Electronic supplementary information (ESI)

Enantioselective cycloaddition of carbonyl ylides with arylallenes using Rh_2(*S*-TCPTTL)$_4$

Janagiraman Krishnamurthi, Hisanori Nambu, Koji Takeda, Masahiro Anada, Akihito Yamano and Shunichi Hashimoto*

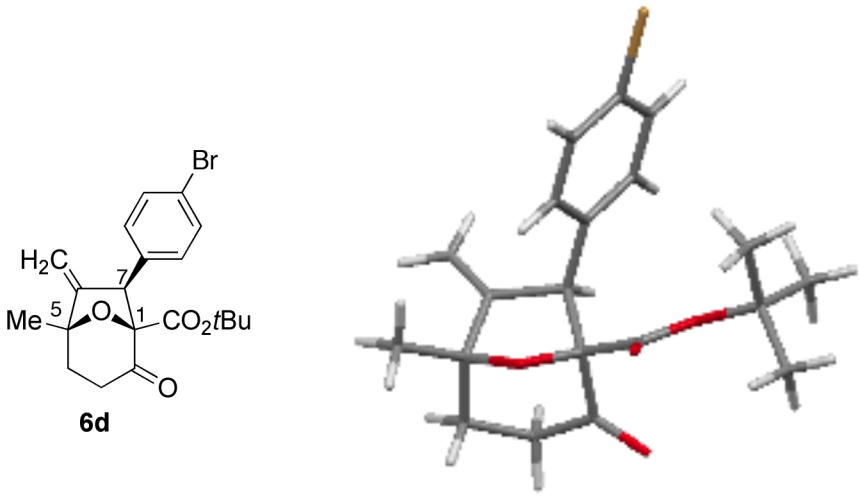


Figure S1. X-ray structure of **6d**.

HOMO and LUMO energies and coefficients for carbonyl ylide and dipolarophiles.

Molecular orbital calculations were performed by Workspace, FUJITSU-SCIGRESS 2.3.0 version after geometry optimization using MM2 (molecular mechanics) followed by MOPAC with AM1 parameters. From frontier molecular orbital (FMO) analysis for these reactions in the absence of a catalyst, the chemo- and regiocontrol observed can be readily rationalized in terms of maximum overlap of the LUMO of carbonyl ylides derived from **4a**–**c** and the HOMO of arylallene dipolarophiles **5a**–**d** and **5f**–**i**. FMO analysis, however, fails to explain the same regiochemistry encountered with a strongly electron-deficient *p*-(trifluoromethyl)phenylallene (**5e**). The calculations suggest that the favored cycloadduct should be the result of interaction between the HOMO of carbonyl ylides derived from **4a**–**c** and the LUMO of **5e** rather than the dipole LUMO-dipolarophile HOMO interaction. One possible explanation for the exclusive formation of **6e**, **6m** and **6q** is that association of Rh_2(*S*-TCPTTL)$_4$ with the carbonyl ylide leads to a significant lowering of the LUMO energy level compared with the

catalyst-free carbonyl ylide and facilitates the interaction between its LUMO and the HOMO of **5e**, which predicts the regiochemistry exactly as observed.

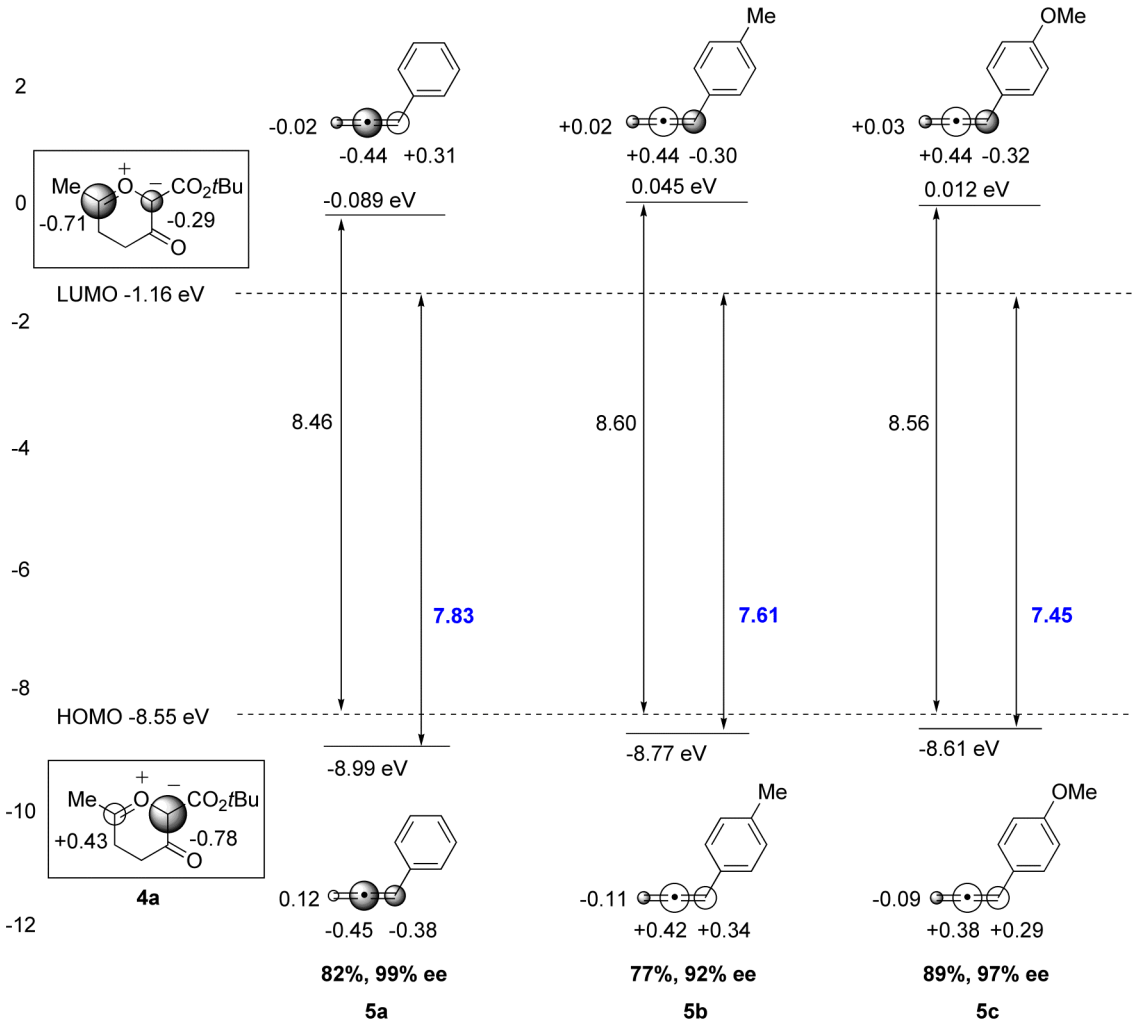


Fig. S2 Frontier orbital energies of carbonyl ylide derived from α-diazo-β-ketoester (**4a**) and arylallenes **5**.

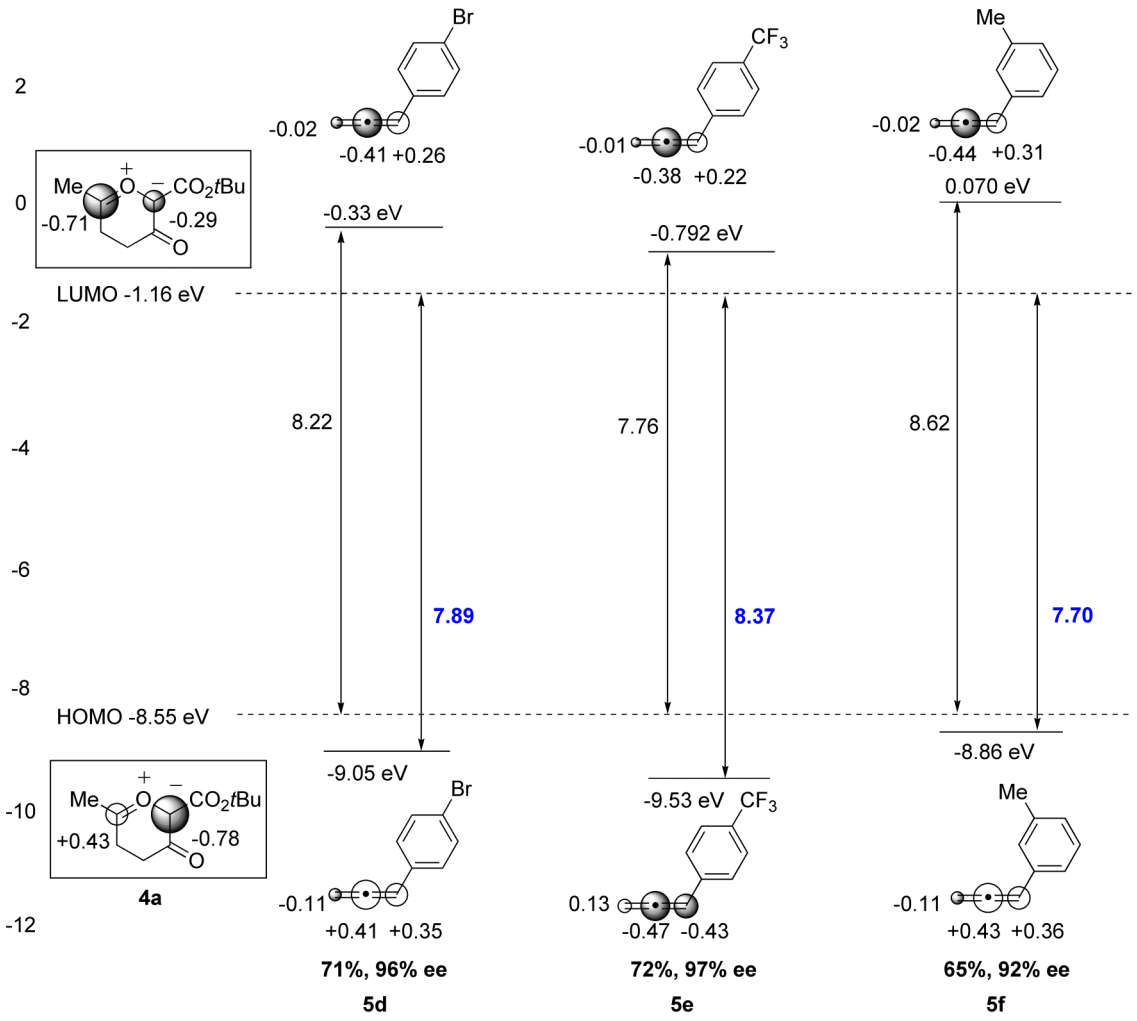


Fig. S3 Frontier orbital energies of carbonyl ylide derived from α-diazo-β-ketoester (**4a**) and arylallenes **5**.

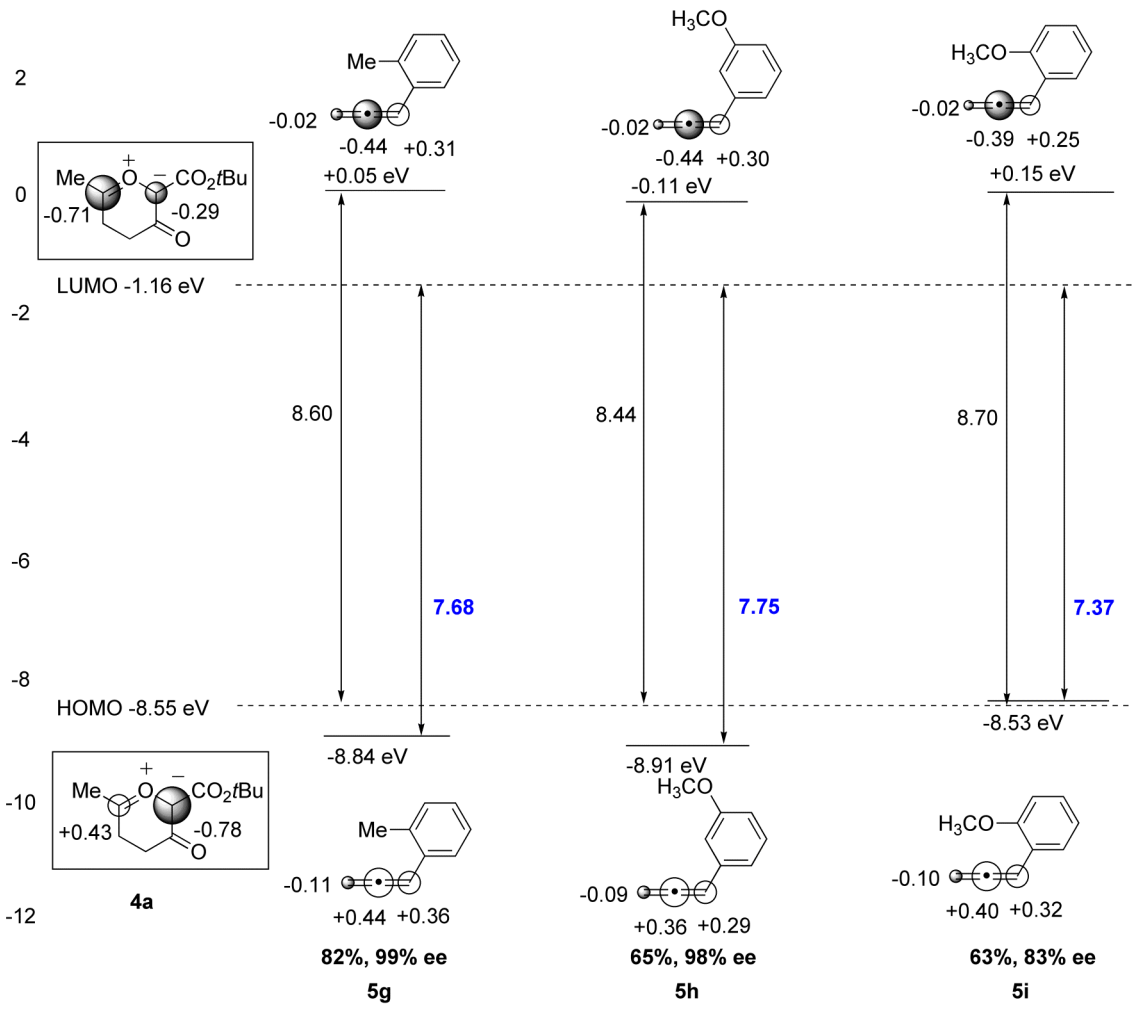


Fig. S4 Frontier orbital energies of carbonyl ylide derived from α-diazo-β-ketoester (**4a**) and arylallenes **5**.

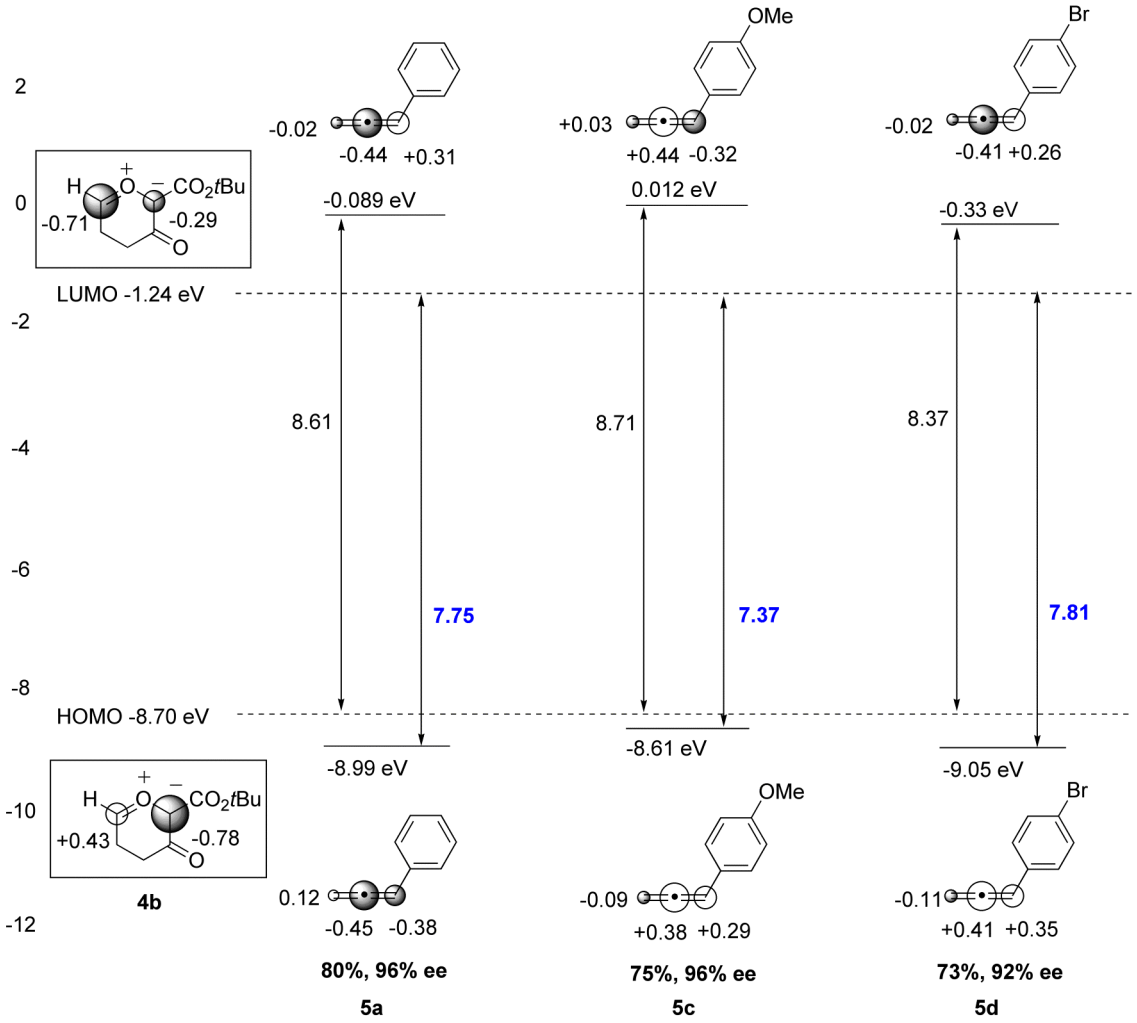


Fig. S5 Frontier orbital energies of carbonyl ylide derived from α-diazo-β-ketoester (**4b**) and arylallenes **5**.

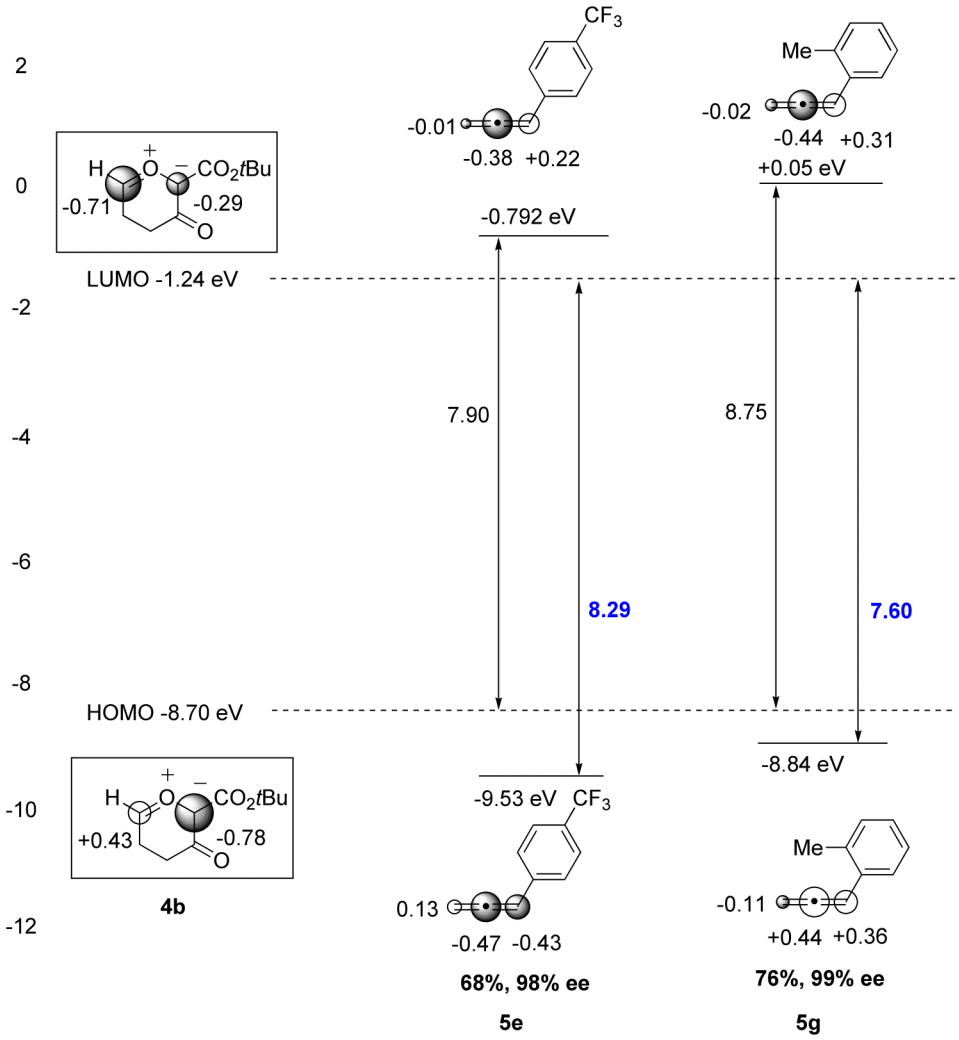


Fig. S6 Frontier orbital energies of carbonyl ylide derived from α-diazo-β-ketoester (**4b**) and arylallenes **5**.

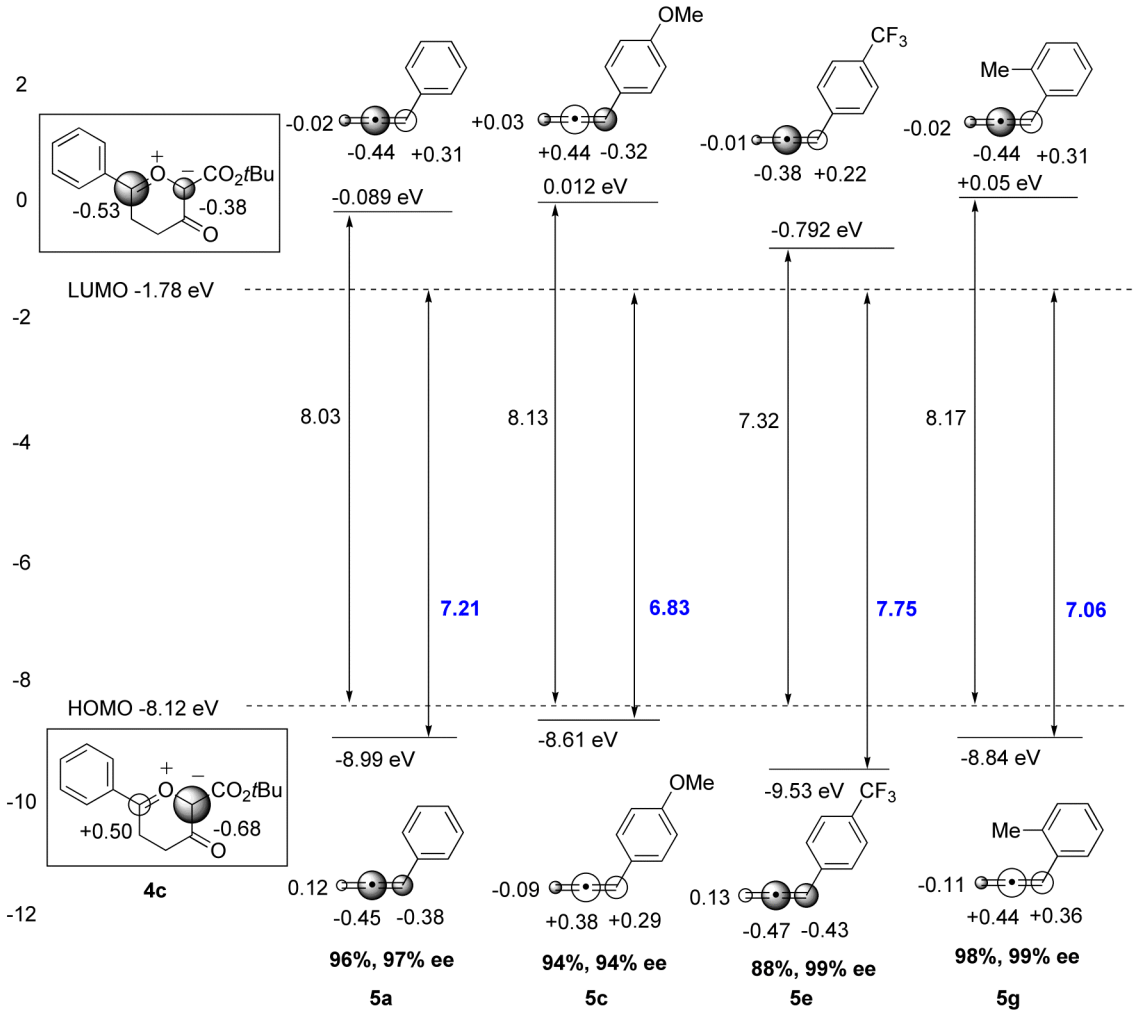
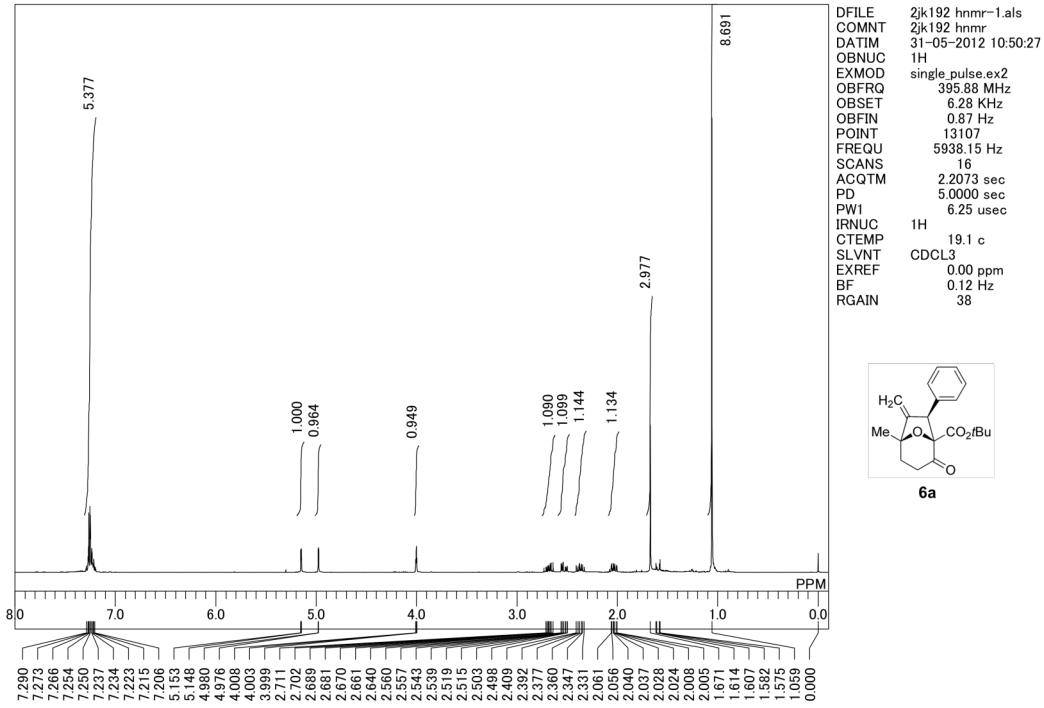


Fig. S7 Frontier orbital energies of carbonyl ylide derived from α-diazo-β-ketoester (**4c**) and arylallenes **5**.



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FREQU 5938.15 Hz
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PD 5.0000 sec
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CTEMP 19.1 c
SLVNT CDCL3
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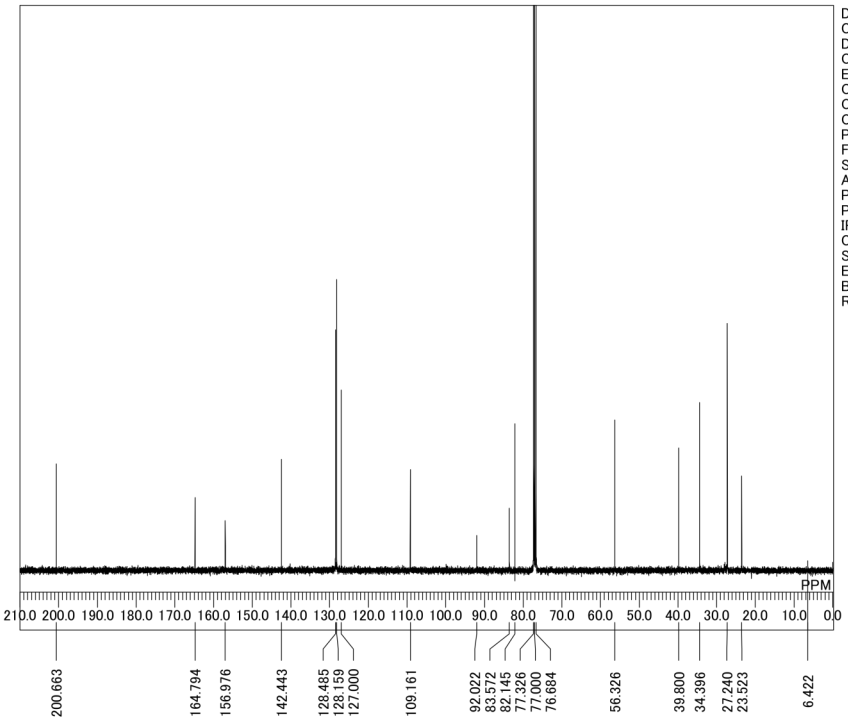
BF 0.12 Hz
RGAIN 38

6a



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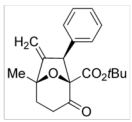
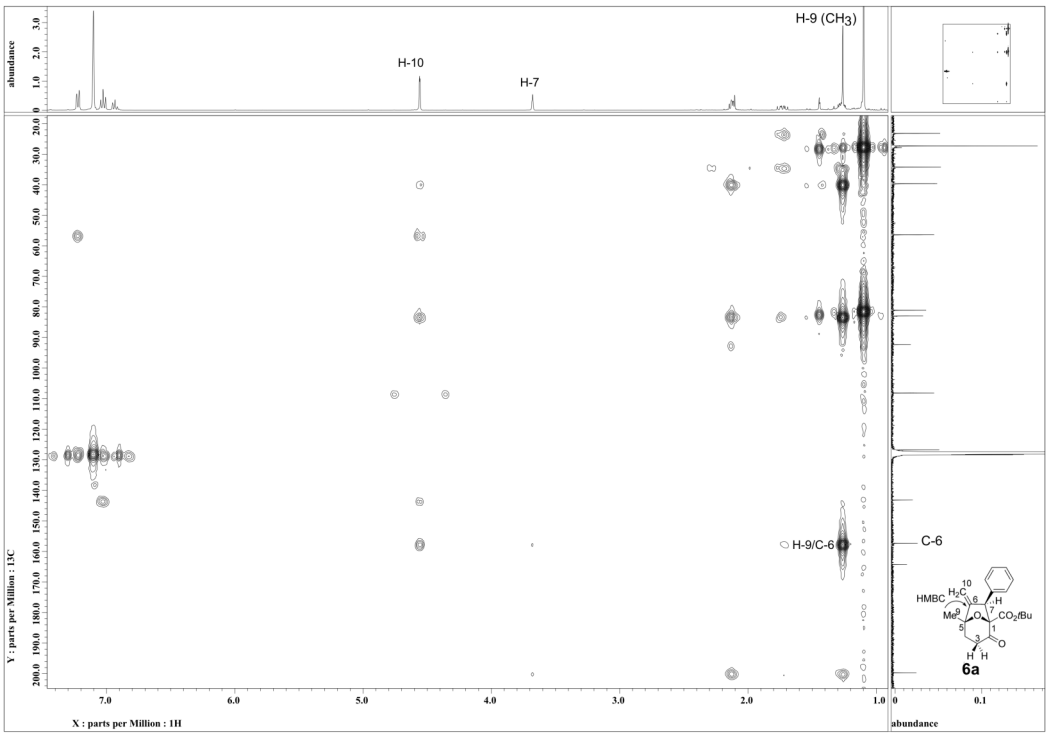


6a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H-7
H-9/C-6
C-6
HMBC
6a
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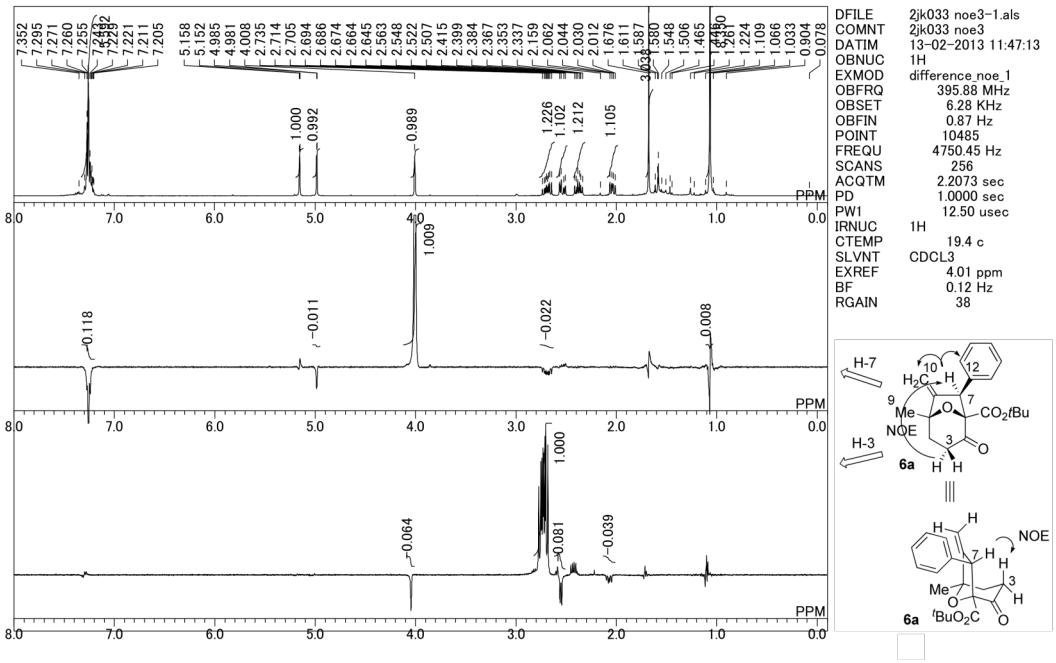
Y : parts per Million : 13C
abundance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PD 1.0000 sec
PW1 12.50 usec
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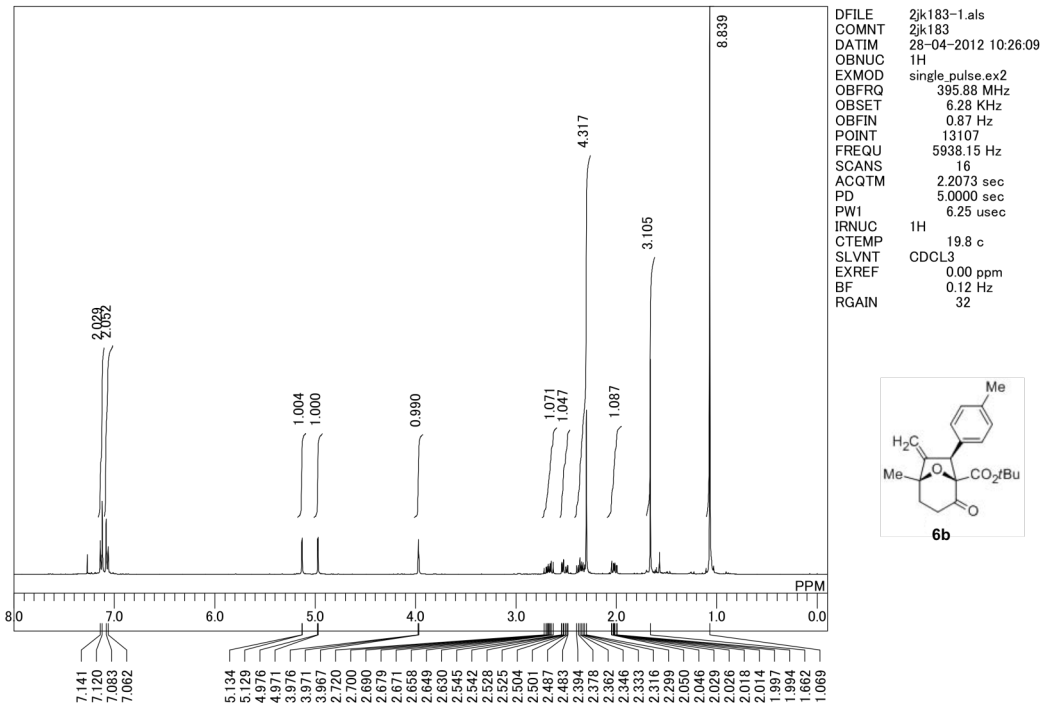
BF 0.12 Hz
RGAIN 38
H-7
H-3
NOE
6a
PPM



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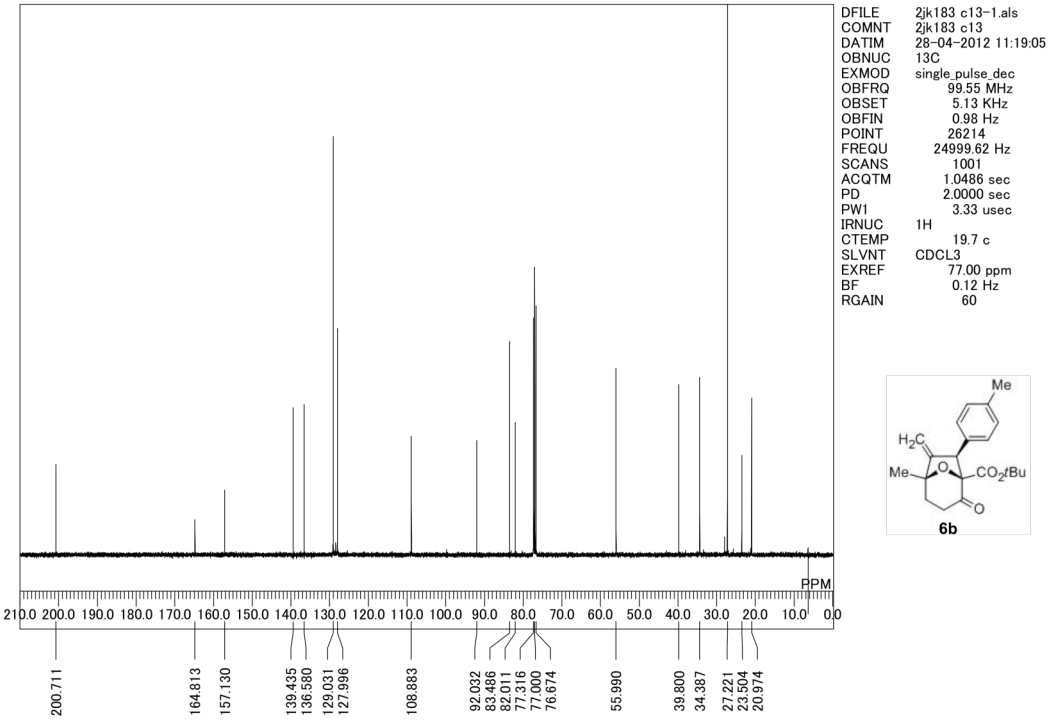
6b



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PD 2.0000 sec
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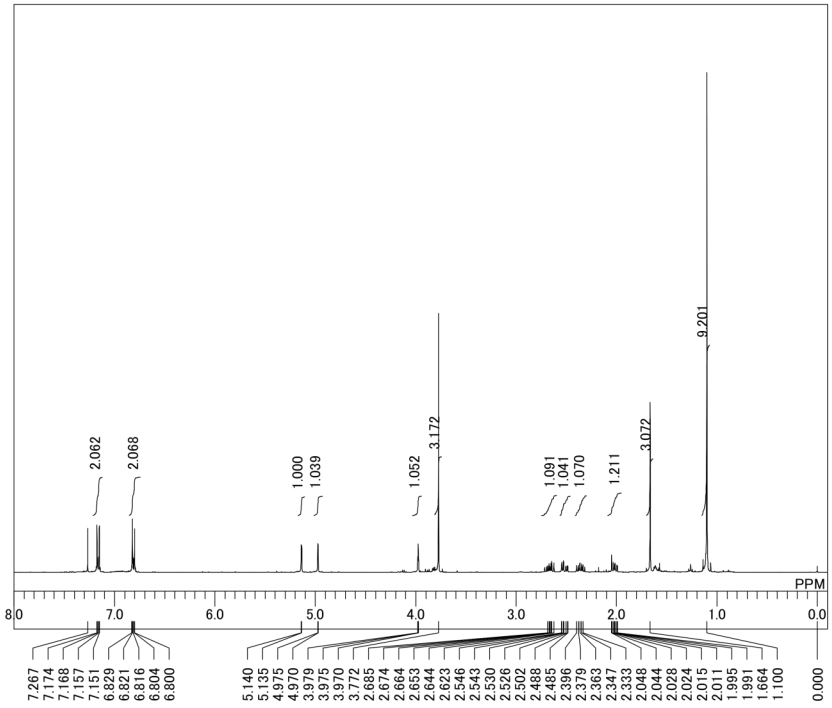
RGAIN 60

6b



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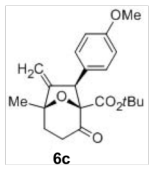
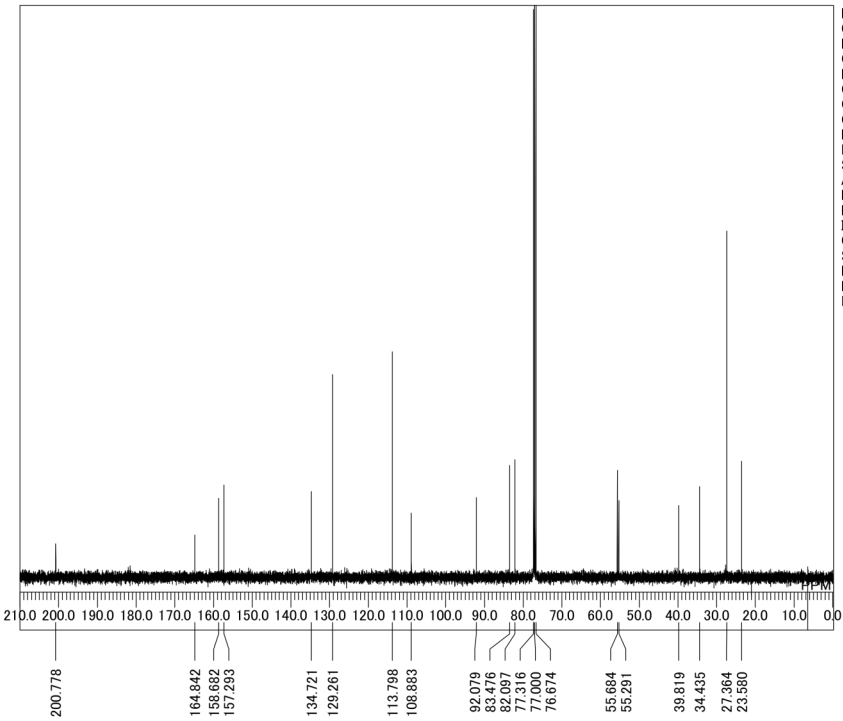
RGAIN 38



6c



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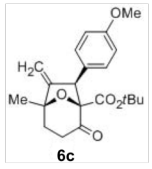
RGAIN 60



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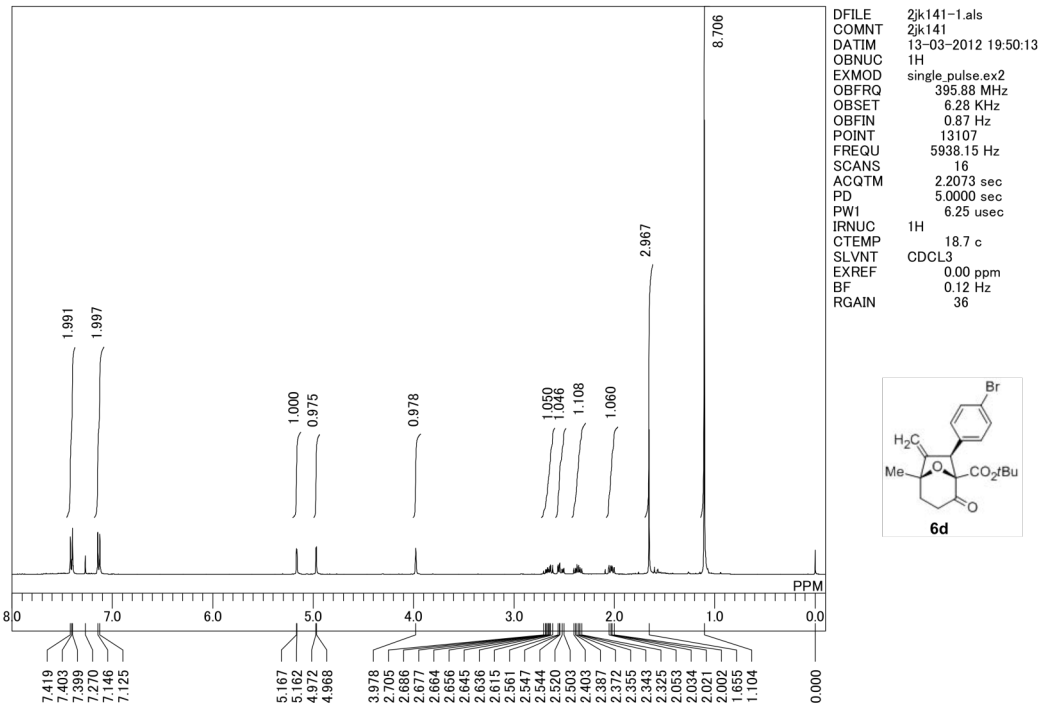
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7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
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7.403
7.399
7.270
7.146
7.125
5.167
5.162
4.972
4.968
3.978
2.705
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2.403
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ACQTM 2.2073 sec
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PW1 6.25 usec
IRNUC 1H
CTEMP 18.7 c
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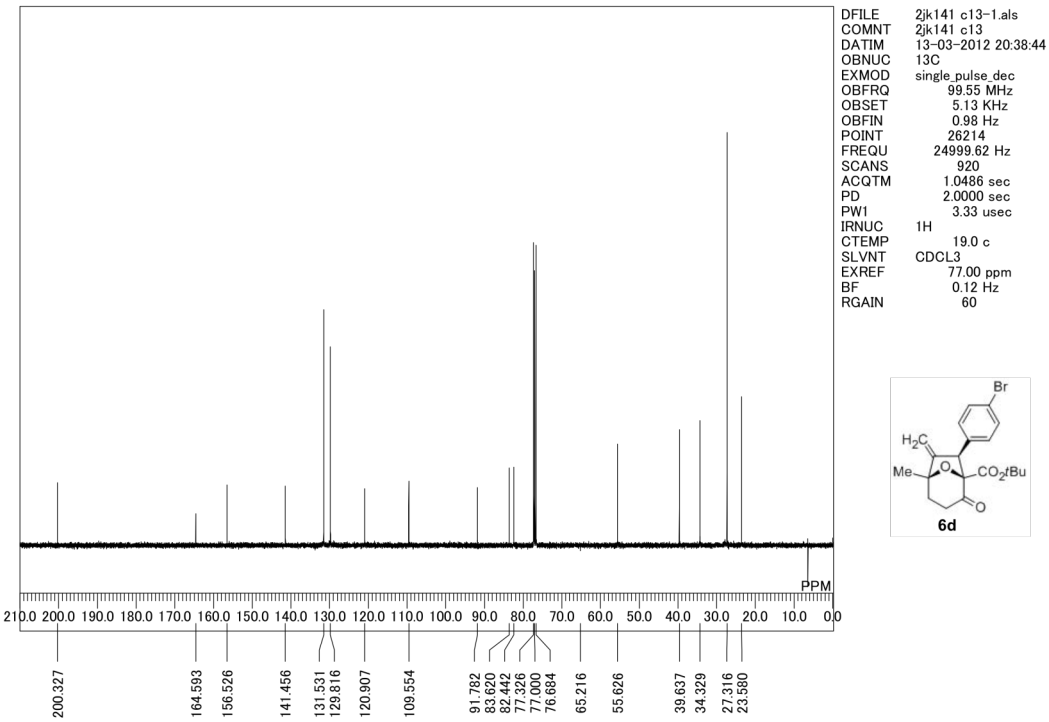
Me
O
CO2tBu
O
6d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164.593
156.526
141.456
131.531
129.816
120.907
109.554
91.782
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82.442
77.326
77.000
76.684
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34.329
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23.580
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PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 19.0 c
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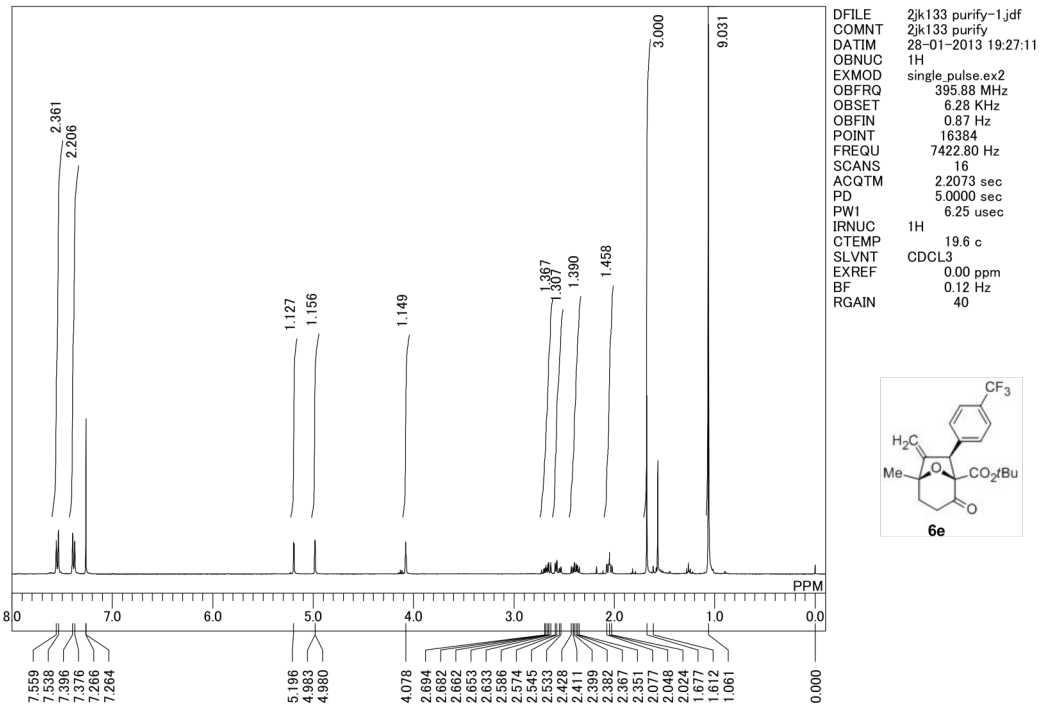
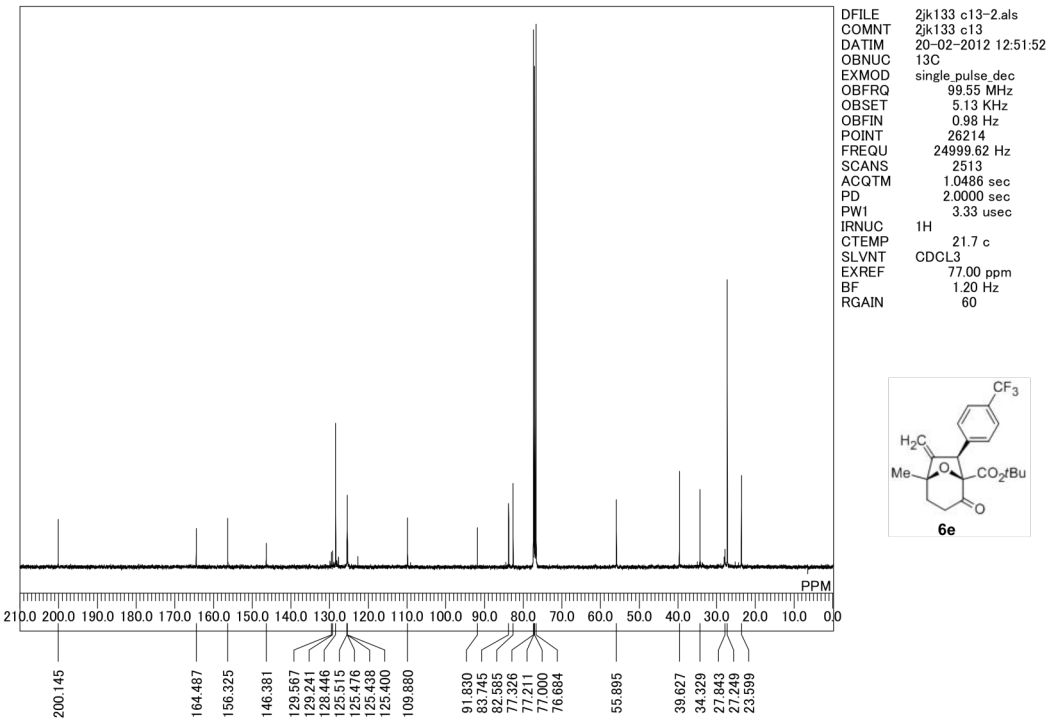
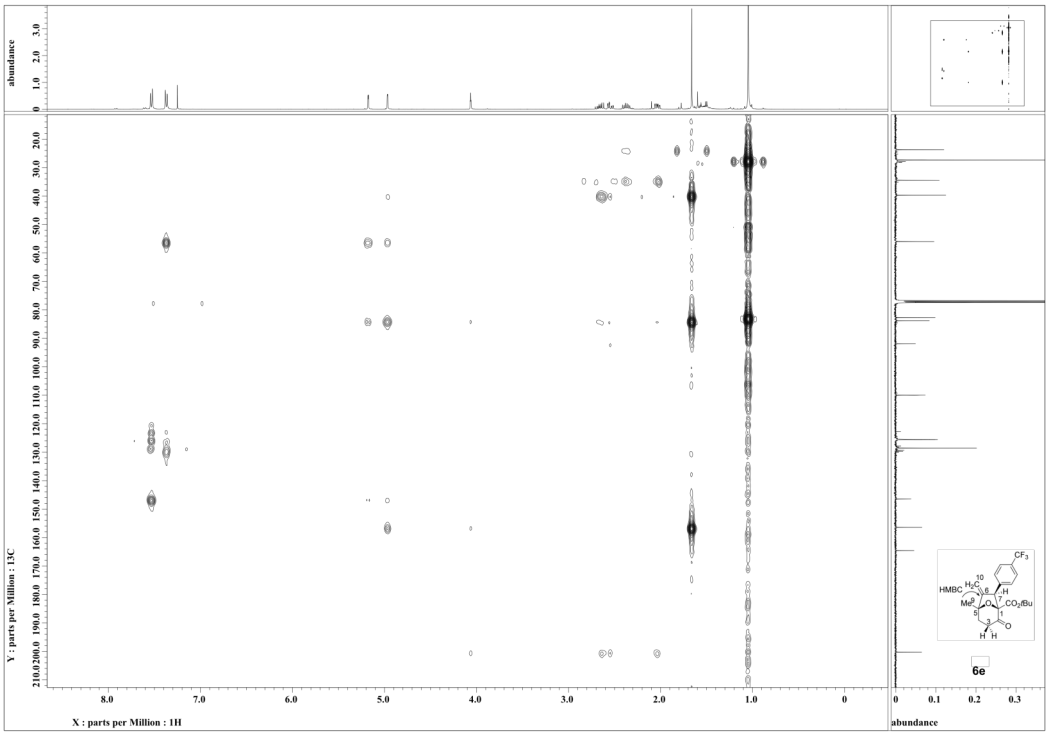
H2C
Me
O
CO2tBu
O
6d



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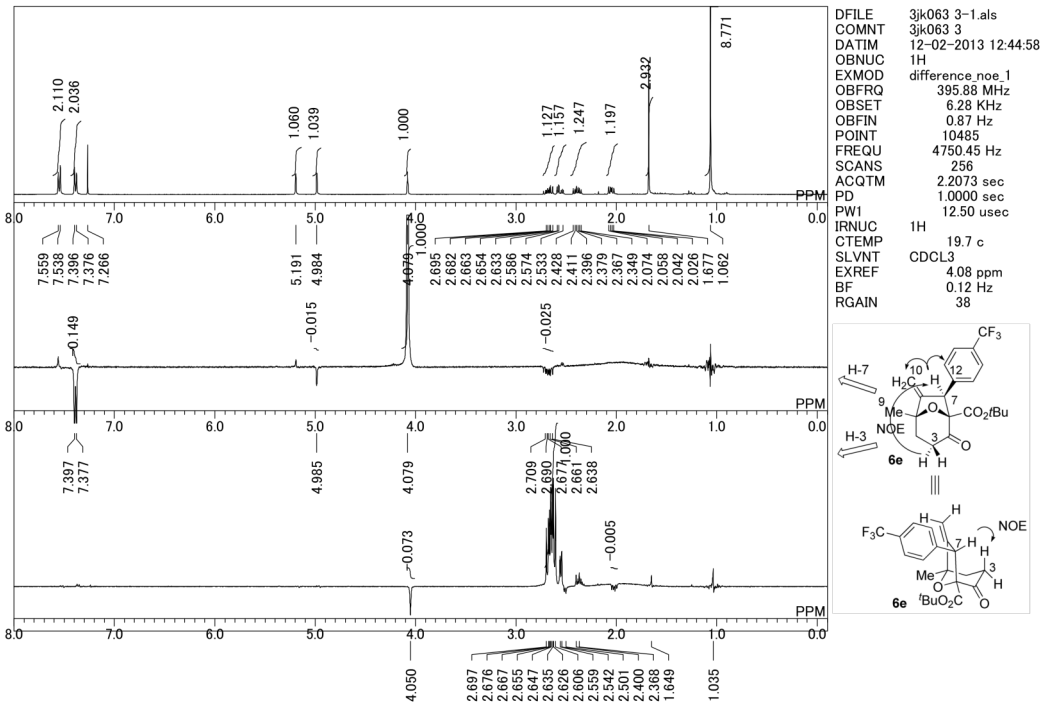
Y : parts per Million : 13C
X : parts per Million : 1H
HMBC
6e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POINT 10485
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ACQTM 2.2073 sec
PD 1.0000 sec
PW1 12.50 usec
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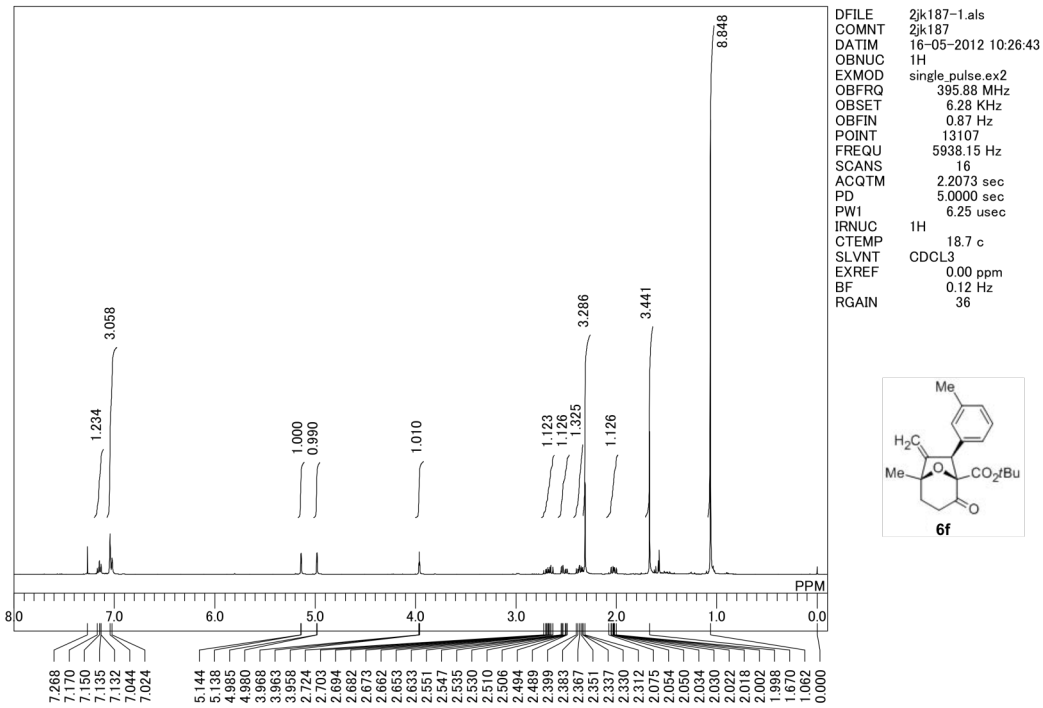
RGAIN 38
H-7
H-3
NOE
6e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OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 16
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.25 usec
IRNUC 1H
CTEMP 18.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 36
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3.058
1.234
1.000
0.990
1.010
1.123
1.126
1.325
1.126
PPM
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7.268 7.170 7.150 7.135 7.132 7.044 7.024
5.144 5.138 4.985 4.980 3.968 3.963 3.958 2.724 2.703 2.694 2.682 2.673 2.662 2.653 2.633 2.551 2.547 2.535 2.530 2.510 2.506 2.494 2.489 2.399 2.383 2.367 2.351 2.337 2.330 2.312 2.075 2.054 2.050 2.034 2.030 2.022 2.018 2.002 1.998 1.670 1.062 0.000
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H2C
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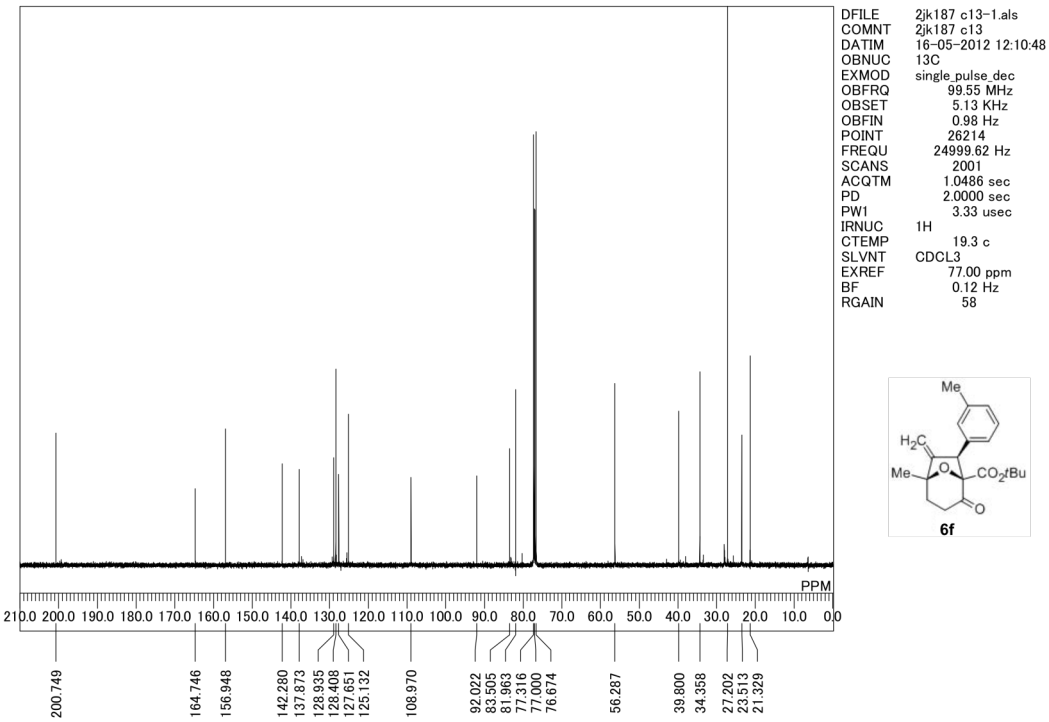
CO2tBu
O
6f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OBFIN 0.98 Hz
POINT 26214
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ACQTM 1.0486 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 19.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
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PPM
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164.746 156.948
142.280 137.873 128.935 128.408 127.651 125.132
108.970
92.022 83.505 81.963 77.316 77.000 76.674
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CO2tBu
O
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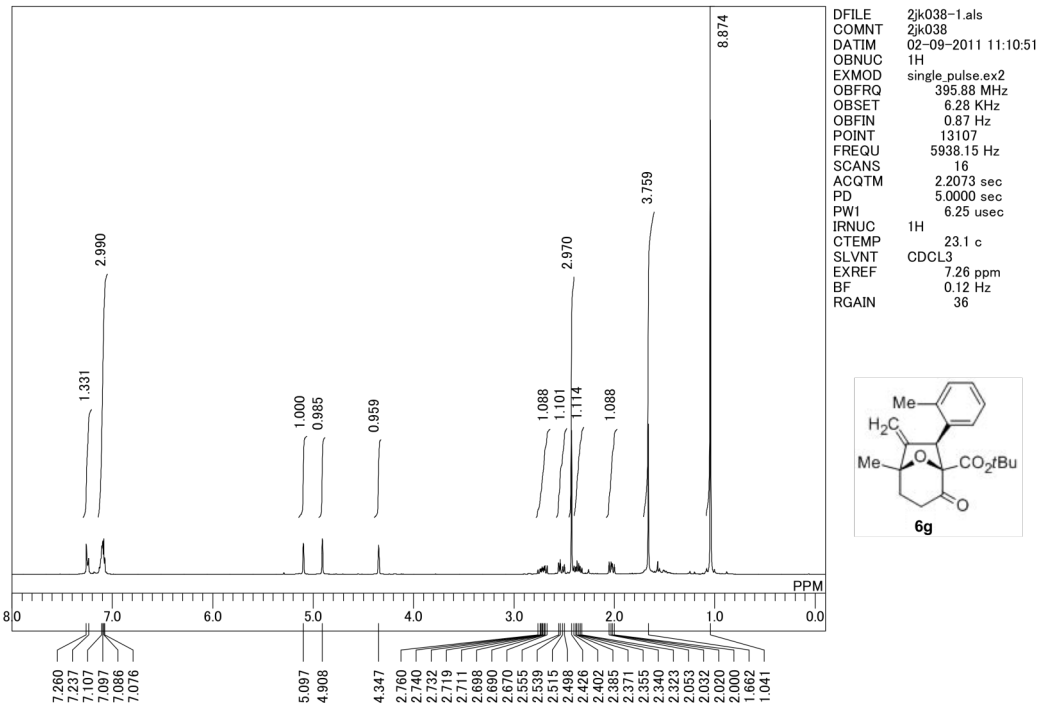

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OBFIN	0.87 Hz
POINT	13107
FREQU	5938.15 Hz
SCANS	16
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	6.25 usec
IRNUC	1H
CTEMP	23.1 c
SLVNT	CDCL3
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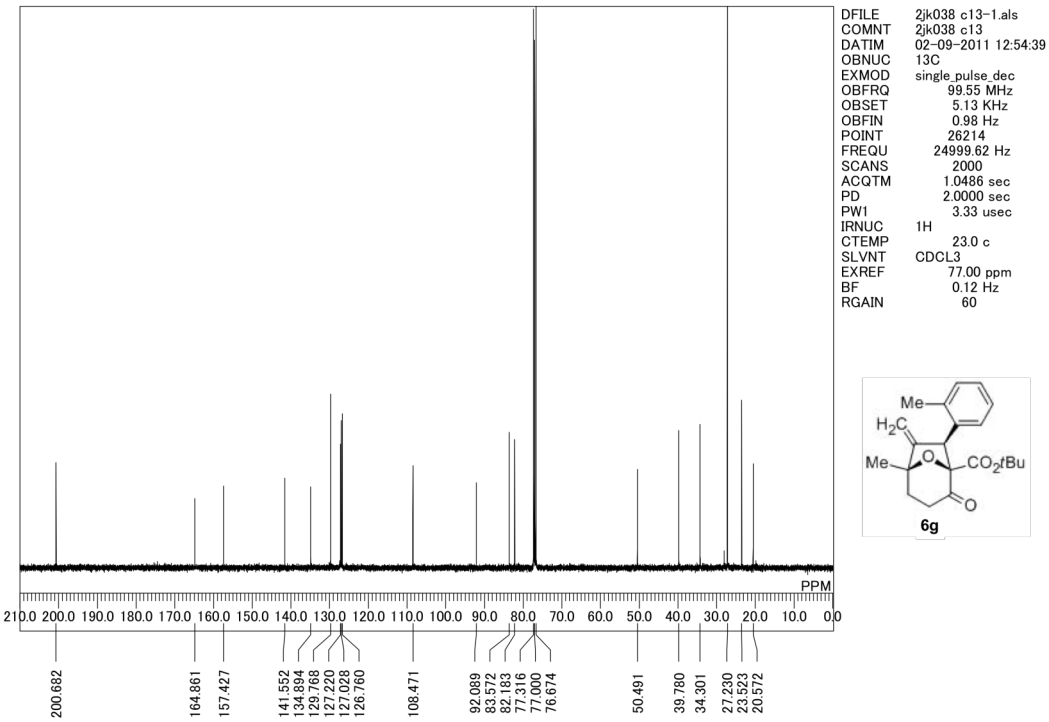
BF	0.12 Hz
RGAIN	36

6g



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PD	2.0000 sec
PW1	3.33 usec
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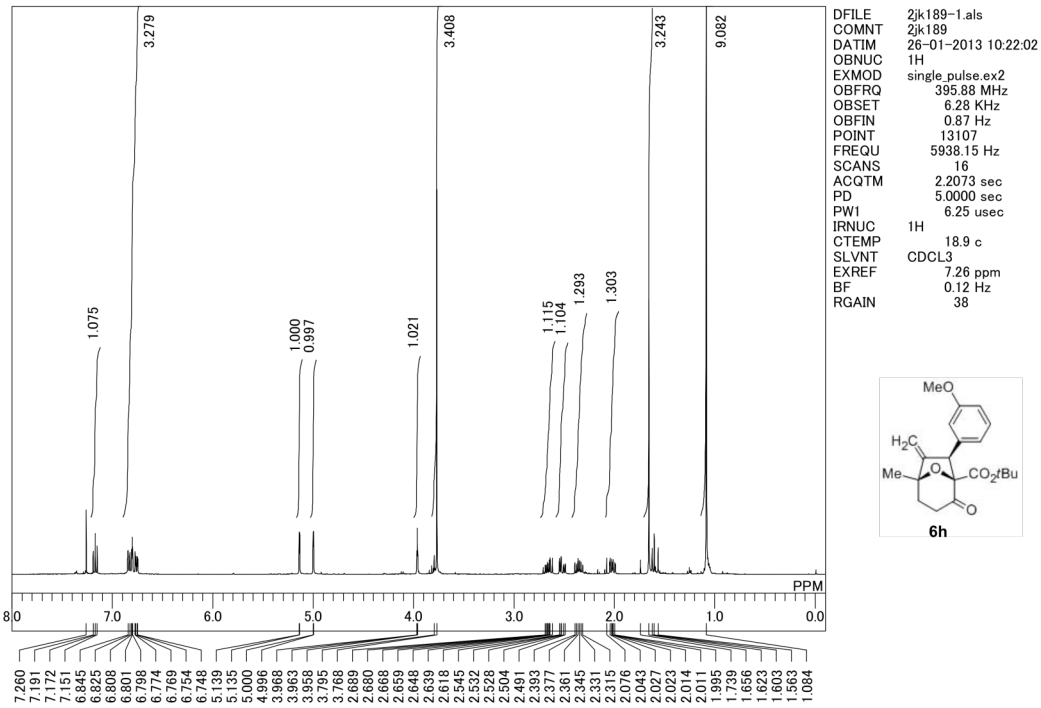
RGAIN	60

6g



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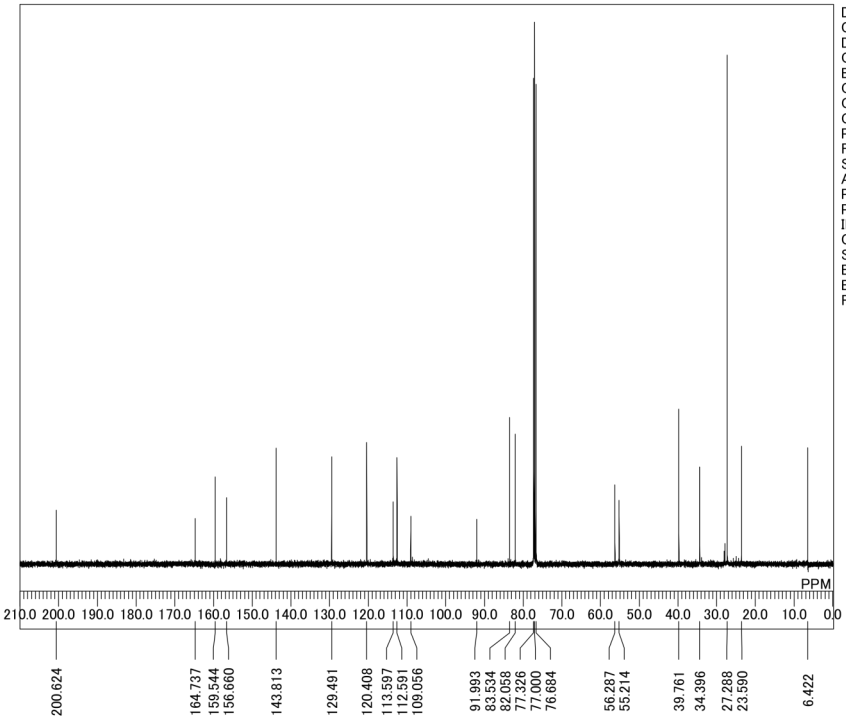
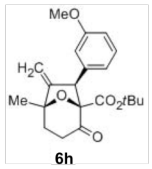
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 16
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.25 usec
IRNUC 1H
CTEMP 18.9 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 38

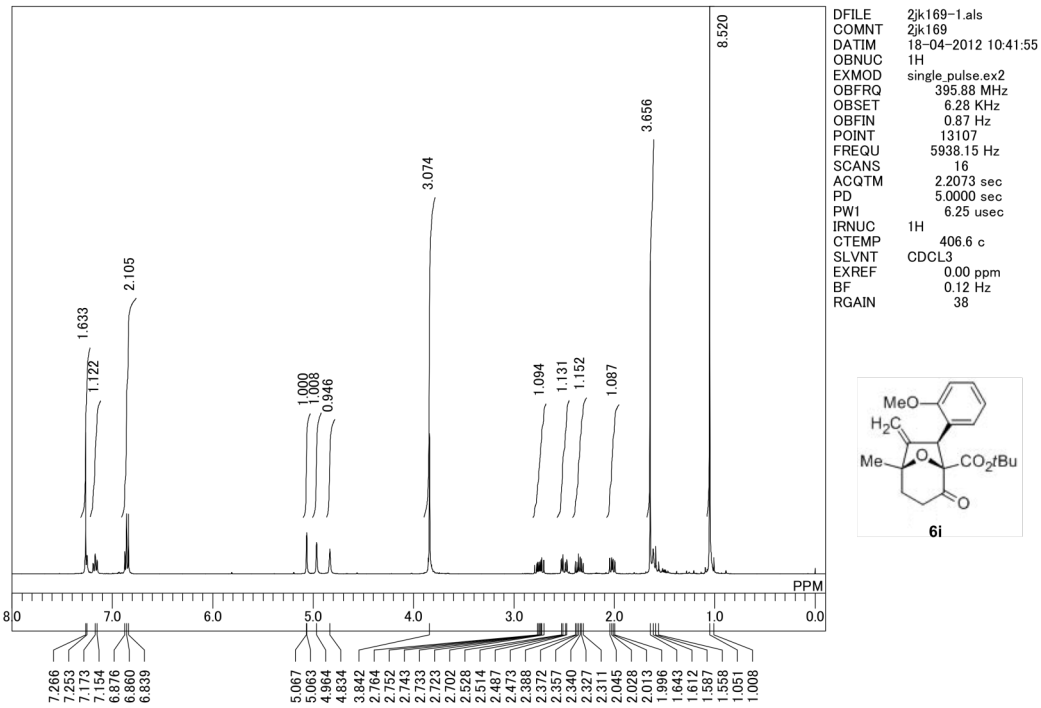
6h

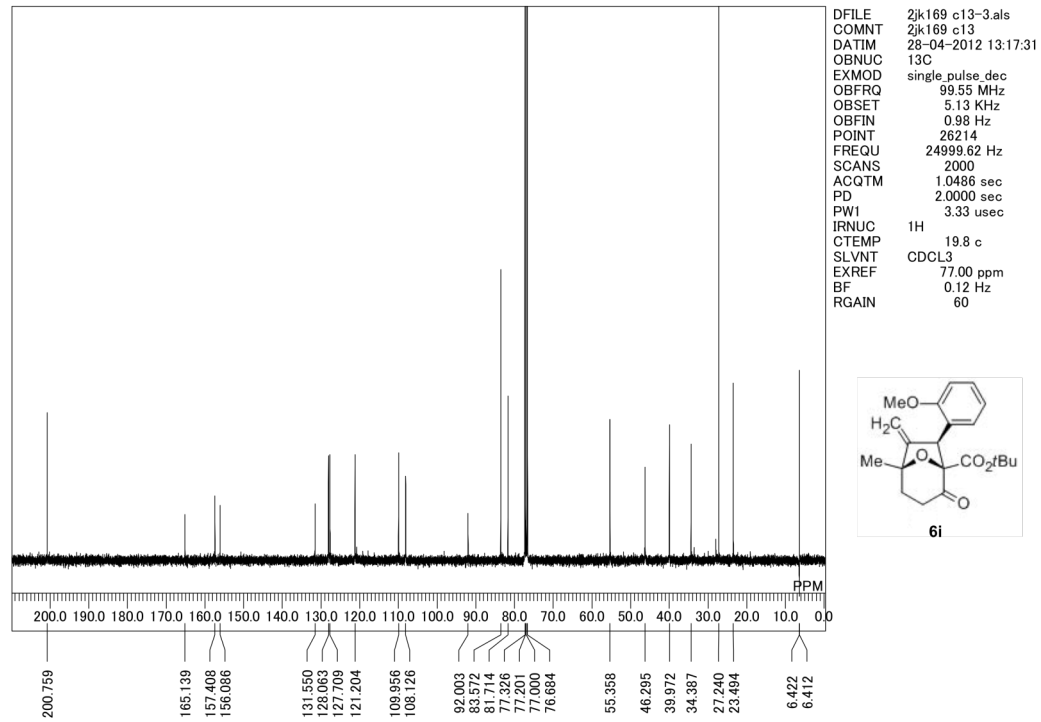


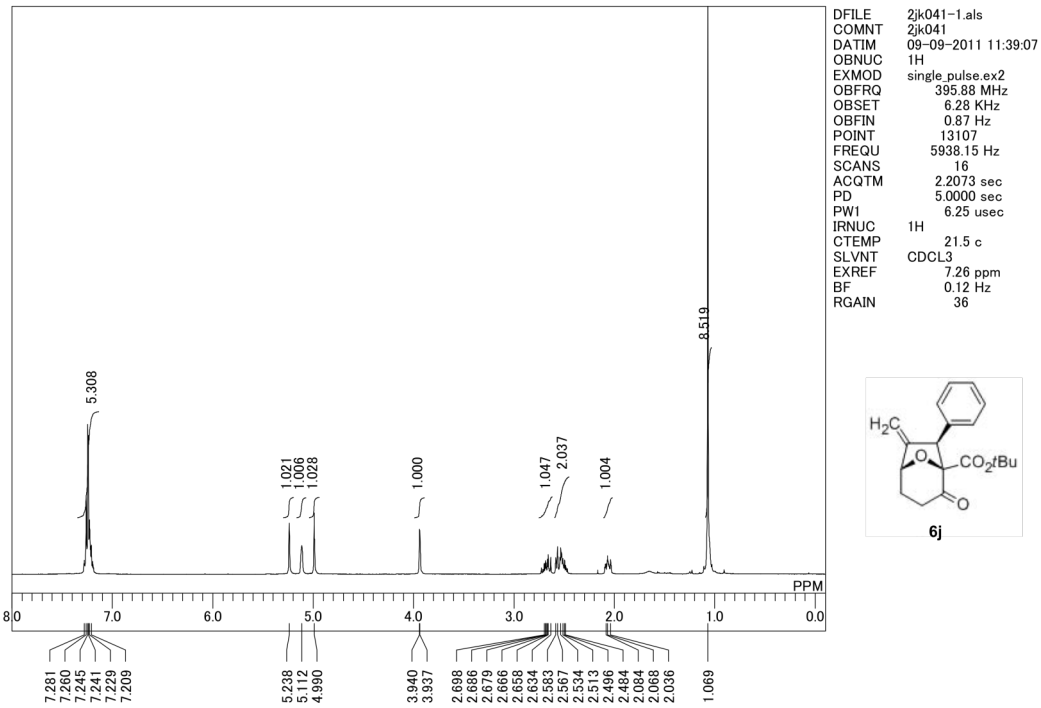
DFILE 2jk189 c13-1.als
COMNT 2jk189 c13
DATIM 26-01-2013 12:13:26
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 2148
ACQTM 1.0486 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

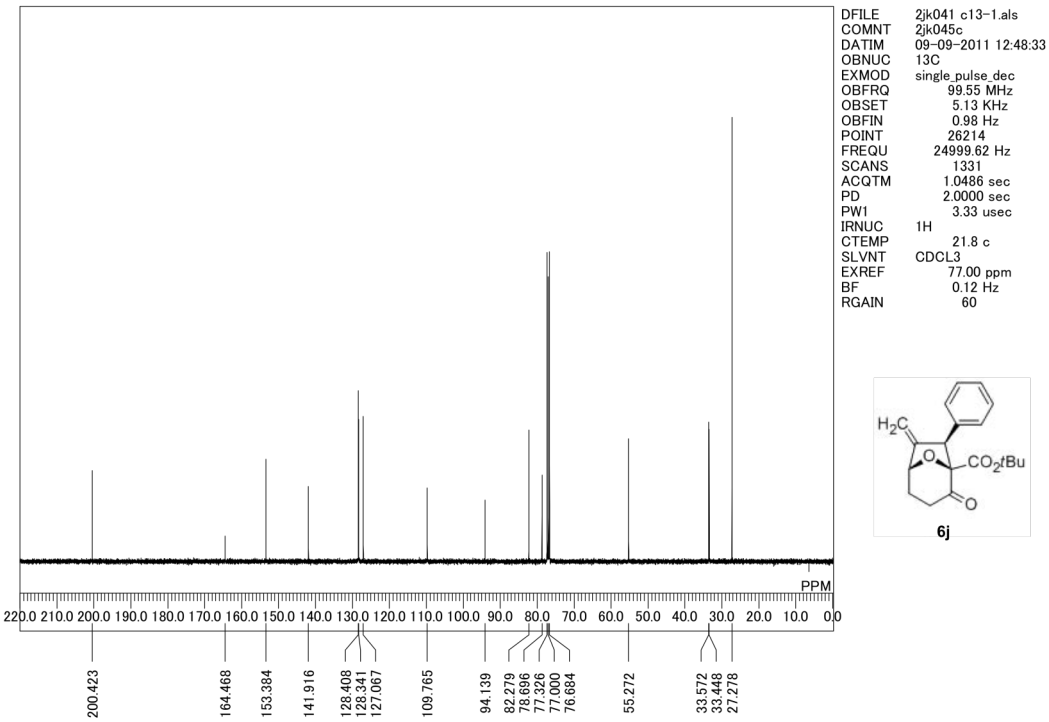


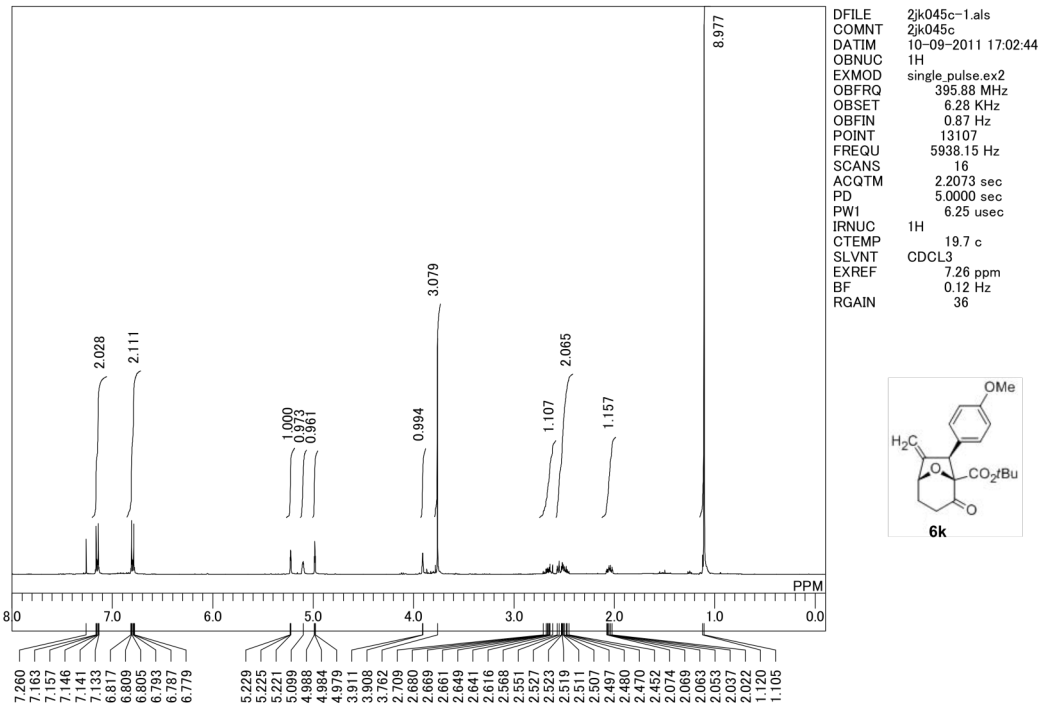
6h


8.520
3.656
3.074
2.105
1.633
1.122
1.000
1.008
0.946
1.094
1.131
1.152
1.087
PPM
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
7.266
7.253
7.173
7.154
6.876
6.860
6.839
5.067
5.063
4.964
4.834
3.842
2.764
2.752
2.743
2.733
2.723
2.702
2.528
2.514
2.487
2.473
2.388
2.372
2.357
2.340
2.327
2.311
2.045
2.028
2.013
1.996
1.643
1.612
1.587
1.558
1.051
1.008
DFILE 2jk169-1.als
COMNT 2jk169
DATIM 18-04-2012 10:41:55
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 16
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.25 usec
IRNUC 1H
CTEMP 406.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 38
MeO
H2C
Me
O
CO2tBu
O
6i


PPM
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0.0
200.759
165.139
157.408
156.086
131.550
128.063
127.709
121.204
109.956
108.126
92.003
83.572
81.714
77.326
77.201
77.000
76.684
55.358
46.295
39.972
34.387
27.240
23.494
6.422
6.412
DFILE 2jk169 c13-3.als
COMNT 2jk169 c13
DATIM 28-04-2012 13:17:31
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 2000
ACQTM 1.0486 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 19.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60
MeO
H2C
Me
O
CO2tBu
O
6i

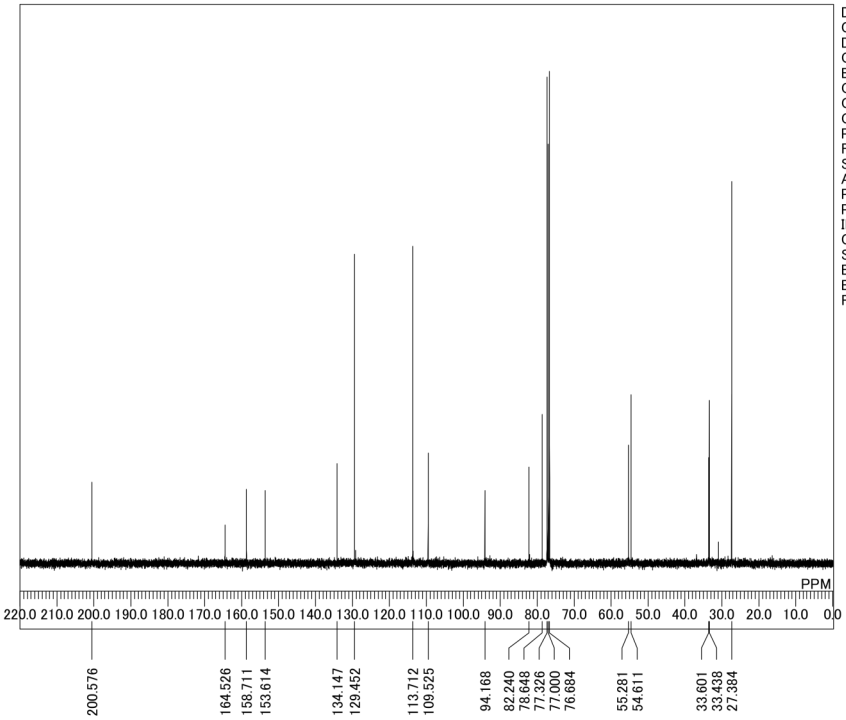

DFILE 2jk041-1.als
COMNT 2jk041
DATIM 09-09-2011 11:39:07
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 16
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.25 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 36
5.308
1.021
1.006
1.028
1.000
1.047
2.037
1.004
8.519
PPM
8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
7.281 7.260 7.245 7.241 7.229 7.209
5.238 5.112 4.990
3.940 3.937
2.698 2.686 2.679 2.666 2.658 2.634 2.583 2.567 2.534 2.513 2.496 2.484 2.084 2.068 2.036
1.069
H2C
O
CO2tBu
O
6j


DFILE 2jk041 c13-1.als
COMNT 2jk045c
DATIM 09-09-2011 12:48:33
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 1331
ACQTM 1.0486 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0.0
200.423
164.468
153.384
141.916
128.408
128.341
127.067
109.765
94.139
82.279
78.696
77.326
77.000
76.684
55.272
33.572
33.448
27.278
H2C
O
CO2tBu
O
6j

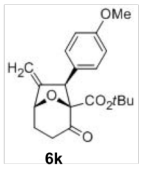


DFILE	2jk045c-1.als
COMNT	2jk045c
DATIM	10-09-2011 17:02:44
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	395.88 MHz
OBSET	6.28 KHz
OBFIN	0.87 Hz
POINT	13107
FREQU	5938.15 Hz
SCANS	16
ACQTM	2.2073 sec
PD	5.0000 sec
PW1	6.25 usec
IRNUC	1H
CTEMP	19.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	36

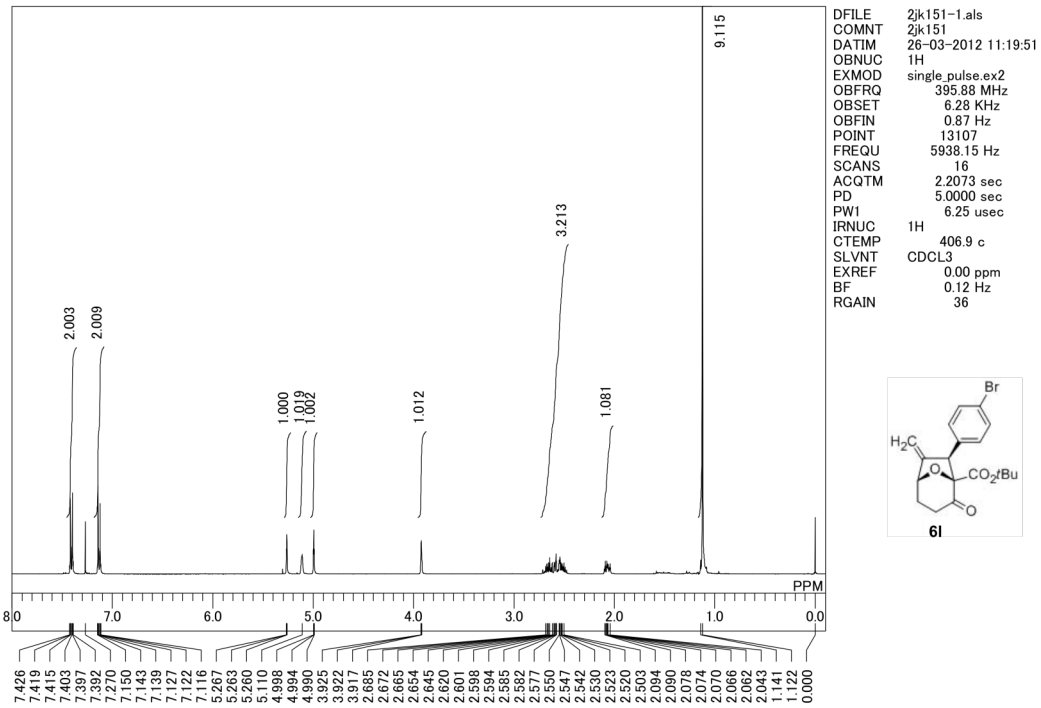
6k



DFILE	2jk045 c13-1.als
COMNT	2jk045 c13
DATIM	13-09-2011 13:27:00
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	99.55 MHz
OBSET	5.13 KHz
OBFIN	0.98 Hz
POINT	26214
FREQU	24999.62 Hz
SCANS	802
ACQTM	1.0486 sec
PD	2.0000 sec
PW1	3.33 usec
IRNUC	1H
CTEMP	21.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

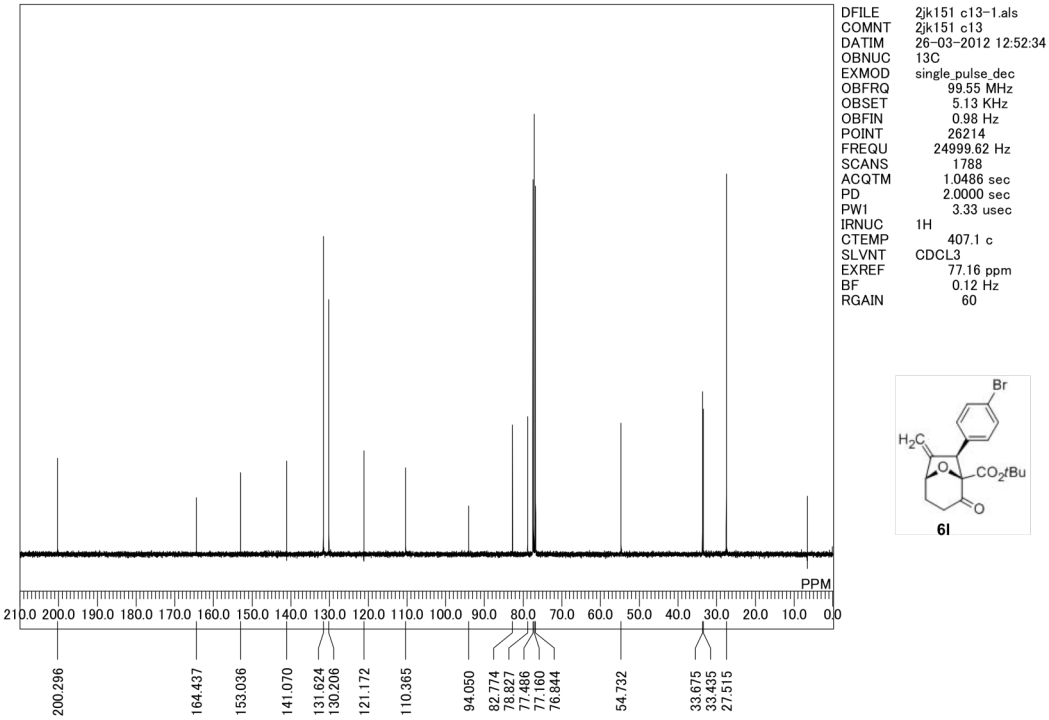


6k



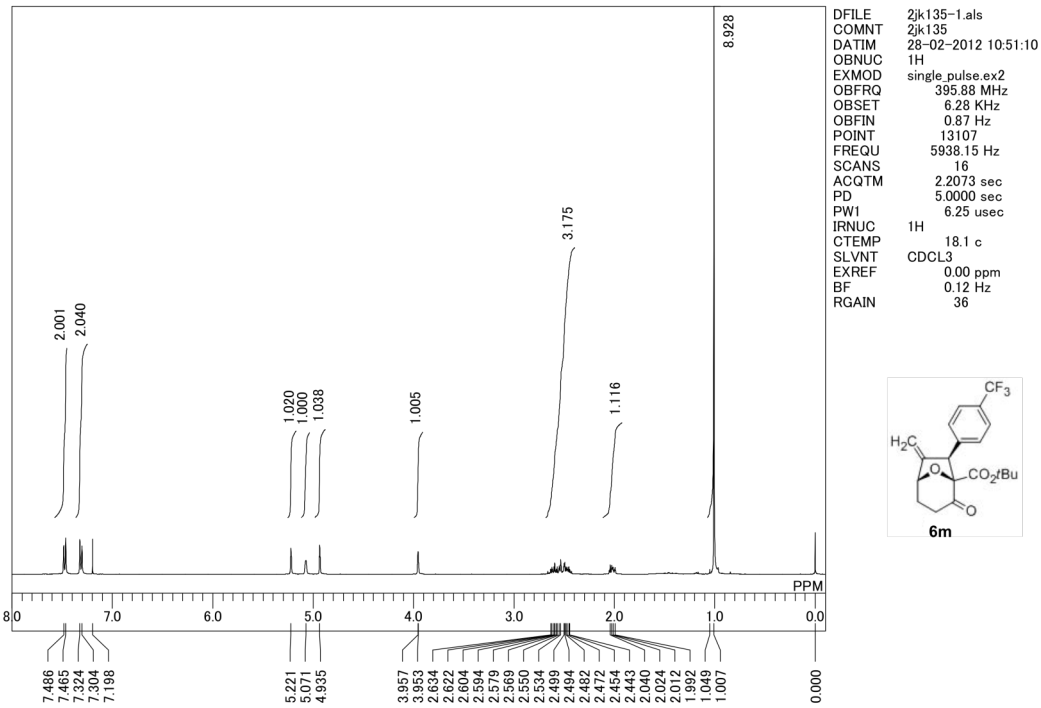
DFILE 2jk151-1.als
COMNT 2jk151
DATIM 26-03-2012 11:19:51
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 16
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.25 usec
IRNUC 1H
CTEMP 406.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 36

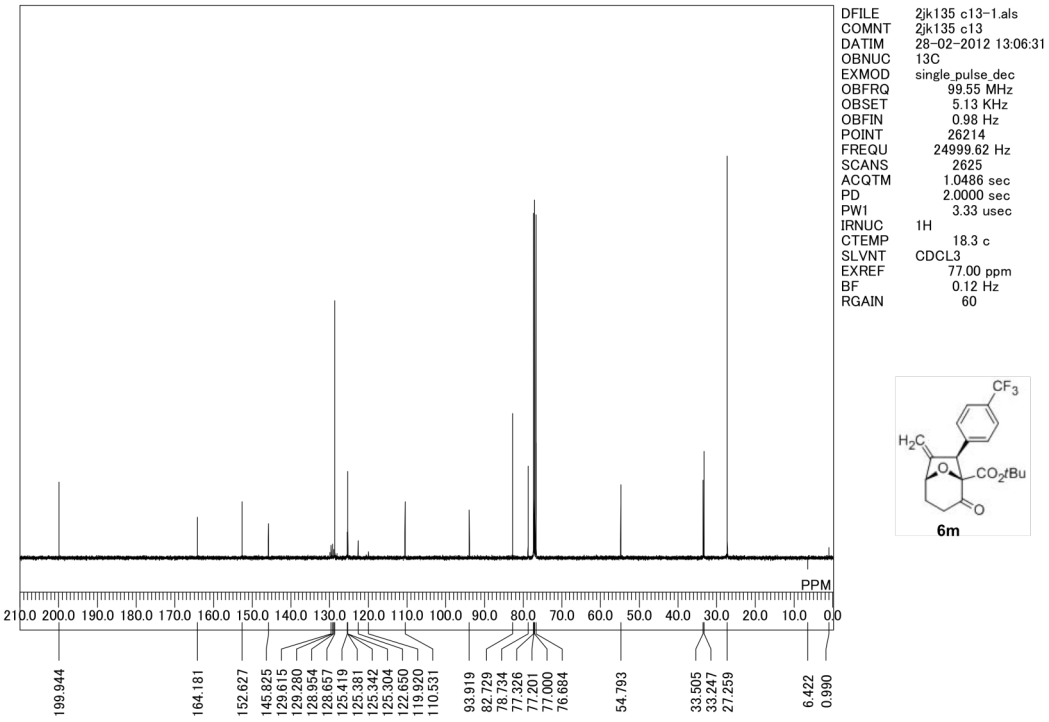
6l

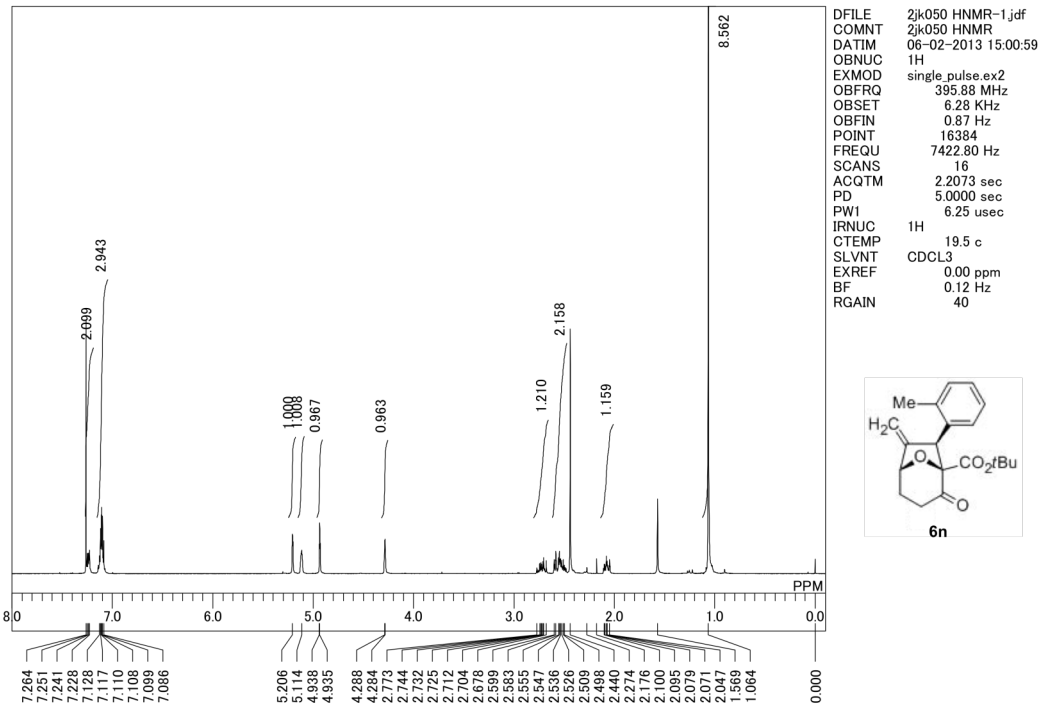


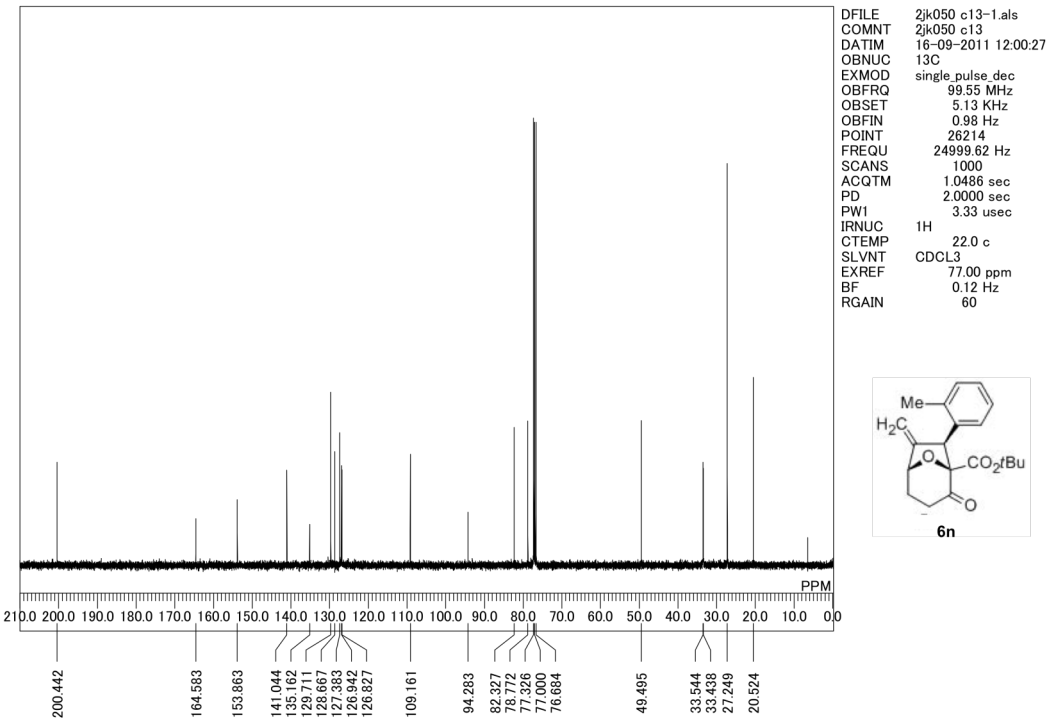
DFILE 2jk151 c13-1.als
COMNT 2jk151 c13
DATIM 26-03-2012 12:52:34
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 1788
ACQTM 1.0486 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 407.1 c
SLVNT CDCL3
EXREF 77.16 ppm
BF 0.12 Hz
RGAIN 60

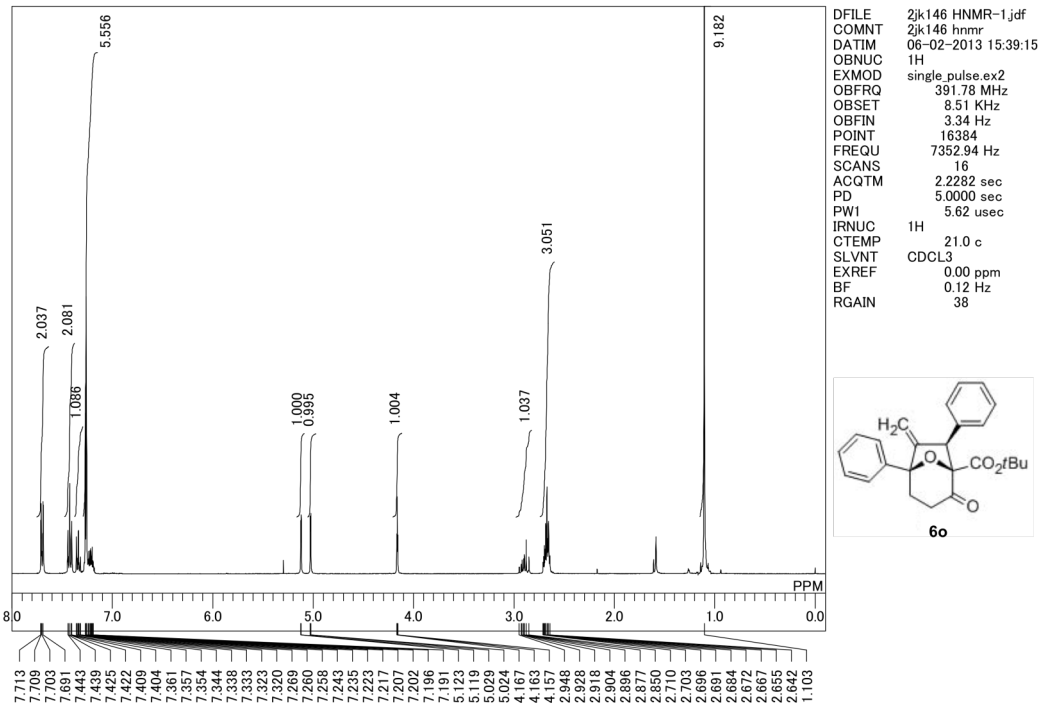
6l

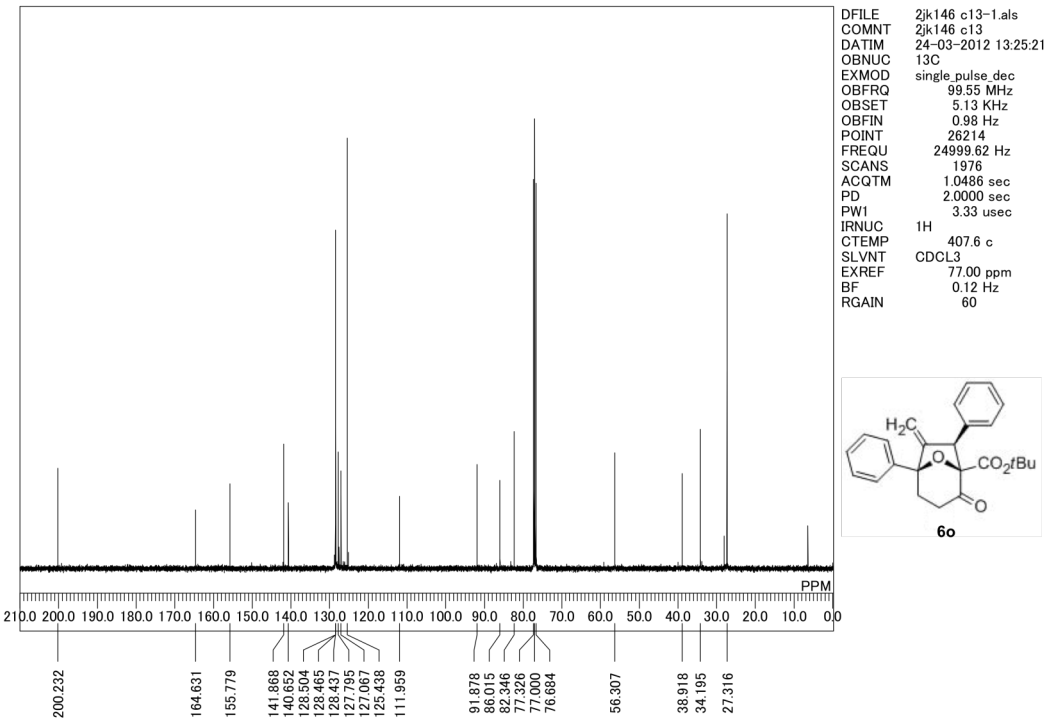

DFILE 2jk135-1.als
COMNT 2jk135
DATIM 28-02-2012 10:51:10
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 16
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.25 usec
IRNUC 1H
CTEMP 18.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 36
8.928
3.175
2.001
2.040
1.020
1.000
1.038
1.005
1.116
PPM
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
7.486
7.465
7.324
7.304
7.198
5.221
5.071
4.935
3.957
3.953
2.634
2.622
2.604
2.594
2.579
2.569
2.550
2.534
2.499
2.494
2.482
2.472
2.454
2.443
2.040
2.024
2.012
1.992
1.049
1.007
0.000
CF3
H2C
O
CO2tBu
O
6m

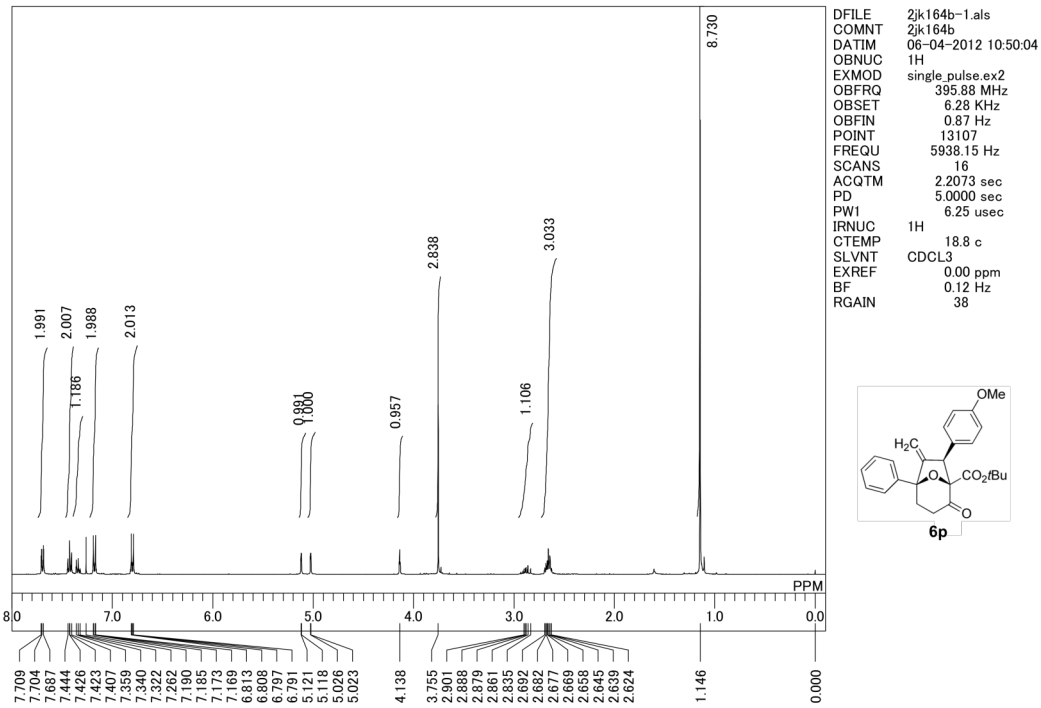

DFILE 2jk135 c13-1.als
COMNT 2jk135 c13
DATIM 28-02-2012 13:06:31
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 2625
ACQTM 1.0486 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 18.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60
PPM
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0.0
199.944
164.181
152.627
145.825
129.615
129.280
128.954
128.657
125.419
125.381
125.342
125.304
122.650
119.920
110.531
93.919
82.729
78.734
77.326
77.201
77.000
76.684
54.793
33.505
33.247
27.259
6.422
0.990
CF3
H2C
O
CO2tBu
O
6m

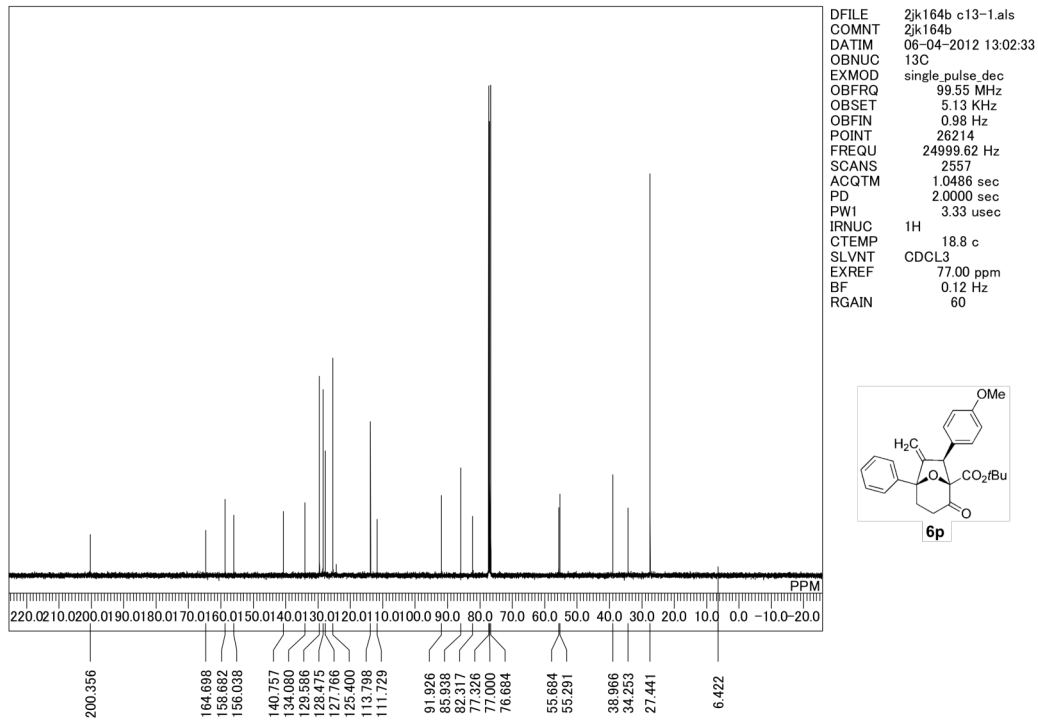

8.562
2.943
2.099
2.158
1.210
1.159
1.000
1.008
0.967
0.963
PPM
8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
7.264 7.251 7.241 7.228 7.128 7.117 7.110 7.108 7.099 7.086
5.206 5.114 4.938 4.935
4.288 4.284 2.773 2.744 2.732 2.725 2.712 2.704 2.678 2.599 2.583 2.555 2.547 2.536 2.526 2.509 2.498 2.440 2.274 2.176 2.100 2.095 2.079 2.071 2.047 1.569 1.064
0.000
DFILE 2jk050 HNMR-1.jdf
COMNT 2jk050 HNMR
DATIM 06-02-2013 15:00:59
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 16384
FREQU 7422.80 Hz
SCANS 16
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.25 usec
IRNUC 1H
CTEMP 19.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40
Me
H2C
O
CO2tBu
O
6n

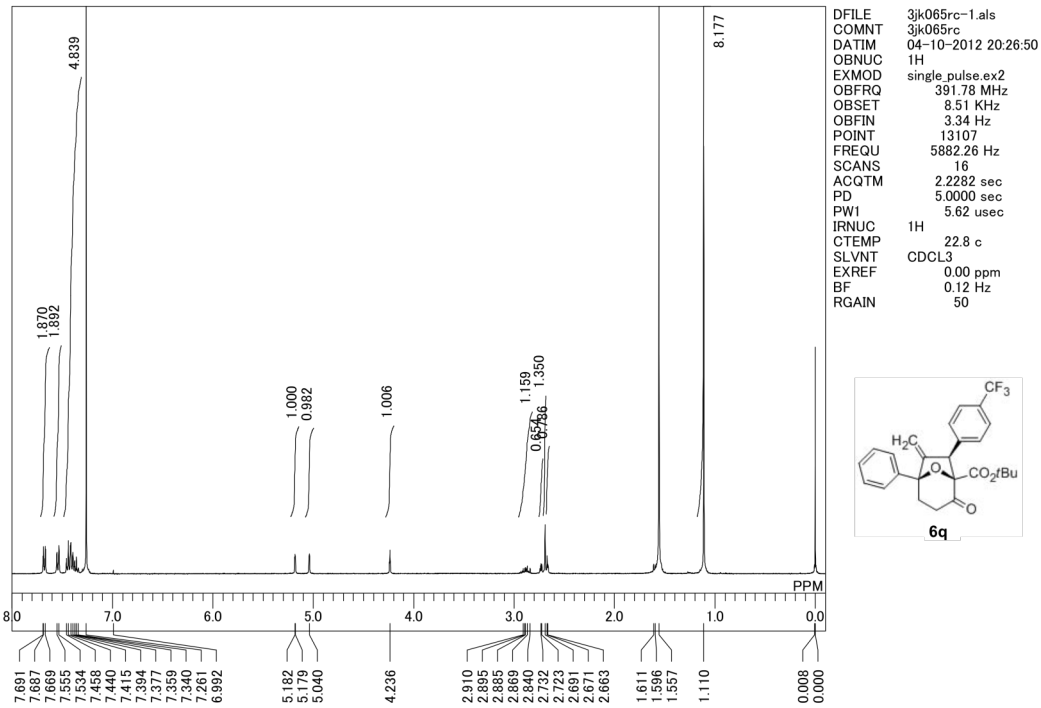

PPM
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0.0
200.442
164.583
153.863
141.044
135.162
129.711
128.667
127.383
126.942
126.827
109.161
94.283
82.327
78.772
77.326
77.000
76.684
49.495
33.544
33.438
27.249
20.524
DFILE 2jk050 c13-1.als
COMNT 2jk050 c13
DATIM 16-09-2011 12:00:27
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 1000
ACQTM 1.0486 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60
Me
H2C
O
CO2tBu
O
6n

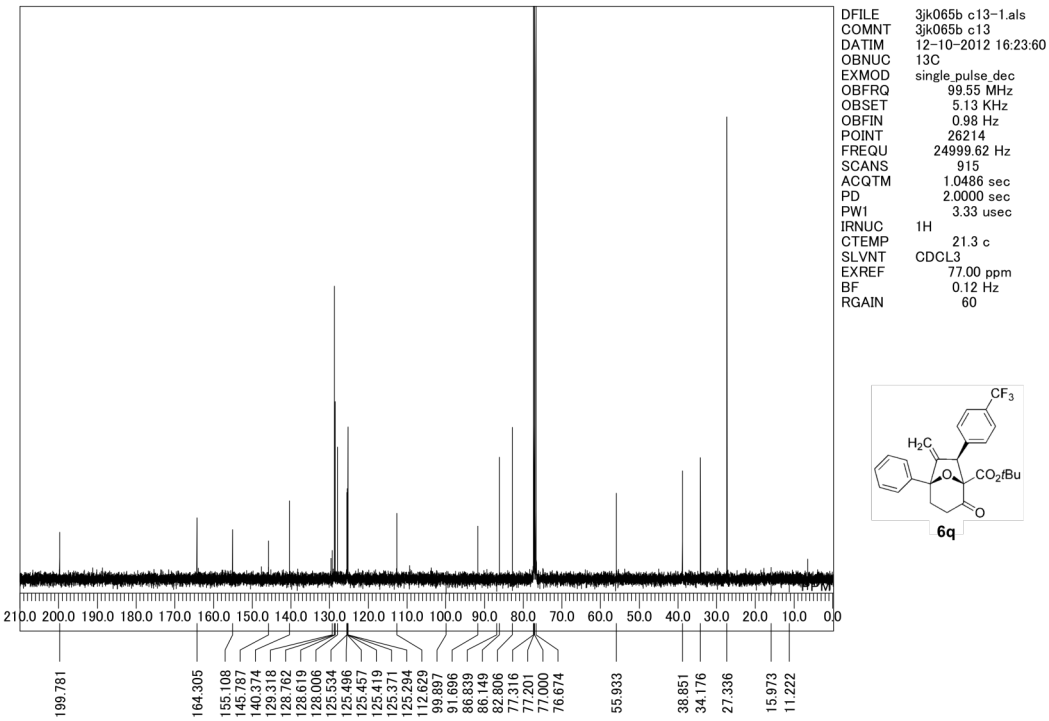

2.037
2.081
1.086
5.556
1.000
0.995
1.004
1.037
3.051
9.182
PPM
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
7.713
7.709
7.703
7.691
7.443
7.439
7.425
7.422
7.409
7.404
7.361
7.357
7.354
7.344
7.338
7.333
7.323
7.320
7.269
7.260
7.258
7.243
7.235
7.223
7.217
7.207
7.202
7.196
7.191
5.123
5.119
5.029
5.024
4.167
4.163
4.157
2.948
2.928
2.918
2.904
2.896
2.877
2.850
2.710
2.703
2.696
2.691
2.684
2.672
2.667
2.655
2.642
1.103
DFILE 2jk146 HNMR-1.jdf
COMNT 2jk146 hnmr
DATIM 06-02-2013 15:39:15
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 16384
FREQU 7352.94 Hz
SCANS 16
ACQTM 2.2282 sec
PD 5.0000 sec
PW1 5.62 usec
IRNUC 1H
CTEMP 21.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 38
H2C
O
CO2tBu
O
6o

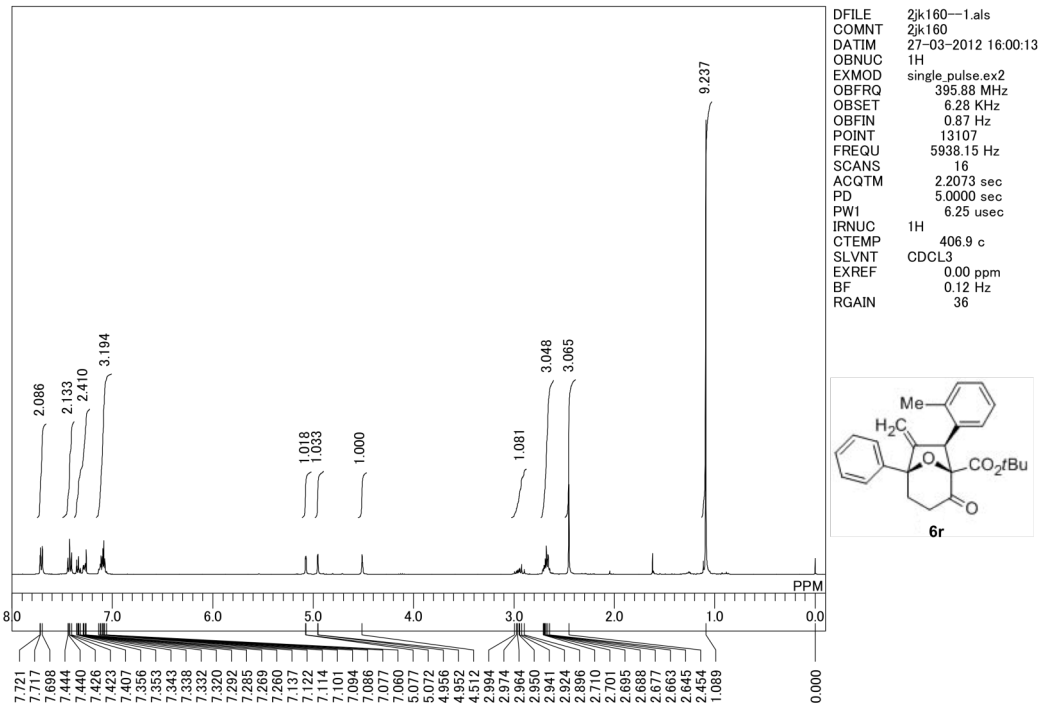

PPM
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0.0
200.232
164.631
155.779
141.868
140.652
128.504
128.465
128.437
127.795
127.067
125.438
111.959
91.878
86.015
82.346
77.326
77.000
76.684
56.307
38.918
34.195
27.316
DFILE 2jk146 c13-1.als
COMNT 2jk146 c13
DATIM 24-03-2012 13:25:21
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 1976
ACQTM 1.0486 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 407.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60
H2C
O
CO2tBu
O
6o


DFILE 2jk164b-1.als
COMNT 2jk164b
DATIM 06-04-2012 10:50:04
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 16
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.25 usec
IRNUC 1H
CTEMP 18.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 38
8.730
3.033
2.838
2.013
1.991
2.007
1.988
1.186
1.106
0.957
0.881
1.000
PPM
8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0
7.709 7.704 7.687 7.444 7.426 7.423 7.407 7.359 7.340 7.322 7.262 7.190 7.185 7.173 7.169 6.813 6.808 6.797 6.791 5.121 5.118 5.026 5.023 4.138 3.755 2.901 2.888 2.879 2.861 2.835 2.692 2.682 2.677 2.669 2.658 2.645 2.639 2.624 1.146 0.000
OMe
H2C
O
CO2tBu
O
6p


DFILE 2jk164b c13-1.als
COMNT 2jk164b
DATIM 06-04-2012 13:02:33
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 2557
ACQTM 1.0486 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 18.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60
PPM
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0.0 -10.0 -20.0
200.356
164.698 158.882 156.038
140.757 134.080 129.586 128.475 127.766 125.400 113.798 111.729
91.926 85.938 82.317 77.326 77.000 76.684
55.684 55.291
38.966 34.253
27.441
6.422
OMe
H2C
O
CO2tBu
O
6p


DFILE 3jk065rc-1.als
COMNT 3jk065rc
DATIM 04-10-2012 20:26:50
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 391.78 MHz
OBSET 8.51 KHz
OBFIN 3.34 Hz
POINT 13107
FREQU 5882.26 Hz
SCANS 16
ACQTM 2.2282 sec
PD 5.0000 sec
PW1 5.62 usec
IRNUC 1H
CTEMP 22.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 50
4.839
8.177
1.870
1.892
1.000
0.982
1.006
1.159
1.350
PPM
7.691 7.687 7.669 7.555 7.534 7.458 7.440 7.415 7.394 7.377 7.359 7.340 7.261 6.992
5.182 5.179 5.040
4.236
2.910 2.895 2.885 2.869 2.840 2.732 2.723 2.691 2.671 2.663
1.611 1.596 1.557
1.110
0.008 0.000
CF3
H2C
O
CO2tBu
O
6q


DFILE 3jk065b c13-1.als
COMNT 3jk065b c13
DATIM 12-10-2012 16:23:60
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 915
ACQTM 1.0486 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60
199.781
164.305
155.108 145.787 140.374 129.318 128.762 128.619 128.006 125.534 125.496 125.457 125.419 125.371 125.294 112.629 99.897 91.696 86.839 86.149 82.806 77.316 77.201 77.000 76.674
55.933
38.851 34.176
27.336
15.973 11.222
CF3
H2C
O
CO2tBu
O
6q


DFILE 2jk160—1.als
COMNT 2jk160
DATIM 27-03-2012 16:00:13
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 16
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.25 usec
IRNUC 1H
CTEMP 406.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 36
9.237
3.194
2.086
2.133
2.410
1.018
1.033
1.000
1.081
3.048
3.065
PPM
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
7.721
7.717
7.698
7.444
7.440
7.426
7.423
7.407
7.356
7.353
7.343
7.338
7.332
7.320
7.292
7.285
7.269
7.260
7.137
7.122
7.114
7.101
7.094
7.086
7.077
7.060
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4.512
2.994
2.974
2.964
2.950
2.941
2.924
2.896
2.710
2.701
2.695
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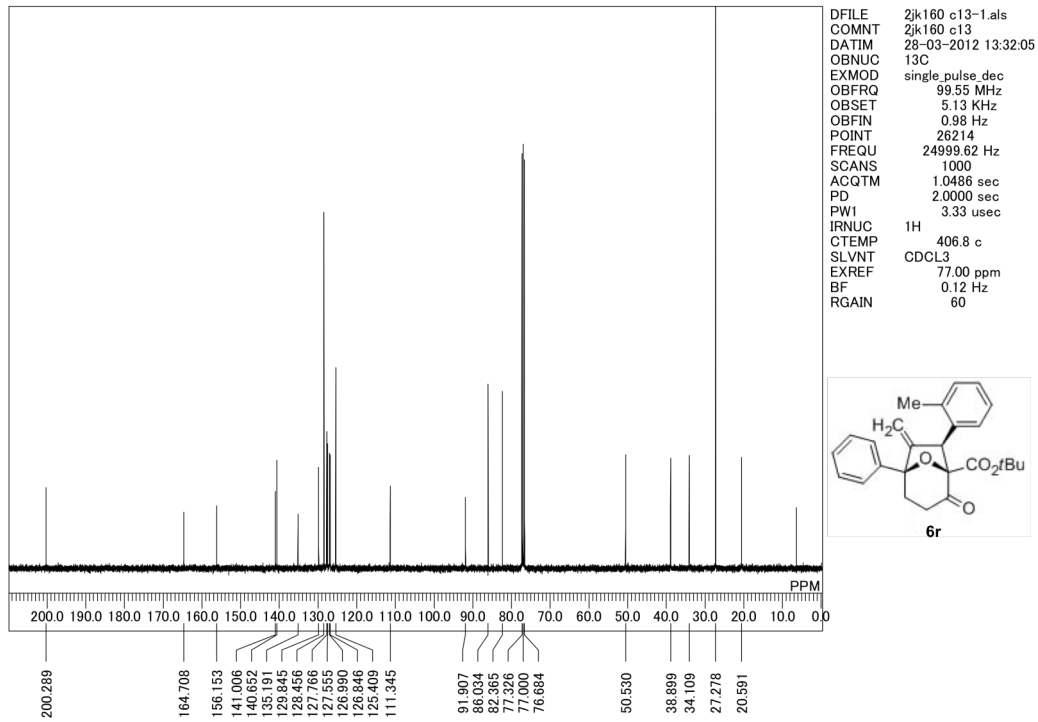
H2C
O
CO2tBu
O
6r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COMNT 2jk160 c13
DATIM 28-03-2012 13:32:05
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 1000
ACQTM 1.0486 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 406.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60
PPM
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0.0
200.289
164.708
156.153
141.006
140.652
135.191
129.845
128.456
127.766
127.555
126.990
126.846
125.409
111.345
91.907
86.034
82.365
77.326
77.000
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38.899
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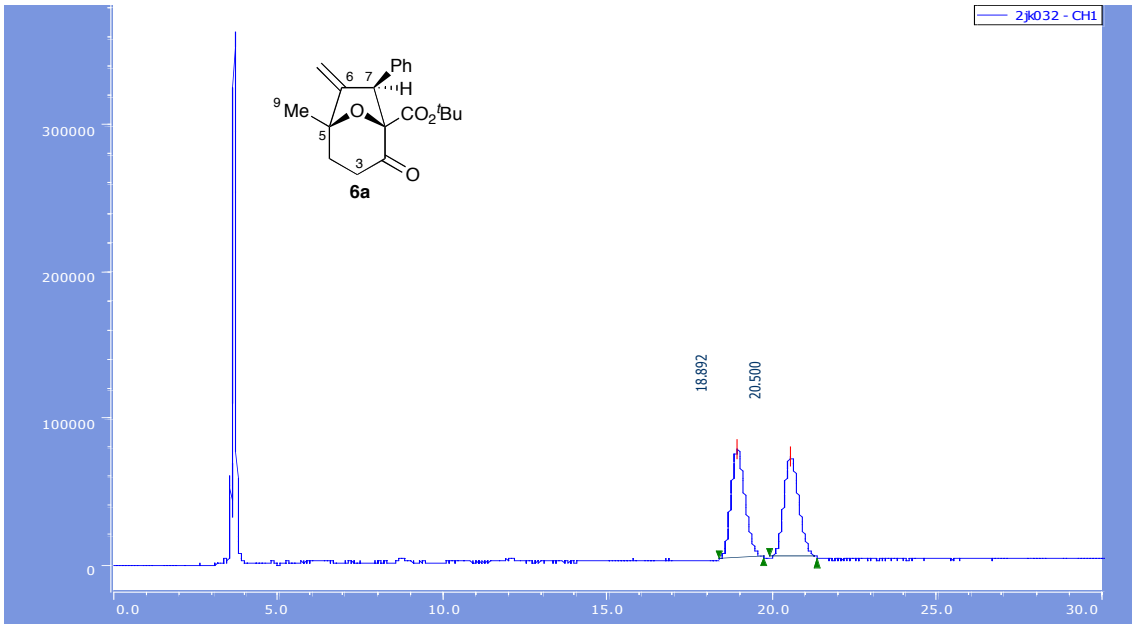
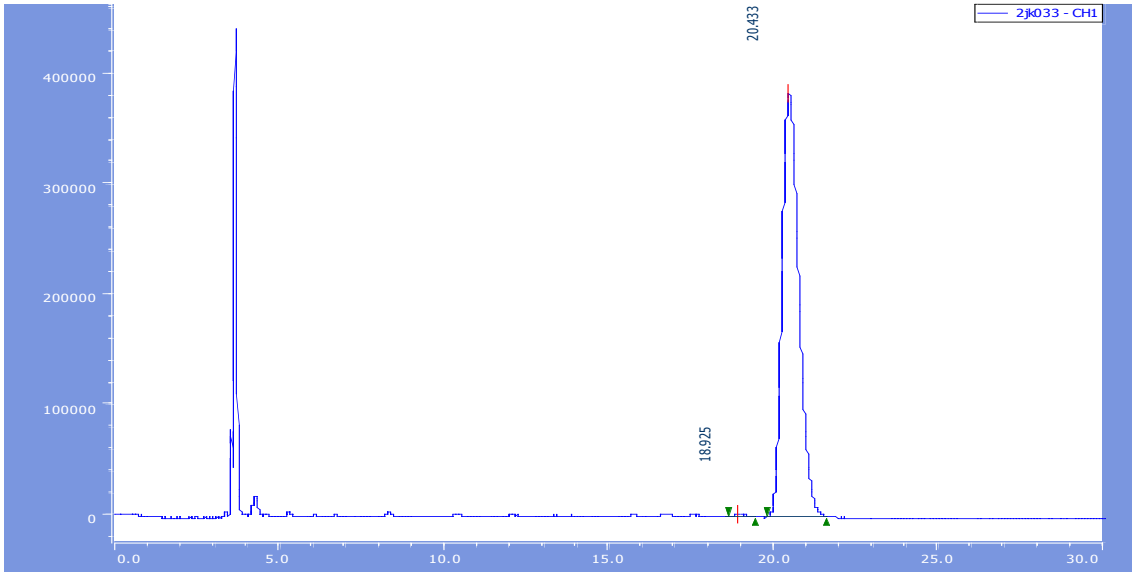
27.278
20.591
Me
H2C
O
CO2tBu
O
6r



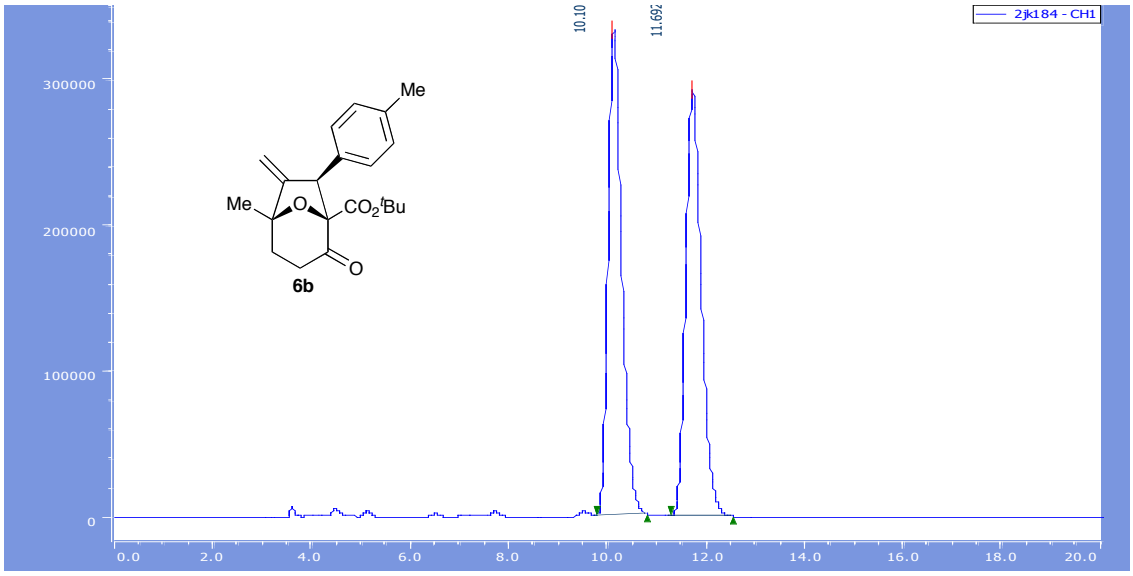


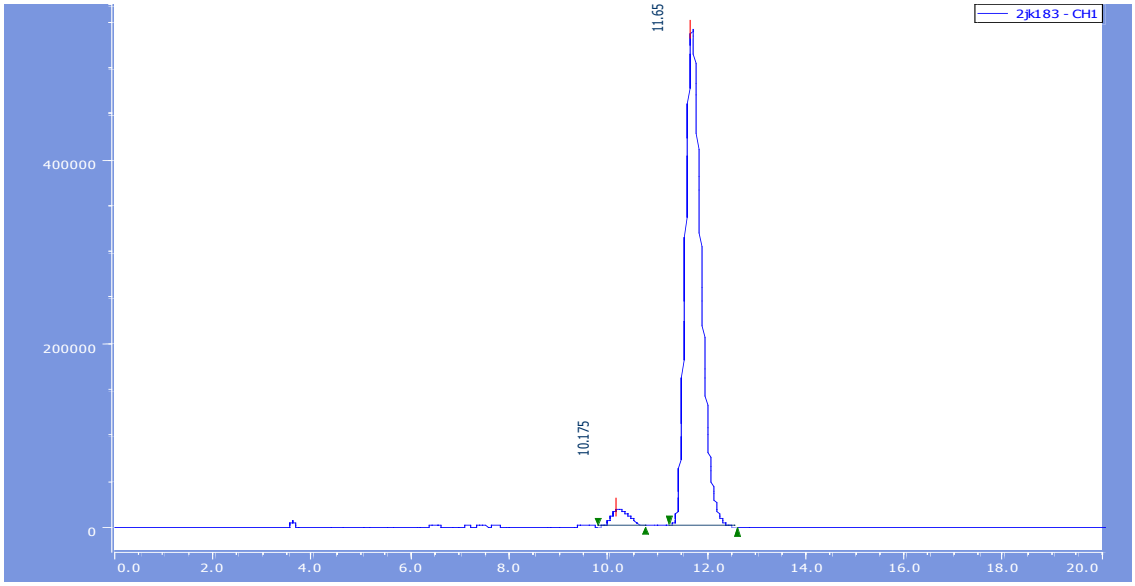
6a (racemate)

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	18.89	2232515	50.02
2	20.50	2231163	49.98

6a

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	18.92	62494	0.46
2	20.43	13630440	99.54



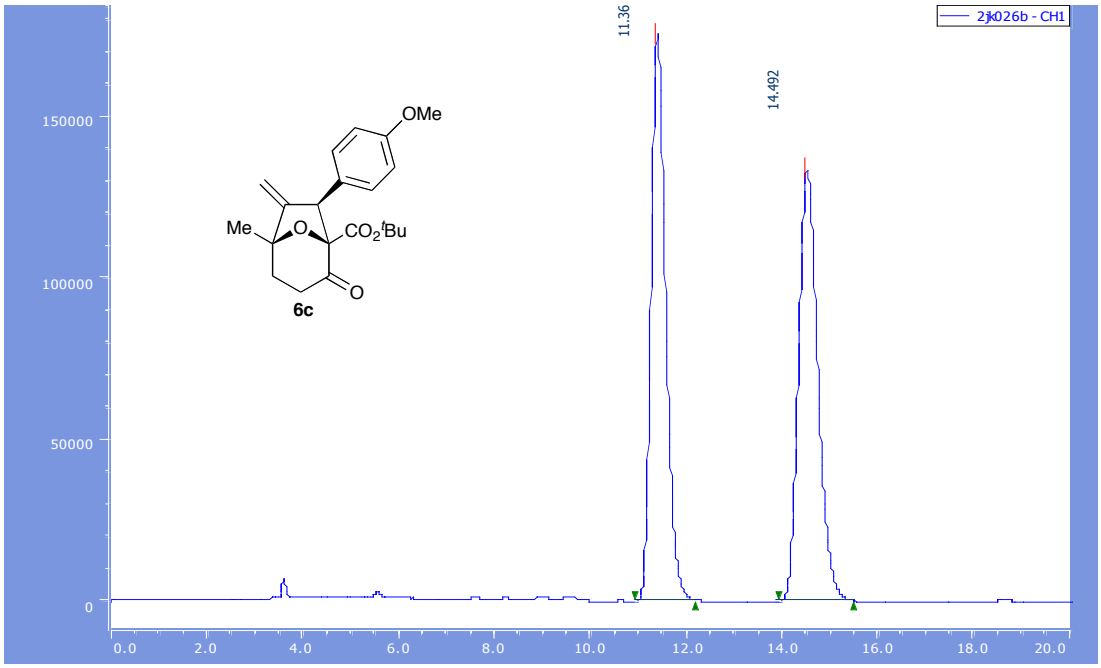


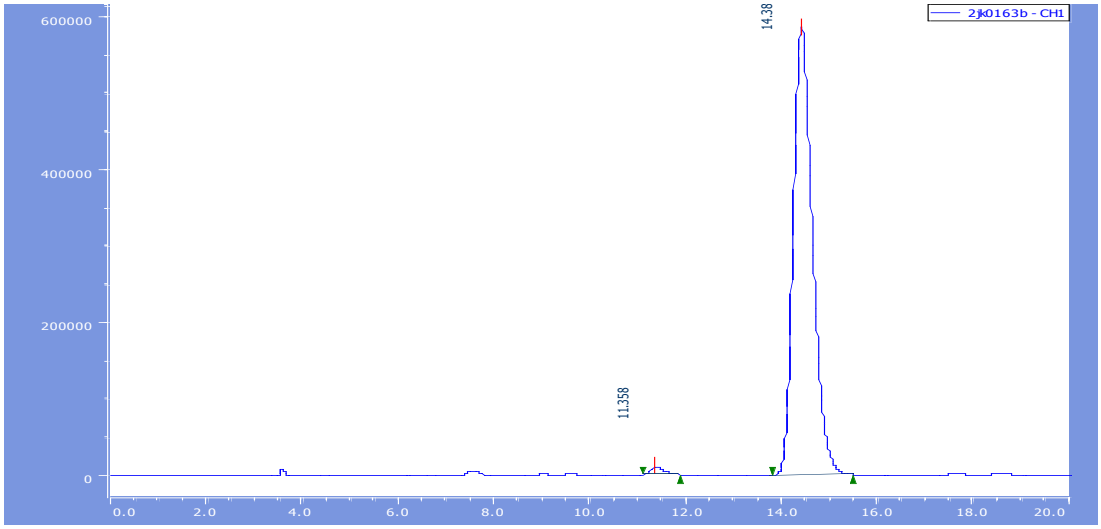
6b (racemate)

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	10.10	6397673	49.78
2	11.69	6455430	50.22

6b

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	10.17	468262	3.73
2	11.65	12070046	96.27



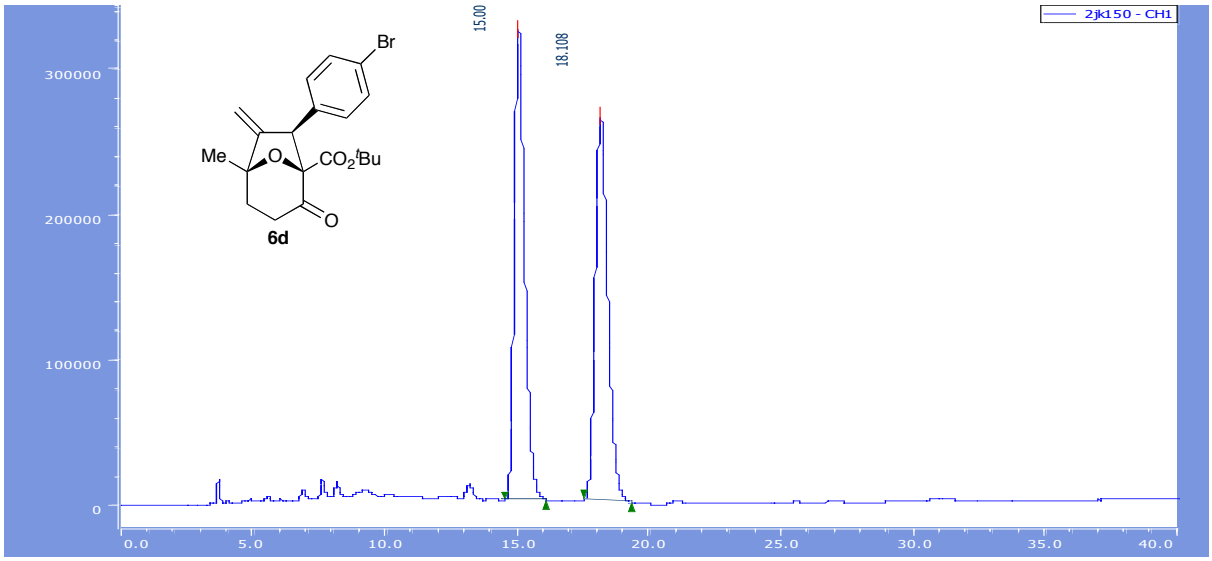


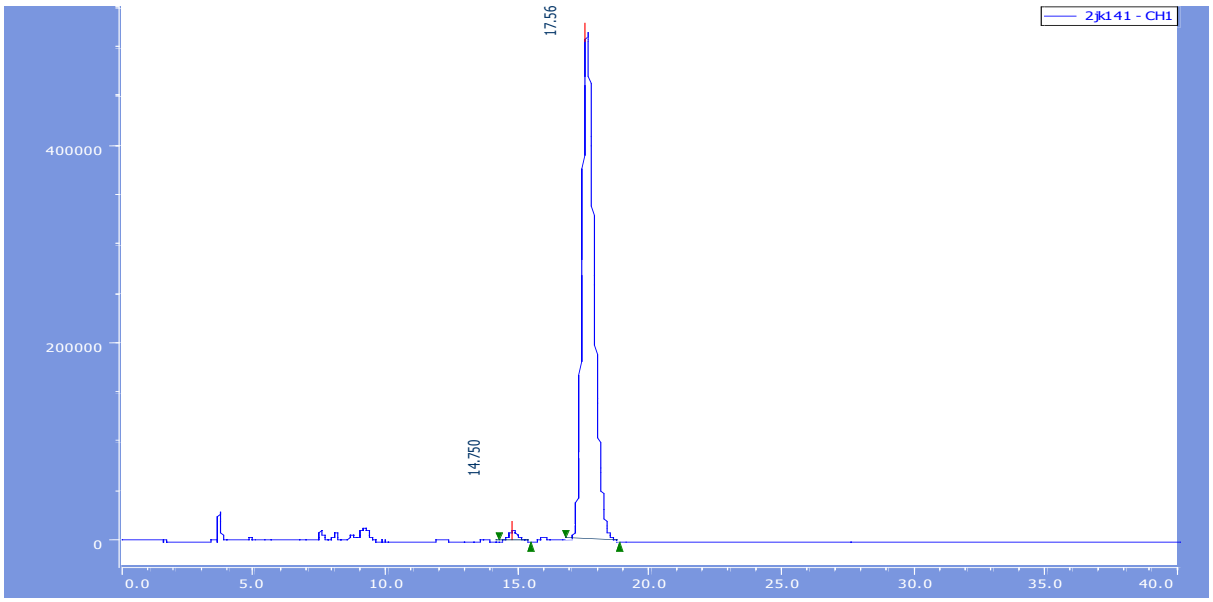
6c (racemate)

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	11.36	3722059	49.82
2	14.49	3748501	50.18

6c

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	11.35	201688	1.21
2	14.38	16437045	98.79



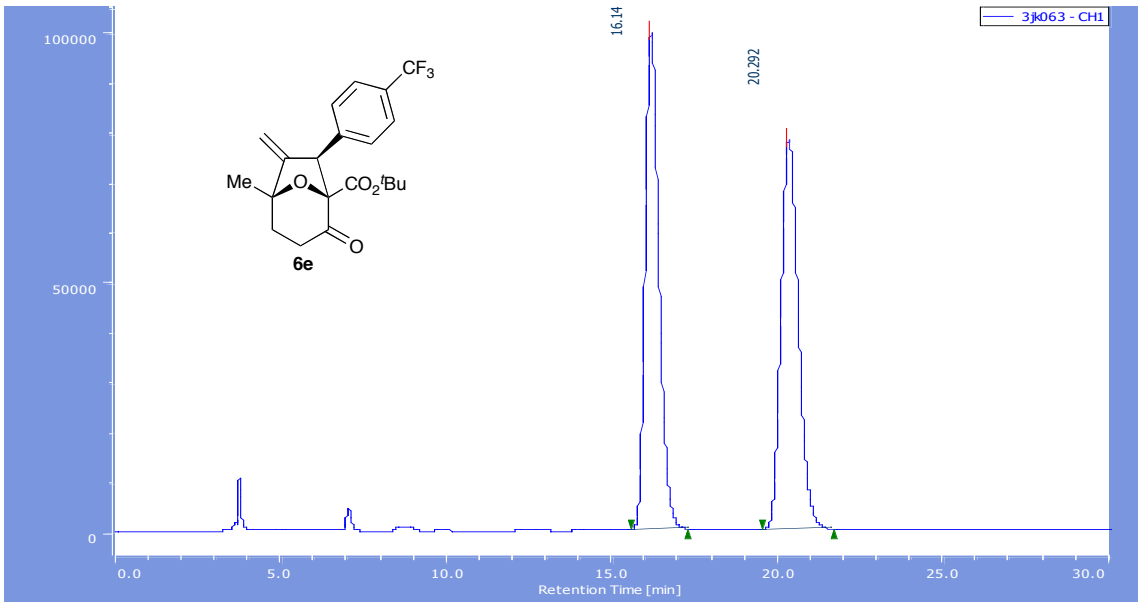


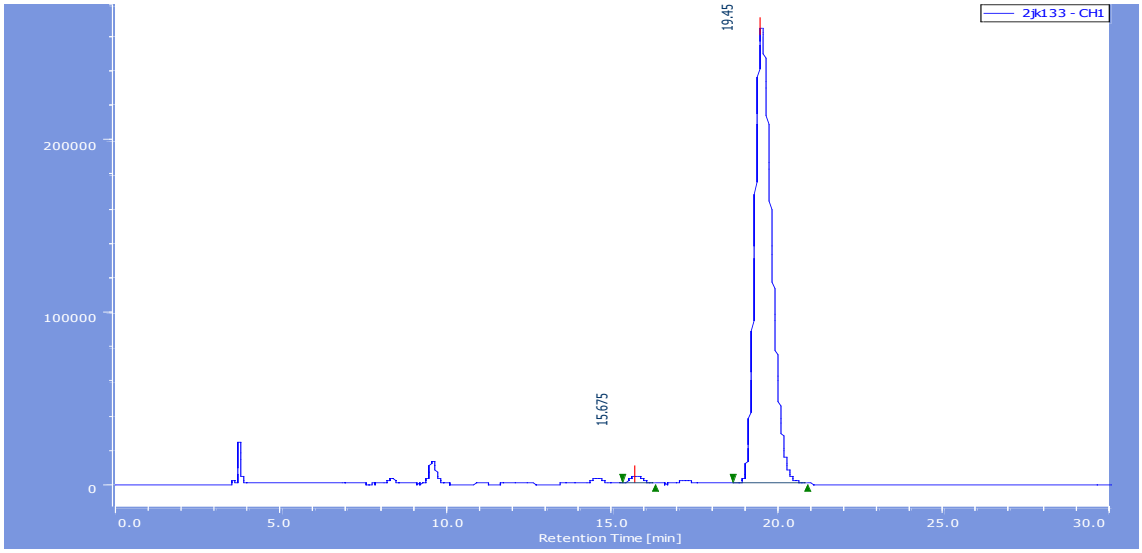
6d (racemate)

PeakNo.	Time (min)	Area [m V. Sec]	Area %
1	15.00	8998222	50.45
2	18.10	8836777	49.55

6d

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	14.75	303835	1.82
2	17.56	16421881	98.18



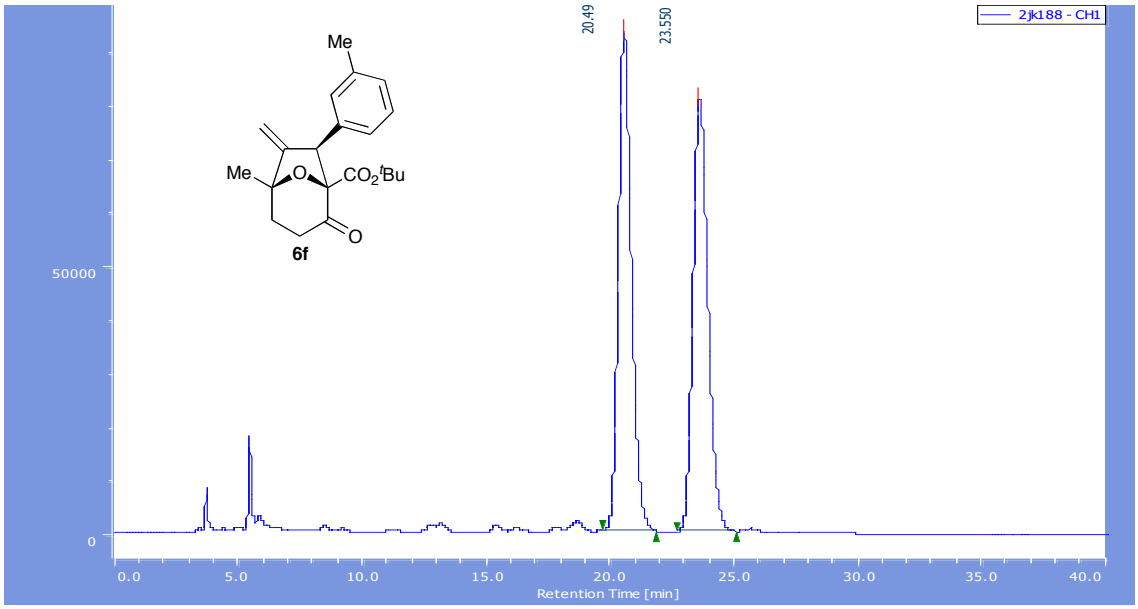


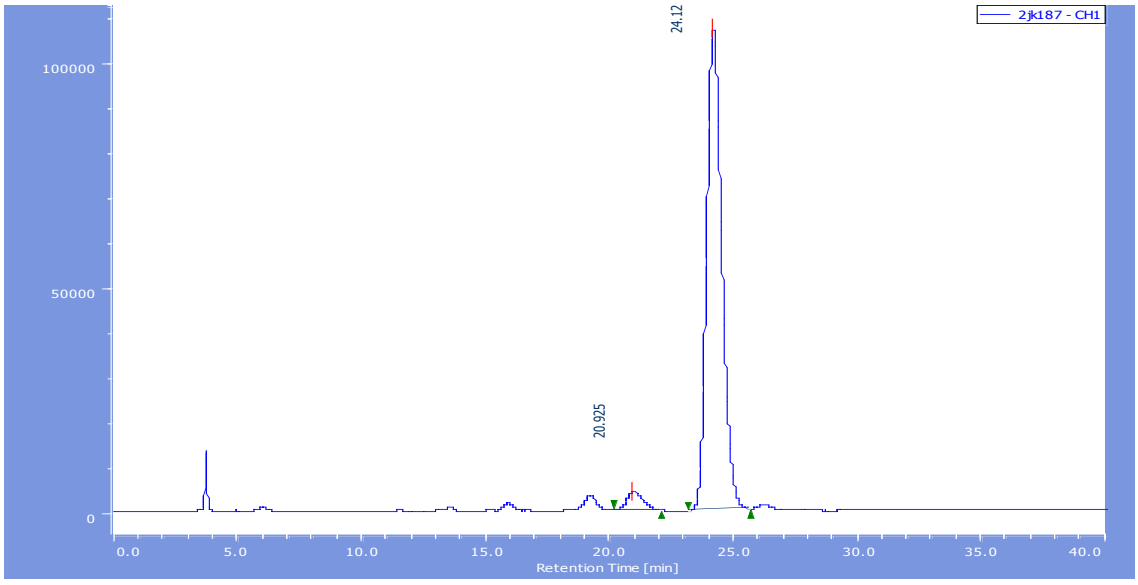
6e (racemate)

PeakNo.	Time (min)	Area [m V. Sec]	Area %
1	16.14	2868587	49.87
2	20.29	2882971	50.13

6e

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	15.67	121816	1.29
2	19.45	9351901	98.71



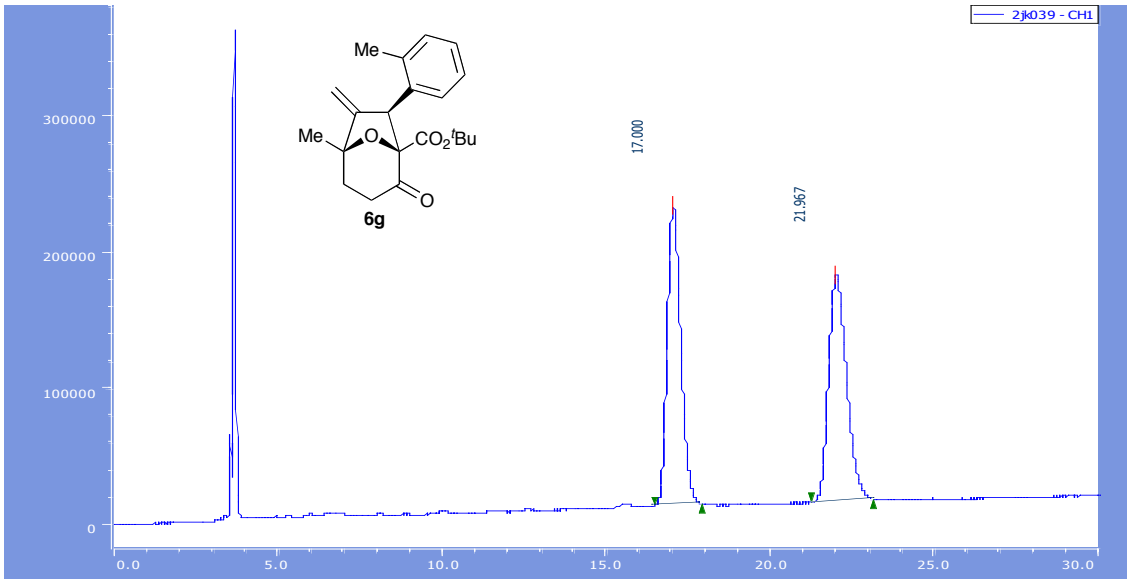


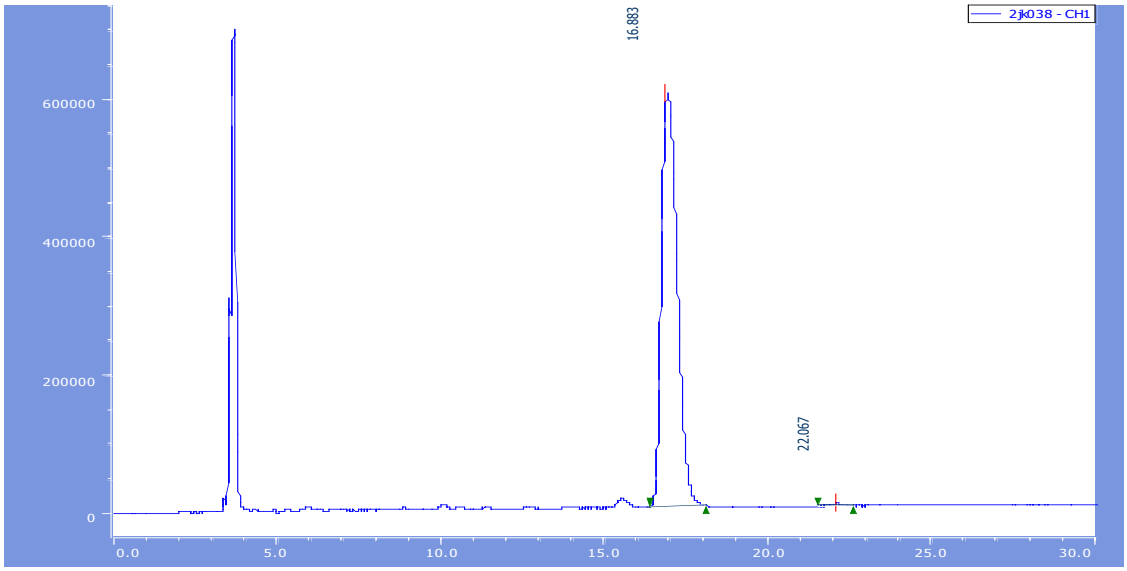
6f (racemate)

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	20.49	3448649	50.40
2	23.55	3394032	49.60

6f

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	20.92	186421	3.94
2	24.12	4541731	96.06



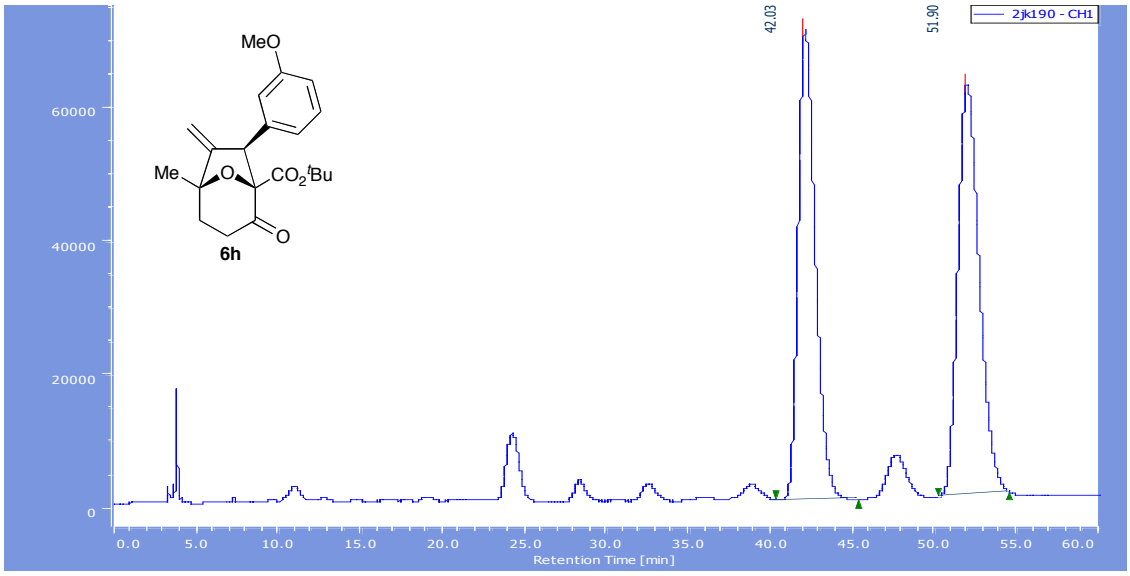


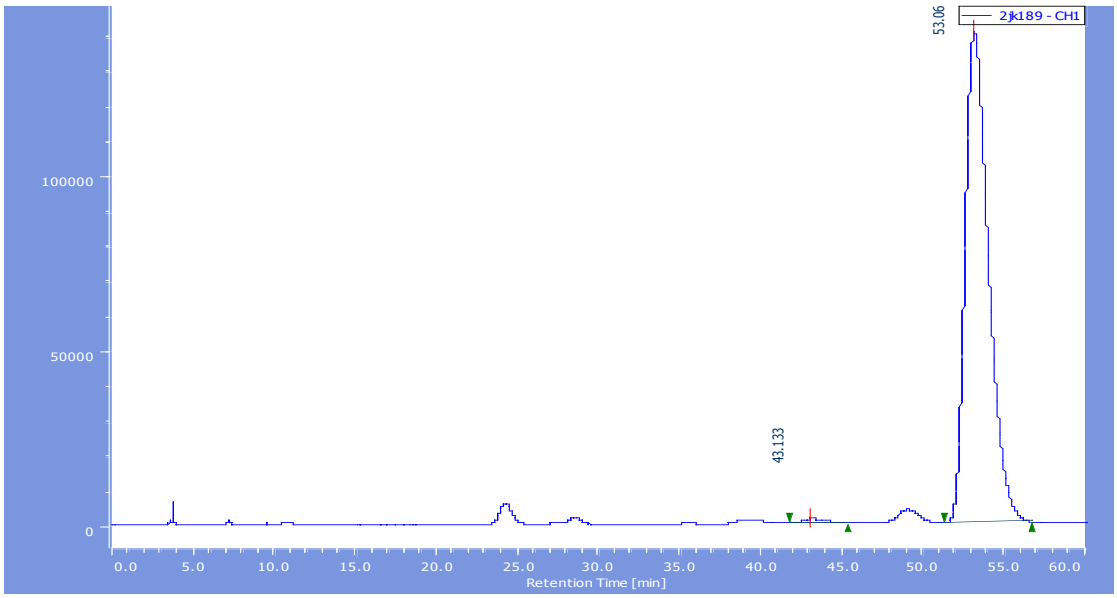
6g (racemate)

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	17.00	6337363	50.04
2	21.96	6326718	49.96

6g

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	16.83	20196325	99.50
2	22.06	100717	0.50



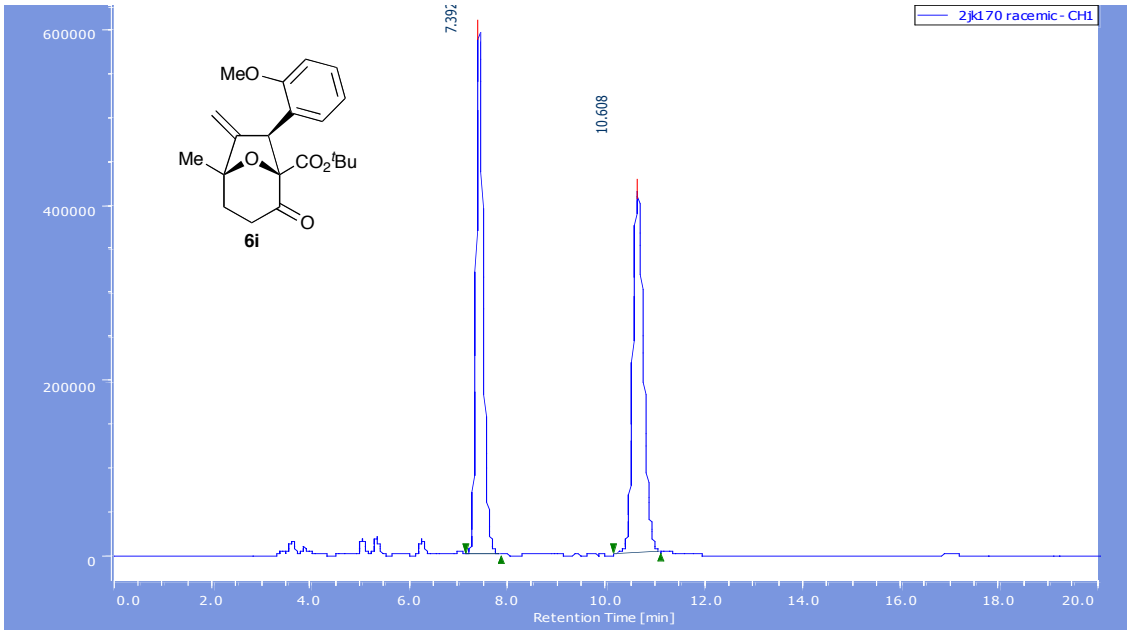


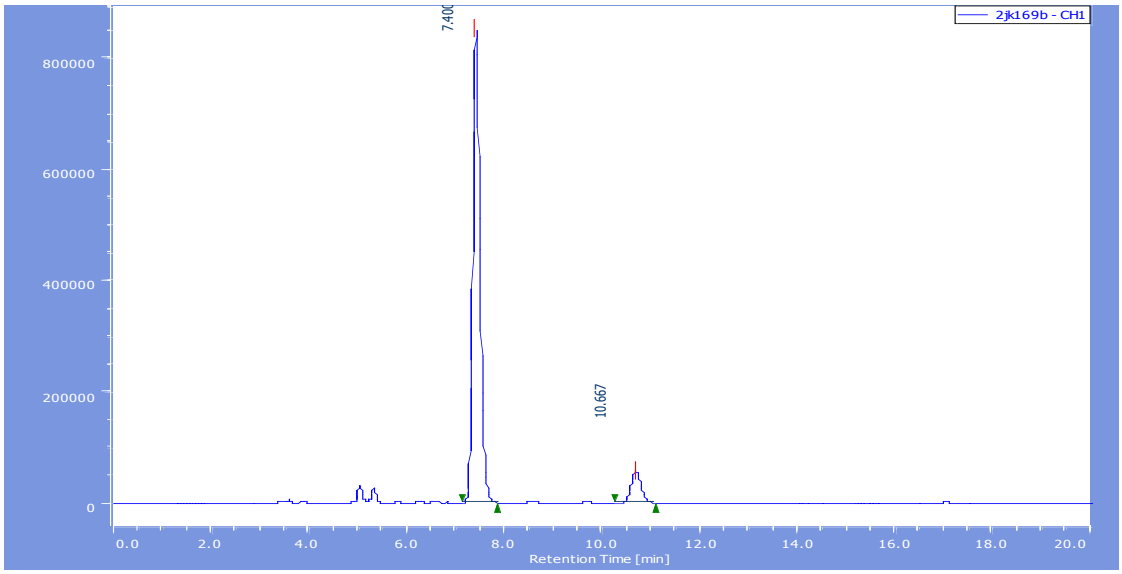
6h (racemate)

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	42.03	5155910	48.21
2	51.90	5539394	51.79

6h

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	43.13	125346	0.88
2	53.06	14052844	99.12



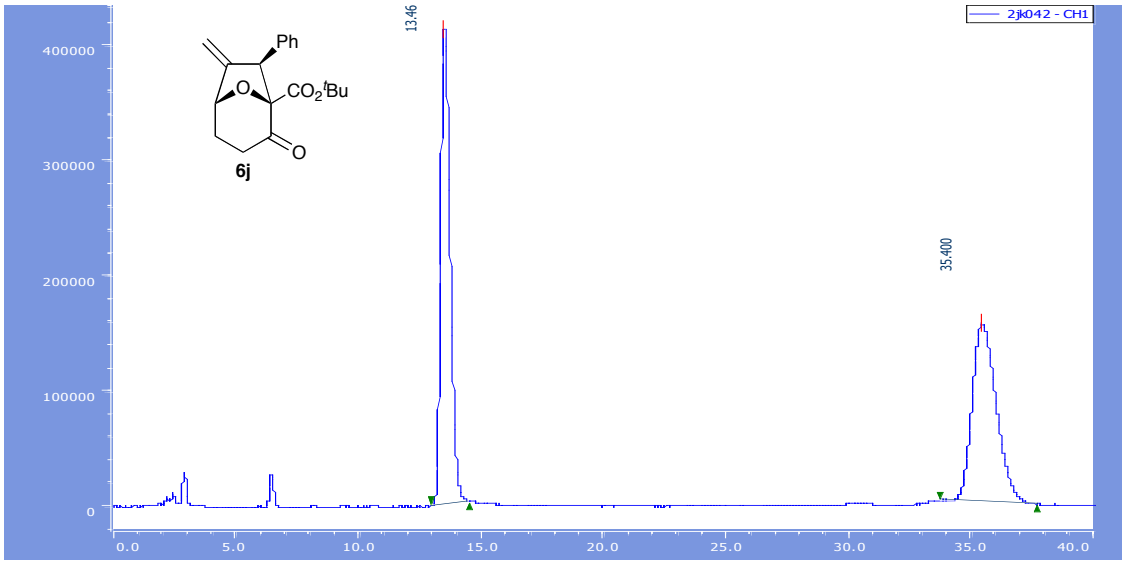


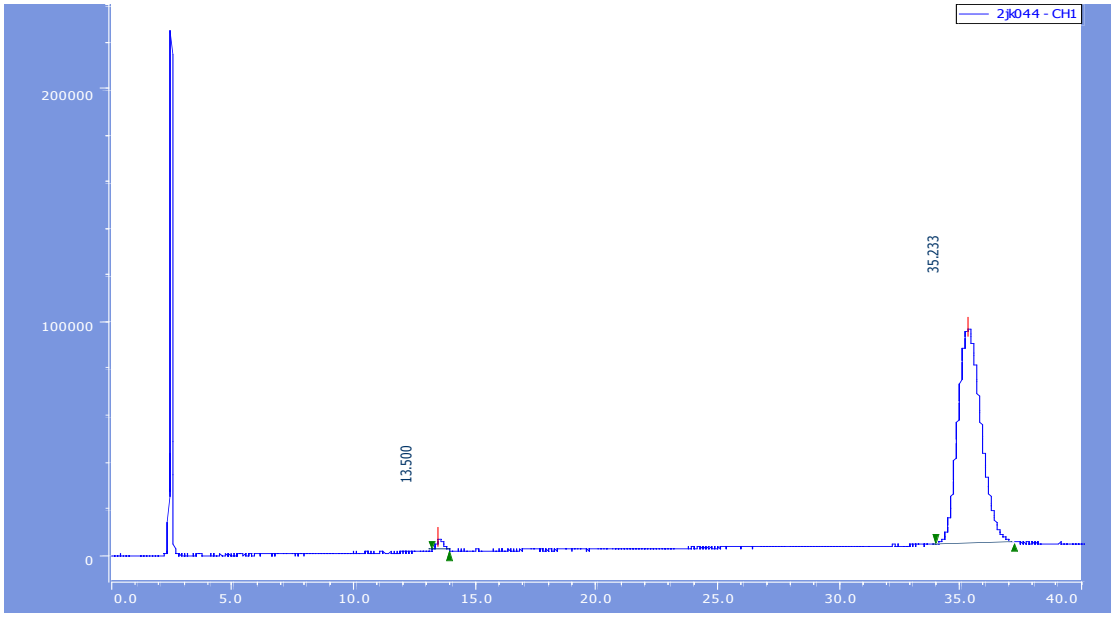
6i (racemate)

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	7.39	6191561	49.62
2	10.60	6285867	50.38

6i

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	7.40	8823821	91.41
2	10.66	828683	8.59



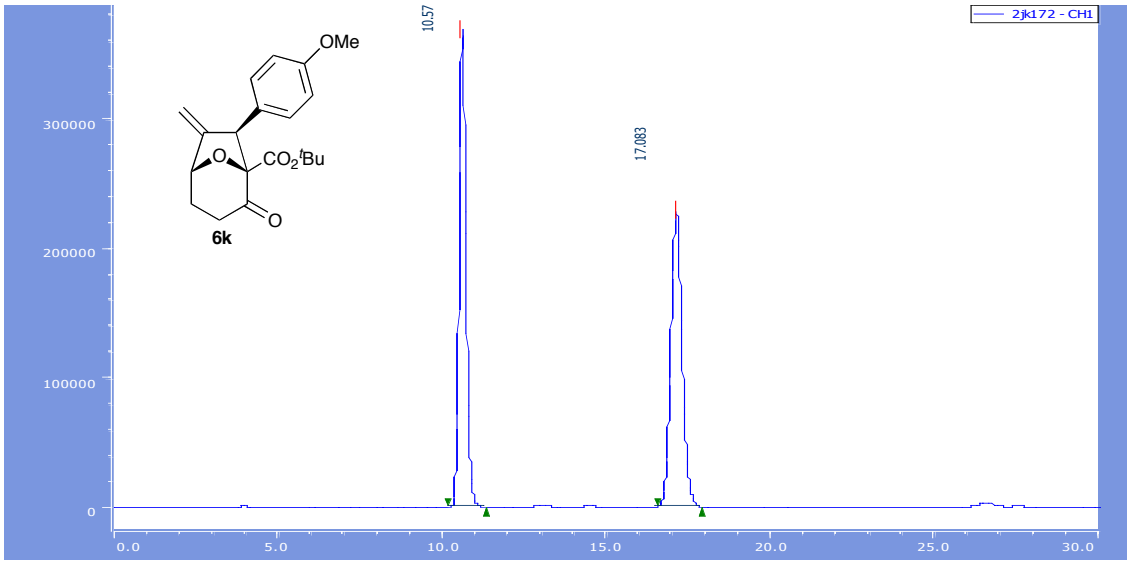


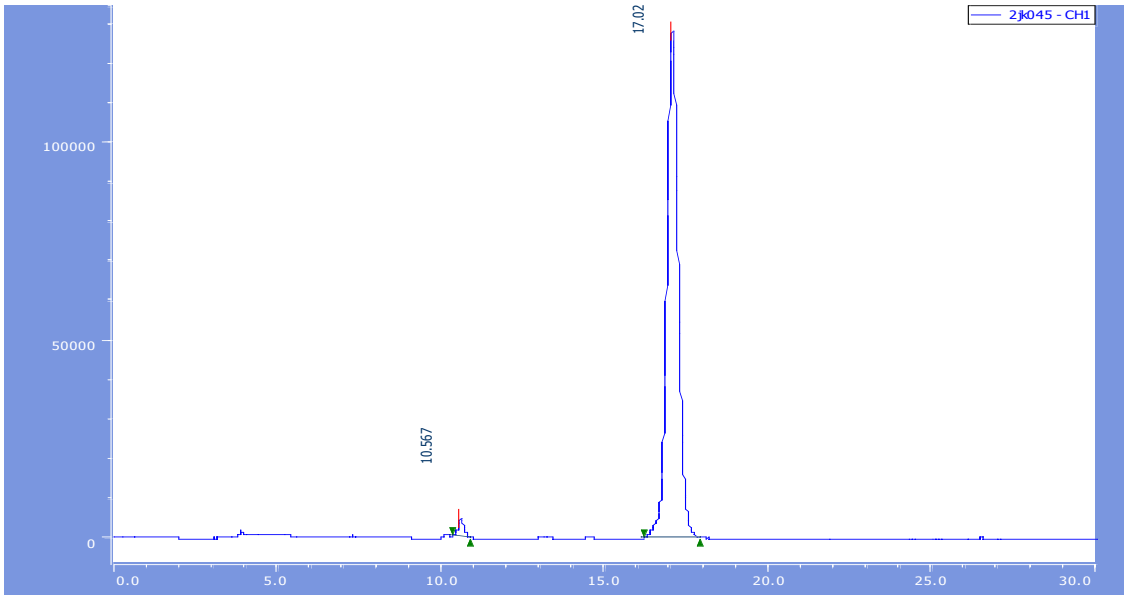
6j (racemate)

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	13.46	11051558	50.85
2	35.40	10683895	49.15

6j

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	13.50	115663	1.83
2	35.23	6221161	98.17



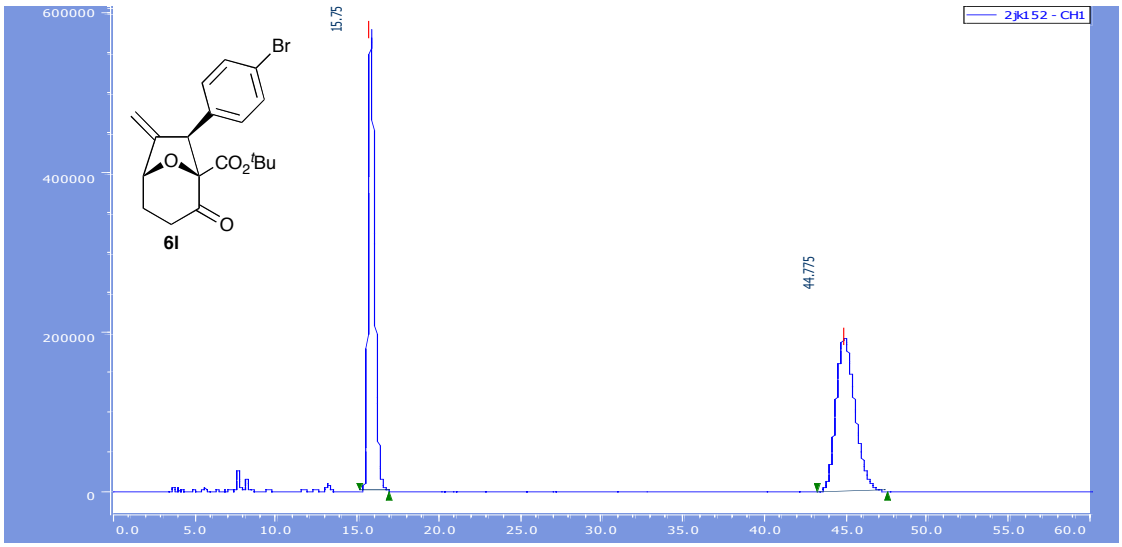


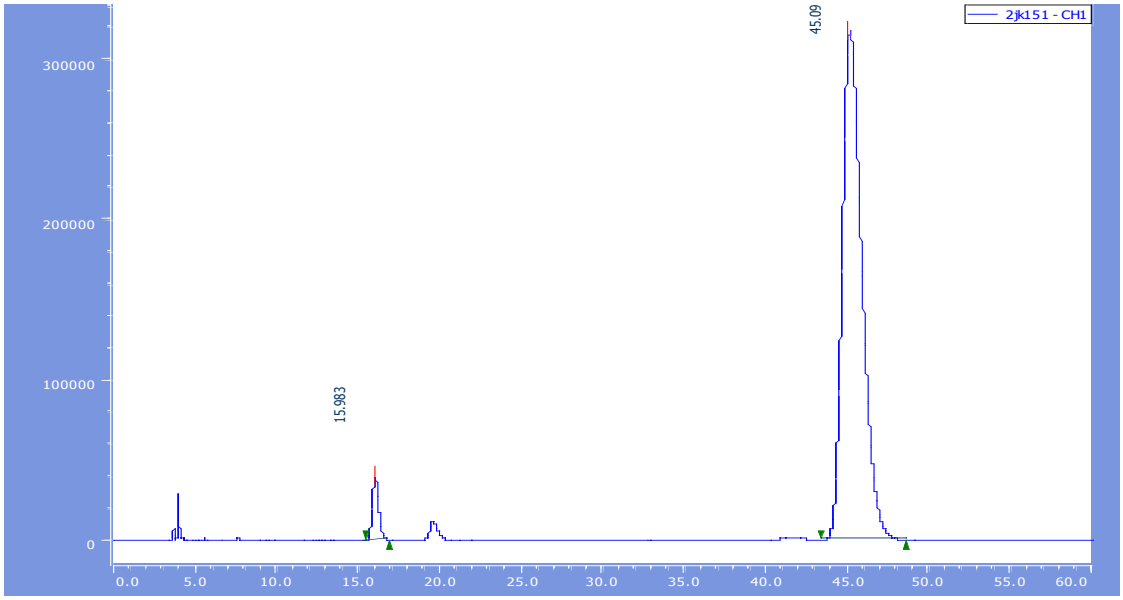
6k (racemate)

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	10.57	5468442	49.67
2	17.08	5540672	50.33

6k

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	10.56	262316	1.96
2	17.00	13089585	98.04



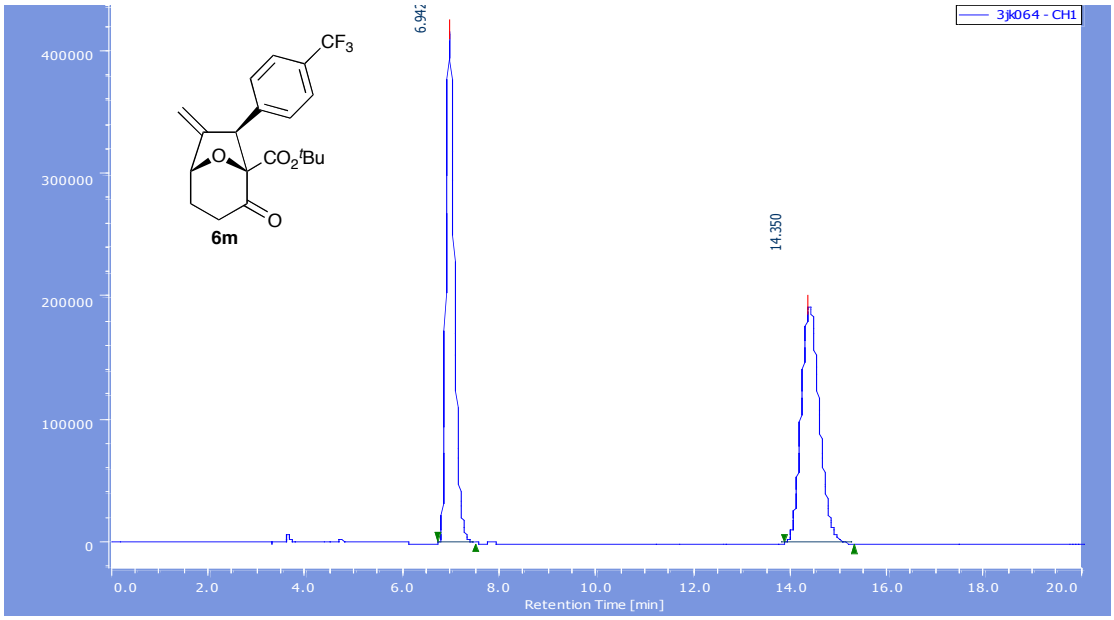


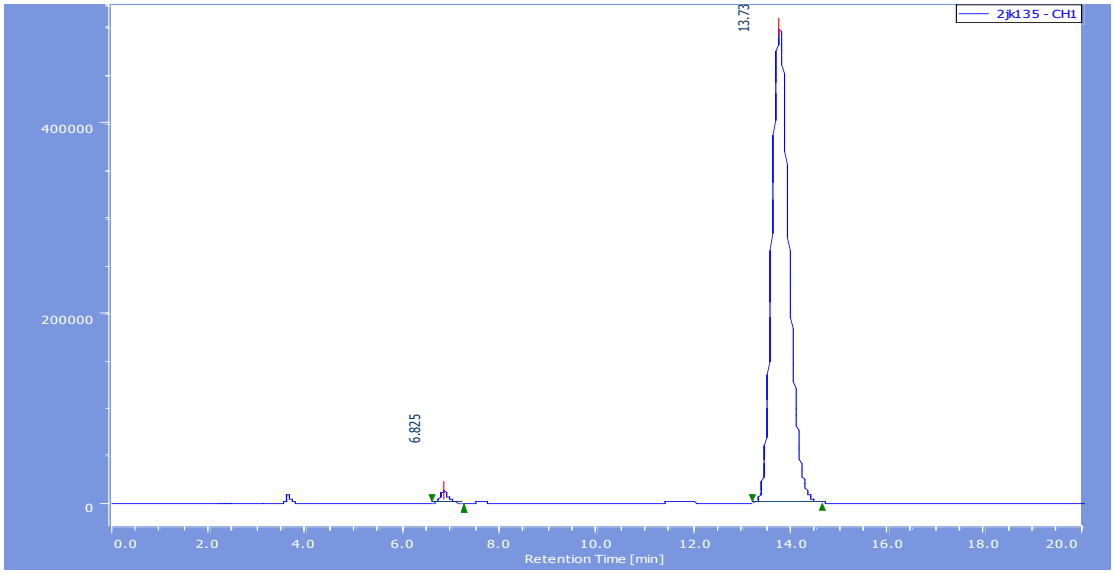
6l (racemate)

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	15.75	15960713	50.12
2	44.77	15884190	49.88

6l

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	15.98	1086556	3.90
2	45.09	26755657	96.10



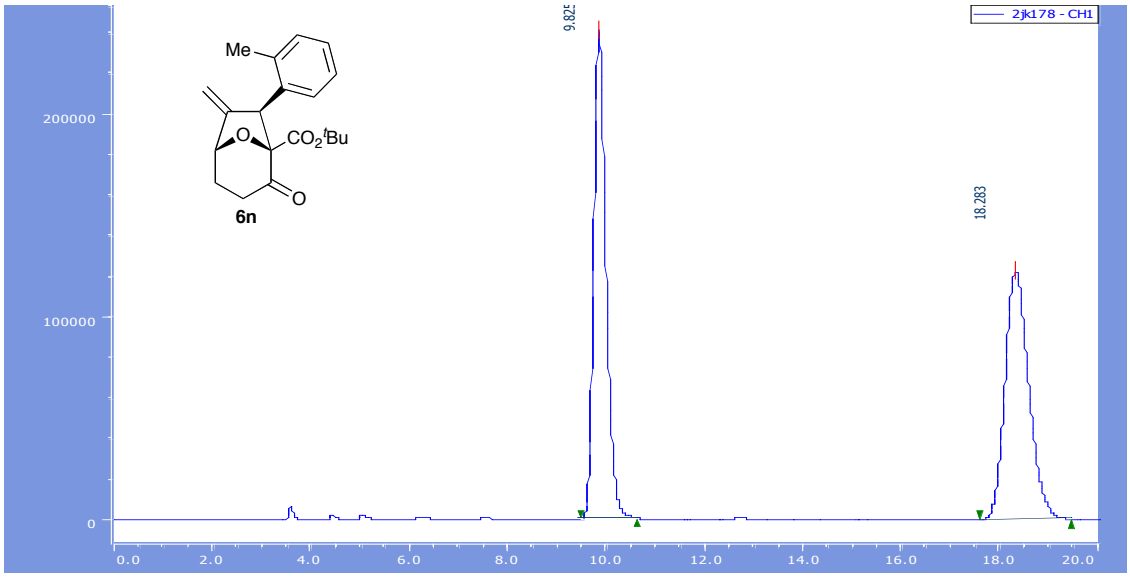


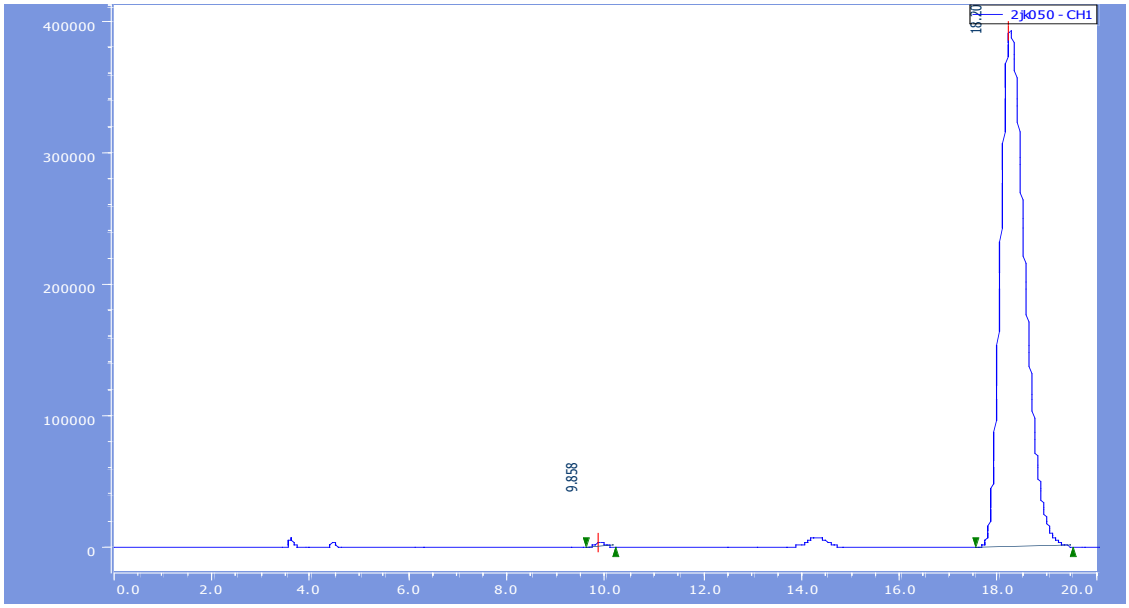
6m (racemate)

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	6.94	5033203	49.61
2	14.35	5112898	50.39

6m

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	6.82	155091	1.23
2	13.73	12417622	98.77



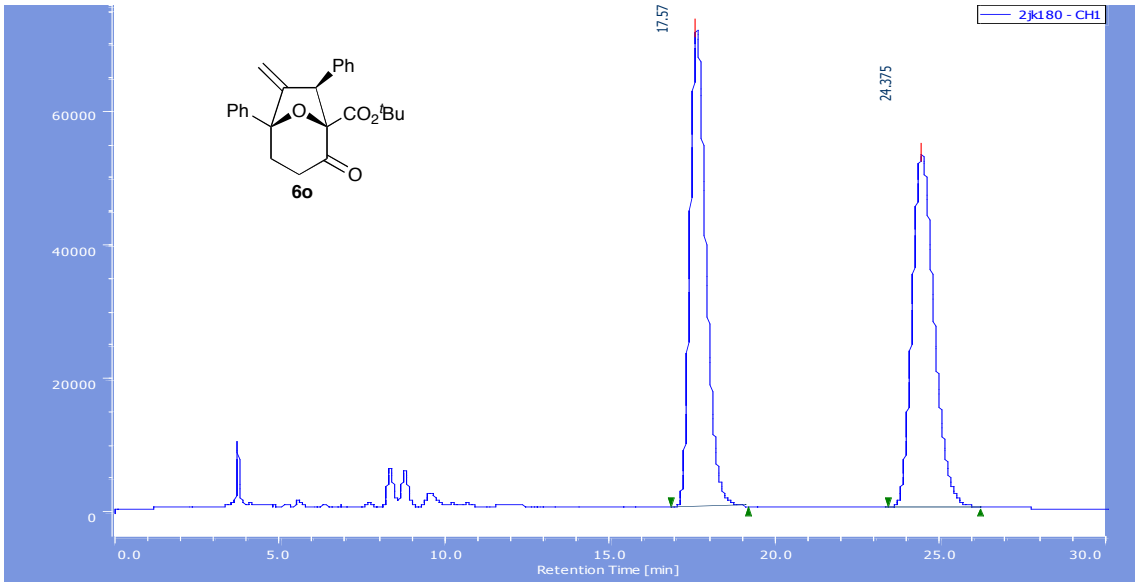


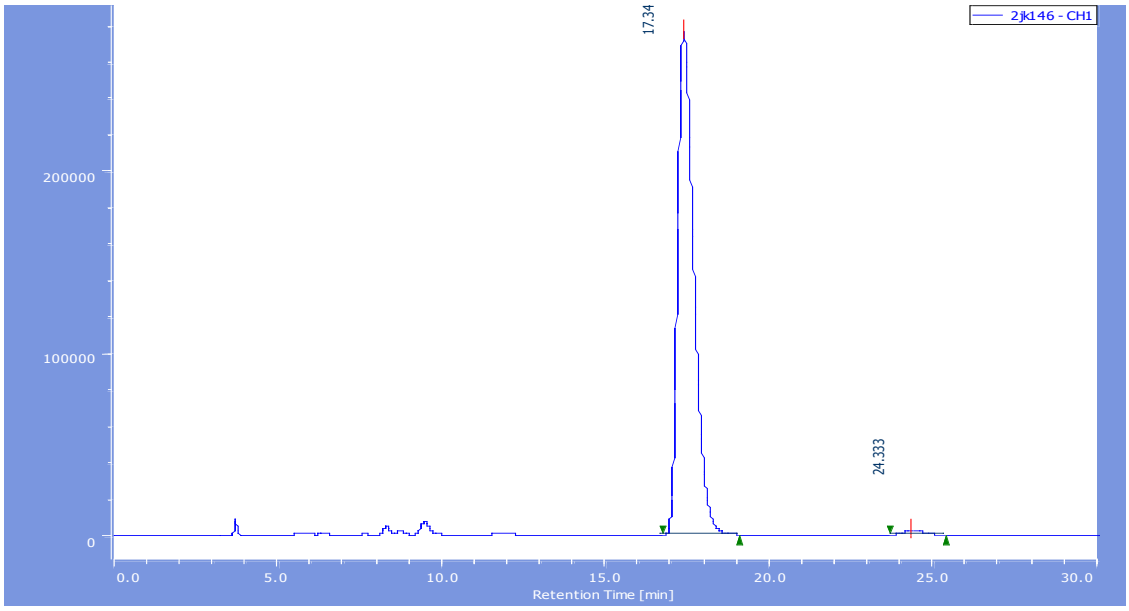
6n (racemate)

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	9.82	4178429	50.25
2	35.40	4136677	49.75

6n

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	9.85	51425	0.37
2	18.20	13715375	99.63



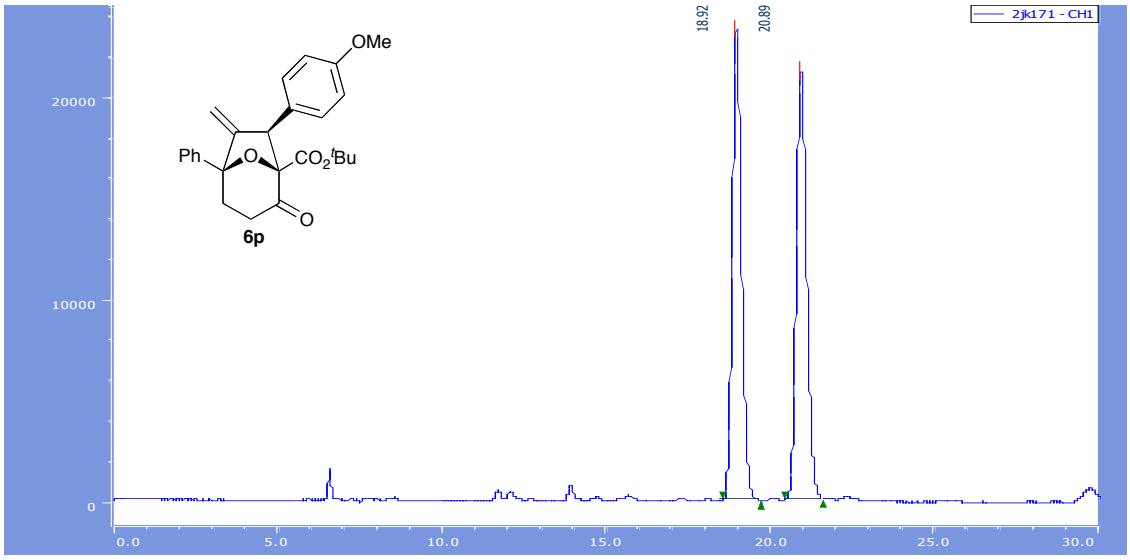


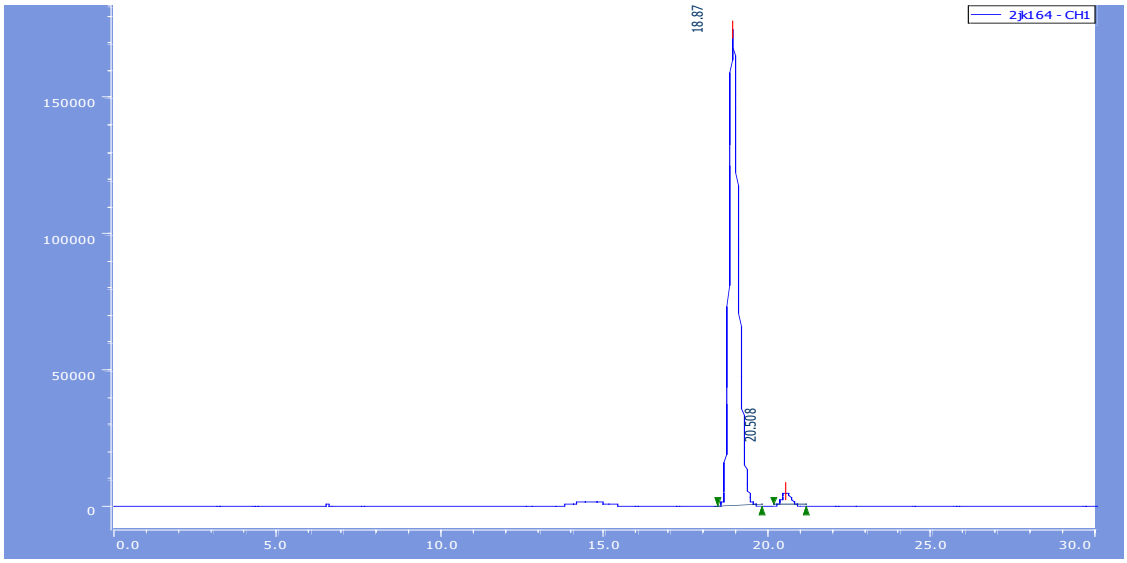
6o (racemate)

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	17.57	2421147	49.53
2	24.37	2467064	50.47

6o

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	17.34	9582901	98.73
2	24.33	123547	1.27



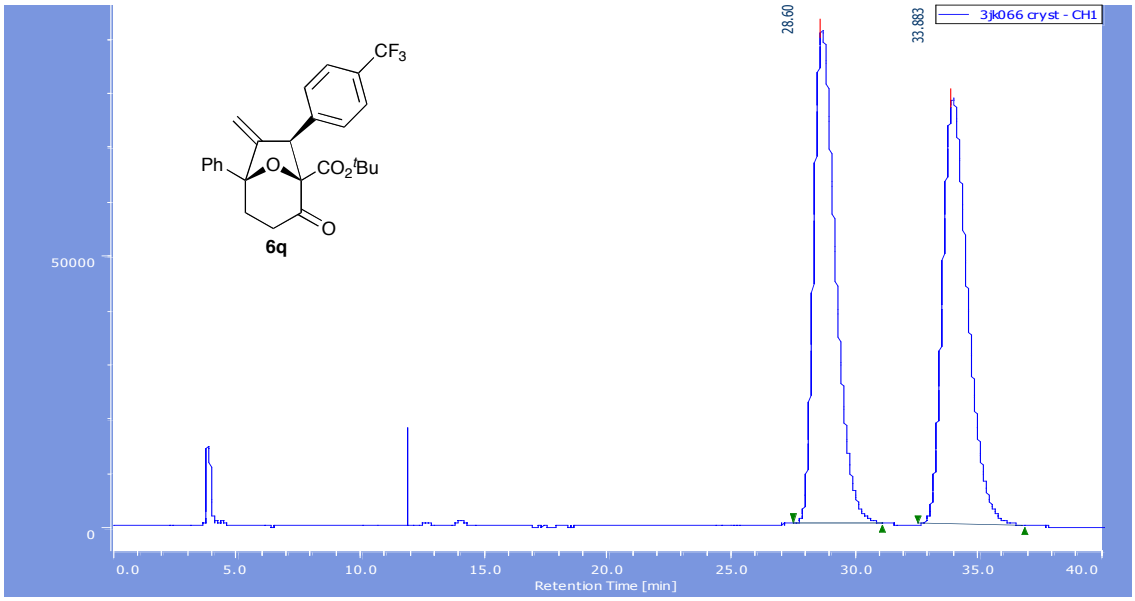


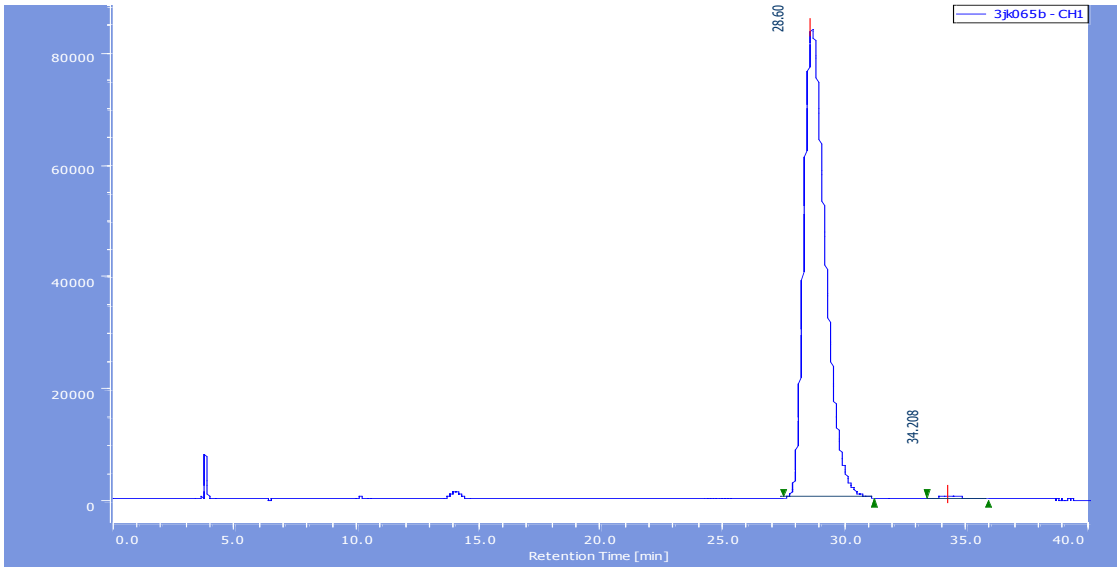
6p (racemate)

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	18.92	461628	49.68
2	20.89	467632	50.32

6p

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	18.87	3639288	97.23
2	20.50	103719	2.77



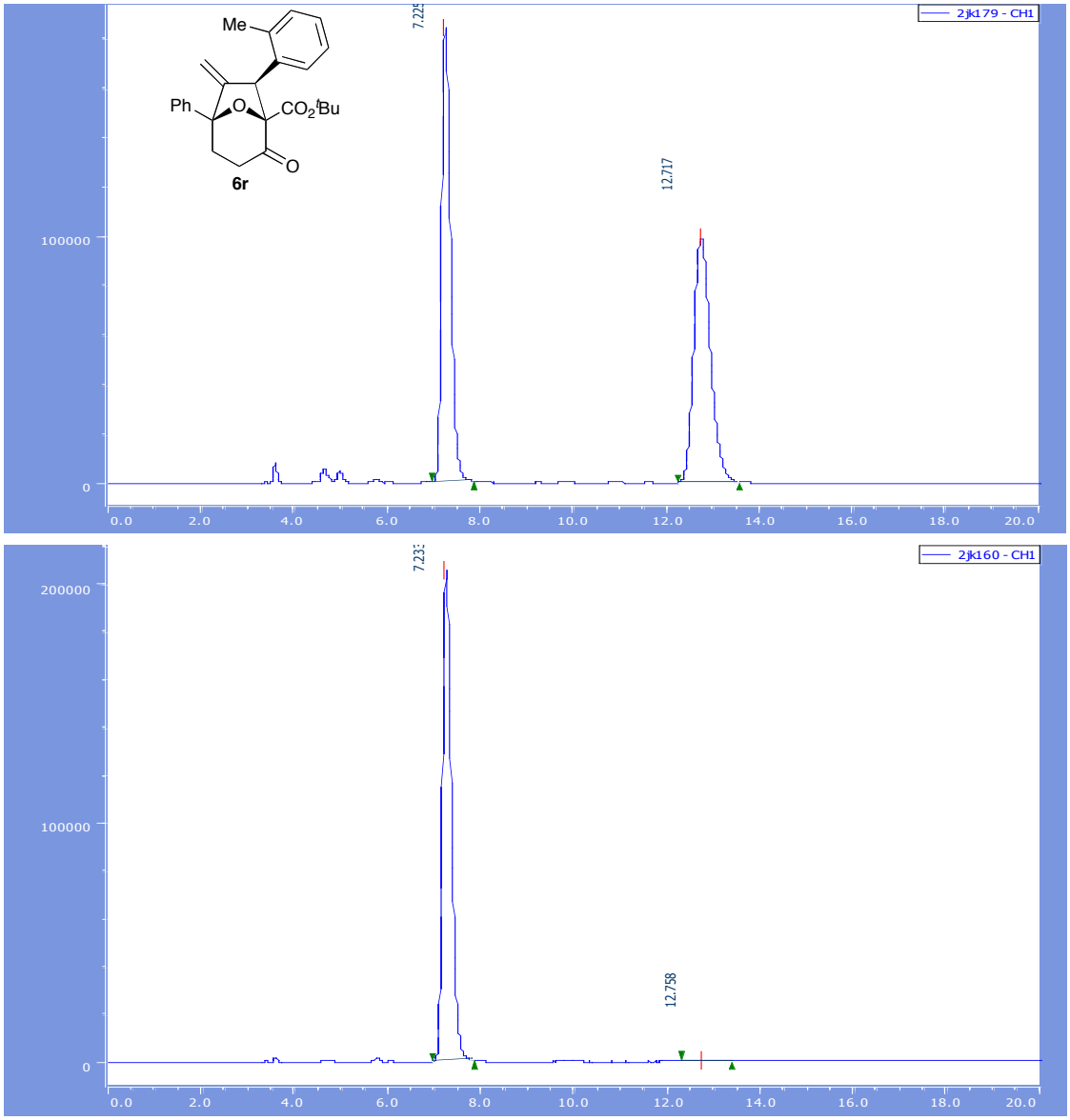


6q (racemate)

PeakNo.	Time (min)	Area [m V. Sec]	Area %
1	28.60	5576543	50.00
2	33.88	5575944	50.00

6q

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	28.60	5157113	99.34
2	34.20	34507	0.66



6r (racemate)

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	7.22	2436267	49.54
2	12.43	2481294	50.46

6r

Peak No.	Time (min)	Area [m V. Sec]	Area %
1	7.23	2727910	99.62
2	12.75	10306	0.38