ChemistryOPEN

Supporting Information

Construction of the Pentacyclic Core and Formal Total Synthesis of (*rac*)-Renieramycin T

Shinya Kimura and Naoki Saito*[a]

open_201800112_sm_miscellaneous_information.pdf

Supporting Information: open.201800112

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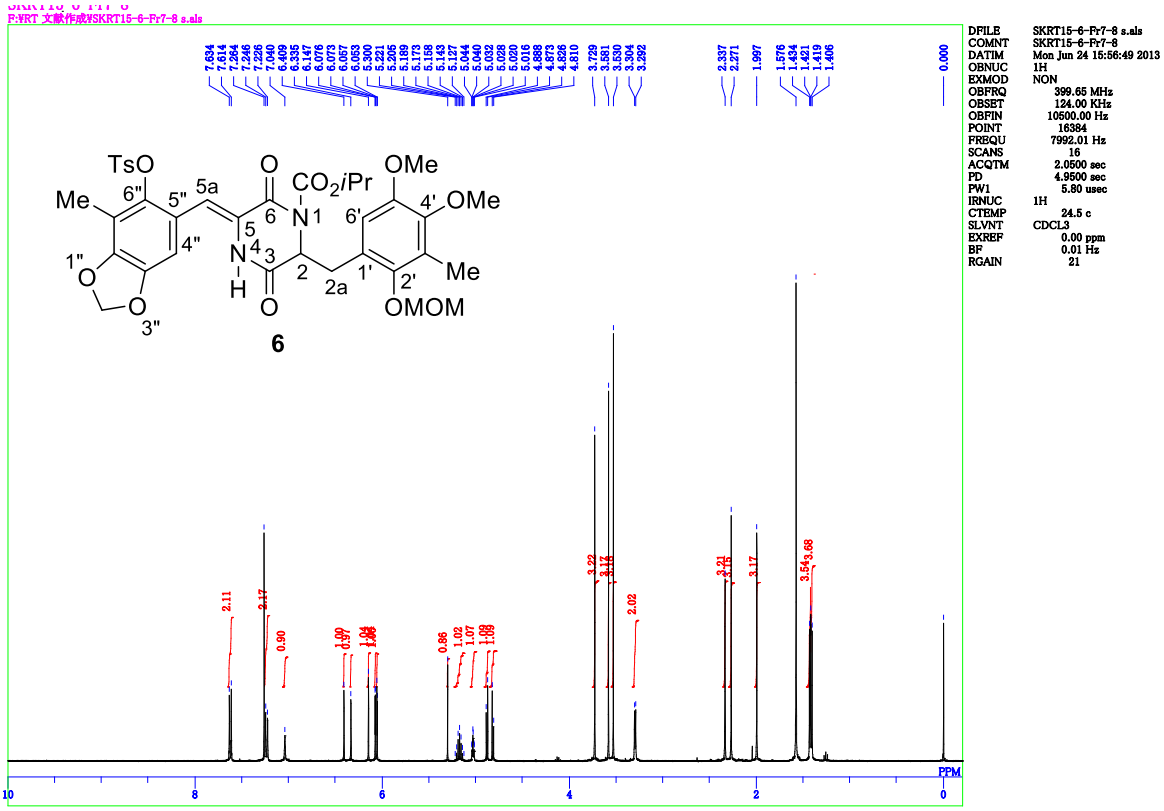
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TsO
Me
6"
5"
5a
O
CO2iPr
OMe
4'
OMe
6
N1
6'
4"
5
N4
3
2
1'
2'
Me
1"
O
H
2a
OMOM
3"
6
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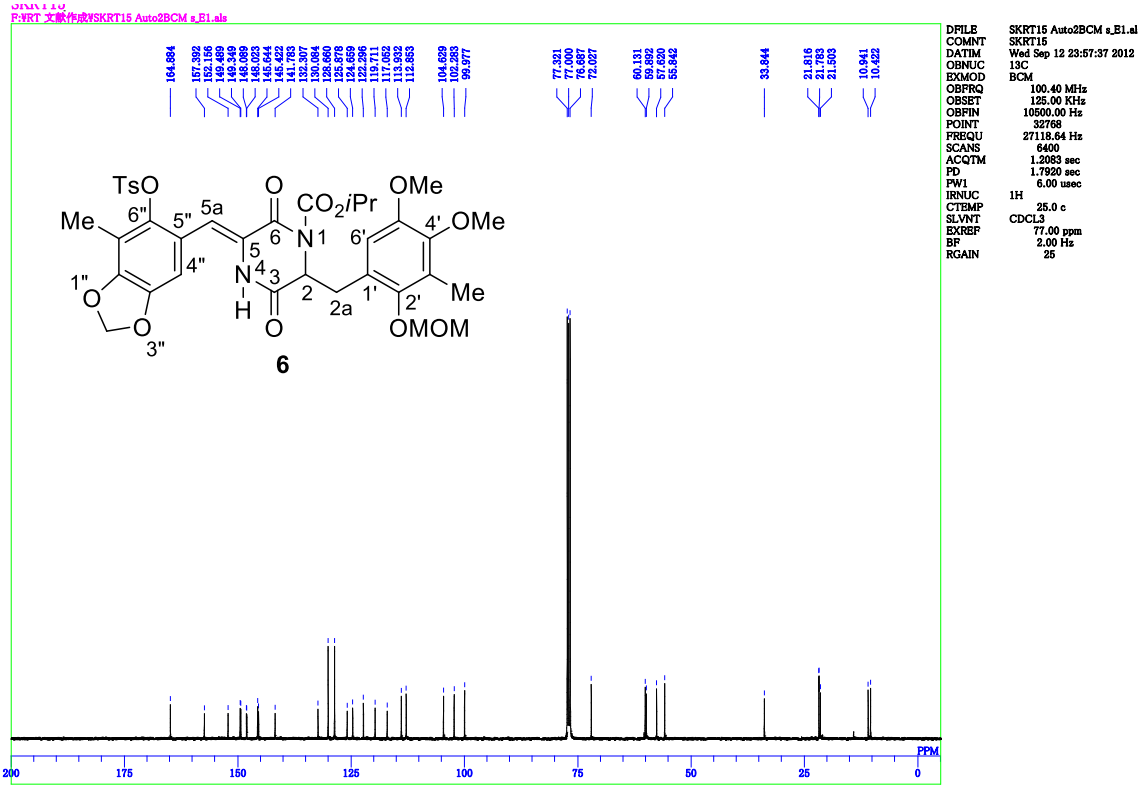
COMNT SKRT15-6-Fr7-8
DATIM Mon Jun 24 15:56:49 2013
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 16
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 5.80 usec
IRNUC 1H
CTEMP 24.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.01 Hz
RGAIN 21
PPM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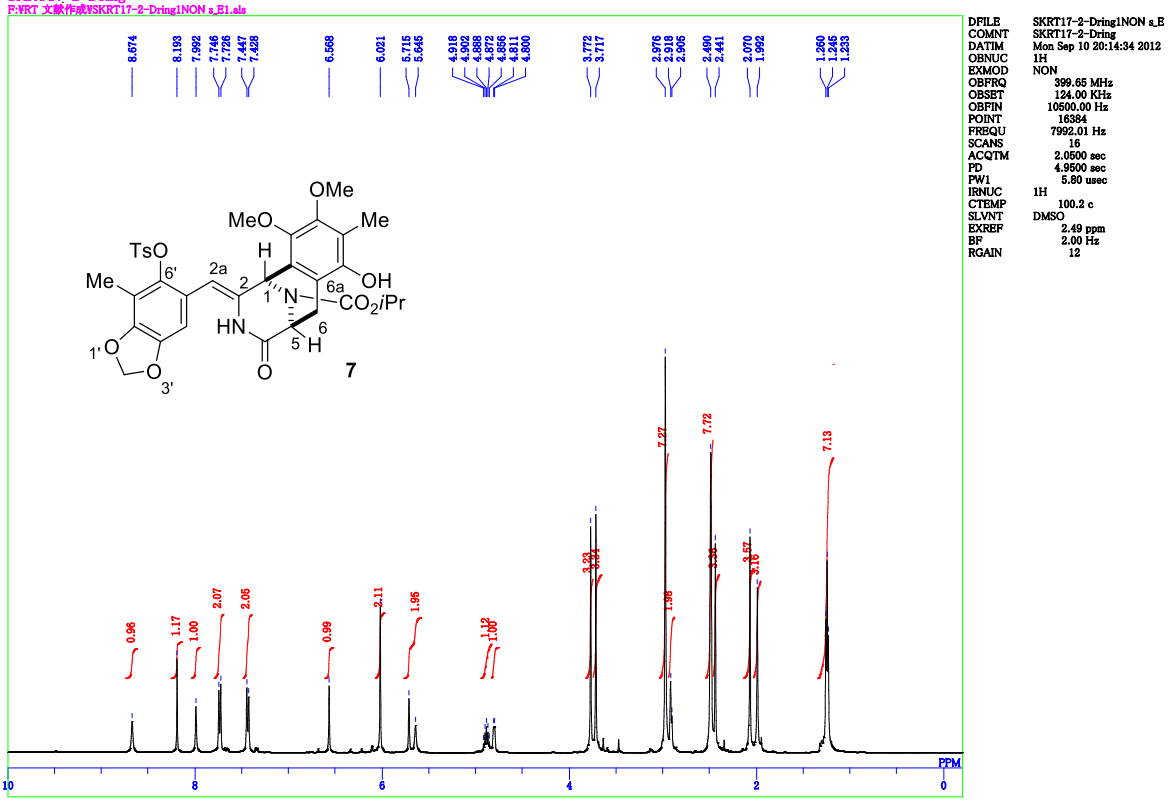
TsO
Me
6"
5"
5a
O
CO2iPr
OMe
4'
OMe
6
N1
6'
4"
5
N4
3
2
1'
2'
Me
1"
O
H
2a
OMOM
3"
6
DFILE SKRT15 Auto2BCM s_E1.al
COMNT SKRT15
DATIM Wed Sep 12 23:57:37 2012
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 6400
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 25.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 2.00 Hz
RGAIN 25
PPM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OMe
MeO
Me
H
TsO
6'
2a
2
1
N
6a
OH
CO2iPr
Me
HN
6
5
H
1'
O
O
3'
O
7
0.96 1.17 1.00 2.07 2.05 0.99 2.11 1.95 1.12 1.00 3.20 3.02 1.98 7.27 3.38 7.72 3.57 3.16 7.13
PPM
10 8 6 4 2 0
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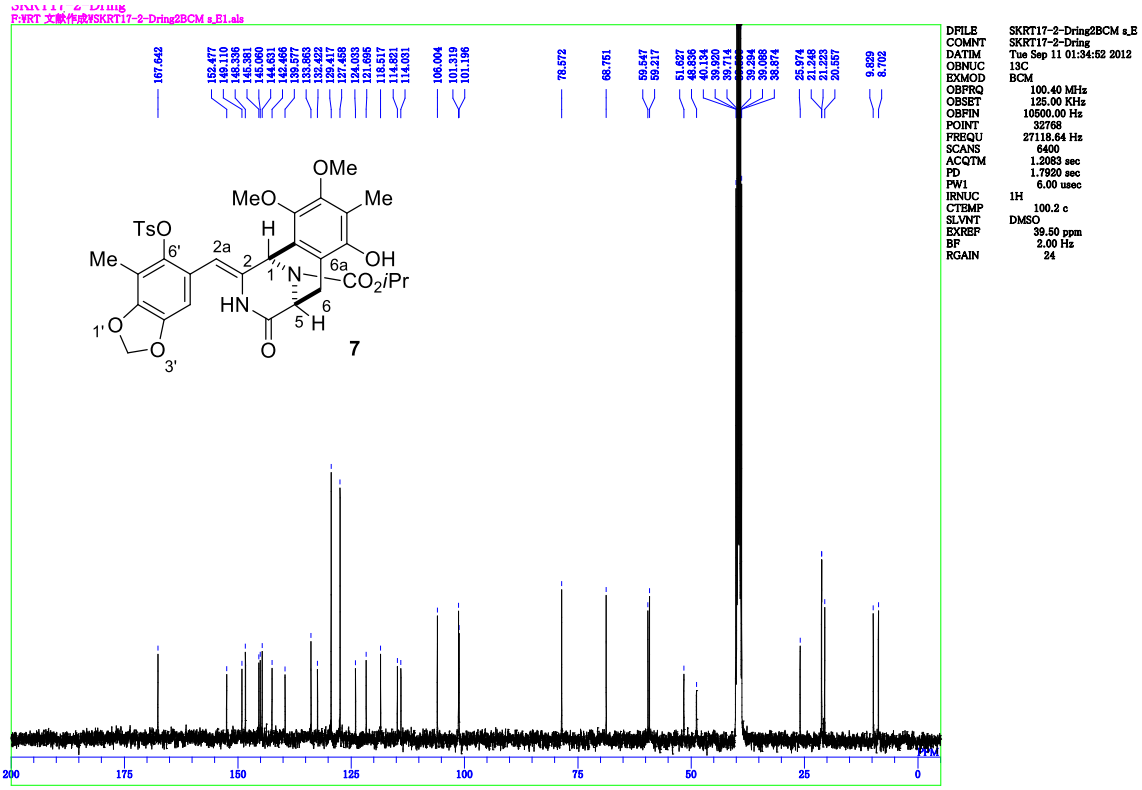
COMNT SKRT17-2-Dring
DATIM Mon Sep 10 20:14:34 2012
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 16
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 5.80 usec
IRNUC 1H
CTEMP 100.2 c
SLVNT DMSO
EXREF 2.49 ppm
BF 2.00 Hz
RGAIN 12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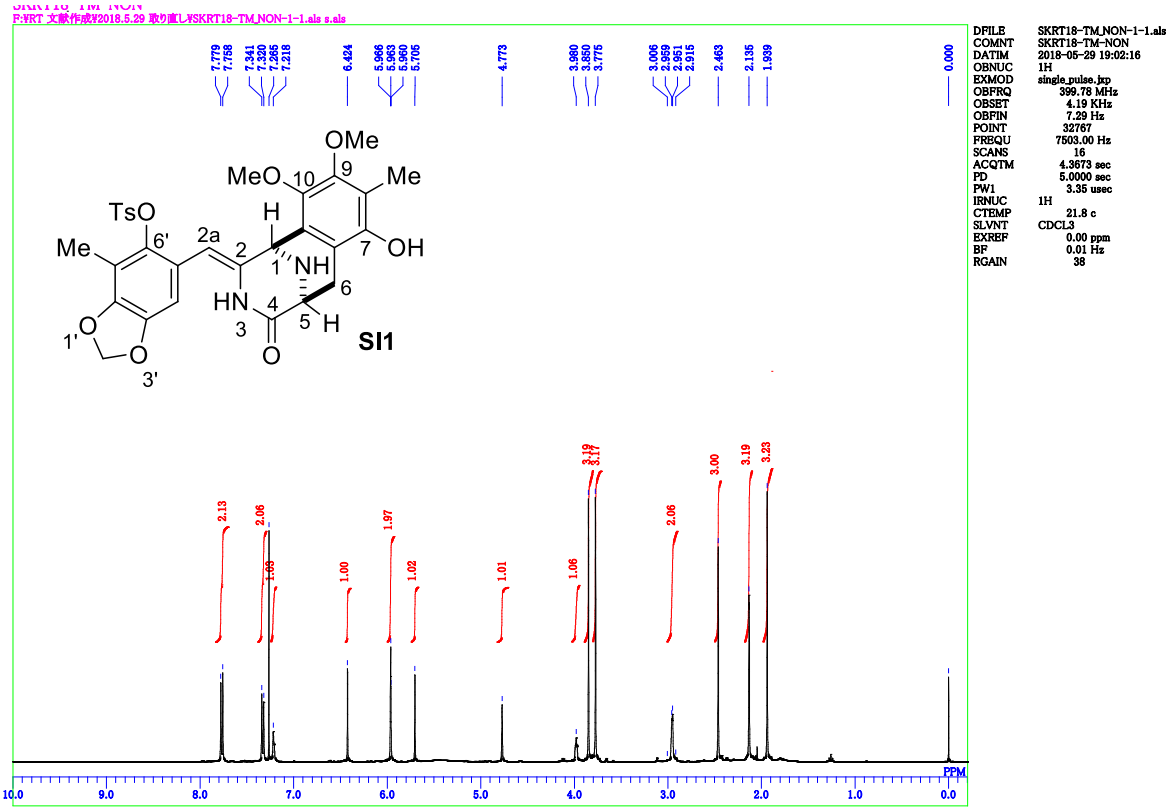
167.642 152.477 149.110 148.336 145.381 145.060 144.631 142.466 139.577 133.865 132.422 129.417 127.468 124.033 121.696 118.517 114.631 114.031 106.004 101.319 101.196 79.572 68.761 59.547 59.217 51.627 48.836 40.134 39.920 39.714 39.504 39.294 39.088 38.874 25.974 21.748 21.223 20.557 9.999 8.702
OMe
MeO
Me
H
TsO
6'
2a
2
1
N
6a
OH
CO2iPr
Me
HN
6
5
H
1'
O
O
3'
O
7
PPM
200 175 150 125 100 75 50 25 0
DFILE SKRT17-2-Dring2BCM s_E
COMNT SKRT17-2-Dring
DATIM Tue Sep 11 01:34:52 2012
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 6400
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 100.2 c
SLVNT DMSO
EXREF 39.50 ppm
BF 2.00 Hz
RGAIN 24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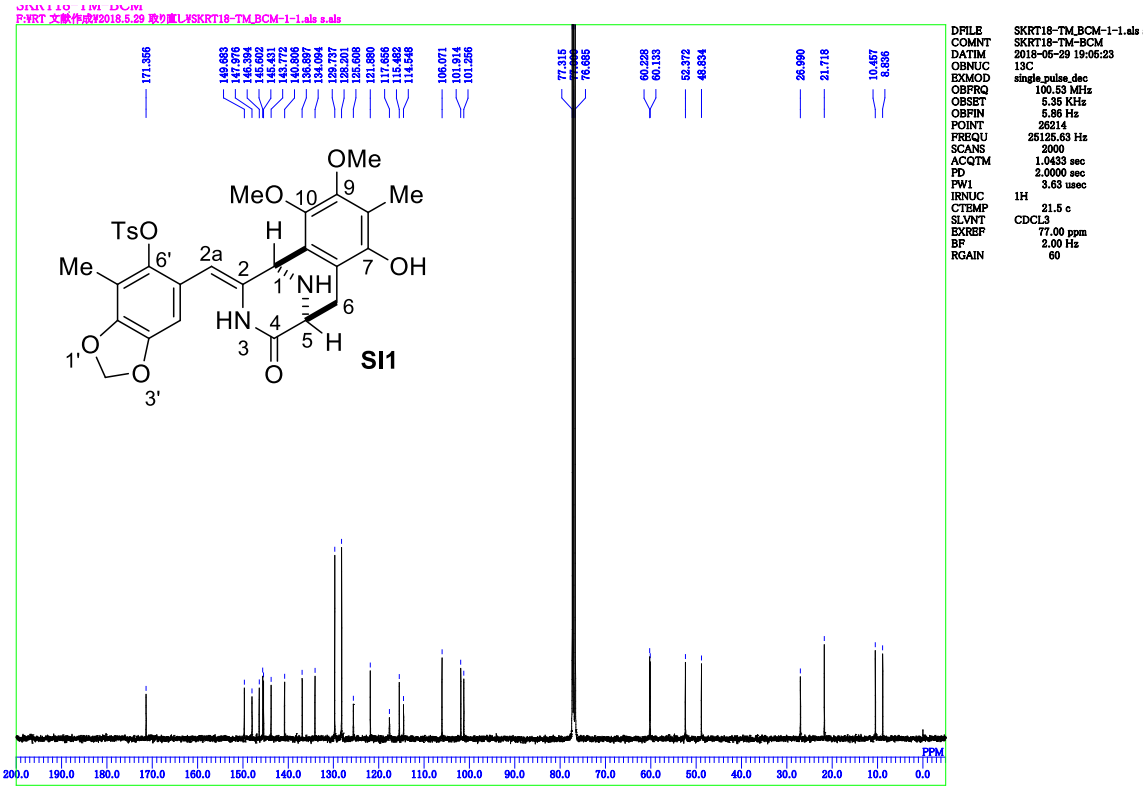
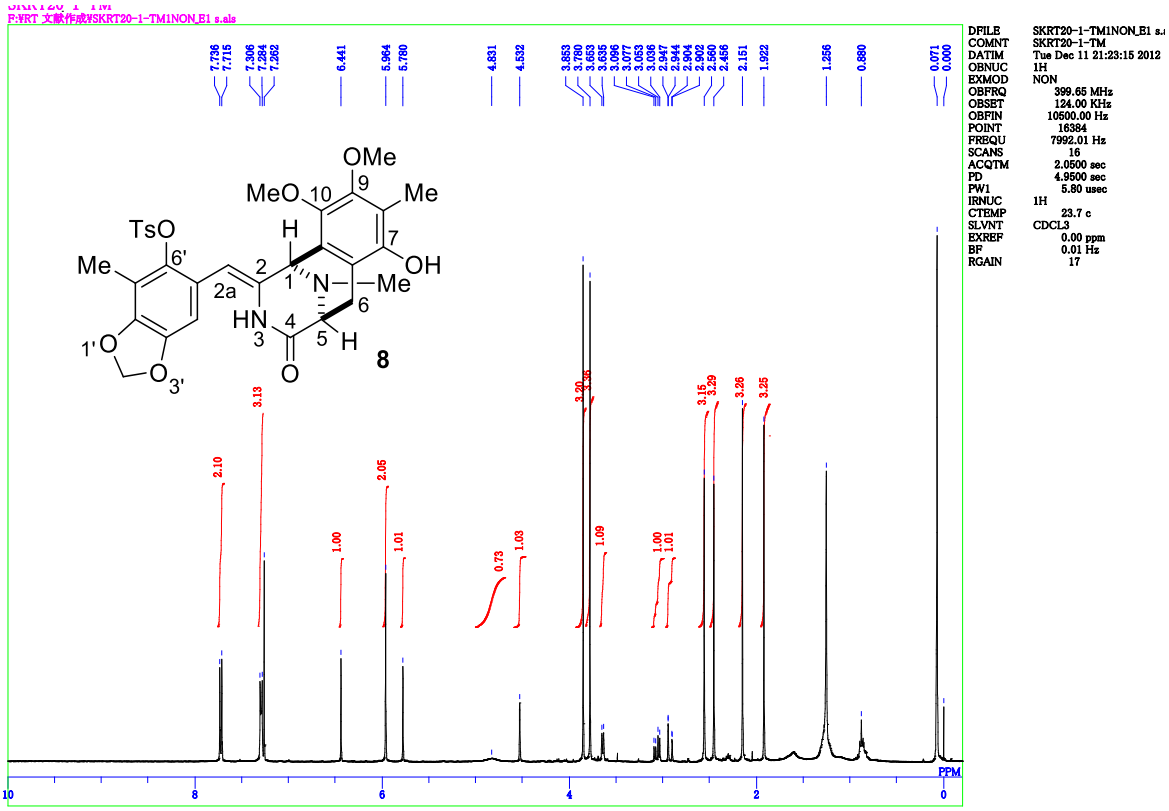
7.758
7.341
7.320
7.285
7.218
6.424
5.966
5.963
5.960
5.706
4.773
3.980
3.850
3.775
3.006
2.959
2.951
2.915
2.463
2.135
1.939
0.000
OMe
MeO
10
9
Me
TsO
H
Me
6'
2a
2
7
OH
1
NH
6
HN
4
5
H
1'
O
3
O
O
3'
SI1
2.13
2.06
1.03
1.00
1.97
1.02
1.01
1.06
3.19
3.17
2.06
3.00
3.19
3.23
PPM
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
DFILE SKRT18-TM_NON-1-1.als
COMNT SKRT18-TM-NON
DATIM 2018-05-29 19:02:16
OBNUC 1H
EXMOD single_pulse.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 32767
FREQU 7503.00 Hz
SCANS 16
ACQTM 4.3673 sec
PD 5.0000 sec
PW1 3.35 usec
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.01 Hz
RGAIN 38


F:¥RT 文献作成¥2018.5.29 取り直し¥SKRT18-TM_BCM-1-1.als s.als
171.356
149.683
147.976
146.394
145.602
145.411
143.772
140.806
136.897
134.094
129.737
128.201
125.988
121.880
117.466
115.469
114.548
106.071
101.914
101.256
77.315
77.000
76.685
60.228
60.133
52.372
48.834
26.990
21.718
10.457
6.838
OMe
MeO
10
9
Me
TsO
H
Me
6'
2a
2
7
OH
1
NH
6
HN
4
5
H
1'
O
3
O
O
3'
SI1
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
DFILE SKRT18-TM_BCM-1-1.als
COMNT SKRT18-TM-BCM
DATIM 2018-05-29 19:06:23
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 2000
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.63 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 2.00 Hz
RGAIN 60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7.306
7.284
7.262
6.441
5.964
5.780
4.831
4.532
3.853
3.780
3.653
3.635
3.096
3.077
3.053
3.036
2.947
2.944
2.904
2.902
2.850
2.495
2.151
1.922
1.256
0.880
0.071
0.000
OMe
MeO
10
9
Me
H
7
OH
TsO
6'
Me
2
2a
1
N
Me
6
HN
4
3
5
H
1'O
O
3'
O
8
2.10
3.13
1.00
2.05
1.01
0.73
1.03
3.20
3.36
1.09
1.00
1.01
3.15
3.29
3.26
3.25
PPM
10
8
6
4
2
0
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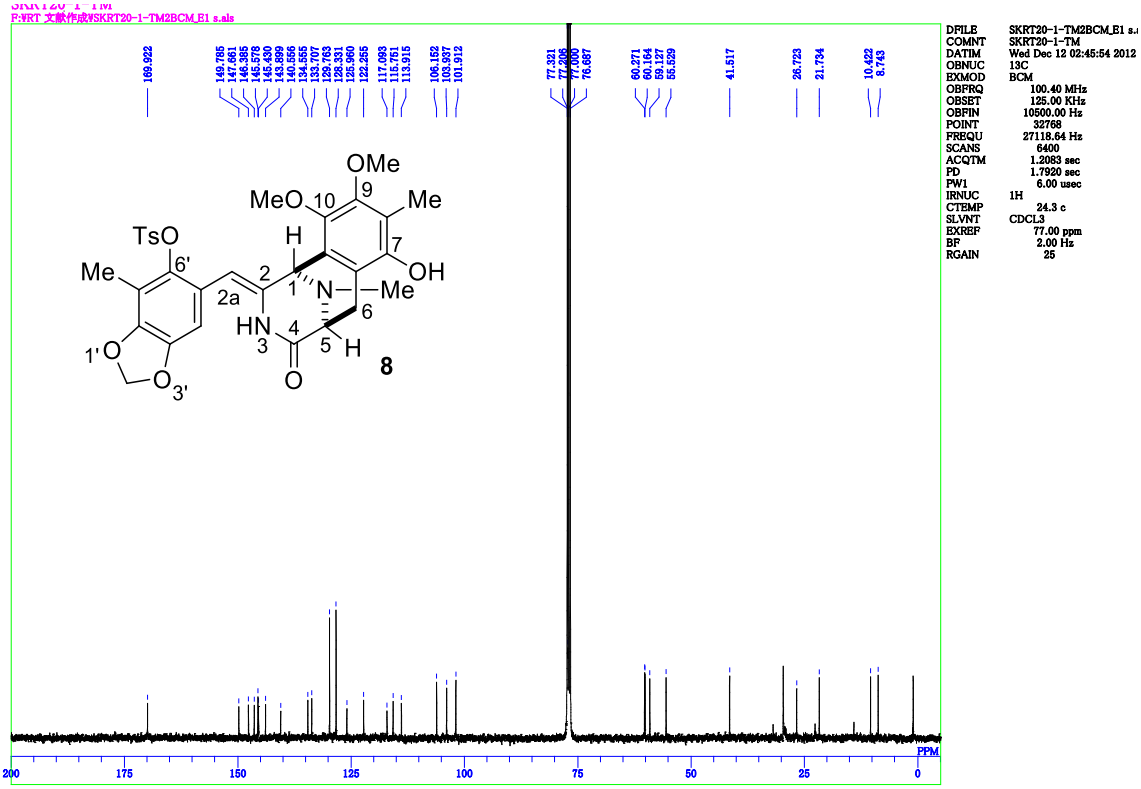
COMNT SKRT20-1-TM
DATIM Tue Dec 11 21:23:15 2012
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 16
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 5.80 usec
IRNUC 1H
CTEMP 23.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.01 Hz
RGAIN 17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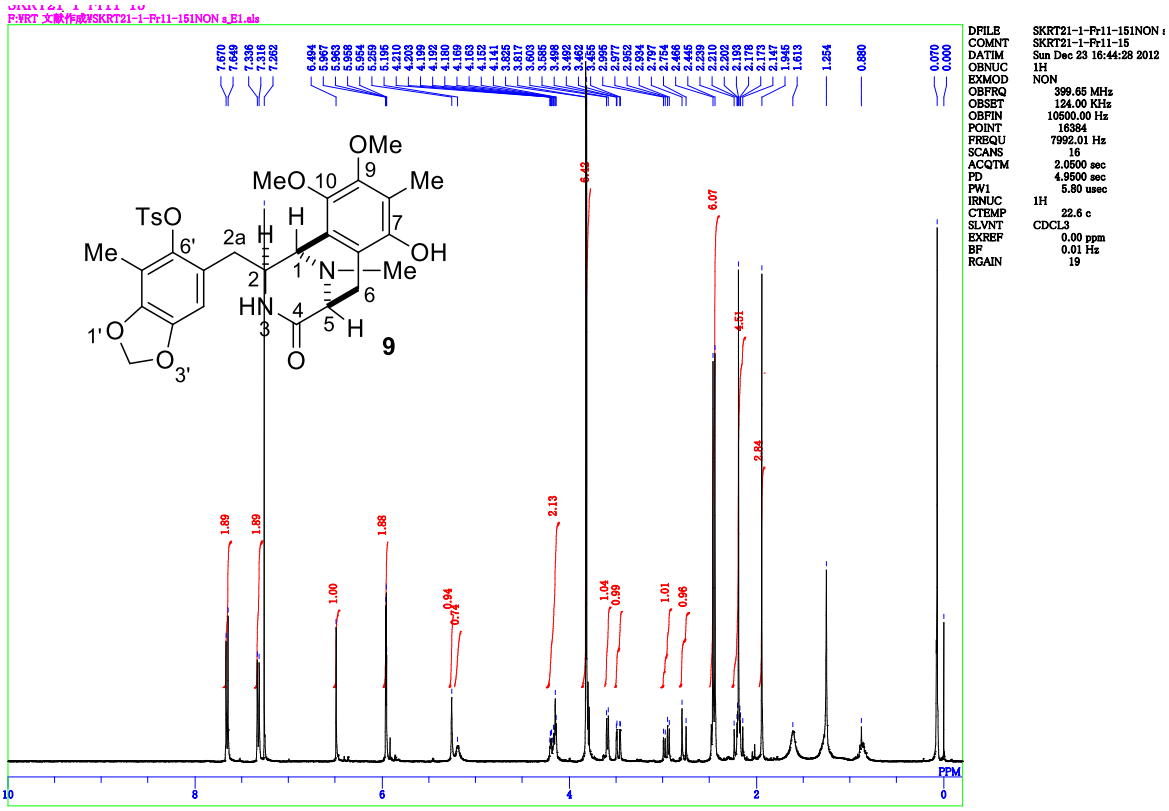
169.922
149.785
147.661
146.386
145.578
145.490
145.399
140.556
134.555
133.707
129.763
128.331
125.960
122.265
117.093
115.751
115.915
106.152
103.937
101.912
77.321
77.000
76.687
60.271
60.164
59.927
55.529
41.517
26.723
21.734
10.422
8.743
OMe
MeO
10
9
Me
H
7
OH
TsO
6'
Me
2
2a
1
N
Me
6
HN
4
3
5
H
1'O
O
3'
O
8
PPM
200
175
150
125
100
75
50
25
0
DFILE SKRT20-1-TM2BCM_E1 s.
COMNT SKRT20-1-TM
DATIM Wed Dec 12 02:45:54 2012
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 6400
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 24.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 2.00 Hz
RGAIN 25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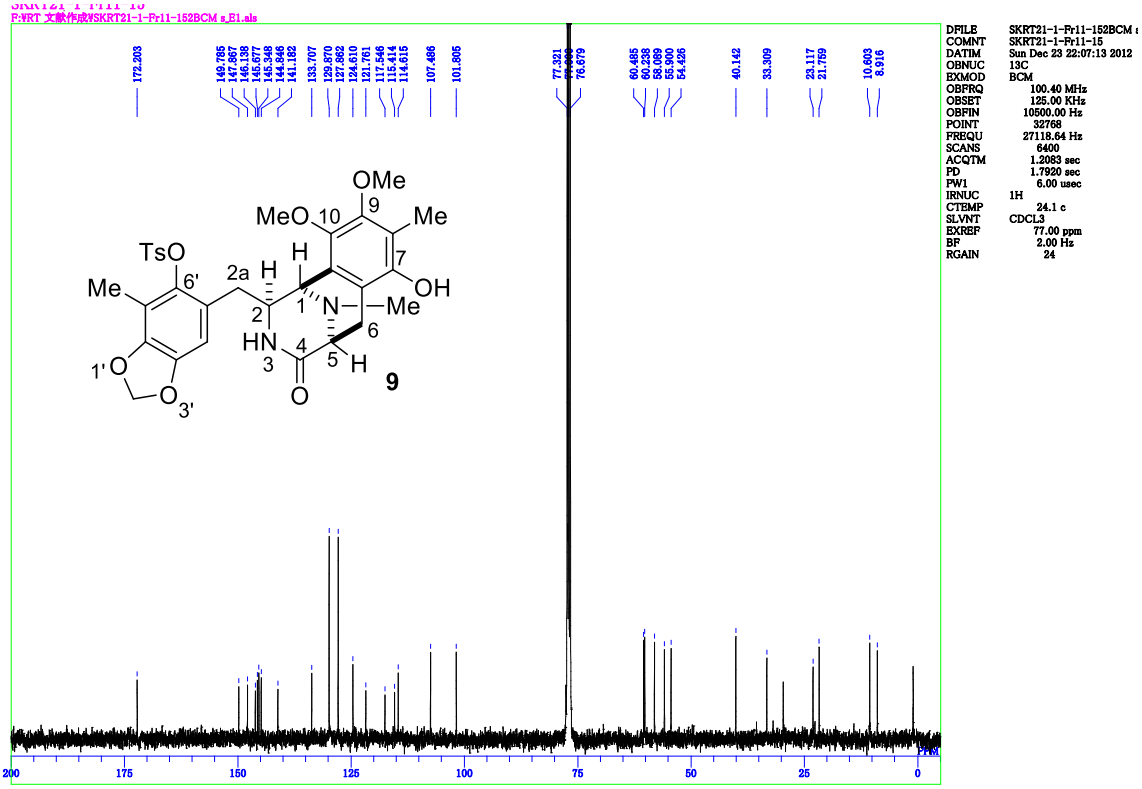
COMNT SKRT21-1-Fr11-15
DATIM Sun Dec 23 16:44:28 2012
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 16
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 5.80 usec
IRNUC 1H
CTEMP 22.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.01 Hz
RGAIN 19
OMe
MeO
Me
TsO
Me
2a
OH
HN
9
PPM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COMNT SKRT21-1-Fr11-15
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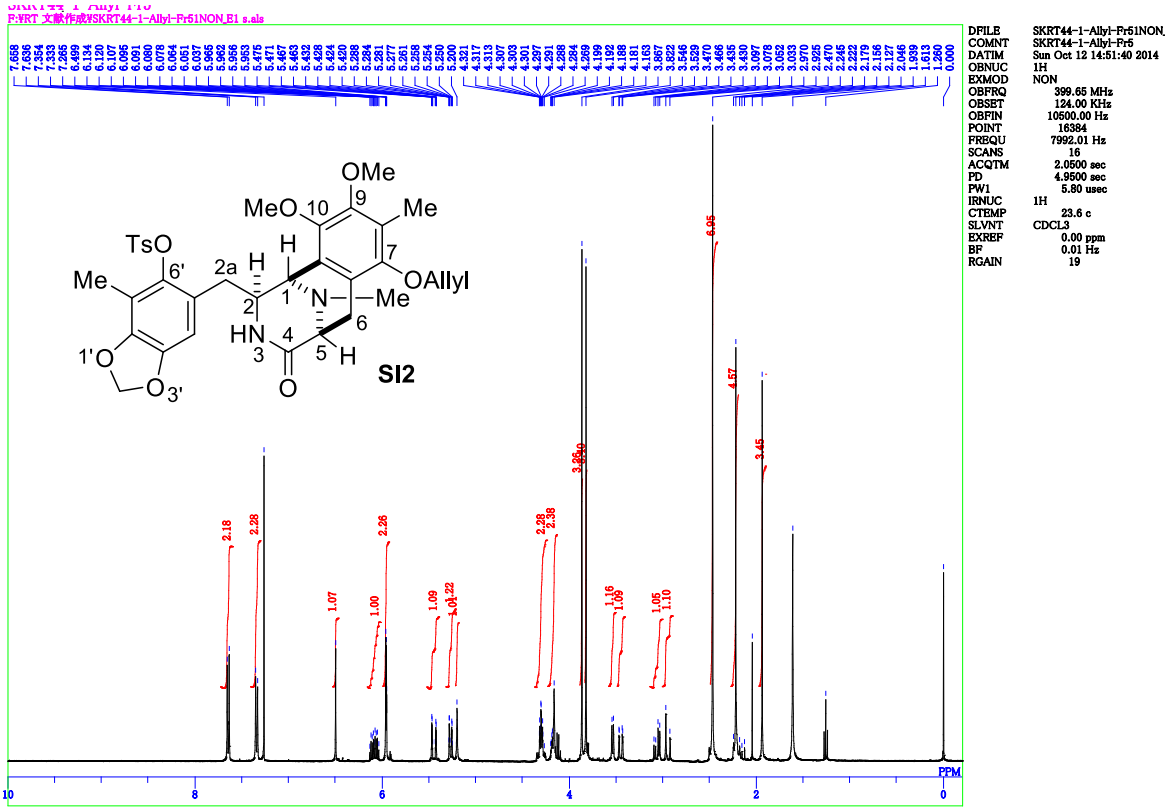
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 6400
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 24.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 2.00 Hz
RGAIN 24
OMe
MeO
Me
TsO
Me
2a
OH
HN
9
PPM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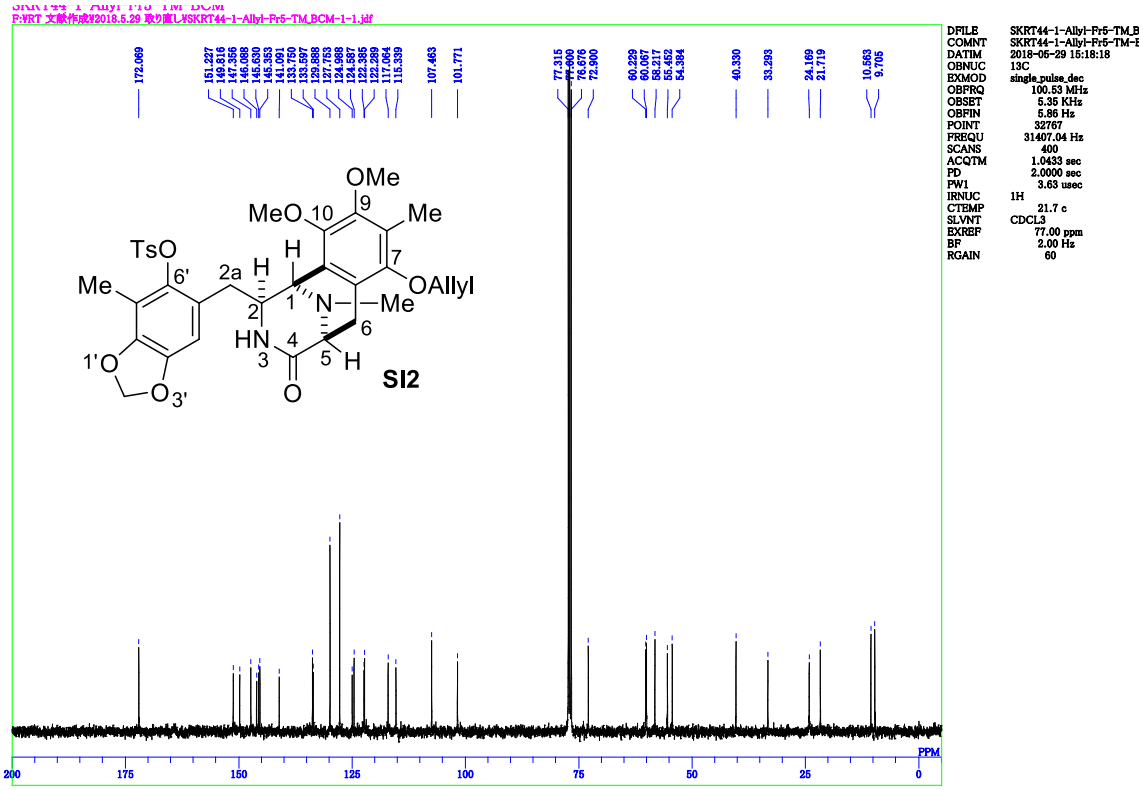
COMNT SKRT44-1-Allyl-Pr5
DATIM Sun Oct 12 14:51:40 2014
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 16
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 5.80 usec
IRNUC 1H
CTEMP 23.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.01 Hz
RGAIN 19
OMe
MeO
Me
TsO
Me
OAllyl
N
HN
O
SI2
PPM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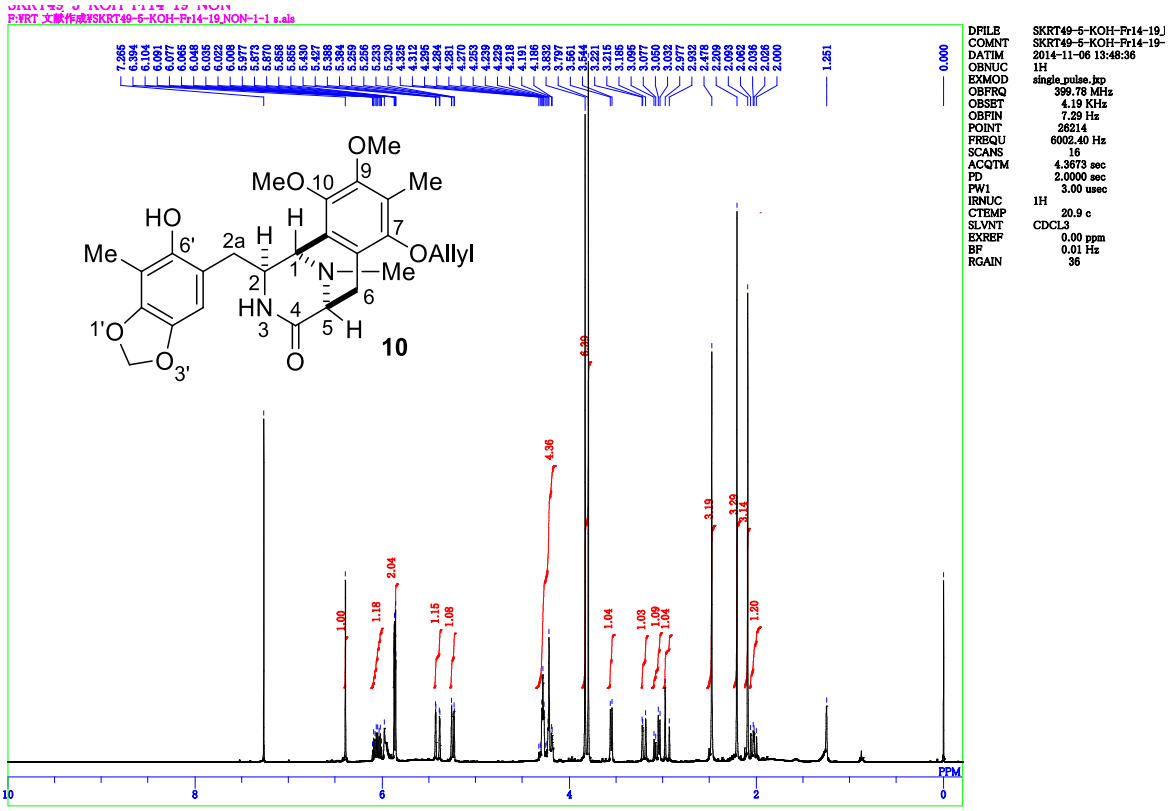
COMNT SKRT44-1-Allyl-Pr5-TM-I
DATIM 2018-05-29 15:18:18
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 32767
FREQU 31407.04 Hz
SCANS 400
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.63 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 2.00 Hz
RGAIN 60
OMe
MeO
Me
TsO
Me
OAllyl
N
HN
O
SI2
PPM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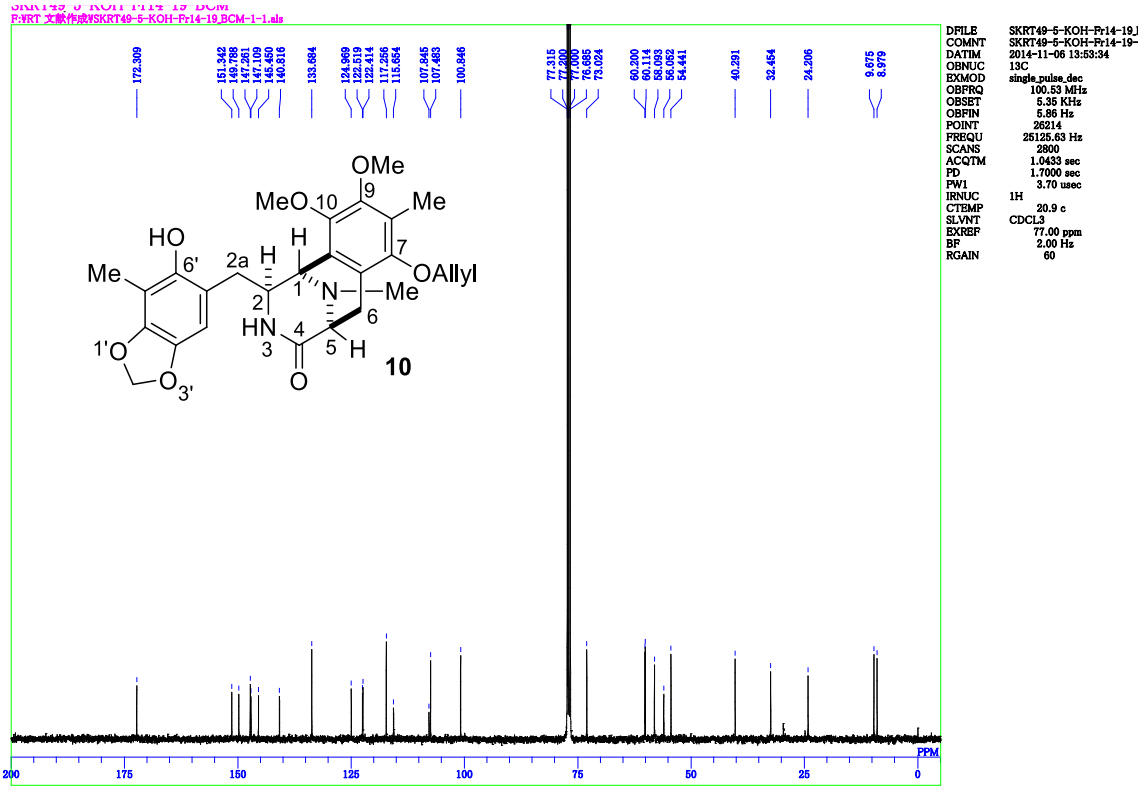
DATIM 2014-11-06 13:48:36
OBNUC 1H
EXMOD single_pulse.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 26214
FREQU 6002.40 Hz
SCANS 16
ACQTM 4.3673 sec
PD 2.0000 sec
PW1 3.00 usec
IRNUC 1H
CTEMP 20.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.01 Hz
RGAIN 36
OMe
MeO
Me
HO
Me
OAllyl
10
9
7
6'
2a
H
1''
N
2
HN
4
6
1'O
3
5
H
O
O3'
10
PPM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COMNT SKRT49-5-KOH-Pr14-19-
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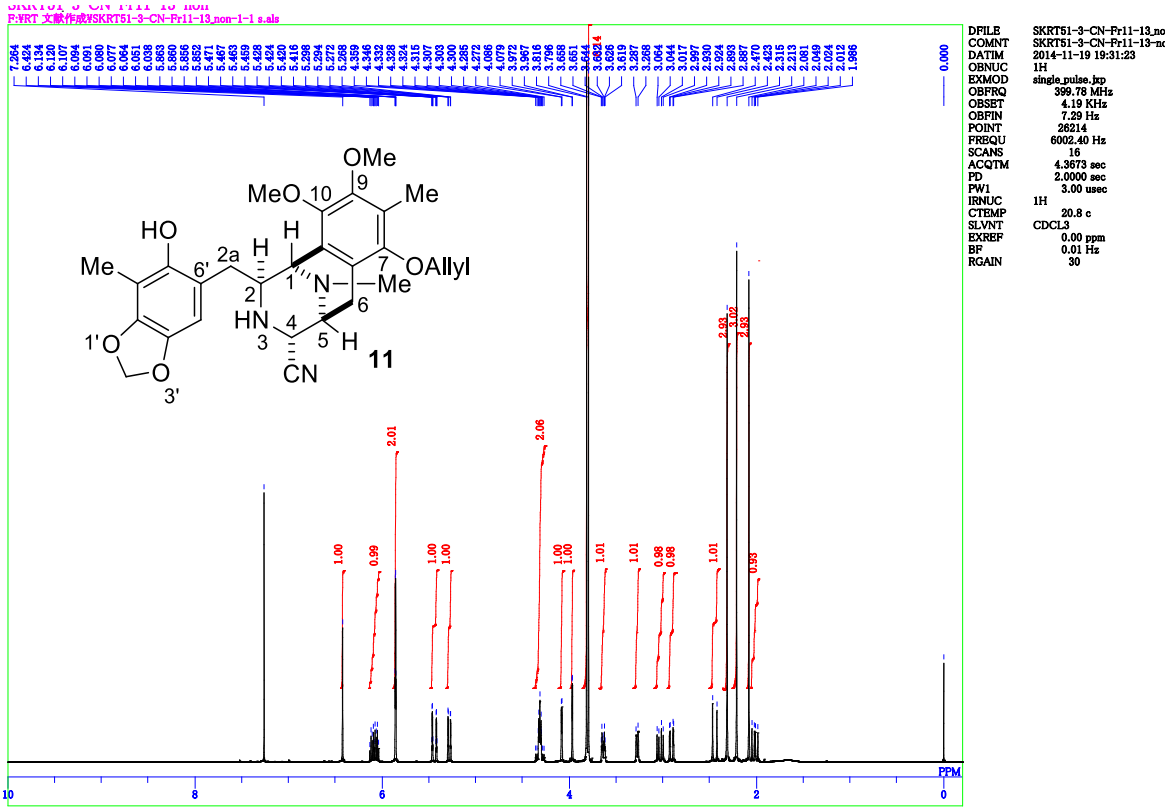
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 2800
ACQTM 1.0433 sec
PD 1.7000 sec
PW1 3.70 usec
IRNUC 1H
CTEMP 20.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 2.00 Hz
RGAIN 60
OMe
MeO
Me
HO
Me
OAllyl
10
9
7
6'
2a
H
1''
N
2
HN
4
6
1'O
3
5
H
O
O3'
10
PPM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COMNT SKRT51-3-CN-Fr11-13-no
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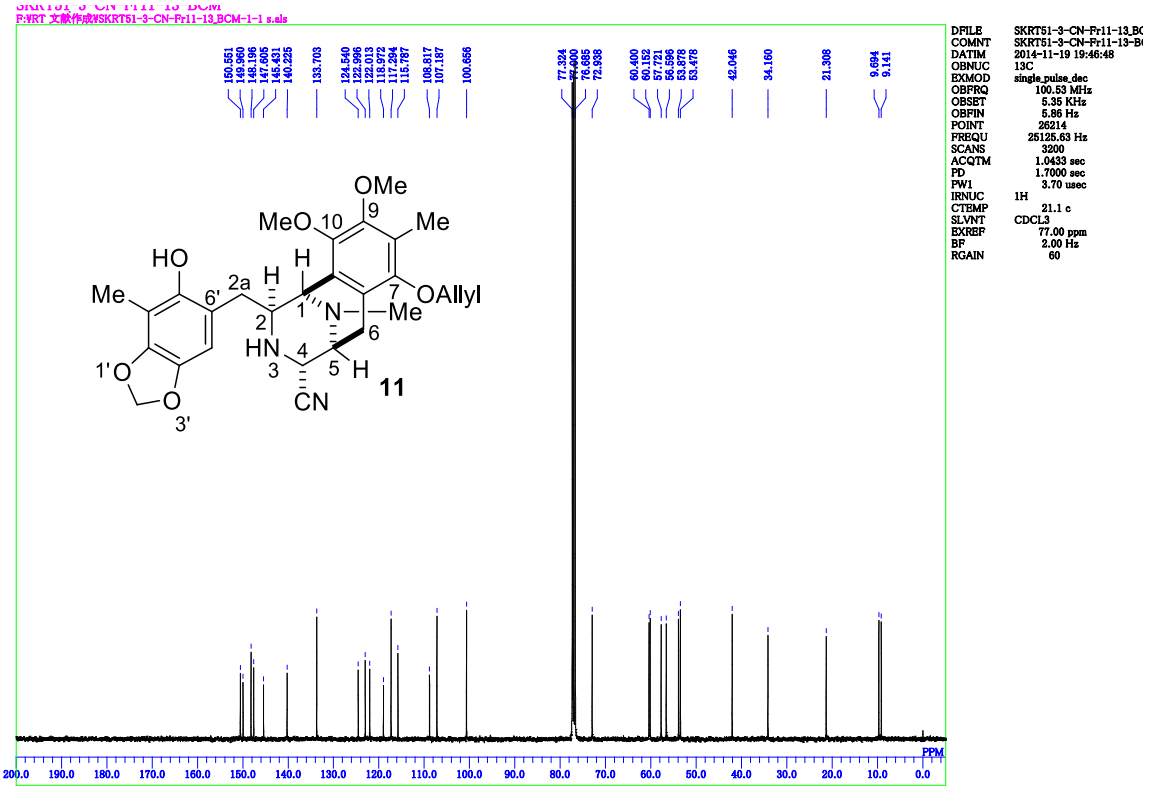
OBNUC 1H
EXMOD single_pulse.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 26214
FREQU 6002.40 Hz
SCANS 16
ACQTM 4.3673 sec
PD 2.0000 sec
PW1 3.00 usec
IRNUC 1H
CTEMP 20.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.01 Hz
RGAIN 30
OMe
MeO
Me
HO
H
Me
2a
6'
7
OAllyl
1''
N
Me
2
6
HN
3
4
5
H
1'O
O
3'
CN
11
PPM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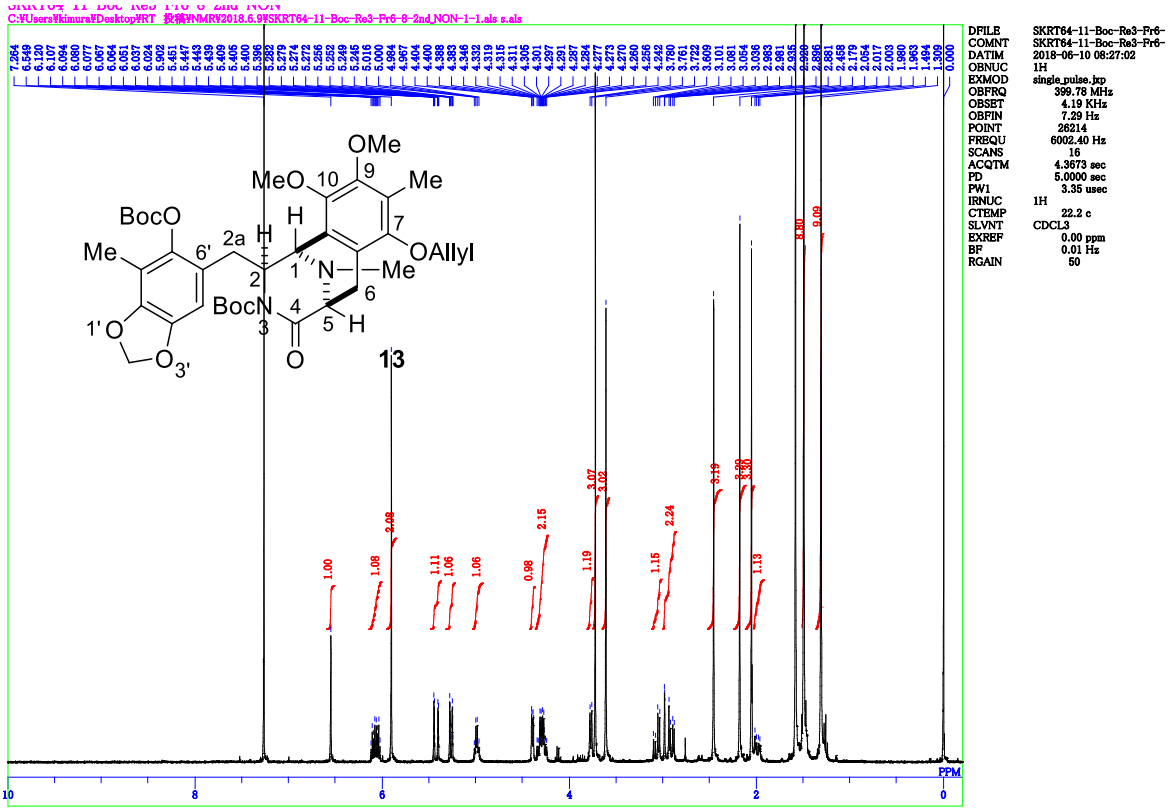
COMNT SKRT51-3-CN-Fr11-13-B
DATIM 2014-11-19 19:46:48
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 3200
ACQTM 1.0433 sec
PD 1.7000 sec
PW1 3.70 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 2.00 Hz
RGAIN 60
OMe
MeO
Me
HO
H
Me
2a
6'
7
OAllyl
1''
N
Me
2
6
HN
3
4
5
H
1'O
O
3'
CN
11
PPM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PD 5.0000 sec
PW1 3.35 usec
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL3
EXREF 0.00 ppm
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OMe
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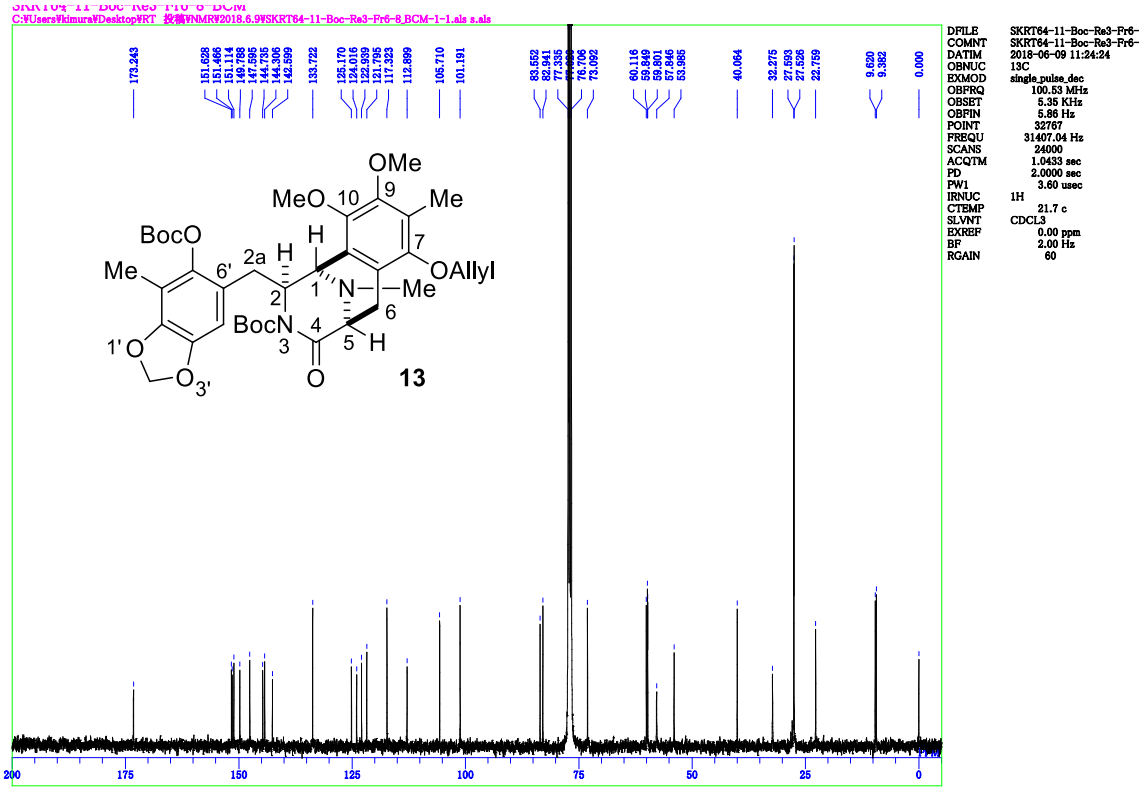
Me
BocO
H
Me
2a
OAllyl
BocN
13
PPM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PW1 3.60 usec
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CTEMP 21.7 c
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2a
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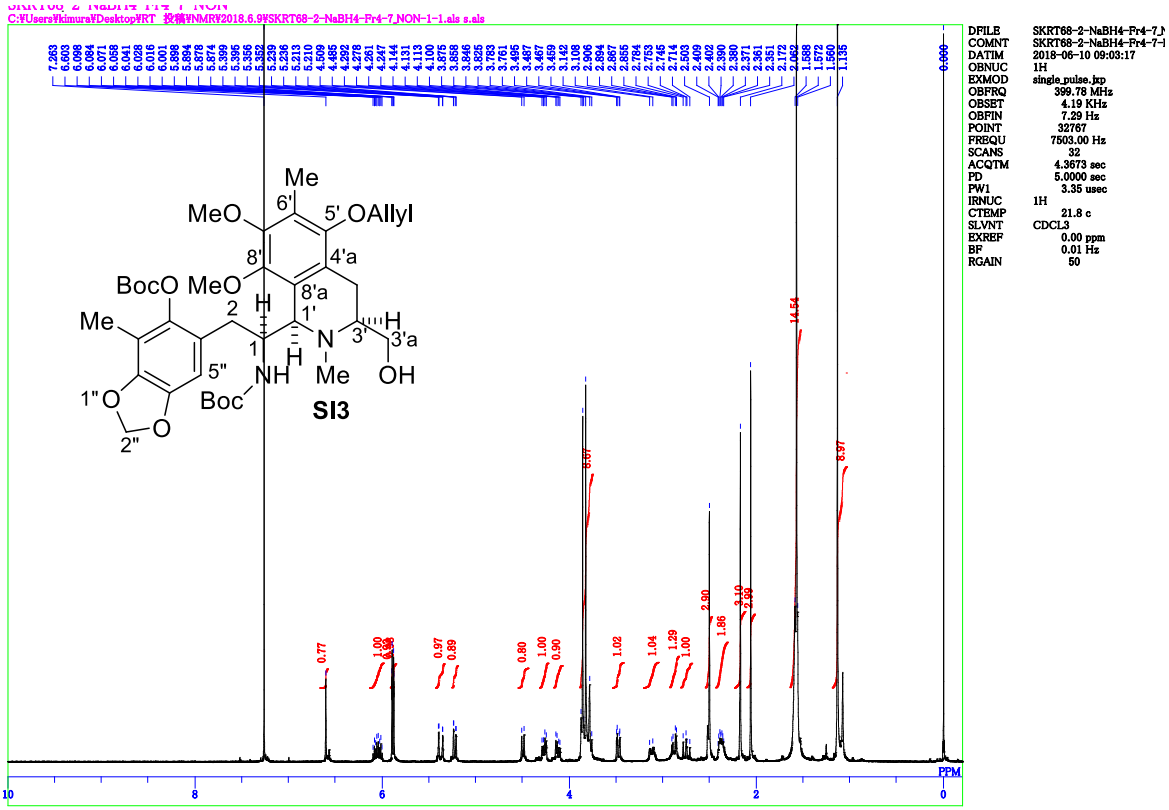
13
PPM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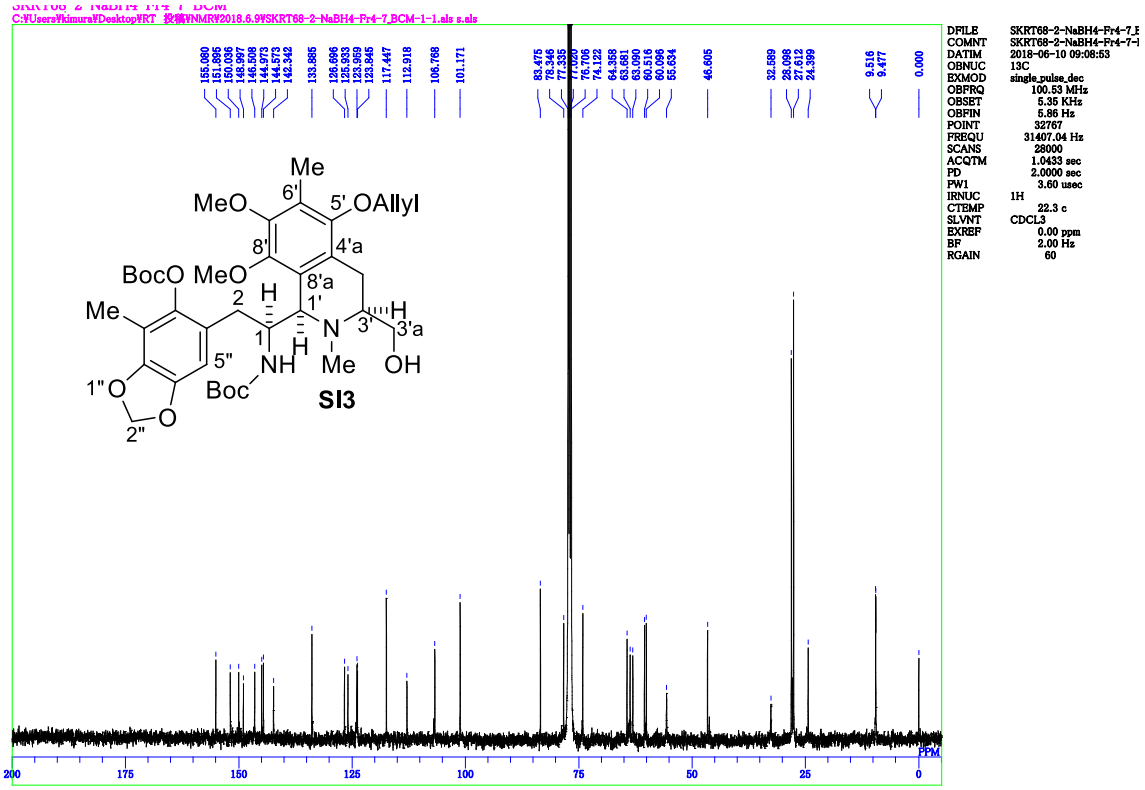
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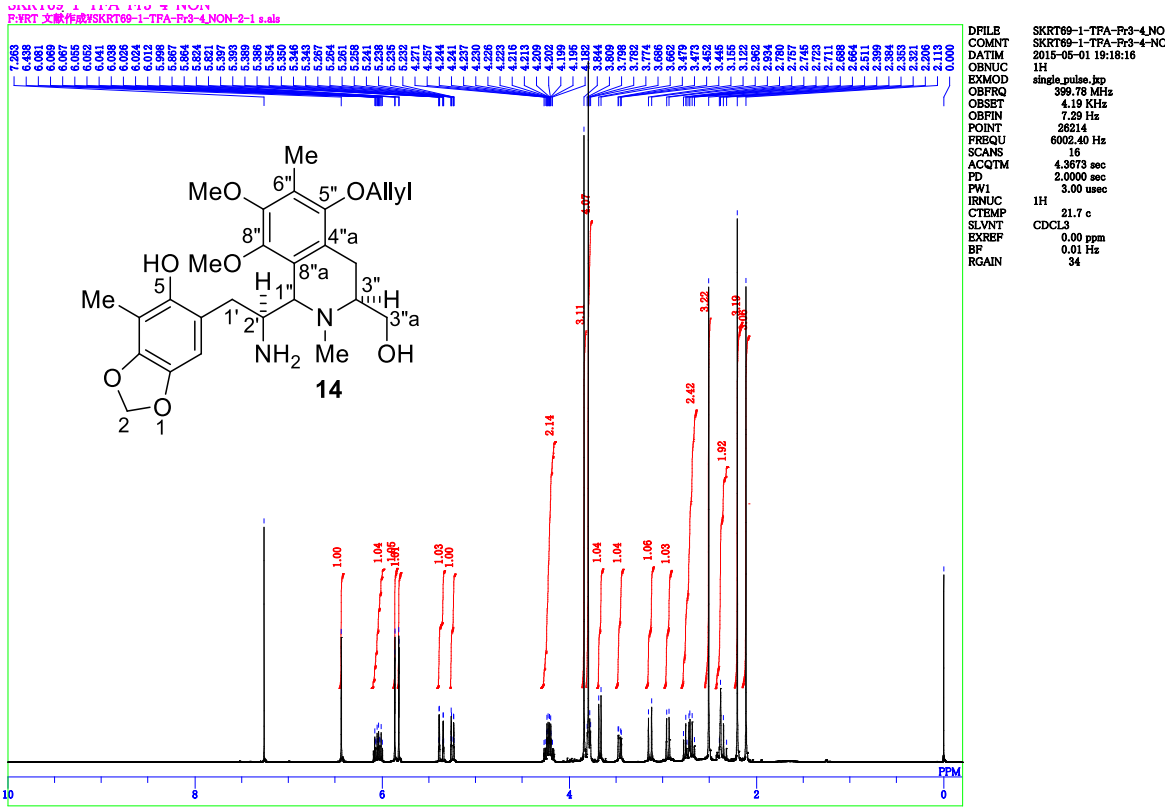
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5"
OAllyl
8"
4"a
HO
MeO
8"a
H
1"
3"
H
Me
5
1'
2'
N
3"a
NH2
Me
OH
O
O
2
1
14
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COMNT SKRT69-1-TFA-Fr3-4-NC
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POINT 26214
FREQU 6002.40 Hz
SCANS 16
ACQTM 4.3673 sec
PD 2.0000 sec
PW1 3.00 usec
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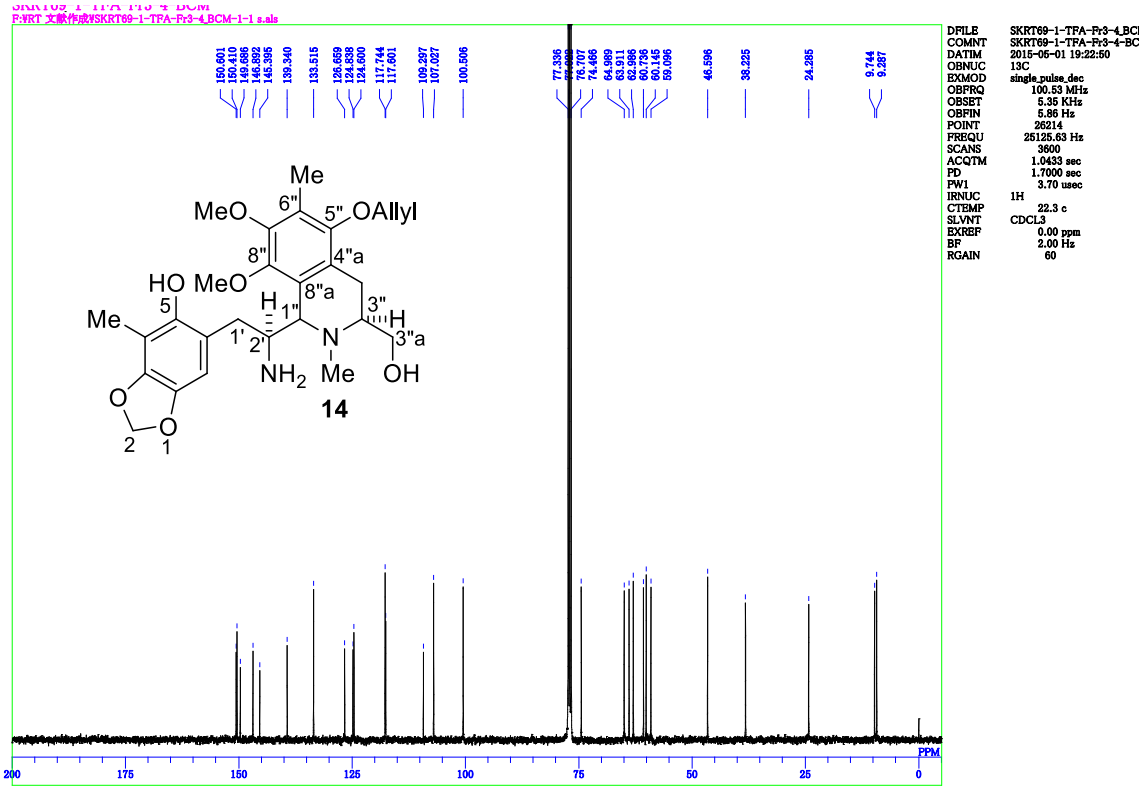
RGAIN 34
PPM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5"
OAllyl
8"
4"a
HO
MeO
8"a
H
1"
3"
H
Me
5
1'
2'
N
3"a
NH2
Me
OH
O
O
2
1
14
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COMNT SKRT69-1-TFA-Fr3-4-BC
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EXMOD single_pulse_dec
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POINT 26214
FREQU 25125.63 Hz
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PD 1.7000 sec
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RGAIN 60
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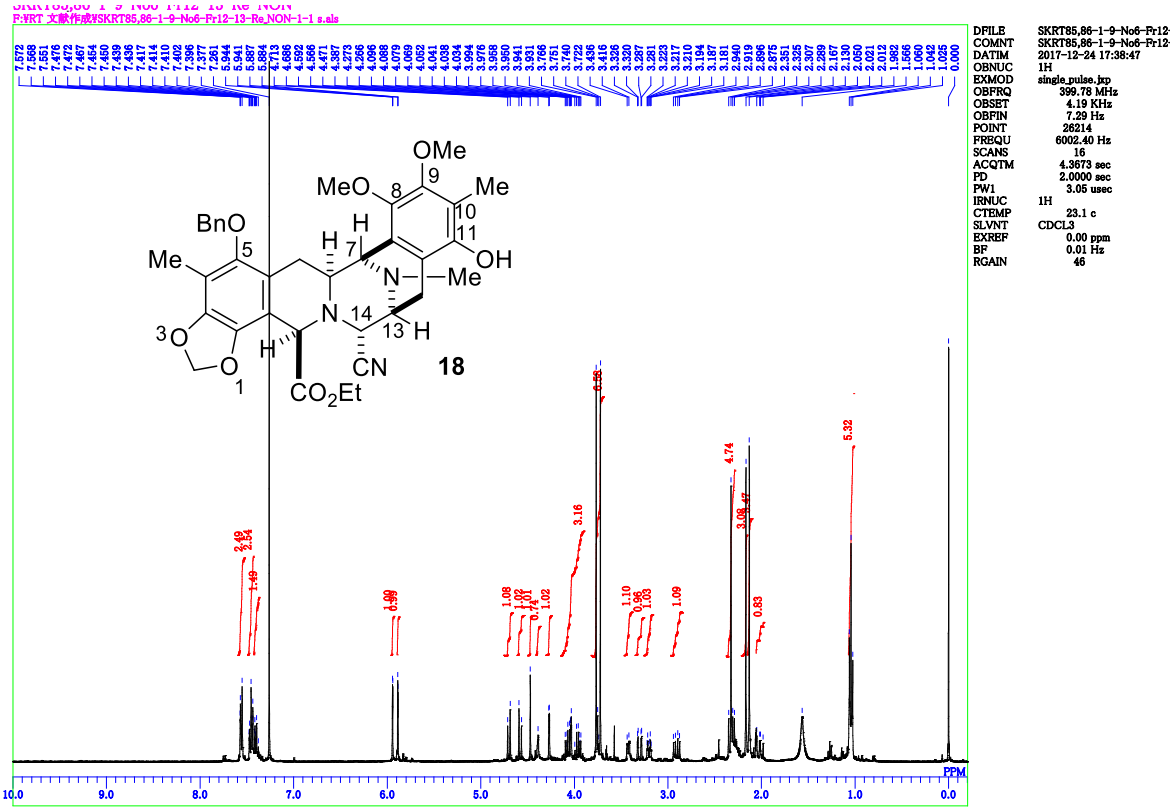
OMe
MeO
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BnO
Me
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N
H
CN
CO2Et
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EXMOD single_pulse.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 26214
FREQU 6002.40 Hz
SCANS 16
ACQTM 4.3673 sec
PD 2.0000 sec
PW1 3.05 usec
IRNUC 1H
CTEMP 23.1 c
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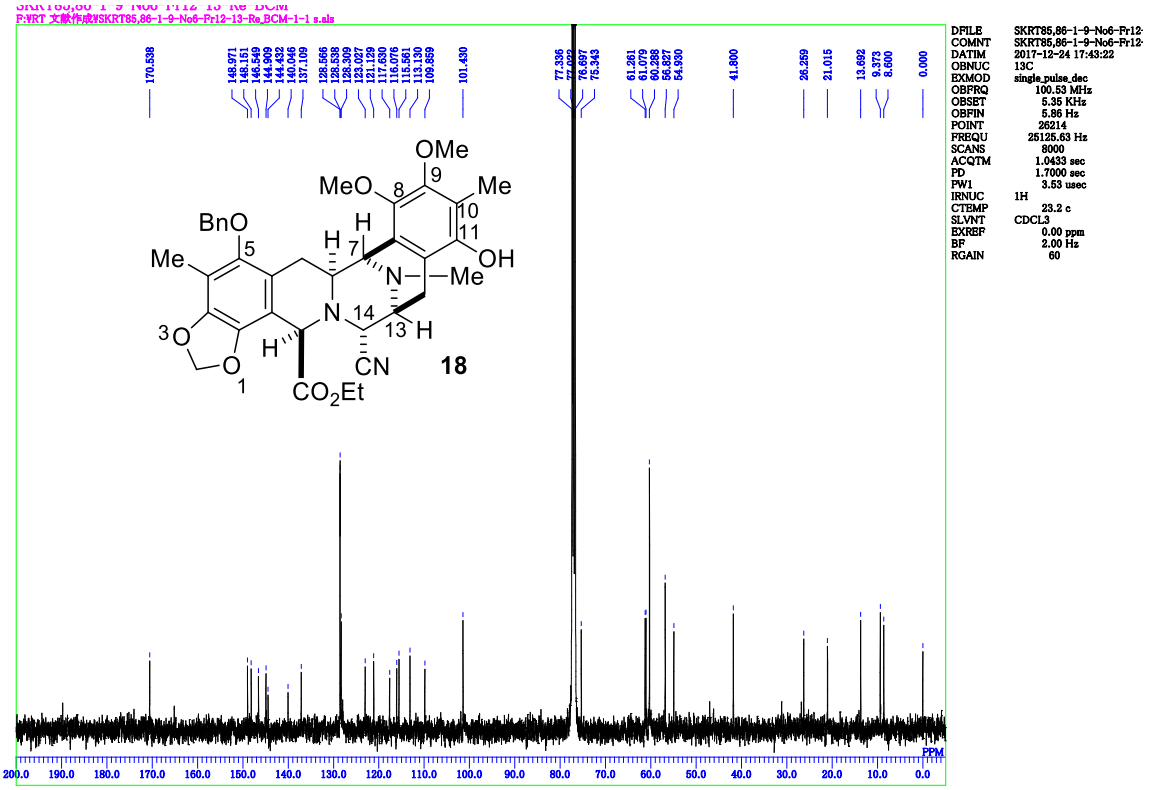
BF 0.01 Hz
RGAIN 46
PPM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Me
BnO
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Me
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H
CN
CO2Et
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OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
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ACQTM 1.0433 sec
PD 1.7000 sec
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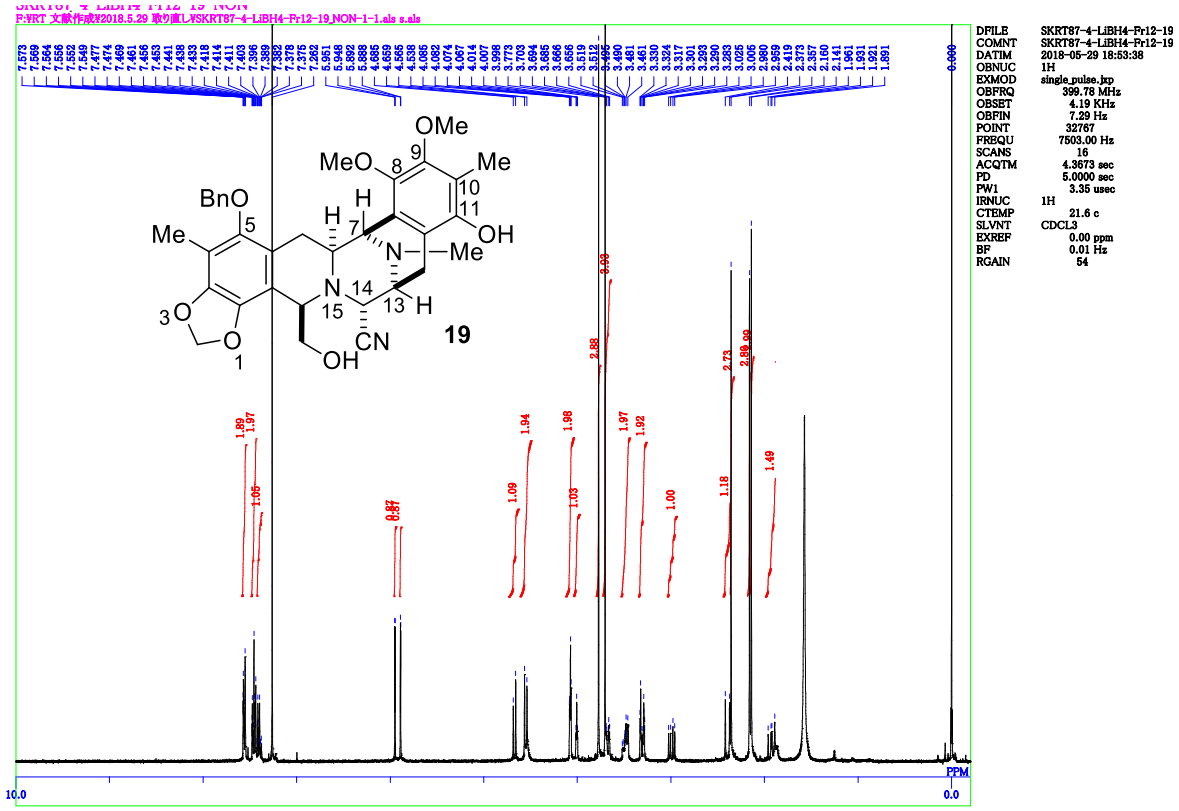
BF 2.00 Hz
RGAIN 60
PPM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EXMOD single_pulse.jxp
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OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 32767
FREQU 7503.00 Hz
SCANS 16
ACQTM 4.3673 sec
PD 5.0000 sec
PW1 3.35 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 0.00 ppm
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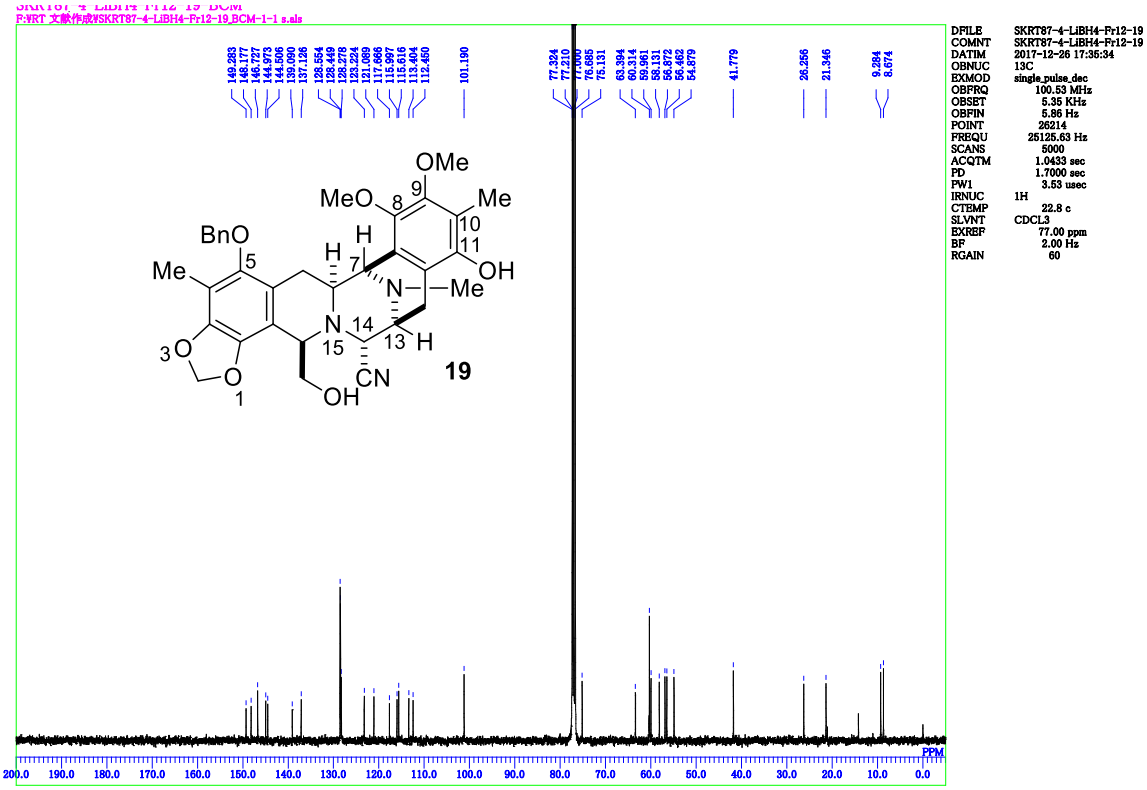
OMe
MeO
Me
BnO
H
OH
N
CN
19
PPM
10.0
0.0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EXMOD single_pulse_dec
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 5000
ACQTM 1.0433 sec
PD 1.7000 sec
PW1 3.53 usec
IRNUC 1H
CTEMP 22.8 c
SLVNT CDCL3
EXREF 77.00 ppm
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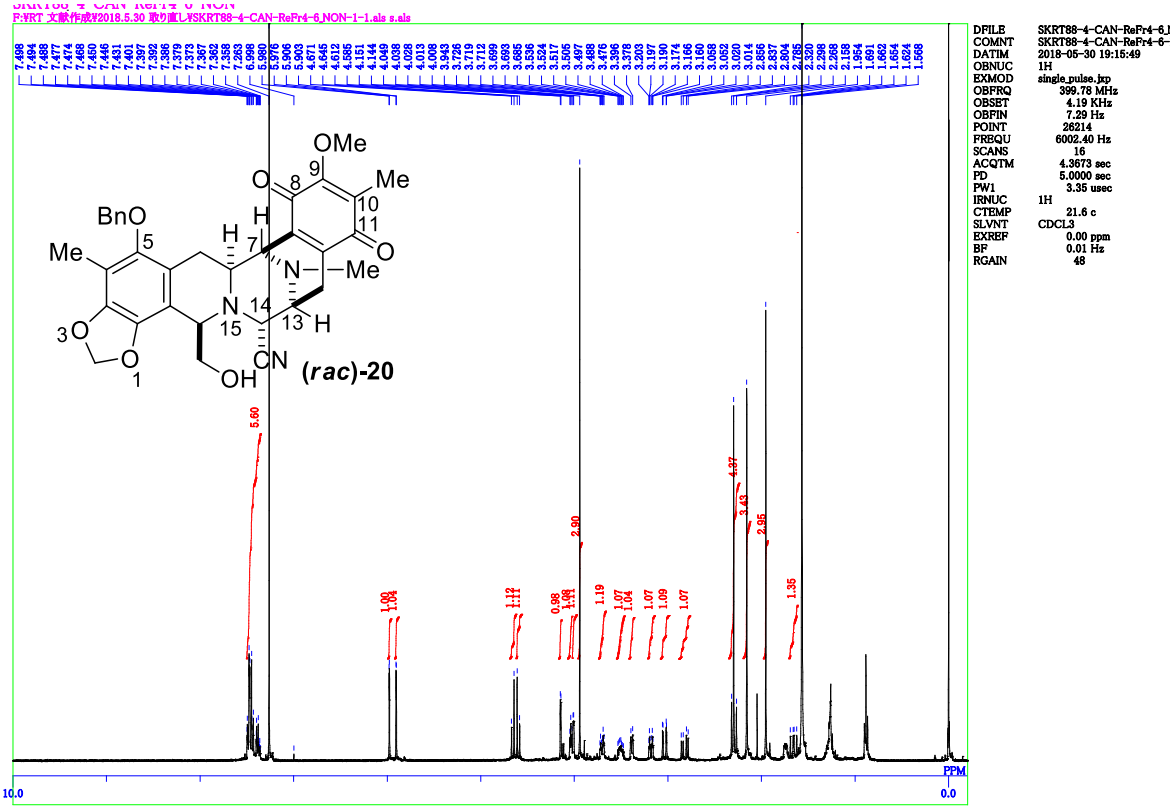
OMe
MeO
Me
BnO
H
OH
N
CN
19
PPM
200.0
0.0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OBFIN 7.29 Hz
POINT 26214
FREQU 6002.40 Hz
SCANS 16
ACQTM 4.3673 sec
PD 5.0000 sec
PW1 3.35 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.01 Hz
RGAIN 48
OMe
Me
BnO
Me
H
H
N
Me
N
H
CN
OH
(rac)-20
PPM
10.0
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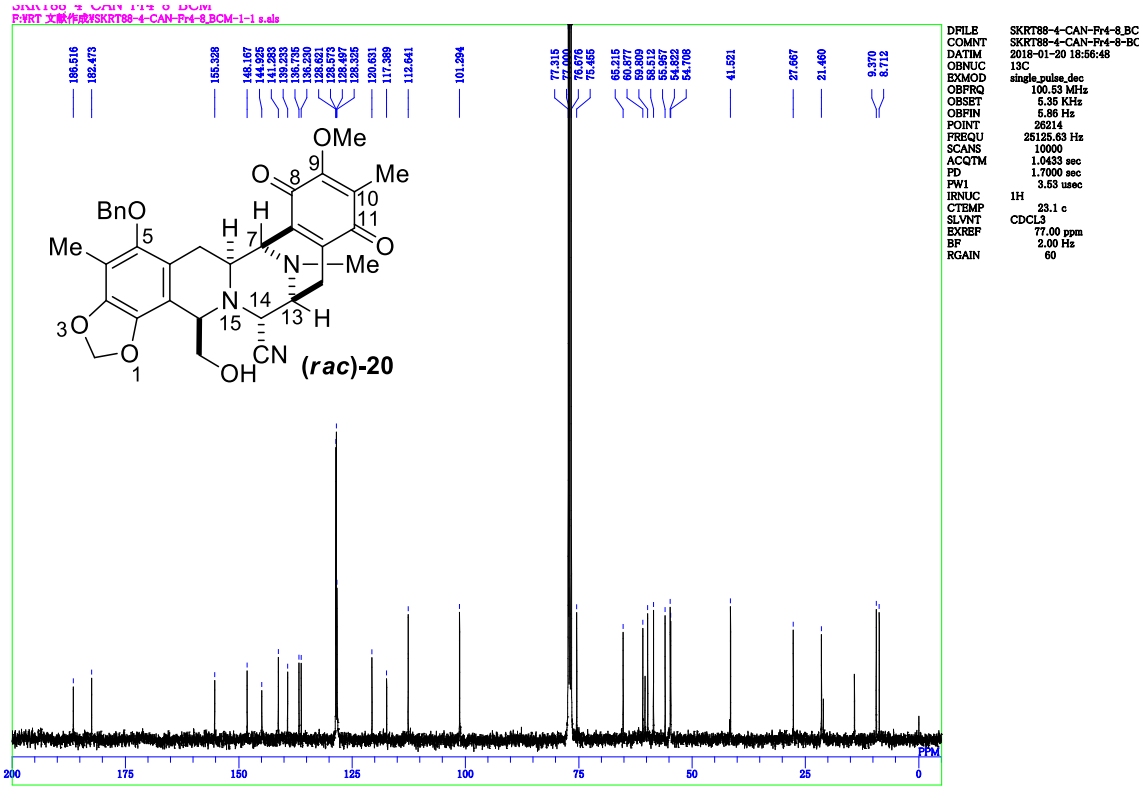
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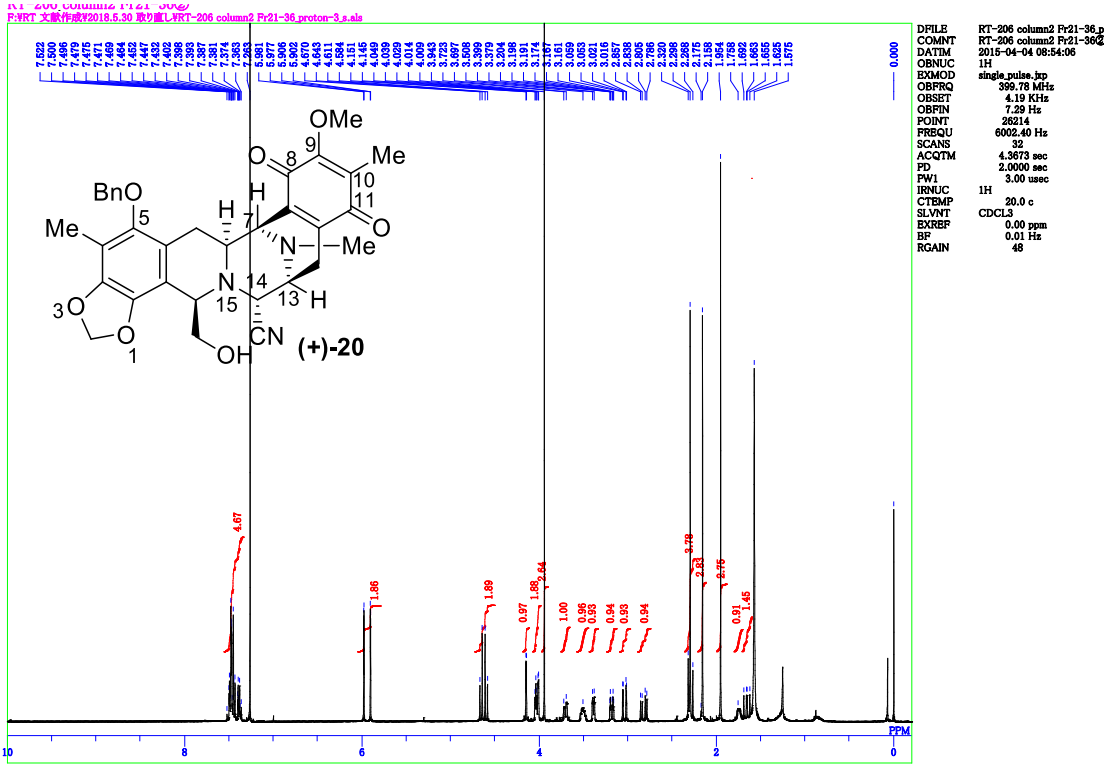
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 10000
ACQTM 1.0433 sec
PD 1.7000 sec
PW1 3.53 usec
IRNUC 1H
CTEMP 23.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 2.00 Hz
RGAIN 60
OMe
Me
BnO
Me
H
H
N
Me
N
H
CN
OH
(rac)-20
PPM
200
175
150
125
100
75
50
25
0

Authentic sample **(+)-20** : M. Yokoya *et al*., *J. Org. Chem.* **2016**, *81*, 4039-4047.



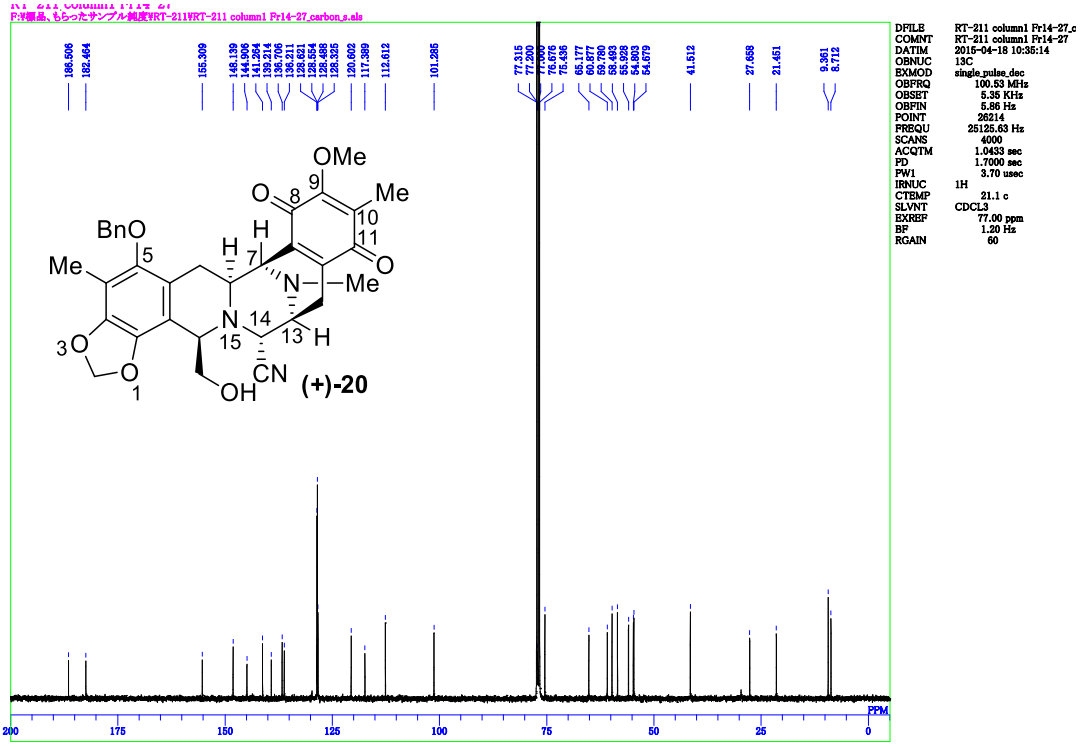
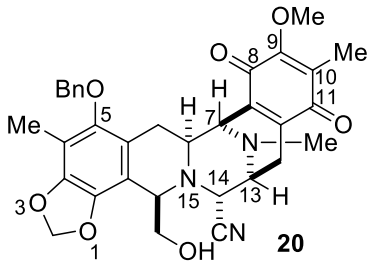


Table 1. Comparison of ^{1}H and ^{13}C NMRassignment for (*rac*)-**20** and (+)-**20**

Atom No.	^{13}C NMR, δ, multi.			^{1}H NMR, δ (multi., integral, J in Hz)			
	(*rac*)-**20**	(+)-**20**		(*rac*)-**20**		(+)-**20**	
2	101.3	101.3	t	5.90	(d, 1H, 1.4)	5.90	(d, 1H, 1.1)
				5.98	(d, 1H, 1.4)	5.98	(d, 1H, 1.1)
3a	144.9	144.9	s				
4	112.6[a]	112.6[a]	s				
5	148.2	148.1	s				
5a	120.6	120.6	s				
6	27.7	27.7	t	α 3.04	(dd, 1H, 15.1, 2.7)	3.04	(dd, 1H, 15.1, 2.3)
				β 1.66	(dd, 1H, 15.1, 12.1)	1.66	(dd, 1H, 15.1, 12.0)
6a	56.0	55.9	d	3.18	(dt, 1H, 12.1, 2.7)	3.18	(dt, 1H, 12.0, 2.3)
7	54.7 or 54.8	54.7 or 54.8	d	4.01	(br d, 1H, 2.7)	4.01	(br d, 1H, 2.3)
7a	136.2	136.2	s				
8	182.5	182.5	s				
9	155.3	155.3	s				
10	128.57	128.6	s				
11	186.5	186.5	s				
11a	141.3	141.3	s				
12	21.5	21.5	t	α 2.82	(dd, 1H, 20.8, 7.6)	2.82	(dd, 1H, 21.0, 7.5)
				β 2.29	(d, 1H, 20.8)	2.29	(d, 1H, 21.0)
13	54.7 or 54.8	54.7 or 54.8	d	3.39	(br d, 1H, 7.6)	3.39	(dd, 1H, 7.5, 2.5)
14	59.8	59.8	d	4.15	(d, 1H, 2.5)	4.15	(d, 1H, 2.5)
16	58.5	58.5	d	4.04	(t, 1H, 4.3)	4.04	(t, 1H, 4.2)
16a	112.6[a]	112.6[a]	s				
16b	139.2	139.2	s				
4-CH_3	9.4	9.4	q	2.16	(s, 3H)	2.16	(s, 3H)
10-CH_3	8.7	8.7	q	1.95	(s, 3H)	1.95	(s, 3H)
9-OCH_3	60.9	60.9	q	3.94	(s, 3H)	3.94	(s, 3H)
C_6H_5	136.7	136.7	s	7.50-7.36	(m, 5H)	7.50-7.36	(m, 5H)
	128.62	128.6	d				
	128.5	128.5	d				
	128.3	128.3	d				
CN	117.4	117.4	s				
$PhCH_2$	75.5	75.4	t	4.60	(d, 1H, 10.6)	4.60	(d, 1H, 10.8)
				4.66	(d, 1H, 10.6)	4.66	(d, 1H, 10.8)
CH_2OH	65.2	65.2	t	3.71	(br d, 1H, 10.8)	3.71	(br d, 1H, 10.9)
				3.51	(m, 1H)	3.54-3.48	(m, 1H)
NCH_3	41.5	41.5	q	2.30	(s, 3H)	2.30	(s, 3H)

[a] overlapped.



20