Supplementary Information

Divergent synthetic approach to 6”-modified α-GalCer analogues

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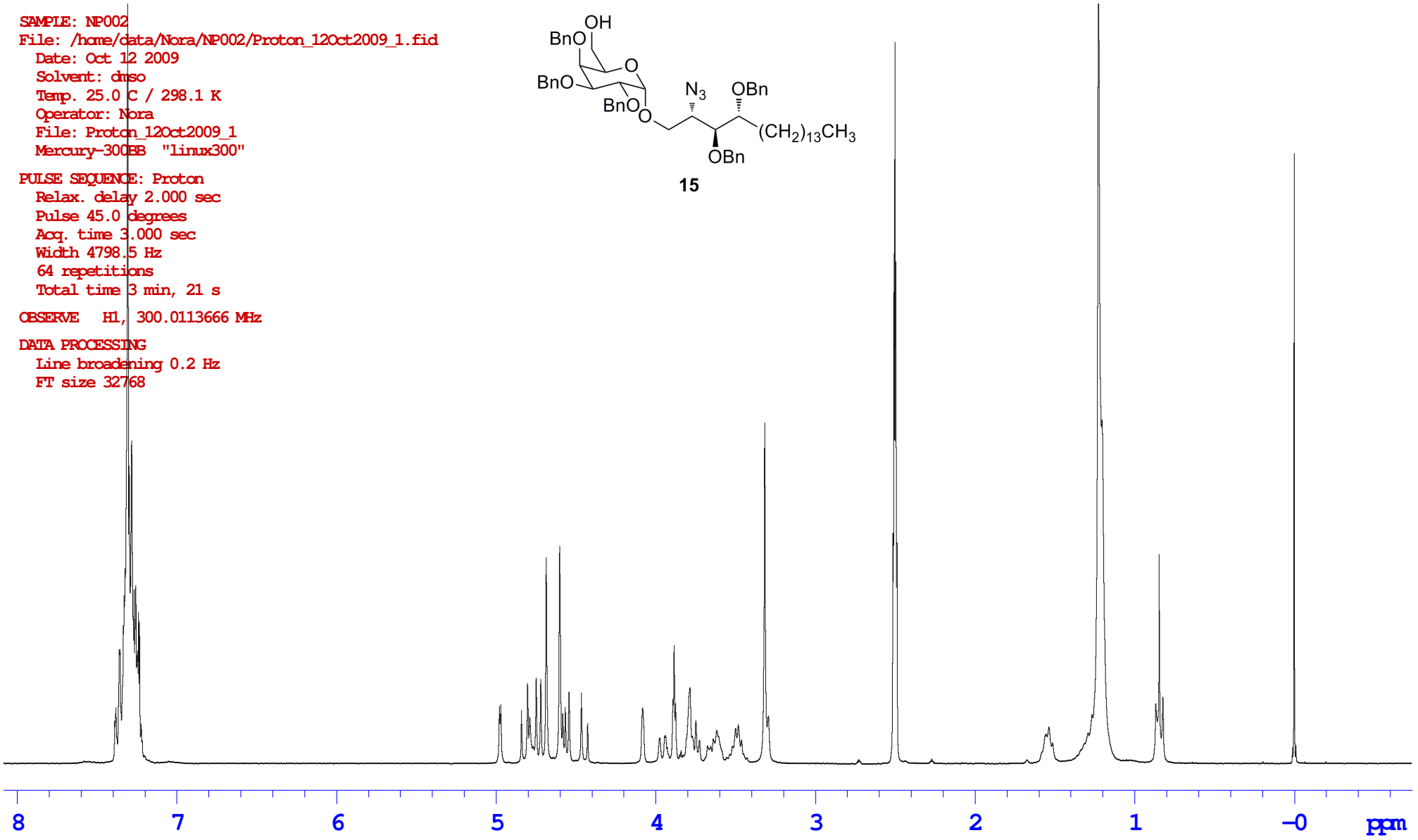

SAMPLE: NP002
File: /home/data/Nora/NP002/Proton_12Oct2009_1.fid
Date: Oct 12 2009
Solvent: dmso
Temp. 25.0 C / 298.1 K
Operator: Nora
File: Proton_12Oct2009_1
Mercury-300BB "linux300"
PULSE SEQUENCE: Proton
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 3.000 sec
Width 4798.5 Hz
64 repetitions
Total time 3 min, 21 s
OBSERVE H1, 300.0113666 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
OH
BnO
O
BnO
BnO
O
N3
OBn
(CH2)13CH3
OBn
15
8
7
6
5
4
3
2
1
–0
ppm

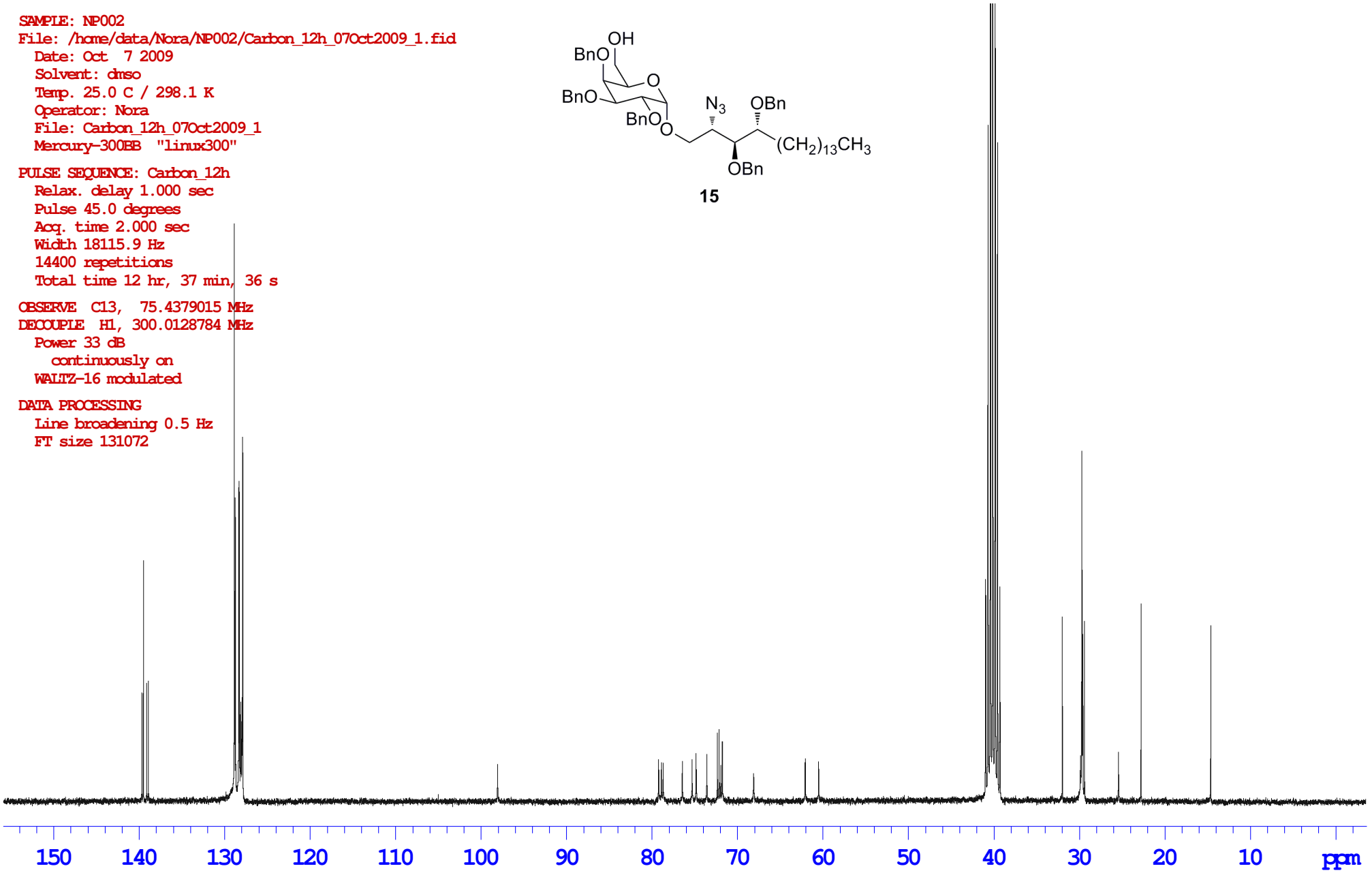
SAMPLE: NP002
File: /home/data/Nora/NP002/Carbon_12h_07Oct2009_1.fid
Date: Oct 7 2009
Solvent: dmso
Temp. 25.0 C / 298.1 K
Operator: Nora
File: Carbon_12h_07Oct2009_1
Mercury-300BB "linux300"

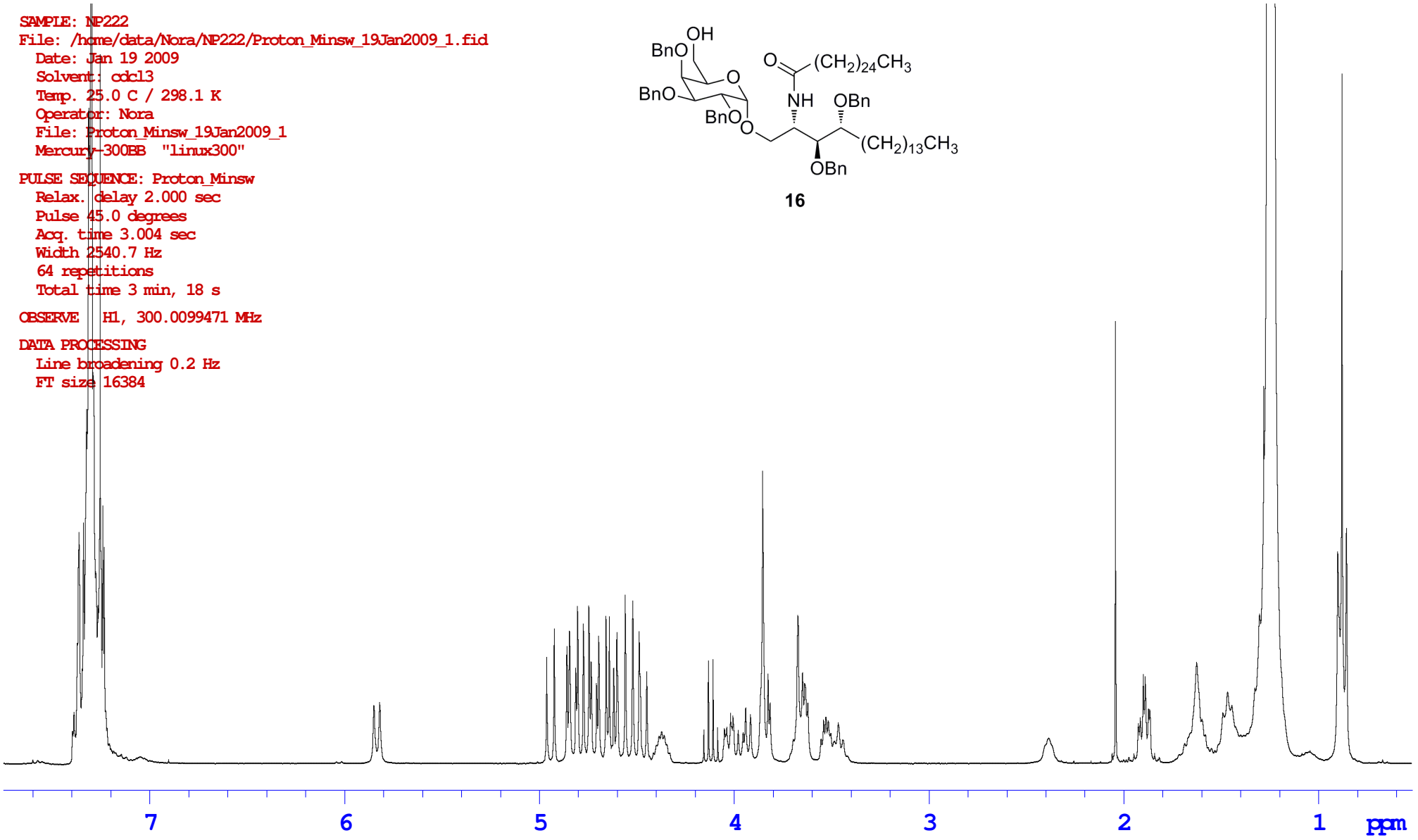
PULSE SEQUENCE: Carbon_12h
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.000 sec
Width 18115.9 Hz
14400 repetitions
Total time 12 hr, 37 min, 36 s

OBSERVE C13, 75.4379015 MHz
DECOUPLE H1, 300.0128784 MHz
Power 33 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072

15

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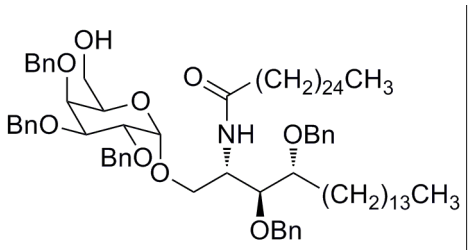
File: /home/data/Nora/NP222/Proton_Minsw_19Jan2009_1.fid
Date: Jan 19 2009
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: Nora
File: Proton_Minsw_19Jan2009_1
Mercury-300BB "linux300"
PULSE SEQUENCE: Proton_Minsw
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 3.004 sec
Width 2540.7 Hz
64 repetitions
Total time 3 min, 18 s
OBSERVE H1, 300.0099471 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
OH
BnO
O
BnO
BnO
O
O
(CH2)24CH3
NH
OBn
(CH2)13CH3
OBn
16
7
6
5
4
3
2
1
ppm

SAMPLE: NP222
File: /home/data/Nora/NP222/Carbon_12h_19Jan2009_1.fid
Date: Jan 19 2009
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: Nora
File: Carbon_12h_19Jan2009_1
Mercury-300BB "linux300"

PULSE SEQUENCE: Carbon_12h
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.000 sec
Width 18115.9 Hz
14400 repetitions
Total time 12 hr, 37 min, 36 s

OBSERVE C13, 75.4375432 MHz
DECOUPLE H1, 300.0114533 MHz
Power 33 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072



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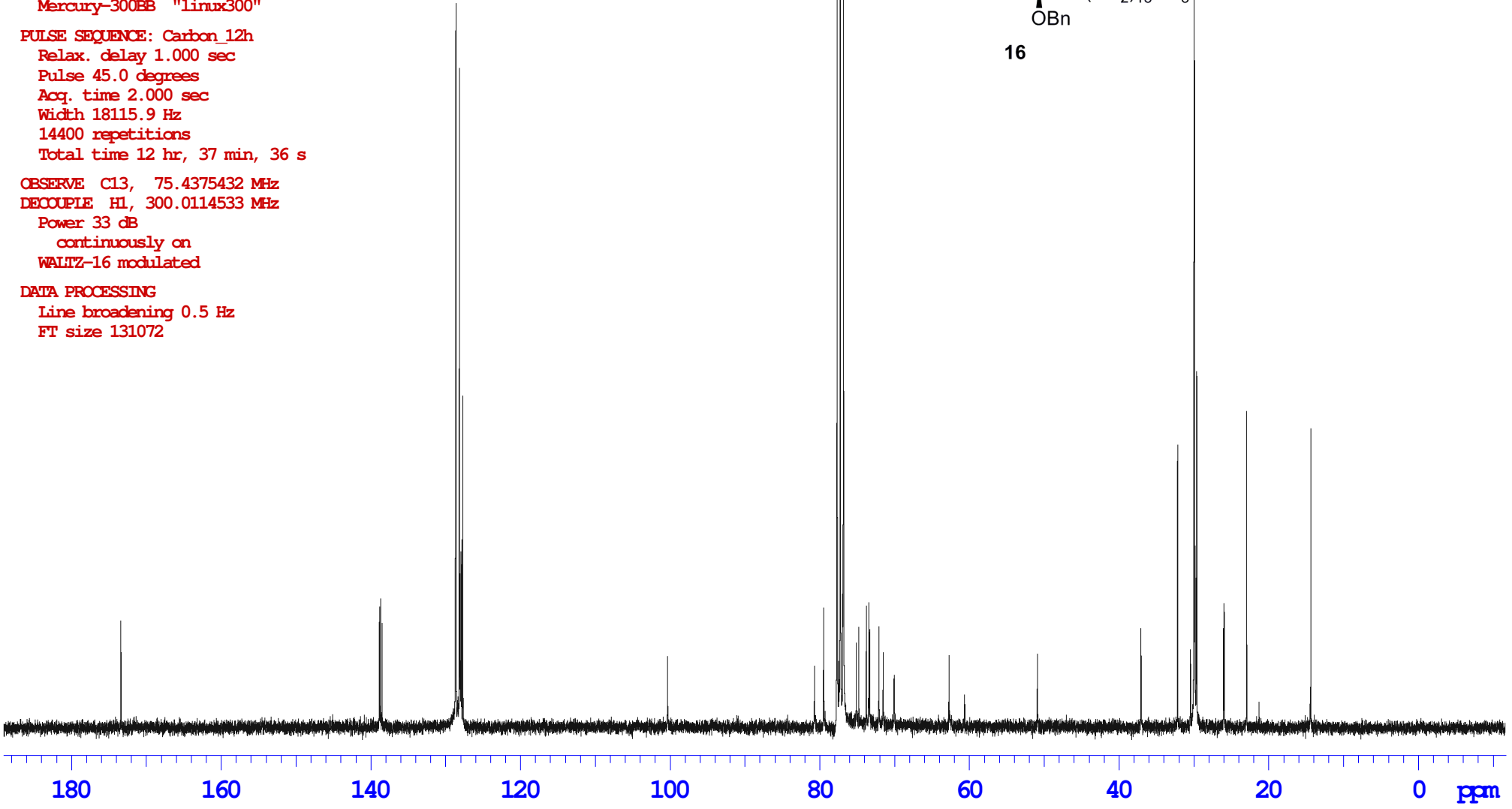

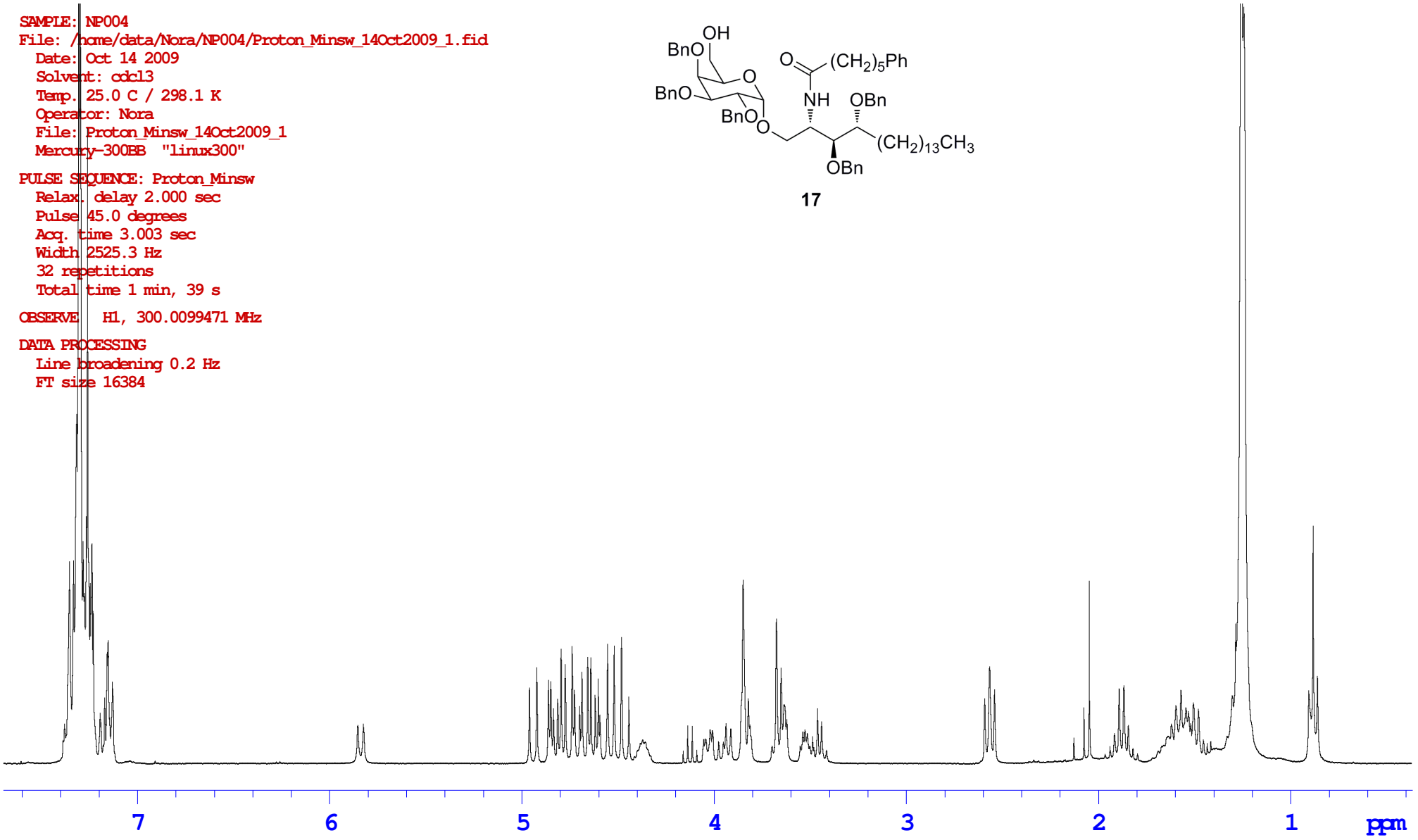
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Date: Oct 14 2009
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: Nora
File: Proton_Minsw_14Oct2009_1
Mercury-300BB "linux300"
PULSE SEQUENCE: Proton_Minsw
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 3.003 sec
Width 2525.3 Hz
32 repetitions
Total time 1 min, 39 s
OBSERVE H1, 300.0099471 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 16384
OH
BnO
O
BnO
BnO
O
O
(CH2)5Ph
NH
OBn
(CH2)13CH3
OBn
17
7
6
5
4
3
2
1
ppm

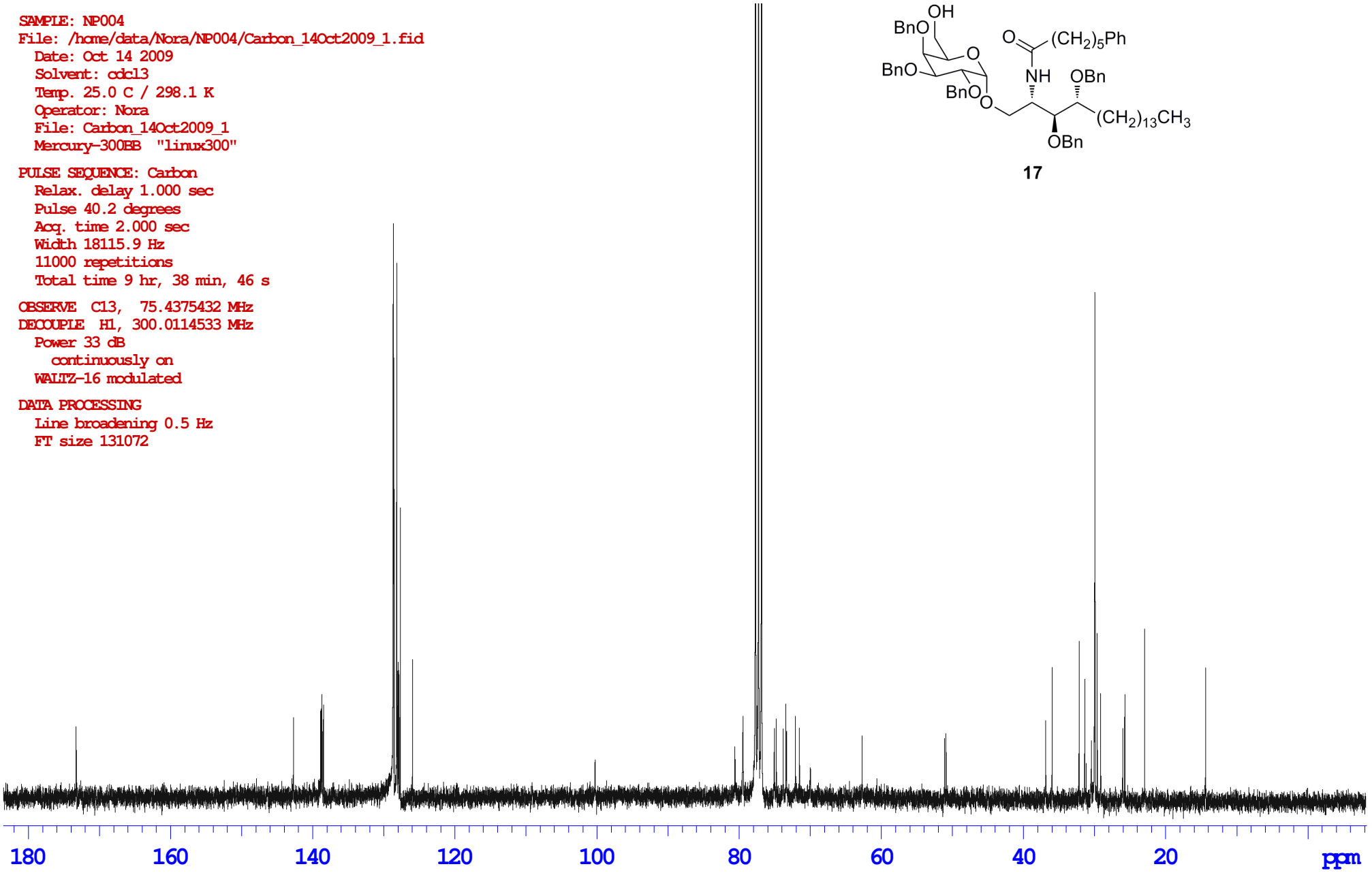
SAMPLE: NP004
File: /home/data/Nora/NP004/Carbon_14Oct2009_1.fid
Date: Oct 14 2009
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: Nora
File: Carbon_14Oct2009_1
Mercury-300BB "linux300"

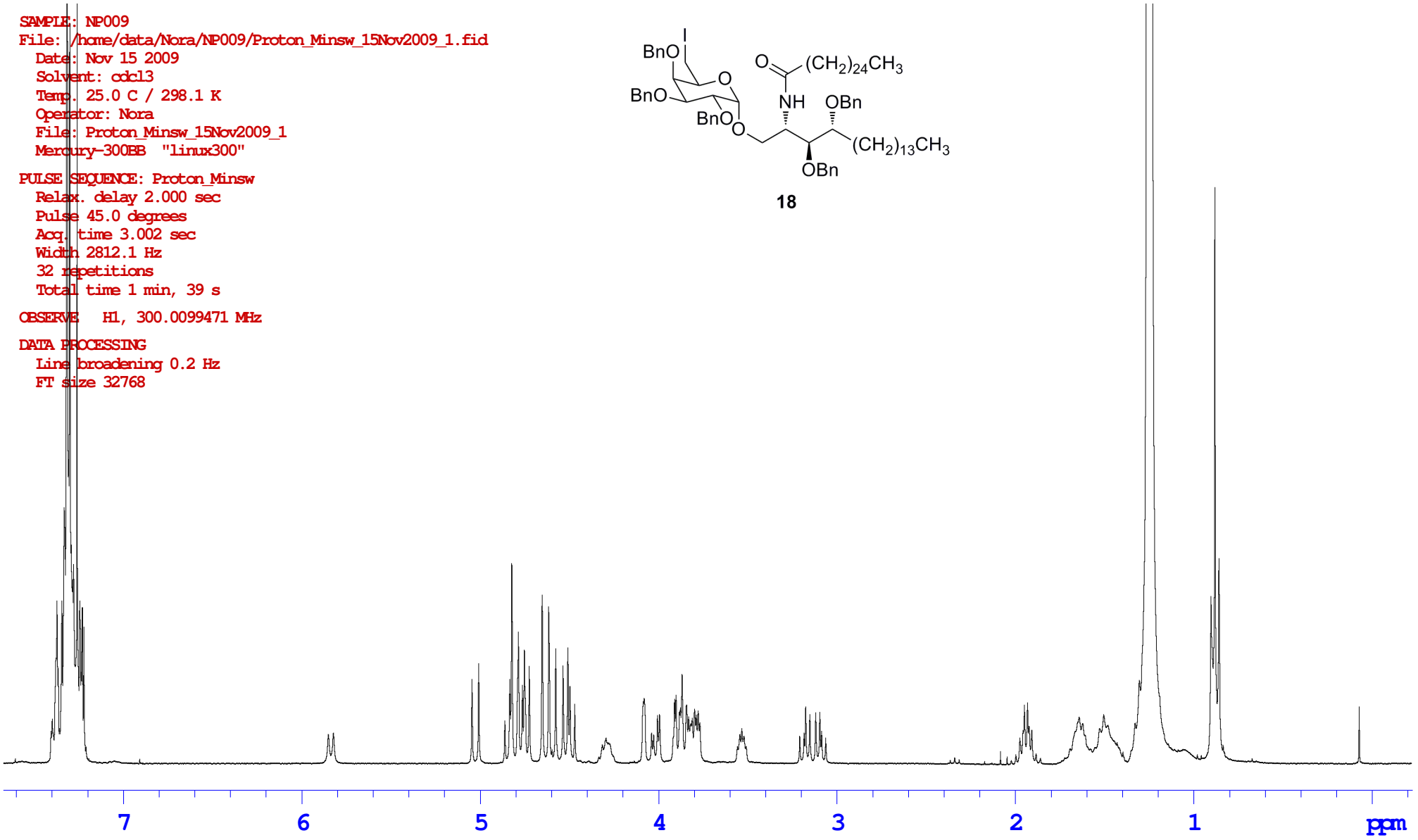
PULSE SEQUENCE: Carbon
Relax. delay 1.000 sec
Pulse 40.2 degrees
Acq. time 2.000 sec
Width 18115.9 Hz
11000 repetitions
Total time 9 hr, 38 min, 46 s

OBSERVE C13, 75.4375432 MHz
DECOUPLE H1, 300.0114533 MHz
Power 33 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072

17

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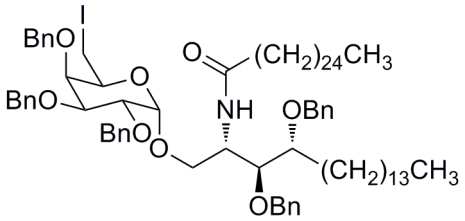
File: /home/data/Nora/NP009/Proton_Minsw_15Nov2009_1.fid
Date: Nov 15 2009
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: Nora
File: Proton_Minsw_15Nov2009_1
Mercury-300BB "linux300"
PULSE SEQUENCE: Proton_Minsw
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 3.002 sec
Width 2812.1 Hz
32 repetitions
Total time 1 min, 39 s
OBSERVE H1, 300.0099471 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
BnO
BnO
BnO
O
O
O
NH
OBn
OBn
(CH2)24CH3
(CH2)13CH3
18
7
6
5
4
3
2
1
ppm

SAMPLE: NP009
File: /home/data/Nora/NP009/Carbon_12h_15Nov2009_1.fid
Date: Nov 15 2009
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: Nora
File: Carbon_12h_15Nov2009_1
Mercury-300BB "linux300"

PULSE SEQUENCE: Carbon_12h
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.000 sec
Width 18115.9 Hz
17000 repetitions
Total time 14 hr, 54 min, 21 s

OBSERVE C13, 75.4378240 MHz
DECOUPLE H1, 300.0114533 MHz
Power 33 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072



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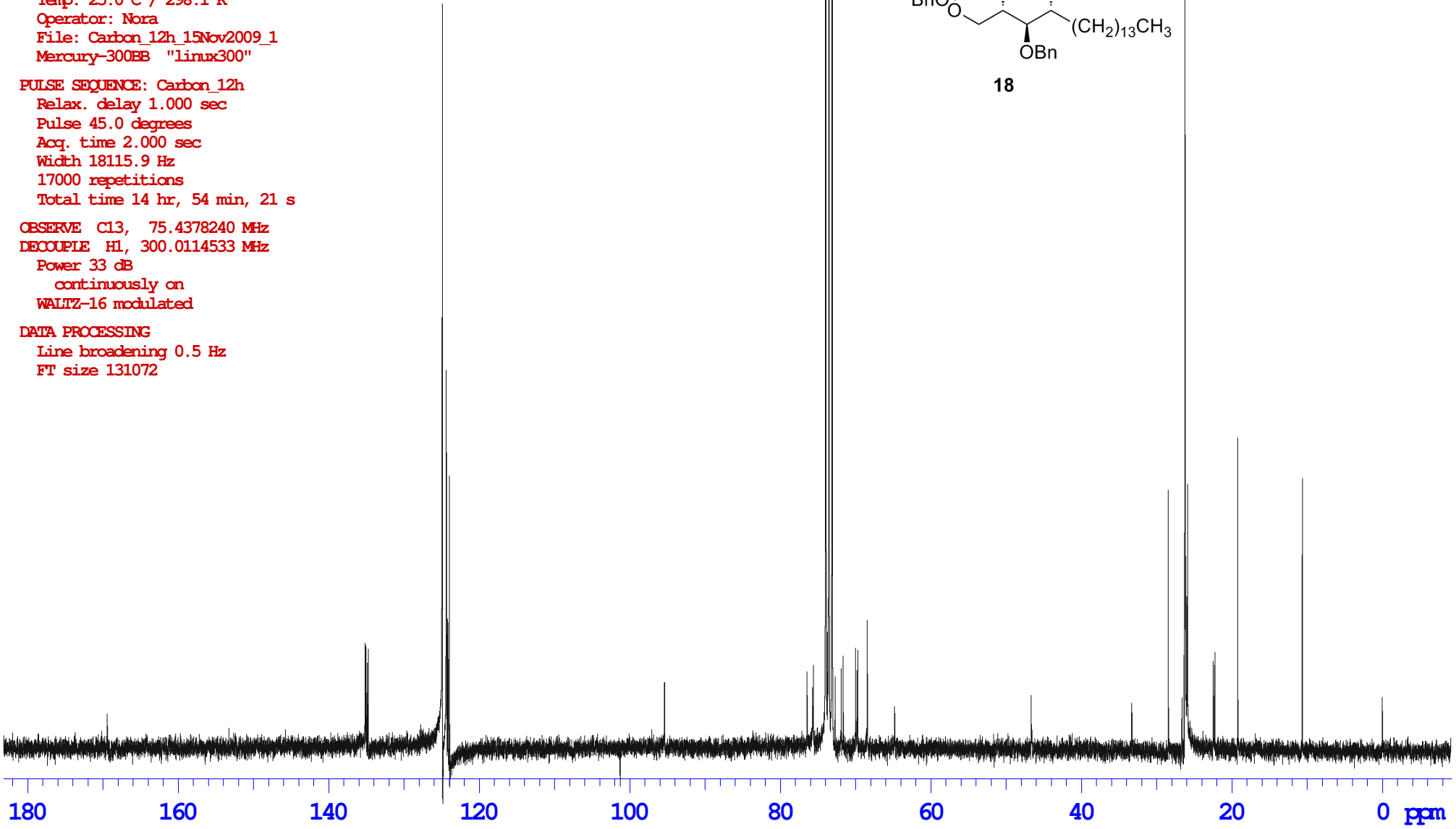

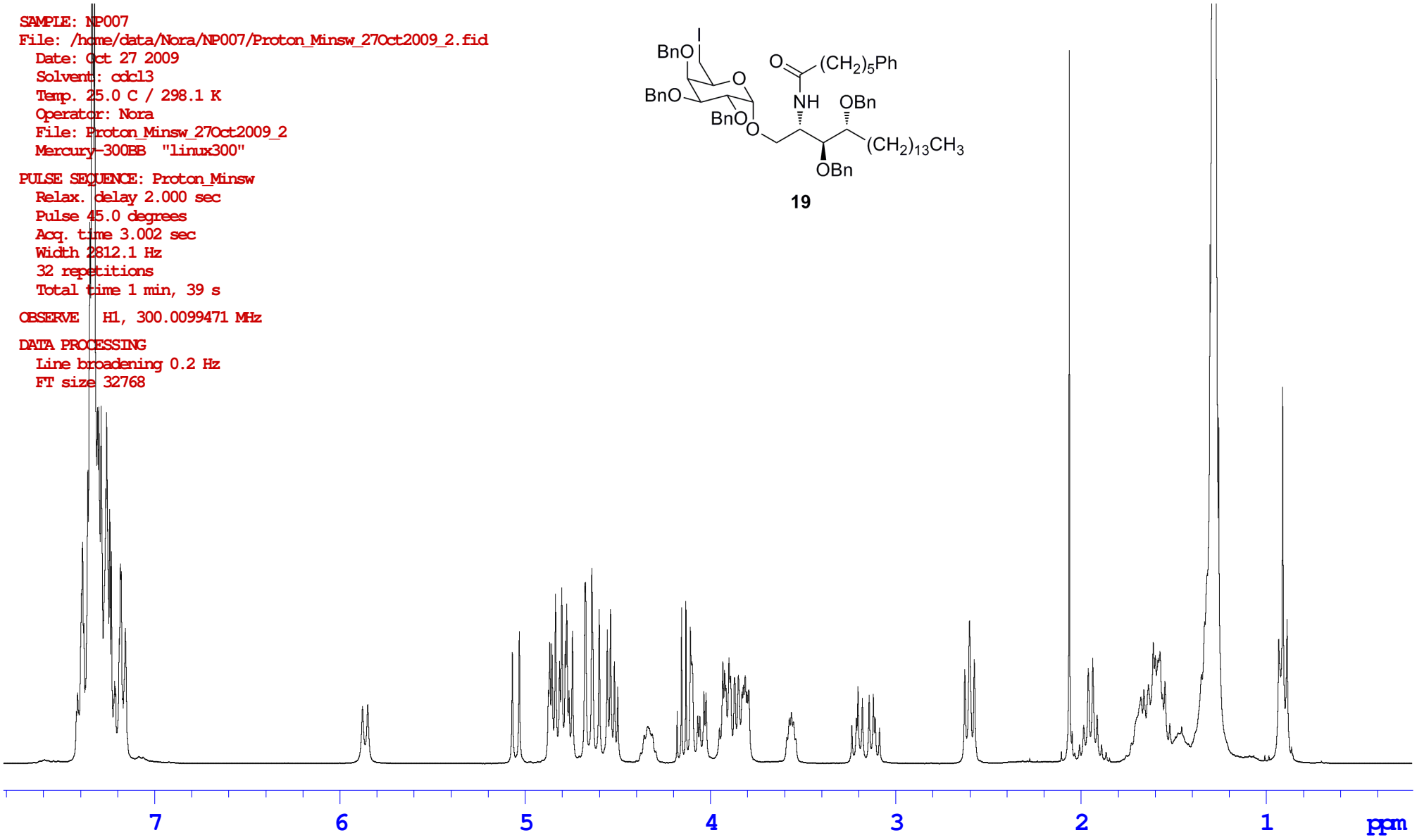
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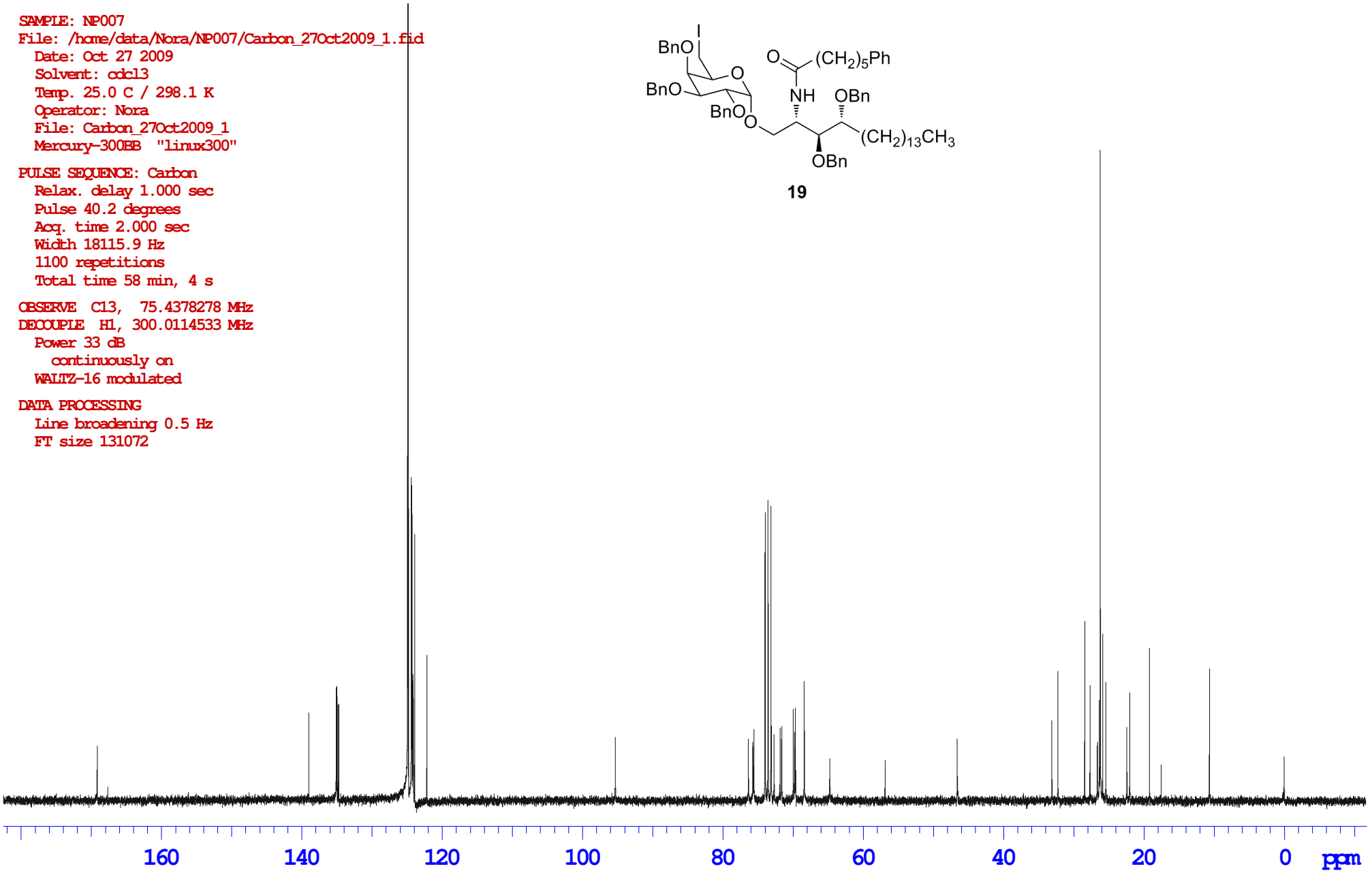
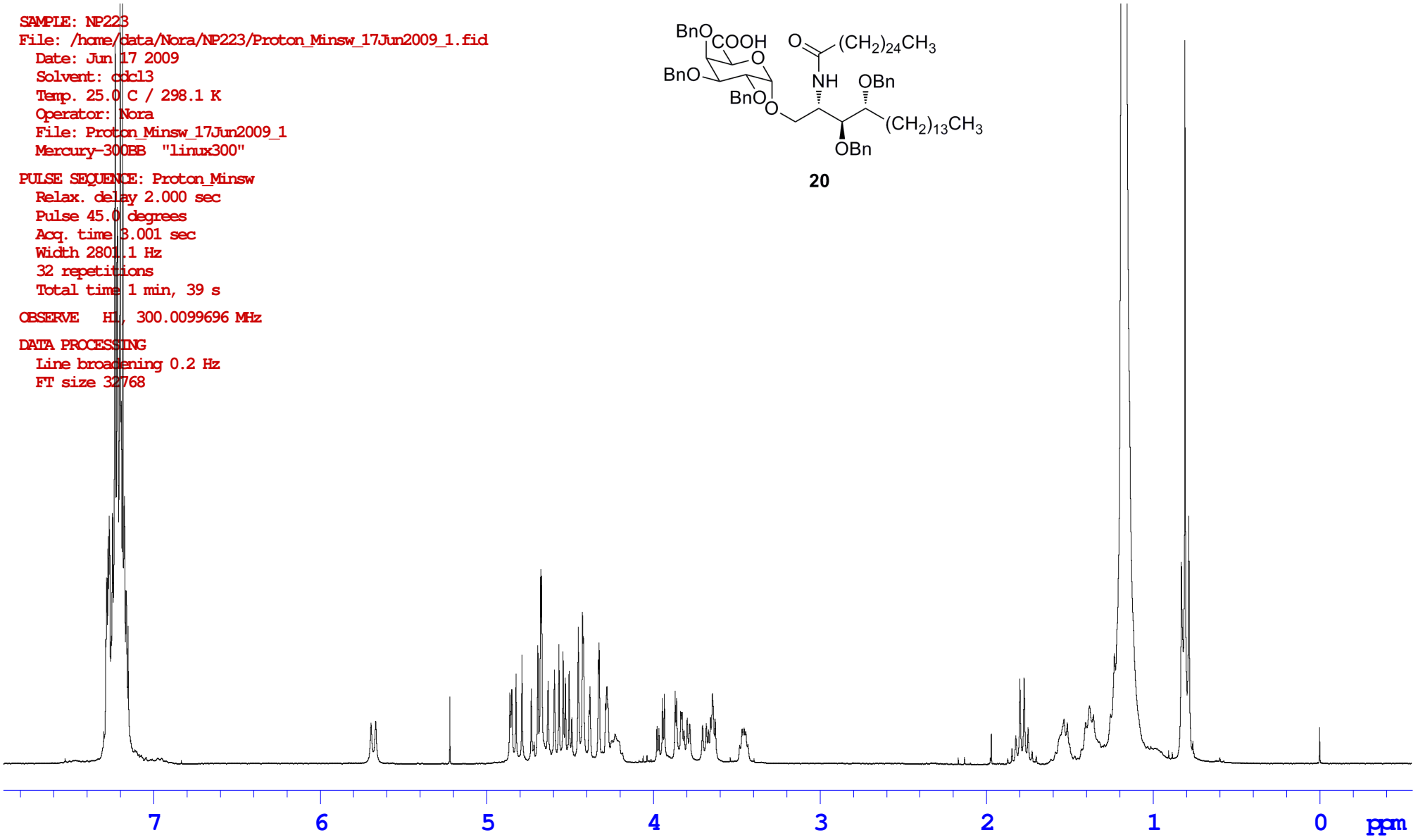
Date: Oct 27 2009
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: Nora
File: Proton_Minsw_27Oct2009_2
Mercury-300BB "linux300"
PULSE SEQUENCE: Proton_Minsw
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 3.002 sec
Width 2812.1 Hz
32 repetitions
Total time 1 min, 39 s
OBSERVE H1, 300.0099471 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
BnO
I
O
BnO
BnO
O
O
(CH2)5Ph
NH
OBn
(CH2)13CH3
OBn
19
7
6
5
4
3
2
1
ppm

BnO
BnO
BnO
O
O
O
NH
OBn
OBn
(CH2)5Ph
(CH2)13CH3

19


SAMPLE: NP007
File: /home/data/Nora/NP007/Carbon_27Oct2009_1.fid
Date: Oct 27 2009
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: Nora
File: Carbon_27Oct2009_1
Mercury-300BB "linux300"
PULSE SEQUENCE: Carbon
Relax. delay 1.000 sec
Pulse 40.2 degrees
Acq. time 2.000 sec
Width 18115.9 Hz
1100 repetitions
Total time 58 min, 4 s
OBSERVE C13, 75.4378278 MHz
DECOUPLE H1, 300.0114533 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
160
140
120
100
80
60
40
20
0
ppm


SAMPLE: NP223
File: /home/data/Nora/NP223/Proton_Minsw_17Jun2009_1.fid
Date: Jun 17 2009
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: Nora
File: Proton_Minsw_17Jun2009_1
Mercury-300BB "linux300"
PULSE SEQUENCE: Proton_Minsw
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 3.001 sec
Width 2801.1 Hz
32 repetitions
Total time 1 min, 39 s
OBSERVE H1, 300.0099696 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
BnO
COOH
O
BnO
BnO
O
O
(CH2)24CH3
NH
OBn
(CH2)13CH3
OBn
20
7
6
5
4
3
2
1
0
ppm

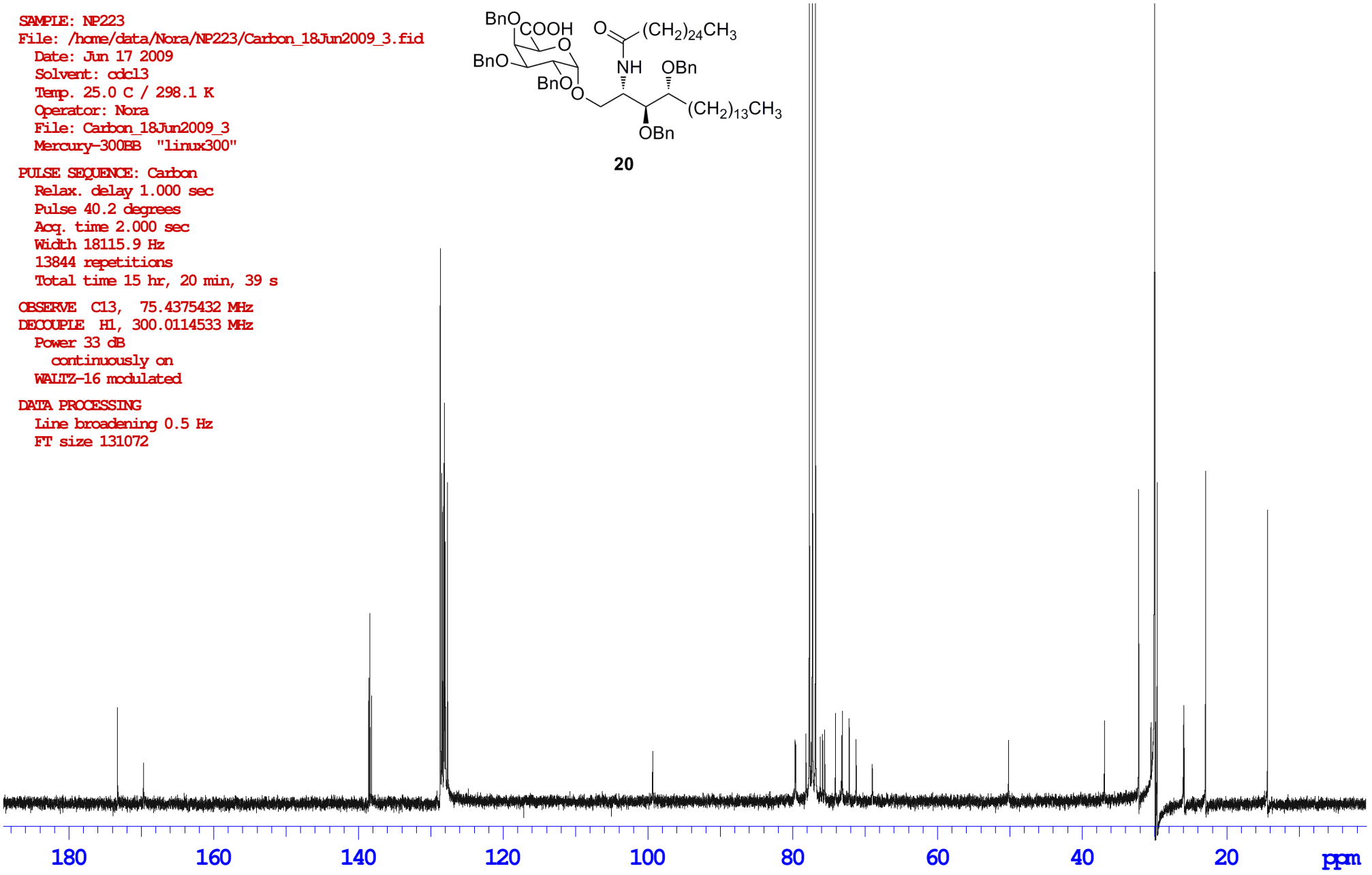
SAMPLE: NP223
File: /home/data/Nora/NP223/Carbon_18Jun2009_3.fid
Date: Jun 17 2009
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: Nora
File: Carbon_18Jun2009_3
Mercury-300BB "linux300"

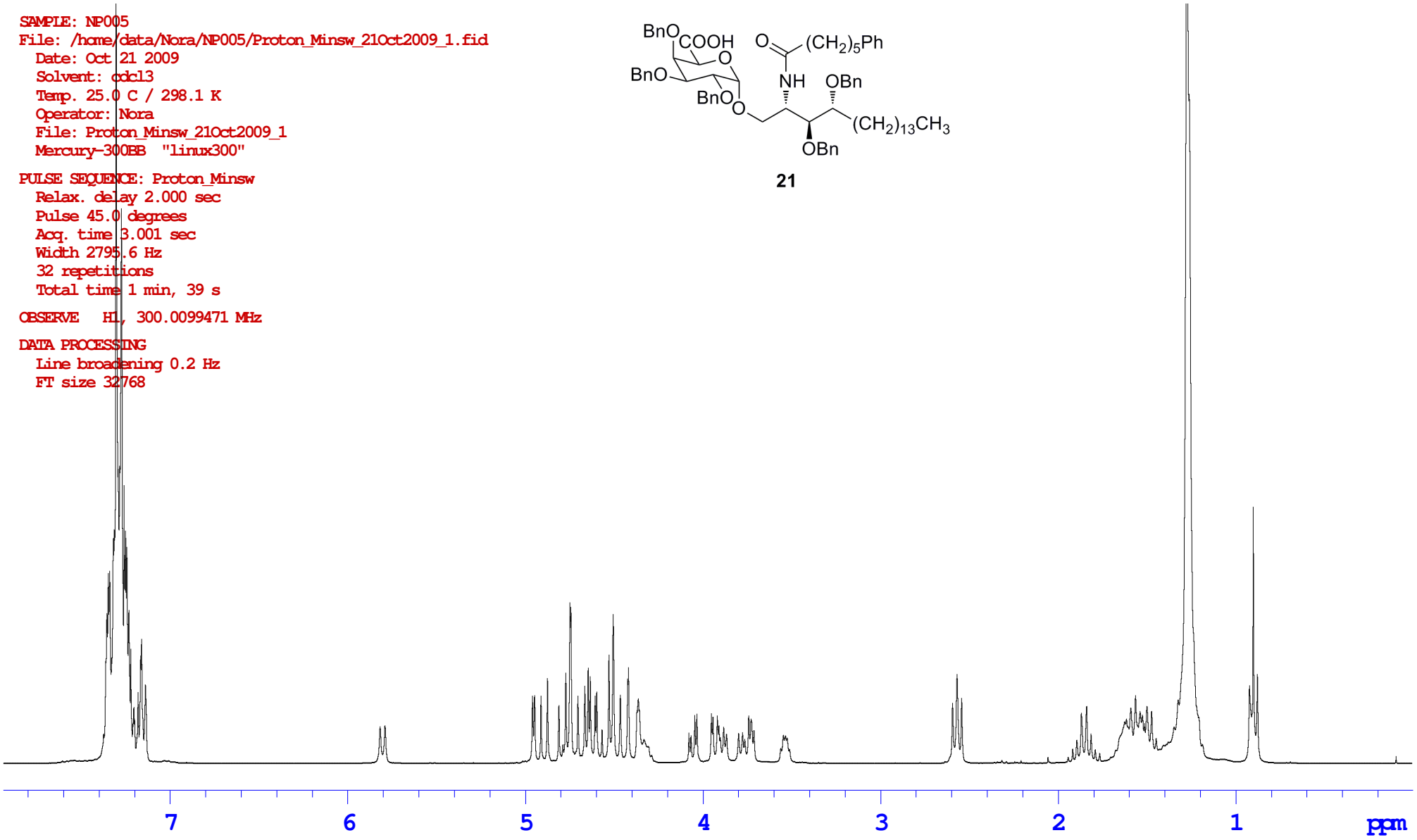
PULSE SEQUENCE: Carbon
Relax. delay 1.000 sec
Pulse 40.2 degrees
Acq. time 2.000 sec
Width 18115.9 Hz
13844 repetitions
Total time 15 hr, 20 min, 39 s

OBSERVE C13, 75.4375432 MHz
DECOUPLE H1, 300.0114533 MHz
Power 33 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072

20

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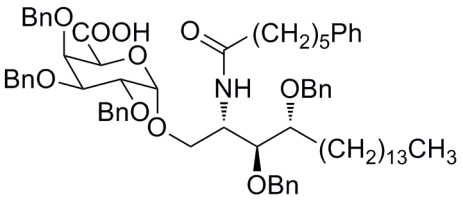
Date: Oct 21 2009
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: Nora
File: Proton_Minsw_21Oct2009_1
Mercury-300BB "linux300"
PULSE SEQUENCE: Proton_Minsw
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 3.001 sec
Width 2795.6 Hz
32 repetitions
Total time 1 min, 39 s
OBSERVE H1, 300.0099471 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
BnO
COOH
O
BnO
BnO
O
O
(CH2)5Ph
NH
OBn
(CH2)13CH3
OBn
21
7
6
5
4
3
2
1
ppm

SAMPLE: NP005
File: /home/data/Nora/NP005/Carbon_12h_21Oct2009_1.fid
Date: Oct 21 2009
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: Nora
File: Carbon_12h_21Oct2009_1
Mercury-300BB "linux300"

PULSE SEQUENCE: Carbon_12h
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.000 sec
Width 18115.9 Hz
11000 repetitions
Total time 9 hr, 38 min, 46 s

OBSERVE C13, 75.4375432 MHz
DECOUPLE H1, 300.0114533 MHz
Power 33 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072



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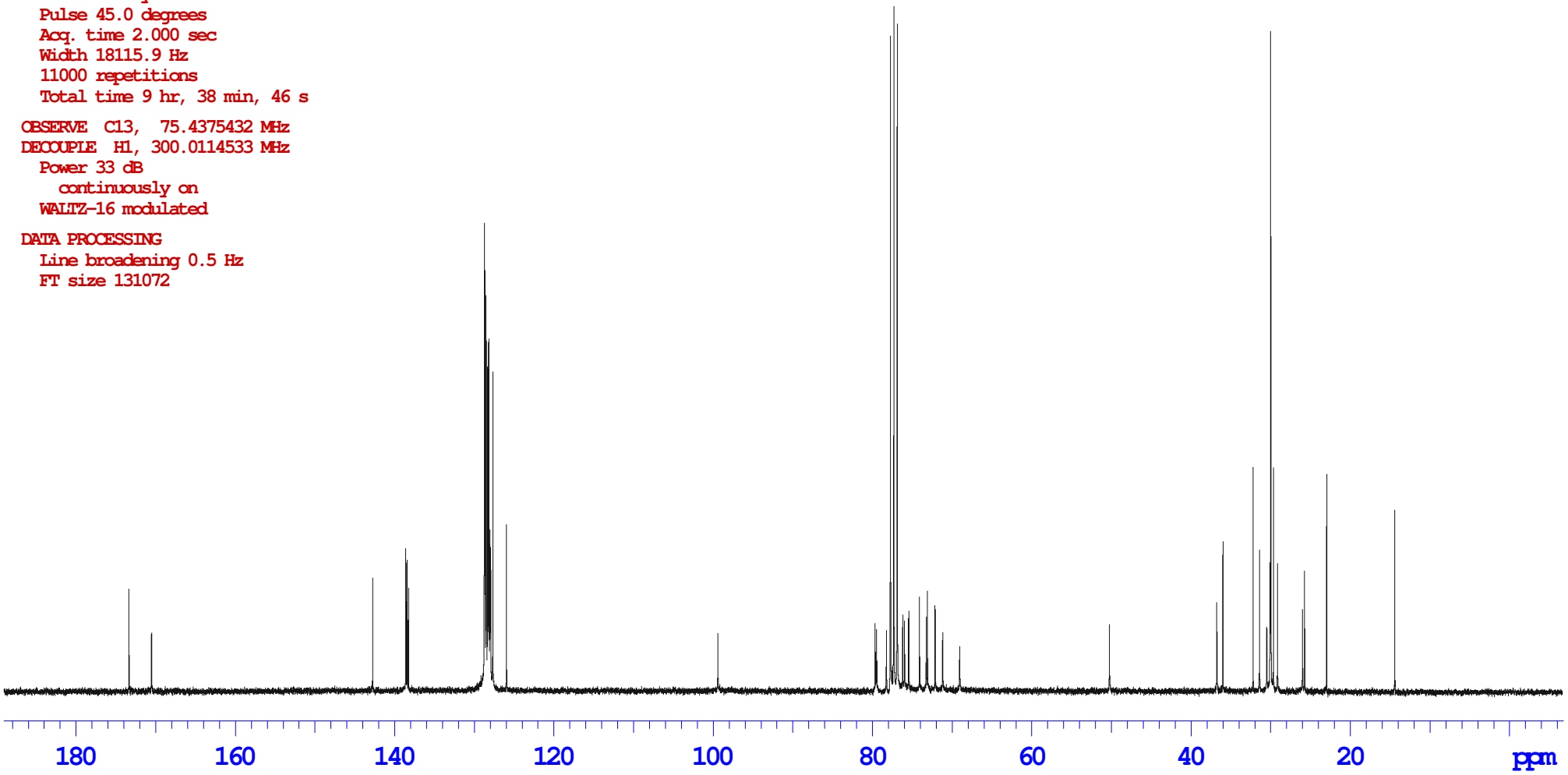


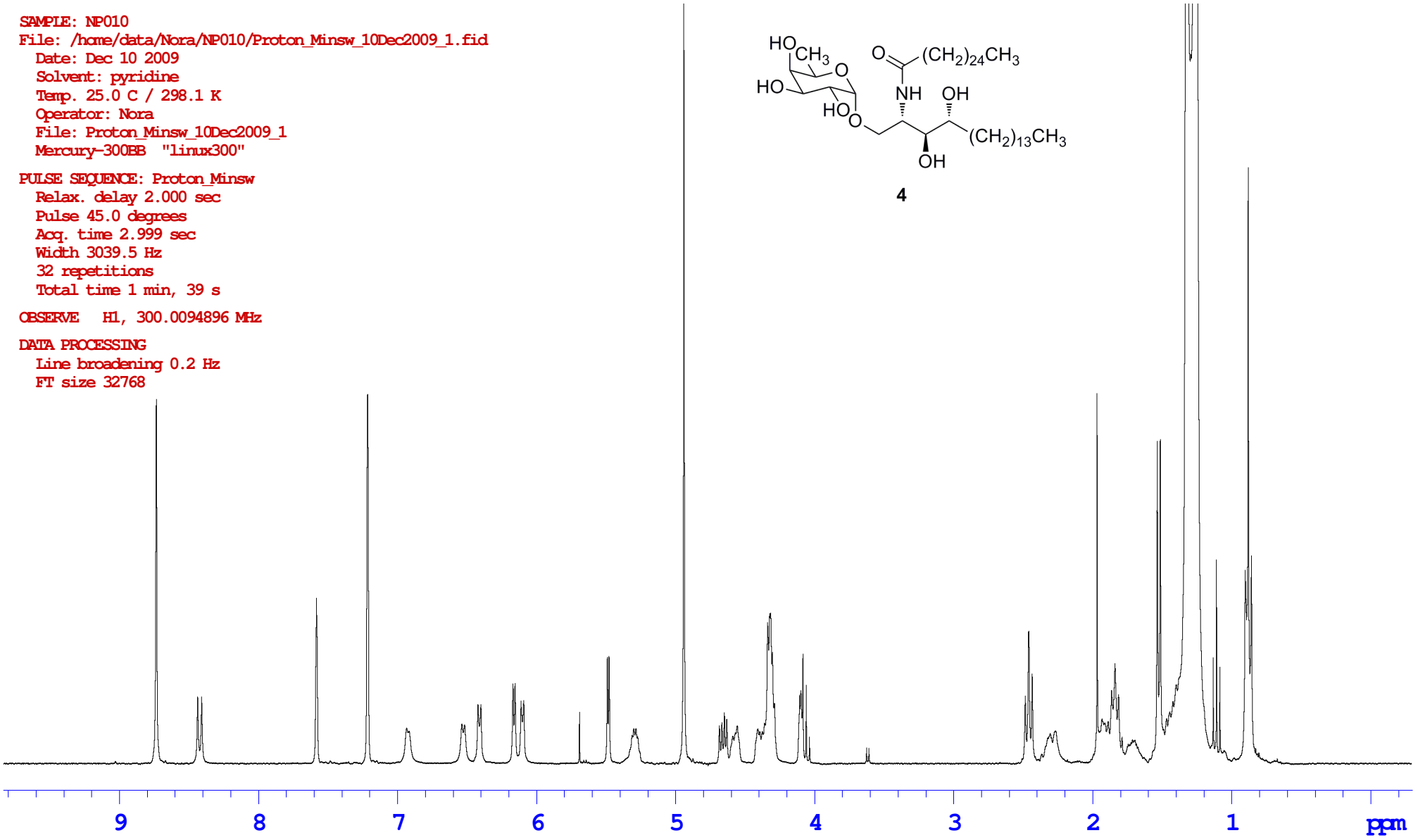
SAMPLE: NP010
File: /home/data/Nora/NP010/Proton_Minsw_10Dec2009_1.fid
Date: Dec 10 2009
Solvent: pyridine
Temp. 25.0 C / 298.1 K
Operator: Nora
File: Proton_Minsw_10Dec2009_1
Mercury-300BB "linux300"

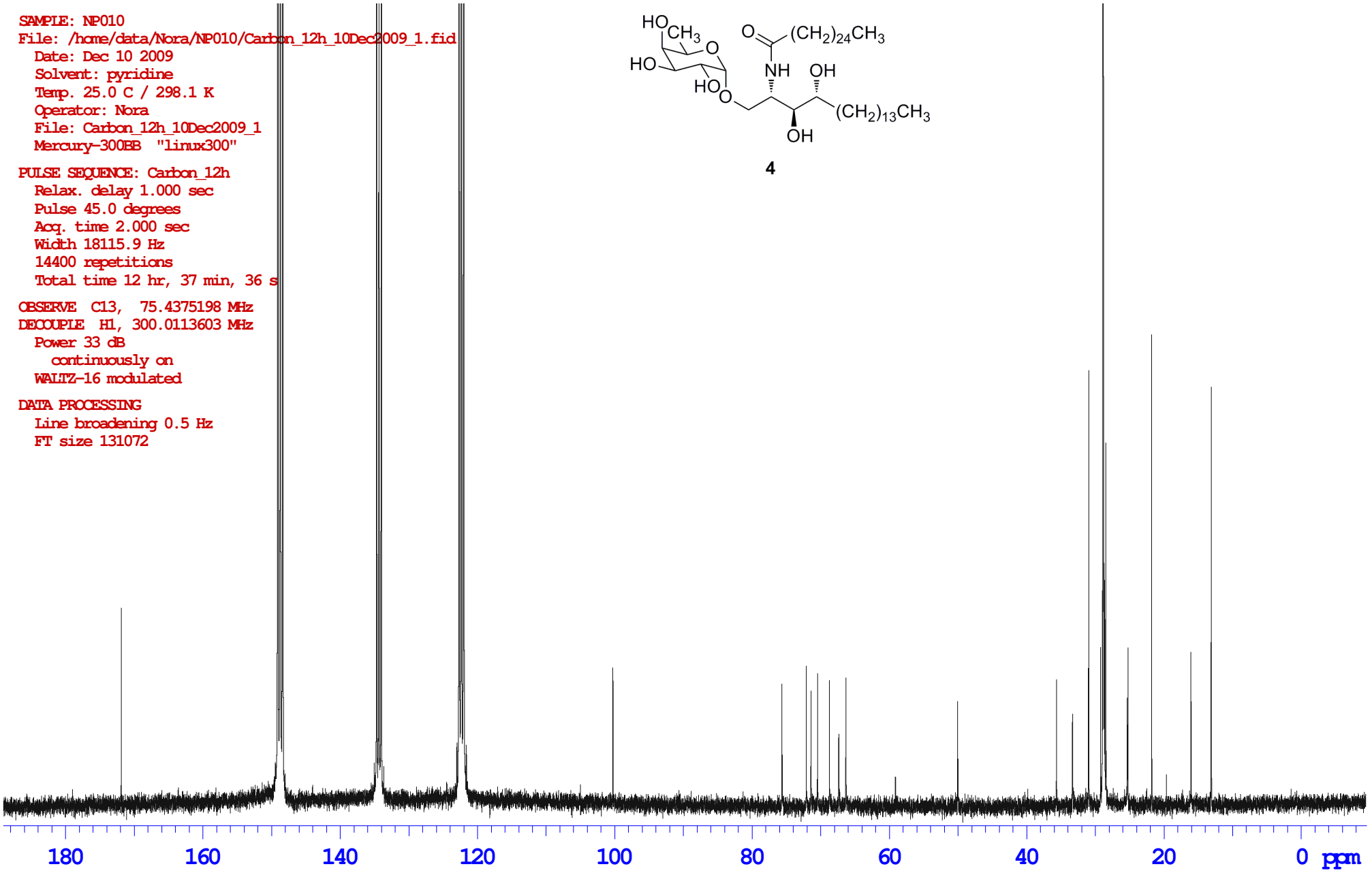
PULSE SEQUENCE: Proton_Minsw
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.999 sec
Width 3039.5 Hz
32 repetitions
Total time 1 min, 39 s

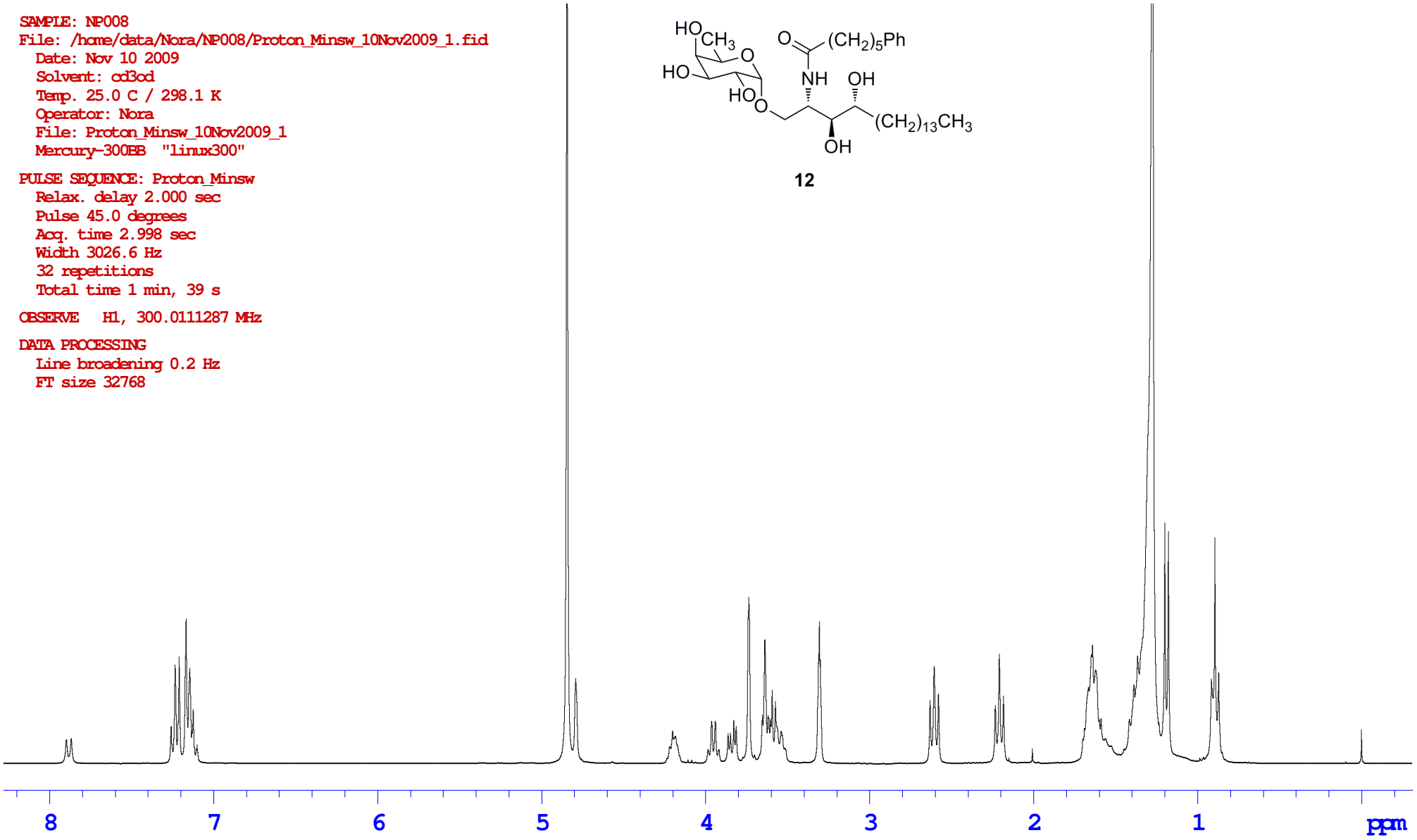
OBSERVE H1, 300.0094896 MHz

DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768

4




SAMPLE: NP010
File: /home/data/Nora/NP010/Carbon_12h_10Dec2009_1.fid
Date: Dec 10 2009
Solvent: pyridine
Temp. 25.0 C / 298.1 K
Operator: Nora
File: Carbon_12h_10Dec2009_1
Mercury-300BB "linux300"
PULSE SEQUENCE: Carbon_12h
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.000 sec
Width 18115.9 Hz
14400 repetitions
Total time 12 hr, 37 min, 36 s
OBSERVE C13, 75.4375198 MHz
DECOUPLE H1, 300.0113603 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
HO
CH3
O
HO
HO
O
O
(CH2)24CH3
NH
OH
(CH2)13CH3
OH
4
180
160
140
120
100
80
60
40
20
0 ppm


SAMPLE: NP008
File: /home/data/Nora/NP008/Proton_Minsw_10Nov2009_1.fid
Date: Nov 10 2009
Solvent: cd3od
Temp. 25.0 C / 298.1 K
Operator: Nora
File: Proton_Minsw_10Nov2009_1
Mercury-300BB "linux300"
PULSE SEQUENCE: Proton_Minsw
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 2.998 sec
Width 3026.6 Hz
32 repetitions
Total time 1 min, 39 s
OBSERVE H1, 300.0111287 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
HO
CH3
O
HO
HO
O
O
(CH2)5Ph
NH
OH
(CH2)13CH3
OH
12
8
7
6
5
4
3
2
1
ppm

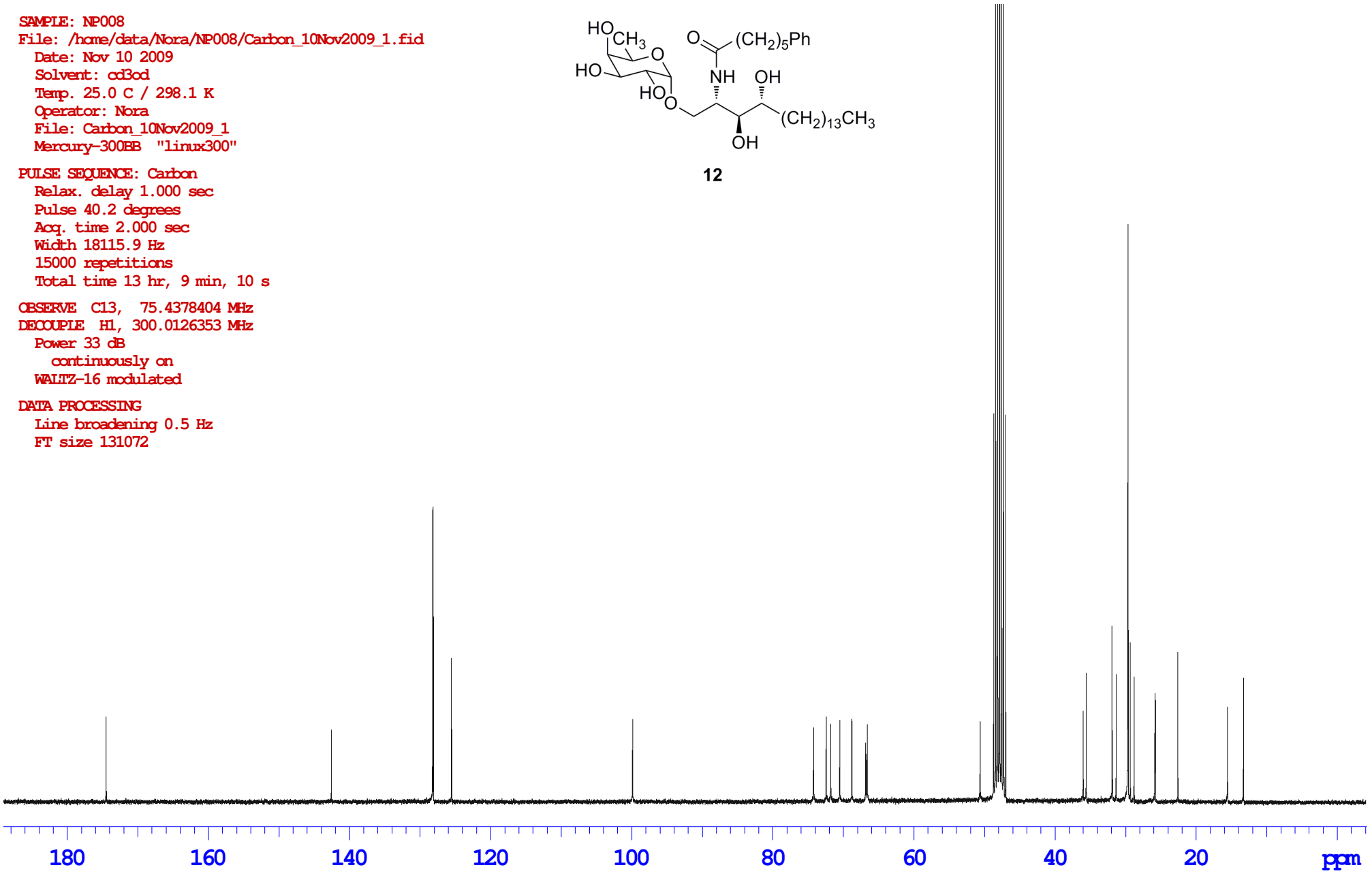
SAMPLE: NP008
File: /home/data/Nora/NP008/Carbon_10Nov2009_1.fid
Date: Nov 10 2009
Solvent: cd3od
Temp. 25.0 C / 298.1 K
Operator: Nora
File: Carbon_10Nov2009_1
Mercury-300BB "linux300"

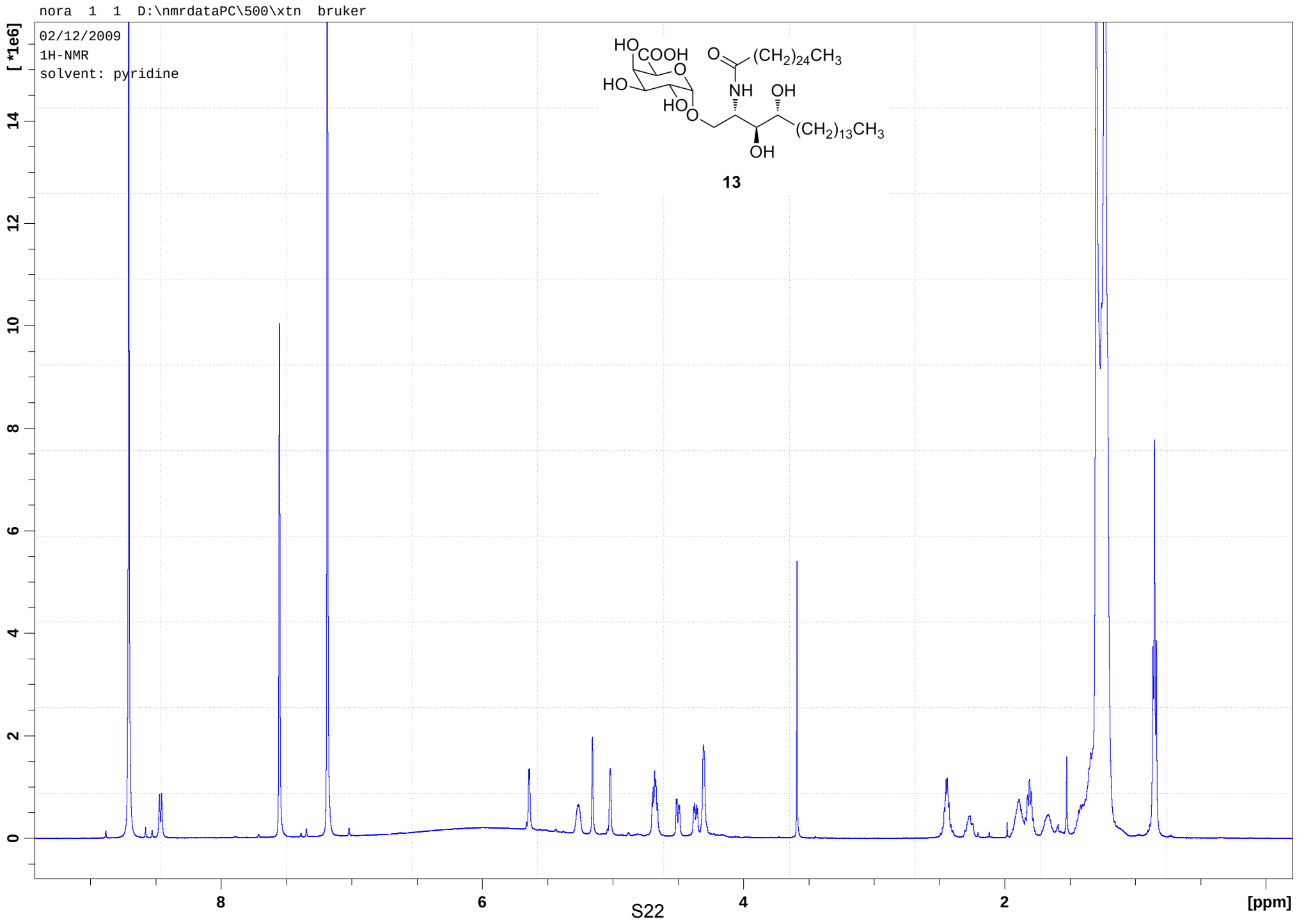
PULSE SEQUENCE: Carbon
Relax. delay 1.000 sec
Pulse 40.2 degrees
Acq. time 2.000 sec
Width 18115.9 Hz
15000 repetitions
Total time 13 hr, 9 min, 10 s

OBSERVE C13, 75.4378404 MHz
DECOUPLE H1, 300.0126353 MHz
Power 33 dB
continuously on
WALTZ-16 modulated

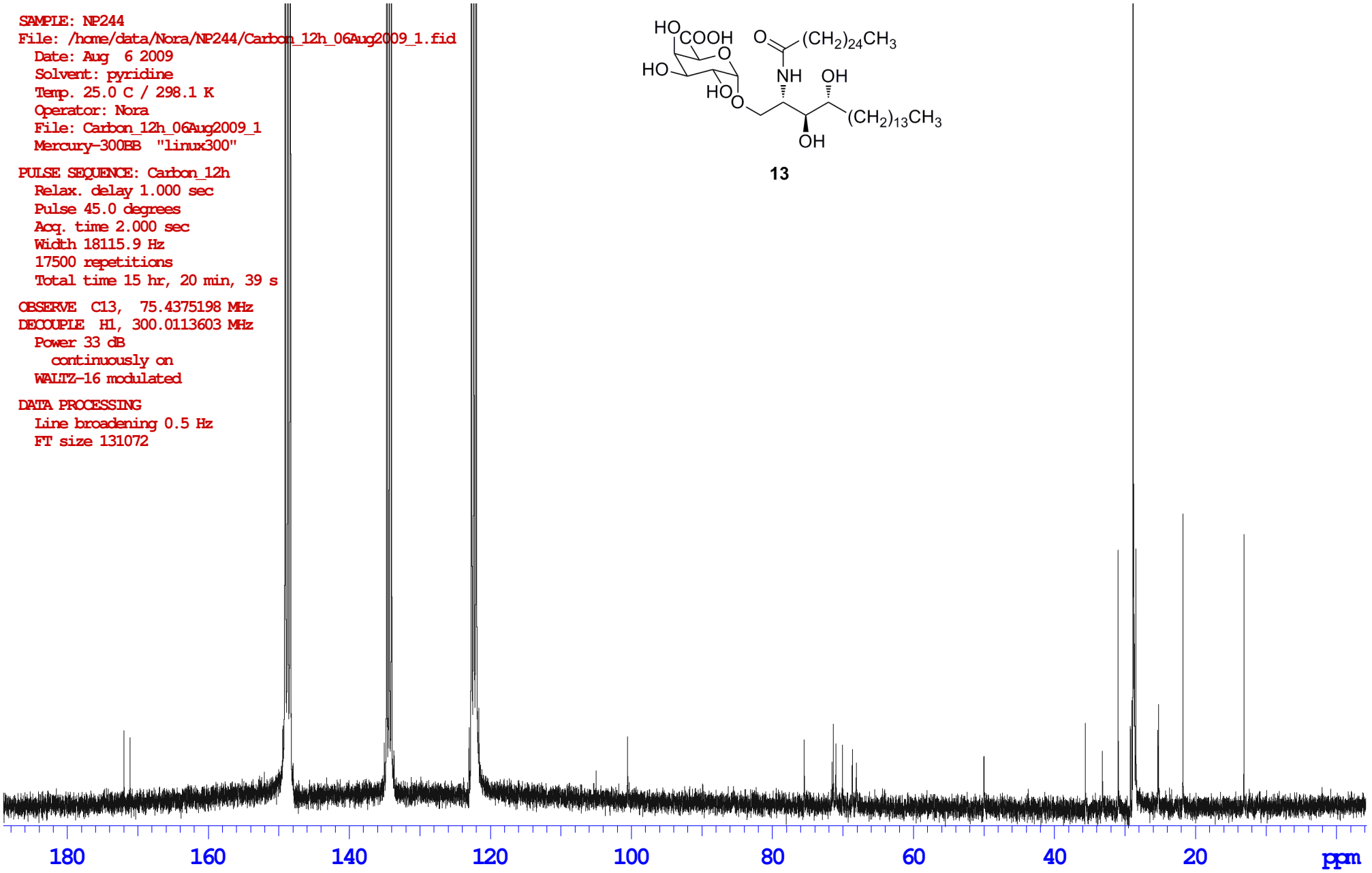
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072

12

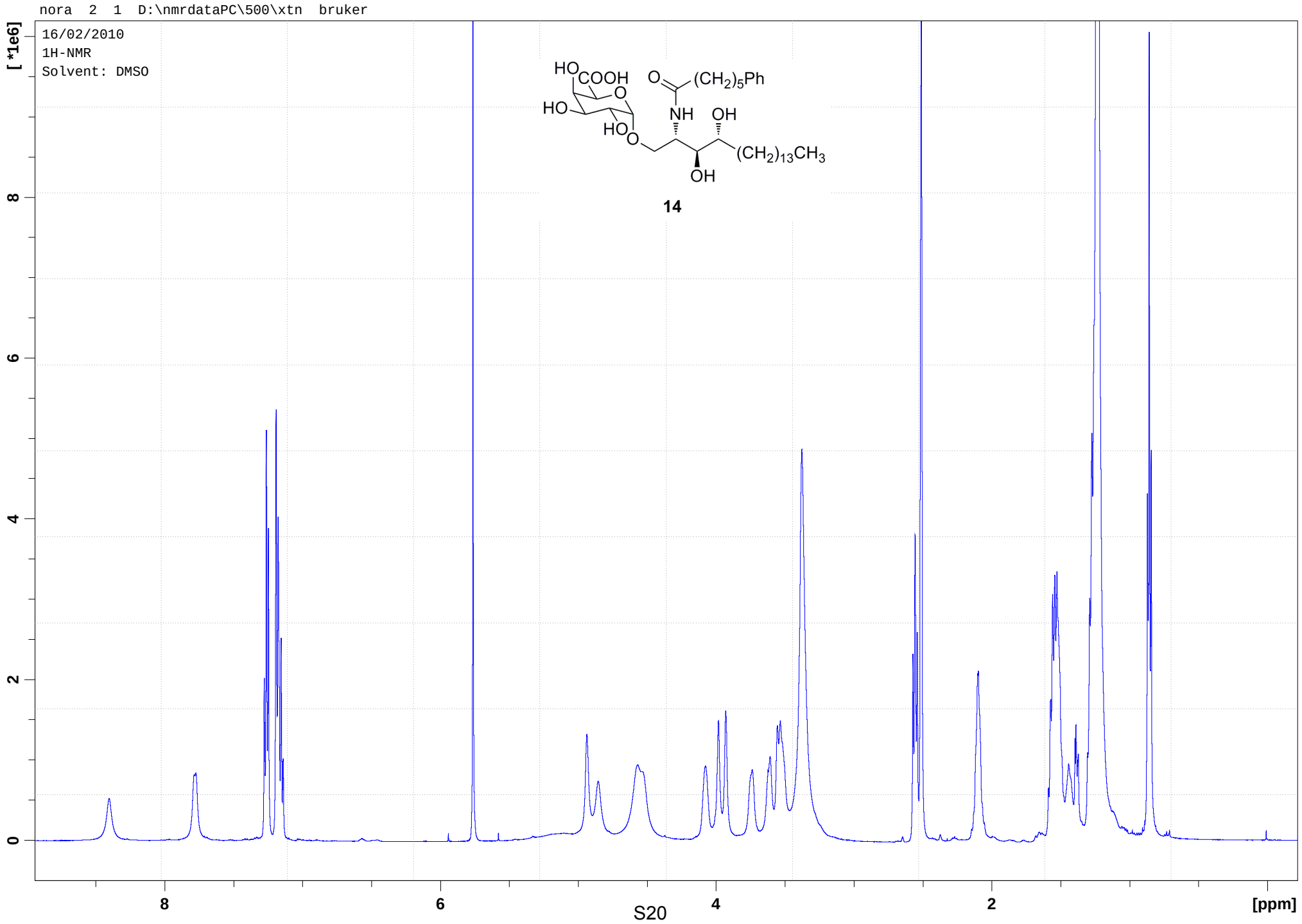



nora 1 1 D:\nmrdataPC\500\xtn bruker
02/12/2009
1H-NMR
solvent: pyridine
[*1e6]
0
2
4
6
8
10
12
14
8
6
4
2
[ppm]
HO
COOH
O
HO
HO
O
O
(CH2)24CH3
NH
OH
(CH2)13CH3
OH

13


SAMPLE: NP244
File: /home/data/Nora/NP244/Carbon_12h_06Aug2009_1.fid
Date: Aug 6 2009
Solvent: pyridine
Temp. 25.0 C / 298.1 K
Operator: Nora
File: Carbon_12h_06Aug2009_1
Mercury-300BB "linux300"
PULSE SEQUENCE: Carbon_12h
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.000 sec
Width 18115.9 Hz
17500 repetitions
Total time 15 hr, 20 min, 39 s
OBSERVE C13, 75.4375198 MHz
DECOUPLE H1, 300.0113603 MHz
Power 33 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
HO
COOH
O
HO
HO
O
O
(CH2)24CH3
NH
OH
(CH2)13CH3
OH
180
160
140
120
100
80
60
40
20
ppm

13


nora 2 1 D:\nmrdataPC\500\xtn bruker
16/02/2010
1H-NMR
Solvent: DMSO
[*1e6]
0
2
4
6
8
8
6
4
2
[ppm]
S20
HO
COOH
O
HO
HO
O
O
(CH2)5Ph
NH
OH
(CH2)13CH3
OH

14

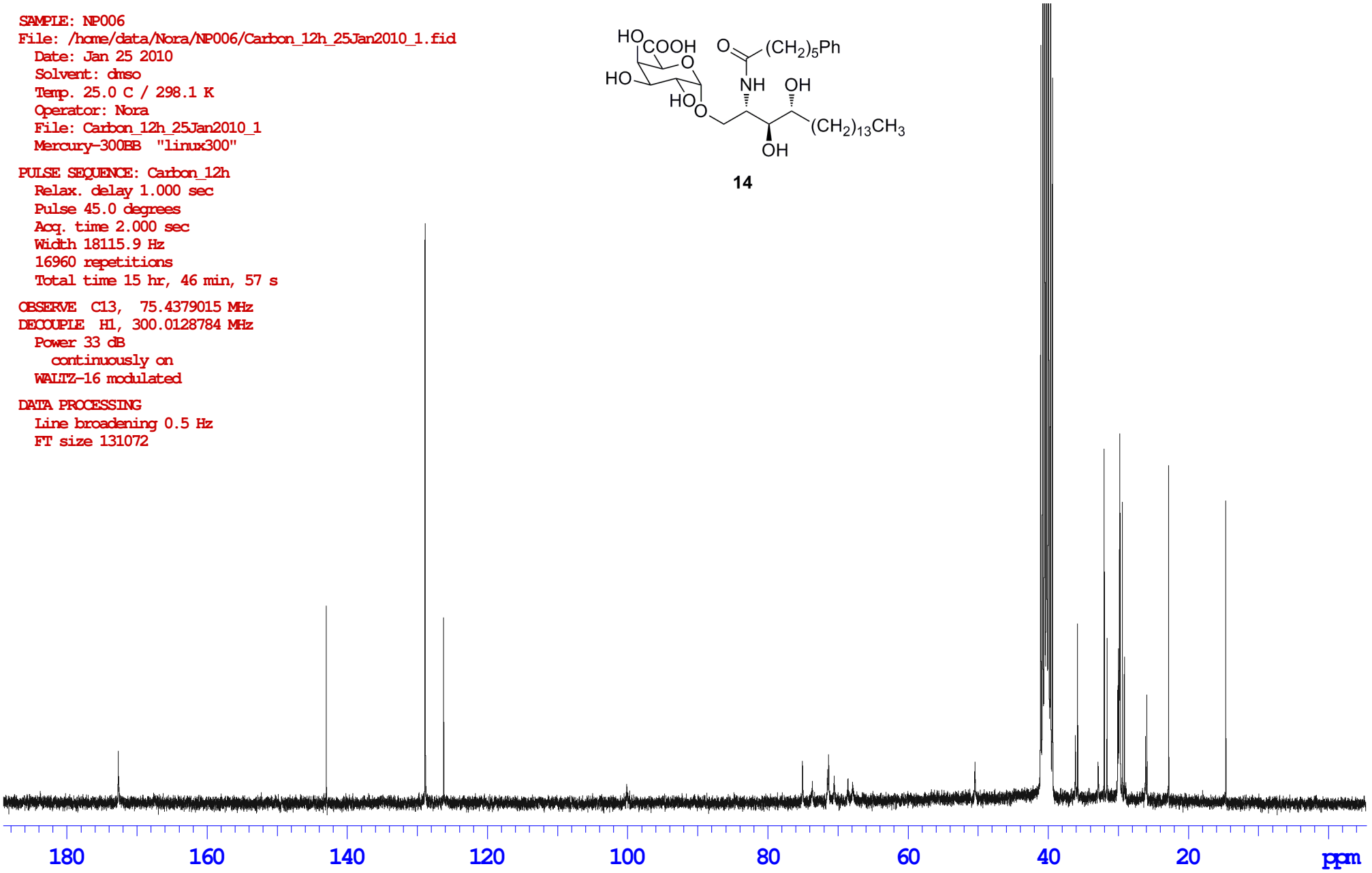
SAMPLE: NP006
File: /home/data/Nora/NP006/Carbon_12h_25Jan2010_1.fid
Date: Jan 25 2010
Solvent: dmso
Temp. 25.0 C / 298.1 K
Operator: Nora
File: Carbon_12h_25Jan2010_1
Mercury-300BB "linux300"

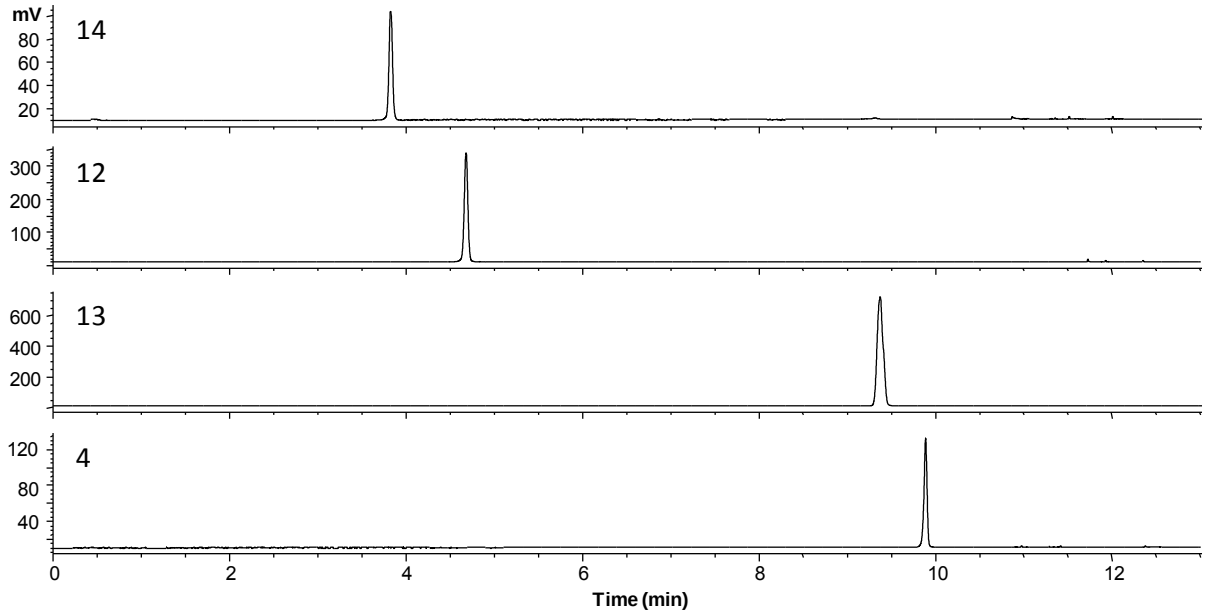
PULSE SEQUENCE: Carbon_12h
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.000 sec
Width 18115.9 Hz
16960 repetitions
Total time 15 hr, 46 min, 57 s

OBSERVE C13, 75.4379015 MHz
DECOUPLE H1, 300.0128784 MHz
Power 33 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072

14




mV
14
12
13
4
Time (min)