Electronic Supporting Information

Chemical synthesis and proinflammatory responses of monophosphoryl lipid A adjuvant candidates

Kaustabh K. Maiti, Michael DeCastro, Abu-Baker M. Abdel-Aal El-Sayed, Matthew I. Foote, Margreet A. Wolfert, and Geert-Jan Boons*

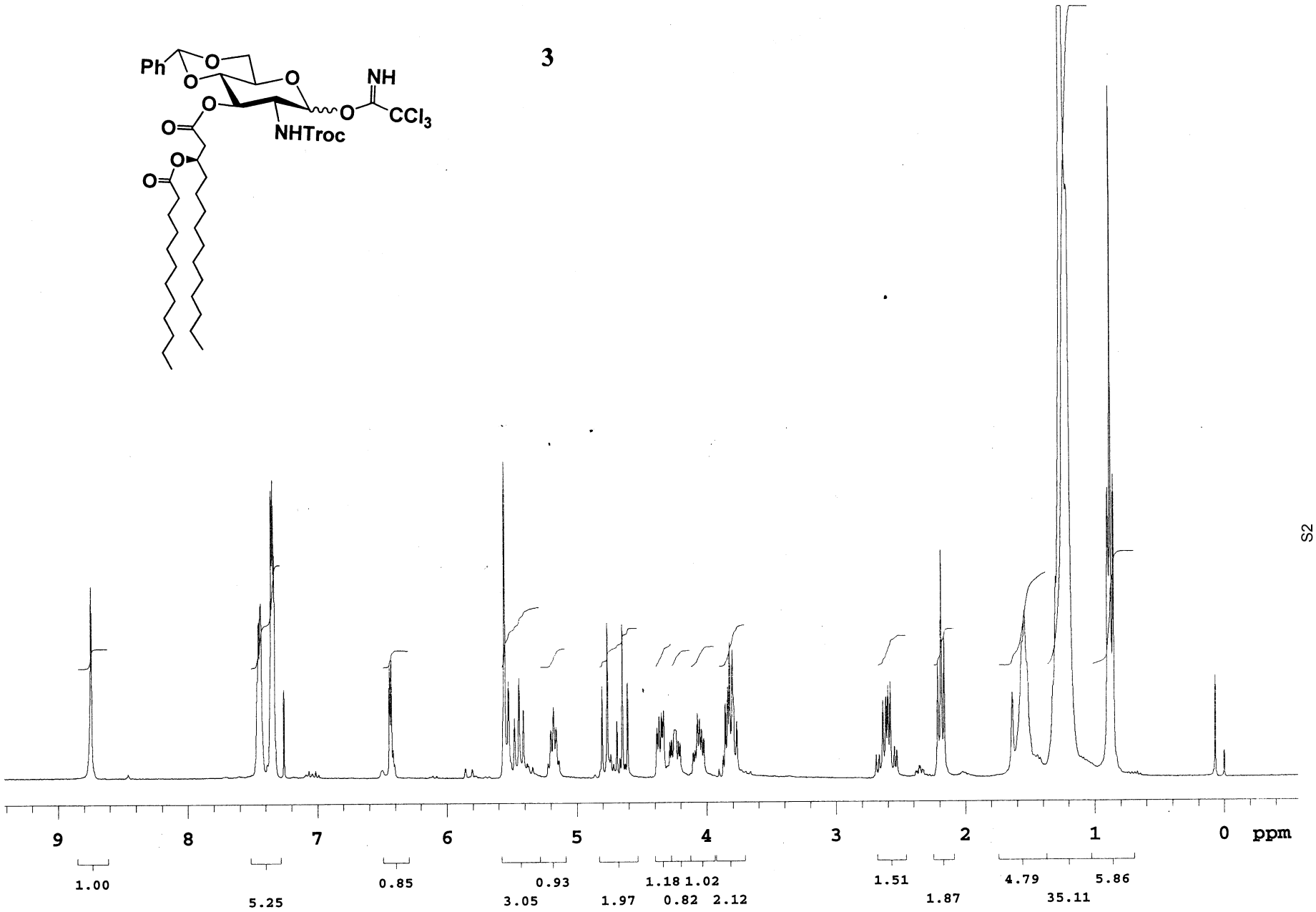
Eur. J. Org. Chem.

This PDF file includes:

NMR spectra synthetic compounds

Ph
O
O
O
O
O
O
NH
O
CCl3
NHTroc

3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7
6
5
4
3
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3.05
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1.18
1.02
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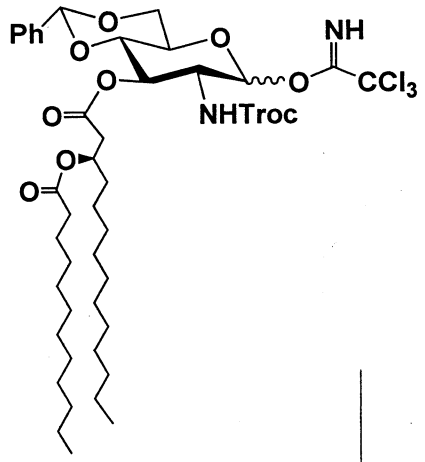
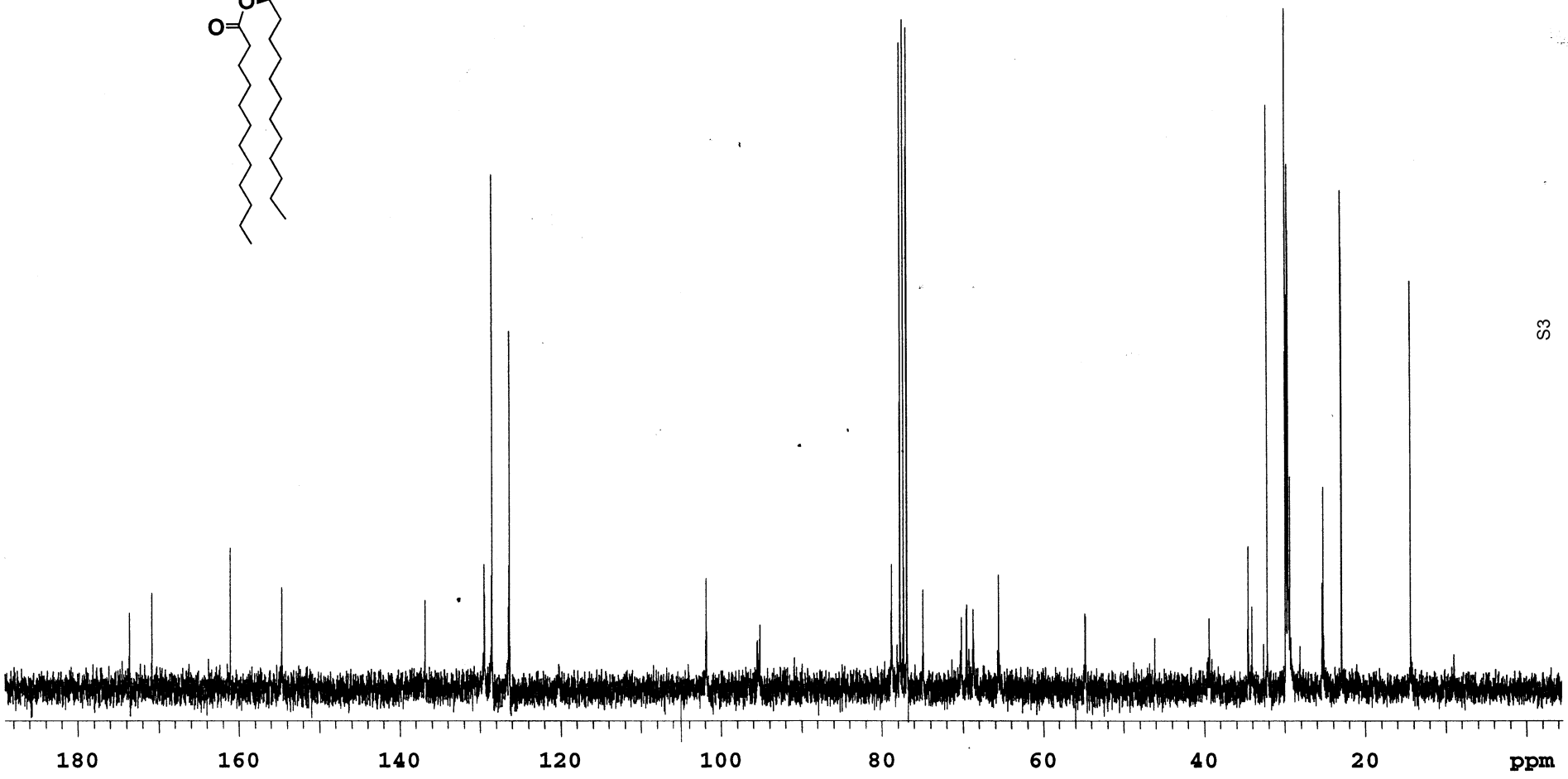
1.87
4.79
35.11
5.86

S2

3





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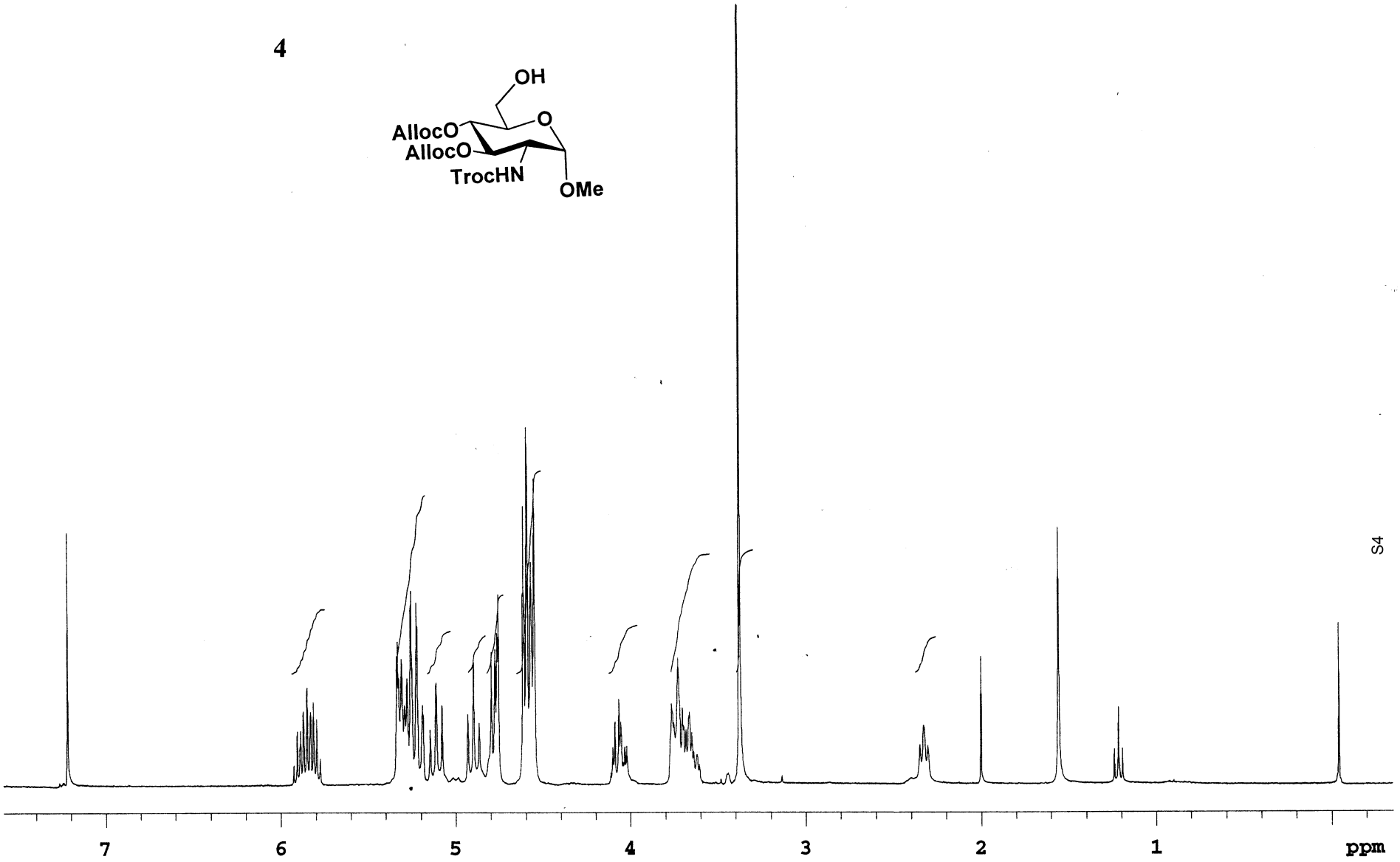

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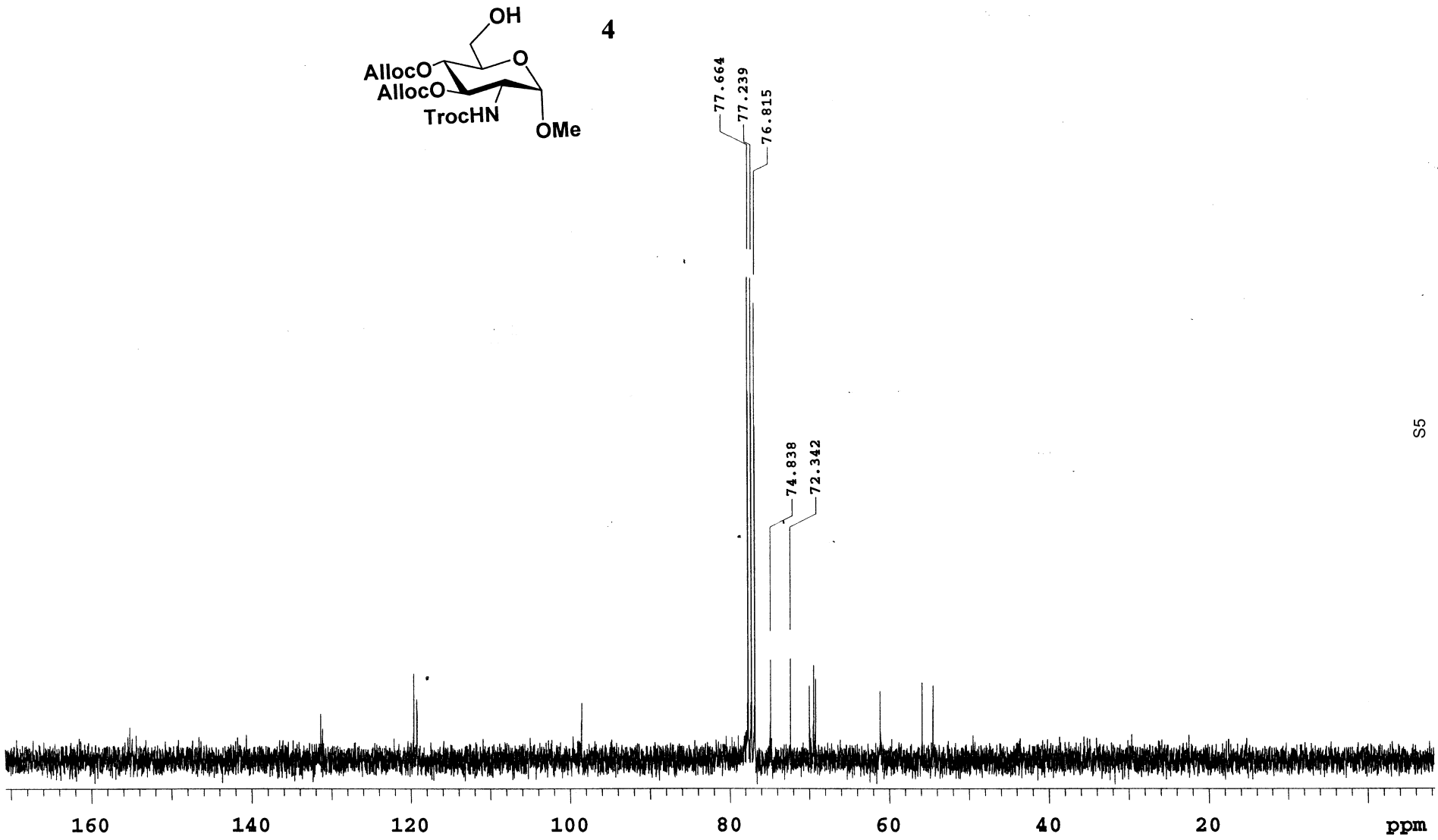
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AllocO
AllocO
TrocHN
O
OMe
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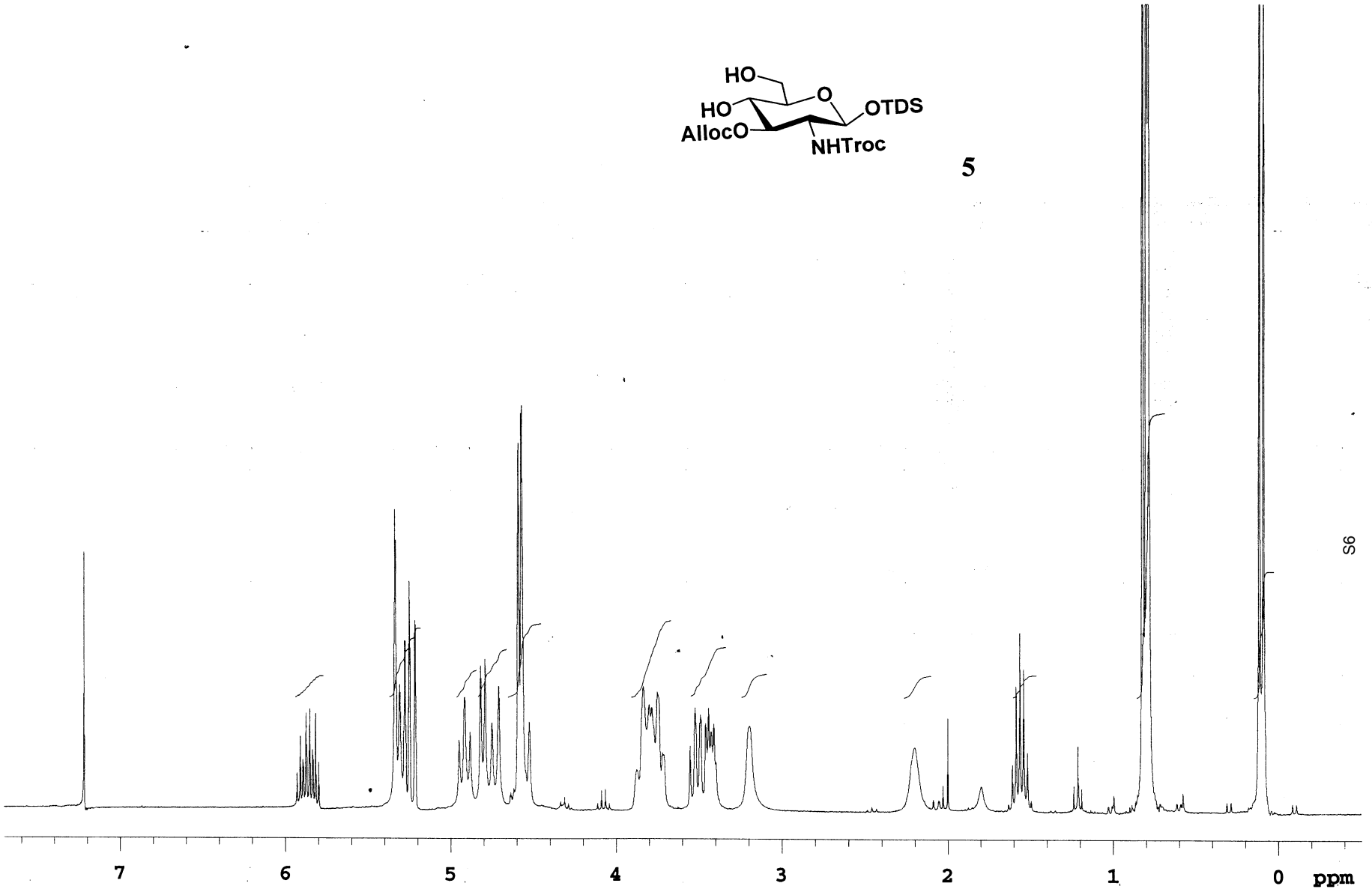
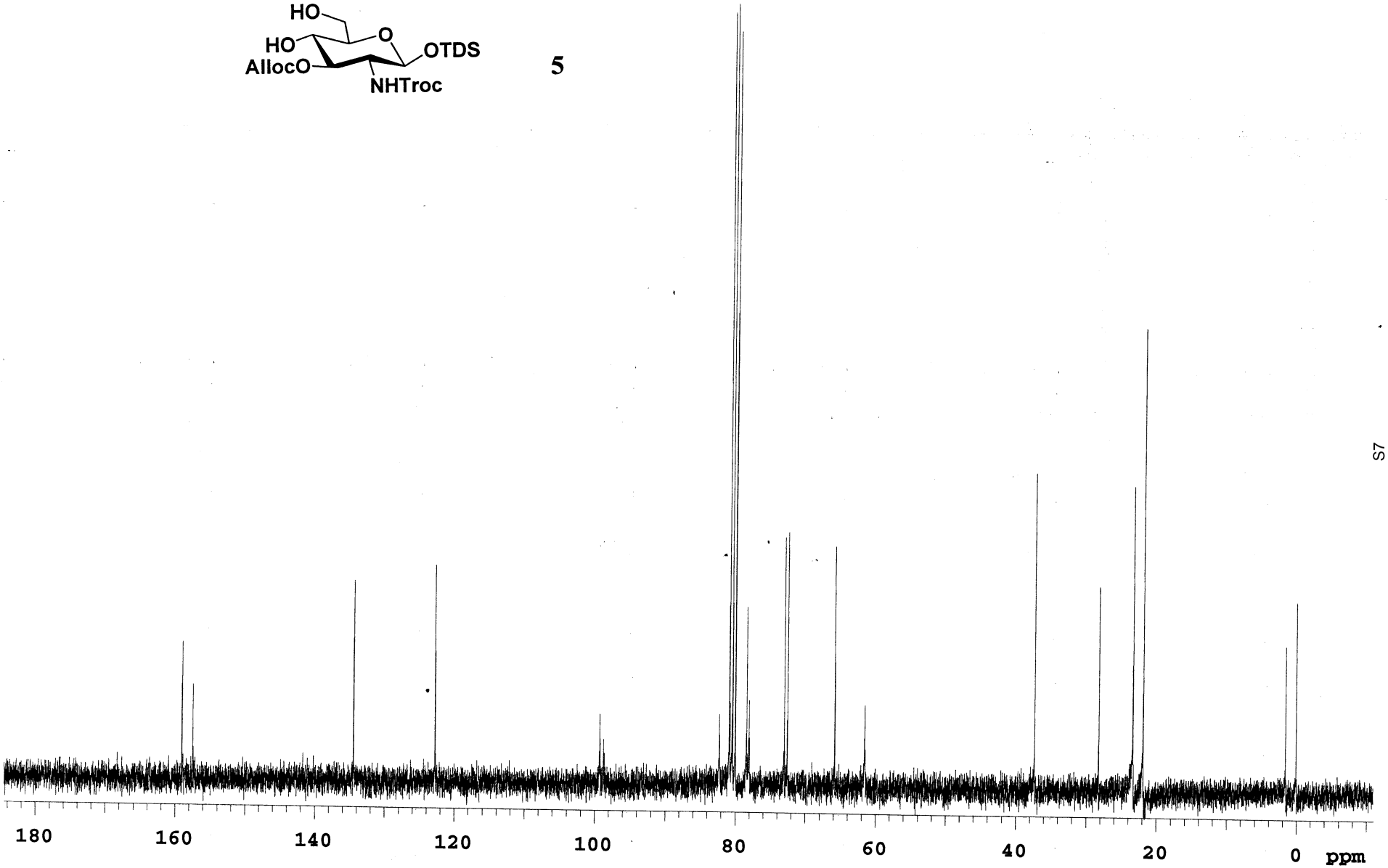
120
100
80
60
40
20
ppm


HO
O
HO
OTDS
AllocO
NHTroc
5
7
6
5
4
3
2
1
0
ppm

5



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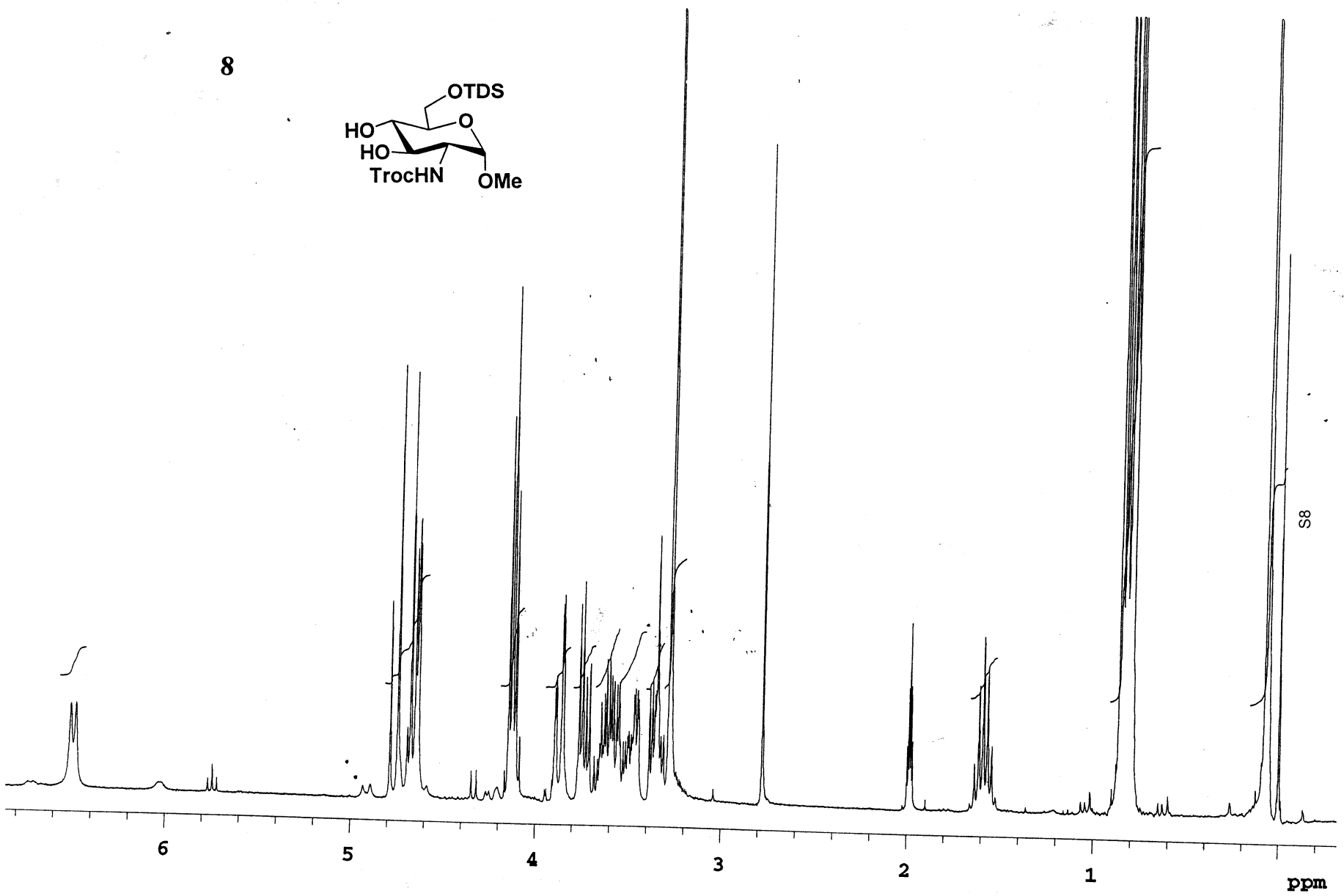
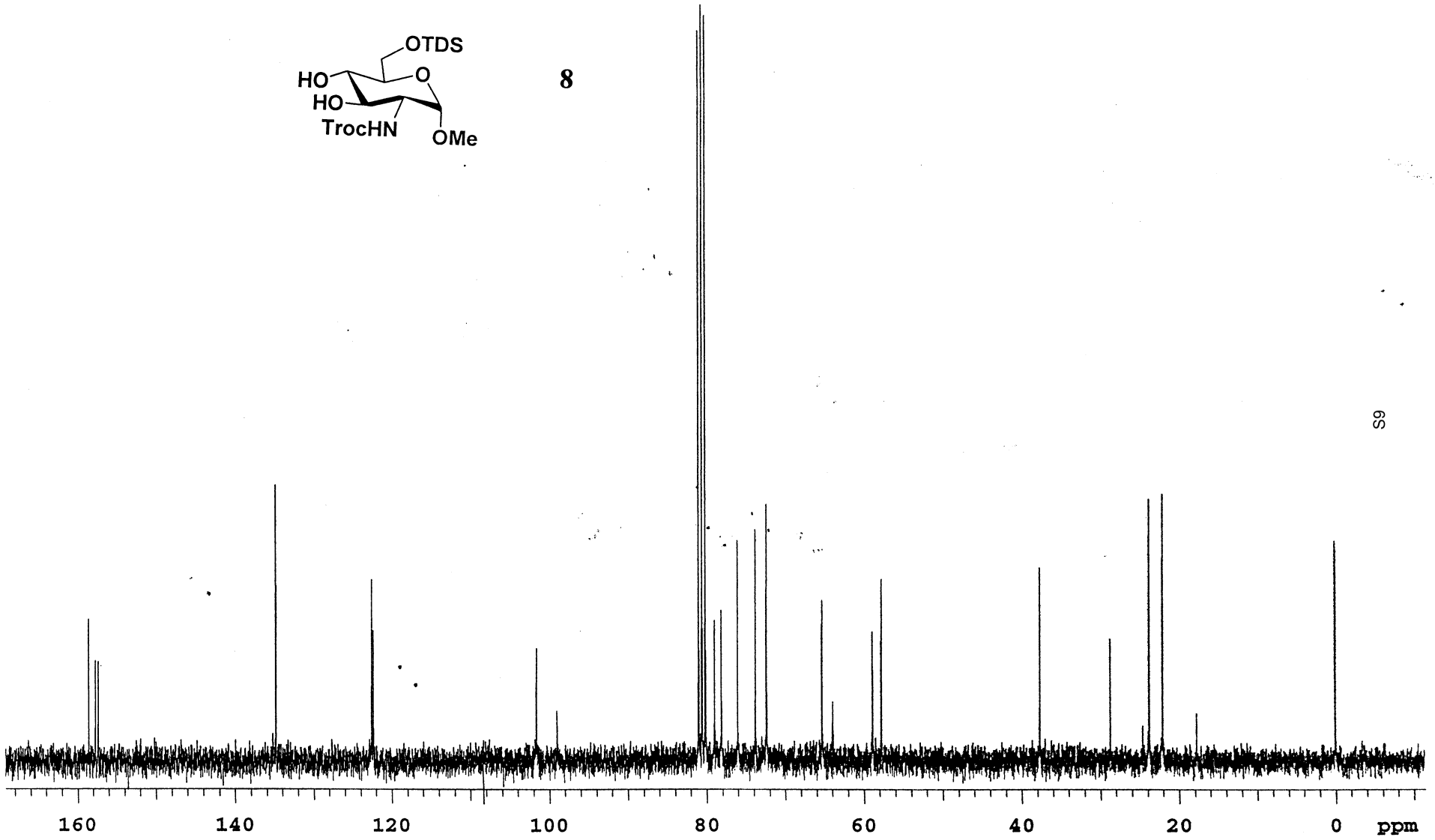
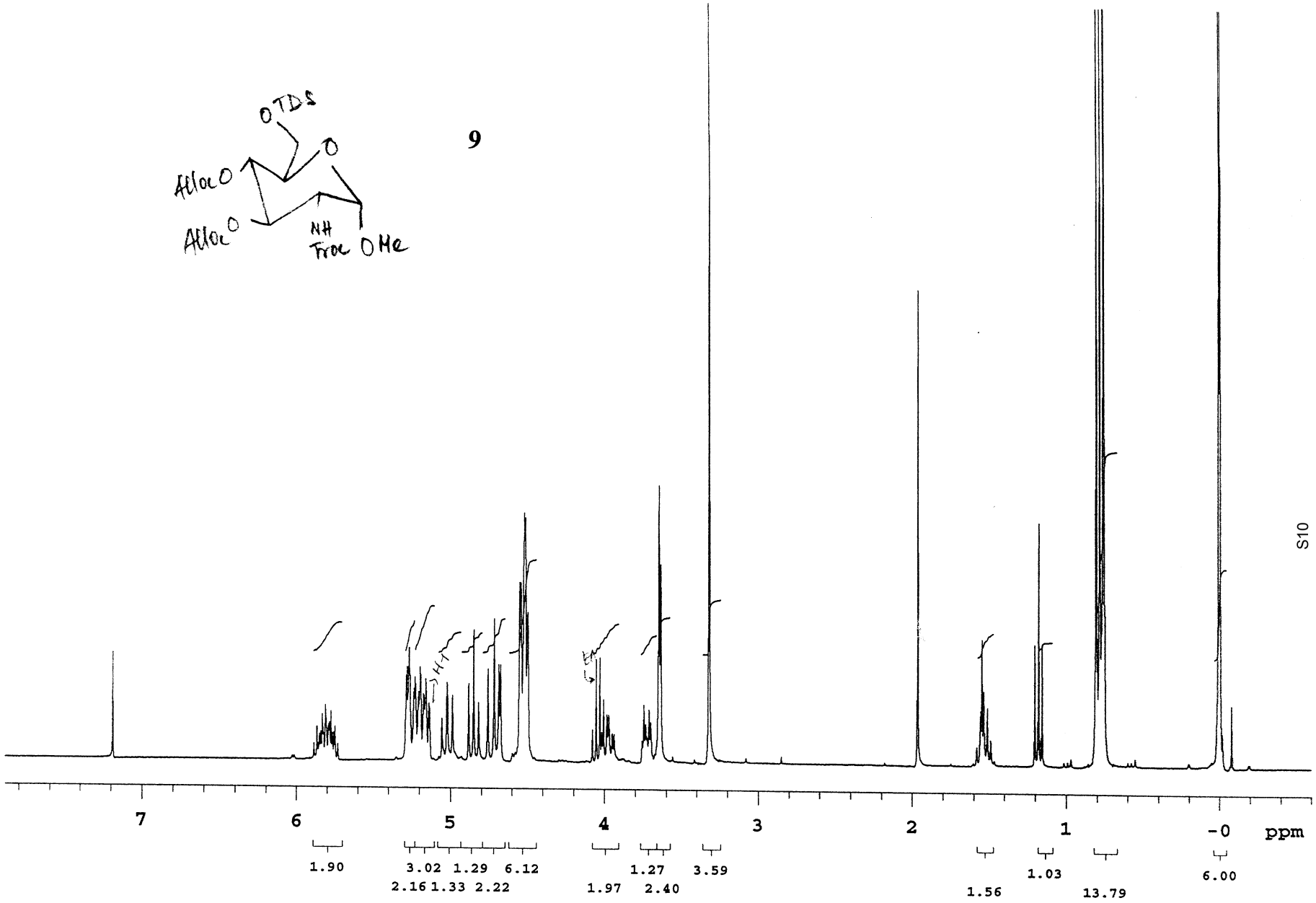



OTDS
HO
O
HO
TrocHN
OMe
8
160
140
120
100
80
60
40
20
0
ppm

OTDS
O
AllocO
AllocO
NH
Troc
OMe

9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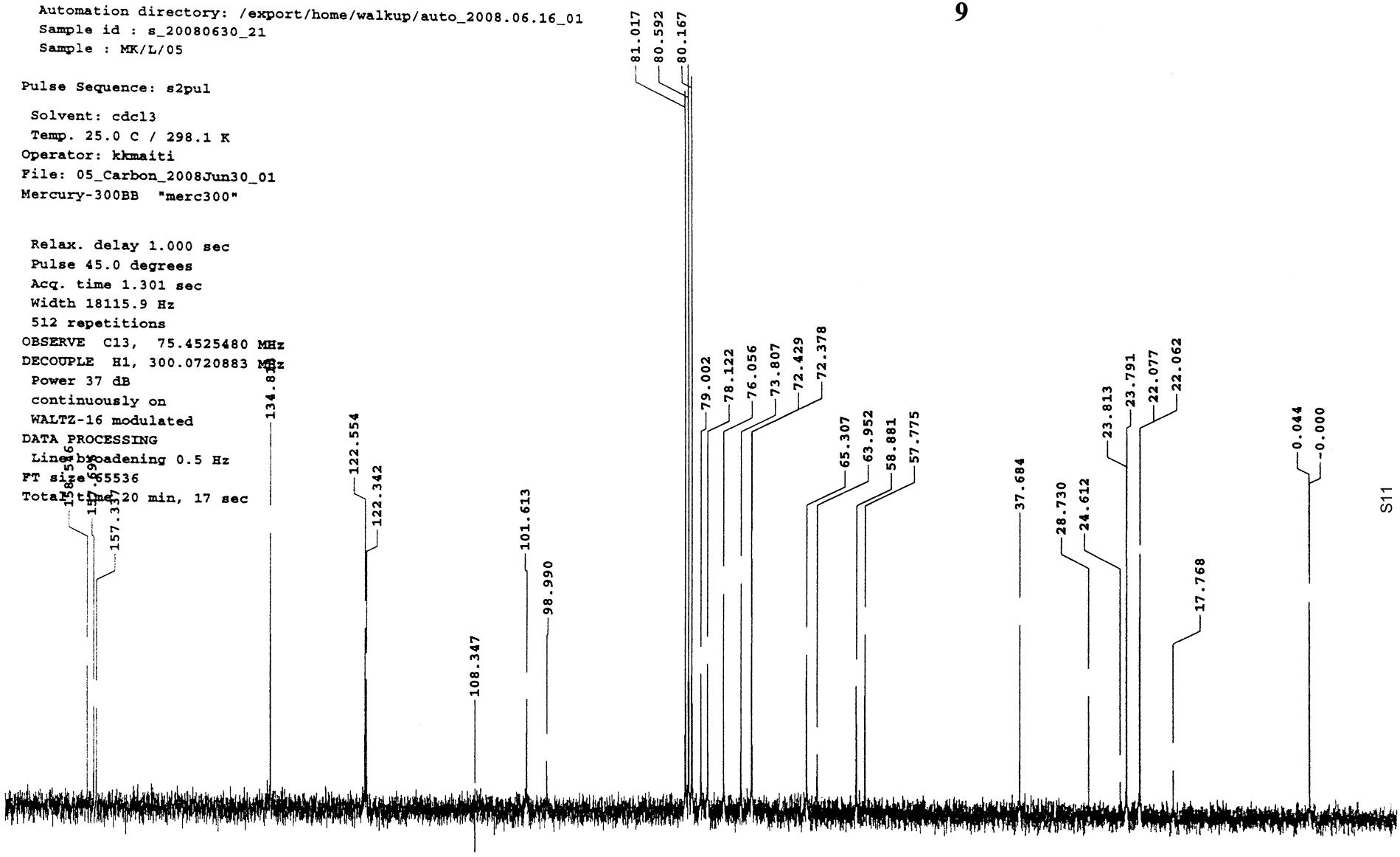
6
5
4
3
2
1
-0
ppm
1.90
3.02
1.29
6.12
2.16
1.33
2.22
1.97
1.27
2.40
3.59
1.56
1.03
13.79
6.00

Automation directory: /export/home/walkup/auto_2008.06.16_01
Sample id : s_20080630_21
Sample : MK/L/05

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: kkmaiti
File: 05_Carbon_2008Jun30_01
Mercury-300BB "merc300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.301 sec
Width 18115.9 Hz
512 repetitions
OBSERVE C13, 75.4525480 MHz
DECOUPLE H1, 300.0720883 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 20 min, 17 sec



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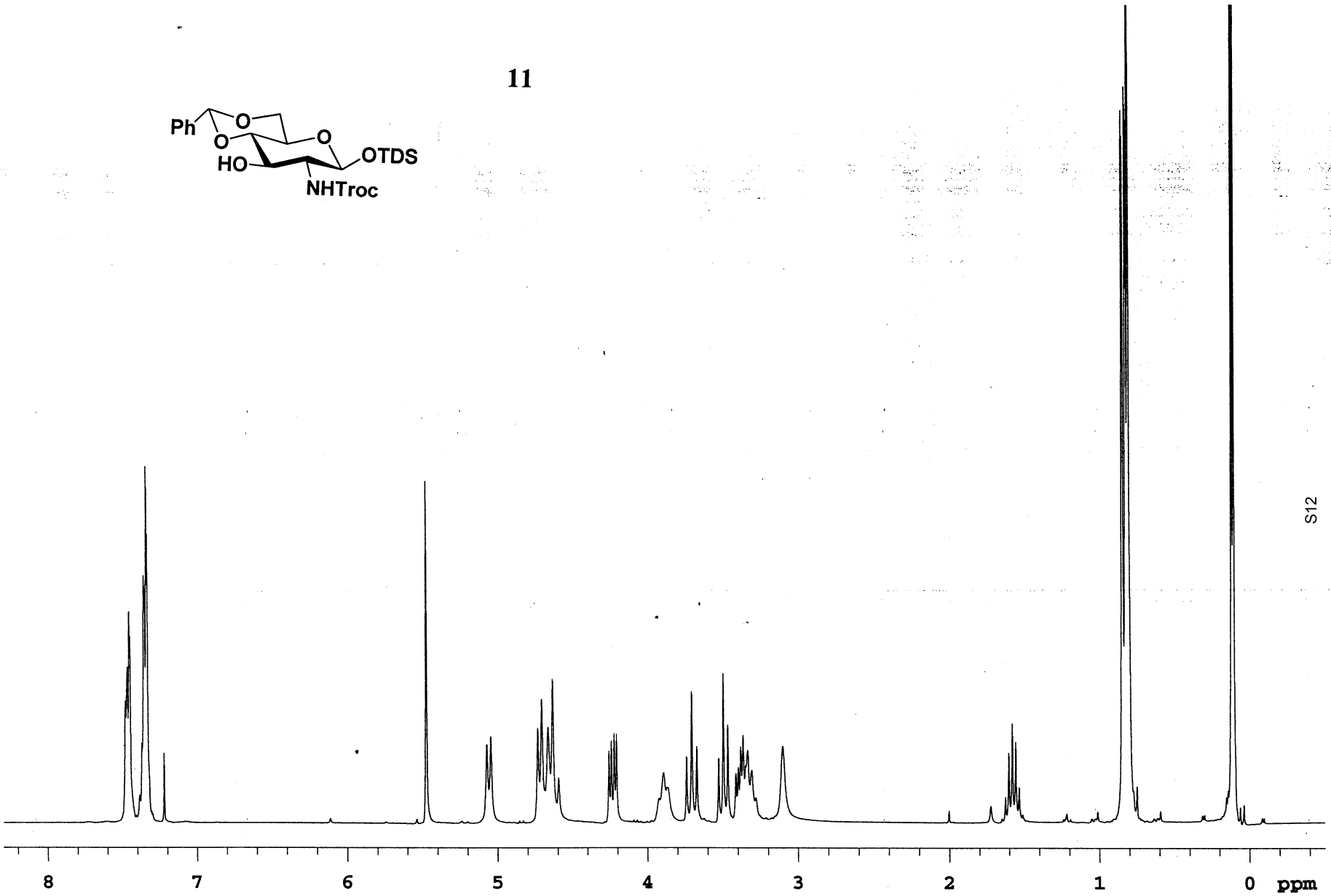

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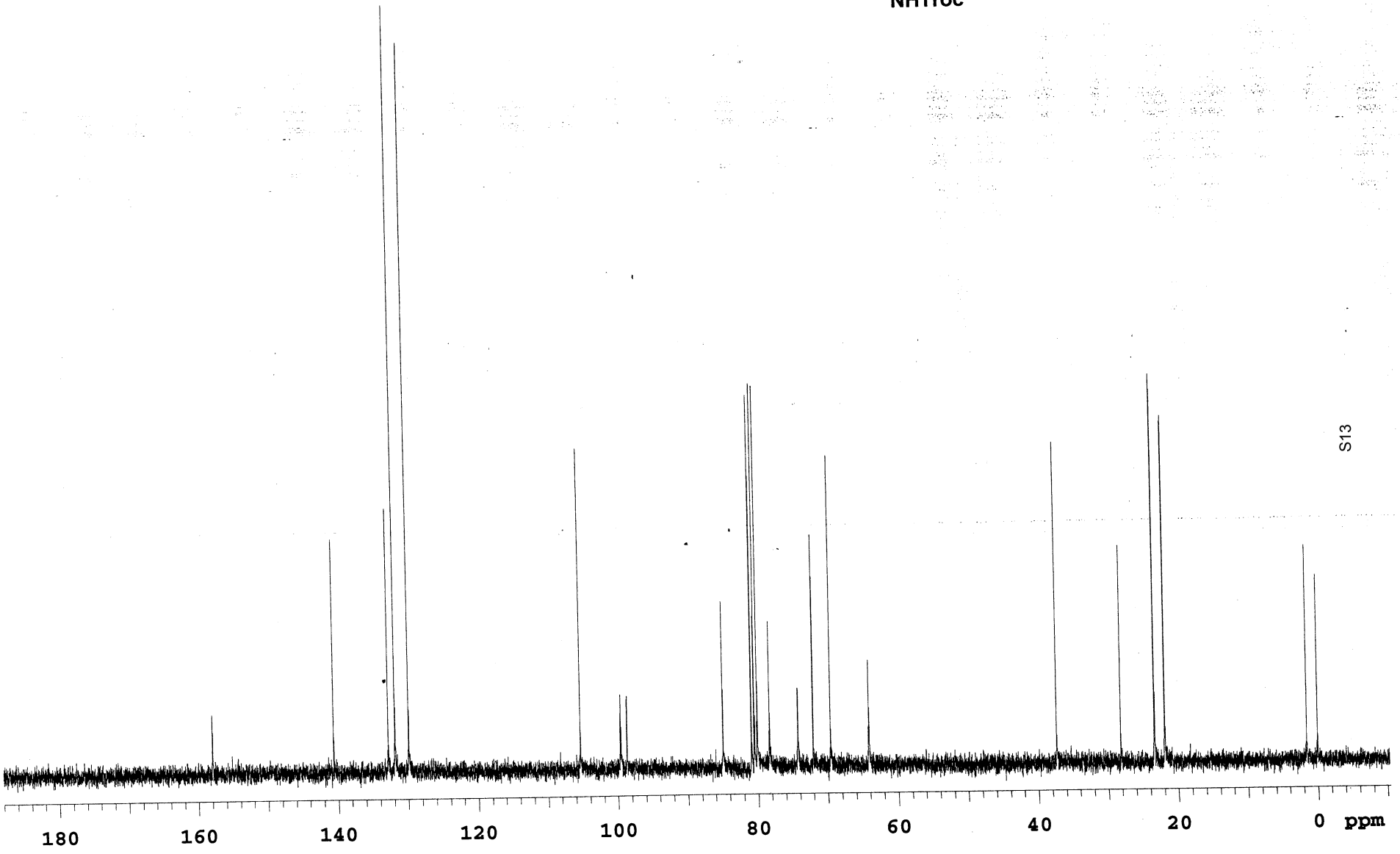

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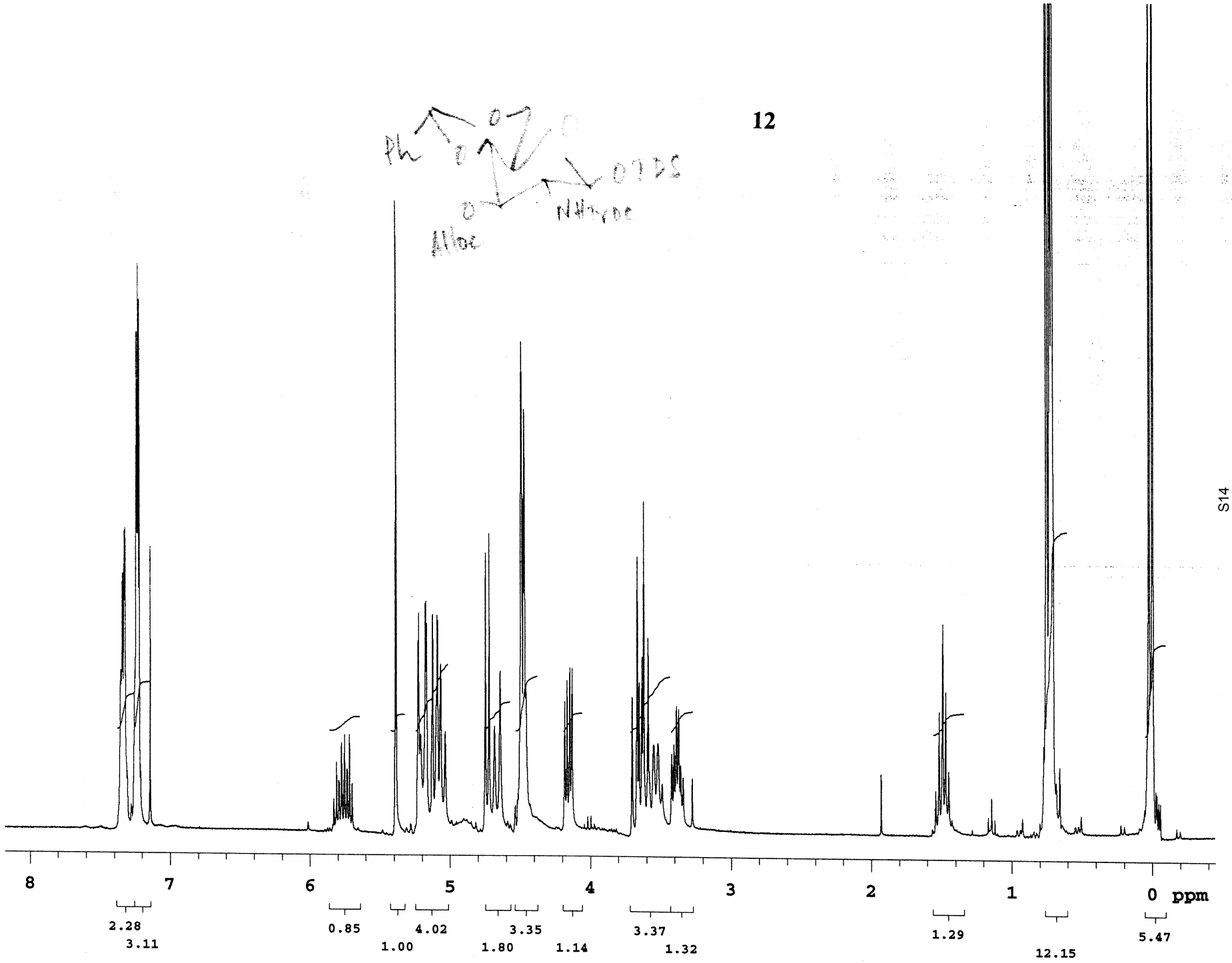
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7
6
5
4
3
2
1
0 ppm
2.28
3.11
0.85
1.00
4.02
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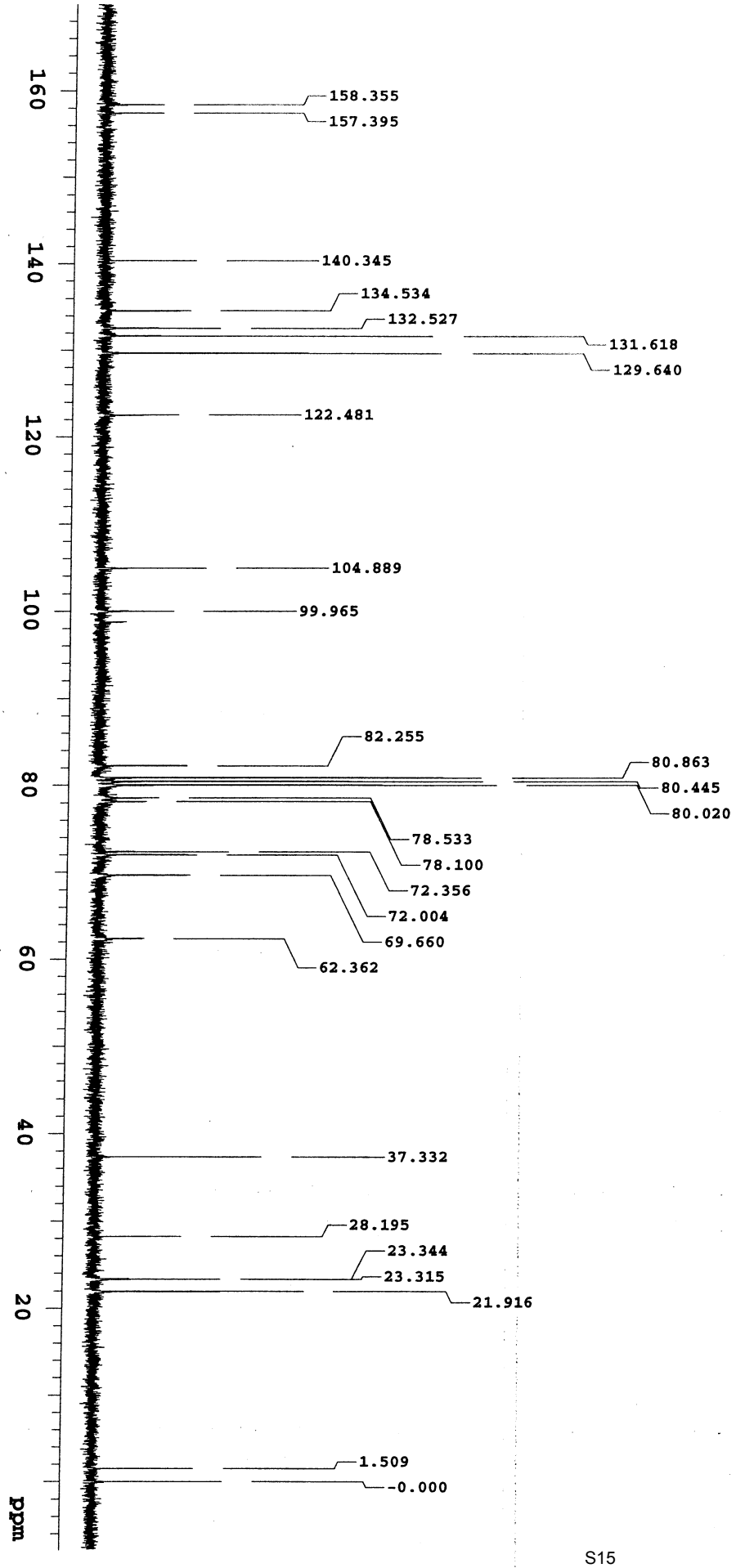
3.35
1.14
3.37
1.32
1.29
12.15
5.47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140
120
100
80
60
40
20
ppm
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140.345
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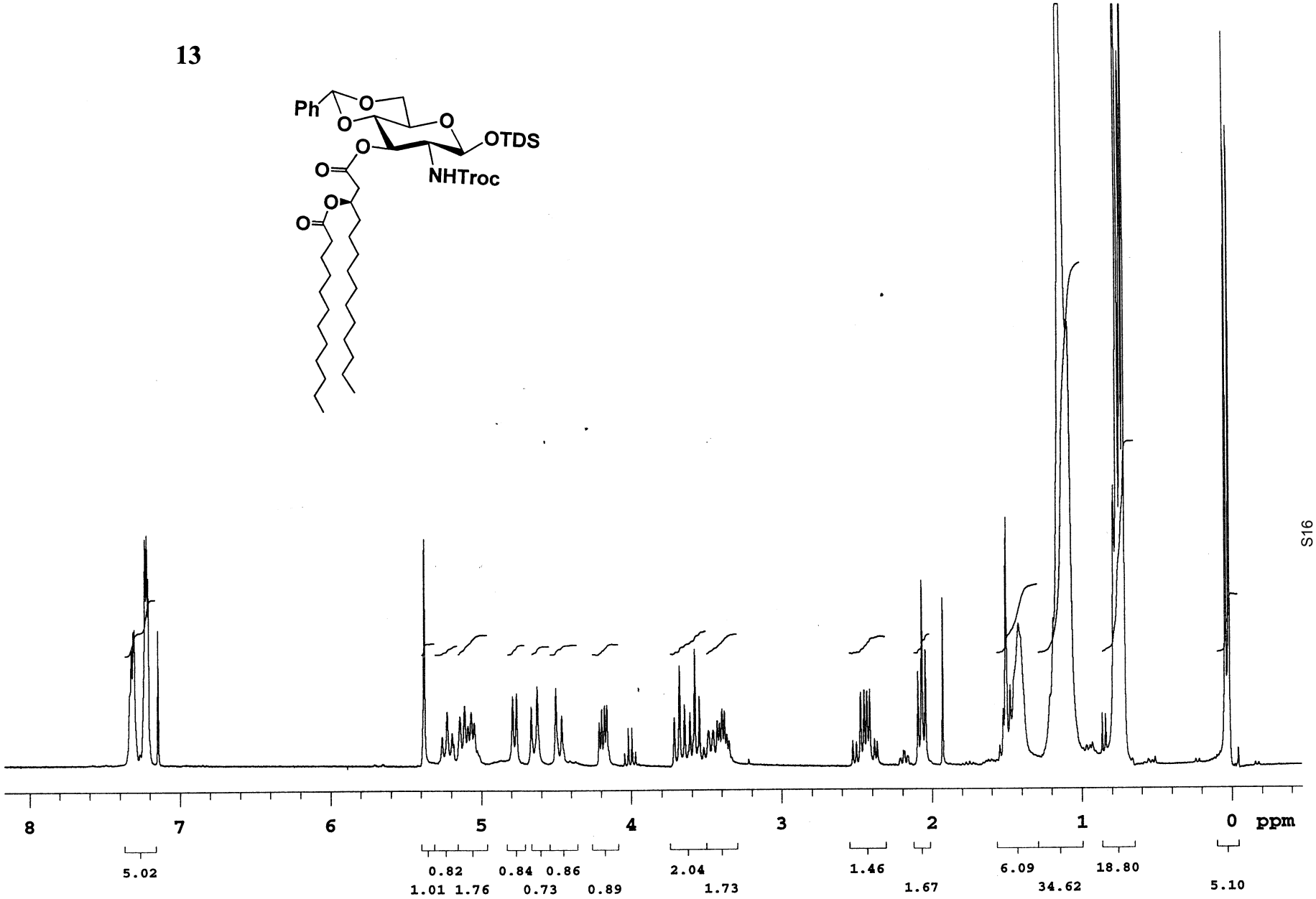
132.527
131.618
129.640
122.481
104.889
99.965
82.255
80.863
80.445
80.020
78.533
78.100
72.356
72.004
69.660
62.362
37.332
28.195
23.344
23.315
21.916
1.509
-0.000

12

13

Ph
O
O
O
O
OTDS
NHTroc
O
O
O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7
6
5
4
3
2
1
0
ppm
5.02
0.82
1.01
1.76
0.84
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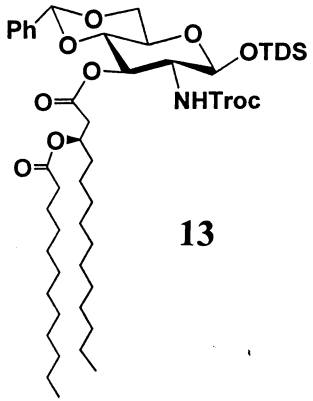
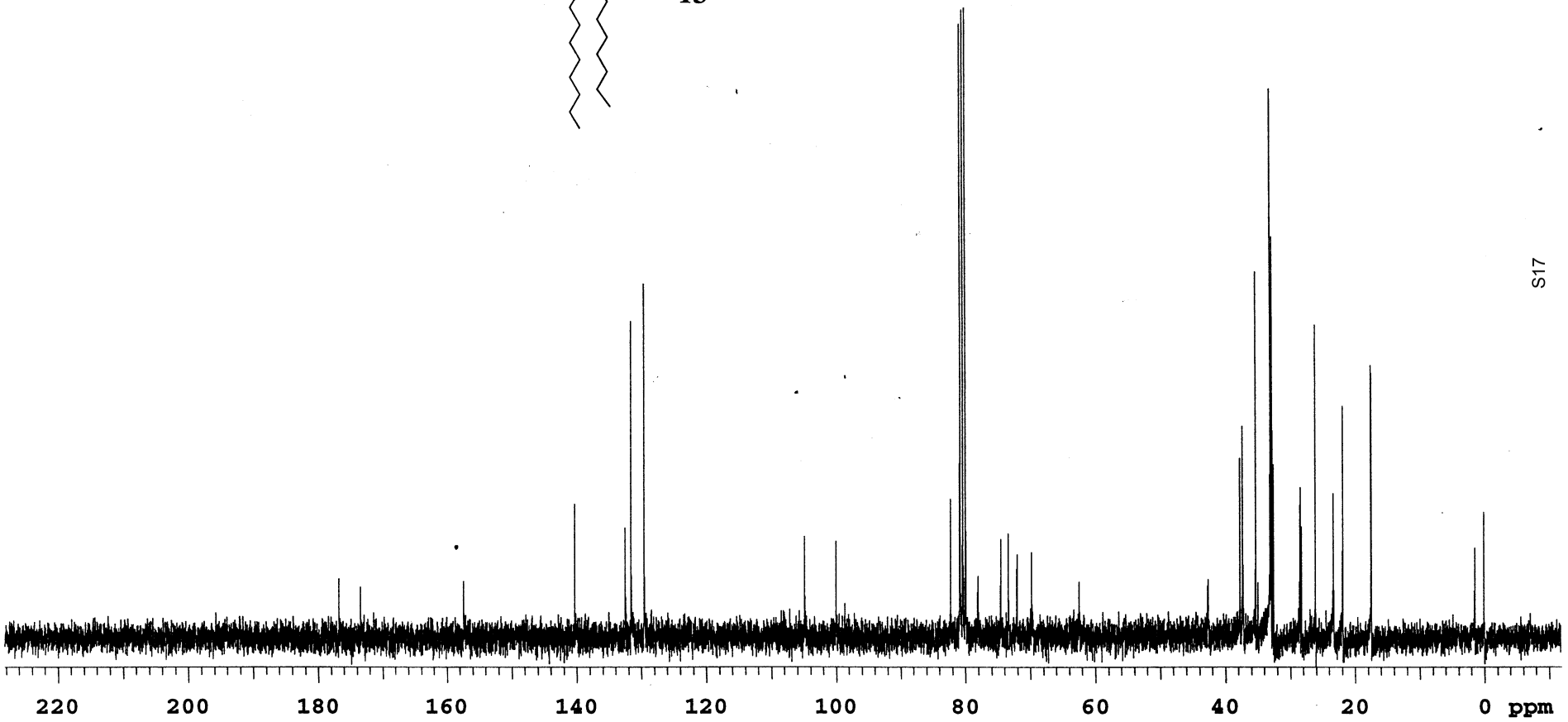
0.73
0.89
2.04
1.73
1.46
1.67
6.09
34.62
18.80
5.10


Ph
O
O
O
O
OTDS
O
NHTroc
O
O
O

13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180
160
140
120
100
80
60
40
20
0 ppm

Std proton

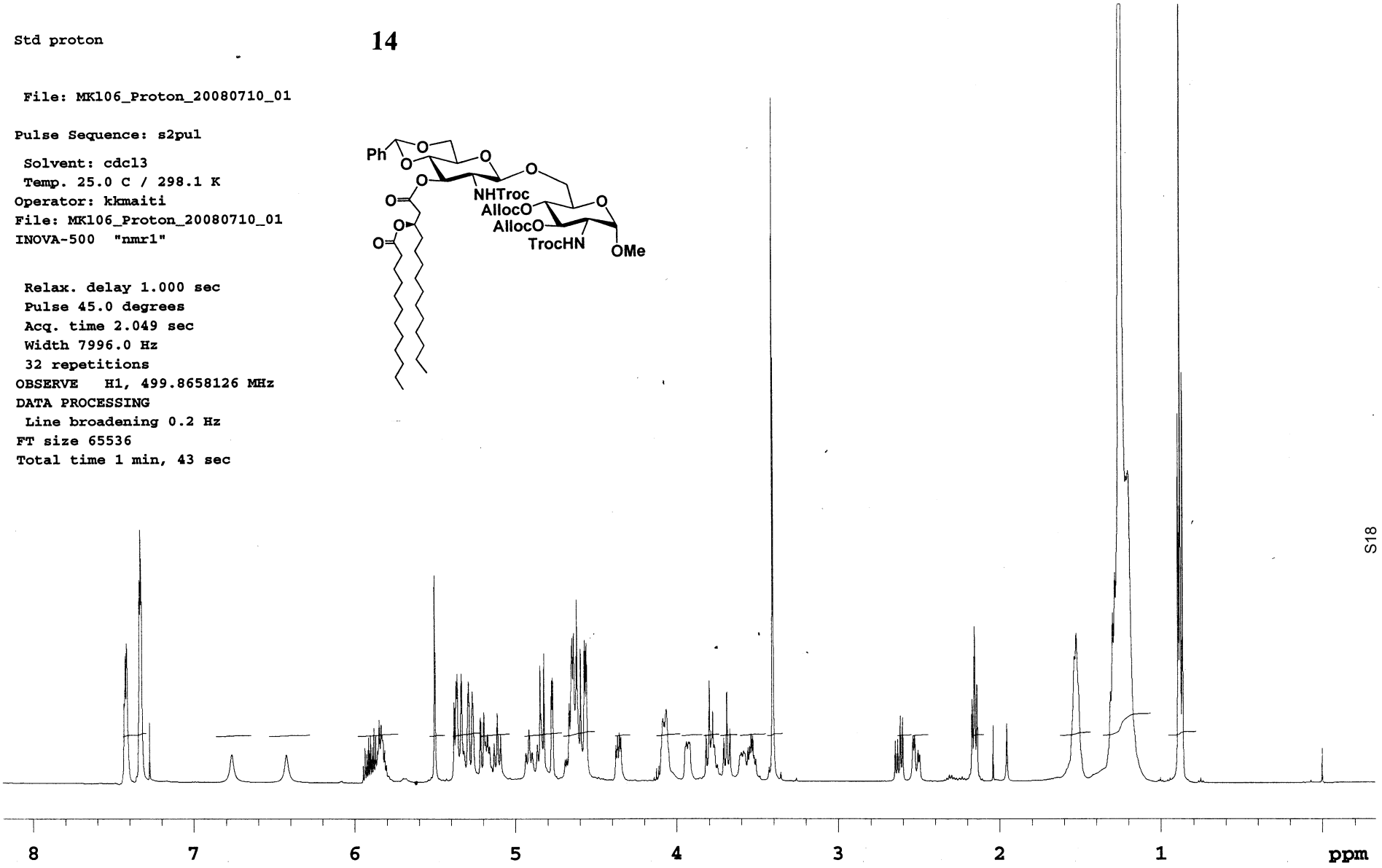
14

File: MK106_Proton_20080710_01

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: kkmaiti
File: MK106_Proton_20080710_01
INOVA-500 "nmr1"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 7996.0 Hz
32 repetitions
OBSERVE H1, 499.8658126 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 1 min, 43 sec



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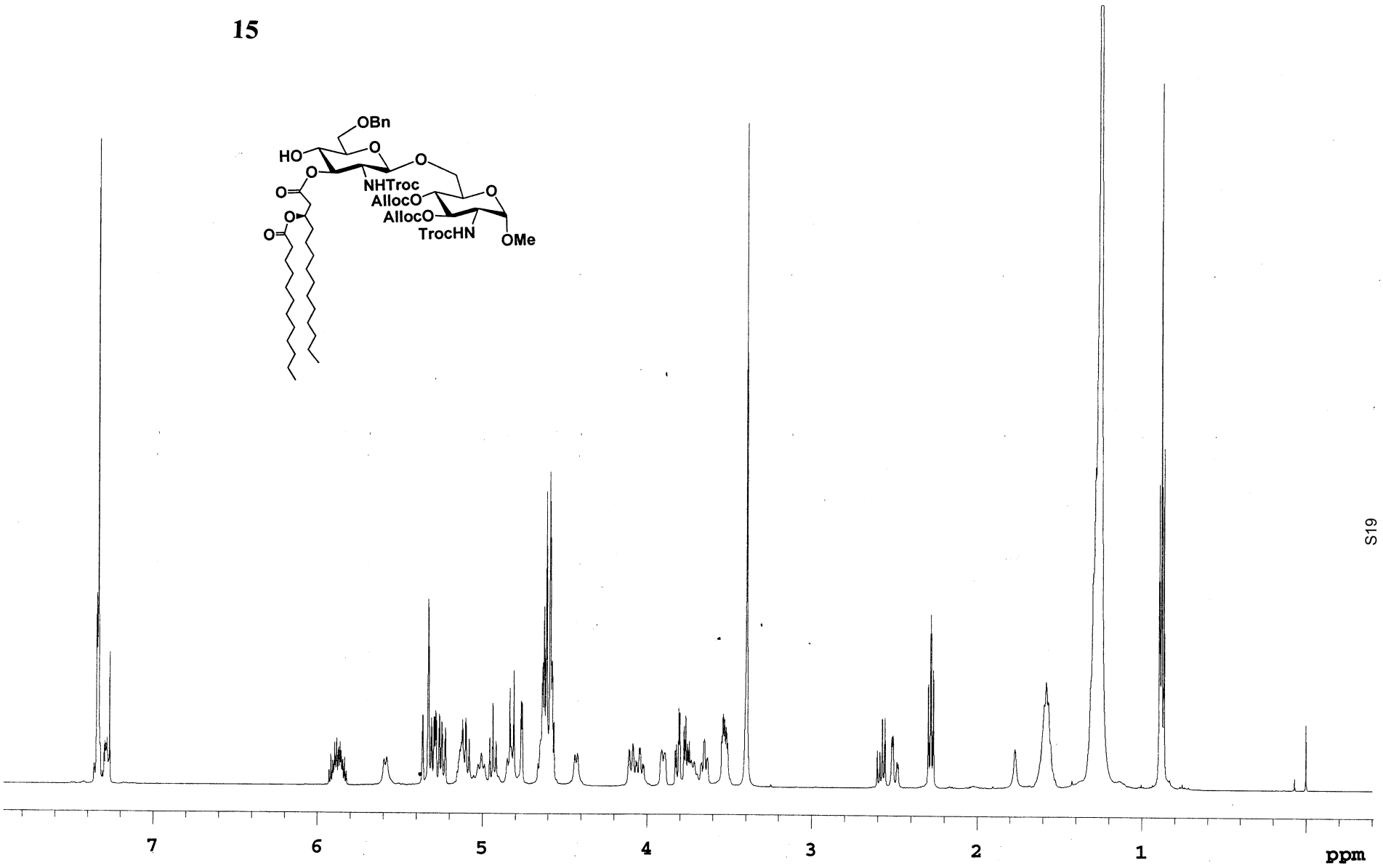

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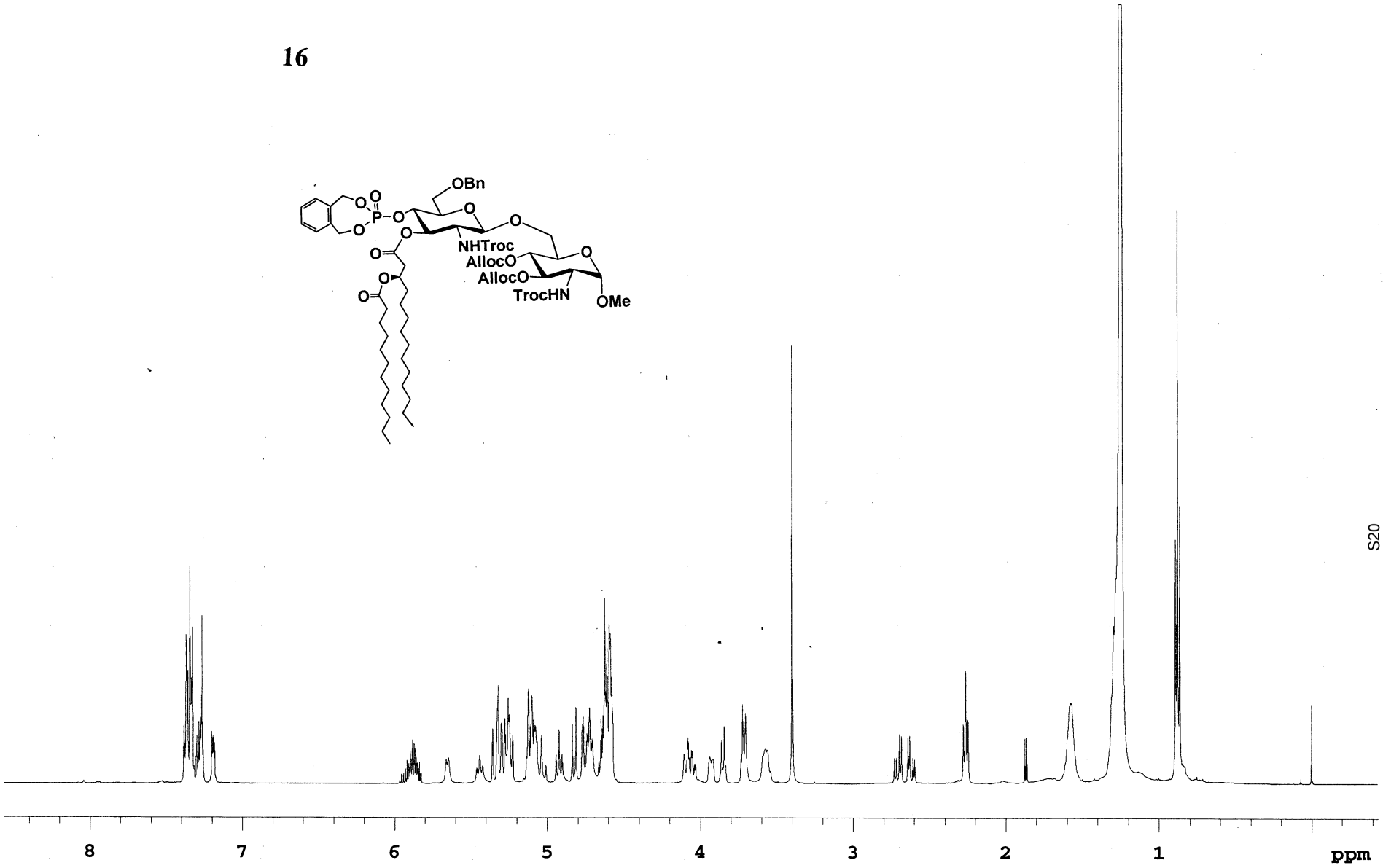

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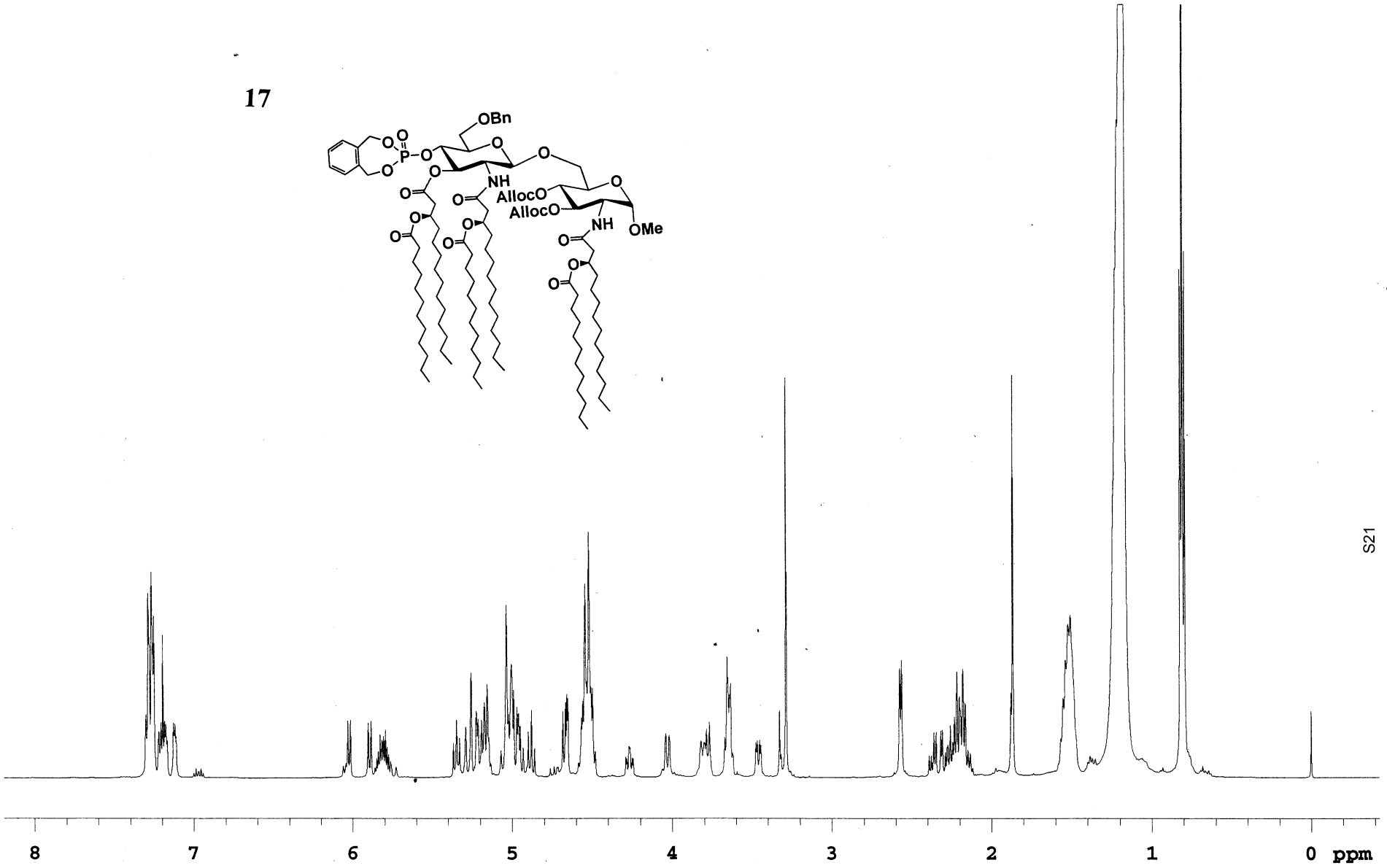

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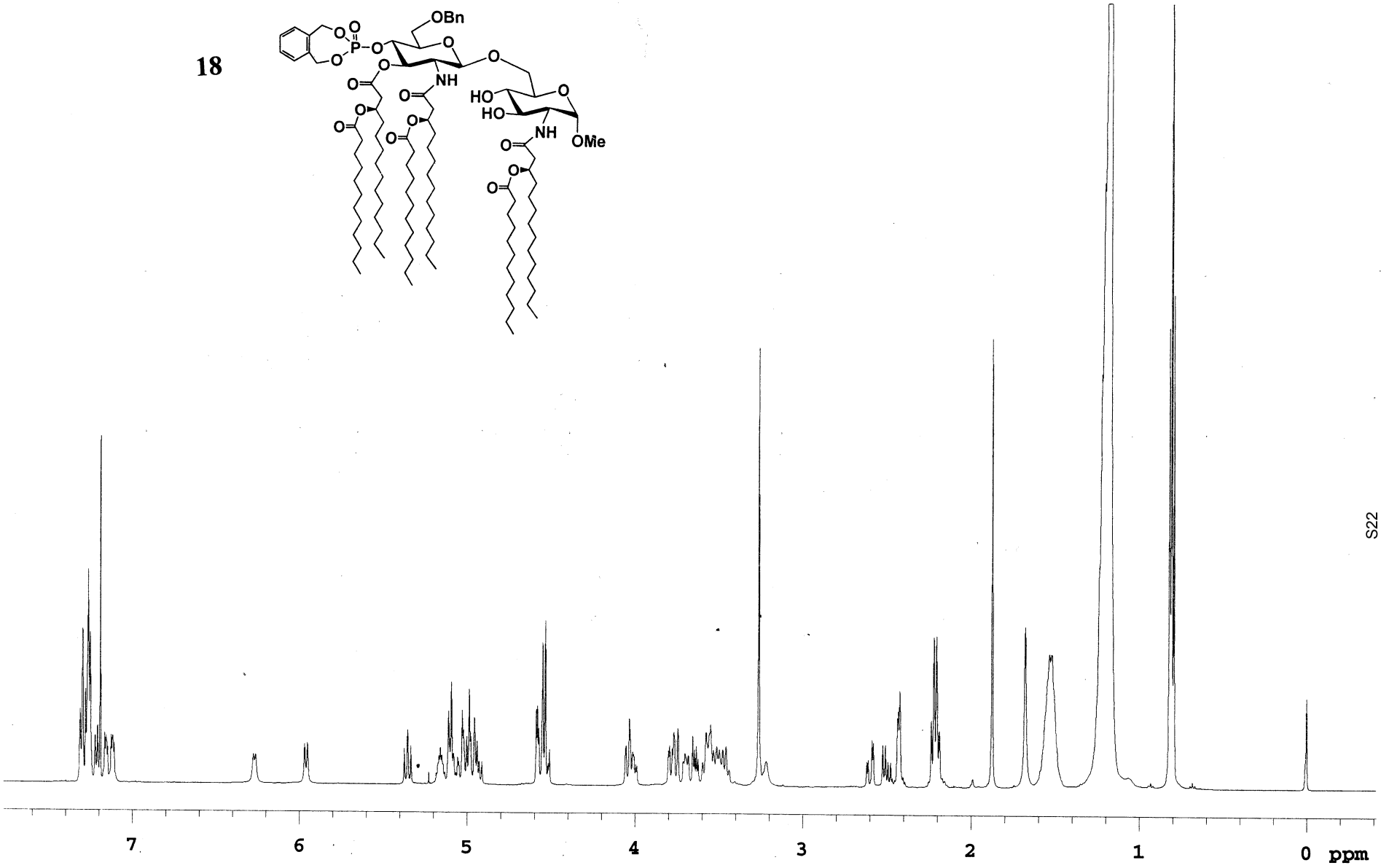

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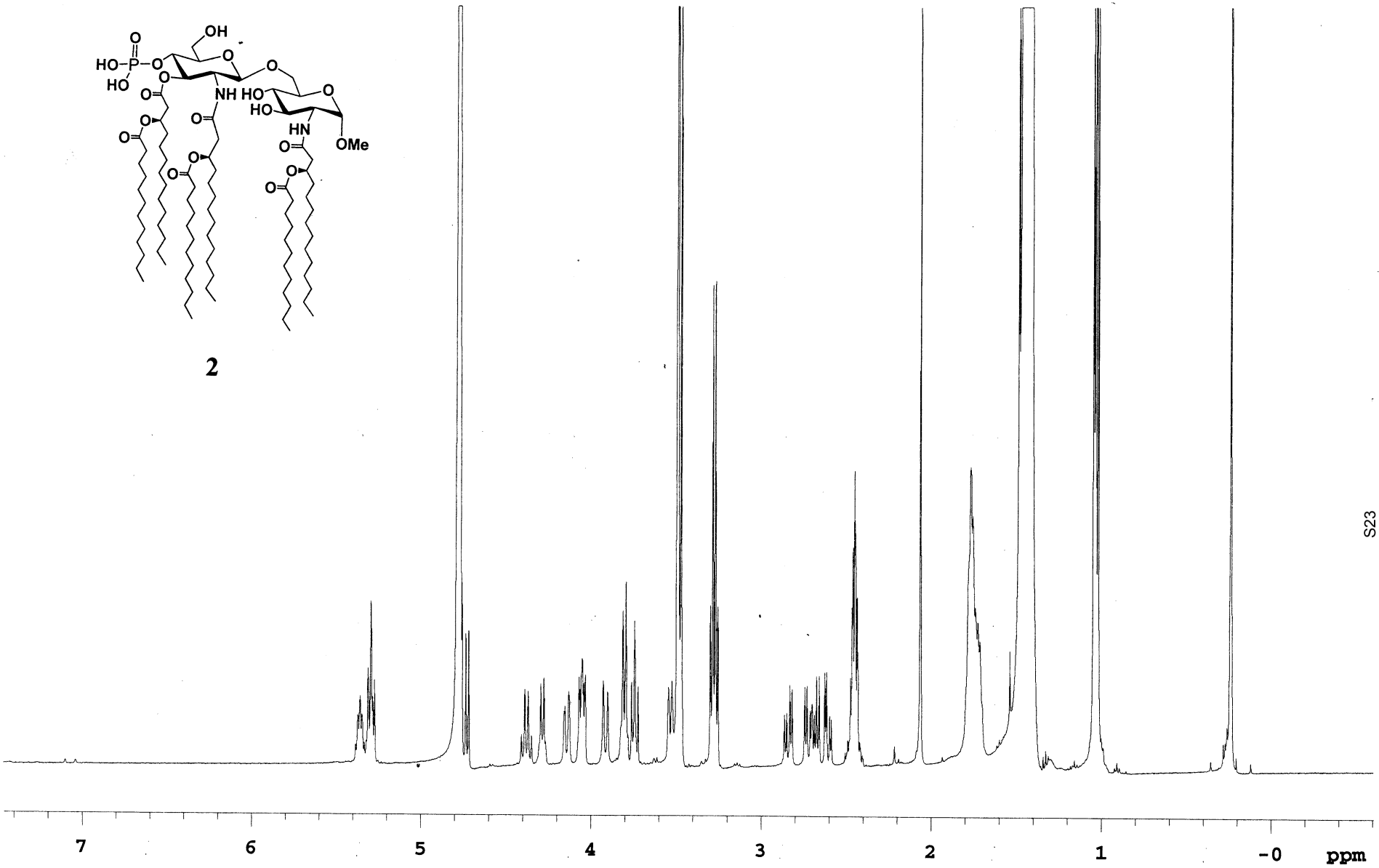
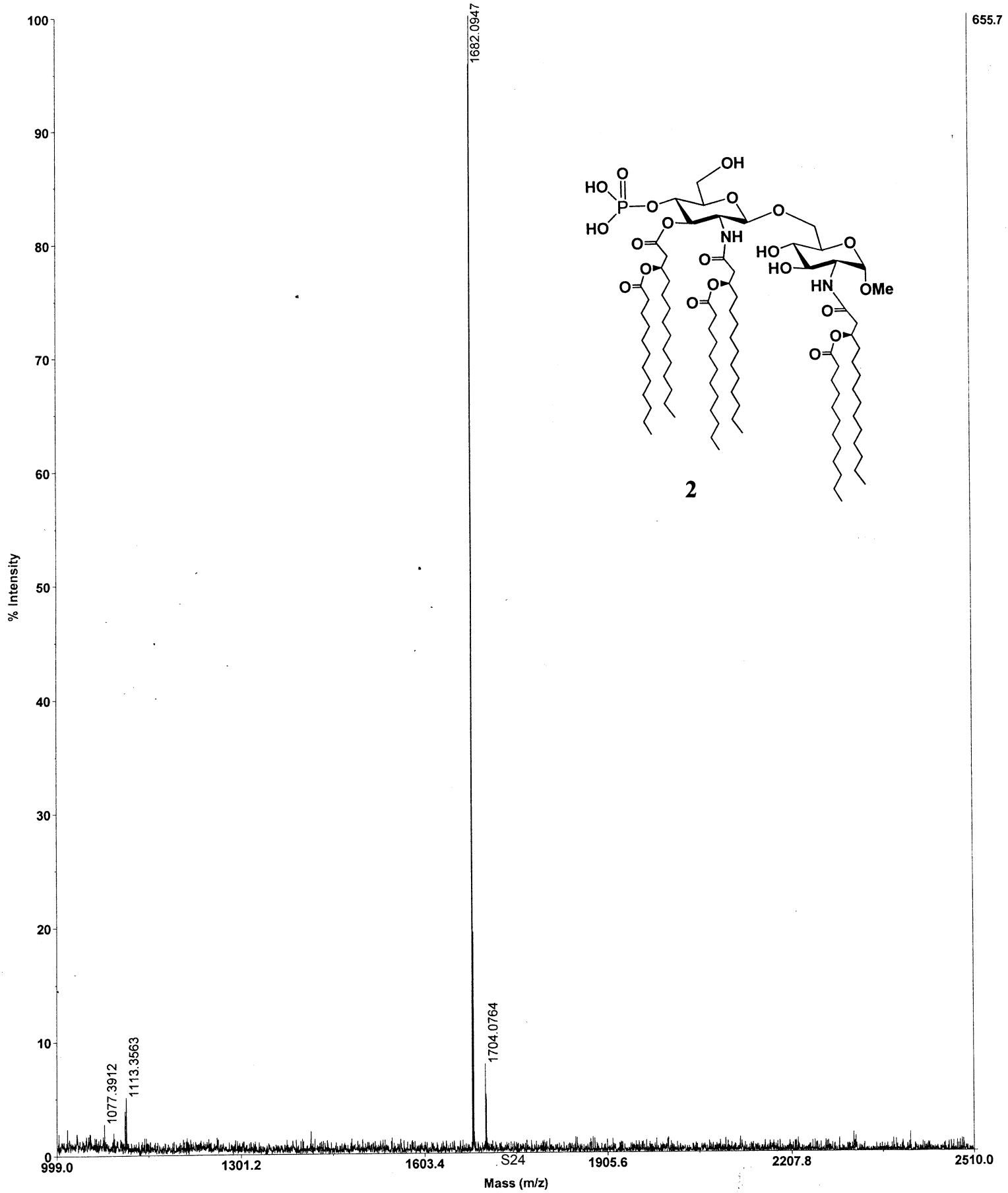


4700 Reflector Spec #1[BP = 1683.1, 656]



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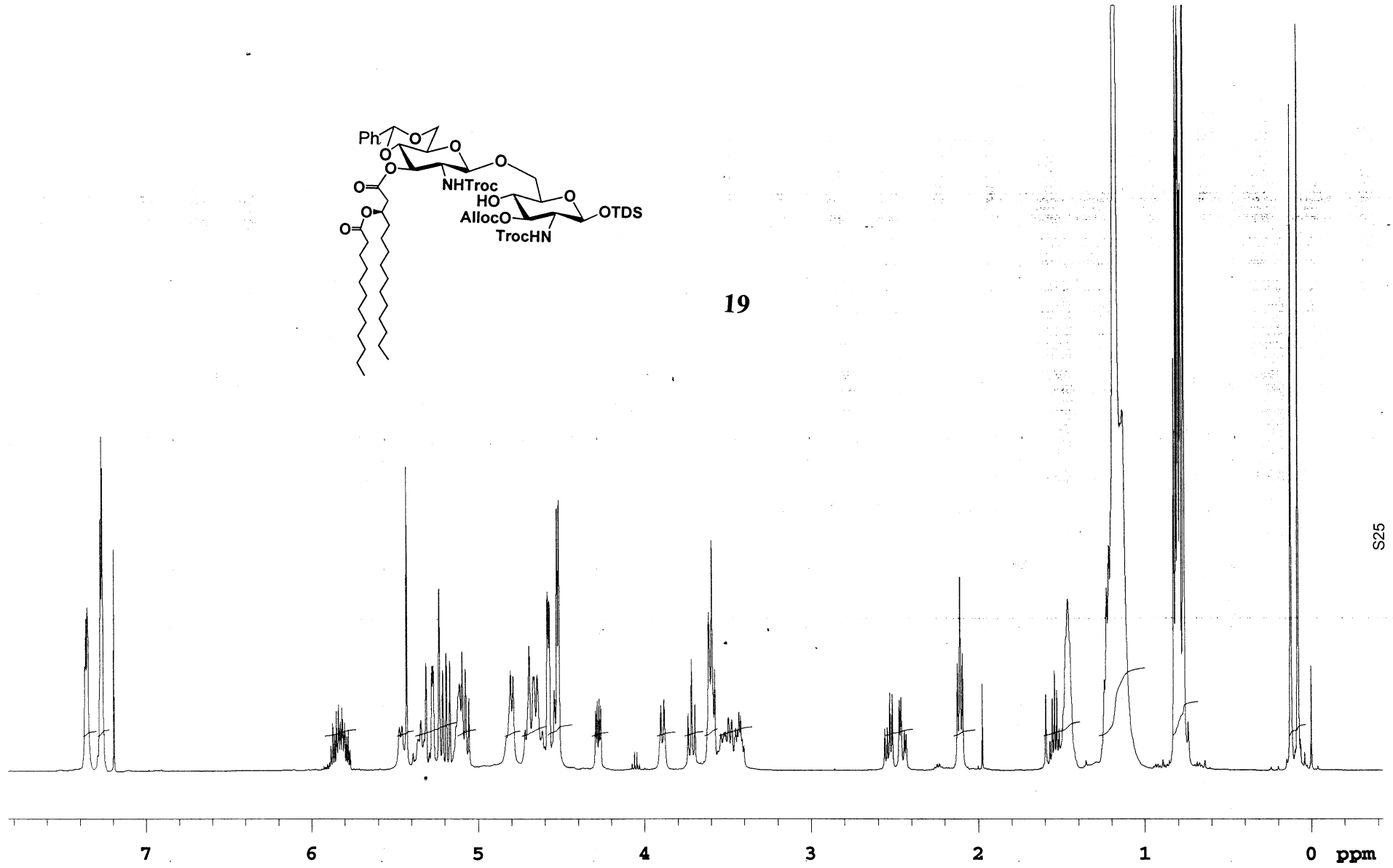
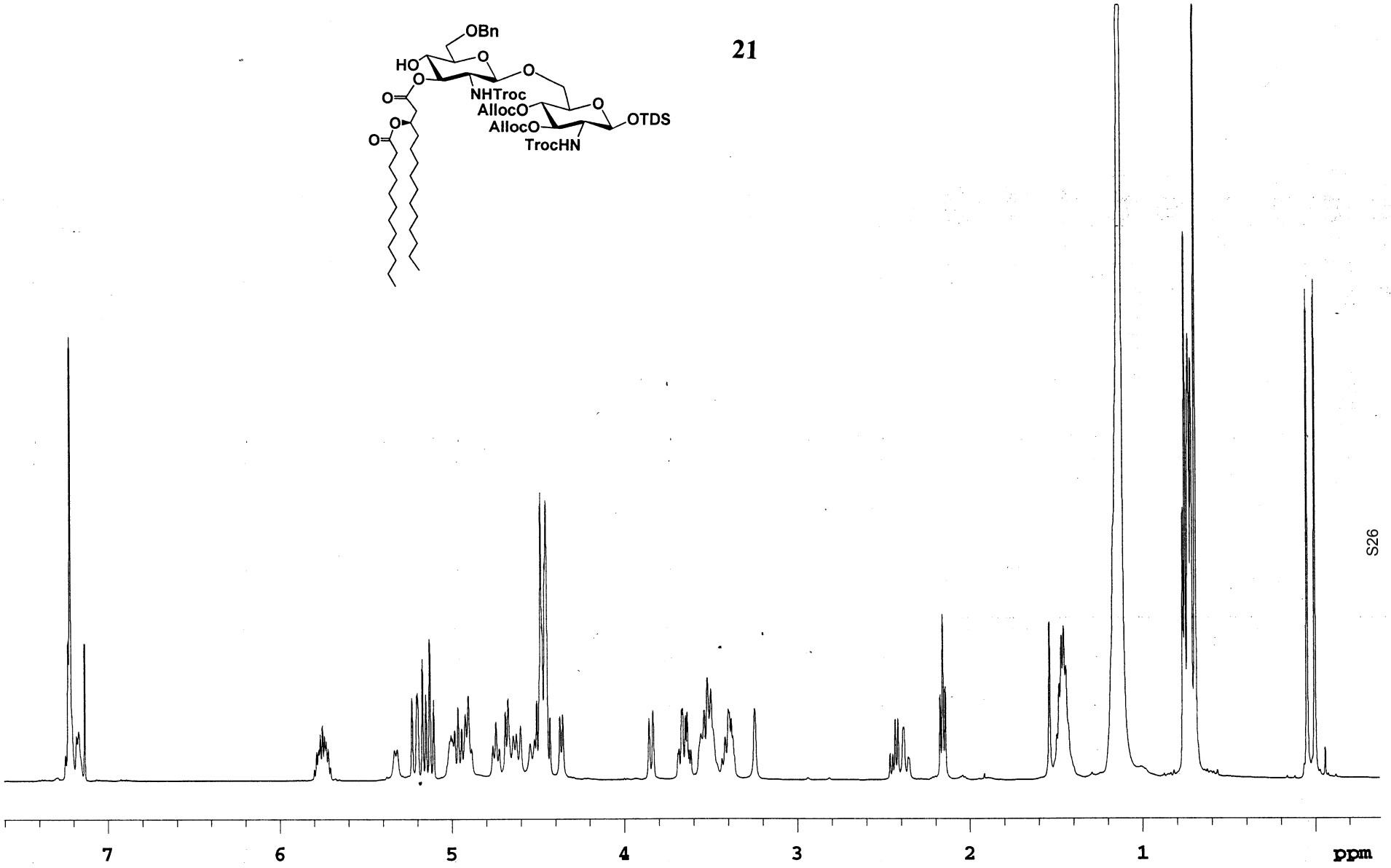

Ph
O
O
O
O
O
O
NHTroc
HO
O
OTDS
AllocO
TrocHN
O
O
O
19
S25
7
6
5
4
3
2
1
0
ppm

OBn
HO
O
O
NHTroc
AllocO
AllocO
TrocHN
OTDS
O
O
O

21


S26
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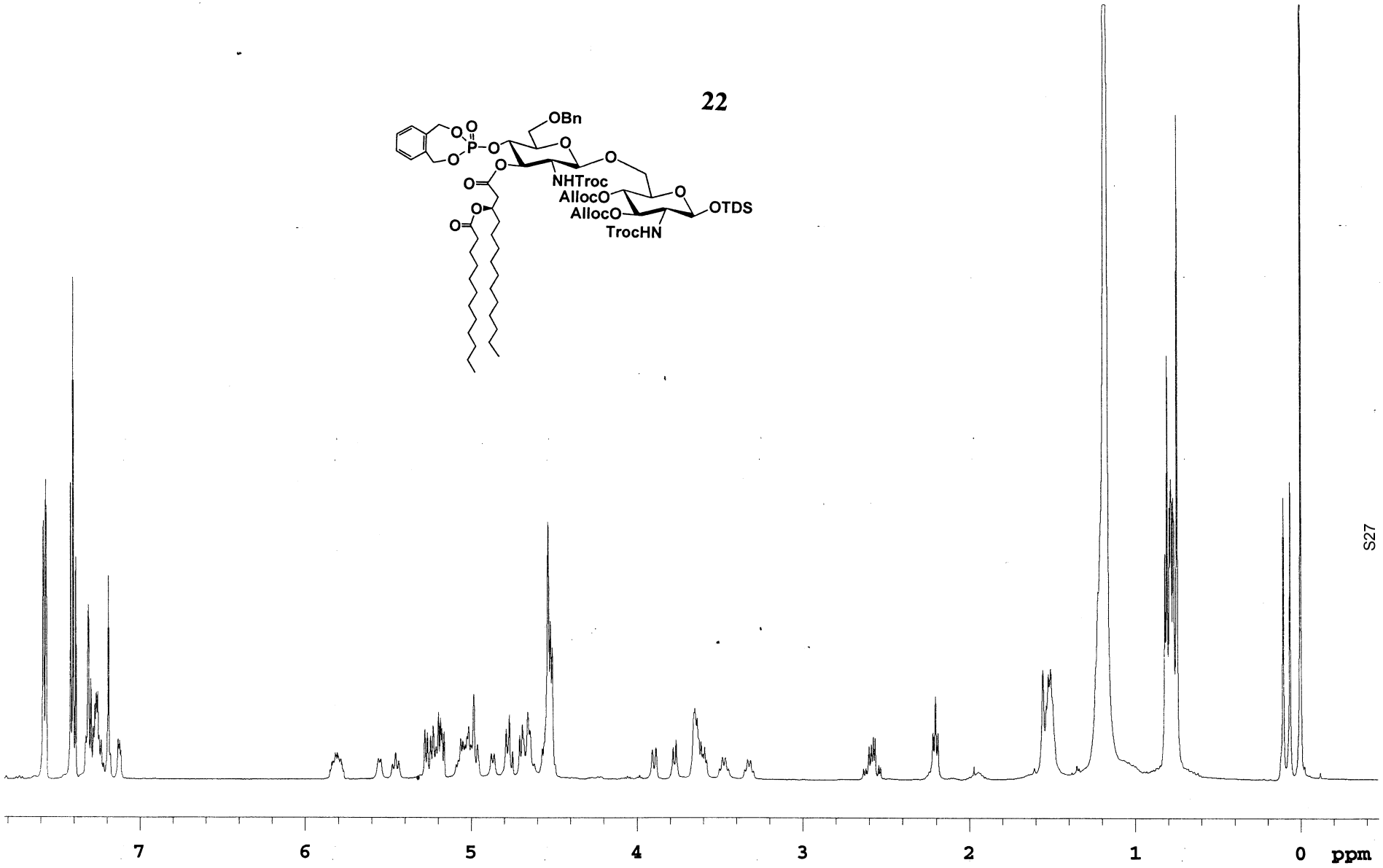
4
3
2
1
ppm

22



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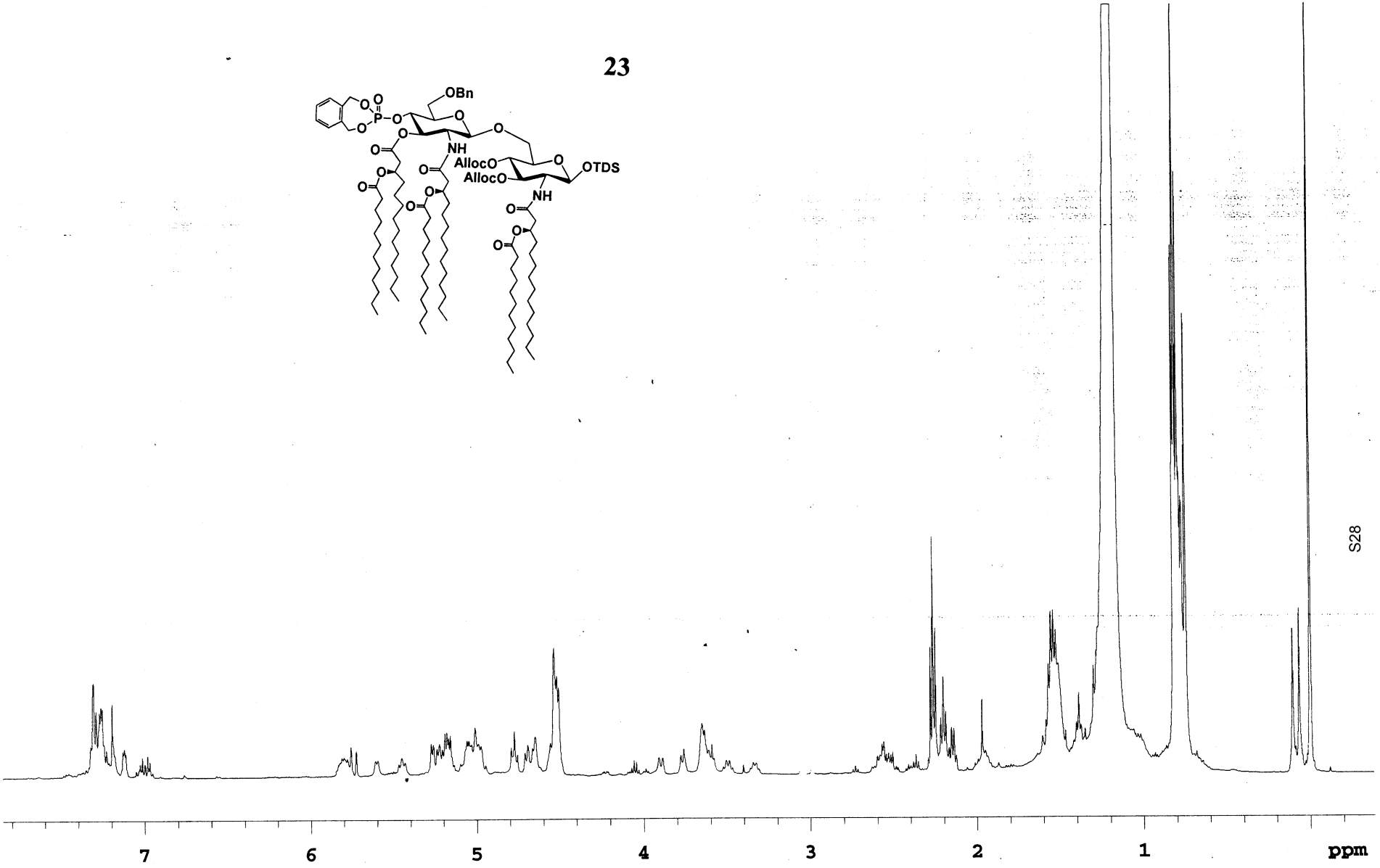

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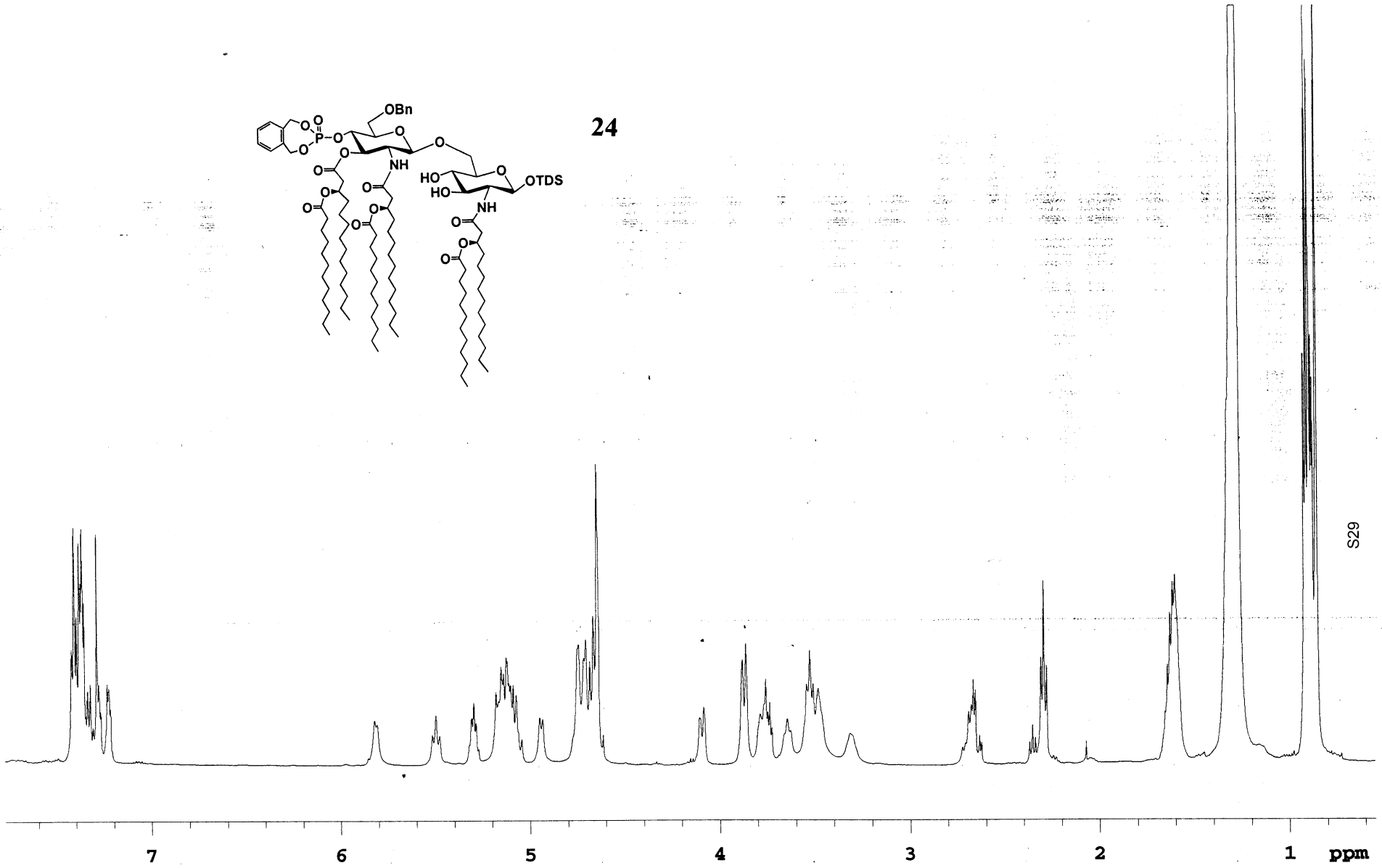

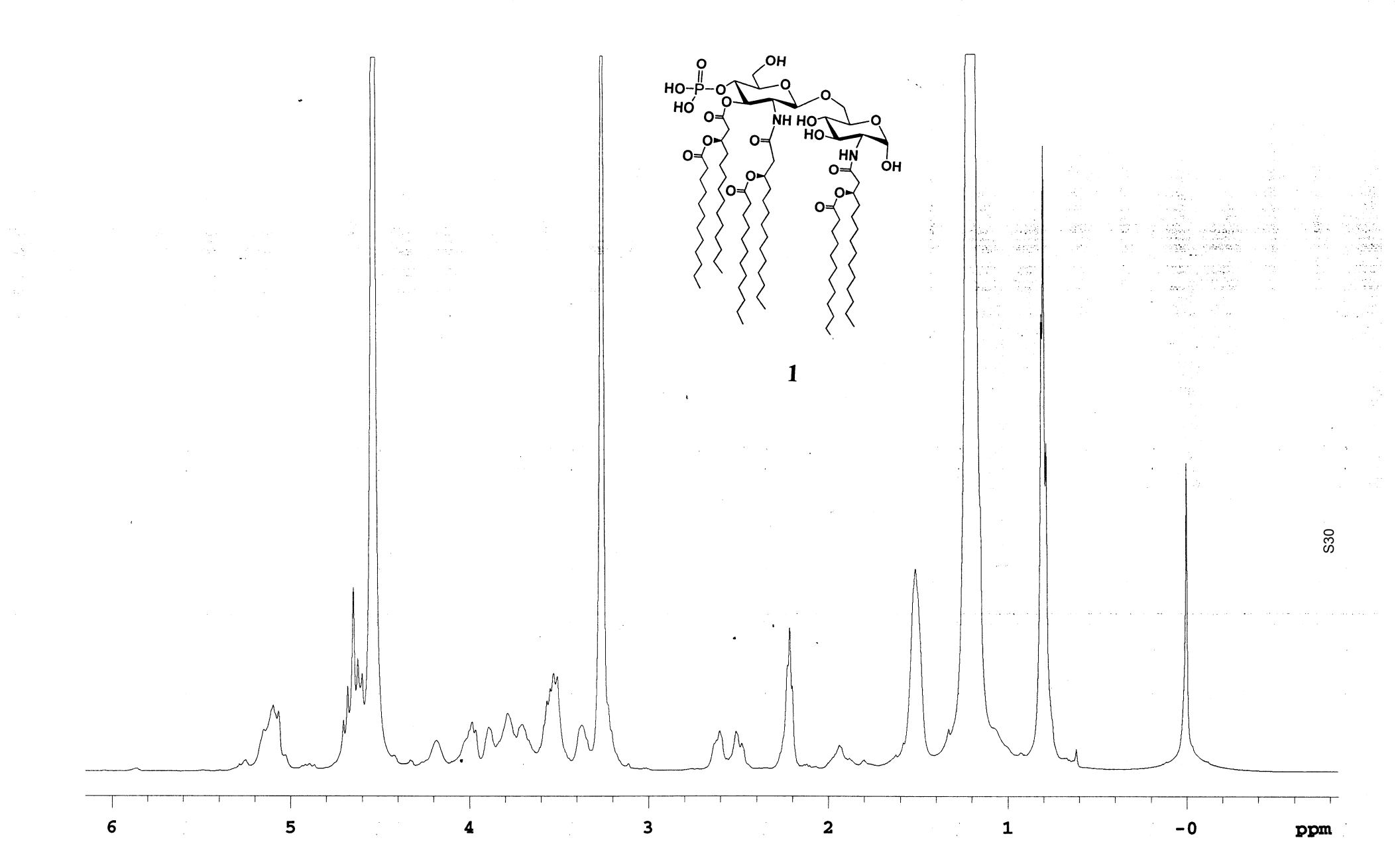

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