Electronic Supplementary Material (ESI) for Analyst.

Supporting Information

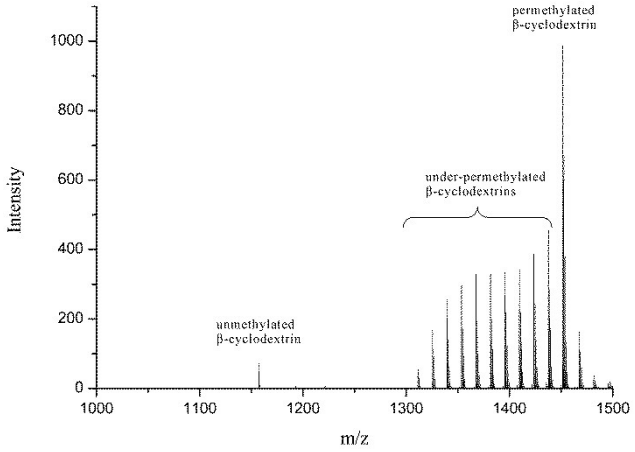

Figure S1. Permethylation of β-cyclodextrins in open-tubular capillary reactor with flow rate of 2.0 μL/min

Reactor: 500 μmi.d., 32 cm length

Table S1. Permethylation rates of β-cyclodextrins under different flow rates

flow rate / μL·min^{-1}	Permethylation rate				SD
	1	2	3	avg	
0.5	0.8777	0.7553	0.7238	0.7856	0.081
1.0	0.8234	0.8464	0.7453	0.8050	0.053
1.5	0.4317	0.4569	0.4916	0.4600	0.030
2.0	0.2826	0.2583	0.2187	0.2532	0.032
3.0	0.0351	0.0531	0.0633	0.0505	0.014

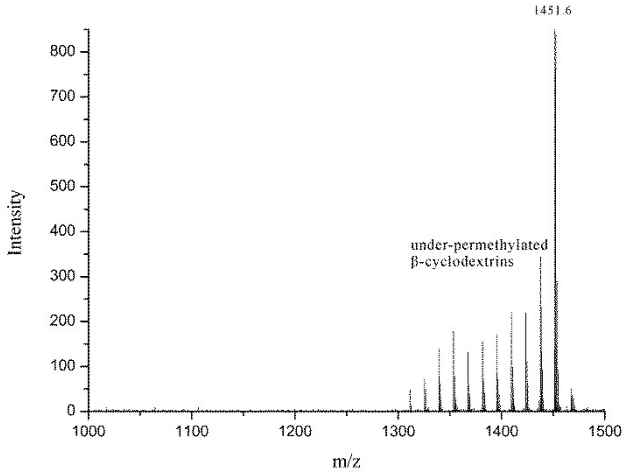

Figure S2. Permethylation of β-cyclodextrins in open-tubular capillary reactor with capillary length of 20 cm

Reactor: 500 μm,; Flow rate: 1.0 μL/min

Table S2. Permethylation rates of β-cyclodextrins in reactors with different capillary lengths

length / cm	Permethylation rate				SD
	1	2	3	avg	
20	0.4374	0.3916	0.3398	0.3896	0.049
24	0.5938	0.5327	0.6188	0.5817	0.044
28	0.6834	0.6124	0.6349	0.6435	0.036
32	0.8234	0.8464	0.7453	0.8050	0.053
35	0.8620	0.8248	0.7894	0.8254	0.036

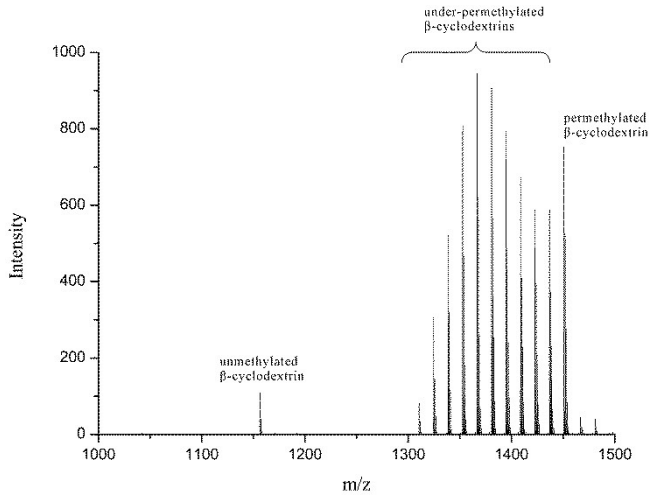

Figure S3. Permethylation of β-cyclodextrins in open-tubular capillary reactor with 320 μm i.d. capillary

Reactor: 32 cm length; Flow rate: 1.0 μL/min

Table S3. Permethylation rates of β-cyclodextrins in reactors with different capillary inner diameters

i.d. / μm	Permethylation rate				SD
	1	2	3	avg	
250	0	0	0	0	0
320	0.1519	0.1176	0.1324	0.1340	0.017
450	0.6201	0.5903	0.6724	0.6276	0.042
500	0.8234	0.8464	0.7453	0.8050	0.053

Table S4. Reusability of the open-tubular capillary reactor

Reusing times	Permethylation rate				SD
	1	2	3	avg	
1	0.7976	0.8534	0.7837	0.8116	0.037
2	0.7778	0.7748	0.8383	0.7970	0.036
3	0.7859	0.7649	0.8485	0.7998	0.043
4	0.8483	0.7516	0.8134	0.8044	0.049

5	0.7353	0.7973	0.8232	0.7853	0.045
6	0.7684	0.7271	0.7934	0.7630	0.033
7	0.6447	0.7219	0.6778	0.6815	0.039
8	0.5857	0.5740	0.5441	0.5679	0.021
9	0.3922	0.3975	0.4440	0.4112	0.028
10	0.2853	0.2520	0.2611	0.2661	0.017

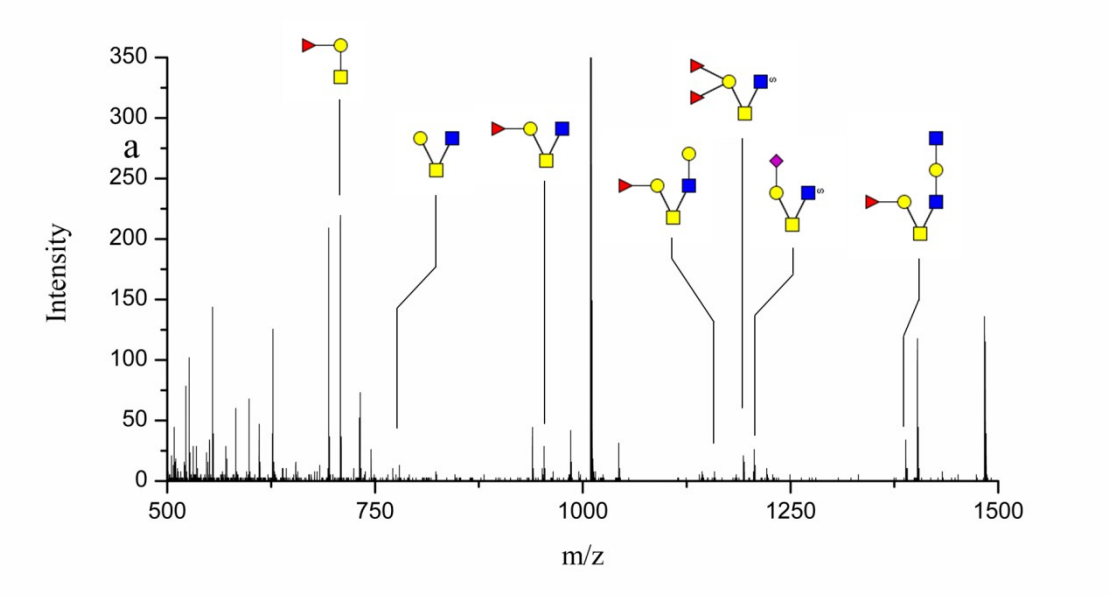

Figure S4. Permethylation of O-glycans from 2.5 μg mucin through open-tubular capillary reactor

Table S5. Data of the assignments showed in Figure 7a

m/z	Signal over noise ratio	Relative Intensity	Accuracy PPM
641.2542	14.88	0.81	3.1126
708.3878	433.34	20.27	-14.2700
738.4007	10.89	0.58	-16.8527
779.4276	22.66	1.20	-16.4158
953.5232	66.76	3.00	-20.1815
1157.6372	20.66	0.83	-28.8626
1193.4287	96.10	3.41	88.5578
1206.4613	55.46	1.93	65.1724
1228.6716	6.93	0.31	-24.9852
1402.7510	322.91	8.54	-14.8919
1473.7816	11.40	0.30	-9.7544

Table S6. Data of the assignments showed in Figure 7b

m/z	Signal over noise ratio	Relative Intensity	Accuracy PPM
708.3489	87.39	80.96	-10.7210
779.3832	16.55	14.68	40.5514
953.4865	41.85	25.96	18.3684

1157.6044	23.78	12.29	-0.5290
1193.6898 (discarded)	17.85	10.28	-130.1574
1206.8125 (discarded)	6.39	3.67	-234.3890
1228.6502	27.98	14.89	-7.5681
1331.6853	51.13	28.19	5.7796
1402.7321	93.99	43.67	-1.4184
1473.7528	42.25	18.40	9.7874

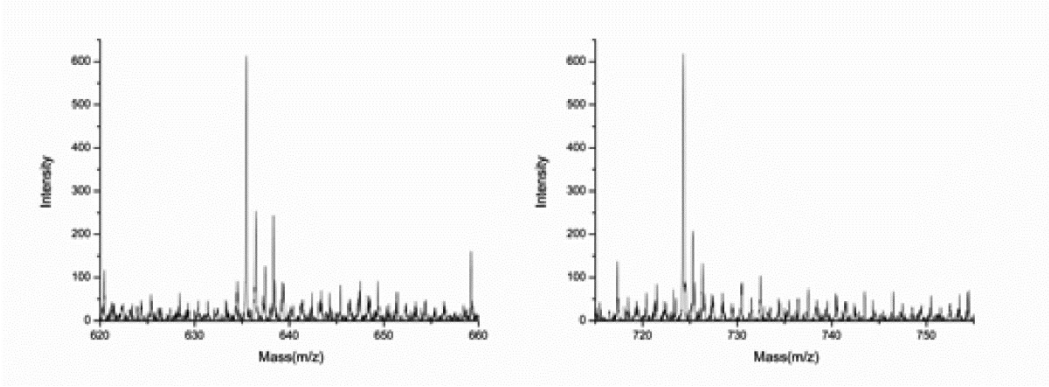

Figure S5. Zoomed-in spectras of Figure 7b