

Figure S1. Schematic representation of the synthesis procedure to obtain dimeric peptides ²⁶[F] and 4. The dimeric peptide (FKKLG)₂-K-Ahx-resin was obtained, and this peptide-resin was divided into two reactors allowing continue to the synthesis to get peptides ²⁶[F] and 4.

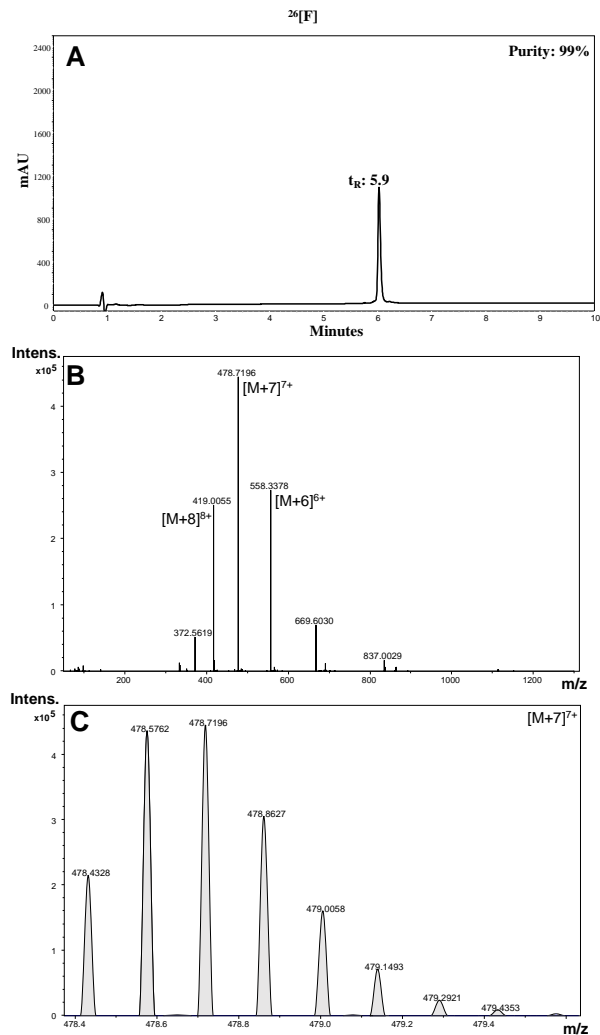


Figure S2. ²⁶[F] characterization: (A) Chromatographic profile; (B) LC-MS spectrum: multicharged species [M+6]⁶⁺; [M+7]⁷⁺ and [M+8]⁸⁺ are observed; (C) the isotopic pattern of multicharged specie [M+7]⁷⁺ is observe in the spectrum zoom.

1

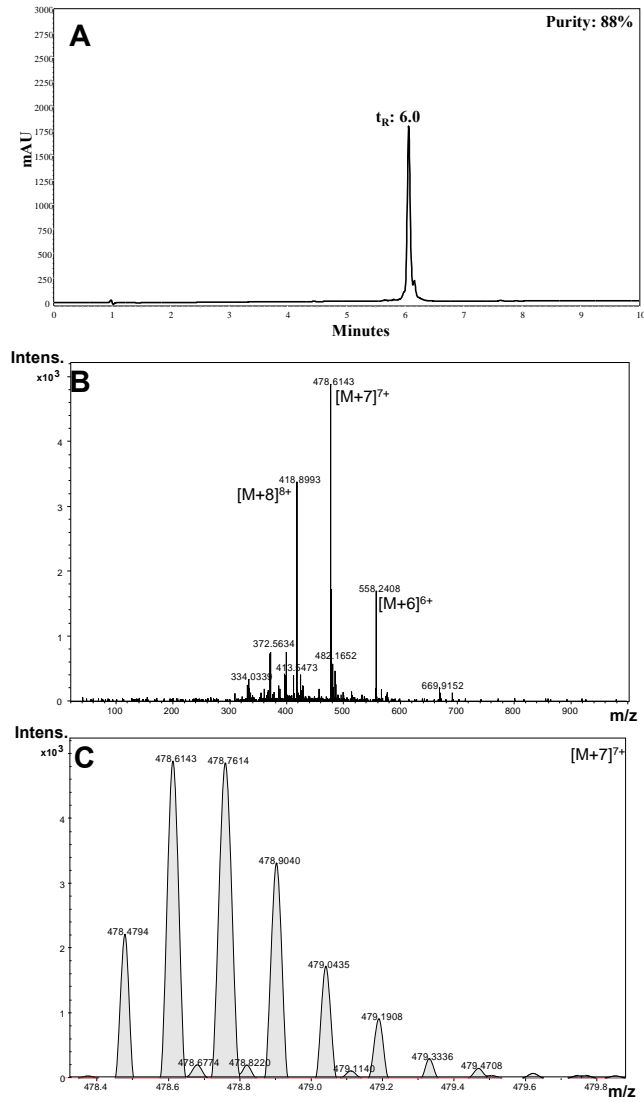


Figure S3. Peptide 1 characterization: (A) Chromatographic profile; (B) LC-MS spectrum: multicharged species $[M+6]^{6+}$; $[M+7]^{7+}$ and $[M+8]^{8+}$ are observed; (C) the isotopic pattern of multicharged specie $[M+7]^{7+}$ is observe in the spectrum zoom.

2

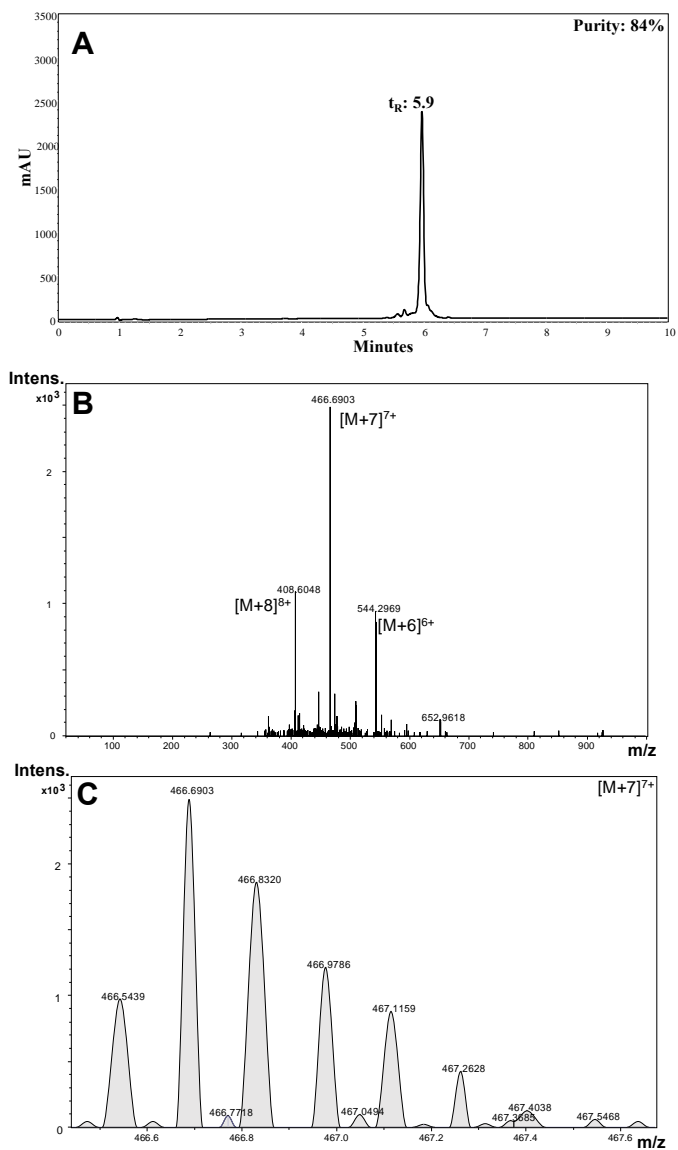


Figure S4. Peptide 2 characterization: (A) Chromatographic profile; (B) LC-MS spectrum: multicharged species $[M+6]^{6+}$; $[M+7]^{7+}$ and $[M+8]^{8+}$ are observed; (C) the isotopic pattern of multicharged specie $[M+7]^{7+}$ is observe in the spectrum zoom.

3

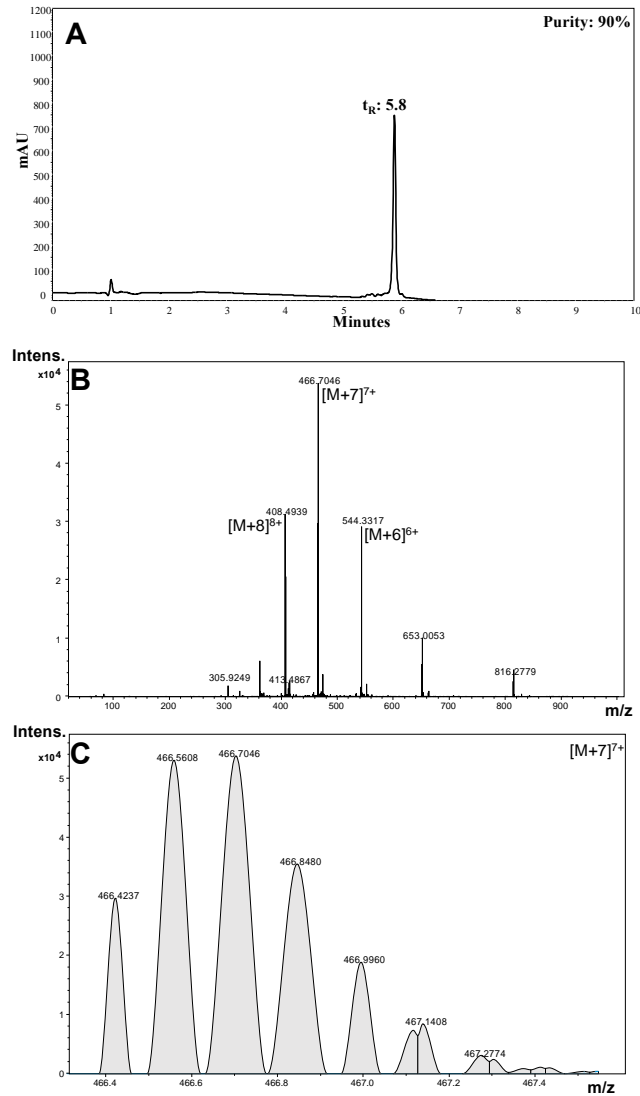


Figure S5. Peptide 3 characterization: (A) Chromatographic profile; (B) LC-MS spectrum: multicharged species $[M+6]^{6+}$; $[M+7]^{7+}$ and $[M+8]^{8+}$ are observed; (C) the isotopic pattern of multicharged specie $[M+7]^{7+}$ is observe in the spectrum zoom.

4

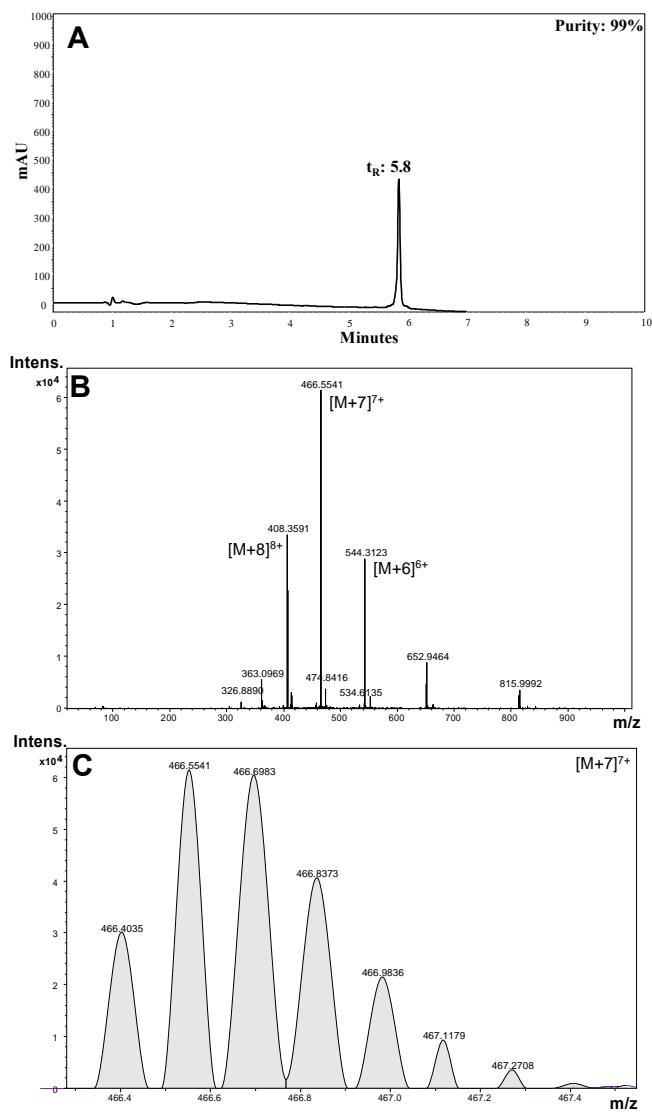


Figure S6. Peptide 4 characterization: (A) Chromatographic profile; (B) LC-MS spectrum: multicharged species $[M+6]^{6+}$; $[M+7]^{7+}$ and $[M+8]^{8+}$ are observed; (C) the isotopic pattern of multicharged specie $[M+7]^{7+}$ is observe in the spectrum zoom.

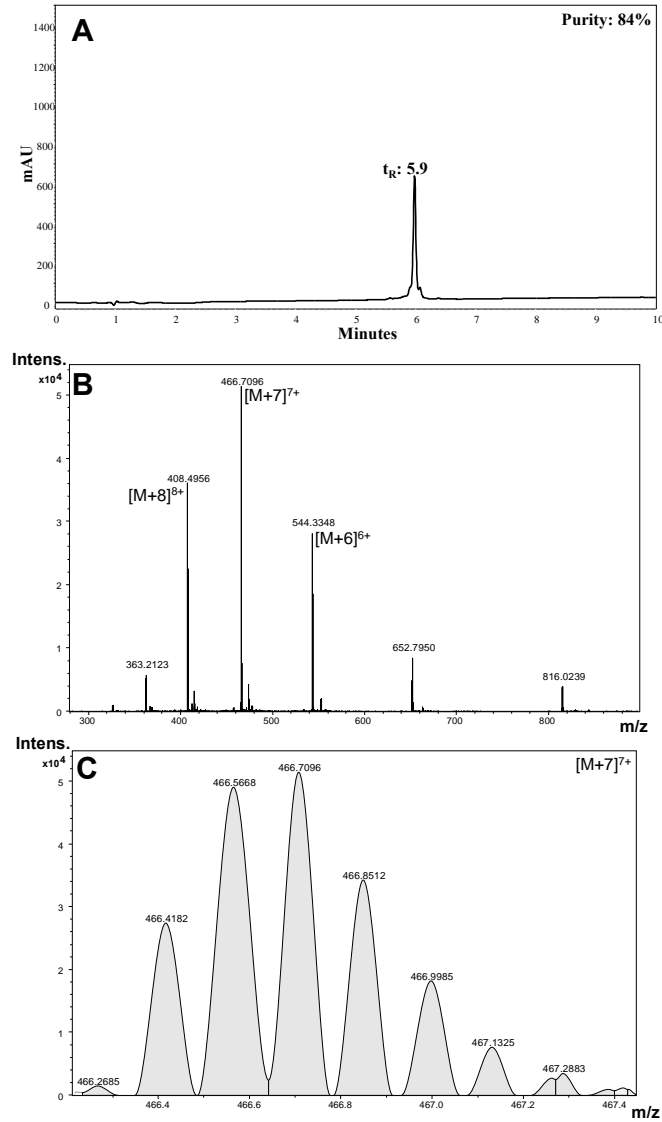


Figure S7. Peptide 5 characterization: (A) Chromatographic profile; (B) LC-MS spectrum: multicharged species $[M+6]^{6+}$; $[M+7]^{7+}$ and $[M+8]^{8+}$ are observed; (C) the isotopic pattern of multicharged specie $[M+7]^{7+}$ is observe in the spectrum zoom.

6

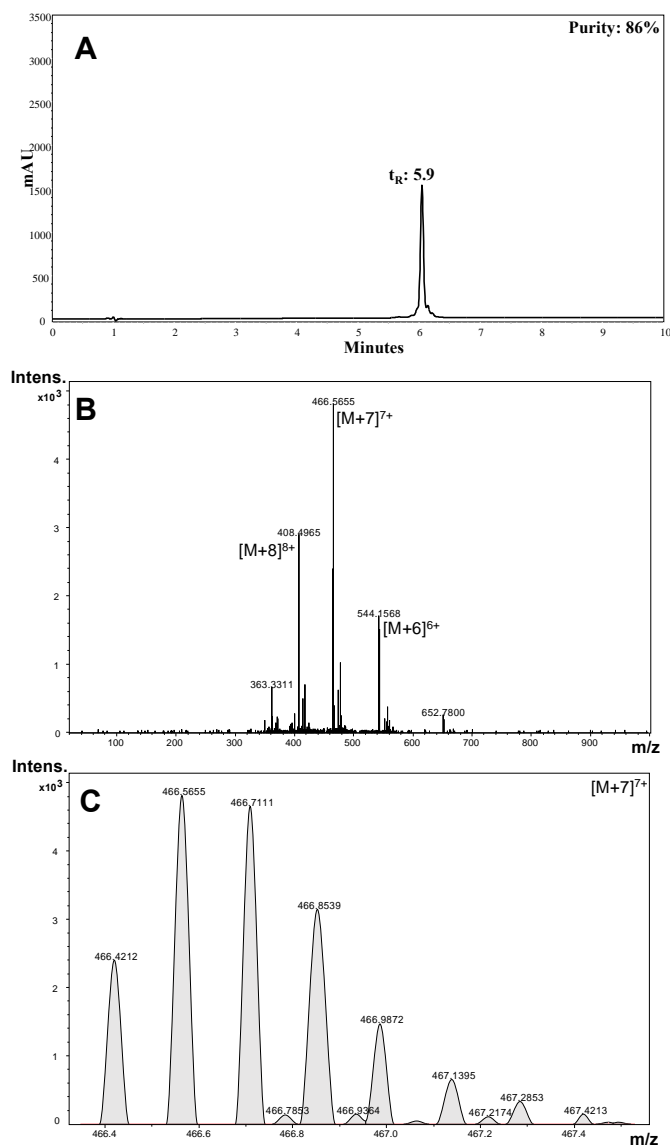


Figure S8. Peptide 6 characterization: (A) Chromatographic profile; (B) LC-MS spectrum: multicharged species $[M+6]^{6+}$; $[M+7]^{7+}$ and $[M+8]^{8+}$ are observed; (C) the isotopic pattern of multicharged specie $[M+7]^{7+}$ is observe in the spectrum zoom.

7

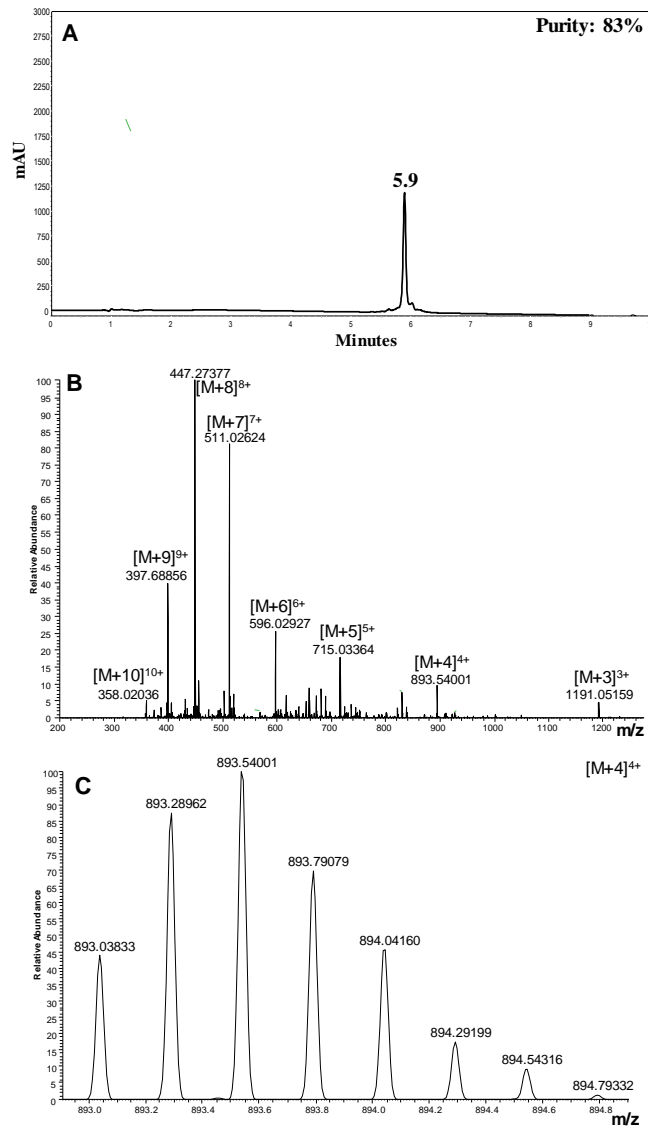


Figure S9. Peptide 7 characterization: (A) Chromatographic profile; (B) LC-MS spectrum: multicharged species $[M+3]^{3+}$; $[M+4]^{4+}$; $[M+5]^{5+}$; $[M+6]^{6+}$; $[M+7]^{7+}$; $[M+8]^{8+}$; $[M+9]^{9+}$ and $[M+10]^{10+}$ species are observed; (C) the isotopic pattern of multicharged species $[M+4]^{4+}$ is observed in the spectrum zoom.

8

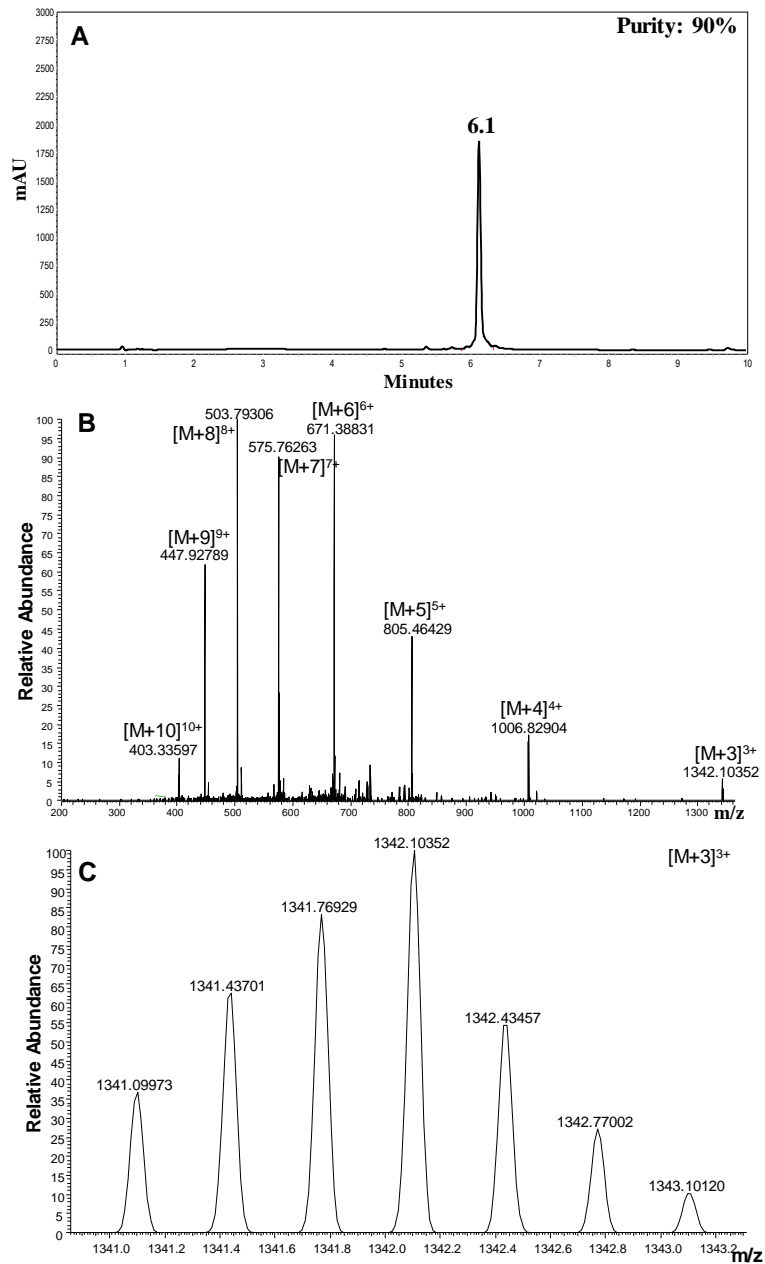


Figure S10. Peptide 8 characterization: (A) Chromatographic profile; (B) LC-MS spectrum: multicharged species $[M+3]^{3+}$; $[M+4]^{4+}$; $[M+5]^{5+}$; $[M+6]^{6+}$; $[M+7]^{7+}$; $[M+8]^{8+}$; $[M+9]^{9+}$ and $[M+10]^{10+}$ are observed; (C) the isotopic pattern of multicharged species $[M+3]^{3+}$ is observed in the spectrum zoom.

9

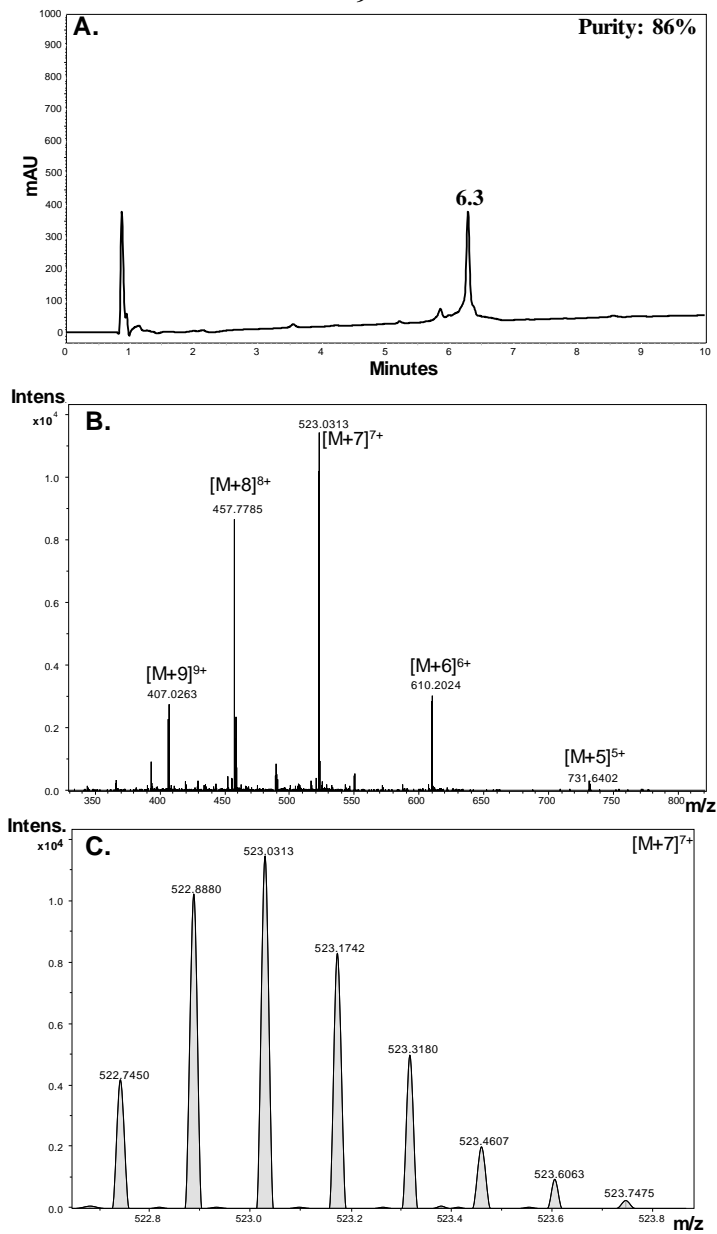


Figure S11. Peptide 9 characterization: (A) Chromatographic profile; (B) LC-MS spectrum: multicharged species [M+5]⁵⁺; [M+6]⁶⁺; [M+7]⁷⁺; [M+8]⁸⁺ and [M+9]⁹⁺ are observed; (C) the isotopic pattern of multicharged species [M+7]⁷⁺ is observed in the spectrum zoom.

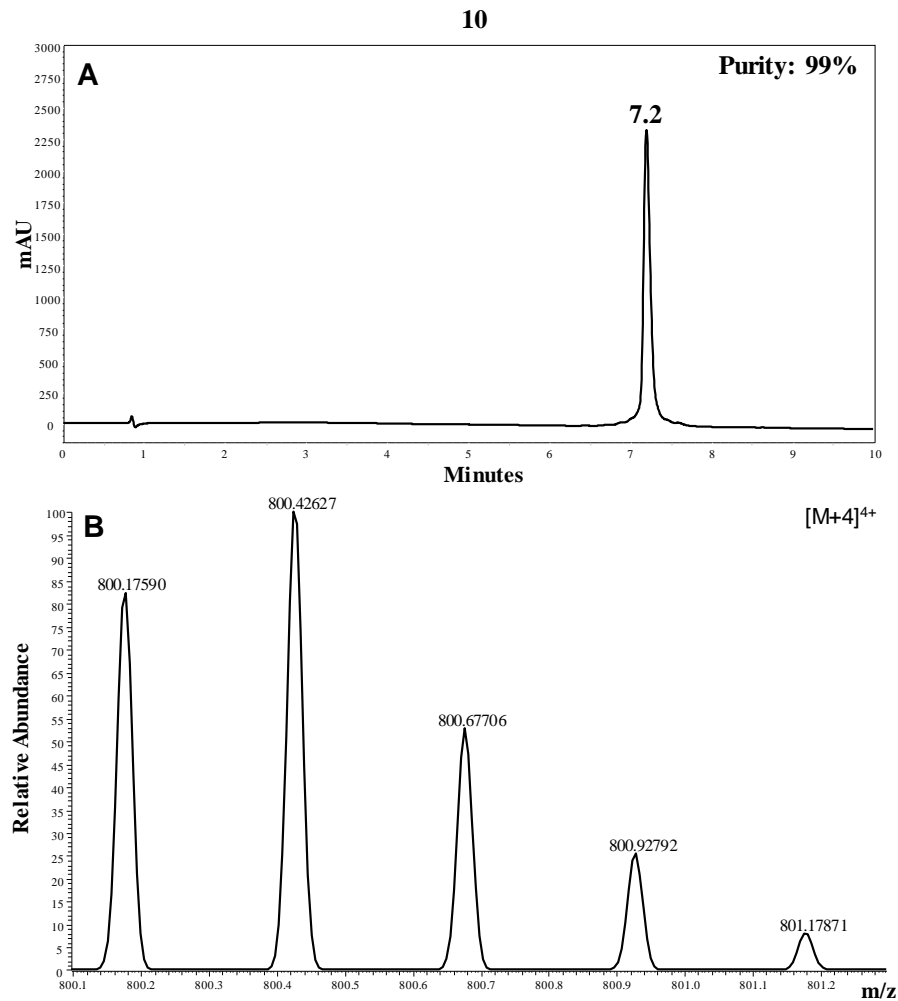


Figure S12. Peptide 10 characterization: (A) Chromatographic profile; (B) Spectrum of isotopic distribution of multicharged specie $[M+4]^{4+}$.

11

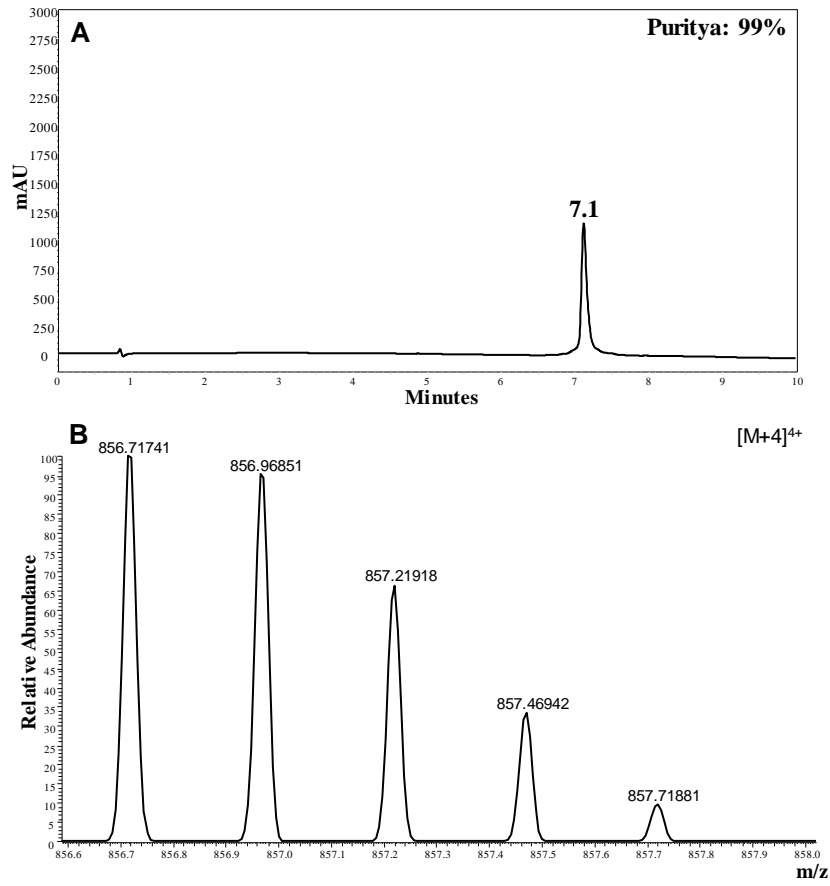


Figure S13. Peptide 11 characterization: (A) Chromatographic profile; (B) Spectrum of isotopic distribution of multicharged specie $[M+4]^{4+}$.

12

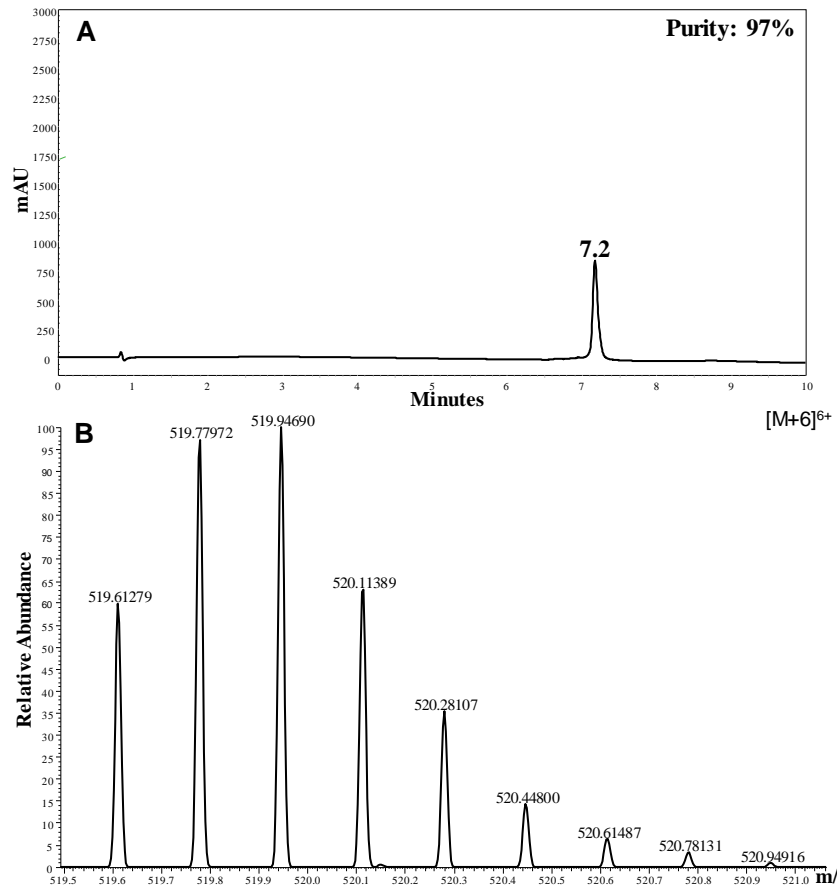


Figure S14. Peptide 12 characterization: (A) Chromatographic profile; (B) Spectrum of isotopic distribution of multicharged specie $[M+6]^{6+}$.

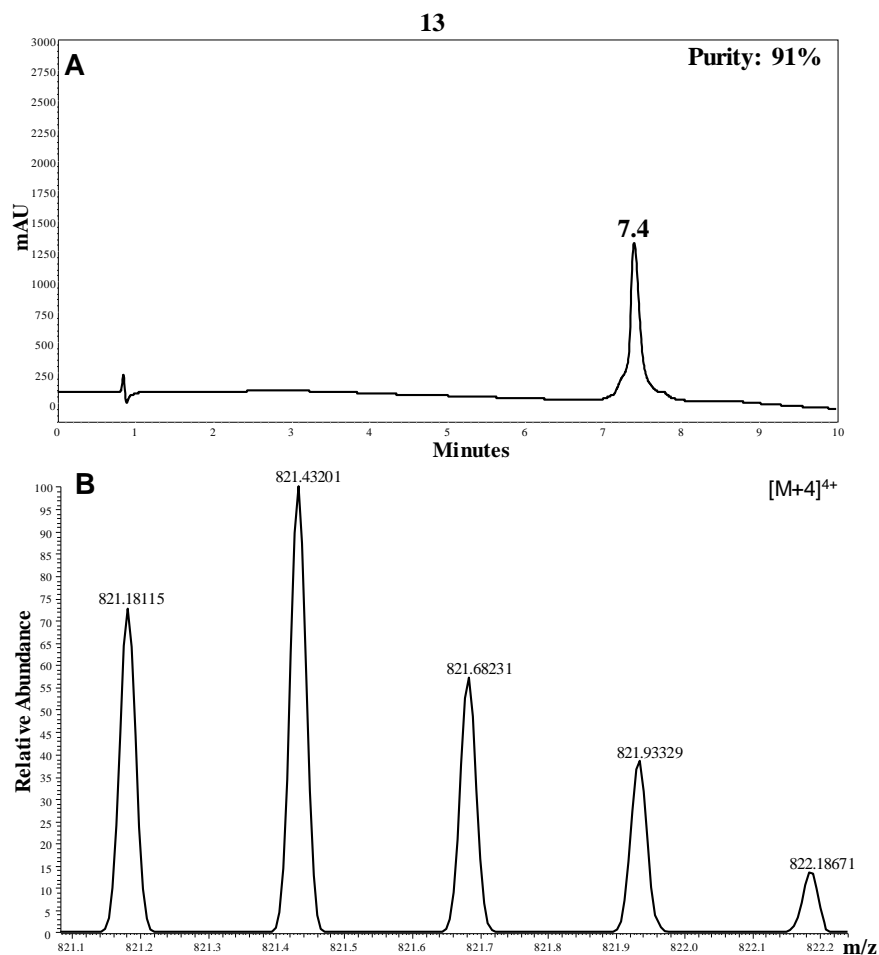


Figure S15. Peptide 13 characterization: (A) Chromatographic profile; (B) Spectrum of isotopic distribution of multicharged specie [M+4]⁴⁺.

Table S1. Characterization of peptide 3 batches by RP-HPLC and LC-MS.* Amount of crude peptide obtained

Code	Amount obtained (mg)*	RP-HPLC		Monoisotopic Mass M ⁺		
		t _R (min)	Purity (%)	Theoretical	Exp	Error (ppm)
Batch 01	10	5.8	90	3257.930	3257.910	6
Batch 02	73	6.1	95	3257.931	3258.081	46
Batch 03	375	6.1	98	3257.931	3258.108	54

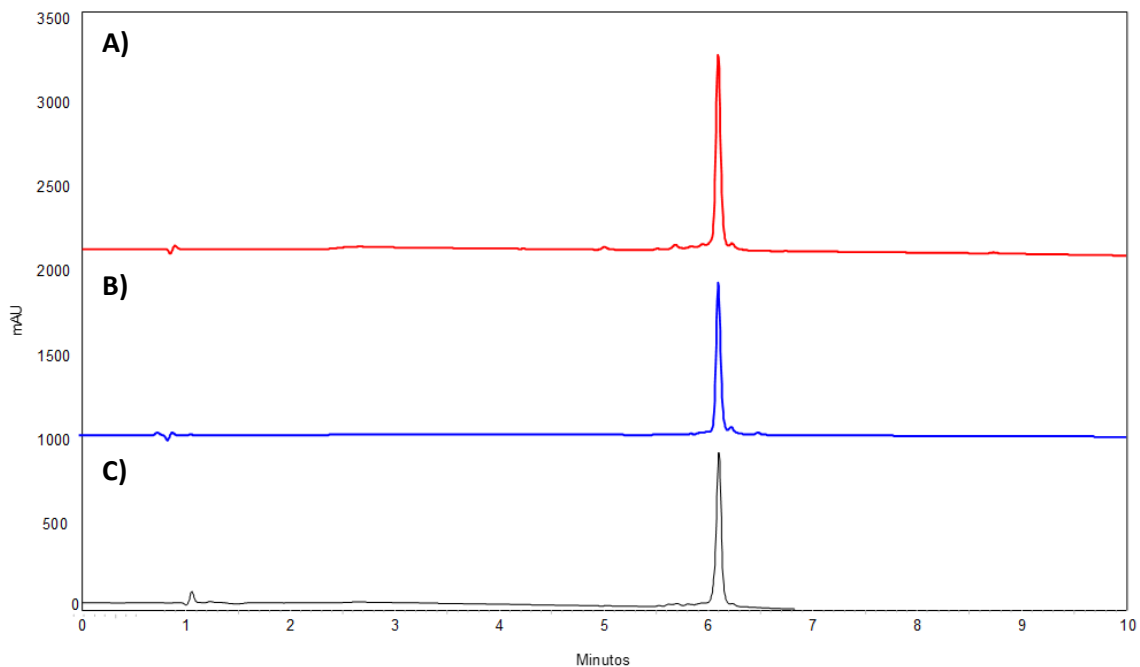


Figure S16. Peptide 3 Chromatographic profile of batches. (A) Batch 03. Chromatographic purity = 98%; (B) Batch 02. Chromatographic purity = 95%; (C) Batch 01. Chromatographic purity = 90%.

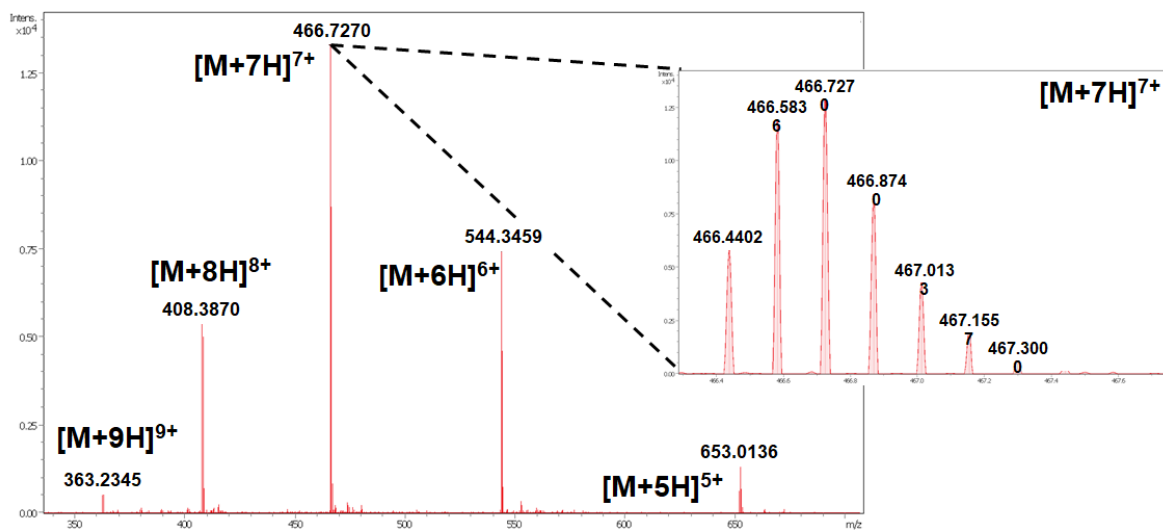


Figure S17. Peptide 3 Mass spectra of batch 02. (A) LC-MS spectrum: multicharged species $[M+2]^{2+}$; $[M+3]^{3+}$ and $[M+4]^{4+}$; are observed; (C) the isotopic pattern of multicharged specie $[M+3]^{3+}$ is observe in the spectrum zoom.

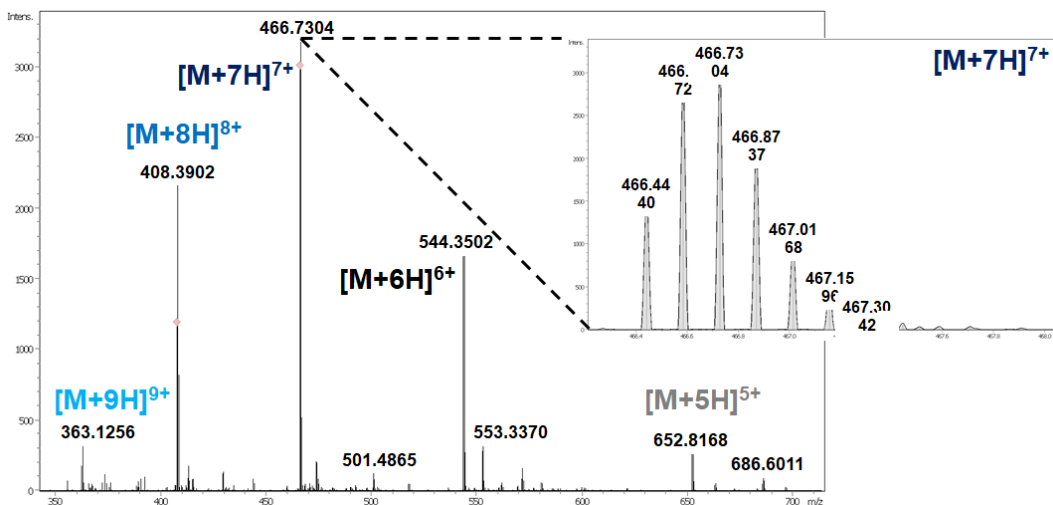


Figure S18. Peptide 3 Mass spectra of batch 03. (A) LC-MS spectrum: multicharged species $[M+5]^{5+}$; $[M+6]^{6+}$; $[M+7]^{7+}$; $[M+8]^{8+}$ and $[M+9]^{9+}$ are observed; (C) the isotopic pattern of multicharged specie $[M+7]^{7+}$ is observe in the spectrum zoom.

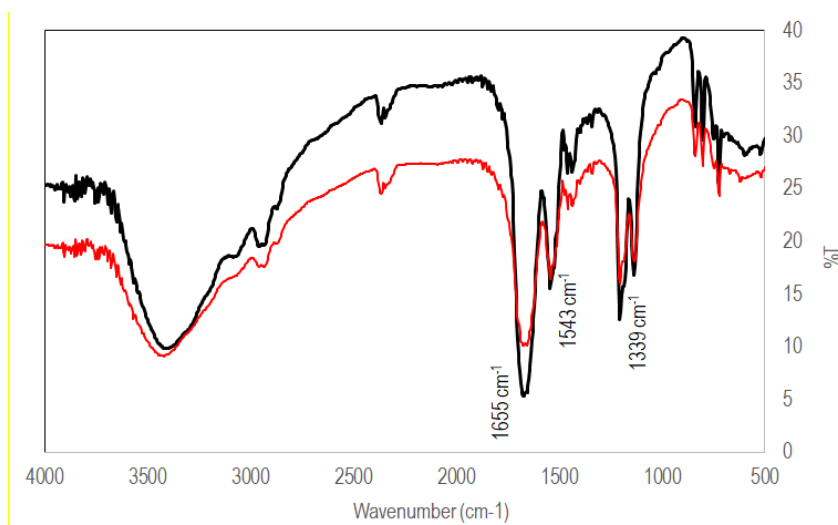


Figure S19. FT-IR analysis of batches 02 (black) and 03 (red) of peptide 3. The signal at 3500 cm^{-1} corresponds to the stretching vibrations of O-H, N-H, and C-H bonds. The peak at 2340 cm^{-1} is characteristic of the symmetric and asymmetric stretching of the C=O bond from CO₂. Between 2000 and 1700 cm^{-1} , overtones and combination bands related to aromatic groups are observed. The fingerprint region, which is unique to each molecule, is identified between 1000 and 500 cm^{-1} . The Amide I region, attributed to C=O bond stretching from the peptide bond, is typically found between 1700 and 1600 cm^{-1} , with both batches displaying a signal at 1655 cm^{-1} . The Amide II region, which corresponds to N-H bending coupled with C-N stretching, appears between 1600 and 1500 cm^{-1} , with a peak at 1543 cm^{-1} in both batches. The Amide III region, involving in-plane N-H bending and C-N bond stretching between the alpha carbon and nitrogen, as well as N-H deformation, is observed between 1350 and 1200 cm^{-1} , with a signal at 1339 cm^{-1} in both batches.

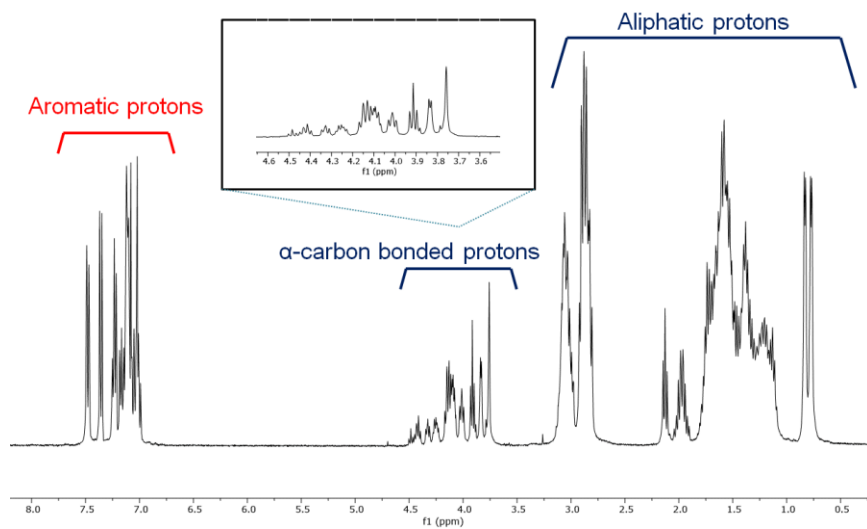


Figure S20. Spectrum ^1H -RMN of peptide 3 batch 02. The three regions are observed corresponding to aromatic, aliphatic and α -carbon protons. The extension shows the signals of protons bonded to α -carbon.

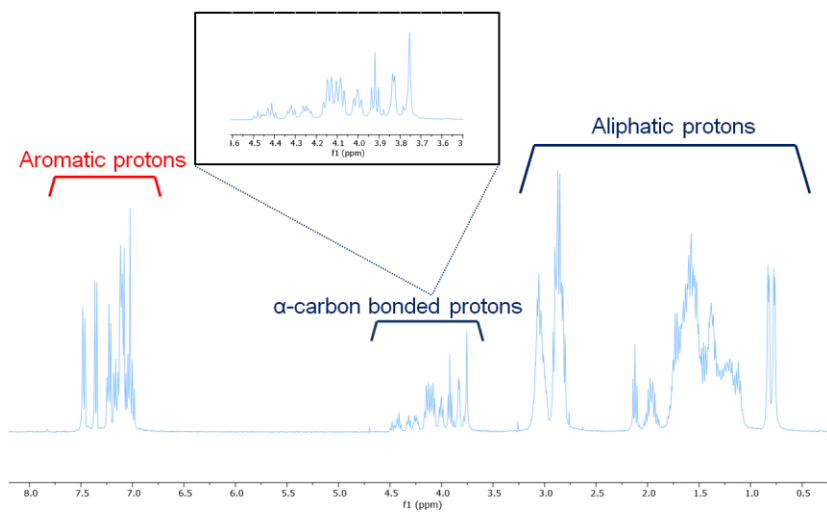


Figure S21. Spectrum ^1H -RMN of peptide 3 batch 03. The three regions are observed corresponding to aromatic, aliphatic and α -carbon protons. The extension shows the signals of protons bonded to α -carbon.

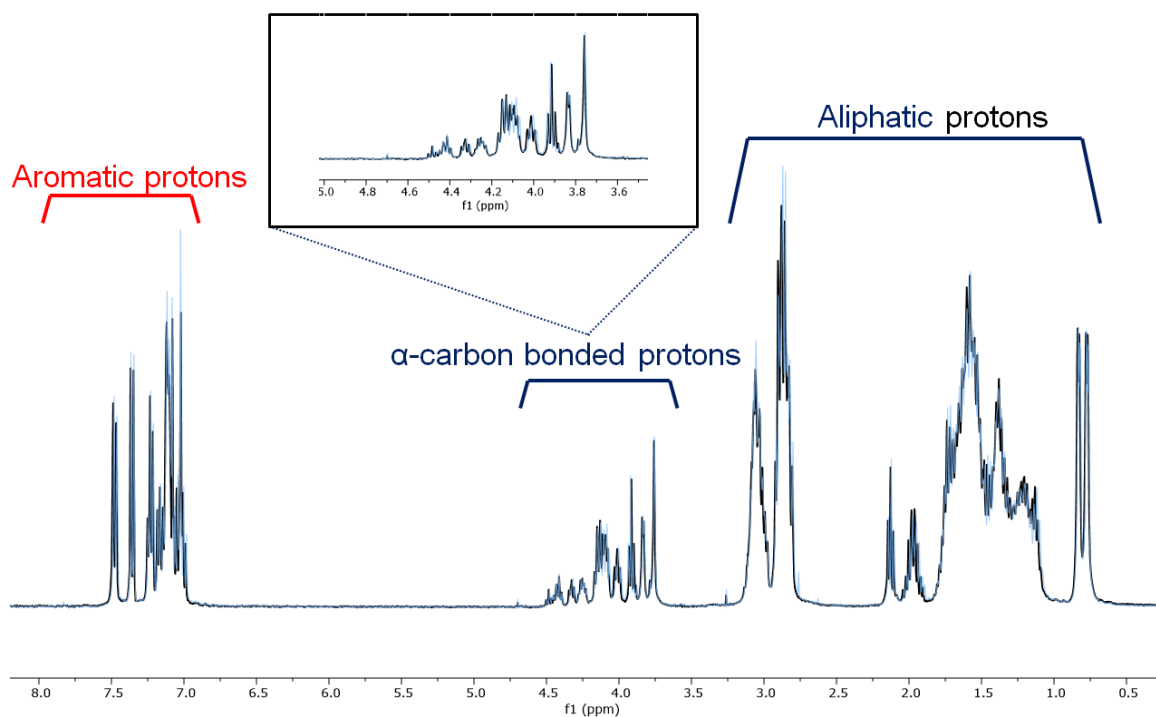


Figure S22. Overlay ^1H -NMR spectrum of peptide batches 02 and 03 of peptide 3. The three regions are observed corresponding to aromatic, aliphatic and α -carbon protons. The extension shows the signals of protons bonded to α -carbon.

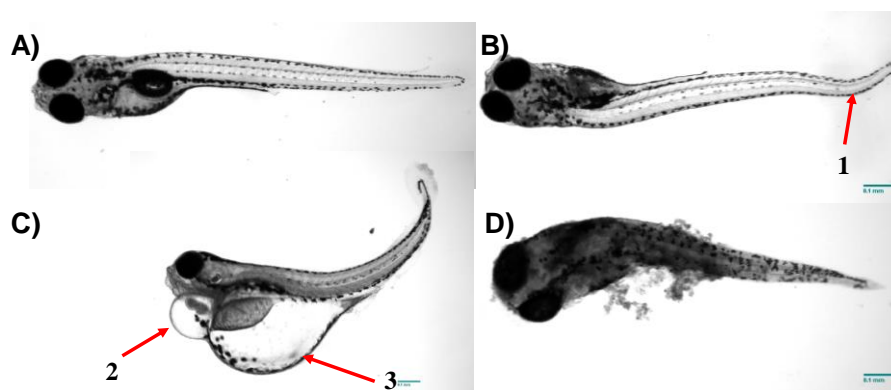


Figure S23. Morphological changes in zebrafish larvae (3 dpf) treated with peptide 3 A) Larva with normal morphology. B) Larva exhibiting a twisted tail (1). C) Larva showing pericardial and yolk sac edema (2 and 3, respectively). D) Dead larvae.