Electronic Supplementary Material (ESI) for ChemComm.

Does Deamidation of Islet Amyloid Polypeptide Accelerate Amyloid Fibril Formation?

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Electronic Supplementary Information

Experimental Procedure

Sample preparation for deamidated hIAPP and fibrils. Wild-type hIAPP lyophilized powder (Sigma Aldrich Company Ltd, Dorset, England) was dissolved in Milli-Q (Direct-Q® 3 UV System, Millipore Corporation, US) H_2O (~pH 7.5) at a concentration of 500 μM and further diluted into 10 uM solution. The 10 μM hIAPP aqueous solution was incubated for 28 days (4 weeks). The incubated solution was then centrifuged at 14,000 rpm for one hour to separate the soluble hIAPP (supernatant) from the insoluble hIAPP fibril (fibrillary pellet). The supernatant solution containing soluble hIAPP was then diluted 20-fold with 49.5:49.5:1 water/acetonitrile/formic acid prior to MS analysis. hIAPP fibrillary pellets from 7-, 14-, 21-, and 28-day solutions were rinsed with 100 μL Milli-Q H_2O three times and then re-dissolved with 20 μL of 47.5:47.5:5 water/acetonitrile/formic acid, and sonicated in water bath at 37 ^oC for one hour. Re-dissolved samples were further diluted with 80 μL of 50:50 water/acetonitrile. The final concentration of formic acid in solutions was 1% (mol/mol).

Sample preparation for seeding mutant peptides. Wild-type hIAPP lyophilized powder and synthetic mutant hIAPPs (Pepscan Company Ltd, The Netherlands) were dissolved in Milli-Q H_2O to a concentration of 500 μM. The seeding experiments were performed by mixing wild-type hIAPP stock solution with 5%, 10%, 25%, or 50% mutant ($(D)_3$hIAPP) or ($(isoD)_3$hIAPP) solutions. Samples were then diluted to a final concentration of 10 μM wild-type hIAPP plus mutant ($(D)_3$hIAPP) or ($(isoD)_3$hIAPP), which were incubated for 1 week at 37^oC. The supernatants and fibrillary pellets were separated and prepared as mentioned above.

FTICR MS analysis. Mass spectra were acquired on a 12 tesla solariX FTICR MS (Bruker Daltonik GmbH, Bremen, Germany). All samples were analyzed in positive ionization mode. For the detection of deamidated hIAPP and dissociated fibrils, an Apollo II electrospray ionization (ESI) source (Bruker Daltonik GmbH, Bremen, Germany) was used with a capillary voltage of 4-4.5 kV. The ESI flow rate was optimized to 100-150 µL/h and the source temperature was set to 200°C. Ions were externally accumulated in a hexapole collision cell before transferred to an infinity cell (ICR cell) for excitation and detection.[1] Data obtained from FTICR-MS were analyzed using Bruker DataAnalysis 4.2 software (Bruker Daltonics, Bremen, Germany). For the CAD experiments, precursor ions were first isolated using the quadrupole mass filter, then collided with argon gas and accumulated in the collision cell. The collision energy was optimized to 2-18 V and the ion accumulation time to 1-3 seconds. Fragments were then transferred to the infinity cell for detection. The most intense isotopic peak from each fragment with signal-to-noise ratio (S/N) over 5 was manually matched with the theoretical *m/z*. All of the fragments were internally calibrated and then assigned with an uncertainty less than 1 part-per-million (ppm).

Quantification of deamidated/ mutant hIAPP in solutions and fibrillary pellets. The monoisotopic peak area of non-deamidated and deamidated/mutant hIAPP peaks were measured using Bruker DataAnalysis 4.2 software. The percentage of deamidated hIAPP (%) was calculated as follows:

$$Deamidated\ (\%) = \frac{Peak\ area\ of\ deamidated\ hIAPP}{Sum\ of\ Peak\ area\ of\ deamidated\ hIAPP\ and\ nondeamidated\ hIAPP}\ x\ 100\%$$

The same calculation method was applied to obtain the percentage of mutant hIAPP against wild-type hIAPP.

Transmission electron microscopy (TEM). The TEM images of the incubated solutions, including 10 μM wild-type hIAPP, 10 μM mutant ($(D)_3$hIAPP), and 10 μM mutant ($(isoD)_3$hIAPP) solution were acquired on a Jeol 2010F TEM operated at 200 kV. 10 μL of incubated solution was transferred onto a carbon-coated grid and settled for one minute, followed by removing the excess solution using filter paper. A 2% (w/v) uranyl acetate solution was used for the negative stain. Multiple images with magnification from x10,000 to x40,000 were acquired.

Thioflavin T (ThT) fluorescence reactivity. The fluorescence reactivity of the 10 μM hIAPP, mutant ($(D)_3$hIAPP), mutant ($(isoD)_3$hIAPP), and hIAPP seeding solutions were measured using a GloMax®-Multi Detection System (Promega; Wisconsin, USA). All samples were placed in a black 96 well-plate and mixed with 10 μM ThT aqueous solution. Fluorescence spectra of the samples were acquired every 45 minutes with excitation at 405 nm and emission measurement at 490 nm, in a similar fashion to Chan *et al*.[2, 3] The intensities obtained from the fluorescence spectrometer were normalized to the signal intensity of the most mature fibril.

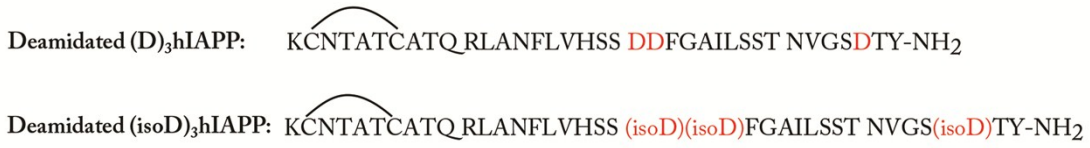

Figure S1. Sequences of synthetic mutant hIAPPs. Asn residues at position 21, 22, and 35 are replaced with aspartic acid – deamidated ($(D)_3$hIAPP) or iso-aspartic acid – deamidated ($(isoD)_3$hIAPP) in order to act as deamidation mimics.

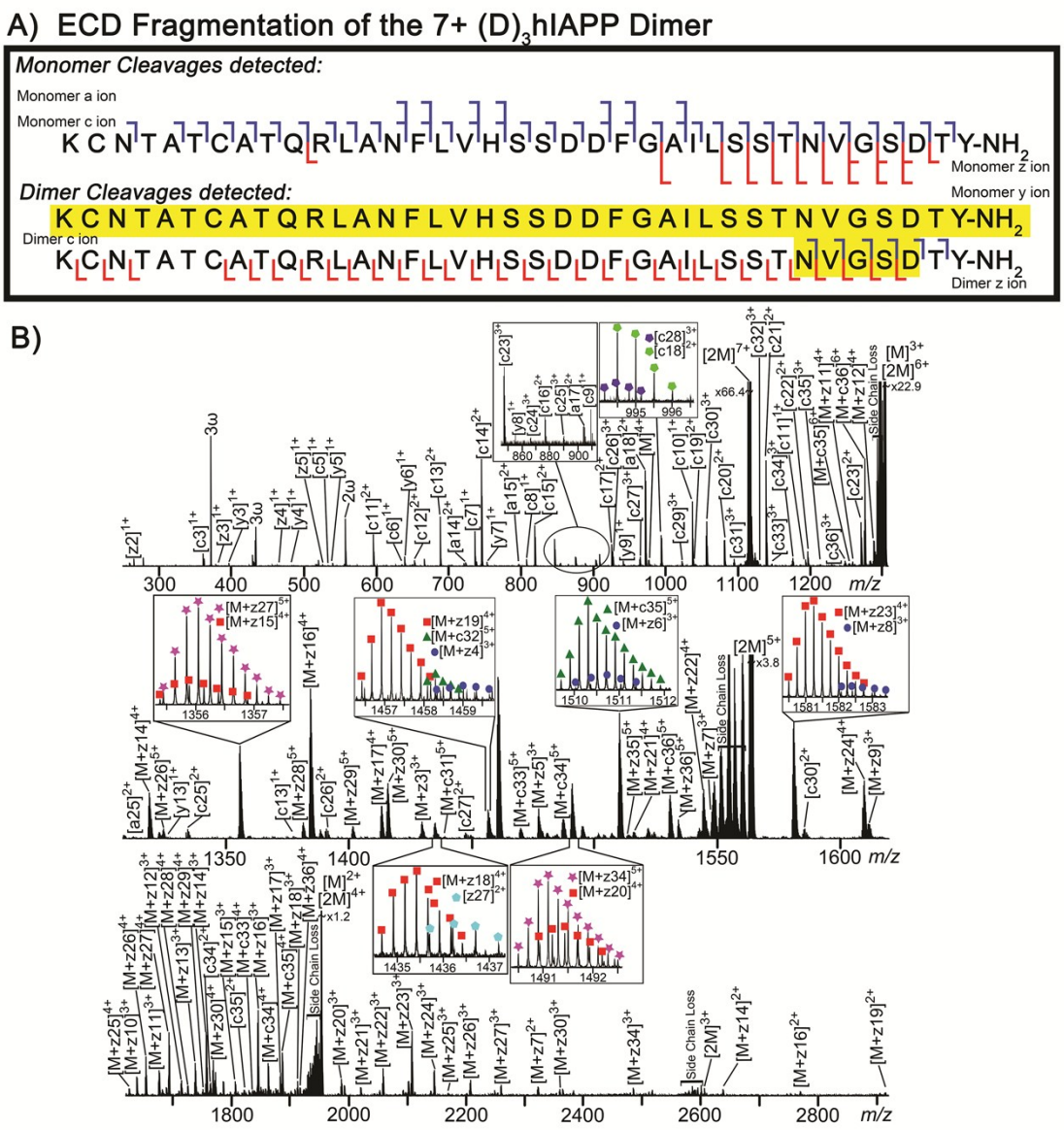

Figure S2. (A) Summary of a-, c-, y-, and z-ion fragments observed in the (B) ECD MS/MS spectrum of the 7+ charge state ($(D)_3$hIAPP) dimer. Highlighted sequence represents the proposed non-covalent interaction region. The side chain losses were not labeled for clarity. The assigned fragments are listed in the ESI Table S1.

Assignment Table of the ECD MS/MS of 7+ charge state ($(D)_3$hIAPP) dimer

Ion	charge	Theoretical m/z	Experimental m/z	Error (ppm)
MH	4+	977.462474	977.461786	-0.704
MH	3+	1302.279143	1302.279165	0.017
MH	2+	1952.915077	1952.915991	0.468
2[MH]	7+	1116.383162	1116.383047	-0.103
2[MH]	6+	1303.115465	1303.115560	0.073
2[MH]	5+	1563.738667	1563.738999	0.212
2[MH]	4+	1954.673471	1954.674185	0.365
2[MH]	3+	2605.897413	2605.897274	-0.053
a14	2+	724.354565	724.354485	-0.110
a15	2+	797.888772	797.888565	-0.259
a17	2+	903.965011	903.965096	0.094
a18	2+	972.494467	972.494518	0.052
a22	2+	1174.553438	1174.553758	0.272
a23	2+	1248.087645	1248.088012	0.294
a25	2+	1312.116934	1312.116940	0.005
c3	1+	362.173076	362.173008	-0.188
c5	1+	534.257869	534.257735	-0.251
c6	1+	636.313372	636.313252	-0.189
c7	1+	737.306908	737.306867	-0.056
c8	1+	808.344022	808.344002	-0.025
c9	1+	909.391701	909.391767	0.073
c10	1+	1037.450279	1037.450336	0.055
c11	2+	597.279333	597.279236	-0.162
c11	1+	1193.551390	1193.551711	0.269
c12	2+	653.821365	653.821279	-0.132
c13	2+	689.339922	689.339851	-0.103
c13	1+	1377.672568	1377.673273	0.512
c14	2+	746.361386	746.361348	-0.051
c15	2+	819.895593	819.895538	-0.067
c16	2+	876.437625	876.437608	-0.019
c17	2+	925.971832	925.971879	0.051
c18	2+	994.501288	994.501285	-0.003
c19	2+	1038.017303	1038.017350	0.045
c20	2+	1081.533317	1081.533397	0.074
c21	2+	1139.046789	1139.046866	0.068
c22	2+	1196.560261	1196.560497	0.197
c23	3+	847.065404	847.065401	-0.004
c23	2+	1270.094468	1270.094648	0.142
c24	3+	866.072559	866.072757	0.229
c25	3+	889.751597	889.751369	-0.256
c25	2+	1334.123849	1334.123808	-0.031
c26	3+	927.446285	927.446297	0.013
c26	2+	1390.665789	1390.665948	0.114
c27	3+	965.140973	965.141253	0.290
c27	2+	1447.709221	1447.709709	0.337
c28	3+	994.151649	994.151665	0.016
c29	3+	1023.162325	1023.162402	0.075
c29	2+	1535.242655	1535.243476	0.535
c30	3+	1056.844885	1056.844929	0.042
c30	2+	1584.763690	1584.764560	0.549
c31	3+	1094.859194	1094.859257	0.058
c31	2+	1642.286550	1642.287622	0.653
c32	3+	1127.881999	1127.882005	0.005
c32	2+	1692.823617	1692.824904	0.760
c33	3+	1146.889154	1146.889155	0.001
c34	3+	1175.899830	1175.899842	0.010
c34	2+	1763.346107	1763.347520	0.801
c35	3+	1214.576411	1214.576892	0.396
c35	2+	1820.859579	1820.859860	0.154
c36	3+	1247.924704	1247.924446	-0.207
y3	1+	397.171777	397.171687	-0.227
y4	1+	484.203806	484.203654	-0.314
y5	1+	541.225270	541.225155	-0.212
y6	1+	640.293684	640.293629	-0.086
y7	1+	754.336612	754.336600	-0.016
y8	1+	855.384291	855.384408	0.137
y9	1+	942.416320	942.416439	0.126
y10	1+	1029.448349	1029.448387	0.037
y13	1+	1326.653591	1326.653604	0.010
z2	1+	266.126109	266.126109	0.000
z3	1+	382.160877	382.160842	-0.092
z4	1+	467.177255	467.177105	-0.321
z5	1+	526.214369	526.214234	-0.257
z27	2+	1435.701402	1435.700996	-0.283
M+c31	5+	1438.883524	1438.884166	0.446
M+c32	5+	1458.496774	1458.496281	-0.338
M+c32	4+	1822.618557	1822.618598	0.022
M+c33	5+	1469.700594	1469.701214	0.422
M+c33	4+	1837.376472	1837.376990	0.282
M+c34	5+	1487.107000	1487.107008	0.005
M+c34	4+	1858.883887	1858.885137	0.672
M+c35	6+	1258.761936	1258.761197	-0.587
M+c35	5+	1510.312868	1510.313214	0.229
M+c35	4+	1887.891222	1887.892061	0.444
M+c36	6+	1275.603219	1275.603240	0.016
M+c36	5+	1530.924420	1530.924741	0.210
M+z3	3+	1429.328685	1429.329245	0.392
M+z4	3+	1458.339361	1458.339476	0.079
M+z5	3+	1477.346514	1477.347034	0.352
M+z6	3+	1510.369320	1510.368816	-0.334
M+z7	3+	1548.049351	1548.049768	0.269
M+z7	2+	2323.077144	2323.078314	0.504
M+z8	3+	1582.400354	1582.401276	0.583
M+z9	3+	1611.746974	1611.746129	-0.524
M+z10	3+	1639.753258	1639.752527	-0.446
M+z11	4+	1258.839125	1258.839009	-0.092
M+z11	3+	1677.447952	1677.448418	0.278
M+z12	4+	1287.110147	1287.110616	0.364
M+z12	3+	1715.811104	1715.812415	0.764
M+z13	4+	1304.869427	1304.869520	0.071
M+z13	3+	1738.821678	1738.820724	-0.549
M+z14	4+	1318.623443	1318.623618	0.133
M+z14	3+	1757.828832	1757.829443	0.348
M+z14	2+	2637.748852	2637.749391	0.204
M+z15	4+	1355.390547	1355.390850	0.224
M+z15	3+	1807.856058	1807.856916	0.475
M+z16	4+	1384.147283	1384.147298	0.011
M+z16	3+	1846.532522	1846.533255	0.397
M+z16	2+	2769.799453	2769.799567	0.041
M+z17	4+	1412.904019	1412.904502	0.342
M+z17	3+	1884.874843	1884.874962	0.063
M+z18	4+	1434.912737	1434.913034	0.207
M+z18	3+	1913.885523	1913.885817	0.154
M+z19	4+	1456.420033	1456.420138	0.072
M+z19	3+	1942.896202	1942.895513	-0.355
M+z19	2+	2914.846456	2914.846774	0.109
M+z20	4+	1491.186124	1491.186700	0.386
M+z20	3+	1988.582507	1988.583390	0.444
M+z21	4+	1515.953231	1515.953433	0.133
M+z21	3+	2021.605320	2021.606146	0.409
M+z22	4+	1544.224252	1544.224820	0.368
M+z22	3+	2059.300020	2059.301231	0.588
M+z23	4+	1580.489984	1580.490360	0.238
M+z23	3+	2108.322843	2108.323867	0.486
M+z24	4+	1609.000716	1609.001073	0.222
M+z24	3+	2146.337153	2146.338370	0.567
M+z25	4+	1627.512008	1627.512728	0.442
M+z25	3+	2170.350335	2170.351553	0.561
M+z26	5+	1324.827880	1324.828098	0.165
M+z26	4+	1655.783031	1655.783641	0.368
M+z26	3+	2208.046833	2208.046628	-0.093
M+z27	5+	1356.048099	1356.048275	0.130
M+z27	4+	1695.060261	1695.060689	0.252
M+z27	3+	2260.080530	2260.081089	0.247
M+z28	5+	1381.659815	1381.660080	0.192
M+z28	4+	1726.824267	1726.824613	0.200
M+z29	5+	1402.069844	1402.070205	0.257
M+z29	4+	1752.086188	1752.086770	0.332
M+z30	5+	1416.076777	1416.077014	0.167
M+z30	4+	1770.096109	1770.096483	0.211
M+z30	3+	2359.792401	2359.792212	-0.080
M+z34	5+	1491.103517	1491.103695	0.119
M+z34	4+	1864.130123	1864.130663	0.290
M+z34	3+	2485.507093	2485.507949	0.344
M+z35	5+	1513.912103	1513.912030	-0.048
M+z36	5+	1534.713228	1534.713352	0.081
M+z36	4+	1918.141132	1918.141936	0.419
			Average Error:	0.124
			Absolute Average Error:	0.236
			Standard Deviation:	0.278

Table S1. List of the assigned fragments from the ECD MS/MS spectrum of the 7+ charge state ($(D)_3$hIAPP) dimer ion. M represents one ($(D)_3$hIAPP) unit.

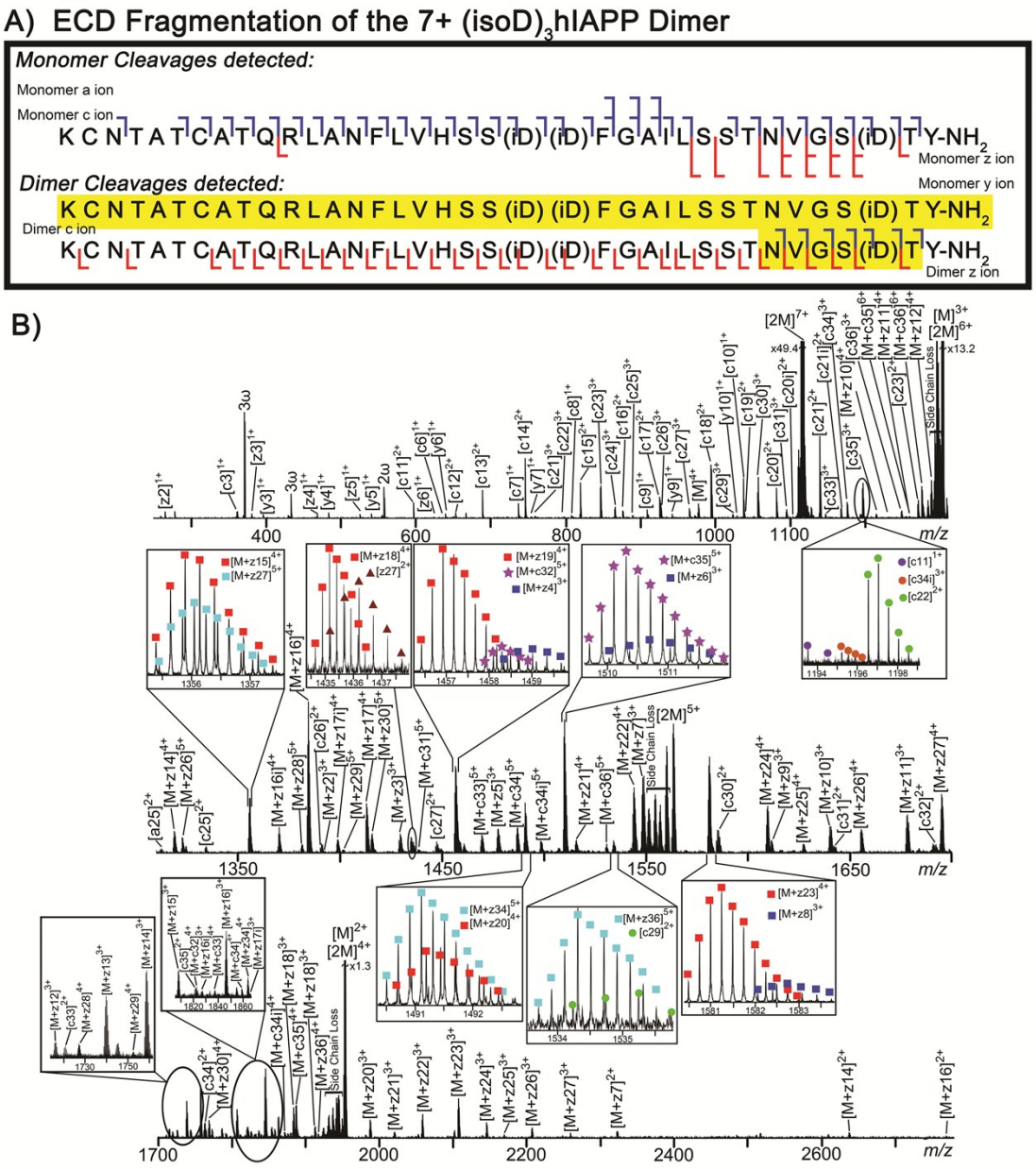

Figure S3. (A) Summary of a-, c-, y-, and z-ion fragments observed in the (B) ECD MS/MS spectrum of the 7+ charge state ($(isoD)_3$hIAPP) dimer. Highlighted sequence represents the proposed non-covalent interaction region. The side chain losses were not labeled for clarity. The assigned fragments are listed in the ESI Table S2.

Assignment Table of the ECD MS/MS of 7+ charge state ((isoD)$_3$hIAPP) dimer

Type	Ion	charge	Theoretical *m/z*	Experimental *m/z*	Error (ppm)
MH	MH	4+	977.211884	977.211677	-0.212
MH	MH	3+	1302.279143	1302.279080	-0.048
MH	MH	2+	1952.915077	1952.915655	0.296
2[MH]	2[MH]	7+	1116.383162	1116.383218	0.050
2[MH]	2[MH]	6+	1303.115465	1303.115597	0.101
2[MH]	2[MH]	5+	1563.738667	1563.738713	0.029
2[MH]	2[MH]	4+	1954.673471	1954.672757	-0.365
a	23	2+	1248.087645	1248.087793	0.119
a	24	2+	1277.099772	1277.099889	0.092
a	25	2+	1312.116934	1312.117109	0.133
c	3	1+	362.173076	362.173033	-0.119
c	6	1+	636.313372	636.313275	-0.152
c	7	1+	737.306908	737.306876	-0.043
c	8	1+	808.344022	808.343966	-0.069
c	9	1+	909.391701	909.391605	-0.106
c	10	1+	1037.450279	1037.450277	-0.002
c	11	2+	597.279333	597.279315	-0.030
c	11	1+	1193.551390	1193.551247	-0.120
c	12	2+	653.821365	653.821303	-0.095
c	13	2+	689.339922	689.339931	0.013
c	14	2+	746.361386	746.361372	-0.019
c	15	2+	819.895593	819.895597	0.005
c	16	2+	876.437625	876.437697	0.082
c	17	2+	925.971832	925.971846	0.015
c	18	2+	994.501288	994.501292	0.004
c	19	2+	1038.017303	1038.017376	0.070
c	20	2+	1081.533317	1081.533351	0.031
c	21	3+	759.700285	759.700251	-0.045
c	21	2+	1139.046789	1139.046820	0.027
c	22	3+	798.042599	798.042537	-0.078
c	22	2+	1196.560261	1196.560293	0.027
c	23	3+	847.065404	847.065386	-0.021
c	23	2+	1270.094468	1270.094418	-0.039
c	24	3+	866.072559	866.072652	0.107
c	25	3+	889.751597	889.751696	0.111
c	25	2+	1334.123849	1334.123757	-0.069
c	26	3+	927.446285	927.446286	0.001
c	26	2+	1390.665789	1390.666501	0.512
c	27	3+	965.140973	965.140963	-0.010
c	27	2+	1447.709221	1447.709778	0.385
c	29	3+	1023.162325	1023.162215	-0.108
c	29	2+	1534.239850	1534.239965	0.075
c	30	3+	1056.844885	1056.844848	-0.035
c	30	2+	1584.763690	1584.763724	0.021
c	31	3+	1094.859194	1094.859270	0.069
c	31	2+	1642.787956	1642.788594	0.388
c	32	3+	1127.881999	1127.881584	-0.368
c	32	2+	1691.319356	1691.320699	0.794
c	33	3+	1146.889154	1146.888901	-0.221
c	33	2+	1720.331490	1720.332143	0.380
c	34	3+	1175.899830	1175.899636	-0.165
c	34	2+	1763.346107	1763.346490	0.217
c	35	3+	1214.576411	1214.576568	0.129
c	35	2+	1820.859579	1820.860110	0.292
c	36	3+	1247.924704	1247.924462	-0.194
c	20i	2+	1110.032142	1110.032427	0.256
c	21i	2+	1167.545613	1167.545686	0.063
c	34i	3+	1195.233316	1195.233951	0.531
y	3	1+	397.171777	397.171827	0.126
y	4	1+	484.203806	484.203771	-0.072
y	5	1+	541.225270	541.225281	0.020
y	6	1+	640.293684	640.293709	0.039
y	7	1+	754.336612	754.336563	-0.065
y	9	1+	942.416320	942.416353	0.035
y	10	1+	1029.448349	1029.448645	0.288
z	2	1+	266.126109	266.126109	0.000
z	3	1+	382.160877	382.160841	-0.094
z	4	1+	469.192905	469.192914	0.019
z	5	1+	526.214369	526.214338	-0.059
z	6	1+	625.282783	625.282678	-0.168
z	27	2+	1435.197489	1435.196299	-0.829
M+c	31	5+	1438.482615	1438.482645	0.021
M+c	32	5+	1458.496774	1458.496042	-0.502
M+c	32	4+	1822.618557	1822.618534	-0.013
M+c	33	5+	1469.700594	1469.699673	-0.627
M+c	33	4+	1837.375068	1837.375877	0.440
M+c	34	5+	1487.105911	1487.106523	0.412
M+c	34	4+	1858.883887	1858.883845	-0.023
M+c	35	6+	1258.761936	1258.761954	0.014
M+c	35	5+	1510.312868	1510.312981	0.075
M+c	35	4+	1887.891222	1887.891506	0.150
M+c	36	6+	1275.603219	1275.603316	0.076
M+c	36	5+	1530.924420	1530.924203	-0.142
M+c	34i	5+	1498.707009	1498.707619	0.407
M+c	34i	4+	1873.131943	1873.132905	0.514
M+z	2	3+	1391.654617	1391.655808	0.856
M+z	3	3+	1429.328685	1429.329444	0.531
M+z	4	3+	1458.339361	1458.340006	0.442
M+z	5	3+	1477.680667	1477.680895	0.154
M+z	6	3+	1510.369320	1510.368775	-0.361
M+z	7	3+	1548.049351	1548.049654	0.196
M+z	7	2+	2323.578098	2323.578660	0.242
M+z	8	3+	1582.400354	1582.401094	0.468
M+z	9	3+	1611.746974	1611.746700	-0.170
M+z	10	4+	1230.568102	1230.567991	-0.090
M+z	10	3+	1639.753258	1639.753036	-0.135
M+z	11	4+	1258.839125	1258.839593	0.372
M+z	11	3+	1677.447952	1677.447828	-0.074
M+z	12	4+	1287.110147	1287.110909	0.592
M+z	12	3+	1715.811104	1715.811618	0.300
M+z	13	4+	1304.869427	1304.869611	0.141
M+z	13	3+	1738.821678	1738.821082	-0.343
M+z	14	4+	1318.623443	1318.623189	-0.193
M+z	14	3+	1757.828832	1757.829081	0.142
M+z	14	2+	2638.247419	2638.246892	-0.200
M+z	15	4+	1355.390547	1355.390795	0.183
M+z	15	3+	1807.856058	1807.856380	0.178
M+z	16	4+	1384.147283	1384.147411	0.092
M+z	16	3+	1846.532522	1846.532903	0.206
M+z	16	2+	2769.295145	2769.294672	-0.171
M+z	17	4+	1412.904019	1412.903882	-0.097
M+z	17	3+	1884.874843	1884.874708	-0.072
M+z	18	4+	1434.912737	1434.912984	0.172
M+z	18	3+	1913.885523	1913.885178	-0.180
M+z	19	4+	1456.420033	1456.420238	0.141
M+z	19	3+	1942.896202	1942.895988	-0.110
M+z	20	4+	1491.186124	1491.186435	0.209
M+z	20	3+	1988.582507	1988.582942	0.219
M+z	21	4+	1515.953231	1515.953379	0.098
M+z	21	3+	2021.605320	2021.605553	0.115
M+z	22	4+	1544.224252	1544.224504	0.163
M+z	22	3+	2059.300020	2059.300910	0.432
M+z	23	4+	1580.489984	1580.489808	-0.111
M+z	23	3+	2108.322843	2108.323448	0.287
M+z	24	4+	1609.000716	1609.000938	0.138
M+z	24	3+	2146.337153	2146.337709	0.259
M+z	25	4+	1627.512008	1627.512645	0.391
M+z	25	3+	2170.350335	2170.351009	0.311
M+z	26	5+	1324.827880	1324.827646	-0.177
M+z	26	4+	1655.783031	1655.783415	0.232
M+z	26	3+	2208.046833	2208.046104	-0.330
M+z	27	5+	1356.048099	1356.048495	0.292
M+z	27	4+	1695.060261	1695.060550	0.170
M+z	27	3+	2260.080530	2260.081289	0.336
M+z	28	5+	1381.659815	1381.660250	0.315
M+z	28	4+	1726.824267	1726.823766	-0.290
M+z	29	5+	1402.069844	1402.069738	-0.076
M+z	29	4+	1752.086188	1752.086786	0.341
M+z	30	5+	1416.076777	1416.076531	-0.174
M+z	30	4+	1769.844153	1769.844536	0.216
M+z	34	5+	1491.103517	1491.103470	-0.032
M+z	34	4+	1864.130123	1864.130353	0.123
M+z	36	5+	1534.713228	1534.713224	-0.003
M+z	36	4+	1918.141132	1918.141912	0.407
M+z	16-i	4+	1370.399228	1370.399399	0.125
M+z	16-i	3+	1827.199153	1827.199447	0.161
M+z	17-i	4+	1399.406577	1399.406737	0.114
M+z	17-i	3+	1865.875642	1865.875456	-0.100
				Average Error:	0.068
				Absolute Average Error:	0.187
				Standard Deviation:	0.245

Table S2. List of the assigned fragments from the ECD MS/MS spectrum of the 7+ charge state ((isoD)$_3$hIAPP) dimer ion. M represents one ((isoD)$_3$hIAPP) unit.

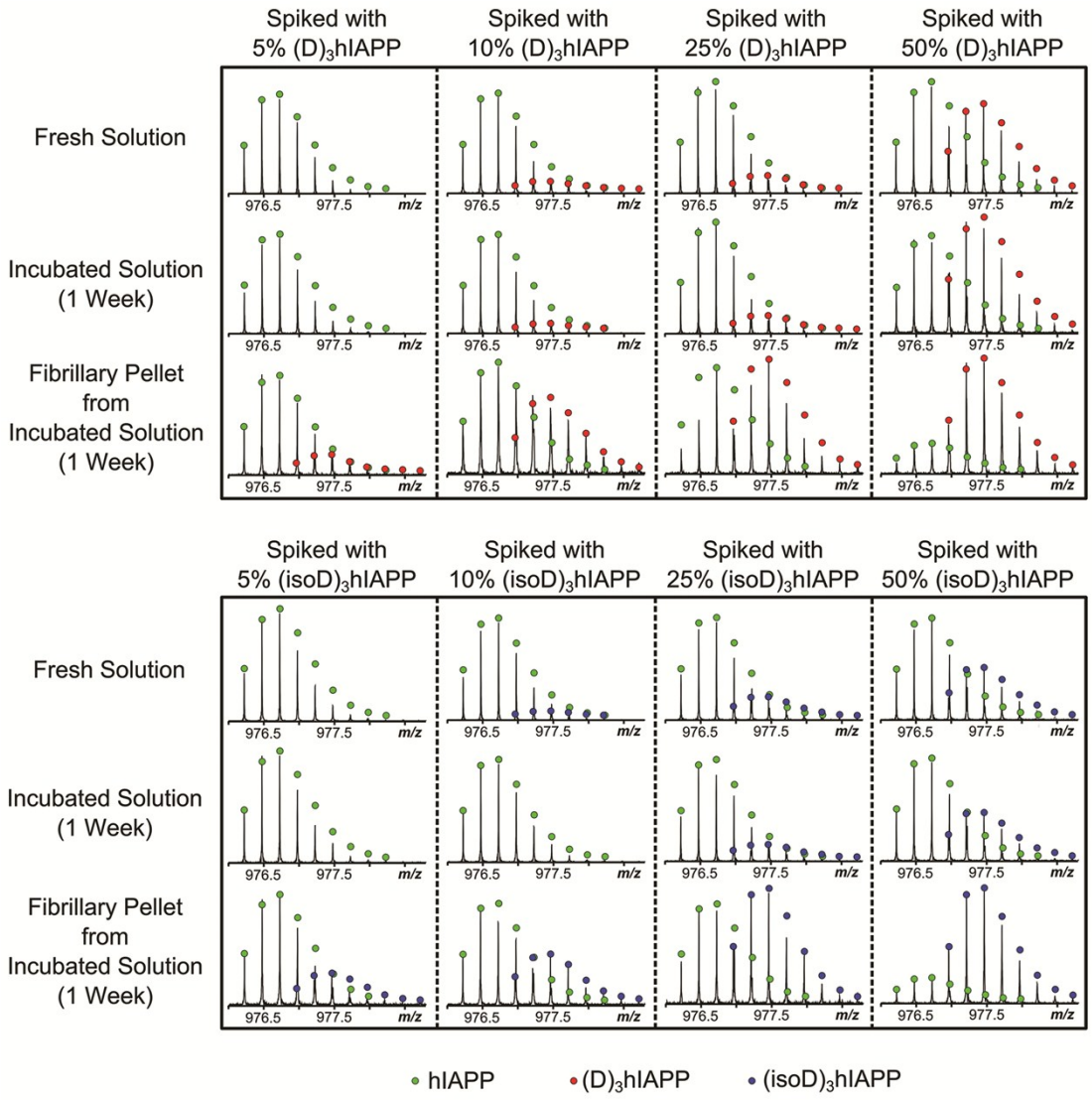

Figure S4. The nESI-MS spectra showing the fresh, the incubated solutions, and the incubated fibrillary pellets of hIAPP mixed with 5%, 10%, 25%, or 50% of mutant ($(D)_3$hIAPP) or ($(isoD)_3$hIAPP).

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