Synthesis of and molecular dynamics simulations on a tetrasaccharide corresponding to the repeating unit of the capsular polysaccharide from *Salmonella enteritidis*

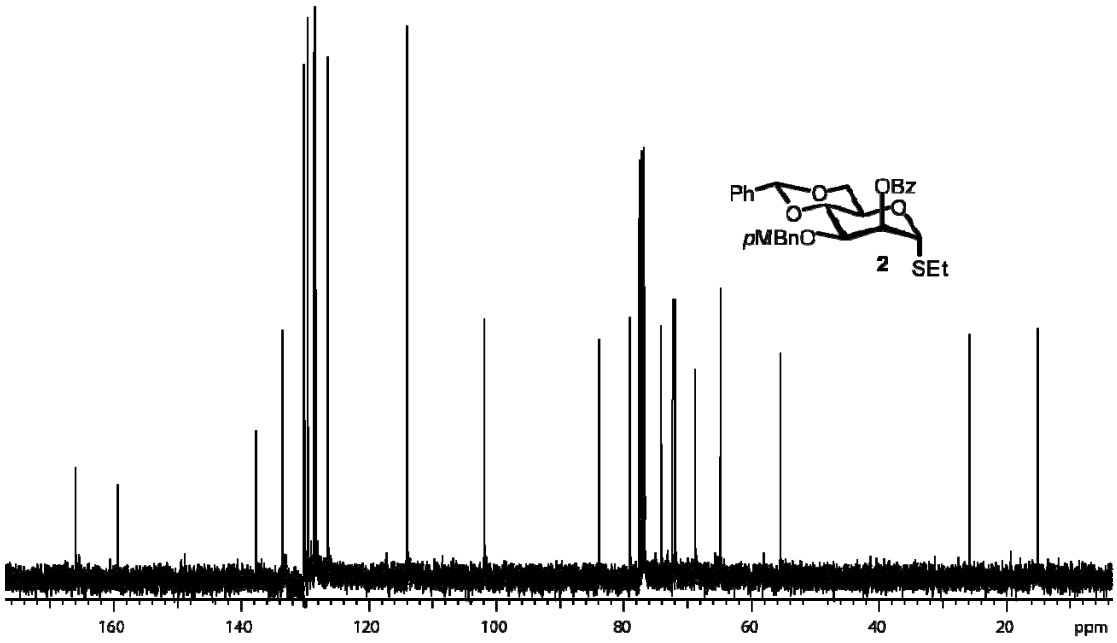
Johan D. M. Olsson,[a] Jens Landström,[a] Jerk Rönnols,[a] Stefan Oscarson[a,b] and Göran Widmalm*[a]

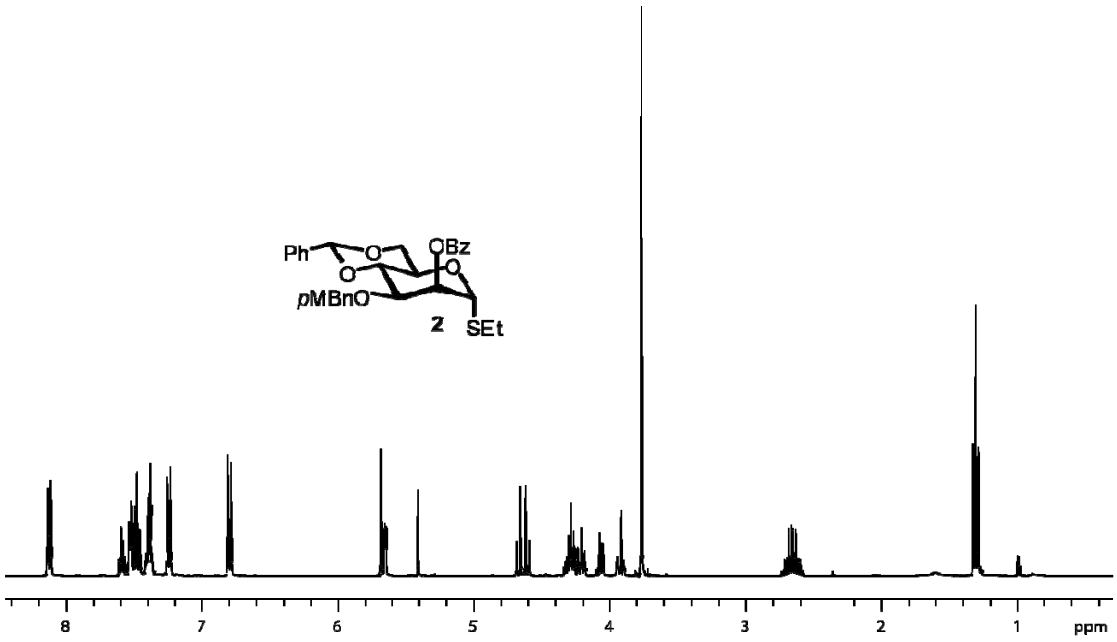
[a] Department of Organic Chemistry, Arrhenius Laboratory, Stockholm University, S-106 91 Stockholm, Sweden. E-mail: gw@organ.su.se

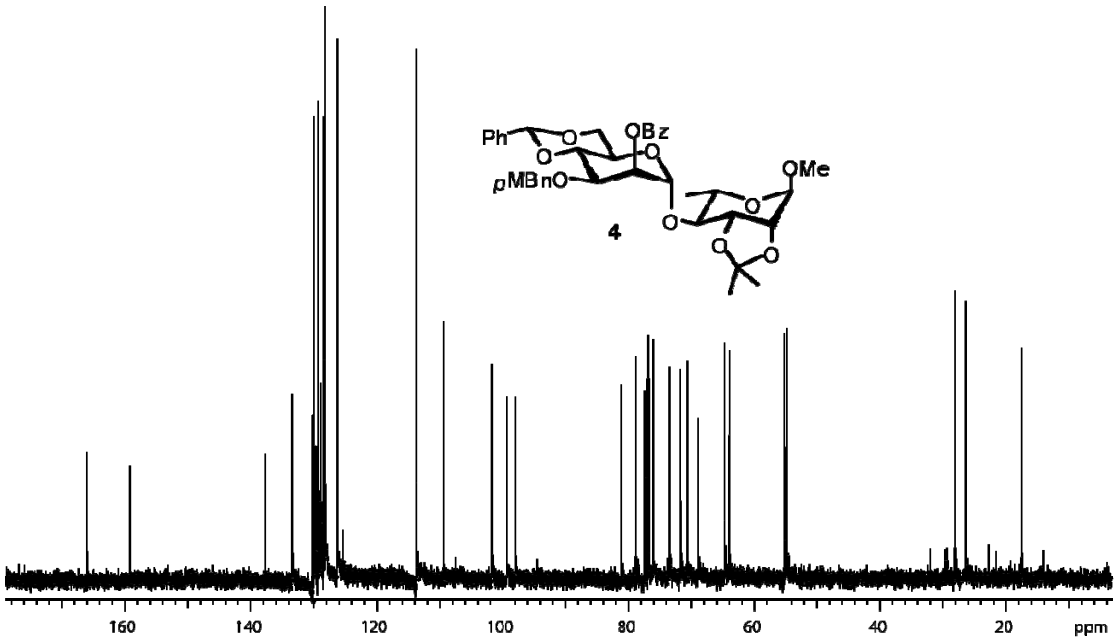
[b] Centre for Synthesis and Chemical Biology, University College Dublin, Belfield, Dublin 4, Ireland.

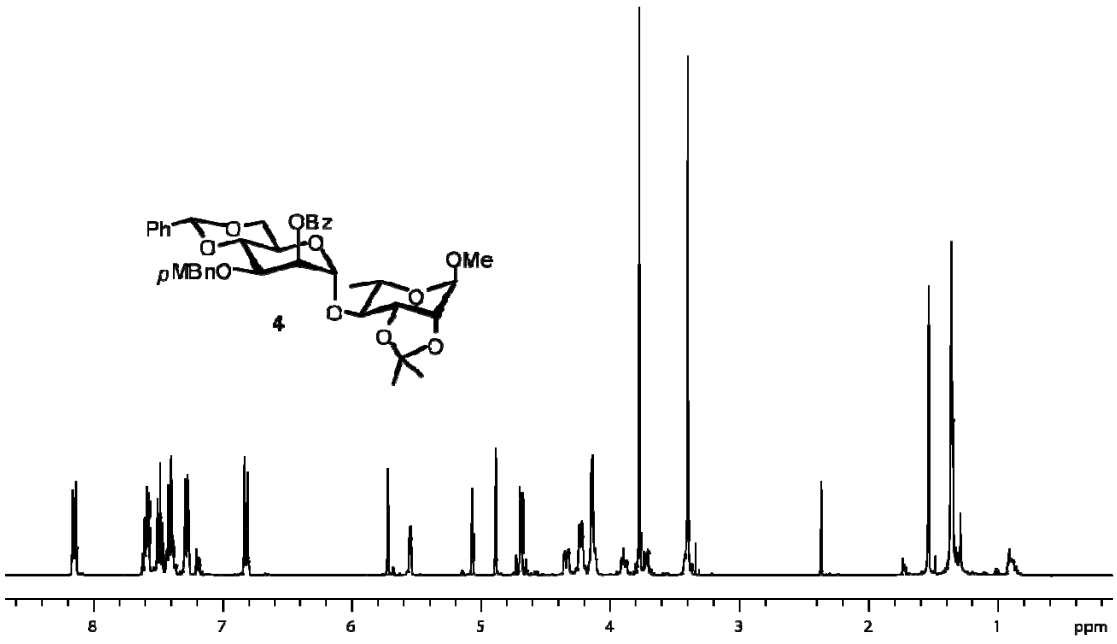
Supporting information

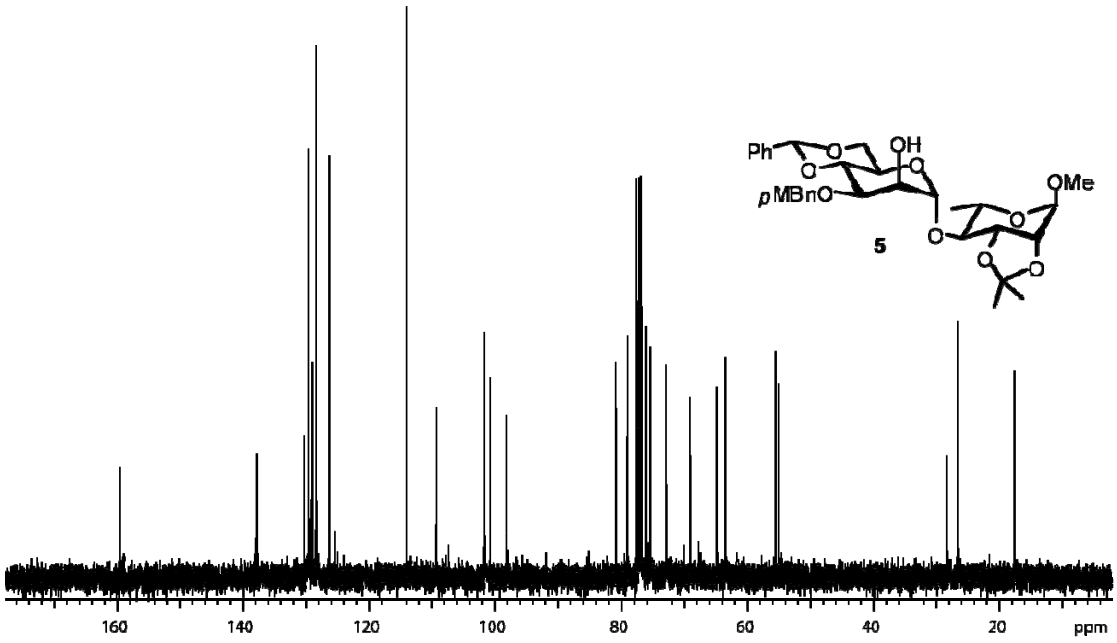
Table of contents

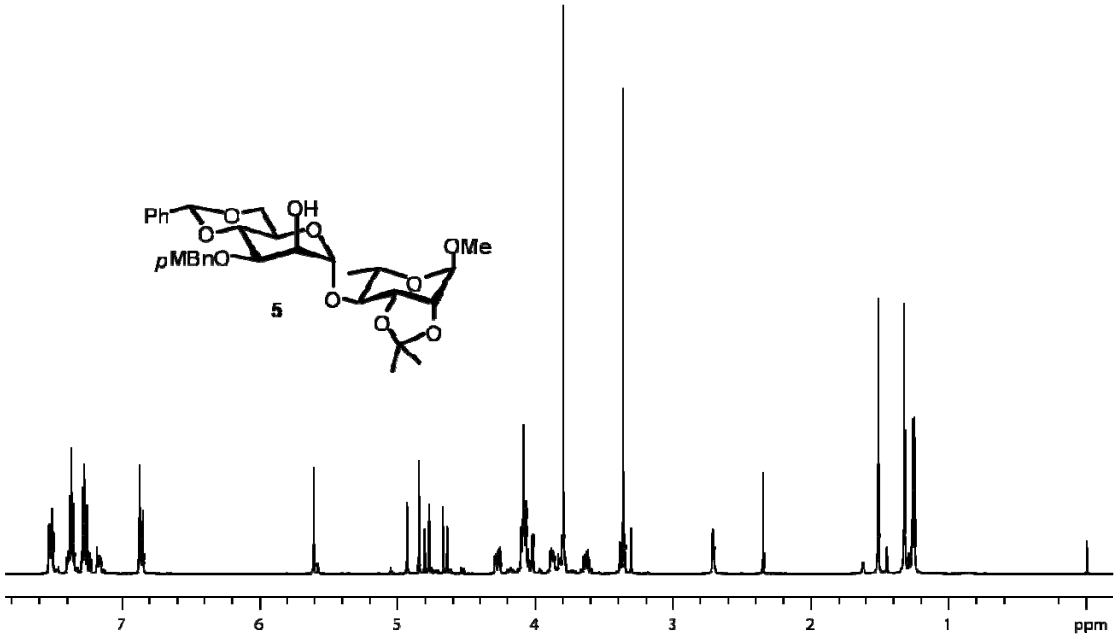

Ph
OBz
pMBnO
2
SEt
160
140
120
100
80
60
40
20
ppm

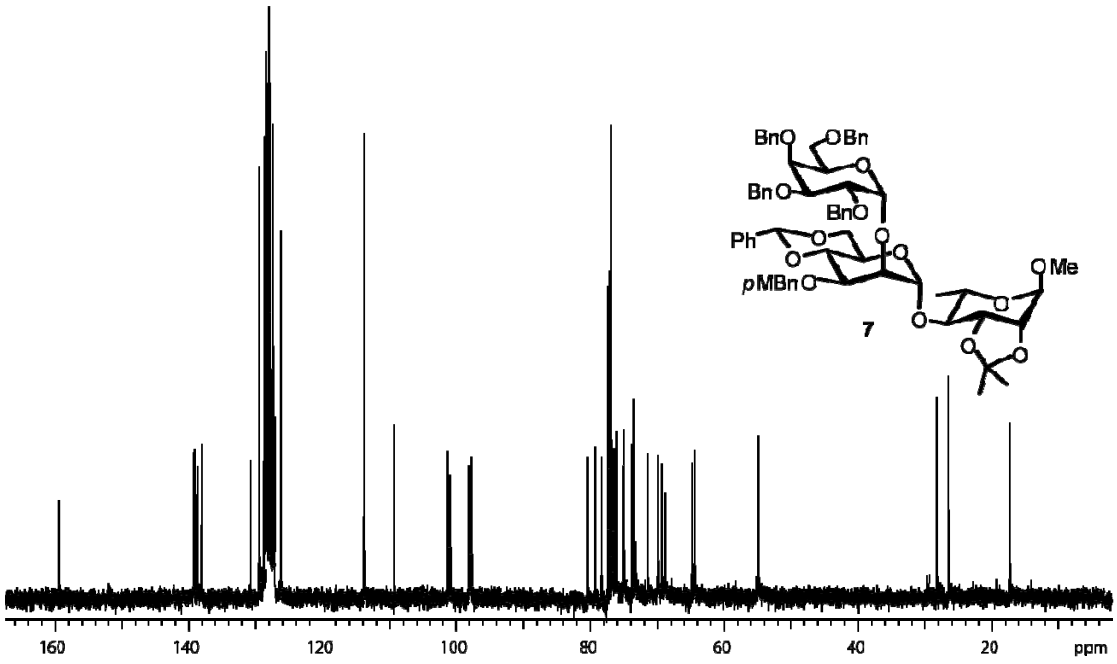

Ph
OBz
pMBnO
2
SEt
8
7
6
5
4
3
2
1
ppm

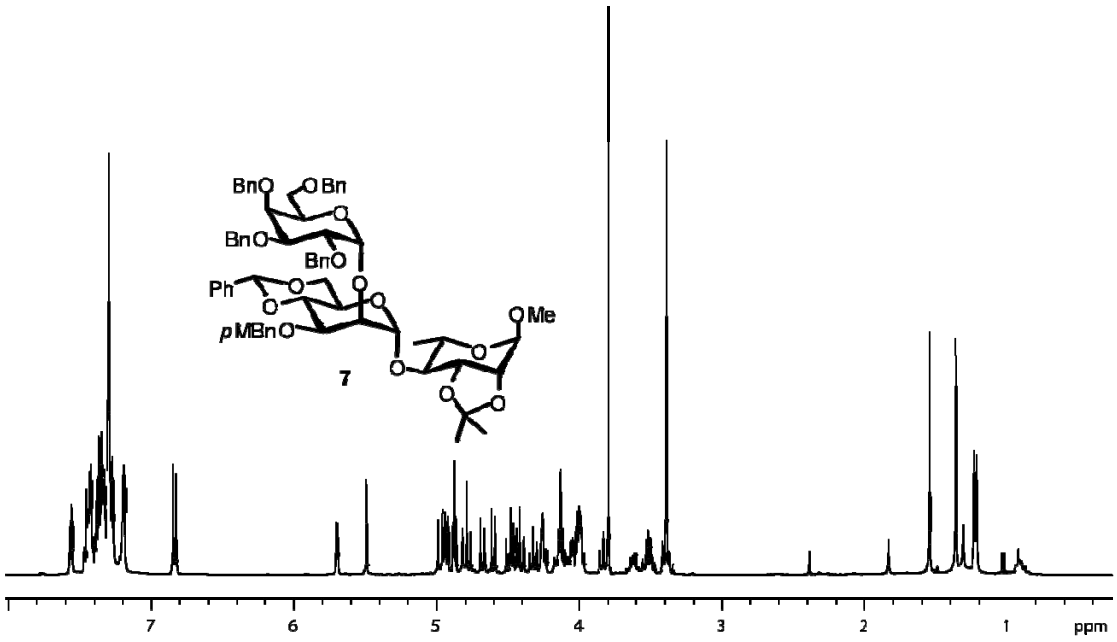

Ph
OBz
pMBnO
OMe
4
160
140
120
100
80
60
40
20
ppm

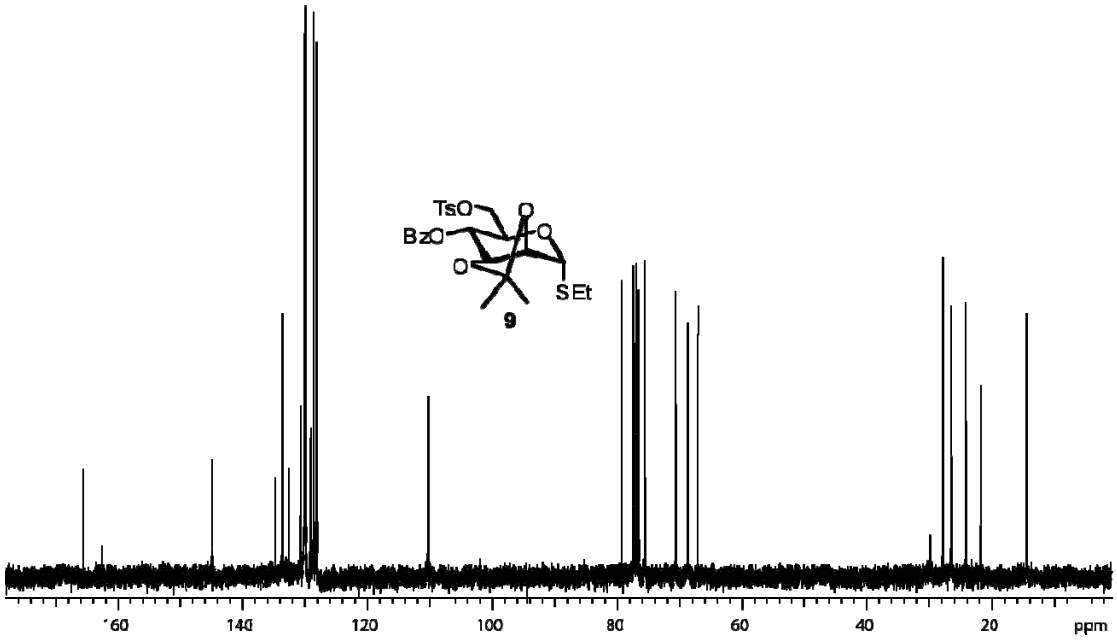

Ph
OBz
pMBnO
OMe
4
8
7
6
5
4
3
2
1
ppm

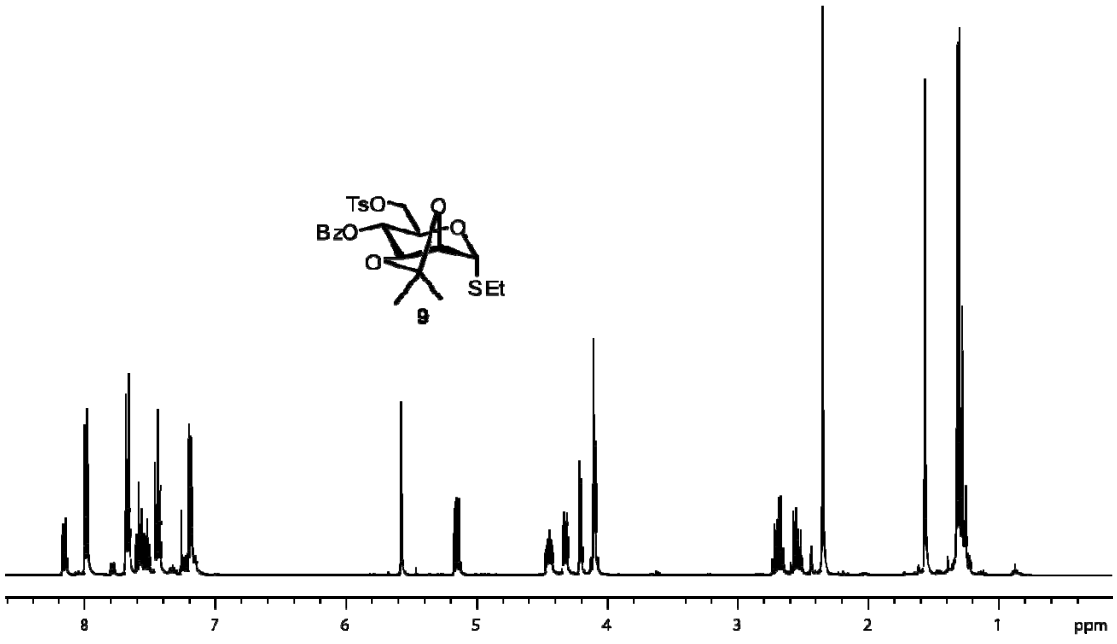

Ph
OH
pMBnO
OMe
5
160
140
120
100
80
60
40
20
ppm

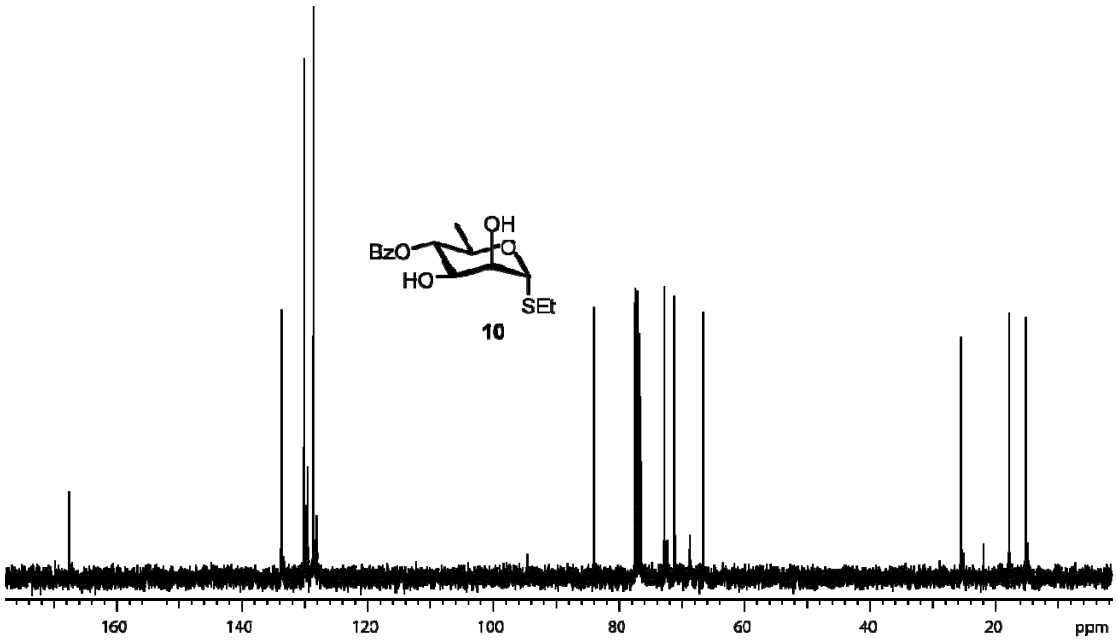

Ph
OH
pMBnO
OMe
5
7
6
5
4
3
2
1
ppm

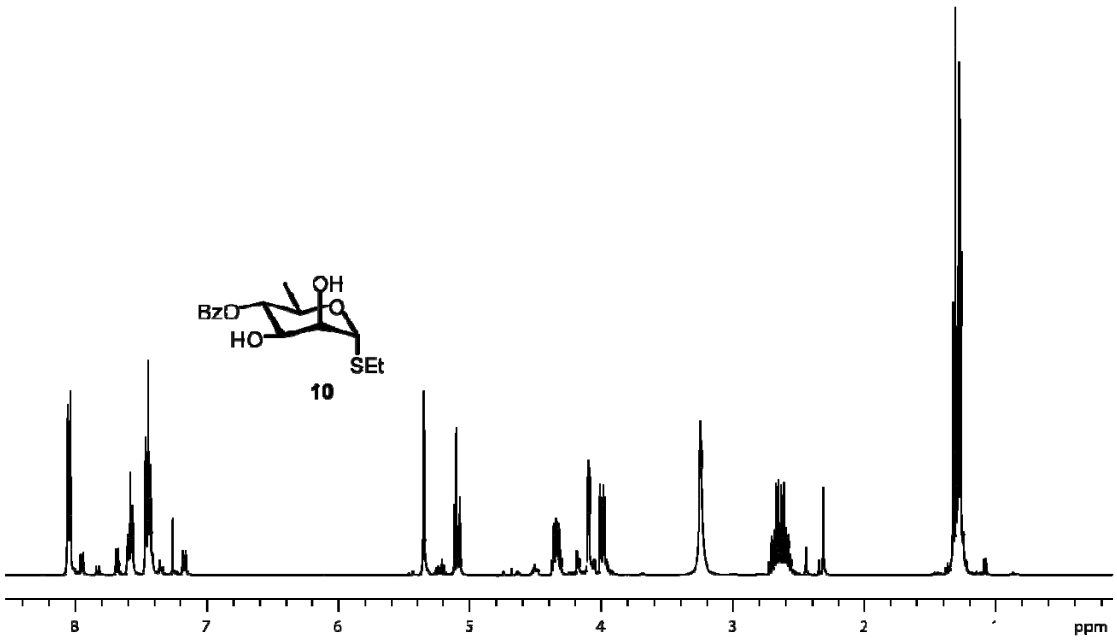

BnO
OBn
BnO
BnO
Ph
O
pMBnO
Me
7
160
140
120
100
80
60
40
20
ppm

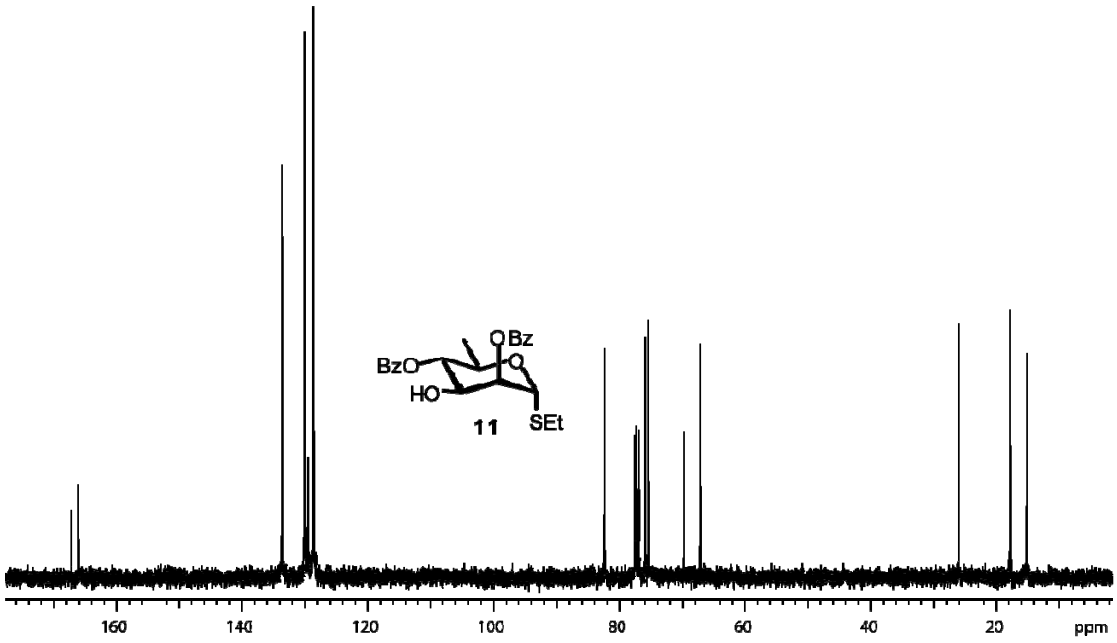

BnO
OBn
BnO
BnO
Ph
O
pMBnO
Me
7
7
6
5
4
3
2
1
ppm

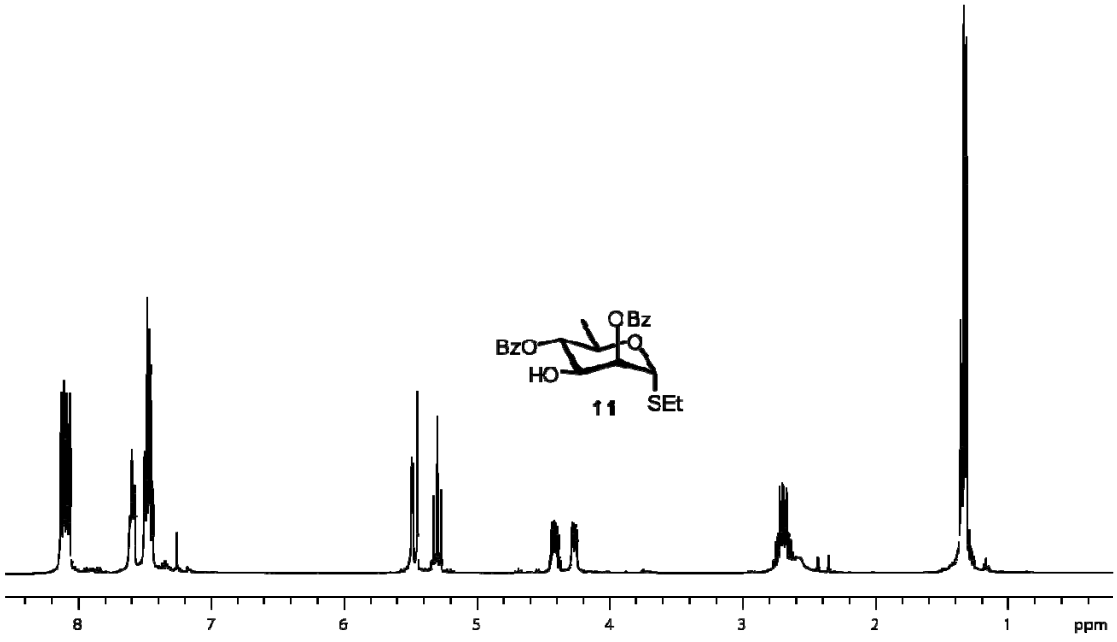

TsO
BzO
O
O
O
SEt
9
160
140
120
100
80
60
40
20
ppm

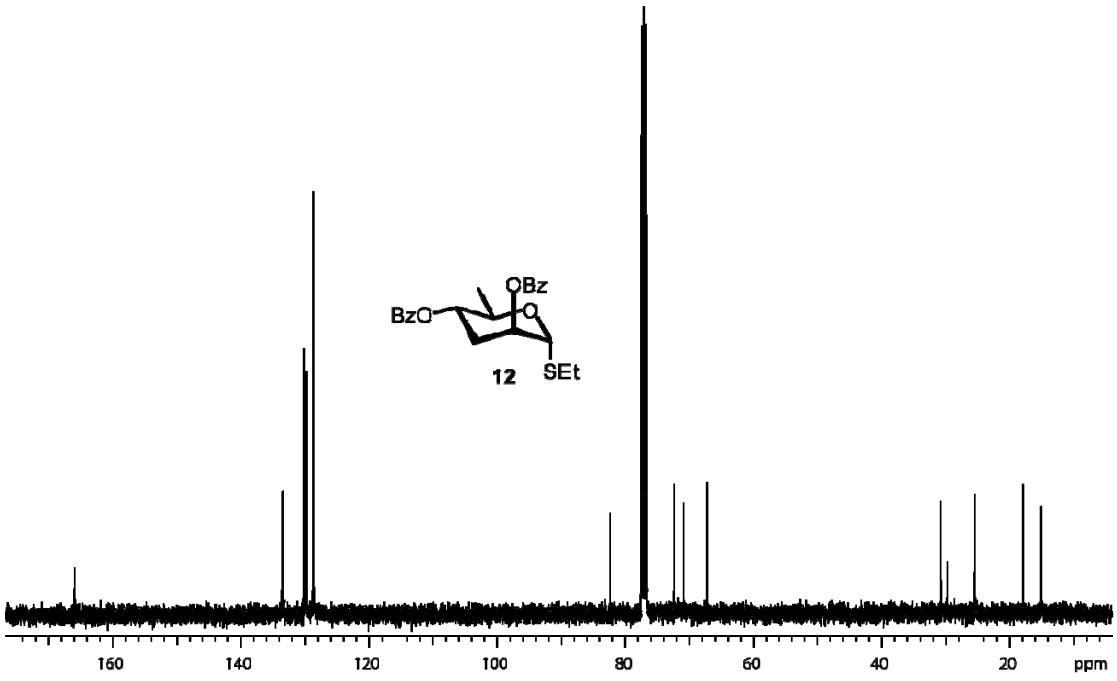

TsO
BzO
O
O
O
SEt
9
8
7
6
5
4
3
2
1
ppm

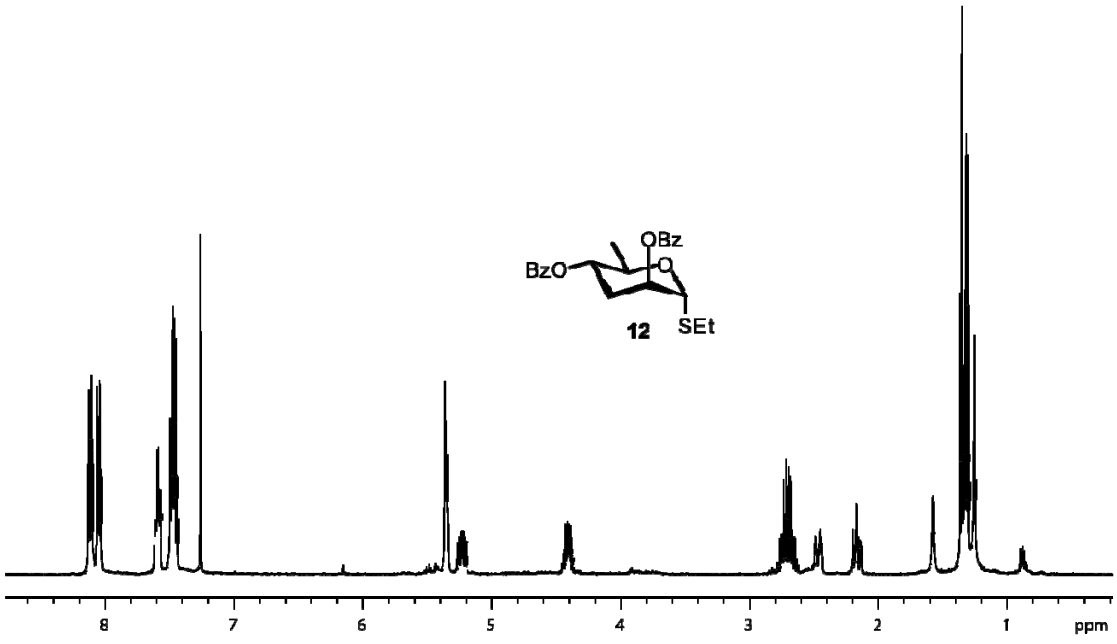

OH
BzO
O
HO
SEt
10
160
140
120
100
80
60
40
20
ppm

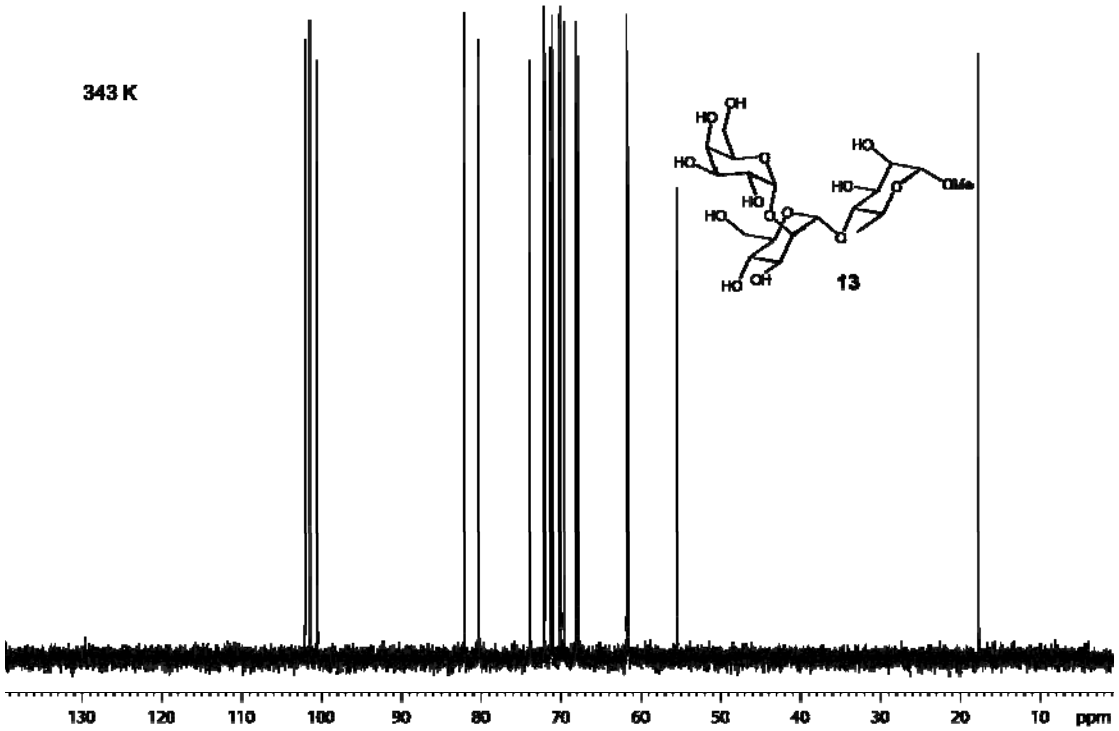

OH
BzO
O
HO
SEt
10
8
7
6
5
4
3
2
ppm

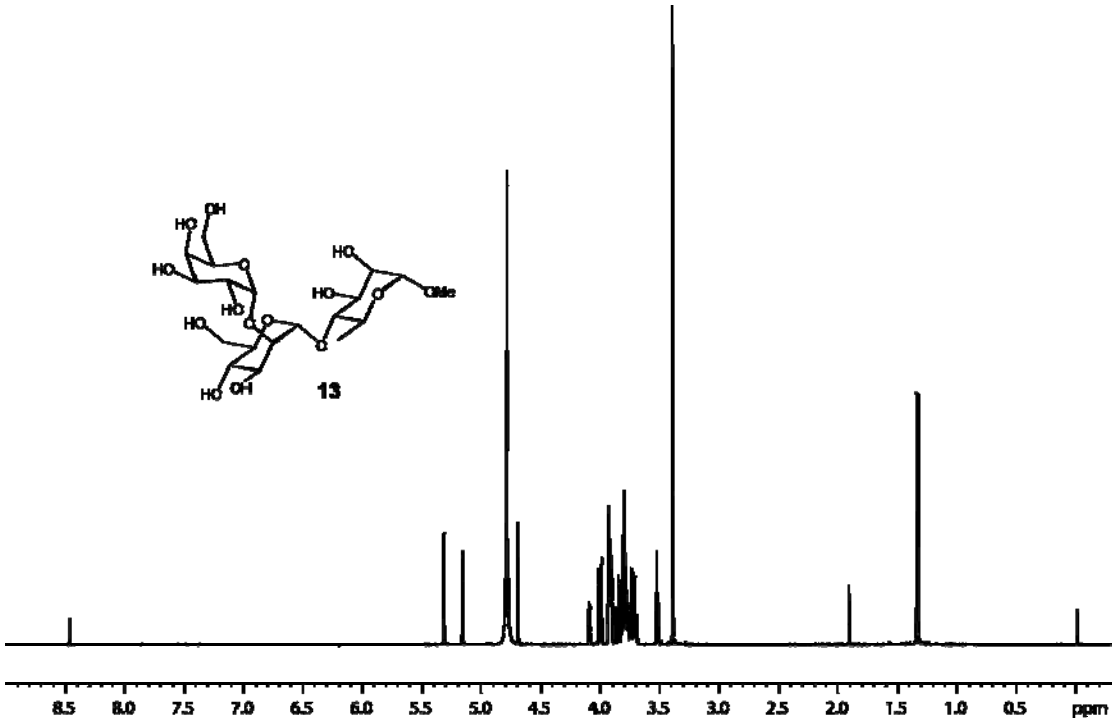

OBz
BzO
HO
SEt
11
160
140
120
100
80
60
40
20
ppm

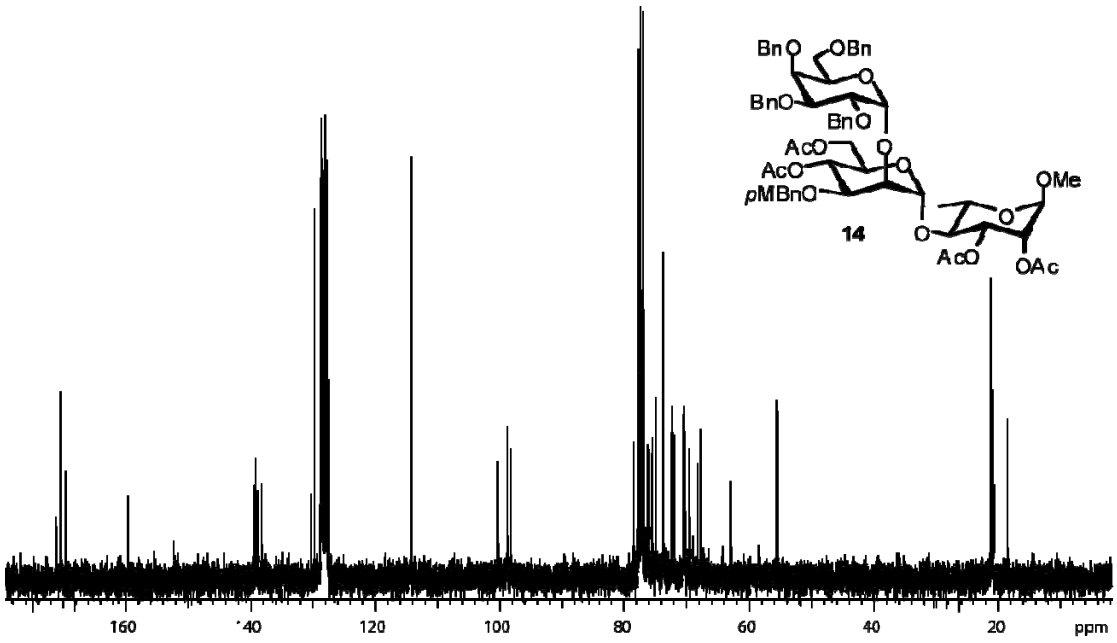

OBz
BzO
HO
SEt
11
8
7
6
5
4
3
2
1
ppm

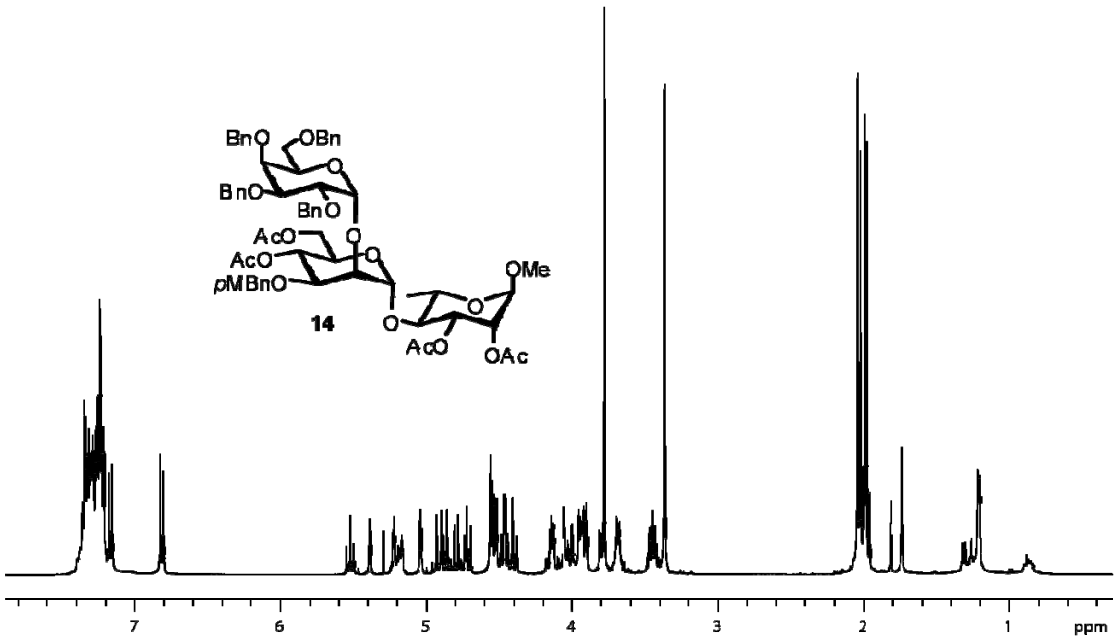

OBz
BzO
O
12
SEt
160
140
120
100
80
60
40
20
ppm

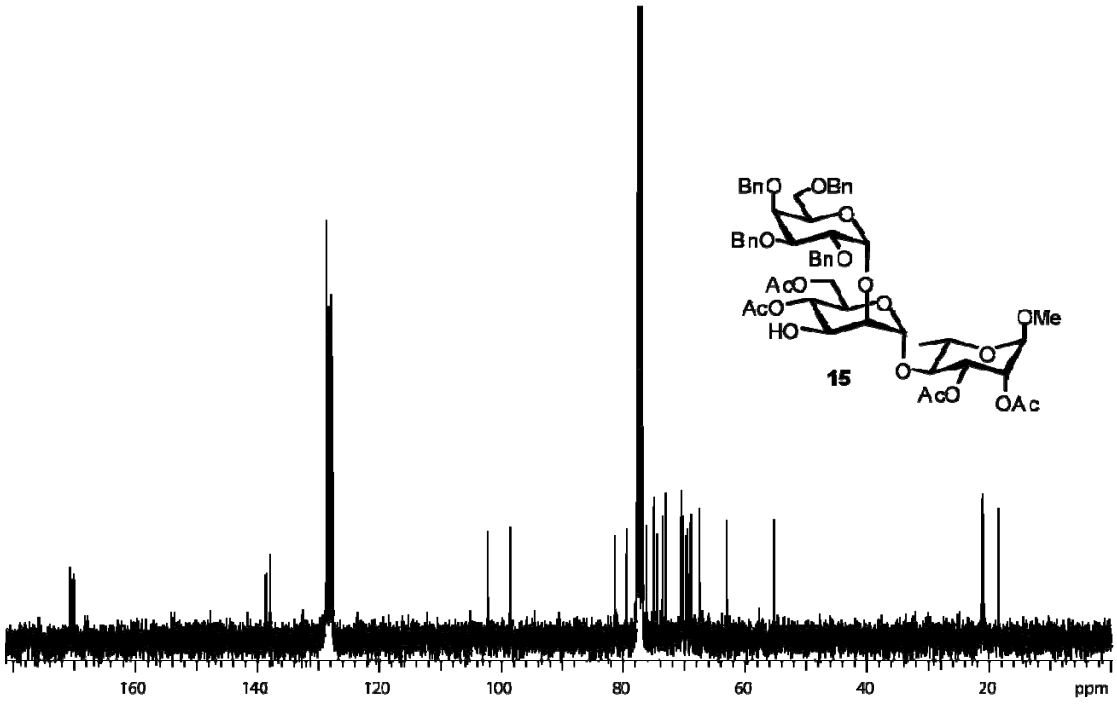

OBz
BzO
O
12
SEt
8
7
6
5
4
3
2
1
ppm

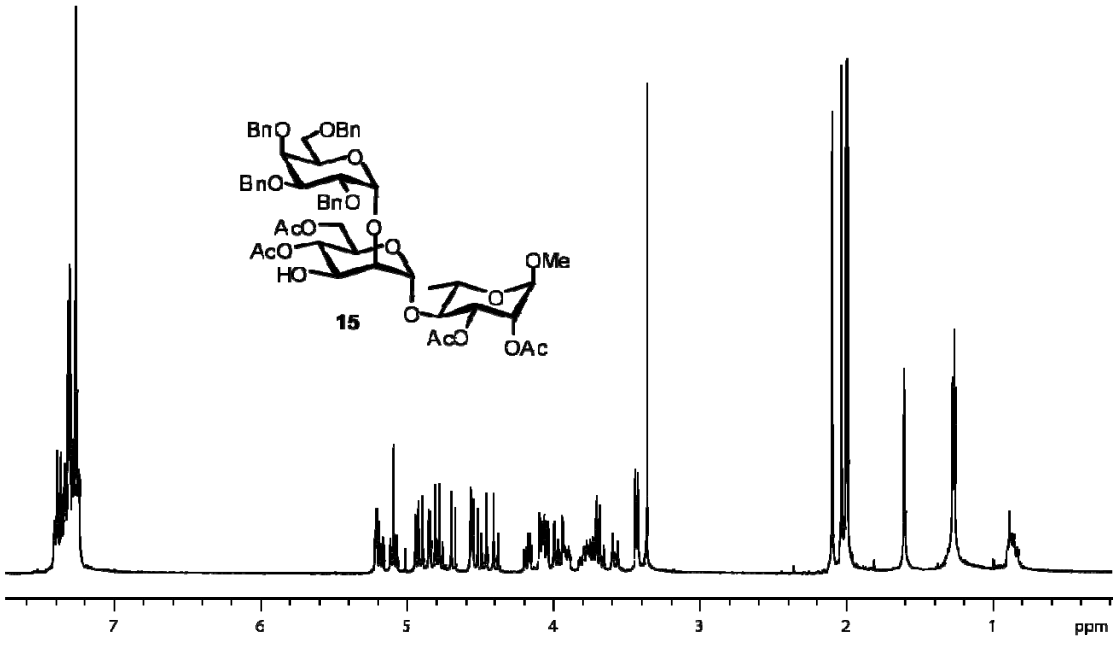

343 K
13
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

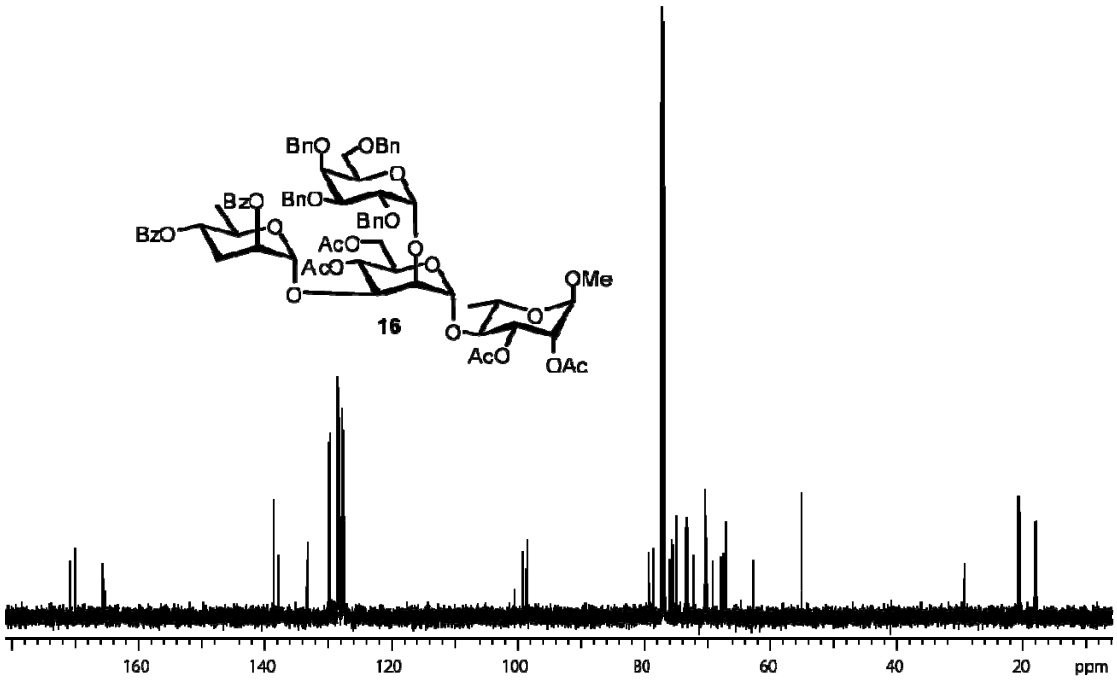

13
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

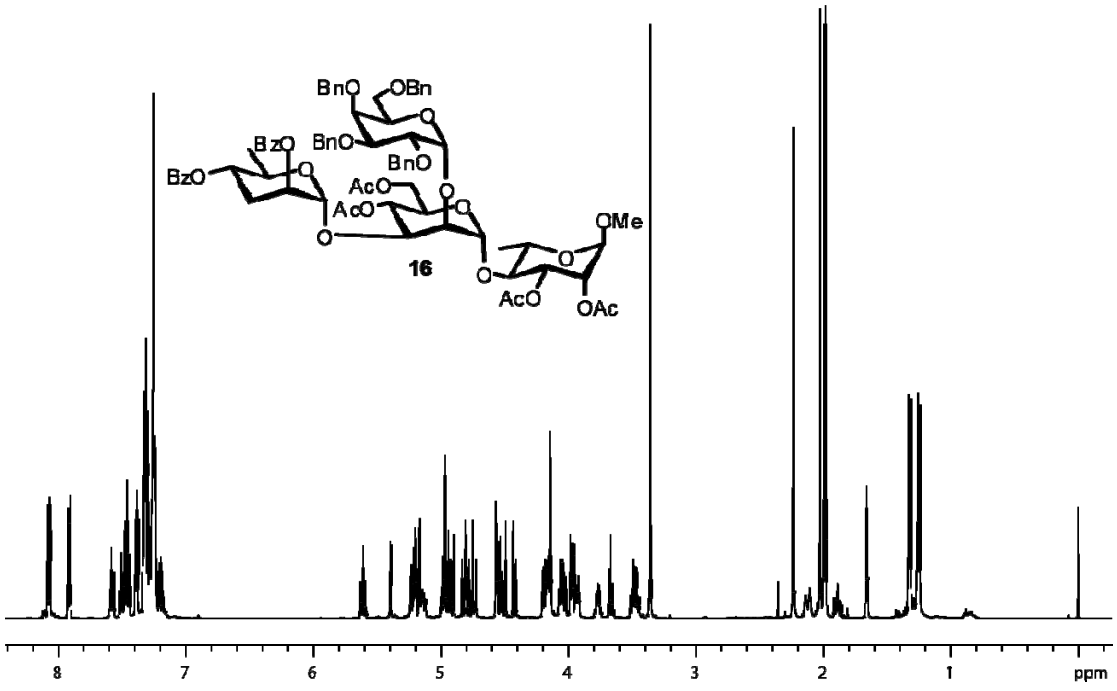

BnO
OBn
BnO
BnO
AcO
AcO
pMBnO
14
OMe
O
AcO
OAc
160
40
120
100
80
60
40
20
ppm

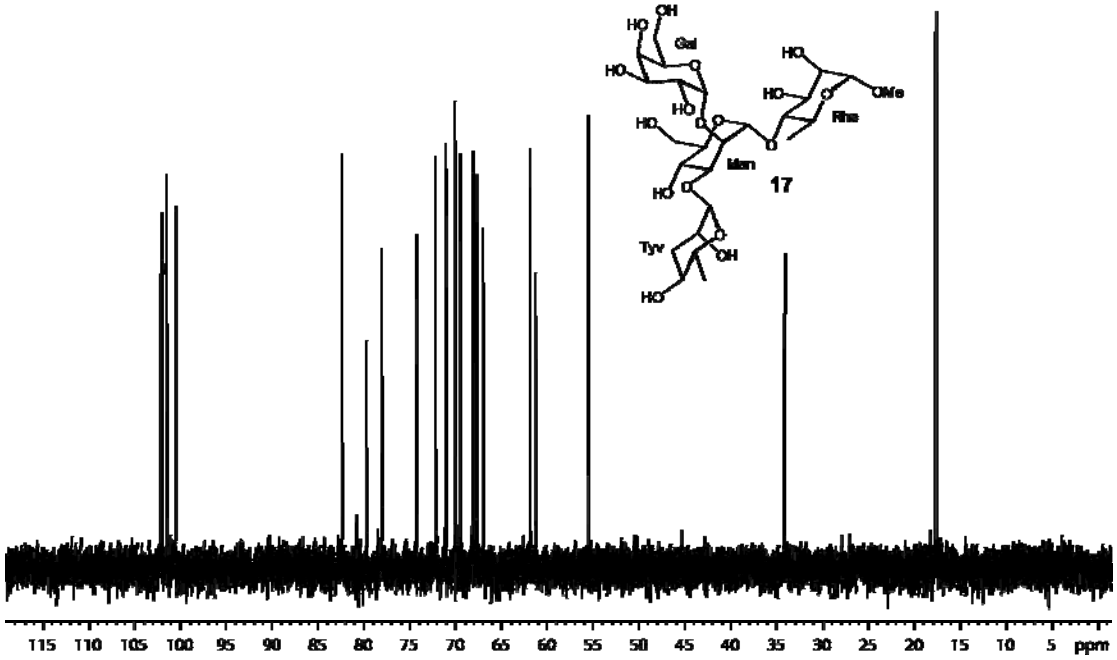

BnO
OBn
BnO
BnO
AcO
AcO
pMBnO
14
OMe
O
AcO
OAc
7
6
5
4
3
2
1
ppm

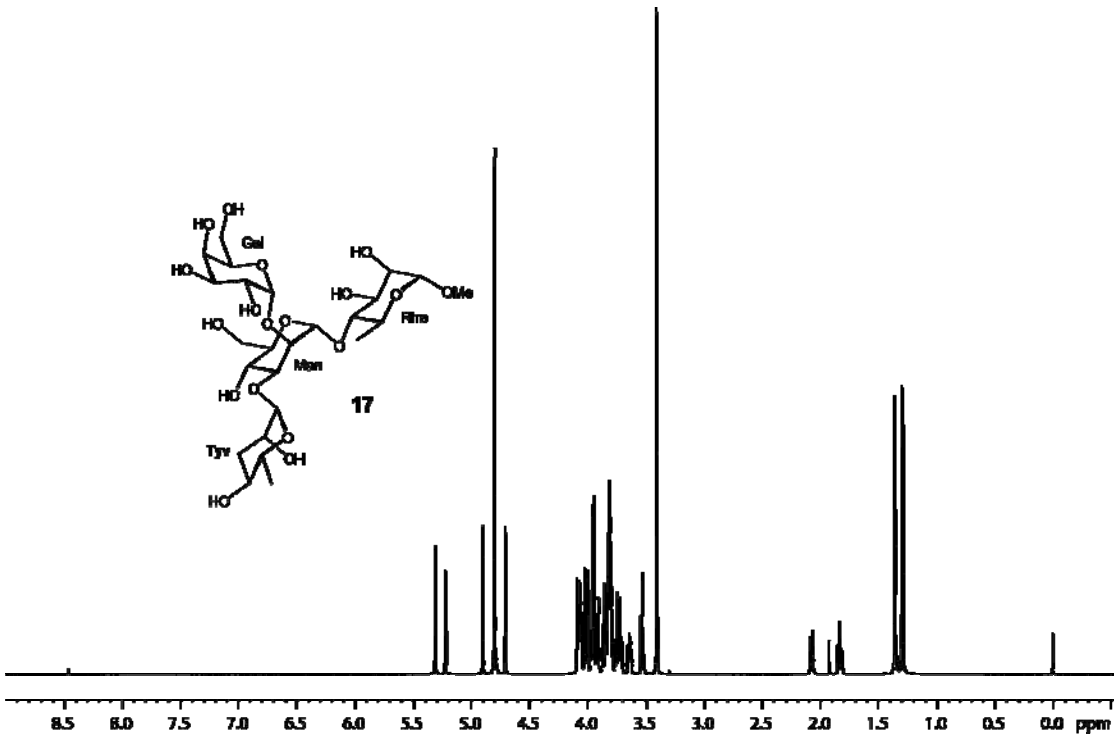

BnO
OBn
BnO
BnO
AcO
AcO
HO
OMe
15
AcO
OAc
160
140
120
100
80
60
40
20
ppm


BnO
OBn
BnO
BnO
AcO
AcO
HO
OMe
15
AcO
OAc
7
6
5
4
3
2
1
ppm


BnO
OBn
BzO
BnO
BnO
BzO
AcO
AcO
O
16
OMe
O
O
AcO
OAc
160
140
120
100
80
60
40
20
ppm


BnO
OBn
BzO
BnO
BnO
BzO
AcO
AcO
O
16
OMe
O
O
AcO
OAc
8
7
6
5
4
3
2
1
ppm


Gal
Rha
Man
17
Tyv
OMe
115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 ppm


Gal
Rha
Man
17
Tyv
OMe
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm

^{1}H and ^{13}C NMR chemical shift assignments and spectra

Table S1. ^{1}H and ^{13}C NMR chemical shifts and $J_{H,H}$ coupling constants in parenthesis of trisaccharide **13** in D_2O at 298 K.

Sugar	1	2	3	4	5	6R/S	OMe
Gal	5.16 (3.9)	3.83 (10.5)	3.92 (3.4)	4.00 (1.1)	4.10 (8.0, 4.3)	3.71, 3.75 (–11.8)	
	102.2	69.6	70.0	70.1	72.2	61.9	
Man	5.32 (1.9)	4.02 (3.3)	3.92 (9.8)	3.81 (10.0)	3.93 (5.1, 2.4)	3.80, 3.86 (–12.4)	
	100.5	80.6	71.3	67.6	73.8	61.3	
Rha	4.70 (1.8)	3.94 (3.5)	3.81 (9.4)	3.53 (9.5)	3.78 (6.3)	1.35	3.40
	101.4	71.1	69.8	82.1	68.2	17.9	55.5

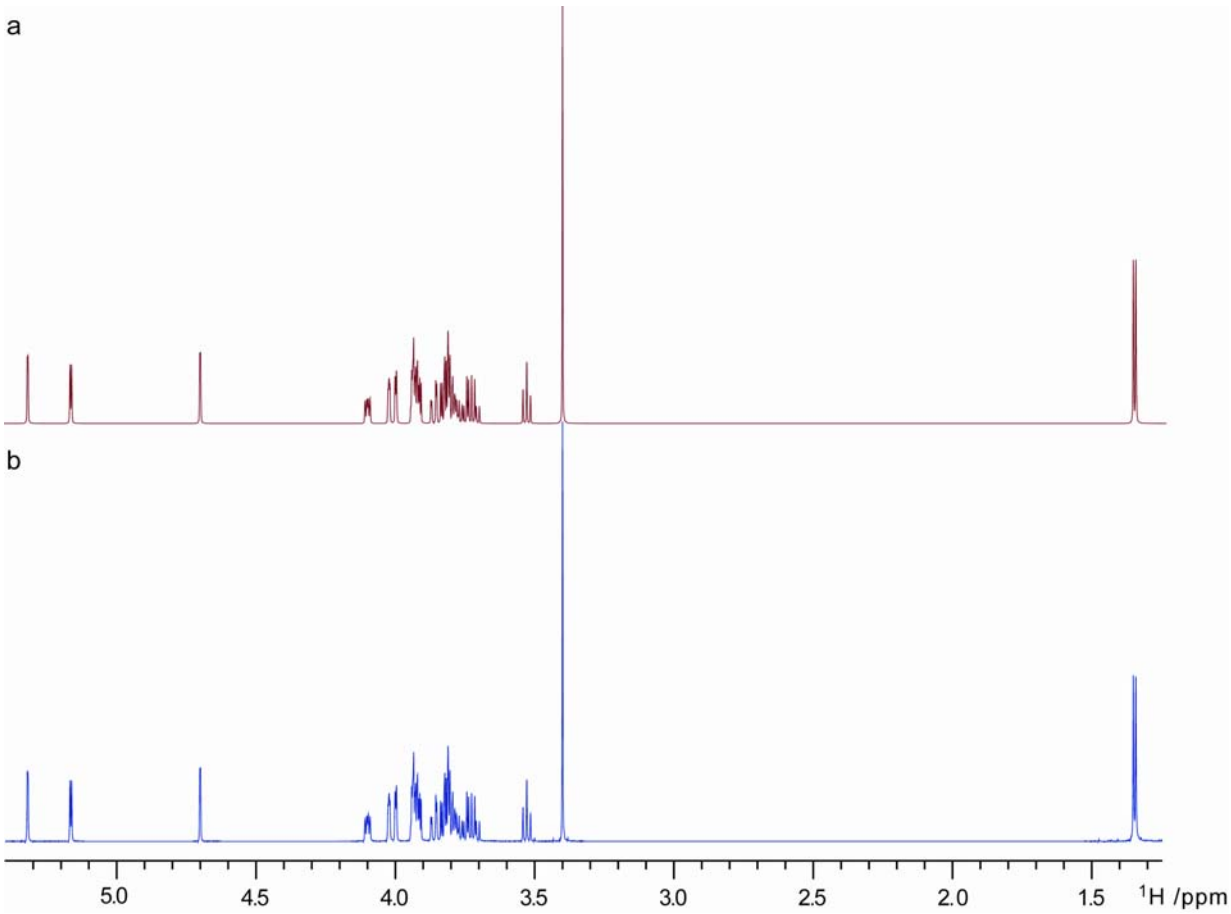


Figure S1. ^{1}H NMR spectra at 700 MHz of trisaccharide **13** from (a) simulation using PERCH and (b) experiment (prepared for PERCH analysis, e.g. solvent peak removed).

Table S2. ^{1}H and ^{13}C NMR chemical shifts and $J_{H,H}$ coupling constants in parenthesis of tetrasaccharide **17** in D_2O at 298 K.

Sugar	1	2	3	4	5	6R/S	OMe
Gal	5.22 (4.0)	3.81 (10.4)	3.91 (3.4)	4.00 (1.1)	4.07 (8.0, 4.3)	3.72, 3.75 (–11.9)	
	101.9	69.5	70.1	70.1	72.2	61.9	
Man	5.31 (2.0)	4.02 (3.0)	4.06 (9.7)	3.95 (10.2)	3.96 (4.7, 2.2)	3.81, 3.86 (–12.3)	
	100.5	79.7	78.1	67.0	74.2	61.3	
Rha	4.71 (1.8)	3.95 (3.5)	3.84 (9.4)	3.53 (9.5)	3.80 (6.3)	1.35	3.40
	101.4	71.1	70.0	82.4	68.1	17.9	55.5
Tyv	4.90 (1.6)	4.09 (3.2, 3.0)	2.07 (eq) (–13.6, 4.5) 1.83 (ax) (11.5)	3.64 (9.3)	3.80 (6.3)	1.29	
	102.1	68.1	34.2	67.7	71.1	17.6	

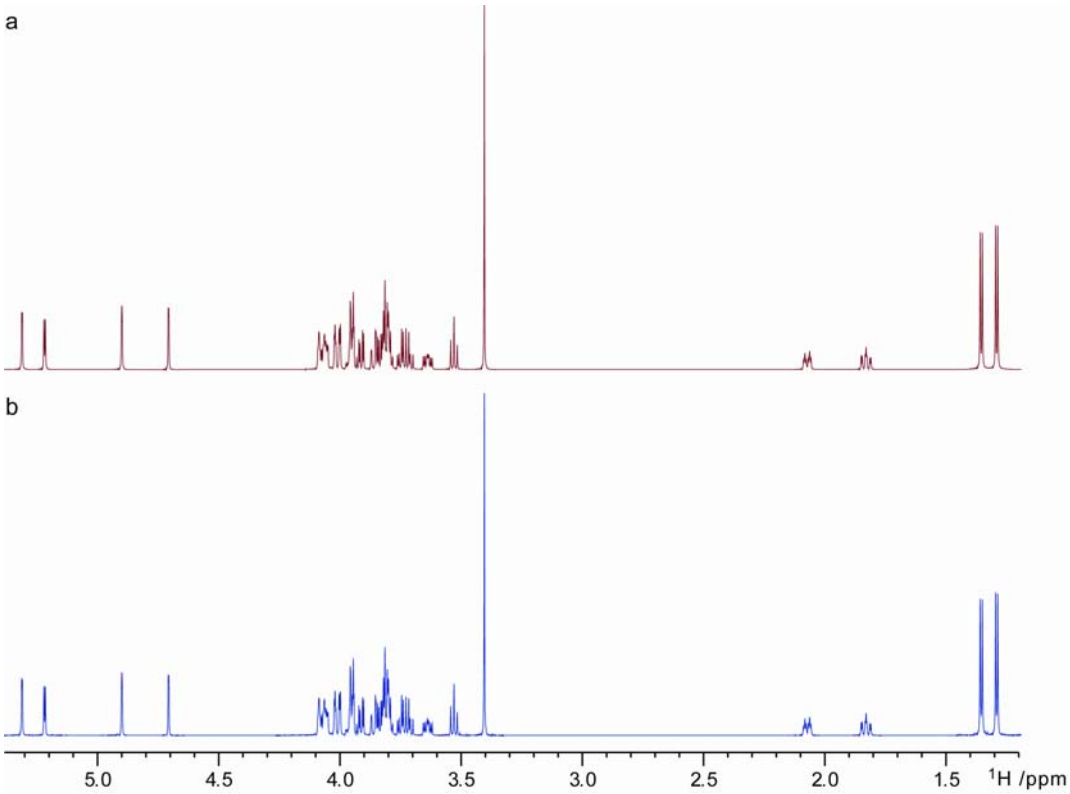


Figure S2. ^{1}H NMR spectra at 700 MHz of tetrasaccharide **17** from (a) simulation using PERCH and (b) experiment (prepared for PERCH analysis, e.g. solvent peak removed).