Supporting Information

TANDEM OXIDATION/CYCLIZATION REACTION OF 4-(ARYLMETHYL)OXY-2-DIAZOBUTYRATE DERIVATIVES

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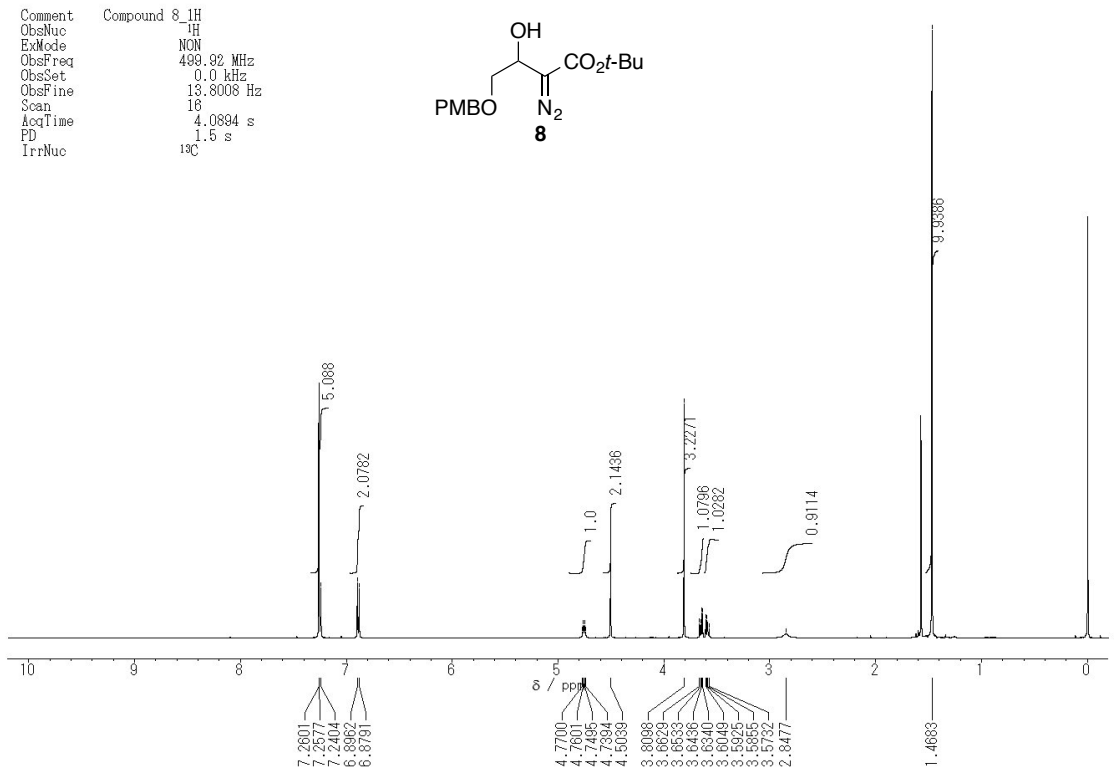
1. General Information

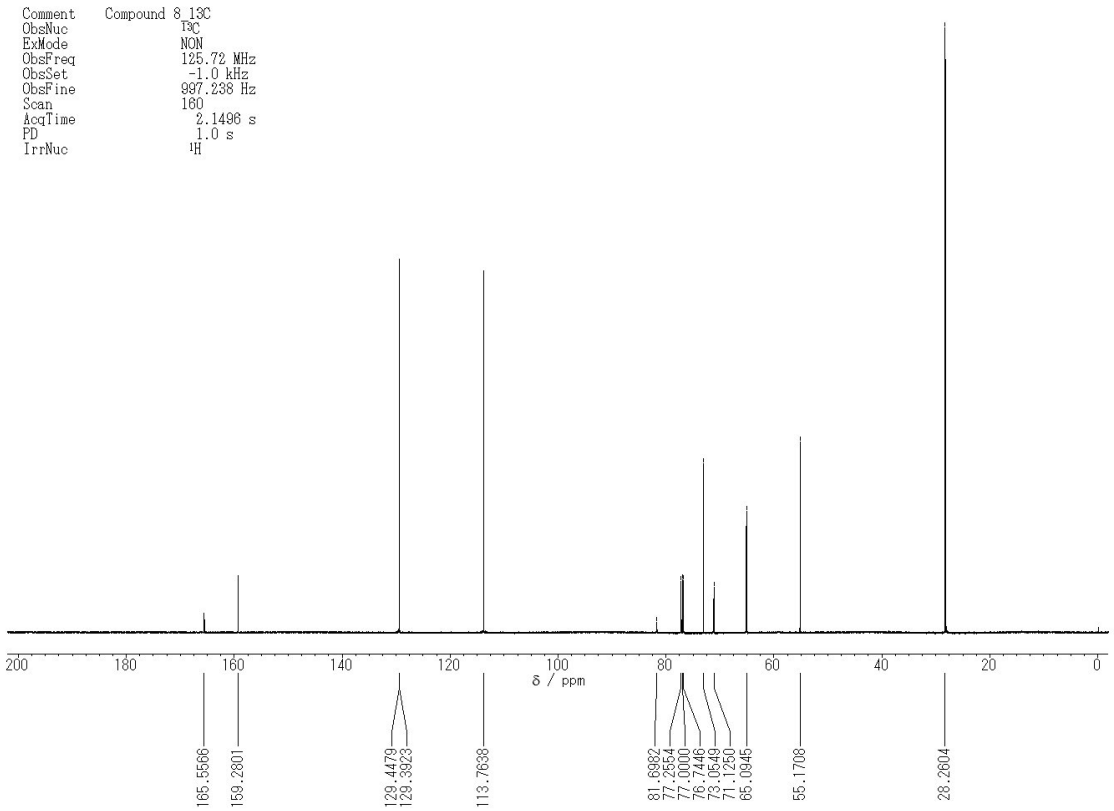
Optical rotations were recorded on a digital polarimeter with a sodium lamp (589 nm). Infrared (IR) spectra were recorded on an FT-IR spectrophotometer and absorbance bands are reported in wavenumber (cm^{-1}). Proton nuclear magnetic resonance (^1H NMR) spectra were recorded with tetramethylsilane (δ_H 0.00), C_6H_6 (δ_H 7.16) or $CHCl_3$ (δ_H 7.26) as an internal standard. Coupling constants (*J*) are reported in hertz (Hz). Abbreviations of multiplicity are as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br broad. Data are presented as follows: chemical shift, multiplicity, coupling constants, integration and assignment. Carbon nuclear magnetic resonance (^{13}C NMR) spectra were recorded with $CDCl_3$ (δ_C 77.0) or C_6D_6 (δ_C 127.0) as an internal standard. High-resolution mass spectra (HRMS) were recorded by electrospray ionization (ESI) using a time-of-flight (TOF) analyzer.

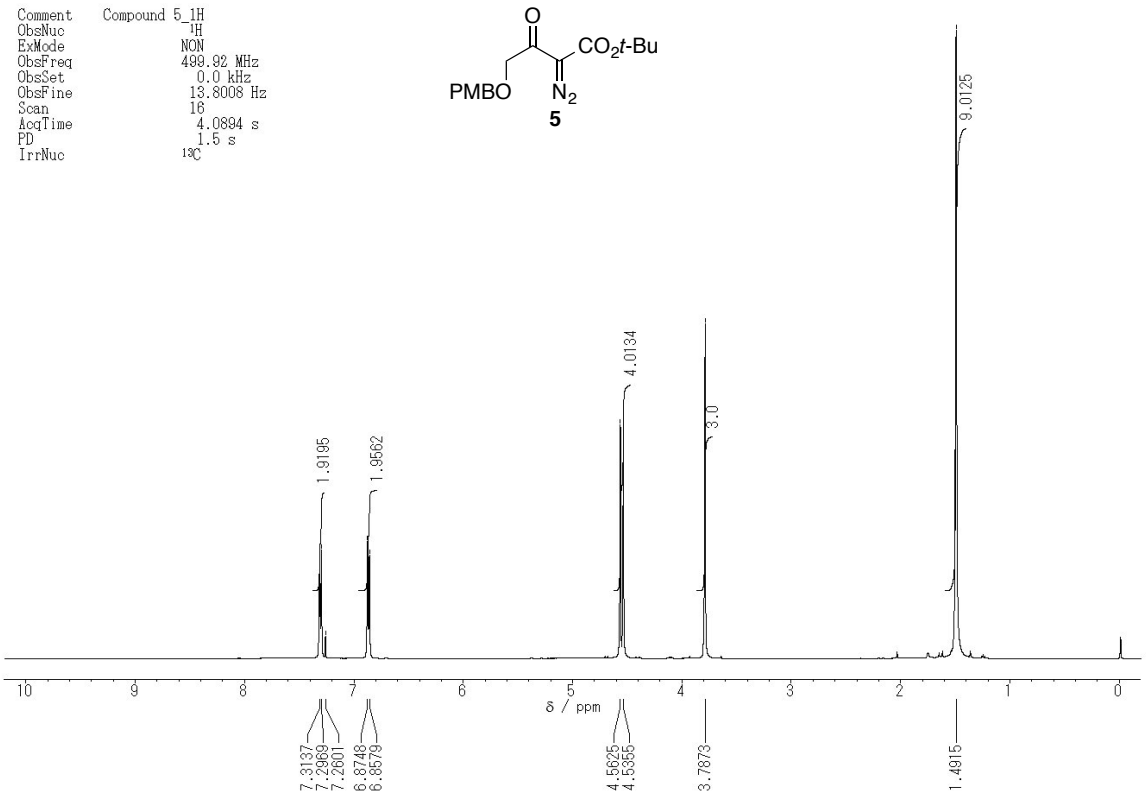
Column chromatography was carried out on silica gel 60 N (63−210 μm or 40−50 μm) or Bio-Beads S-X3 (40−80 μm). Analytical thin layer chromatography (TLC) was carried out with 0.25 mm silica gel plates. Visualization was accomplished with ultraviolet light and anisaldehyde or phosphomolybdic acid stain, followed by heating.

Reagents and solvents were purified by standard means or used as received unless otherwise noted. Dehydrated dichloromethane (CH_2Cl_2) and tetrahydrofuran (THF, stabilizer free) were purchased. Diisopropylamine and 1,1,1,3,3,3-hexamethyldisilazane (HMDS) were distilled from calcium hydride. All reactions were conducted under an argon atmosphere unless otherwise noted.

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



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CO2t-Bu
PMBO
N2
5
9.0125
4.0134
3.0
1.9195
1.9562
δ / ppm
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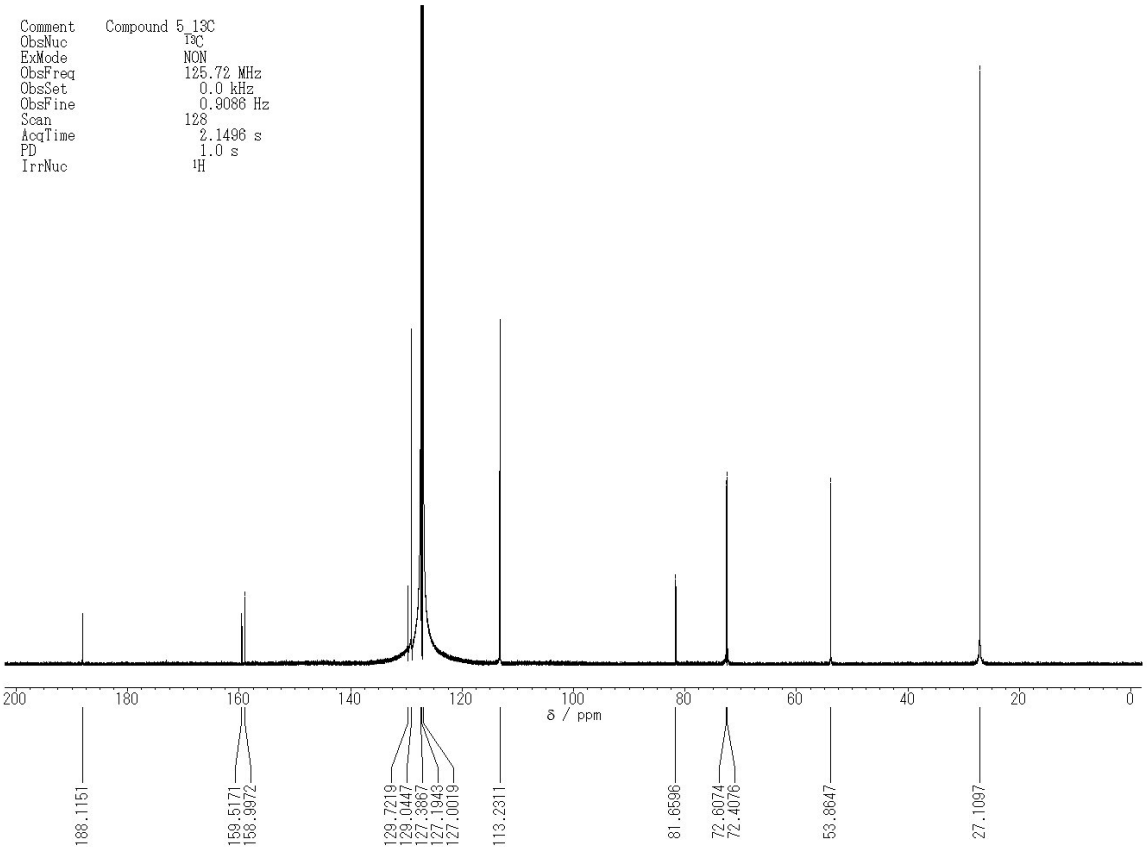
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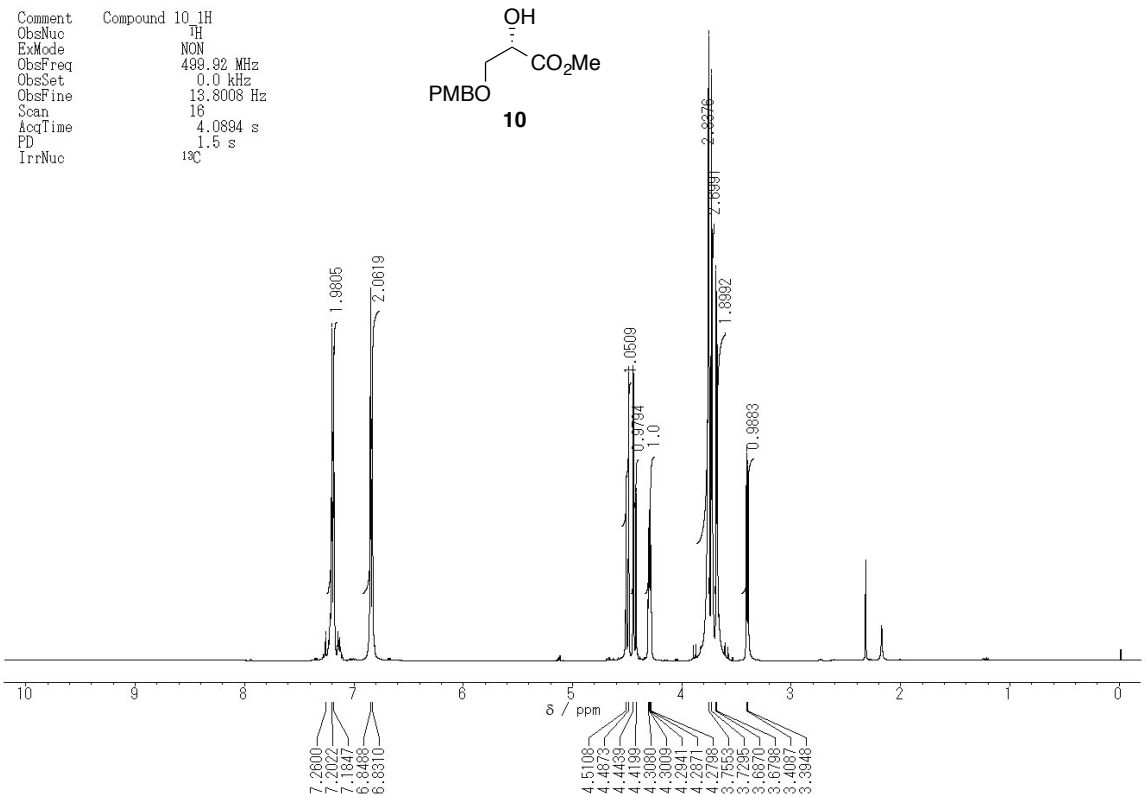
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PMBO
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1.0509
0.9794
1.0
2.8876
2.8691
1.8992
0.9883
δ / ppm
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7.2022
7.1847
6.8488
6.8310
4.5108
4.4873
4.4439
4.4199
4.3080
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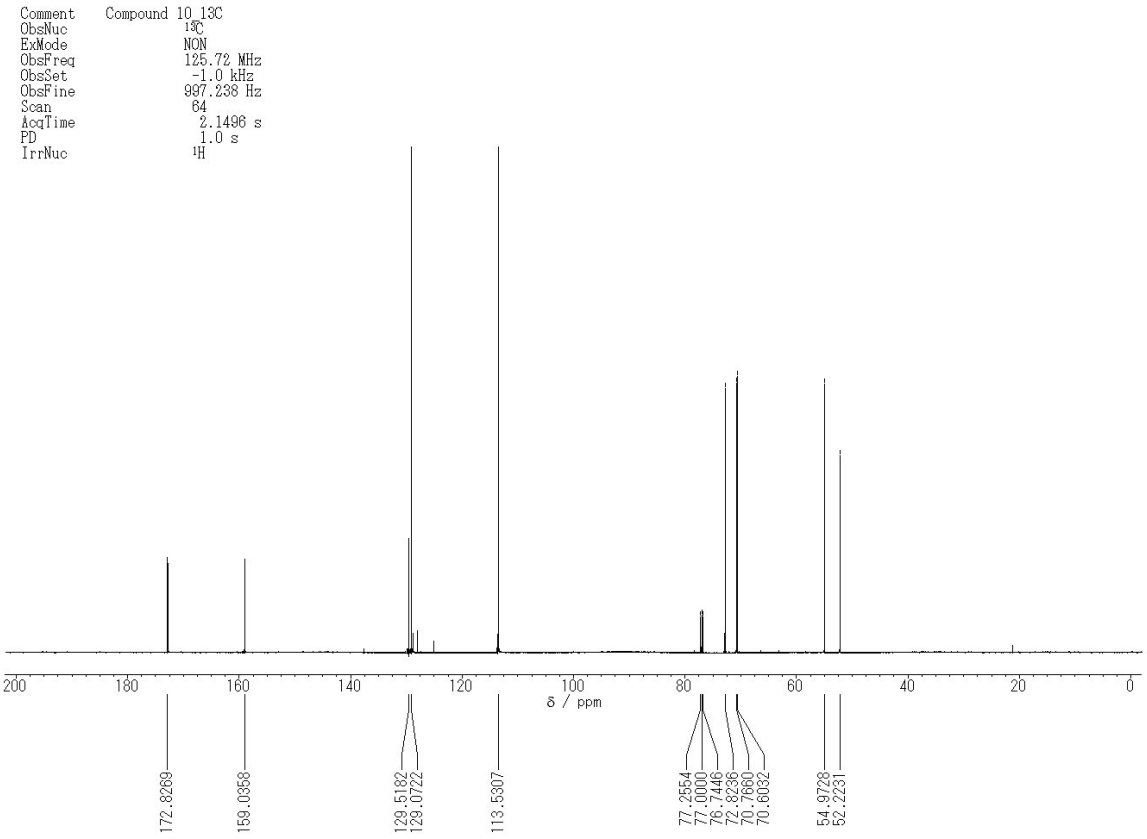
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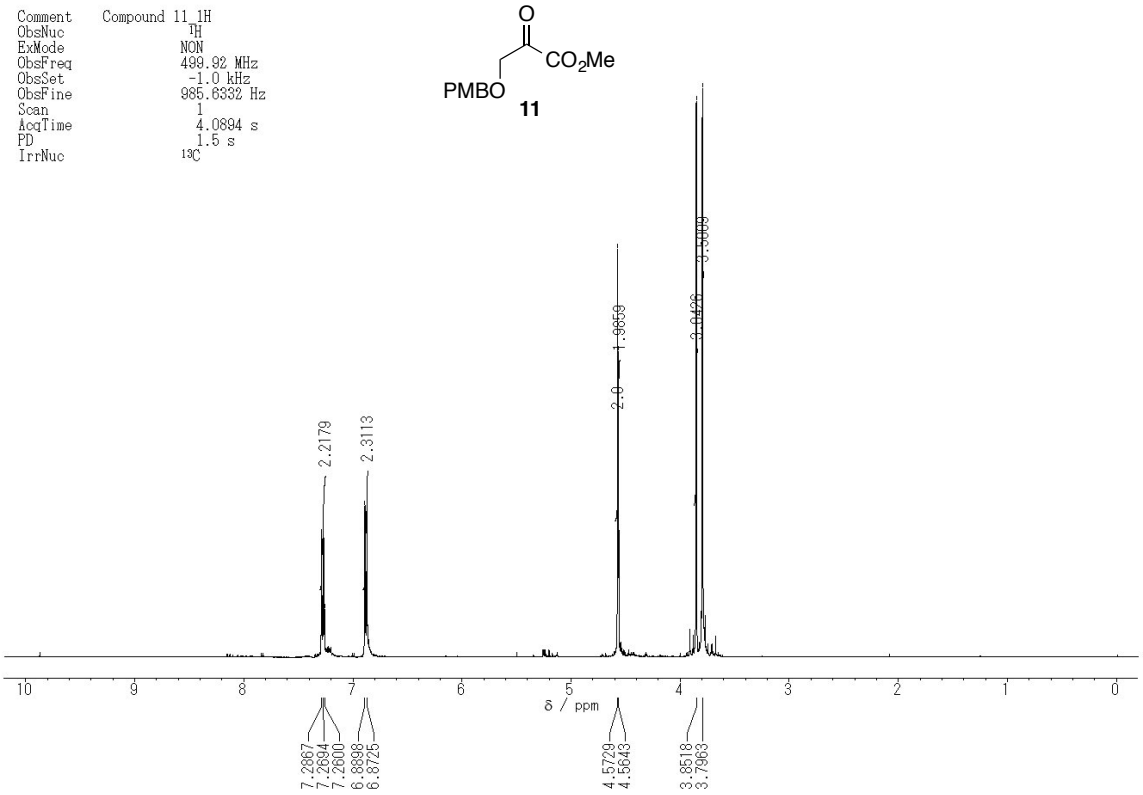
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O
CO2Me
PMBO
11
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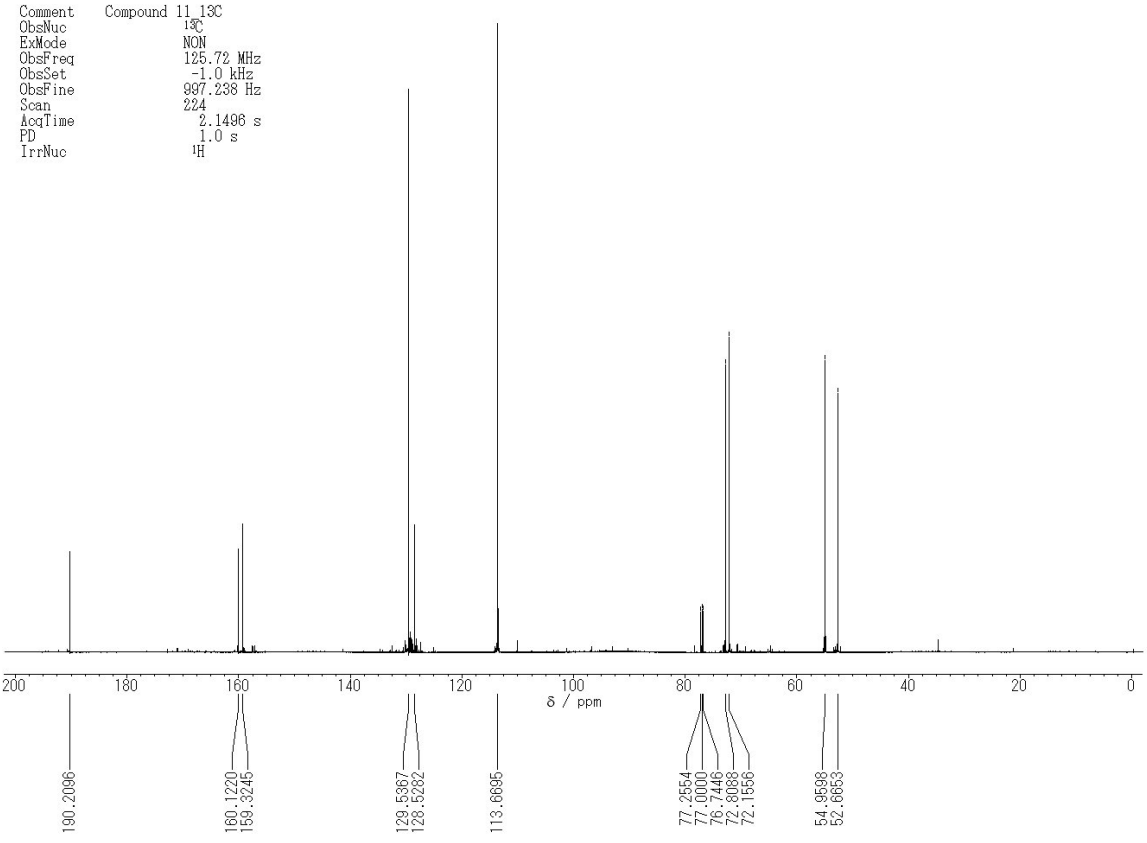
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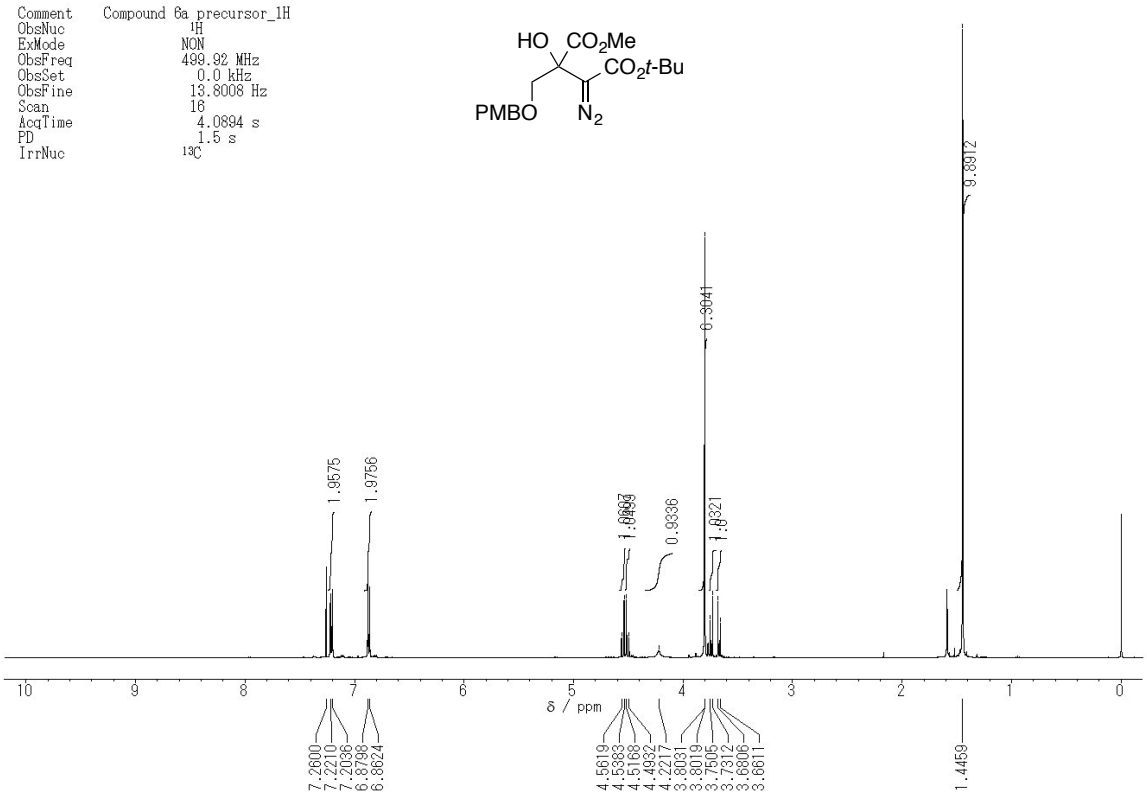
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HO CO2Me
CO2t-Bu
PMBO N2
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6.9041
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0.9336
δ / ppm
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7.2210
7.2036
6.8798
6.8624
4.5619
4.5383
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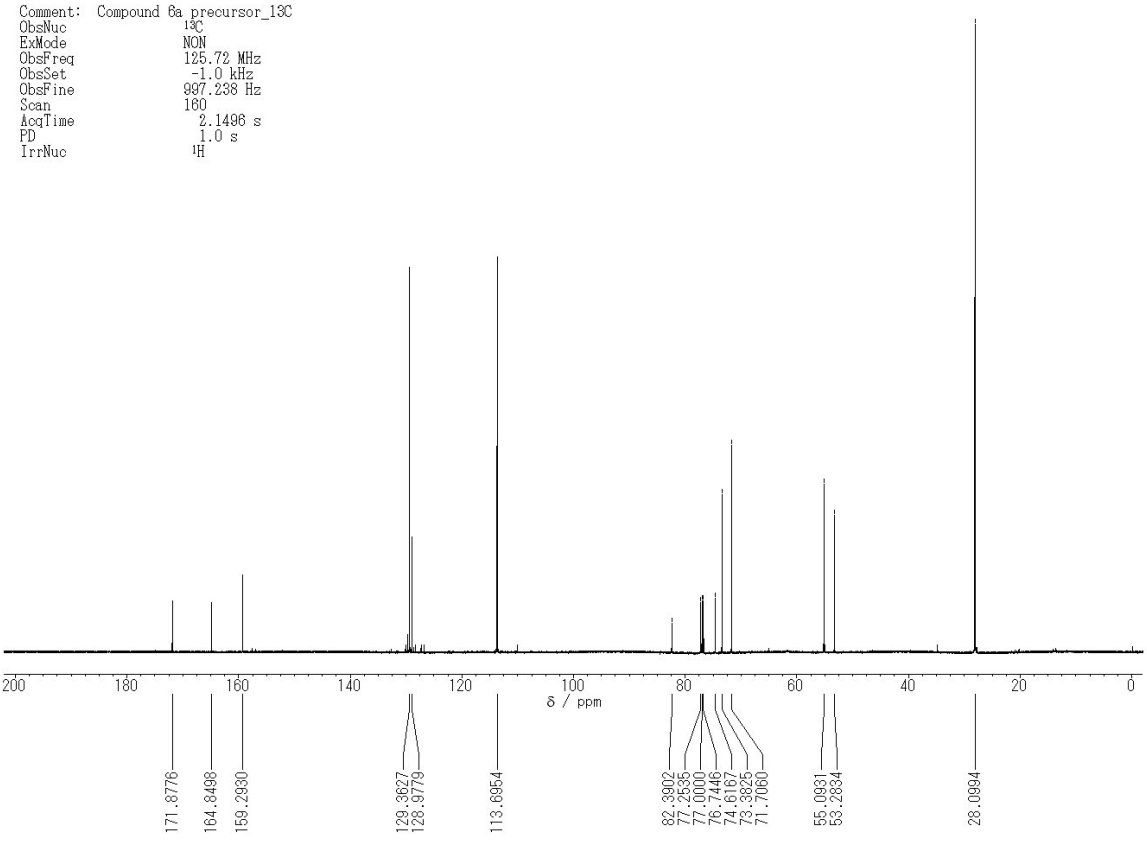
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IrrNuc 1H
δ / ppm
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159.2930
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128.9779
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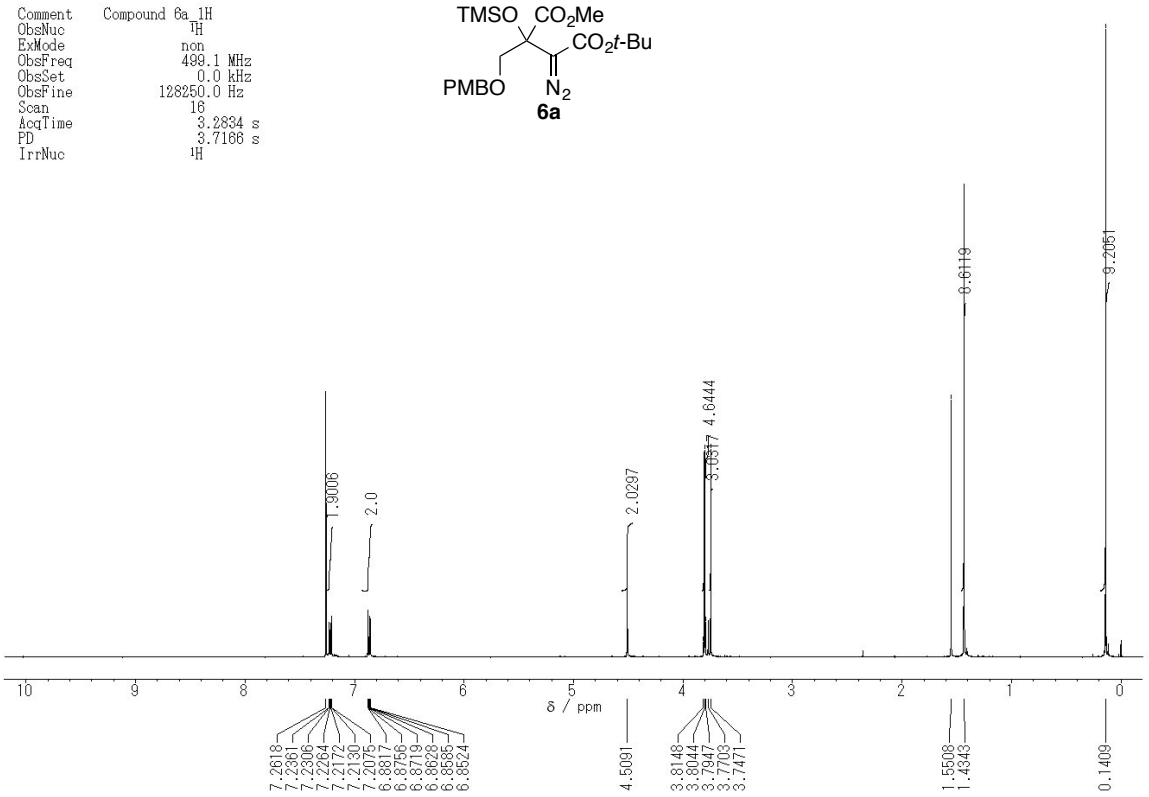
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TMSO
CO2Me
CO2t-Bu
PMBO
N2
6a
1.3006
2.0
2.0297
4.6444
6.6119
9.2051
δ / ppm
7.2618
7.2361
7.2306
7.2264
7.2172
7.2130
7.2075
6.8817
6.8756
6.8719
6.8628
6.8585
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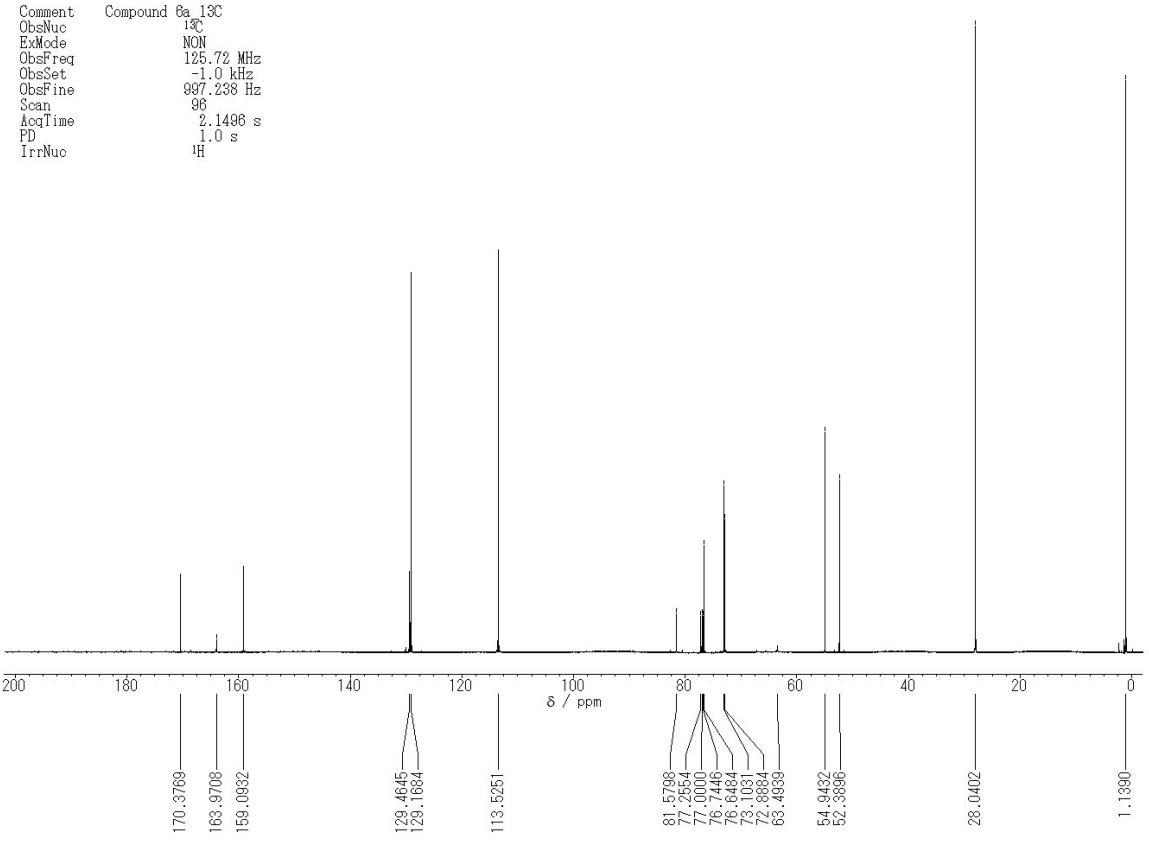
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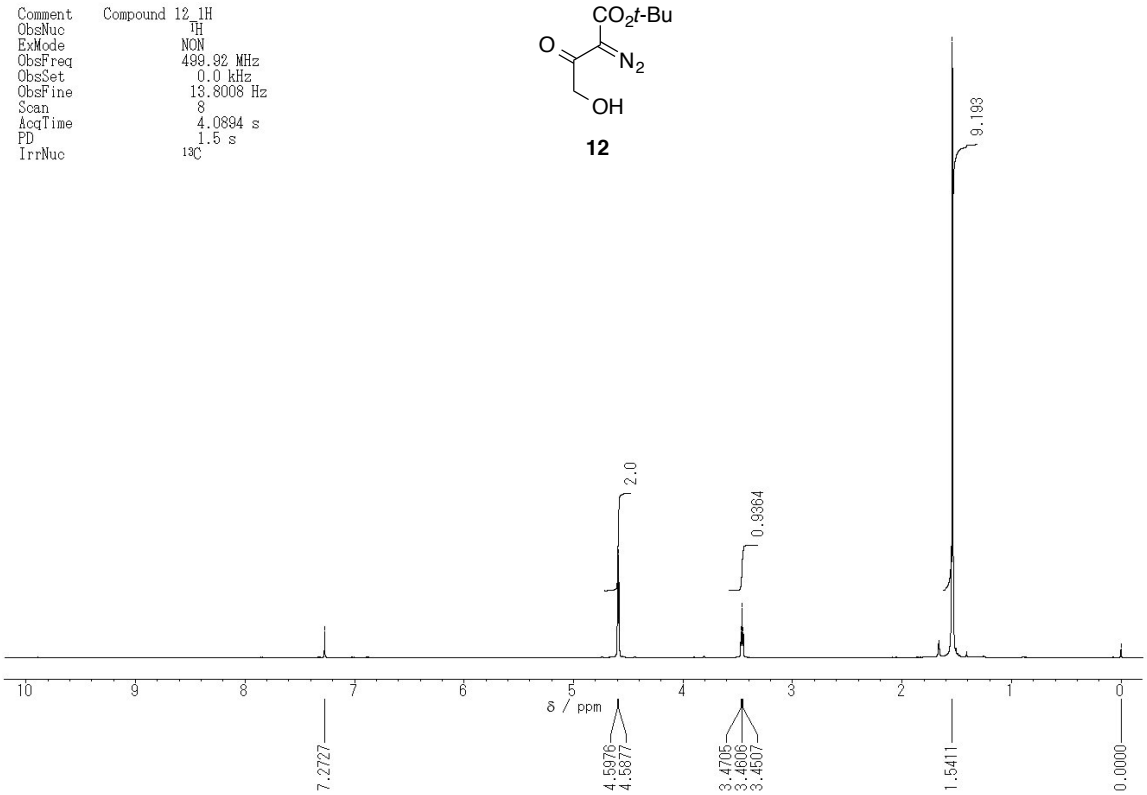
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CO2t-Bu
O
N2
OH
12
9.193
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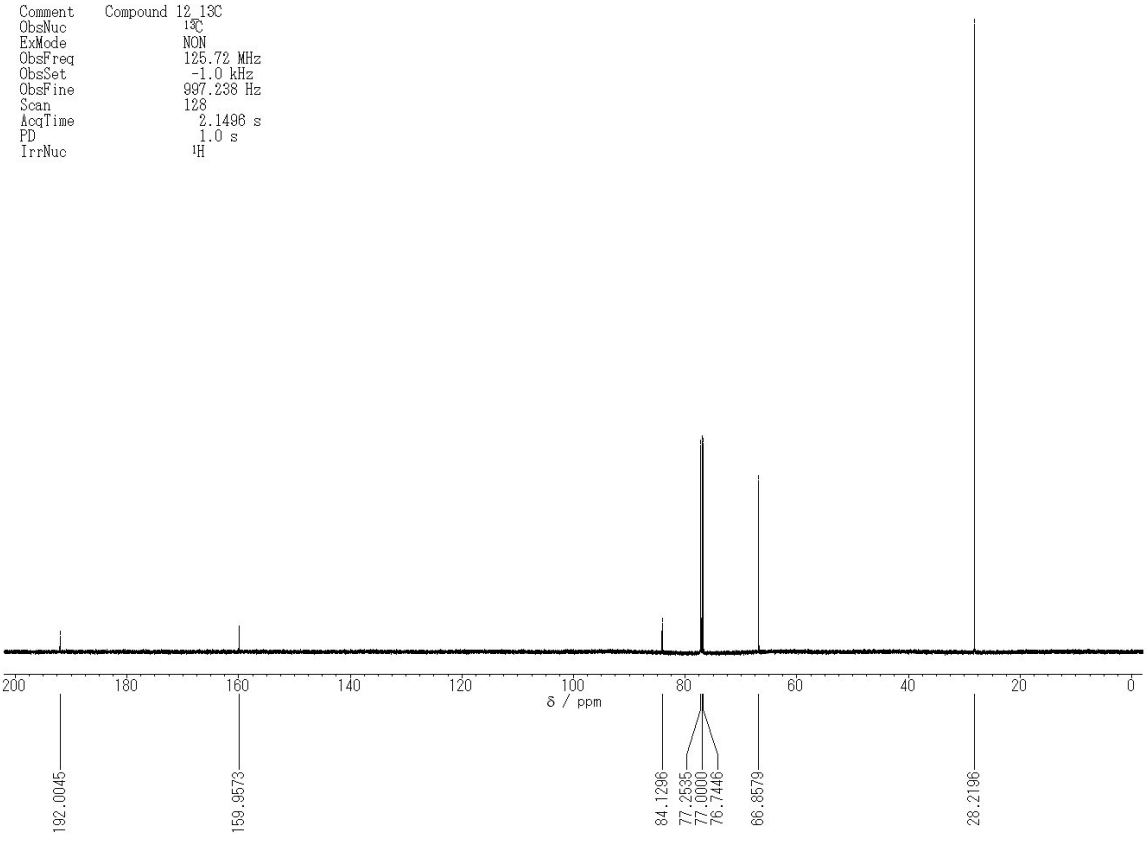
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PD 1.0 s
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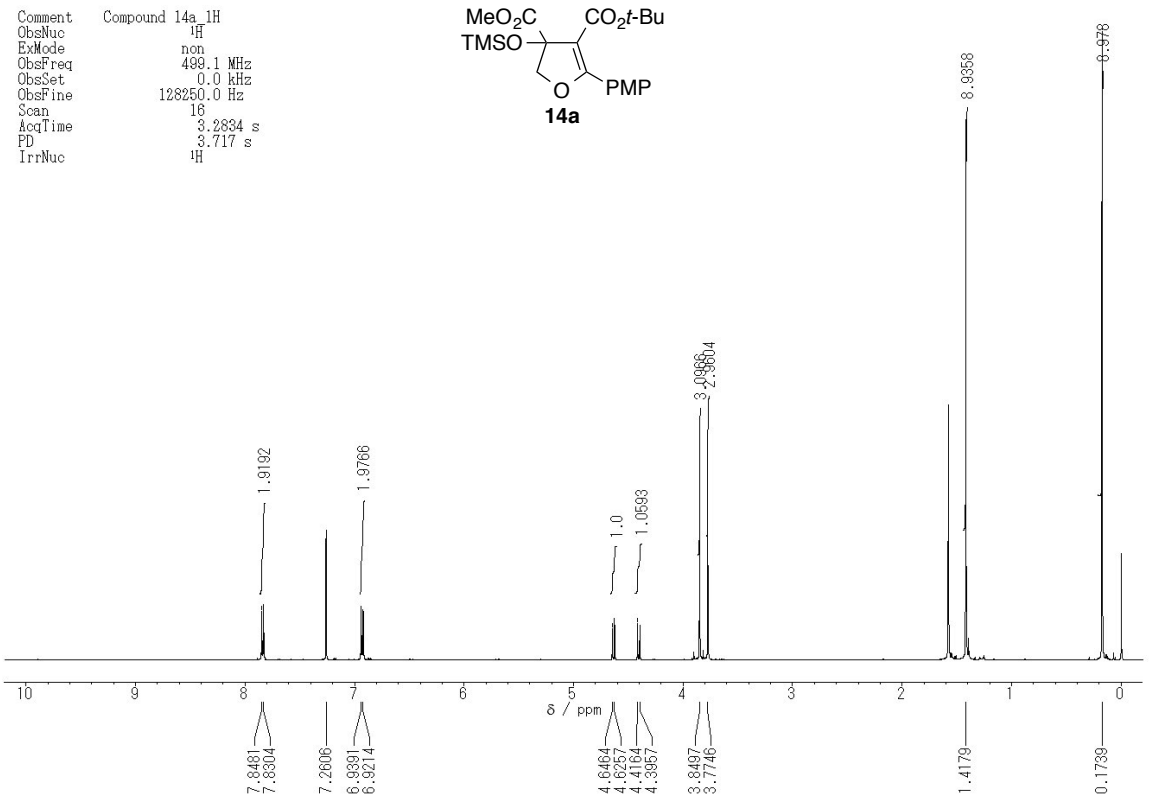
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IrrNuc 1H
MeO2C
CO2t-Bu
TMSO
O
PMP
14a
1.9192
1.9766
1.0
1.0593
3.0966
2.9604
8.9358
8.978
δ / ppm
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7.8304
7.2606
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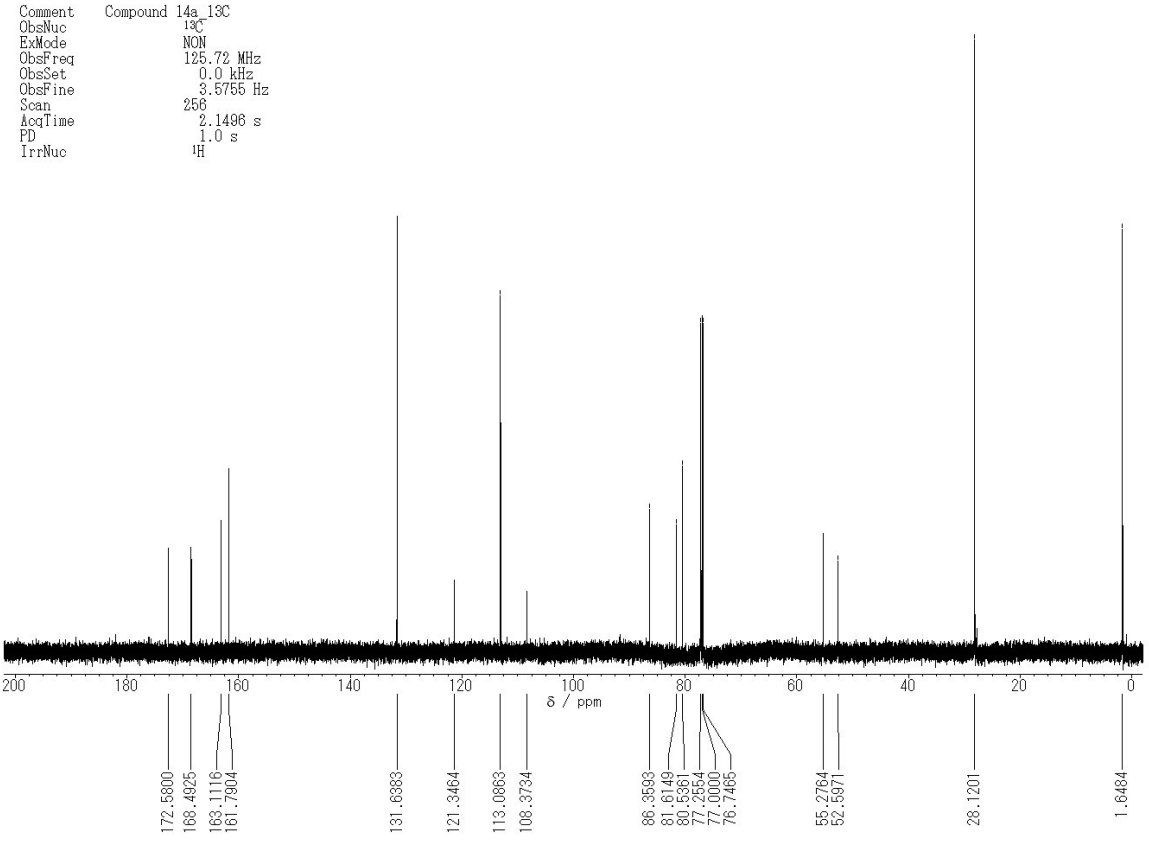
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δ / ppm
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168.4925
163.1116
161.7904
131.6383
121.3464
113.0863
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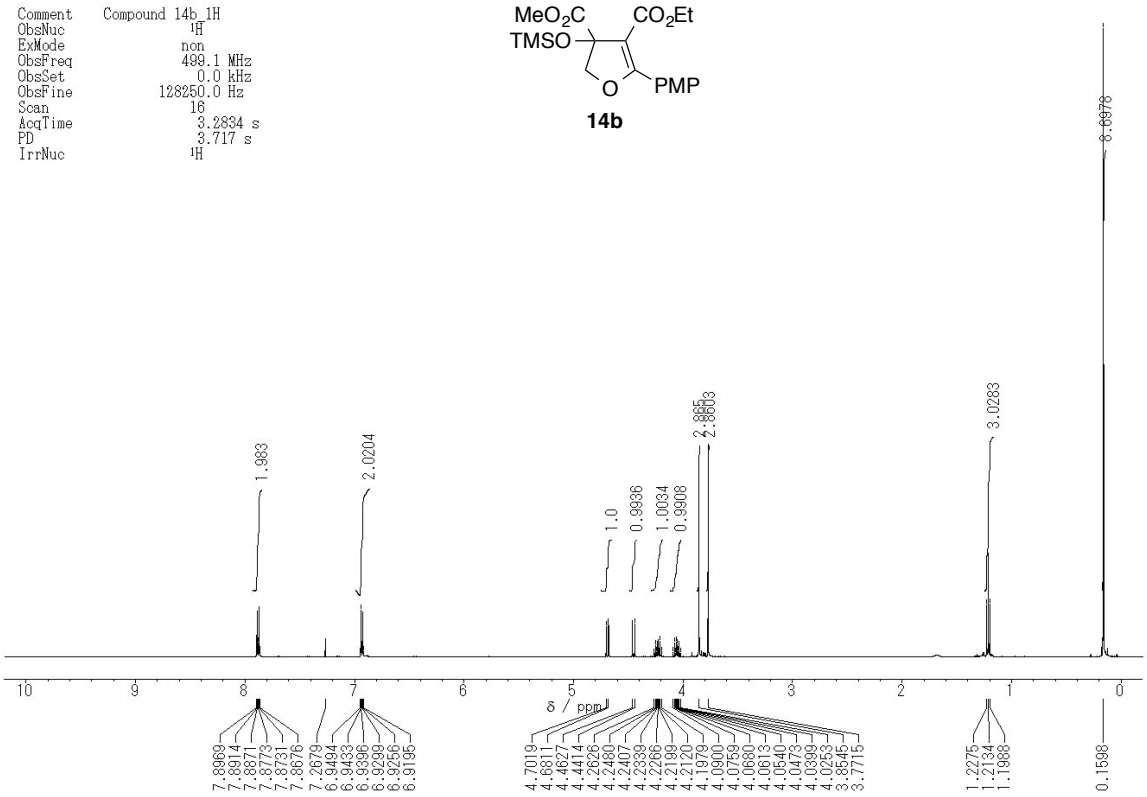
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MeO2C
CO2Et
TMSO
O
PMP
14b
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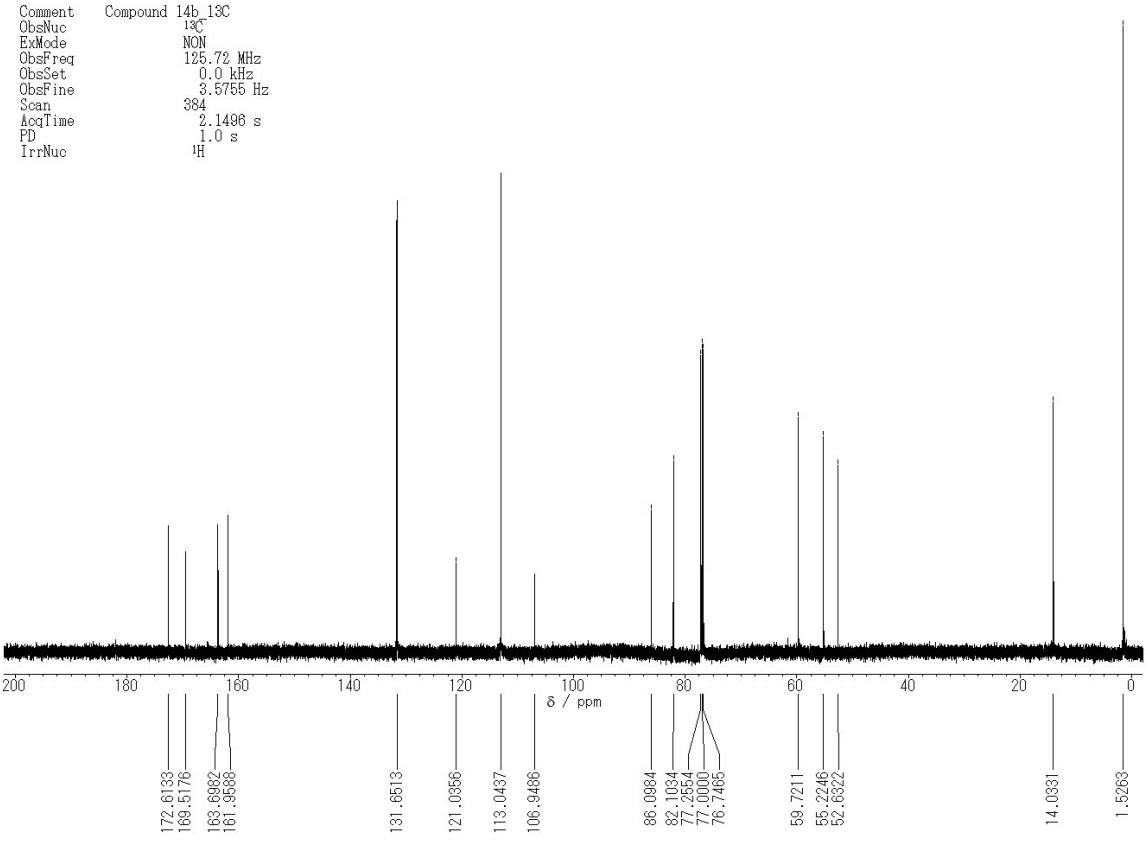
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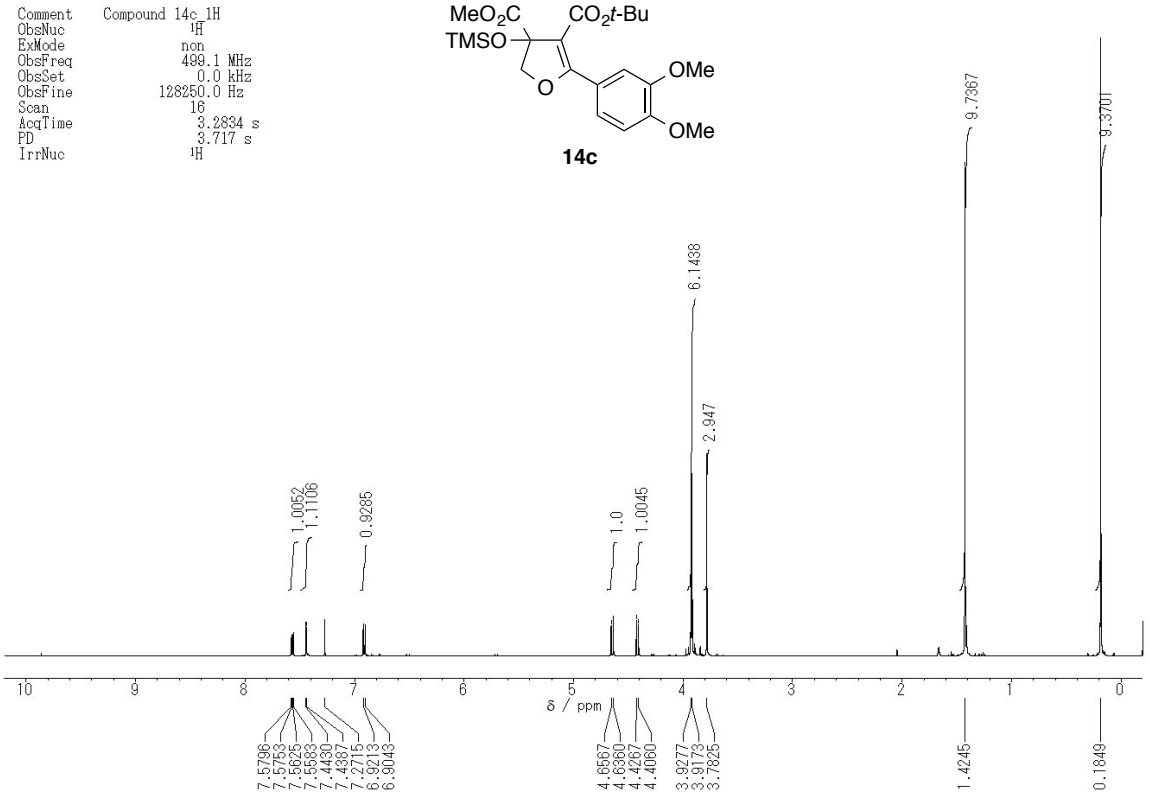
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MeO2C
CO2t-Bu
TMSO
O
OMe
OMe
14c
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1.0045
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7.2715
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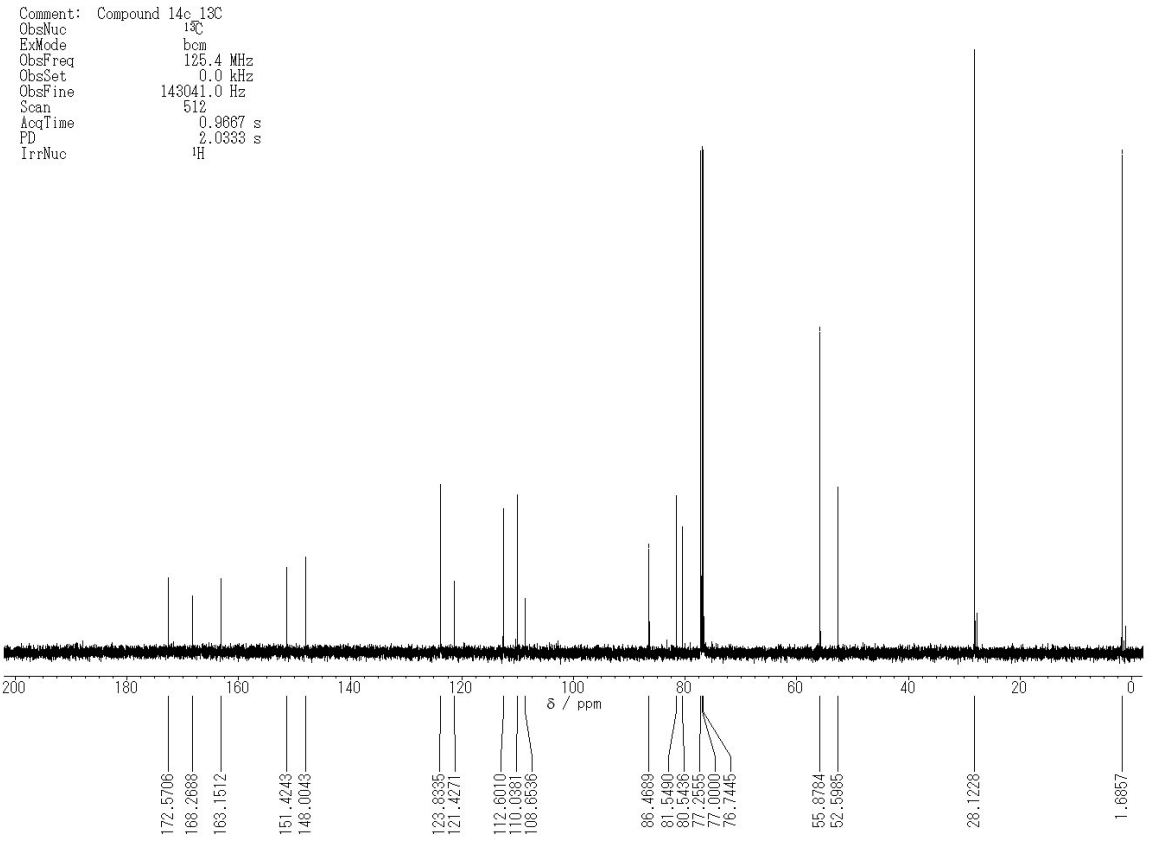
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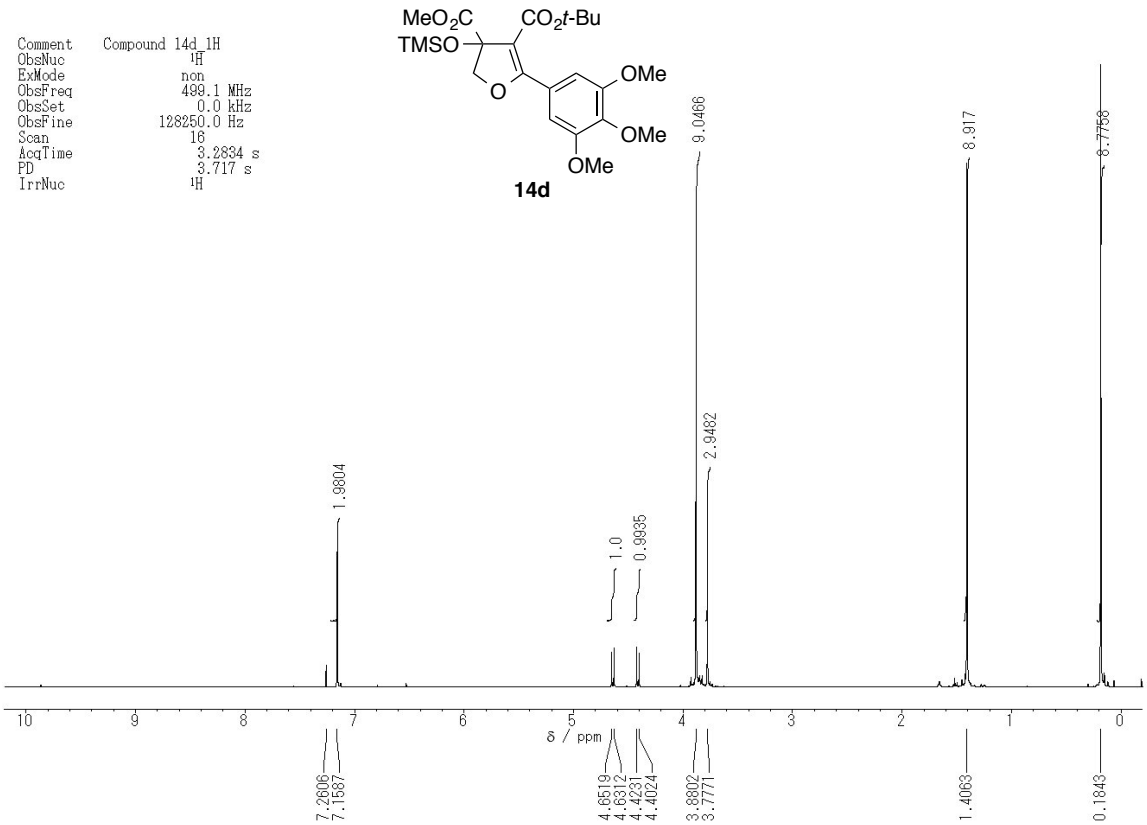
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MeO2C
CO2t-Bu
TMSO
O
OMe
OMe
OMe
14d
9.0466
8.917
8.7758
2.9482
1.9804
1.0
0.9935
δ / ppm
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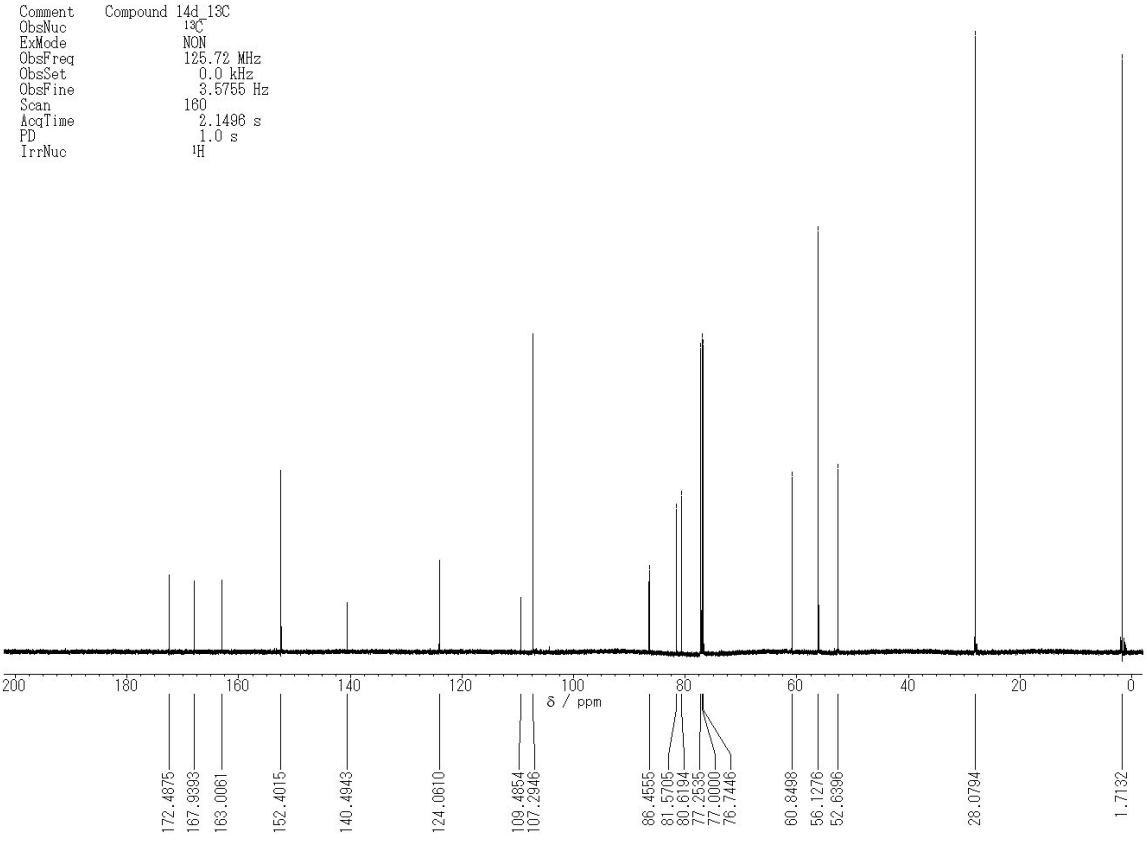
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δ / ppm
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163.0061
152.4015
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109.4854
107.2946
86.4555
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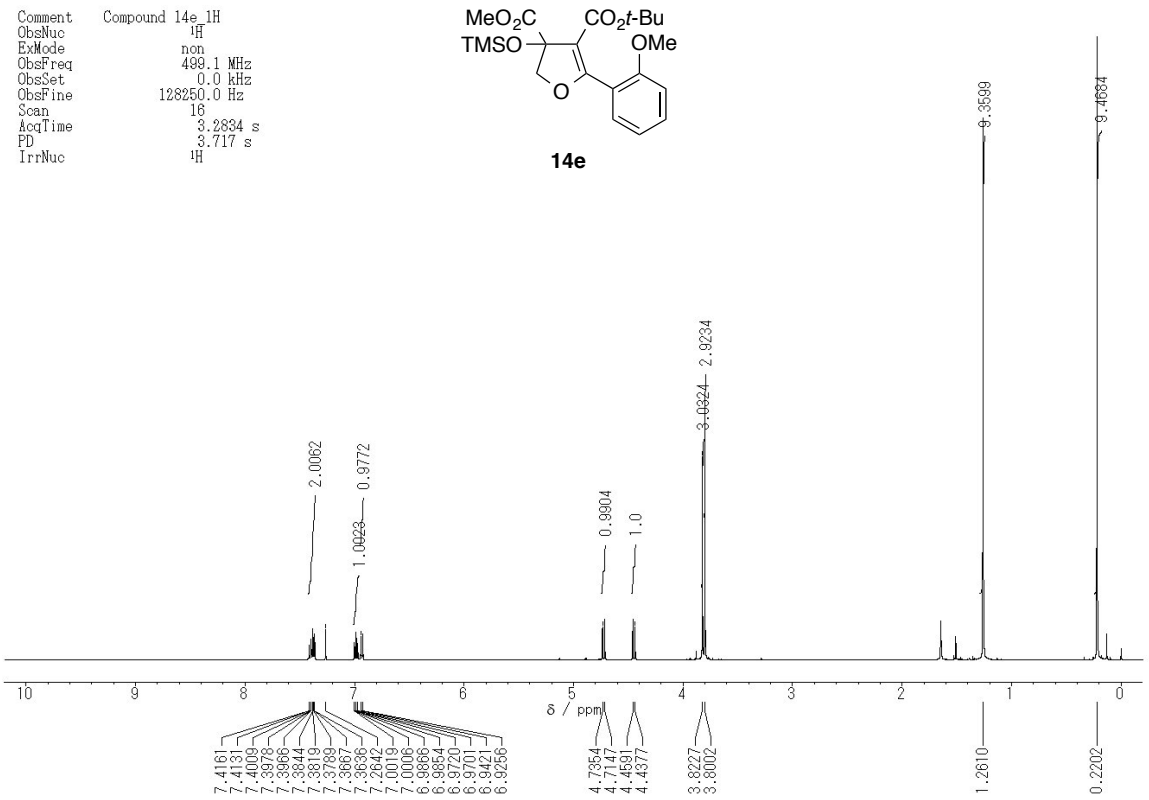
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MeO2C
CO2t-Bu
TMSO
OMe
O
14e
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0.9772
1.0023
0.9904
1.0
3.0324
2.9234
9.3599
9.4684
δ / ppm
7.4161
7.4131
7.4009
7.3978
7.3966
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7.3636
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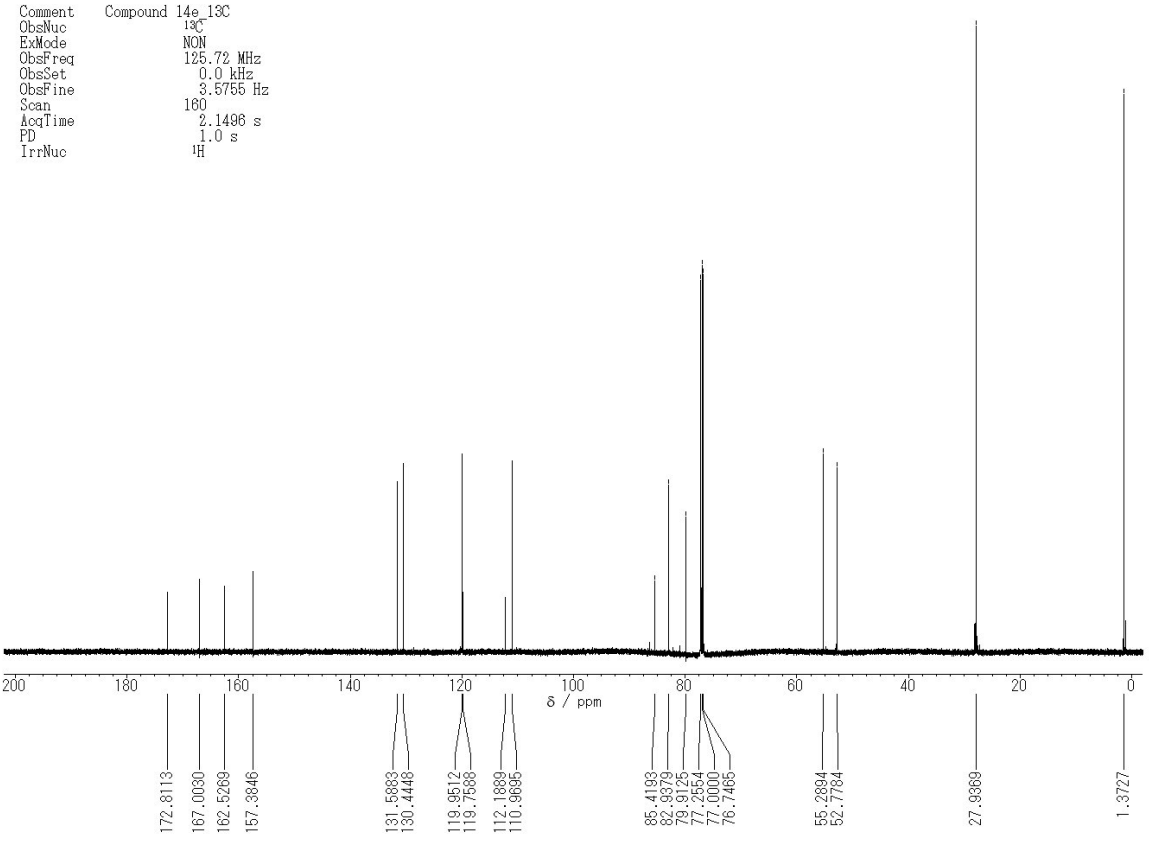
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δ / ppm
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167.0030
162.5269
157.3846
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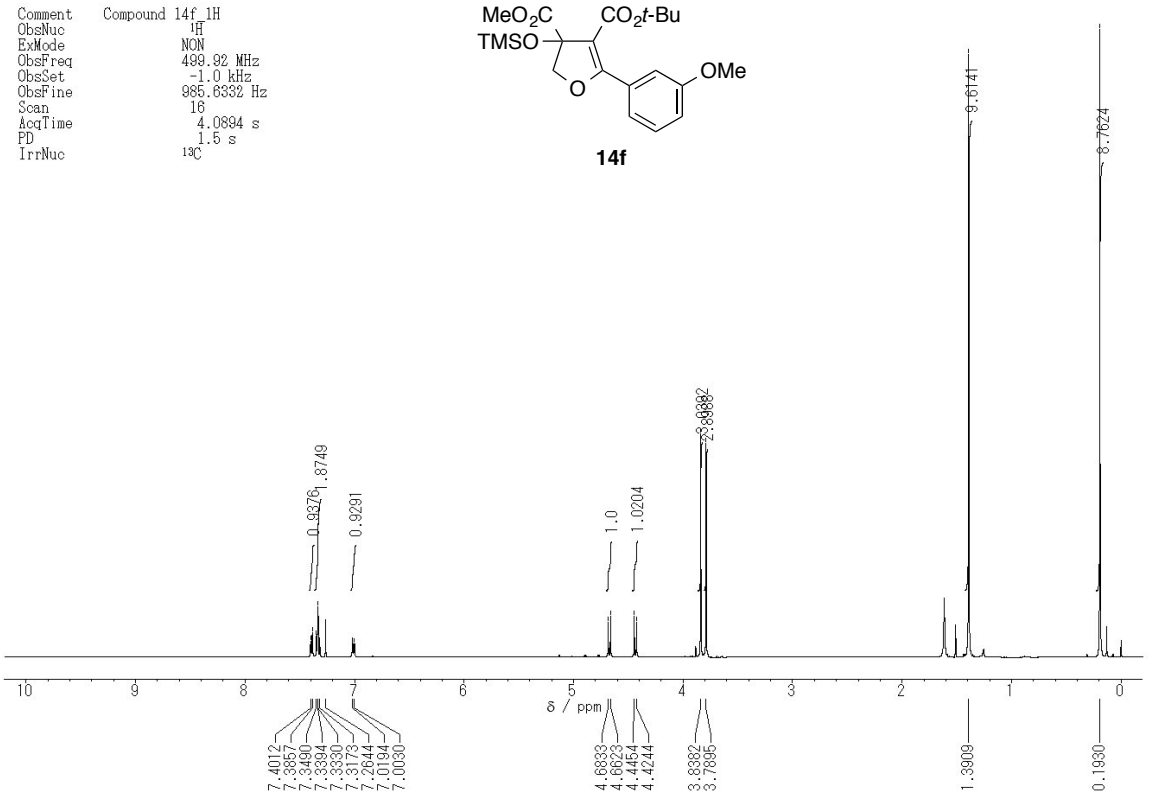
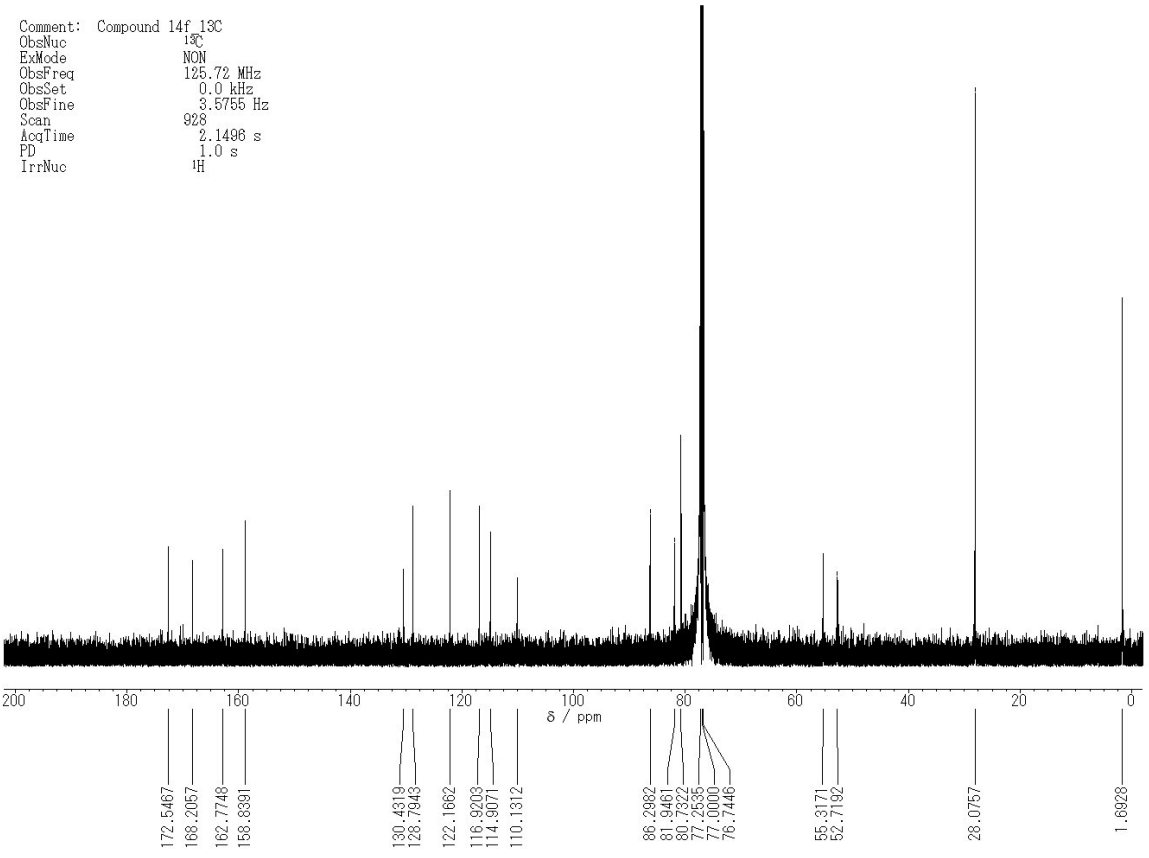
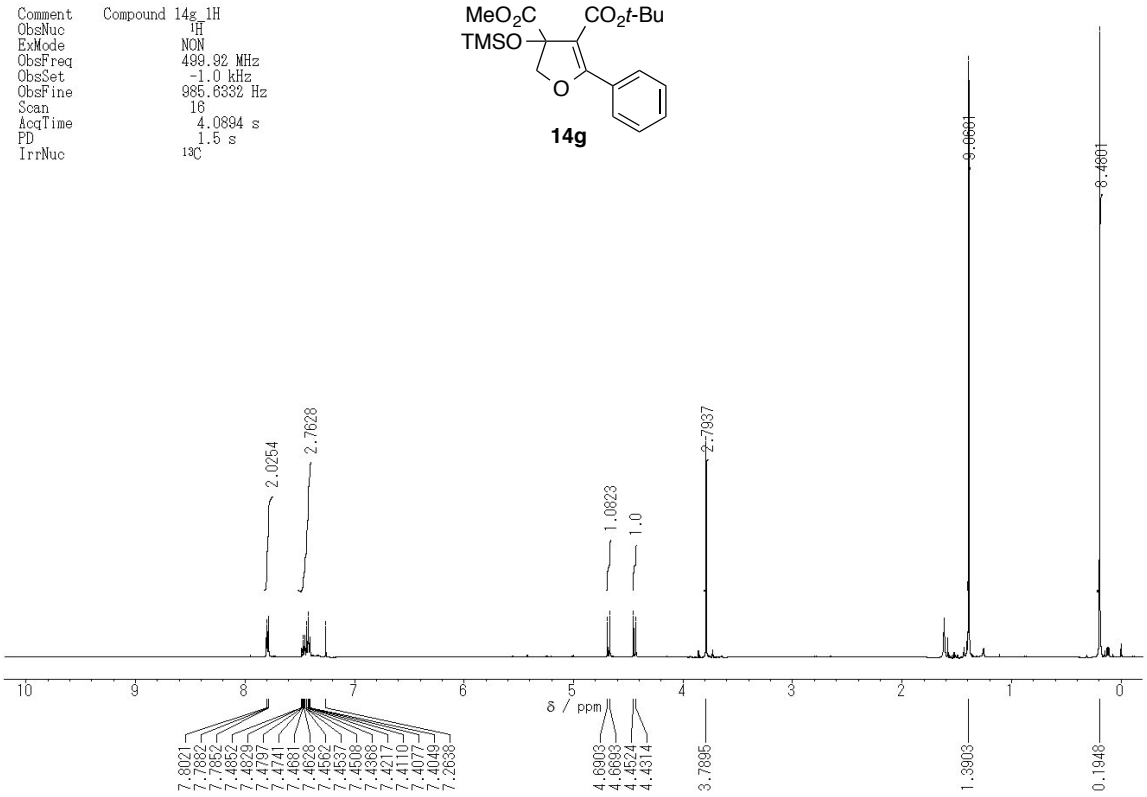
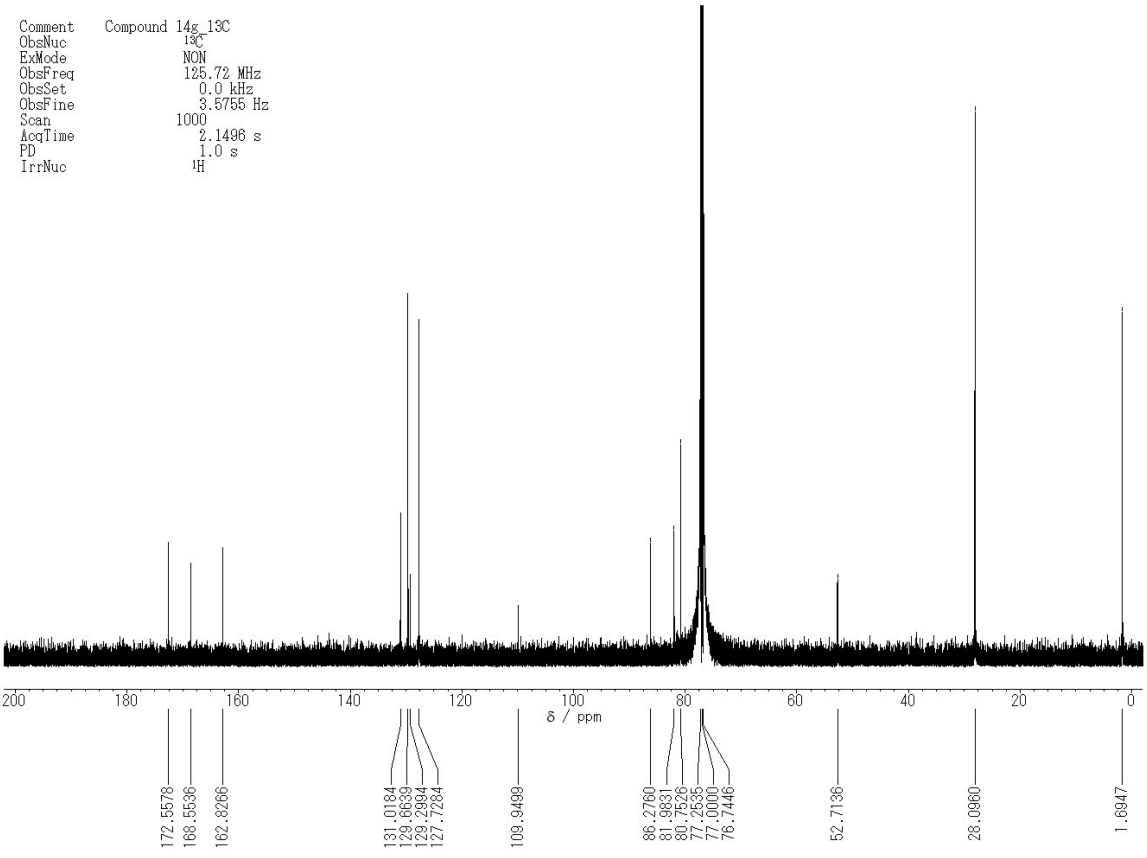
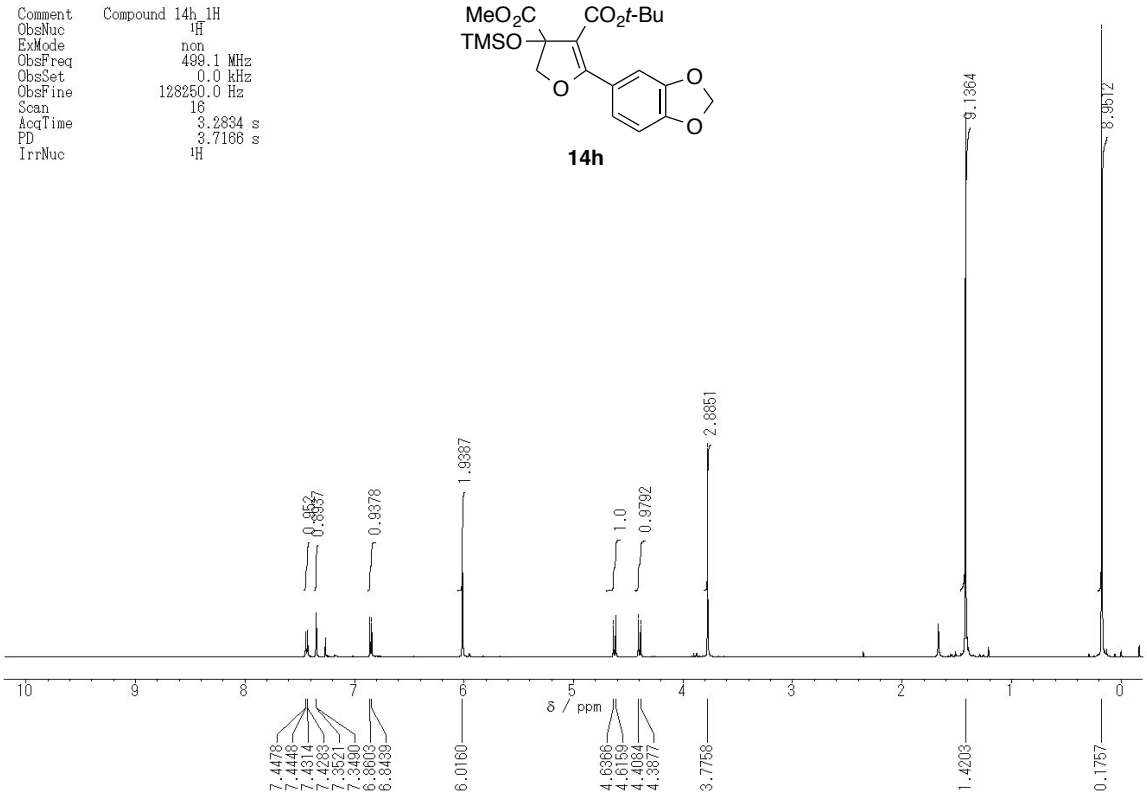
27.9369
1.3727

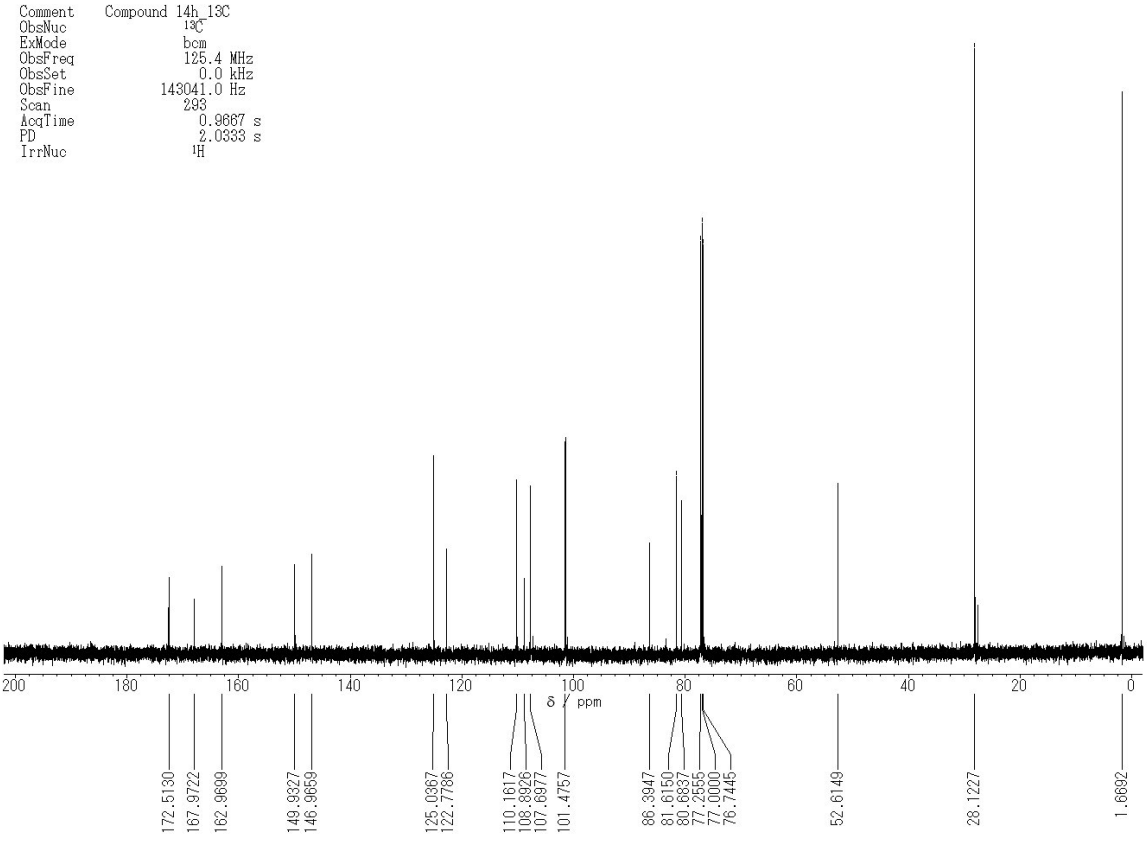

Comment Compound 14f_1H
ObsNuc 1H
ExMode NON
ObsFreq 499.92 MHz
ObsSet -1.0 kHz
ObsFine 985.6332 Hz
Scan 16
AcqTime 4.0894 s
PD 1.5 s
IrrNuc 13C
MeO2C
CO2t-Bu
TMSO
O
OMe
14f
0.9276
1.8749
0.9291
1.0
1.0204
9.6141
8.7624
δ / ppm
7.4012
7.3857
7.3490
7.3394
7.3330
7.3173
7.2644
7.0194
7.0030
4.6833
4.6623
4.4454
4.4244
3.8382
3.7895
1.3909
0.1930

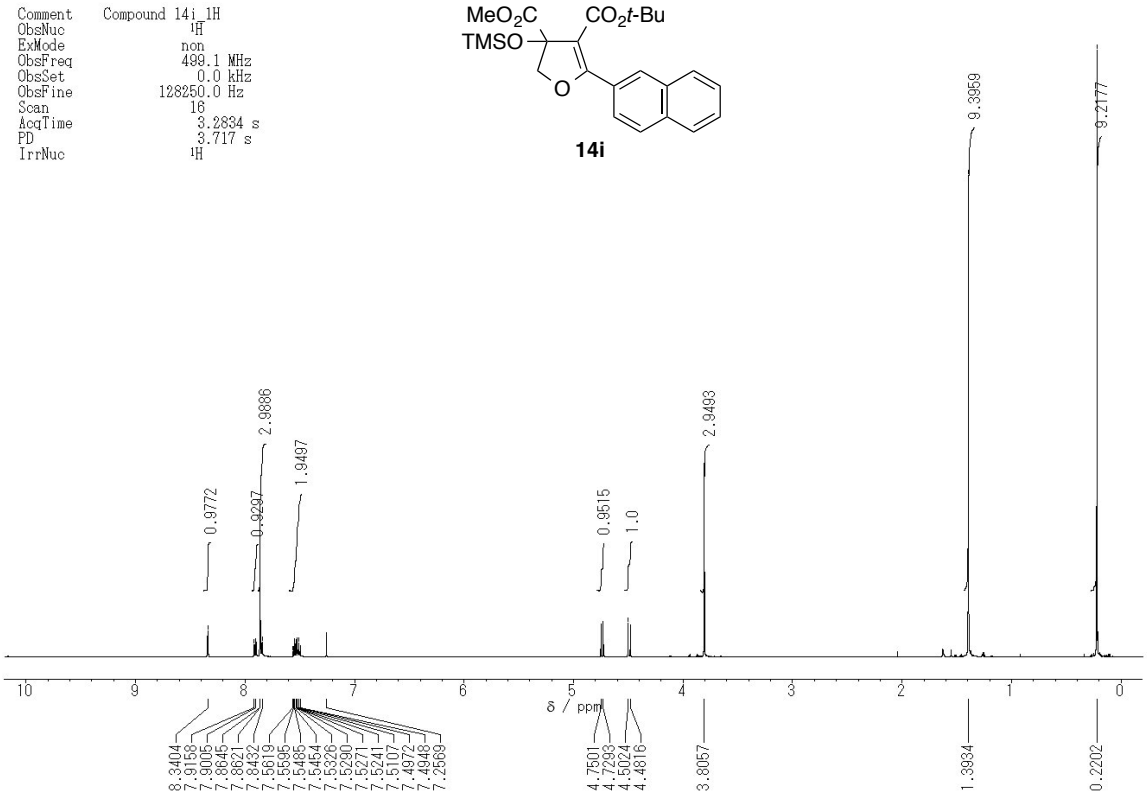

Comment: Compound 14f_13C
ObsNuc 13C
ExMode NON
ObsFreq 125.72 MHz
ObsSet 0.0 kHz
ObsFine 3.5755 Hz
Scan 928
AcqTime 2.1496 s
PD 1.0 s
IrrNuc 1H
δ / ppm
172.5467
168.2057
162.7748
158.8391
130.4319
128.7943
122.1662
116.9203
114.9071
110.1312
86.2982
81.9461
80.7322
77.2535
77.0000
76.7446
55.3171
52.7192
28.0757
1.6928

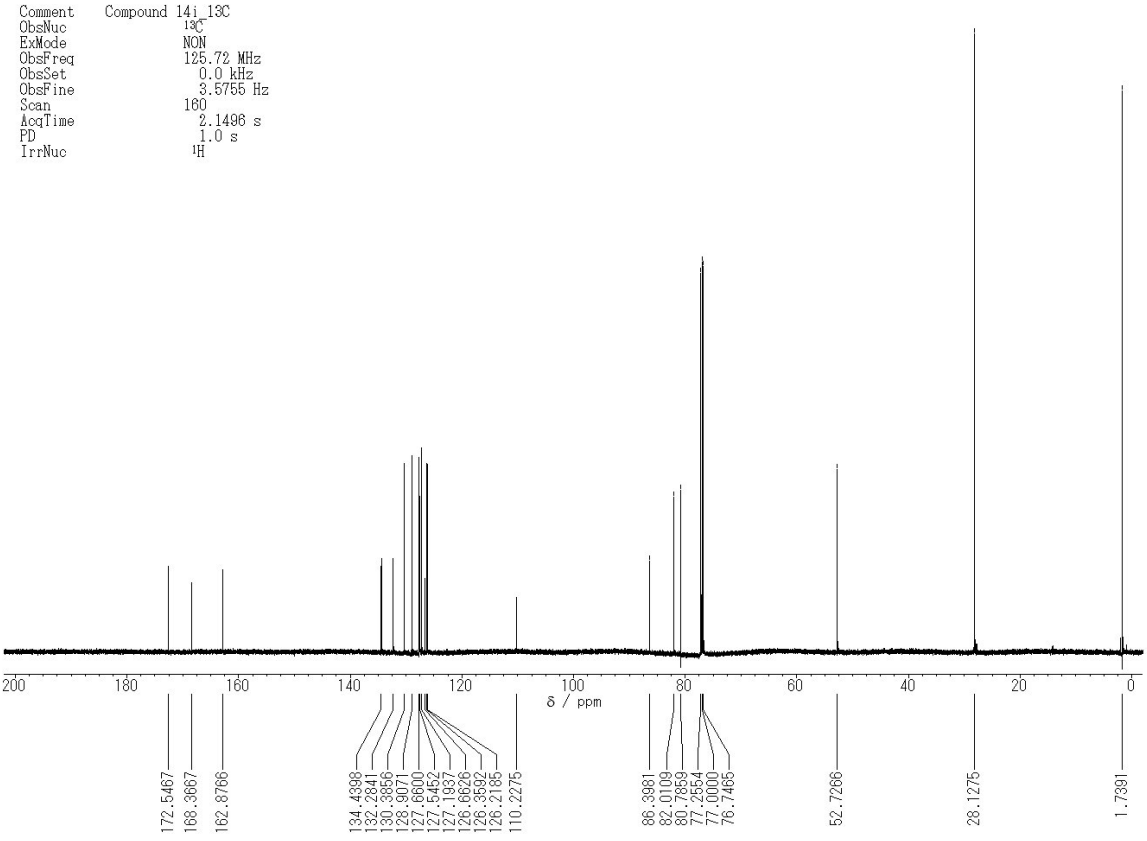

Comment Compound 14g_1H
ObsNuc 1H
ExMode NON
ObsFreq 499.92 MHz
ObsSet -1.0 kHz
ObsFine 985.6332 Hz
Scan 16
AcqTime 4.0894 s
PD 1.5 s
IrrNuc 13C
MeO2C
CO2t-Bu
TMSO
O
14g
2.0254
2.7628
1.0823
1.0
2.7937
9.0681
8.4801
δ / ppm
7.8021
7.7882
7.7862
7.4852
7.4829
7.4797
7.4741
7.4681
7.4628
7.4562
7.4537
7.4508
7.4368
7.4217
7.4110
7.4077
7.4049
7.2638
4.6903
4.6693
4.4524
4.4314
3.7895
1.3903
0.1948

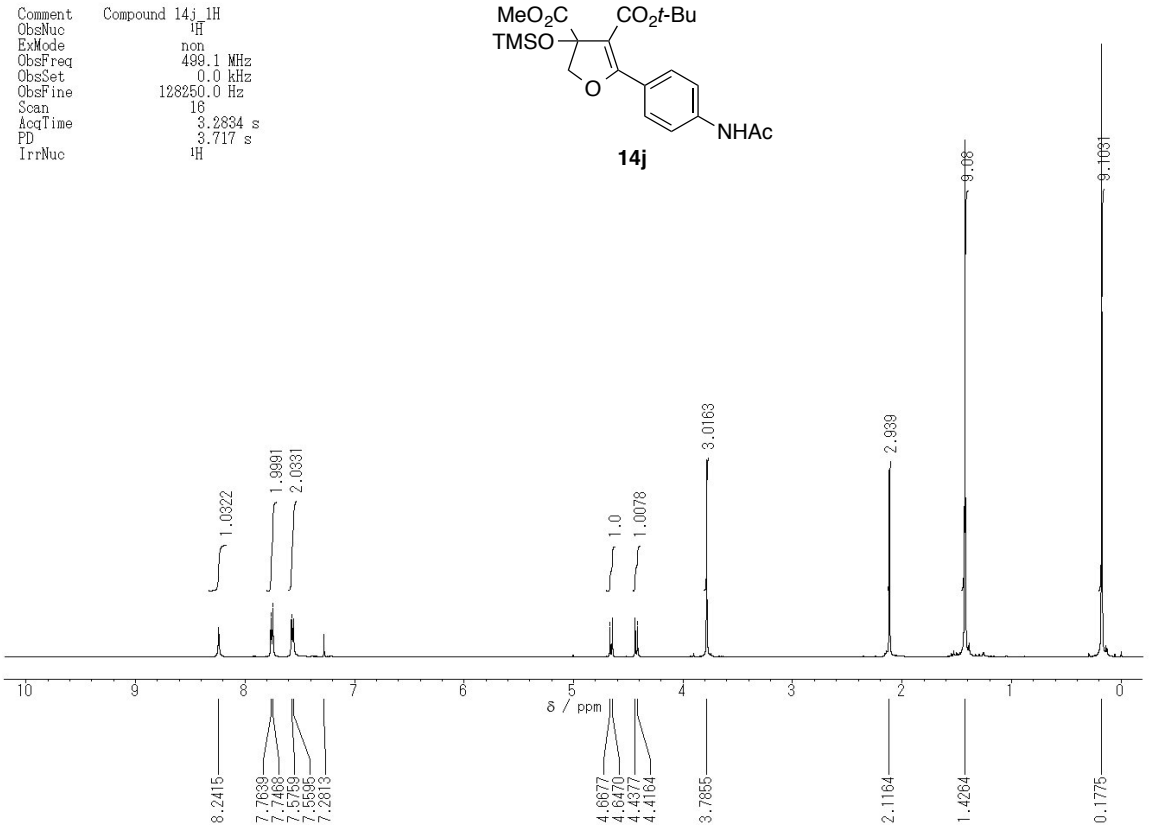

Comment Compound 14g_13C
ObsNuc 13C
ExMode NON
ObsFreq 125.72 MHz
ObsSet 0.0 kHz
ObsFine 3.5755 Hz
Scan 1000
AcqTime 2.1496 s
PD 1.0 s
IrrNuc 1H
δ / ppm
172.5578
168.5536
162.8266
131.0184
129.6639
129.2994
127.7284
109.9499
86.2760
81.9831
80.7526
77.2535
77.0000
76.7446
52.7136
28.0960
1.6947

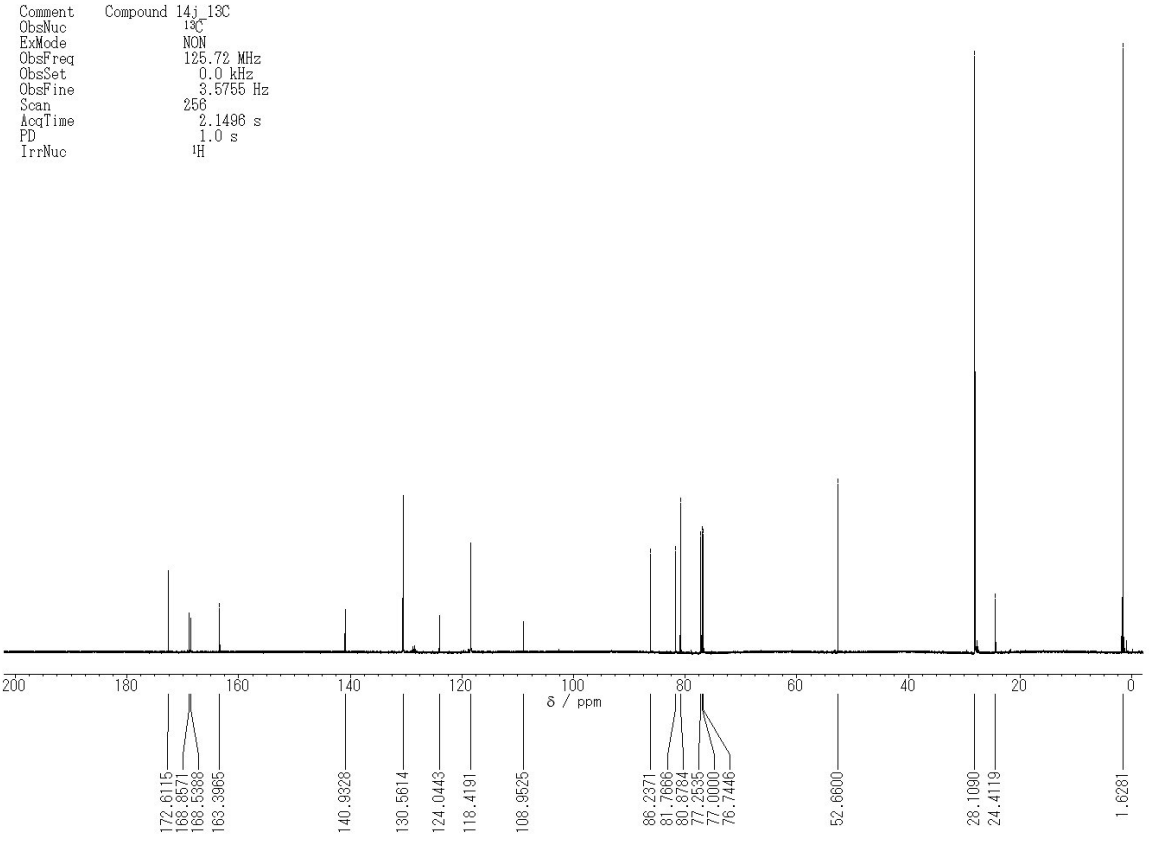

Comment Compound 14h_1H
ObsNuc 1H
ExMode non
ObsFreq 499.1 MHz
ObsSet 0.0 kHz
ObsFine 128250.0 Hz
Scan 16
AcqTime 3.2834 s
PD 3.7166 s
IrrNuc 1H
MeO2C
CO2t-Bu
TMSO
14h
9.1364
8.9512
2.8851
1.9387
0.9952
0.8937
0.9378
1.0
0.9792
δ / ppm
7.4478
7.4448
7.4314
7.4283
7.3521
7.3490
6.8603
6.8439
6.0160
4.6366
4.6159
4.4084
4.3877
3.7758
1.4203
0.1757

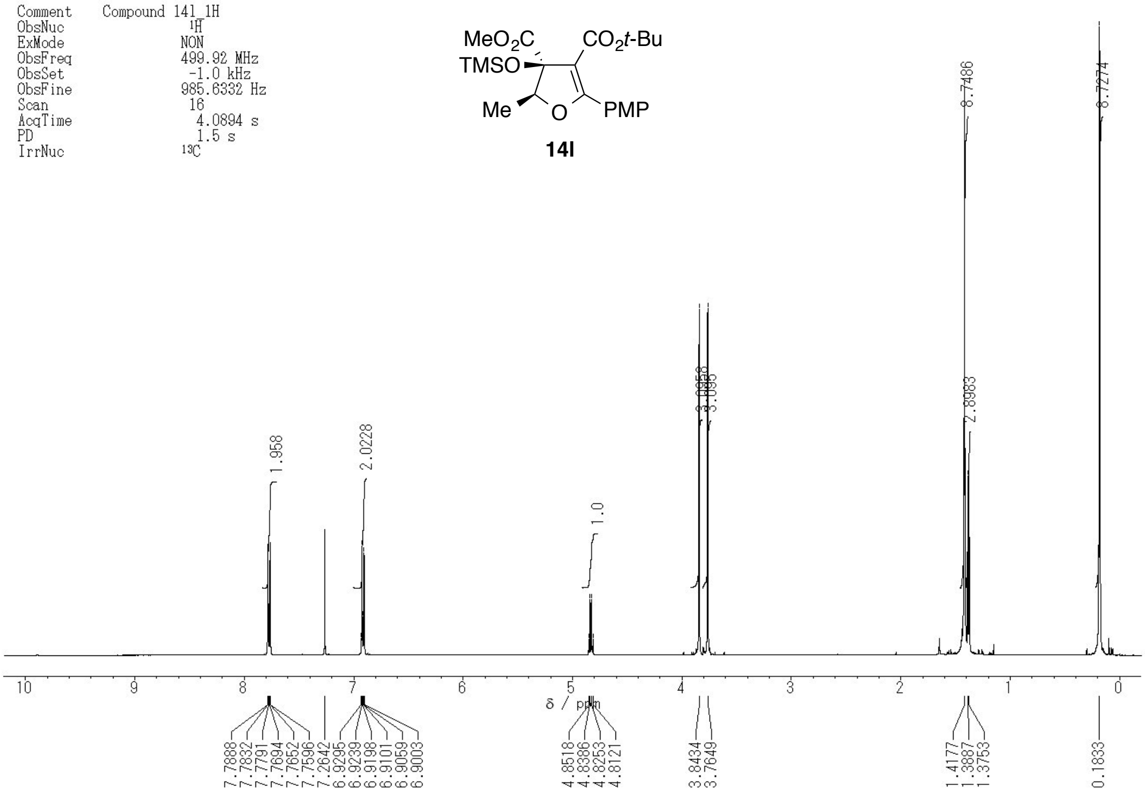

Comment Compound 14h_13C
ObsNuc 13C
ExMode bcm
ObsFreq 125.4 MHz
ObsSet 0.0 kHz
ObsFine 143041.0 Hz
Scan 293
AcqTime 0.9667 s
PD 2.0333 s
IrrNuc 1H
δ / ppm
172.5130
167.9722
162.9699
149.9327
146.9659
125.0367
122.7786
110.1617
108.8926
107.6977
101.4757
86.3947
81.6150
80.6837
77.2555
77.0000
76.7445
52.6149
28.1227
1.6692


Comment Compound 14i_1H
ObsNuc 1H
ExMode non
ObsFreq 499.1 MHz
ObsSet 0.0 kHz
ObsFine 128250.0 Hz
Scan 16
AcqTime 3.2834 s
PD 3.717 s
IrrNuc 1H
MeO2C
CO2t-Bu
TMSO
O
14i
0.9772
0.9297
2.9886
1.9497
0.9515
1.0
2.9493
9.3959
9.2177
δ / ppm
8.3404
7.9158
7.9005
7.8645
7.8621
7.8432
7.5619
7.5595
7.5465
7.5454
7.5326
7.5290
7.5271
7.5241
7.5107
7.4972
7.4948
7.2569
4.7501
4.7293
4.5024
4.4816
3.8057
1.3934
0.2202

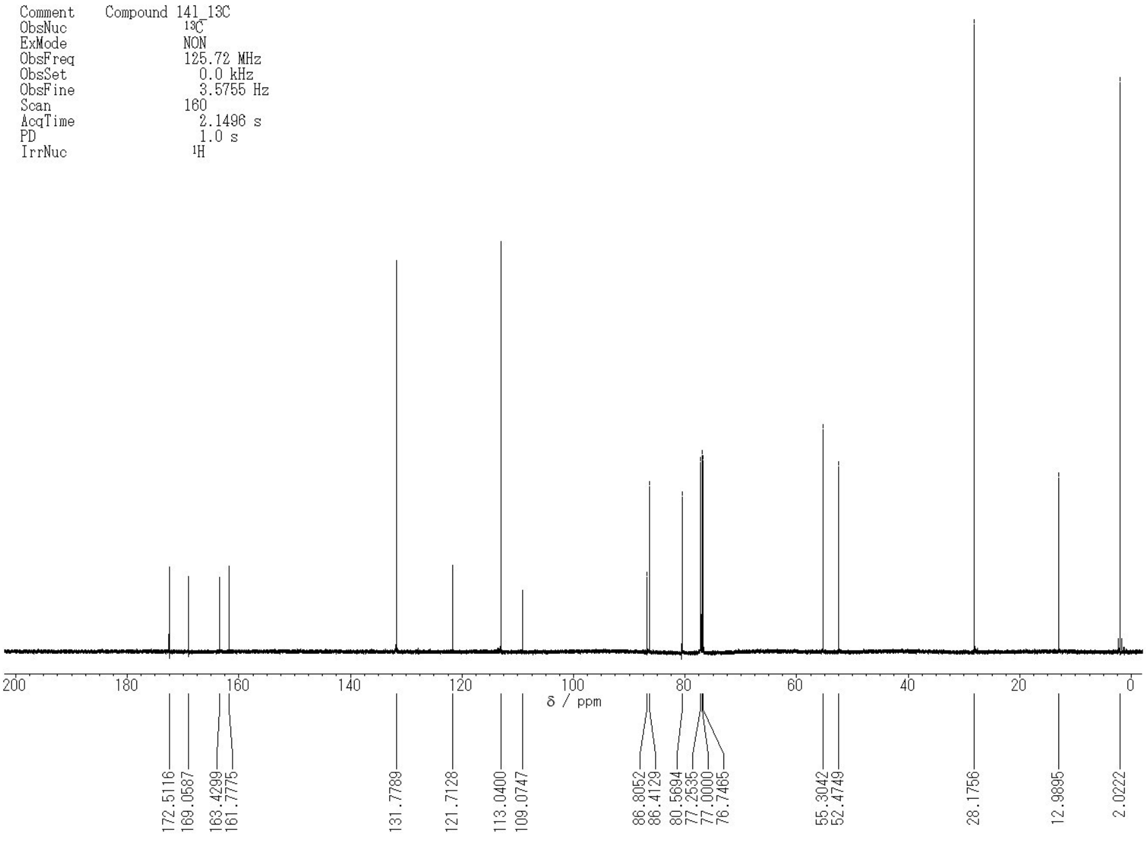

Comment Compound 14i_13C
ObsNuc 13C
ExMode NON
ObsFreq 125.72 MHz
ObsSet 0.0 kHz
ObsFine 3.5755 Hz
Scan 160
AcqTime 2.1496 s
PD 1.0 s
IrrNuc 1H
δ / ppm
172.5467
168.3667
162.8766
134.4398
132.2841
130.3866
128.9071
127.6600
127.5452
127.1937
126.6626
126.3692
126.2185
110.2275
86.3981
82.0109
80.7859
77.2554
77.0000
76.7465
52.7266
28.1275
1.7391

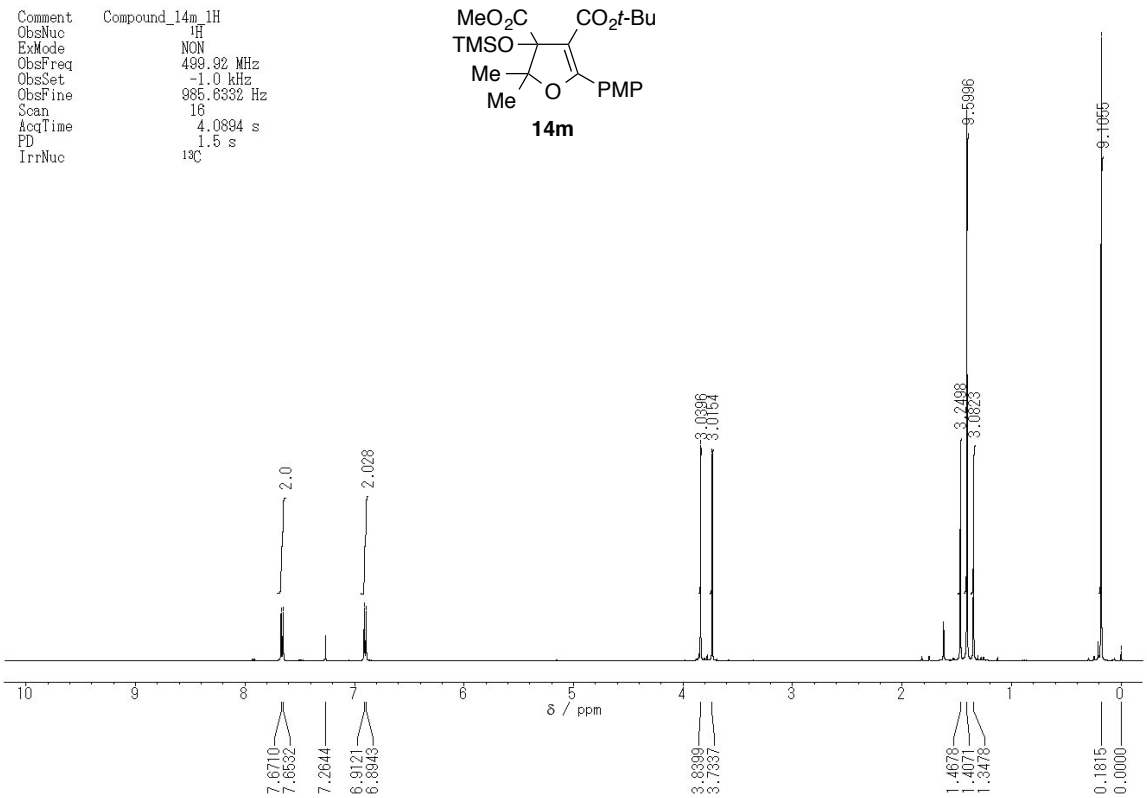

Comment Compound 14j_1H
ObsNuc 1H
ExMode non
ObsFreq 499.1 MHz
ObsSet 0.0 kHz
ObsFine 128250.0 Hz
Scan 16
AcqTime 3.2834 s
PD 3.717 s
IrrNuc 1H
MeO2C
CO2t-Bu
TMSO
O
NHAc
14j
1.0322
1.9991
2.0331
1.0
1.0078
3.0163
2.939
9.08
9.1031
δ / ppm
8.2415
7.7639
7.7468
7.5759
7.5595
7.2813
4.6677
4.6470
4.4377
4.4164
3.7855
2.1164
1.4264
0.1775

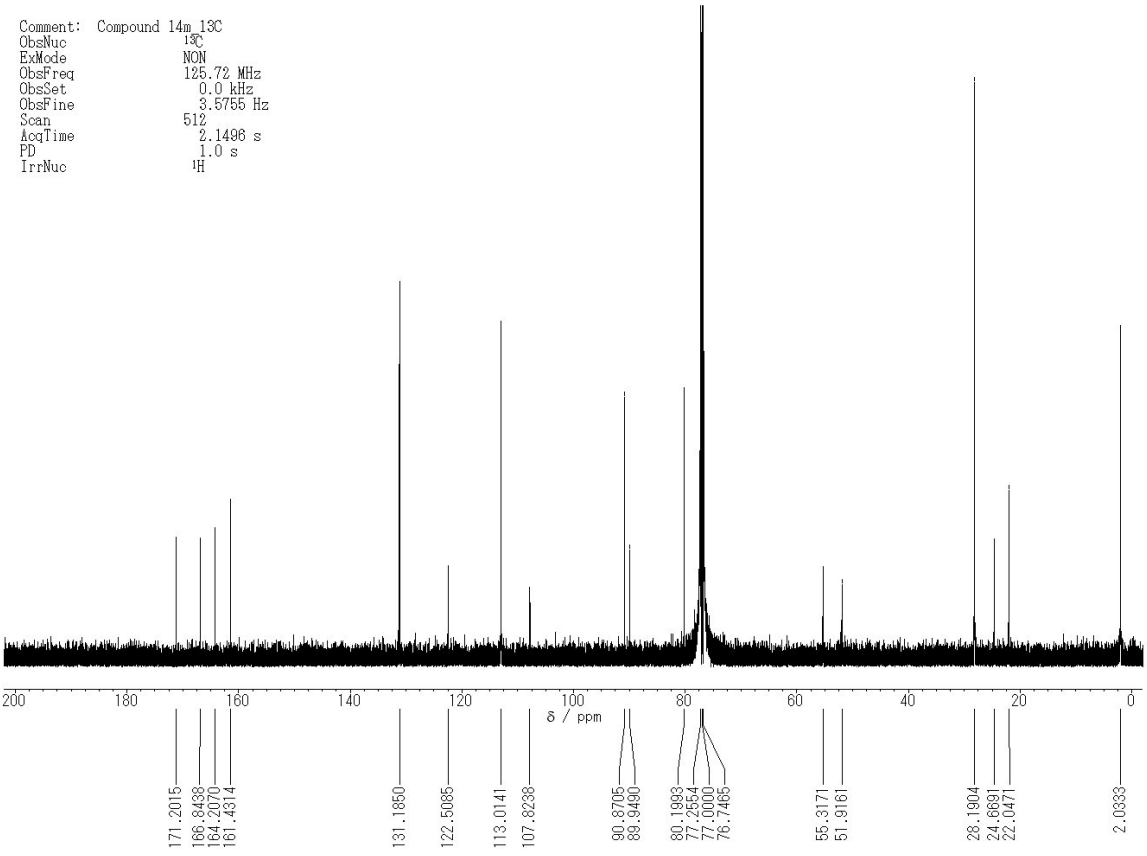

Comment Compound 14j_13C
ObsNuc 13C
ExMode NON
ObsFreq 125.72 MHz
ObsSet 0.0 kHz
ObsFine 3.5755 Hz
Scan 256
AcqTime 2.1496 s
PD 1.0 s
IrrNuc 1H
δ / ppm
172.6115
168.8571
168.5388
163.3965
140.9328
130.5614
124.0443
118.4191
108.9525
86.2371
81.7666
80.8784
77.2535
77.0000
76.7446
52.6600
28.1090
24.4119
1.6281

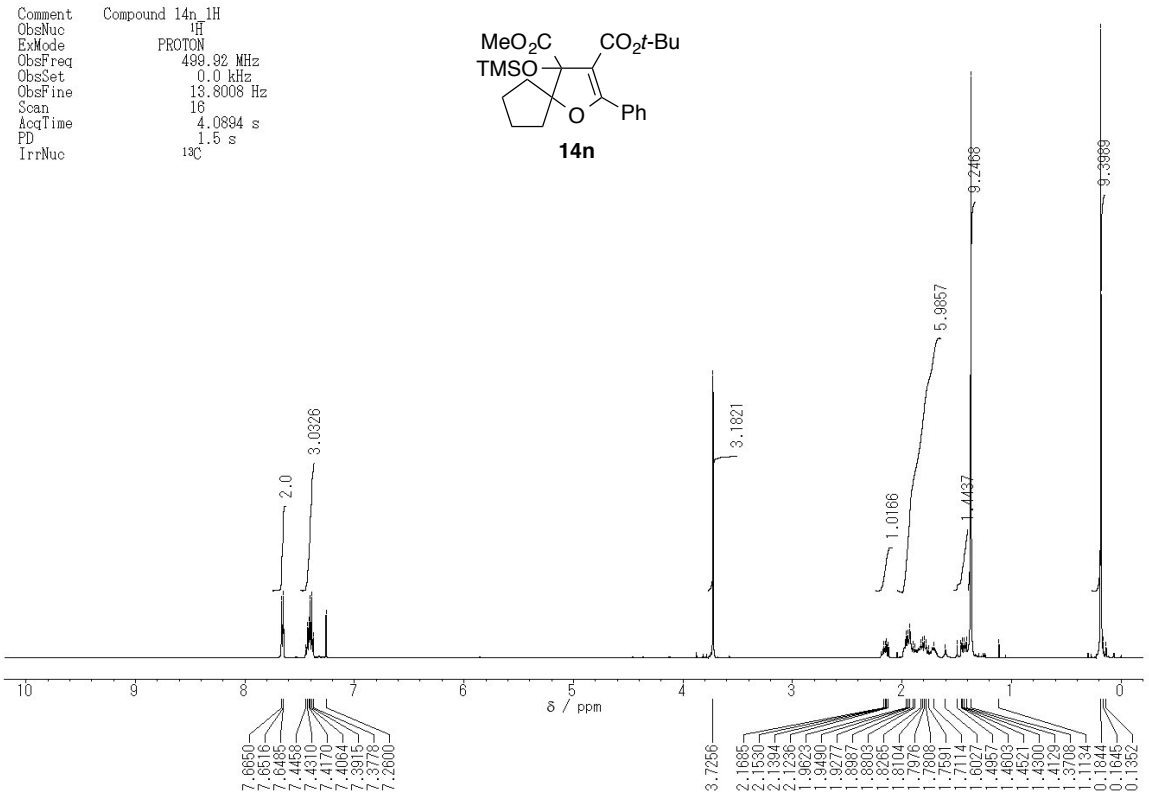

Comment Compound 14l_1H
ObsNuc 1H
ExMode NON
ObsFreq 499.92 MHz
ObsSet -1.0 kHz
ObsFine 985.6332 Hz
Scan 16
AcqTime 4.0894 s
PD 1.5 s
IrrNuc 13C
MeO2C
CO2t-Bu
TMSO
Me
O
PMP
1.958
2.0228
1.0
3.0058
3.095
2.8983
8.7486
8.7274
δ / ppm
7.7888
7.7832
7.7791
7.7694
7.7652
7.7596
7.2642
6.9295
6.9239
6.9198
6.9101
6.9059
6.9003
4.8518
4.8386
4.8253
4.8121
3.8434
3.7649
1.4177
1.3887
1.3753
0.1833

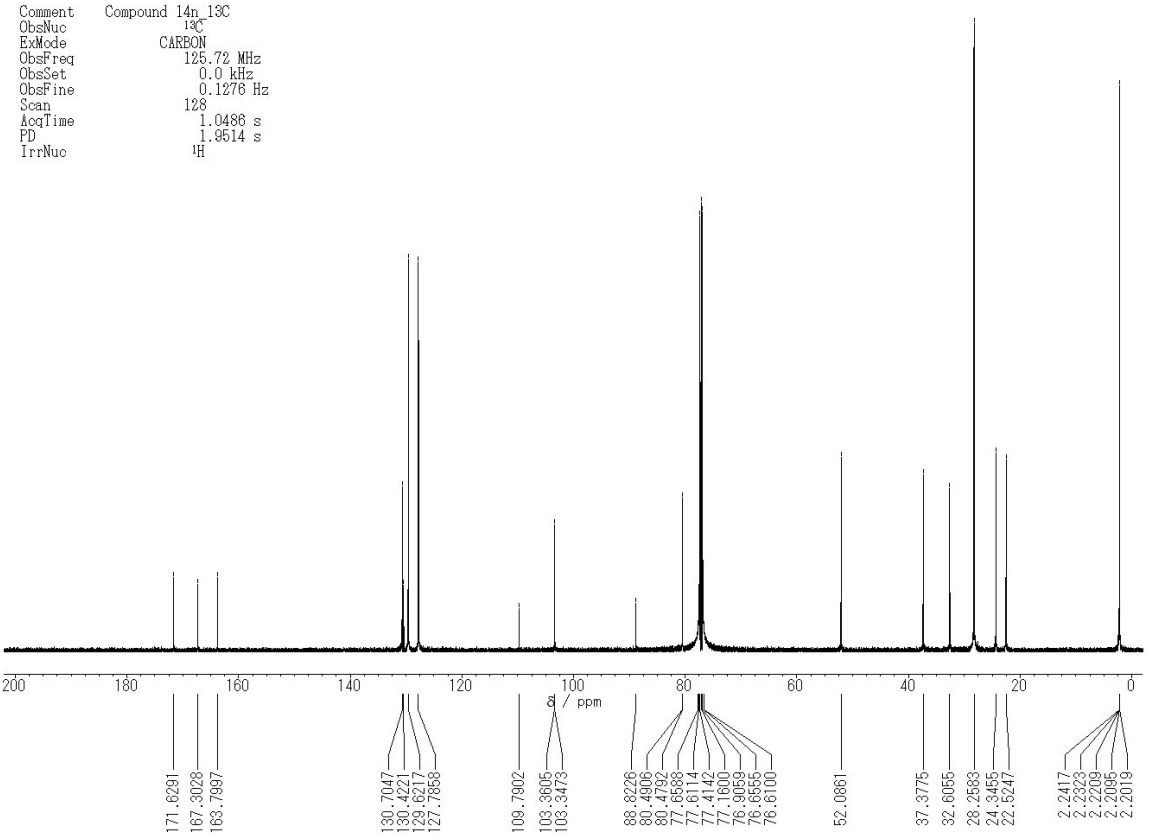
14l

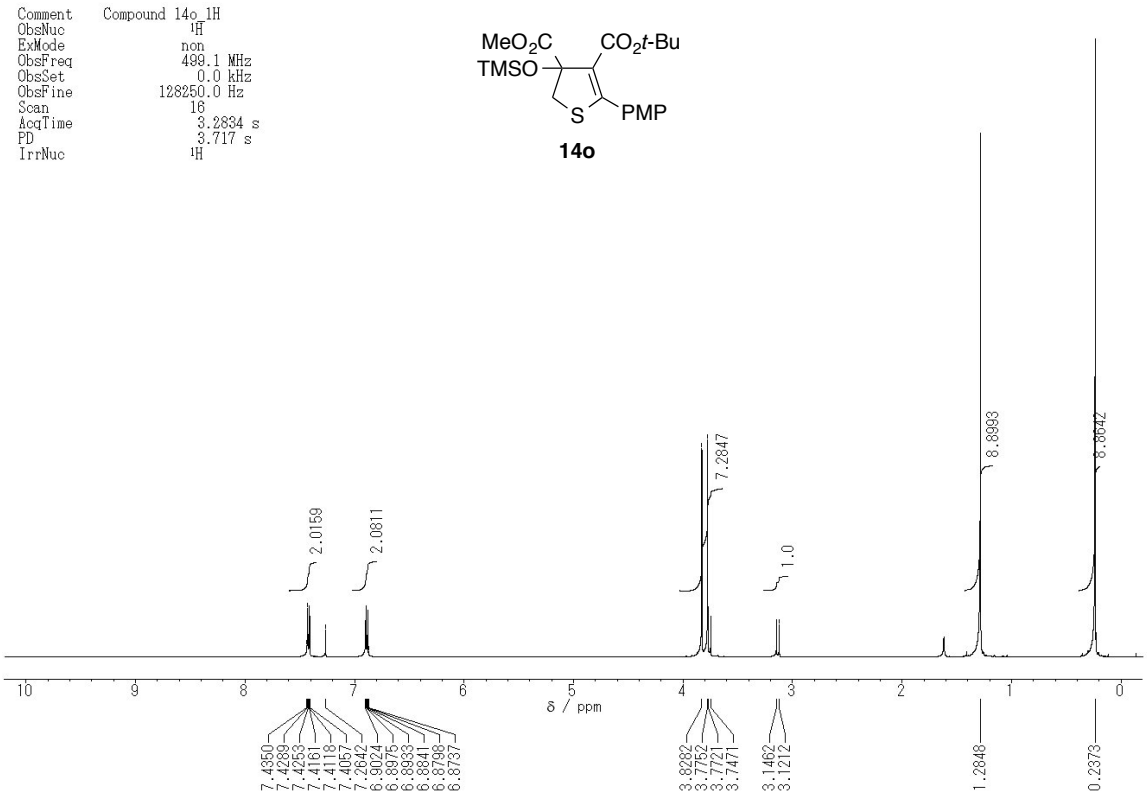

Comment Compound 14l_13C
ObsNuc 13C
ExMode NON
ObsFreq 125.72 MHz
ObsSet 0.0 kHz
ObsFine 3.5755 Hz
Scan 160
AcqTime 2.1496 s
PD 1.0 s
IrrNuc 1H
δ / ppm
172.5116
169.0587
163.4299
161.7775
131.7789
121.7128
113.0400
109.0747
86.8052
86.4129
80.5694
77.2535
77.0000
76.7465
55.3042
52.4749
28.1756
12.9895
2.0222

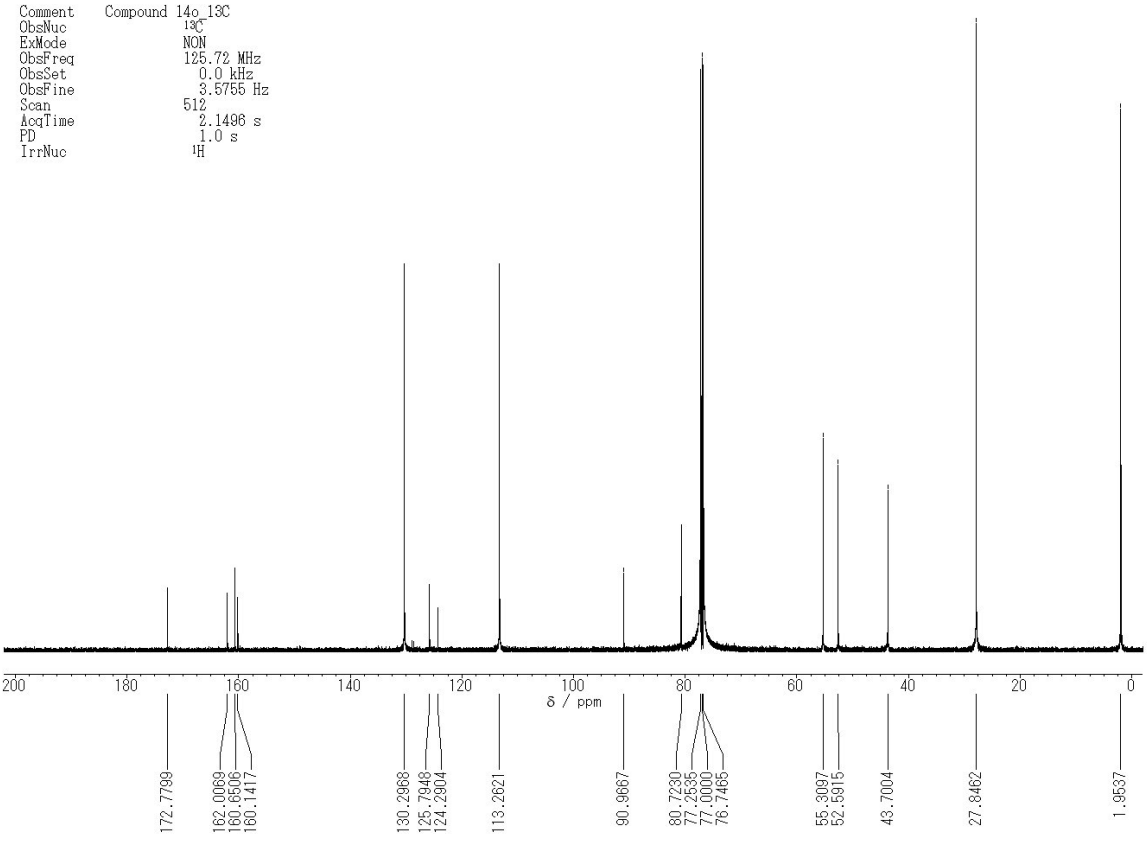

Comment Compound_14m_1H
ObsNuc 1H
ExMode NON
ObsFreq 499.92 MHz
ObsSet -1.0 kHz
ObsFine 985.6332 Hz
Scan 16
AcqTime 4.0894 s
PD 1.5 s
IrrNuc 13C
MeO2C
CO2t-Bu
TMSO
Me
Me
O
PMP
14m
9.5996
9.1055
3.2498
3.1023
3.0396
3.0154
2.028
2.0
δ / ppm
7.6710
7.6532
7.2644
6.9121
6.8943
3.8399
3.7337
1.4678
1.4071
1.3478
0.1815
0.0000

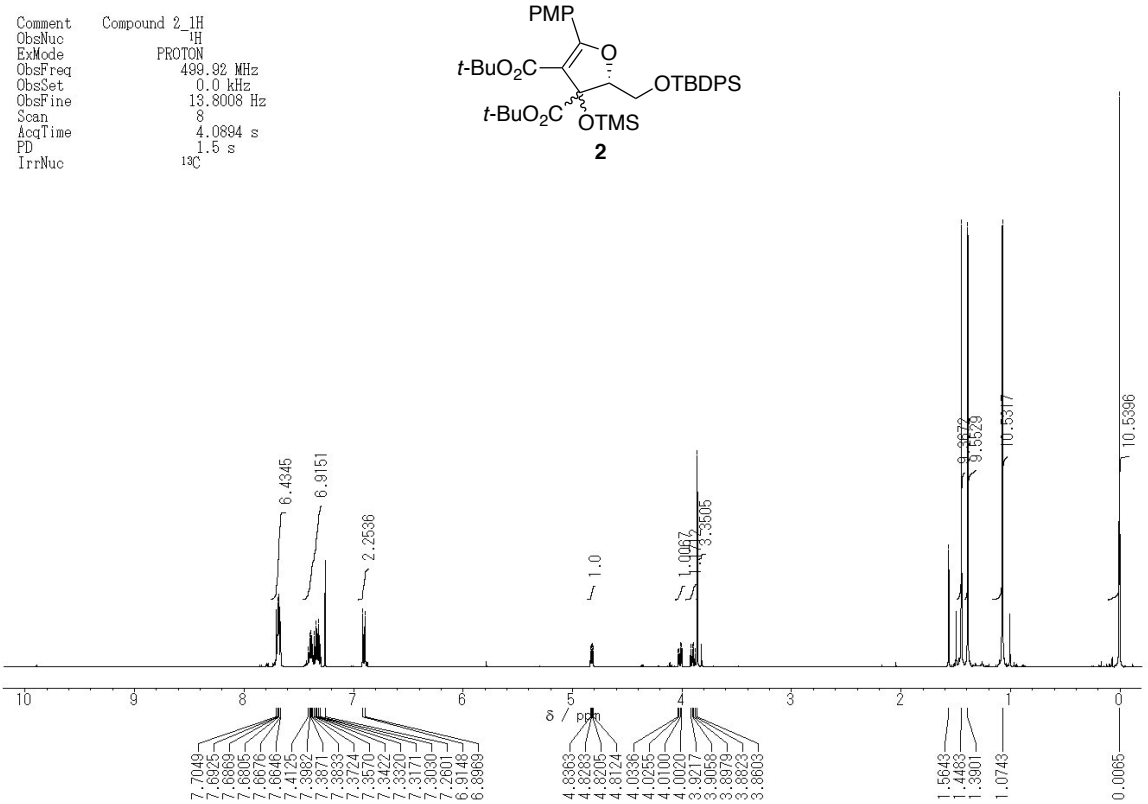

Comment: Compound 14m 13C
ObsNuc 13C
ExMode NON
ObsFreq 125.72 MHz
ObsSet 0.0 kHz
ObsFine 3.5755 Hz
Scan 512
AcqTime 2.1496 s
PD 1.0 s
IrrNuc 1H
δ / ppm
171.2015
166.8438
164.2070
161.4314
131.1850
122.5085
113.0141
107.8238
90.8705
89.9490
80.1993
77.2554
77.0000
76.7465
55.3171
51.9161
28.1904
24.6691
22.0471
2.0333

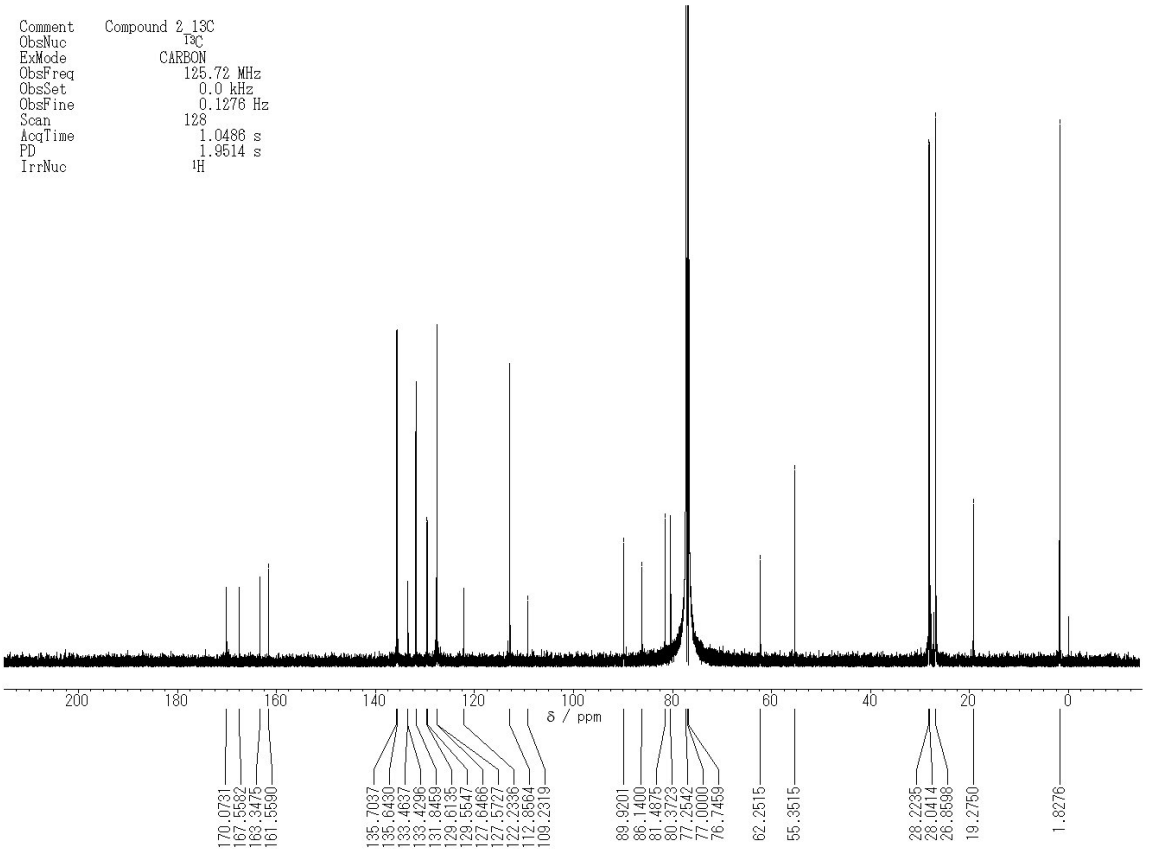

Comment Compound 14n_1H
ObsNuc 1H
ExMode PROTON
ObsFreq 499.92 MHz
ObsSet 0.0 kHz
ObsFine 13.8008 Hz
Scan 16
AcqTime 4.0894 s
PD 1.5 s
IrrNuc 13C
MeO2C
CO2t-Bu
TMSO
O
Ph
14n
2.0
3.0326
3.1821
1.0166
5.9857
1.4437
9.2468
9.3969
δ / ppm
7.6650
7.6516
7.6485
7.4458
7.4310
7.4170
7.4064
7.3915
7.3778
7.2600
3.7256
2.1685
2.1530
2.1394
2.1236
1.9623
1.9490
1.9277
1.8987
1.8803
1.8265
1.8104
1.7976
1.7808
1.7591
1.7114
1.6027
1.4957
1.4603
1.4521
1.4300
1.4129
1.3708
1.1134
0.1844
0.1645
0.1352

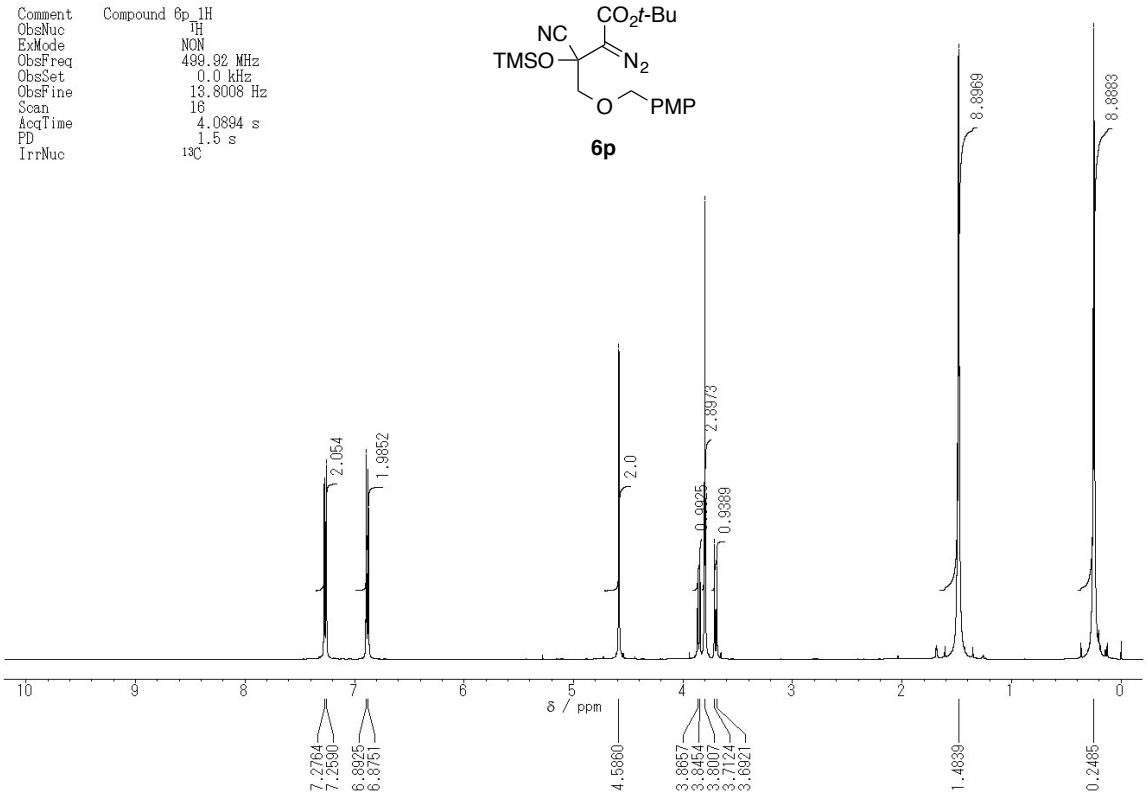

Comment Compound 14n_13C
ObsNuc 13C
ExMode CARBON
ObsFreq 125.72 MHz
ObsSet 0.0 kHz
ObsFine 0.1276 Hz
Scan 128
AcqTime 1.0486 s
PD 1.9514 s
IrrNuc 1H
δ / ppm
171.6291
167.3028
163.7997
130.7047
130.4221
129.6217
127.7858
109.7902
103.3605
103.3473
88.8226
80.4906
80.4792
77.6688
77.6114
77.4142
77.1600
76.9059
76.6555
76.6100
52.0861
37.3775
32.6055
28.2583
24.3455
22.5247
2.2417
2.2323
2.2209
2.2096
2.2019

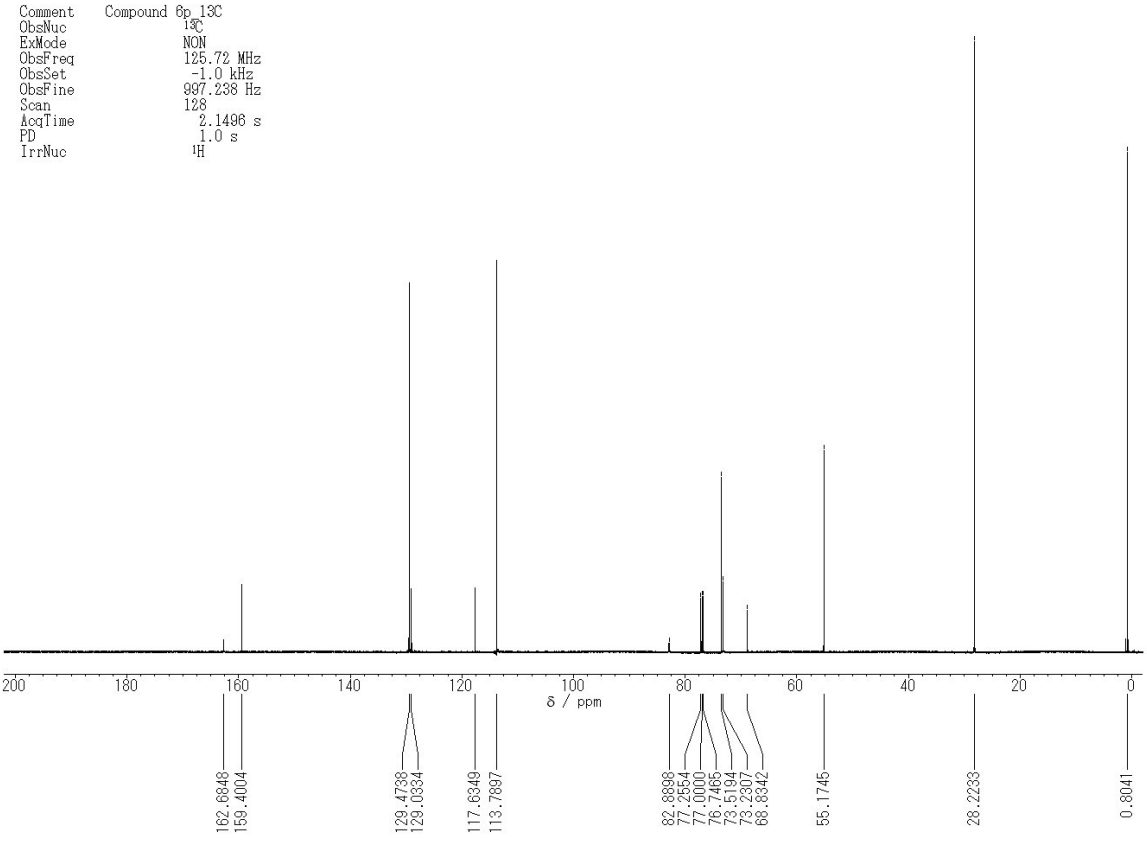

Comment Compound 14o_1H
ObsNuc 1H
ExMode non
ObsFreq 499.1 MHz
ObsSet 0.0 kHz
ObsFine 128250.0 Hz
Scan 16
AcqTime 3.2834 s
PD 3.717 s
IrrNuc 1H
MeO2C
CO2t-Bu
TMSO
S
PMP
14o
2.0159
2.0811
7.2847
1.0
8.8993
8.8642
δ / ppm
7.4350
7.4289
7.4253
7.4161
7.4118
7.4057
7.2642
6.9024
6.8975
6.8933
6.8841
6.8798
6.8737
3.8282
3.7752
3.7721
3.7471
3.1462
3.1212
1.2848
0.2373

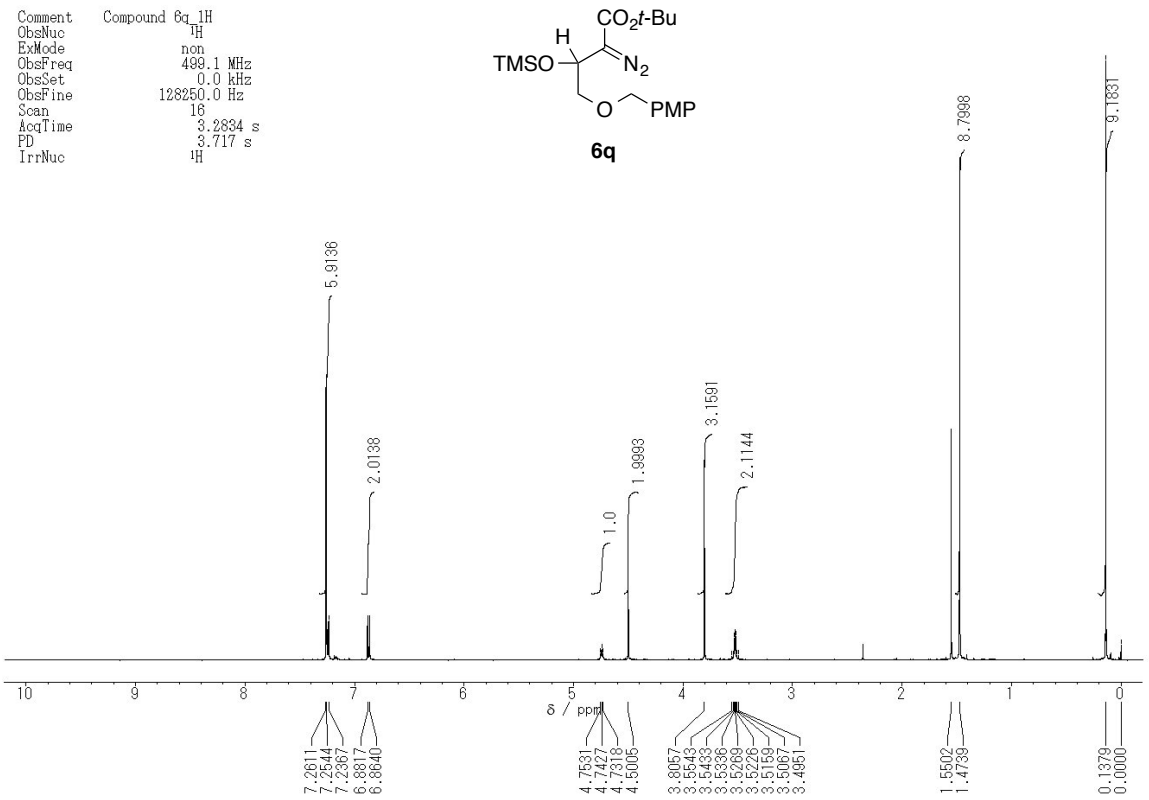

Comment Compound 14o_13C
ObsNuc 13C
ExMode NON
ObsFreq 125.72 MHz
ObsSet 0.0 kHz
ObsFine 3.5755 Hz
Scan 512
AcqTime 2.1496 s
PD 1.0 s
IrrNuc 1H
δ / ppm
172.7799
162.0069
160.6506
160.1417
130.2968
125.7948
124.2904
113.2621
90.9667
80.7230
77.2535
77.0000
76.7465
55.3097
52.5915
43.7004
27.8462
1.9537

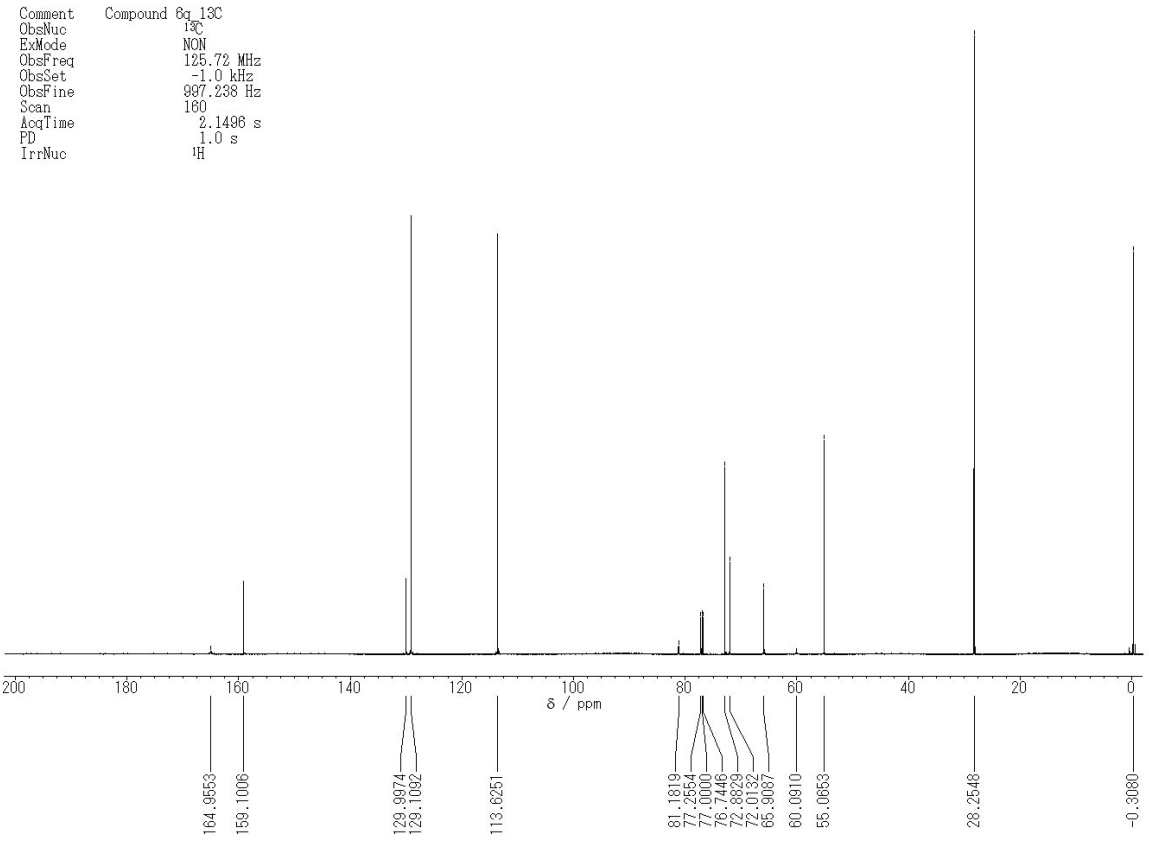

Comment Compound 2_1H
ObsNuc 1H
ExMode PROTON
ObsFreq 499.92 MHz
ObsSet 0.0 kHz
ObsFine 13.8008 Hz
Scan 8
AcqTime 4.0894 s
PD 1.5 s
IrrNuc 13C
PMP
t-BuO2C
O
OTBDPS
t-BuO2C
OTMS
2
6.4345
6.9151
2.2536
1.0
1.0067
1.4712
3.3505
9.3672
9.5529
10.5317
10.5396
δ / ppm
7.7049
7.6926
7.6869
7.6805
7.6676
7.6646
7.4125
7.3982
7.3871
7.3833
7.3724
7.3670
7.3422
7.3320
7.3171
7.3030
7.2601
6.9148
6.8969
4.8363
4.8283
4.8205
4.8124
4.0336
4.0255
4.0100
4.0020
3.9217
3.9058
3.8979
3.8823
3.8603
1.5643
1.4483
1.3901
1.0743
0.0065

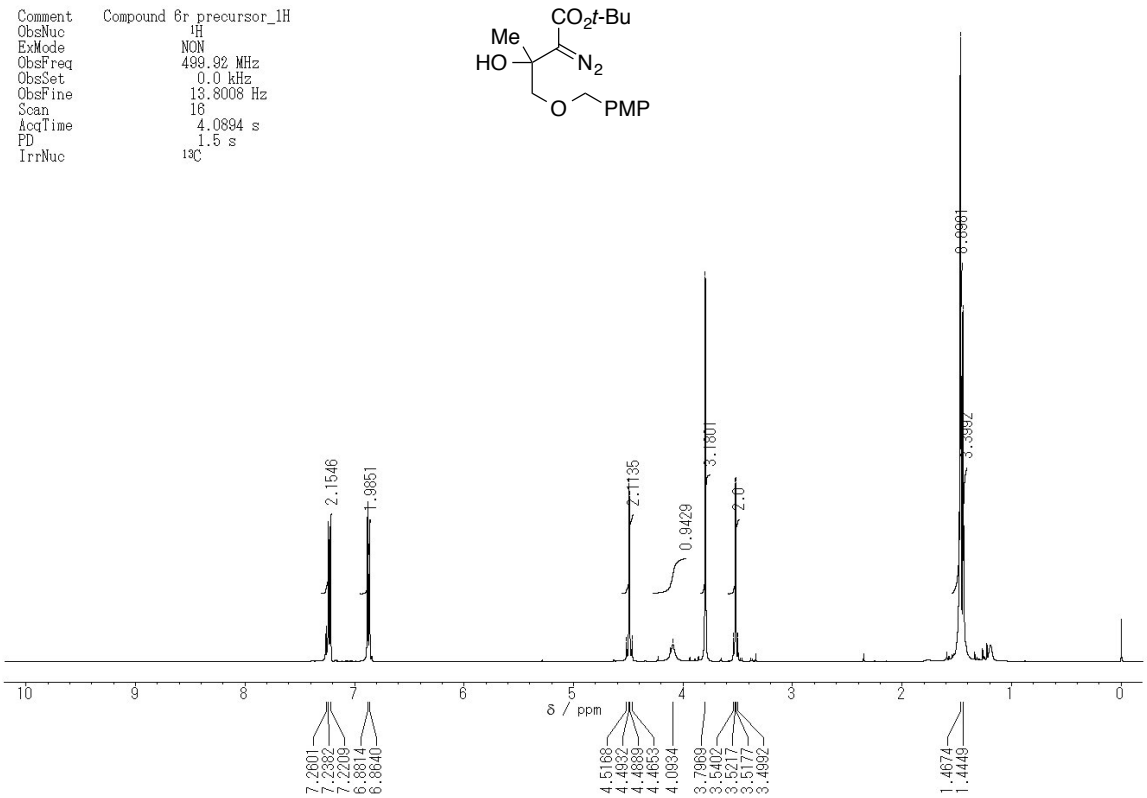

Comment Compound 2_13C
ObsNuc 13C
ExMode CARBON
ObsFreq 125.72 MHz
ObsSet 0.0 kHz
ObsFine 0.1276 Hz
Scan 128
AcqTime 1.0486 s
PD 1.9514 s
IrrNuc 1H
δ / ppm
170.0731
167.5582
163.3475
161.5590
135.7037
135.6430
133.4637
133.4296
131.8459
129.6135
129.5547
127.6466
127.5727
122.2336
112.8564
109.2319
89.9201
86.1400
81.4875
80.3723
77.2542
77.0000
76.7459
62.2515
55.3515
28.2235
28.0414
26.8598
19.2750
1.8276

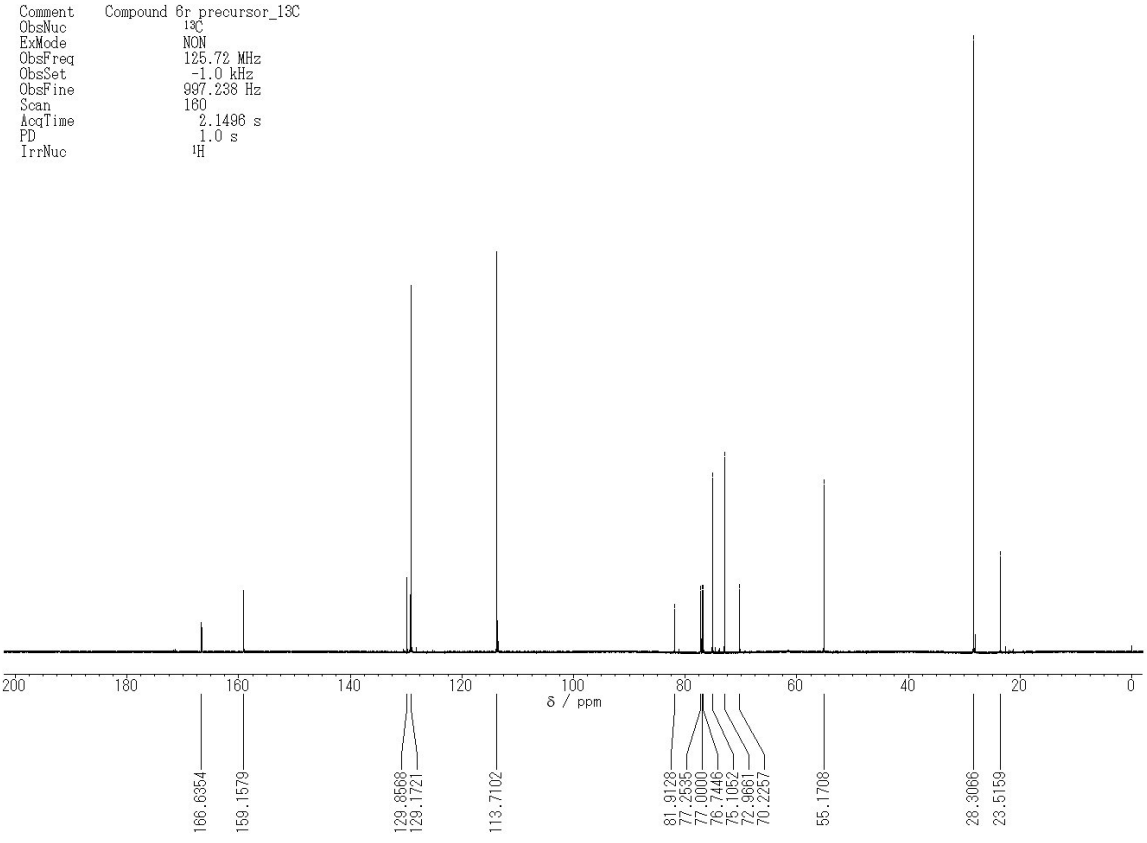

Comment Compound 6p_1H
ObsNuc 1H
ExMode NON
ObsFreq 499.92 MHz
ObsSet 0.0 kHz
ObsFine 13.8008 Hz
Scan 16
AcqTime 4.0894 s
PD 1.5 s
IrrNuc 13C
NC
CO2t-Bu
TMSO
N2
O
PMP
6p
2.054
1.9852
2.0
2.8973
0.9925
0.9389
8.8969
8.8883
δ / ppm
7.2764
7.2590
6.8925
6.8751
4.5860
3.8657
3.8454
3.8007
3.7124
3.6921
1.4839
0.2485

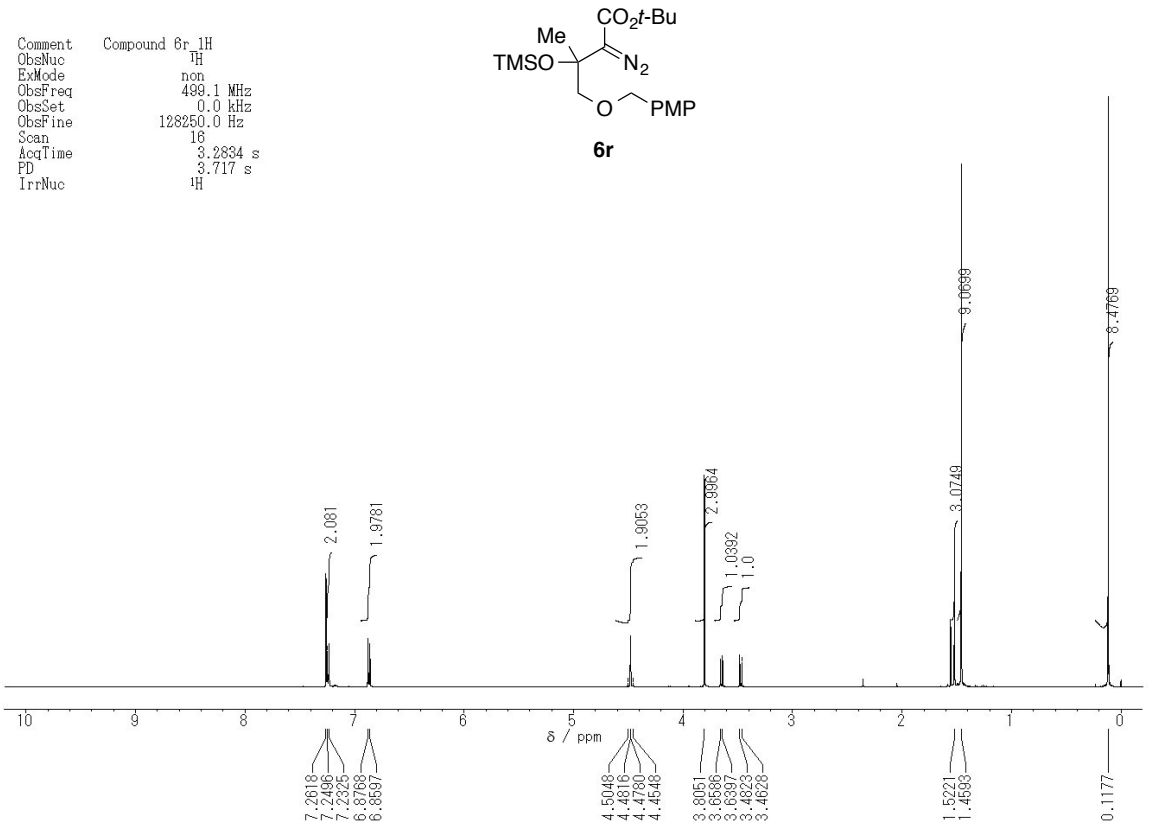

Comment Compound 6p_13C
ObsNuc 13C
ExMode NON
ObsFreq 125.72 MHz
ObsSet -1.0 kHz
ObsFine 997.238 Hz
Scan 128
AcqTime 2.1496 s
PD 1.0 s
IrrNuc 1H
δ / ppm
162.6848
159.4004
129.4738
129.0334
117.6349
113.7897
82.8898
77.2554
77.0000
76.7465
73.5194
73.2307
68.8342
55.1745
28.2233
0.8041

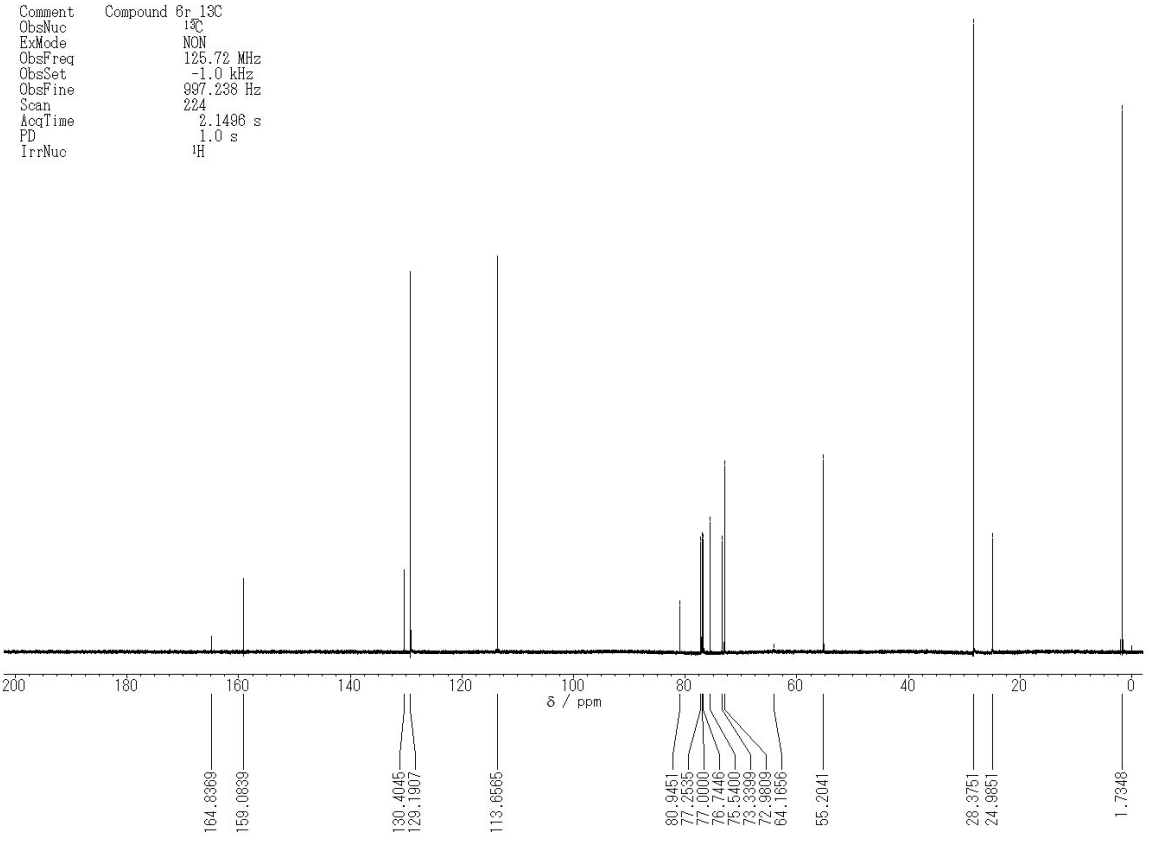

Comment Compound 6q_1H
ObsNuc 1H
ExMode non
ObsFreq 499.1 MHz
ObsSet 0.0 kHz
ObsFine 128250.0 Hz
Scan 16
AcqTime 3.2834 s
PD 3.717 s
IrrNuc 1H
CO2t-Bu
H
TMSO
N2
O
PMP
6q
5.9136
2.0138
1.0
1.9993
3.1591
2.1144
8.7998
9.1831
δ / ppm
7.2611
7.2544
7.2367
6.8817
6.8640
4.7531
4.7427
4.7318
4.5005
3.8057
3.5543
3.5433
3.5336
3.5269
3.5226
3.5159
3.5067
3.4951
1.5502
1.4739
0.1379
0.0000

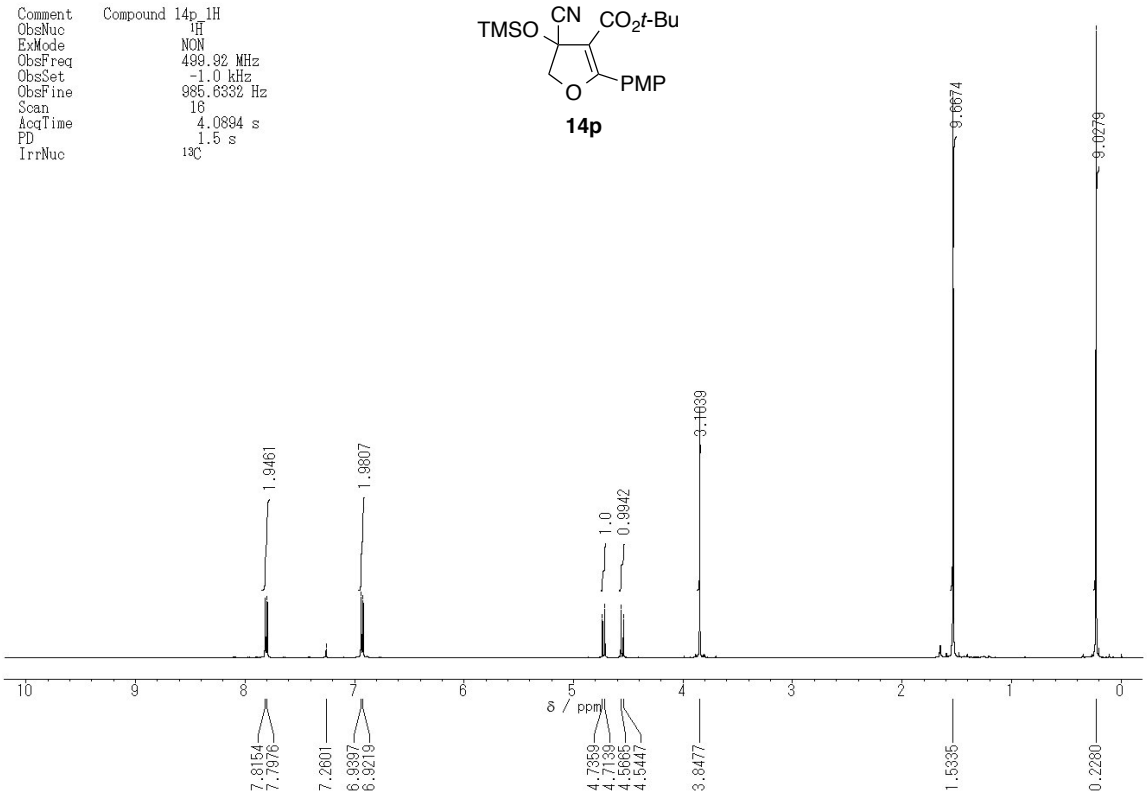

Comment Compound 6q_13C
ObsNuc 13C
ExMode NON
ObsFreq 125.72 MHz
ObsSet -1.0 kHz
ObsFine 997.238 Hz
Scan 160
AcqTime 2.1496 s
PD 1.0 s
IrrNuc 1H
δ / ppm
164.9553
159.1006
129.9974
129.1092
113.6251
81.1819
77.2554
77.0000
76.7446
72.8829
72.0132
65.9087
60.0910
55.0653
28.2548
-0.3080

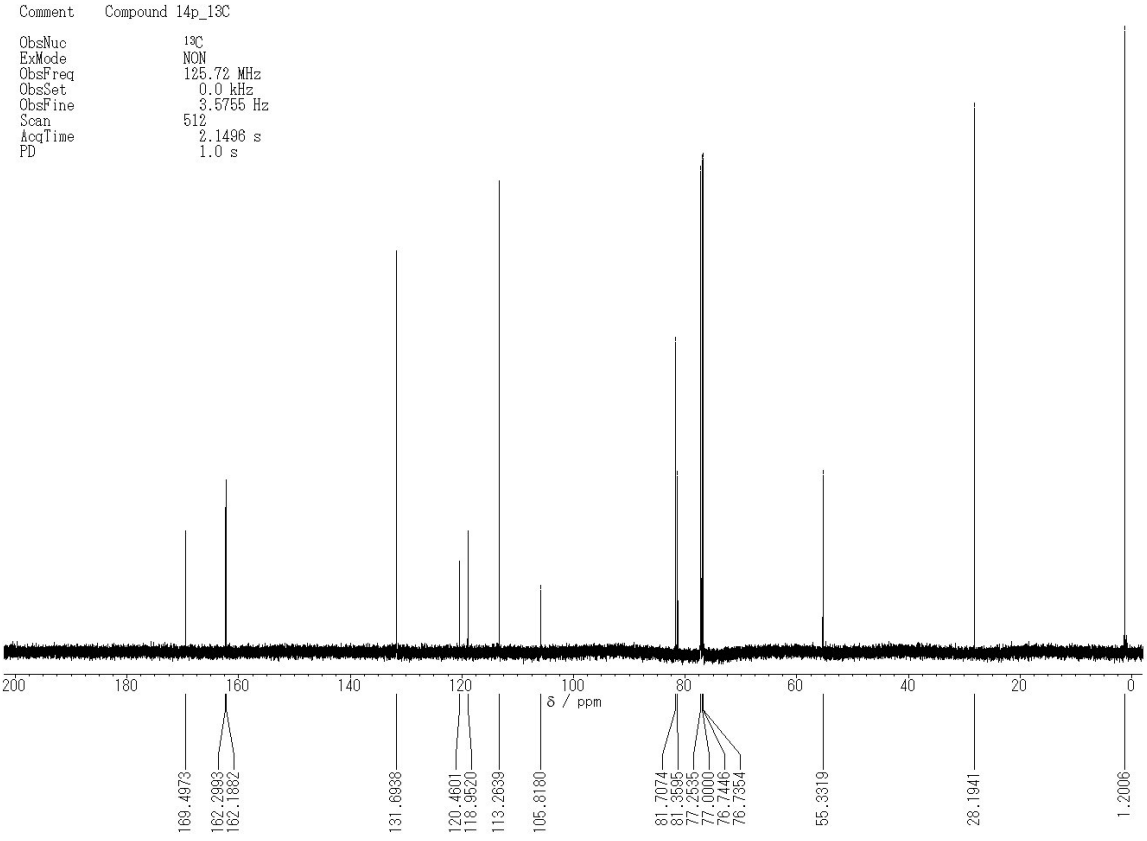

Comment Compound 6r precursor_1H
ObsNuc 1H
ExMode NON
ObsFreq 499.92 MHz
ObsSet 0.0 kHz
ObsFine 13.8008 Hz
Scan 16
AcqTime 4.0894 s
PD 1.5 s
IrrNuc 13C
CO2t-Bu
Me
HO
N2
O
PMP
2.1546
1.9851
2.1135
0.9429
3.1801
2.0
8.8961
3.3892
δ / ppm
7.2601
7.2382
7.2209
6.8814
6.8640
4.5168
4.4932
4.4689
4.4653
4.0934
3.7969
3.5402
3.5217
3.5177
3.4992
1.4674
1.4449

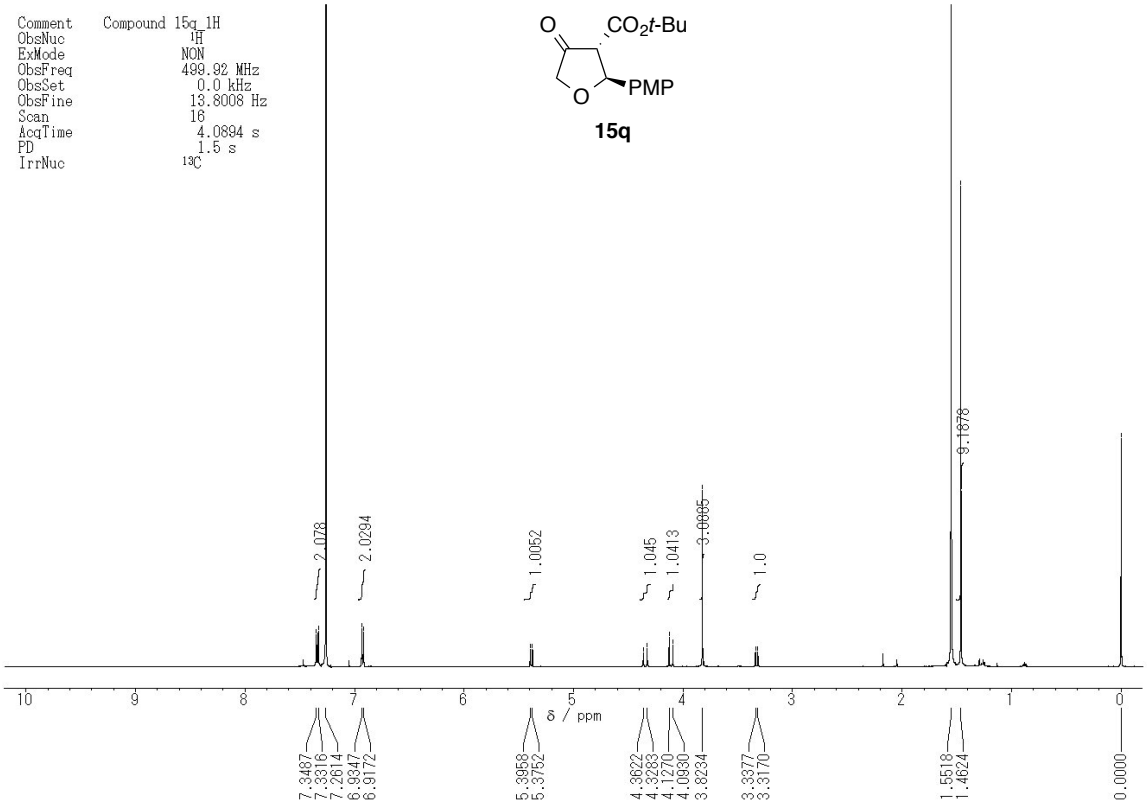

Comment Compound 6r precursor_13C
ObsNuc 13C
ExMode NON
ObsFreq 125.72 MHz
ObsSet -1.0 kHz
ObsFine 997.238 Hz
Scan 160
AcqTime 2.1496 s
PD 1.0 s
IrrNuc 1H
δ / ppm
166.6354
159.1579
129.8568
129.1721
113.7102
81.9128
77.2535
77.0000
76.7446
75.1052
72.9661
70.2257
55.1708
28.3066
23.5159

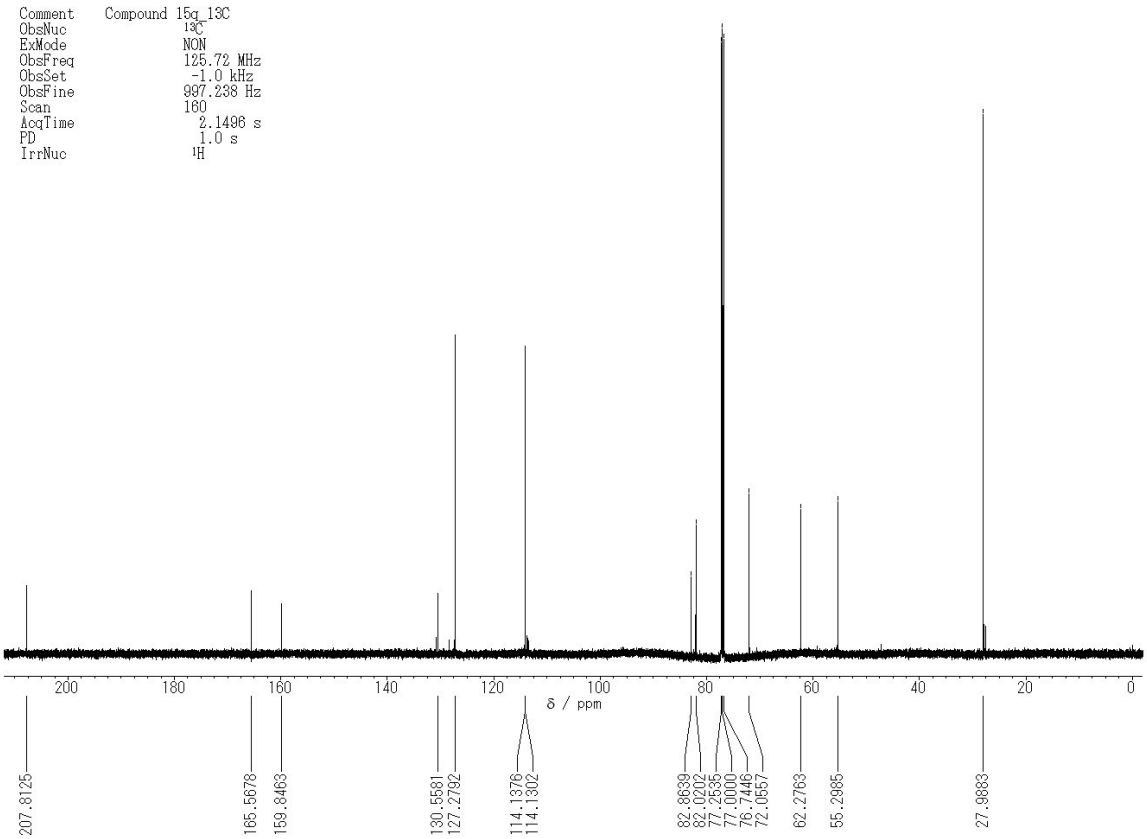

Comment Compound 6r_1H
ObsNuc 1H
ExMode non
ObsFreq 499.1 MHz
ObsSet 0.0 kHz
ObsFine 128250.0 Hz
Scan 16
AcqTime 3.2834 s
PD 3.717 s
IrrNuc 1H
Me
TMSO
CO2t-Bu
N2
O
PMP
6r
2.081
1.9781
1.9053
2.9964
1.0392
1.0
3.0749
9.0699
8.4769
δ / ppm
7.2618
7.2496
7.2325
6.8768
6.8597
4.5048
4.4816
4.4780
4.4548
3.8051
3.6586
3.6397
3.4823
3.4628
1.5221
1.4593
0.1177

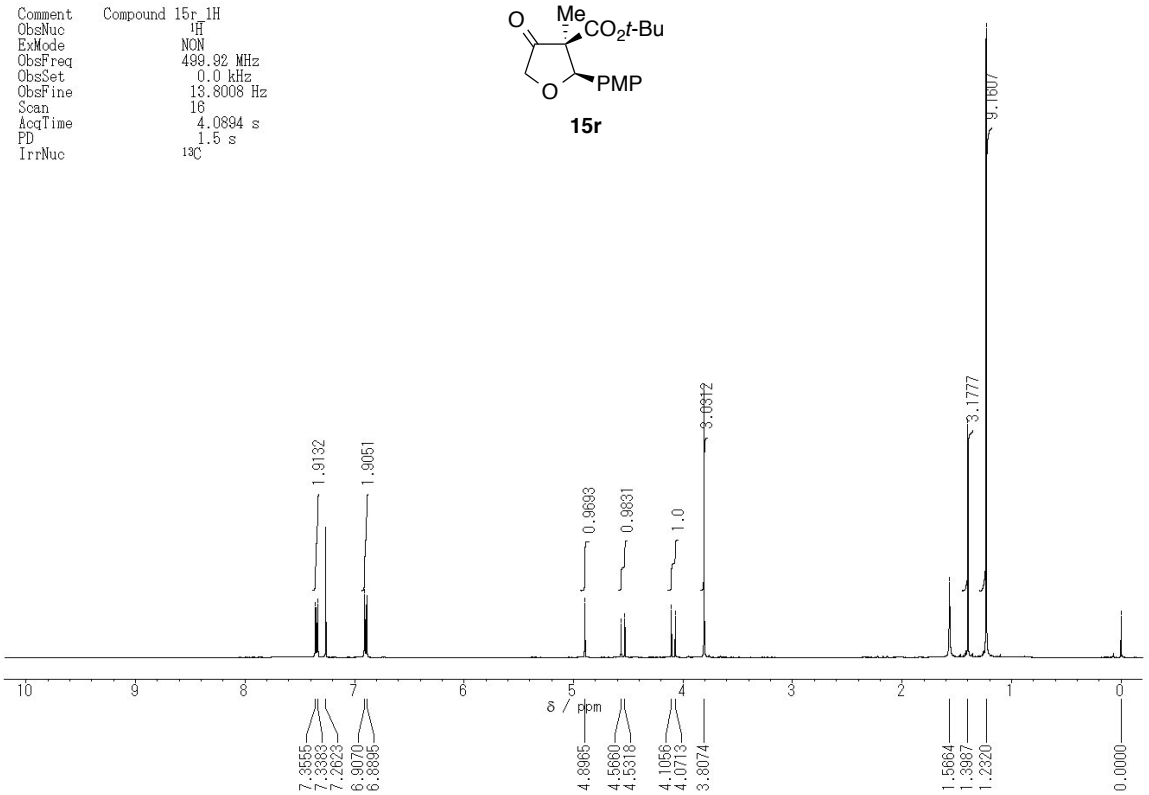

Comment Compound 6r_13C
ObsNuc 13C
ExMode NON
ObsFreq 125.72 MHz
ObsSet -1.0 kHz
ObsFine 997.238 Hz
Scan 224
AcqTime 2.1496 s
PD 1.0 s
IrrNuc 1H
δ / ppm
164.8369
159.0839
130.4045
129.1907
113.6565
80.9451
77.2536
77.0000
76.7446
76.5400
73.3399
72.9809
64.1656
55.2041
28.3751
24.9851
1.7348

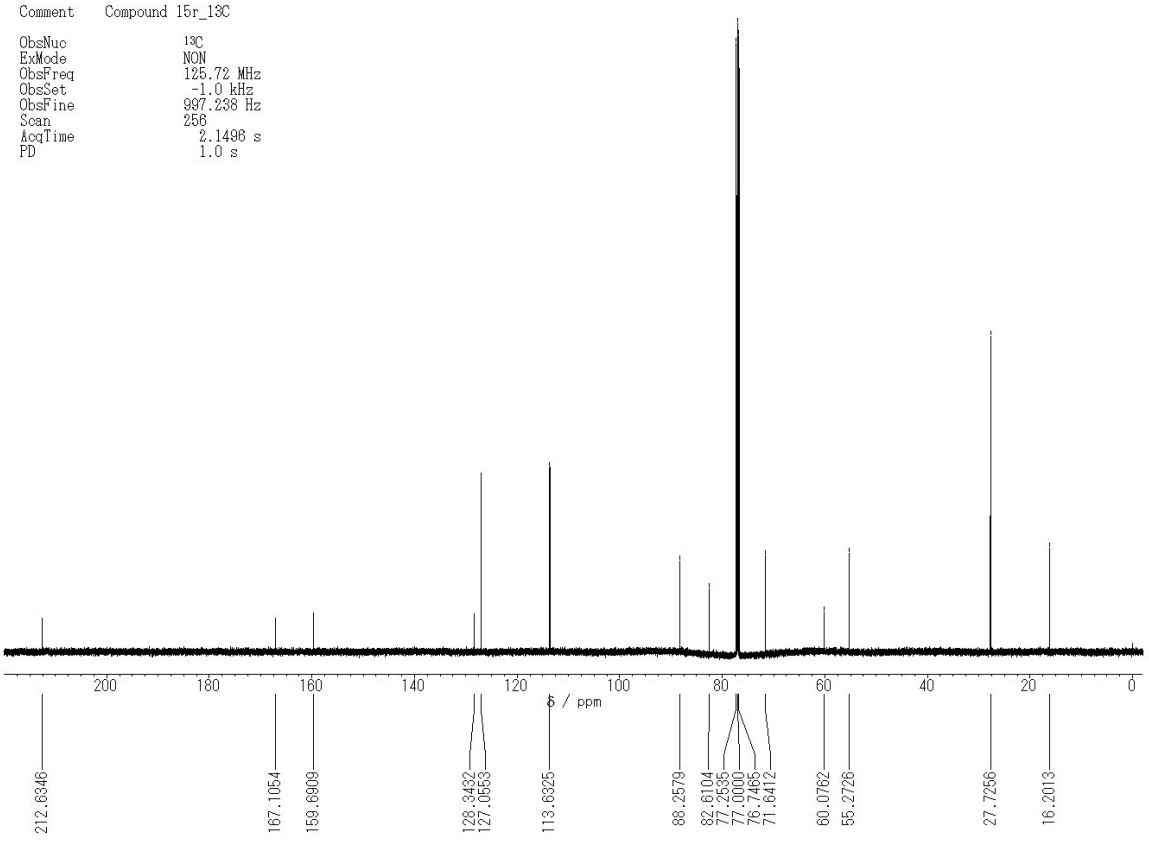

Comment Compound 14p_1H
ObsNuc 1H
ExMode NON
ObsFreq 499.92 MHz
ObsSet -1.0 kHz
ObsFine 985.6332 Hz
Scan 16
AcqTime 4.0894 s
PD 1.5 s
IrrNuc 13C
TMSO
CN
CO2t-Bu
PMP
O
14p
1.9461
1.9807
1.0
0.9942
3.1039
9.6674
9.0279
δ / ppm
7.8154
7.7976
7.2601
6.9397
6.9219
4.7359
4.7139
4.5665
4.5447
3.8477
1.5335
0.2280

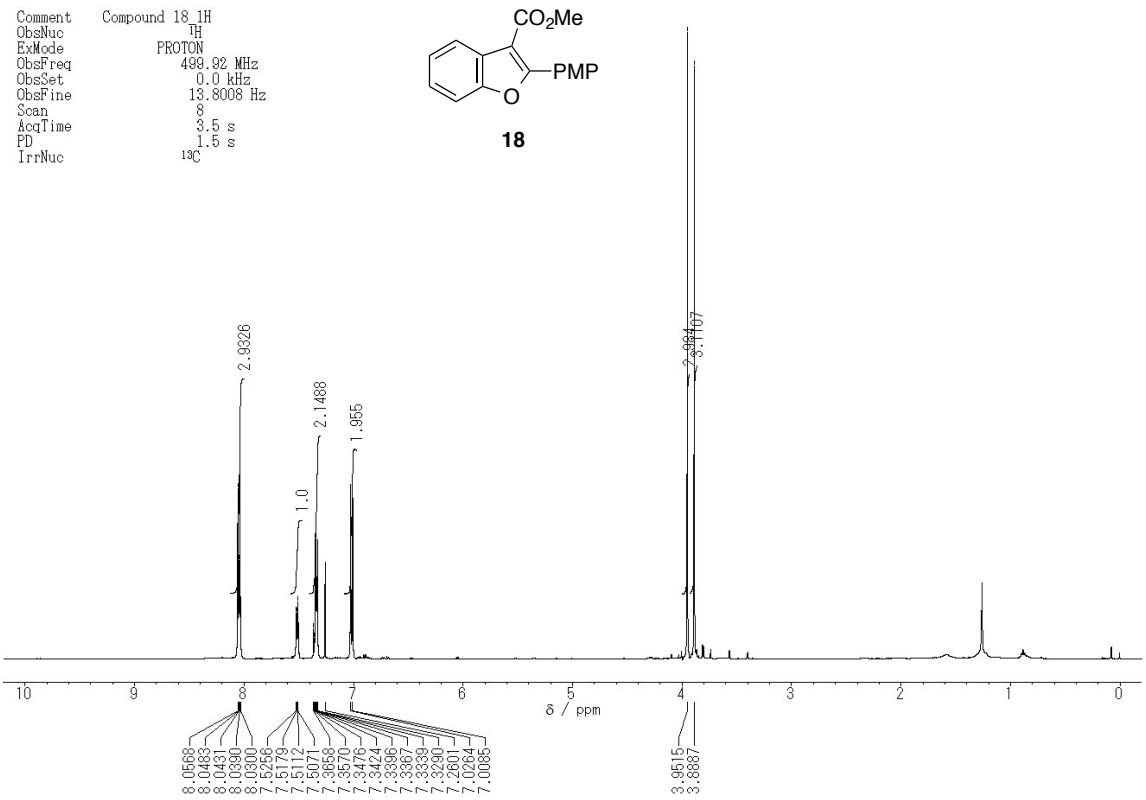

Comment Compound 14p_13C
ObsNuc 13C
ExMode NON
ObsFreq 125.72 MHz
ObsSet 0.0 kHz
ObsFine 3.5755 Hz
Scan 512
AcqTime 2.1496 s
PD 1.0 s
δ / ppm
169.4973
162.2993
162.1882
131.6938
120.4601
118.9520
113.2639
105.8180
81.7074
81.3696
77.2535
77.0000
76.7446
76.7354
55.3319
28.1941
1.2006

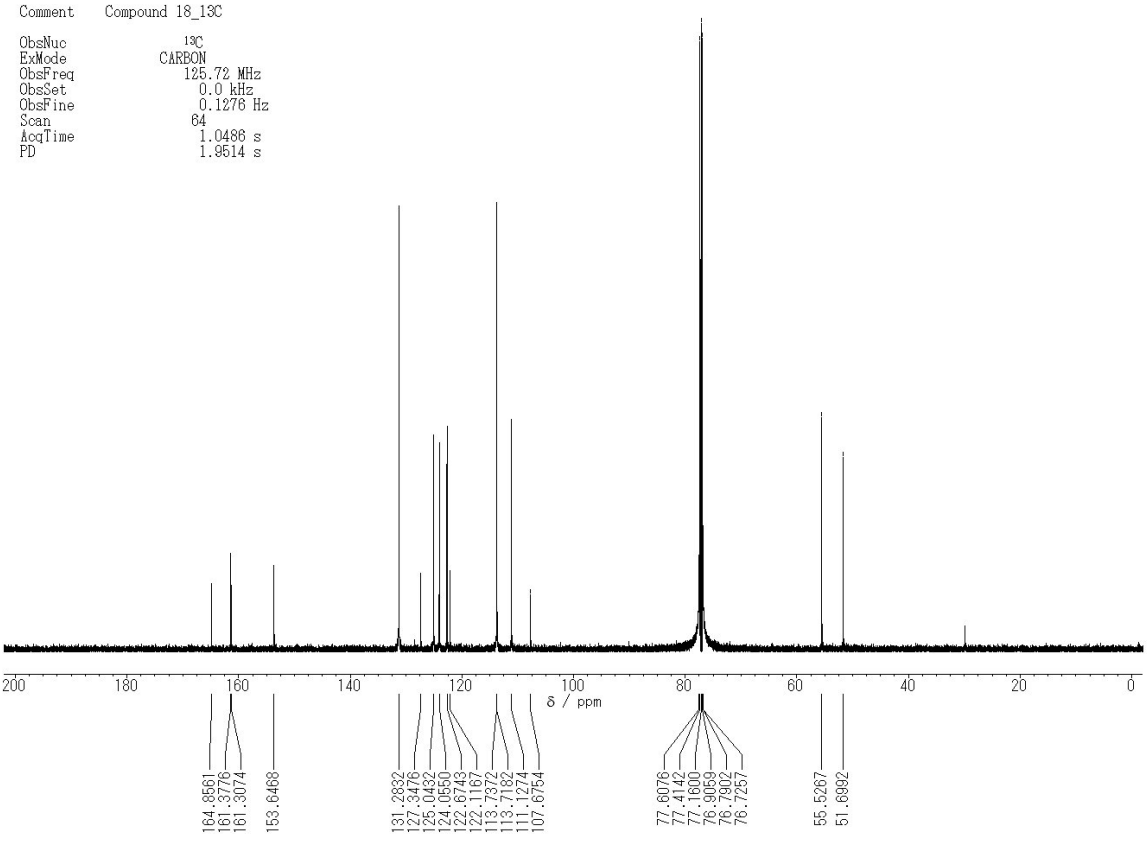

Comment Compound 15q_1H
ObsNuc 1H
ExMode NON
ObsFreq 499.92 MHz
ObsSet 0.0 kHz
ObsFine 13.8008 Hz
Scan 16
AcqTime 4.0894 s
PD 1.5 s
IrrNuc 13C
O
CO2t-Bu
O
PMP
15q
2.078
2.0294
1.0052
1.045
1.0413
3.0085
1.0
9.1878
δ / ppm
7.3487
7.3316
7.2614
6.9347
6.9172
5.3958
5.3752
4.3622
4.3283
4.1270
4.0930
3.8234
3.3377
3.3170
1.5518
1.4624
0.0000

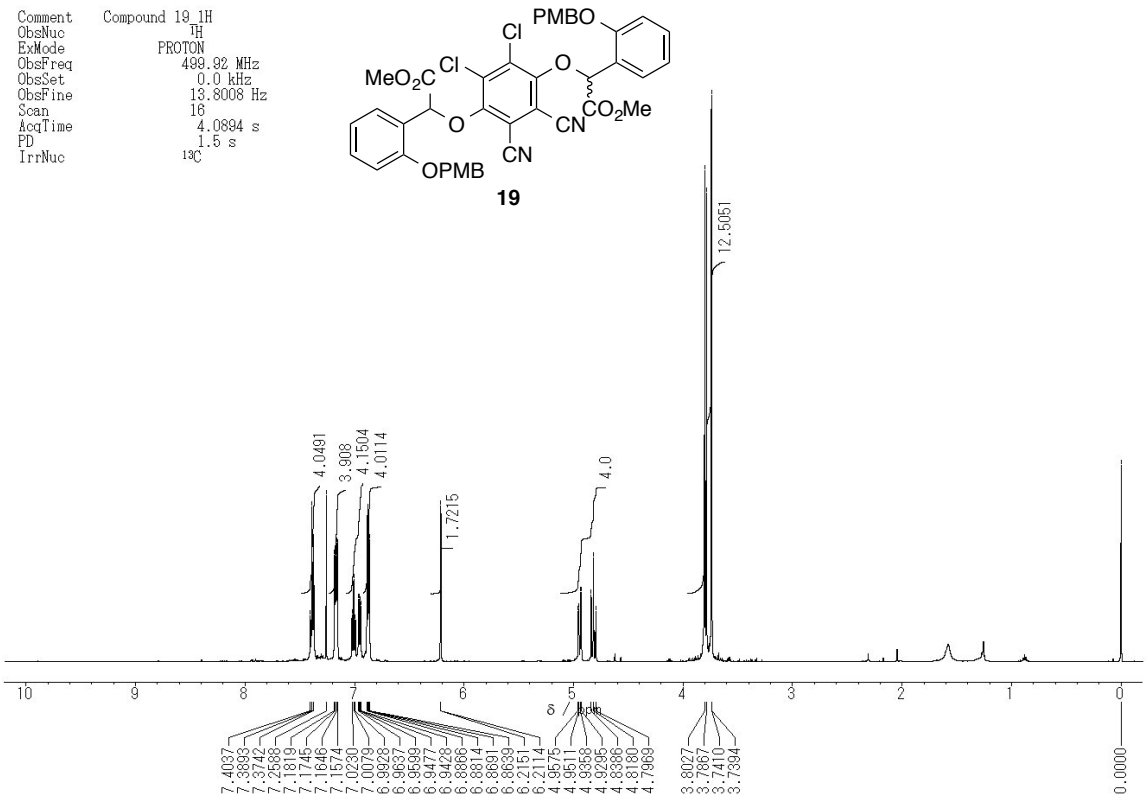

Comment Compound 15q_13C
ObsNuc 13C
ExMode NON
ObsFreq 125.72 MHz
ObsSet -1.0 kHz
ObsFine 997.238 Hz
Scan 160
AcqTime 2.1496 s
PD 1.0 s
IrrNuc 1H
δ / ppm
207.8125
165.5678
159.8463
130.5581
127.2792
114.1376
114.1302
82.8639
82.0202
77.2535
77.0000
76.7446
72.0557
62.2763
55.2985
27.9883

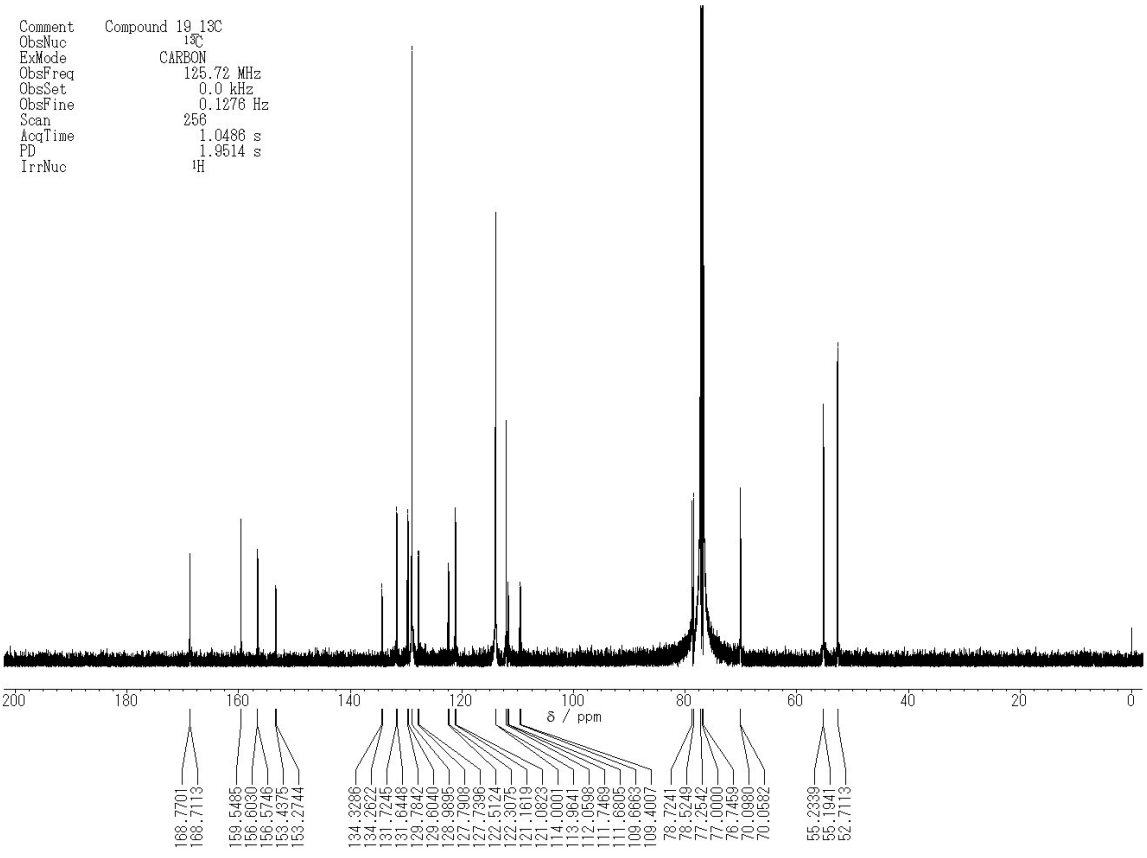

Comment Compound 15r_1H
ObsNuc 1H
ExMode NON
ObsFreq 499.92 MHz
ObsSet 0.0 kHz
ObsFine 13.8008 Hz
Scan 16
AcqTime 4.0894 s
PD 1.5 s
IrrNuc 13C
Me
CO2t-Bu
PMP
15r
1.9132
1.9051
0.9693
0.9831
1.0
3.0312
3.1777
9.1607
δ / ppm
7.3555
7.3383
7.2623
6.9070
6.8895
4.8965
4.5660
4.5318
4.1056
4.0713
3.8074
1.5664
1.3987
1.2320
0.0000


Comment Compound 15r_13C
ObsNuc 13C
ExMode NON
ObsFreq 125.72 MHz
ObsSet -1.0 kHz
ObsFine 997.238 Hz
Scan 256
AcqTime 2.1496 s
PD 1.0 s
δ / ppm
212.6346
167.1054
159.6909
128.3432
127.0563
113.6325
88.2579
82.6104
77.2535
77.0000
76.7465
71.6412
60.0762
55.2726
27.7256
16.2013


Comment Compound 18_1H
ObsNuc 1H
ExMode PROTON
ObsFreq 499.92 MHz
ObsSet 0.0 kHz
ObsFine 13.8008 Hz
Scan 8
AcqTime 3.5 s
PD 1.5 s
IrrNuc 13C
CO2Me
PMP
O
18
2.9326
2.1488
1.955
1.0
3.9515
3.8887
δ / ppm
8.0568
8.0483
8.0431
8.0390
8.0300
7.5256
7.5179
7.5112
7.5071
7.3658
7.3570
7.3476
7.3424
7.3396
7.3367
7.3339
7.3290
7.2601
7.0264
7.0085


Comment Compound 18_13C
ObsNuc 13C
ExMode CARBON
ObsFreq 125.72 MHz
ObsSet 0.0 kHz
ObsFine 0.1276 Hz
Scan 64
AcqTime 1.0486 s
PD 1.9514 s
δ / ppm
164.8561
161.3776
161.3074
153.6468
131.2832
127.3476
125.0432
124.0550
122.6743
122.1167
113.7372
113.7182
111.1274
107.6764
77.6076
77.4142
77.1600
76.9069
76.7902
76.7257
55.5267
51.6892


Comment Compound 19_1H
ObsNuc 1H
ExMode PROTON
ObsFreq 499.92 MHz
ObsSet 0.0 kHz
ObsFine 13.8008 Hz
Scan 16
AcqTime 4.0894 s
PD 1.5 s
IrrNuc 13C
PMBO
Cl
MeO2C
Cl
O
CO2Me
CN
O
CN
OPMB
19
12.5051
4.0491
3.908
4.1504
4.0114
1.7215
4.0
δ / ppm
0.0000


Comment Compound 19_13C
ObsNuc 13C
ExMode CARBON
ObsFreq 125.72 MHz
ObsSet 0.0 kHz
ObsFine 0.1276 Hz
Scan 256
AcqTime 1.0486 s
PD 1.9514 s
IrrNuc 1H
δ / ppm

3. NOESY Correlation Diagrams for β-Ketoesters 15q and 15r, and Copies of NOESY Spectra