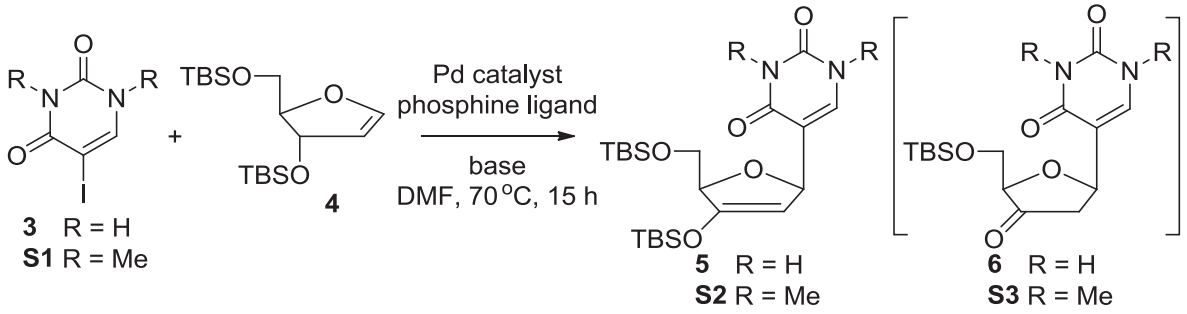
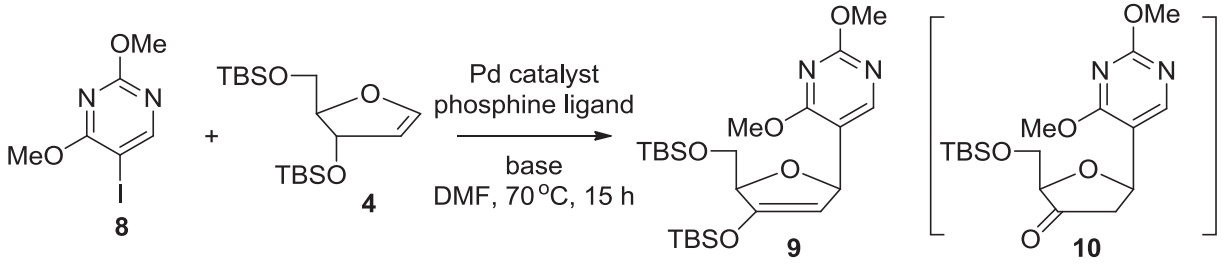
Table S1. Optimization for the Heck reaction of 5-iodouracil (**3**) with glycal **4**



entry[a]	aryl iodide	Pd catalyst (0.1 equiv)	phosphine ligand (0.2 equiv)	base (1.5 equiv)	yield (%)[b,c]
1	**S1**	$Pd(OAc)_2$	PPh_3	Et_3N	44 (22)
2	**S1**	$Pd(OAc)_2$	XantPhos	Et_3N	N.R.
3	**S1**	$Pd(OAc)_2$	DPPF	Et_3N	N.R.
4	**S1**	$Pd(OAc)_2$	DPPE	Et_3N	38 (13)
5	**S1**	Pd_2dba_3	PPh_3	Et_3N	69
6	**S1**	Pd_2dba_3	XantPhos	Et_3N	83
7	**S1**	Pd_2dba_3	DPPF	Et_3N	62
8	**S1**	Pd_2dba_3	DPPE	Et_3N	N.R.
9	**S1**	$Pd(PPh_3)_4$	-----	Et_3N	36
10	**3**	$Pd(OAc)_2$	PPh_3	Et_3N	33 (6)
11	**3**	Pd_2dba_3	PPh_3	Et_3N	35
12	**3**	Pd_2dba_3	XantPhos	Et_3N	35 (2)
13	**3**	Pd_2dba_3	XantPhos	DIPEA	65
14	**3**	Pd_2dba_3	XantPhos	DBU	N.R.
15	**3**	Pd_2dba_3	XantPhos	DABCO	N.R.
16	**3**	Pd_2dba_3	XantPhos	*n*-Bu_3N	83

[a] reagents and conditions: glycal **4** (1.0 equiv), aryl iodide (**3** or **S1**: 2.0 equiv), catalyst (0.1 equiv), ligand (0.2 equiv), base (1.5 equiv), reaction concentration = 0.1 M. The reactions were conducted on 0.5 mmol scale; [b] Isolated yield (**5** or **S2**, respectively); [c] Numbers in parentheses represent the yield of the ketone product (**6** or **S3**, respectively)

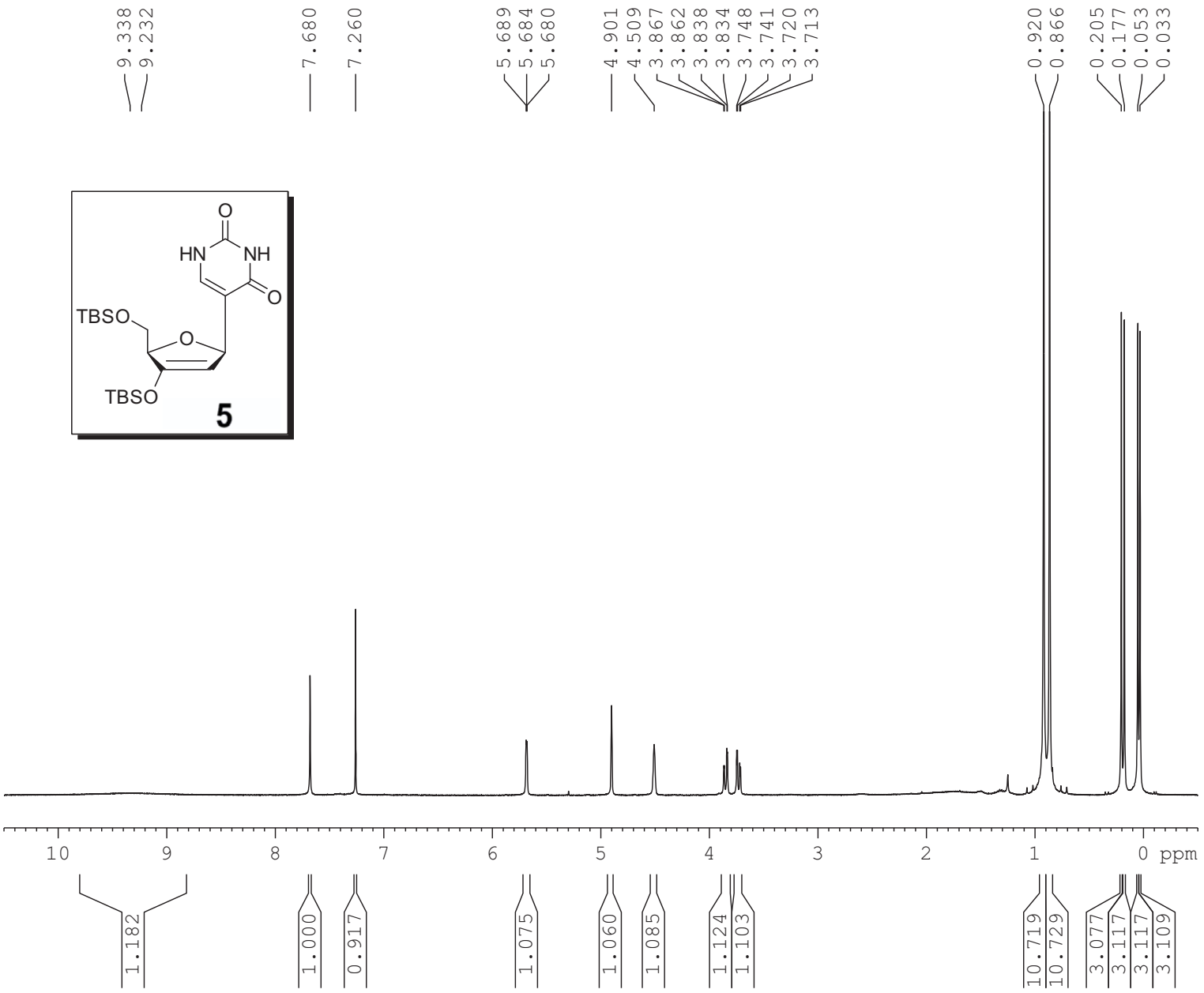
Table S2. Optimization for the Heck reaction of 2,4-dimethoxy-5-iodopyrimidine (**8**) with glycal **4**



entry[a]	aryl iodide	Pd catalyst (0.1 equiv)	phosphine ligand (0.2 equiv)	base (1.5 equiv)	yield (%)[b,c]
1	**8**	$Pd(OAc)_2$	PPh_3	Et_3N	7 (83)
2	**8**	Pd_2dba_3	PPh_3	Et_3N	77 (trace)
3	**8**	Pd_2dba_3	XantPhos	Et_3N	trace
4	**8**	Pd_2dba_3	PPh_3	*n*-Bu_3N	76 (trace)
5	**8**	Pd_2dba_3	XantPhos	*n*-Bu_3N	trace
6	**8**	$Pd(OAc)_2$	XantPhos	Et_3N	6 (71)
7	**8**	Pd_2dba_3	PPh_3	Ag_2CO_3	N.R.

[a] reagents and conditions: glycal **4** (1.0 equiv), aryl iodide **8** (2.0 equiv), catalyst (0.1 equiv), ligand (0.2 equiv), base (1.5 equiv), reaction concentration = 0.1 M. The reactions were conducted on 0.5 mmol scale; [b] Isolated yield (**9**); [c] Numbers in parentheses represent the yield of the ketone product **10**

1H



Current Data Parameters
NAME TD003-1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
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Time 22.19
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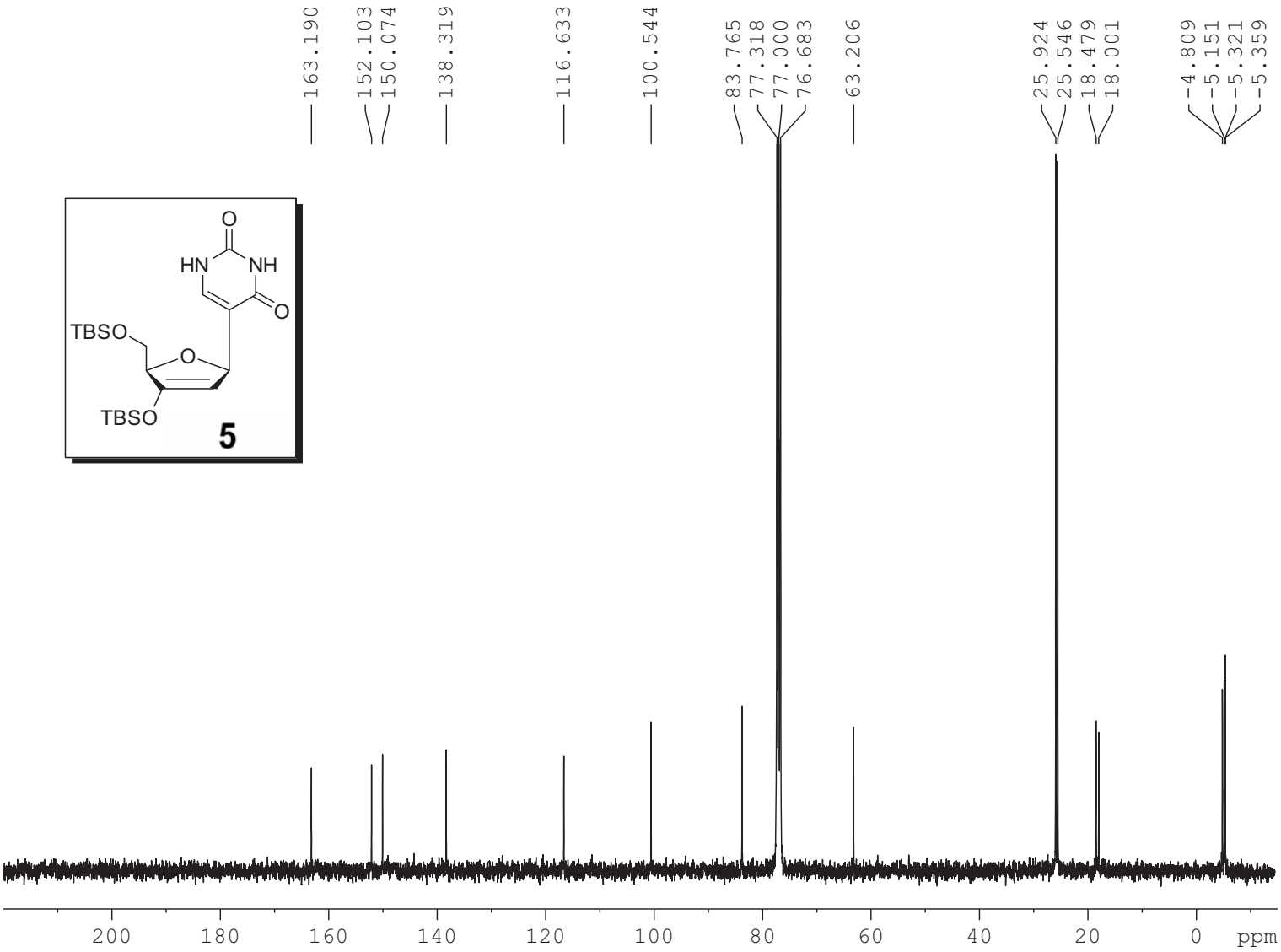
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 12
DS 0
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719147 sec
RG 99.72
DW 69.333 usec
DE 10.50 usec
TE 297.6 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 12.90 usec
PLW1 15.00000000 W

F2 - Processing parameters
SI 16384
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

13C



Current Data Parameters
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EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
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PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2000
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SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 298.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
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NUC1 13C
P1 10.00 usec
PLW1 47.50000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 15.00000000 W
PLW12 0.33750001 W
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F2 - Processing parameters
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GB 0
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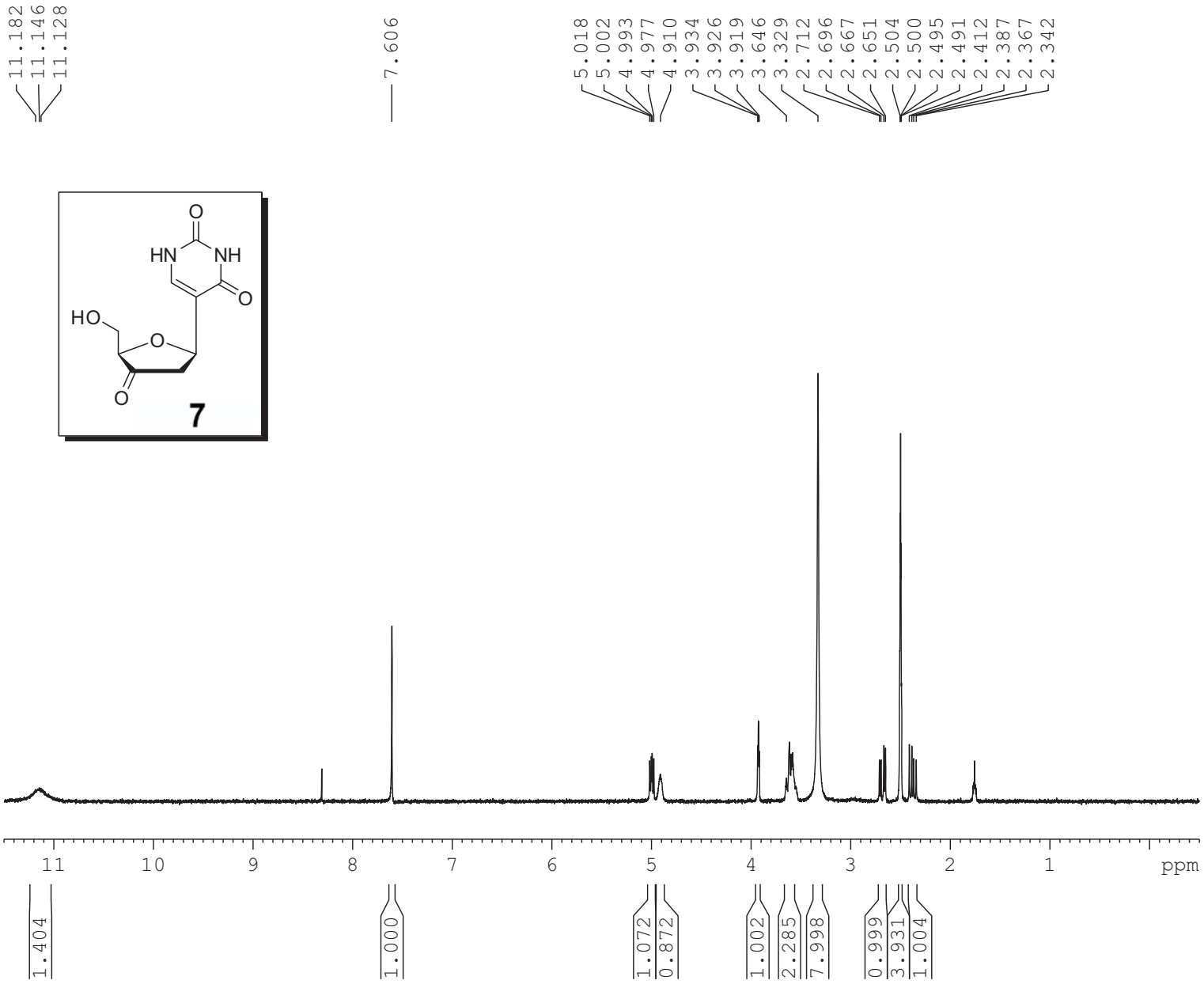
1H



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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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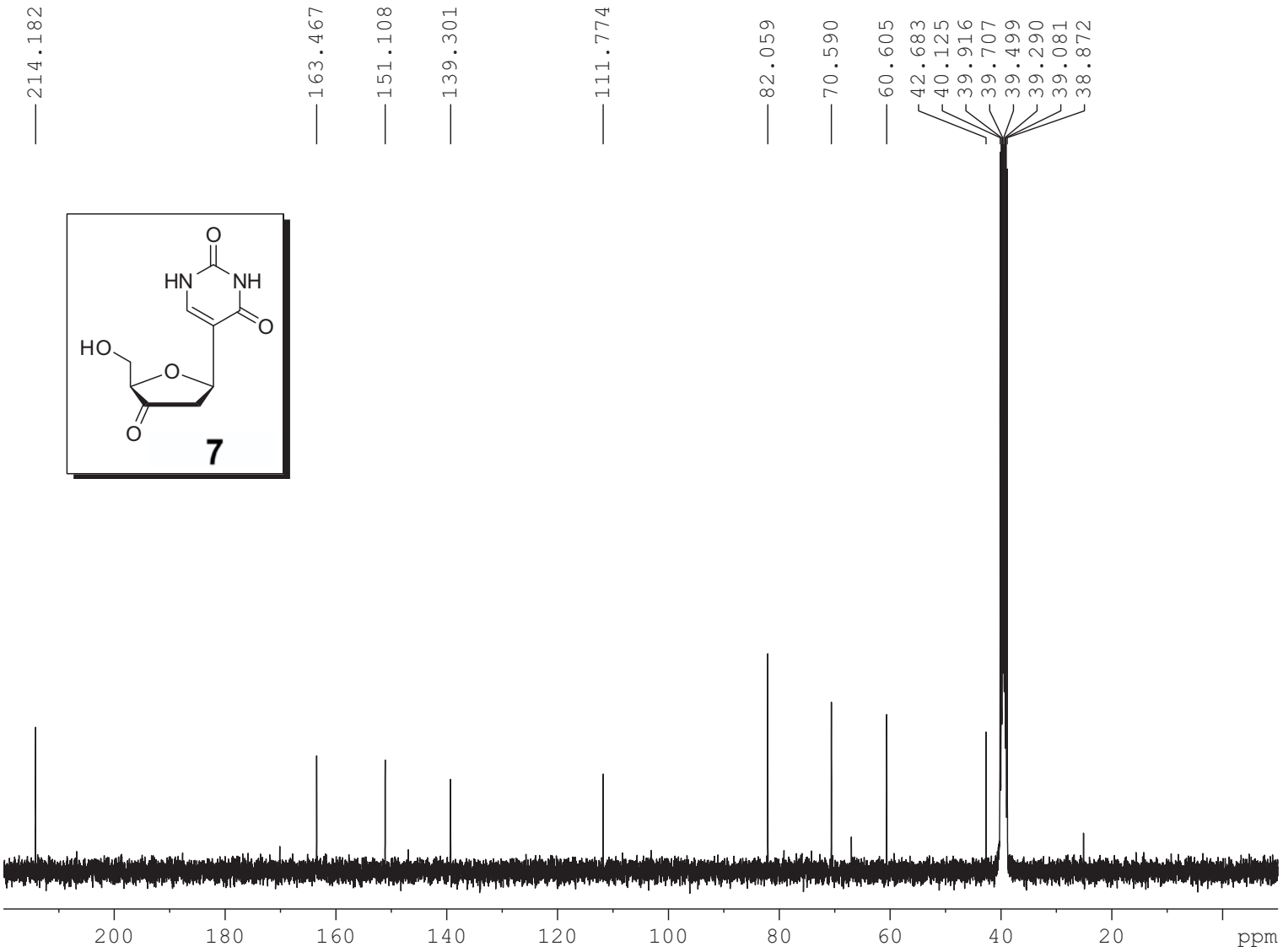
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 1
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2609921 sec
RG 256
DW 69.000 usec
DE 6.50 usec
TE 297.2 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.40 usec
PL1 1.80 dB
SFO1 400.1324008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300027 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

13C



Current Data Parameters
NAME TD031
EXPNO 3
PROCNO 1

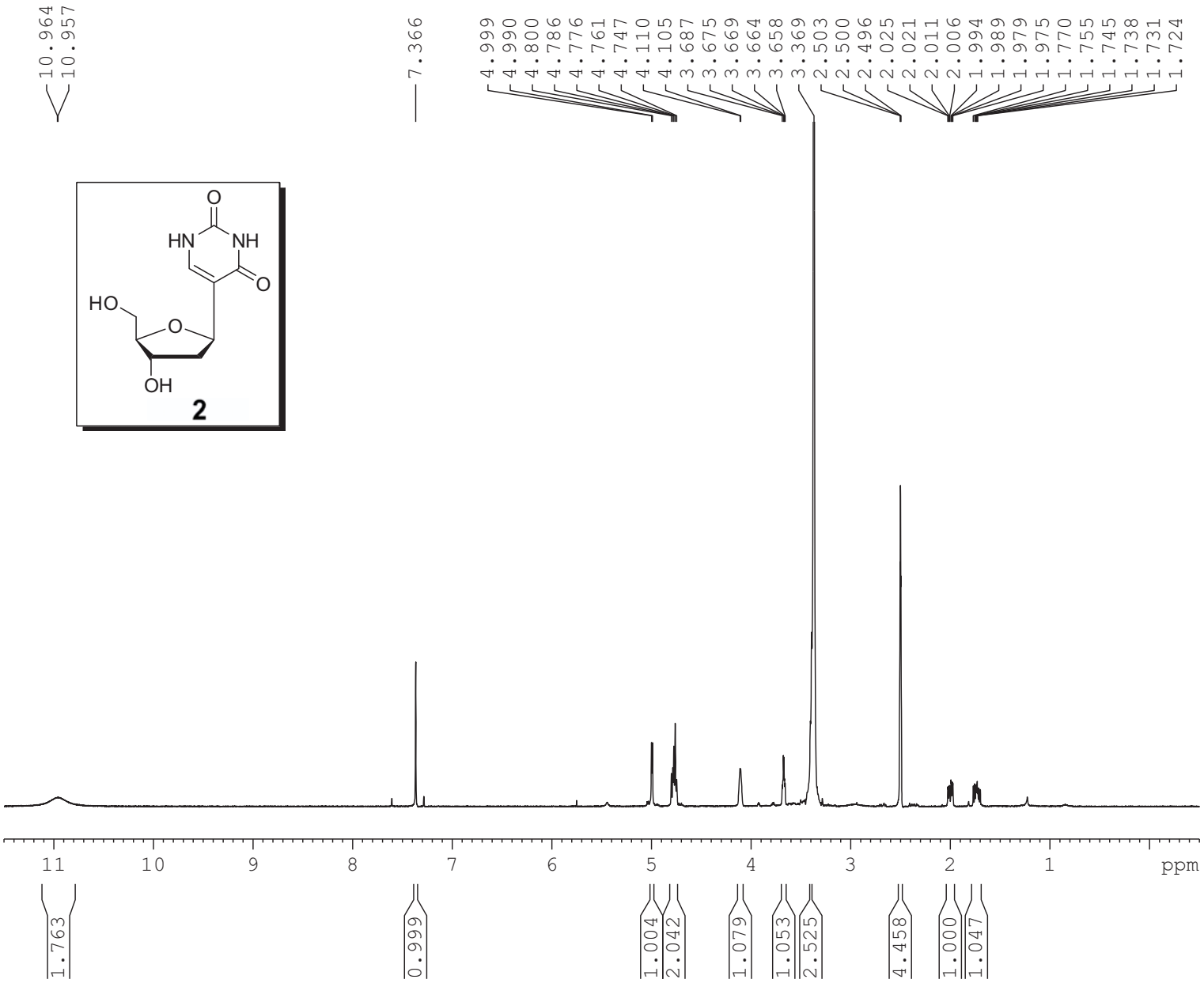
F2 - Acquisition Parameters
Date_ 20160713
Time 23.58
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 2000
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 8192
DW 20.800 usec
DE 6.50 usec
TE 297.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.80 dB
PL12 17.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6128194 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1H



Current Data Parameters
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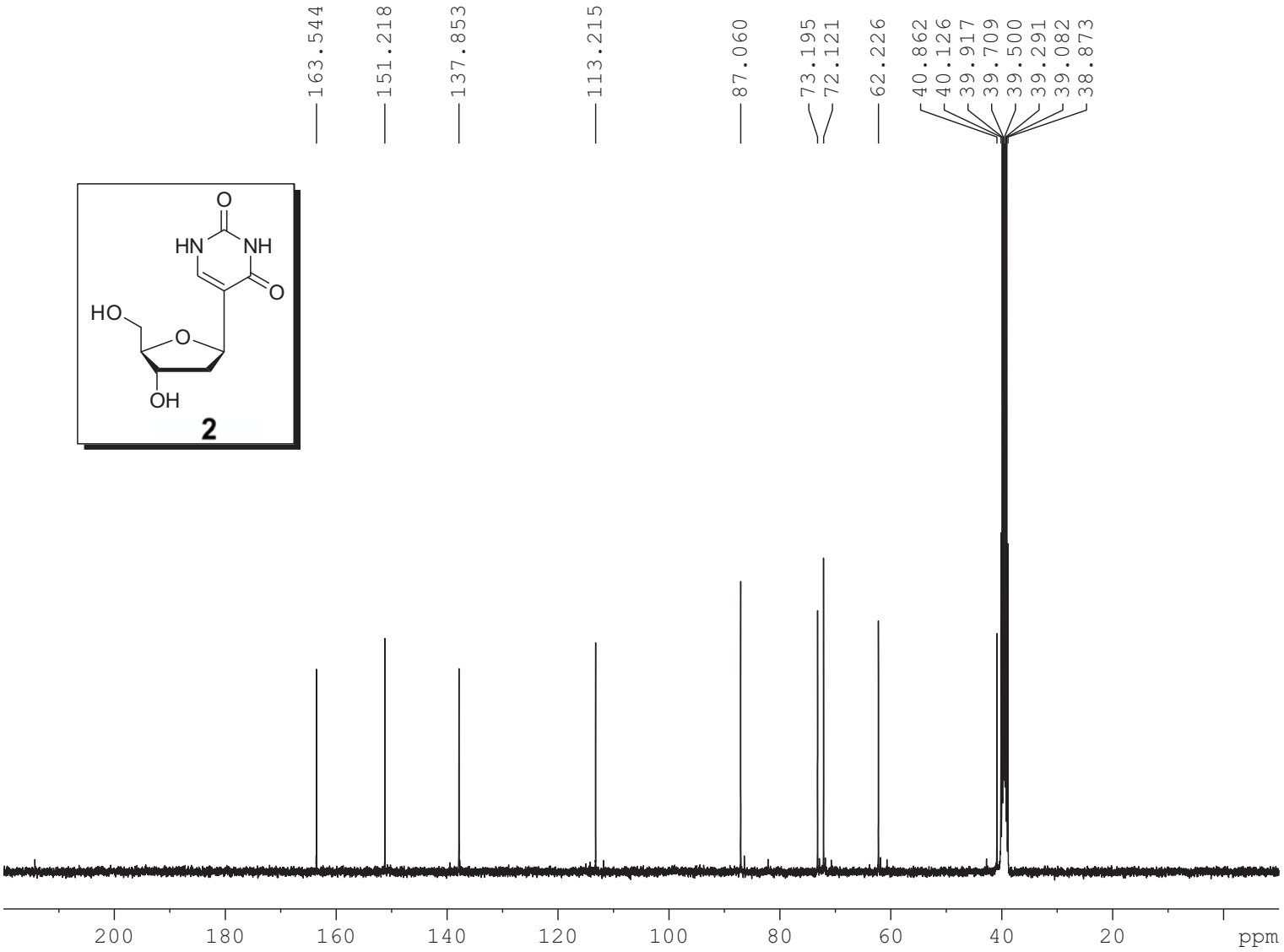
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160115
Time 20.54
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 12
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2609921 sec
RG 228.1
DW 69.000 usec
DE 6.50 usec
TE 297.5 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.40 usec
PL1 1.80 dB
SFO1 400.1324008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300030 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

13C



Current Data Parameters
NAME TD010
EXPNO 6
PROCNO 1

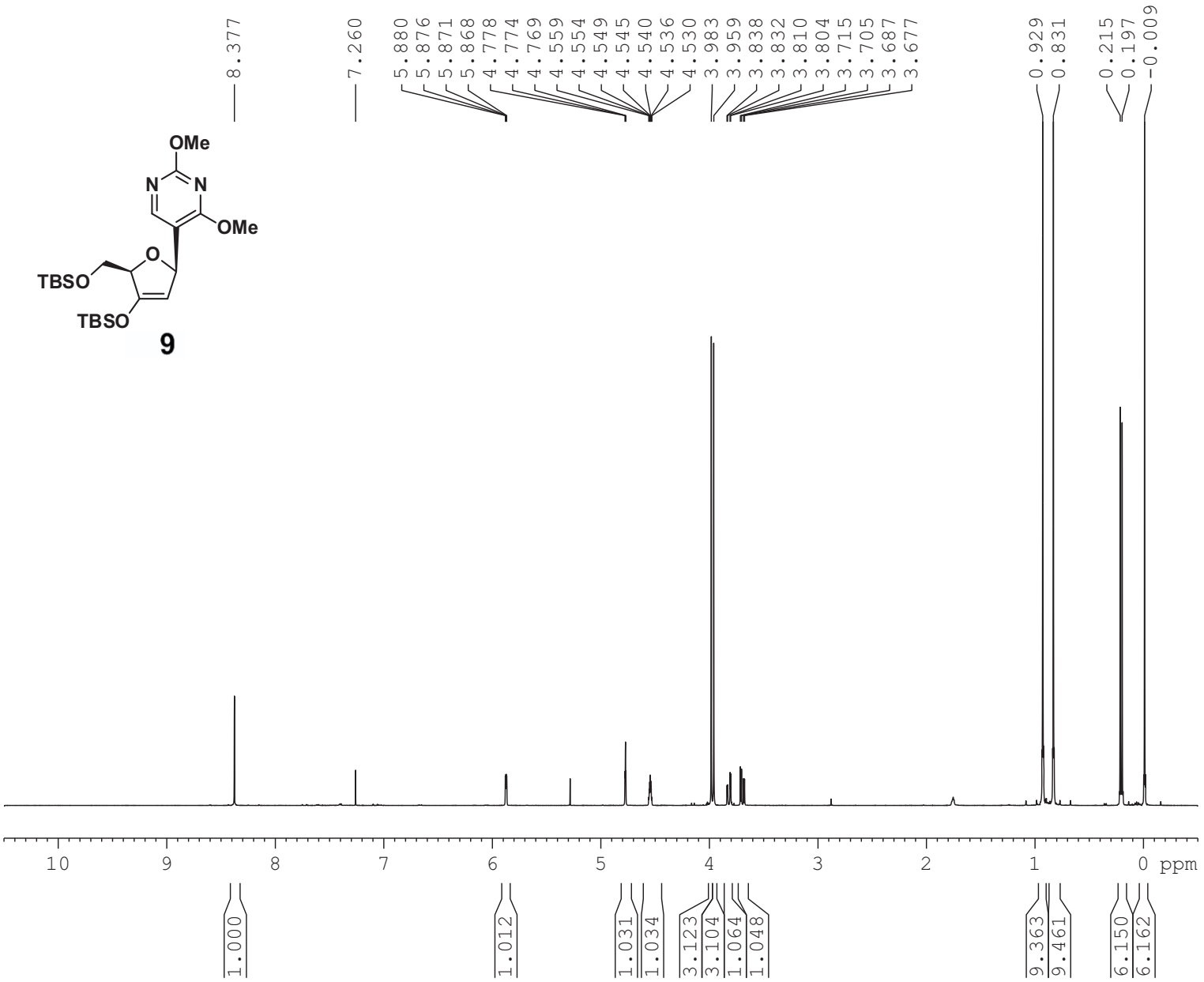
F2 - Acquisition Parameters
Date_ 20160121
Time 1.20
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 3000
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 8192
DW 20.800 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.80 dB
PL12 17.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6128092 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1H of P data



Current Data Parameters
NAME TQ111
EXPNO 101
PROCNO 1

F2 - Acquisition Parameters
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Time 17.19
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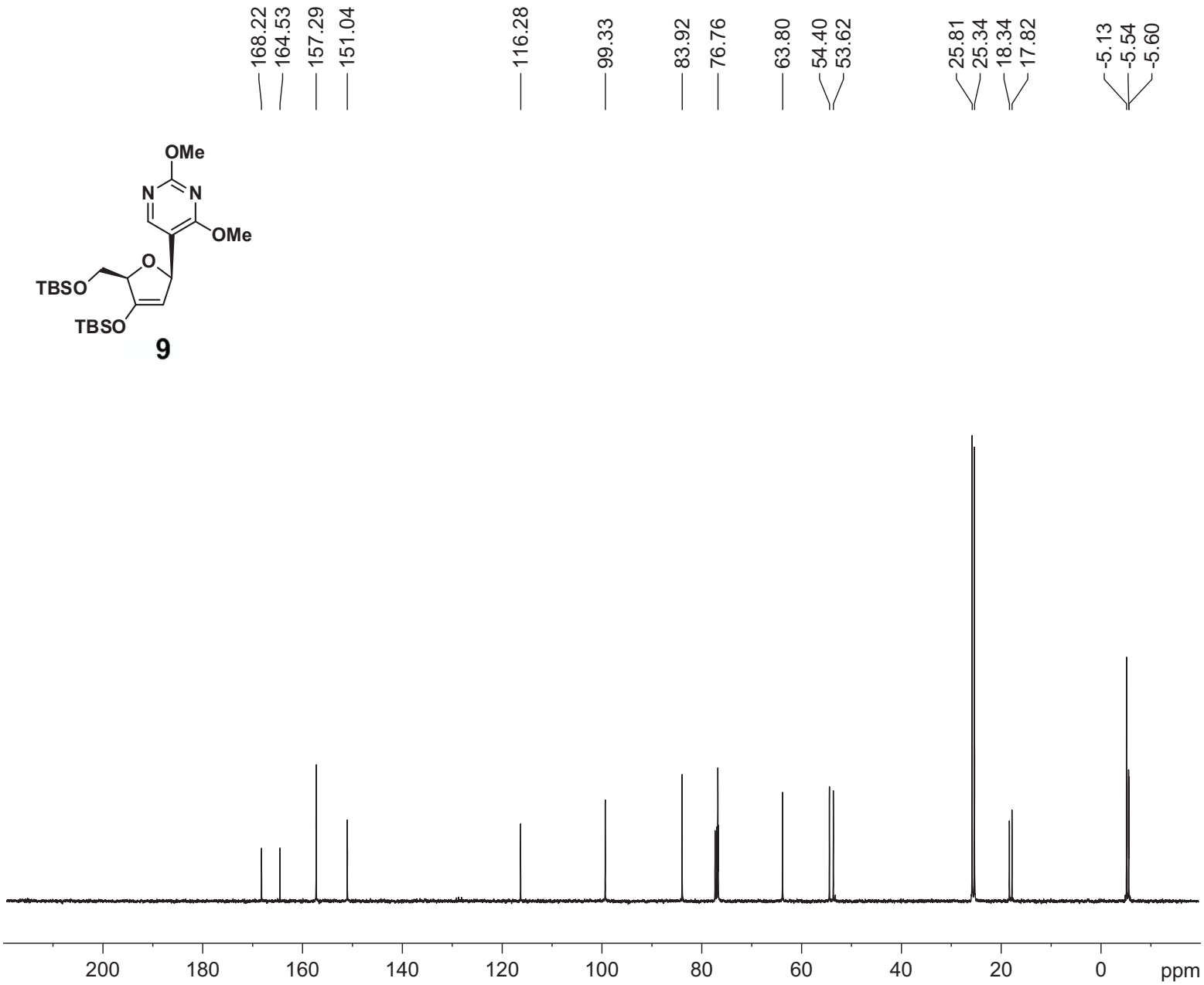
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719147 sec
RG 45.15
DW 69.333 usec
DE 10.50 usec
TE 297.8 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 12.90 usec
PLW1 15.00000000 W

F2 - Processing parameters
SI 16384
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

13C of P data



Current Data Parameters
NAME TQ111
EXPNO 105
PROCNO 1

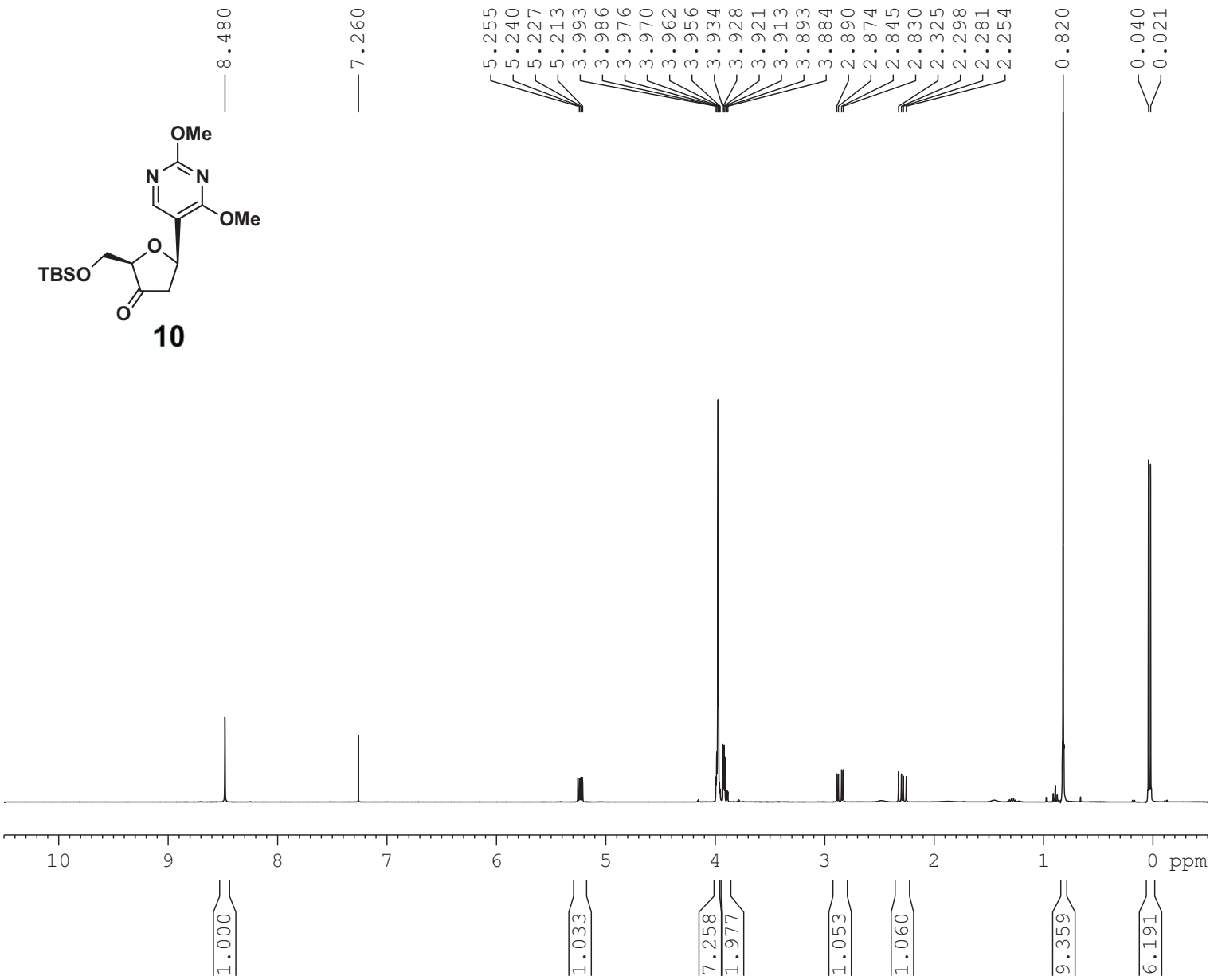
F2 - Acquisition Parameters
Date_ 20161231
Time 17.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 59
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 47.50000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 15.00000000 W
PLW12 0.33750001 W
PLW13 0.27338001 W

F2 - Processing parameters
SI 32768
SF 100.6127817 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

1H of P data



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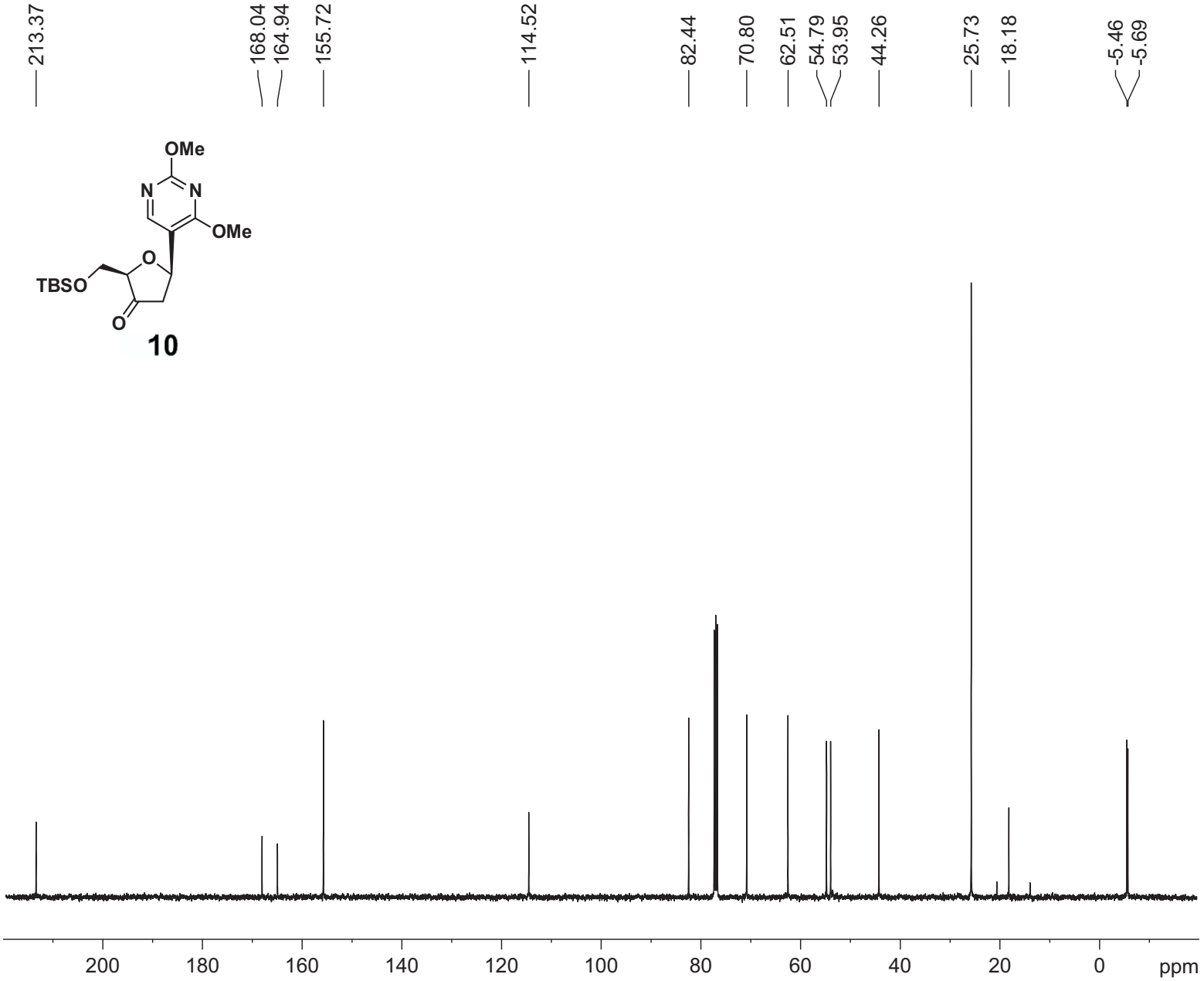
NAME TQ115
EXPNO 101
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170101
Time 21.51
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719147 sec
RG 45.15
DW 69.333 usec
DE 10.50 usec
TE 297.6 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 12.90 usec
PLW1 15.00000000 W

F2 - Processing parameters
SI 16384
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

13C of P data



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NAME TQ115
EXPNO 103
PROCNO 1

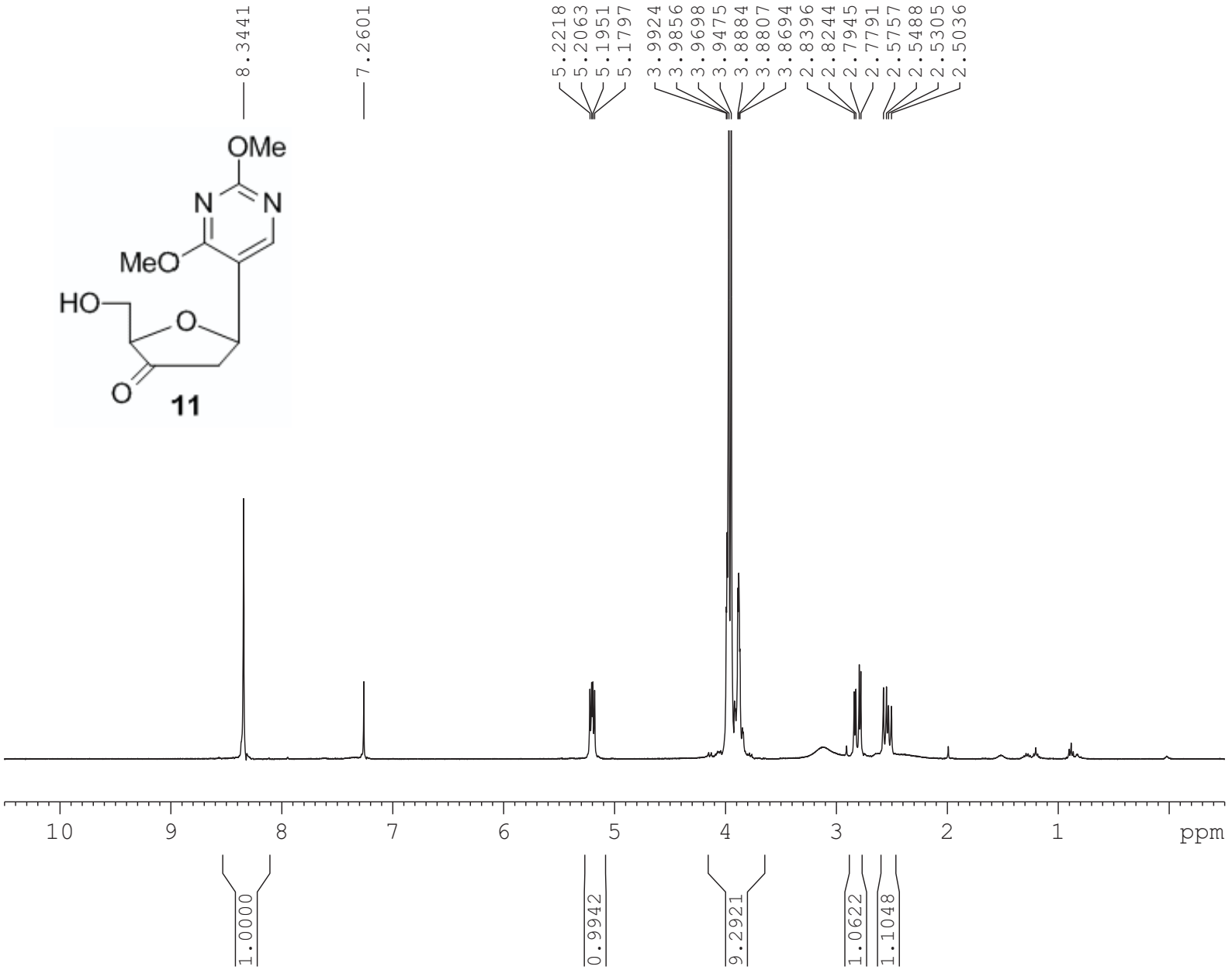
F2 - Acquisition Parameters
Date_ 20170101
Time 21.56
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 201
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 47.50000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 15.00000000 W
PLW12 0.33750001 W
PLW13 0.27338001 W

F2 - Processing parameters
SI 32768
SF 100.6127736 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

1H of P 25-30



Current Data Parameters
NAME TQ133
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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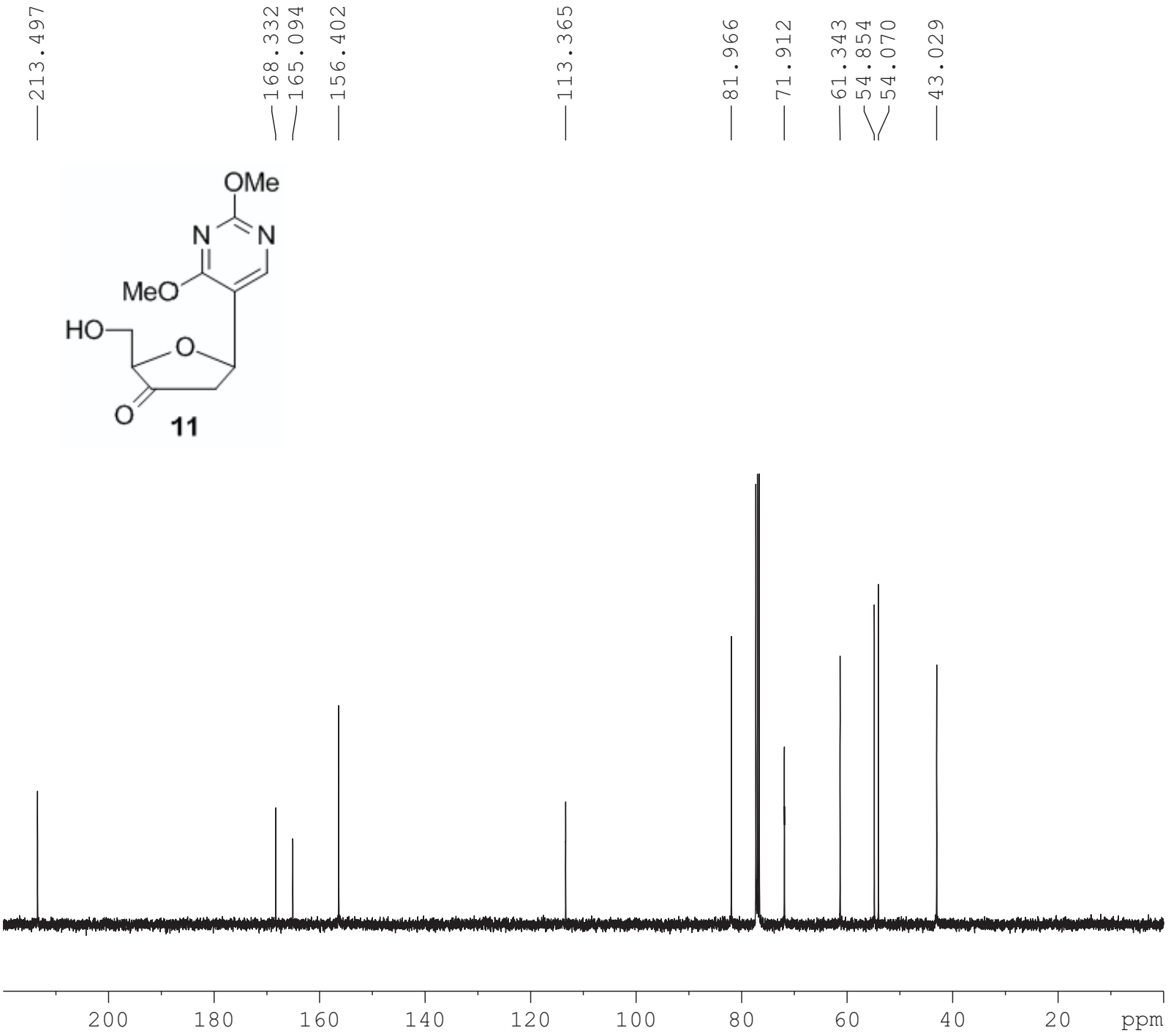
Time 16.42
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 1
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2609921 sec
RG 71.8
DW 69.000 usec
DE 6.50 usec
TE 296.8 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.40 usec
PL1 1.80 dB
SFO1 400.1324008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

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Current Data Parameters
NAME                 TQ133
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_             20170213
Time                 16.48
INSTRUM              spect
PROBHD   5 mm BBO BB-1H
PULPROG             zgpg30
TD                   32768
SOLVENT              CDCl3
NS                     262
DS                       0
SWH              24038.461 Hz
FIDRES           0.733596 Hz
AQ               0.6815744 sec
RG                    8192
DW                  20.800 usec
DE                   6.50 usec
TE                  297.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                   9.00 usec
PL1                  7.00 dB
SFO1          100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                  1.80 dB
PL12                17.00 dB
PL13                20.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF            100.6127797 MHz
WDW                    EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.00
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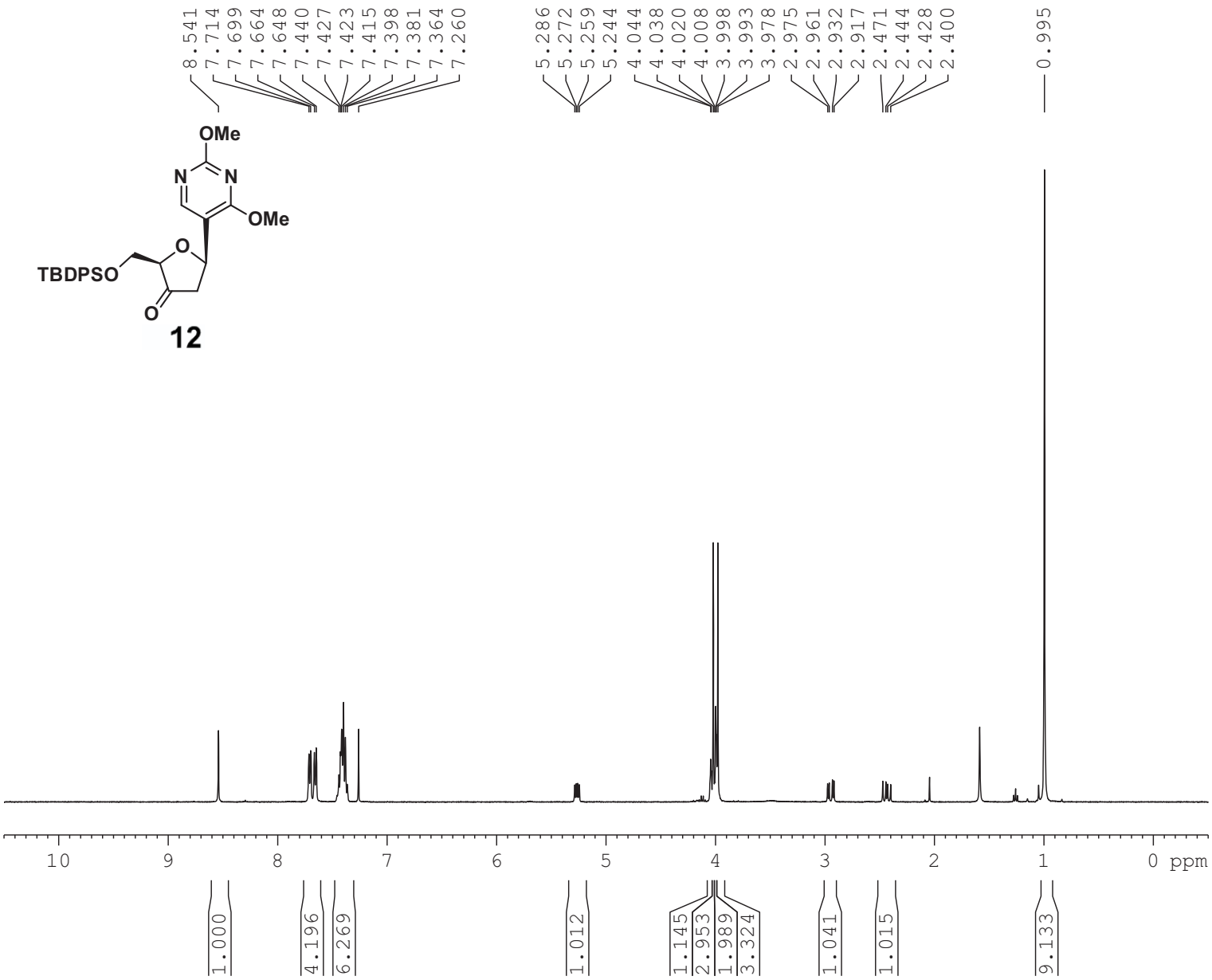
1H of P data



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NAME TQ160
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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Time 19.48
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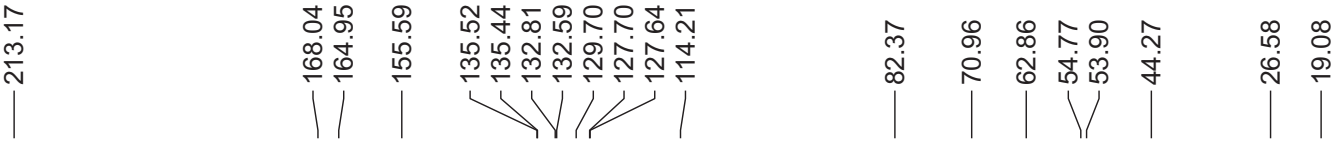
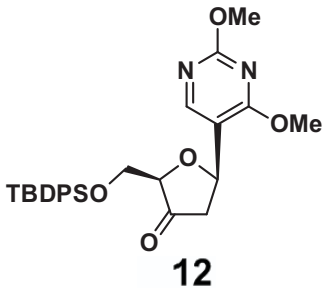
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2609921 sec
RG 228.1
DW 69.000 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.40 usec
PL1 1.80 dB
SFO1 400.1324008 MHz

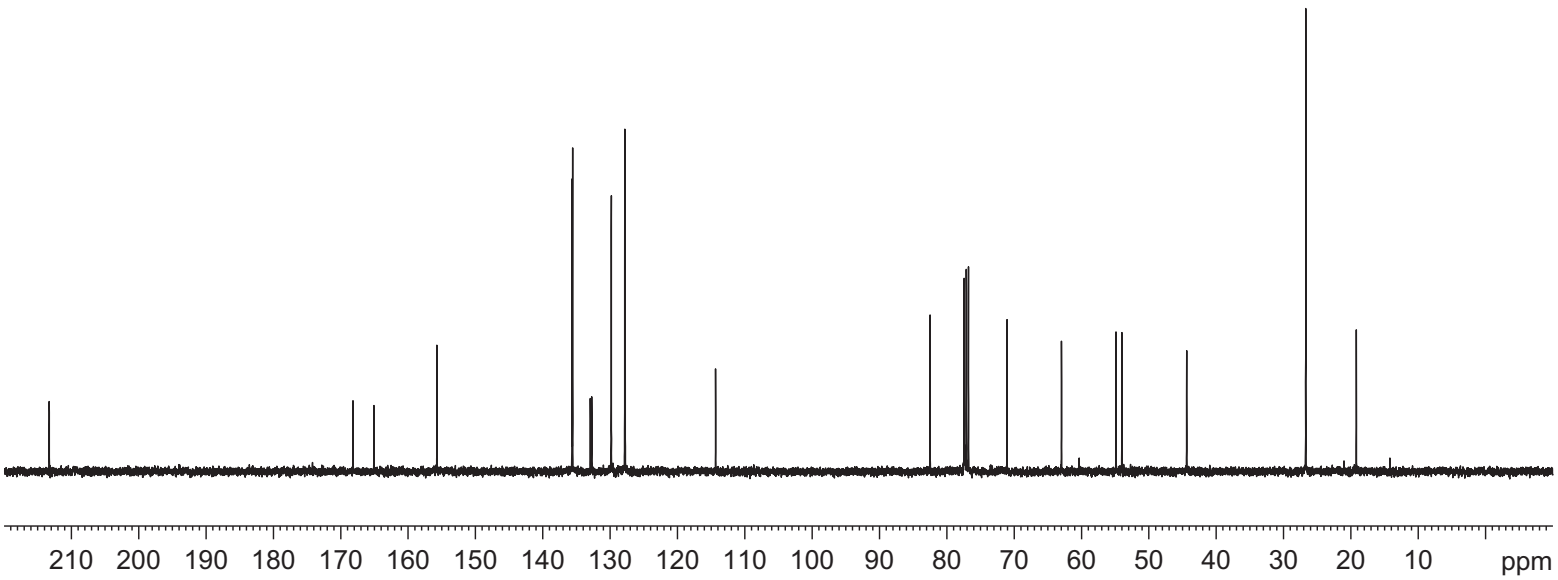
F2 - Processing parameters
SI 16384
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

13C of P data





12



Current Data Parameters
NAME TQ160
EXPNO 4
PROCNO 1

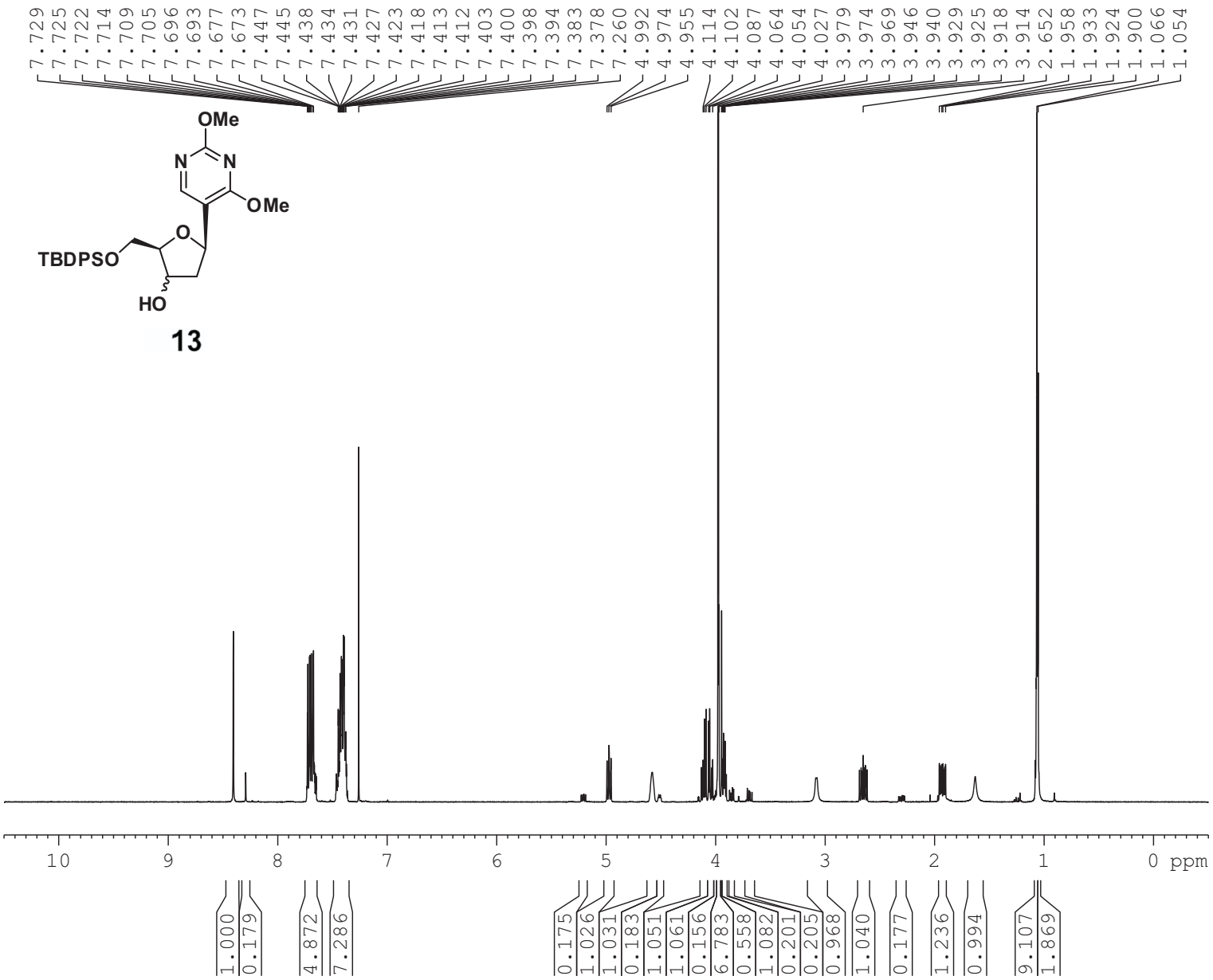
F2 - Acquisition Parameters
Date_ 20170317
Time 19.57
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 81
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 4096
DW 20.800 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PL1 7.00 dB
SFO1 100.6233325 MHz

======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.80 dB
PL12 17.00 dB
PL13 20.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1H of P data



Current Data Parameters
NAME TQ161
EXPNO 101
PROCNO 1

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Time 16.42
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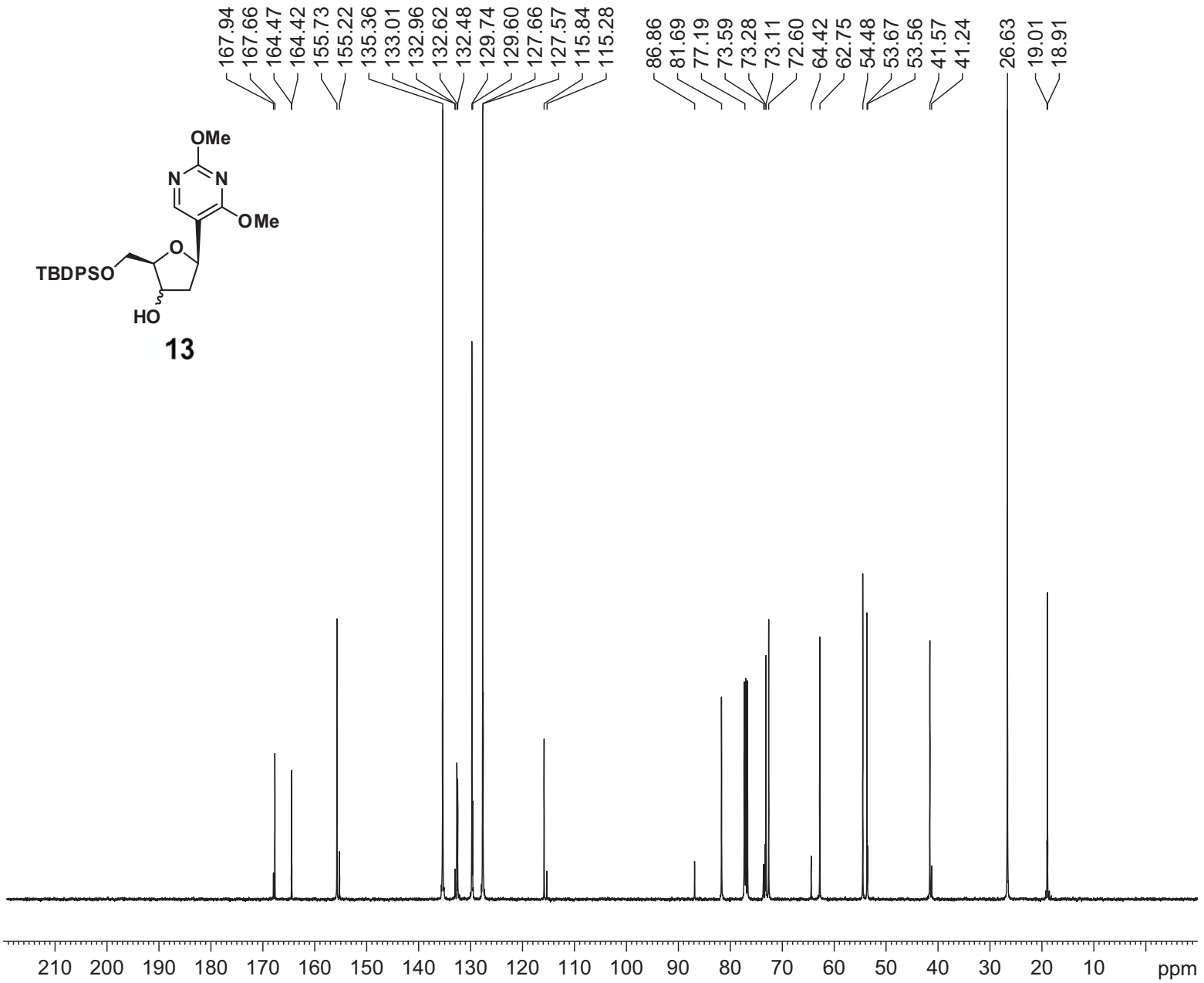
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719147 sec
RG 89.08
DW 69.333 usec
DE 10.50 usec
TE 298.3 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 12.90 usec
PLW1 15.00000000 W

F2 - Processing parameters
SI 16384
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

13C of P data



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EXPNO 104
PROCNO 1

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PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 793
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 298.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

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NUC1 13C
P1 10.00 usec
PLW1 47.50000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
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PLW2 15.00000000 W
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PLW13 0.27338001 W

F2 - Processing parameters
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SSB 0
LB 2.00 Hz
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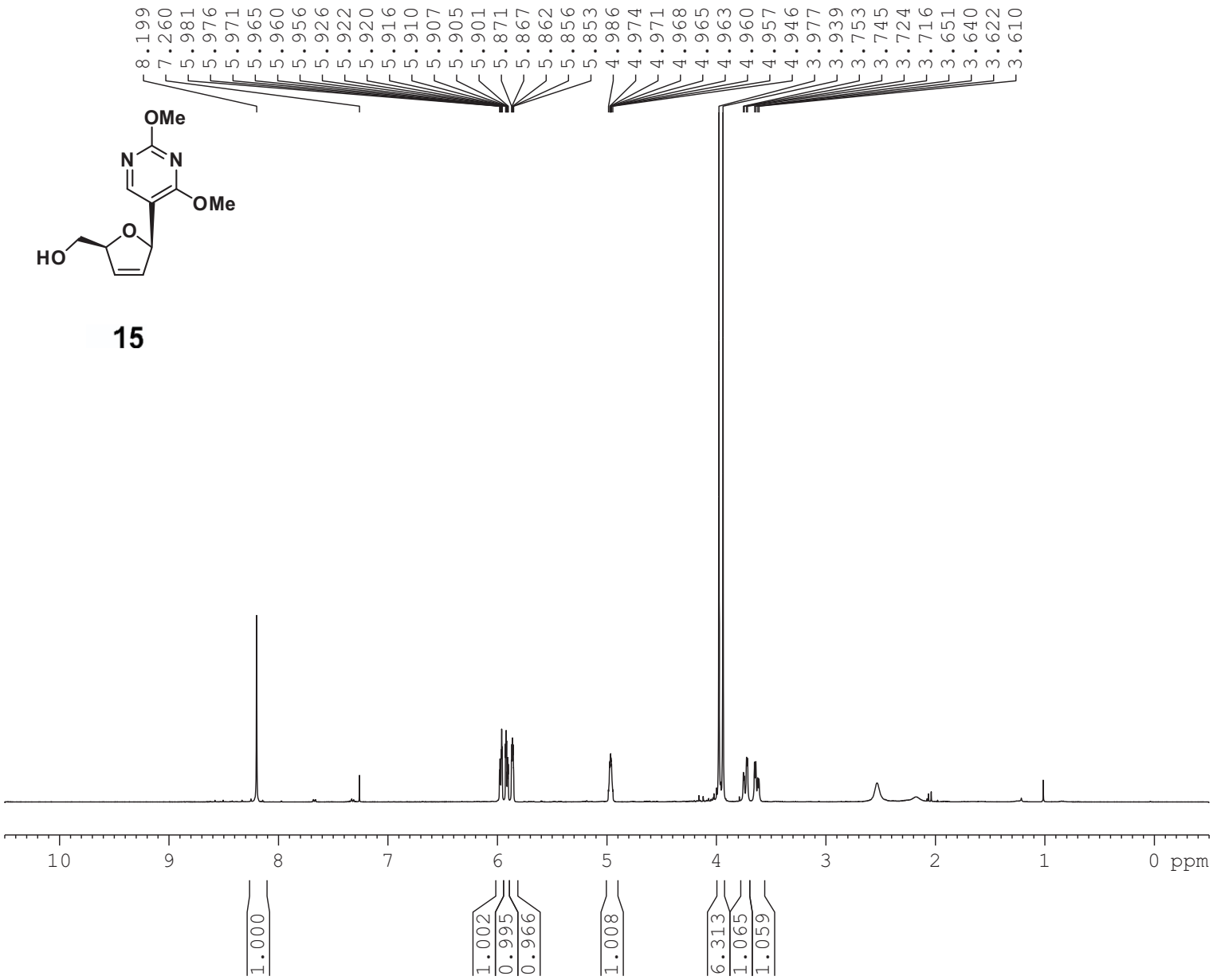
1H of P data



15

Current Data Parameters
NAME TQ167
EXPNO 110
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170331
Time 3.29
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719147 sec
RG 57.42
DW 69.333 usec
DE 10.50 usec
TE 298.2 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 12.90 usec
PLW1 15.00000000 W

F2 - Processing parameters
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SF 400.1300097 MHz
WDW EM
SSB 0
LB 0 Hz
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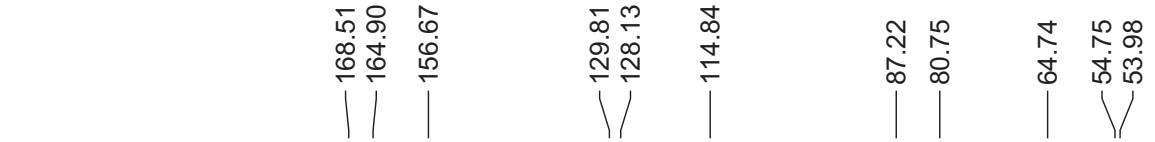
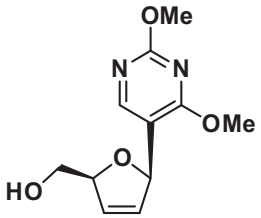
PC 1.00

13C of P data





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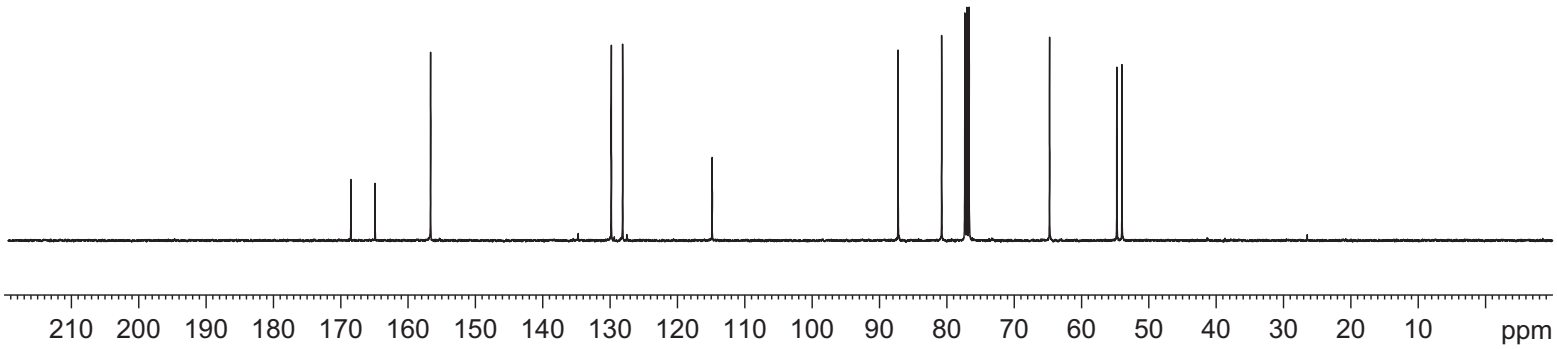

Current Data Parameters
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EXPNO 109
PROCNO 1

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PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1000
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 298.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 47.50000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 15.00000000 W
PLW12 0.33750001 W
PLW13 0.27338001 W

F2 - Processing parameters
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SF 100.6127766 MHz
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SSB 0
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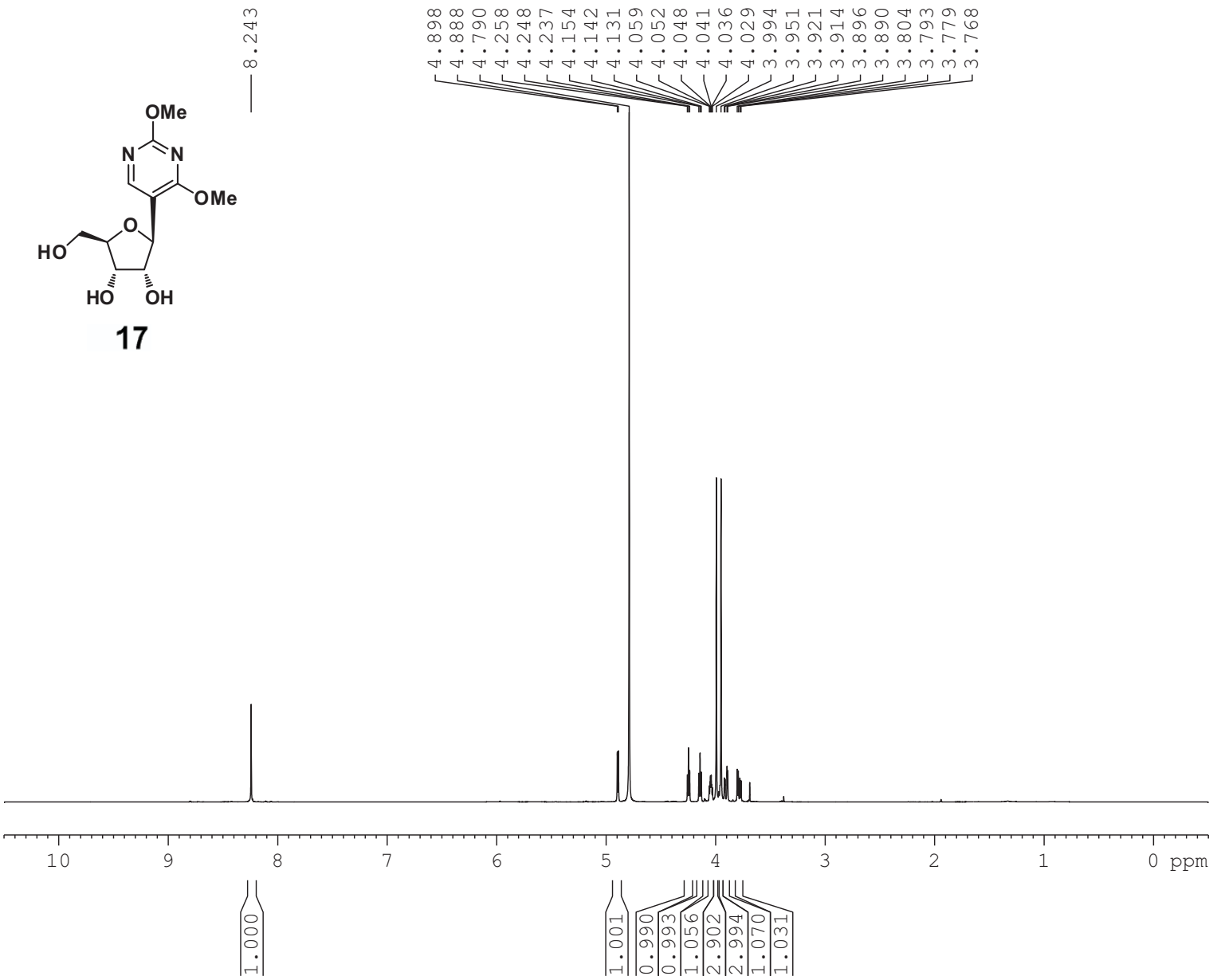
GB 0
PC 1.00

1H of TQ170 P2 data



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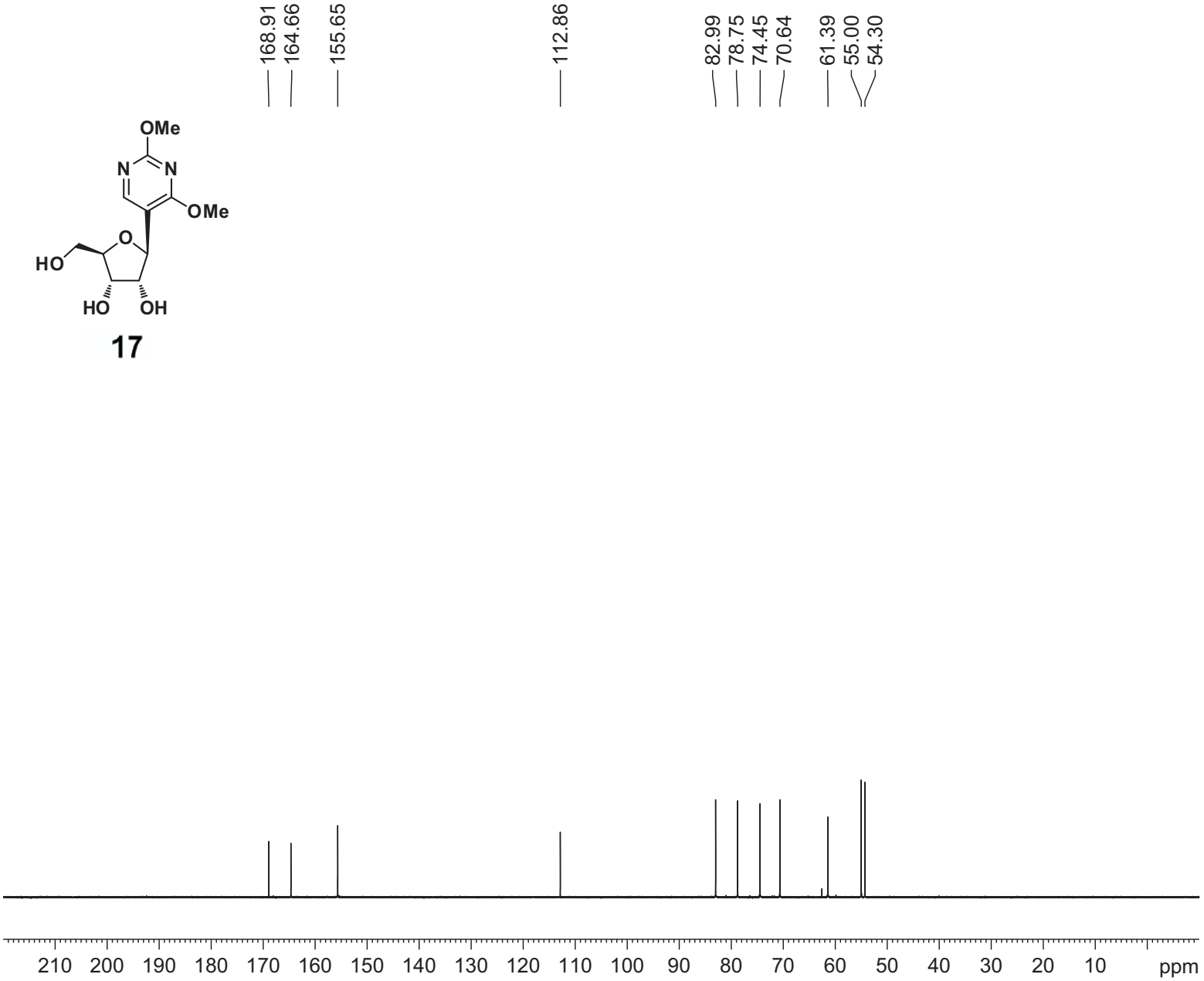
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170330
Time 16.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT D2O
NS 64
DS 0
SWH 6510.417 Hz
FIDRES 0.099341 Hz
AQ 5.0331650 sec
RG 35.9
DW 76.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.30 usec
PL1 -0.50 dB
SFO1 500.1322506 MHz

F2 - Processing parameters
SI 65536
SF 500.1299561 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

13C of TQ170 P2 data



Current Data Parameters
NAME TCC-2017030185
EXPNO 6
PROCNO 1

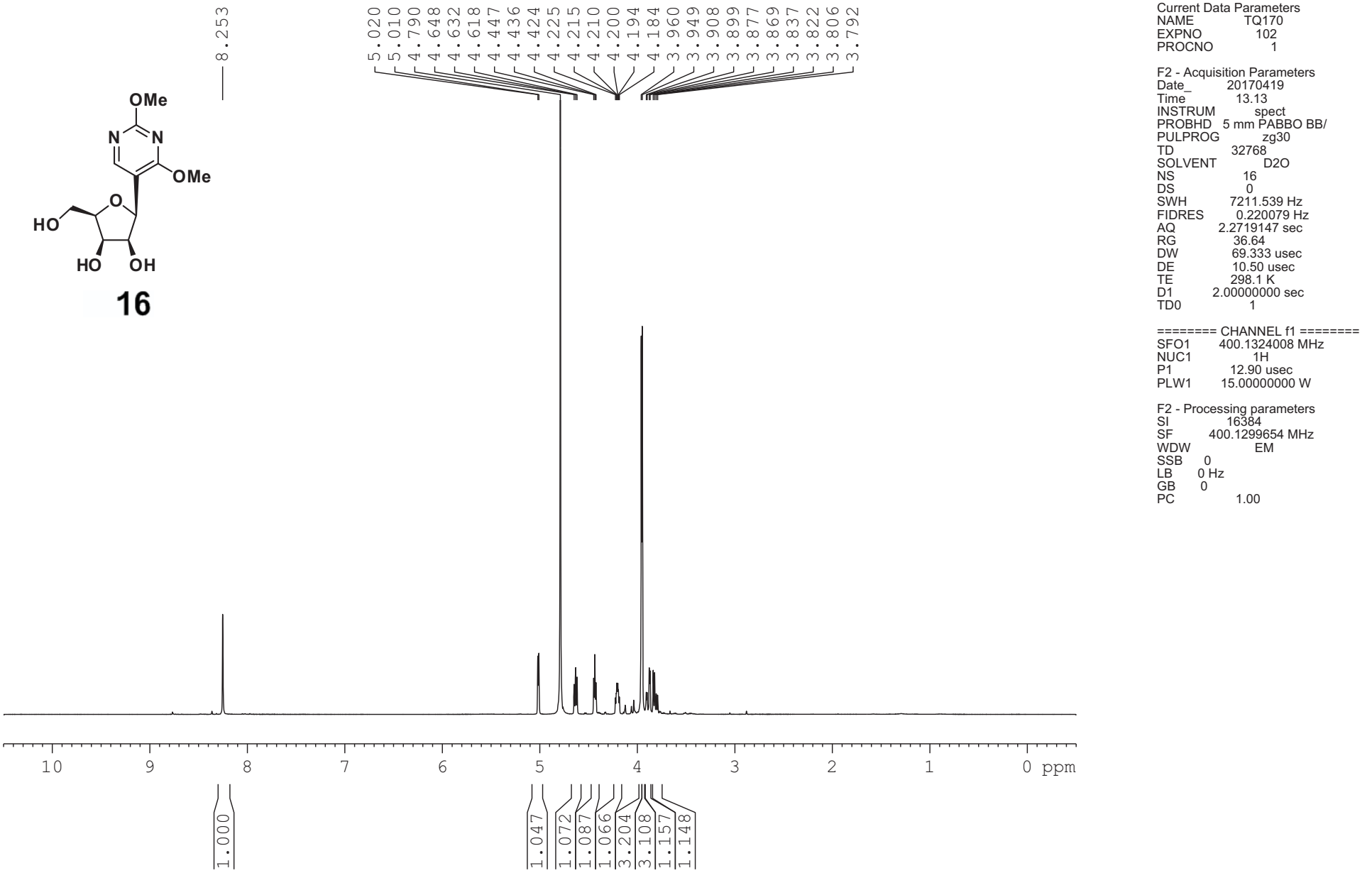
F2 - Acquisition Parameters
Date_ 20170331
Time 2.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 400
DS 0
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813440 sec
RG 5792.6
DW 16.500 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.10 usec
PL1 6.00 dB
SFO1 125.7709936 MHz

======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.50 dB
PL12 16.00 dB
PL13 19.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 65536
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1H of P1 data



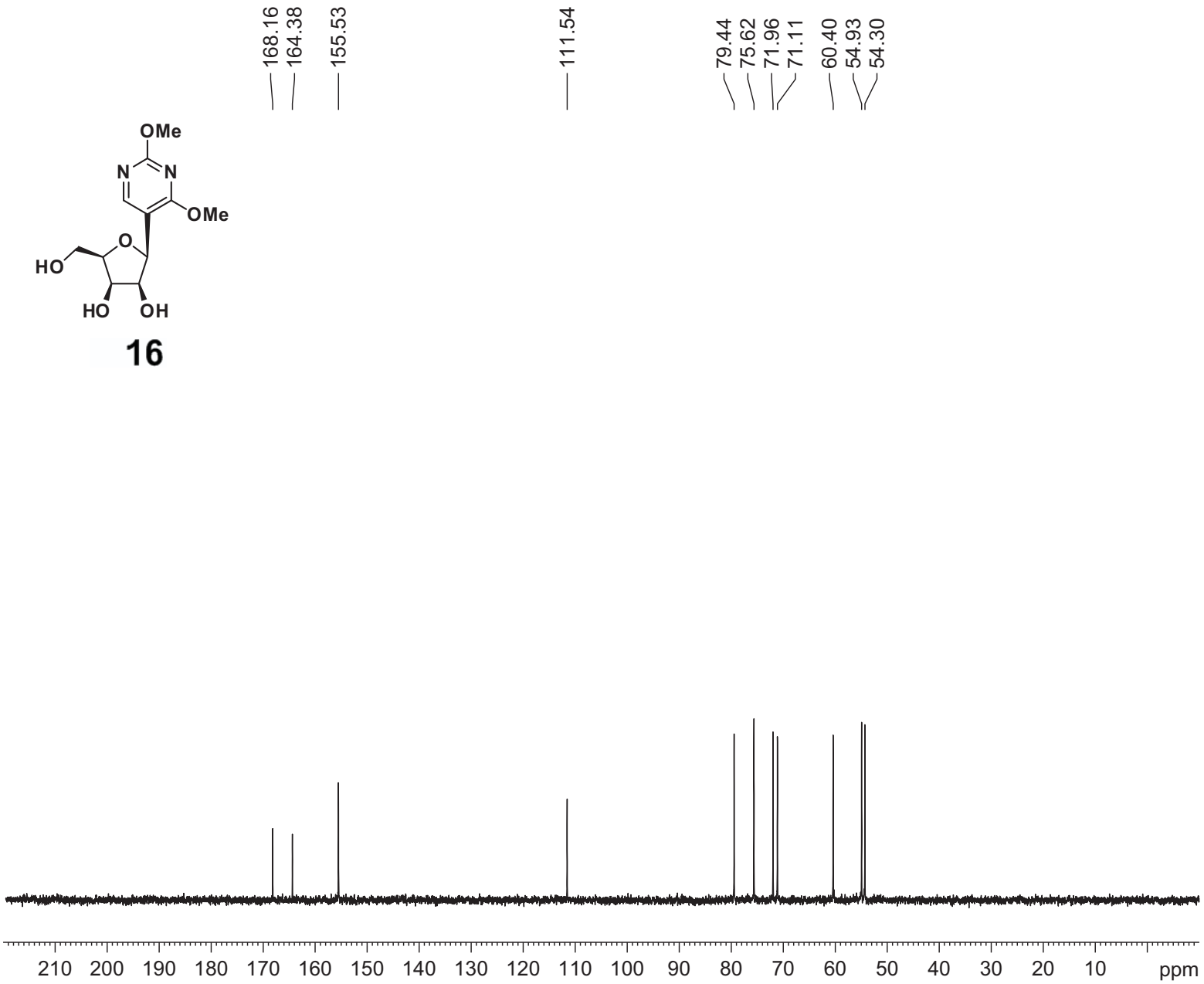
Current Data Parameters
NAME TQ170
EXPNO 102
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170419
Time 13.13
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT D2O
NS 16
DS 0
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719147 sec
RG 36.64
DW 69.333 usec
DE 10.50 usec
TE 298.1 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324008 MHz
NUC1 1H
P1 12.90 usec
PLW1 15.00000000 W

F2 - Processing parameters
SI 16384
SF 400.1299654 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

13C of P1 data



Current Data Parameters
NAME TQ170
EXPNO 104
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170419
Time 13.19
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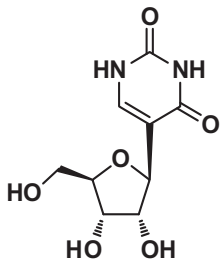
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT D2O
NS 100
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6815744 sec
RG 198.09
DW 20.800 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 47.50000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 15.00000000 W
PLW12 0.33750001 W
PLW13 0.27338001 W

F2 - Processing parameters
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

1H of TQ172 P data



Pseudouridine (1-1)

7.516

4.462
4.452
3.926
3.916
3.907
3.870
3.859
3.849
3.704
3.697
3.691
3.686
3.679
3.603
3.596
3.579
3.573
3.458
3.450
3.434
3.427
2.504
2.500
2.497

Current Data Parameters
NAME TCC-2017040199
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170407
Time 16.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 128
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 128
DW 62.400 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.30 usec
PL1 -0.50 dB
SFO1 500.1335009 MHz

F2 - Processing parameters
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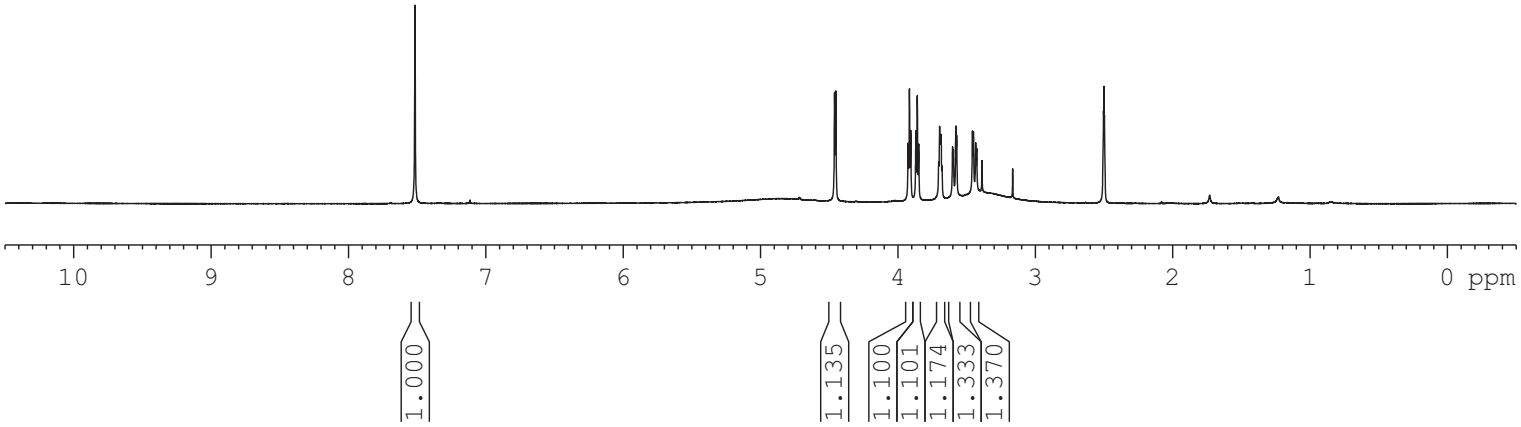
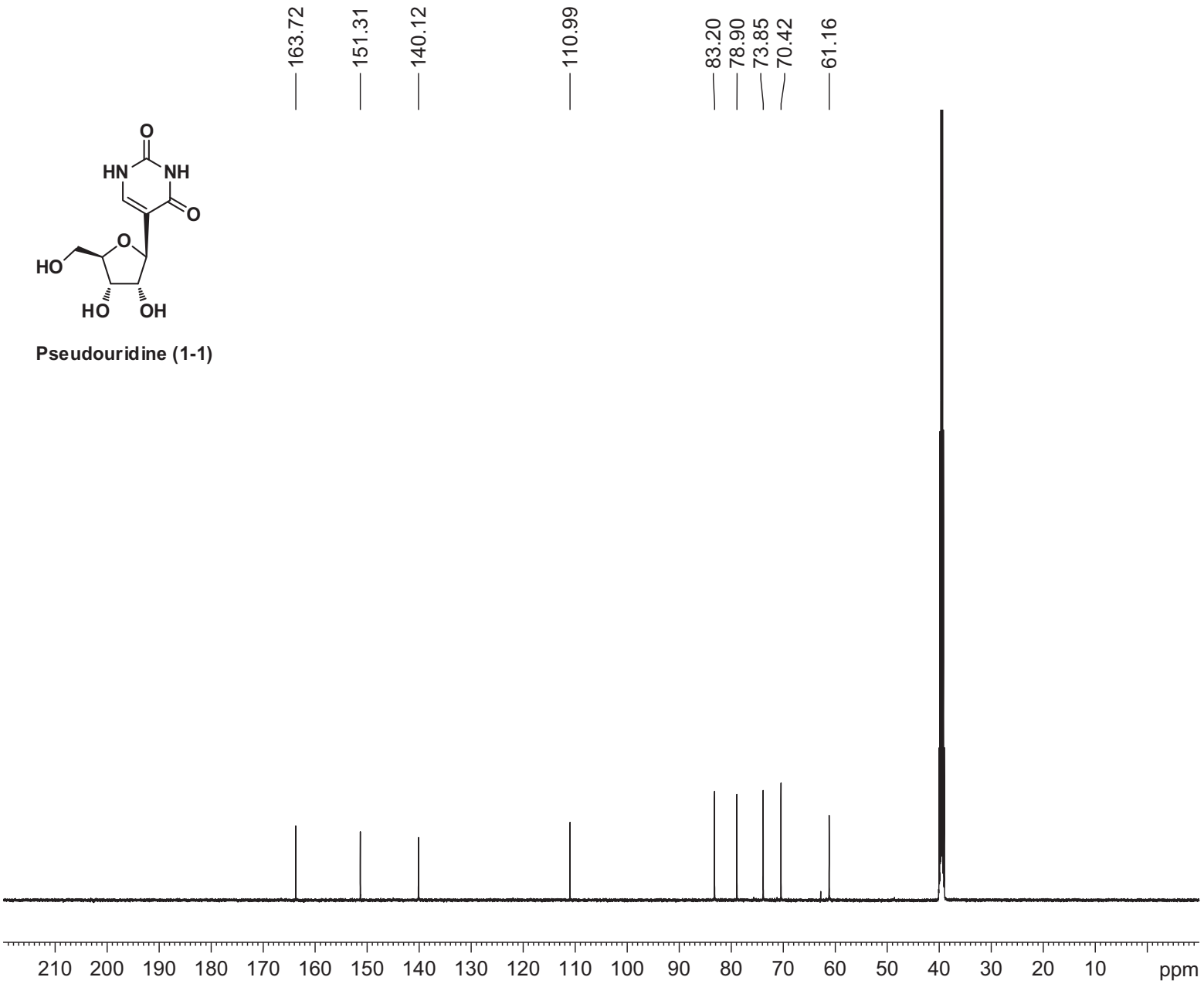
SF 500.1300045 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00



13C of TQ172 P data



Pseudouridine (1-1)

Current Data Parameters
NAME TCC-2017040199
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170408
Time 8.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 3000
DS 0
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813440 sec
RG 8192
DW 16.500 usec
DE 6.50 usec
TE 300.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

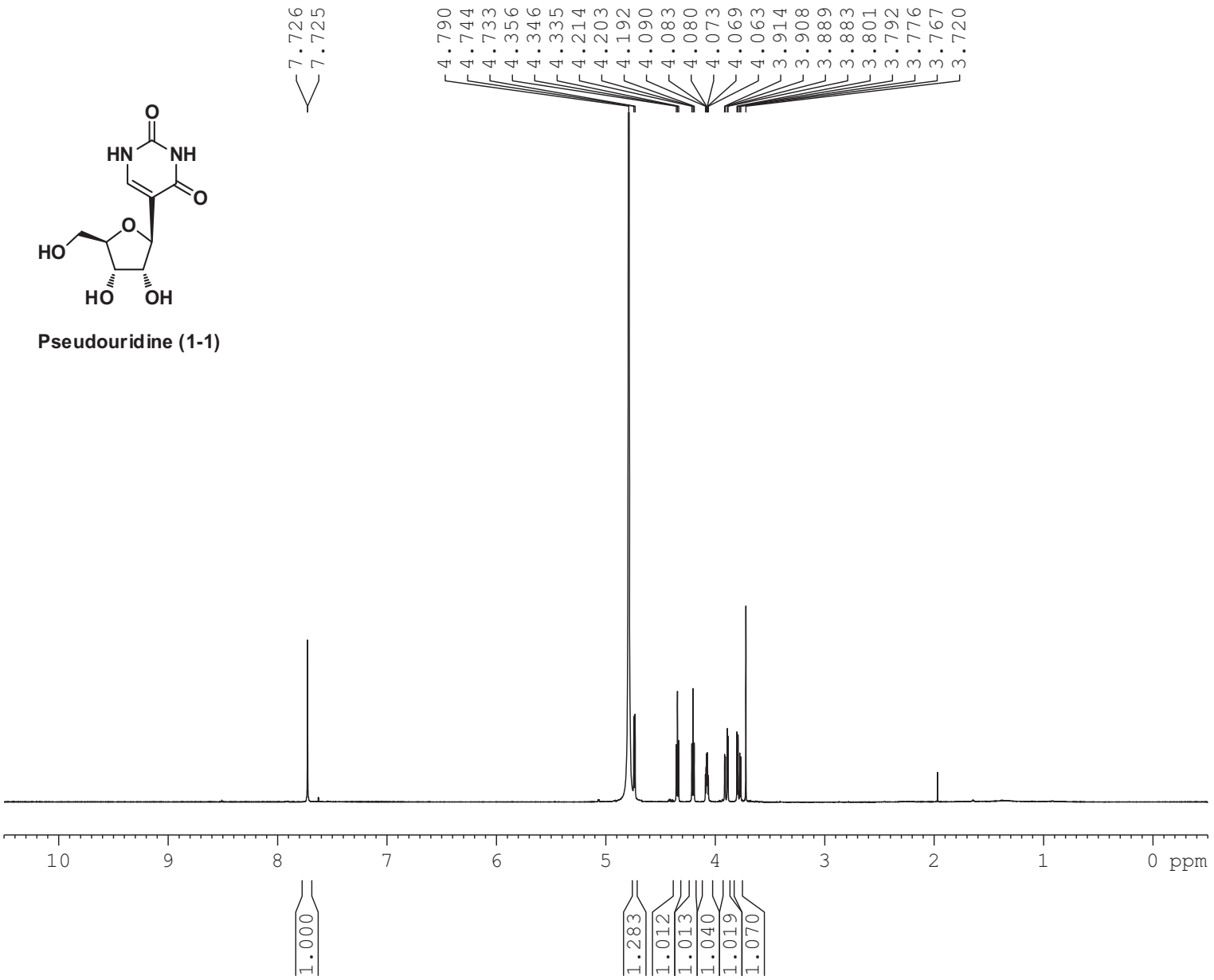
======== CHANNEL f1 ========
NUC1 13C
P1 10.10 usec
PL1 6.00 dB
SFO1 125.7709936 MHz

======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.50 dB
PL12 16.00 dB
PL13 19.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 65536
SF 125.7578470 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1H of TQ172 P data

Pseudouridine (1-1)



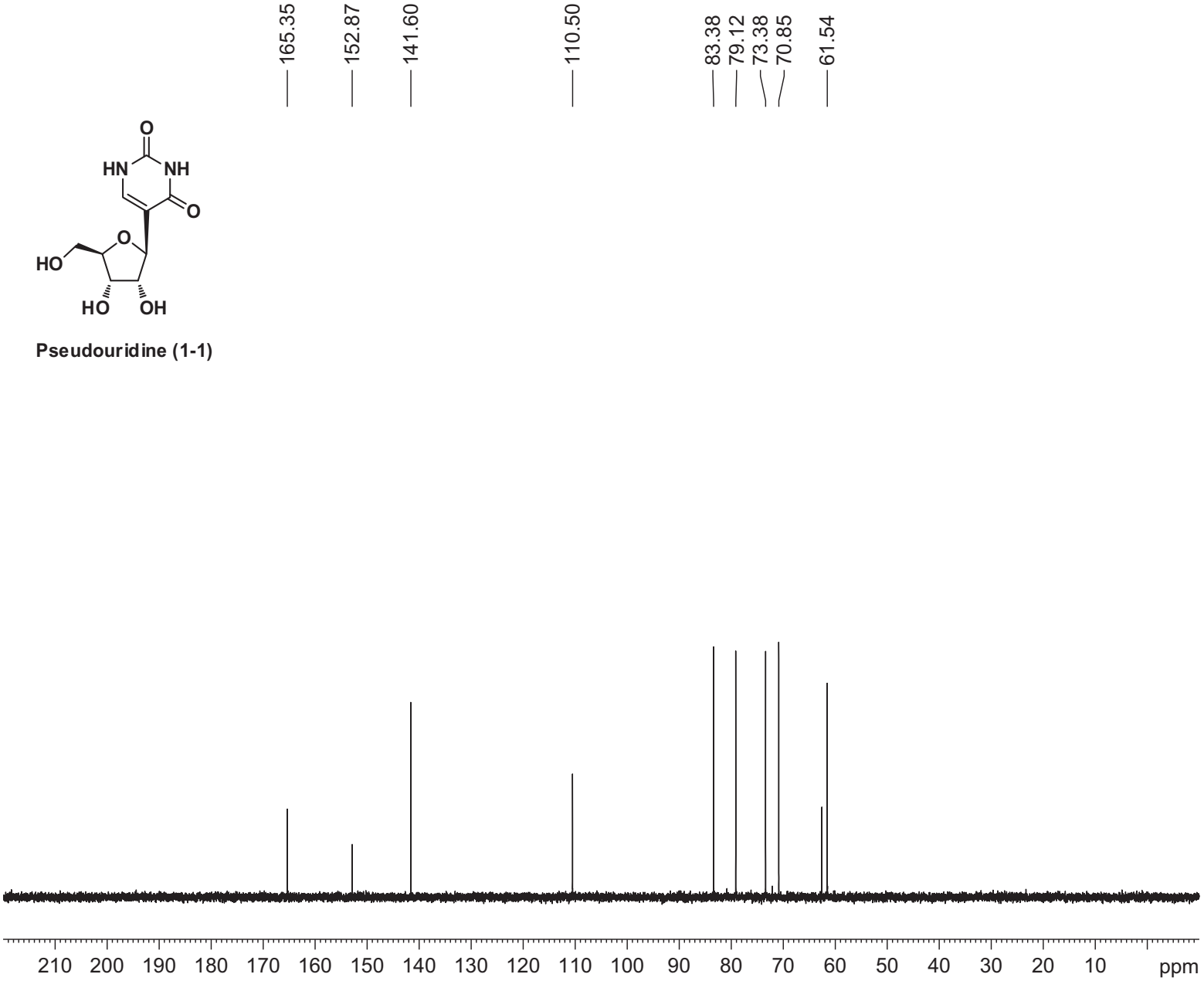
Current Data Parameters
NAME TCC-2017040198
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170420
Time 8.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT D2O
NS 128
DS 0
SWH 6510.417 Hz
FIDRES 0.198682 Hz
AQ 2.5165825 sec
RG 128
DW 76.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 13.30 usec
PL1 -0.50 dB
SFO1 500.1322506 MHz

F2 - Processing parameters
SI 32768
SF 500.1299558 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00

13C of TQ172 P data



Pseudouridine (1-1)

Current Data Parameters
NAME TCC-2017040198
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170420
Time 6.18
INSTRUM spect
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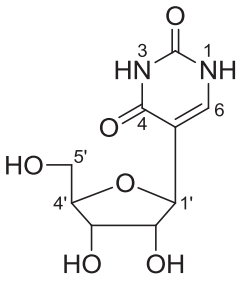
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 4000
DS 0
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813440 sec
RG 8192
DW 16.500 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.10 usec
PL1 6.00 dB
SFO1 125.7709936 MHz

======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.50 dB
PL12 16.00 dB
PL13 19.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 65536
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



pseudouridine (Ψ, **1**)

Table S3. Spectral data comparison: ^{1}H NMR in DMSO-d_6 (δ, ppm)

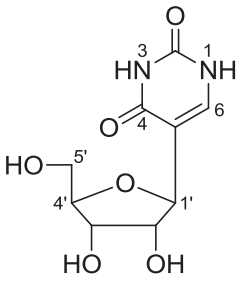
peak (^{1}H, δ)	Smith[a]	Noyori[b] (100 MHz)	**This work** (500 MHz)
1	7.546 (s, 1H, H-6)	7.52 (d, *J* = 6.0 Hz, 1H, H-6)	7.52 (s, 1H, H-6)
2	4.471 (*J* = 4.5 Hz,1H, H-1')	4.51 (d, *J* = 4.0 Hz, 1H, H-1')	4.46 (d, *J* = 4.5 Hz,1H, H-1')
3	3.925 (*J* = 4.5 Hz, 1H, H-2')	3.3-4.0 (m, 5H, H-2', H-3', H-4', H-5')	3.92 (t, *J* = 4.8 Hz, 1H, H-2')
4	3.877 (*J* = 5.3, 4.5 Hz, 1H, H-3')	-----	3.86 (t, *J* = 5.3 Hz, 1H, H-3')
5	3.706 (*J* = 5.3, 3.5, 3.0 Hz, 1H, H-4')	-----	3.69 (dt, *J* = 5.5, 3.2 Hz, 1H, H-4')
6	3.601 (dd, *J* = 11.9, 3.0 Hz, 1H, H-5')	-----	3.59 (dd, *J* = 11.9, 3.1 Hz, 1H, H-5a')
7	3.450 (dd, *J* = 11.9, 3.5 Hz, 1H, H-5')	-----	3.44 (dd, *J* = 11.9, 3.7 Hz, 1H, H-5')
8	4.855 (d, *J* = 4.8 Hz, 1H, OH), 4.743 (dd, *J* = 6.2, 4.8 Hz, 1H, OH), 4.655 (d, *J* = 5.3 Hz, 1H, OH)	11.07 (br s, 1H, H-3), 10.85 (d, *J* = 6.0 Hz, 1H, H-1), 4.54 (br s, 3H, 3 OH)	-----

[a] Deslauriers, R.; Smith, I. C. P. *Can. J. Chem.* **1973**, *51* 833;
[b] Sato, T.; Hayakawa, Y.; Noyori, R. *Bull. Chem. Soc. Jpn.* **1984**, *57*, 2515

Table S4. Spectral data comparison: ^{13}C NMR in DMSO-d_6 (δ, ppm)

peak (^{13}C, δ)	Wenkert[a] (15 MHz)	Townsend[b] (25 MHz)	Noyori[c] (20 MHz)	**This work**[d] (125 MHz)
1	164.8 (C-4)	163.95 (C-4)	163.57	163.7
2	152.3 (C-2)	151.36 (C-2)	150.98	151.3
3	141.0 (C-6)	140.14 (C-6)	139.62	140.1 (CH)
4	110.0 (C-5)	111.27 (C-5)	111.05	111.0
5	82.9 (C-4')	83.60 (C-4')	83.47	83.2 (CH)
6	78.6 (C-1')	79.13 (C-1')	78.88	78.9 (CH)
7	72.9 (C-2')	74.10 (C-2')	73.93	73.9 (CH)
8	71.3 (C-3')	70.80 (C-3')	70.62	70.4 (CH)
9	61.1 (C-5')	61.55 (C-5')	61.36	61.2 (CH_2)

[a] Wenkert, B.; Hagaman, E. W.; Gutowski, G. E.; *Biochem. Biophys. Res. Commun.* **1973**, *51*, 318;
[b] Chenon, M.-T.; Pugmire, R. L.; Grant, D. M.; Panzica, R. P.; Townsend, L. B. *J. Heterocycl. Chem.* **1973**, *10*, 427;
[c] Sato, T.; Hayakawa, Y.; Noyori, R. *Bull. Chem. Soc. Jpn.* **1984**, *57*, 2515;
[d] The numbers of protons directly attached to the individual carbons were determined by ^{13}C NMR DEPT experiments



pseudouridine (Ψ, **1**)

Table S5. Spectral data comparison: ^{1}H NMR in D_2O (δ, ppm)

peak (^{1}H, δ)	Sasaki[a] (100 MHz)	Smith[b,c] (100 MHz)	Chow[d,e] (500 MHz)	**This work** (500 MHz)
1	7.63 (s, 1H, H-6)	7.660 (d, *J* = 0.8 Hz, 1H, H-6)	7.50 (d, *J* = 3.0 Hz, 1H)	7.73 (d, *J* = 0.6 Hz, 1H, H-6)
2	4.67 (d, *J* = 5.0 Hz, 1H, H-1')	4.674 (dd, *J* = 5.0, 0.8 Hz, 1H, H-1')	4.51 (d, *J* = 5.5 Hz, 1H)	4.74 (d, *J* = 5.6 Hz,1H, H-1')
3	4.27 (t, *J* = 5.0 Hz, 1H, H-2')	4.279 (t, *J* = 5.0 Hz, 1H, H-2')	4.13 (t, *J* = 5.5 Hz, 1H)	4.35 (t, *J* = 5.5 Hz, 1H, H-2')
4	4.13 (t, *J* = 5.2 Hz, 1H, H-3')	4.141 (t, *J* = 5.2 Hz, 1H, H-3')	3.98 (t, *J* = 5.5 Hz, 1H)	4.20 (t, *J* = 5.4 Hz, 1H, H-3')
5	4.00 (ddd, *J* = 5.2, 4.5, 3.5 Hz, 1H, H-4')	4.009 (*J* = 5.2, 4.6, 3.2 Hz, 1H, H-4')	3.85 (m, 1H)	4.08 (dt, *J* = 5.3, 3.3 Hz, 1H, H-4')
6	3.82 (dd, *J* = 12.5, 3.5 Hz, 1H, H-5')	3.840 (dd, *J* = 12.7, 3.2 Hz, 1H, H-5')	3.68 (dd, *J* = 12.0, 3.0 Hz, 1H)	3.90 (dd, *J* = 12.5, 3.2 Hz, 1H, H-5')
7	3.73 (dd, *J* = 12.5, 4.5 Hz, 1H, H-5')	3.726 (dd, *J* = 12.7, 4.6 Hz, 1H, H-5')	3.56 (dd, *J* = 13.0, 5.0 Hz, 1H)	3.78 (dd, *J* = 12.5, 4.9 Hz, 1H, H-5')

[a] Sasaki, K.; Kusakabe, Y.; Esumi, S. *J. Antibiot.* **1972**, *25*, 151;
[b] Frank E. Hruska, Arthur A. Grey, Ian C. P. Smith, *J. Am. Chem. Soc.* **1970**, *92*, 4088;
[c] Deslauriers, R.; Smith, I. C. P. *Can. J. Chem.* **1973**, *51* 833;
[d] Desaulniers, J.-P.; Chang, Y.-C.; Aduri, R.; Abeysirigunawardena, S. C.; SantaLucia J.; Chow, C. S. *Org. Biomol. Chem.* **2008**, *6*, 3892;
[e] [1,3-^{15}N]-pseudouridine

Table S6. Spectral data comparison: ^{13}C NMR in D_2O (δ, ppm)

peak (^{13}C, δ)	Chattopadhyaya[a] (125 MHz)	Chow[b,c] (125 MHz)	**This work**[d] (125 MHz)
1	165.7 (C-4)	165.36	165.4
2	153.2 (C-2)	(141.63)	152.9
3	141.9 (C-6)	141.54	141.6 (CH)
4	110.9 (C-5)	110.59	110.5
5	-----	83.46	83.4 (CH)
6	-----	79.27	79.1 (CH)
7	-----	73.45	73.4 (CH)
8	-----	70.92	70.9 (CH)
9	-----	61.60	61.5 (CH_2)

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[c] [1,3-^{15}N]-pseudouridine;
[d] The numbers of protons directly attached to the individual carbons were determined by ^{13}C NMR DEPT experiments