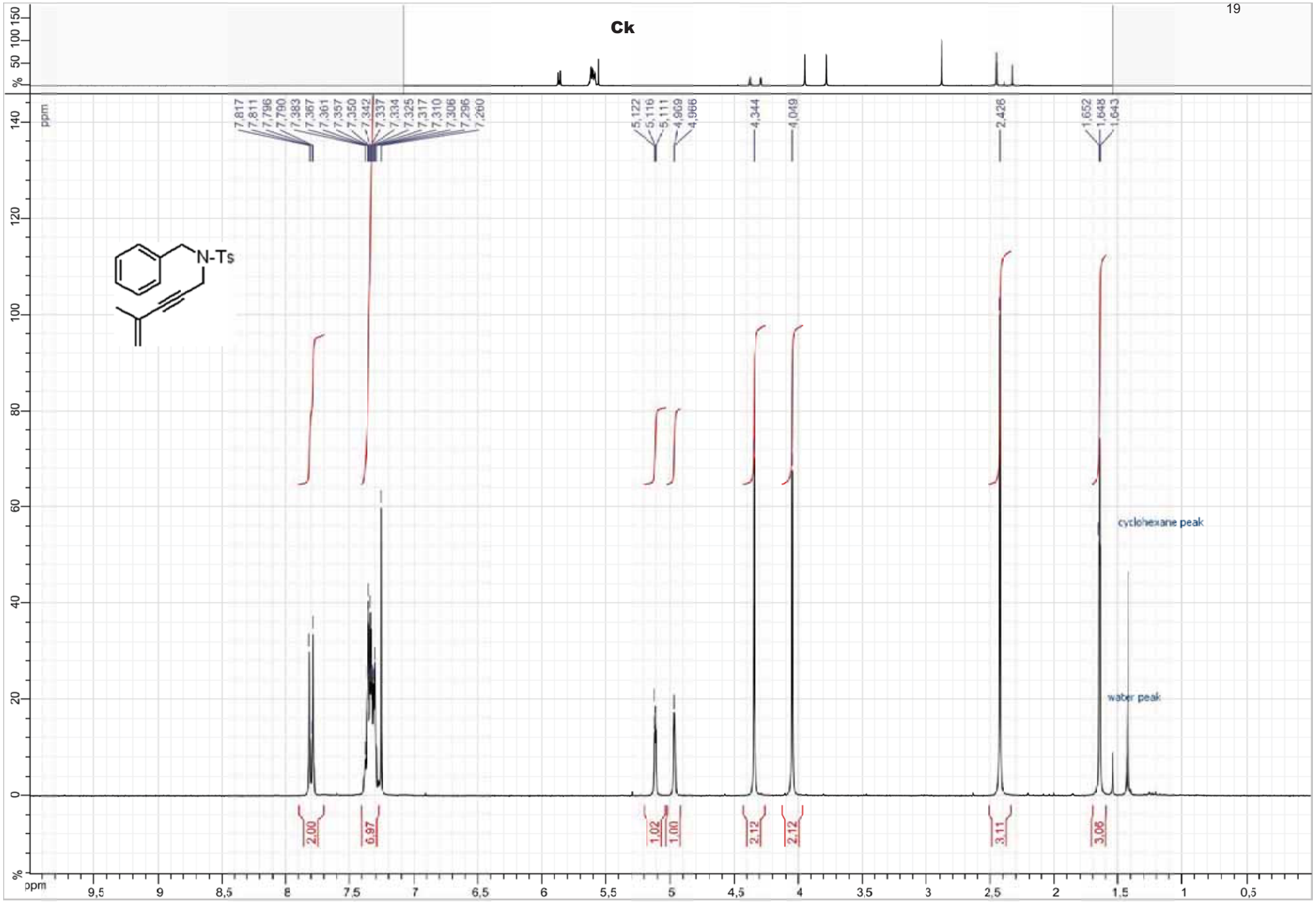

ppm
7,365
7,260
6,342
6,319
6,297
4,613
4,287
2,361
2,325
2,162
1,747
1,543
OBn
4,76
1,00
1,99
1,89
2,01
1,97
2,17
4,13

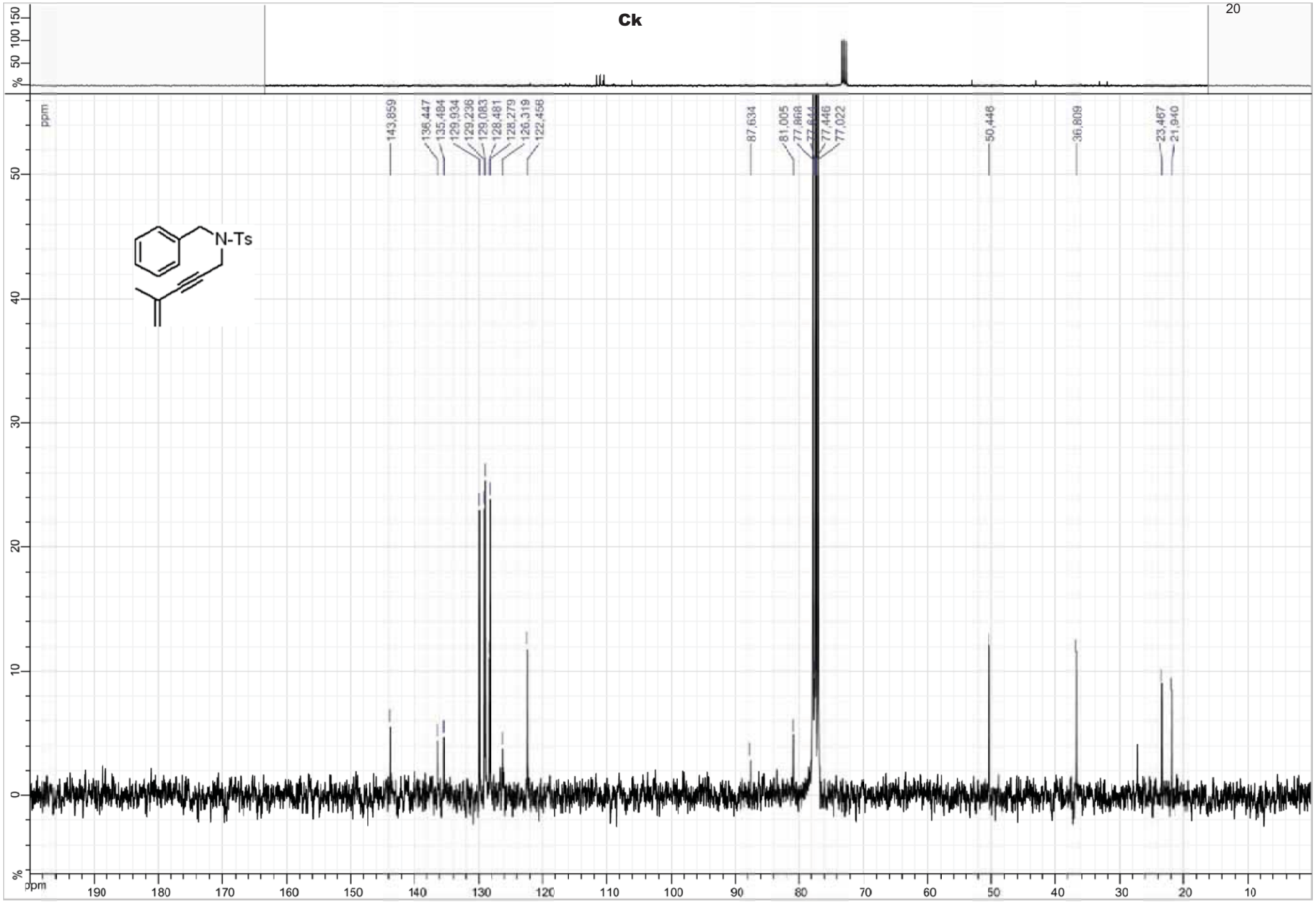
Ci

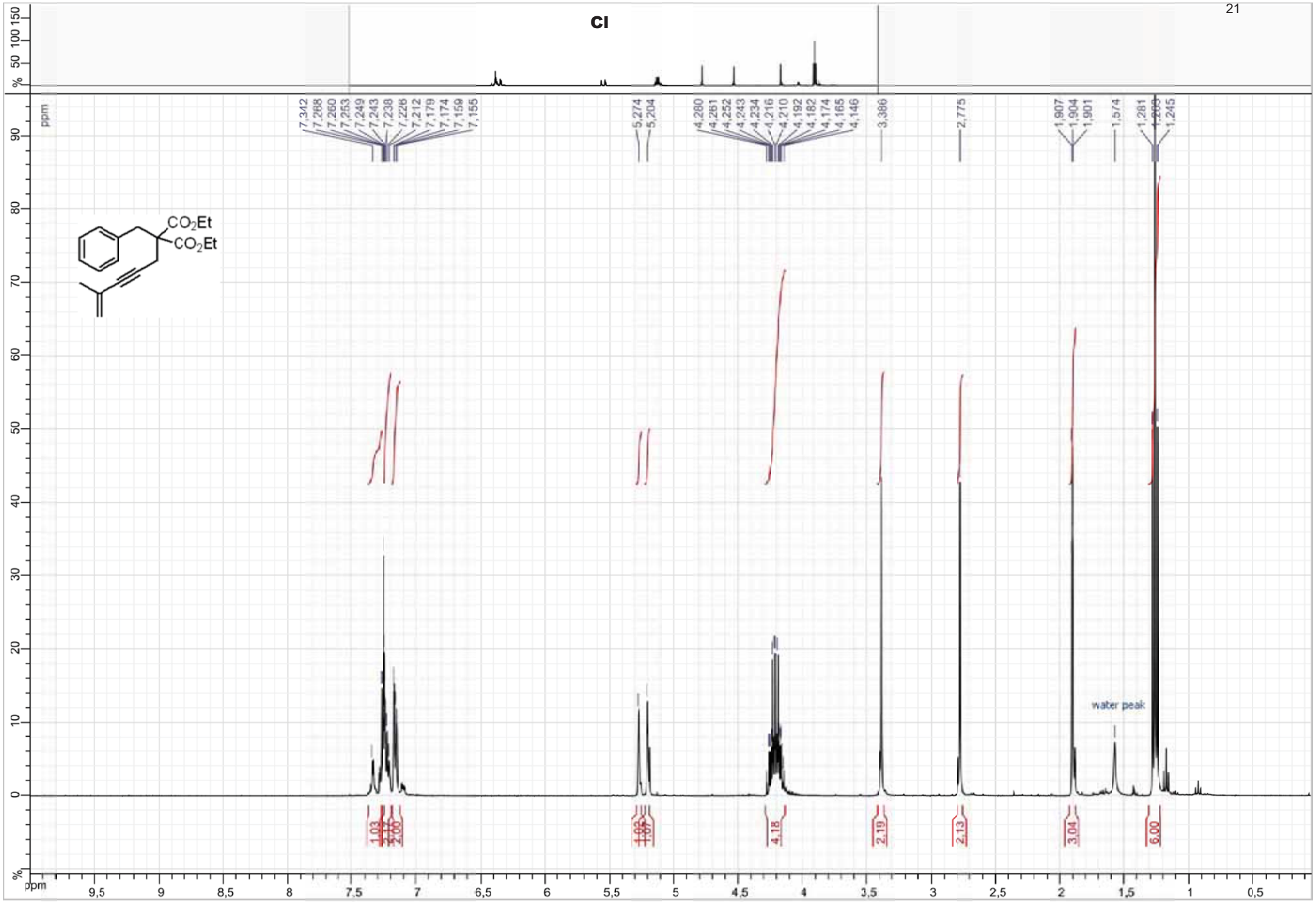

ppm
140,525
137,720
128,420
128,134
127,788
126,365
90,007
82,198
77,464
77,245
77,043
76,622
71,483
58,084
34,182
32,135
29,187
26,559
26,508
OBn

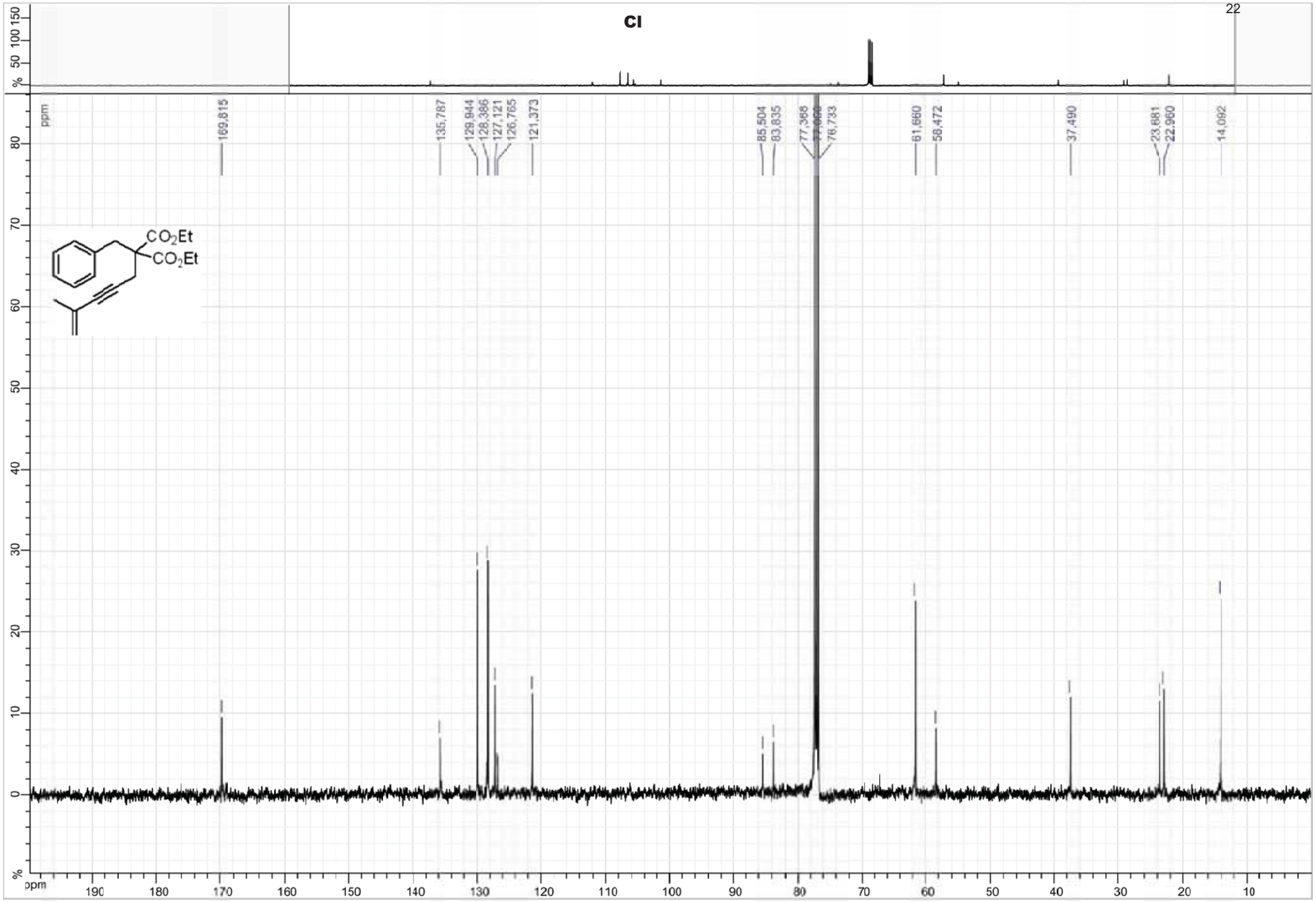
Cj


ppm
7,382
7,367
7,350
7,331
7,313
7,308
7,301
7,293
7,260
6,140
6,119
6,098
4,613
4,594
4,293
4,210
2,328
2,313
2,297
2,204
2,191
2,184
2,176
2,155
1,628
1,617
1,607
1,542
1,533
1,522
1,501
OBn
degradation
residue
degradation
residue
5,29
1,00
2,06
2,04
2,14
2,00
3,07
5,15
ppm

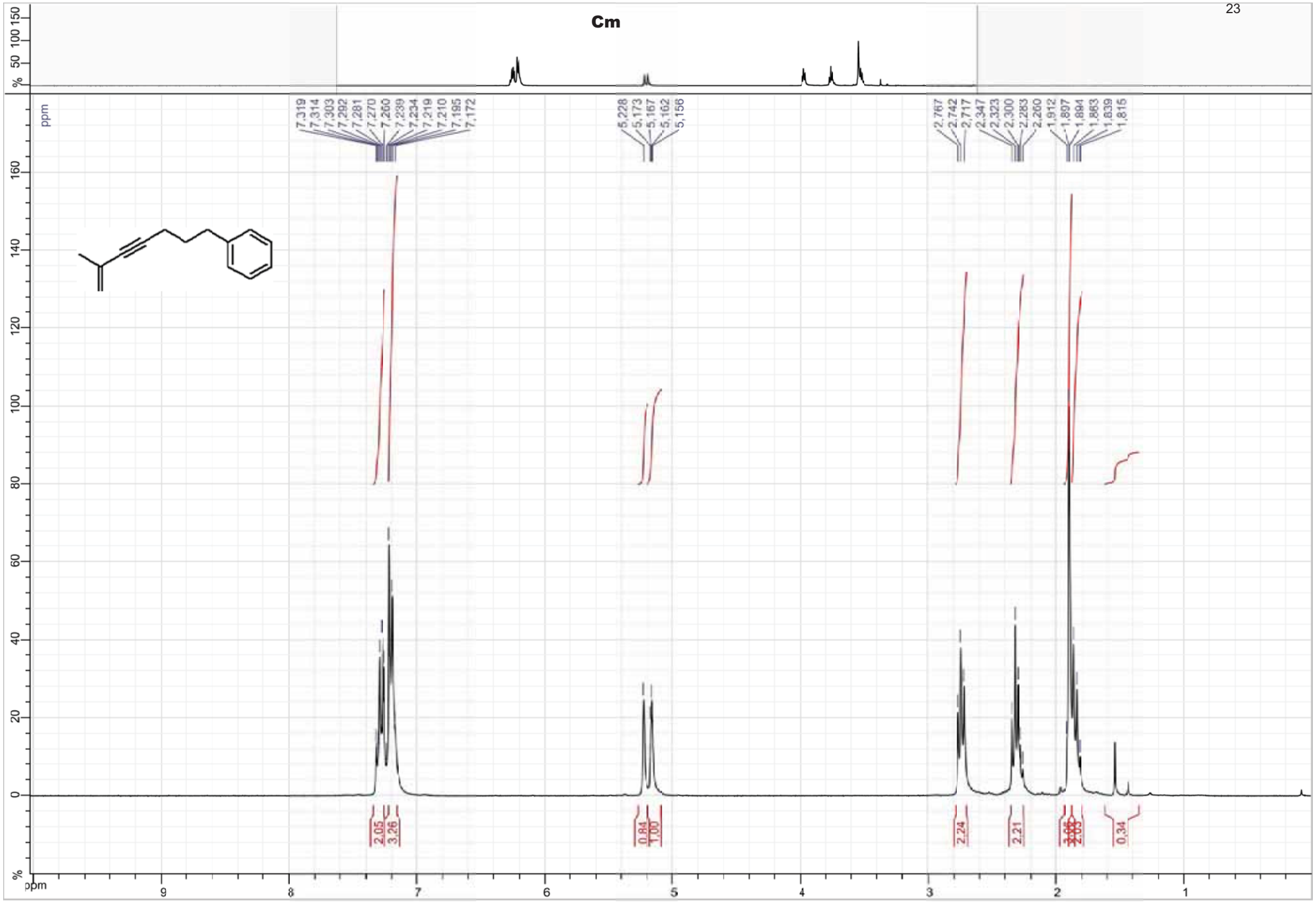

Ck
ppm
7,817
7,811
7,796
7,790
7,383
7,367
7,361
7,357
7,350
7,342
7,337
7,334
7,325
7,317
7,310
7,306
7,296
7,260
5,122
5,116
5,111
4,969
4,966
4,344
4,049
2,428
1,652
1,648
1,643
N-Ts
cyclohexane peak
water peak
2,00
6,97
1,02
1,00
2,12
2,12
3,11
3,06

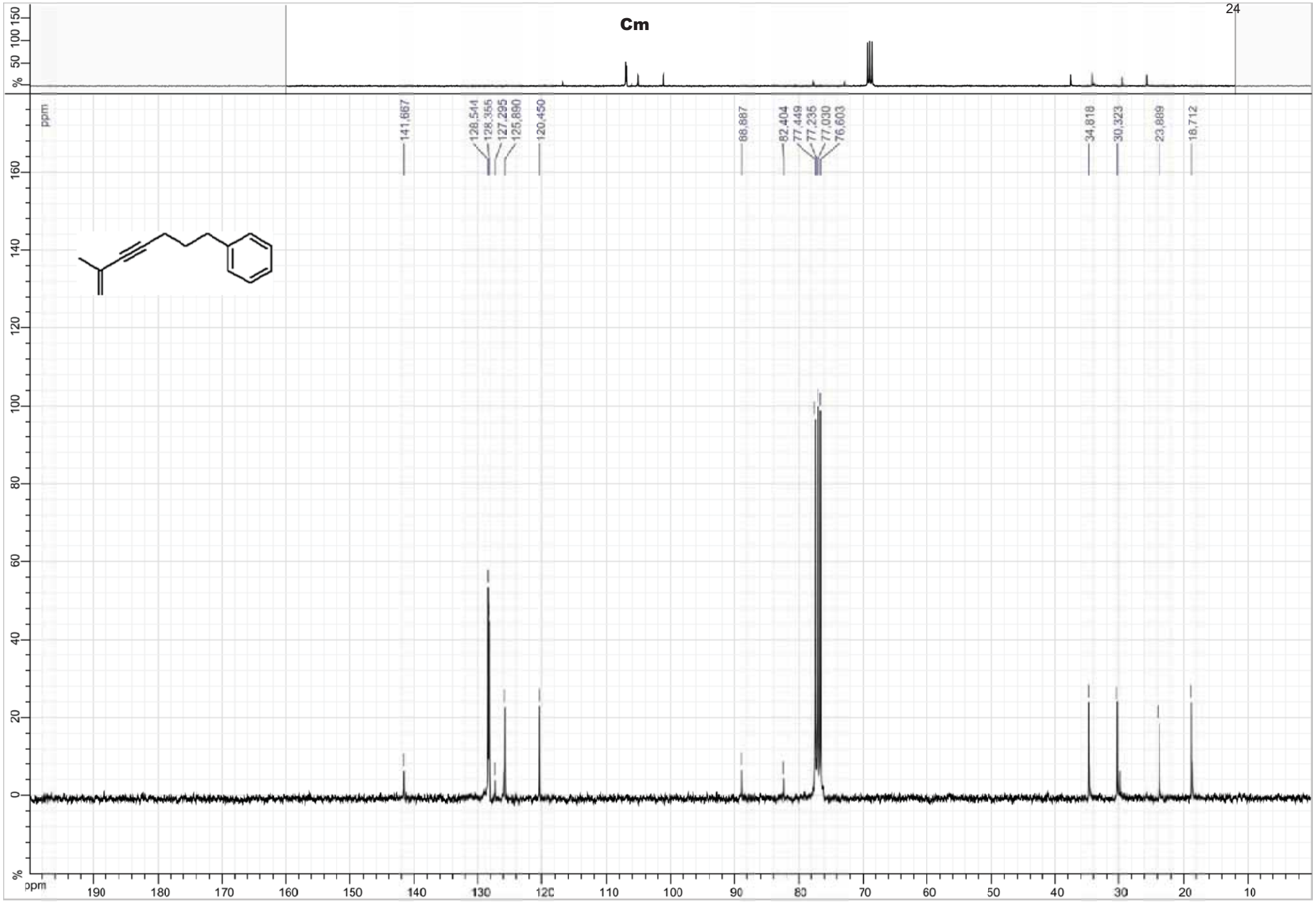

Ck
ppm
143,859
136,447
135,484
129,934
129,236
129,083
128,481
128,279
126,319
122,458
87,634
81,005
77,868
77,644
77,446
77,022
50,448
36,809
23,467
21,940
N-Ts

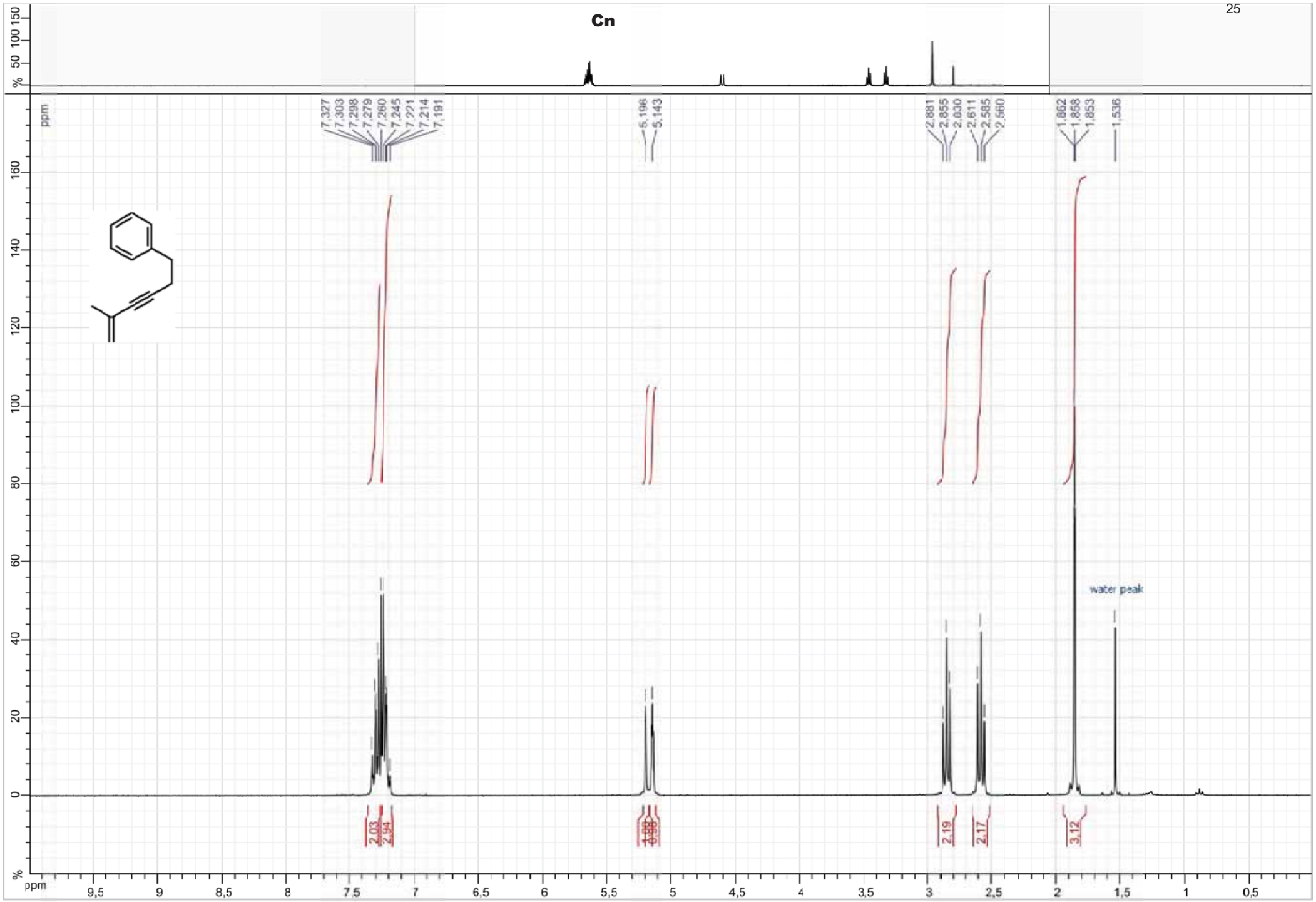

CI
ppm
7,342
7,268
7,260
7,253
7,249
7,243
7,238
7,226
7,212
7,179
7,174
7,159
7,155
5,274
5,204
4,280
4,261
4,252
4,243
4,234
4,216
4,210
4,192
4,182
4,174
4,165
4,146
3,386
2,775
1,907
1,904
1,901
1,574
1,281
1,245
CO2Et
CO2Et
water peak
1,03
2,00
4,18
2,19
2,13
3,04
6,00
ppm

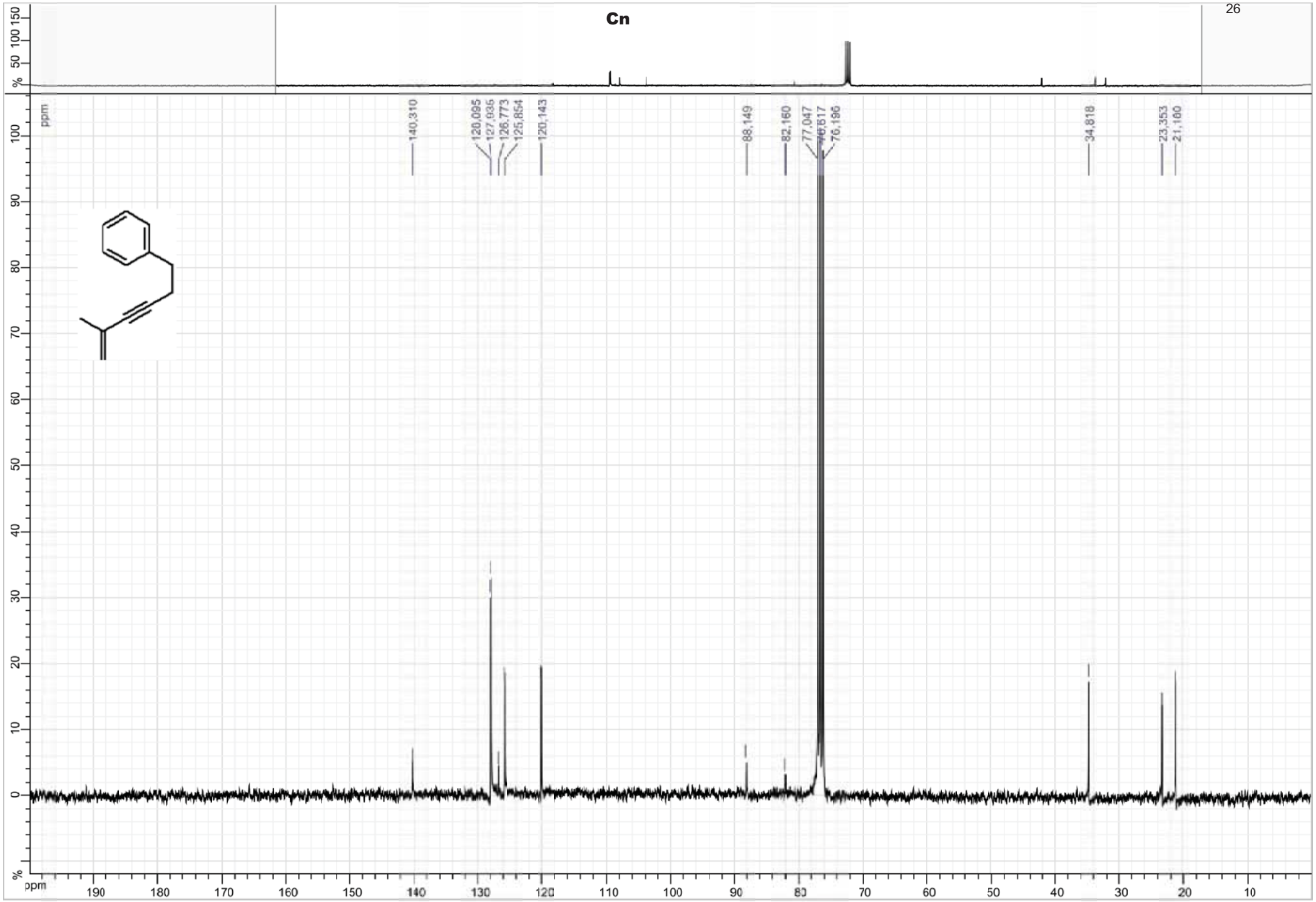

Cl
22
ppm
169,815
135,787
129,944
128,386
127,121
126,765
121,373
85,504
83,835
77,368
76,733
61,660
58,472
37,490
23,681
22,960
14,092
CO2Et
CO2Et

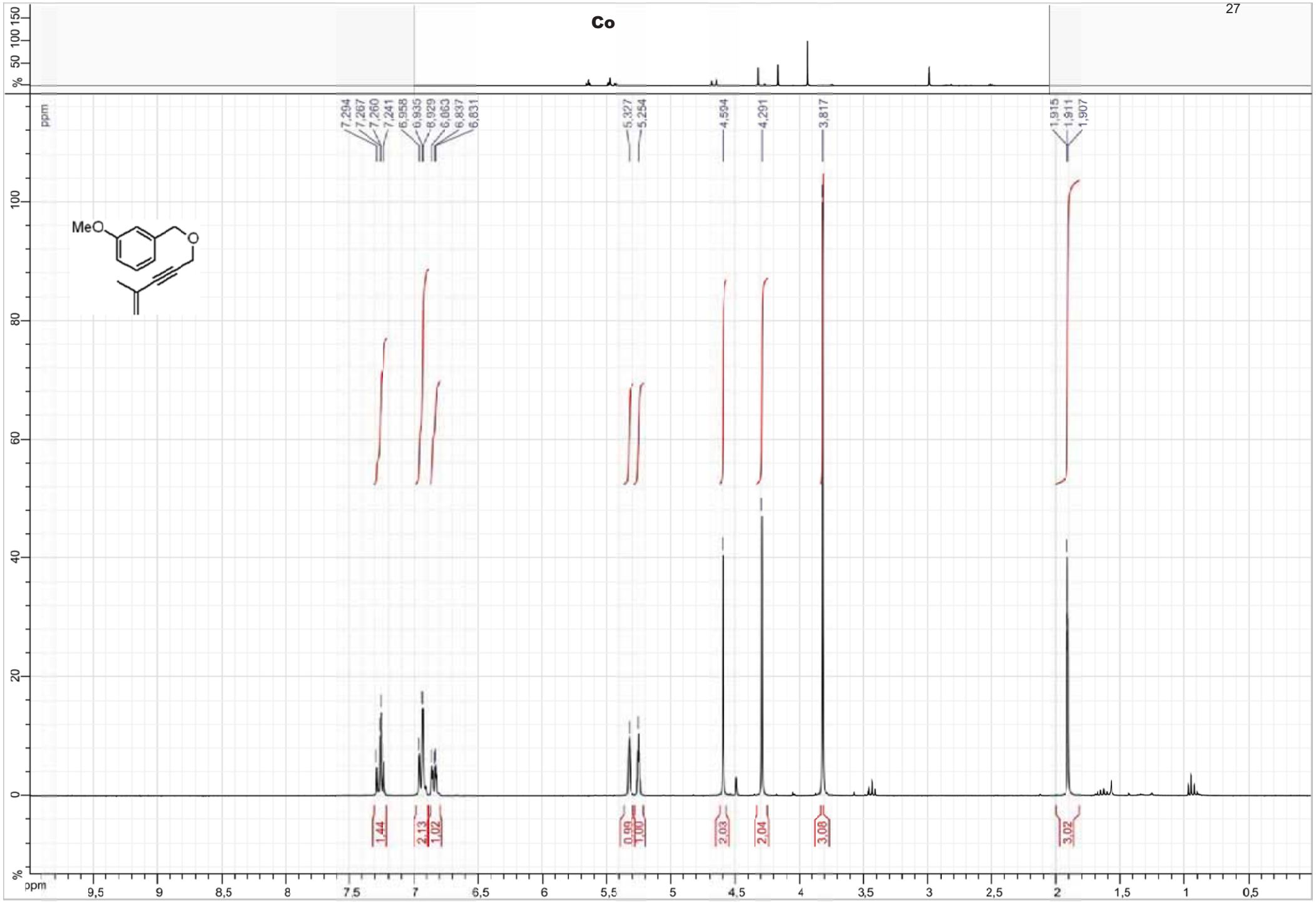
Cm

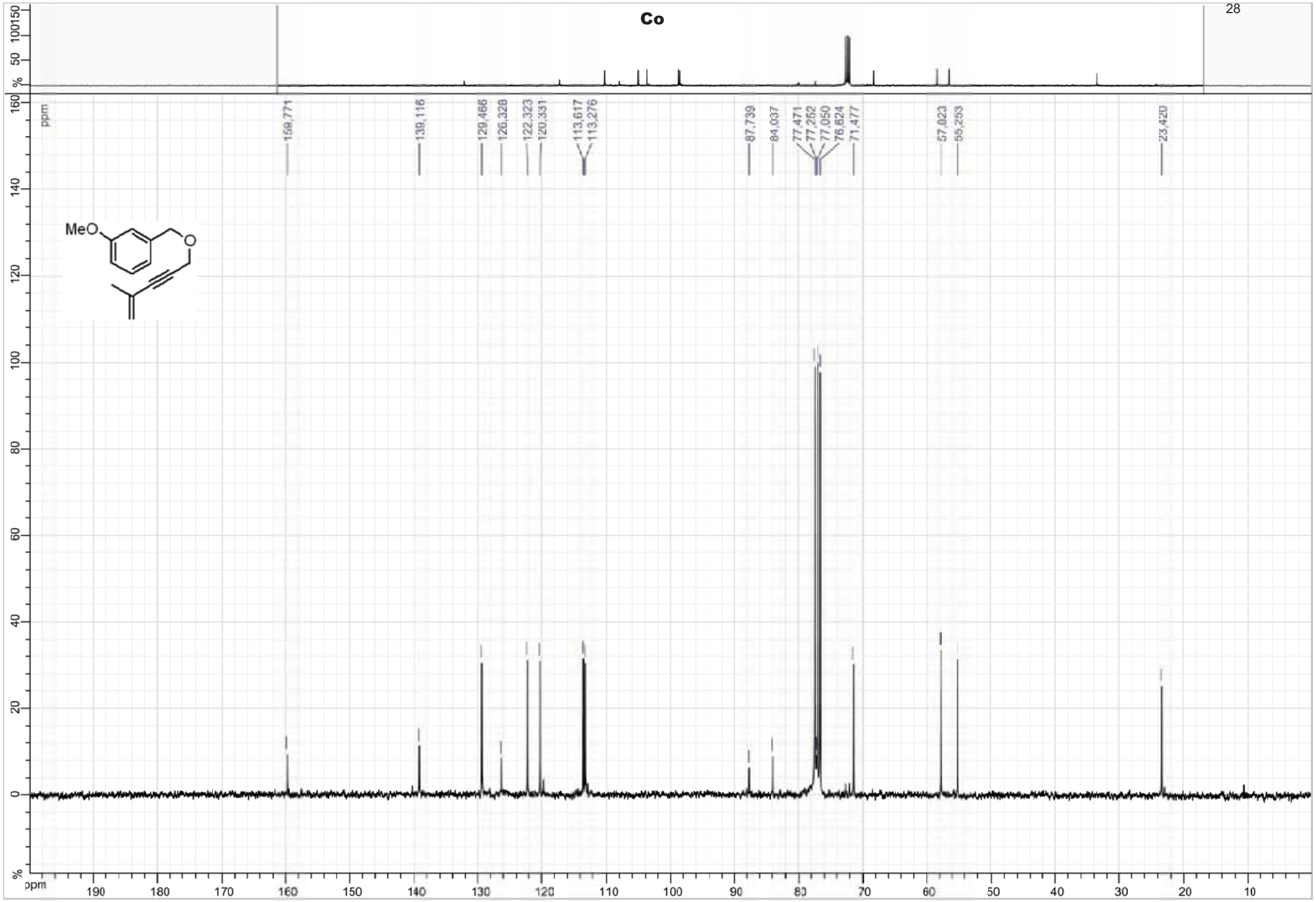

ppm
7,319
7,314
7,303
7,292
7,281
7,270
7,260
7,239
7,234
7,219
7,210
7,195
7,172
5,228
5,173
5,167
5,162
5,156
2,767
2,742
2,717
2,347
2,323
2,300
2,283
2,260
1,912
1,897
1,894
1,863
1,839
1,815
2,05
3,26
0,84
1,00
2,24
2,21
3,05
0,34
ppm
9
8
7
6
5
4
3
2
1


Cm
24
ppm
141,667
128,541
128,355
127,295
125,890
120,450
88,887
82,404
77,449
77,235
77,030
76,603
34,818
30,323
23,889
18,712

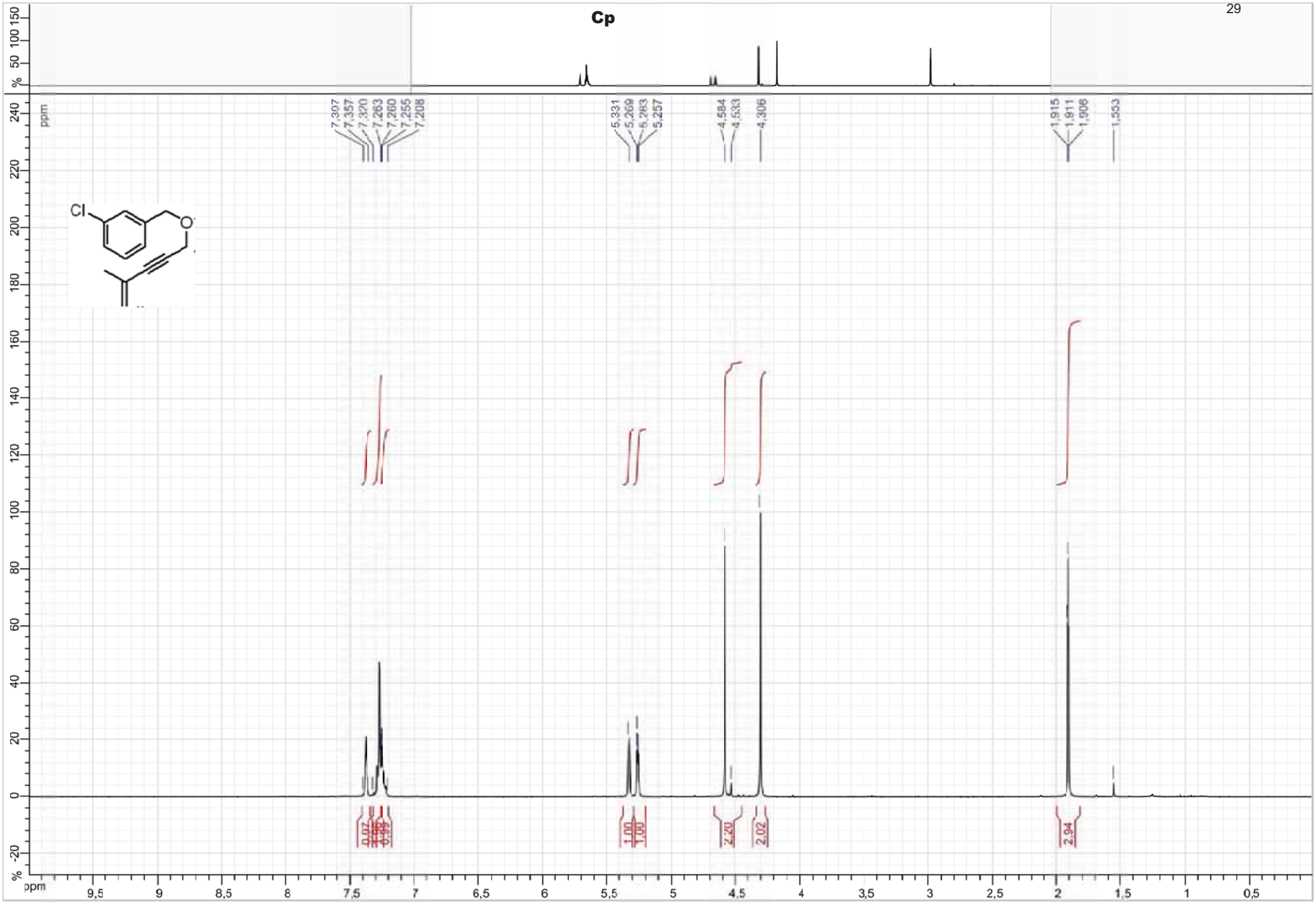

Cn
ppm
7,327
7,303
7,298
7,279
7,260
7,245
7,221
7,214
7,191
5,196
5,143
2,881
2,855
2,830
2,611
2,585
2,560
1,862
1,858
1,853
1,536
water peak
2,03
2,94
2,19
2,17
3,12

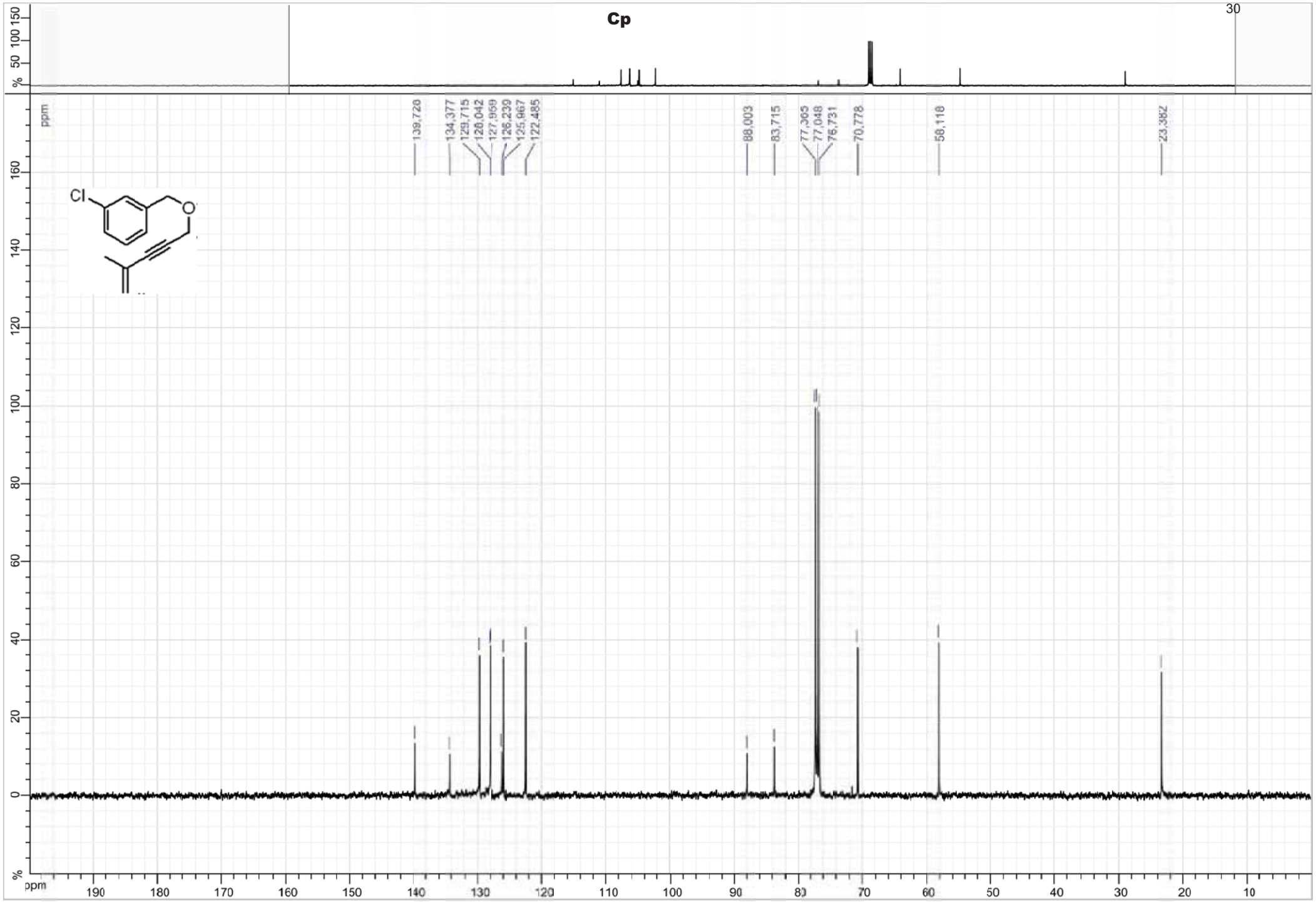

Cn
ppm
140,310
128,095
127,936
126,773
125,854
120,143
88,149
82,160
77,047
76,817
76,196
34,818
23,353
21,160

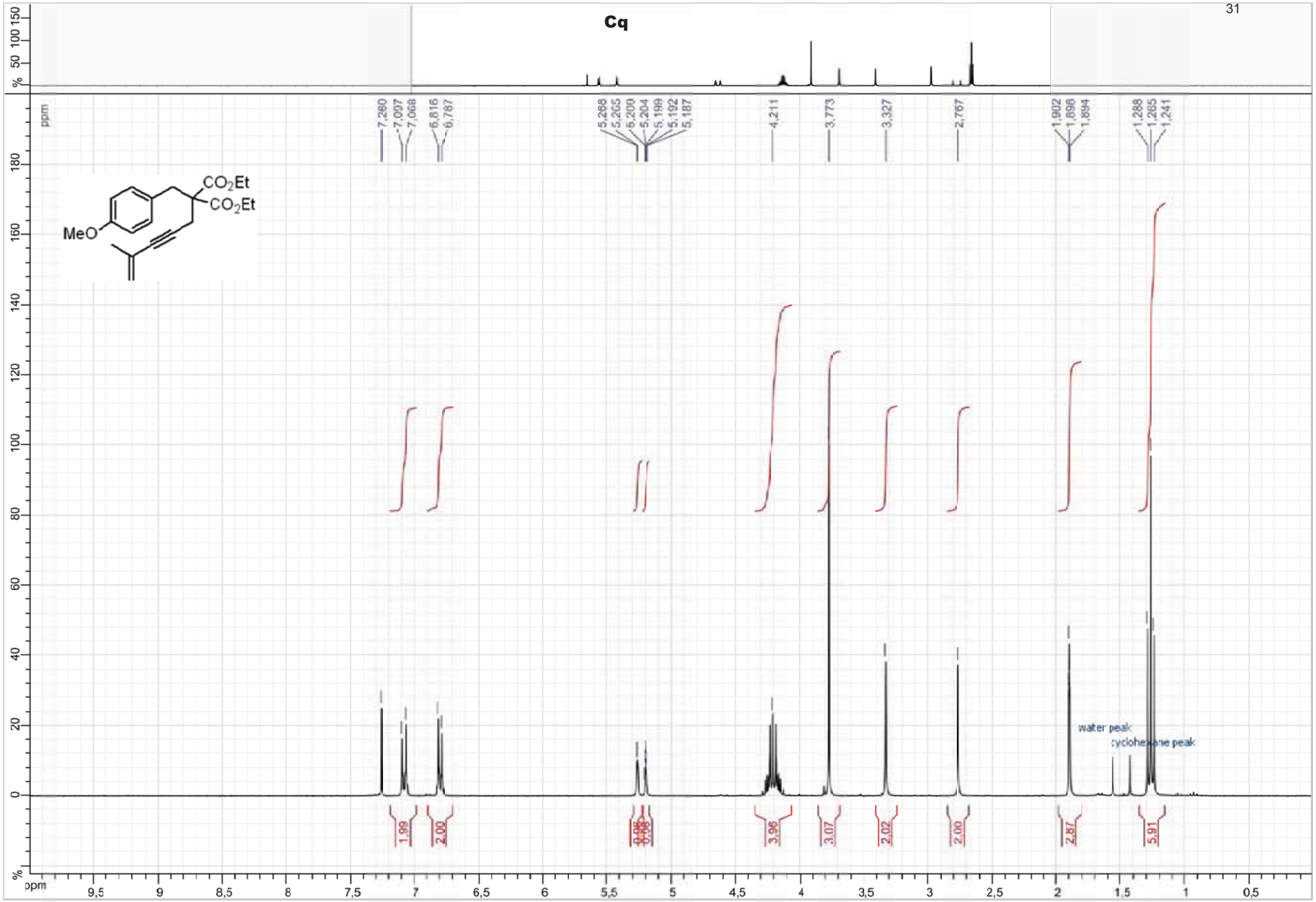

Co
MeO
O
ppm
7,294
7,267
7,260
7,241
6,958
6,935
6,929
6,863
6,837
6,831
5,327
5,254
4,594
4,291
3,817
1,915
1,911
1,907
1,44
2,13
1,02
0,99
1,00
2,03
2,04
3,08
3,02

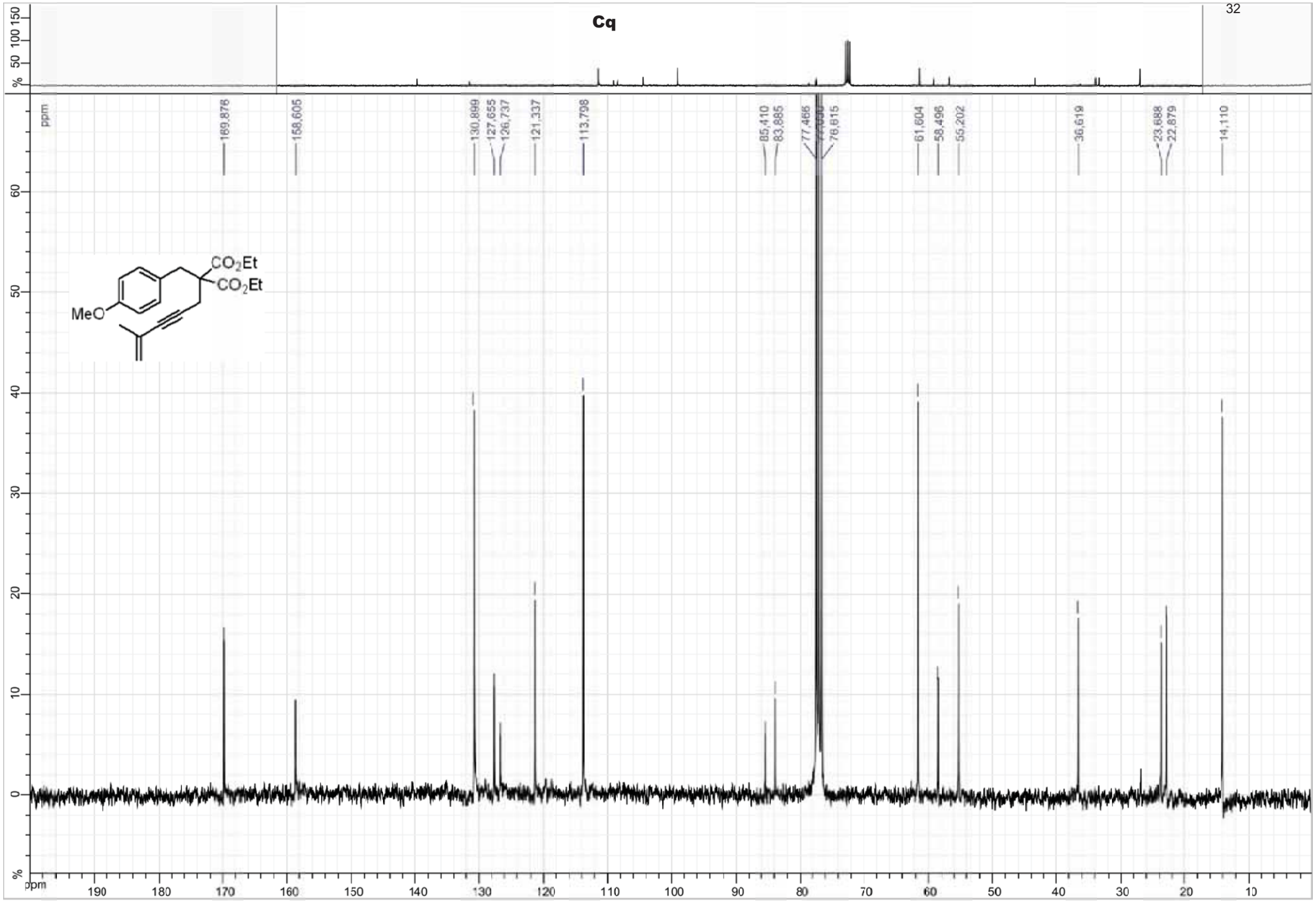

Co
ppm
159,771
139,116
129,466
126,328
122,323
120,331
113,617
113,276
87,739
84,037
77,471
77,252
77,050
76,624
71,477
57,023
55,253
23,420
MeO
O
%
ppm

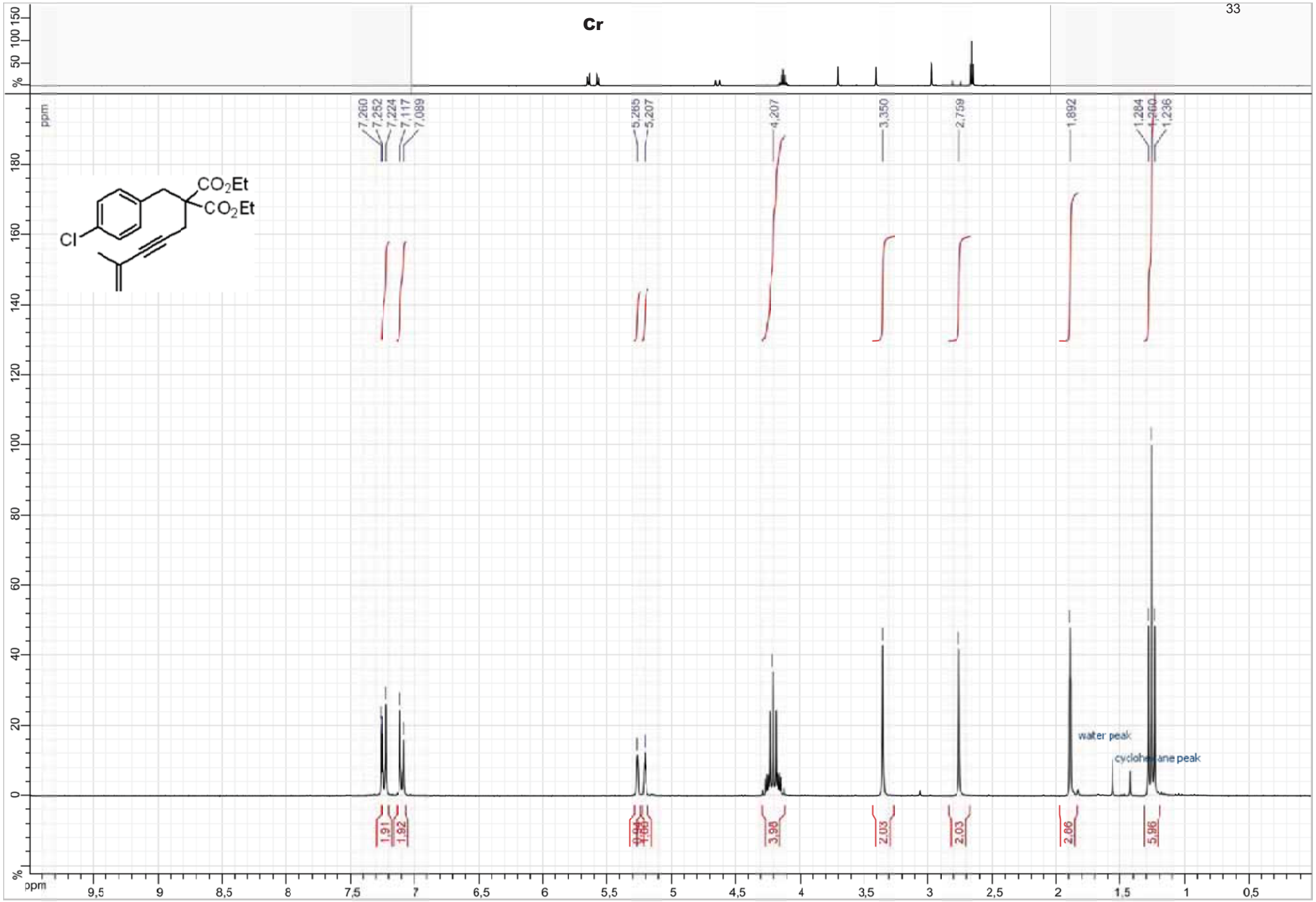
Cp

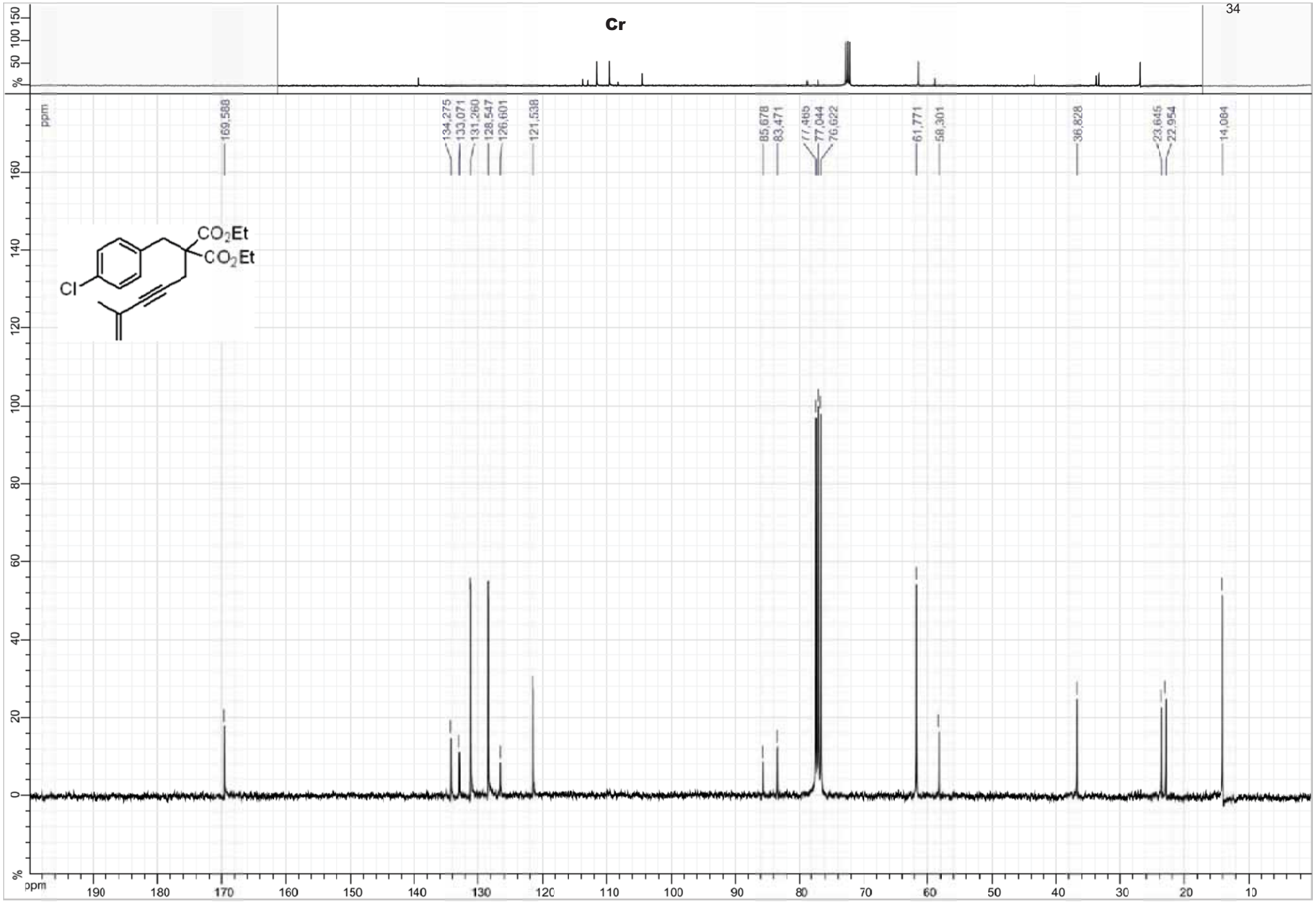

7,307
7,357
7,320
7,263
7,260
7,255
7,208
5,331
5,269
5,263
5,257
4,584
4,533
4,306
1,915
1,911
1,908
1,553
Cl
O
1,00
1,00
2,20
2,02
2,94
ppm
9,5
9
8,5
8
7,5
7
6,5
6
5,5
5
4,5
4
3,5
3
2,5
2
1,5
1
0,5


Cp
30
ppm
139,720
134,377
129,715
128,042
127,959
126,239
125,967
122,485
88,003
83,715
77,365
77,048
76,731
70,778
58,118
23,382
Cl
O

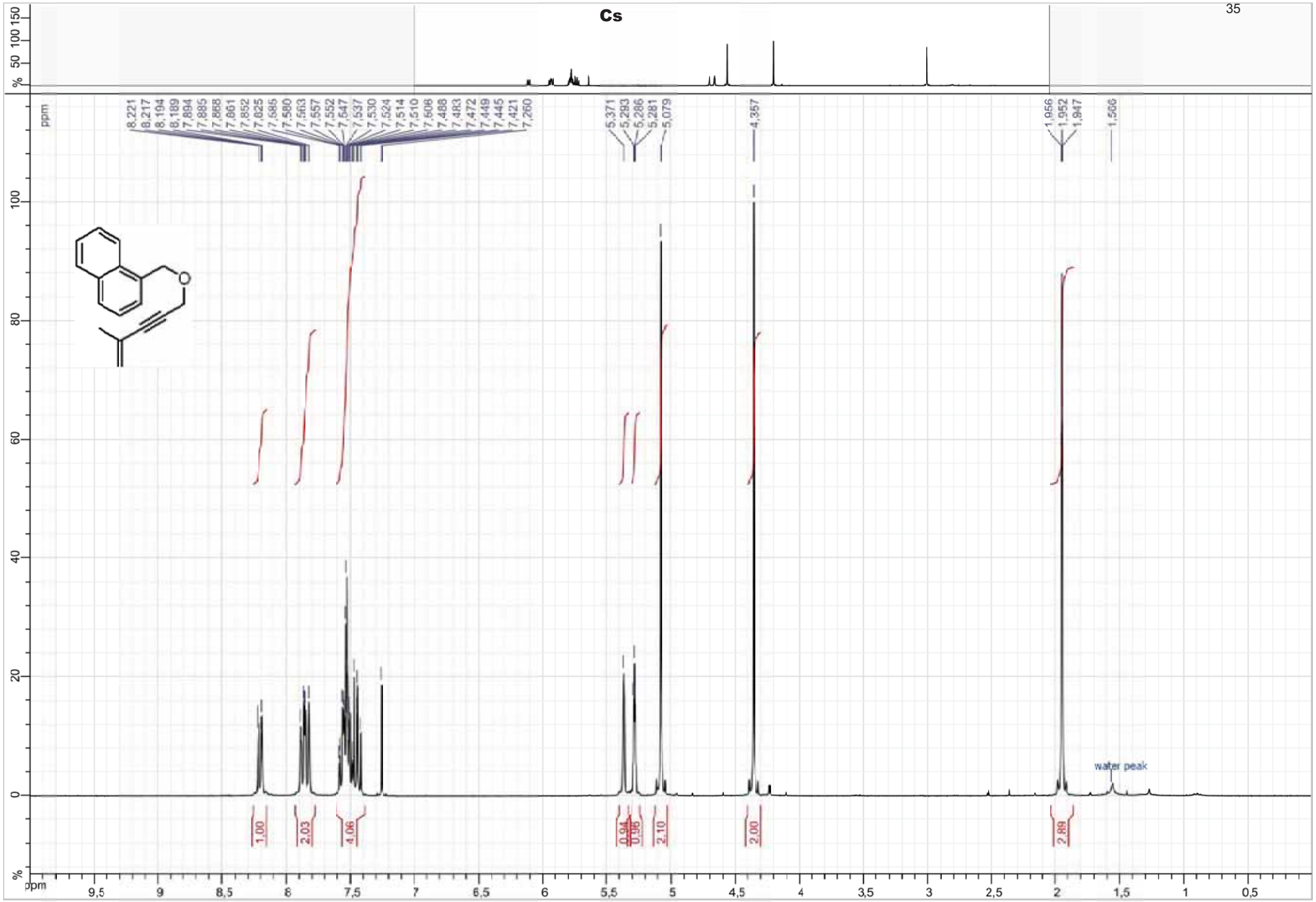

Cq
ppm
7,260
7,097
7,068
6,816
6,787
5,268
5,265
5,200
5,204
5,199
5,192
5,187
4,211
3,773
3,327
2,767
1,902
1,898
1,894
1,288
1,265
1,241
MeO
CO2Et
CO2Et
water peak
cyclohexane peak
1,99
2,00
3,96
3,07
2,02
2,00
2,87
5,91


Cq
ppm
169,876
158,605
130,899
127,655
126,737
121,337
113,798
85,410
83,885
77,466
76,615
61,604
58,496
55,202
36,619
23,688
22,879
14,110
CO2Et
CO2Et
MeO

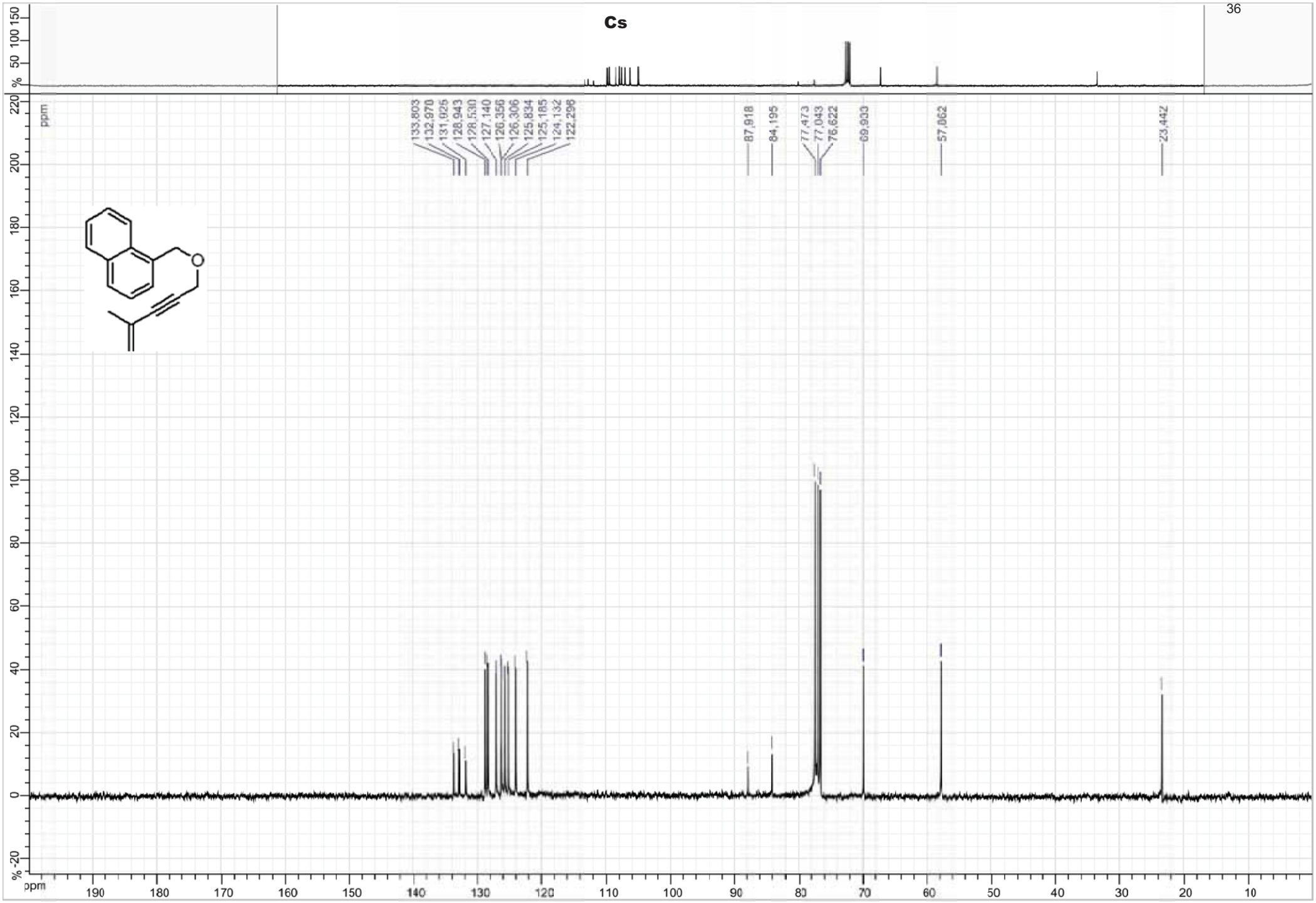

Cr
ppm
7,260
7,252
7,224
7,117
7,089
5,265
5,207
4,207
3,350
2,759
1,892
1,284
1,260
1,236
CO2Et
CO2Et
Cl
water peak
cyclohexane peak
1,91
1,92
3,98
2,03
2,03
2,86
5,96
ppm

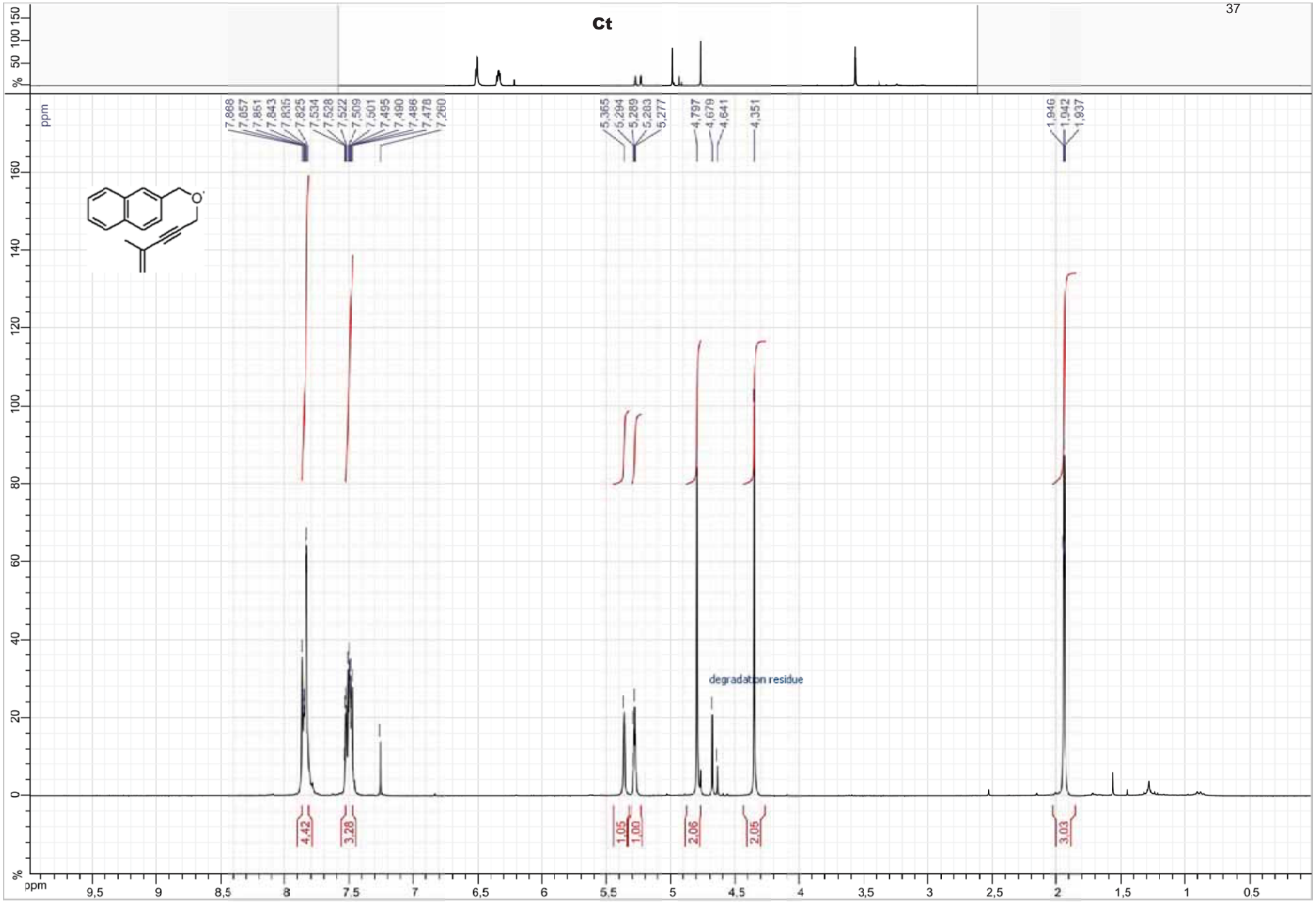

Cr
ppm
169,588
134,275
133,071
131,260
128,547
126,601
121,538
85,678
83,471
77,466
77,044
76,622
61,771
58,301
36,828
23,645
22,954
14,084
CO2Et
CO2Et
Cl

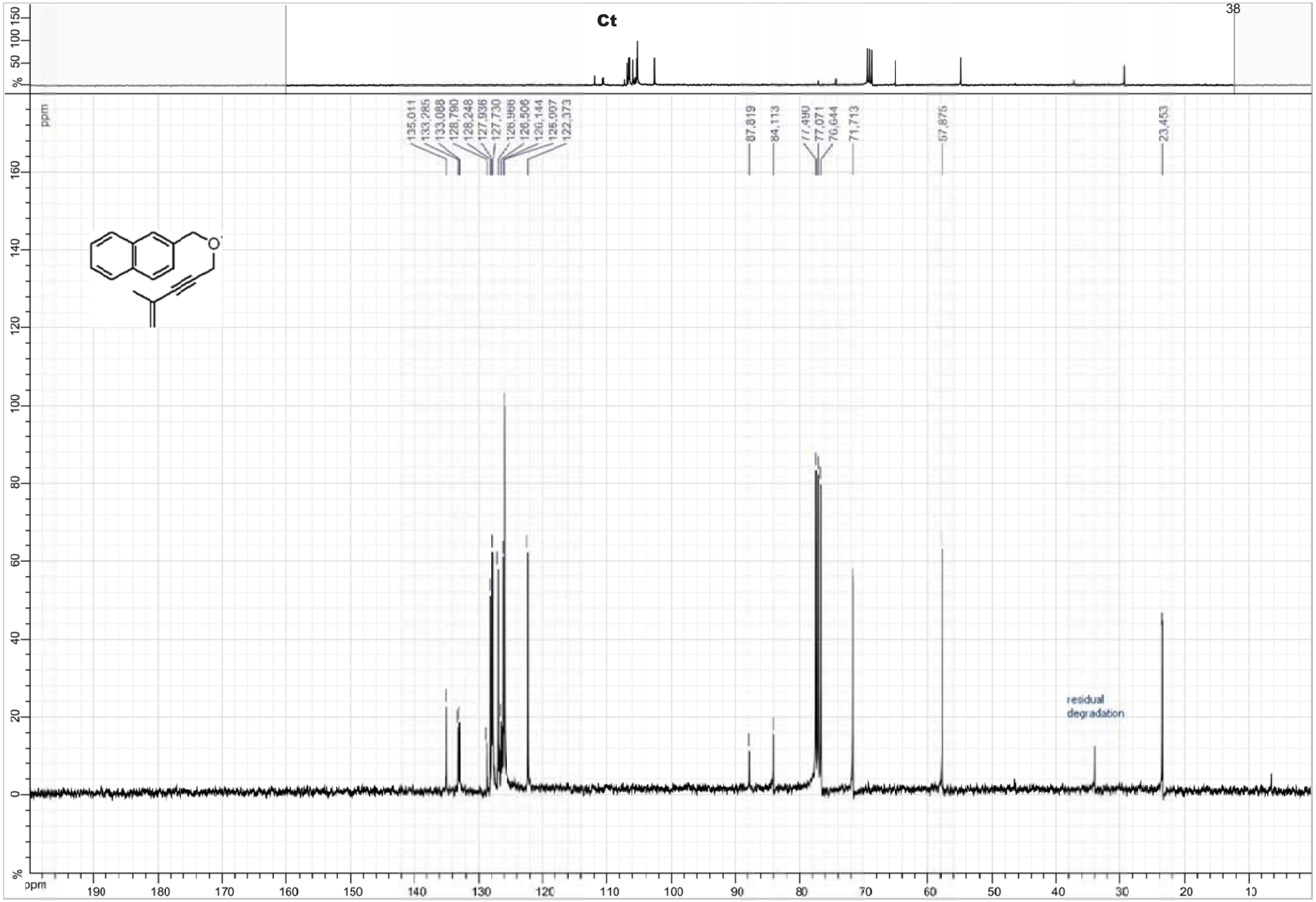
Cs

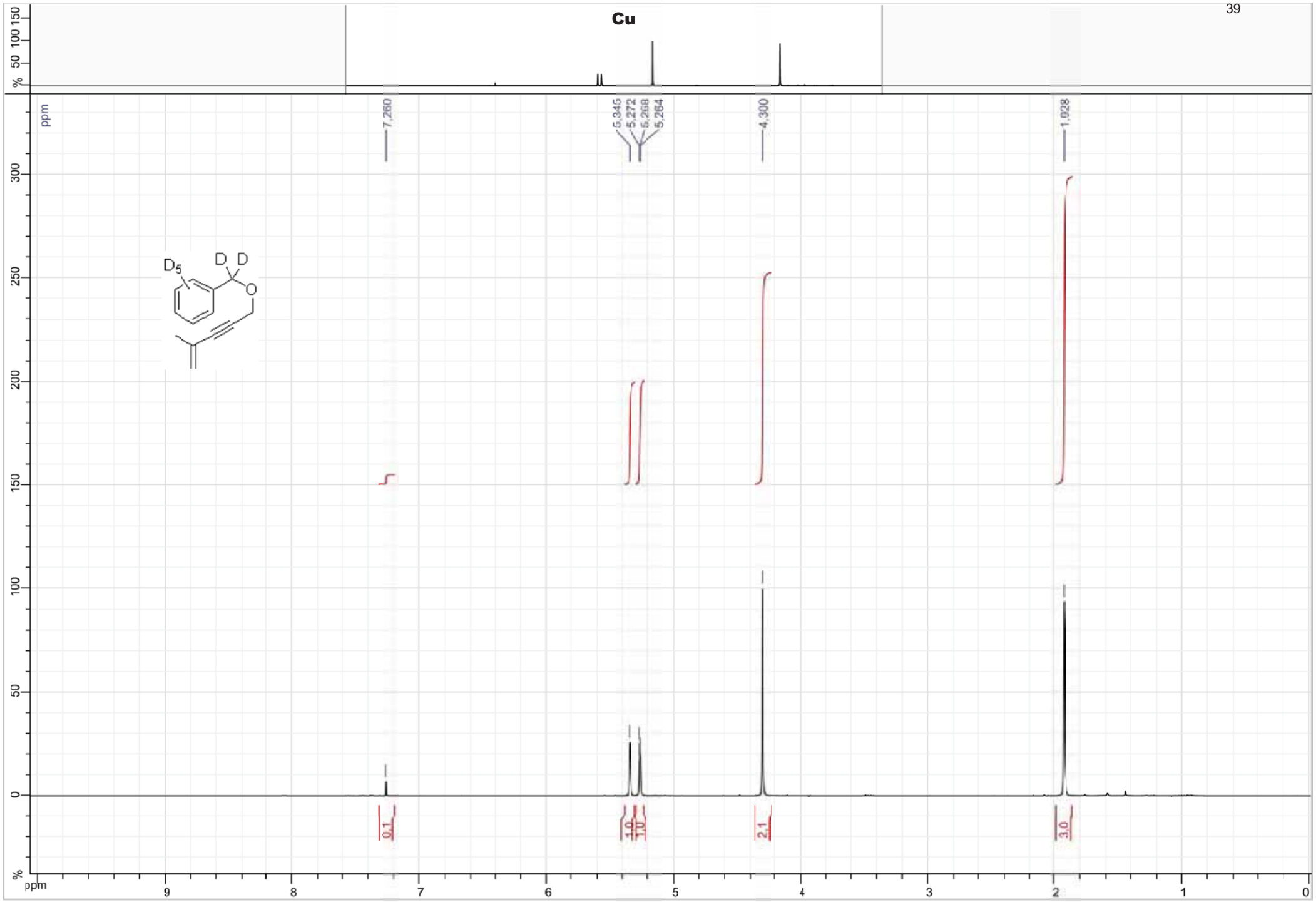

ppm
8,221
8,217
8,194
8,189
7,894
7,885
7,868
7,861
7,852
7,825
7,585
7,580
7,563
7,557
7,552
7,547
7,537
7,530
7,524
7,514
7,510
7,506
7,488
7,483
7,472
7,449
7,445
7,421
7,260
5,371
5,293
5,286
5,281
5,079
4,357
1,956
1,952
1,847
1,566
water peak
1,00
2,03
4,06
0,94
0,96
2,10
2,00
2,89

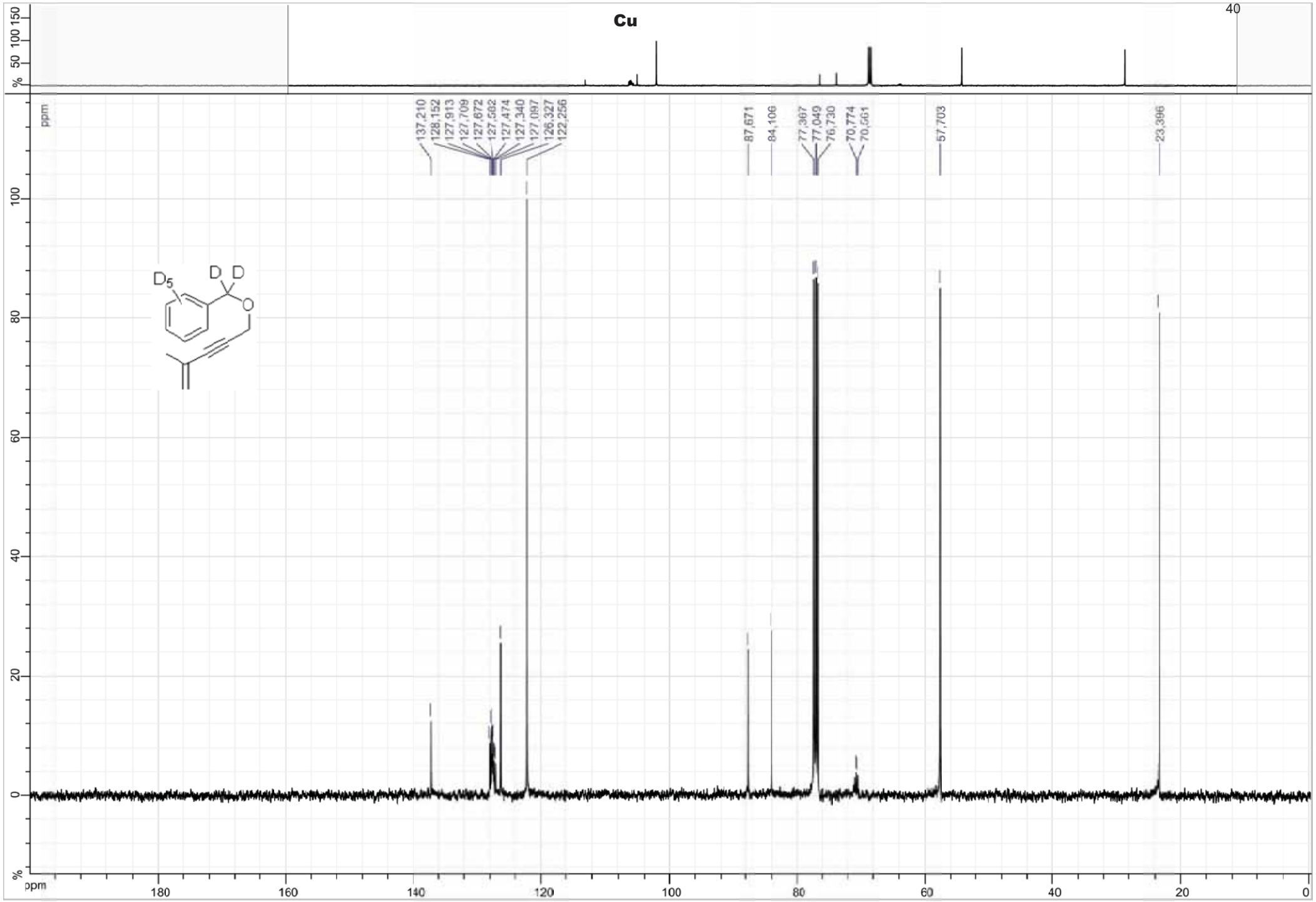
Cs


ppm
133,803
132,970
131,925
128,943
128,530
127,140
126,356
126,306
125,834
125,185
124,132
122,296
87,918
84,195
77,473
77,043
76,622
69,933
57,062
23,442
O

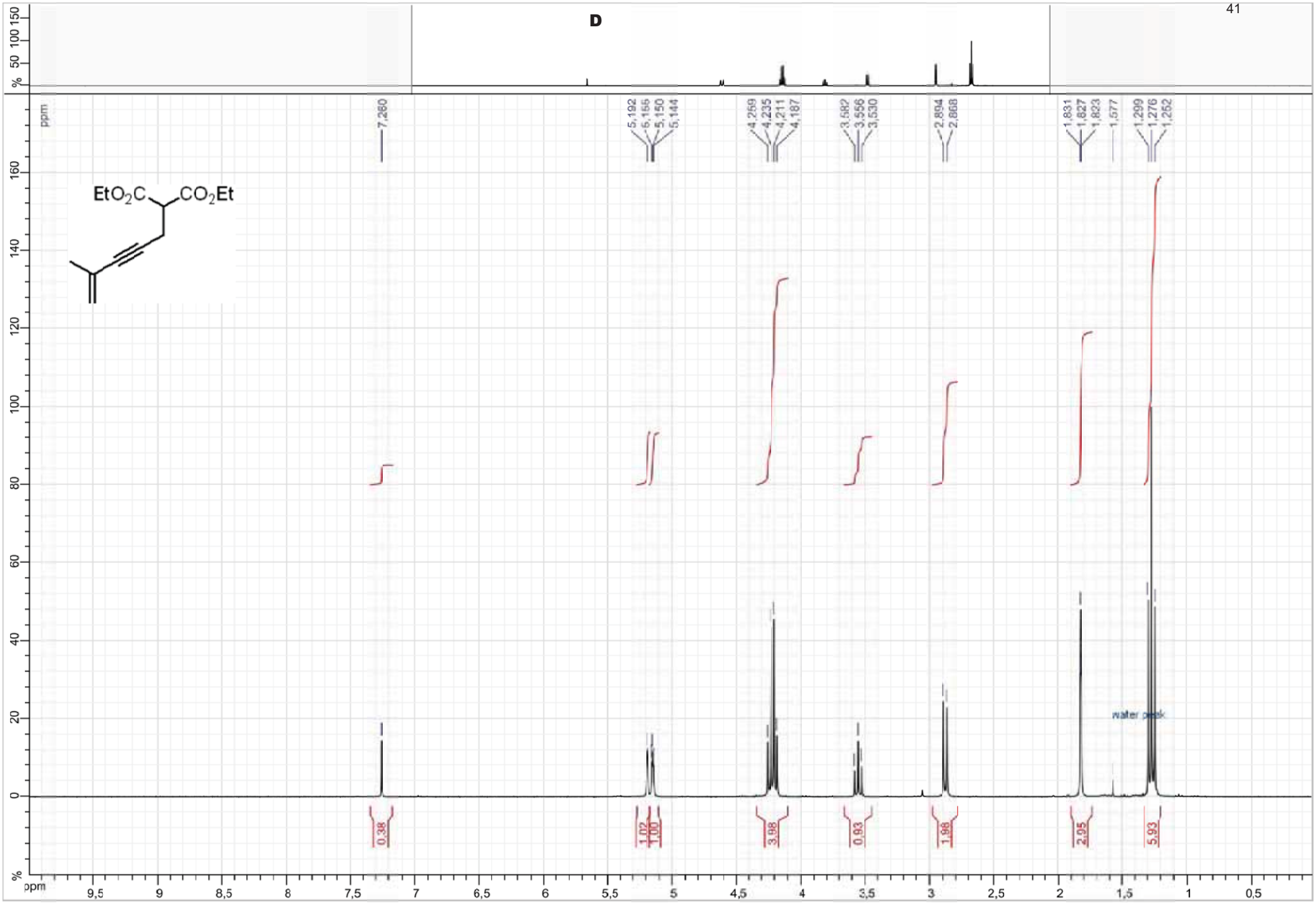

Ct
ppm
7,868
7,857
7,851
7,843
7,835
7,825
7,534
7,528
7,522
7,509
7,501
7,495
7,490
7,486
7,478
7,260
5,365
5,294
5,289
5,283
5,277
4,797
4,679
4,641
4,351
1,946
1,942
1,937
degradation residue
4,42
3,28
1,05
1,00
2,06
2,05
3,03


Ct
ppm
135,011
133,285
133,088
128,790
128,248
127,936
127,730
126,966
126,506
126,144
125,097
122,373
87,819
84,113
77,490
77,071
76,644
71,713
57,875
23,453
residual
degradation
ppm

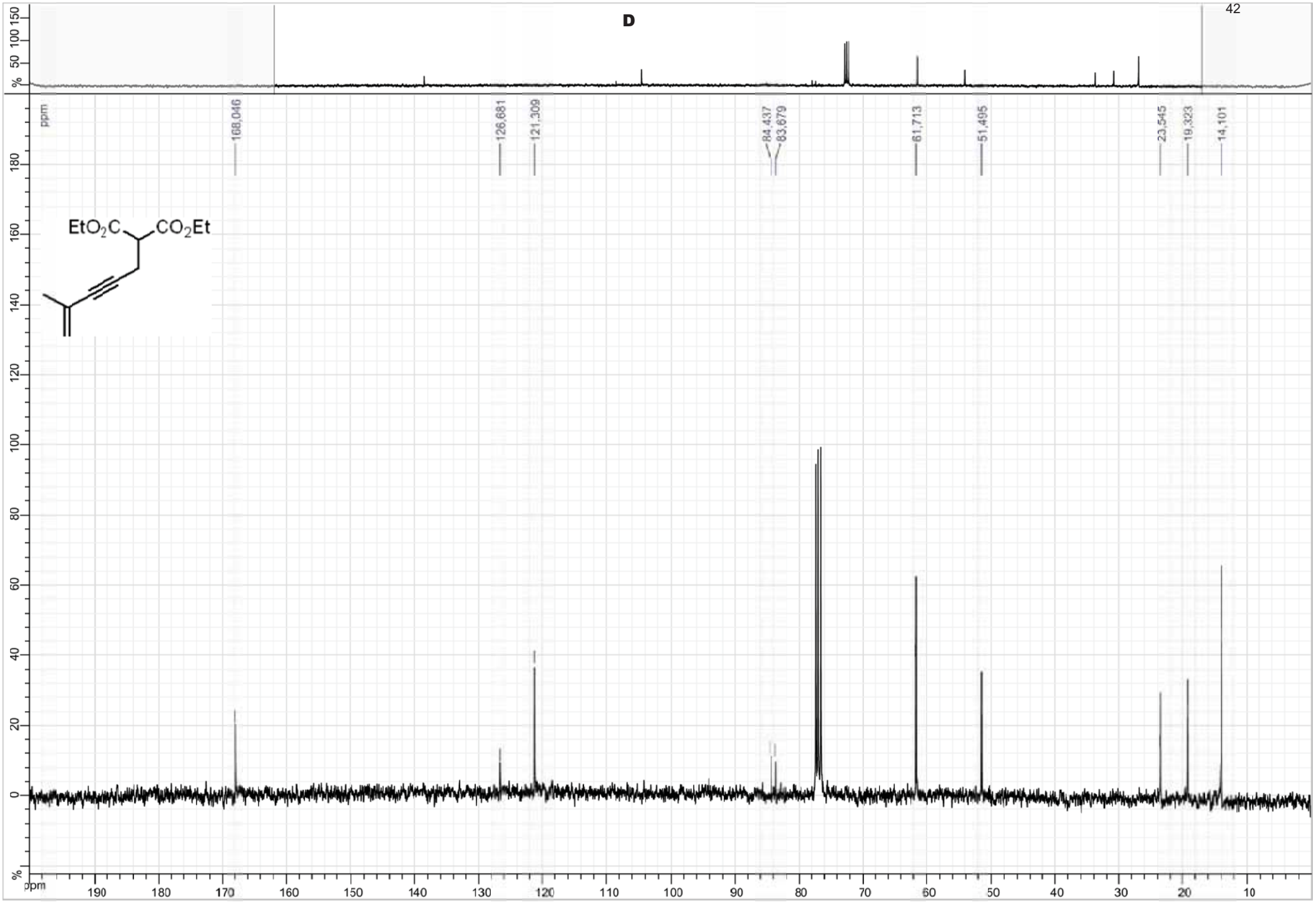

Cu
ppm
7,260
5,345
5,272
5,268
5,264
4,300
1,928
D5
D D
O
0,1
1,0
1,0
2,1
3,0
%
ppm

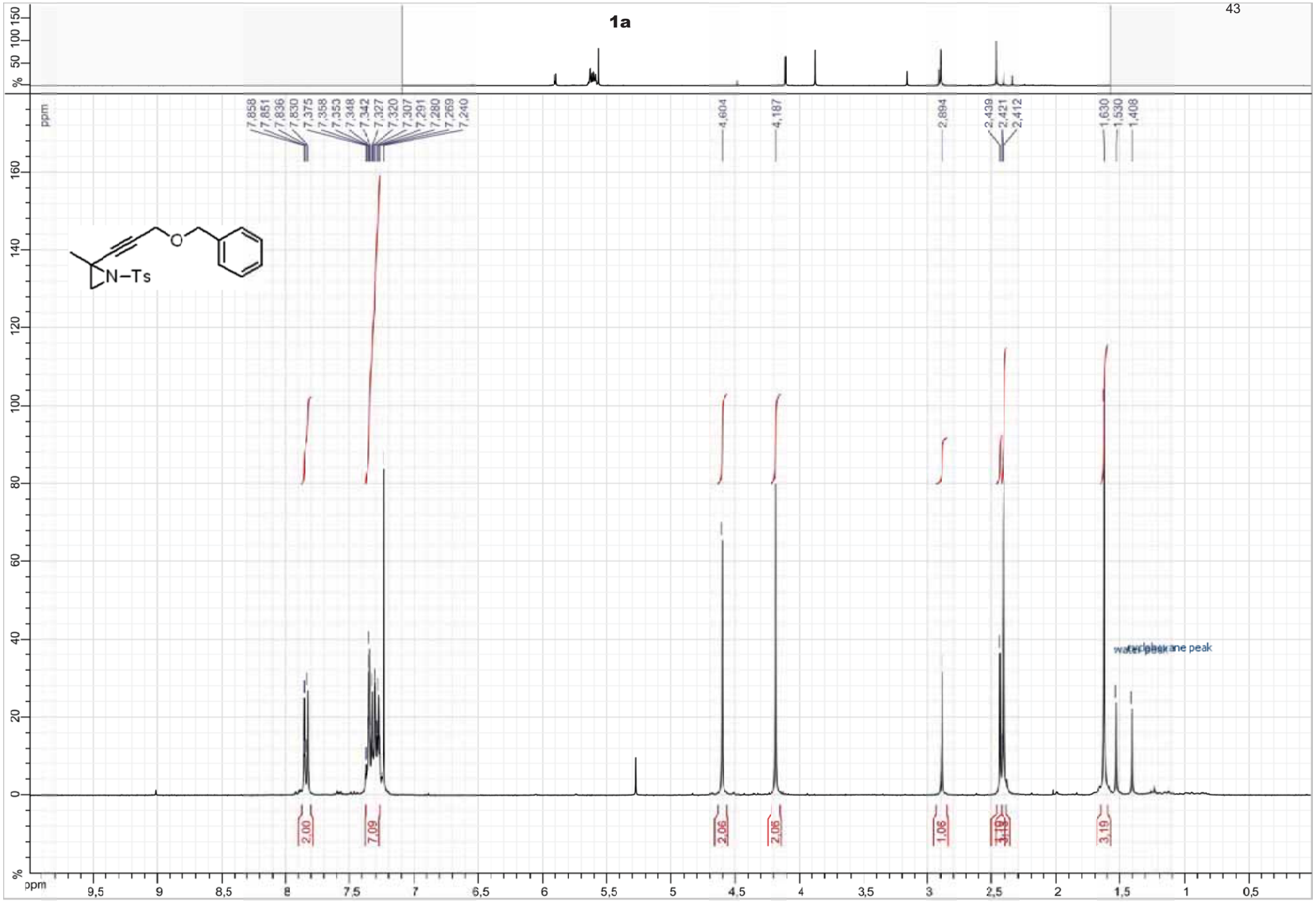

Cu
40
ppm
137,210
128,152
127,913
127,709
127,672
127,562
127,474
127,340
127,097
126,327
122,256
87,671
84,106
77,367
77,049
76,730
70,774
70,561
57,703
23,396
D5
D
D
O
%
ppm
180
160
140
120
100
80
60
40
20
0

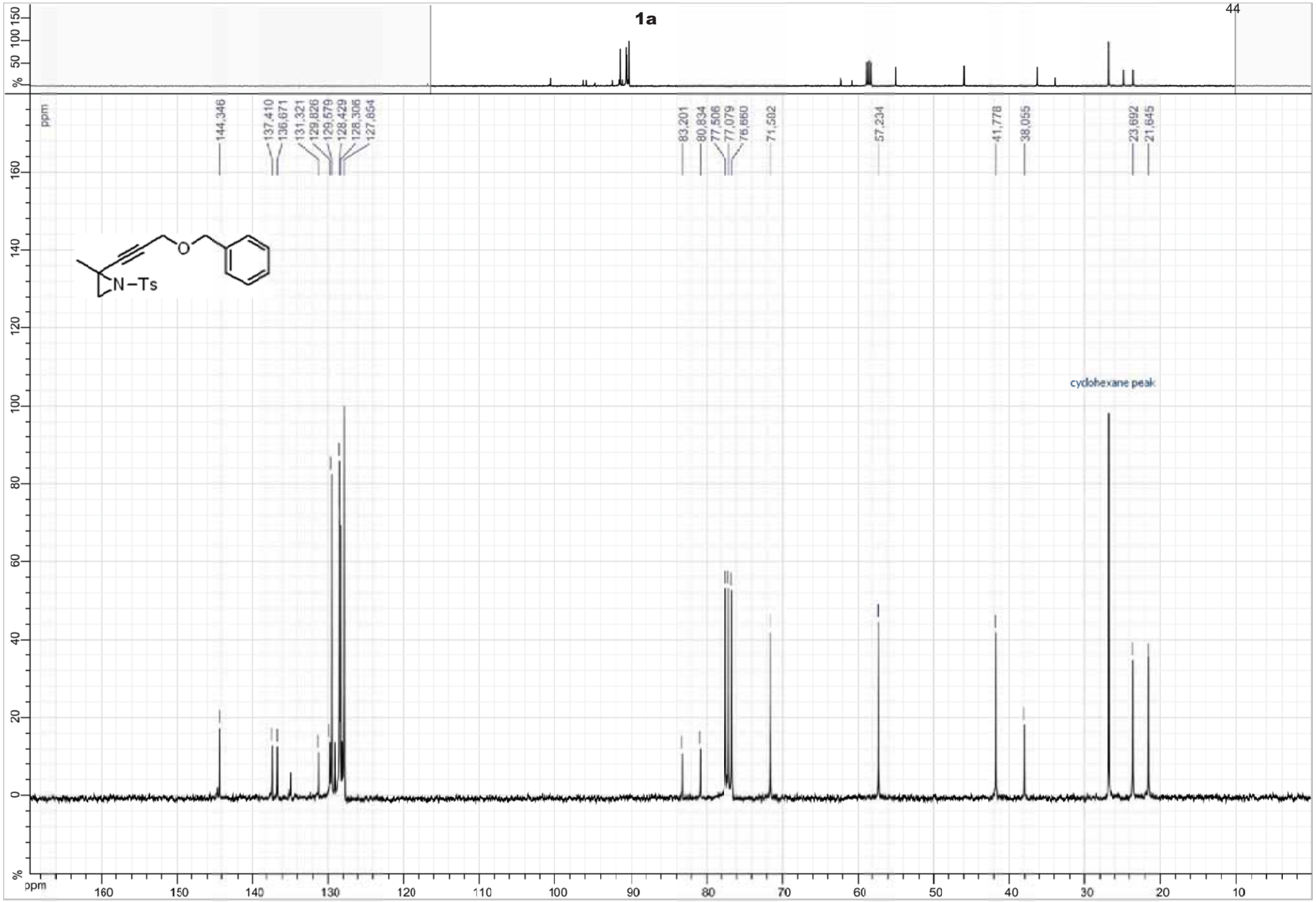
D

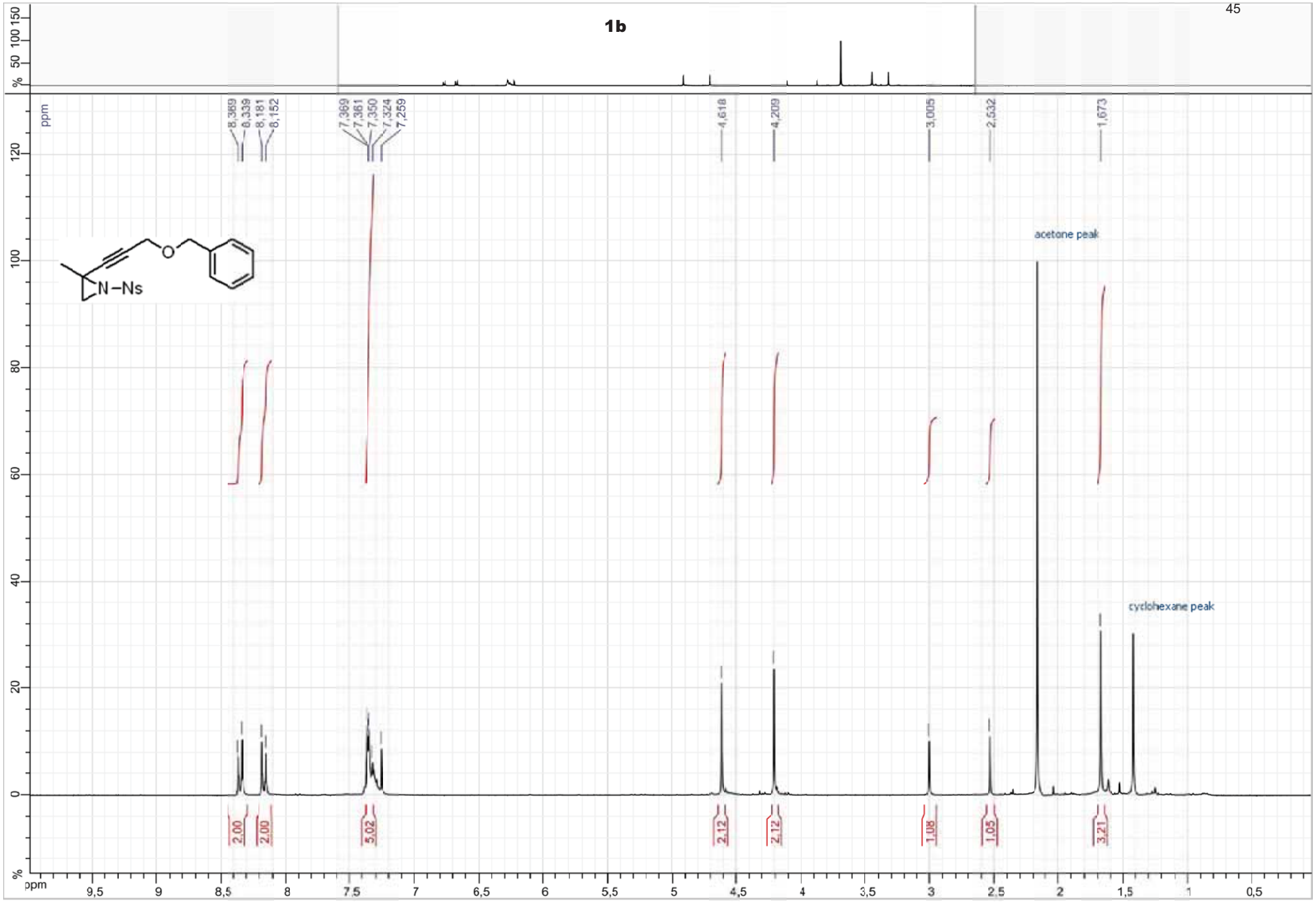

EtO2C
CO2Et
7,260
5,192
5,155
5,150
5,144
4,259
4,235
4,211
4,187
3,582
3,556
3,530
2,894
2,868
1,831
1,827
1,823
1,577
1,299
1,276
1,252
water peak
0,38
1,02
1,00
3,98
0,93
1,98
2,95
5,93
ppm

D

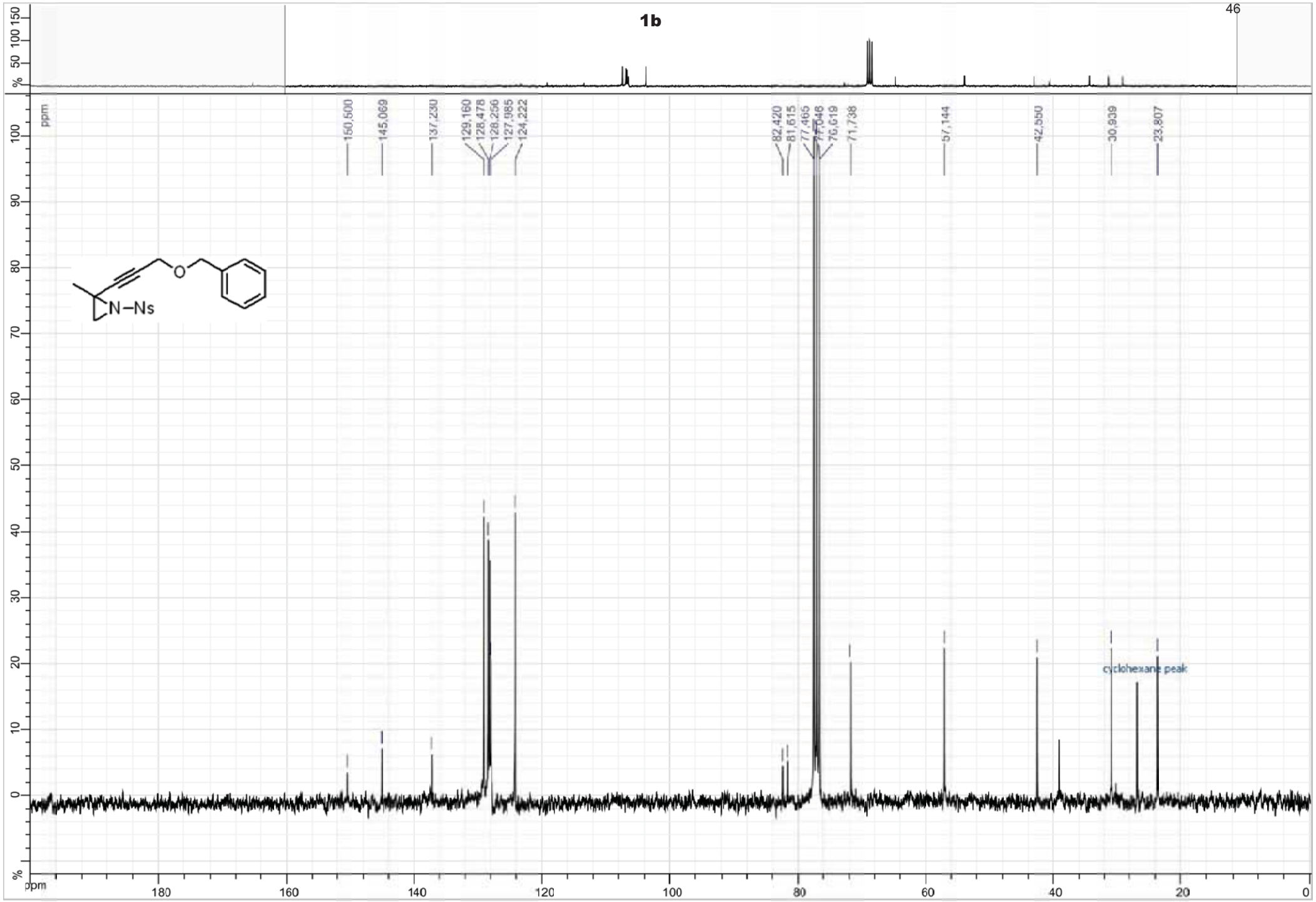

ppm
168,046
126,681
121,309
84,437
83,679
61,713
51,495
23,545
19,323
14,101
EtO2C
CO2Et


1a
7,858
7,851
7,836
7,830
7,375
7,358
7,353
7,348
7,342
7,327
7,320
7,307
7,291
7,280
7,269
7,240
4,604
4,187
2,894
2,439
2,421
2,412
1,630
1,530
1,408
N–Ts
O
2,00
7,09
2,06
2,06
1,06
3,19
3,19
ppm

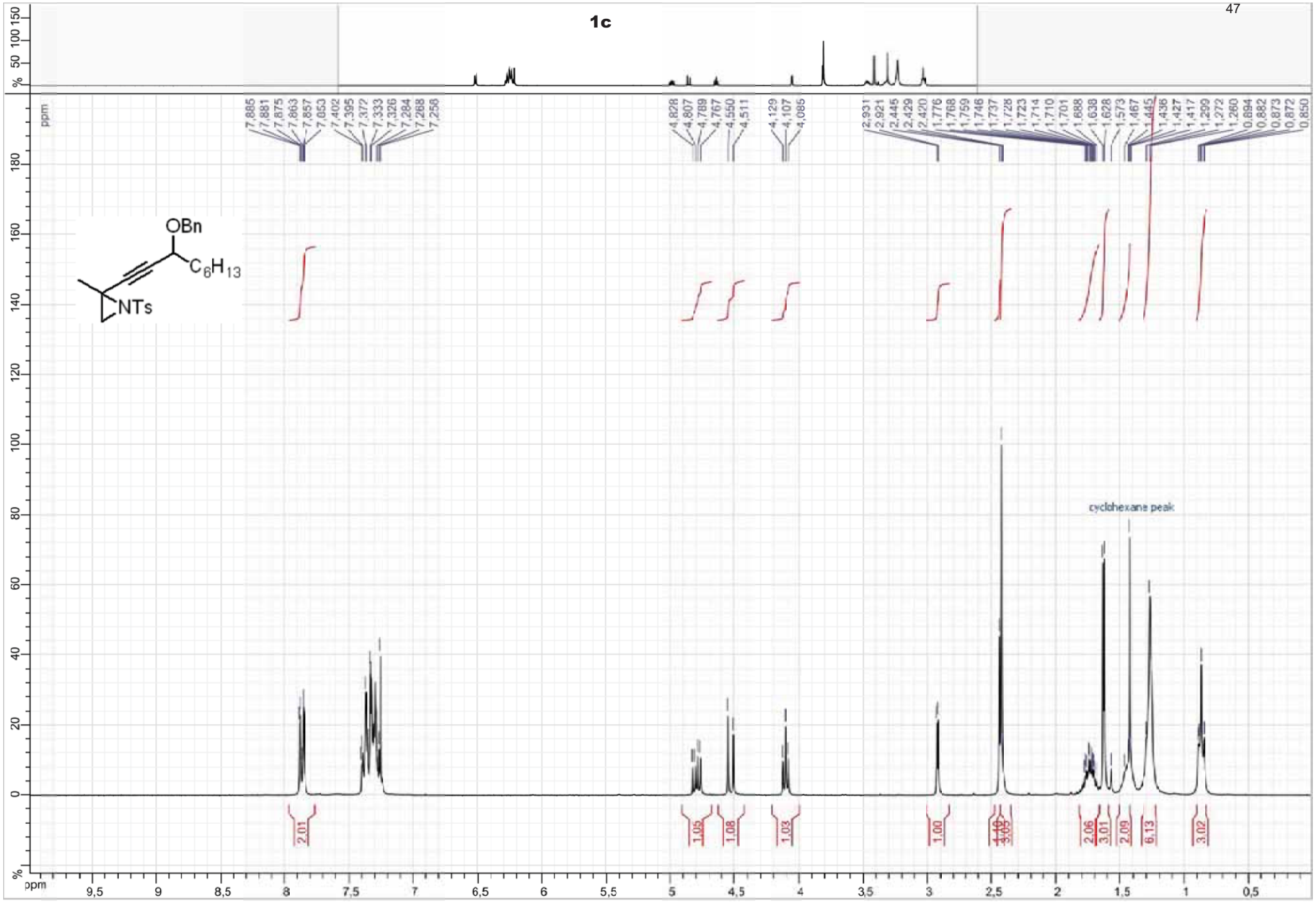

1a
ppm
144,346
137,410
136,671
131,321
129,826
129,579
128,429
128,306
127,854
83,201
80,834
77,506
77,079
76,660
71,582
57,234
41,778
38,055
23,692
21,645
N–Ts
cyclohexane peak

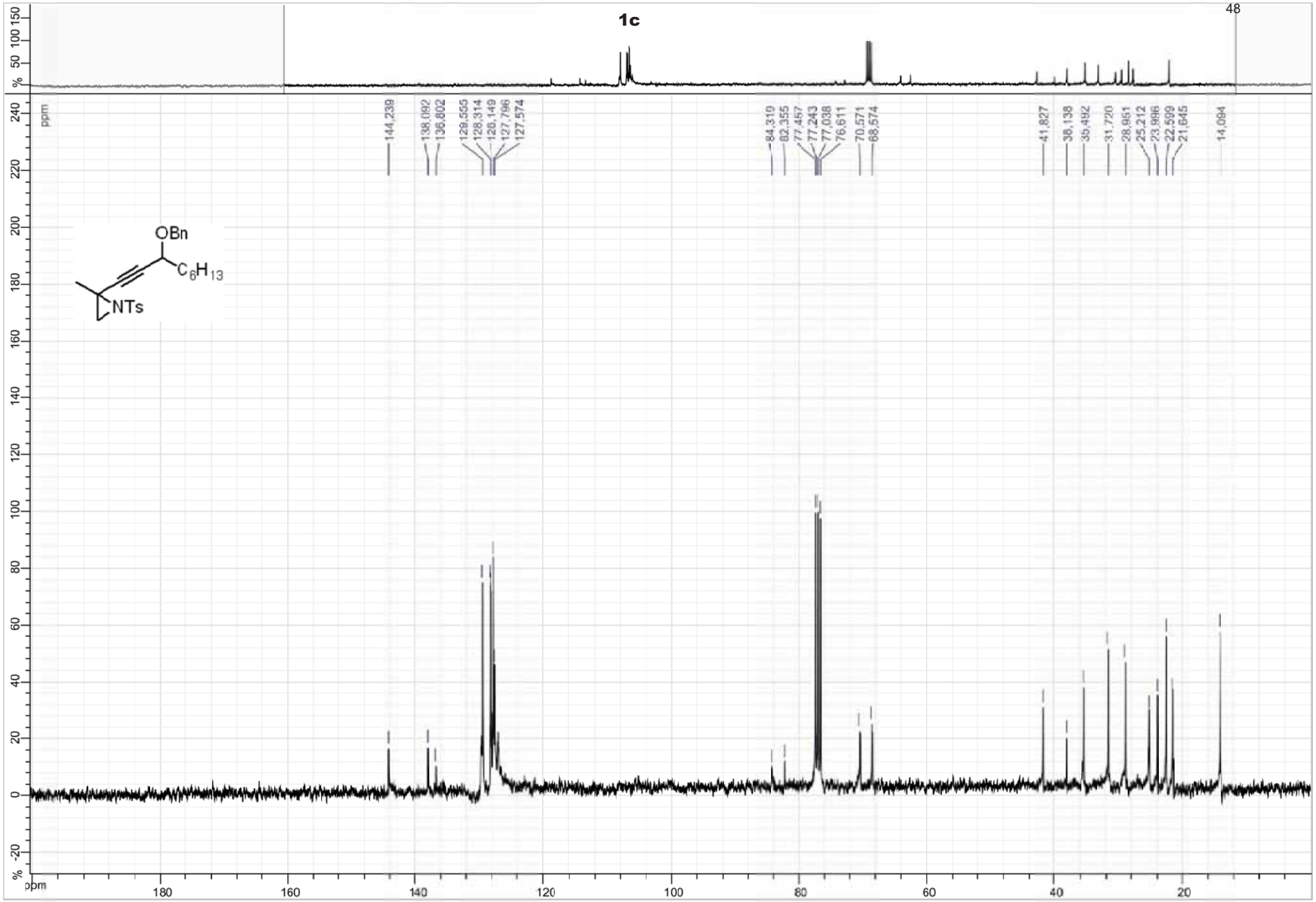

1b
ppm
8,369
8,339
8,181
8,152
7,369
7,361
7,350
7,324
7,259
4,618
4,209
3,005
2,532
1,673
N–Ns
acetone peak
cyclohexane peak
2,00
2,00
5,02
2,12
2,12
1,08
1,05
3,21

1b

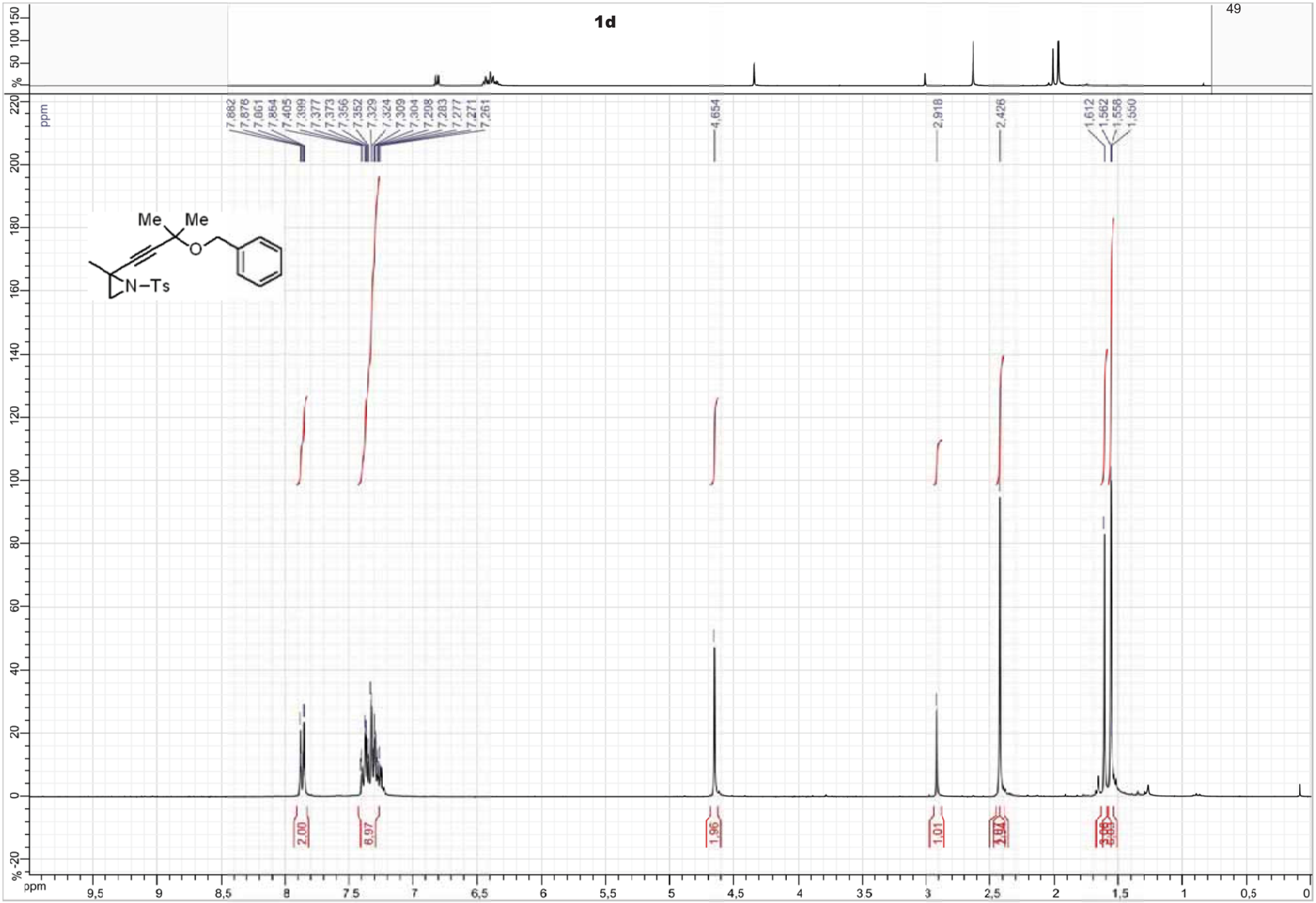

150,500
145,069
137,230
129,160
128,478
128,256
127,985
124,222
82,420
81,615
77,465
77,046
76,619
71,738
57,144
42,550
30,939
23,907
cyclohexane peak
N–Ns
ppm
180
160
140
120
100
80
60
40
20
0

1c

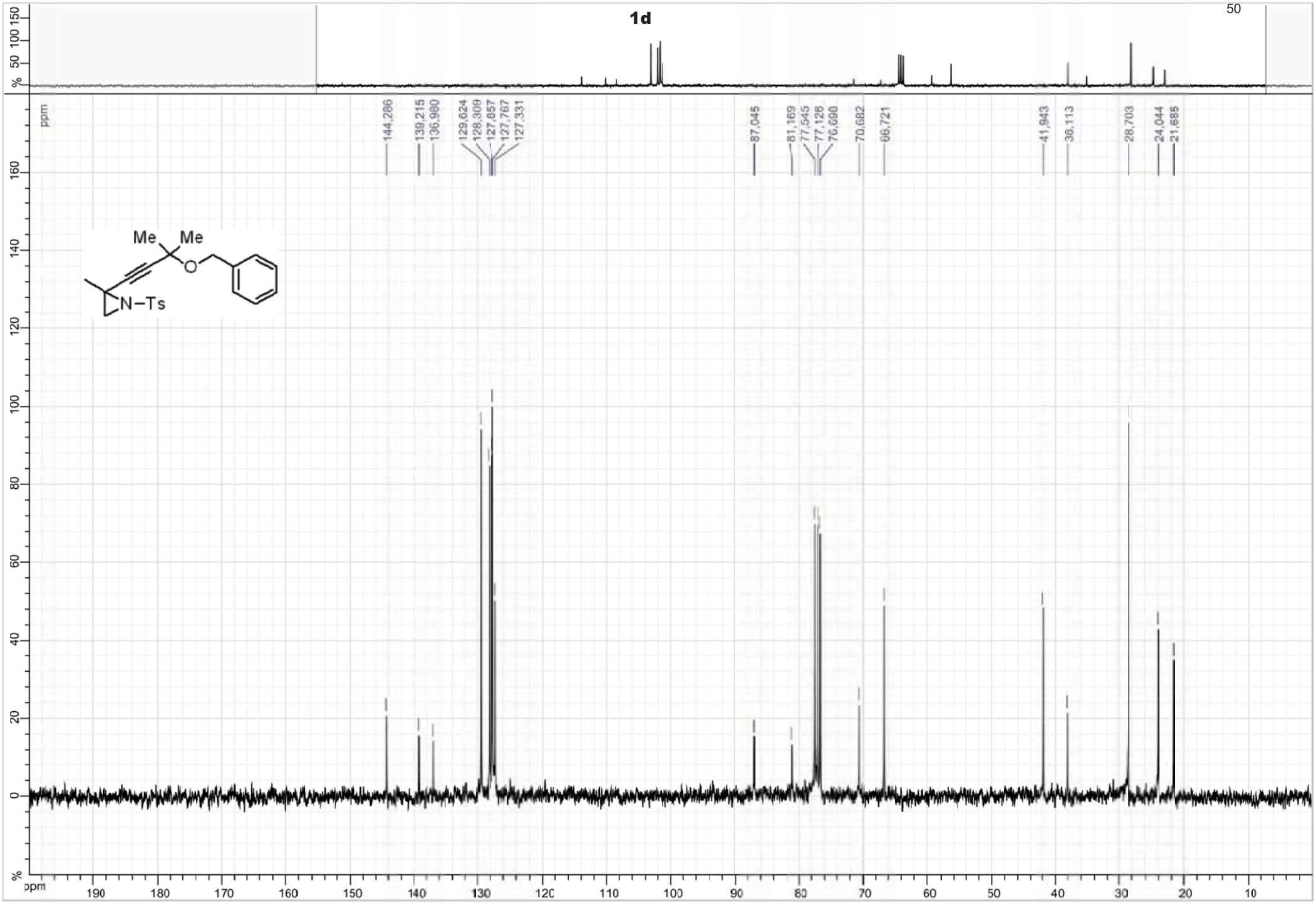


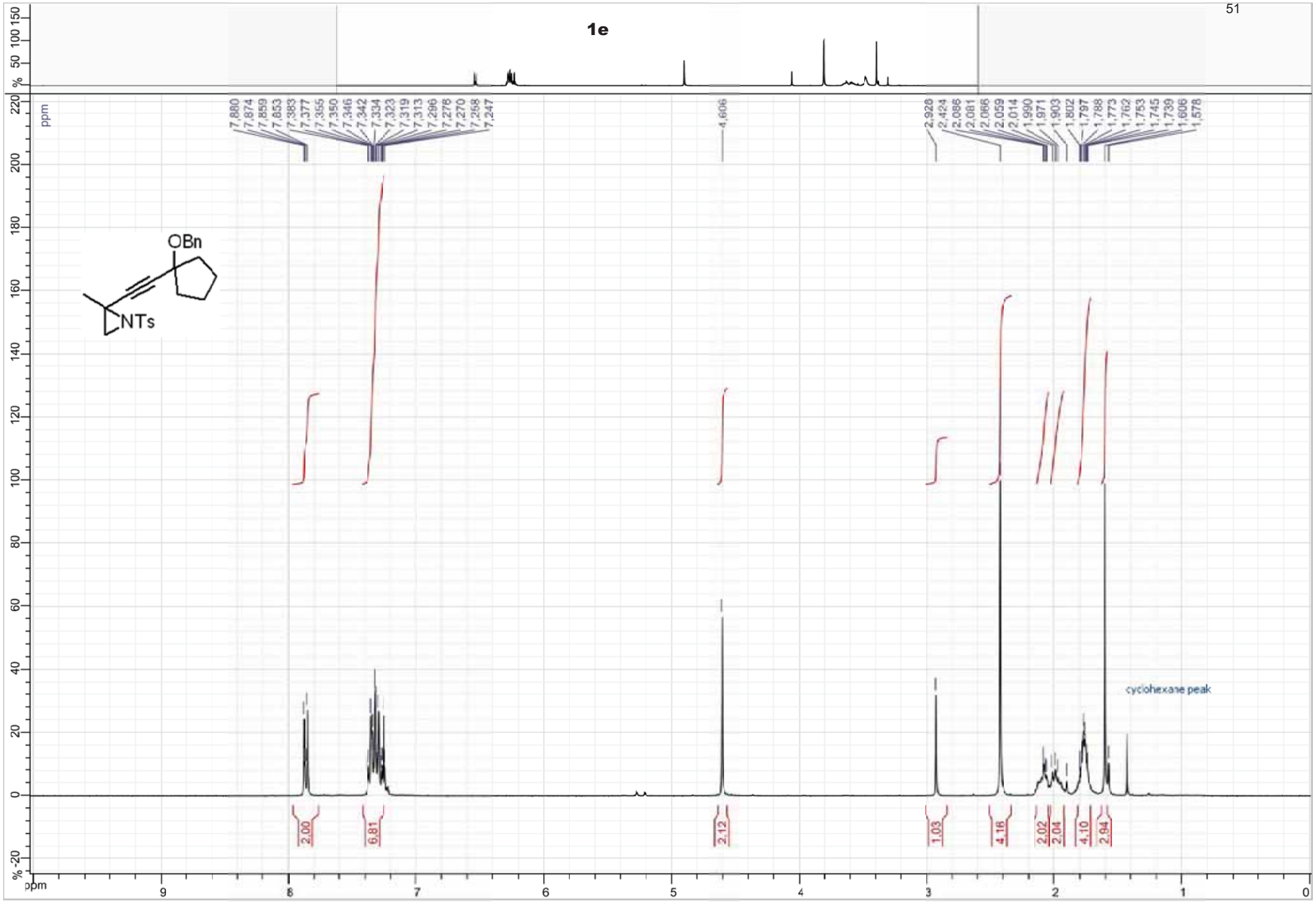

1c
144.239
138.092
136.802
129.555
128.314
128.149
127.796
127.574
84.319
82.355
77.457
77.243
77.038
76.611
70.571
68.574
41.827
38.138
35.492
31.720
28.951
25.212
23.996
22.599
21.645
14.094
OBn
C6H13
NTs
ppm

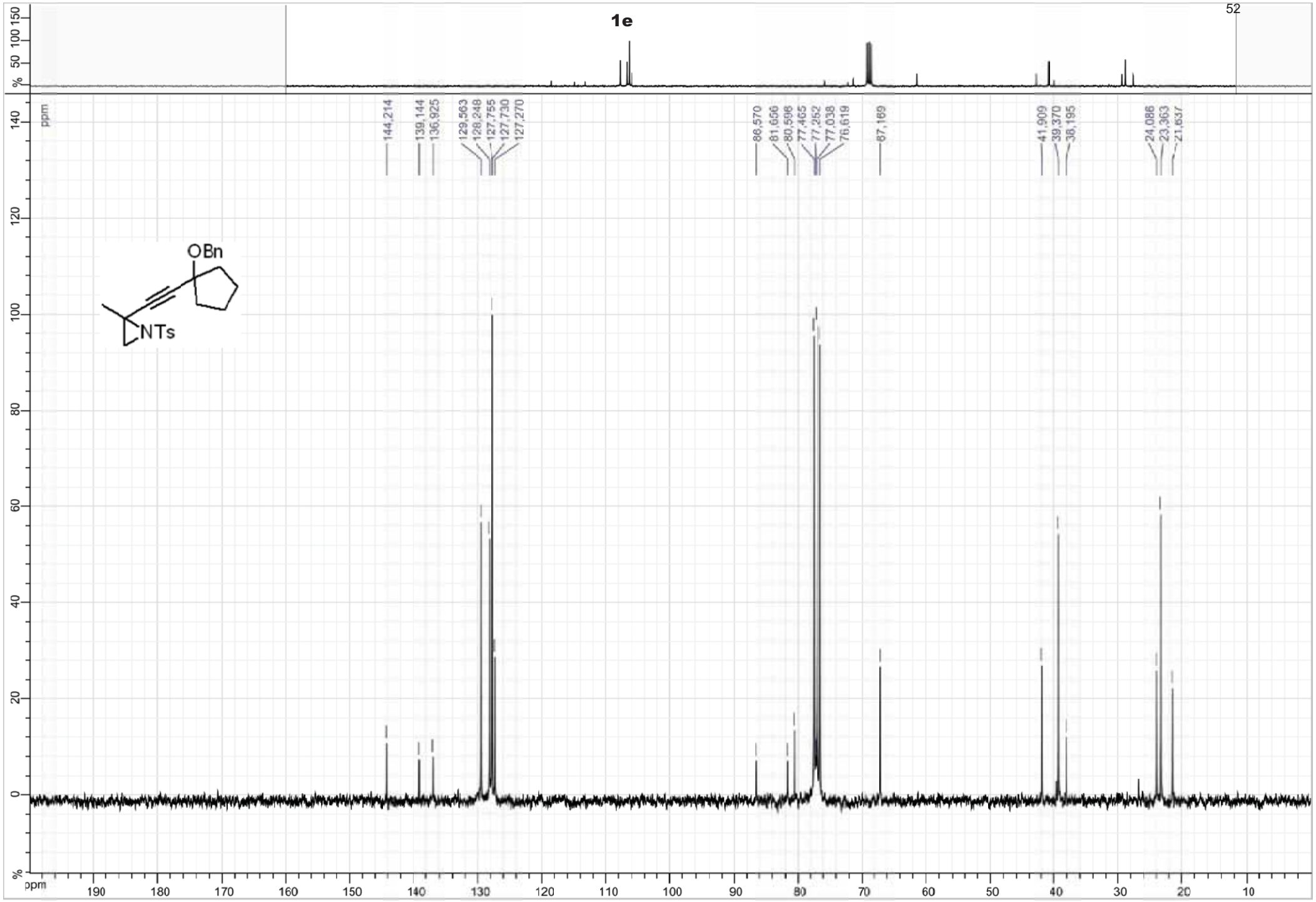
1d


7,882
7,878
7,861
7,854
7,405
7,399
7,377
7,373
7,356
7,352
7,329
7,324
7,309
7,304
7,298
7,283
7,277
7,271
7,261
4,654
2,918
2,426
1,612
1,562
1,558
1,550
Me
Me
O
N–Ts
2,00
8,97
1,96
1,01
3,94
3,05
ppm

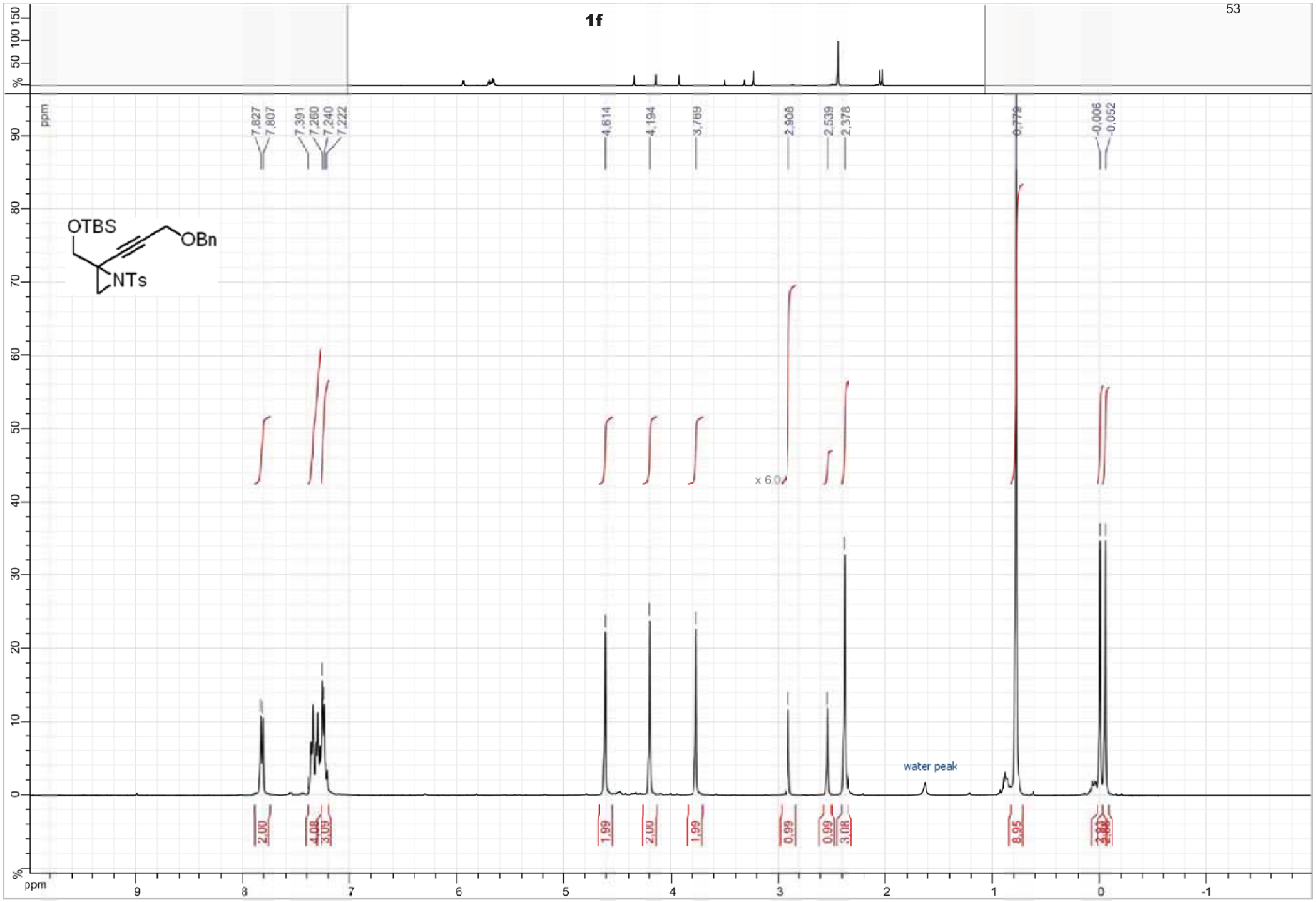
1d

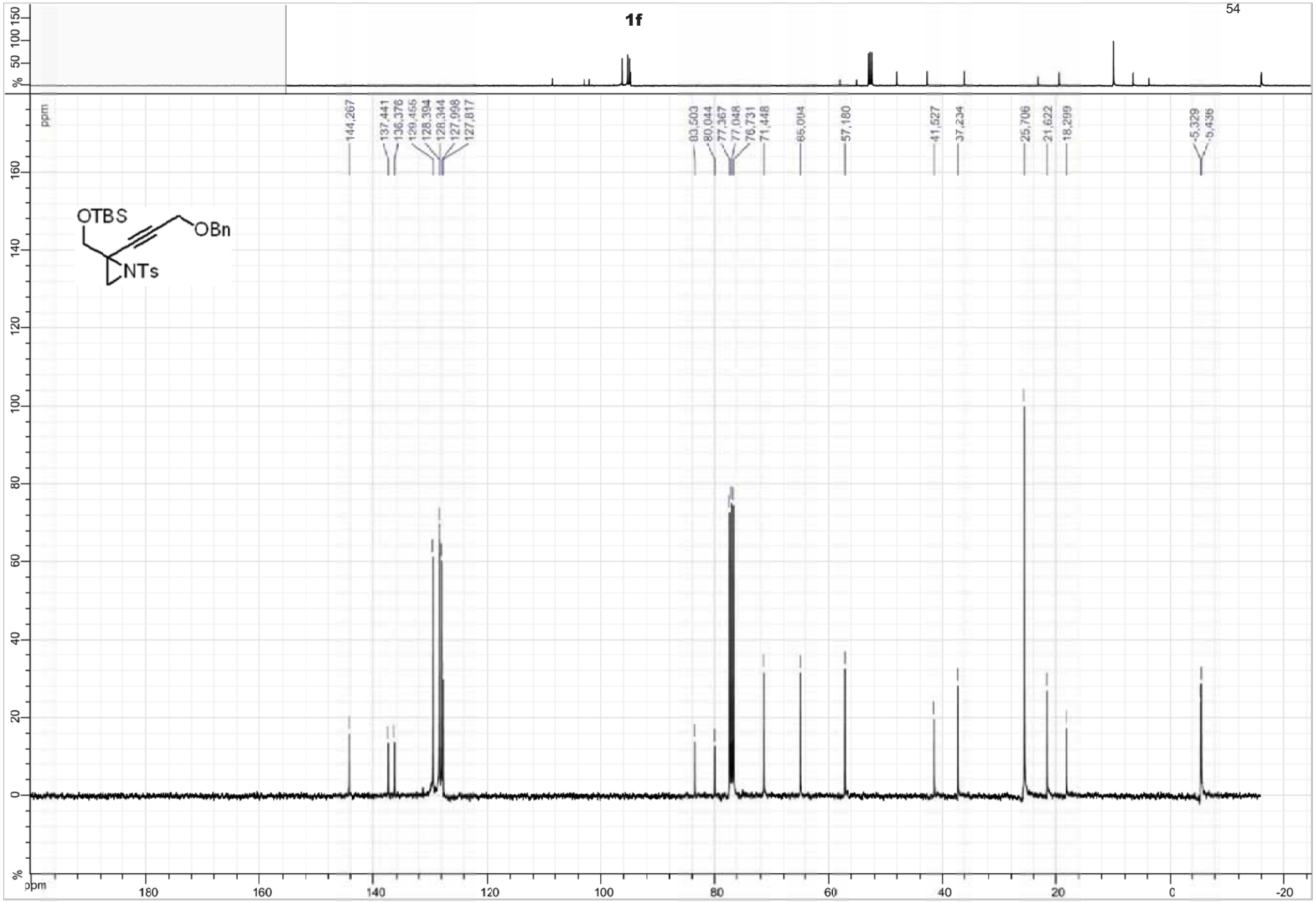

144,286
139,215
136,980
129,624
128,309
127,857
127,767
127,331
87,045
81,169
77,545
77,128
76,690
70,682
66,721
41,943
38,113
28,703
24,044
21,685
Me
Me
O
N–Ts
ppm


1e
OBn
NTs
cyclohexane peak
7,880
7,874
7,859
7,853
7,383
7,377
7,355
7,350
7,346
7,342
7,334
7,323
7,319
7,313
7,296
7,278
7,270
7,258
7,247
4,606
2,928
2,424
2,086
2,081
2,066
2,059
2,014
1,990
1,971
1,903
1,802
1,797
1,788
1,773
1,762
1,753
1,745
1,739
1,606
1,578
2,00
6,81
2,12
1,03
4,16
2,02
2,04
4,10
2,04
ppm


1e
52
144,214
139,144
136,925
129,563
128,248
127,755
127,730
127,270
86,570
81,656
80,598
77,465
77,252
77,038
76,619
67,169
41,909
39,370
38,195
24,086
23,363
21,637
OBn
NTs
ppm

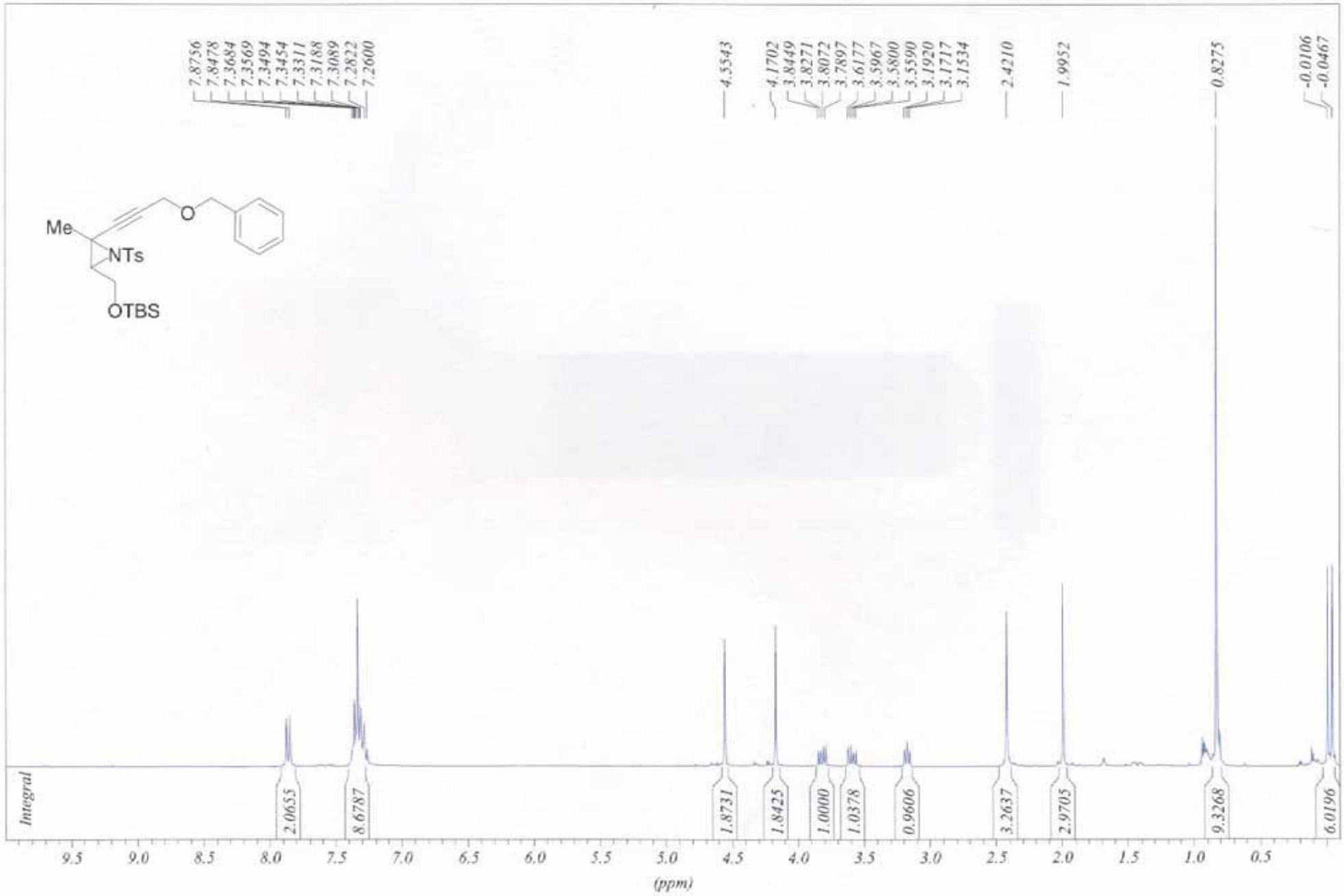
1f


OTBS
OBn
NTs
7,827
7,807
7,391
7,260
7,240
7,222
4,614
4,194
3,769
2,906
2,539
2,378
0,779
-0,006
-0,052
x 6.0
water peak
2,00
4,08
3,09
1,99
2,00
1,99
0,99
0,99
3,08
8,95
ppm


1f
144,267
137,441
136,376
129,465
128,394
128,344
127,998
127,817
83,503
80,044
77,367
77,048
76,731
71,448
66,094
57,180
41,527
37,234
25,706
21,622
18,299
-5,329
-5,436
OTBS
OBn
NTs
ppm

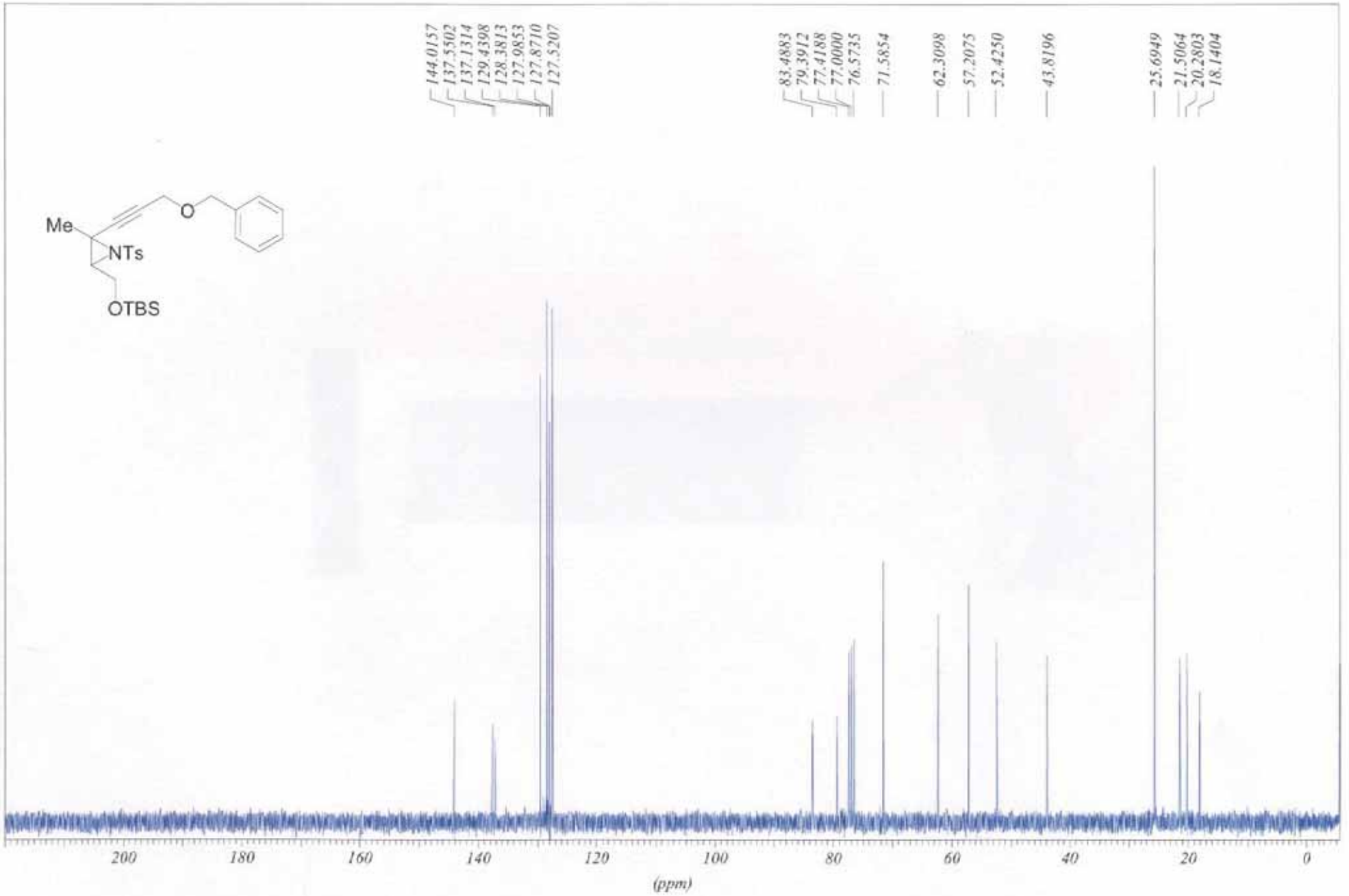
1g

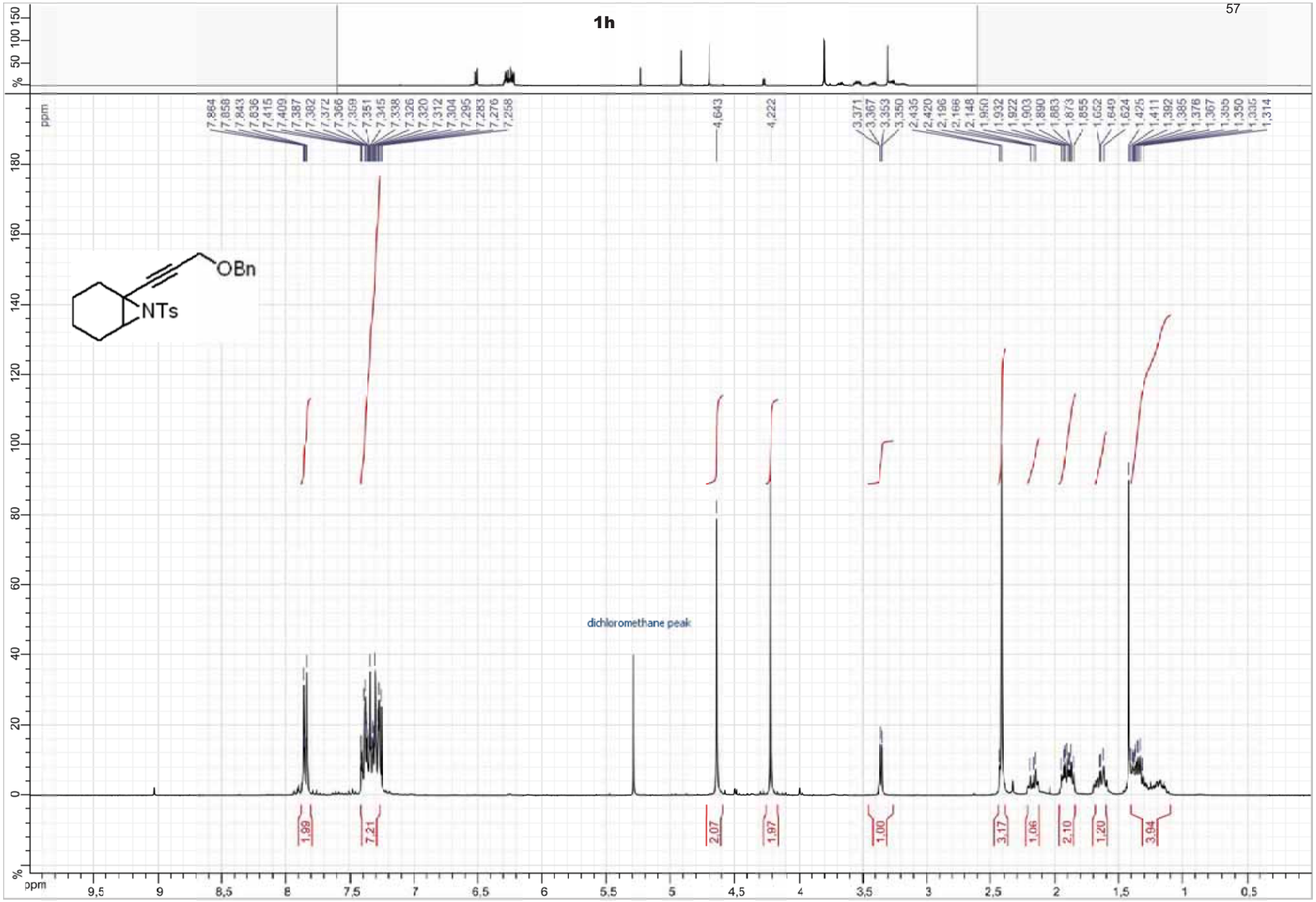
Proton CDCl3 300 MHz



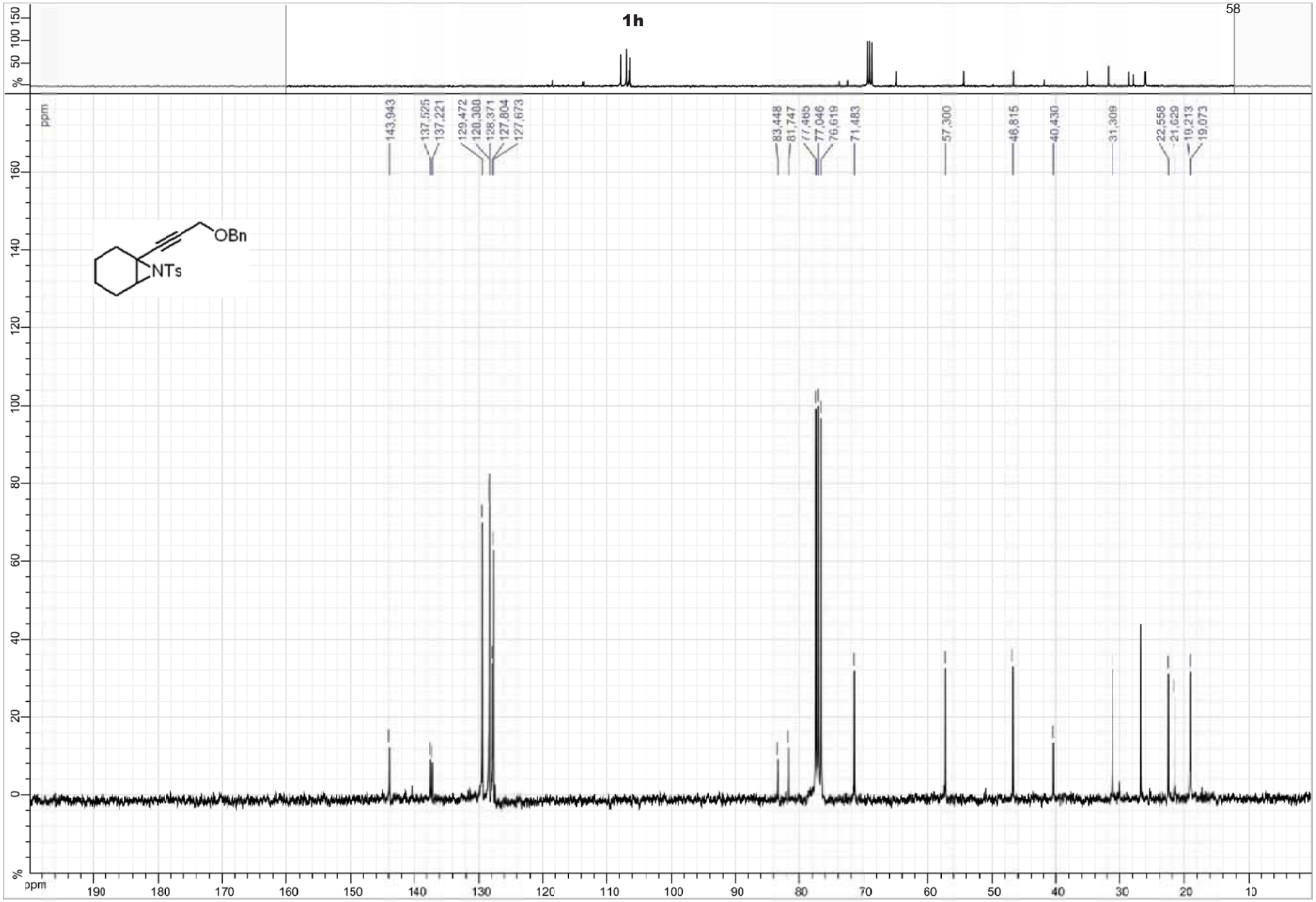
1g

Carbon CDCl3 300 MHz

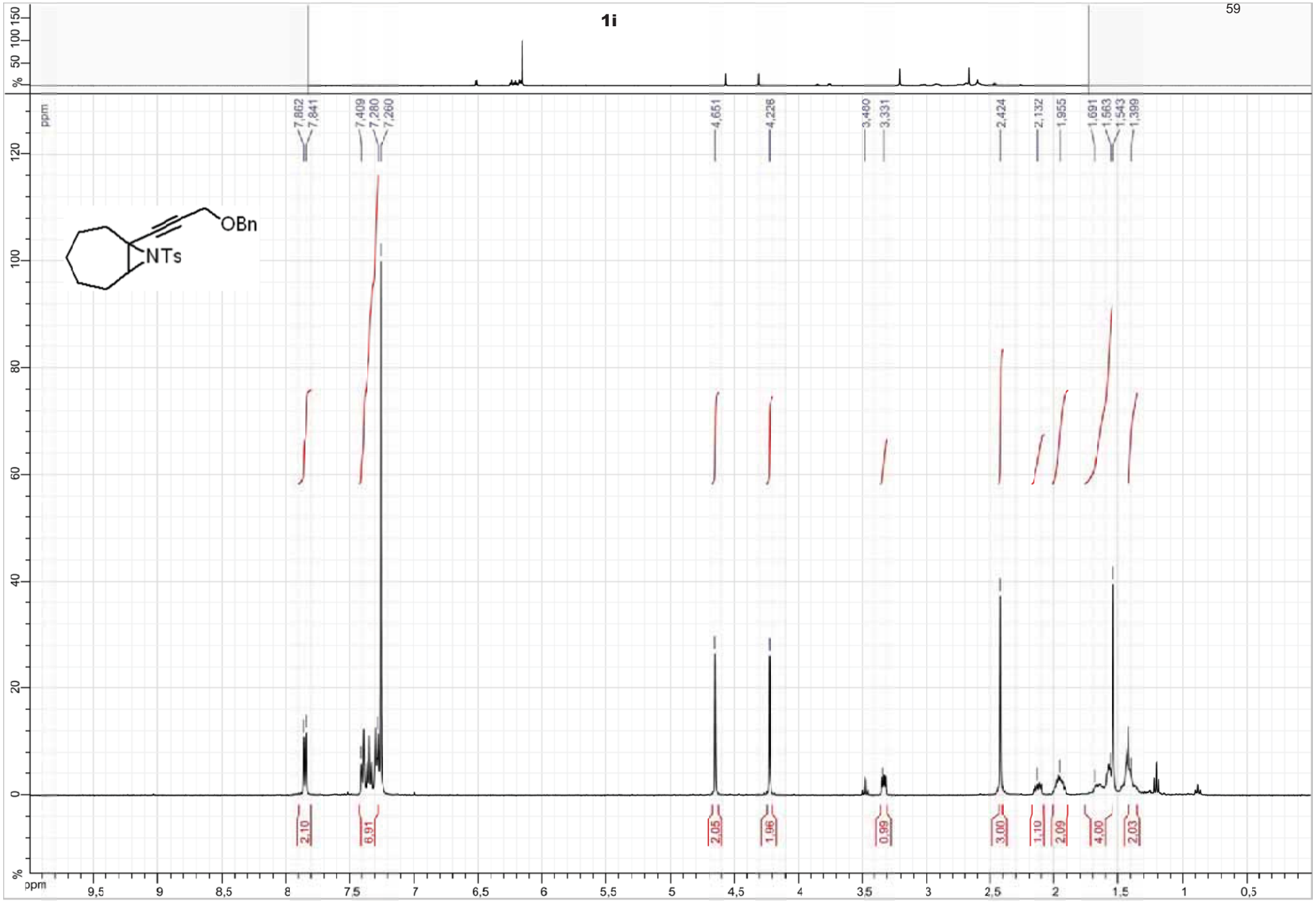



1h
OBn
NTs
dichloromethane peak
7,864
7,858
7,843
7,836
7,415
7,409
7,387
7,382
7,372
7,366
7,359
7,351
7,345
7,338
7,326
7,320
7,312
7,304
7,295
7,283
7,276
7,258
4,643
4,222
3,371
3,367
3,353
3,350
2,435
2,420
2,196
2,166
2,148
1,950
1,932
1,922
1,903
1,890
1,883
1,873
1,855
1,652
1,649
1,624
1,425
1,411
1,392
1,385
1,376
1,367
1,355
1,350
1,335
1,314
1,99
7,21
2,07
1,97
1,00
3,17
1,06
2,10
1,20
3,94
ppm

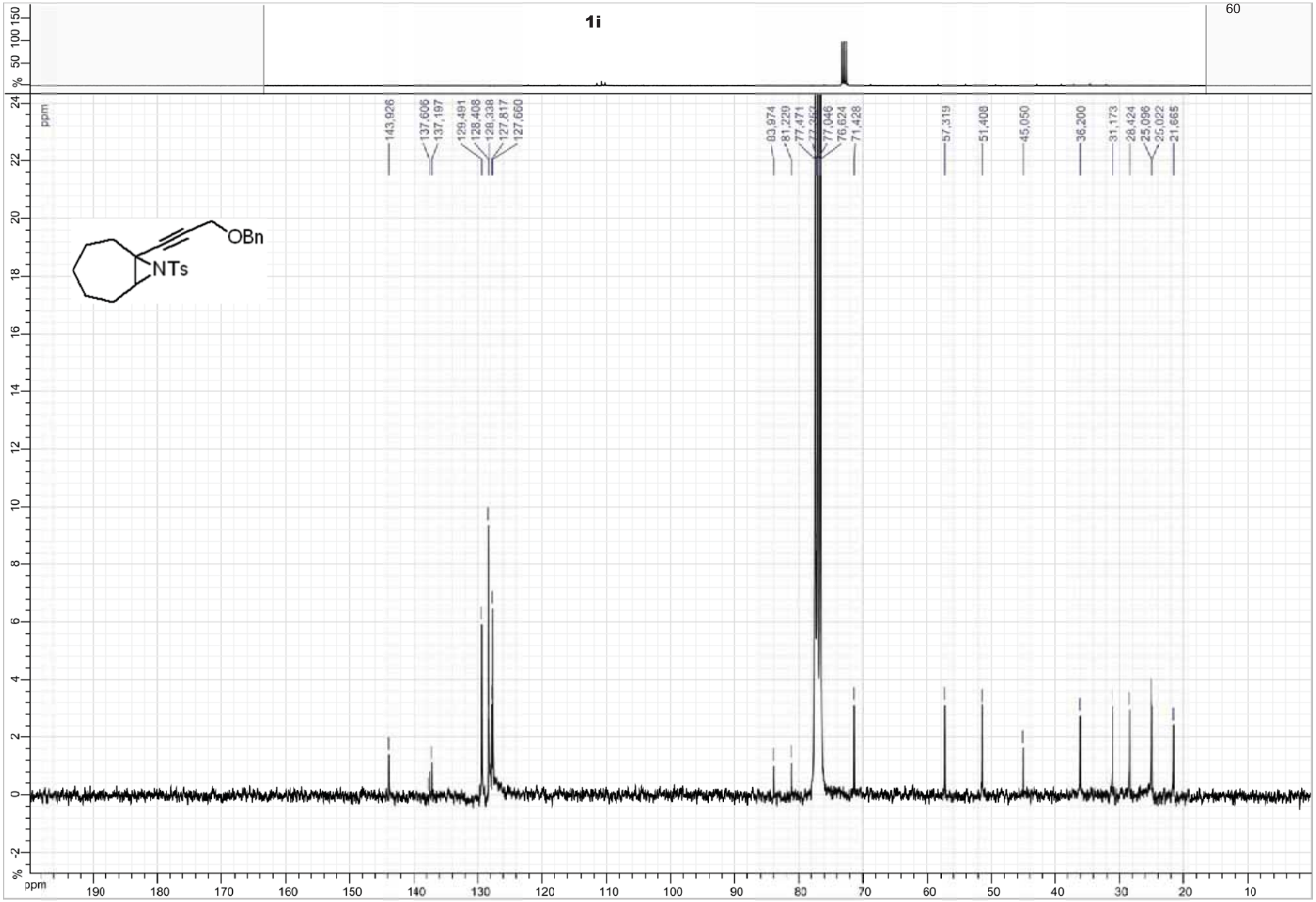
1h

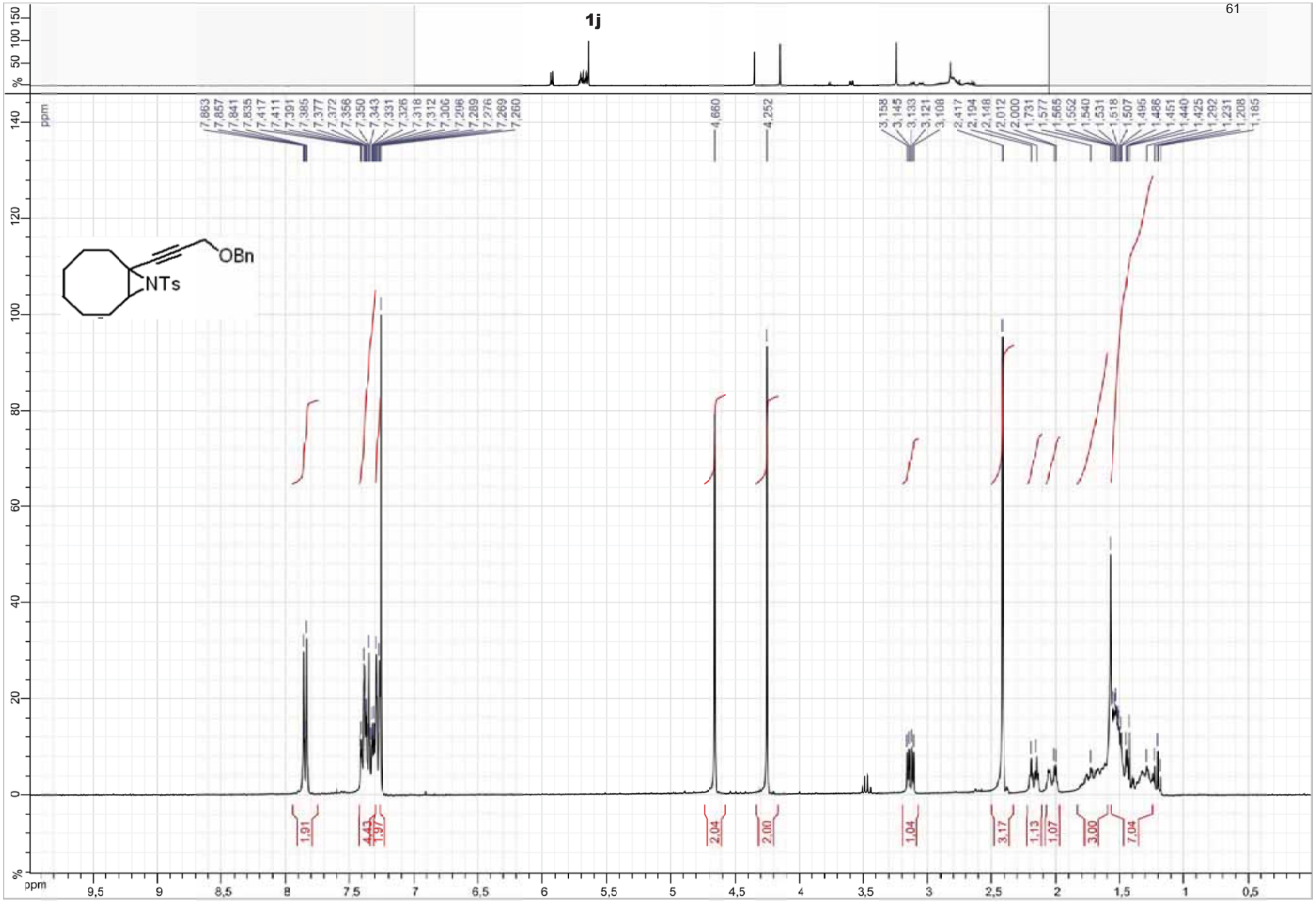

143,943
137,525
137,221
129,472
128,388
128,371
127,804
127,673
83,448
81,747
77,465
77,046
76,619
71,483
57,300
46,815
40,430
31,309
22,558
21,629
19,213
19,073
OBn
NTs
ppm

1i

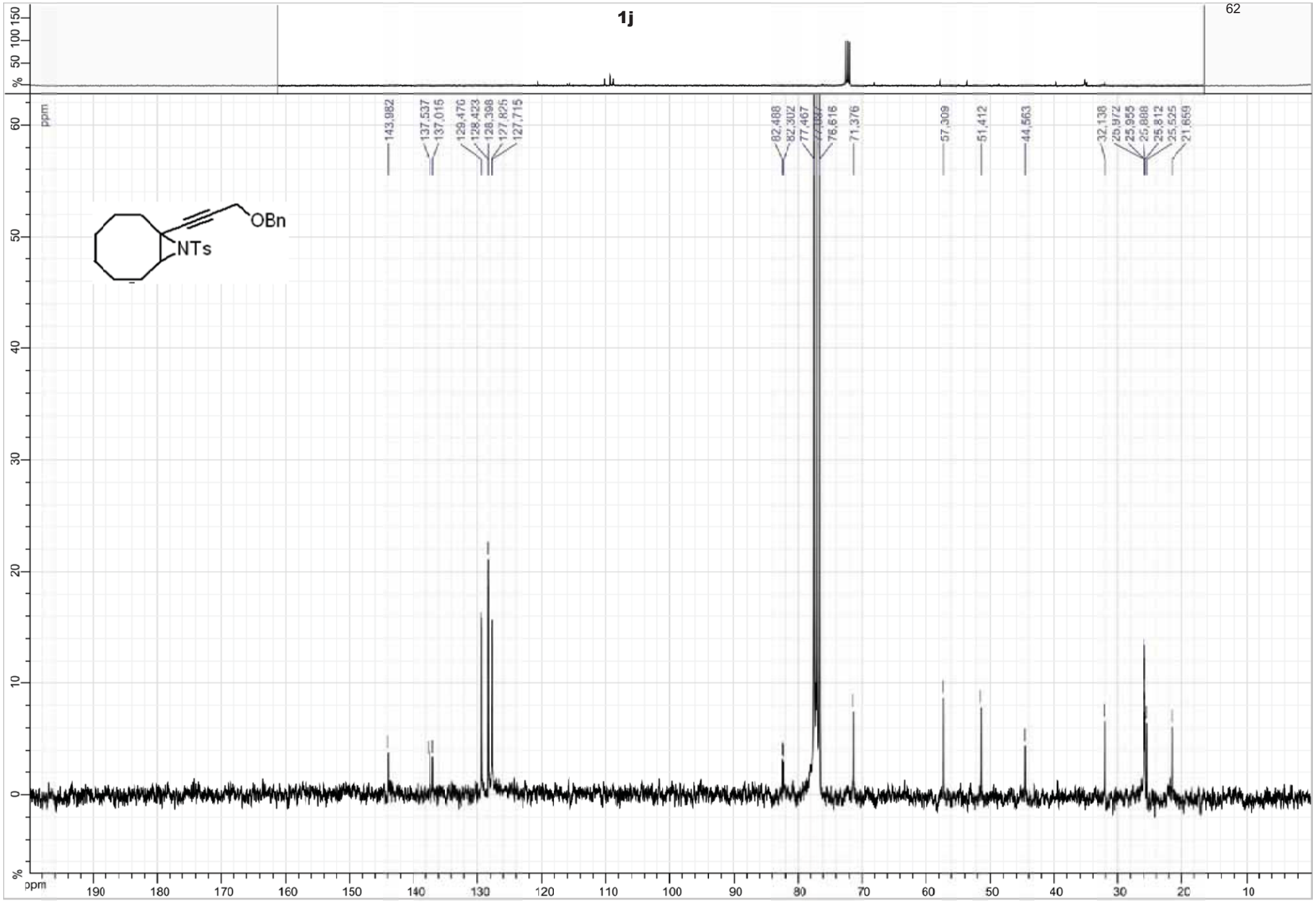

7,862
7,841
7,409
7,280
7,260
4,651
4,226
3,480
3,331
2,424
2,132
1,955
1,691
1,563
1,543
1,399
OBn
NTs
2,10
6,91
2,05
1,96
0,99
3,00
1,10
2,09
4,00
2,03
ppm

1i

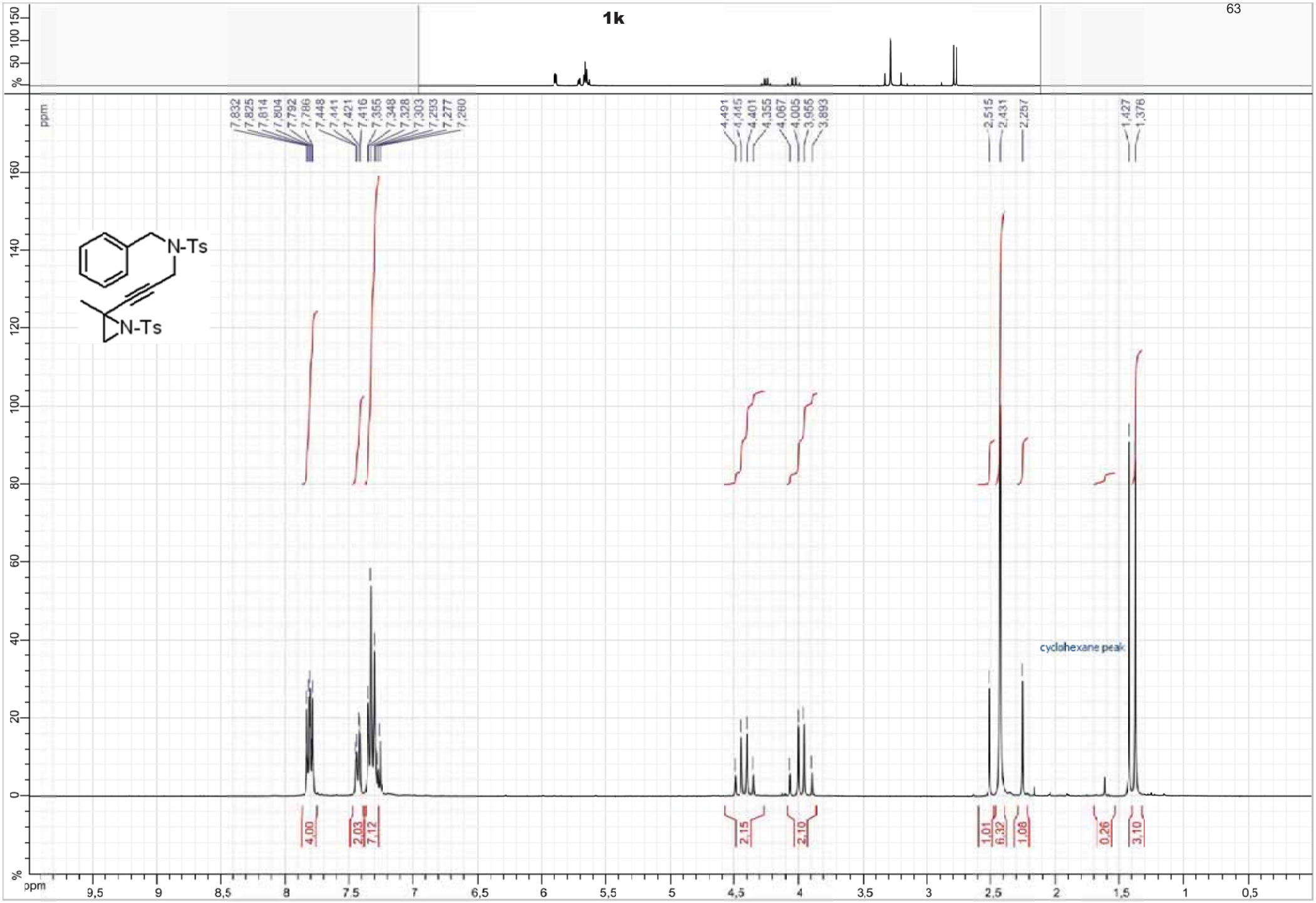

143,926
137,606
137,197
129,491
128,408
128,338
127,817
127,660
83,974
81,229
77,471
77,046
76,624
71,428
57,319
51,408
45,050
36,200
31,173
28,424
25,096
25,022
21,665
OBn
NTs
ppm


1j
OBn
NTs
7,863
7,857
7,841
7,835
7,417
7,411
7,391
7,385
7,377
7,372
7,358
7,350
7,343
7,331
7,326
7,318
7,312
7,306
7,296
7,289
7,276
7,269
7,260
4,660
4,252
3,158
3,145
3,133
3,121
3,108
2,417
2,194
2,148
2,012
2,000
1,731
1,577
1,565
1,552
1,540
1,531
1,518
1,507
1,495
1,486
1,451
1,440
1,425
1,292
1,231
1,208
1,185
1,91
4,43
1,97
2,04
2,00
1,04
3,17
1,13
1,07
3,00
7,04
ppm

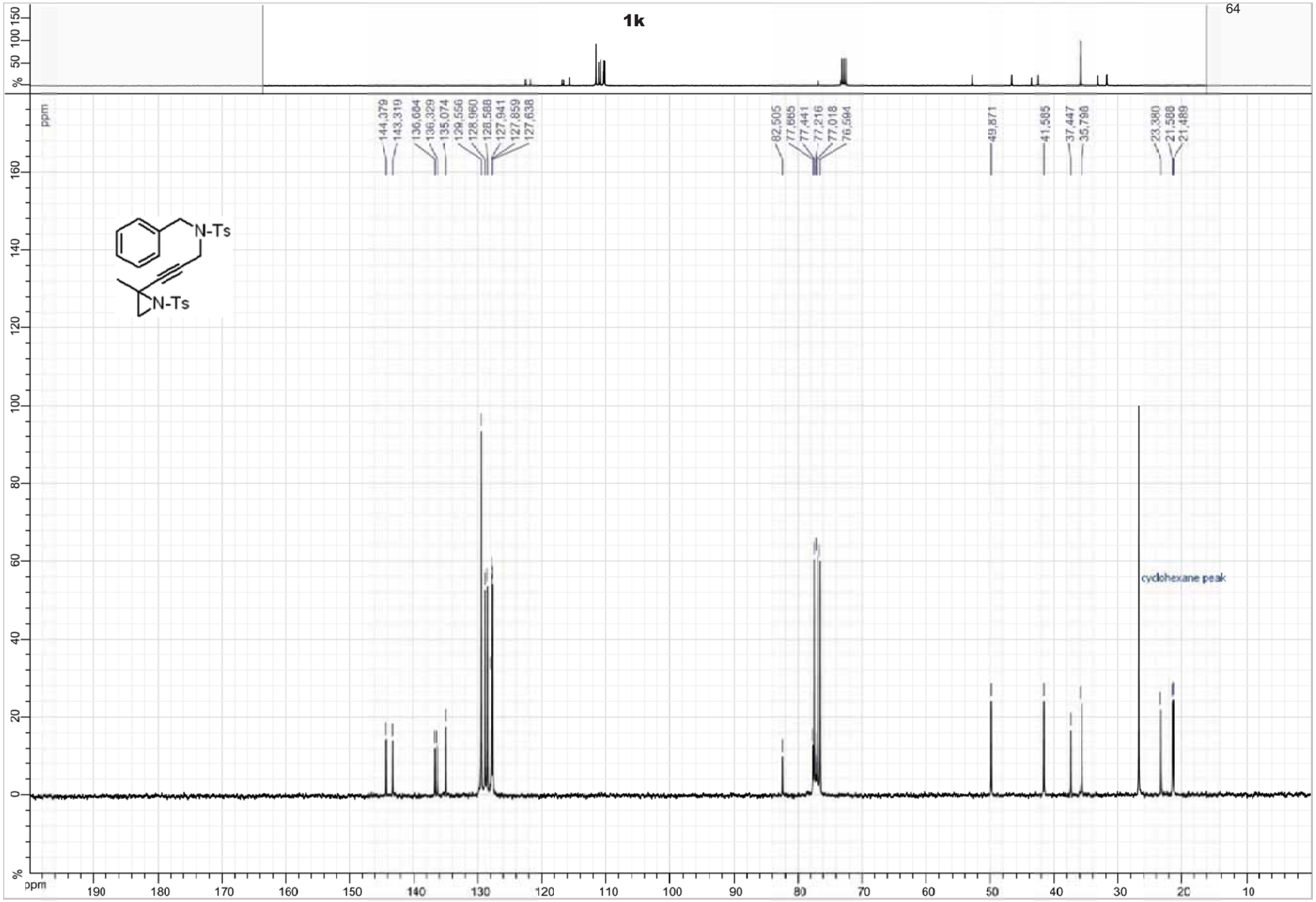
1j


143,982
137,537
137,015
129,476
128,423
128,398
127,825
127,715
82,488
82,302
77,467
76,616
71,376
57,309
51,412
44,563
32,138
26,972
25,955
25,888
25,812
25,525
21,659
OBn
NTs
ppm

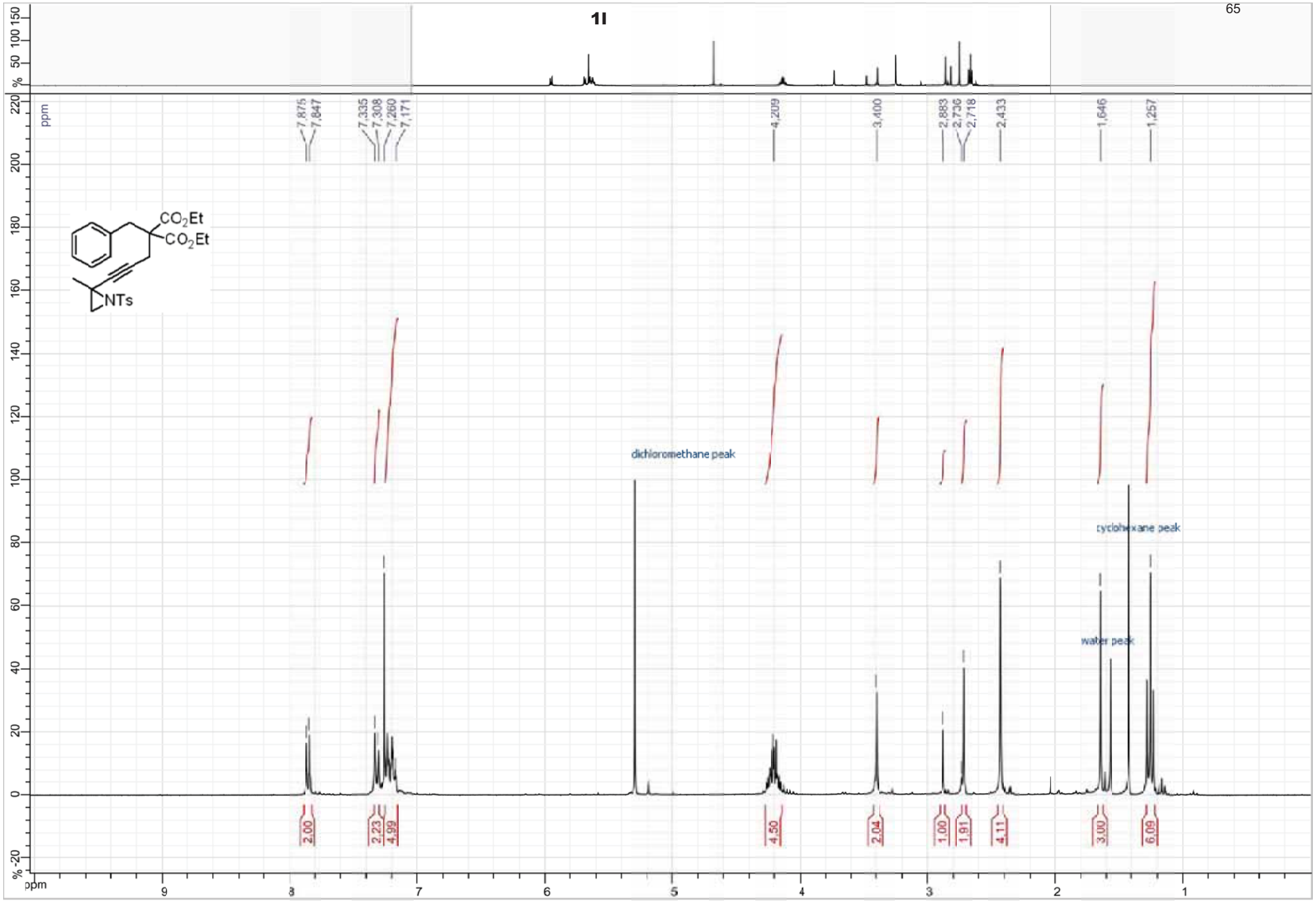
1k


7,832
7,825
7,814
7,804
7,792
7,786
7,448
7,441
7,421
7,416
7,355
7,348
7,328
7,303
7,293
7,277
7,260
4,491
4,445
4,401
4,355
4,067
4,005
3,955
3,893
2,515
2,431
2,257
1,427
1,376
N-Ts
N-Ts
cyclohexane peak
4,00
2,03
7,12
2,15
2,10
1,01
6,32
1,06
0,26
3,10
ppm

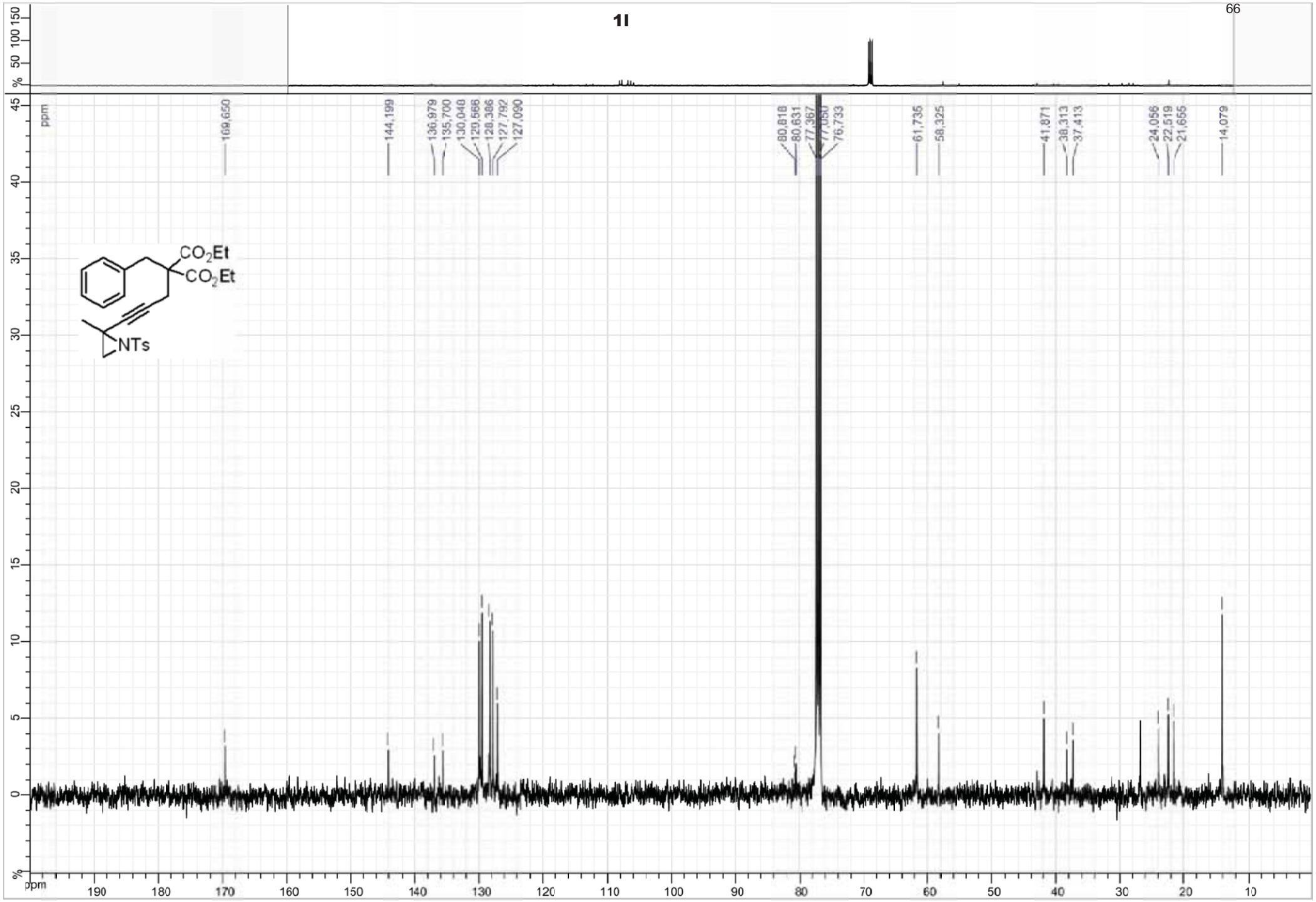
1k

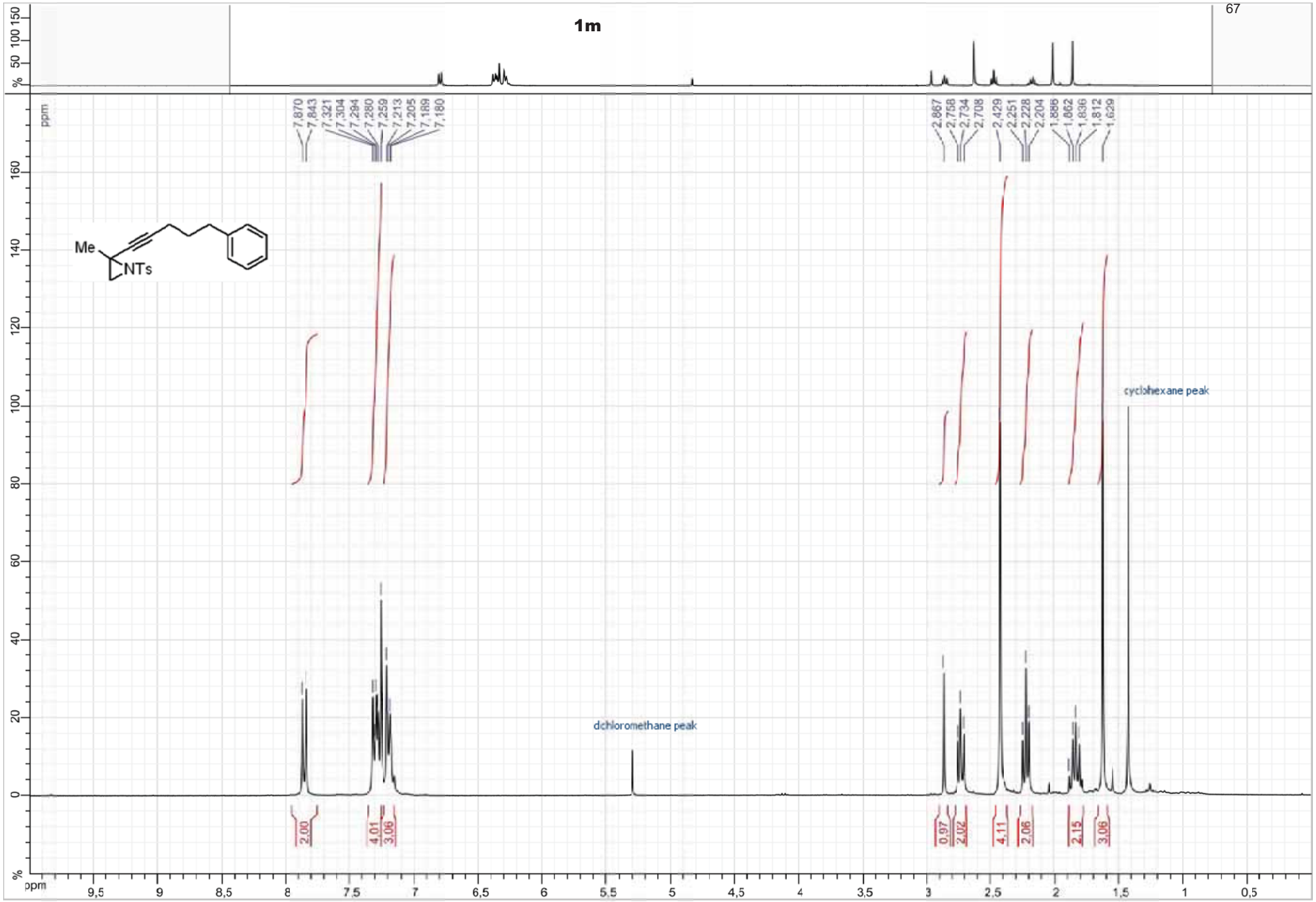

144,379
143,319
136,684
136,329
135,074
129,556
128,960
128,588
127,941
127,859
127,638
82,505
77,665
77,441
77,216
77,018
76,594
49,871
41,585
37,447
35,796
23,380
21,588
21,489
N-Ts
N-Ts
cyclohexane peak
ppm

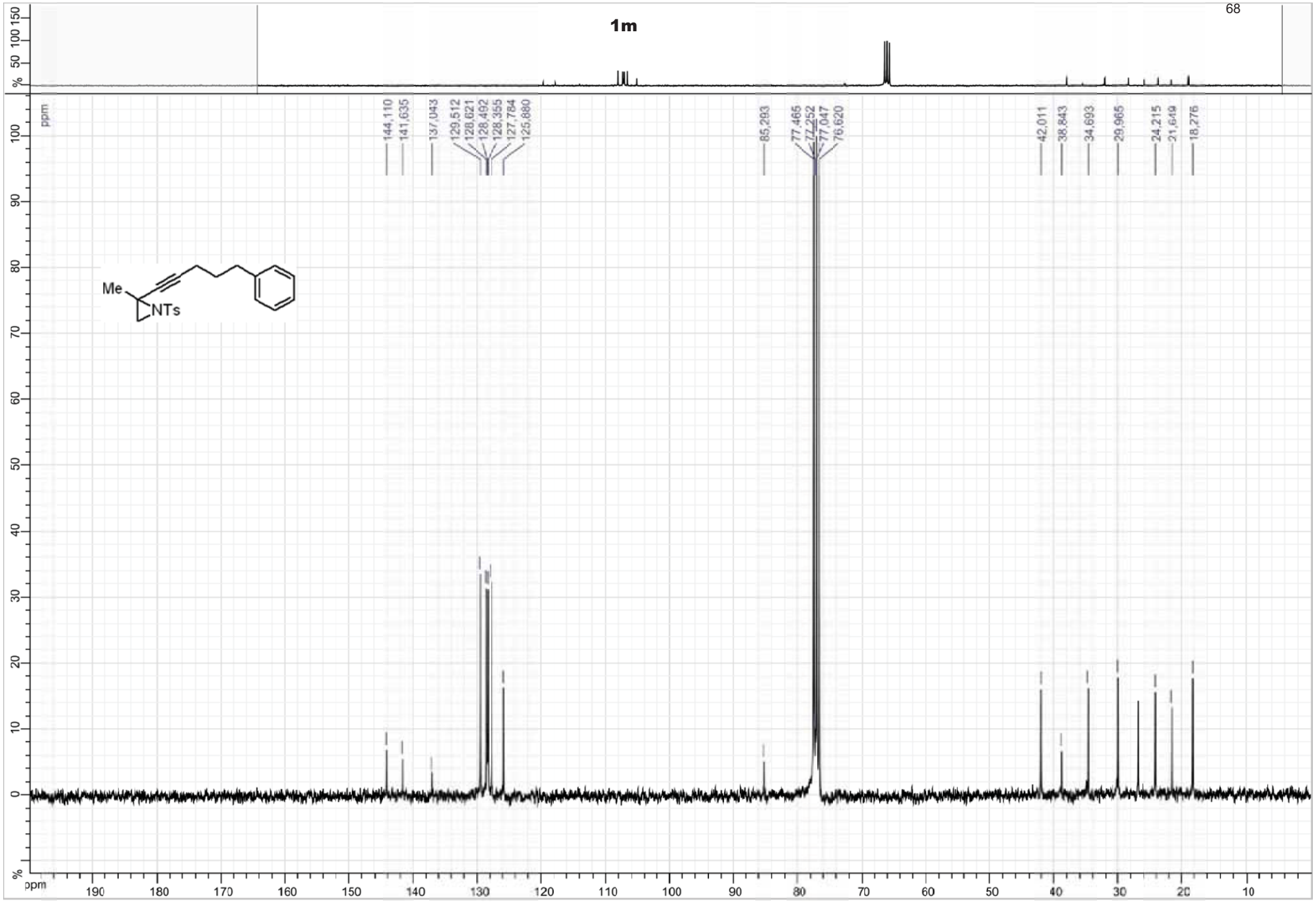
1l


ppm
7.875
7.847
7.335
7.308
7.260
7.171
4.209
3.400
2.883
2.736
2.718
2.433
1.646
1.257
CO2Et
CO2Et
NTs
dichloromethane peak
cyclohexane peak
water peak
2.00
2.23
4.99
4.50
2.04
1.00
1.91
4.11
3.00
6.09

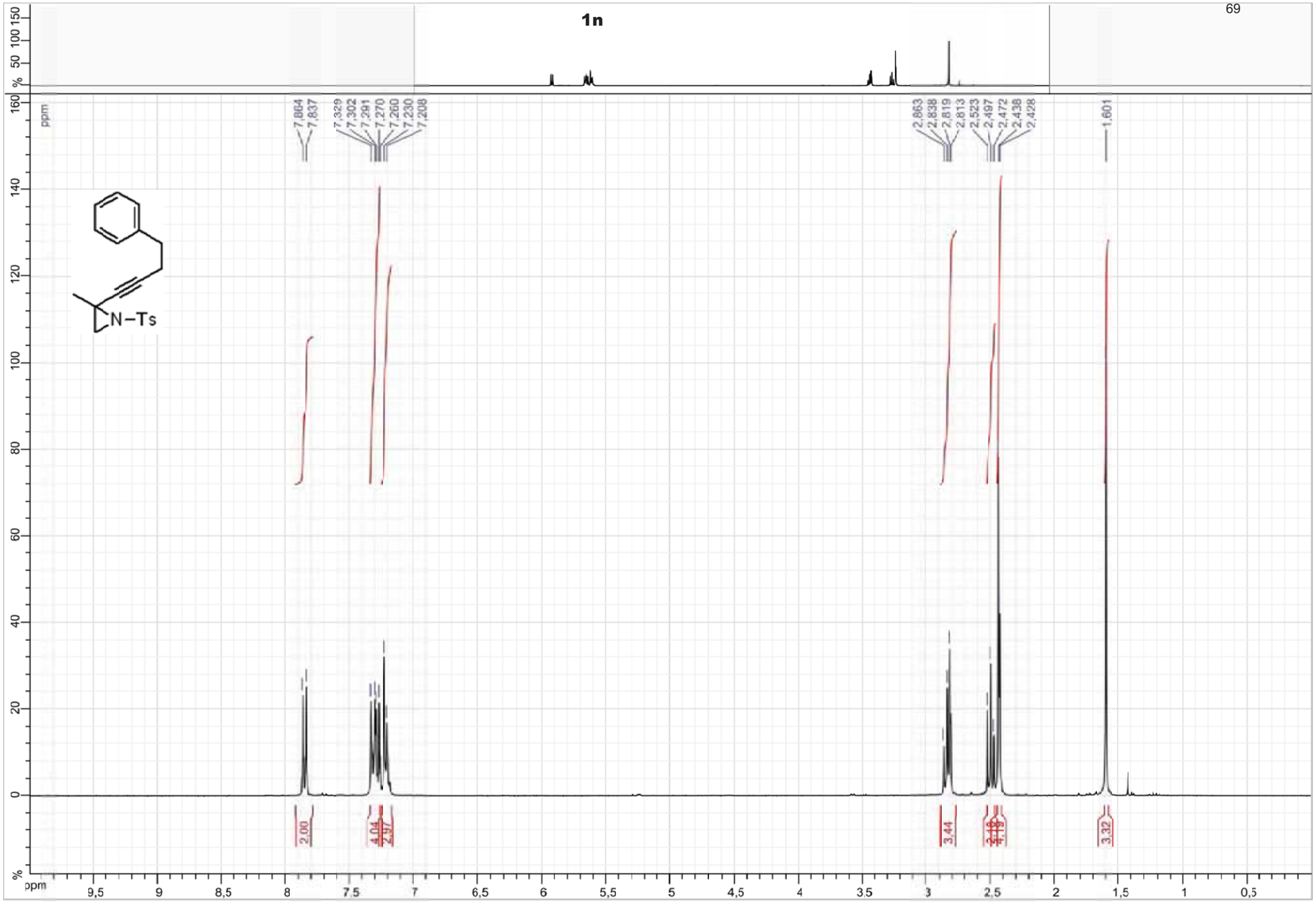
1l

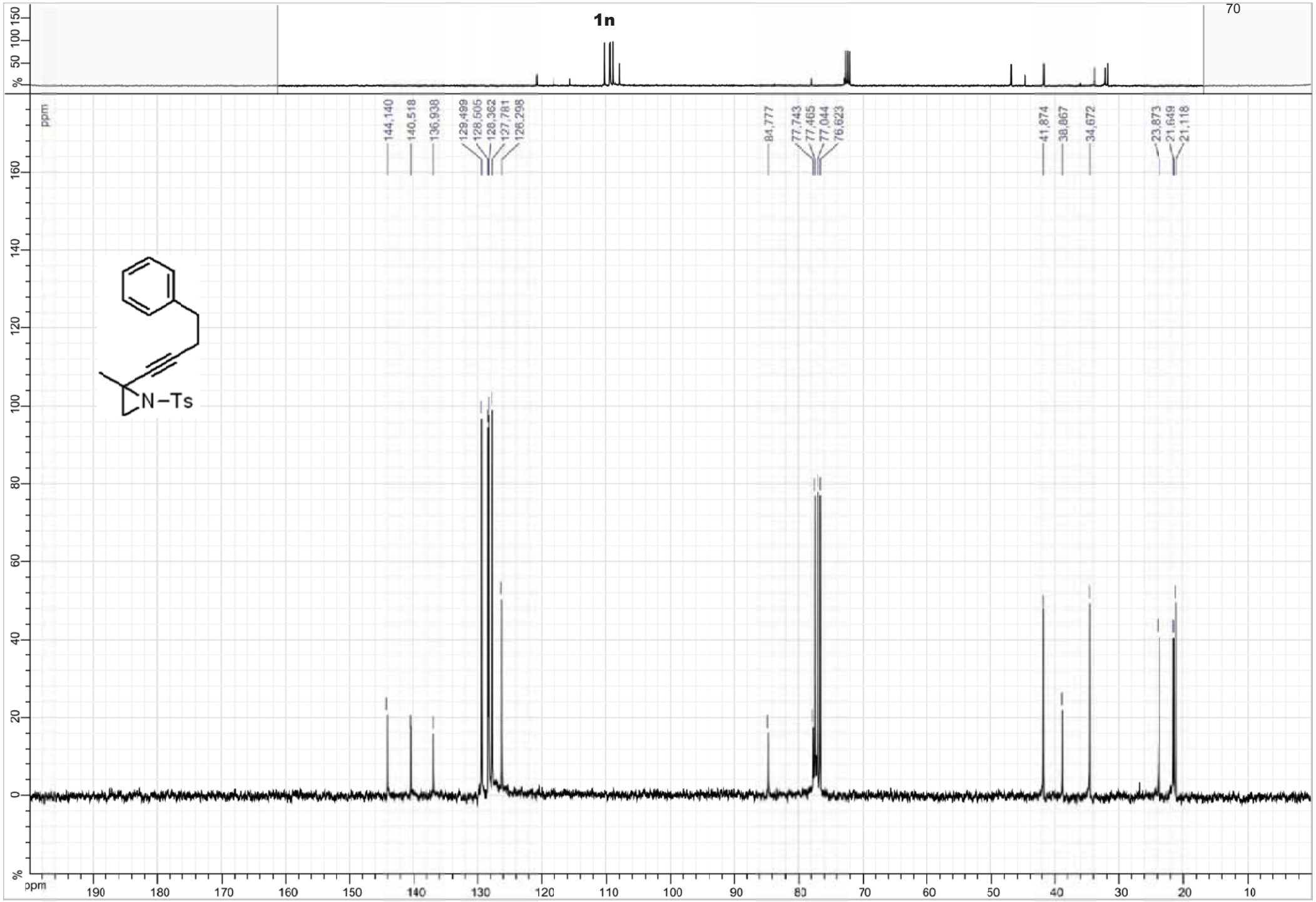

ppm
169,650
144,199
136,979
135,700
130,048
129,566
128,386
127,792
127,090
80,818
80,631
77,367
77,050
76,733
61,735
58,325
41,871
38,313
37,413
24,056
22,519
21,655
14,079
CO2Et
CO2Et
NTs


1m
ppm
7,870
7,843
7,321
7,304
7,294
7,280
7,259
7,213
7,205
7,189
7,180
2,867
2,758
2,734
2,708
2,429
2,251
2,228
2,204
1,886
1,862
1,836
1,812
1,629
Me
NTs
dichloromethane peak
cyclohexane peak
2,00
4,01
3,06
0,97
2,02
4,11
2,06
2,15
3,06
ppm

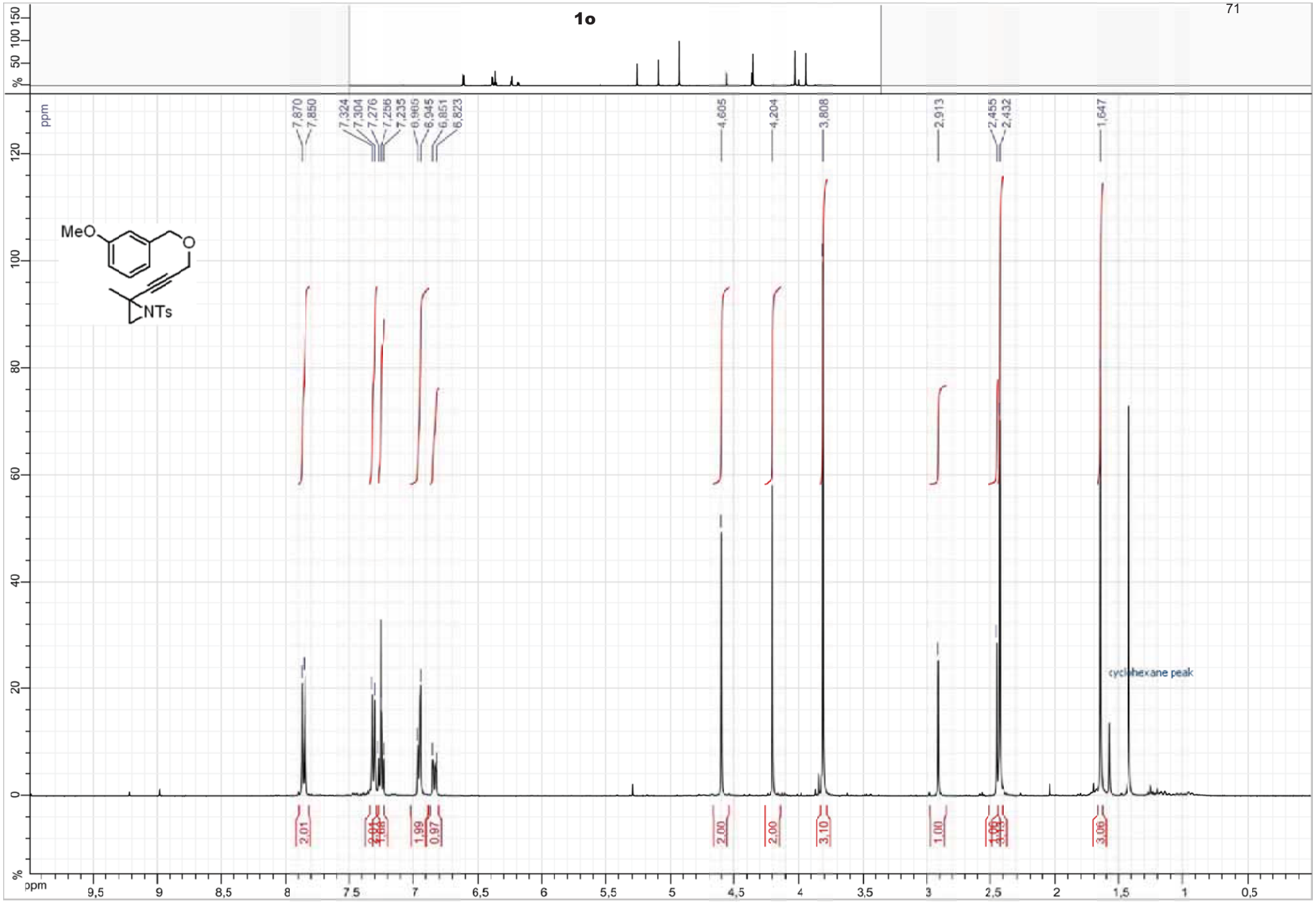

1m
144,110
141,635
137,043
129,512
128,621
128,492
128,355
127,784
125,880
85,293
77,465
77,252
77,047
76,620
42,011
38,843
34,693
29,965
24,215
21,649
18,276
Me
NTs
ppm

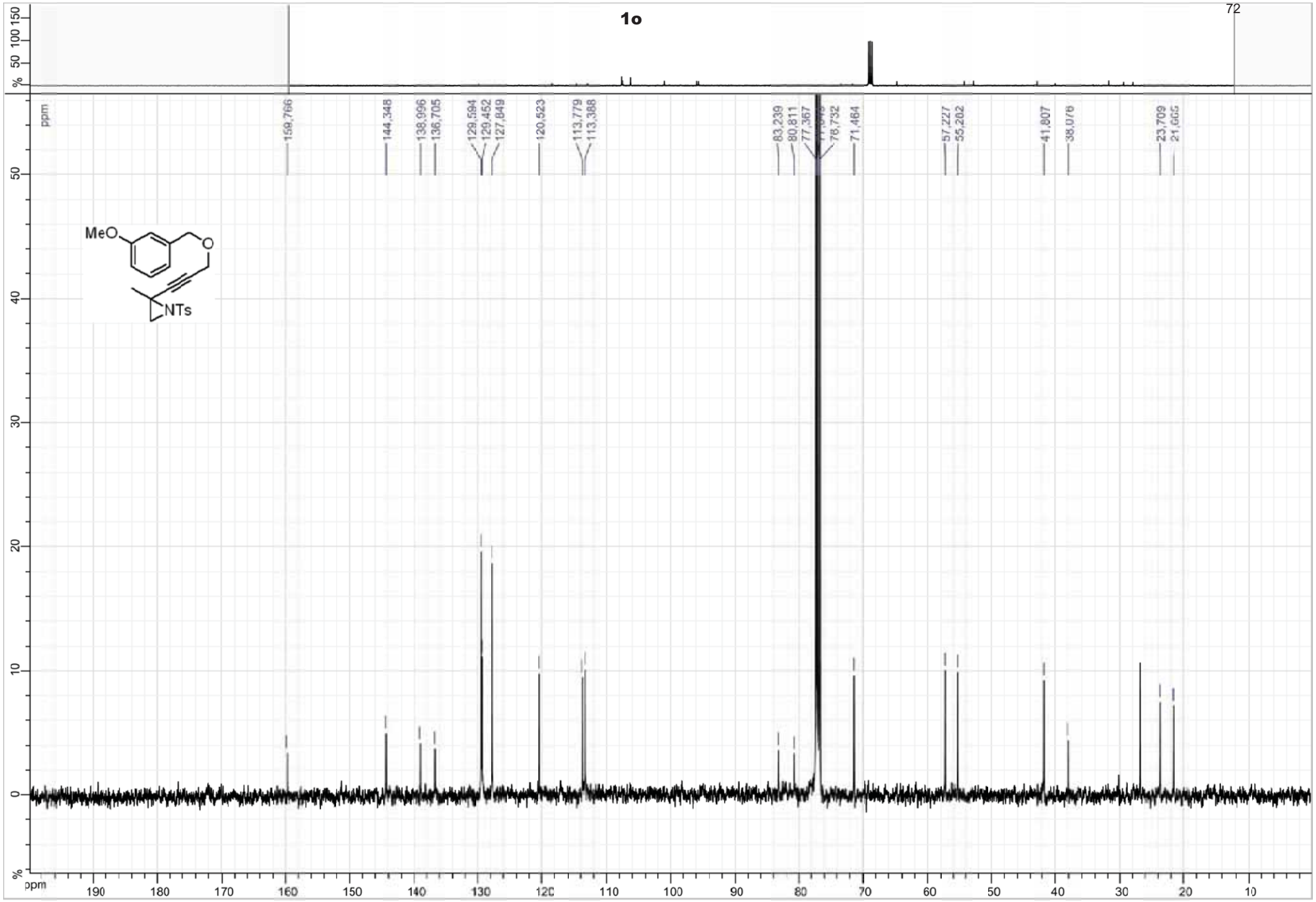
1n

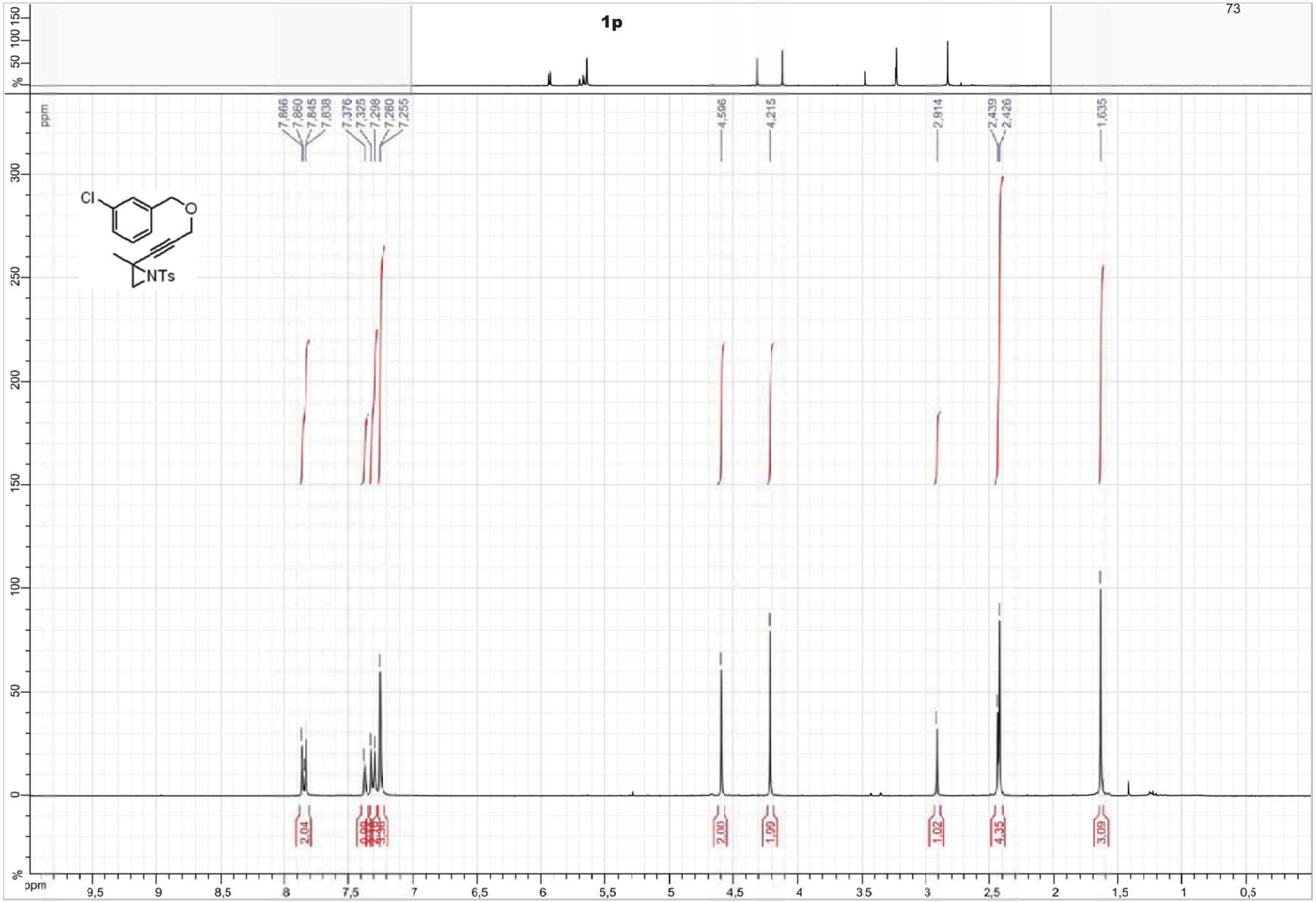

7,864
7,837
7,329
7,302
7,291
7,270
7,260
7,230
7,208
2,863
2,838
2,819
2,813
2,523
2,497
2,472
2,438
2,428
1,601
N–Ts
2,00
4,04
2,97
3,44
3,18
4,19
3,32
ppm

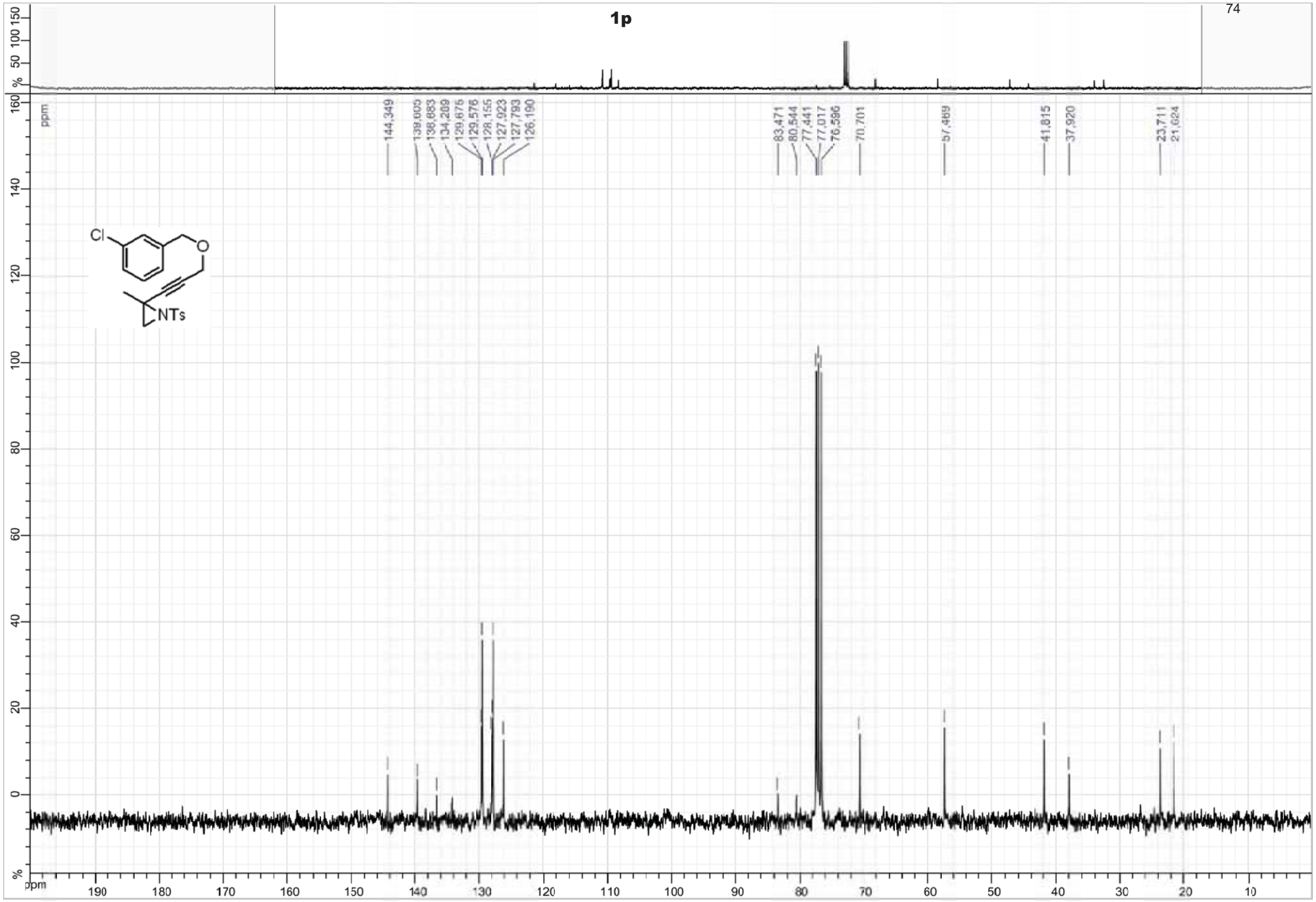

1n
144,140
140,518
136,938
129,499
128,505
128,362
127,781
126,298
84,777
77,743
77,465
77,044
76,623
41,874
38,867
34,672
23,873
21,649
21,118
N–Ts
ppm

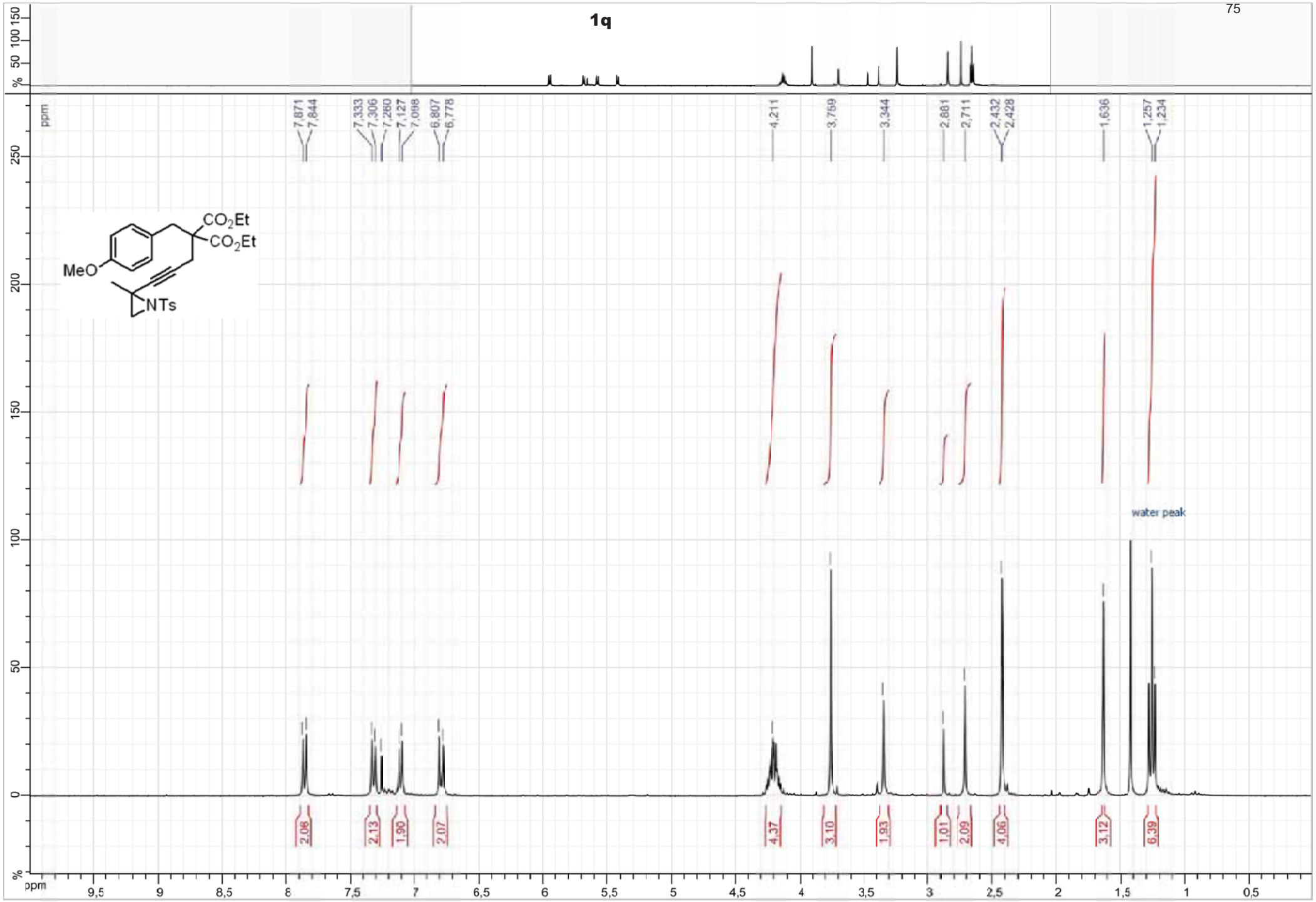
1o

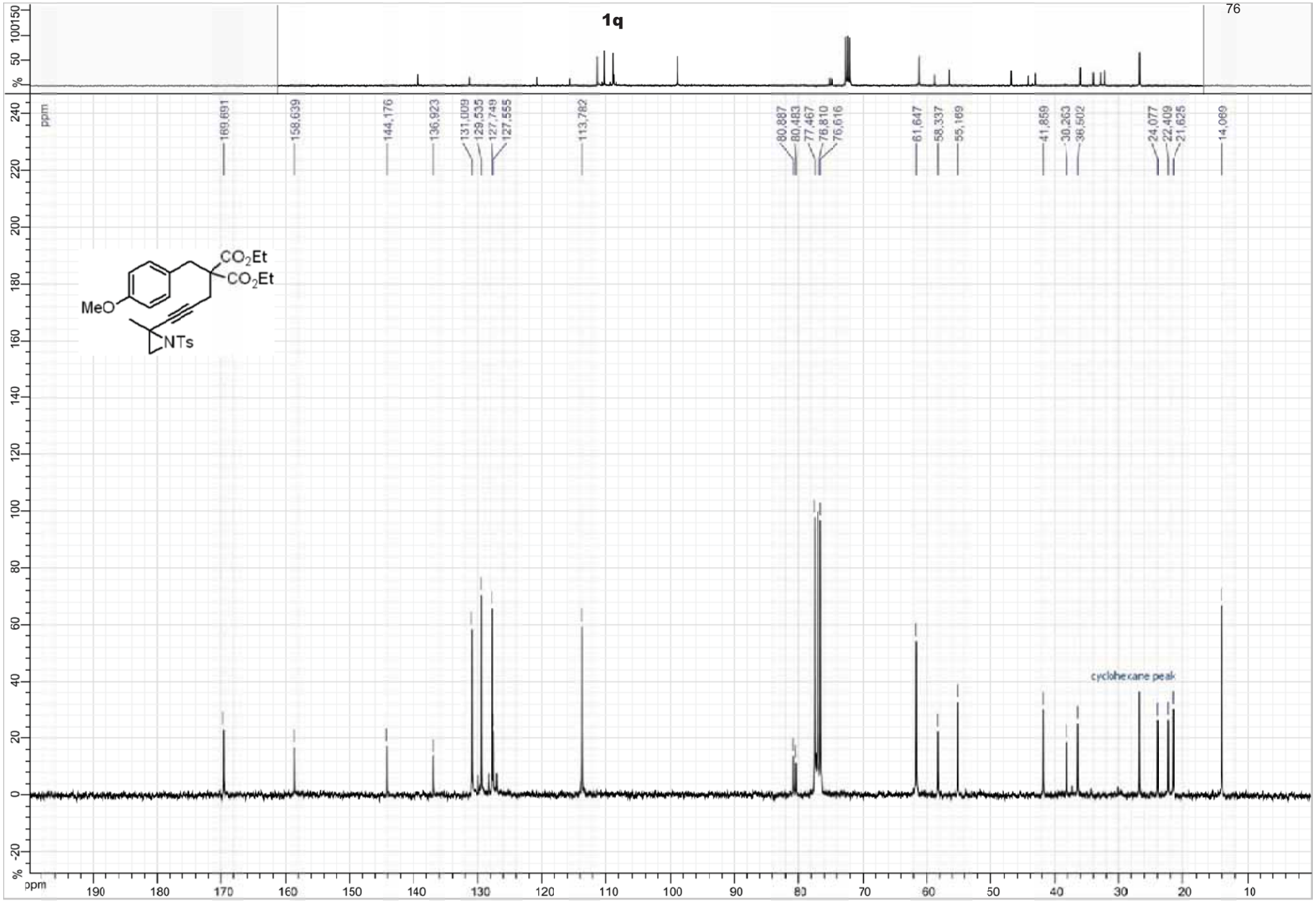

MeO
O
NTs
ppm
7,870
7,850
7,324
7,304
7,276
7,256
7,235
6,965
6,945
6,851
6,823
4,605
4,204
3,808
2,913
2,455
2,432
1,647
cyclohexane peak
2,01
1,88
1,99
0,97
2,00
2,00
3,10
1,00
3,06


1o
72
159,766
144,348
138,996
136,705
129,594
129,452
127,849
120,523
113,779
113,388
83,239
80,811
77,367
78,732
71,464
57,227
55,262
41,807
38,076
23,709
21,665
MeO
O
NTs
ppm

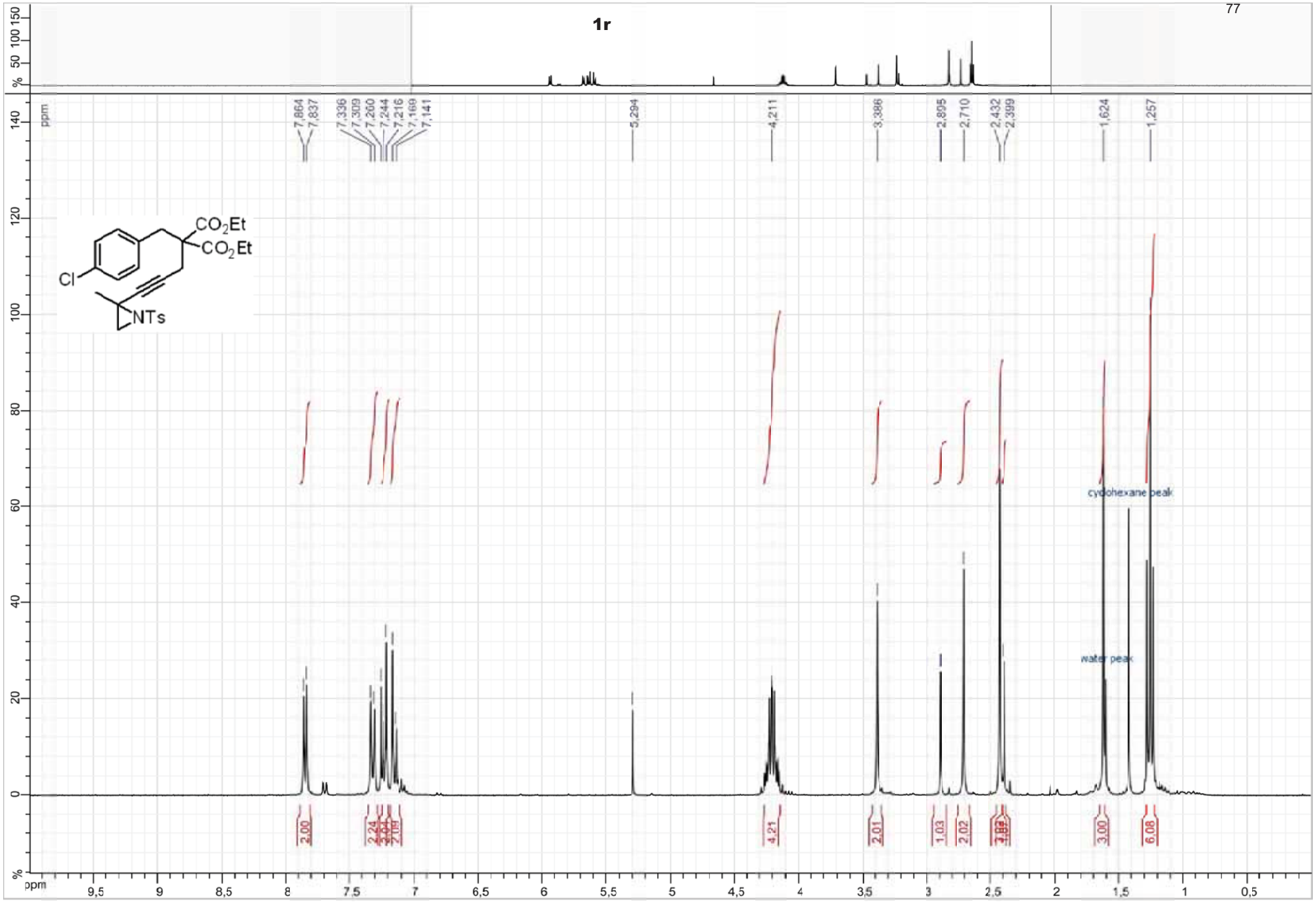

1p
ppm
7,866
7,860
7,845
7,838
7,376
7,325
7,298
7,260
7,255
4,596
4,215
2,914
2,439
2,426
1,635
Cl
O
NTs
2,04
2,00
1,99
1,02
4,35
3,09
9,5
9
8,5
8
7,5
7
6,5
6
5,5
5
4,5
4
3,5
3
2,5
2
1,5
1
0,5


1p
144,349
139,005
138,683
134,269
129,675
129,576
128,155
127,923
127,793
126,190
83,471
80,544
77,441
77,017
76,596
70,701
57,469
41,815
37,920
23,711
21,624
Cl
O
NTs
ppm

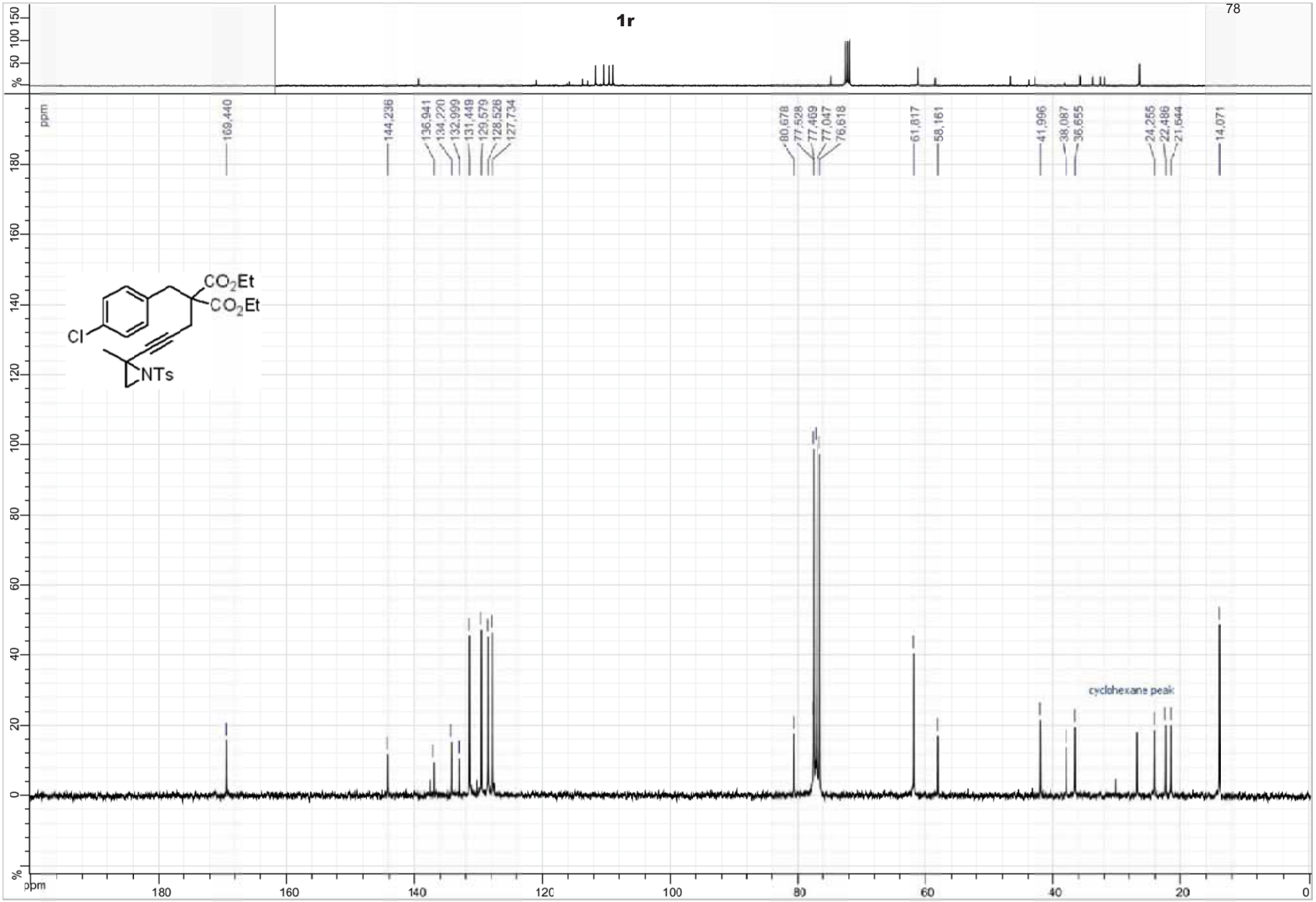

1q
ppm
7,871
7,844
7,333
7,306
7,260
7,127
7,098
6,807
6,778
4,211
3,759
3,344
2,881
2,711
2,432
2,428
1,636
1,257
1,234
water peak
MeO
CO2Et
CO2Et
NTs
2,06
2,13
1,90
2,07
4,37
3,10
1,93
1,01
2,09
4,06
3,12
6,39

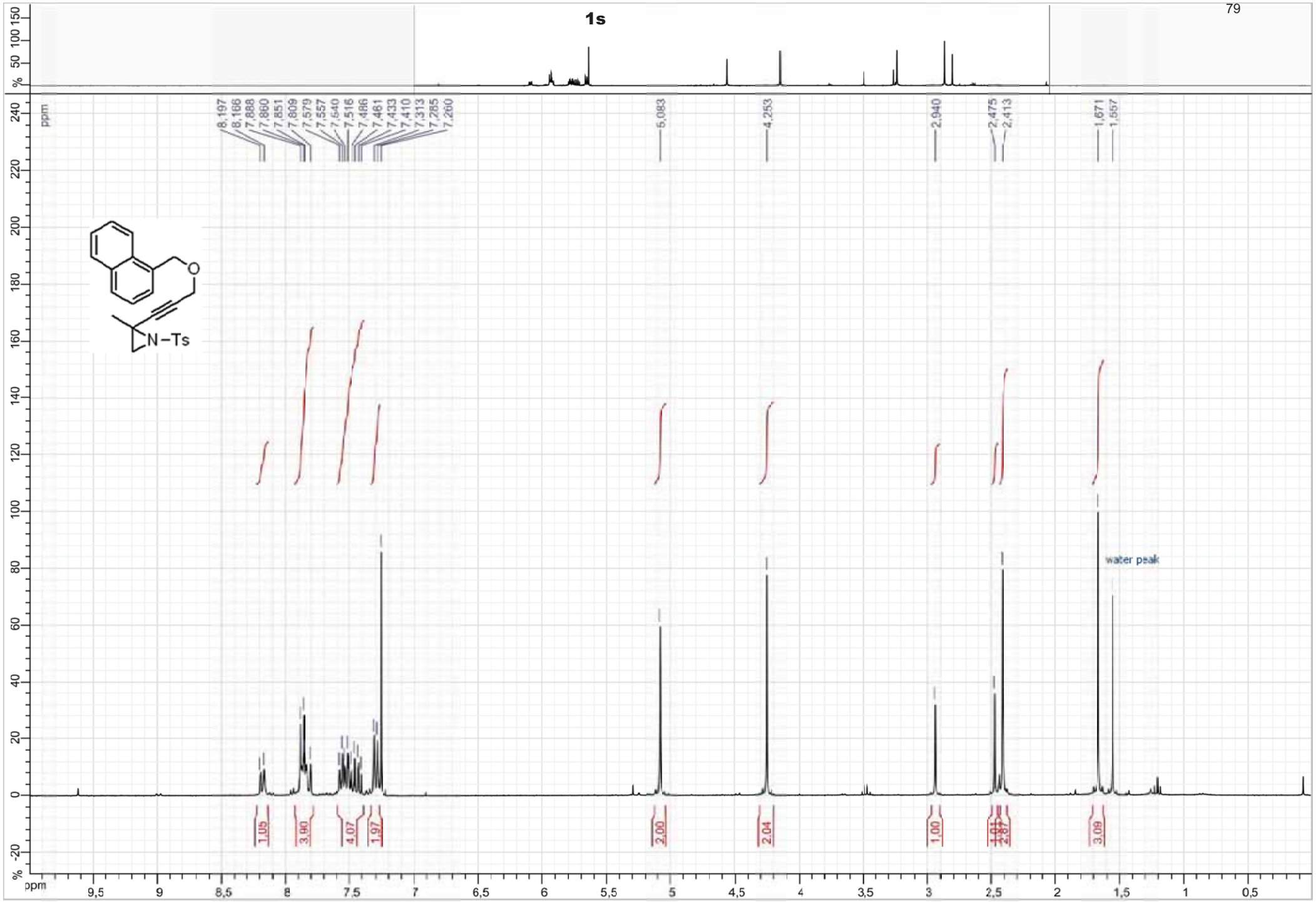

1q
ppm
169.691
158.639
144.176
136.923
131.009
129.535
127.749
127.555
113.782
80.887
80.483
77.467
76.810
76.616
61.647
58.337
55.169
41.859
30.263
30.502
24.077
22.409
21.625
14.069
MeO
CO2Et
CO2Et
NTs
cyclohexane peak

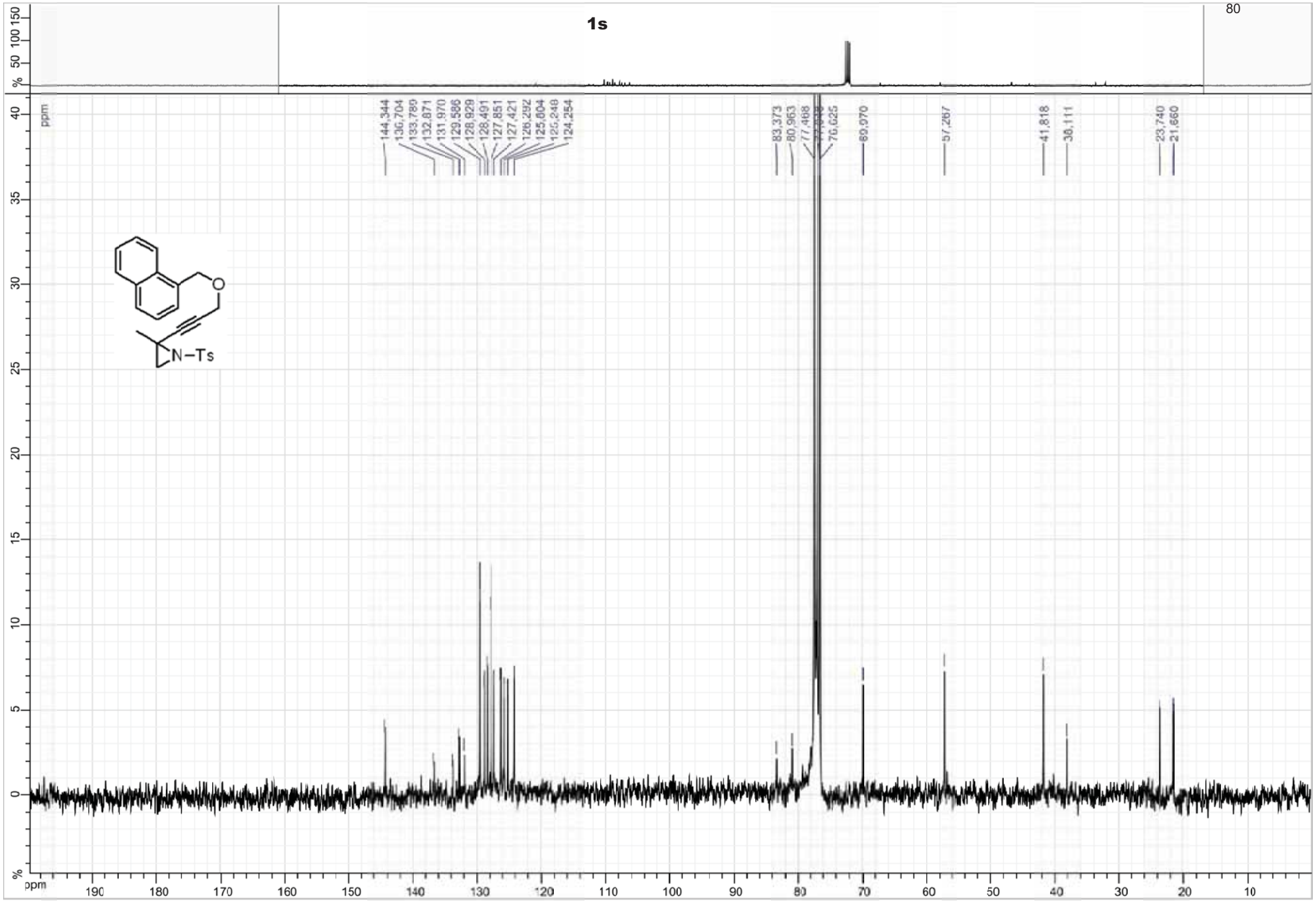
1r

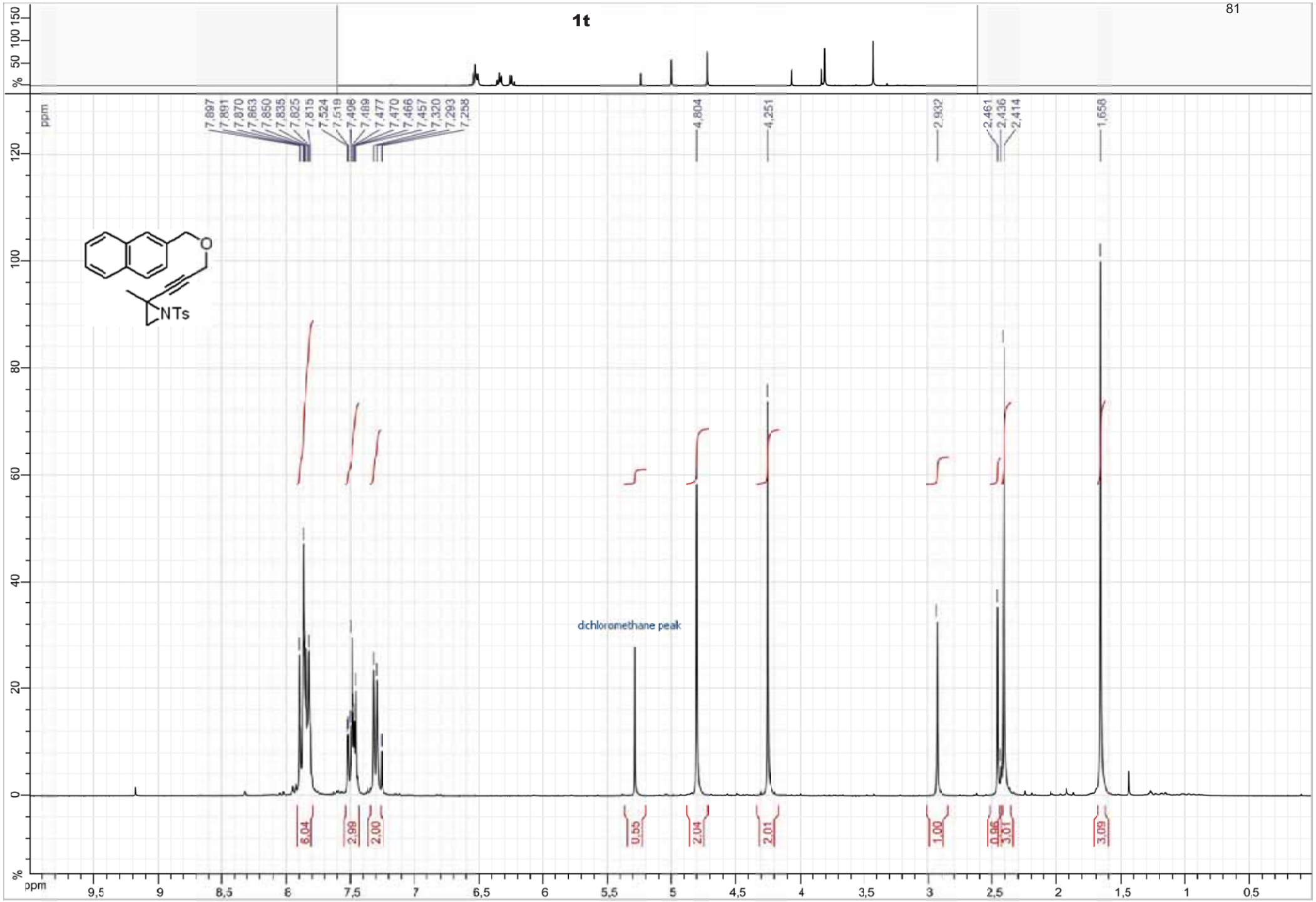

7,864
7,837
7,336
7,309
7,260
7,244
7,216
7,169
7,141
5,294
4,211
3,386
2,895
2,710
2,432
2,399
1,624
1,257
CO2Et
CO2Et
Cl
NTs
cyclohexane peak
water peak
2,00
4,21
2,01
1,03
2,02
3,00
6,08
ppm

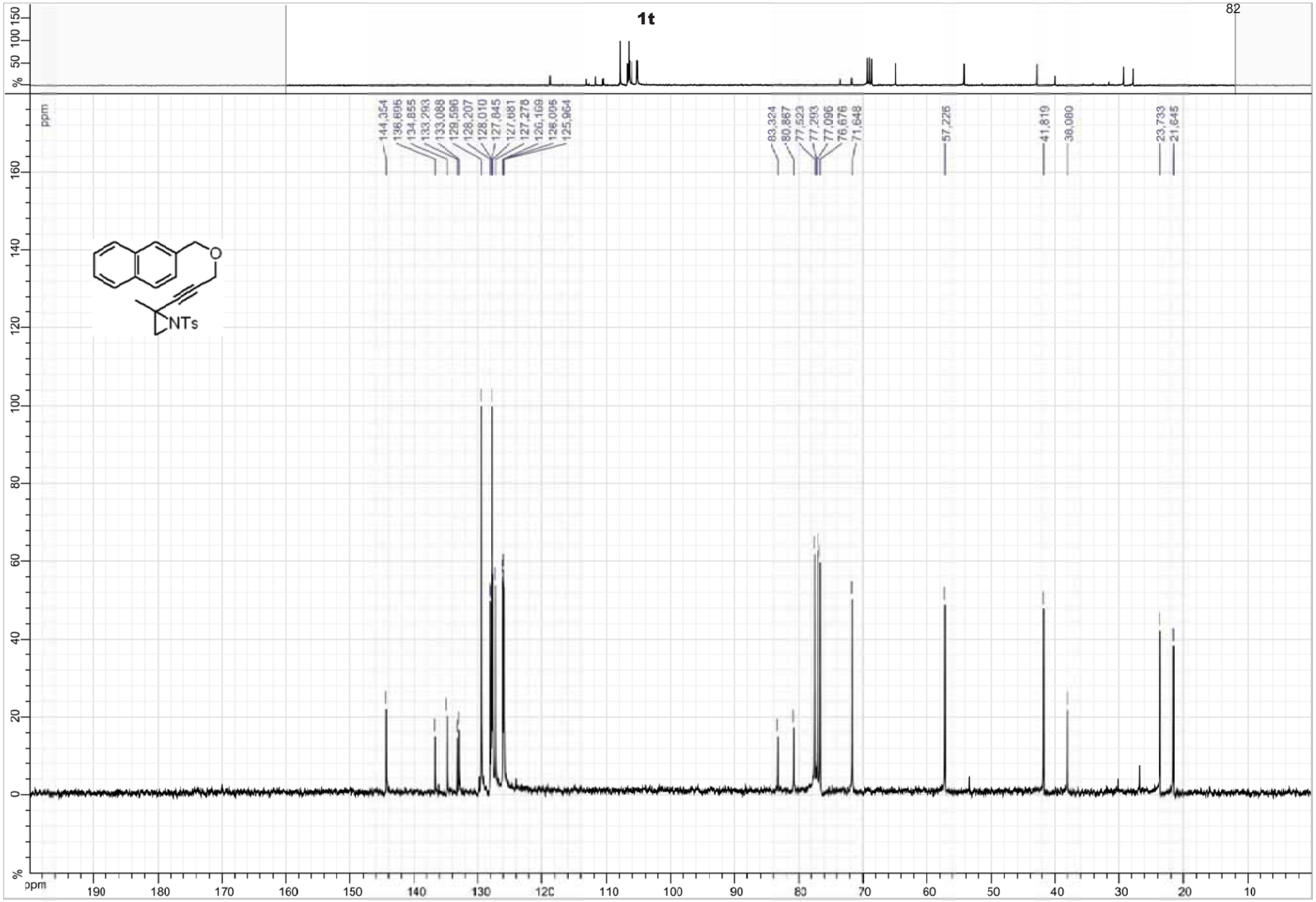
1r


ppm
169,440
144,236
136,941
134,220
132,999
131,449
129,579
128,526
127,734
80,678
77,528
77,469
77,047
76,618
61,817
58,161
41,996
38,087
36,655
24,255
22,486
21,644
14,071
CO2Et
CO2Et
Cl
NTs
cyclohexane peak
180
160
140
120
100
80
60
40
20
0

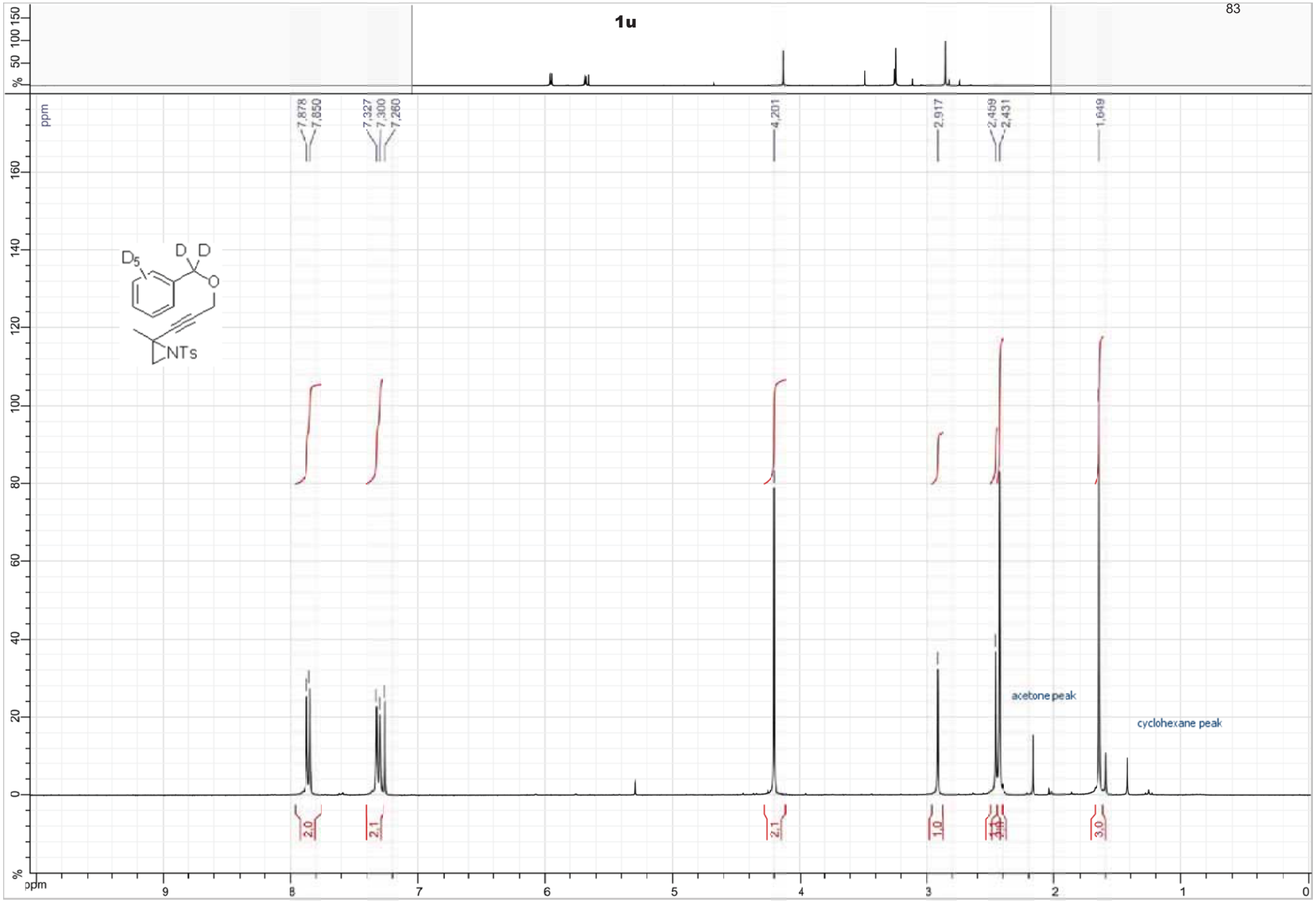

1s
ppm
8,197
8,166
7,888
7,860
7,851
7,809
7,579
7,557
7,540
7,516
7,486
7,461
7,433
7,410
7,313
7,285
7,260
5,083
4,253
2,940
2,475
2,413
1,671
1,557
N–Ts
water peak
1,05
3,90
4,07
1,97
2,00
2,04
1,00
3,09


1s
144,344
136,704
133,789
132,871
131,970
129,586
128,929
128,491
127,851
127,421
126,292
125,604
125,248
124,254
83,373
80,963
77,468
76,625
69,970
57,267
41,818
38,111
23,740
21,660
N–Ts
ppm

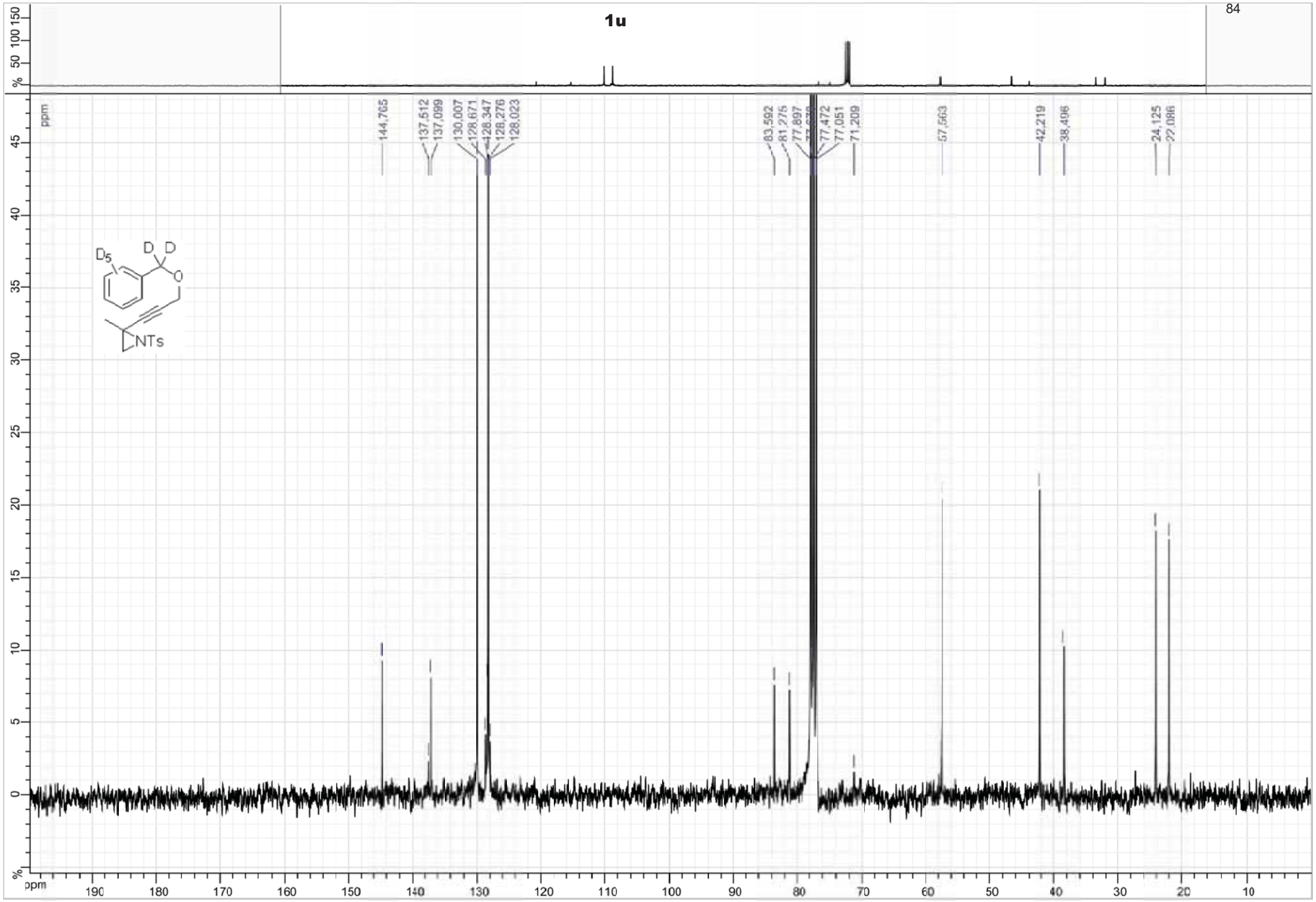

1t
ppm
7,897
7,891
7,870
7,863
7,850
7,835
7,825
7,815
7,524
7,519
7,498
7,489
7,477
7,470
7,466
7,457
7,320
7,293
7,258
4,804
4,251
2,932
2,461
2,436
2,414
1,658
NTs
dichloromethane peak
6,04
2,99
2,00
0,55
2,04
2,01
1,00
0,96
3,01
3,09

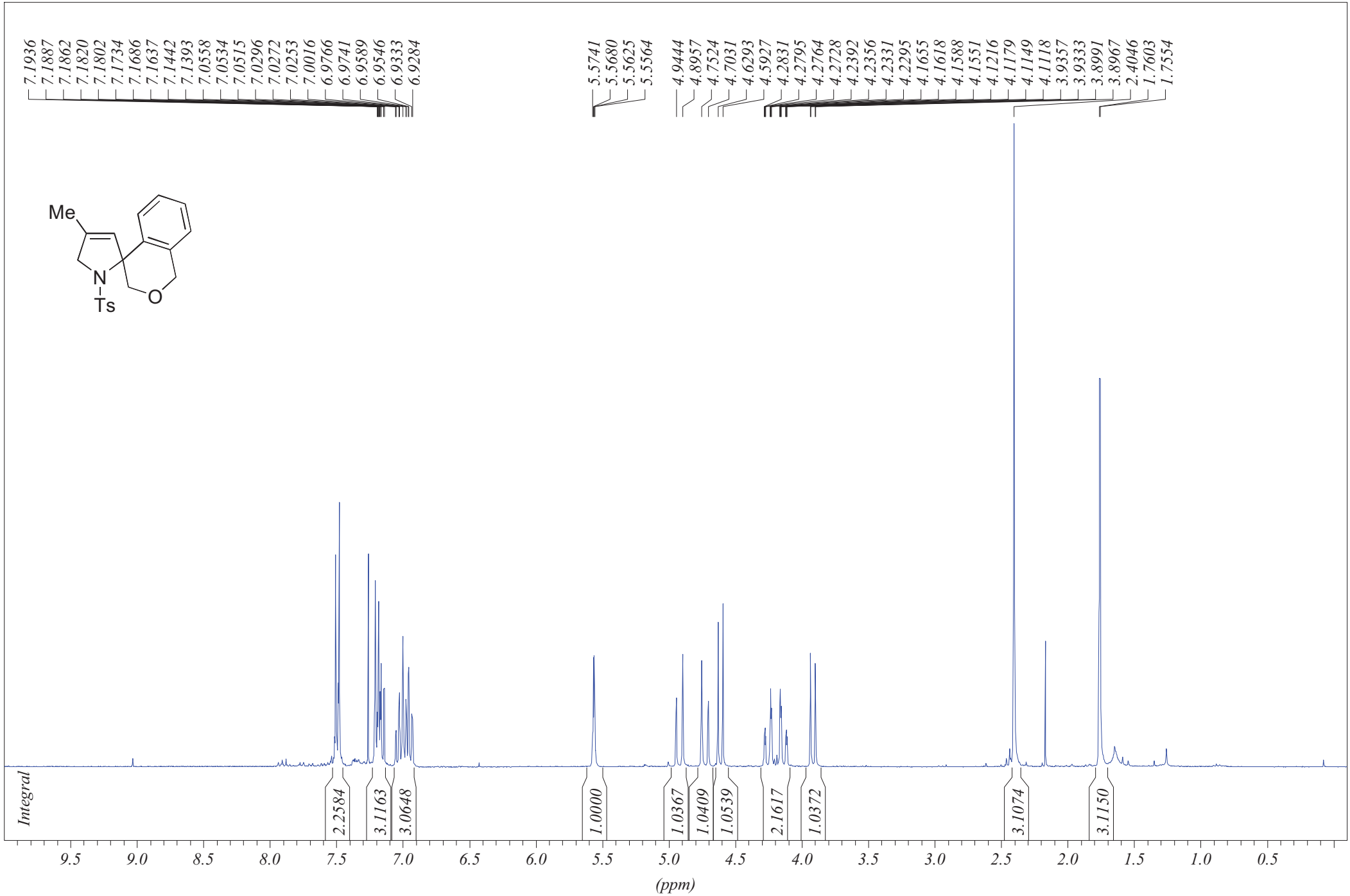

1t
82
144,354
136,695
134,855
133,293
133,088
129,596
128,207
128,010
127,845
127,681
127,278
126,169
126,095
125,964
83,324
80,887
77,523
77,293
77,096
76,676
71,648
57,226
41,819
38,080
23,733
21,645
NTs
ppm

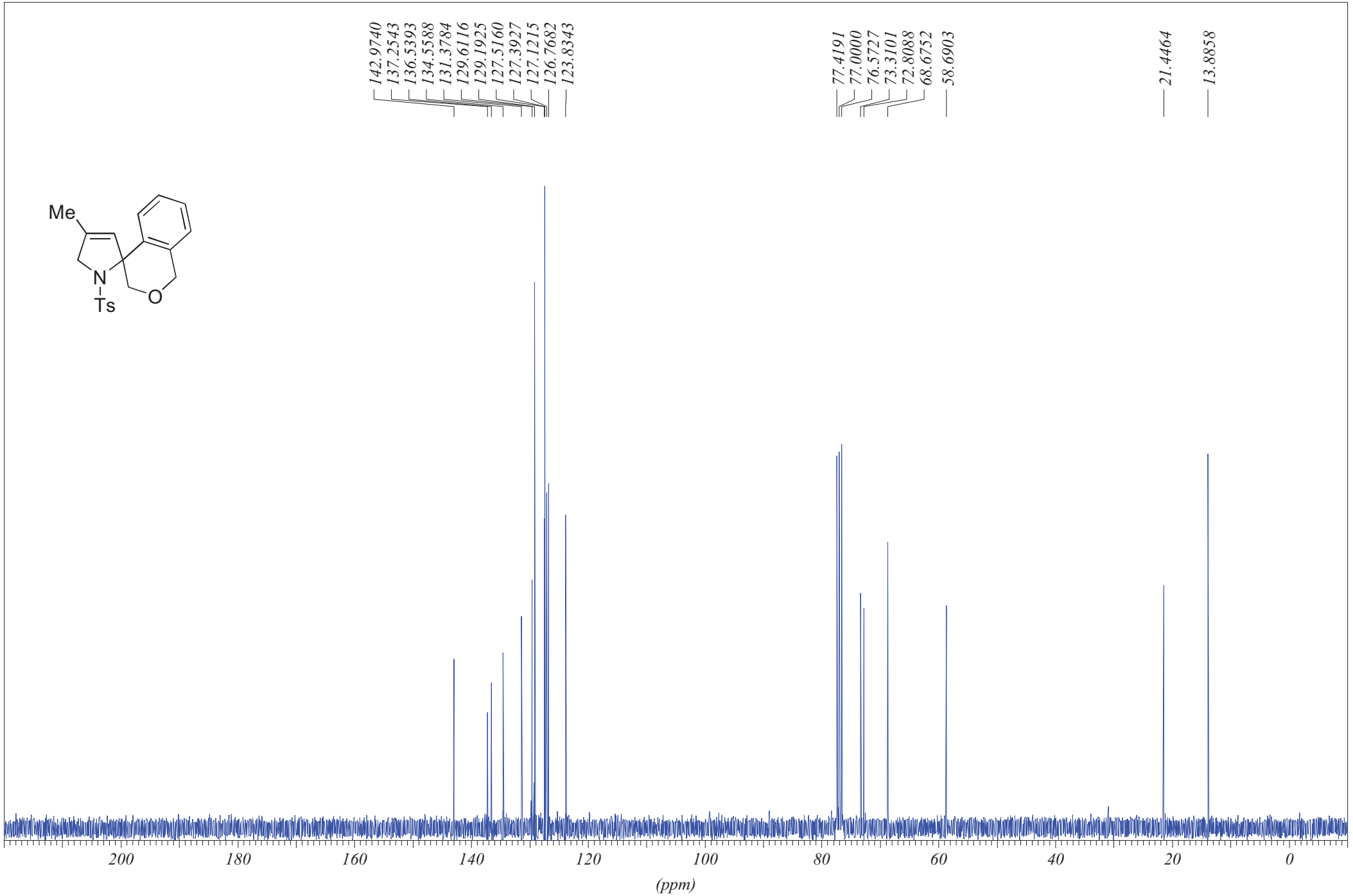
1u

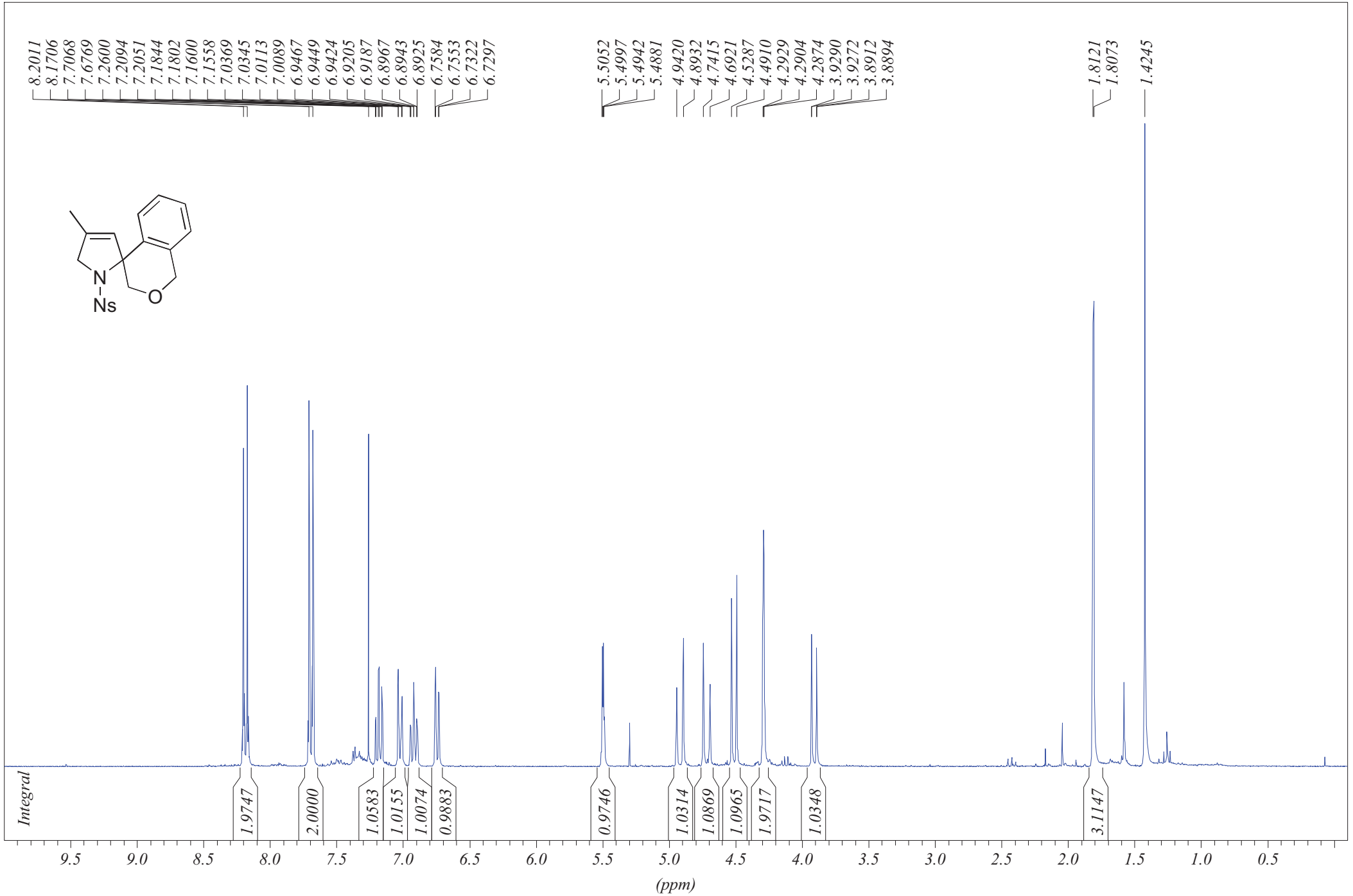

ppm
7.878
7.850
7.327
7.300
7.260
4.201
2.917
2.469
2.431
1.649
D5
D
D
O
NTs
acetone peak
cyclohexane peak
2.0
2.1
2.1
1.0
3.0
ppm

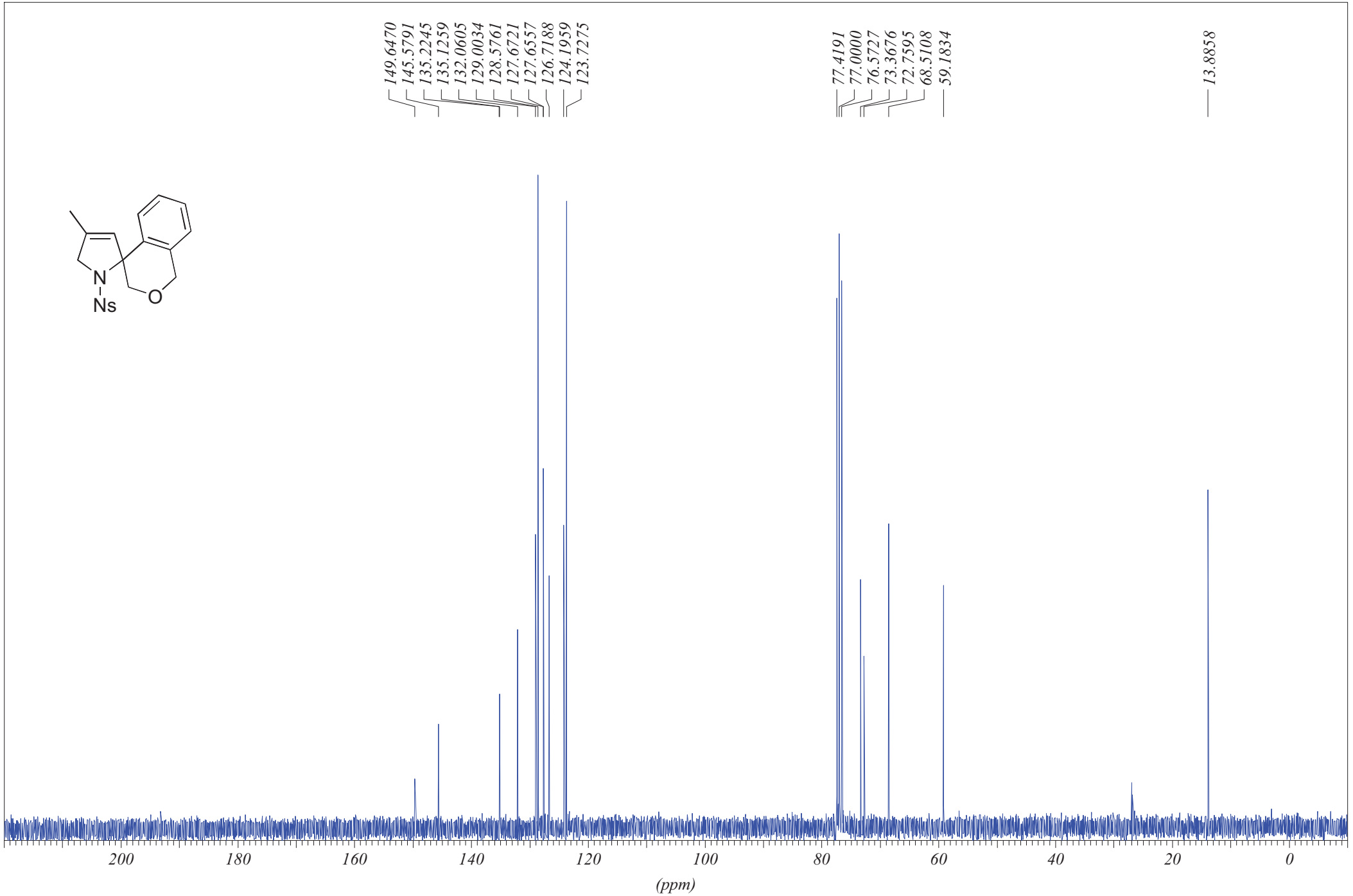
1u

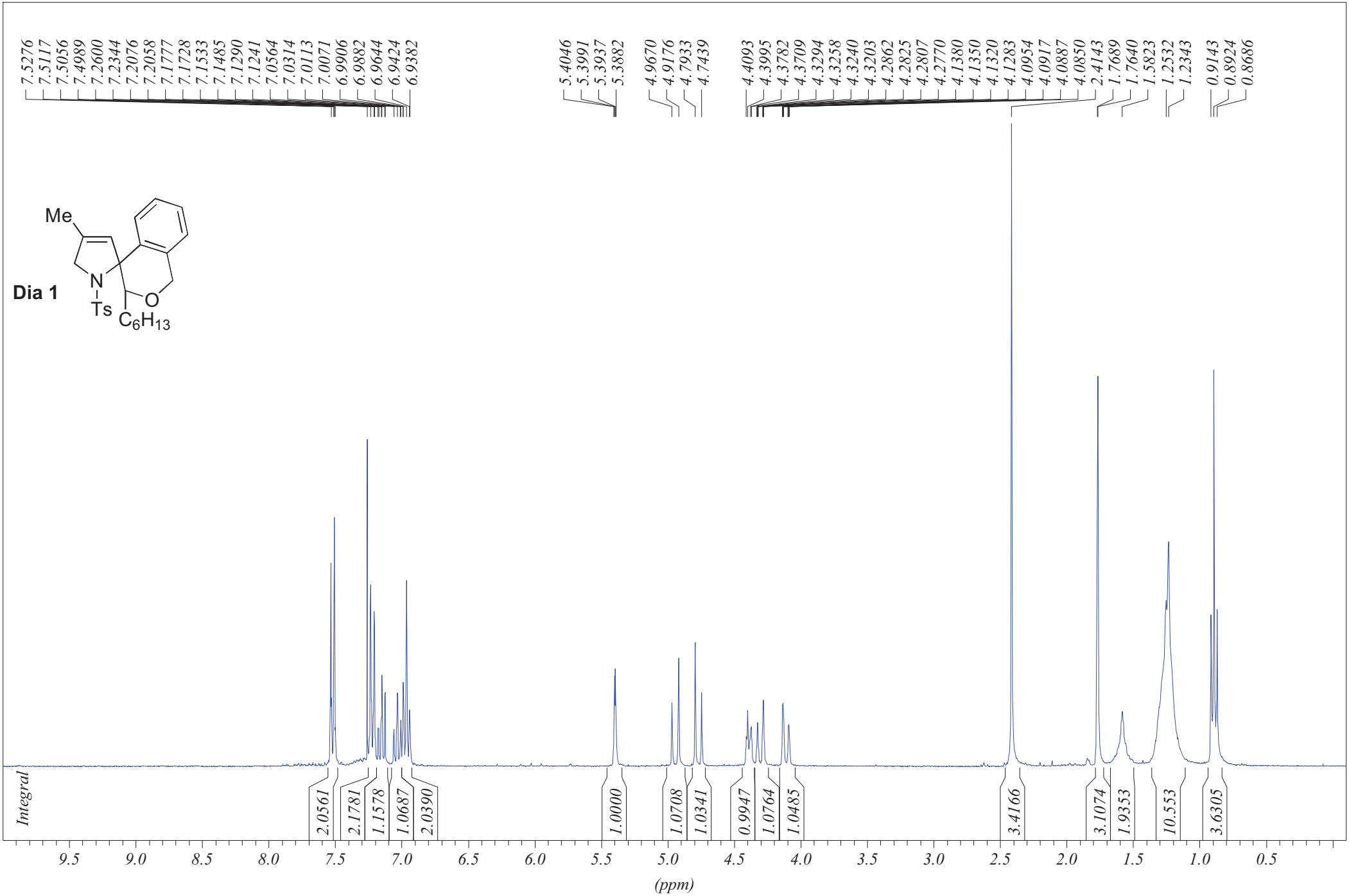

144,765
137,512
137,099
130,007
128,671
128,347
128,276
128,023
83,592
81,275
77,897
77,472
77,051
71,209
57,563
42,219
38,496
24,125
22,086
D5
D D
O
NTs
ppm

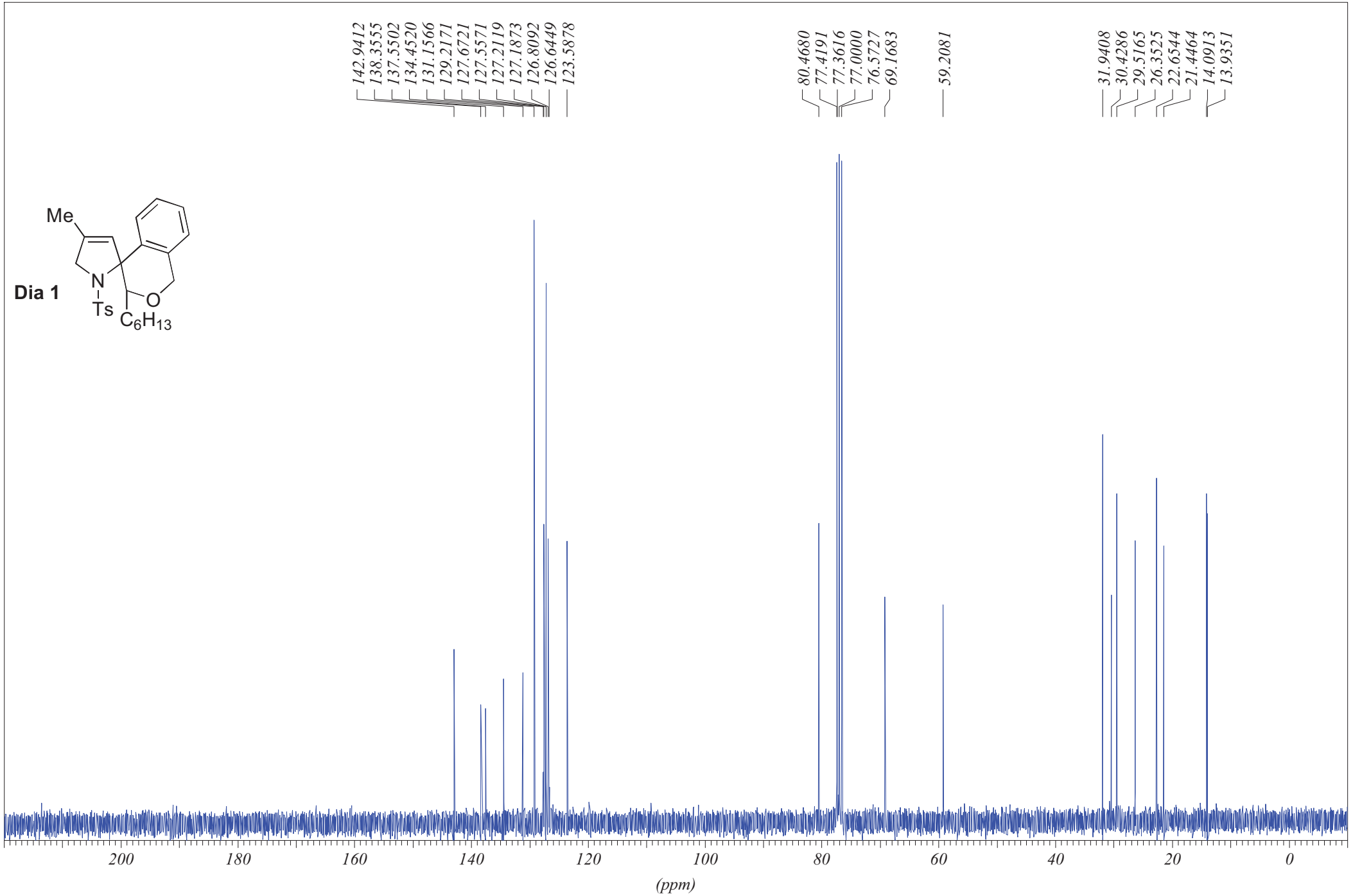

7.1936
7.1887
7.1862
7.1820
7.1802
7.1734
7.1686
7.1637
7.1442
7.1393
7.0558
7.0534
7.0515
7.0296
7.0272
7.0253
7.0016
6.9766
6.9741
6.9589
6.9546
6.9333
6.9284
5.5741
5.5680
5.5625
5.5564
4.9444
4.8957
4.7524
4.7031
4.6293
4.5927
4.2831
4.2795
4.2764
4.2728
4.2392
4.2356
4.2331
4.2295
4.1655
4.1618
4.1588
4.1551
4.1216
4.1179
4.1149
4.1118
3.9357
3.9333
3.8991
3.8967
2.4046
1.7603
1.7554
Me
N
Ts
O
Integral
2.2584
3.1163
3.0648
1.0000
1.0367
1.0409
1.0539
2.1617
1.0372
3.1074
3.1150
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
(ppm)

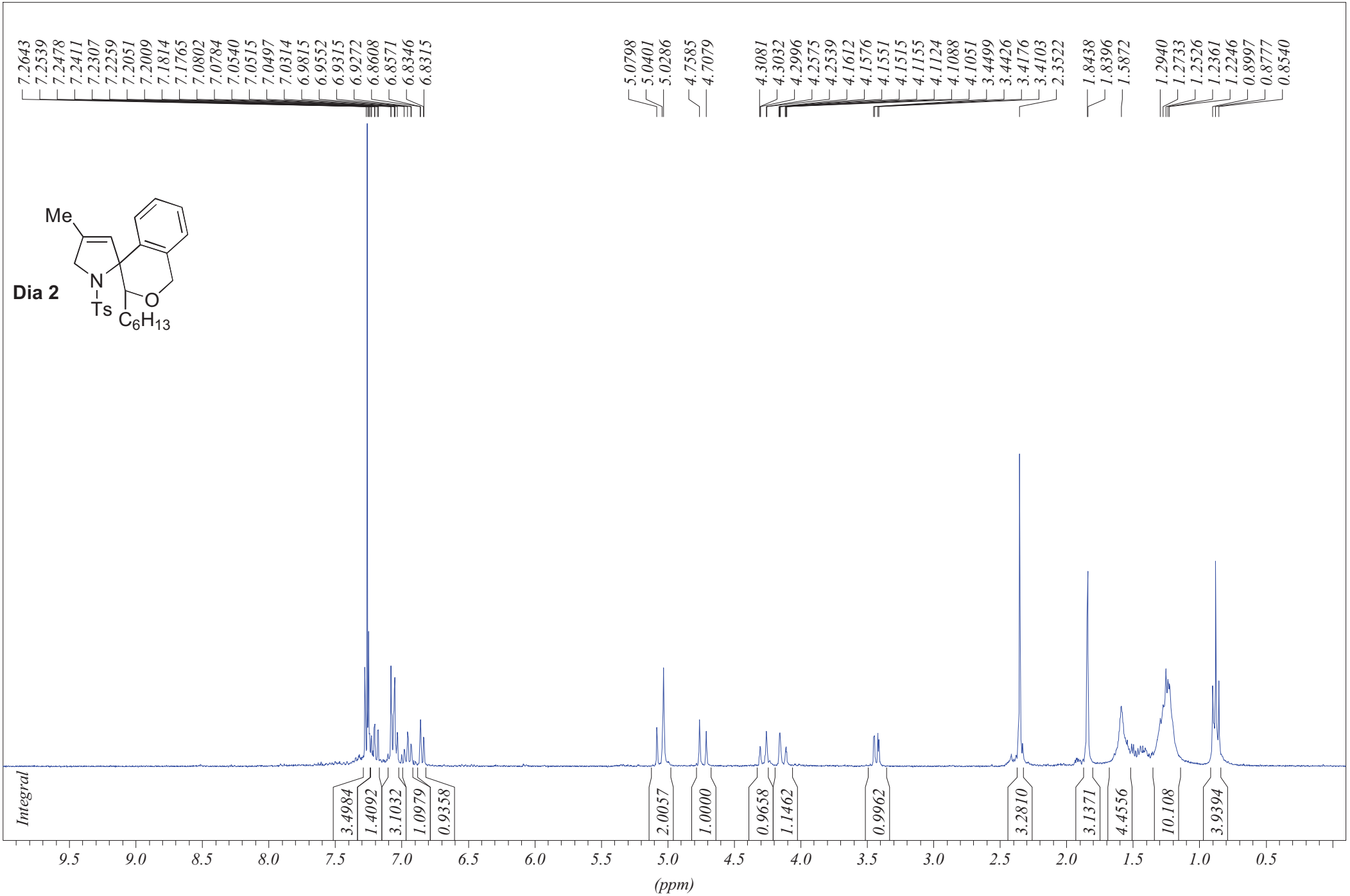

142.9740
137.2543
136.5393
134.5588
131.3784
129.6116
129.1925
127.5160
127.3927
127.1215
126.7682
123.8343
77.4191
77.0000
76.5727
73.3101
72.8088
68.6752
58.6903
21.4464
13.8858
Me
N
Ts
O
200
180
160
140
120
100
80
60
40
20
0
(ppm)

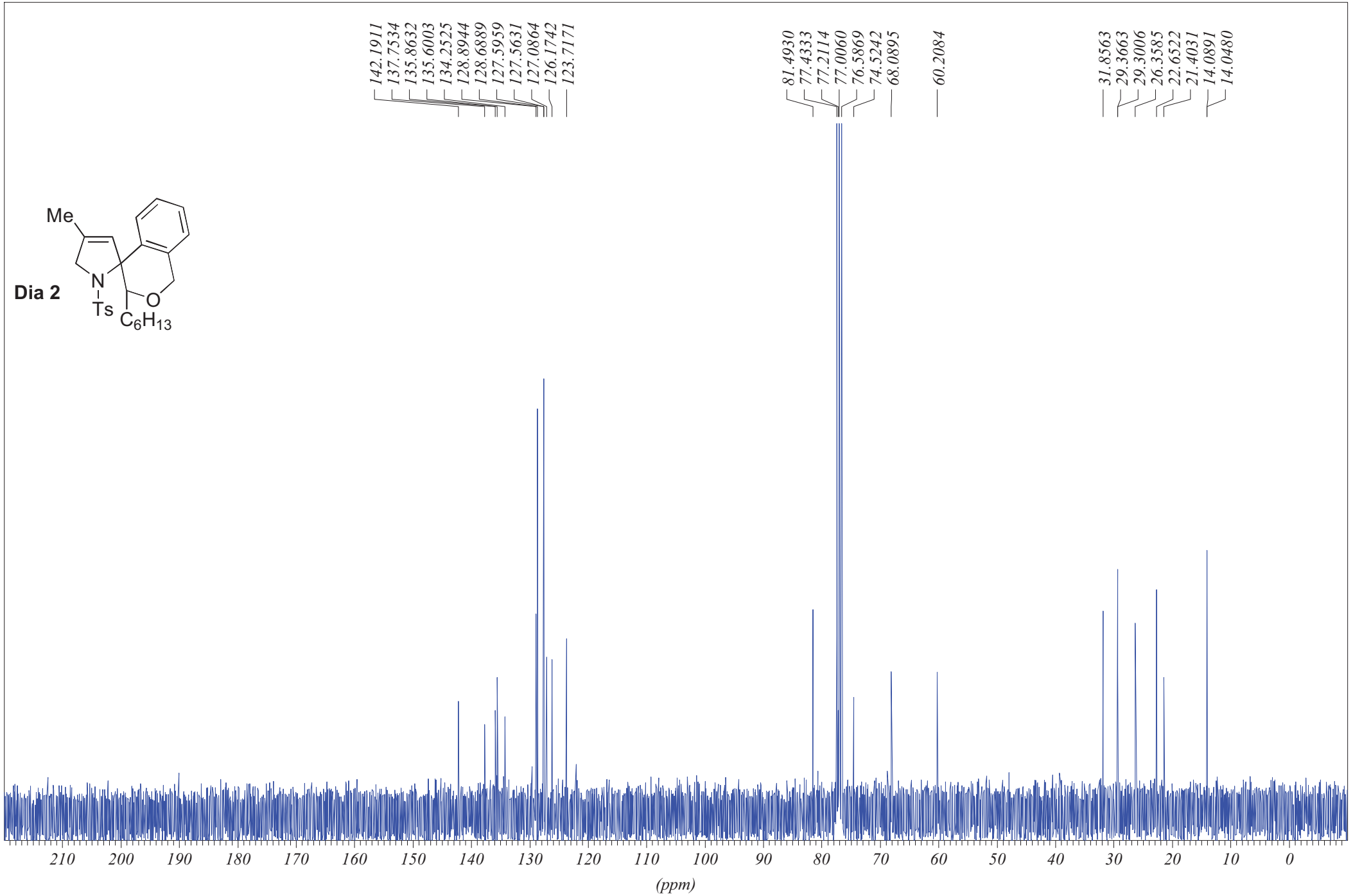

8.2011
8.1706
7.7068
7.6769
7.2600
7.2094
7.2051
7.1844
7.1802
7.1600
7.1558
7.0369
7.0345
7.0113
7.0089
6.9467
6.9449
6.9424
6.9205
6.9187
6.8967
6.8943
6.8925
6.7584
6.7553
6.7322
6.7297
5.5052
5.4997
5.4942
5.4881
4.9420
4.8932
4.7415
4.6921
4.5287
4.4910
4.2929
4.2904
4.2874
3.9290
3.9272
3.8912
3.8894
1.8121
1.8073
1.4245
N
Ns
O
Integral
1.9747
2.0000
1.0583
1.0155
1.0074
0.9883
0.9746
1.0314
1.0869
1.0965
1.9717
1.0348
3.1147
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
(ppm)

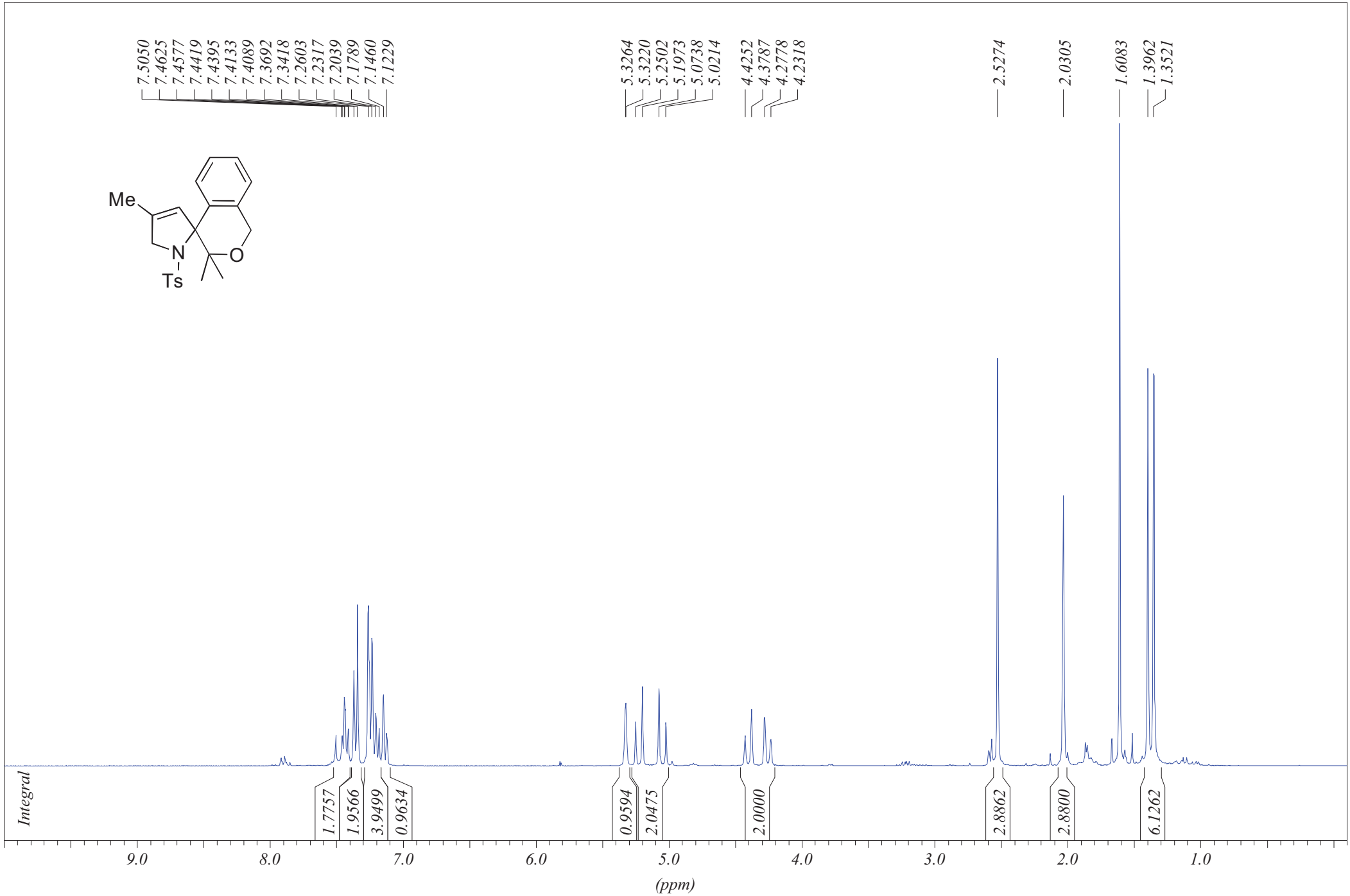

149.6470
145.5791
135.2245
135.1259
132.0605
129.0034
128.5761
127.6721
127.6557
126.7188
124.1959
123.7275
77.4191
77.0000
76.5727
73.3676
72.7595
68.5108
59.1834
13.8858
N
Ns
O
200
180
160
140
120
100
80
60
40
20
0
(ppm)

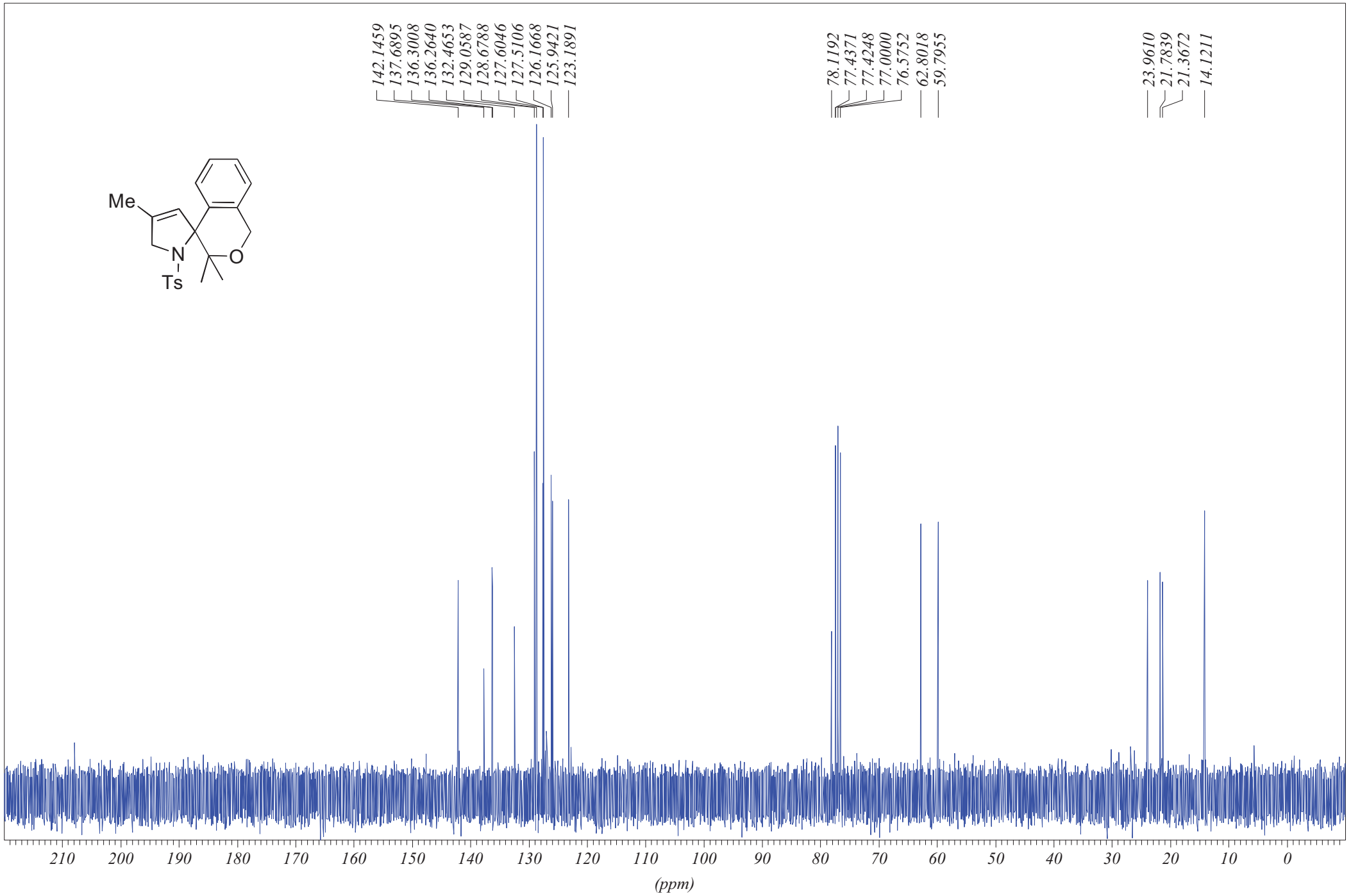

7.5276
7.5117
7.5056
7.4989
7.2600
7.2344
7.2076
7.2058
7.1777
7.1728
7.1533
7.1485
7.1290
7.1241
7.0564
7.0314
7.0113
7.0071
6.9906
6.9882
6.9644
6.9424
6.9382
5.4046
5.3991
5.3937
5.3882
4.9670
4.9176
4.7933
4.7439
4.4093
4.3995
4.3782
4.3709
4.3294
4.3258
4.3240
4.3203
4.2862
4.2825
4.2807
4.2770
4.1380
4.1350
4.1320
4.1283
4.0954
4.0917
4.0887
4.0850
2.4143
1.7689
1.7640
1.5823
1.2532
1.2343
0.9143
0.8924
0.8686
Me
N
Ts
O
C_6H_{13}
Dia 1
Integral
2.0561
2.1781
1.1578
1.0687
2.0390
1.0000
1.0708
1.0341
0.9947
1.0764
1.0485
3.4166
3.1074
1.9353
10.553
3.6305
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
(ppm)

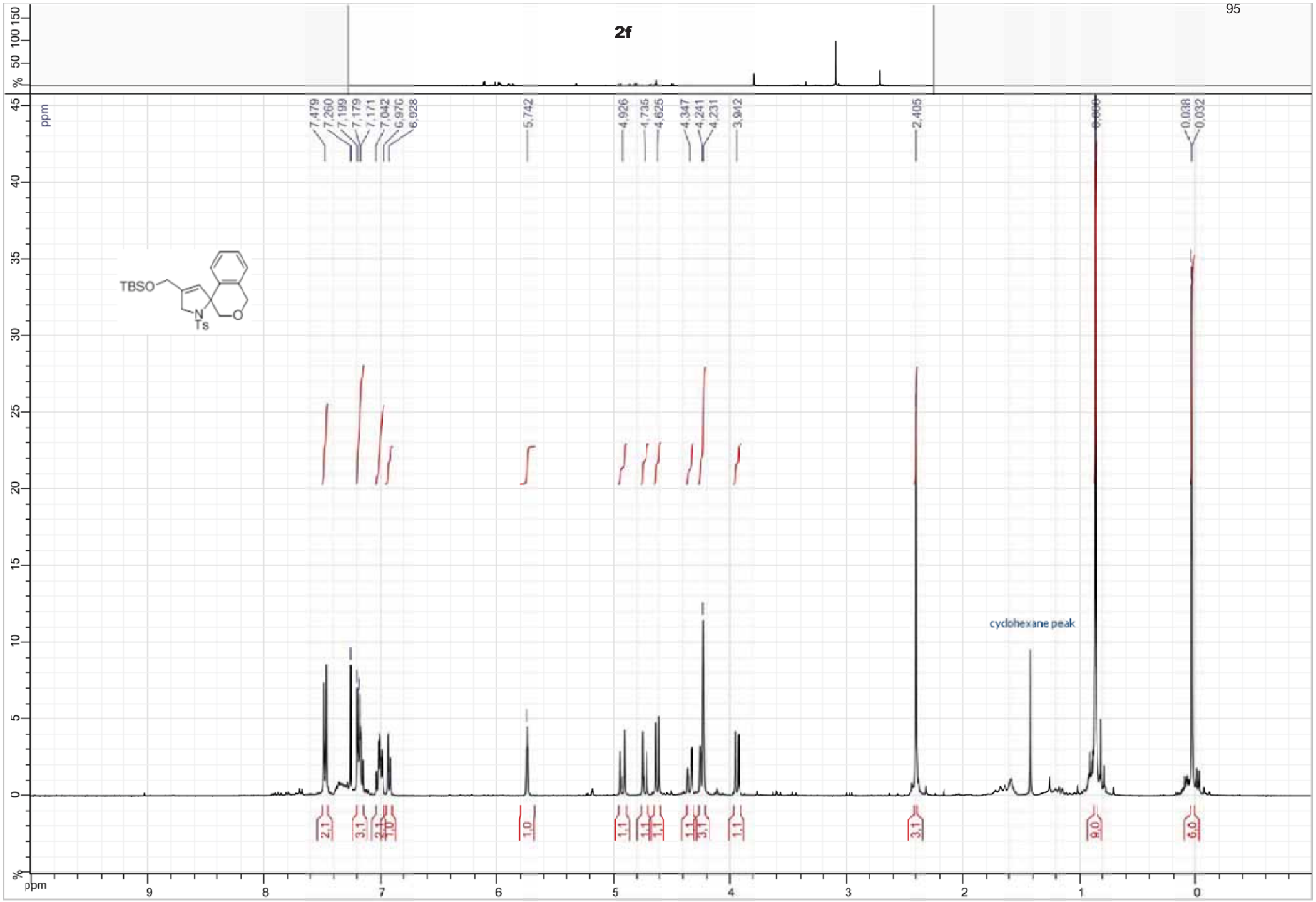

142.9412
138.3555
137.5502
134.4520
131.1566
129.2171
127.6721
127.5571
127.2119
127.1873
126.8092
126.6449
123.5878
80.4680
77.4191
77.3616
77.0000
76.5727
69.1683
59.2081
31.9408
30.4286
29.5165
26.3525
22.6544
21.4464
14.0913
13.9351
Me
N
Ts
O
C6H13
Dia 1
200
180
160
140
120
100
80
60
40
20
0
(ppm)


7.2643
7.2539
7.2478
7.2411
7.2307
7.2259
7.2051
7.2009
7.1814
7.1765
7.0802
7.0784
7.0540
7.0515
7.0497
7.0314
6.9815
6.9552
6.9315
6.9272
6.8608
6.8571
6.8346
6.8315
5.0798
5.0401
5.0286
4.7585
4.7079
4.3081
4.3032
4.2996
4.2575
4.2539
4.1612
4.1576
4.1551
4.1515
4.1155
4.1124
4.1088
4.1051
3.4499
3.4426
3.4176
3.4103
2.3522
1.8438
1.8396
1.5872
1.2940
1.2733
1.2526
1.2361
1.2246
0.8997
0.8777
0.8540
Me
N
Ts
O
C6H13
Dia 2
Integral
3.4984
1.4092
3.1032
1.0979
0.9358
2.0057
1.0000
0.9658
1.1462
0.9962
3.2810
3.1371
4.4556
10.108
3.9394
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
(ppm)

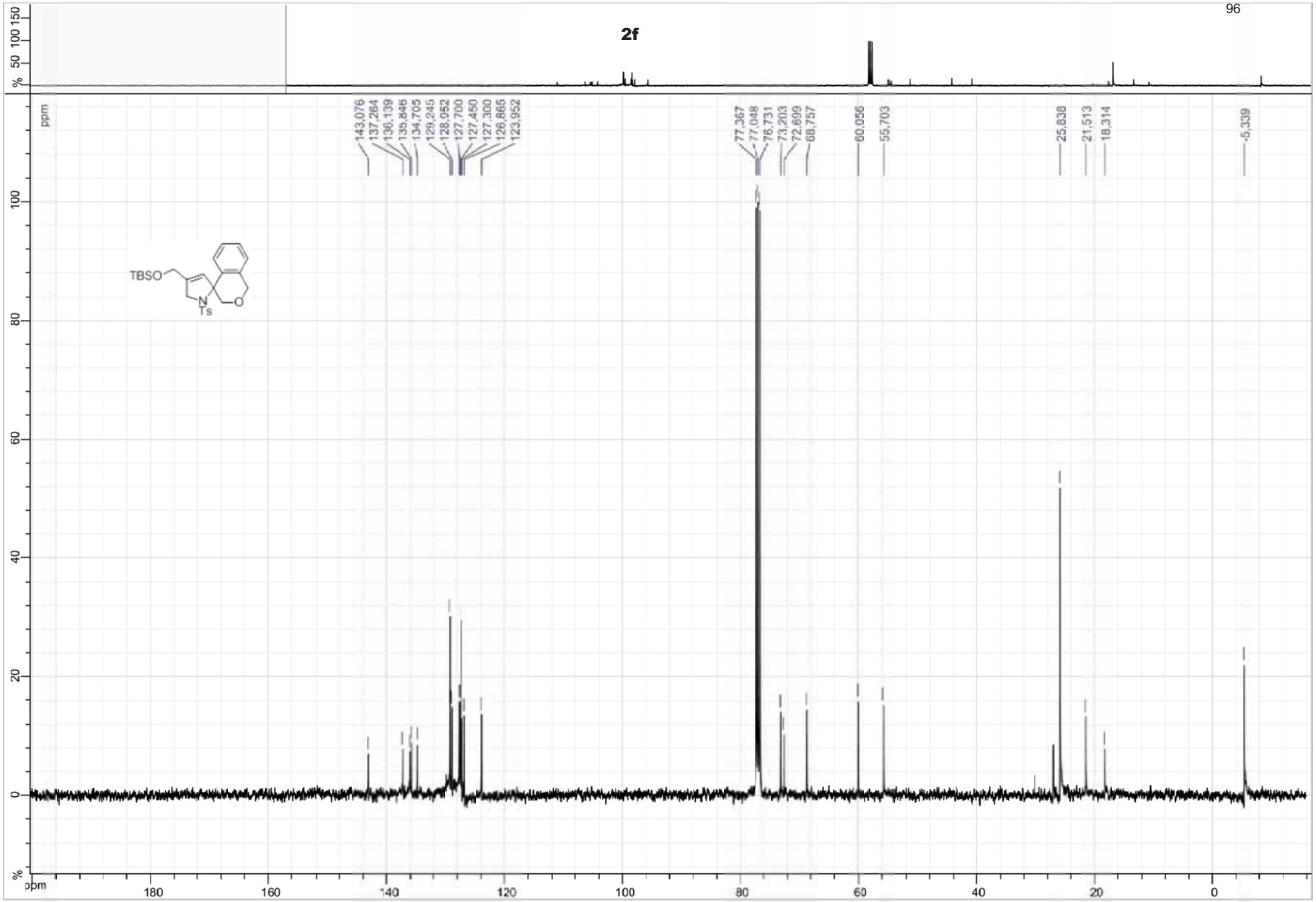

142.1911
137.7534
135.8632
135.6003
134.2525
128.8944
128.6889
127.5959
127.5631
127.0864
126.1742
123.7171
81.4930
77.4333
77.2114
77.0060
76.5869
74.5242
68.0895
60.2084
31.8563
29.3663
29.3006
26.3585
22.6522
21.4031
14.0891
14.0480
Me
N
Ts
O
C_6H_{13}
Dia 2
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

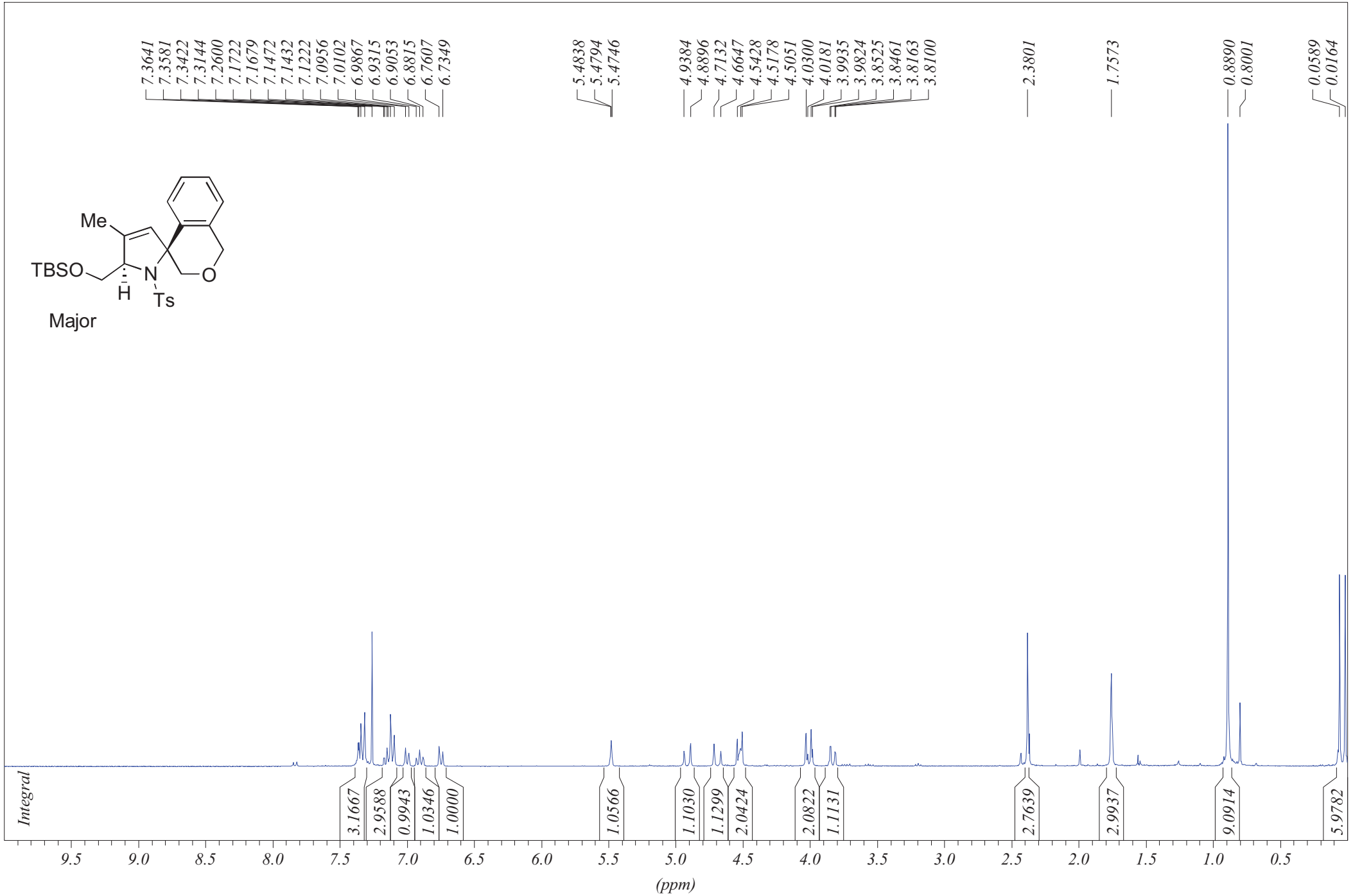

7.5050
7.4625
7.4577
7.4419
7.4395
7.4133
7.4089
7.3692
7.3418
7.2603
7.2317
7.2039
7.1789
7.1460
7.1229
5.3264
5.3220
5.2502
5.1973
5.0738
5.0214
4.4252
4.3787
4.2778
4.2318
2.5274
2.0305
1.6083
1.3962
1.3521
Me
N
Ts
O
Integral
1.7757
1.9566
3.9499
0.9634
0.9594
2.0475
2.0000
2.8862
2.8800
6.1262
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
(ppm)

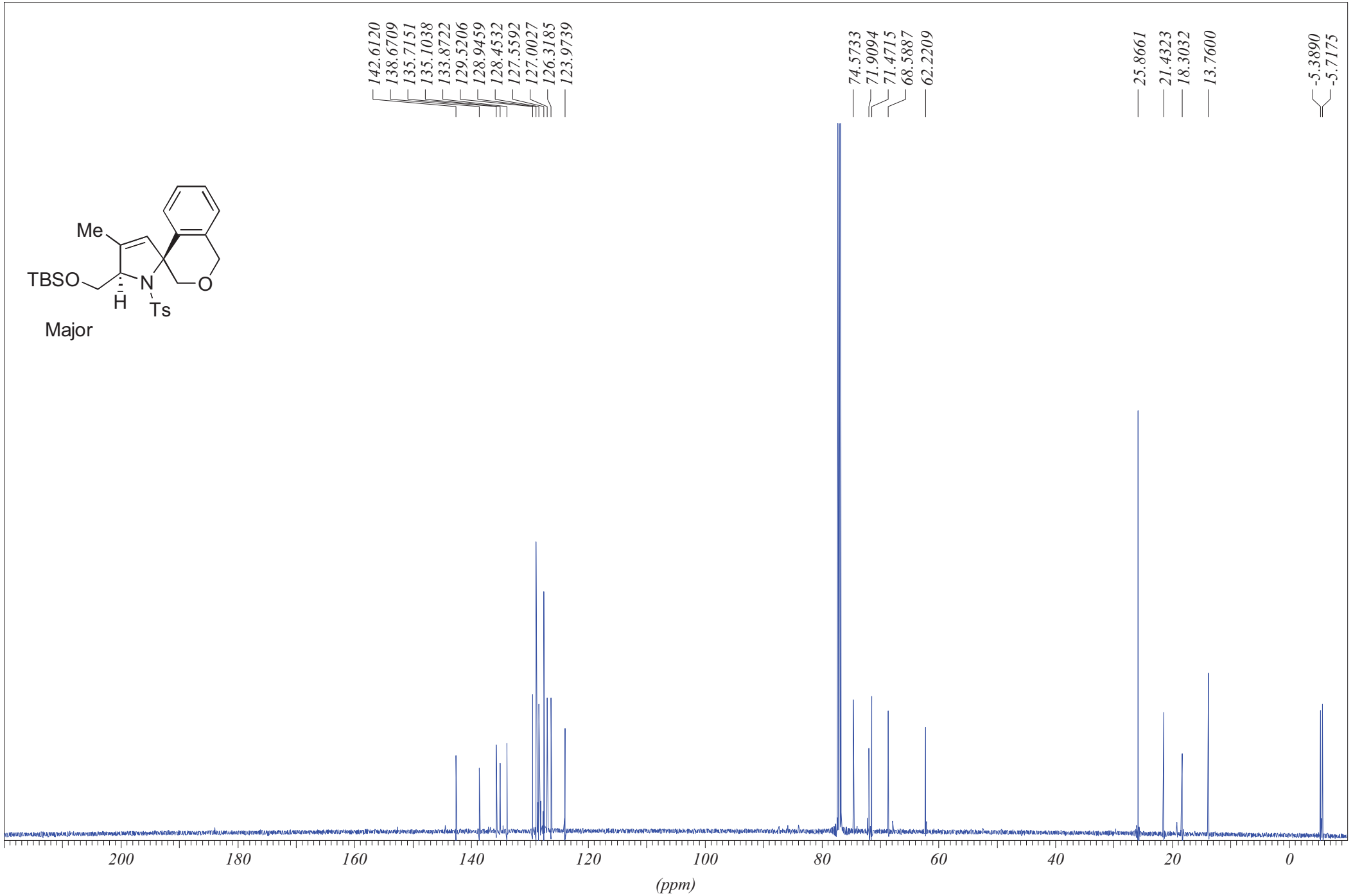

142.1459
137.6895
136.3008
136.2640
132.4653
129.0587
128.6788
127.6046
127.5106
126.1668
125.9421
123.1891
78.1192
77.4371
77.4248
77.0000
76.5752
62.8018
59.7955
23.9610
21.7839
21.3672
14.1211
Me
N
Ts
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

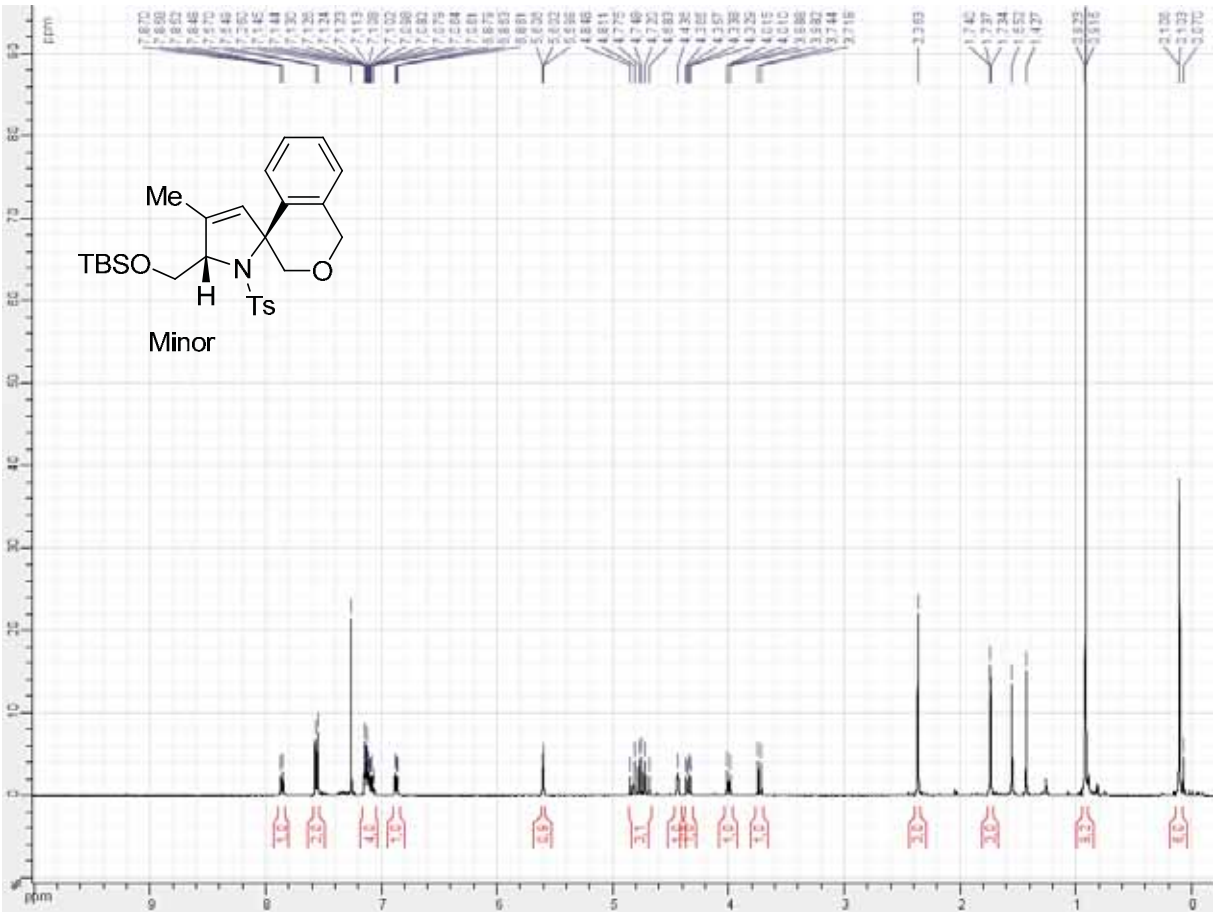

2f
ppm
7,479
7,260
7,199
7,179
7,171
7,042
6,976
6,928
5,742
4,926
4,735
4,625
4,347
4,241
4,231
3,942
2,405
0,038
0,032
TBSO
N
Ts
O
cyclohexane peak
2,1
3,1
2,1
1,0
1,0
1,1
1,1
1,1
1,1
3,1
1,1
3,1
9,0
6,0

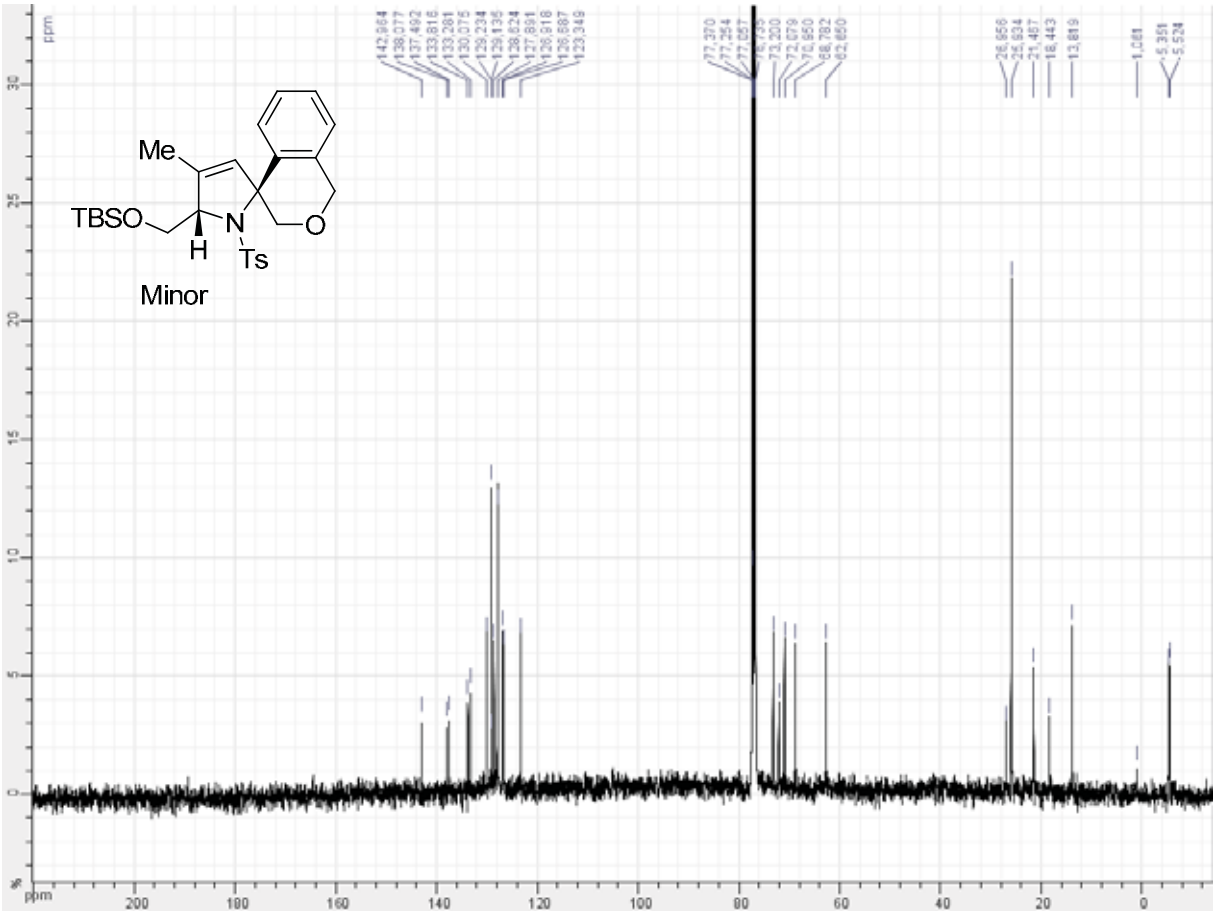
2f

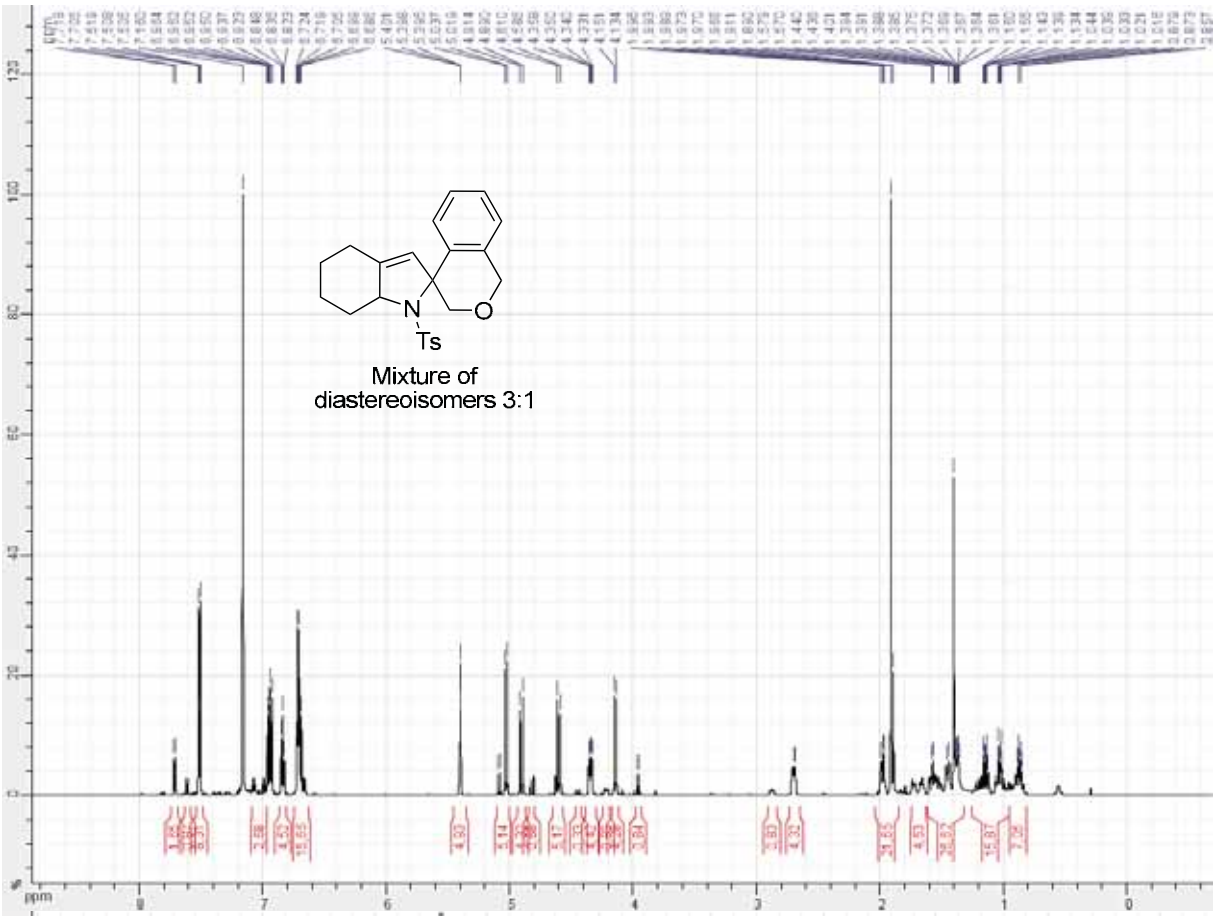

143,076
137,264
136,139
135,846
134,705
129,245
128,952
127,700
127,450
127,300
126,865
123,952
77,367
77,048
76,731
73,203
72,699
68,757
60,056
55,703
25,838
21,513
18,314
-5,339
TBSO
N
Ts
O
ppm
%

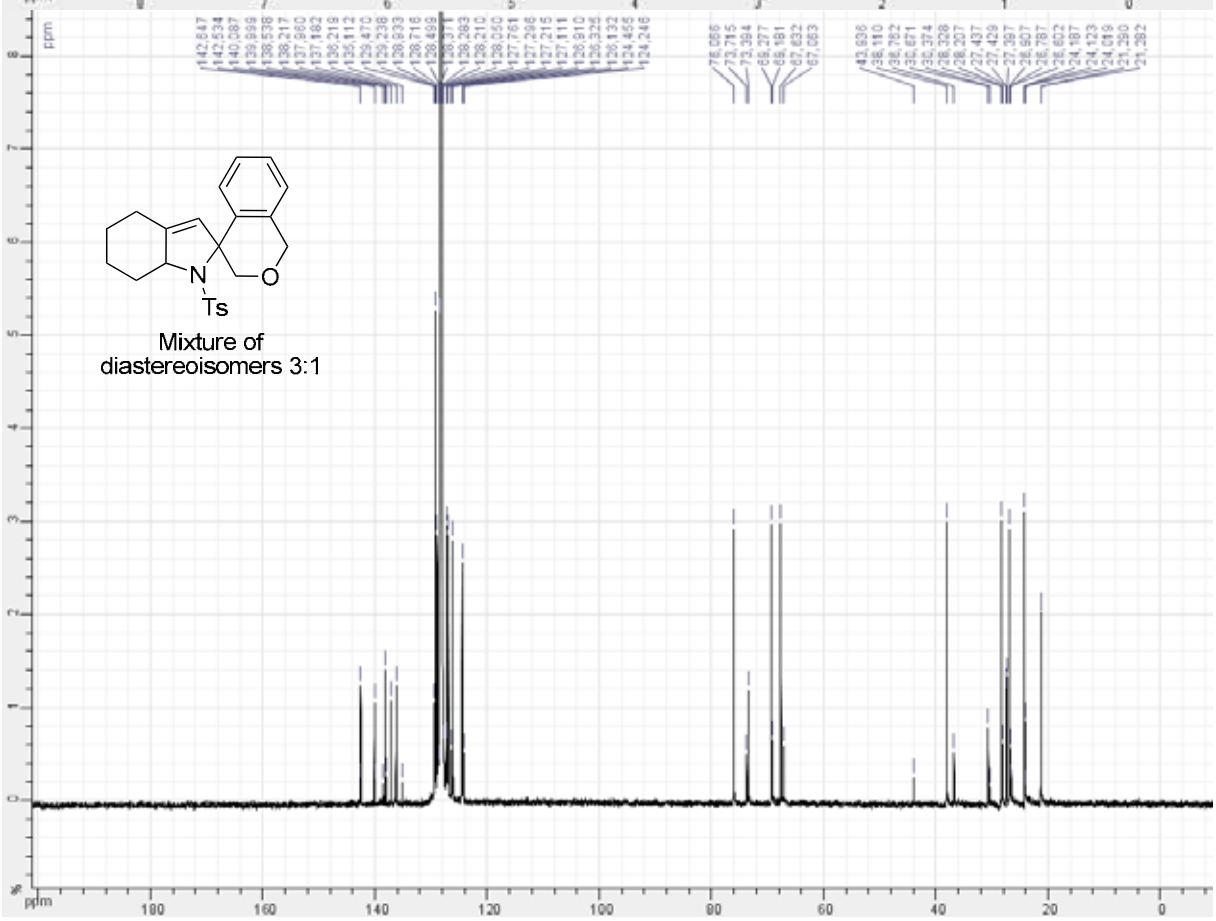

7.3641
7.3581
7.3422
7.3144
7.2600
7.1722
7.1679
7.1472
7.1432
7.1222
7.0956
7.0102
6.9867
6.9315
6.9053
6.8815
6.7607
6.7349
5.4838
5.4794
5.4746
4.9384
4.8896
4.7132
4.6647
4.5428
4.5178
4.5051
4.0300
4.0181
3.9935
3.9824
3.8525
3.8461
3.8163
3.8100
2.3801
1.7573
0.8890
0.8001
0.0589
0.0164
Me
TBSO
N
O
H
Ts
Major
Integral
3.1667
2.9588
0.9943
1.0346
1.0000
1.0566
1.1030
1.1299
2.0424
2.0822
1.1131
2.7639
2.9937
9.0914
5.9782
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
(ppm)

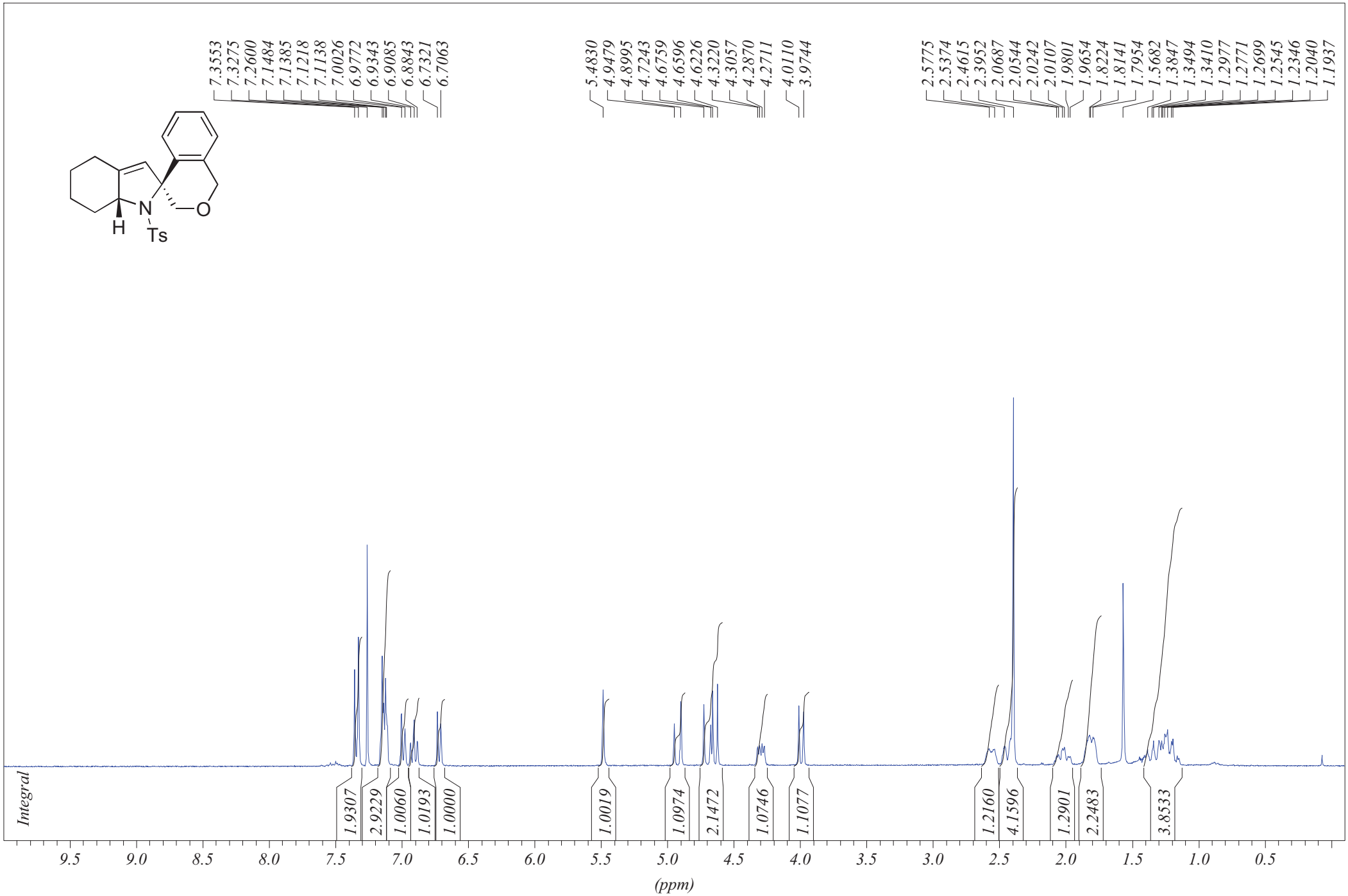

142.6120
138.6709
135.7151
135.1038
133.8722
129.5206
128.9459
128.4532
127.5592
127.0027
126.3185
123.9739
74.5733
71.9094
71.4715
68.5887
62.2209
25.8661
21.4323
18.3032
13.7600
-5.3890
-5.7175
Me
TBSO
N
O
H
Ts
Major
200
180
160
140
120
100
80
60
40
20
0
(ppm)

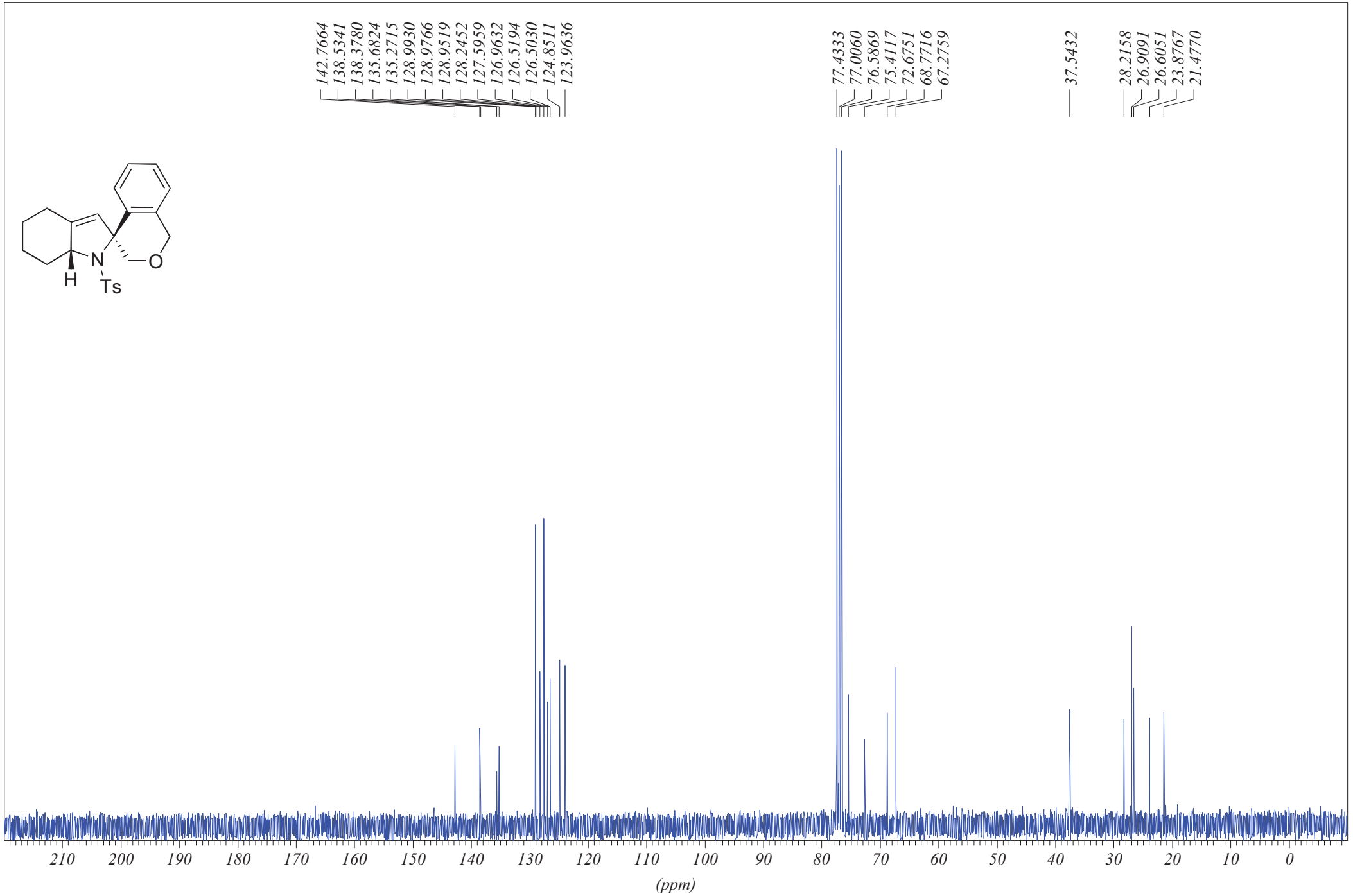

Me
TBSO
N
O
H
Ts
Minor


Me
TBSO
N
O
H
Ts
Minor

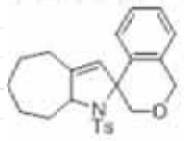

N
O
Ts
Mixture of
diastereoisomers 3:1


N
O
Ts
Mixture of
diastereoisomers 3:1


7.3553
7.3275
7.2600
7.1484
7.1385
7.1218
7.1138
7.0026
6.9772
6.9343
6.9085
6.8843
6.7321
6.7063
5.4830
4.9479
4.8995
4.7243
4.6759
4.6596
4.6226
4.3220
4.3057
4.2870
4.2711
4.0110
3.9744
2.5775
2.5374
2.4615
2.3952
2.0687
2.0544
2.0242
2.0107
1.9801
1.9654
1.8224
1.8141
1.7954
1.5682
1.3847
1.3494
1.3410
1.2977
1.2771
1.2699
1.2545
1.2346
1.2040
1.1937
N
O
H
Ts
Integral
1.9307
2.9229
1.0060
1.0193
1.0000
1.0019
1.0974
2.1472
1.0746
1.1077
1.2160
4.1596
1.2901
2.2483
3.8533
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
(ppm)

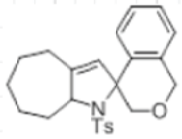

142.7664
138.5341
138.3780
135.6824
135.2715
128.9930
128.9766
128.9519
128.2452
127.5959
126.9632
126.5194
126.5030
124.8511
123.9636
77.4333
77.0060
76.5869
75.4117
72.6751
68.7716
67.2759
37.5432
28.2158
26.9091
26.6051
23.8767
21.4770
N
H
Ts
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

2i



mixture of diastereoisomers 1:1

2i



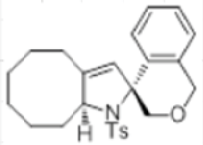
mixture of diastereoisomers 1:1

142,942
142,691
141,589
140,975
138,461
138,432
138,230
137,668
135,567
135,346
133,642
129,235
128,948
128,359
127,922
127,823
127,638
127,547
127,209
126,983
126,950
126,394
124,008
123,645

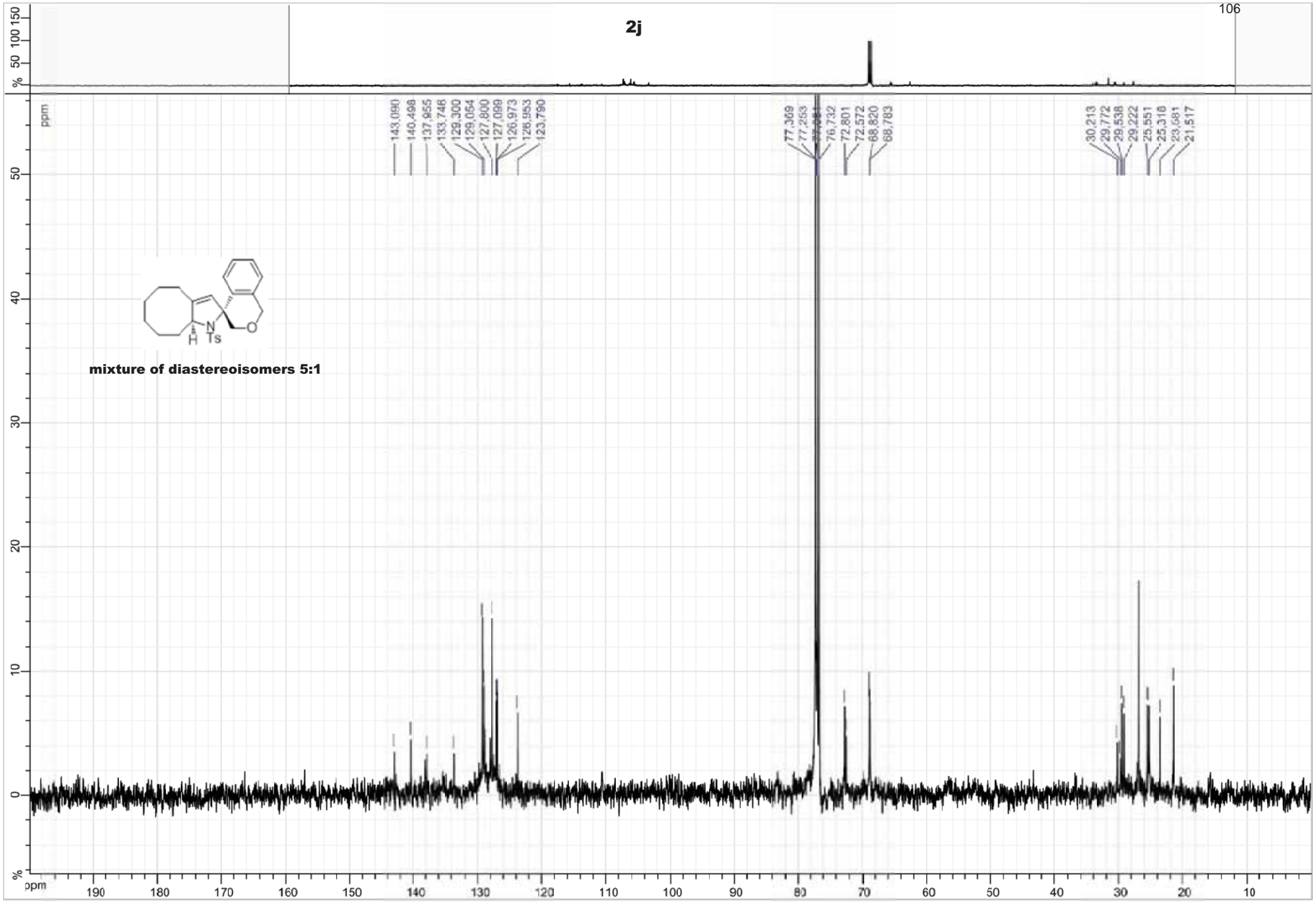
77,368
77,050
76,731
75,303
72,771
72,600
72,293
70,418
70,373
68,722
68,671

35,722
35,334
30,194
29,837
29,687
28,055
27,868
27,777
26,699
25,296
24,674
21,465

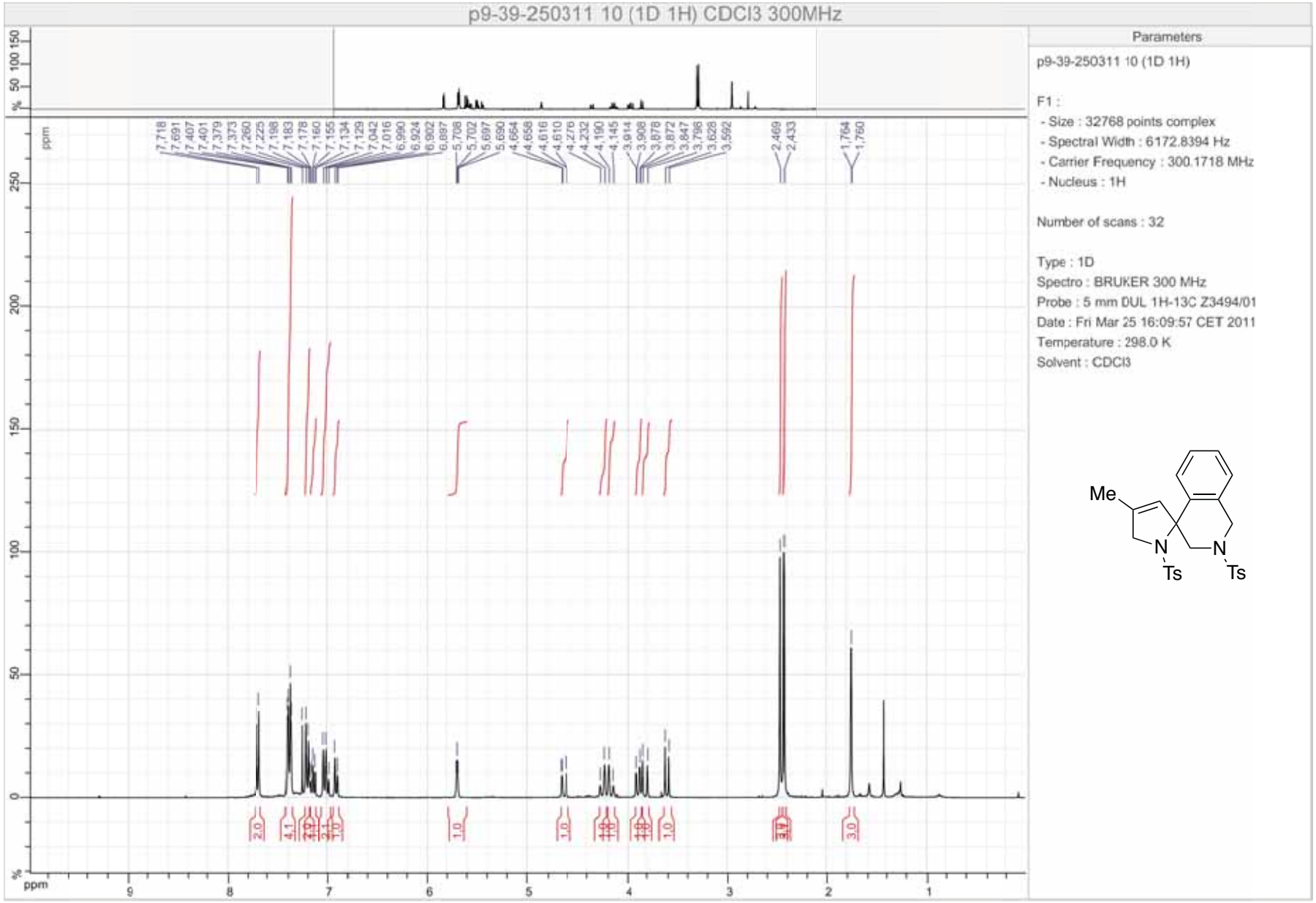
2j



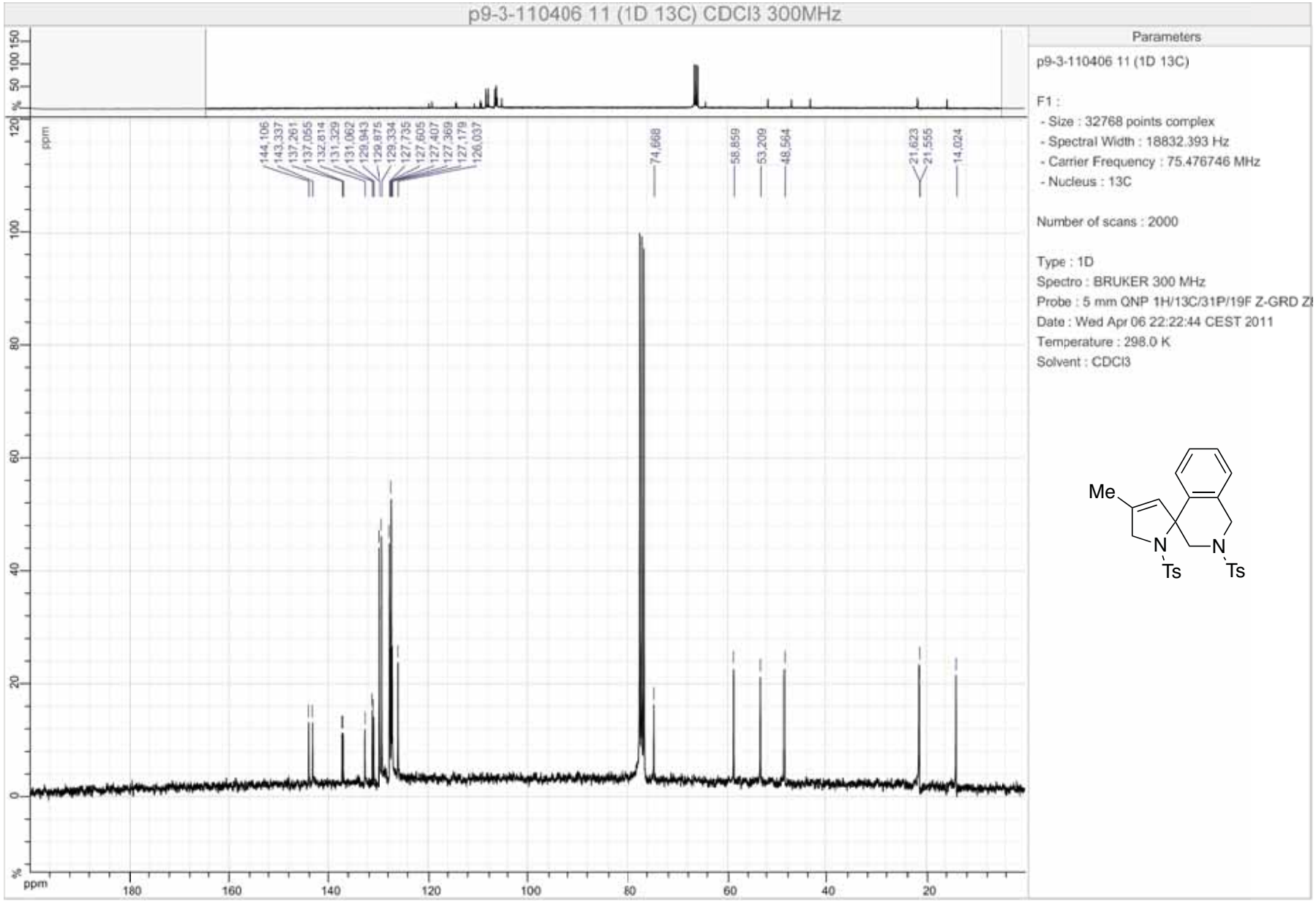
mixture of diastereoisomers 5:1


2j
106
143,090
140,498
137,955
133,746
129,300
129,054
127,800
127,099
126,973
126,953
123,790
77,369
77,253
76,732
72,801
72,572
68,820
68,783
30,213
29,772
29,538
29,222
25,551
25,316
23,681
21,517
ppm
mixture of diastereoisomers 5:1

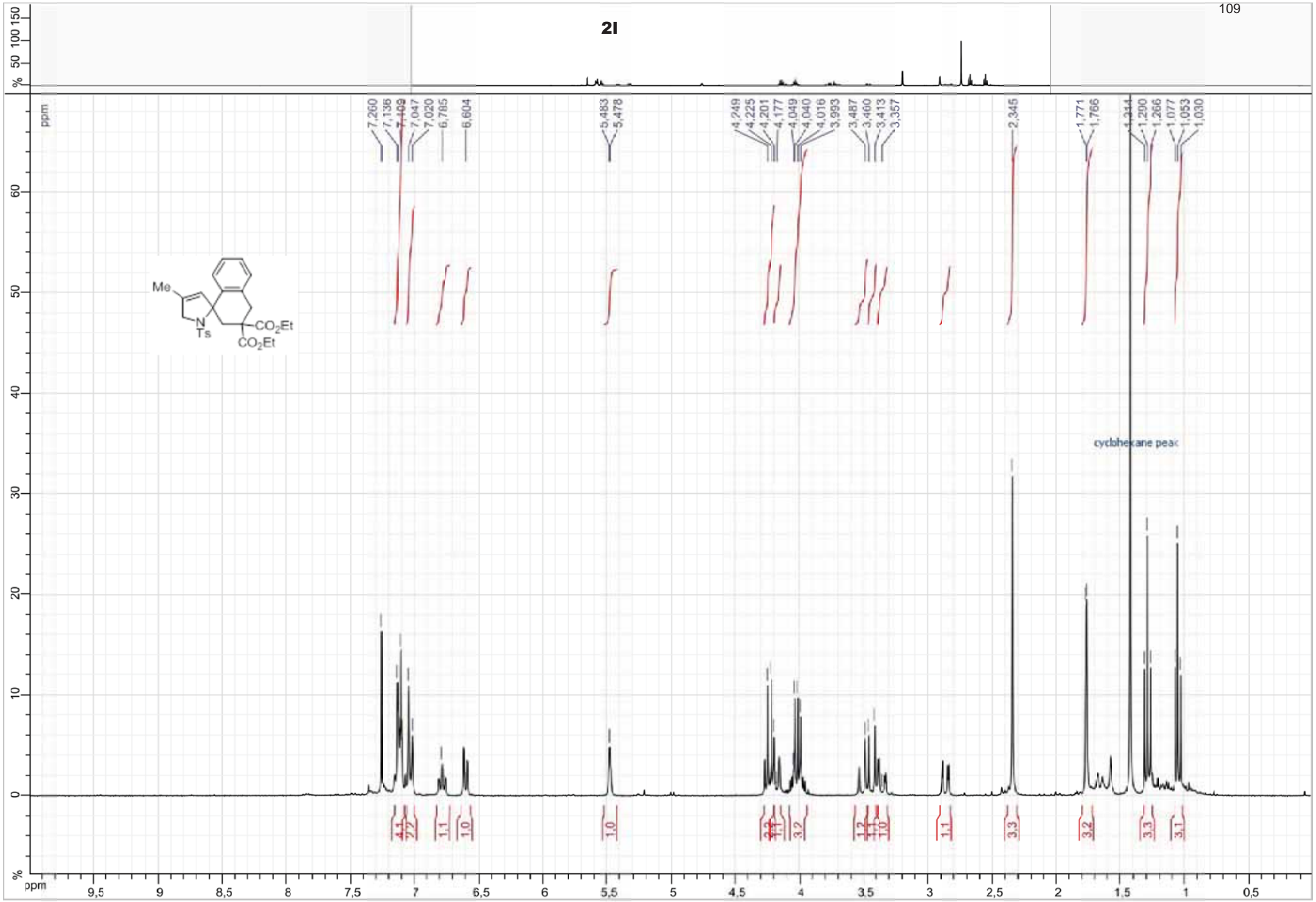
2k

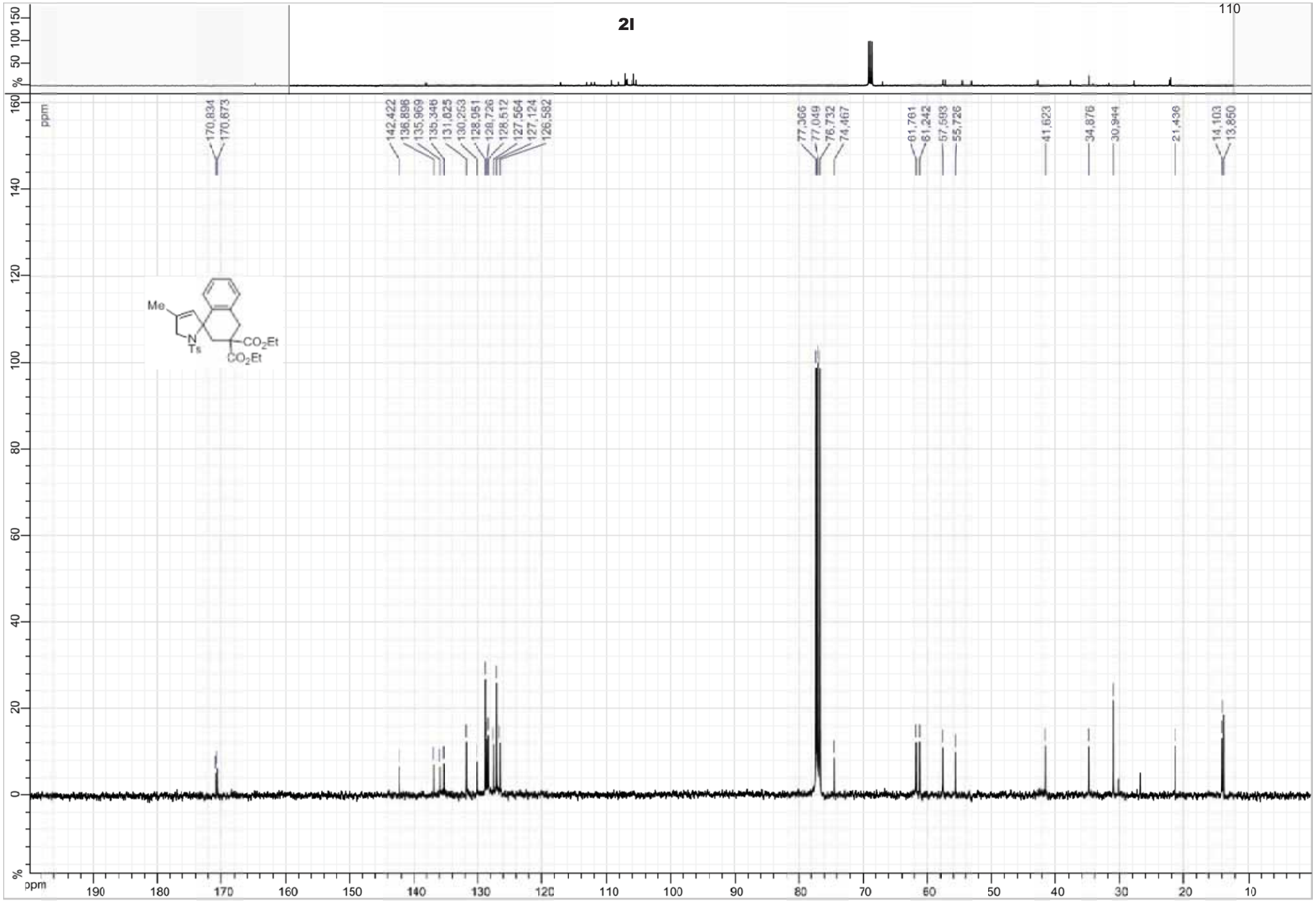

p9-39-250311 10 (1D 1H) CDCl3 300MHz
Parameters
p9-39-250311 10 (1D 1H)
F1 :
- Size : 32768 points complex
- Spectral Width : 6172.8394 Hz
- Carrier Frequency : 300.1718 MHz
- Nucleus : 1H
Number of scans : 32
Type : 1D
Spectro : BRUKER 300 MHz
Probe : 5 mm DUL 1H-13C Z3494/01
Date : Fri Mar 25 16:09:57 CET 2011
Temperature : 298.0 K
Solvent : CDCl3
Me
N
Ts
N
Ts

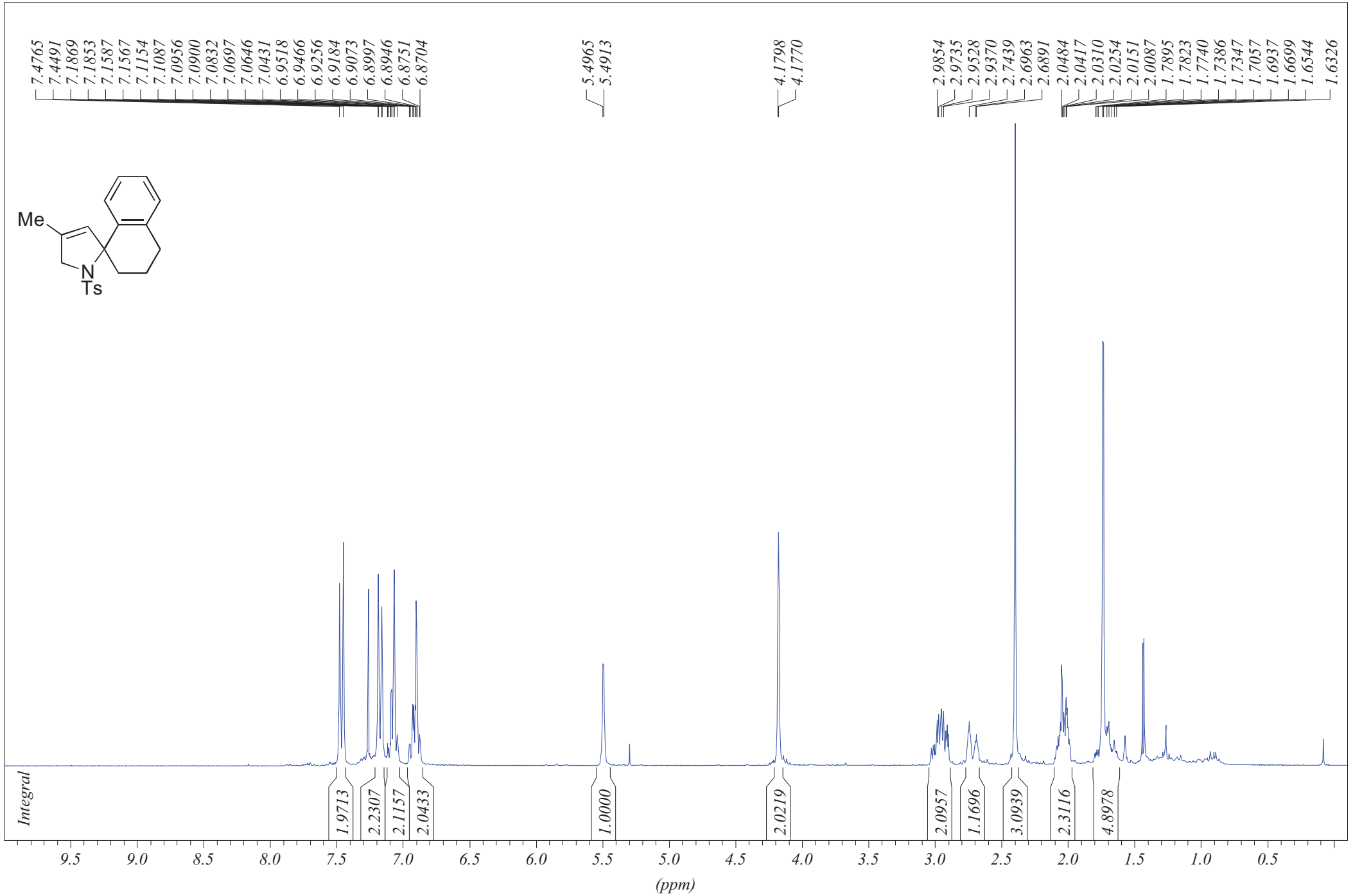
2k

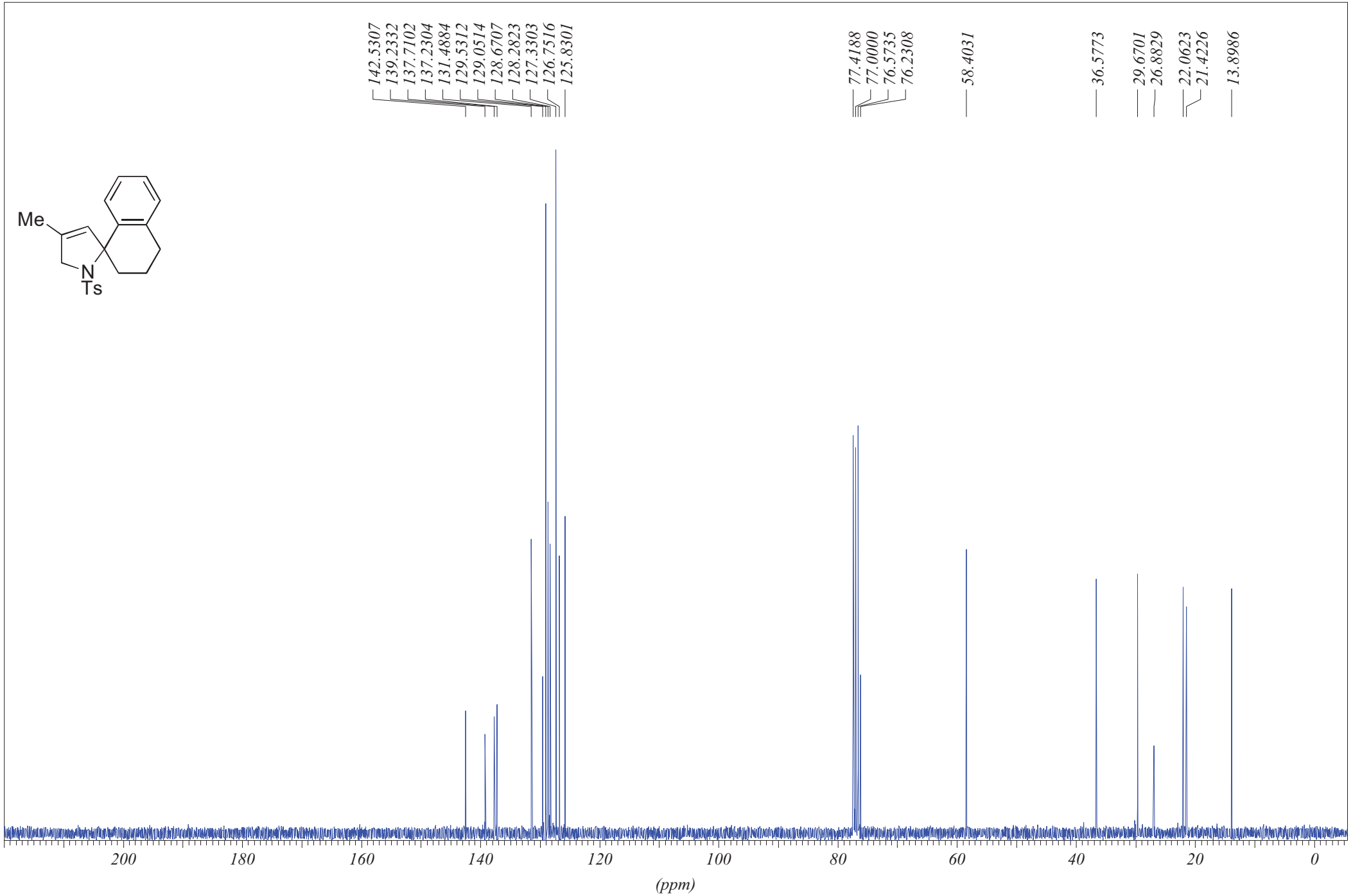

p9-3-110406 11 (1D 13C) CDCl3 300MHz
Parameters
p9-3-110406 11 (1D 13C)
F1 :
- Size : 32768 points complex
- Spectral Width : 18832.393 Hz
- Carrier Frequency : 75.476746 MHz
- Nucleus : 13C
Number of scans : 2000
Type : 1D
Spectro : BRUKER 300 MHz
Probe : 5 mm QNP 1H/13C/31P/19F Z-GRD Z
Date : Wed Apr 06 22:22:44 CEST 2011
Temperature : 298.0 K
Solvent : CDCl3
144,106
143,337
137,261
137,055
132,814
131,329
131,062
129,943
129,875
129,334
127,735
127,605
127,407
127,369
127,179
126,037
74,668
58,859
53,209
48,564
21,623
21,555
14,024
Me
N
Ts
N
Ts
ppm
180
160
140
120
100
80
60
40
20

2l

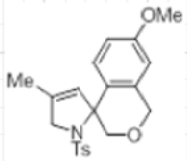

ppm
7,260
7,136
7,109
7,047
7,020
6,785
6,604
5,483
5,478
4,249
4,225
4,201
4,177
4,049
4,040
4,016
3,993
3,487
3,460
3,413
3,357
2,345
1,771
1,766
1,344
1,290
1,266
1,077
1,053
1,030
Me
N
Ts
CO2Et
CO2Et
cyclohexane peak
4,1
7,2
1,1
1,0
1,0
2,2
1,1
3,2
1,2
1,1
1,0
1,1
3,3
3,2
3,3
3,1
ppm
9,5
9
8,5
8
7,5
7
6,5
6
5,5
5
4,5
4
3,5
3
2,5
2
1,5
1
0,5


2l
110
ppm
170,834
170,673
142,422
136,896
135,969
135,346
131,825
130,253
128,951
128,726
128,512
127,564
127,124
126,582
77,366
77,049
76,732
74,467
61,761
61,242
57,593
55,726
41,623
34,876
30,944
21,436
14,103
13,850
Me
N
Ts
CO2Et
CO2Et


7.4765
7.4491
7.1869
7.1853
7.1587
7.1567
7.1154
7.1087
7.0956
7.0900
7.0832
7.0697
7.0646
7.0431
6.9518
6.9466
6.9256
6.9184
6.9073
6.8997
6.8946
6.8751
6.8704
5.4965
5.4913
4.1798
4.1770
2.9854
2.9735
2.9528
2.9370
2.7439
2.6963
2.6891
2.0484
2.0417
2.0310
2.0254
2.0151
2.0087
1.7895
1.7823
1.7740
1.7386
1.7347
1.7057
1.6937
1.6699
1.6544
1.6326
Me
N
Ts
Integral
1.9713
2.2307
2.1157
2.0433
1.0000
2.0219
2.0957
1.1696
3.0939
2.3116
4.8978
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
(ppm)

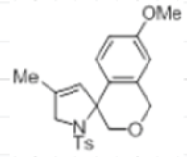

142.5307
139.2332
137.7102
137.2304
131.4884
129.5312
129.0514
128.6707
128.2823
127.3303
126.7516
125.8301
77.4188
77.0000
76.5735
76.2308
58.4031
36.5773
29.6701
26.8829
22.0623
21.4226
13.8986
Me
N
Ts
200
180
160
140
120
100
80
60
40
20
0
(ppm)

2o



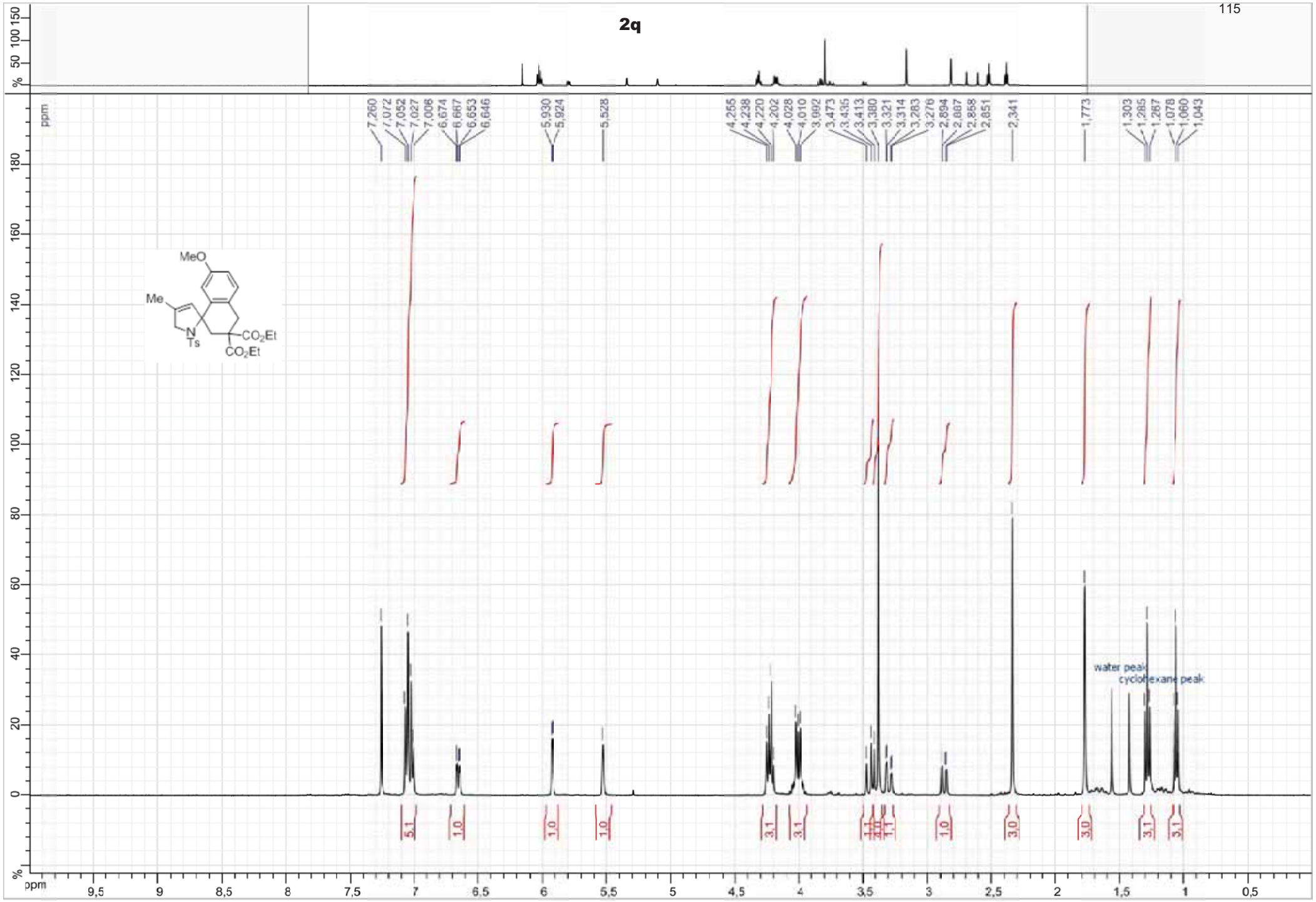
major regioisomer (mixture)

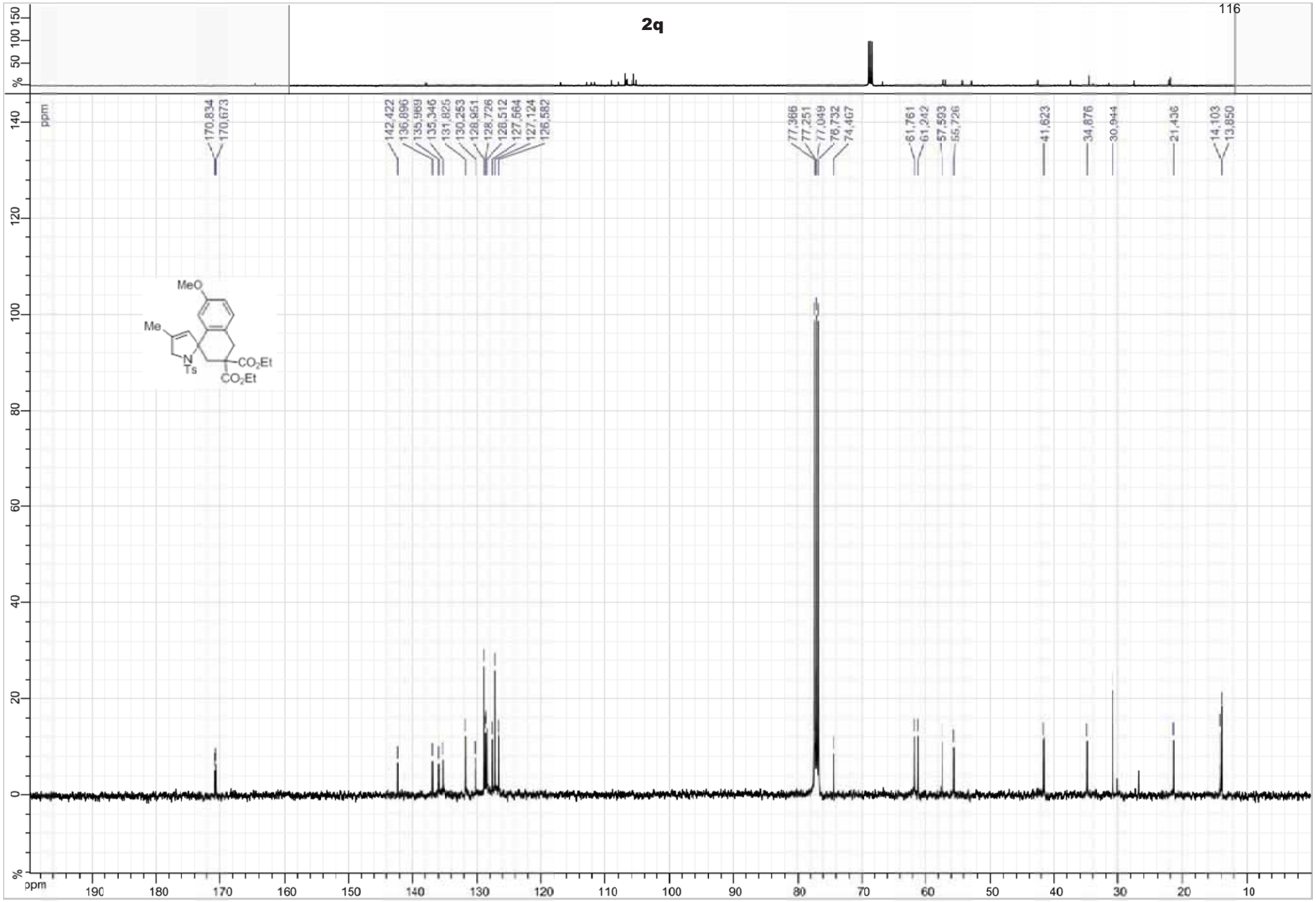
2o



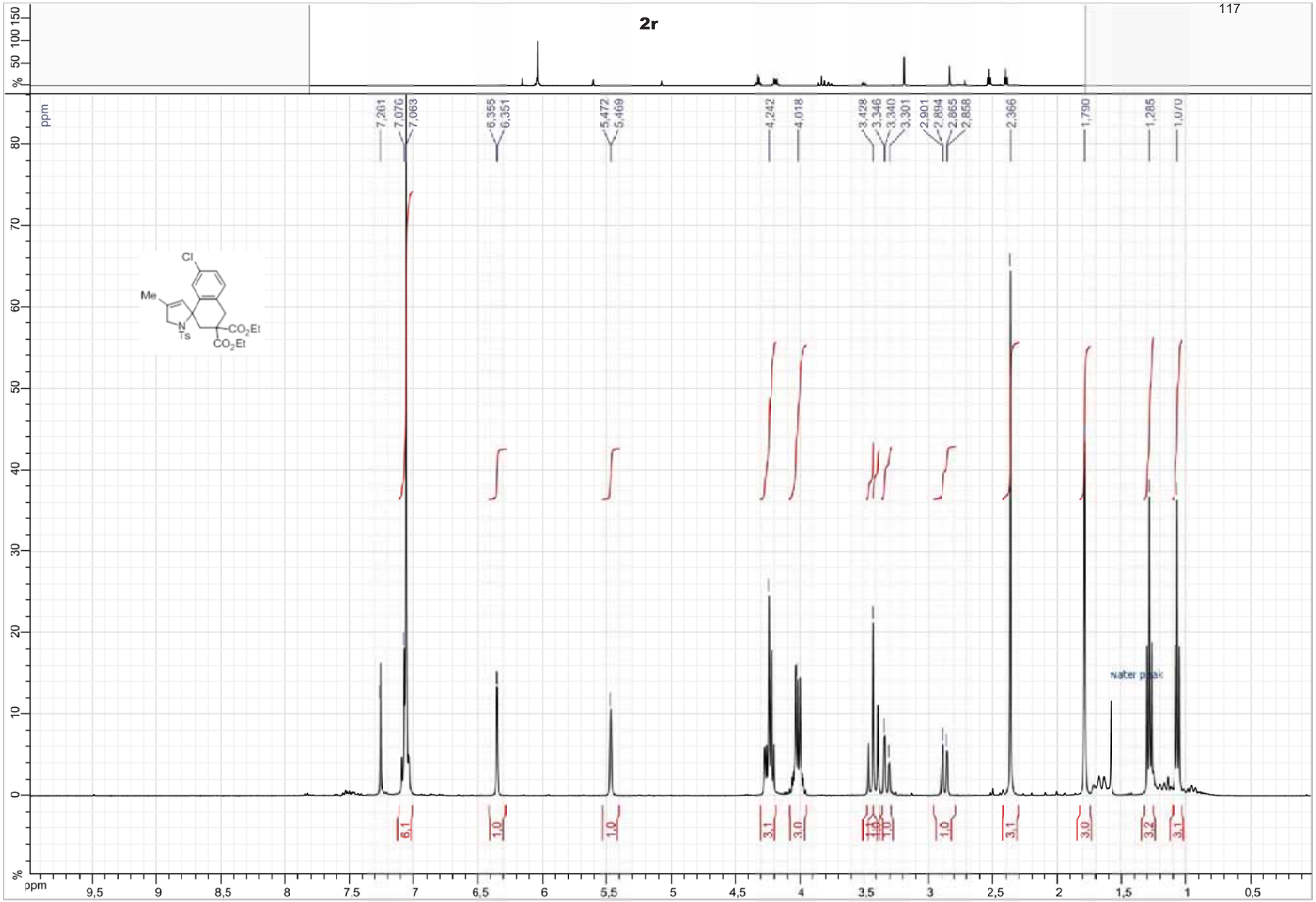
major regioisomer (mixture)

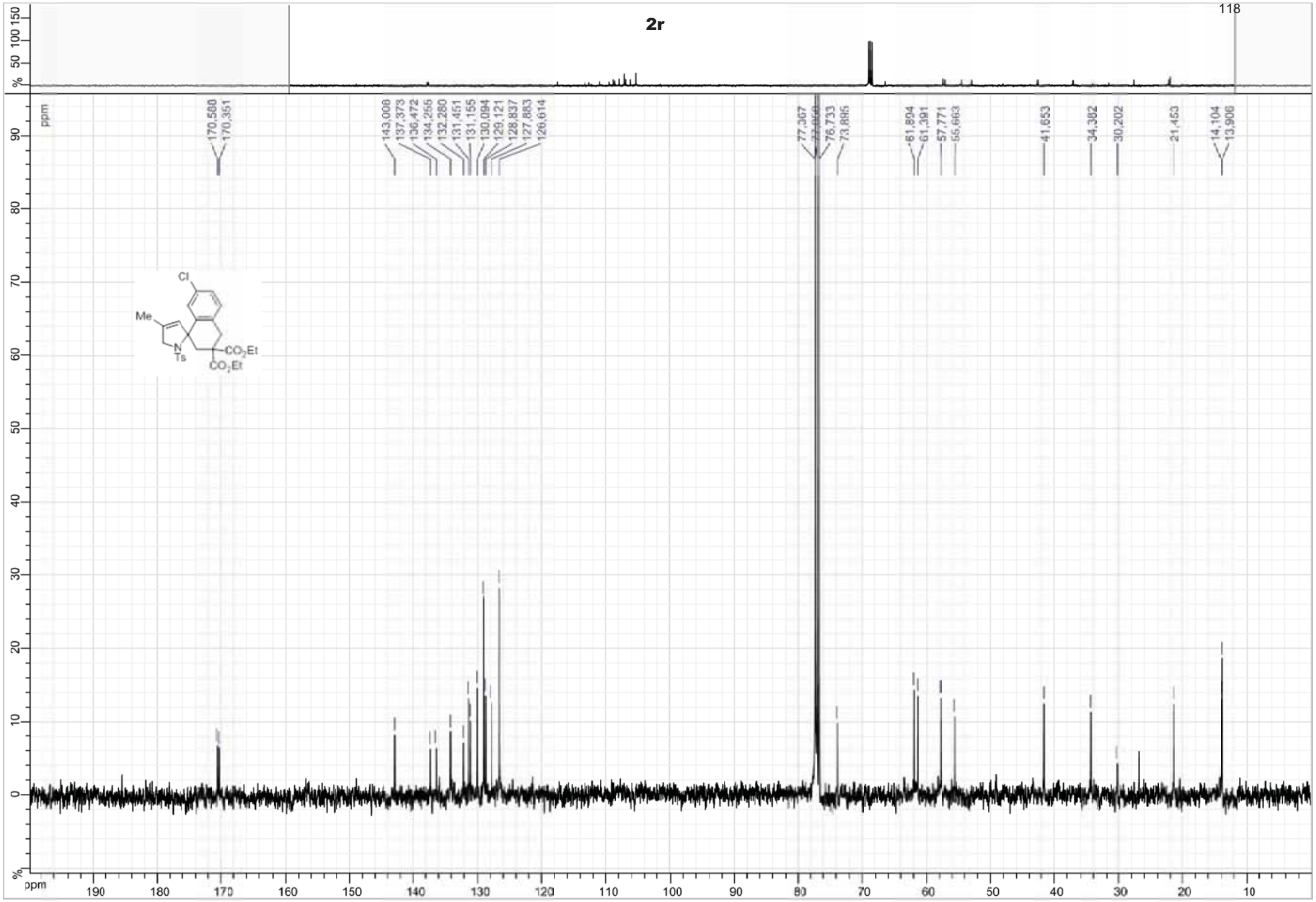
2q


MeO
Me
N
Ts
CO2Et
CO2Et
ppm
7,260
7,072
7,052
7,027
7,006
6,674
6,667
6,653
6,646
5,930
5,924
5,528
4,255
4,238
4,220
4,202
4,028
4,010
3,992
3,473
3,435
3,413
3,380
3,321
3,314
3,283
3,276
2,894
2,887
2,858
2,851
2,341
1,773
1,303
1,285
1,267
1,078
1,060
1,043
water peak
cyclohexane peak
5,1
1,0
1,0
1,0
3,1
3,1
1,1
4,0
1,1
1,0
3,0
3,0
3,1
3,1

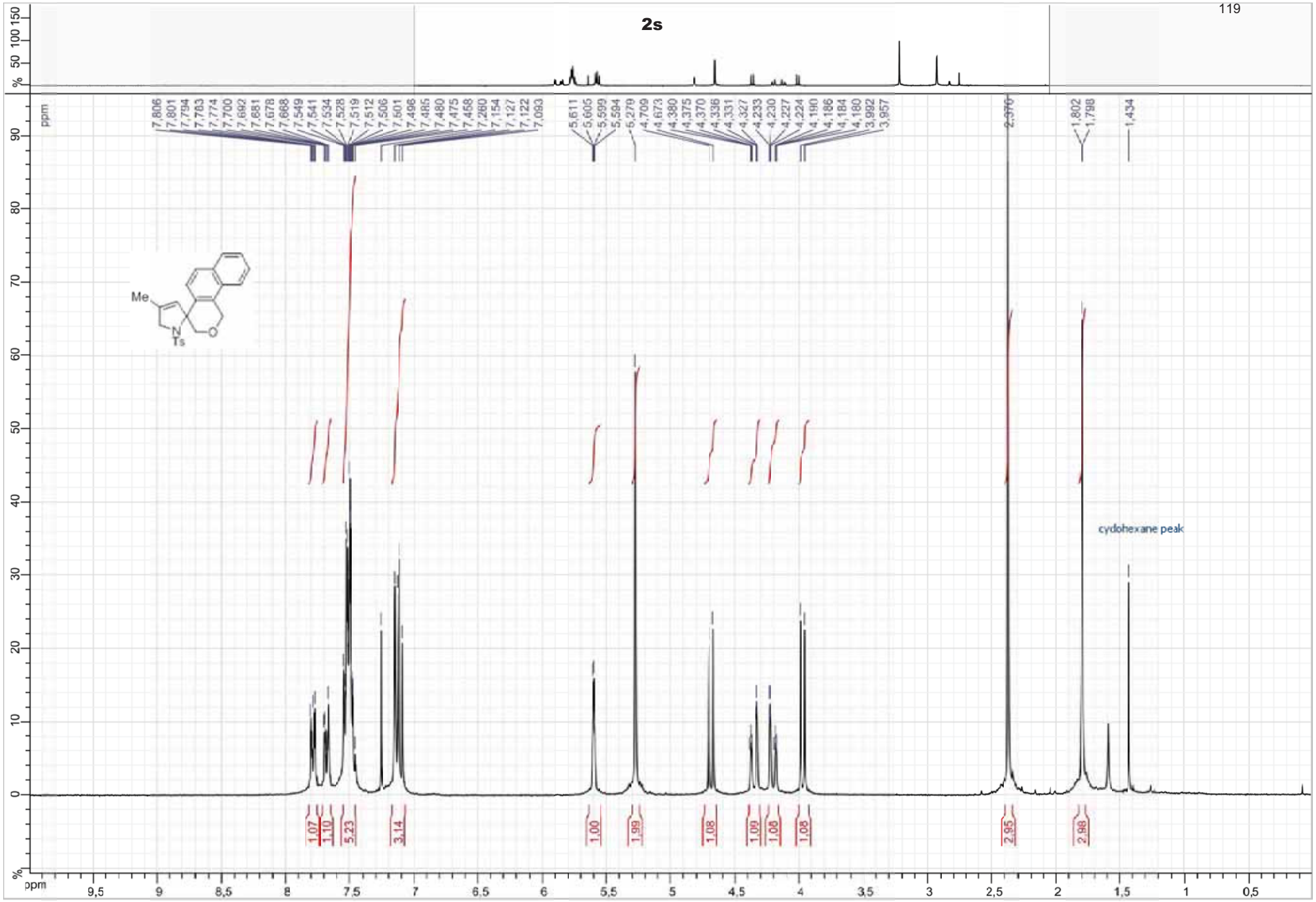

2q
116
170,834
170,673
142,422
136,896
135,969
135,346
131,825
130,253
128,951
128,726
128,512
127,564
127,124
126,582
77,366
77,251
77,049
76,732
74,467
61,761
61,242
57,593
55,726
41,623
34,876
30,944
21,436
14,103
13,850
MeO
Me
N
Ts
CO2Et
CO2Et
ppm

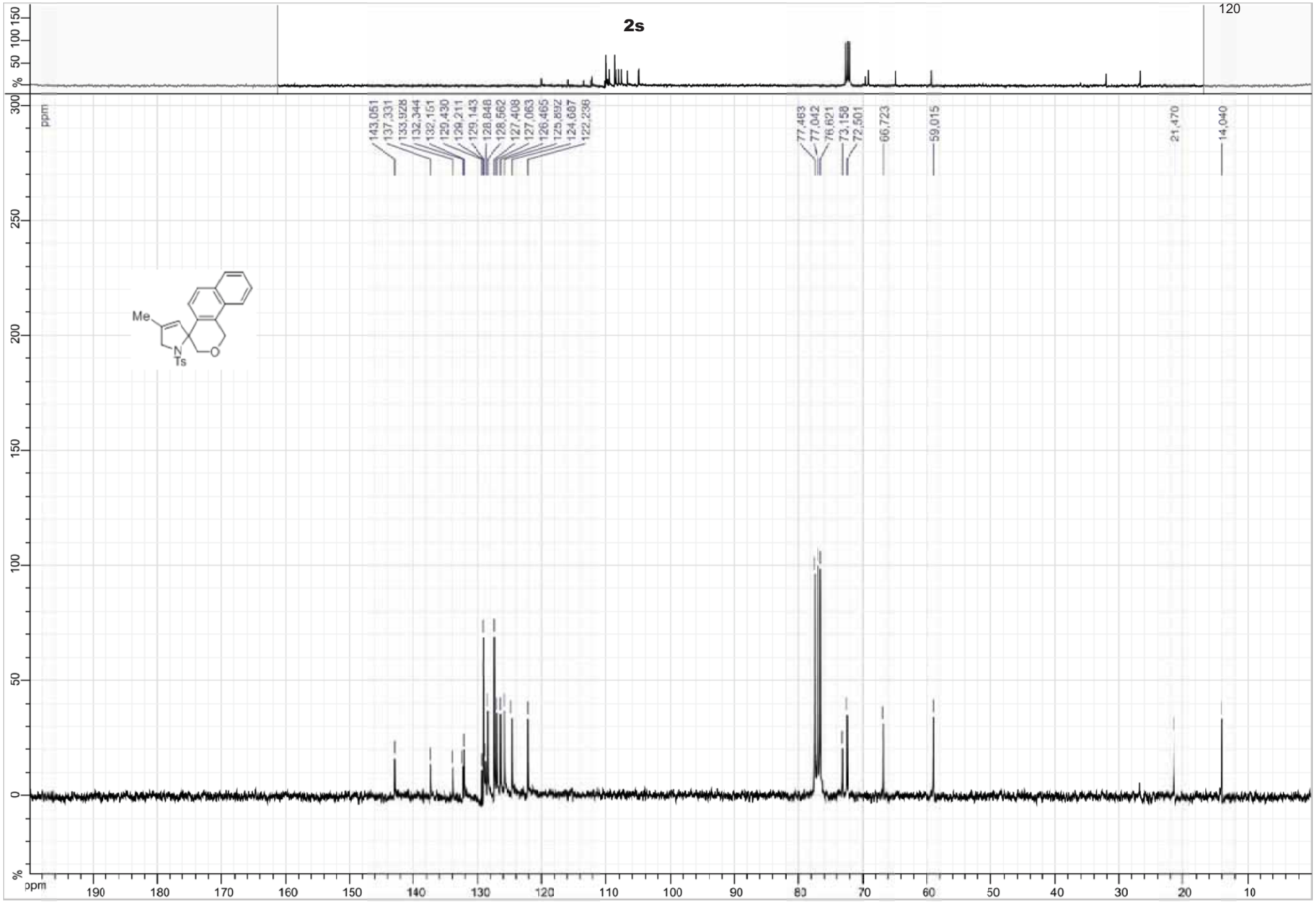
2r

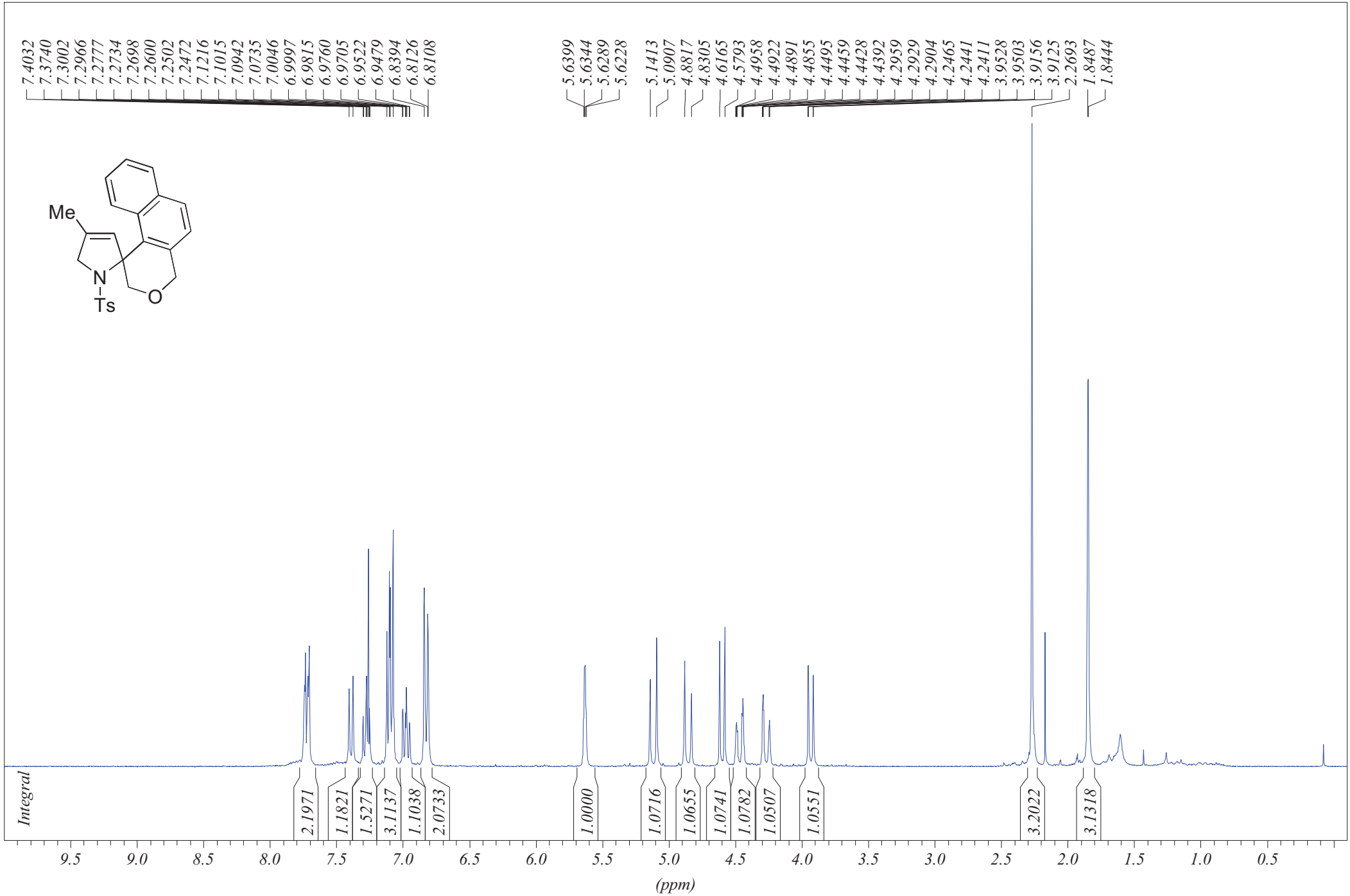

ppm
7,261
7,076
7,063
6,355
6,351
5,472
5,469
4,242
4,018
3,428
3,346
3,340
3,301
2,901
2,894
2,865
2,858
2,366
1,790
1,285
1,070
Cl
Me
N
Ts
CO2Et
CO2Et
water peak
6,1
1,0
1,0
3,1
3,0
1,0
1,0
1,0
3,1
3,0
3,2
3,1

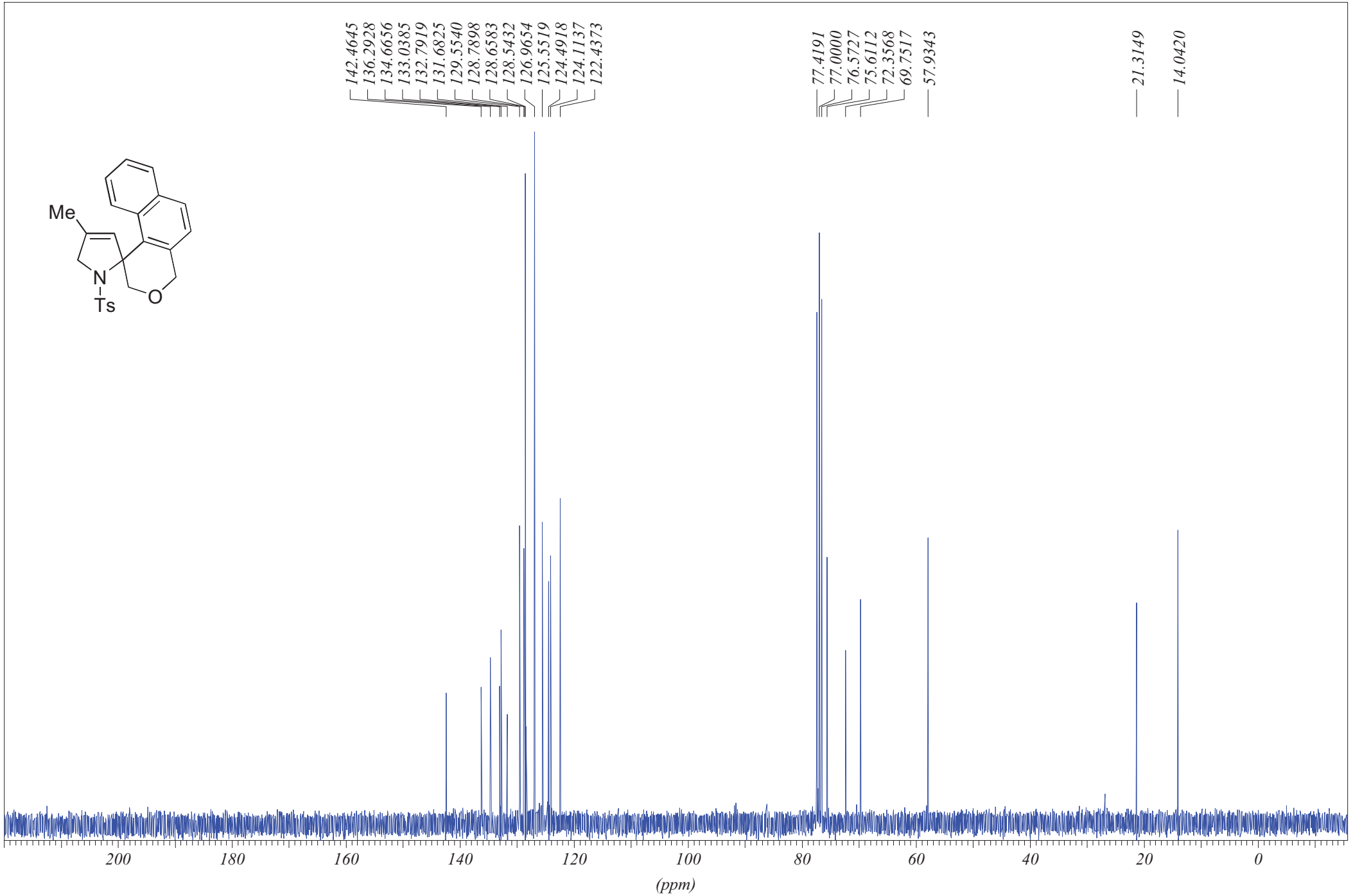

2r
118
ppm
170,588
170,351
143,008
137,373
136,472
134,255
132,280
131,451
131,155
130,094
129,121
128,837
127,883
126,614
77,367
76,733
73,895
61,894
61,391
57,771
56,663
41,653
34,382
30,202
21,453
14,104
13,906
Cl
Me
N
Ts
CO2Et
CO2Et

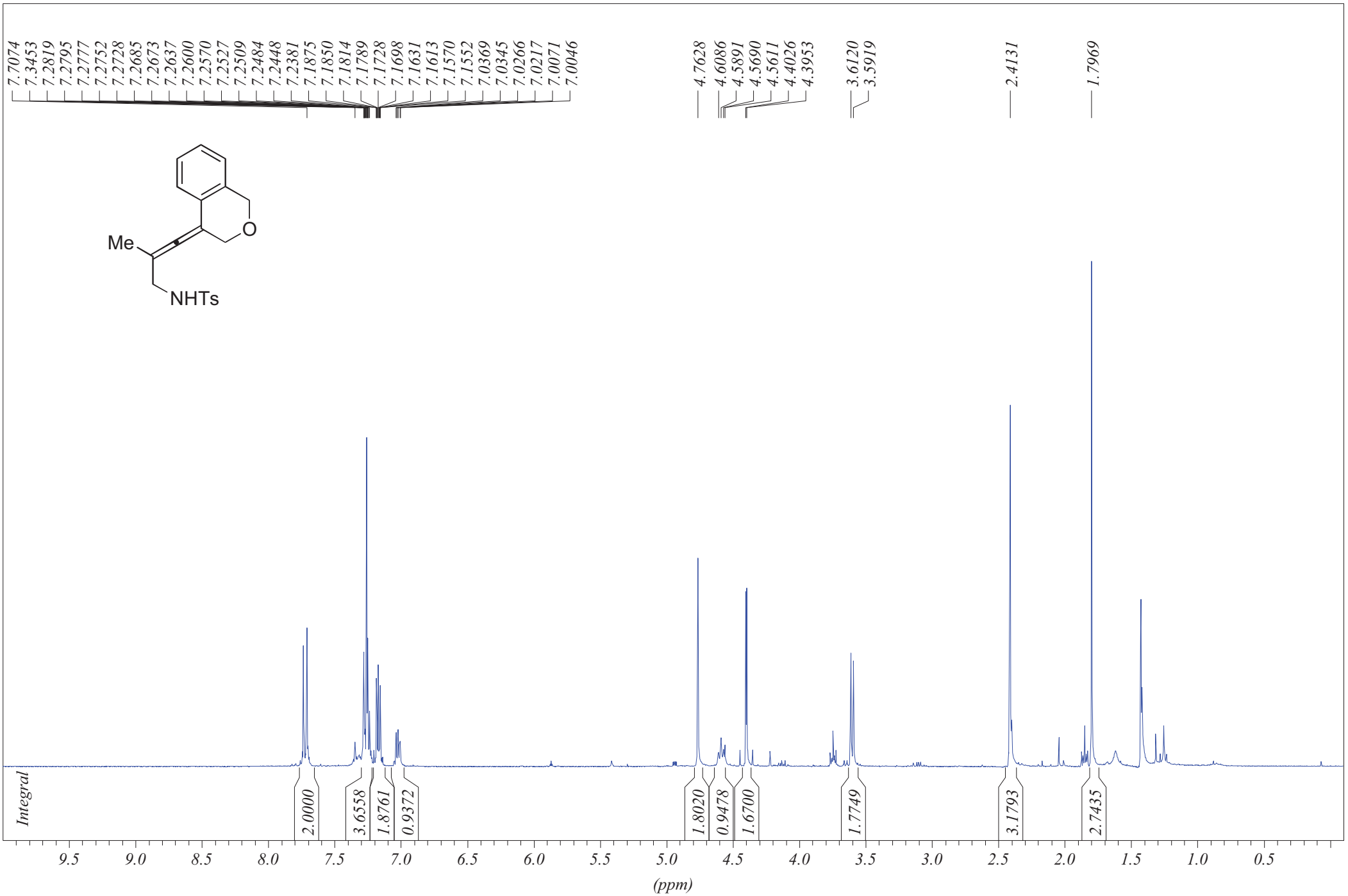
2s

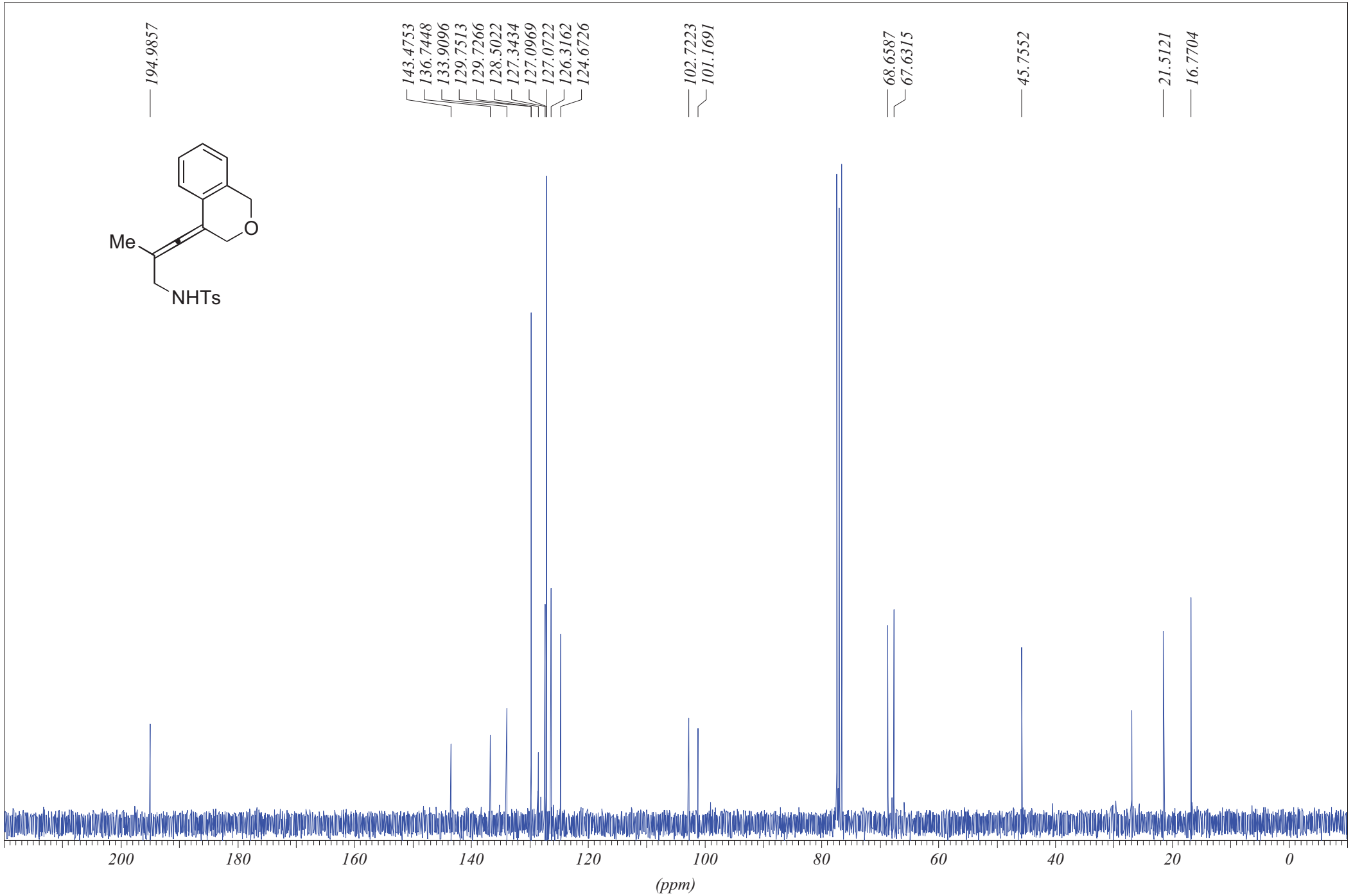

7,806
7,801
7,794
7,783
7,774
7,700
7,692
7,681
7,678
7,668
7,549
7,541
7,534
7,528
7,519
7,512
7,506
7,501
7,496
7,485
7,480
7,475
7,458
7,260
7,154
7,127
7,122
7,093
5,611
5,605
5,599
5,594
5,279
4,709
4,673
4,380
4,375
4,370
4,336
4,331
4,327
4,233
4,230
4,227
4,224
4,190
4,186
4,184
4,180
3,992
3,957
2,376
1,802
1,798
1,434
Me
N
Ts
O
cydohexane peak
1,07
1,10
5,23
3,14
1,00
1,99
1,08
1,09
1,06
1,08
2,95
2,98
ppm


2s
120
ppm
143,051
137,331
133,928
132,344
132,151
129,430
129,211
129,143
128,848
128,562
127,408
127,063
126,465
125,892
124,687
122,236
77,463
77,042
76,621
73,158
72,501
66,723
59,015
21,470
14,040
Me
N
Ts
O

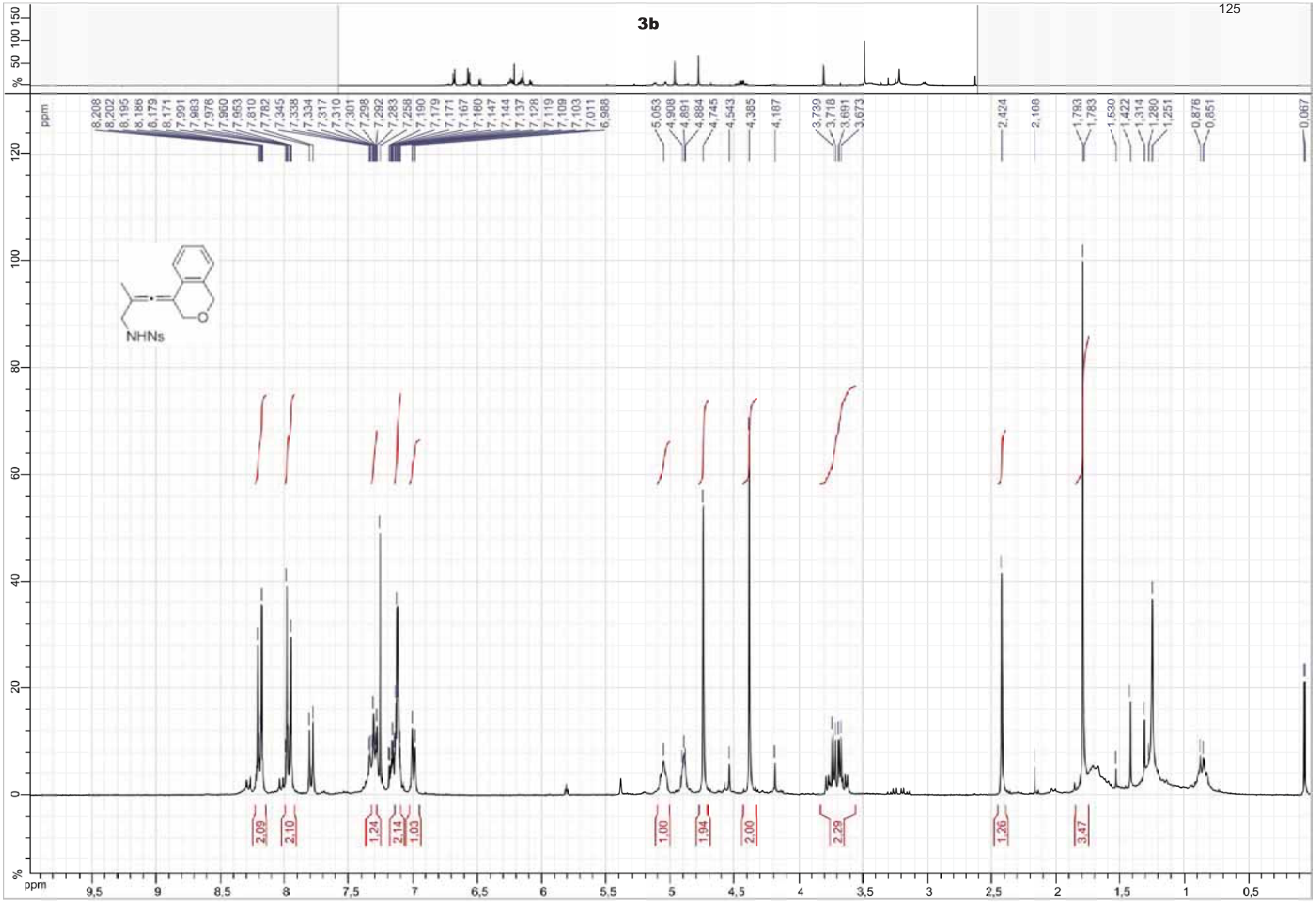

7.4032
7.3740
7.3002
7.2966
7.2777
7.2734
7.2698
7.2600
7.2502
7.2472
7.1216
7.1015
7.0942
7.0735
7.0046
6.9997
6.9815
6.9760
6.9705
6.9522
6.9479
6.8394
6.8126
6.8108
5.6399
5.6344
5.6289
5.6228
5.1413
5.0907
4.8817
4.8305
4.6165
4.5793
4.4958
4.4922
4.4891
4.4855
4.4495
4.4459
4.4428
4.4392
4.2959
4.2929
4.2904
4.2465
4.2441
4.2411
3.9528
3.9503
3.9156
3.9125
2.2693
1.8487
1.8444
Me
N
Ts
O
Integral
2.1971
1.1821
1.5271
3.1137
1.1038
2.0733
1.0000
1.0716
1.0655
1.0741
1.0782
1.0507
1.0551
3.2022
3.1318
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
(ppm)

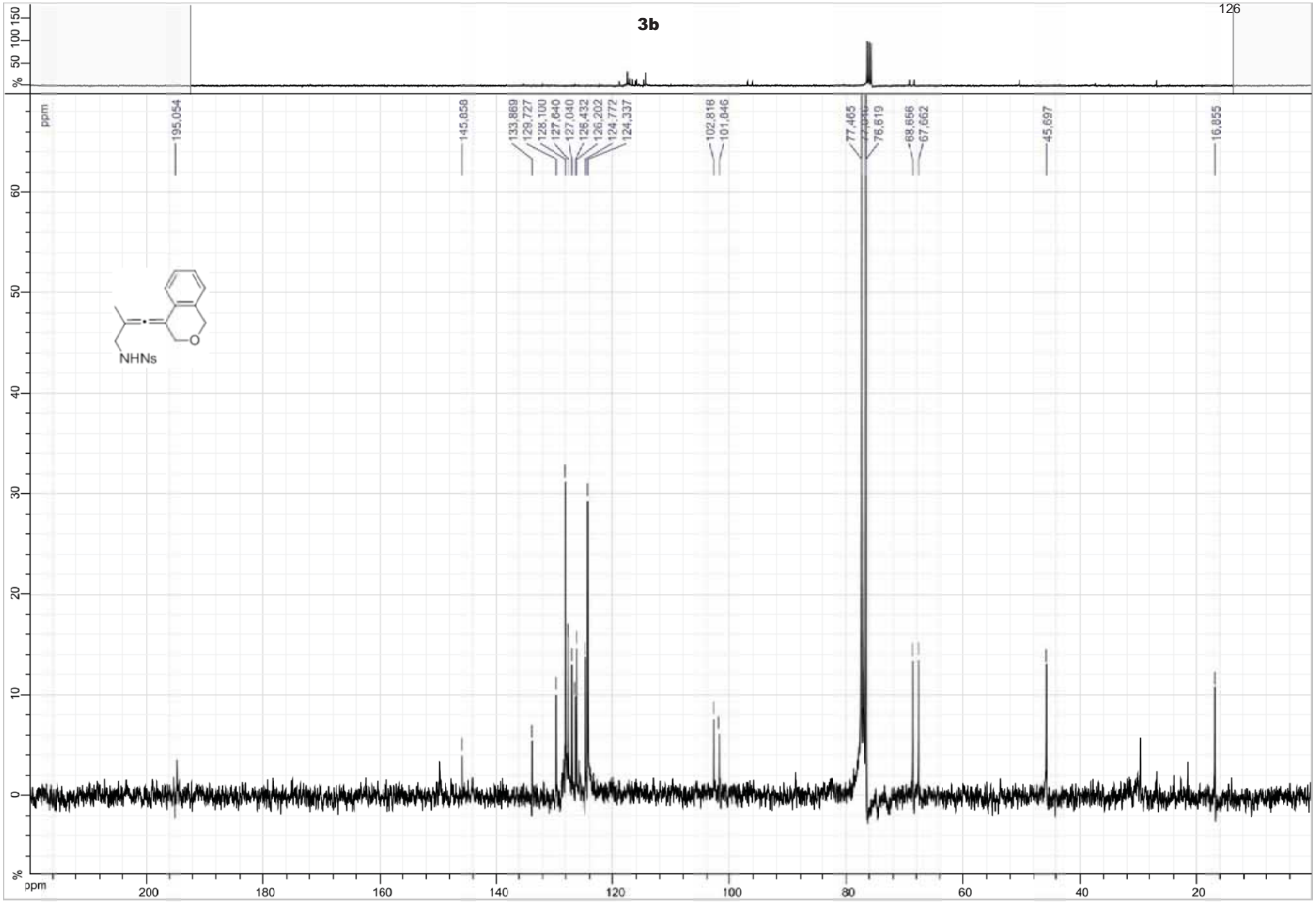

142.4645
136.2928
134.6656
133.0385
132.7919
131.6825
129.5540
128.7898
128.6583
128.5432
126.9654
125.5519
124.4918
124.1137
122.4373
77.4191
77.0000
76.5727
75.6112
72.3568
69.7517
57.9343
21.3149
14.0420
Me
N
Ts
O
200
180
160
140
120
100
80
60
40
20
0
(ppm)


7.7074
7.3453
7.2819
7.2795
7.2777
7.2752
7.2728
7.2685
7.2673
7.2637
7.2600
7.2570
7.2527
7.2509
7.2484
7.2448
7.2381
7.1875
7.1850
7.1814
7.1789
7.1728
7.1698
7.1631
7.1613
7.1570
7.1552
7.0369
7.0345
7.0266
7.0217
7.0071
7.0046
4.7628
4.6086
4.5891
4.5690
4.5611
4.4026
4.3953
3.6120
3.5919
2.4131
1.7969
Me
O
NHTs
Integral
2.0000
3.6558
1.8761
0.9372
1.8020
0.9478
1.6700
1.7749
3.1793
2.7435
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
(ppm)

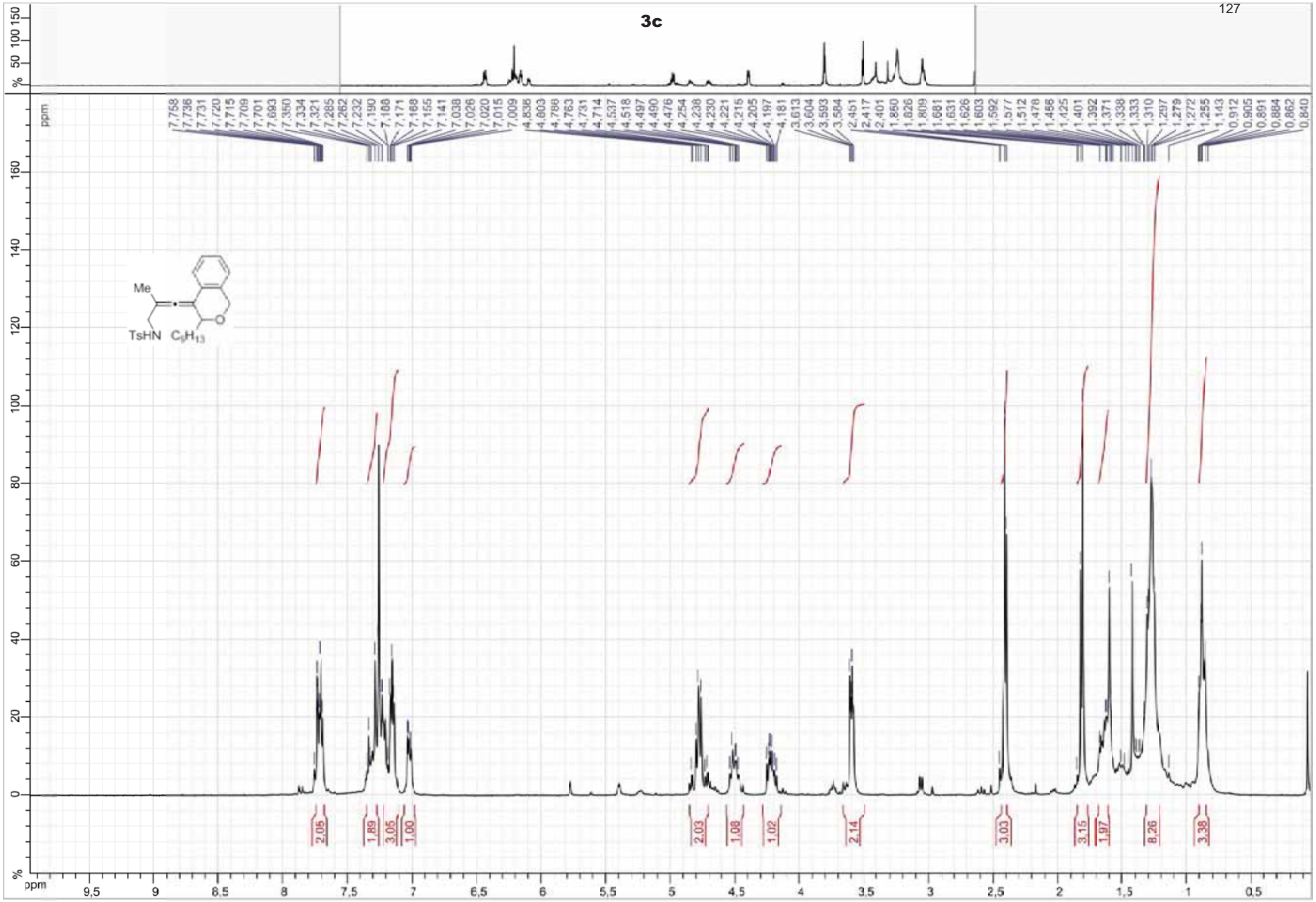

194.9857
143.4753
136.7448
133.9096
129.7513
129.7266
128.5022
127.3434
127.0969
127.0722
126.3162
124.6726
102.7223
101.1691
68.6587
67.6315
45.7552
21.5121
16.7704
Me
O
NHTs
200
180
160
140
120
100
80
60
40
20
0
(ppm)

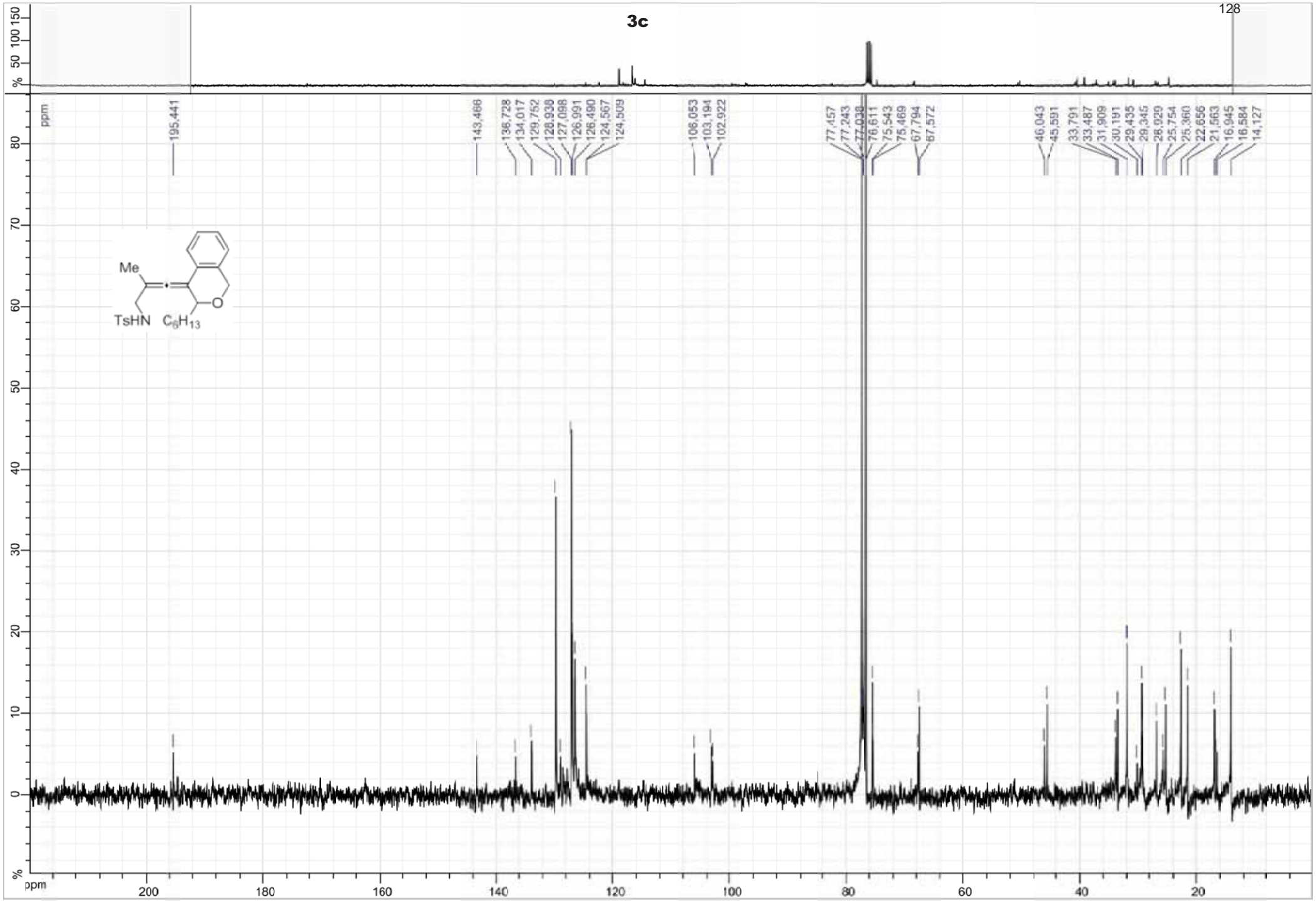
3b

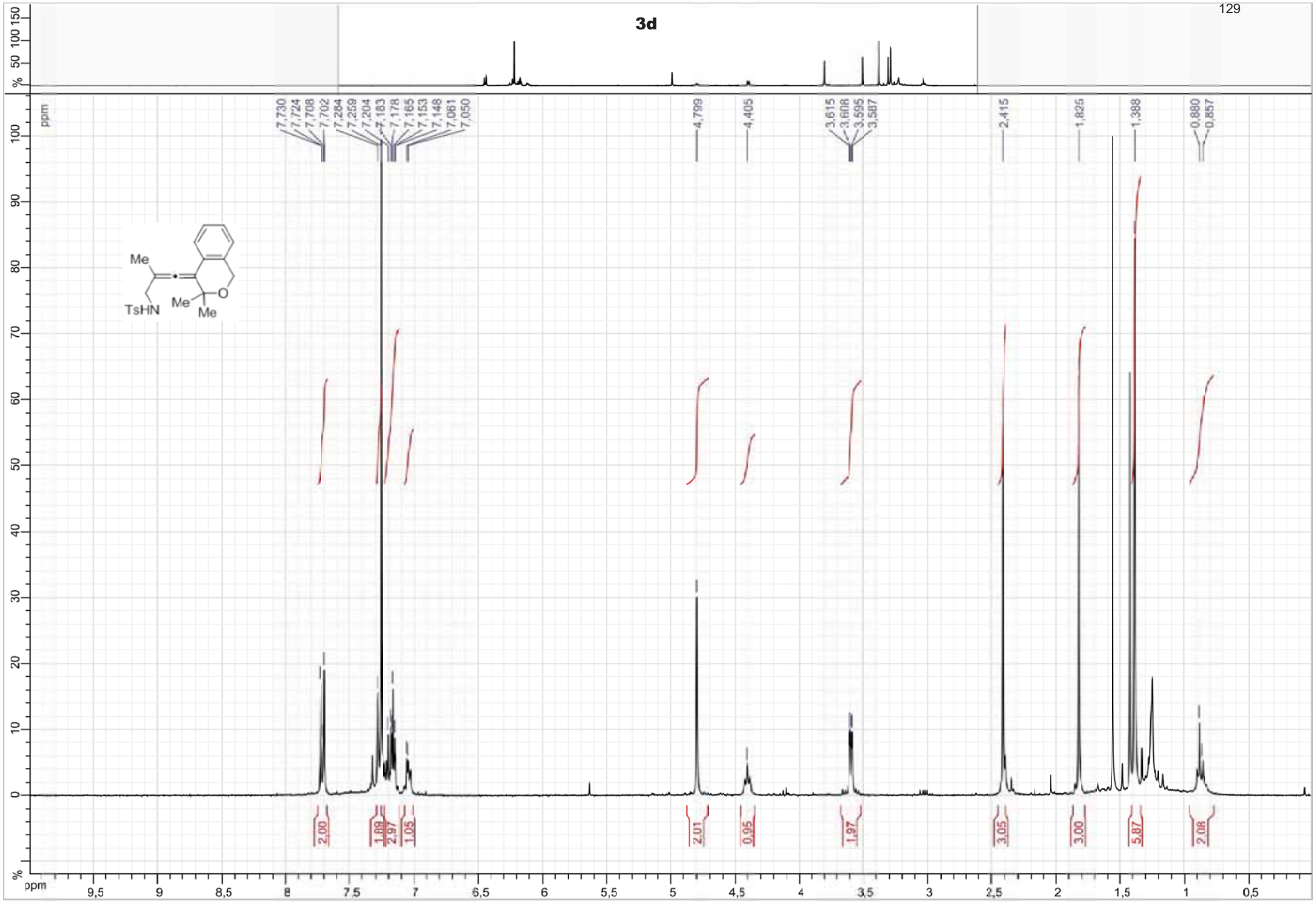

8,208
8,202
8,195
8,186
8,179
8,171
7,991
7,983
7,976
7,960
7,953
7,810
7,782
7,345
7,338
7,334
7,317
7,310
7,301
7,298
7,292
7,283
7,258
7,190
7,179
7,171
7,167
7,160
7,147
7,144
7,137
7,128
7,119
7,109
7,103
7,011
6,988
5,053
4,908
4,891
4,884
4,745
4,543
4,385
4,187
3,739
3,718
3,691
3,673
2,424
2,166
1,793
1,783
1,530
1,422
1,314
1,280
1,251
0,876
0,851
0,067
NHNs
2,09
2,10
1,24
2,14
1,03
1,00
1,94
2,00
2,29
1,26
3,47
ppm

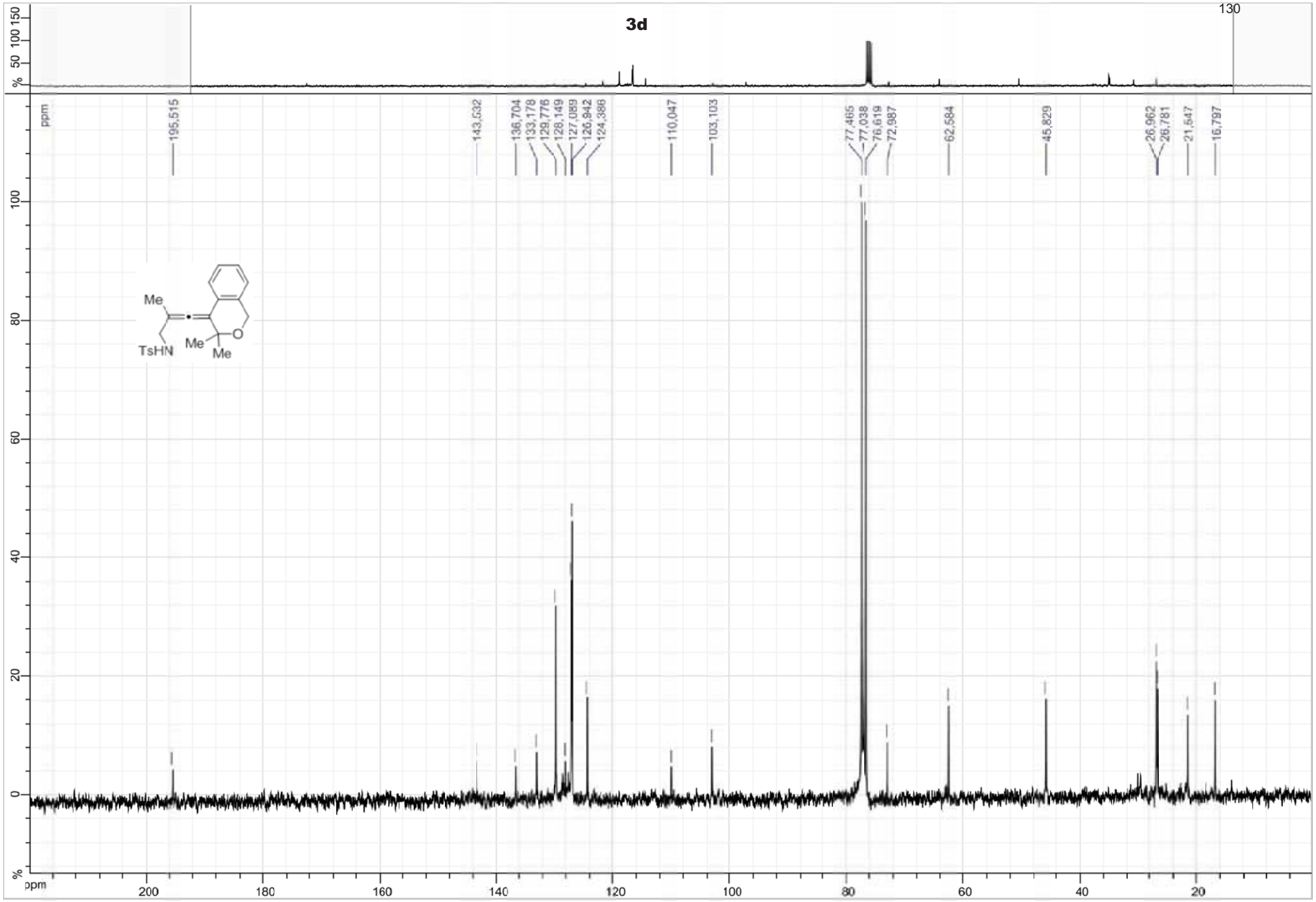

3b
126
ppm
195,054
145,858
133,869
129,727
128,100
127,640
127,040
126,432
126,202
124,772
124,337
102,818
101,646
77,465
76,819
68,656
67,662
45,697
16,855
NHNs
200
180
160
140
120
100
80
60
40
20

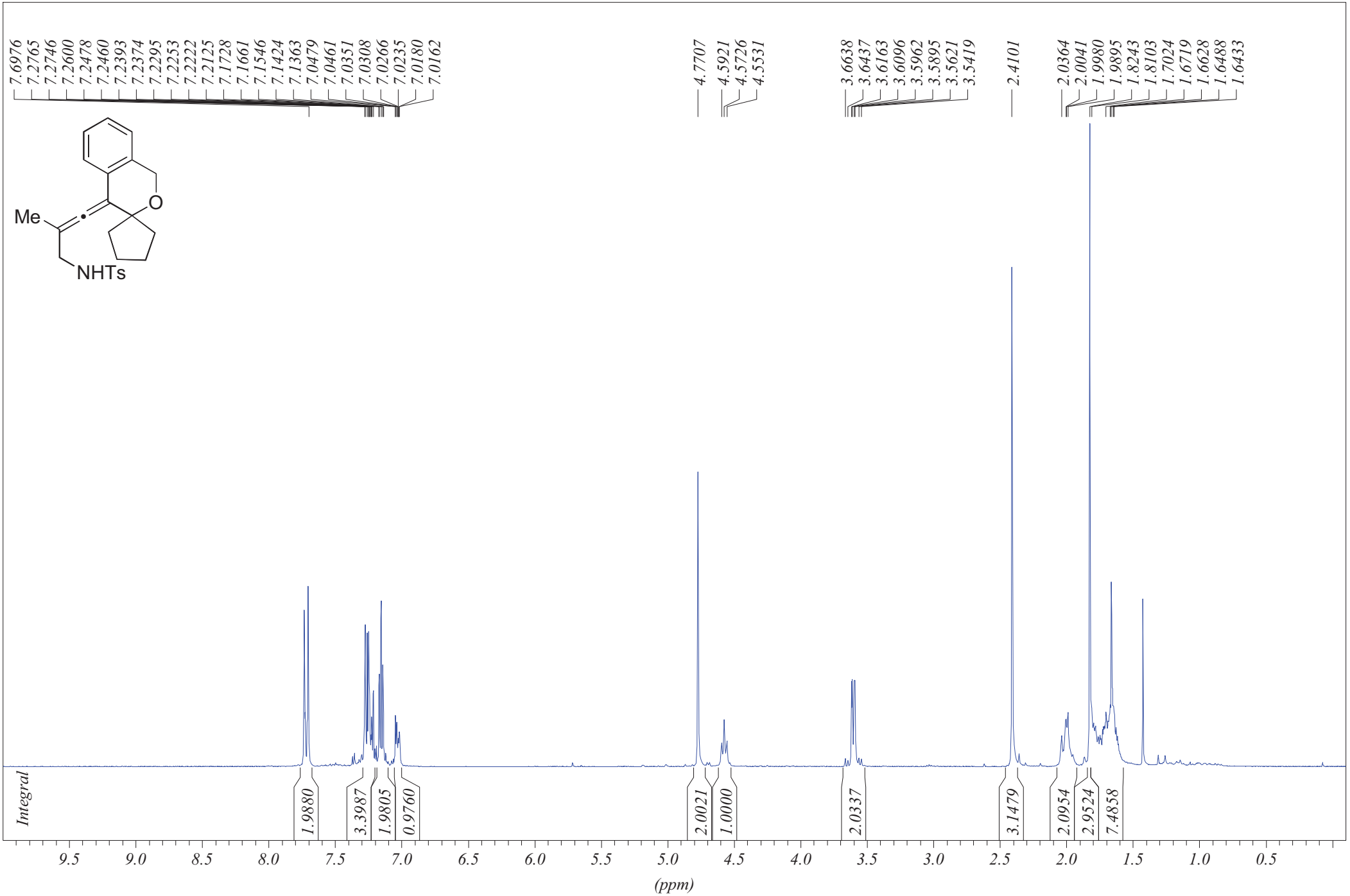
3c

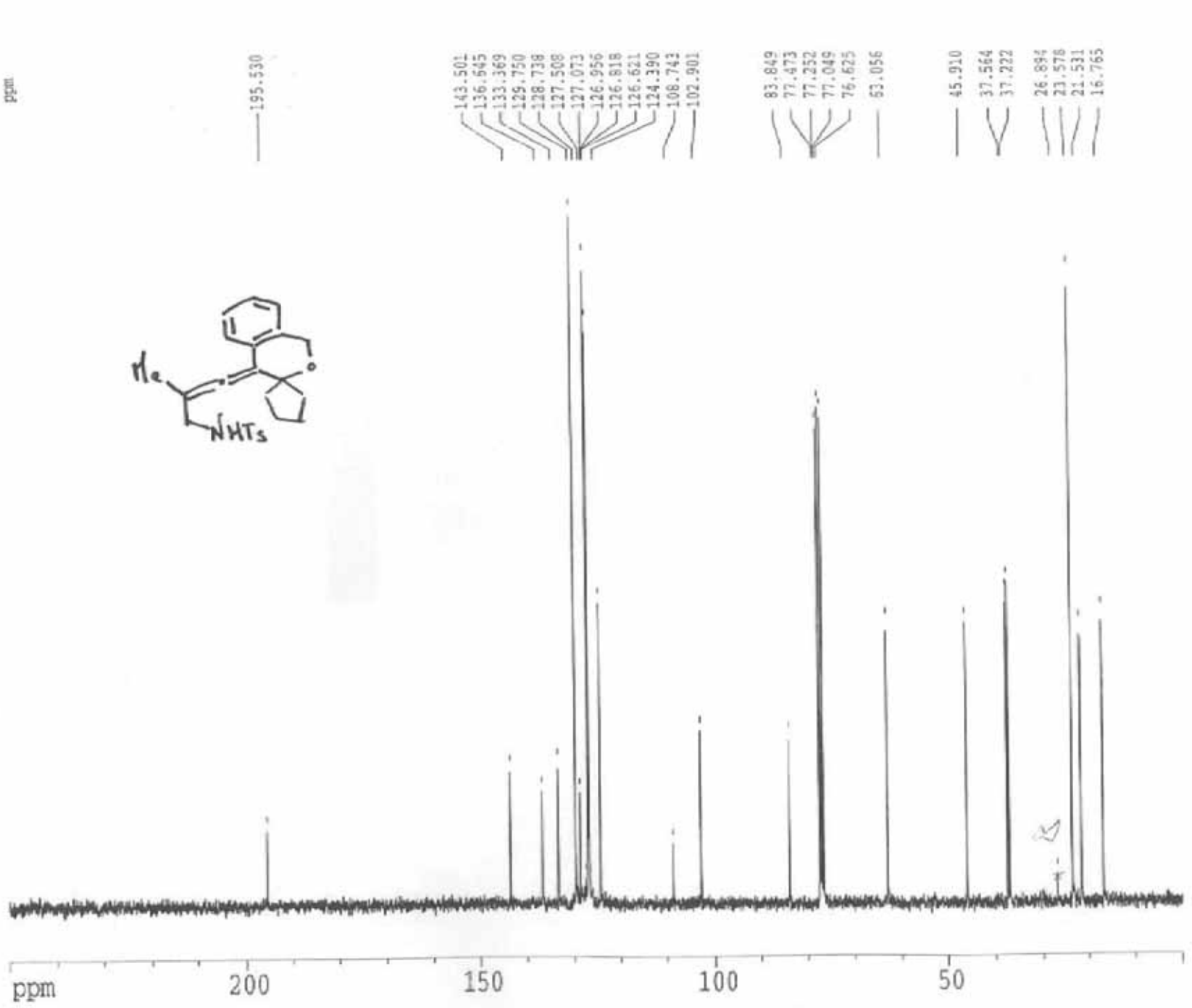

Me
TsHN
C_6H_{13}
ppm
2,05
1,89
3,05
1,00
2,03
1,08
1,02
2,14
3,03
3,15
1,97
8,26
3,38


3c
128
ppm
195,441
143,466
136,728
134,017
129,752
128,938
127,098
126,991
126,490
124,567
124,509
106,053
103,194
102,922
77,457
77,243
77,038
76,611
75,543
75,469
67,794
67,572
46,043
45,591
33,791
33,487
31,909
30,191
29,435
29,345
28,929
25,754
25,360
22,656
21,563
16,945
16,584
14,127
Me
TsHN
C6H13
O
%
ppm
200
180
160
140
120
100
80
60
40
20


3d
7,730
7,724
7,708
7,702
7,284
7,259
7,204
7,183
7,178
7,165
7,153
7,148
7,061
7,050
4,799
4,405
3,615
3,608
3,595
3,587
2,415
1,825
1,388
0,880
0,857
Me
TsHN
Me
O
Me
2,00
1,89
2,97
1,05
2,01
0,95
1,97
3,05
3,00
5,87
2,08
ppm


3d
130
ppm
195,515
143,532
136,704
133,178
129,776
128,149
127,089
126,942
124,386
110,047
103,103
77,465
77,038
76,619
72,987
62,584
45,829
26,962
26,781
21,547
16,797
Me
TsHN
Me
Me
O
%
ppm
200
180
160
140
120
100
80
60
40
20


7.6976
7.2765
7.2746
7.2600
7.2478
7.2460
7.2393
7.2374
7.2295
7.2253
7.2222
7.2125
7.1728
7.1661
7.1546
7.1424
7.1363
7.0479
7.0461
7.0351
7.0308
7.0266
7.0235
7.0180
7.0162
4.7707
4.5921
4.5726
4.5531
3.6638
3.6437
3.6163
3.6096
3.5962
3.5895
3.5621
3.5419
2.4101
2.0364
2.0041
1.9980
1.9895
1.8243
1.8103
1.7024
1.6719
1.6628
1.6488
1.6433
Me
O
NHTs
Integral
1.9880
3.3987
1.9805
0.9760
2.0021
1.0000
2.0337
3.1479
2.0954
2.9524
7.4858
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
(ppm)



Current Data Parameters
NAME p3_48._20.05.10
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100510
Time 11.14
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgdc30
TD 32768
SOLVENT CDCl3
NS 1600
DS 4
SWH 20325.203 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 16384
DW 24.600 usec
DE 10.00 usec
TE 297.0 K
D1 0.60000002 sec
d11 0.03000000 sec

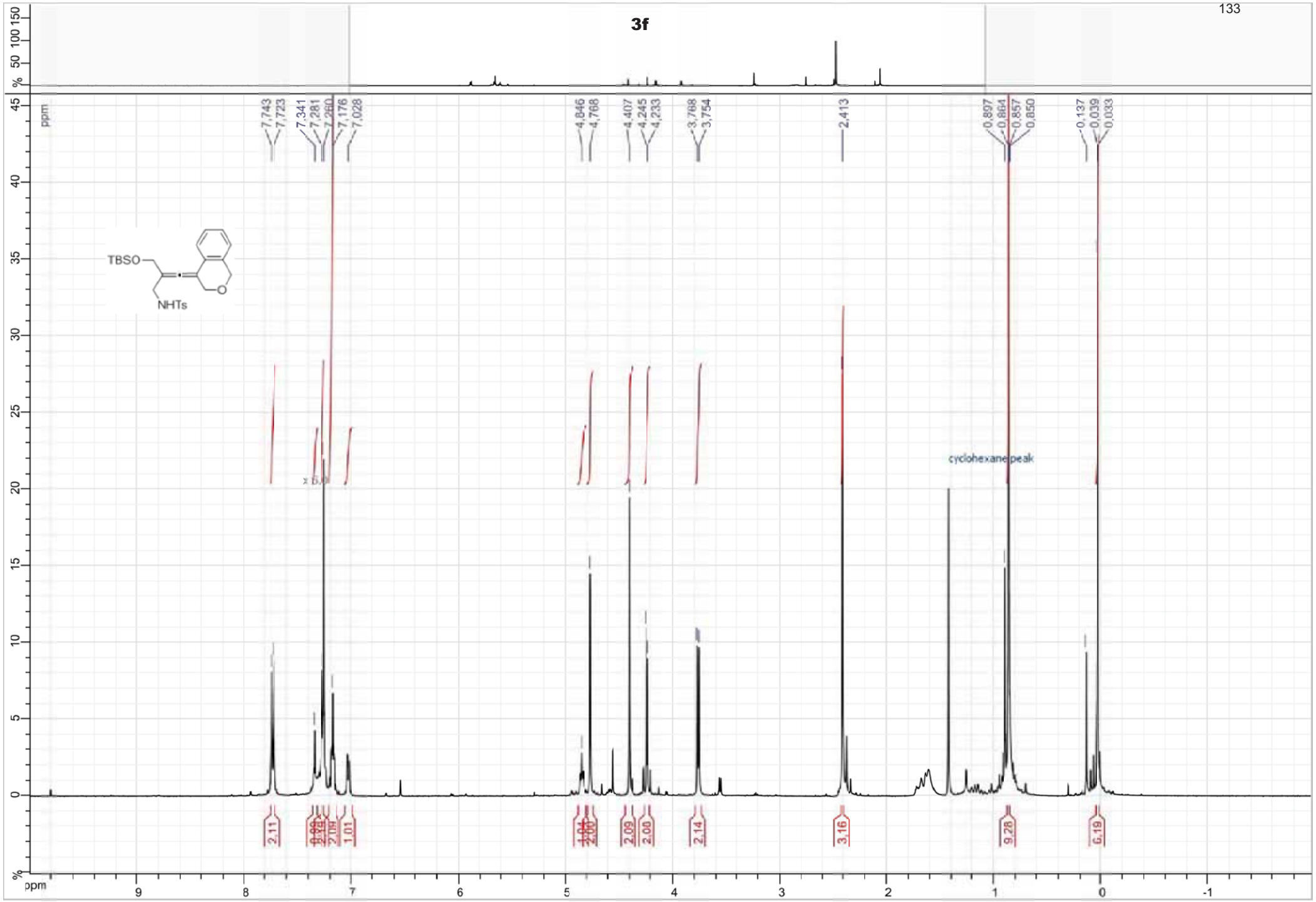
======== CHANNEL f1 ========
NUC1 13C
P1 11.50 usec
PL1 2.00 dB
SFO1 75.4867925 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 109.00 usec
PL2 2.00 dB
PL12 22.92 dB
SFO2 300.1713607 MHz

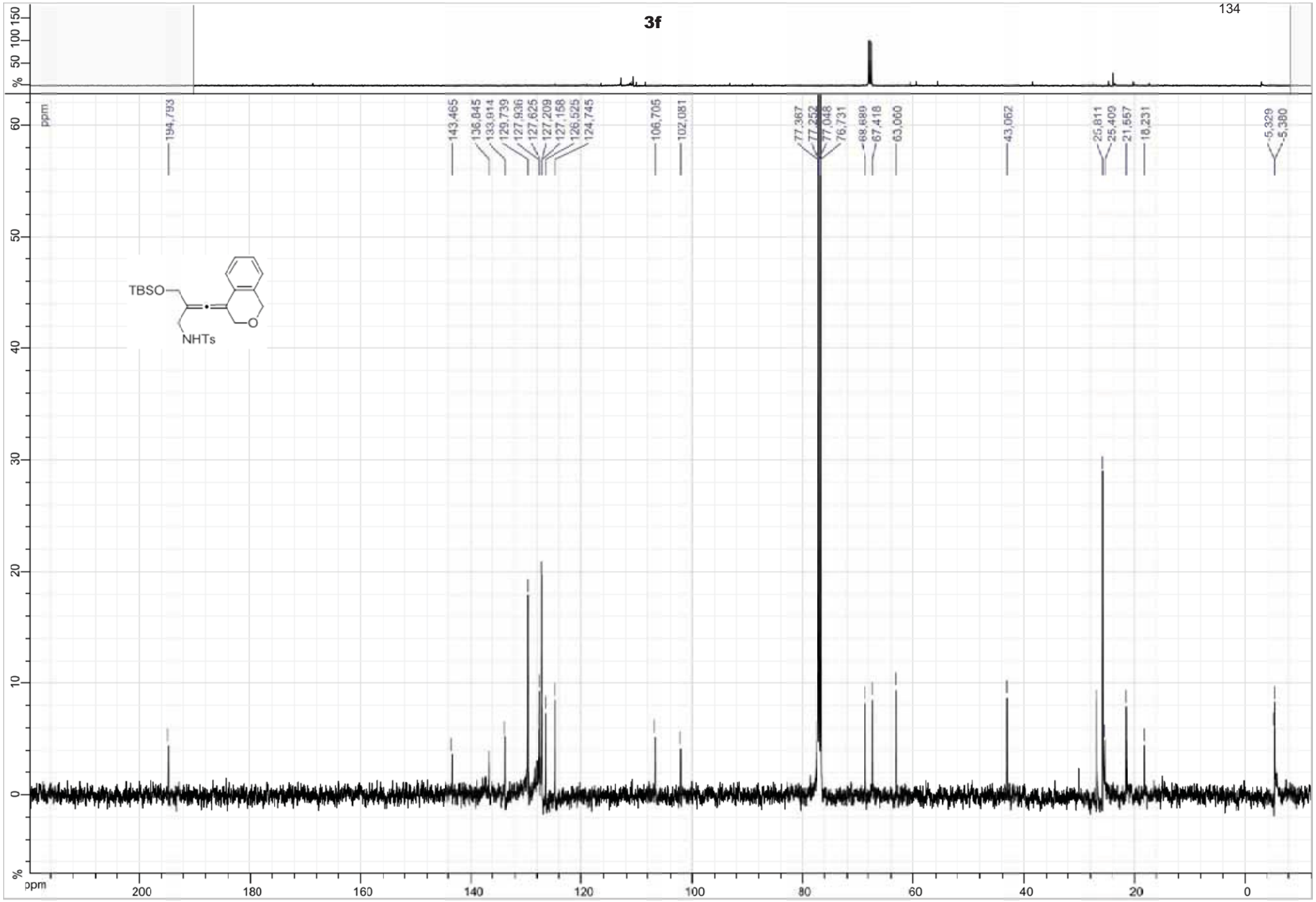
F2 - Processing parameters
SI 32768
SF 75.4778106 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

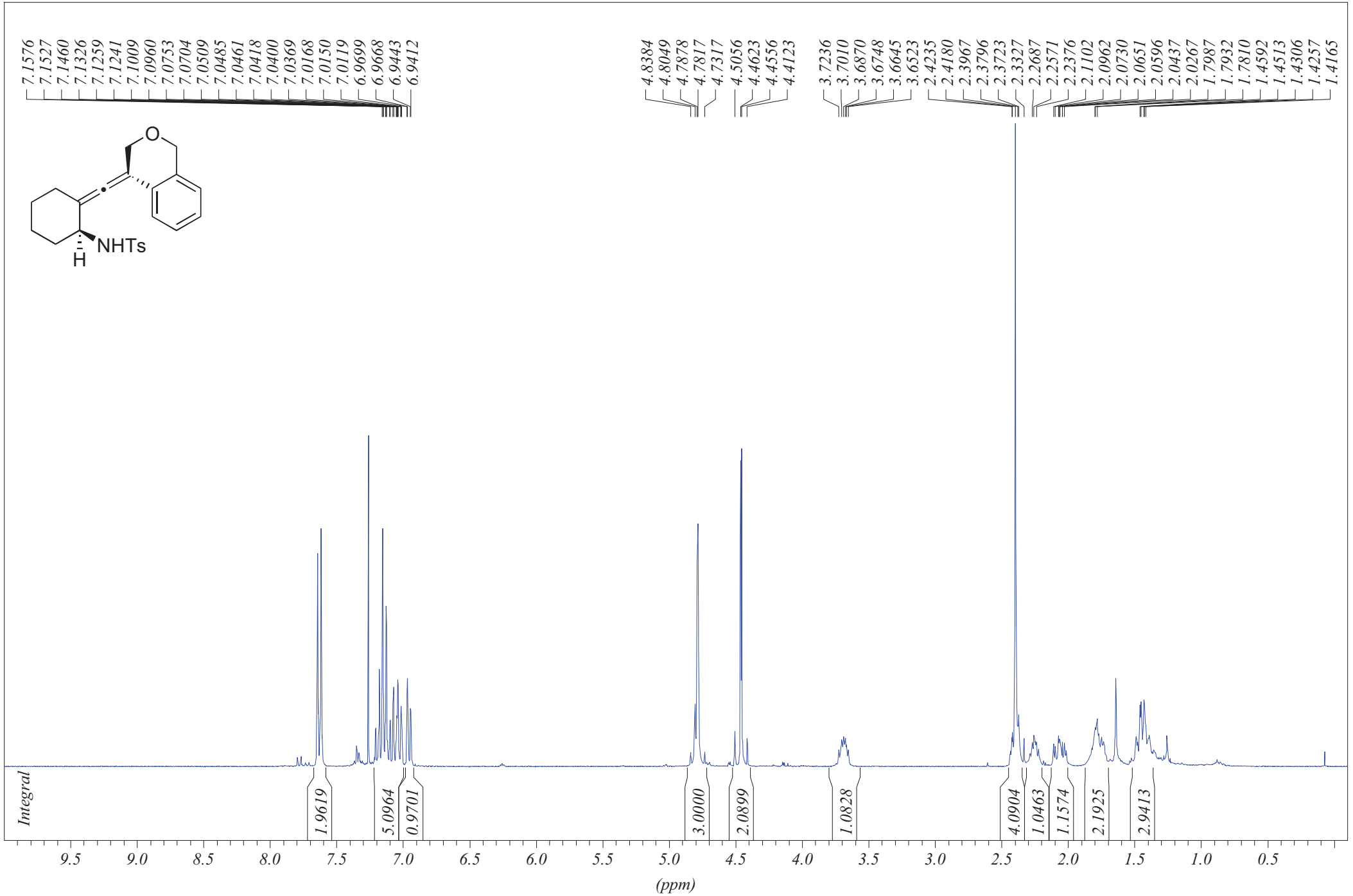
1D NMR plot parameters
CX 22.00 cm
CY 13.00 cm
F1P 250.000 ppm
F1 18869.45 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 11.36364 ppm/c
HZCM 857.70245 Hz/cm

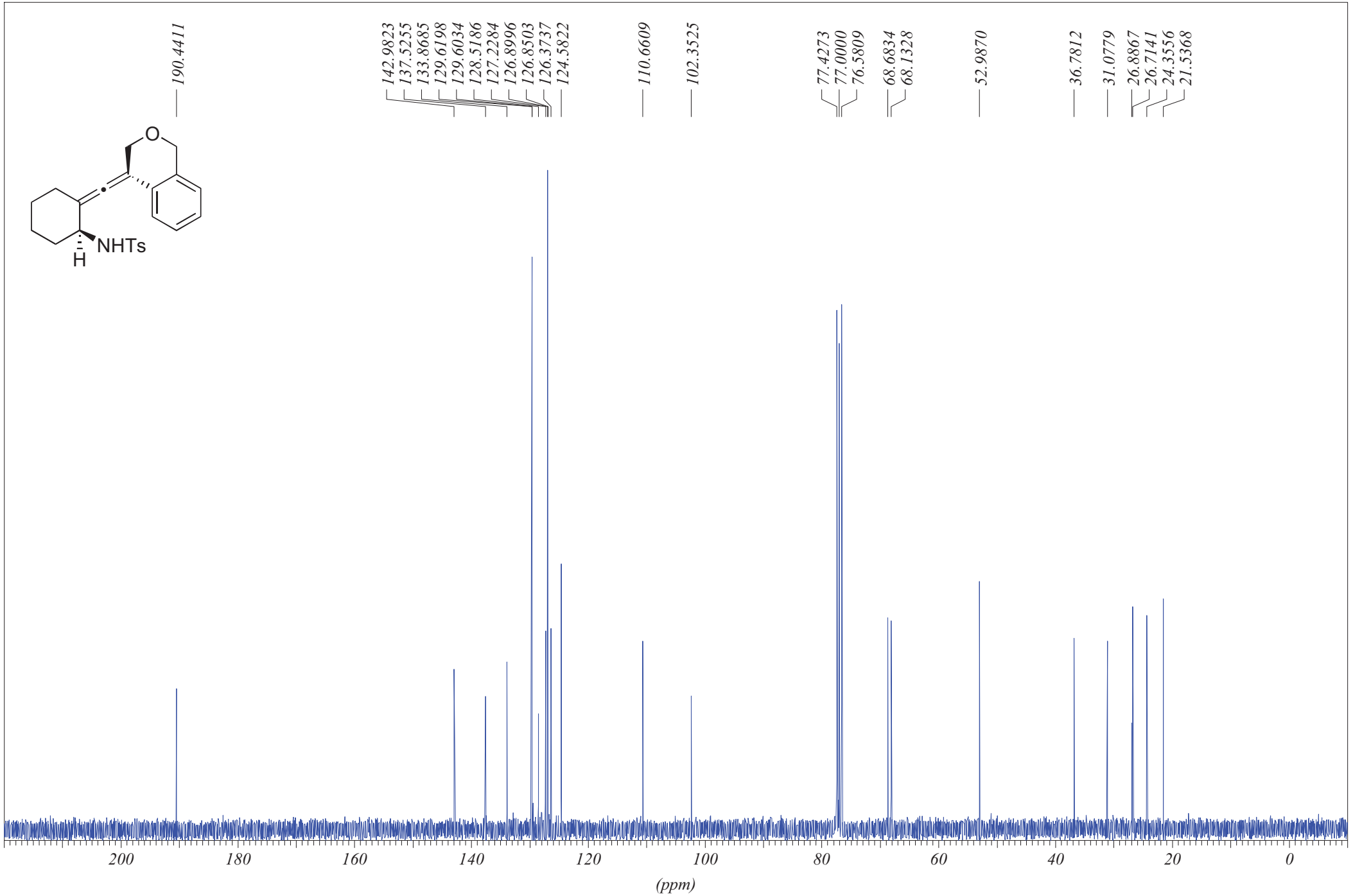
3f

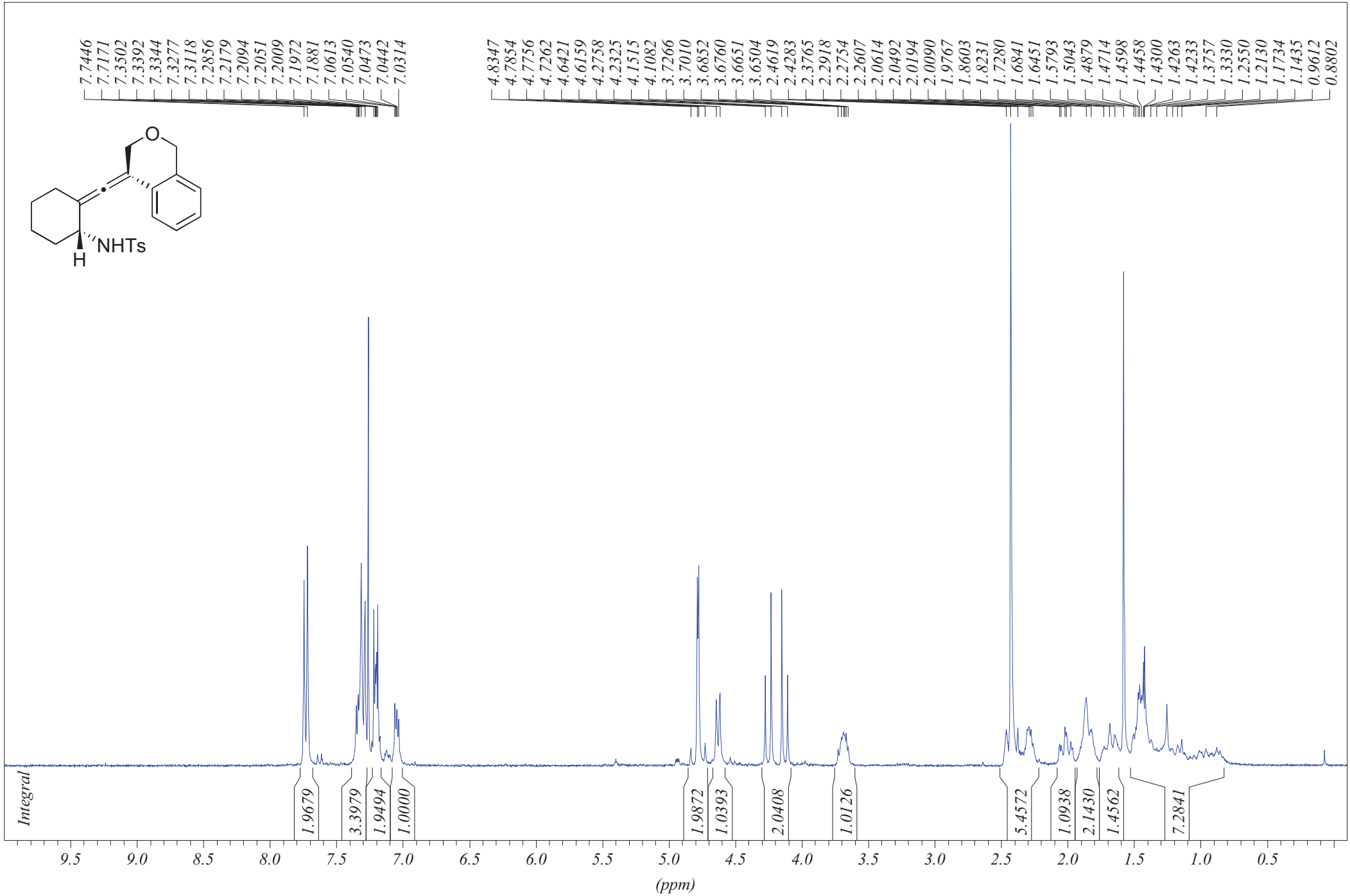

TBSO
NHTs
7,743
7,723
7,341
7,281
7,260
7,176
7,028
4,846
4,768
4,407
4,245
4,233
3,768
3,754
2,413
0,897
0,864
0,857
0,850
0,137
0,039
0,033
cyclohexane peak
2,11
3,16
9,26
6,19
ppm

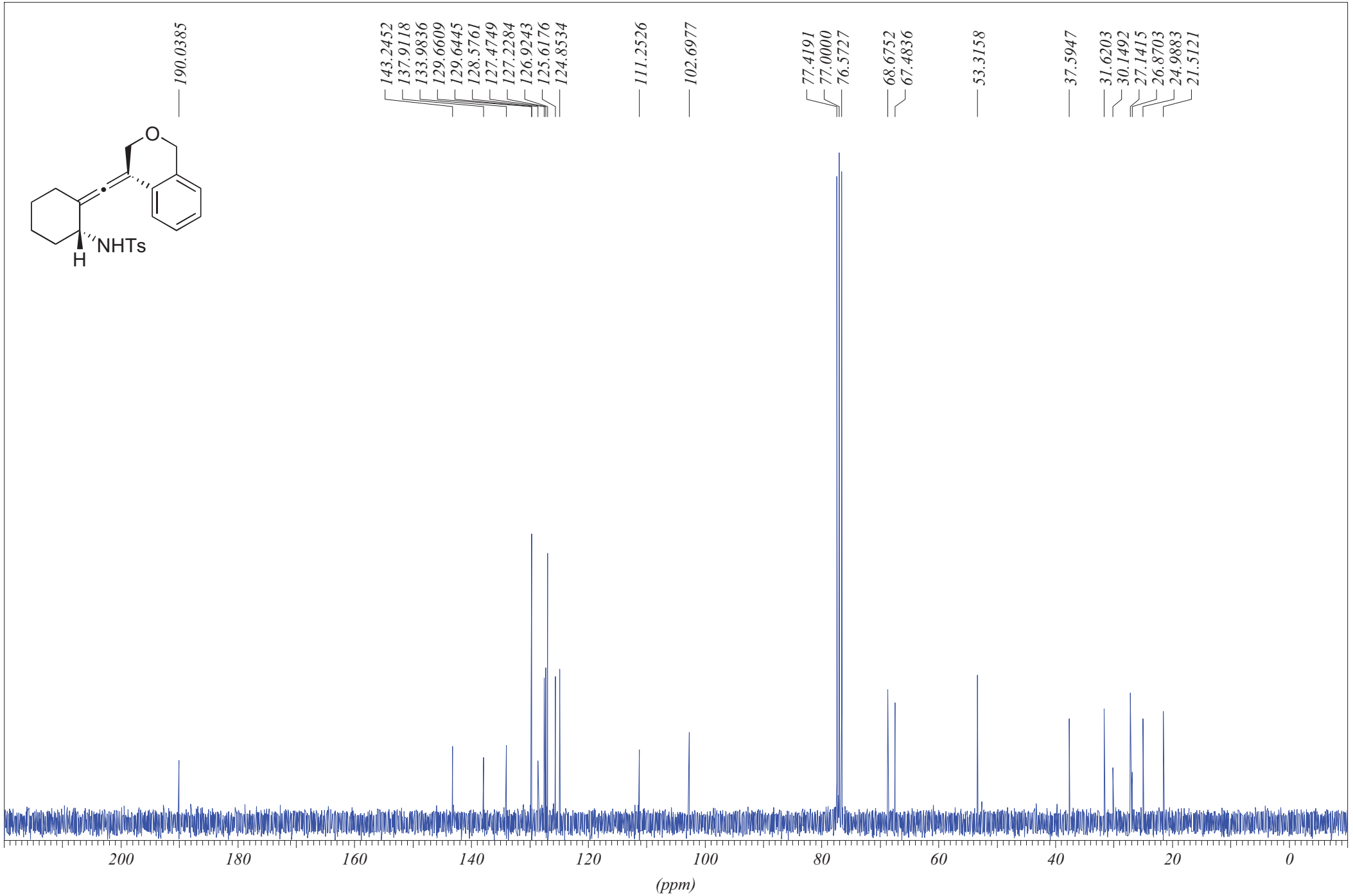
3f

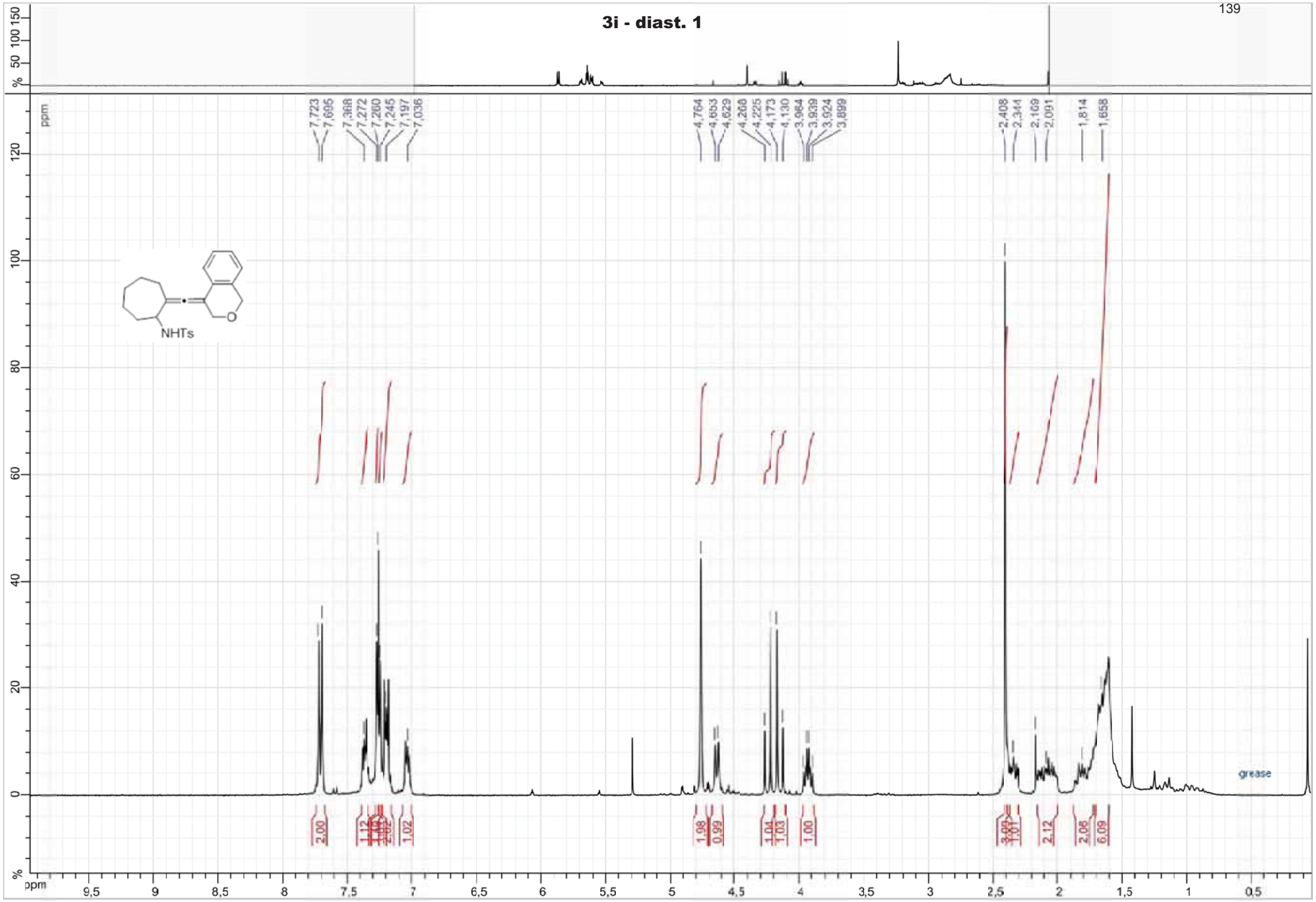

ppm
194,793
143,465
136,845
133,914
129,739
127,936
127,625
127,209
127,158
126,525
124,745
106,705
102,081
77,367
77,252
77,048
76,731
68,689
67,418
63,060
43,062
25,811
25,409
21,557
18,231
-5,329
-5,380
TBSO
NHTs
O
%
ppm
200
180
160
140
120
100
80
60
40
20
0


7.1576
7.1527
7.1460
7.1326
7.1259
7.1241
7.1009
7.0960
7.0753
7.0704
7.0509
7.0485
7.0461
7.0418
7.0400
7.0369
7.0168
7.0150
7.0119
6.9699
6.9668
6.9443
6.9412
4.8384
4.8049
4.7878
4.7817
4.7317
4.5056
4.4623
4.4556
4.4123
3.7236
3.7010
3.6870
3.6748
3.6645
3.6523
2.4235
2.4180
2.3967
2.3796
2.3723
2.3327
2.2687
2.2571
2.2376
2.1102
2.0962
2.0730
2.0651
2.0596
2.0437
2.0267
1.7987
1.7932
1.7810
1.4592
1.4513
1.4306
1.4257
1.4165
O
NHTs
H
Integral
1.9619
5.0964
0.9701
3.0000
2.0899
1.0828
4.0904
1.0463
1.1574
2.1925
2.9413
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
(ppm)

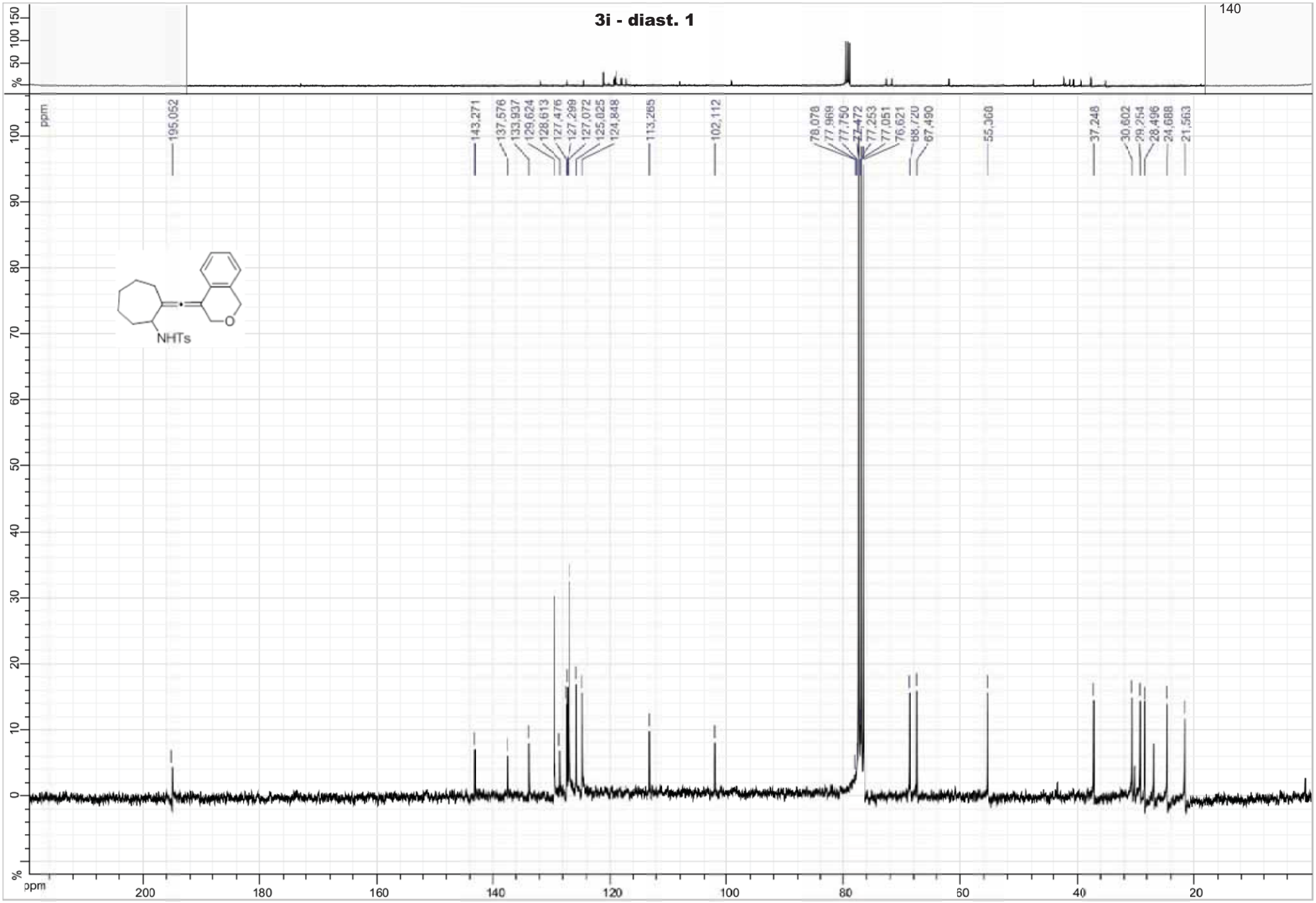

190.4411
142.9823
137.5255
133.8685
129.6198
129.6034
128.5186
127.2284
126.8996
126.8503
126.3737
124.5822
110.6609
102.3525
77.4273
77.0000
76.5809
68.6834
68.1328
52.9870
36.7812
31.0779
26.8867
26.7141
24.3556
21.5368
O
NHTs
H
200
180
160
140
120
100
80
60
40
20
0
(ppm)


7.7446
7.7171
7.3502
7.3392
7.3344
7.3277
7.3118
7.2856
7.2179
7.2094
7.2051
7.2009
7.1972
7.1881
7.0613
7.0540
7.0473
7.0442
7.0314
4.8347
4.7854
4.7756
4.7262
4.6421
4.6159
4.2758
4.2325
4.1515
4.1082
3.7266
3.7010
3.6852
3.6760
3.6651
3.6504
2.4619
2.4283
2.3765
2.2918
2.2754
2.2607
2.0614
2.0492
2.0194
2.0090
1.9767
1.8603
1.8231
1.7280
1.6841
1.6451
1.5793
1.5043
1.4879
1.4714
1.4598
1.4458
1.4300
1.4263
1.4233
1.3757
1.3330
1.2550
1.2130
1.1734
1.1435
0.9612
0.8802
O
NHTs
H
Integral
1.9679
3.3979
1.9494
1.0000
1.9872
1.0393
2.0408
1.0126
5.4572
1.0938
2.1430
1.4562
7.2841
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
(ppm)

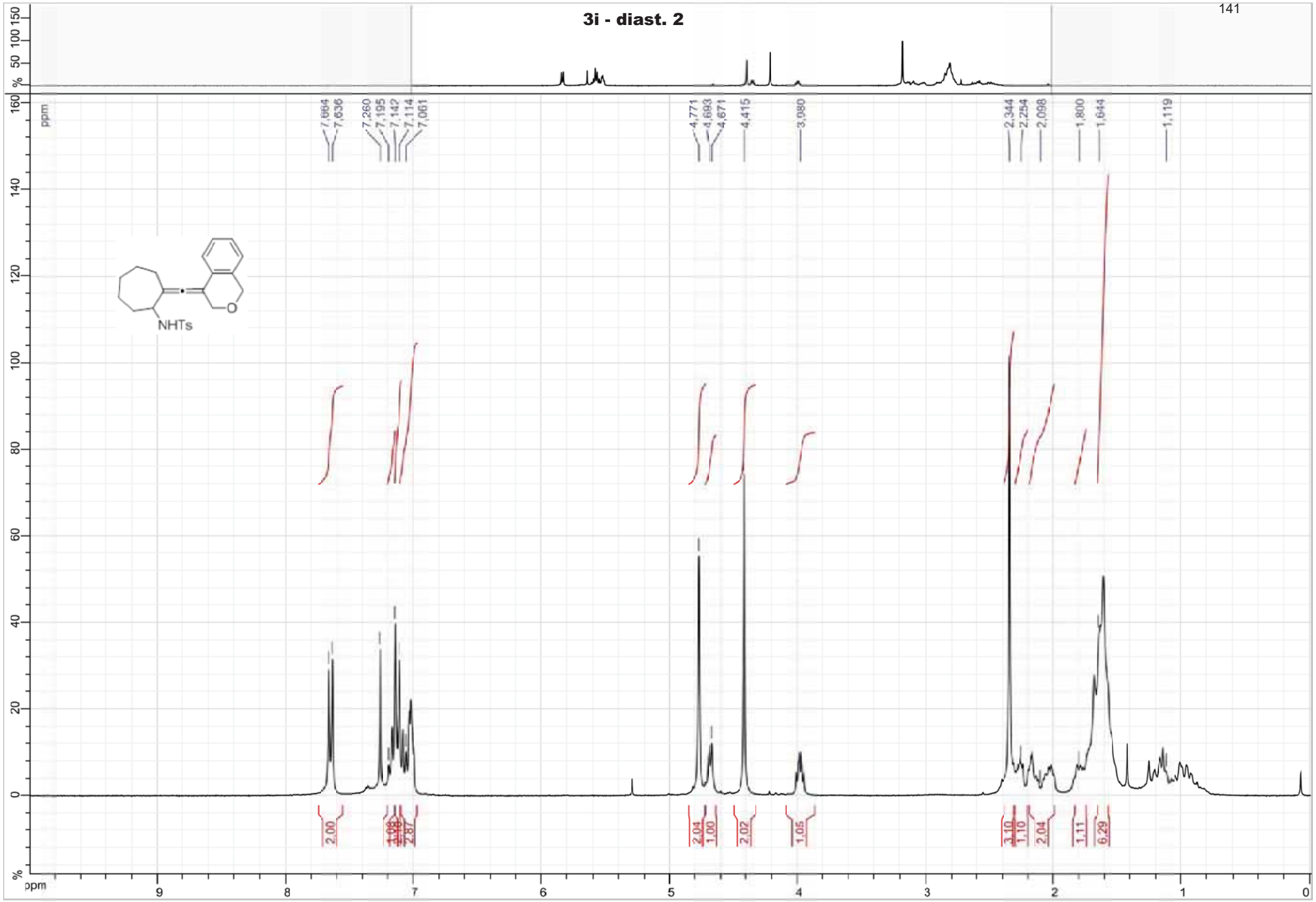

190.0385
143.2452
137.9118
133.9836
129.6609
129.6445
128.5761
127.4749
127.2284
126.9243
125.6176
124.8534
111.2526
102.6977
77.4191
77.0000
76.5727
68.6752
67.4836
53.3158
37.5947
31.6203
30.1492
27.1415
26.8703
24.9883
21.5121
O
NHTs
H
200
180
160
140
120
100
80
60
40
20
0
(ppm)


3i - diast. 1
ppm
7,723
7,695
7,368
7,272
7,260
7,245
7,197
7,036
4,764
4,653
4,629
4,266
4,225
4,173
4,130
3,964
3,939
3,924
3,899
2,408
2,344
2,169
2,091
1,814
1,658
NHTs
O
grease
2,00
1,12
1,02
1,08
0,99
1,04
1,03
1,00
2,12
2,06
6,09

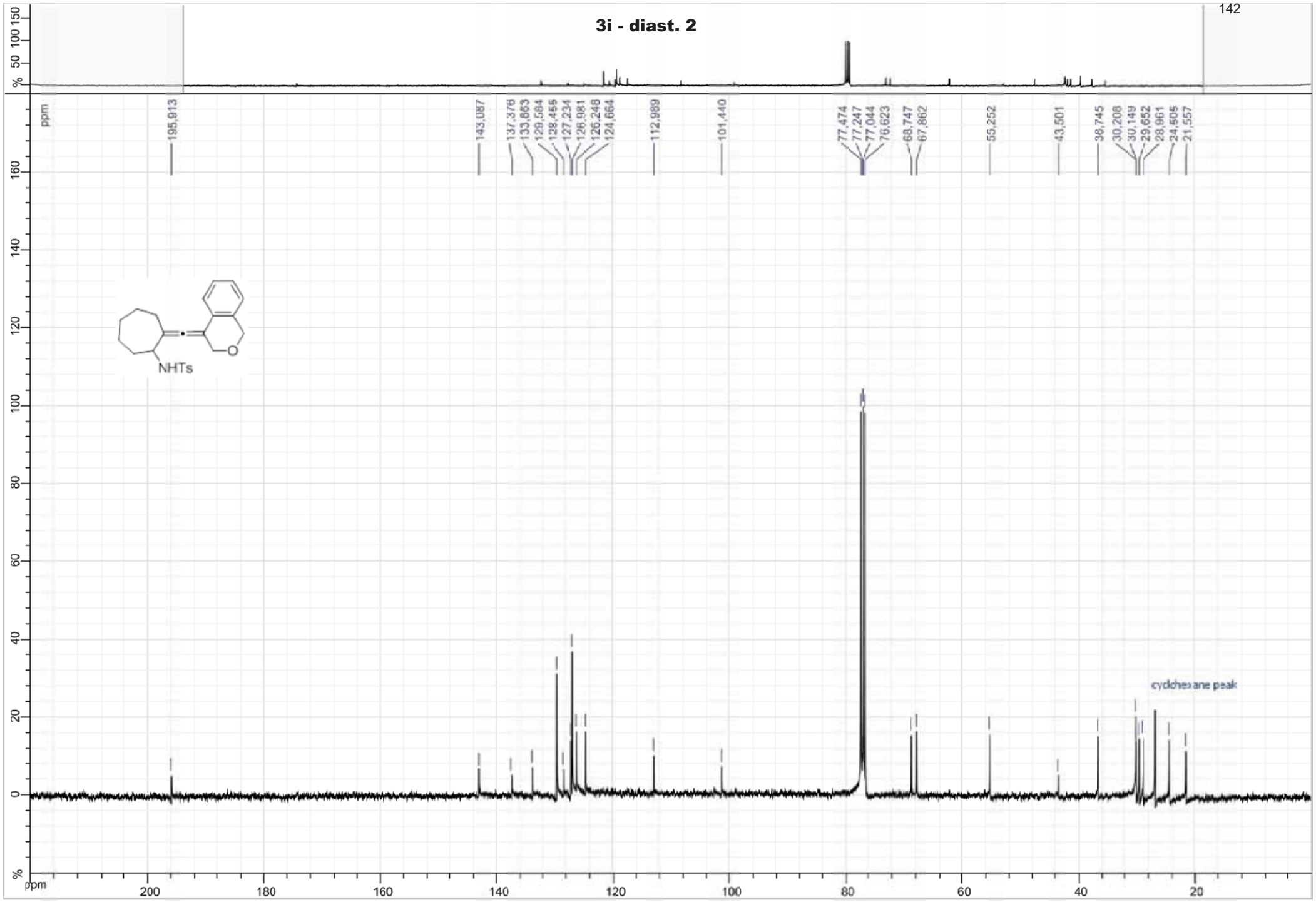
3i - diast. 1


ppm
195,052
143,271
137,576
133,937
129,624
128,613
127,476
127,299
127,072
125,025
124,848
113,265
102,112
78,078
77,969
77,750
77,472
77,253
77,051
76,621
68,720
67,490
55,368
37,248
30,602
29,254
28,496
24,688
21,563
NHTs
O
200
180
160
140
120
100
80
60
40
20

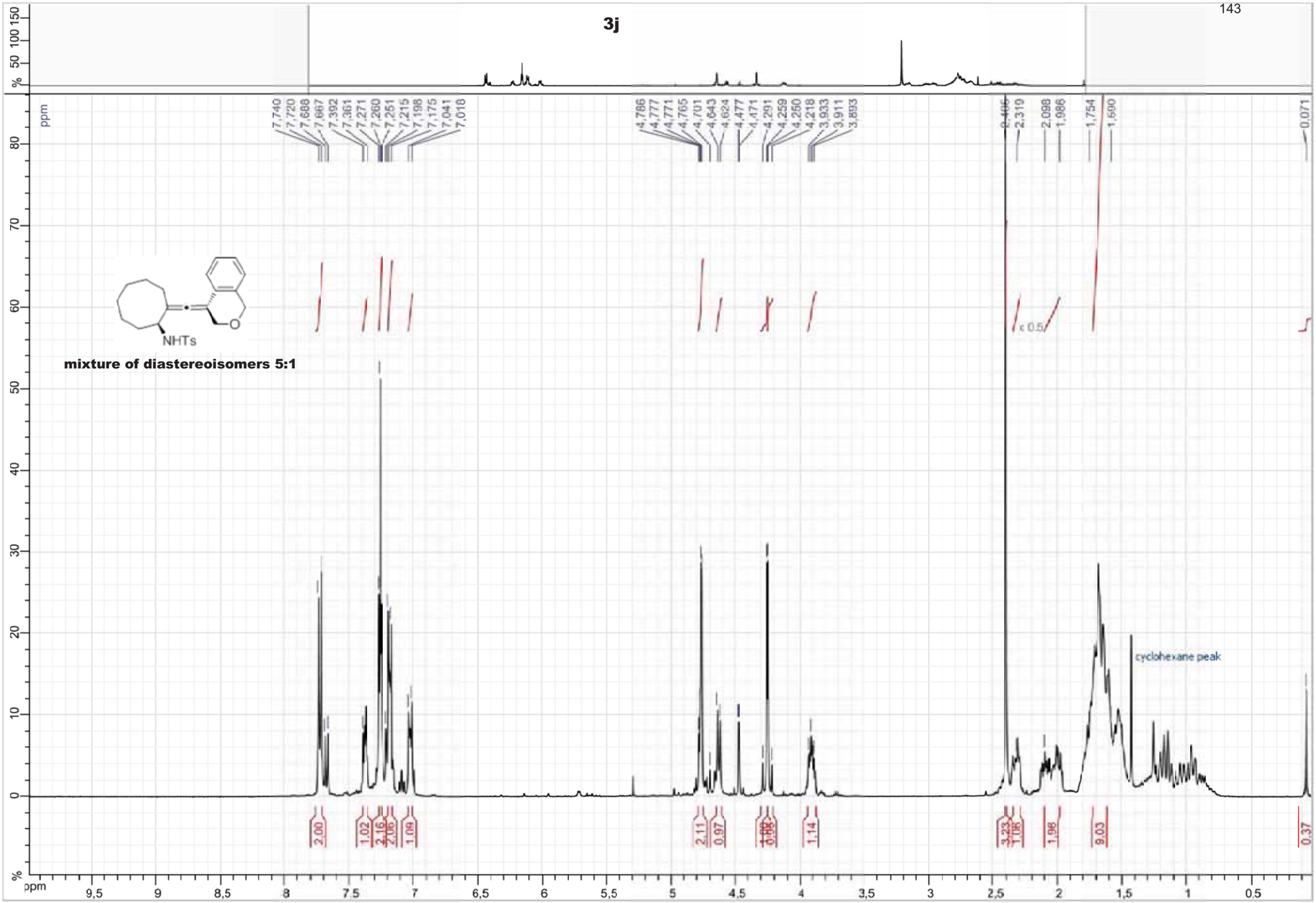
3i - diast. 2


7,664
7,636
7,260
7,195
7,142
7,114
7,061
4,771
4,693
4,671
4,415
3,980
2,344
2,254
2,098
1,800
1,644
1,119
NHTs
2,00
2,04
1,00
2,02
1,05
3,10
1,10
2,04
1,11
6,29
ppm

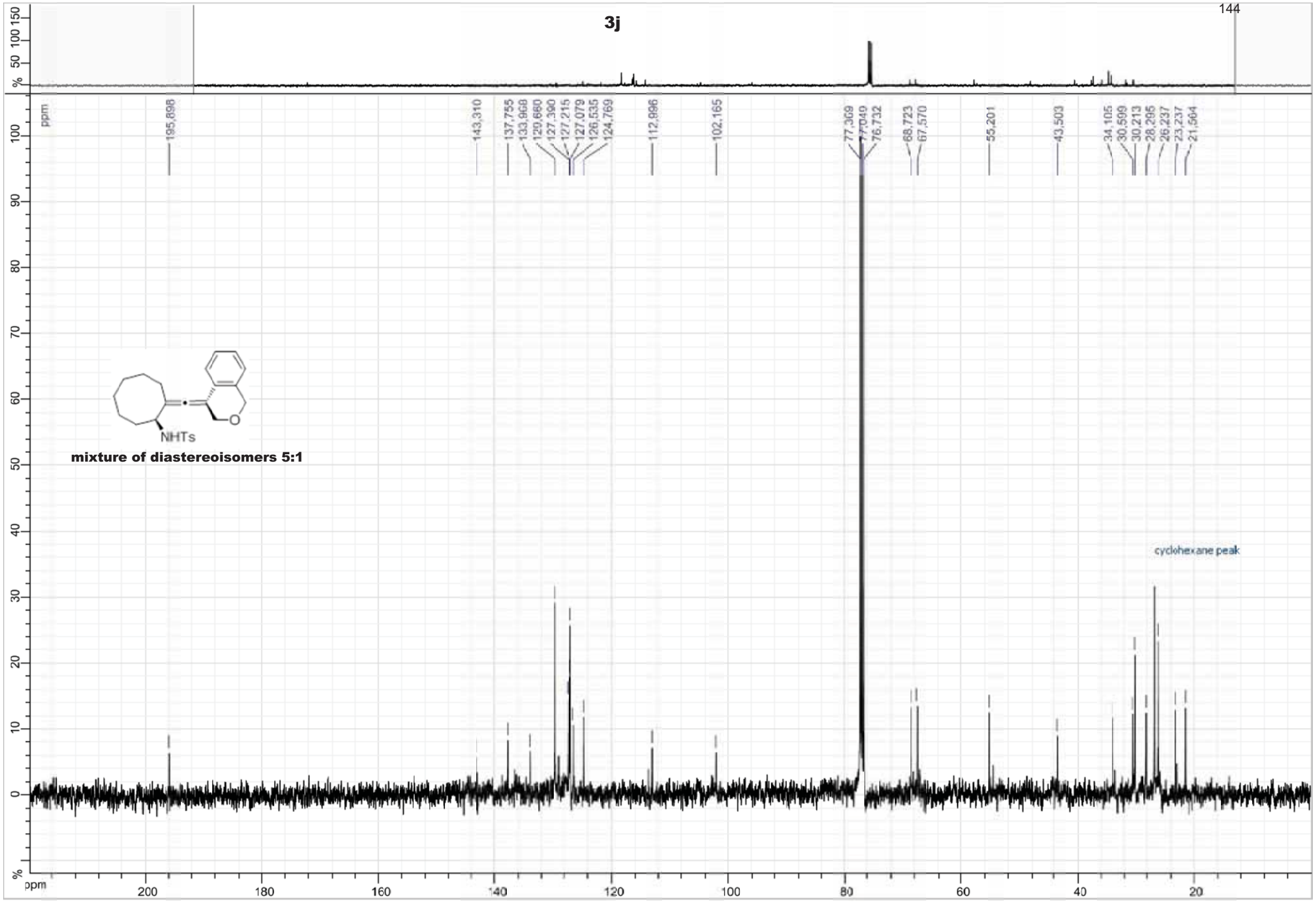
3i - diast. 2


ppm
195,913
143,087
137,376
133,863
129,584
128,455
127,234
126,981
126,248
124,664
112,989
101,440
77,474
77,247
77,044
76,623
68,747
67,862
55,252
43,501
36,745
30,208
30,149
29,652
28,961
24,505
21,557
NHTs
cyclohexane peak
ppm

3j

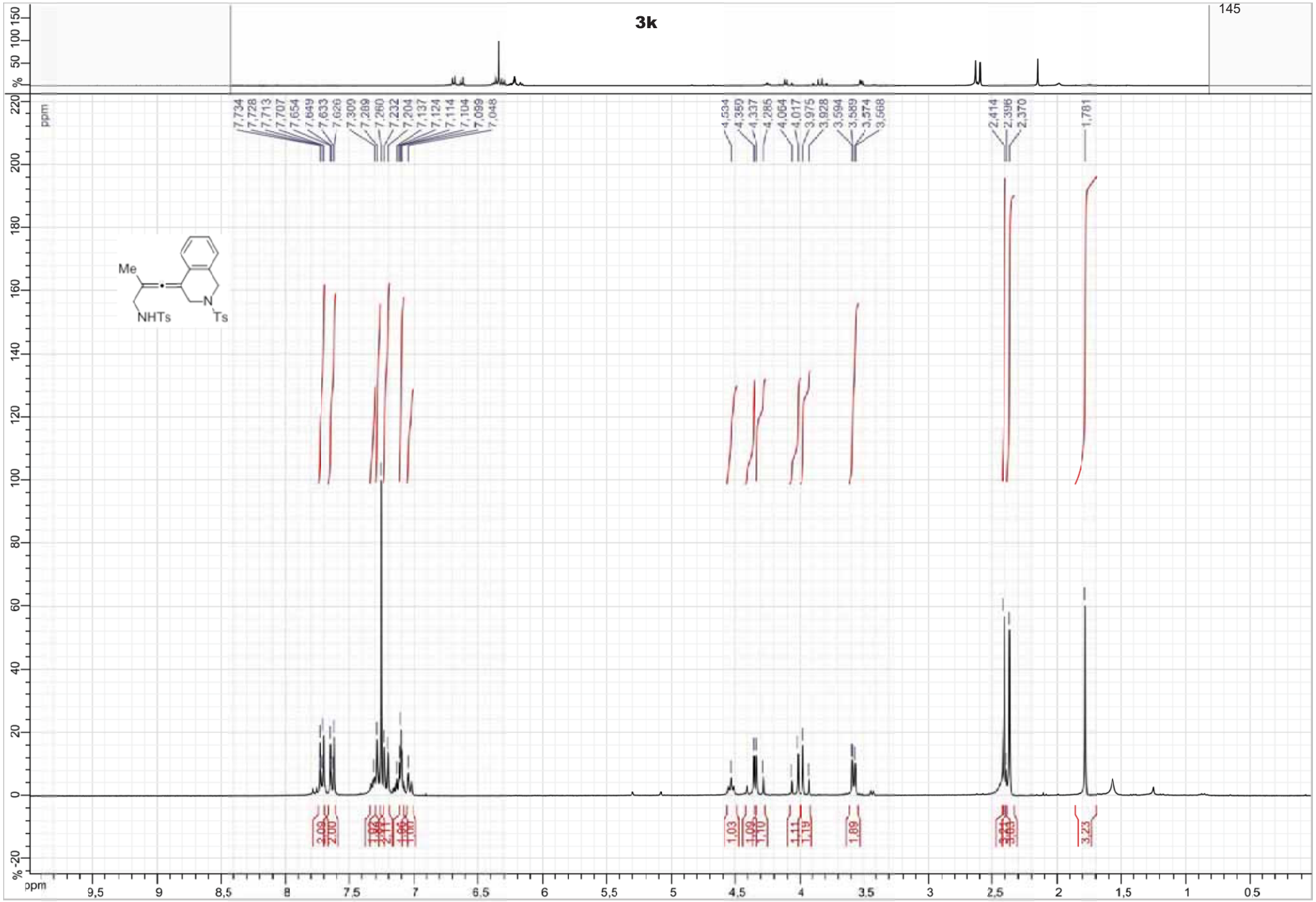


3j

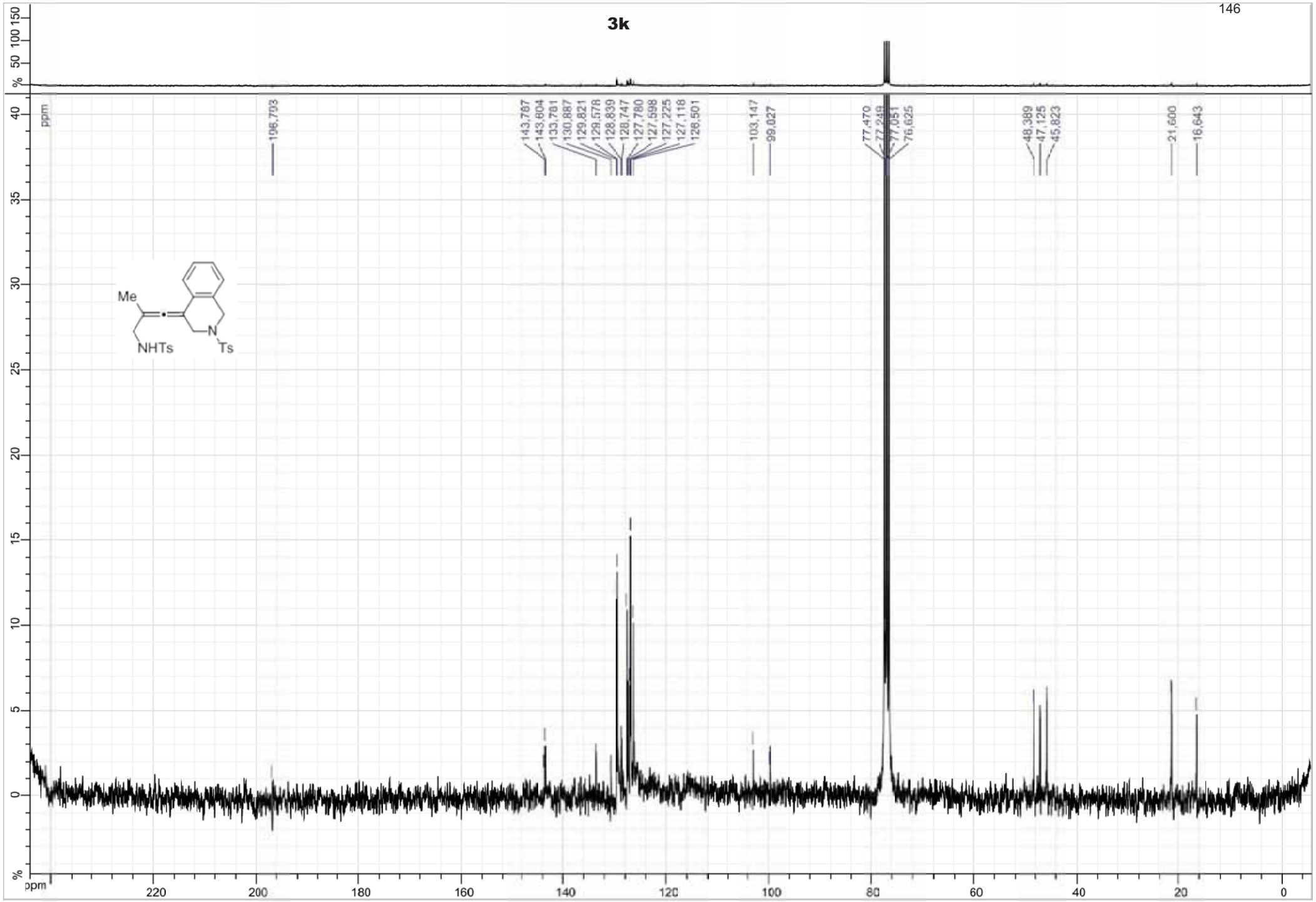


mixture of diastereoisomers 5:1

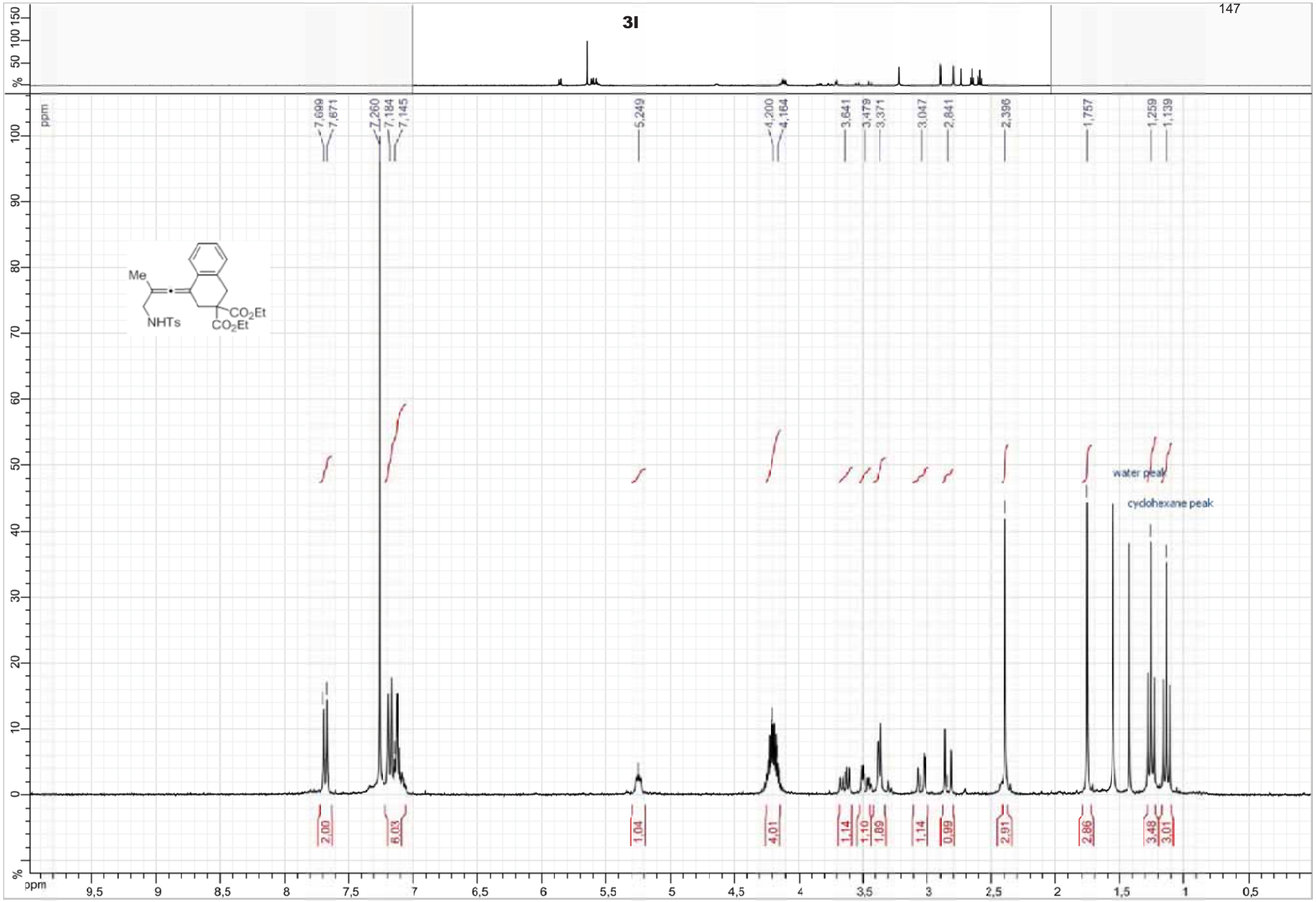
3k


7,734
7,728
7,713
7,707
7,654
7,649
7,633
7,626
7,309
7,289
7,260
7,232
7,204
7,137
7,124
7,114
7,104
7,099
7,048
4,534
4,350
4,337
4,285
4,064
4,017
3,975
3,928
3,594
3,589
3,574
3,568
2,414
2,396
2,370
1,781
Me
NHTs
N
Ts
2,09
2,00
1,03
1,09
1,10
1,11
1,19
1,89
3,23
ppm

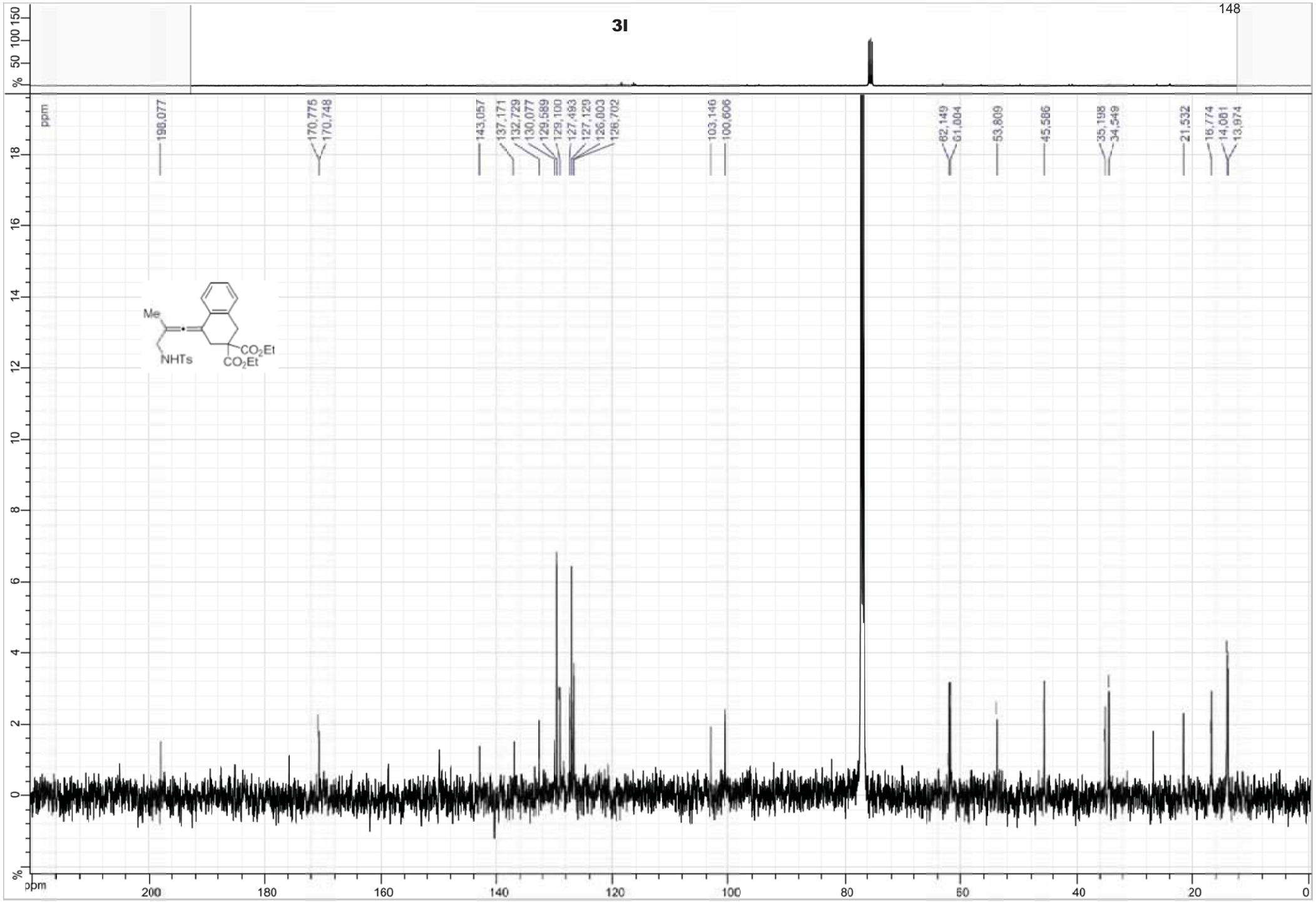
3k


196,793
143,767
143,604
133,781
130,887
129,821
129,578
128,839
128,747
127,780
127,598
127,225
127,118
128,501
103,147
99,027
77,470
77,249
77,051
76,625
48,389
47,125
45,823
21,600
16,643
Me
NHTs
N
Ts
ppm

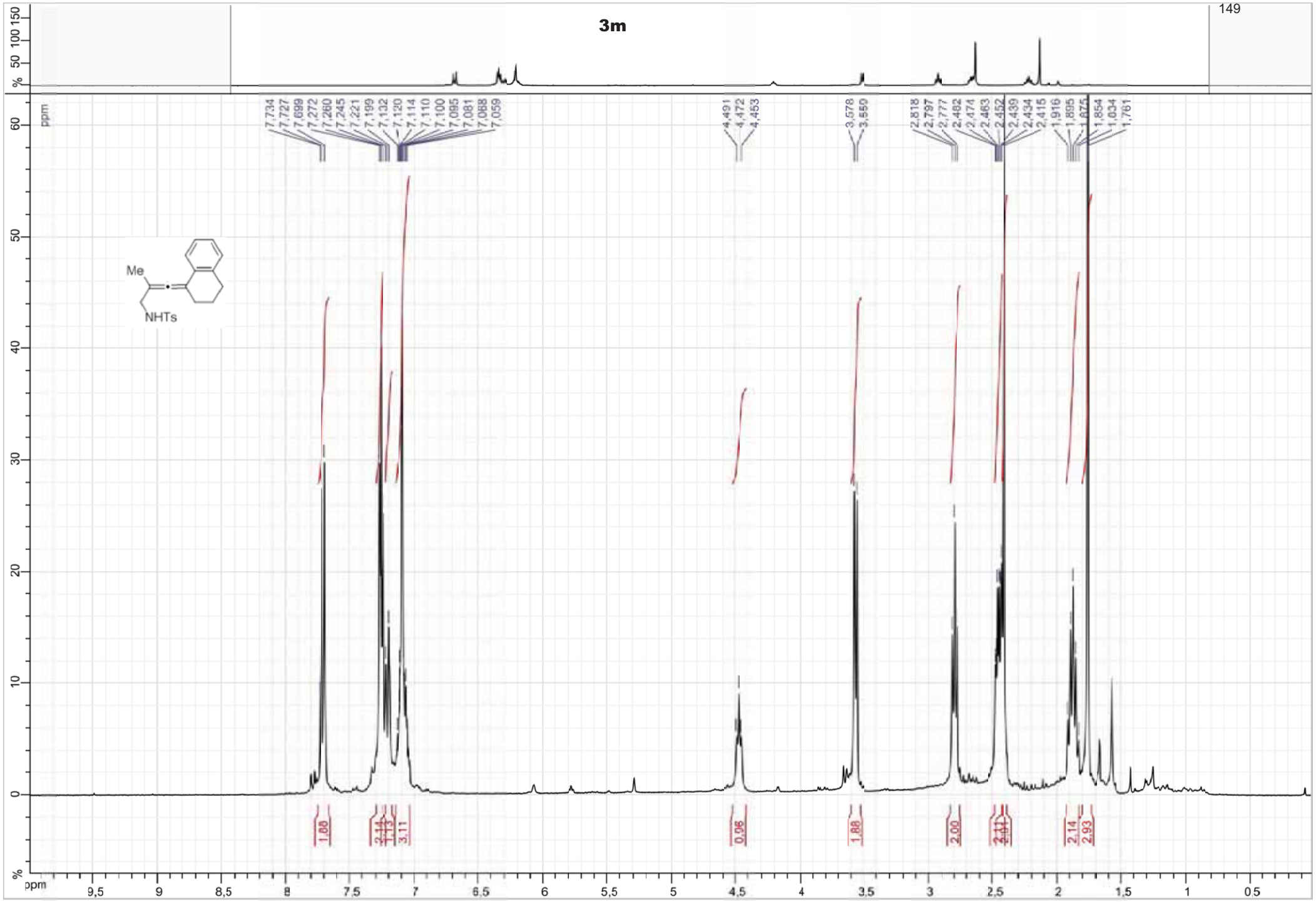
3l

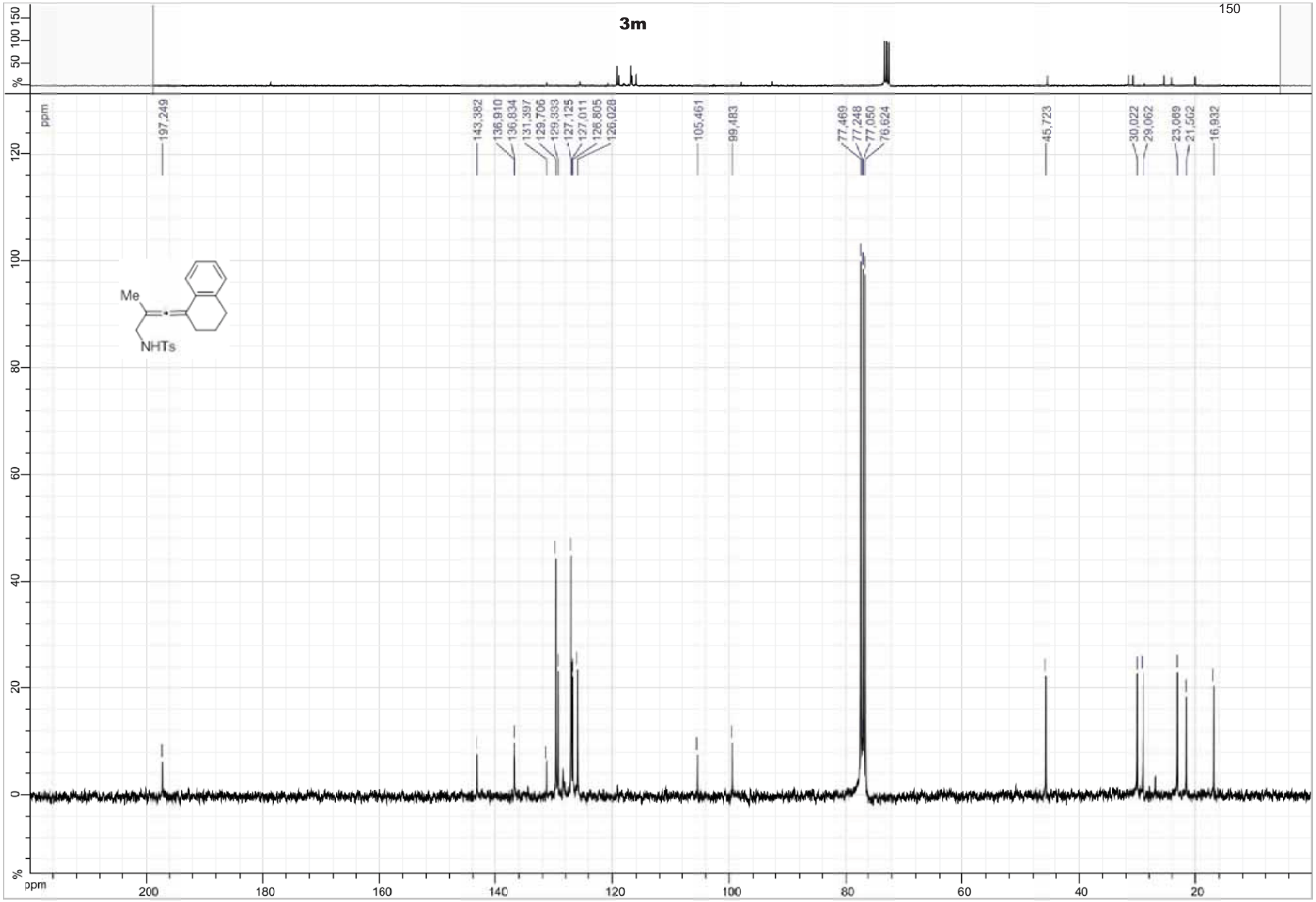

ppm
7,699
7,671
7,260
7,184
7,145
5,249
4,200
4,164
3,641
3,479
3,371
3,047
2,841
2,396
1,757
1,259
1,139
Me
NHTs
CO2Et
CO2Et
water peak
cyclohexane peak
2,00
6,03
1,04
4,01
1,14
1,10
1,89
1,14
0,99
2,91
2,86
3,48
3,01

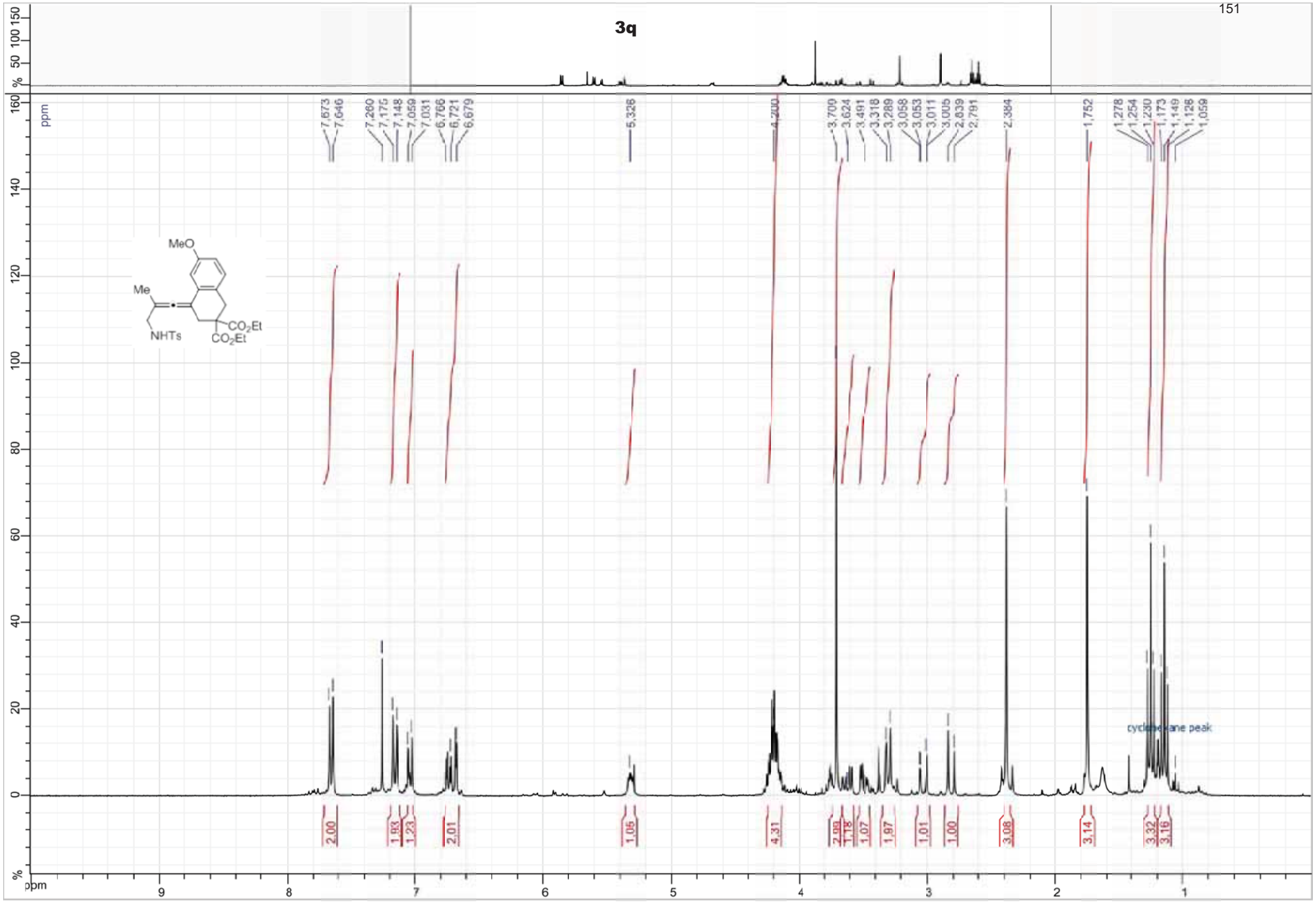
3l

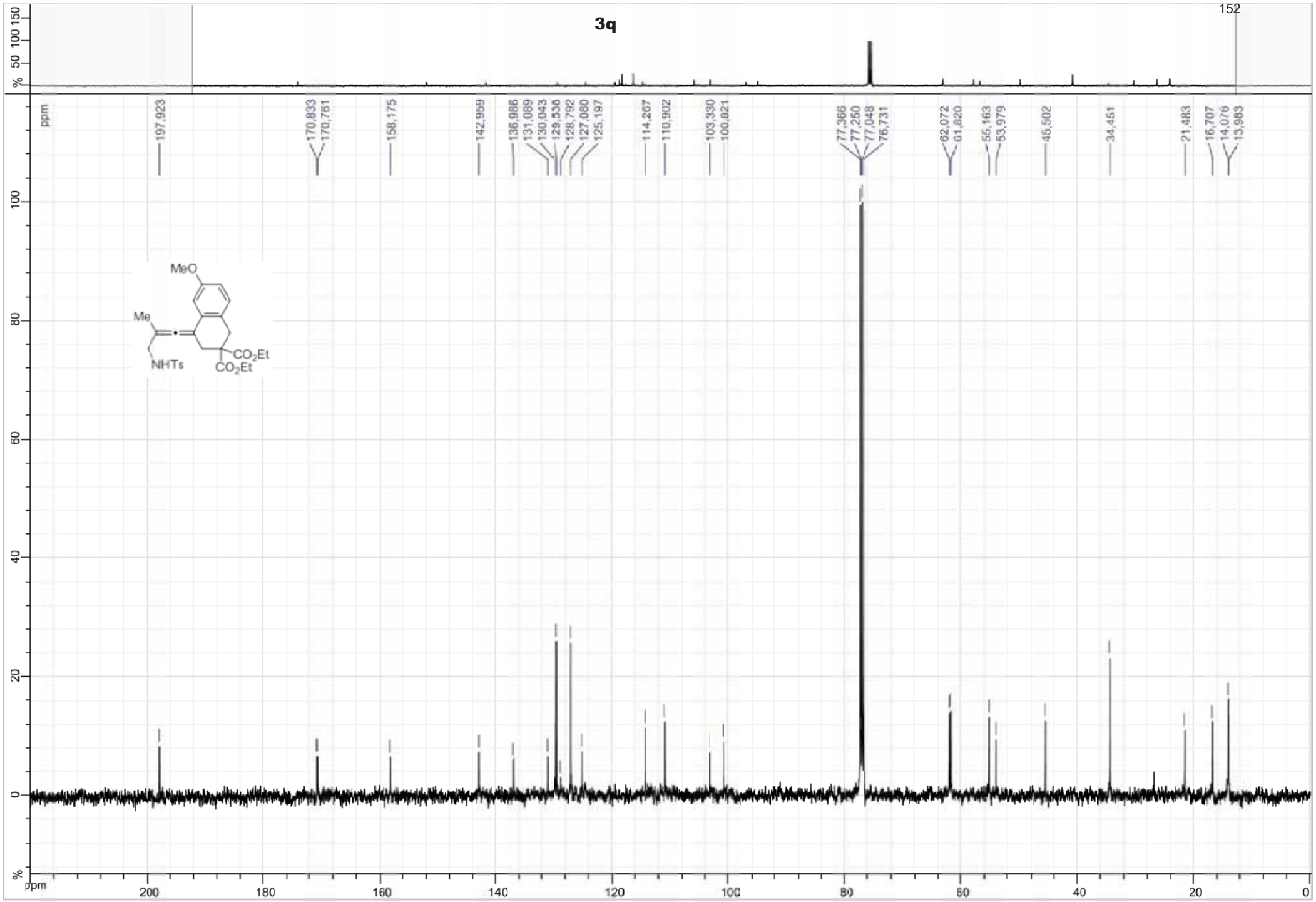

198,077
170,775
170,748
143,057
137,171
132,729
130,077
129,589
129,100
127,493
127,129
126,003
126,702
103,146
100,606
82,149
61,004
53,809
45,586
35,198
34,549
21,532
16,774
14,061
13,974
Me
NHTs
CO2Et
CO2Et
ppm

3m

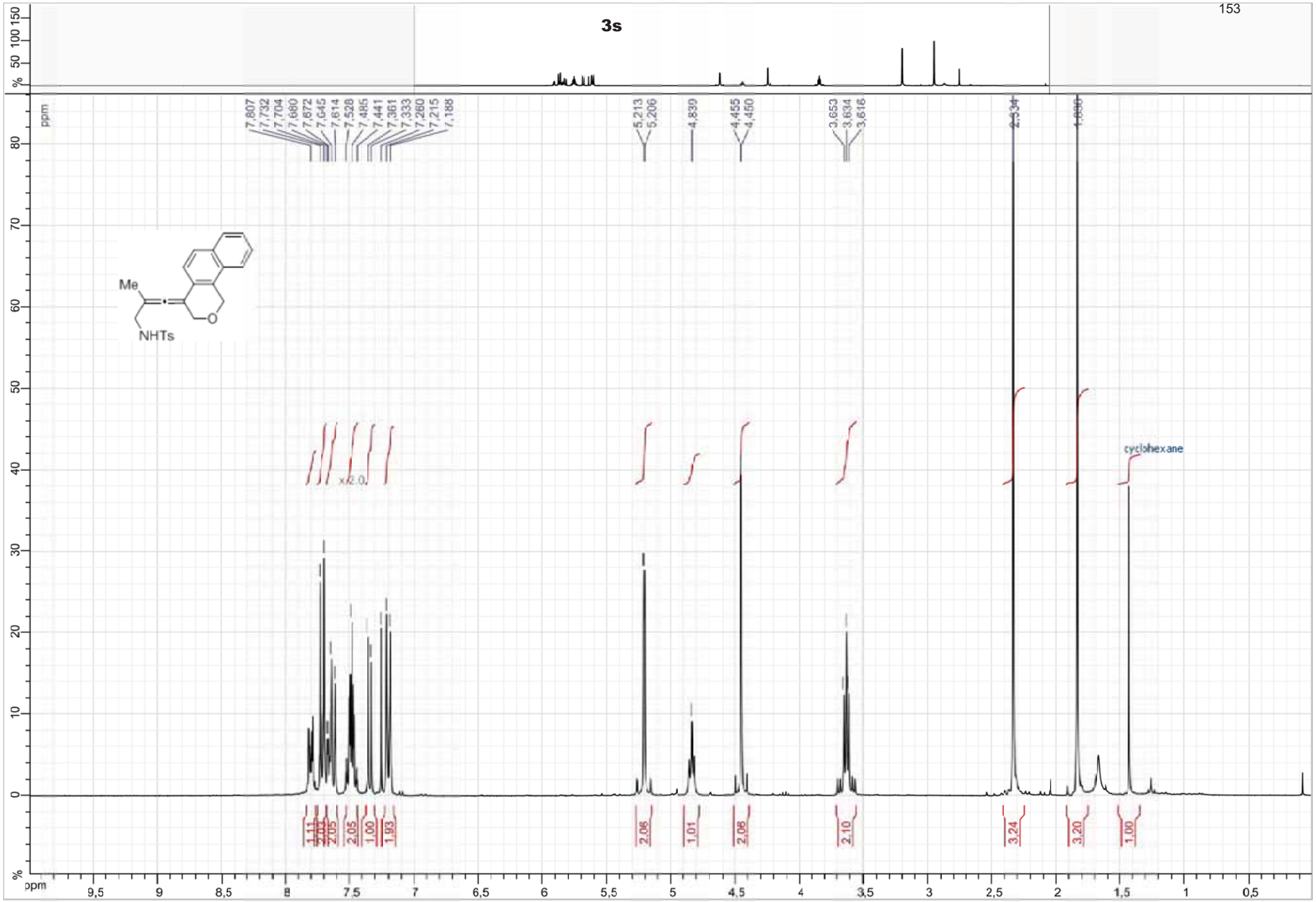

7,734
7,727
7,699
7,272
7,260
7,245
7,221
7,199
7,132
7,120
7,114
7,110
7,100
7,095
7,081
7,068
7,059
4,491
4,472
4,453
3,578
3,559
2,818
2,797
2,777
2,462
2,474
2,463
2,452
2,439
2,434
2,415
1,916
1,895
1,875
1,854
1,834
1,761
Me
NHTs
1,00
3,11
0,96
1,88
2,00
2,14
2,93
ppm
9,5
9
8,5
8
7,5
7
6,5
6
5,5
5
4,5
4
3,5
3
2,5
2
1,5
1
0,5


3m
150
100
50
%
ppm
197,249
143,382
136,910
136,834
131,397
129,706
129,333
127,125
127,011
126,805
126,028
105,461
99,483
77,469
77,248
77,050
76,624
45,723
30,022
29,062
23,089
21,562
16,932
Me
NHTs
120
100
80
60
40
20
0
%
ppm
200
180
160
140
120
100
80
60
40
20

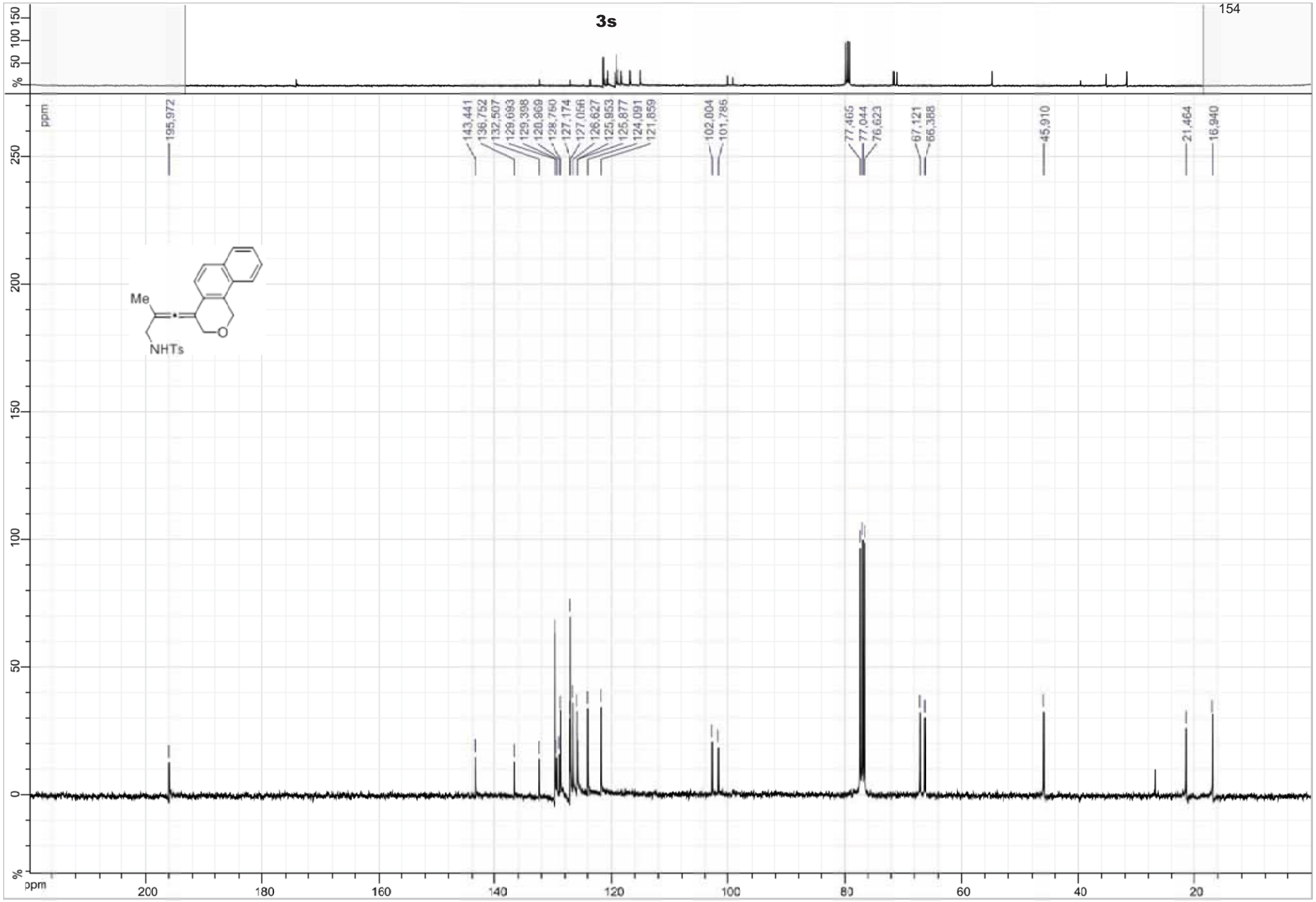

3q
ppm
7,673
7,646
7,260
7,175
7,148
7,059
7,031
6,766
6,721
6,679
5,326
4,200
3,700
3,624
3,491
3,318
3,289
3,058
3,053
3,011
3,005
2,839
2,791
2,384
1,752
1,278
1,254
1,230
1,173
1,149
1,128
1,059
MeO
Me
NHTs
CO2Et
CO2Et
2,00
1,83
1,23
2,01
1,05
4,31
2,09
1,18
1,07
1,97
1,01
1,00
3,08
3,14
3,32
3,16
cyclohexane peak
ppm

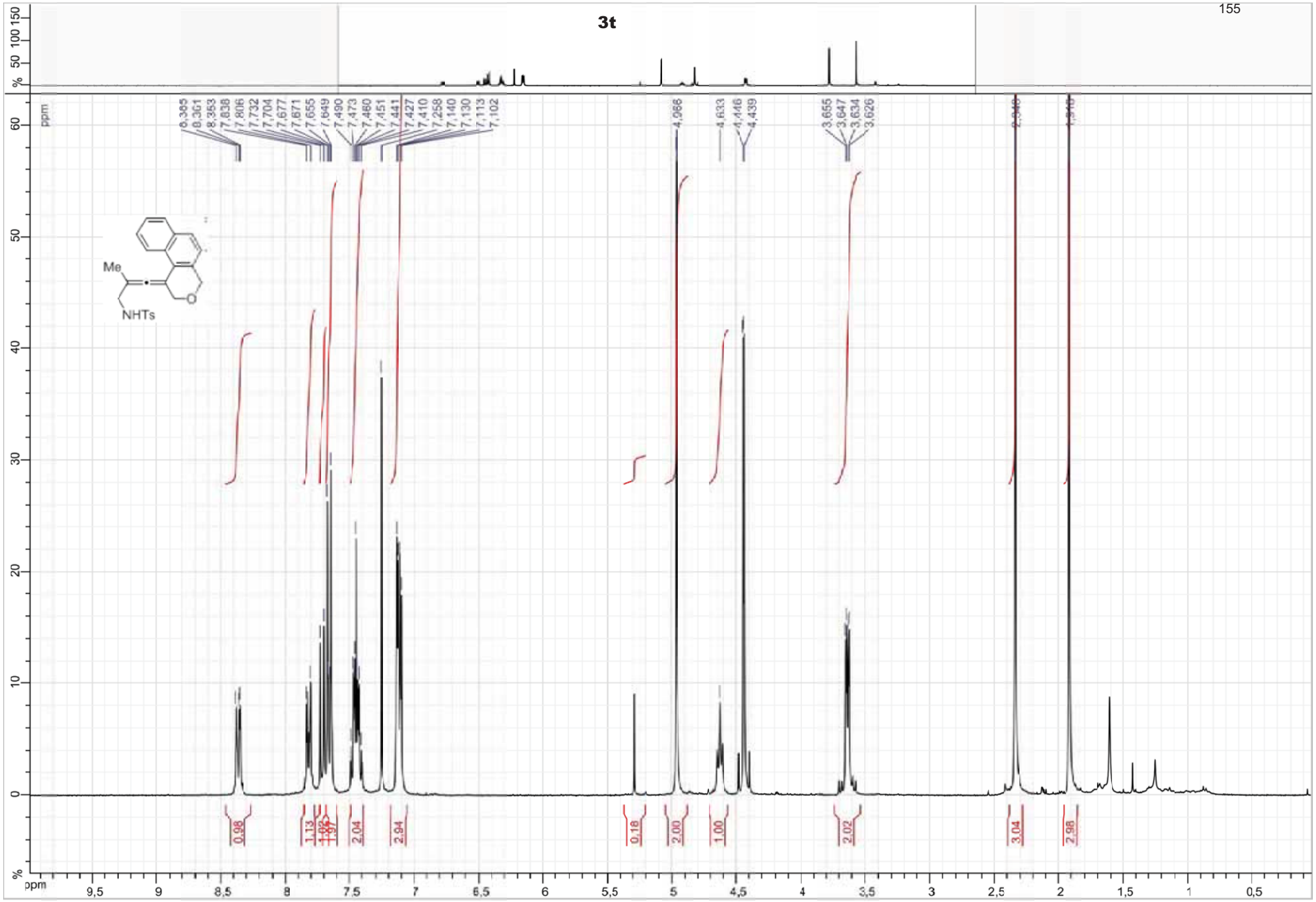

3q
152
ppm
197,923
170,833
170,761
158,175
142,959
136,986
131,089
130,043
129,536
128,792
127,080
125,197
114,267
110,902
103,330
100,821
77,366
77,250
77,048
76,731
62,072
61,820
55,163
53,979
45,502
34,451
21,483
16,707
14,076
13,983
MeO
Me
NHTs
CO2Et
CO2Et
%
ppm

3s


7,807
7,732
7,704
7,680
7,672
7,645
7,614
7,528
7,485
7,441
7,361
7,333
7,260
7,215
7,188
5,213
5,206
4,839
4,455
4,450
3,653
3,634
3,618
2,334
1,680
cyclohexane
Me
NHTs
O
x2.0
2,06
1,01
2,06
2,10
3,24
3,20
1,00
ppm

3s


ppm
195,972
143,441
136,752
132,507
129,693
129,398
128,969
128,760
127,174
127,056
126,627
125,953
125,877
124,091
121,859
102,004
101,785
77,465
77,044
76,623
67,121
66,388
45,910
21,464
16,940
Me
NHTs
O
%
ppm
200
180
160
140
120
100
80
60
40
20


3t
ppm
8,365
8,361
8,353
7,838
7,806
7,732
7,704
7,677
7,671
7,655
7,649
7,490
7,473
7,460
7,451
7,441
7,427
7,410
7,258
7,140
7,130
7,113
7,102
4,966
4,633
4,446
4,439
3,655
3,647
3,634
3,626
2,348
1,918
Me
NHTs
0,98
1,13
1,03
1,97
2,04
2,94
0,18
2,00
1,00
2,02
3,04
2,98
ppm
9,5
9
8,5
8
7,5
7
6,5
6
5,5
5
4,5
4
3,5
3
2,5
2
1,5
1
0,5

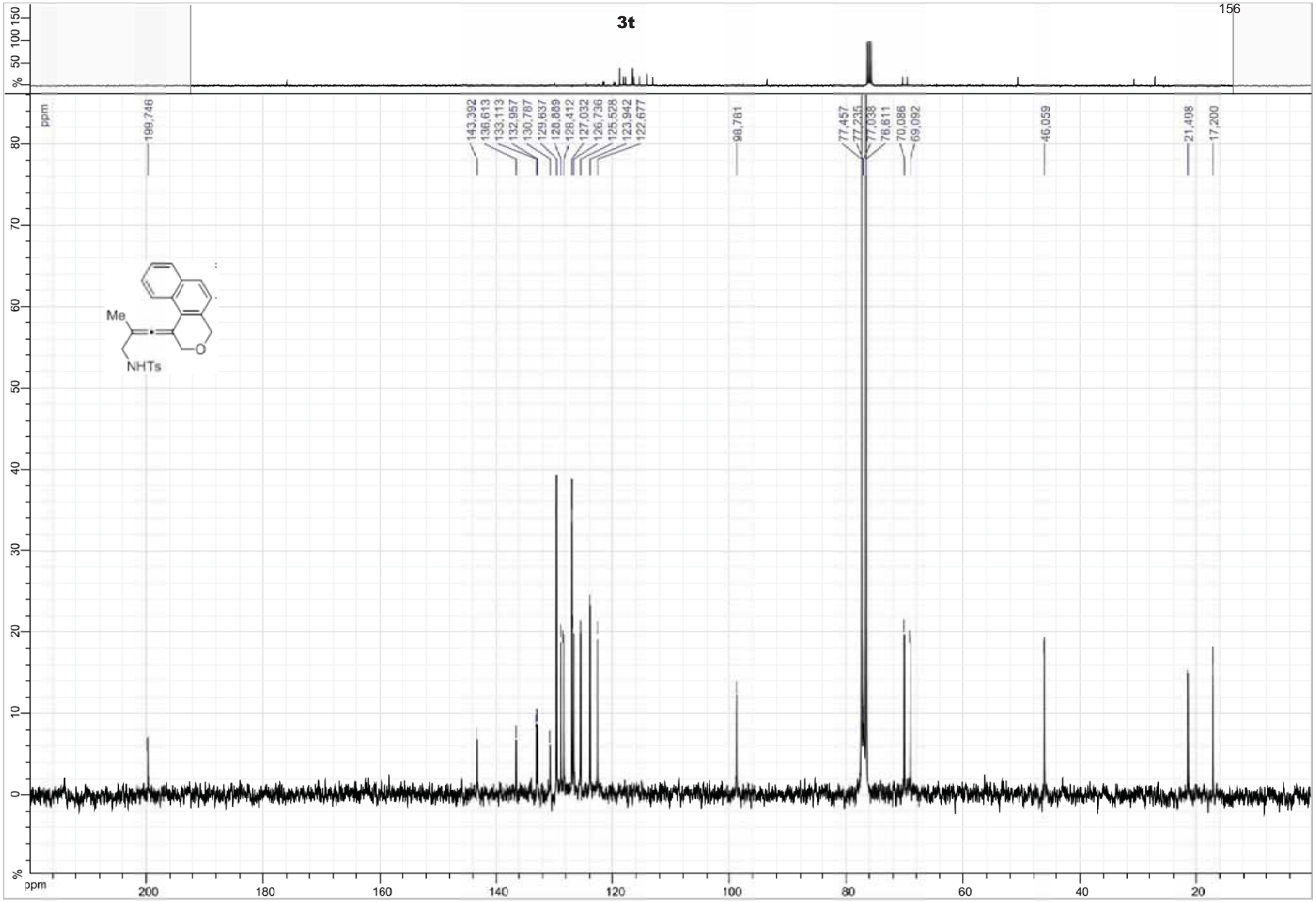
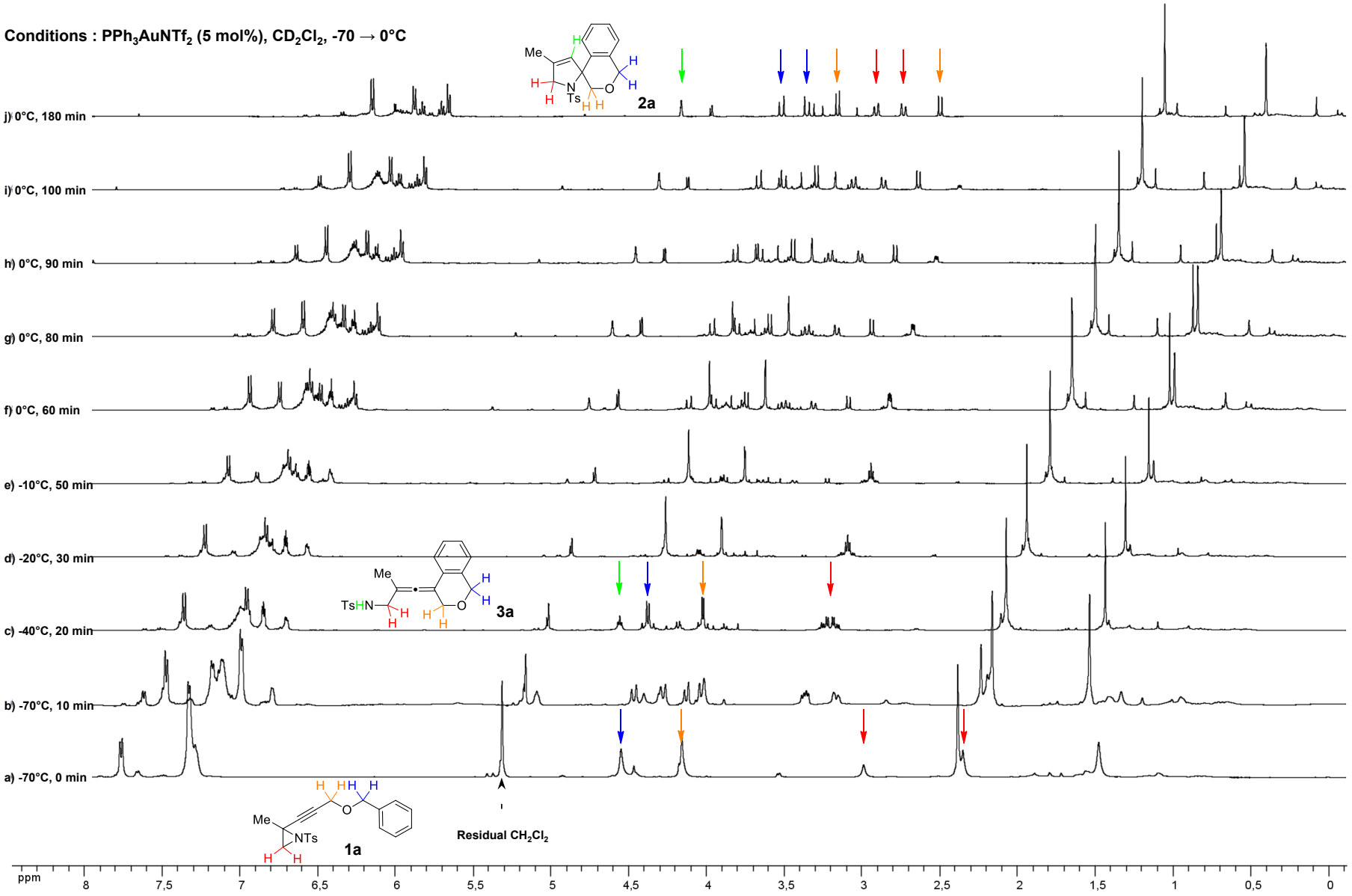
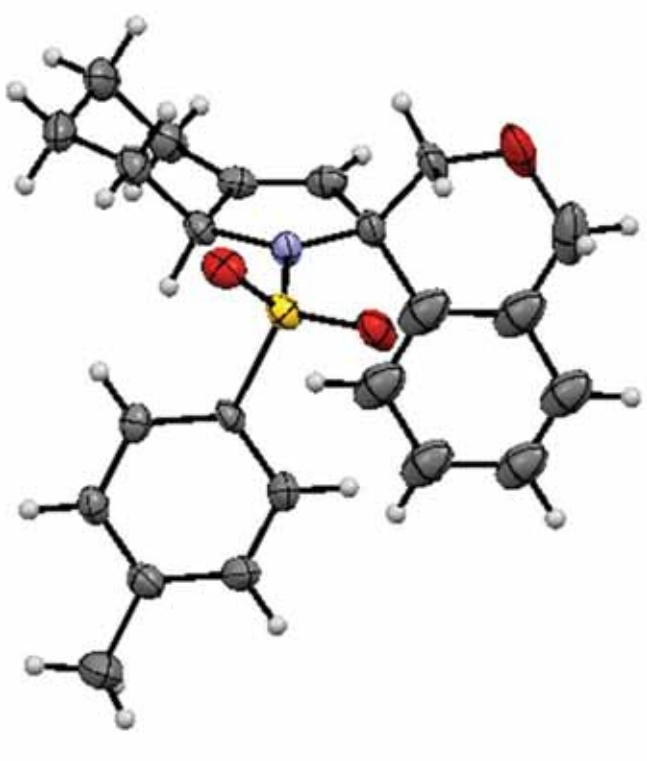

3t
156
199.746
143.392
136.613
133.113
132.957
130.787
129.637
128.889
128.412
127.032
126.736
125.528
123.942
122.677
98.781
77.457
77.235
77.038
76.611
70.086
69.092
46.059
21.498
17.200
Me
NHTs
O
ppm
200
180
160
140
120
100
80
60
40
20

Figure 2: ^{1}H NMR spectra of the transient intermediates **3a** in gold(I)-catalyzed formation of spiro[isochroman-4,2-pyrroline] **2a** (The ppm scale was calibrated on residual CH_2Cl_2 of the **a** spectrum).





Structure of
2h-maj determined from
X-ray diffraction

Table of crystal data and refinement details for **2h-maj**:

Table 1. Crystal data and structure refinement for ppnk110706.

Identification code	ppnk110706
Empirical formula	C23 H25 N O3 S
Formula weight	395.50
Temperature	173(2) K
Wavelength	0.71073 A
Crystal system, space group	Tetragonal, I -4
Unit cell dimensions	a = 22.5705(17) A alpha = 90 deg. b = 22.5705(17) A beta = 90 deg. c = 8.0007(6) A gamma = 90 deg.
Volume	4075.8(5) A^3
Z, Calculated density	8, 1.289 Mg/m^3
Absorption coefficient	0.182 mm^-1
F(000)	1680
Crystal size	0.50 x 0.08 x 0.06 mm
Theta range for data collection	1.80 to 28.02 deg.
Limiting indices	-26<=h<=29, -29<=k<=22, -10<=l<=9
Reflections collected / unique	25172 / 4936 [R(int) = 0.0911]
Completeness to theta = 28.02	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9891 and 0.9143
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4936 / 0 / 224
Goodness-of-fit on F^2	1.017
Final R indices [I>2sigma(I)]	R1 = 0.0597, wR2 = 0.1203
R indices (all data)	R1 = 0.1159, wR2 = 0.1413
Absolute structure parameter	0.12(12)
Largest diff. peak and hole	0.808 and -0.515 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for ppnk110706.
U(eq) is defined as one third of the trace of the orthogonalized

Uij tensor.

	x	y	z	U(eq)
C(1)	8220(2)	4428(2)	4041(4)	28(1)
C(2)	8601(2)	4978(2)	4343(5)	35(1)
C(3)	9073(2)	4834(2)	5659(5)	41(1)
C(4)	9468(2)	4326(2)	5093(5)	42(1)
C(5)	9101(2)	3763(2)	4825(5)	38(1)
C(6)	8598(2)	3897(2)	3642(5)	30(1)
C(7)	8441(2)	3643(2)	2242(5)	31(1)
C(8)	7918(2)	3930(2)	1404(4)	29(1)
C(9)	8072(2)	4161(2)	-355(4)	39(1)
C(10)	7488(2)	3479(2)	-1778(6)	60(1)
C(11)	7193(2)	3303(2)	-198(7)	54(1)
C(12)	7397(2)	3506(2)	1342(7)	54(1)
C(13)	7140(2)	3297(2)	2810(7)	54(1)
C(14)	6660(2)	2910(2)	2790(7)	54(1)
C(15)	6445(2)	2736(2)	1234(7)	54(1)
C(16)	6700(2)	2915(2)	-194(7)	54(1)
C(17)	6828(2)	4760(2)	4247(4)	24(1)
C(18)	6954(2)	5027(2)	5765(5)	29(1)
C(19)	6600(2)	4907(2)	7139(4)	30(1)
C(20)	6119(2)	4529(2)	7007(4)	33(1)
C(21)	5990(2)	4278(2)	5471(5)	37(1)
C(22)	6341(2)	4388(2)	4091(5)	32(1)
C(23)	5733(2)	4394(2)	8496(6)	51(1)
O(1)	8078(1)	3686(1)	-1520(4)	52(1)
O(2)	7501(1)	5499(1)	2740(3)	36(1)
O(3)	6948(1)	4779(1)	1021(3)	38(1)
S(1)	7277(1)	4911(1)	2496(1)	28(1)
N(1)	7833(1)	4455(1)	2539(4)	25(1)

Table 3. Bond lengths [A] and angles [deg] for ppnk110706.

C(1)-N(1)	1.486(4)
C(1)-C(6)	1.506(5)
C(1)-C(2)	1.531(5)
C(1)-H(1)	1.0000
C(2)-C(3)	1.533(5)
C(2)-H(2A)	0.9900
C(2)-H(2B)	0.9900
C(3)-C(4)	1.521(6)
C(3)-H(3A)	0.9900
C(3)-H(3B)	0.9900
C(4)-C(5)	1.532(6)
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(5)-C(6)	1.508(5)
C(5)-H(5A)	0.9900
C(5)-H(5B)	0.9900
C(6)-C(7)	1.306(5)
C(7)-C(8)	1.504(5)
C(7)-H(7)	0.9500
C(8)-N(1)	1.505(4)
C(8)-C(12)	1.518(6)

C(8)-C(9)	1.541(5)
C(9)-O(1)	1.420(5)
C(9)-H(9A)	0.9900
C(9)-H(9B)	0.9900
C(10)-O(1)	1.426(5)
C(10)-C(11)	1.482(7)
C(10)-H(10A)	0.9900
C(10)-H(10B)	0.9900
C(11)-C(12)	1.392(7)
C(11)-C(16)	1.417(6)
C(12)-C(13)	1.392(7)
C(13)-C(14)	1.392(6)
C(13)-H(13)	0.9500
C(14)-C(15)	1.393(7)
C(14)-H(14)	0.9500
C(15)-C(16)	1.342(7)
C(15)-H(15)	0.9500
C(16)-H(16)	0.9500
C(17)-C(18)	1.384(5)
C(17)-C(22)	1.390(5)
C(17)-S(1)	1.762(3)
C(18)-C(19)	1.385(5)
C(18)-H(18)	0.9500
C(19)-C(20)	1.385(5)
C(19)-H(19)	0.9500
C(20)-C(21)	1.385(5)
C(20)-C(23)	1.507(5)
C(21)-C(22)	1.381(5)
C(21)-H(21)	0.9500
C(22)-H(22)	0.9500
C(23)-H(23A)	0.9800
C(23)-H(23B)	0.9800
C(23)-H(23C)	0.9800
O(2)-S(1)	1.434(2)
O(3)-S(1)	1.426(3)
S(1)-N(1)	1.626(3)

N(1)-C(1)-C(6)	101.1(3)
N(1)-C(1)-C(2)	115.1(3)
C(6)-C(1)-C(2)	111.2(3)
N(1)-C(1)-H(1)	109.7
C(6)-C(1)-H(1)	109.7
C(2)-C(1)-H(1)	109.7
C(1)-C(2)-C(3)	109.1(3)
C(1)-C(2)-H(2A)	109.9
C(3)-C(2)-H(2A)	109.9
C(1)-C(2)-H(2B)	109.9
C(3)-C(2)-H(2B)	109.9
H(2A)-C(2)-H(2B)	108.3
C(4)-C(3)-C(2)	111.3(3)
C(4)-C(3)-H(3A)	109.4
C(2)-C(3)-H(3A)	109.4
C(4)-C(3)-H(3B)	109.4
C(2)-C(3)-H(3B)	109.4
H(3A)-C(3)-H(3B)	108.0
C(3)-C(4)-C(5)	110.4(3)
C(3)-C(4)-H(4A)	109.6
C(5)-C(4)-H(4A)	109.6
C(3)-C(4)-H(4B)	109.6
C(5)-C(4)-H(4B)	109.6
H(4A)-C(4)-H(4B)	108.1

C(6)-C(5)-C(4)	109.3(3)
C(6)-C(5)-H(5A)	109.8
C(4)-C(5)-H(5A)	109.8
C(6)-C(5)-H(5B)	109.8
C(4)-C(5)-H(5B)	109.8
H(5A)-C(5)-H(5B)	108.3
C(7)-C(6)-C(1)	112.1(3)
C(7)-C(6)-C(5)	130.9(3)
C(1)-C(6)-C(5)	116.9(3)
C(6)-C(7)-C(8)	114.0(3)
C(6)-C(7)-H(7)	123.0
C(8)-C(7)-H(7)	123.0
C(7)-C(8)-N(1)	99.8(3)
C(7)-C(8)-C(12)	110.6(3)
N(1)-C(8)-C(12)	114.7(3)
C(7)-C(8)-C(9)	112.1(3)
N(1)-C(8)-C(9)	108.3(3)
C(12)-C(8)-C(9)	111.0(3)
O(1)-C(9)-C(8)	110.2(3)
O(1)-C(9)-H(9A)	109.6
C(8)-C(9)-H(9A)	109.6
O(1)-C(9)-H(9B)	109.6
C(8)-C(9)-H(9B)	109.6
H(9A)-C(9)-H(9B)	108.1
O(1)-C(10)-C(11)	112.6(4)
O(1)-C(10)-H(10A)	109.1
C(11)-C(10)-H(10A)	109.1
O(1)-C(10)-H(10B)	109.1
C(11)-C(10)-H(10B)	109.1
H(10A)-C(10)-H(10B)	107.8
C(12)-C(11)-C(16)	117.4(5)
C(12)-C(11)-C(10)	121.3(4)
C(16)-C(11)-C(10)	121.3(5)
C(13)-C(12)-C(11)	119.9(4)
C(13)-C(12)-C(8)	120.6(4)
C(11)-C(12)-C(8)	119.5(5)
C(12)-C(13)-C(14)	121.8(5)
C(12)-C(13)-H(13)	119.1
C(14)-C(13)-H(13)	119.1
C(13)-C(14)-C(15)	117.3(5)
C(13)-C(14)-H(14)	121.4
C(15)-C(14)-H(14)	121.4
C(16)-C(15)-C(14)	121.8(4)
C(16)-C(15)-H(15)	119.1
C(14)-C(15)-H(15)	119.1
C(15)-C(16)-C(11)	121.7(5)
C(15)-C(16)-H(16)	119.1
C(11)-C(16)-H(16)	119.1
C(18)-C(17)-C(22)	120.2(3)
C(18)-C(17)-S(1)	119.7(3)
C(22)-C(17)-S(1)	120.0(3)
C(17)-C(18)-C(19)	119.5(3)
C(17)-C(18)-H(18)	120.2
C(19)-C(18)-H(18)	120.2
C(18)-C(19)-C(20)	120.8(3)
C(18)-C(19)-H(19)	119.6
C(20)-C(19)-H(19)	119.6
C(21)-C(20)-C(19)	119.0(3)
C(21)-C(20)-C(23)	119.8(4)
C(19)-C(20)-C(23)	121.2(3)
C(22)-C(21)-C(20)	121.0(4)

C(22)-C(21)-H(21)	119.5
C(20)-C(21)-H(21)	119.5
C(21)-C(22)-C(17)	119.4(3)
C(21)-C(22)-H(22)	120.3
C(17)-C(22)-H(22)	120.3
C(20)-C(23)-H(23A)	109.5
C(20)-C(23)-H(23B)	109.5
H(23A)-C(23)-H(23B)	109.5
C(20)-C(23)-H(23C)	109.5
H(23A)-C(23)-H(23C)	109.5
H(23B)-C(23)-H(23C)	109.5
C(9)-O(1)-C(10)	109.5(3)
O(3)-S(1)-O(2)	119.40(17)
O(3)-S(1)-N(1)	106.67(16)
O(2)-S(1)-N(1)	108.12(14)
O(3)-S(1)-C(17)	108.59(15)
O(2)-S(1)-C(17)	105.88(17)
N(1)-S(1)-C(17)	107.72(17)
C(1)-N(1)-C(8)	112.4(2)
C(1)-N(1)-S(1)	119.8(2)
C(8)-N(1)-S(1)	125.8(2)

Symmetry transformations used to generate equivalent atoms: