

Supplementary Part

3D Sub-Nanometer Analysis of Glucose in an Aqueous Solution by Cryo-Atom Probe Tomography

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Table A 1: Possible combinations of different C_xH_y , $C_xO_yH_z$ and H_xO_y molecules in a supersaturated glucose solution considering only the single charged state. If double-charged molecules were taken into account, the combination possibilities would increase enormously.

m/q	molecules		m/q	molecules	
1	H		96	C6OH8	C5O2H4 C4O3
2	H2		97	C6OH9	C5O2H5 C4O3H
3	H3		98	C6OH10	C5O2H6 C4O3H2
12	C		99	C6OH11	C5O2H7 C4O3H3
15	CH3		100	C6OH12	C5O2H8 C4O3H4
16	O		102		C5O2H10 C4O3H6
17	HO		103		C5O2H11 C4O3H7
18	H2O		104	C6O2	C5O2H12 C4O3H8
19	H3O		105	C6O2H	C5O2H13 C4O3H9
20	H4O		106	C6O2H2	C4O3H10
21	H5O		107	C6O2H3	C4O3H11
27	C2H3		108	C6O2H4	C5O3
28	C2H4	CO	109	C6O2H5	C5O3H
29	C2H5	COH	110	C6O2H6	C5O3H2
30	C2H6	COH2	111	C6O2H7	C5O3H3
31	C2H7	COH3	112	C6O2H8	C5O3H4
32	O2	COH4	113	C6O2H9	C4O4H C5O3H5
33	O2H		114	C6O2H10	C4O4H2 C5O3H6
34	(OH)2		115	C6O2H11	C4O4H3 C5O3H7
35	(OH)(OH2)		116	C6O2H12	C4O4H4 C5O3H8
36	C3		117	C6O2H13	C4O4H5 C5O3H9
37	(H2O)(H3O)	C3H	118		C4O4H6 C5O3H10
39	C3H3		119		C4O4H7 C5O3H11
40	C3H4	C2O	120	C6O3	C4O4H8 C5O3H12
41	C3H5	C2OH	121	C6O3H	C4O4H9 C5O3H13
42	C3H6	C2OH2	122	C6O3H2	C4O4H10
43	C3H7	C2OH3	123	C6O3H3	
44	C3H8	C2OH4 CO2	124	C6O3H4	C5O4
45	C3H9	C2OH5 CO2H	125	C6O3H5	C5O4H
46		C2OH6 CO2H2	126	C6O3H6	C5O4H2
47		C2OH7 CO2H3	127	C6O3H7	C5O4H3
48	C4	CO2H4	128	C6O3H8	C5O4H4
49	C4H	CO2H5	129	C6O3H9	C5O4H5
52	C4H4	C3O	130	C6O3H10	C5O4H6
54	C4H6	C3OH2	131	C6O3H11	C5O4H7
55	(H2O)2(H3O)	C4H7 C3OH3	132	C6O3H12	C5O4H8
56	C4H8	C3OH4 C2O2	133	C6O3H13	C5O4H9
57	C4H9	C3OH5 C2O2H	134		C5O4H10
58	C4H10	C3OH6 C2O2H2	135		C5O4H11
59	C4H11	C3OH7 C2O2H3	136	C6O4	C5O4H12

60	C5		C3OH8	C2O2H4		137	C6O4H	C5O4H13
61	C5H		C3OH9	C2O2H5		138	C6O4H2	C5O4H14
62	C5H2			C2O2H6		139	C6O4H3	
63	C5H3			C2O2H7		140	C6O4H4	C5O5
64	C5H4			C4O		141	C6O4H5	C5O5H
65	C5H5			C4OH		142	C6O4H6	C5O5H2
66	C5H6			C4OH2		143	C6O4H7	C5O5H3
67	C5H7			C4OH3		144	C6O4H8	C5O5H4
68	C5H8			C4OH4	C3O2	145	C6O4H9	C5O5H5
69	C5H9			C4OH5	C3O2H	147	C6O4H11	C5O5H7
70	C5H10			C4OH6	C3O2H2	148	C6O4H12	C5O5H8
71	C5H11			C4OH7	C3O2H3	149	C6O4H13	C5O5H9
72	C5H12	C6		C4OH8	C3O2H4	150	C6O4H14	C5O5H10
73	(H2O)3(H3O)	C6H	C5H13	C4OH9	C3O2H5	151		C5O5H11
77	C5OH	C6H5			C3O2H9	152	C6O5	C5O5H12
78	C5OH2	C6H6				153	C6O5H	C5O5H13
79	C5OH3	C6H7				157	C6O5H5	
80	C5OH4	C6H8	C4O2			163	C6O5H11	
81	C5OH5	C6H9	C4O2H			169	C6O6H	
82	C5OH6	C6H10	C4O2H2			171	C6O6H3	
83	C5OH7	C6H11	C4O2H3			175	C6O6H7	
84	C5OH8	C6H12	C4O2H4			181	C6O6H13	
85	C5OH9	C6H13	C4O2H5			187	C5O5H10+H2O+H3O	
86	C5OH10	C6H14	C4O2H6			189	C5O5H12+H2O+H3O	
87	C5OH11	C6H15	C4O2H7			193	C5O5H15+H3O+H3O	
91	(H2O)4(H3O)	C6OH3	C4O2H11			199	C6O6H12+H3O	
92		C6OH4		C5O2		205	C5O5H11+H2O+H2O+H2O	
93	C6OH5	C5O2H				207	C5O5H13+H2O+H2O+H2O	
94	C6OH6	C5O2H2				211	C5O5H14+H3O+H3O+H3O	
95	C6OH7	C5O2H3				217	C6O6H12+H2O+H3O	

Table A 2: Possible combinations of different C_xH_y , $C_xO_yH_z$ molecules for a bulk glucose specimen considering only the single charged state.

m/q	molecules			Approach 1	Approach 2
				C_xH_y	$C_xO_yH_z$
1	H			H	H
2	H ₂			H ₂	H ₂
3	H ₃			H ₃	H ₃
12	C			C	C
13	CH			CH	CH
14	CH ₂			CH ₂	CH ₂
15	CH ₃			CH ₃	CH ₃
16	O			O	O
17	OH			OH	OH
18	OH ₂			OH ₂	OH ₂
19	OH ₃			OH ₃	OH ₃
20	OH ₄			OH ₄	OH ₄
21	OH ₅			OH ₅	OH ₅
26	C ₂ H ₂			C ₂ H ₂	C ₂ H ₂
27	C ₂ H ₃			C ₂ H ₃	C ₂ H ₃
28	C ₂ H ₄	CO		C ₂ H ₄	CO
29	C ₂ H ₅	COH		C ₂ H ₅	COH
30	C ₂ H ₆	COH ₂		C ₂ H ₆	COH ₂
31	C ₂ H ₇	COH ₃		C ₂ H ₇	COH ₃
32	O ₂	COH ₄		O ₂	O ₂
36	C ₃			C ₃	C ₃
37	C ₃ H			C ₃ H	C ₃ H
38	C ₃ H ₂			C ₃ H ₂	C ₃ H ₂
39	C ₃ H ₃			C ₃ H ₃	C ₃ H ₃
40	C ₃ H ₄	C ₂ O		C ₃ H ₄	C ₂ O
41	C ₃ H ₅	C ₂ OH		C ₃ H ₅	C ₂ OH
42	C ₃ H ₆	C ₂ OH ₂		C ₃ H ₆	C ₂ OH ₂
43	C ₃ H ₇	C ₂ OH ₃		C ₃ H ₇	C ₂ OH ₃
44	C ₃ H ₈	C ₂ OH ₄	CO ₂	C ₃ H ₈	CO ₂
45		C ₂ OH ₅	CO ₂ H	C ₂ OH ₅	CO ₂ H
46		C ₂ OH ₆	CO ₂ H ₂	C ₂ OH ₆	CO ₂ H ₂
47		C ₂ OH ₇	CO ₂ H ₃	C ₂ OH ₇	CO ₂ H ₃
48	C ₄		CO ₂ H ₄	C ₄	CO ₂ H ₄
49	C ₄ H		CO ₂ H ₅	C ₄ H	CO ₂ H ₅
50	C ₄ H ₂			C ₄ H ₂	C ₄ H ₂
51	C ₄ H ₃			C ₄ H ₃	C ₄ H ₃
52	C ₄ H ₄	C ₃ O		C ₄ H ₄	C ₃ O
53	C ₄ H ₅	C ₃ OH		C ₄ H ₅	C ₃ OH
54	C ₄ H ₆	C ₃ OH ₂		C ₄ H ₆	C ₃ OH ₂
55	C ₄ H ₇	C ₃ OH ₃		C ₄ H ₇	C ₃ OH ₃
56	C ₄ H ₈	C ₃ OH ₄	C ₂ O ₂	C ₄ H ₈	C ₂ O ₂
57	C ₄ H ₉	C ₃ OH ₅	C ₂ O ₂ H	C ₄ H ₉	C ₂ O ₂ H

58	C4H10	C3OH6	C2O2H2		C4H10	C2O2H2
59		C3OH7	C2O2H3		C3OH7	C2O2H3
60	C5	C3OH8	C2O2H4		C5	C2O2H4
61	C5H	C3OH9	C2O2H5		C5H	C2O2H5
62	C5H2		C2O2H6		C5H2	C2O2H6
63	C5H3		C2O2H7		C5H3	C2O2H7
64	C5H4		C4O		C5H4	C4O
65	C5H5		C4OH		C5H5	C4OH
66	C5H6		C4OH2		C5H6	C4OH2
67	C5H7		C4OH3		C5H7	C4OH3
68	C5H8		C4OH4	C3O2	C5H8	C3O2
69	C5H9		C4OH5	C3O2H	C5H9	C3O2H
70	C5H10		C4OH6	C3O2H2	C5H10	C3O2H2
71	C5H11		C4OH7	C3O2H3	C5H11	C3O2H3
72	C5H12	C6	C4OH8	C3O2H4	C6	C3O2H4
73		C6H	C4OH9	C3O2H5	C6H	C3O2H5
74		C6H2		C3O2H9	C6H2	C3O2H9
75		C6H3			C6H3	C6H3

Table A 3: For the first approach only C_xH_y molecules are considered. In the second approach C_xH_y molecules and common protonated water cluster with the formula $(H_2O)_nH^+$ with $n = 1 - 5$ were assumed.

Approach 1				Approach 2			
m/q	molecules	m/q	molecules	m/q	molecules	m/q	molecules
1	H	96	C6OH8	1	H	96	C6OH8
2	H2	97	C6OH9	2	H2	97	C6OH9
3	H3	98	C6OH10	3	H3	98	C6OH10
12	C	99	C6OH11	12	C	99	C6OH11
15	CH3	100	C6OH12	15	CH3	100	C6OH12
16	O	102	C6OH14	16	O	102	C6OH14
17	HO	103	C5O2H11	17	HO	103	C5O2H11
18	H2O	104	C6O2	18	H2O	104	C6O2
19	H3O	105	C6O2H	19	H3O	105	C6O2H
20	H4O	106	C6O2H2	20	H4O	106	C6O2H2
21	H5O	107	C6O2H3	21	H5O	107	C6O2H3
27	C2H3	108	C6O2H4	27	C2H3	108	C6O2H4
28	C2H4	109	C6O2H5	28	C2H4	109	C6O2H5
29	C2H5	110	C6O2H6	29	C2H5	110	C6O2H6
30	C2H6	111	C6O2H7	30	C2H6	111	C6O2H7
31	C2H7	112	C6O2H8	31	C2H7	112	C6O2H8
32	O2	113	C6O2H9	32	O2	113	C6O2H9
33	O2H	114	C6O2H10	33	O2H	114	C6O2H10
34	(OH)2	115	C6O2H11	34	(OH)2	115	C6O2H11
35	(OH)(OH2)	116	C6O2H12	35	(OH)(OH2)	116	C6O2H12
36	C3	117	C6O2H13	36	C3	117	C6O2H13
37	C3H	118	C6O2H14	37	(H2O)(H3O)	118	C6O2H14
39	C3H3	119	C5O3H11	39	C3H3	119	C5O3H11
40	C3H4	120	C6O3	40	C3H4	120	C6O3
41	C3H5	121	C6O3H	41	C3H5	121	C6O3H
42	C3H6	122	C6O3H2	42	C3H6	122	C6O3H2
43	C3H7	123	C6O3H3	43	C3H7	123	C6O3H3
44	C3H8	124	C6O3H4	44	C3H8	124	C6O3H4
45	C3H9	125	C6O3H5	45	C3H9	125	C6O3H5
46	C2OH6	126	C6O3H6	46	C2OH6	126	C6O3H6
47	C2OH7	127	C6O3H7	47	C2OH7	127	C6O3H7
48	C4	128	C6O3H8	48	C4	128	C6O3H8
49	C4H	129	C6O3H9	49	C4H	129	C6O3H9
52	C4H4	130	C6O3H10	52	C4H4	130	C6O3H10
54	C4H6	131	C6O3H11	54	C4H6	131	C6O3H11
55	C4H7	132	C6O3H12	55	(H2O)2(H3O)	132	C6O3H12
56	C4H8	133	C6O3H13	56	C4H8	133	C6O3H13
57	C4H9	134	C5O4H10	57	C4H9	134	C5O4H10
58	C4H10	135	C5O4H11	58	C4H10	135	C5O4H11

59	C4H11	136	C6O4	59	C4H11	136	C6O4
60	C5	137	C6O4H	60	C5	137	C6O4H
61	C5H	138	C6O4H2	61	C5H	138	C6O4H2
62	C5H2	139	C6O4H3	62	C5H2	139	C6O4H3
63	C5H3	140	C6O4H4	63	C5H3	140	C6O4H4
64	C5H4	141	C6O4H5	64	C5H4	141	C6O4H5
65	C5H5	142	C6O4H6	65	C5H5	142	C6O4H6
66	C5H6	143	C6O4H7	66	C5H6	143	C6O4H7
67	C5H7	144	C6O4H8	67	C5H7	144	C6O4H8
68	C5H8	145	C6O4H9	68	C5H8	145	C6O4H9
69	C5H9	147	C6O4H11	69	C5H9	147	C6O4H11
70	C5H10	148	C6O4H12	70	C5H10	148	C6O4H12
71	C5H11	149	C6O4H13	71	C5H11	149	C6O4H13
72	C5H12	150	C6O4H14	72	C5H12	150	C6O4H14
73	C5H13	151	C5O5H11	73	(H2O)3(H3O)	151	C5O5H11
77	C6H5	152	C6O5	77	C6H5	152	C6O5
78	C6H6	153	C6O5H	78	C6H6	153	C6O5H
79	C6H7	157	C6O5H5	79	C6H7	157	C6O5H5
80	C6H8	163	C6O5H11	80	C6H8	163	C6O5H11
81	C6H9	169	C6O6H	81	C6H9	169	C6O6H
82	C6H10	171	C6O6H3	82	C6H10	171	C6O6H3
83	C6H11	175	C6O6H7	83	C6H11	175	C6O6H7
84	C6H12	181	C6O6H13	84	C6H12	181	C6O6H13
85	C6H13	187	C5O5H10+H2O+H3O	85	C6H13	187	C5O5H10+H2O+H3O
86	C6H14	189	C5O5H12+H2O+H3O	86	C6H14	189	C5O5H12+H2O+H3O
87	C6H15	193	C5O5H15+H3O+H3O	87	C6H15	193	C5O5H15+H3O+H3O
91	C6OH3	199	C6O6H12+H3O	91	(H2O)4(H3O)	199	C6O6H12+H3O
92	C6OH4	205	C5O5H11+H2O+H2O+H2O	92	C6OH4	205	C5O5H11+H2O+H2O+H2O
93	C6OH5	207	C5O5H13+H2O+H2O+H2O	93	C6OH5	207	C5O5H13+H2O+H2O+H2O
94	C6OH6	211	C5O5H14+H3O+H3O+H3O	94	C6OH6	211	C5O5H14+H3O+H3O+H3O
95	C6OH7	217	C6O6H12+H2O+H3O	95	C6OH7	217	C6O6H12+H2O+H3O

Table A 4: In the third approach $C_xO_yH_z$ molecules and common protonated water cluster with the formula $(H_2O)_nH^+$ with $n = 1 - 5$ were assumed. In the fourth approach, the same molecules were assumed as in the third approach, in addition, the overlap with water peaks in the mass-to-charge state ratio of 27-31, 36 and 41-48 $u e^{-1}$ were calculated. The overlapping regions are marked in red.

Approach 3				Approach 4				
m/q	molecules	m/q	molecules	m/q	molecules	m/q	molecules	
1	H	96	C6OH8	1	H	96	C6OH8	
2	H2	97	C6OH9	2	H2	97	C6OH9	
3	H3	98	C6OH10	3	H3	98	C6OH10	
12	C	99	C6OH11	12	C	99	C6OH11	
15	CH3	100	C6OH12	15	CH3	100	C6OH12	
16	O	102	C4O3H6	16	O	102	C4O3H6	
17	HO	103	C4O3H7	17	HO	103	C4O3H7	
18	H2O	104	C6O2	18	H2O	104	C6O2	
19	H3O	105	C6O2H	19	H3O	105	C6O2H	
20	H4O	106	C6O2H2	20	H4O	106	C6O2H2	
21	H5O	107	C6O2H3	21	H5O	107	C6O2H3	
27	C2H3	108	C6O2H4	27	C2H3	(OH2)3	108	C6O2H4
28	CO	109	C6O2H5	28	CO	(OH2)(OH3)2	109	C6O2H5
29	COH	110	C6O2H6	29	COH	(OH)3(OH2)2	110	C6O2H6
30	COH2	111	C6O2H7	30	COH2	(OH2)5	111	C6O2H7
31	COH3	112	C6O2H8	31	COH3	(OH)(OH3)4	112	C6O2H8
32	O2	113	C6O2H9	32	O2		113	C6O2H9
33	O2H	114	C6O2H10	33	O2H		114	C6O2H10
34	(OH)2	115	C6O2H11	34	(OH)2		115	C6O2H11
35	(OH)(OH2)	116	C6O2H12	35	(OH)(OH2)		116	C6O2H12
36	C3	117	C6O2H13	36	C3	(OH2)2	117	C6O2H13
37	(H2O)(H3O)	118	C6O2H14	37	(H2O)(H3O)		118	C6O2H14
39	C3H3	119	C5O3H11	39	C3H3		119	C5O3H11
40	C2O	120	C6O3	40	C2O		120	C6O3
41	C2OH	121	C6O3H	41	C2OH	(OH)2O3	121	C6O3H
42	C2OH2	122	C6O3H2	42	C2OH2	(OH)4O	122	C6O3H2
43	C2OH3	123	C6O3H3	43	C2OH3	(OH)4(OH2)	123	C6O3H3
44	C02	124	C6O3H4	44	C02	(OH)2(OH2)3	124	C6O3H4
45	C02H	125	C6O3H5	45	C02H	(OH2)5	125	C6O3H5
46	C02H2	126	C6O3H6	46	C02H2	(OH2)3(OH3)	126	C6O3H6
47	C02H3	127	C6O3H7	47	C02H3	(OH2)(OH3)4	127	C6O3H7
48	C02H4	128	C6O3H8	48	C02H4	(OH3)5H	128	C6O3H8
49	C02H5	129	C6O3H9	49	C02H5		129	C6O3H9
52	C3O	130	C6O3H10	52	C3O		130	C6O3H10
54	C3OH2	131	C6O3H11	54	C3OH2		131	C6O3H11
55	(H2O)2(H3O)	132	C6O3H12	55	(H2O)2(H3O)		132	C6O3H12
56	C2O2	133	C6O3H13	56	C2O2		133	C6O3H13
57	C2O2H	134	C5O4H10	57	C2O2H		134	C5O4H10

58	C2O2H2	135	C5O4H11	58	C2O2H2	135	C5O4H11
59	C2O2H3	136	C6O4	59	C2O2H3	136	C6O4
60	C2O2H4	137	C6O4H	60	C2O2H4	137	C6O4H
61	C2O2H5	138	C6O4H2	61	C2O2H5	138	C6O4H2
62	C2O2H6	139	C6O4H3	62	C2O2H6	139	C6O4H3
63	C2O2H7	140	C6O4H4	63	C2O2H7	140	C6O4H4
64	C4O	141	C6O4H5	64	C4O	141	C6O4H5
65	C4OH	142	C6O4H6	65	C4OH	142	C6O4H6
66	C4OH2	143	C6O4H7	66	C4OH2	143	C6O4H7
67	C4OH3	144	C6O5H8	67	C4OH3	144	C6O5H8
68	C3O2	145	C6O4H9	68	C3O2	145	C6O4H9
69	C3O2H	147	C6O4H11	69	C3O2H	147	C6O4H11
70	C3O2H2	148	C6O4H12	70	C3O2H2	148	C6O4H12
71	C3O2H3	149	C6O4H13	71	C3O2H3	149	C6O4H13
72	C3O2H4	150	C6O4H14	72	C3O2H4	150	C6O4H14
73	(H2O)3(H3O)	151	C5O5H11	73	(H2O)3(H3O)	151	C5O5H11
77	C3O2H9	152	C6O5	77	C3O2H9	152	C6O5
78	C5OH2	153	C6O5H	78	C5OH2	153	C6O5H
79	C5OH3	157	C6O5H5	79	C5OH3	157	C6O5H5
80	C4O2	163	C6O5H11	80	C4O2	163	C6O5H11
81	C4O2H	169	C6O6H	81	C4O2H	169	C6O6H
82	C4O2H2	171	C6O6H3	82	C4O2H2	171	C6O6H3
83	C4O2H3	175	C6O6H7	83	C4O2H3	175	C6O6H7
84	C4O2H4	181	C6O6H13	84	C4O2H4	181	C6O6H13
85	C4O2H5	187	C5O5H10+H2O+H3O	85	C4O2H5	187	C5O5H10+H2O+H3O
86	C4O2H6	189	C5O5H12+H2O+H3O	86	C4O2H6	189	C5O5H12+H2O+H3O
87	C4O2H7	193	C5O5H15+H3O+H3O	87	C4O2H7	193	C5O5H15+H3O+H3O
91	(H2O)4(H3O)	199	C6O6H12+H3O	91	(H2O)4(H3O)	199	C6O6H12+H3O
92	C6OH4	205	C5O5H11+H2O+H2O+H2O	92	C6OH4	205	C5O5H11+H2O+H2O+H2O
93	C6OH5	207	C5O5H13+H2O+H2O+H2O	93	C6OH5	207	C5O5H13+H2O+H2O+H2O
94	C6OH6	211	C5O5H14+H3O+H3O+H3O	94	C6OH6	211	C5O5H14+H3O+H3O+H3O
95	C6OH7	217	C6O6H12+H2O+H3O	95	C6OH7	217	C6O6H12+H2O+H3O

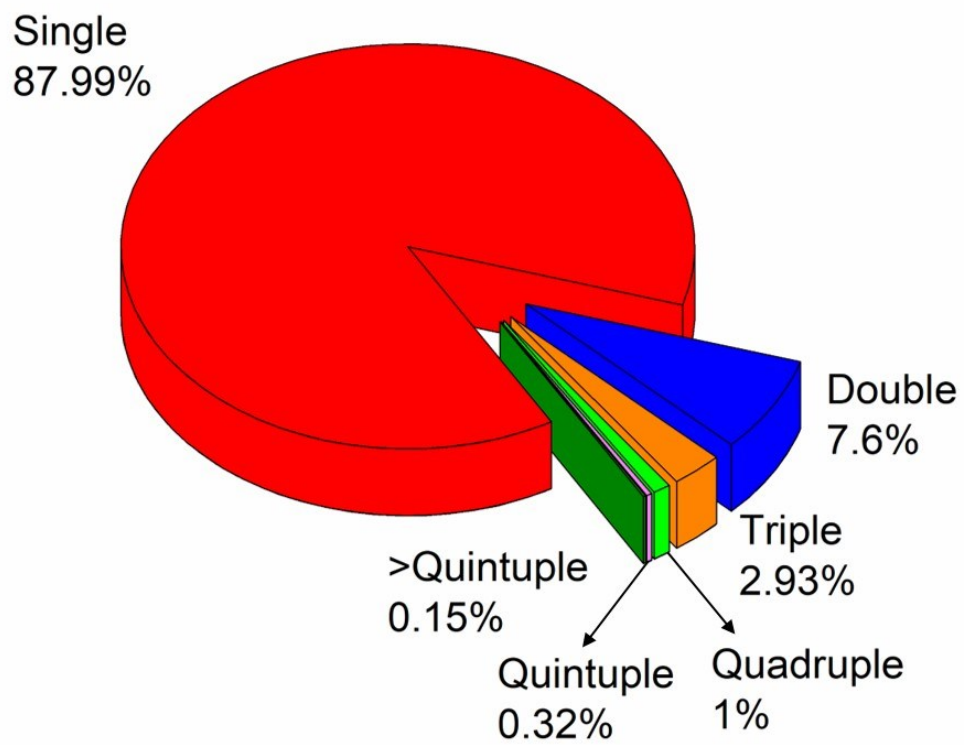


Figure A 1: The number of multi-hit events of the measurement in percentage.

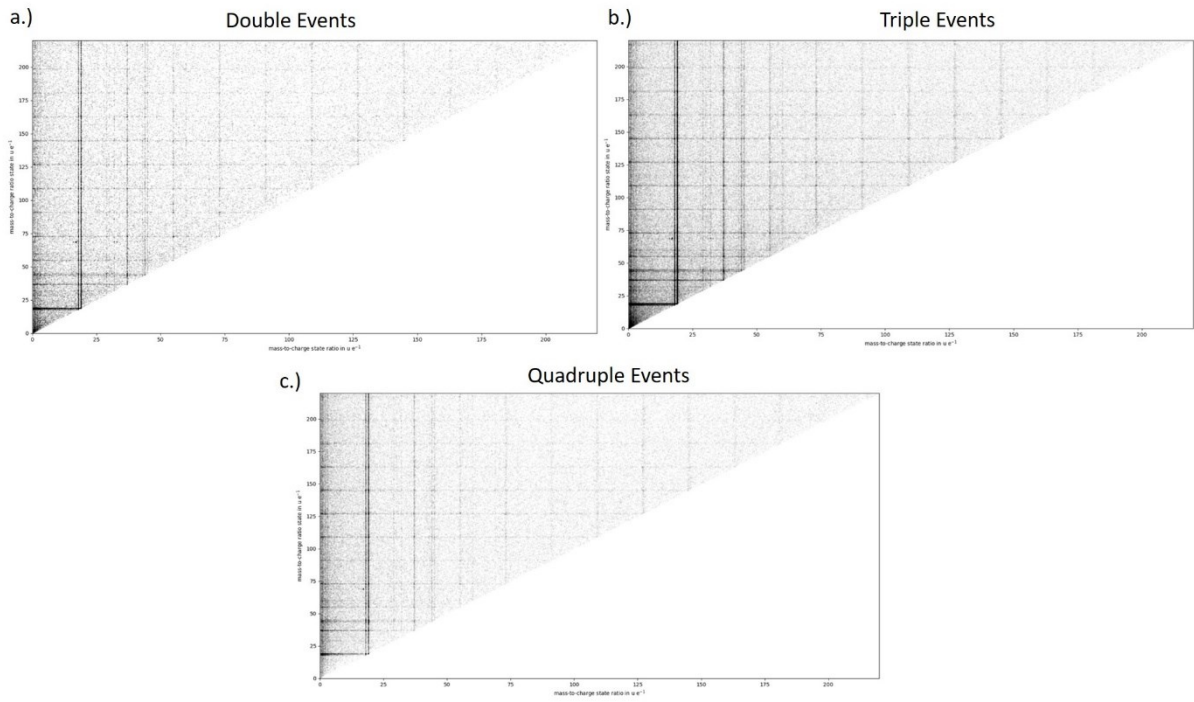


Figure A 2: Correlation histogram for a supersaturated glucose solution, the measured mass-to-charge state ratio m_2/q_2 of the second event is plotted versus the measured mass-to-charge state ratio m_1/q_1 of the first event. In b.) the correlation histogram of triple events and in c.) for quadruple events are shown.

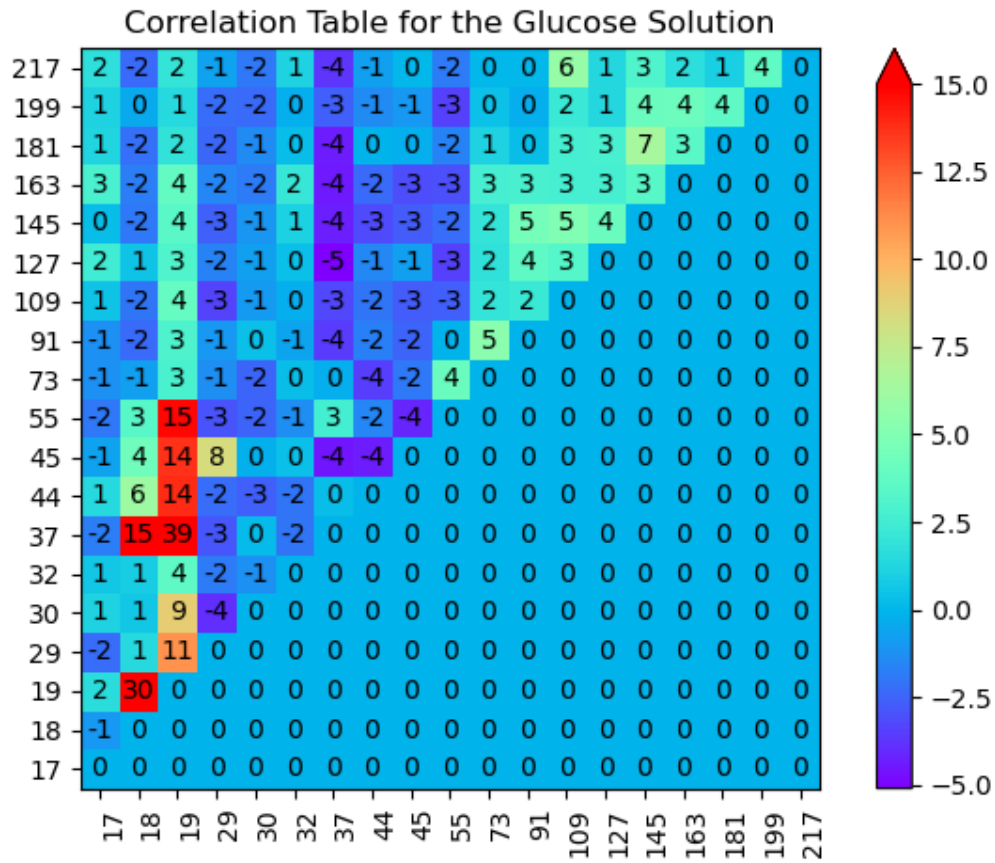


Figure A 3: Correlation table $d_{(ij)}$ according to (32) for ion-pairs in the evaporation of glucose solution. The values represent the excess (or deficit) of the number of the co-evaporation events, compared to the uncorrelated co-evaporation of double events.

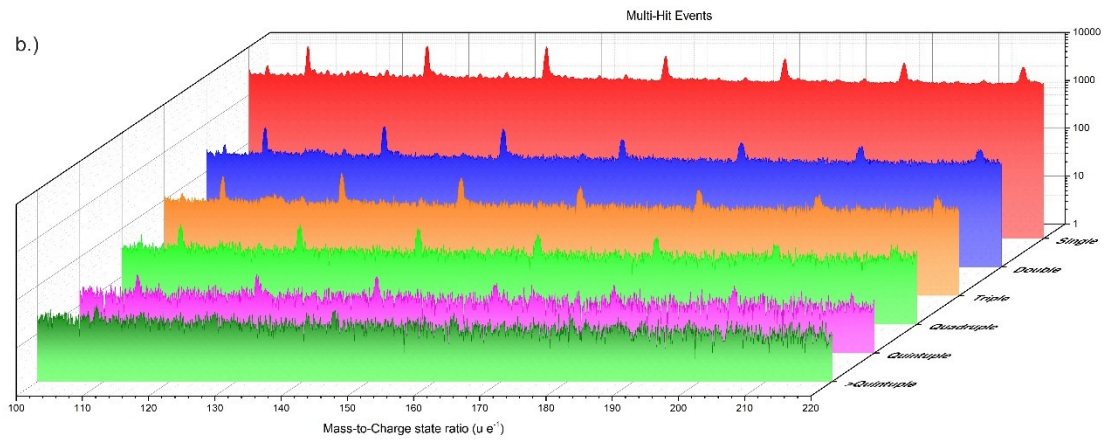
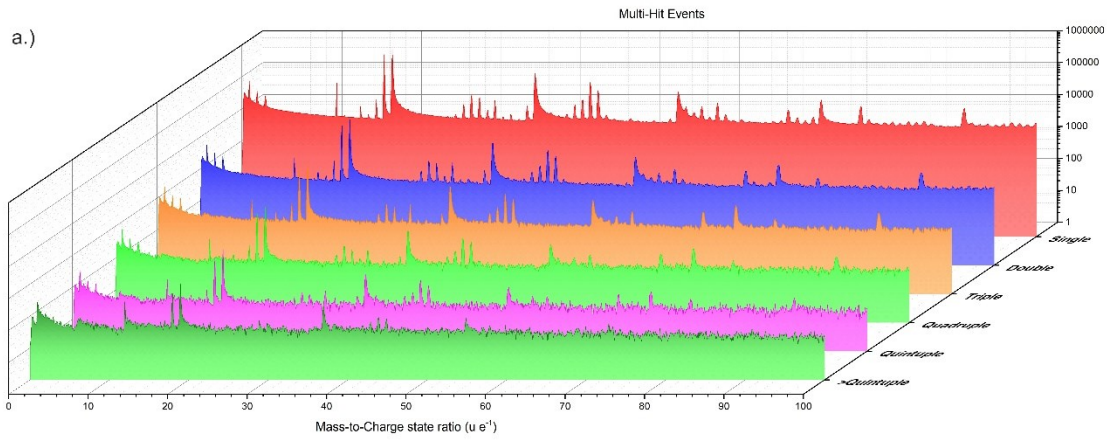


Figure A 4: Mass spectrum split into single, double, triple, quadruple, quintuple and >quintuple events in a.) from a mass-to-charge state ratio of 0-100 $u e^{-1}$ and in b.) from 100-220 $u e^{-1}$.

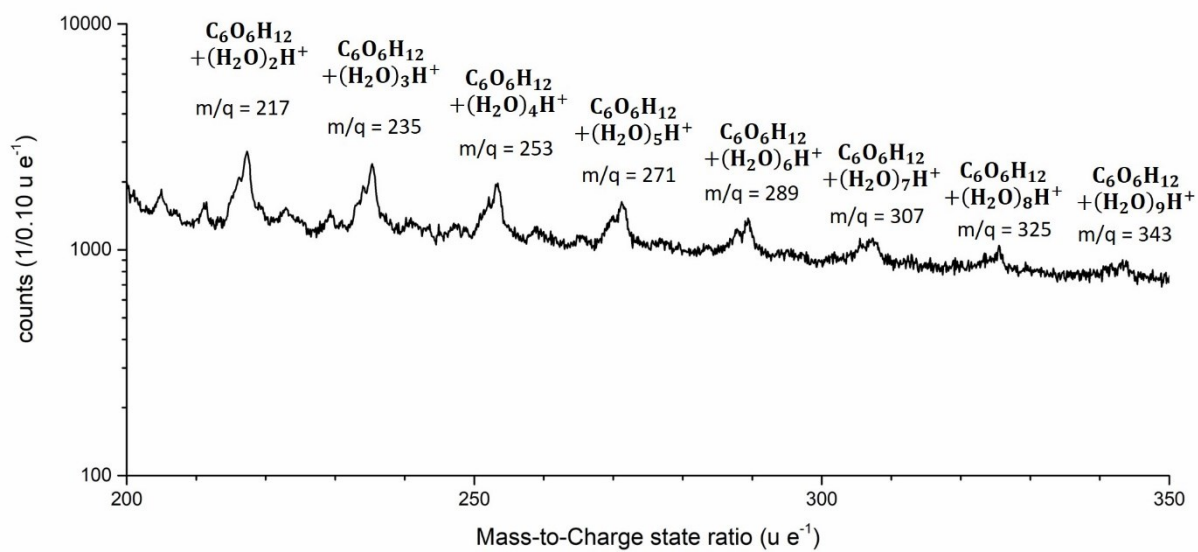


Figure A 5: Mass spectrum of a supersaturated glucose solution in a larger mass-to-charge state ratio from 200-350 u e⁻¹ in a logarithmic plot. Larger glucose+water cluster with the formula C₆O₆H₁₂+(H₂O)_nH⁺ with n = 2 – 9 are visible.