Electronic Supplementary Material (ESI) for Analyst.

Supporting information

Rapid Resolution of Carbohydrate Isomers via Multi-site Derivatization Ion Mobility-Mass Spectrometry

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Figure S1. Singly-labelled disaccharides' drift time overlay at m/z 516.121.

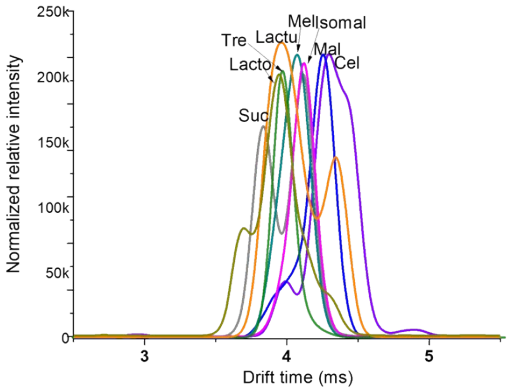
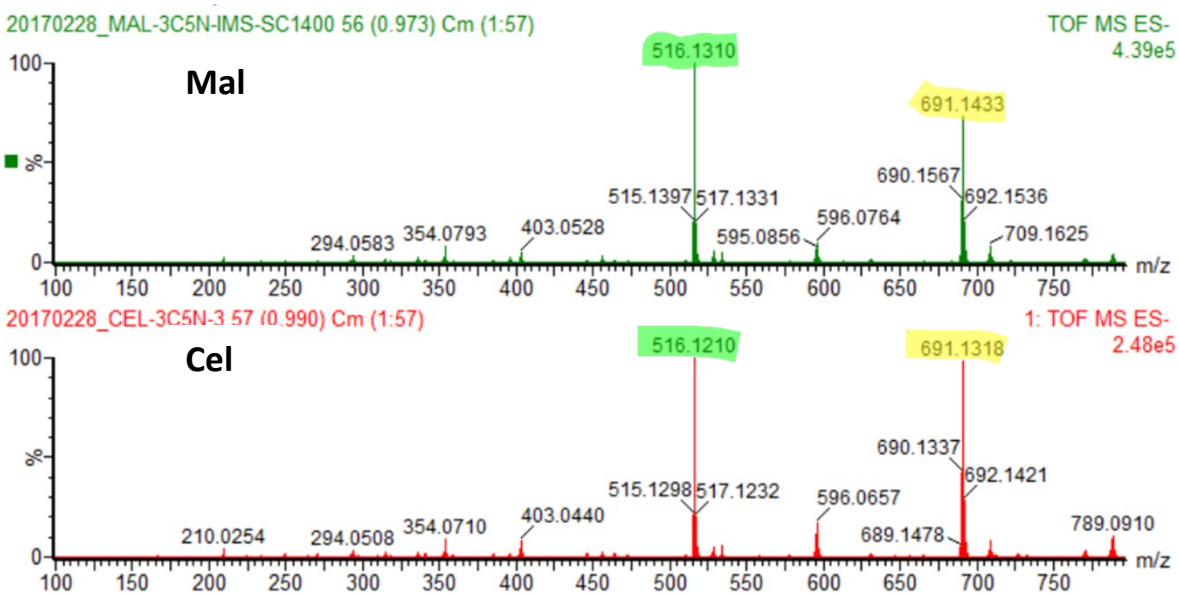
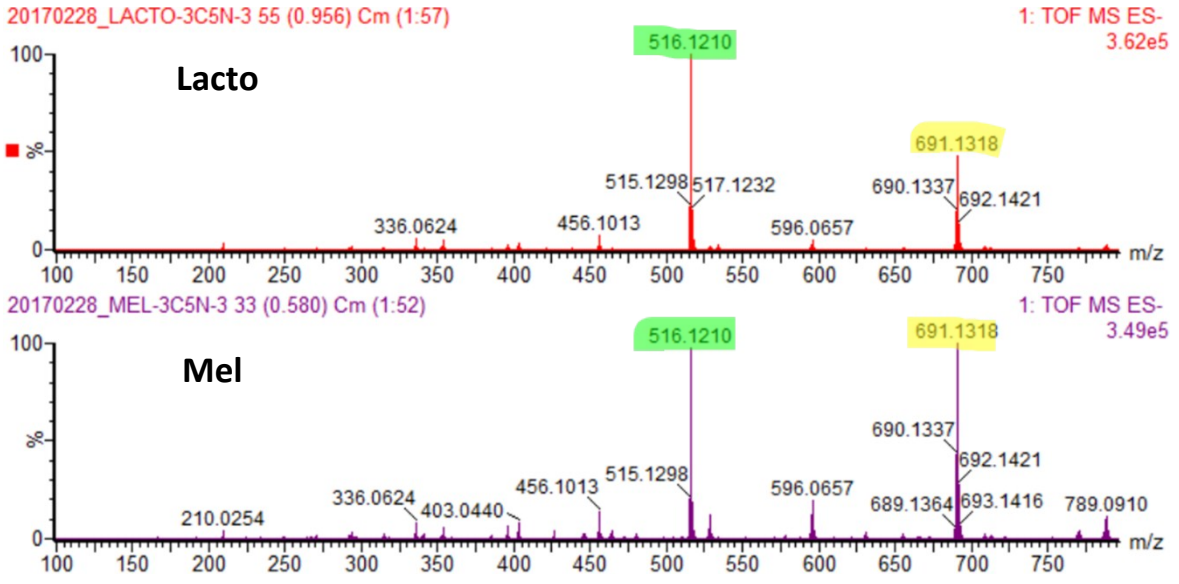
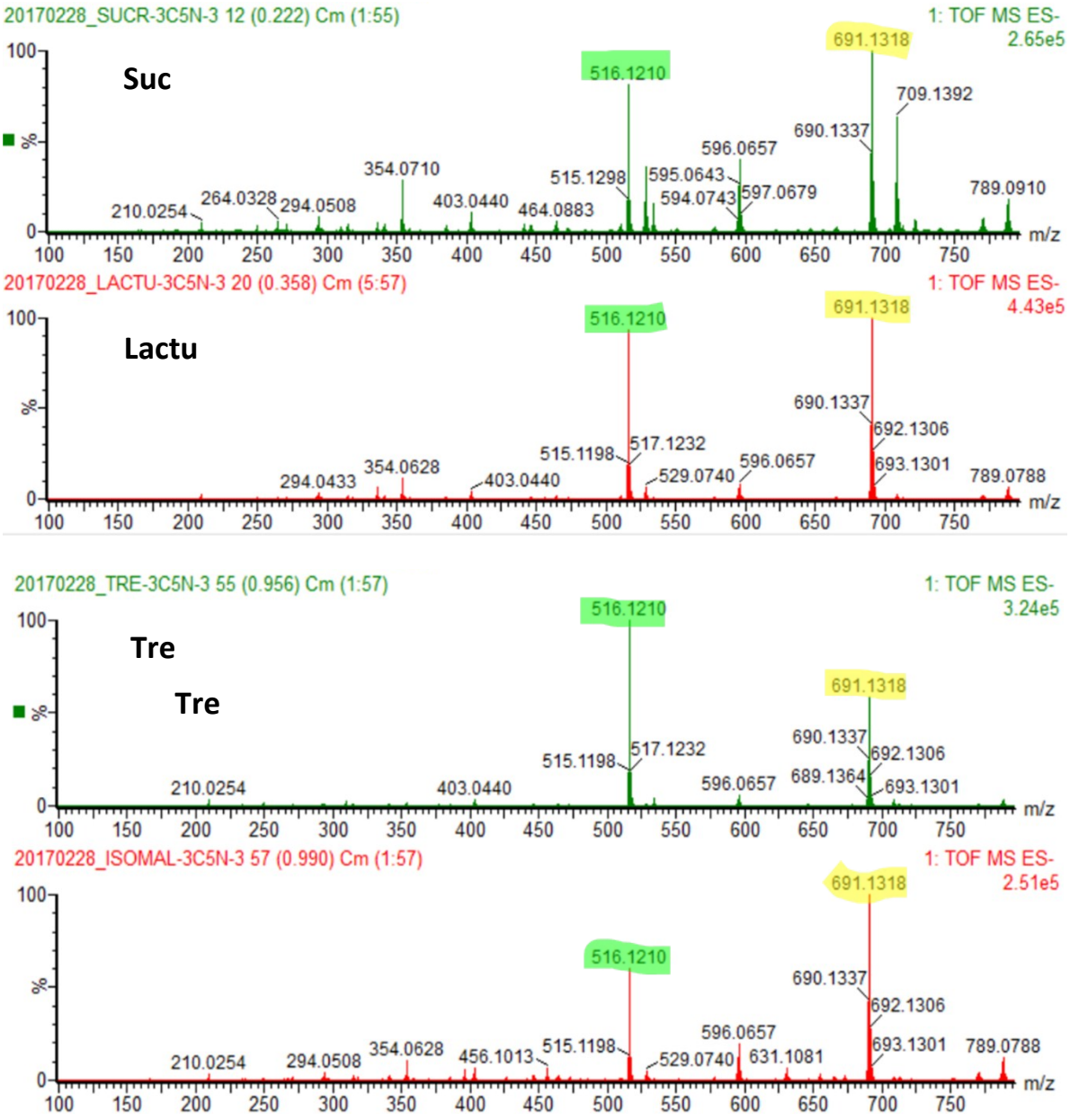


Figure S2. Mass spectra of reaction mixtures for individual sugar isomers reacted with 3C5NBA. The reaction was incubated at room temperature in aqueous solution, at a sugar:3C5NBA ratio of 1:2. For disaccharides, the incubation time was 5 minutes, whereas for monosaccharides it was 30 minutes. The time required for each measurement was approximately 15 minutes, including all the rinsing steps, etc. (Note: Singly-labeled species, *m/z* 516.1310 highlighted in green; the doubly labelled species, *m/z* 691.1433 highlighted in yellow)






20170228_SUCR-3C5N-3 12 (0.222) Cm (1:55)
1: TOF MS ES-
2.65e5
Suc
691.1318
516.1210
709.1392
690.1337
596.0657
354.0710
515.1298
595.0643
597.0679
594.0743
789.0910
210.0254
264.0328
294.0508
403.0440
464.0883
20170228_LACTU-3C5N-3 20 (0.358) Cm (5:57)
1: TOF MS ES-
4.43e5
Lactu
516.1210
691.1318
690.1337
692.1306
515.1198
517.1232
693.1301
596.0657
354.0628
529.0740
294.0433
403.0440
789.0788
20170228_TRE-3C5N-3 55 (0.956) Cm (1:57)
1: TOF MS ES-
3.24e5
Tre
Tre
516.1210
691.1318
690.1337
515.1198
517.1232
692.1306
210.0254
403.0440
596.0657
689.1364
693.1301
20170228_ISOMAL-3C5N-3 57 (0.990) Cm (1:57)
1: TOF MS ES-
2.51e5
691.1318
516.1210
690.1337
692.1306
596.0657
210.0254
294.0508
354.0628
456.1013
515.1198
529.0740
631.1081
693.1301
789.0788
%
m/z

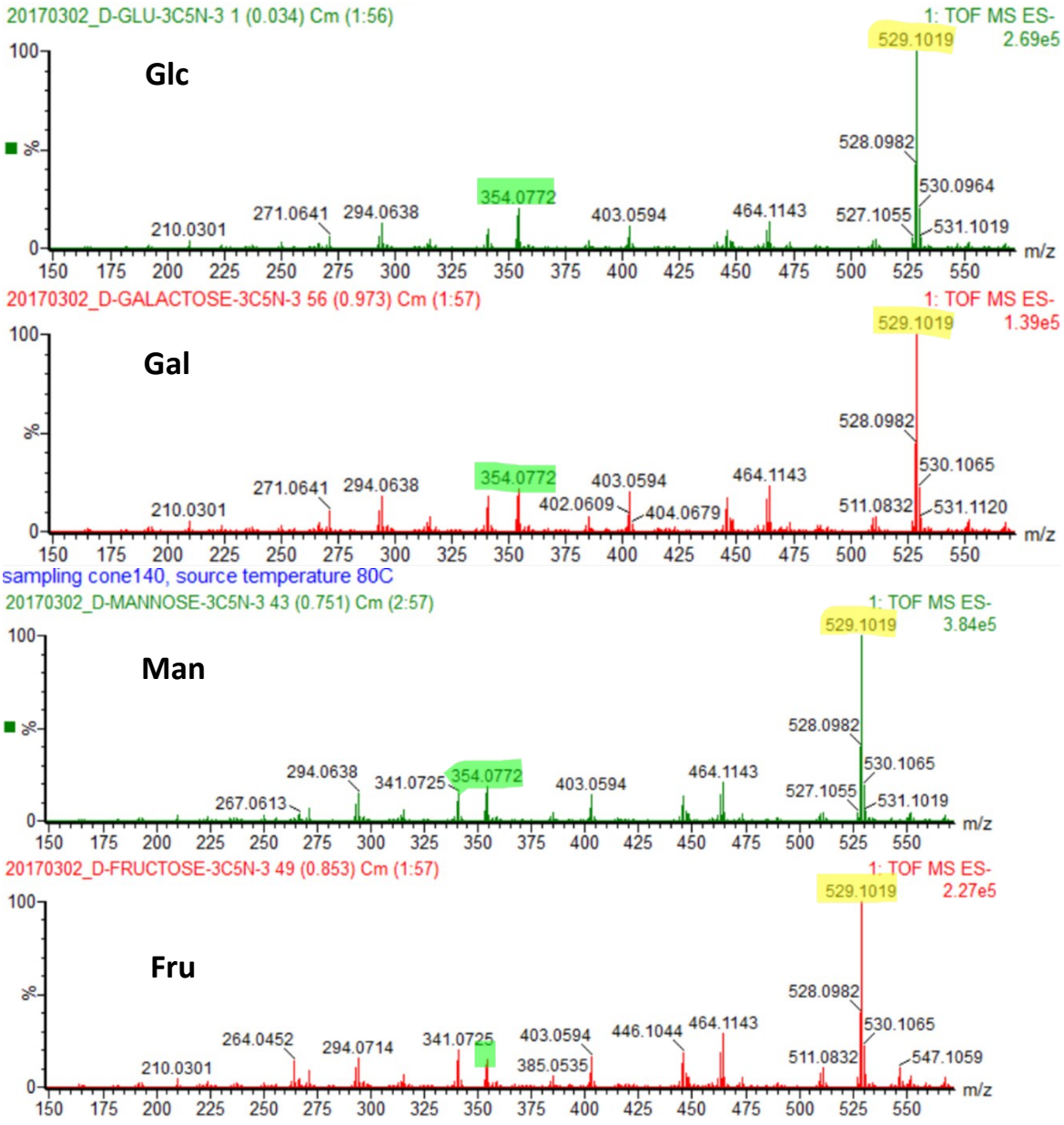
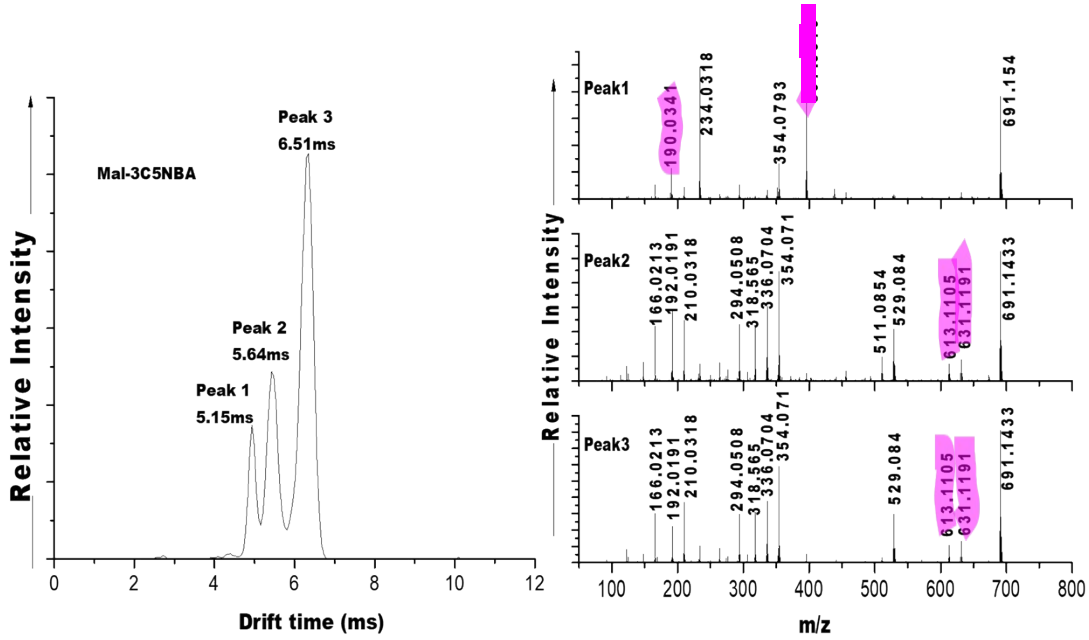
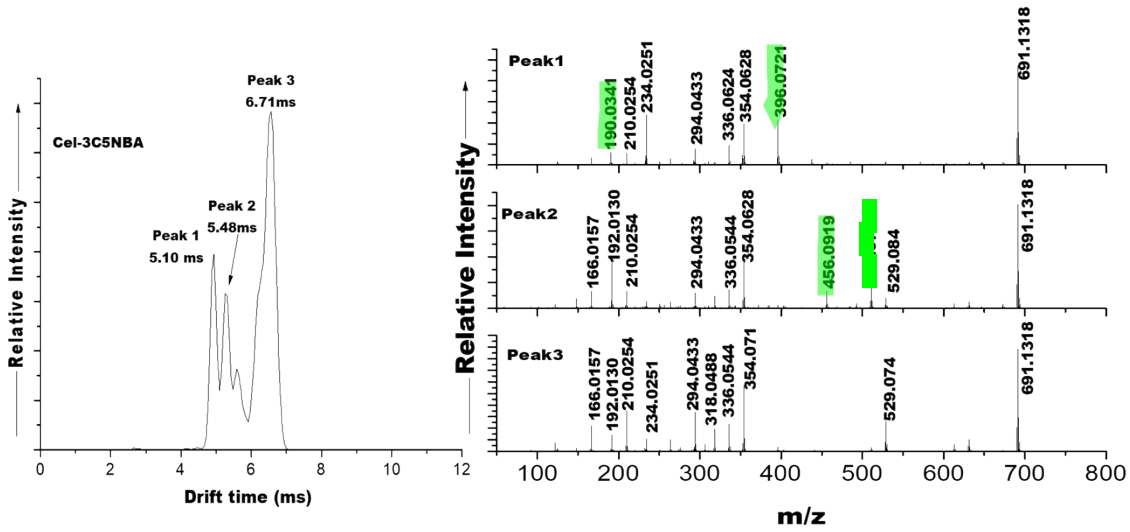
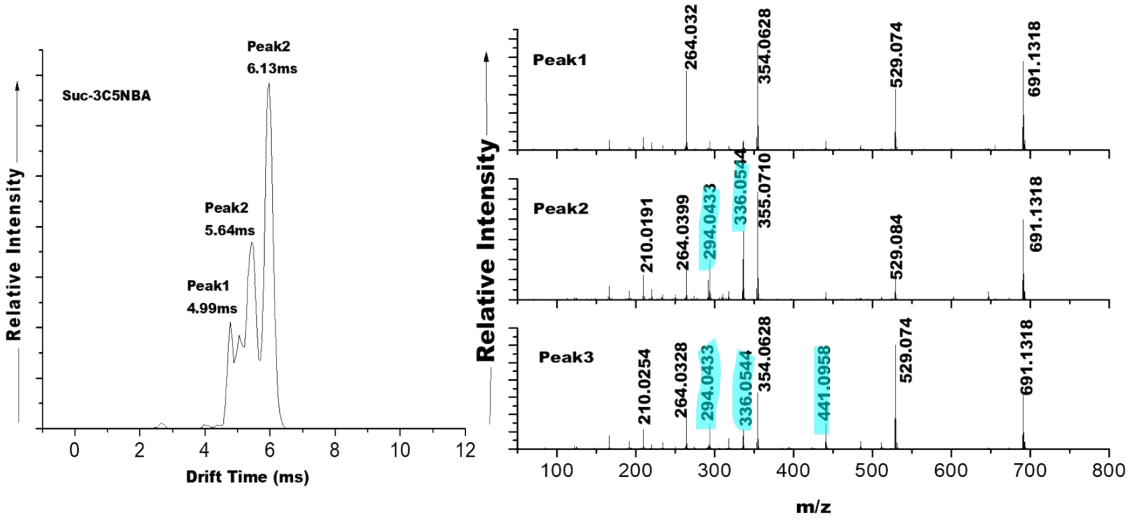
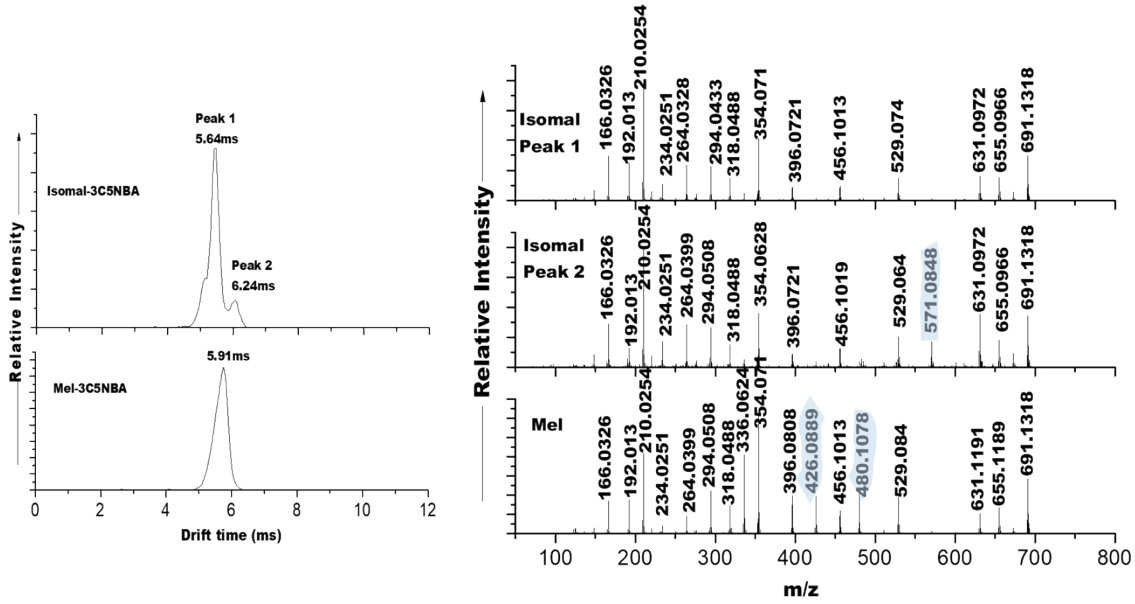

20170302_D-GLU-3C5N-3 1 (0.034) Cm (1:56)
1: TOF MS ES-
529.1019
2.69e5
Glc
528.0982
530.0964
354.0772
271.0641
294.0638
403.0594
464.1143
527.1055
531.1019
210.0301
m/z
20170302_D-GALACTOSE-3C5N-3 56 (0.973) Cm (1:57)
1: TOF MS ES-
529.1019
1.39e5
Gal
528.0982
530.1065
354.0772
271.0641
294.0638
403.0594
464.1143
402.0609
404.0679
511.0832
531.1120
210.0301
m/z
sampling cone140, source temperature 80C
20170302_D-MANNOSE-3C5N-3 43 (0.751) Cm (2:57)
1: TOF MS ES-
529.1019
3.84e5
Man
528.0982
530.1065
294.0638
341.0725
354.0772
403.0594
464.1143
527.1055
531.1019
267.0613
m/z
20170302_D-FRUCTOSE-3C5N-3 49 (0.853) Cm (1:57)
1: TOF MS ES-
529.1019
2.27e5
Fru
528.0982
530.1065
264.0452
294.0714
341.0725
403.0594
446.1044
464.1143
385.0535
511.0832
547.1059
210.0301
m/z

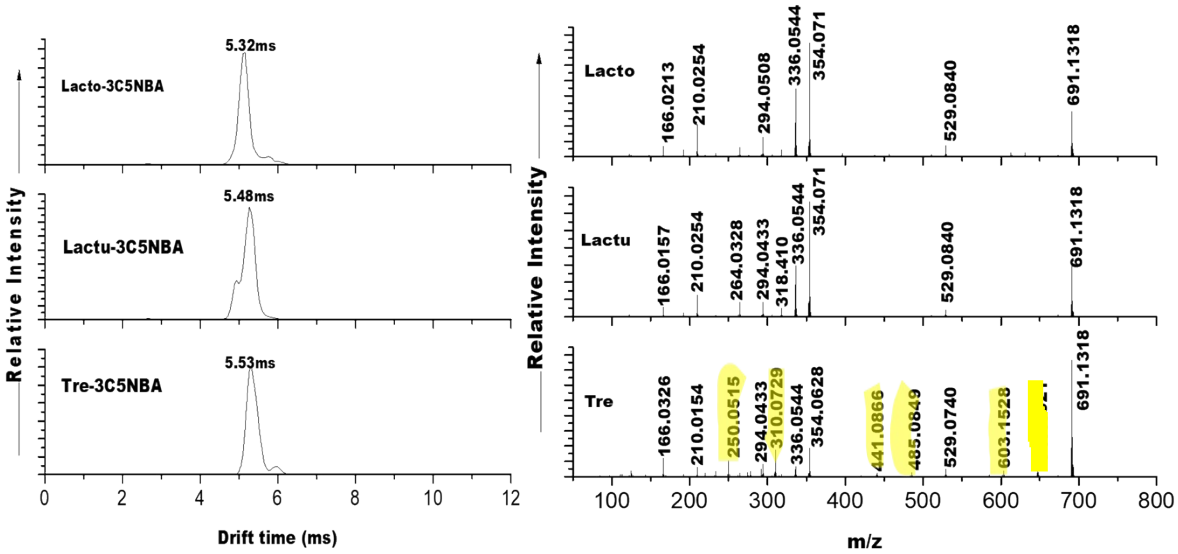
Figure S3. Fragmentation spectra of mobility-resolved 3C5NBA derivatives of each disaccharide at m/z 691.132 (collision energy=40 V), and monosaccharide at m/z 529.10. All characteristic fragments for each species are highlighted. The disaccharide neutral exact mass is 342.1162 Da, 3C5NBA is 211.0288 and the doubly-labelled neutral product is 692.1316. Abbreviations: maltose (Mal), melibiose (Mel), cellobiose (Cel), isomaltose (Isomal), lactose (Lacto), sucrose (Suc), trehalose (Tre).

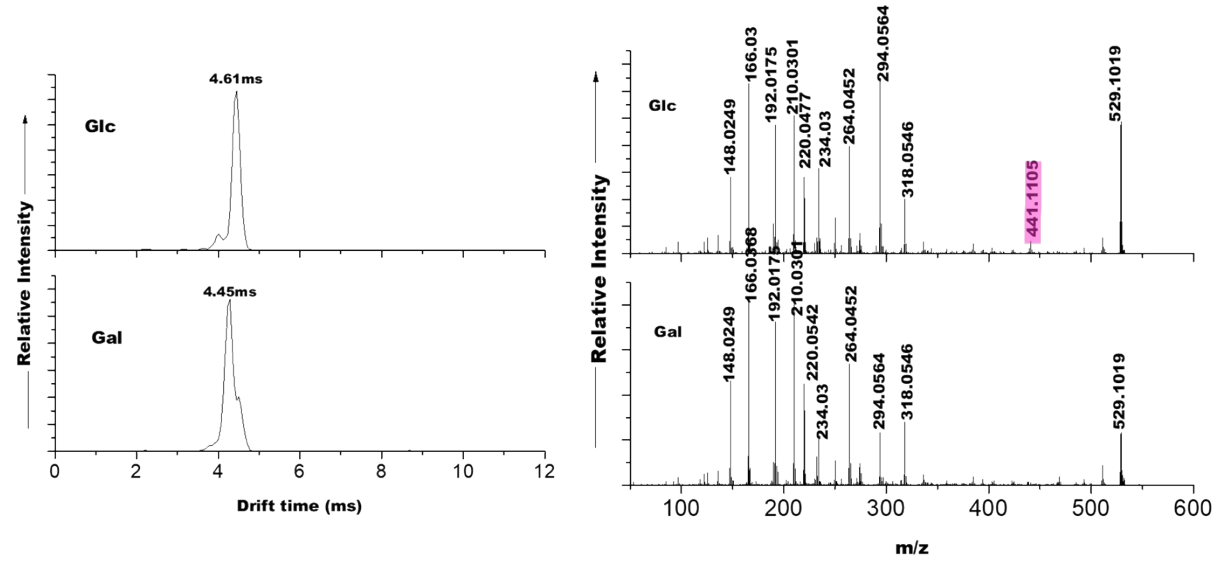





Suc-3C5NBA
Peak2
6.13ms
Peak2
5.64ms
Peak1
4.99ms
Relative Intensity
Drift Time (ms)
Peak1
264.032
354.0628
529.074
691.1318
Peak2
210.0191
264.0399
294.0433
336.0544
355.0710
529.084
691.1318
Peak3
210.0254
264.0328
294.0433
336.0544
354.0628
441.0958
529.074
691.1318
Relative Intensity
m/z


Isomal-3C5NBA
Peak 1
5.64ms
Peak 2
6.24ms
Mel-3C5NBA
5.91ms
Relative Intensity
Drift time (ms)
Isomal
Peak 1
166.0326
192.013
210.0254
234.0251
264.0328
294.0433
318.0488
354.071
396.0721
456.1013
529.074
631.0972
655.0966
691.1318
Isomal
Peak 2
166.0326
192.013
210.0254
234.0251
264.0399
294.0508
318.0488
354.0628
396.0721
456.1019
529.064
571.0848
631.0972
655.0966
691.1318
Mel
166.0326
192.013
210.0254
234.0251
264.0399
294.0508
318.0488
336.0624
354.071
396.0808
426.0889
456.1013
480.1078
529.084
631.1191
655.1189
691.1318
Relative Intensity
m/z


5.32ms
Lacto-3C5NBA
5.48ms
Lactu-3C5NBA
5.53ms
Tre-3C5NBA
Relative Intensity
0
2
4
6
8
10
12
Drift time (ms)
Lacto
166.0213
210.0254
294.0508
336.0544
354.071
529.0840
691.1318
Lactu
166.0157
210.0254
264.0328
294.0433
318.410
336.0544
354.071
529.0840
691.1318
Tre
166.0326
210.0154
250.0515
294.0433
310.0729
336.0544
354.0628
441.0866
485.0849
529.0740
603.1528
691.1318
Relative Intensity
100
200
300
400
500
600
700
800
m/z


4.61ms
Glc
4.45ms
Gal
Relative Intensity
0
2
4
6
8
10
12
Drift time (ms)
Glc
148.0249
166.03
192.0175
210.0301
220.0477
234.03
264.0452
294.0564
318.0546
441.1105
529.1019
Gal
148.0249
166.0368
192.0175
210.0301
220.0542
234.03
264.0452
294.0564
318.0546
529.1019
Relative Intensity
100
200
300
400
500
600
m/z

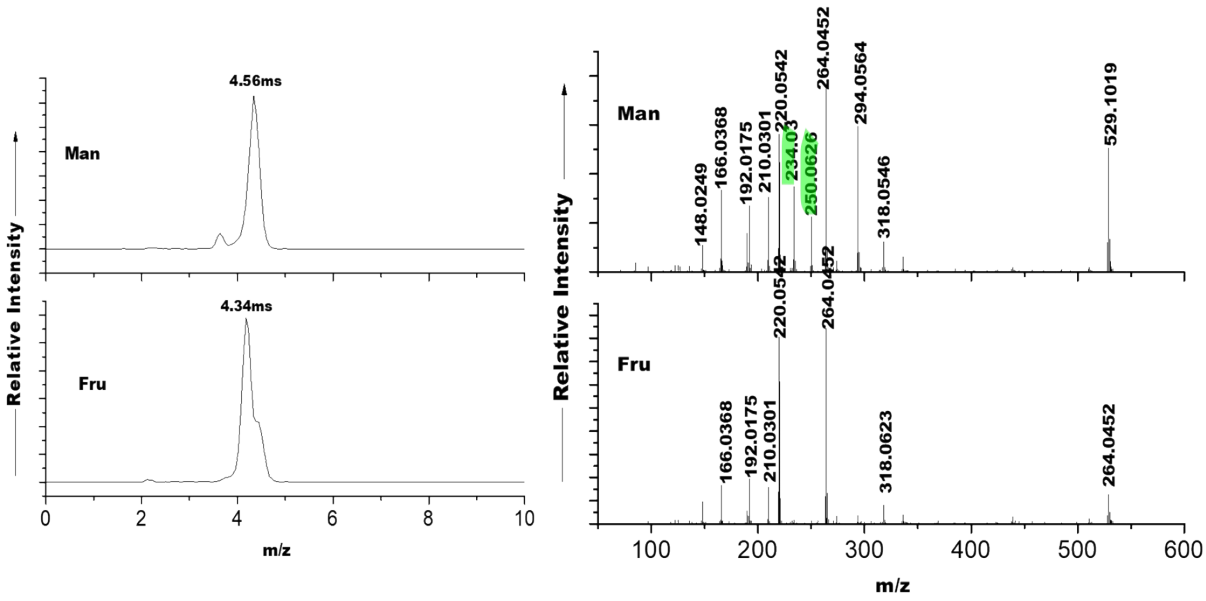

4.56ms
Man
4.34ms
Fru
Relative Intensity
0
2
4
6
8
10
m/z
Man
148.0249
166.0368
192.0175
210.0301
220.0542
234.03
250.0626
264.0452
294.0564
318.0546
529.1019
Fru
166.0368
192.0175
210.0301
220.0542
264.0452
318.0623
264.0452
Relative Intensity
100
200
300
400
500
600
m/z

Figure S4. Energy-resolved ion activation experiments of $[M+2x3C5NBA)-H]^-$ species. The graph on the left shows monosaccharide fragmentation at m/z 529.10, and the one on the right disaccharide fragmentation at m/z 691.132. Fragment ion yield= Total fragment ion intensity/ (Total fragment ion intensity+ Precursor ion intensity).