CHEMISTRY
AN ASIAN JOURNAL

Supporting Information

Synthetic Mucin-Like Glycopeptides as Versatile Tools to Measure Effects of Glycan Structure/Density/Position on the Interaction with Adhesion/Growth-Regulatory Galectins in Arrays

Gerard Artigas,[a] Hiroshi Hinou,*[a, b] Fayna Garcia-Martin,[a] Hans-Joachim Gabius,*[c] and Shin-Ichiro Nishimura*[a, b]

asia_201601420_sm_miscellaneous_information.pdf

Table S1. Sequences of the MUC1-derived (glyco)peptide and glyco amino acid used in this work.

1	5-oxo-hexanoyl-PEG-GVTSAPDTRPAPGSTAPPAHGVT-NH_2
2	5-oxo-hexanoyl-PEG-GVT(GalNAc α 1→)SAPDTRPAPGSTAPPAHGVT-NH_2
3	5-oxo-hexanoyl-PEG-GVTS(GalNAc α 1→)APDTRPAPGSTAPPAHGVT-NH_2
4	5-oxo-hexanoyl-PEG-GVTSAPDT(GalNAc α 1→)RPAPGSTAPPAHGVT-NH_2
5	5-oxo-hexanoyl-PEG-GVTSAPDTRPAPGS(GalNAc α 1→)TAPPAHGVT-NH_2
6	5-oxo-hexanoyl-PEG-GVTSAPDTRPAPGST(GalNAc α 1→)APPAHGVT-NH_2
7	5-oxo-hexanoyl-PEG-GVT(Gal β (1→3)GalNAc α 1→)SAPDTRPAPGSTAPPAHGVT-NH_2
8	5-oxo-hexanoyl-PEG-GVTS(Gal β (1→3)GalNAc α 1→)APDTRPAPGSTAPPAHGVT-NH_2
9	5-oxo-hexanoyl-PEG-GVTSAPDT(Gal β (1→3)GalNAc α 1→)RPAPGSTAPPAHGVT-NH_2
10	5-oxo-hexanoyl-PEG-GVTSAPDTRPAPGS(Gal β (1→3)GalNAc α 1→)TAPPAHGVT-NH_2
11	5-oxo-hexanoyl-PEG-GVTSAPDTRPAPGST(Gal β (1→3)GalNAc α 1→)APPAHGVT-NH_2
12	5-oxo-hexanoyl-PEG-GVT(Gal β (1 → 3)[GlcNAc β (1 → 6)]GalNAc α 1 →)SAPDTRPAPGSTAPPAHGVT-NH_2
13	5-oxo-hexanoyl-PEG-GVTS(Gal β (1 → 3)[GlcNAc β (1 → 6)]GalNAc α 1 →)APDTRPAPGSTAPPAHGVT-NH_2
14	5-oxo-hexanoyl-PEG-GVTSAPDT(Gal β (1 → 3)[GlcNAc β (1 → 6)]GalNAc α 1 →)RPAPGSTAPPAHGVT-NH_2

15	5-oxo-hexanoyl-PEG-GVTSAPDTRPAPGS(Gal β (1→3)[GlcNAc β (1→6)]GalNAc α 1→)TAPPAHGVT-NH_2
16	5-oxo-hexanoyl-PEG-GVTSAPDTRPAPGST(Gal β (1→3)[GlcNAc β (1→6)]GalNAc α 1→)APPAHGVT-NH_2
17	5-oxo-hexanoyl-PEG-GVT(GalNAc α 1 →)S(GalNAc α 1 →)APDTRPAPGSTAPPAHGVT-NH_2
18	5-oxo-hexanoyl-PEG-GVT(GalNAc α 1 →)SAPDT(GalNAc α 1 →)RPAPGSTAPPAHGVT-NH_2
19	5-oxo-hexanoyl-PEG-GVT(GalNAc α 1 →)SAPDTRPAPGST(GalNAc α 1 →)APPAHGVT-NH_2
20	5-oxo-hexanoyl-PEG-GVTS(GalNAc α 1 →)APDT(GalNAc α 1 →)RPAPGSTAPPAHGVT-NH_2
21	5-oxo-hexanoyl-PEG-GVTS(GalNAc α 1 →)APDTRPAPGST(GalNAc α 1 →)APPAHGVT-NH_2
22	5-oxo-hexanoyl-PEG-GVTSAPDT(GalNAc α 1 →)RPAPGST(GalNAc α 1 →)APPAHGVT-NH_2
23	5-oxo-hexanoyl-PEG-GVTSAPDTRPAPGS(GalNAc α 1 →)T(GalNAc α 1 →)APPAHGVT-NH_2
24	5-oxo-hexanoyl-PEG-GVTS(GalNAc α 1→)APDT(GalNAc α 1→)RPAPGS(GalNAc α 1→)TAPPAHGVT-NH_2
25	5-oxo-hexanoyl-PEG-GVTS(GalNAc α 1 →)APDT(Gal β (1 → 3)GalNAc α 1 →)RPAPGS(GalNAc α 1→)TAPPAHGVT-NH_2
26	5-oxo-hexanoyl-PEG-T(GalNAc α 1→)-NH_2
27	5-oxo-hexanoyl-PEG-S(GalNAc α 1→)-NH_2
28	5-oxo-hexanoyl-PEG-T(Gal β (1→3)GalNAc α 1→)-NH_2
29	5-oxo-hexanoyl-PEG-S(Gal β (1→3)GalNAc α 1→)-NH_2
30	5-oxo-hexanoyl-PEG-T(Gal β (1→3)[GlcNAc β (1→6)]GalNAc α 1→)-NH_2

31	5-oxo-hexanoyl-PEG-S(Gal β (1→3)[GlcNAc β (1→6)]GalNAc α 1→)-NH_2
32	5-oxo-hexanoyl-PPAHGVTSAPDTRPAPGSTA-NH_2
33	5-oxo-hexanoyl-PPAHGVTSAPDT(Neu5Ac α (2 → 3)Gal β (1 → 3)GalNAc α 1 →)RPAPGSTA-NH_2
34	5-oxo-hexanoyl-PPAHGVT(GalNAc α 1→)S(Gal β (1→3)[GlcNAc β (1→6)[Neu5Ac α (2→3)Gal β (1→3)]GalNAc α →)APDT(Neu5Ac α (2→3)Gal β (1→3)GalNAc α 1→)RPAPGS(Gal β (1→3)[GlcNAc β (1→6)[Neu5Ac α (2→3)Gal β (1→3)]GalNAc α →)T(GalNAc α 1→)A-NH_2
35	5-oxo-hexanoyl-PPAHGVTS(Gal β (1→3)[GlcNAc β (1→6)[Neu5Ac α (2→3)Gal β (1 →3)]GalNAc α →)APDT(Neu5Ac α (2→3)Gal β (1→3)GalNAc α 1→)RPAPGS(Gal β (1→3)[GlcNAc β (1→6)[Neu5Ac α (2→3)Gal β (1→3)]GalNAc α →)TA-NH_2

Synthesis and characterization of compounds **1**, **3**, **4**, **20**, **24**, and **25**;[1] **2**, **5**, and **6**;[2] **32**-**35**.[3]

[1] F. Garcia-Martin, T. Matsushita, H. Hinou and S.-I. Nishimura, *Chemistry-a European Journal* **2014**, *20*, 15891-15902.

[2] H. Coelho, T. Matsushita, G. Artigas, H. Hinou, F. J. Canada, R. Lo-Man, C. Leclerc, E. J. Cabrita, J. Jimenez-Barbero, S.-I. Nishimura, F. Garcia-Martin and F. Marcelo, *Journal of the American Chemical Society* **2015**, *137*, 12438-12441.

[3] S. Rangappa, G. Artigas, R. Miyoshi, Y. Yokoi, S. Hayakawa, F. Garcia-Martin, H. Hinou and S.-I. Nishimura, *Medchemcomm* **2016**, *7*, 1102-1122.

Table S2. Grades of fluorescence intensity* for compound **1**~**35** after treatment with galectins and galectin related proteins (90 μg/mL) tested in this study.

#	Chimera-type		tandem-repeat-type				Proto-type			
	Gal-3	Gal-3tr	Gal-4	Gal-4N	Gal-4C	Gal-8	Gal-1	GRIFIN	Gal-5	GRP
1				+						+
2										
3										
4										+
5					+			+		+
6				+	+					
7			+	+	+					
8			++++	+	+	+++		+++		
9			+++	+	+			+		+
10			++++++	++	++	++++++		+++	+	
11			++++++	+++	++	+++++		+	+	
12				+		+				
13				+						
14										
15			+++	++	+	++		+		
16				+		+				
17				+						
18										
19				+	+					
20										
21				+++	+					
22					+					
23				+	+					
24										
25			+	+	+					
26										
27										
28			+++++		+					
29			++++	+	+	+		+		
30			+							
31			+++++		+	+		++		
32										
33			+	+		+++++				+
34	+			++		++++++				
35	+++			+++		++++++				

*Each grade are assigned from average value of relative fluorescence intensity (RFU) of the four 100 mM spots for each compound on microarray; + 0.50-1.49 (x 10^{-7}), ++ 1.50-2.49 (x 10^{-7}), +++ 2.50-3.49 (x 10^{-7}), ++++ 3.50-4.49 (x 10^{-7}), +++++ 4.50-5.49 (x 10^{-7}), ++++++ > 5.50 (x 10^{-7}).

Characterization of peptides/glycopeptides

General procedure for the UPLC analyses of purified glycopeptides

All measurements were performed using a Waters Acquity Ultra Performance LC system equipped with binary solvent delivery pump, an auto sampler and a UV detector. Synthetic peptides and glycopeptides purified by semi-preparative RP-HPLC were analyzed on an Acquity UPLC BEN®C18 column (1.7 μm, 2.1 × 50 mm, Waters) at a flow rate of 0.2 mL/min. All separations were monitored at 220 nm. Column temperature was 40 °C. A gradient of water with 0.1% TFA (eluent A) and acetonitrile with 0.1% TFA (eluent B) were used for the mobile phase. The ratio of B was linearly increased from 2 to 30% in 12.6 min, then the column was washed with 90% B and equilibrium with 2% B. MALDI-TOF MS data were recorded by Ultraflex I (Bruker Daltnics) using DHB [10 mg in 1 mL of 50% acetonitrile with 0.1% TFA (1:1 mixture of eluent A and B for RP-HPLC).

Compound 7. Analytical UPLC: t_R = 7.269 min, peak area ratio 100%. MALDI-TOFMS: $C_{117}H_{189}N_{31}O_{46}$ $[M+H]^+$ calcd (m/z) 2765.3403, found (m/z) 2765.1069. Amino acid ratios (numbers in parenthesis are theorical values): Asp (1) 1.0, Thr (4) 3.7, Ser (2) 1.8, Gly (3) 3.0, Ala (4) 4.1, Val (2) 1.9, His (1) 1.0, Arg (1) 1.0, Pro (5) 5.4.

Compound 8. Analytical UPLC: t_R = 7.624 min, peak area ratio 100%. MALDI-TOFMS: $C_{117}H_{189}N_{31}O_{46}$ $[M+H]^+$ calcd (m/z) 2765.3403, found (m/z) 2765.0138. Amino acid ratios (numbers in parenthesis are theorical values): Asp (1) 1.0, Thr (4) 3.8, Ser (2) 1.8, Gly (3) 3.0, Ala (4) 4.1, Val (2) 2.0, His (1) 1.0, Arg (1) 1.0, Pro (5) 5.3.

Compound 9. Analytical UPLC: t_R = 7.504 min, peak area ratio 100%. MALDI-TOFMS: $C_{117}H_{189}N_{31}O_{46}$ $[M+H]^+$ calcd (m/z) 2765.3403, found (m/z) 2764.9759. Amino acid ratios (numbers in parenthesis are theorical values): Asp (1) 1.0, Thr (4) 3.8, Ser (2) 1.8, Gly (3) 3.0, Ala (4) 4.0, Val (2) 1.9, His (1) 1.1, Arg (1) 1.0, Pro (5) 5.4.

Compound 10. Analytical UPLC: t_R = 7.616 min, peak area ratio 100%. MALDI-TOF-HRMS: $C_{117}H_{189}N_{31}O_{46}$ $[M+H]^+$ calcd (m/z) 2765.3403, found (m/z) 2765.1383. Amino acid ratios (numbers in parenthesis are theorical values): Asp (1) 1.0, Thr (4) 3.7, Ser (2) 1.8, Gly (3) 3.0, Ala (4) 4.1, Val (2) 1.9, His (1) 1.1, Arg (1) 1.0, Pro (5) 5.4.

Compound 11. Analytical UPLC: t_R = 7.538 min, peak area ratio 100%. MALDI-TOFMS: $C_{117}H_{189}N_{31}O_{46}$ $[M+H]^+$ calcd (m/z) 2765.3403, found (m/z) 2765.2273. Amino acid ratios (numbers in parenthesis are theorical values): Asp (1) 1.0, Thr (4) 3.7, Ser (2) 1.8, Gly (3) 3.0, Ala (4) 4.1, Val (2) 1.9, His (1) 1.1, Arg (1) 1.0, Pro (5) 5.4.

Compound 12. Analytical UPLC: t_R = 7.088 min, peak area ratio 98.4%. MALDI-TOFMS: $C_{117}H_{189}N_{31}O_{46}$ $[M+H]^+$ calcd (m/z) 2968.4197, found (m/z) 2967.9384. Amino acid ratios (numbers in parenthesis are theorical values): Asp (1) 1.0, Thr (4) 3.8, Ser (2) 1.8, Gly (3) 3.0, Ala (4) 4.1, Val (2) 2.0, His (1) 1.0, Arg (1) 1.0, Pro (5) 5.2.

Compound 13. Analytical UPLC: t_R = 7.431 min, peak area ratio 100%. MALDI-TOFMS: $C_{117}H_{189}N_{31}O_{46}$ $[M+H]^+$ calcd (m/z) 2968.4197, found (m/z) 2968.4793. Amino acid ratios (numbers in parenthesis are theorical values): Asp (1) 1.0, Thr (4) 3.8, Ser (2) 1.8, Gly (3) 3.0, Ala (4) 4.2, Val (2) 2.0, His (1) 1.0, Arg (1) 1.0, Pro (5) 5.2.

Compound 14. Analytical UPLC: t_R = 7.229 min, peak area ratio 100%. MALDI-TOFMS: $C_{117}H_{189}N_{31}O_{46}$ $[M+H]^+$ calcd (m/z) 2968.4197, found (m/z) 2967.9460. Amino acid ratios (numbers in parenthesis are theorical values): Asp (1) 1.0, Thr (4) 3.8, Ser (2) 1.8, Gly (3) 3.1, Ala (4) 4.1, Val (2) 2.0, His (1) 1.1, Arg (1) 1.0, Pro (5) 5.2.

Compound 15. Analytical UPLC: t_R = 7.441 min, peak area ratio 98.5%. MALDI-TOFMS: $C_{117}H_{189}N_{31}O_{46}$ $[M+H]^+$ calcd (m/z) 2968.4197, found (m/z) 2968.0959. Amino acid ratios (numbers in parenthesis are theorical values): Asp (1) 1.0, Thr (4) 3.8, Ser (2)

1.8, Gly (3) 3.0, Ala (4) 4.1, Val (2) 2.0, His (1) 1.1, Arg (1) 1.0, Pro (5) 5.2.

Compound 16. Analytical UPLC: t_R = 7.341 min, peak area ratio 100%. MALDI-TOFMS: $C_{117}H_{189}N_{31}O_{46}$ $[M+H]^+$ calcd (m/z) 2968.4197, found (m/z) 2968.3110. Amino acid ratios (numbers in parenthesis are theorical values): Asp (1) 1.0, Thr (4) 3.8, Ser (2) 1.8, Gly (3) 3.0, Ala (4) 4.1, Val (2) 2.0, His (1) 1.1, Arg (1) 1.0, Pro (5) 5.3.

Compound 17. Analytical UPLC: t_R = 7.305 min, peak area ratio 100%. MALDI-TOFMS: $C_{119}H_{192}N_{32}O_{46}$ $[M+H]^+$ calcd (m/z) 2806.3668, found (m/z) 2806.0823 Amino acid ratios (numbers in parenthesis are theorical values): Asp (1) 1.0, Thr (4) 3.7, Ser (2) 1.8, Gly (3) 3.0, Ala (4) 4.1, Val (2) 2.0, His (1) 1.0, Arg (1) 1.0, Pro (5) 5.3.

Compound 18. Analytical UPLC: t_R = 7.363 min, peak area ratio 100%. MALDI-TOFMS: $C_{119}H_{192}N_{32}O_{46}$ $[M+H]^+$ calcd (m/z) 2806.3668, found (m/z) 2805.9321. Amino acid ratios (numbers in parenthesis are theorical values): Asp (1) 1.0, Thr (4) 3.7, Ser (2) 1.8, Gly (3) 3.0, Ala (4) 4.1, Val (2) 2.0, His (1) 1.0, Arg (1) 1.0, Pro (5) 5.4.

Compound 19. Analytical UPLC: t_R = 7.355 min, peak area ratio 100%. MALDI-TOFMS: $C_{119}H_{192}N_{32}O_{46}$ $[M+H]^+$ calcd (m/z) 2806.3668, found (m/z) 2806.1062. Amino acid ratios (numbers in parenthesis are theorical values): Asp (1) 1.0, Thr (4) 3.7, Ser (2) 1.8, Gly (3) 3.0, Ala (4) 4.1, Val (2) 1.9, His (1) 1.0, Arg (1) 1.0, Pro (5) 5.4.

Compound 21. Analytical UPLC: t_R = 7.591 min, peak area ratio 100%. MALDI-TOFMS: $C_{119}H_{192}N_{32}O_{46}$ $[M+H]^+$ calcd (m/z) 2806.3668, found (m/z) 2806.1647. Amino acid ratios (numbers in parenthesis are theorical values): Asp (1) 1.0, Thr (4) 3.7, Ser (2) 1.8, Gly (3) 3.0, Ala (4) 4.1, Val (2) 2.0, His (1) 1.0, Arg (1) 1.0, Pro (5) 5.4.

Compound 22. Analytical UPLC: t_R = 7.718 min, peak area ratio 99.6%. MALDI-TOFMS: $C_{119}H_{192}N_{32}O_{46}$ $[M+H]^+$ calcd (m/z) 2806.3668, found (m/z) 2806.4264. Amino

acid ratios (numbers in parenthesis are theorical values): Asp (1) 1.0, Thr (4) 3.8, Ser (2) 1.8, Gly (3) 3.0, Ala (4) 4.1, Val (2) 1.9, His (1) 1.1, Arg (1) 1.0, Pro (5) 5.4.

Compound 23. Analytical UPLC: t_R = 7.484 min, peak area ratio 100%. MALDI-TOFMS: $C_{119}H_{192}N_{32}O_{46}$ $[M+H]^+$ calcd (m/z) 2806.3668, found (m/z) 2806.0616. Amino acid ratios (numbers in parenthesis are theorical values): Asp (1) 1.0, Thr (4) 3.7, Ser (2) 1.8, Gly (3) 3.0, Ala (4) 4.1, Val (2) 2.0, His (1) 1.0, Arg (1) 1.0, Pro (5) 5.4.

Compound 26. Analytical UPLC: t_R = 4.014 min, peak area ratio 100%. MALDI-TOFMS: $C_{24}H_{42}N_4O_{12}$ $[M+H]^+$ calcd (m/z) 578.2799, $[M+Na]^+$ found (m/z) 601.8256. Amino acid ratios (numbers in parenthesis are theorical values): Thr (1) 1.0.

Compound 27. Analytical UPLC: t_R = 3.654 min, peak area ratio 100%. MALDI-TOFMS: $C_{23}H_{40}N_4O_{12}$ $[M+H]^+$ calcd (m/z) 564.2643, $[M+Na]^+$ found (m/z) 587.7875. Amino acid ratios (numbers in parenthesis are theorical values): Ser (1) 1.0.

Compound 28. Analytical UPLC: t_R = 3.748 min, peak area ratio 100%. MALDI-TOFMS: $C_{30}H_{52}N_4O_{17}$ $[M+H]^+$ calcd (m/z) 740.3327, $[M+Na]^+$ found (m/z) 763.1747. Amino acid ratios (numbers in parenthesis are theorical values): Thr (1) 1.0.

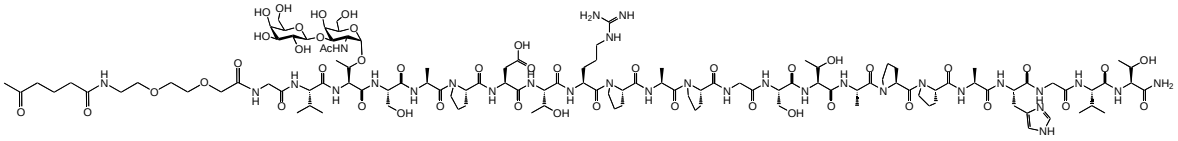
Compound 29. Analytical UPLC: t_R = 3. min, peak area ratio 100%. MALDI-TOFMS: $C_{29}H_{50}N_4O_{17}$ $[M+H]^+$ calcd (m/z) 726.3171, $[M+Na]^+$ found (m/z) 749.1237. Amino acid ratios (numbers in parenthesis are theorical values): Ser (1) 1.0.

Compound 30. Analytical UPLC: t_R = 3.382 min, peak area ratio 95.5%. MALDI-TOFMS: $C_{38}H_{65}N_5O_{22}$ $[M+H]^+$ calcd (m/z) 943.4121, $[M+Na]^+$ found (m/z) 966.5864. Amino acid ratios (numbers in parenthesis are theorical values): Thr (1) 1.0.

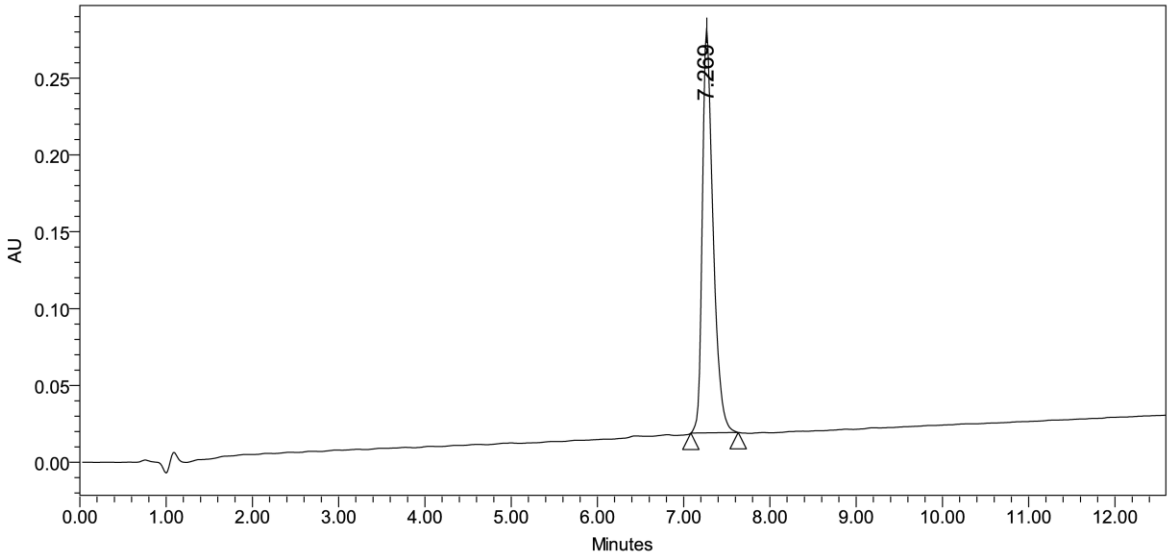
Compound 31. Analytical UPLC: t_R = 3.464 min, peak area ratio 100%. MALDI-TOFMS:

$C_{37}H_{63}N_5O_{22}$ $[M+H]^+$ calcd (m/z) 929.3965, $[M+Na]^+$ found (m/z) 952.2437. Amino acid ratios (numbers in parenthesis are theorical values): Ser (1) 1.0.

Figure S1. (a) UPLC profile and (b) MALDI-TOF MS of **Compound 7.**



(a) UPLC profile



(b) MALDI-TOF MS

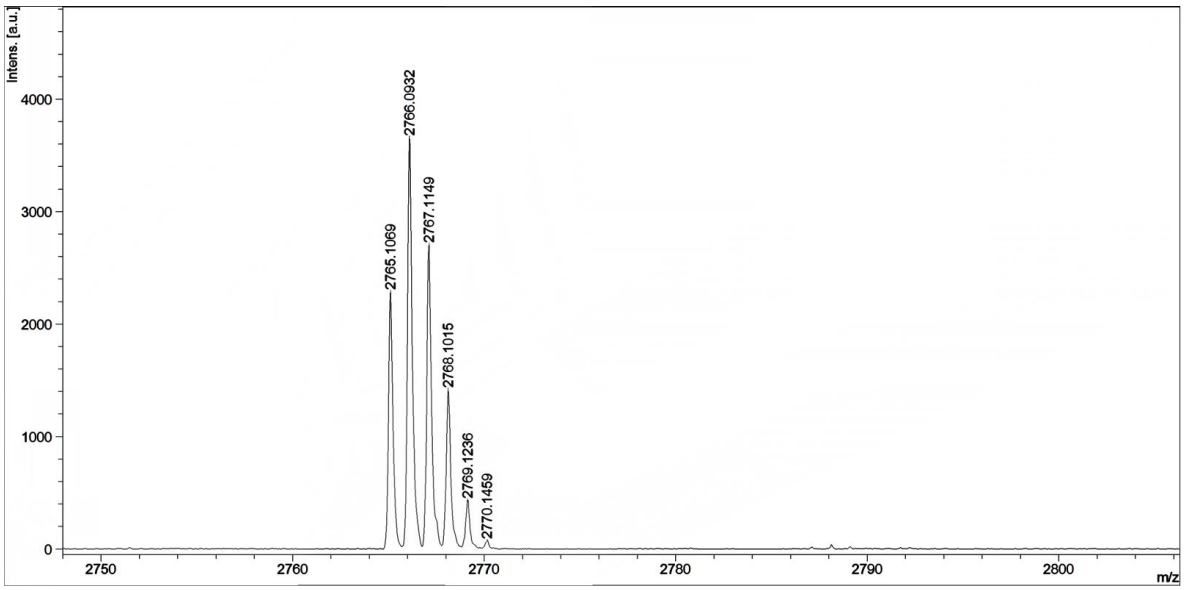
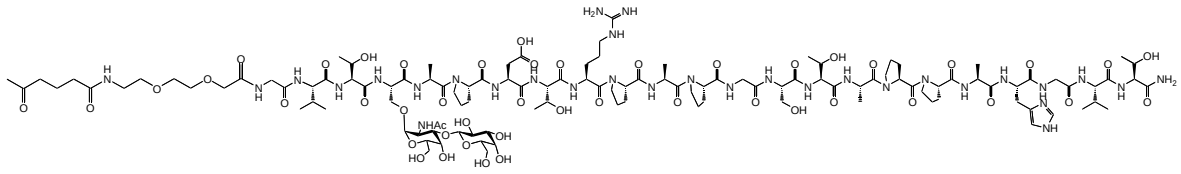
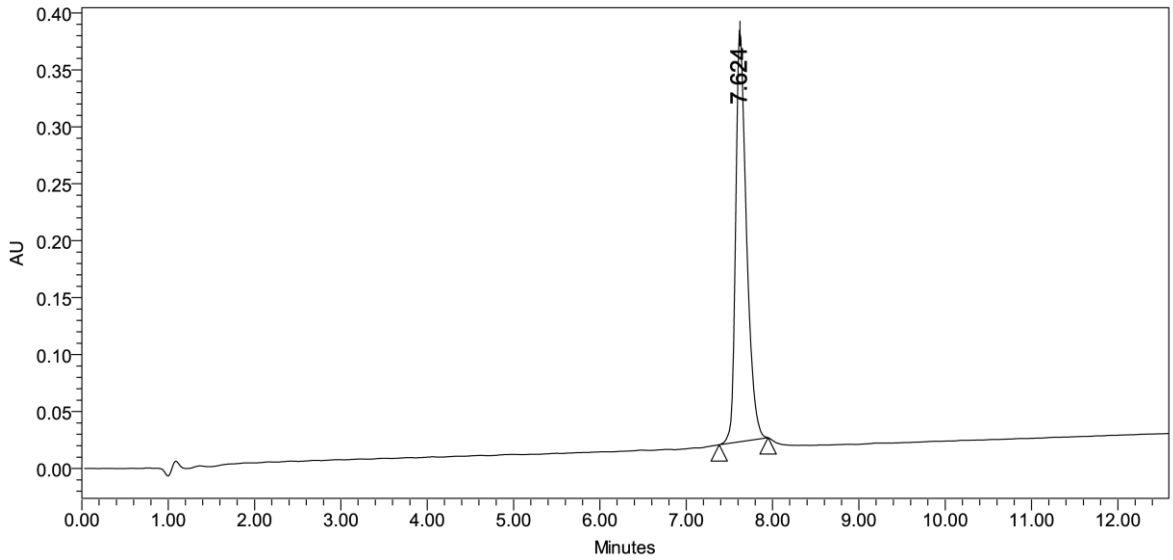


Figure S2. (a) UPLC profile and (b) MALDI-TOF MS of **Compound 8.**



(a) UPLC profile



(b) MALDI-TOF MS

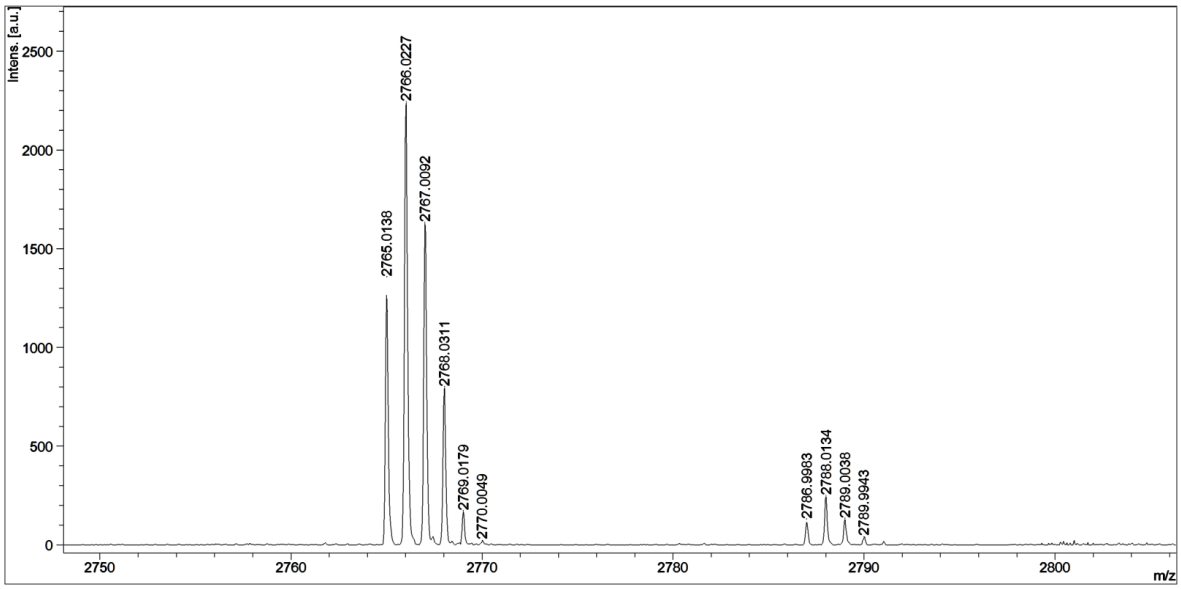
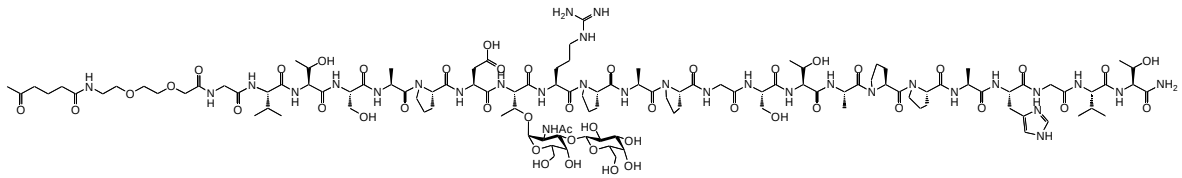
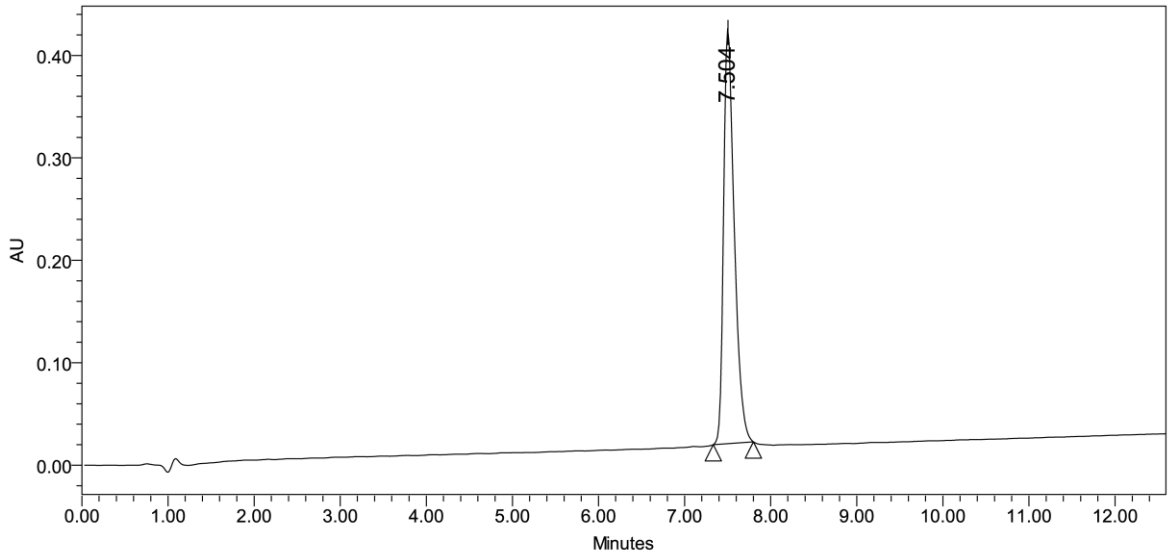


Figure S3. (a) UPLC profile and (b) MALDI-TOF MS of **Compound 9.**



(a) UPLC profile



(b) MALDI-TOF MS

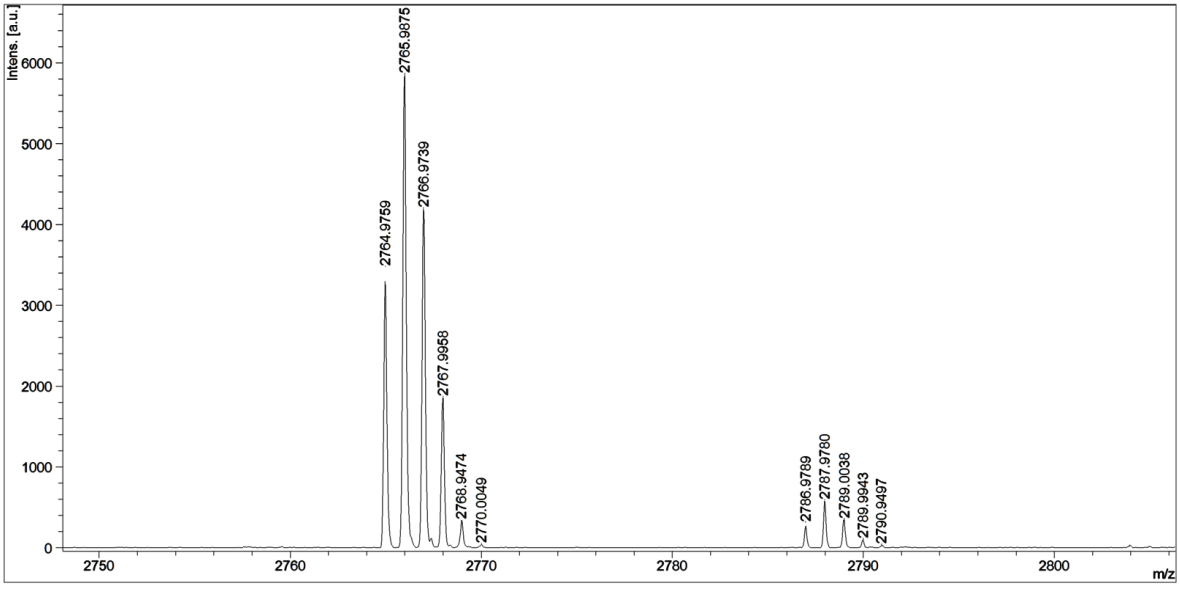
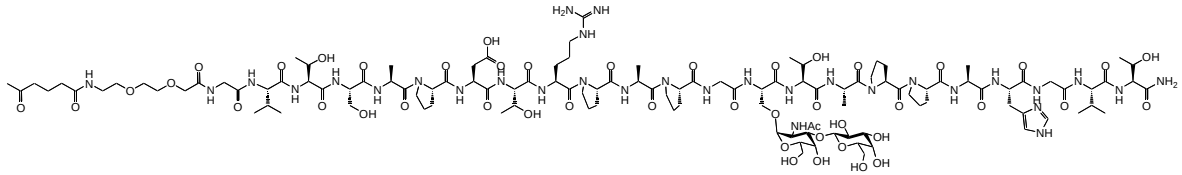
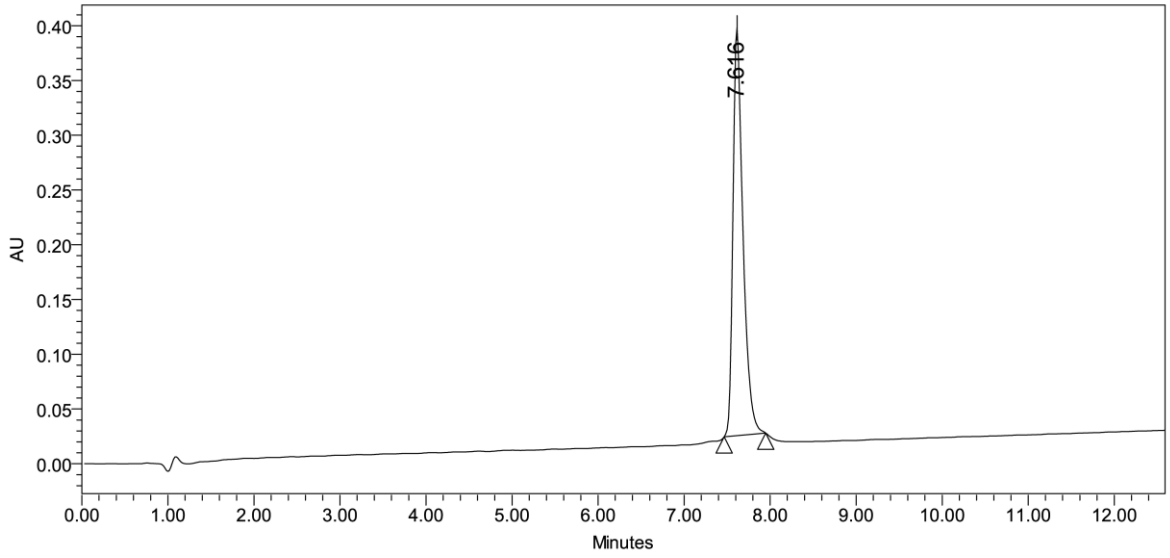


Figure S4. (a) UPLC profile and (b) MALDI-TOF MS of **Compound 10.**



(a) UPLC profile



(b) MALDI-TOF MS

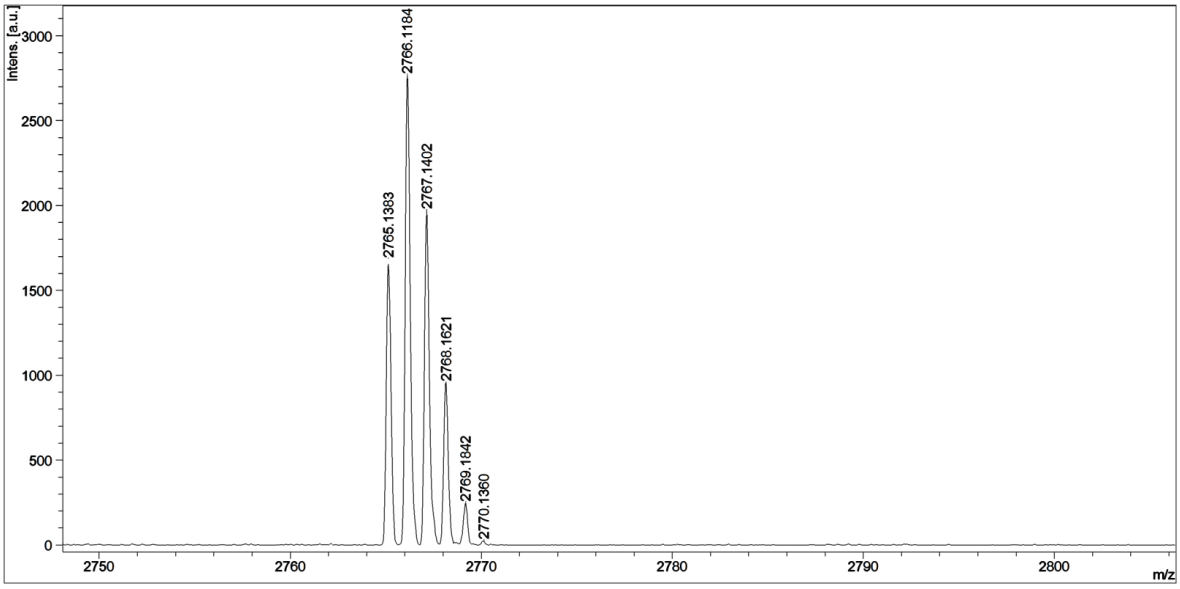
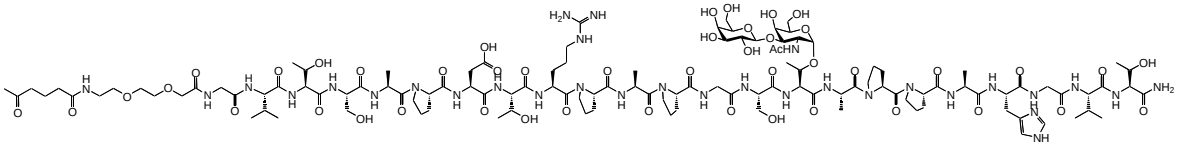
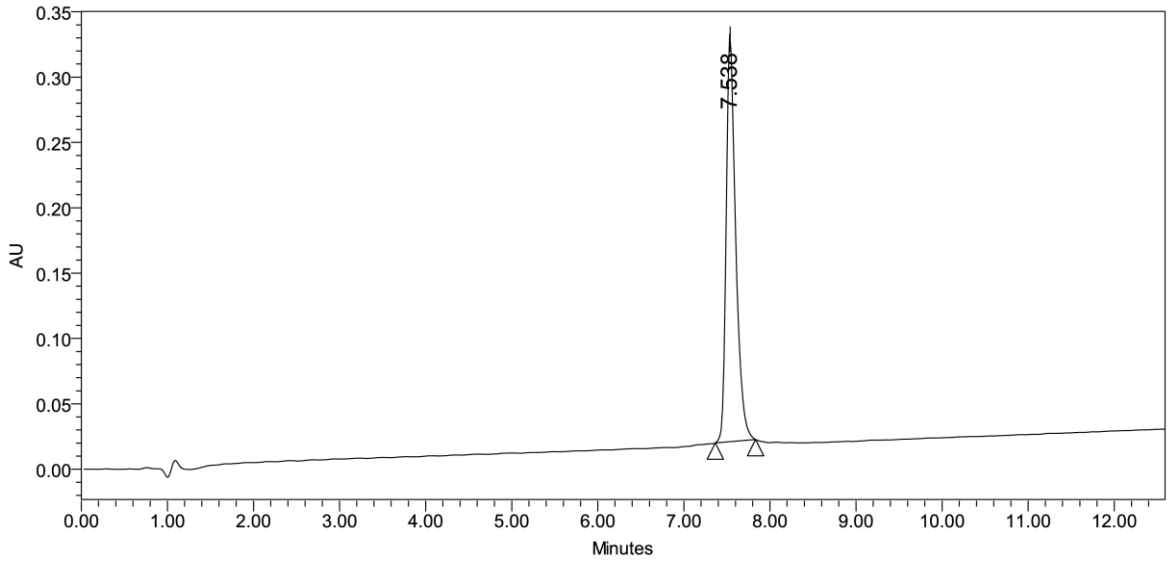


Figure S5. (a) UPLC profile and (b) MALDI-TOF MS of **Compound 11.**



(a) UPLC profile



(b) MALDI-TOF MS

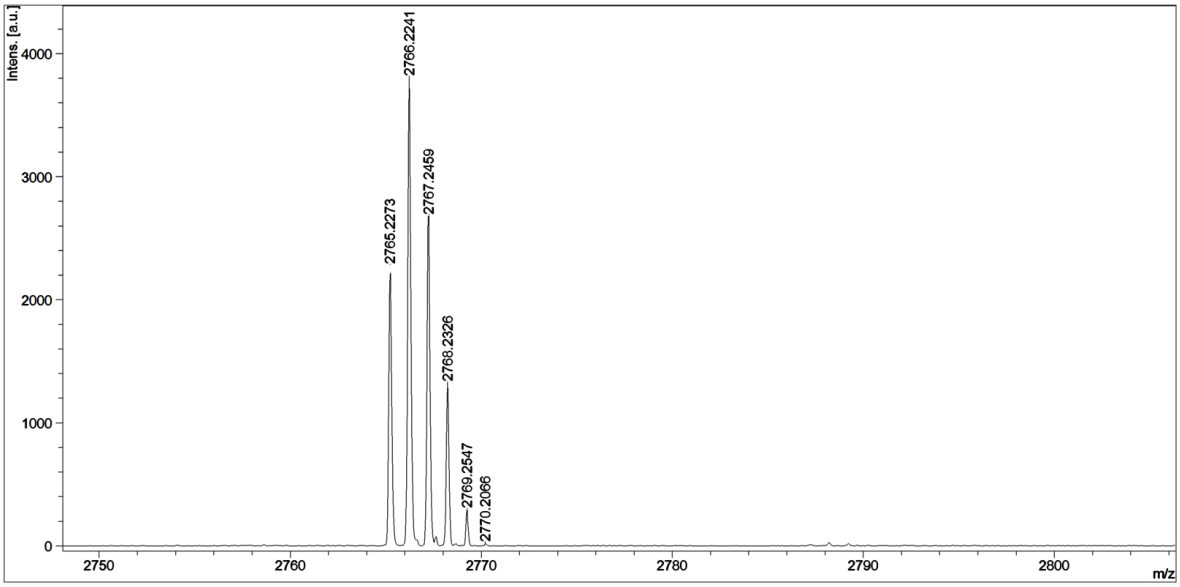
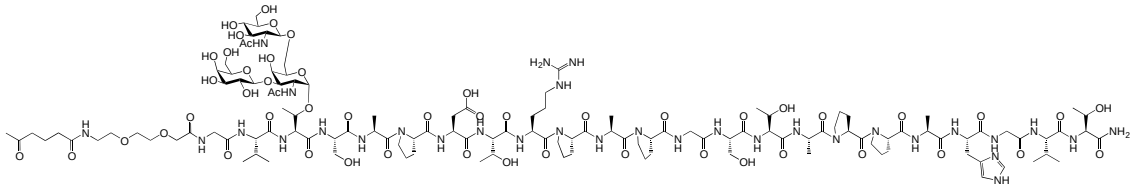
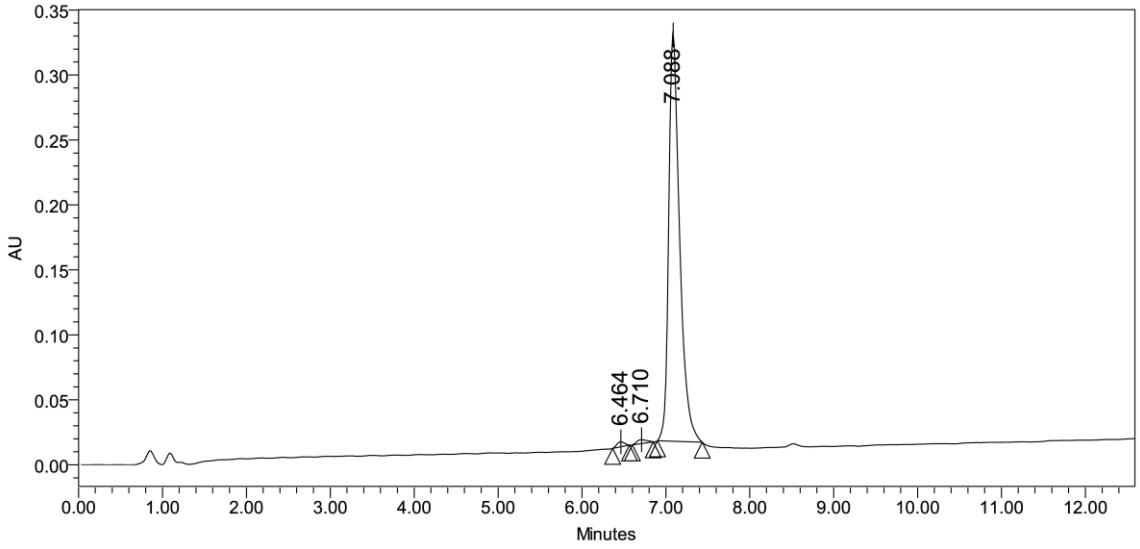


Figure S6. (a) UPLC profile and (b) MALDI-TOF MS of **Compound 12.**



(a) UPLC profile



(b) MALDI-TOF MS

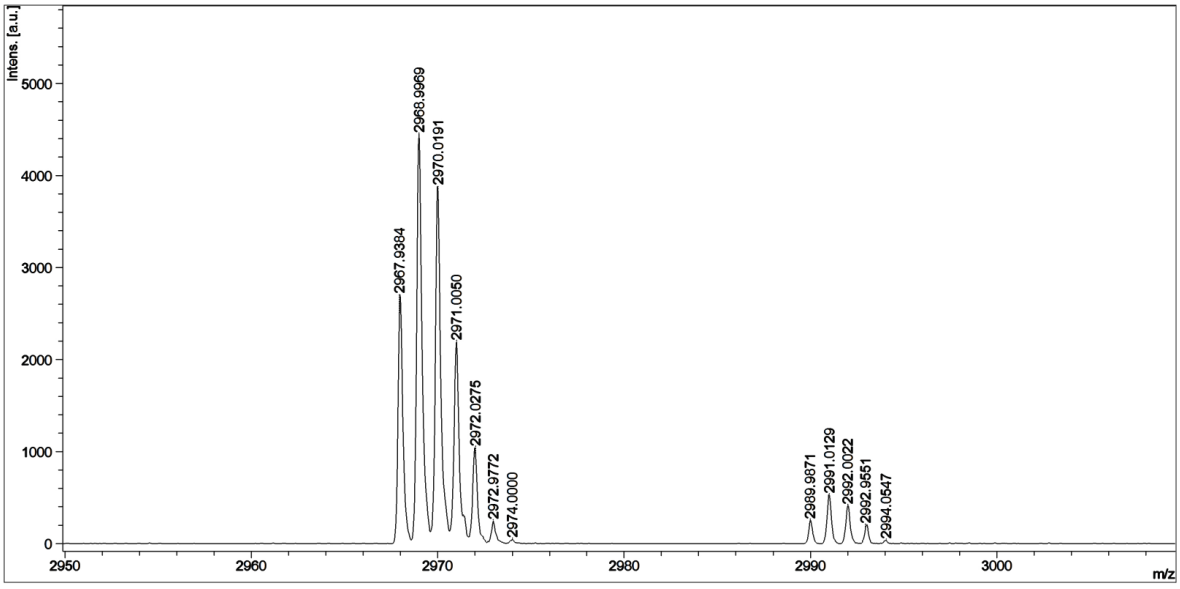
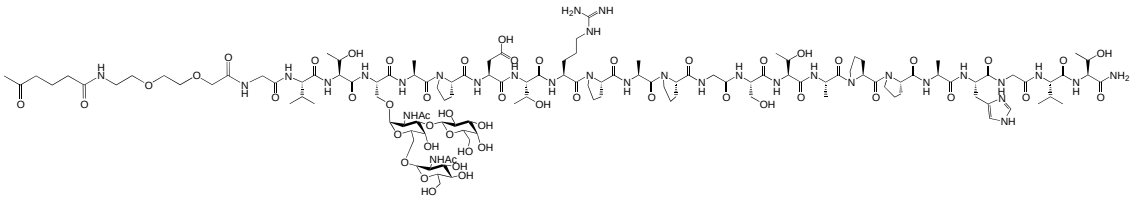
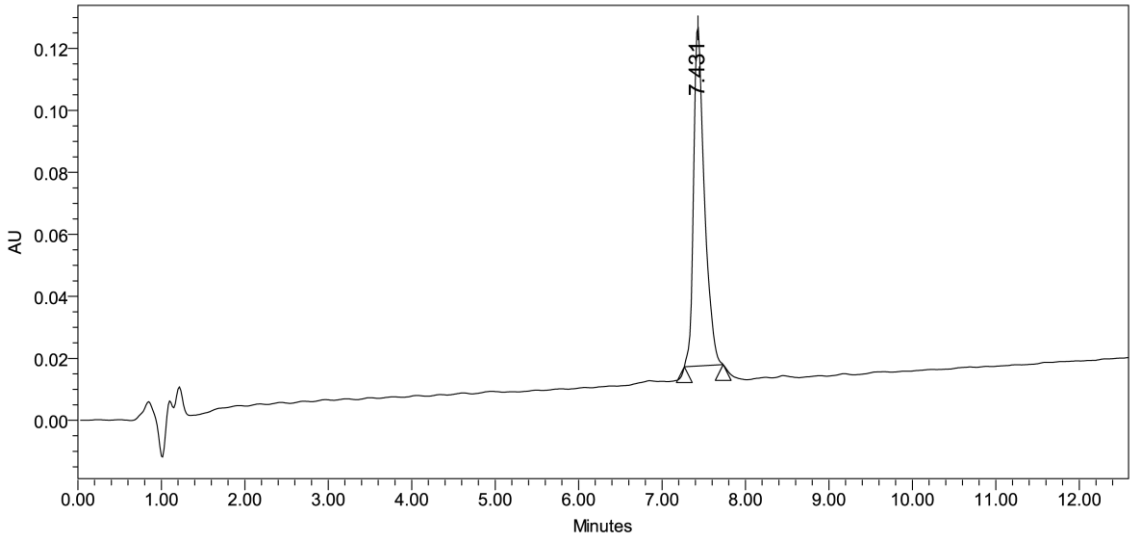


Figure S7. (a) UPLC profile and (b) MALDI-TOF MS of **Compound 13.**



(a) UPLC profile



(b) MALDI-TOF MS

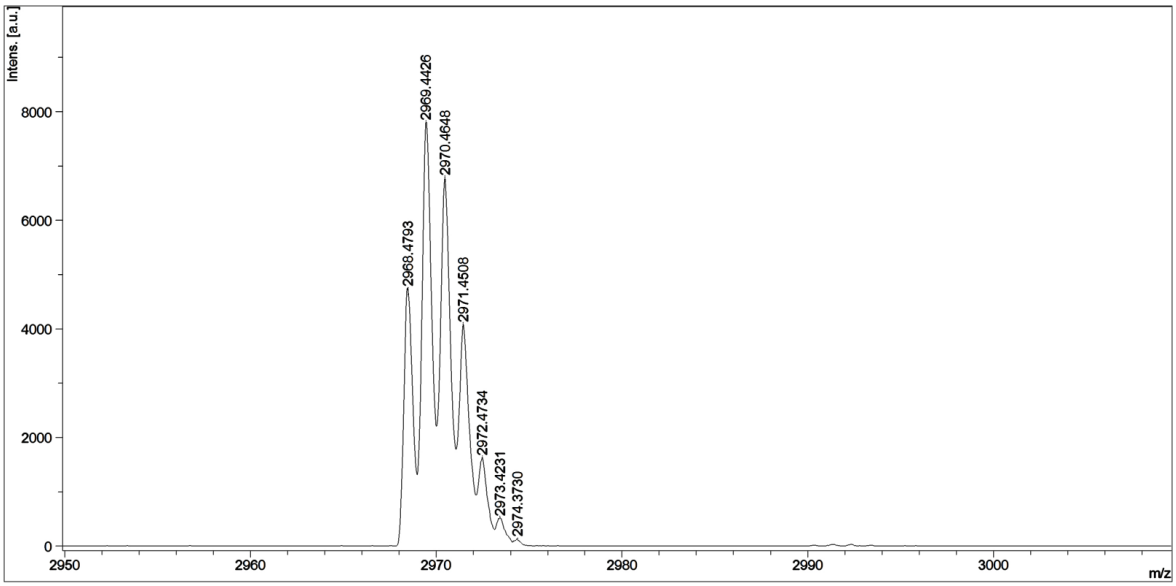
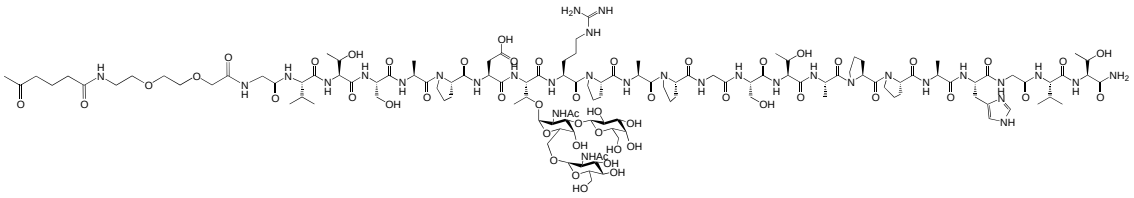
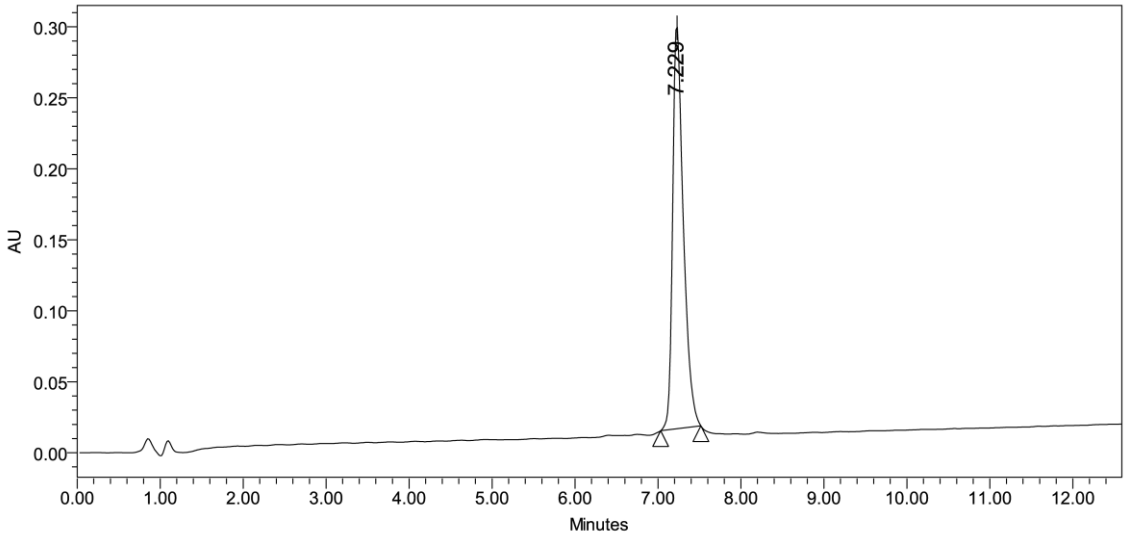


Figure 8. (a) UPLC profile and (b) MALDI-TOF MS of **Compound 14.**



(a) UPLC profile



(b) MALDI-TOF MS

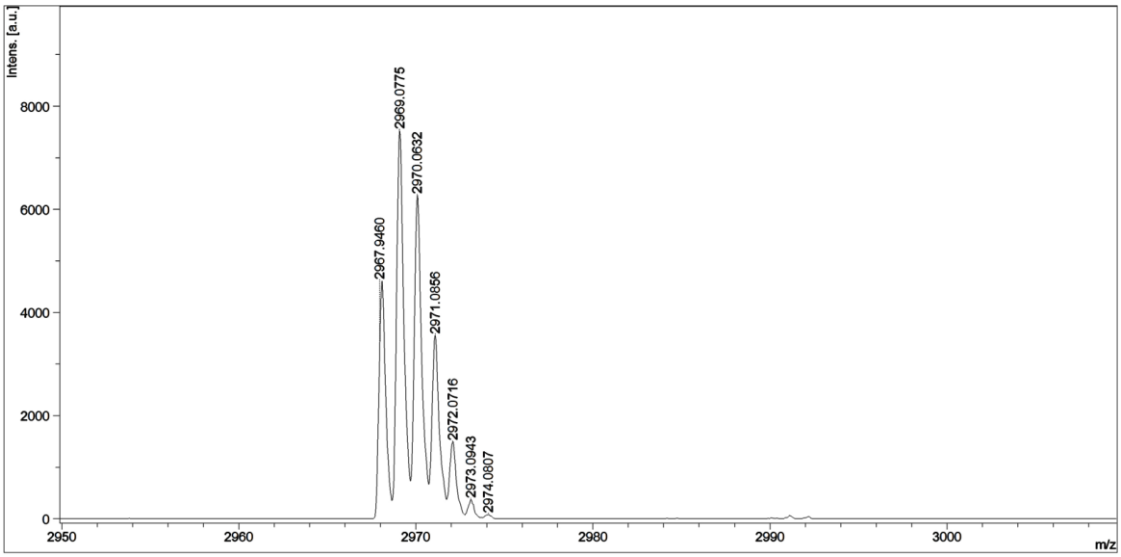
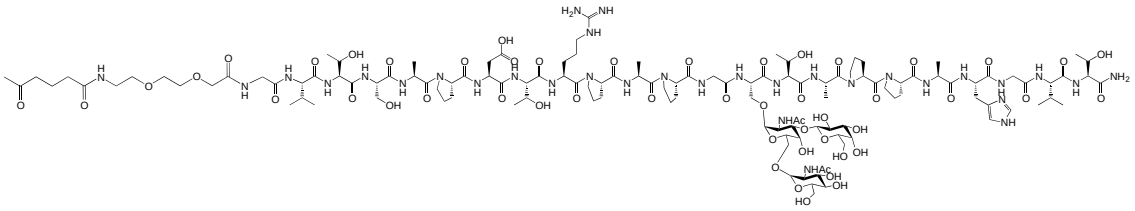
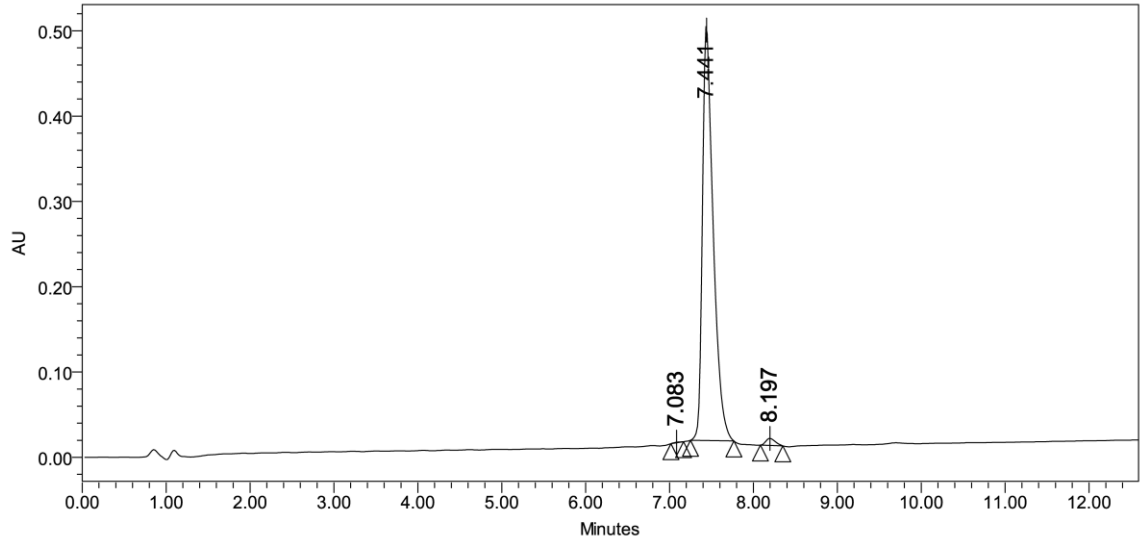


Figure S9. (a) UPLC profile and (b) MALDI-TOF MS of **Compound 15.**



(a) UPLC profile



(b) MALDI-TOF MS

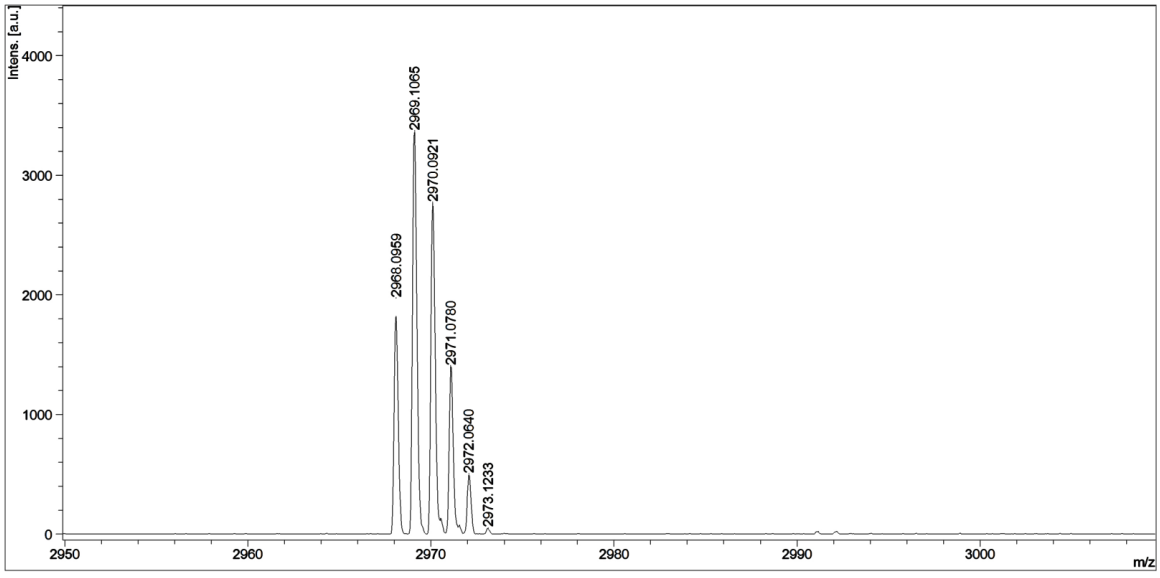
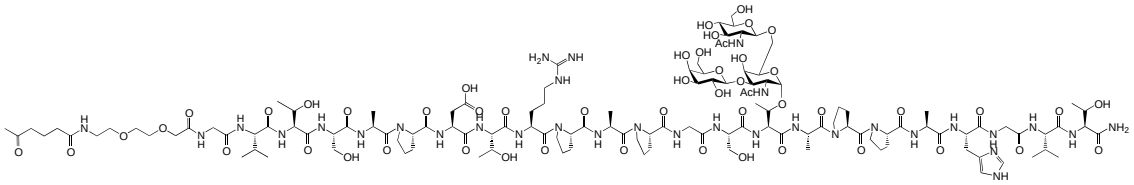
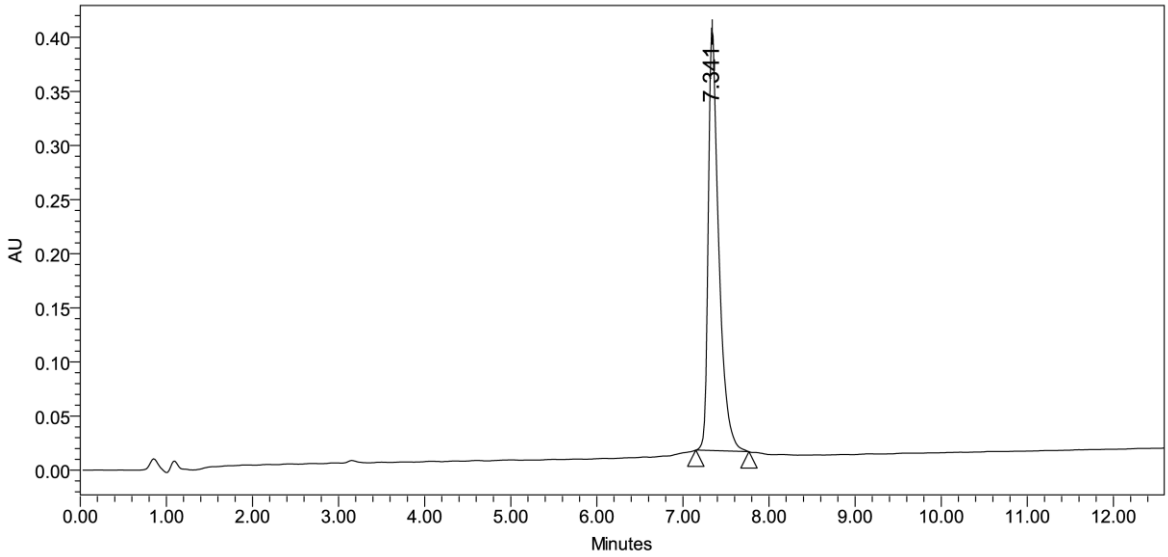


Figure S10. (a) UPLC profile and (b) MALDI-TOF MS of **Compound 16.**



(a) UPLC profile



(b) MALDI-TOF MS

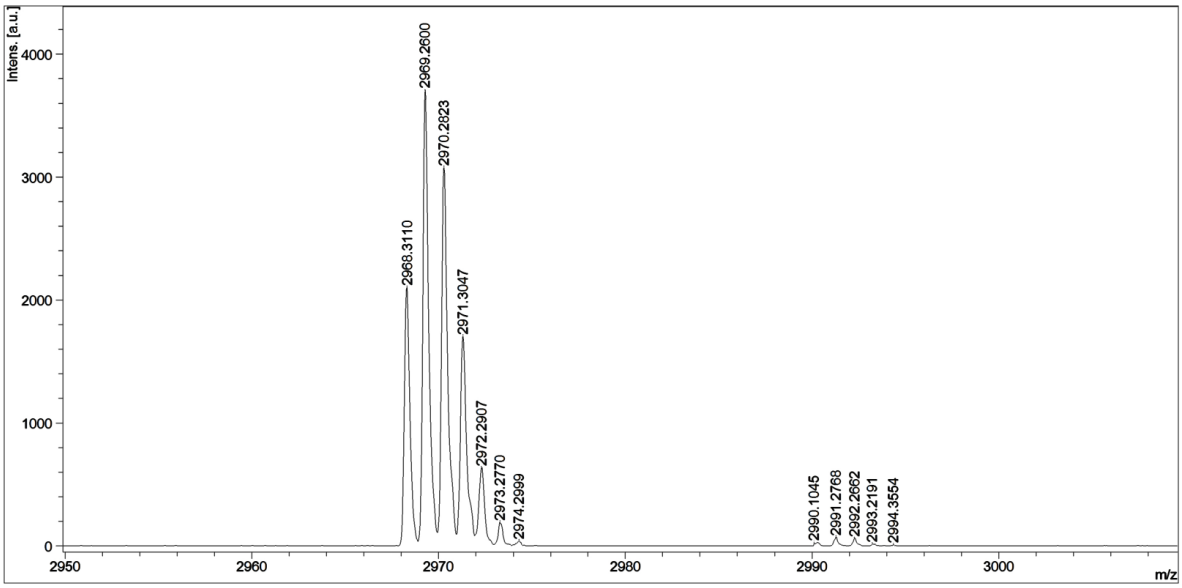
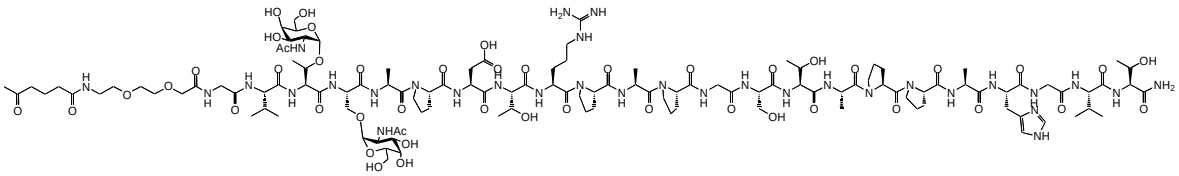
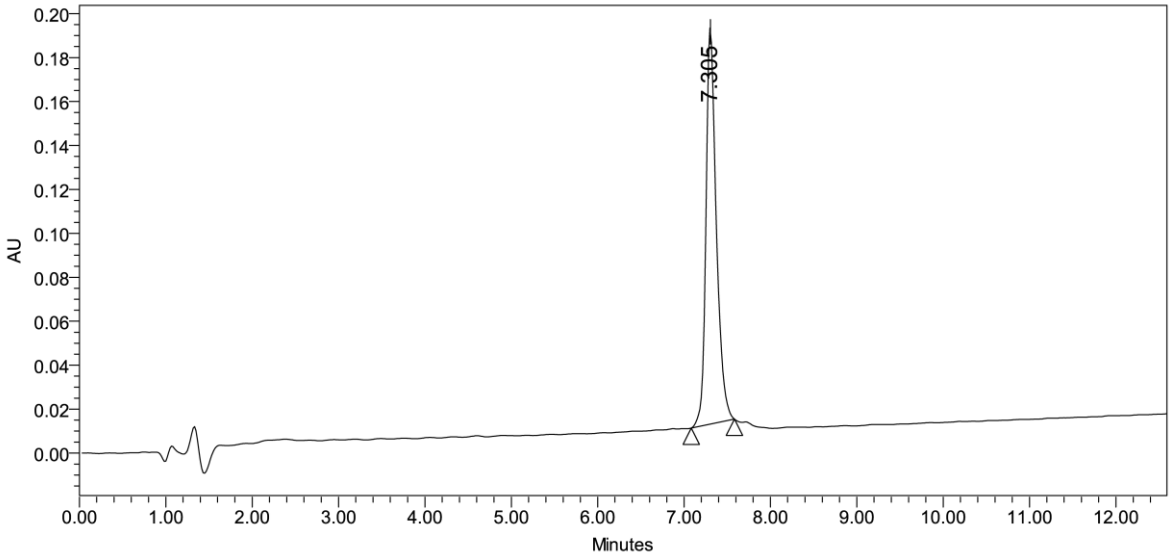


Figure S11. (a) UPLC profile and (b) MALDI-TOF MS of **Compound 17.**



(a) UPLC profile



(b) MALDI-TOF MS

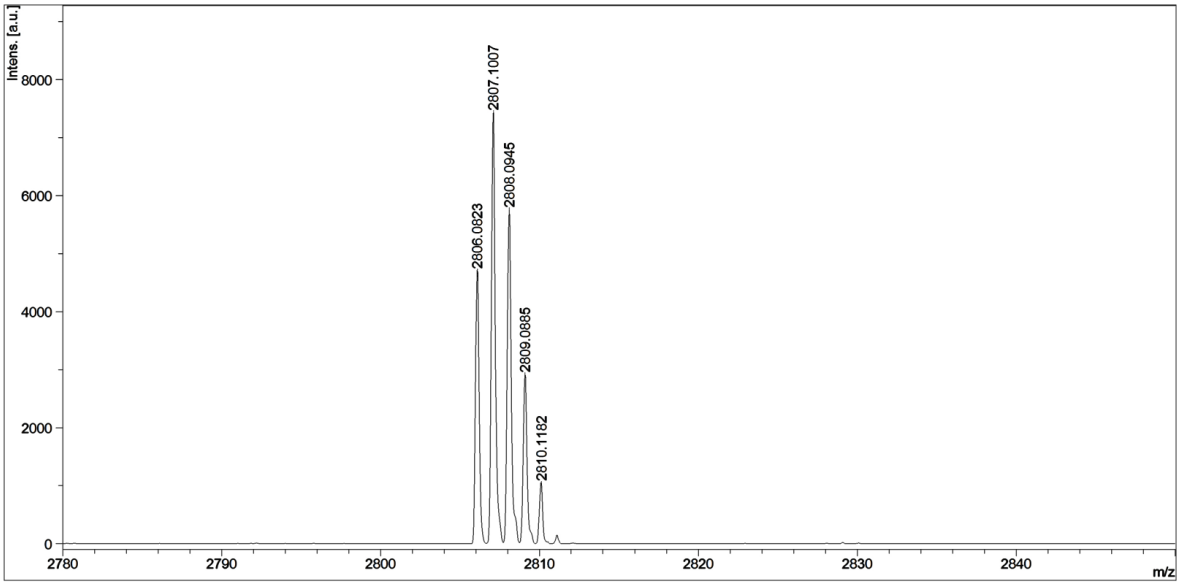
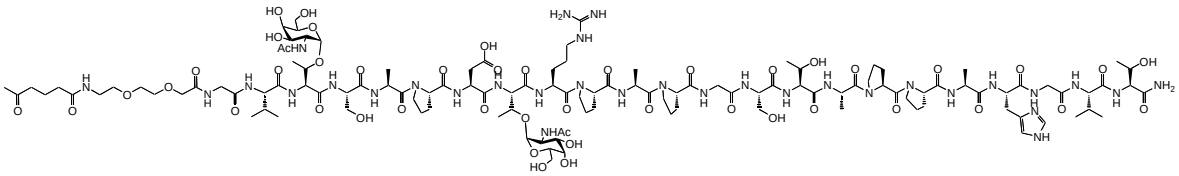
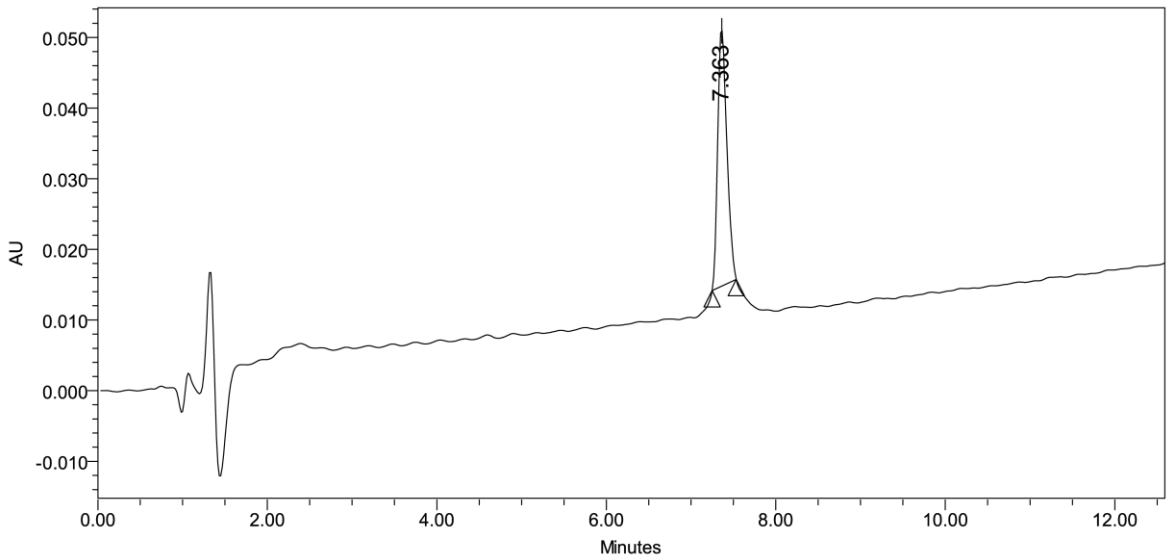


Figure S12. (a) UPLC profile and (b) MALDI-TOF MS of **Compound 18.**



(a) UPLC profile



(b) MALDI-TOF MS

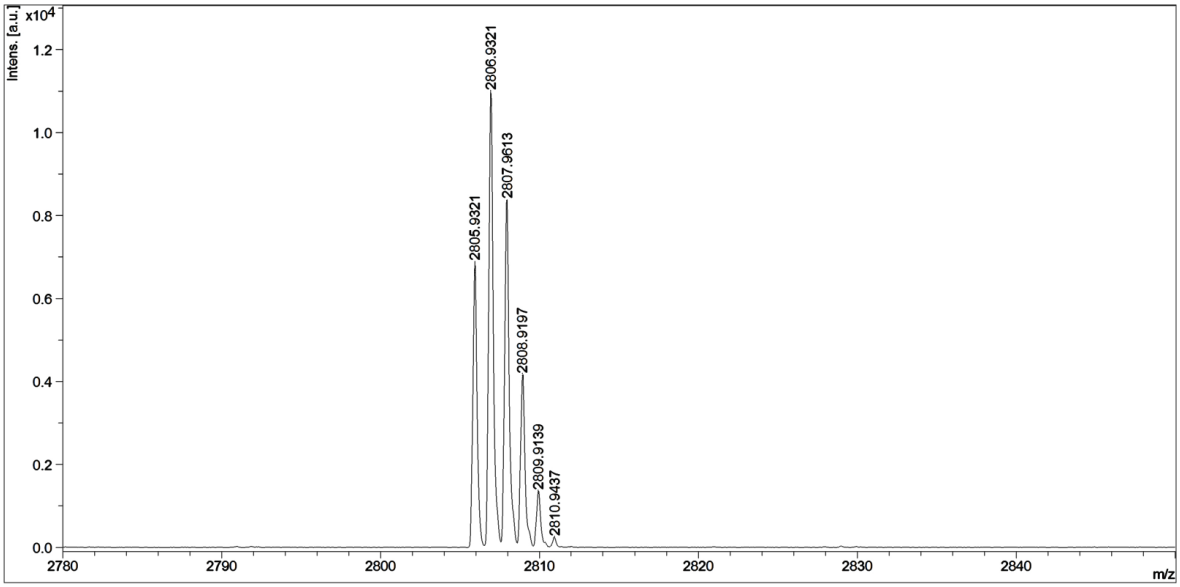
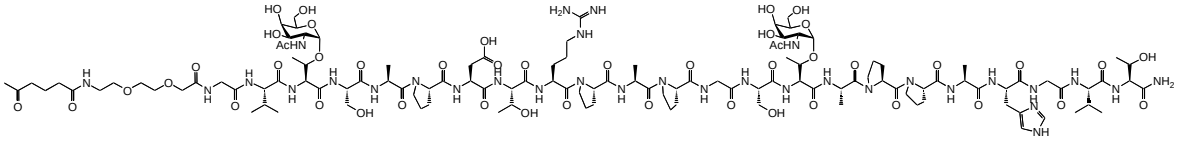
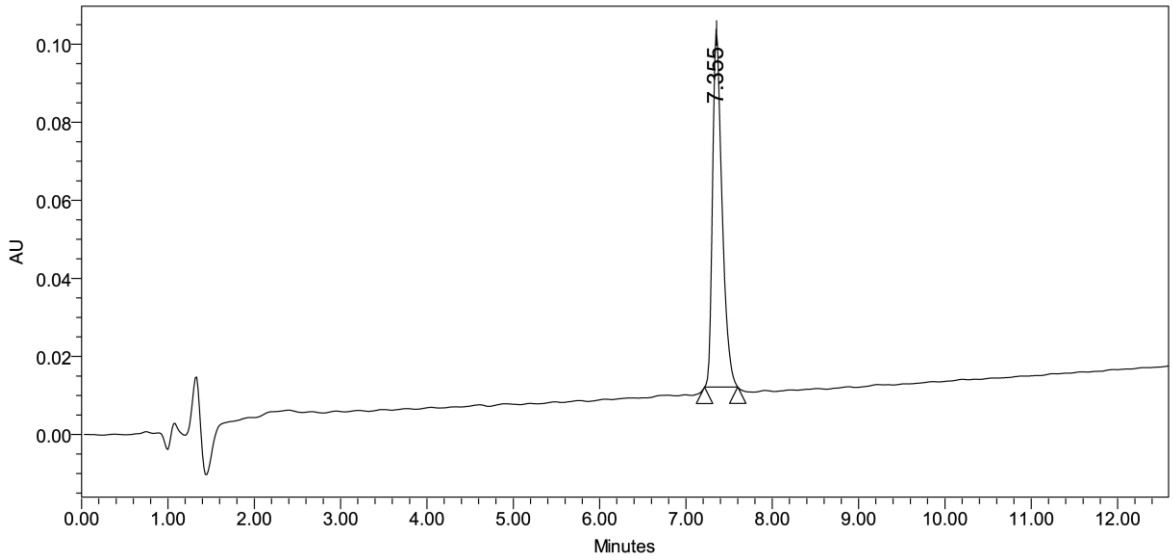


Figure S13. (a) UPLC profile and (b) MALDI-TOF MS of **Compound 19.**



(a) UPLC profile



(b) MALDI-TOF MS

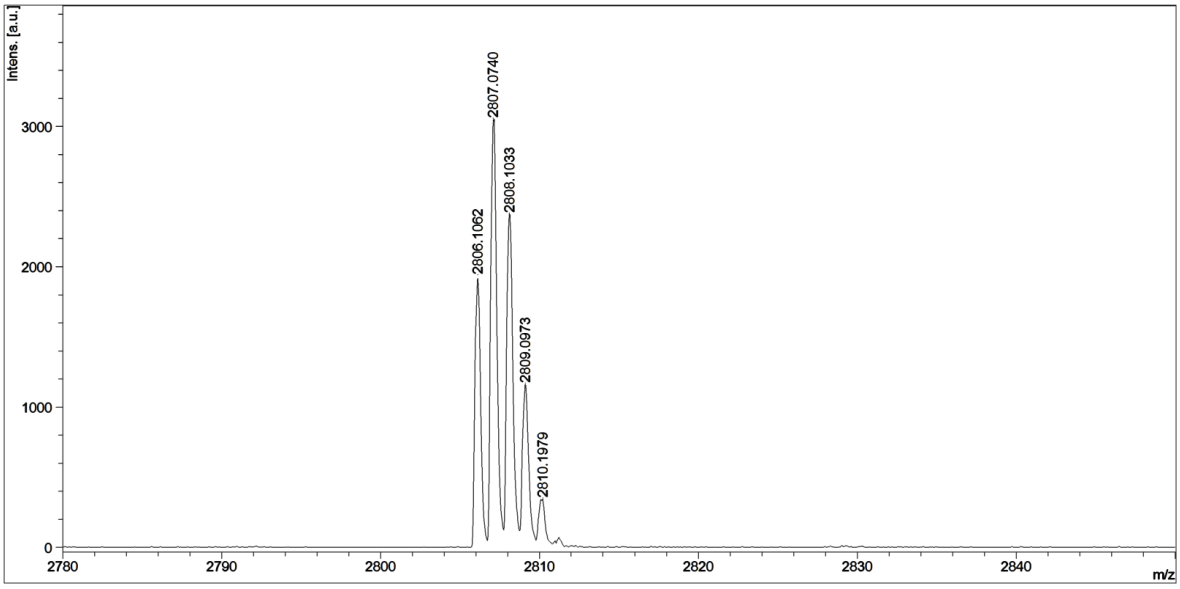
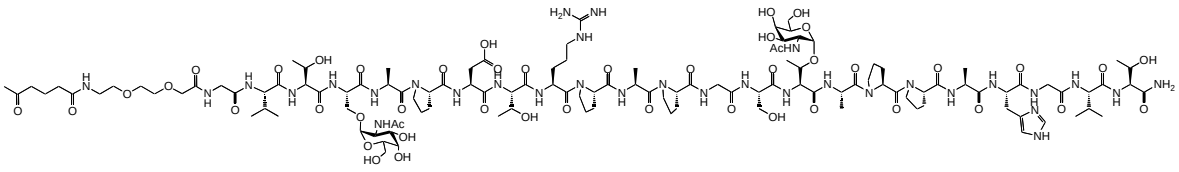
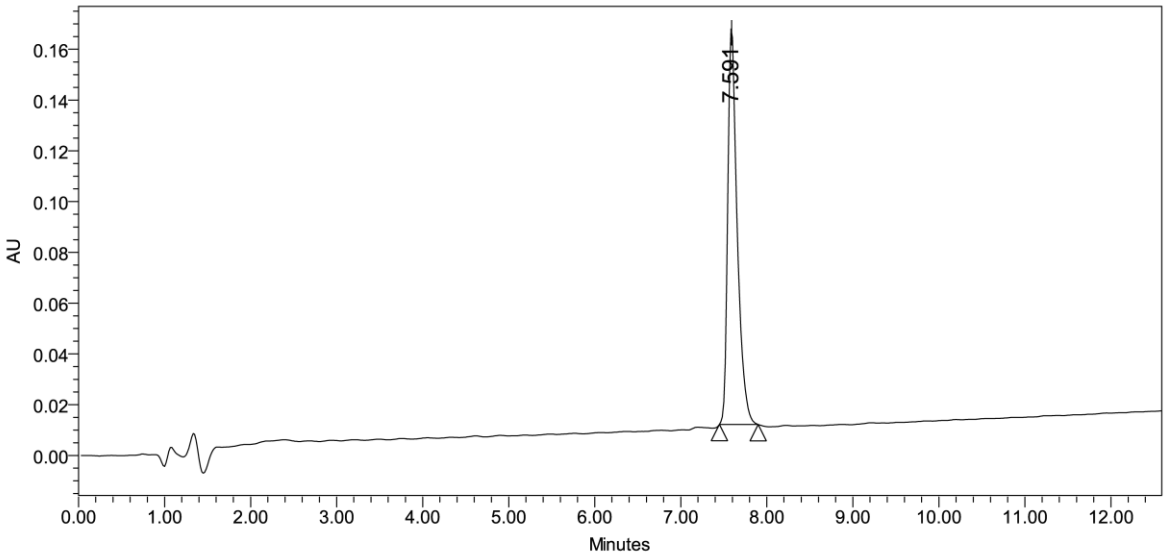


Figure S14. (a) UPLC profile and (b) MALDI-TOF MS of **Compound 21.**



(a) UPLC profile



(b) MALDI-TOF MS

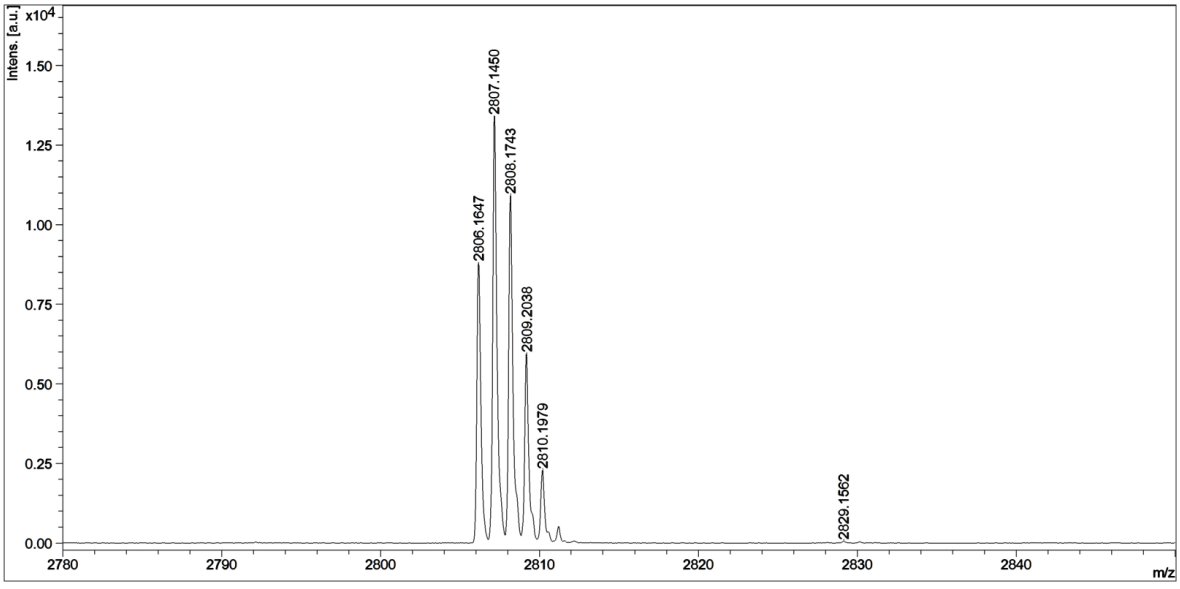
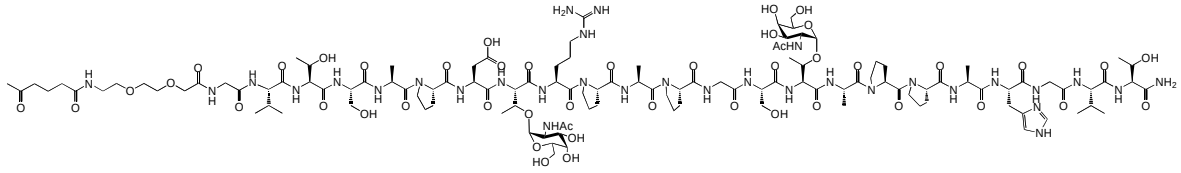
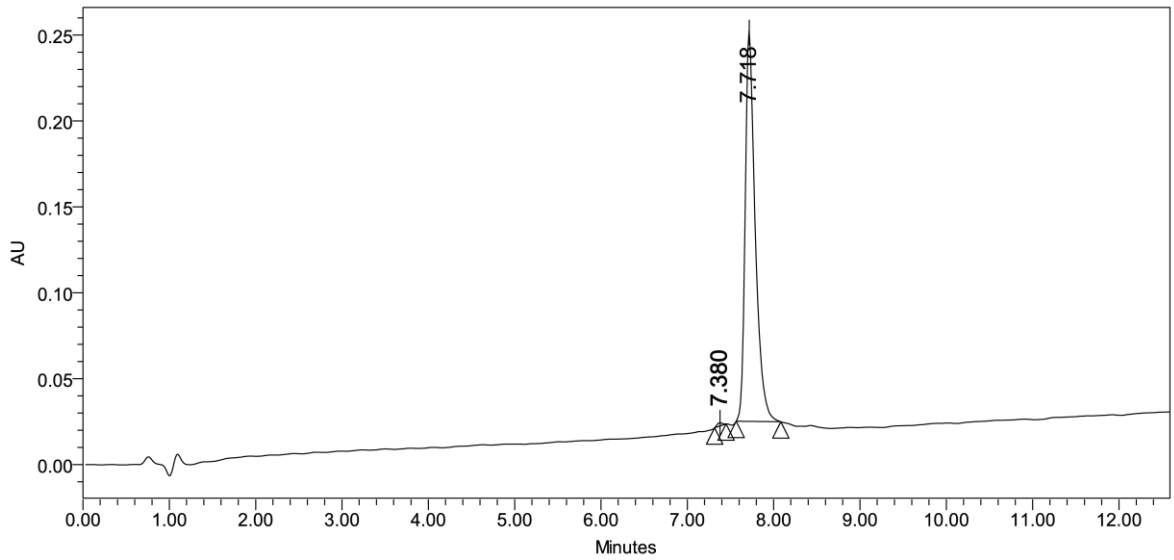


Figure S15. (a) UPLC profile and (b) MALDI-TOF MS of **Compound 22.**



(a) UPLC profile



(b) MALDI-TOF MS

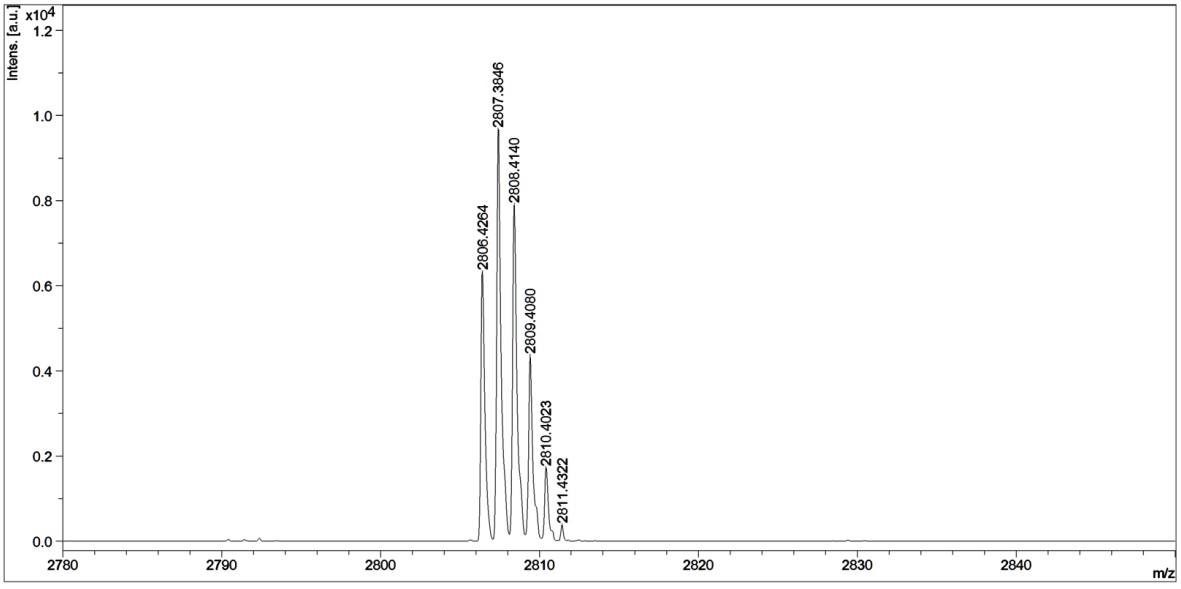
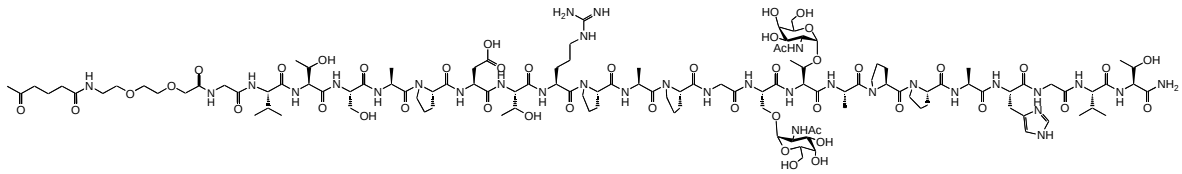
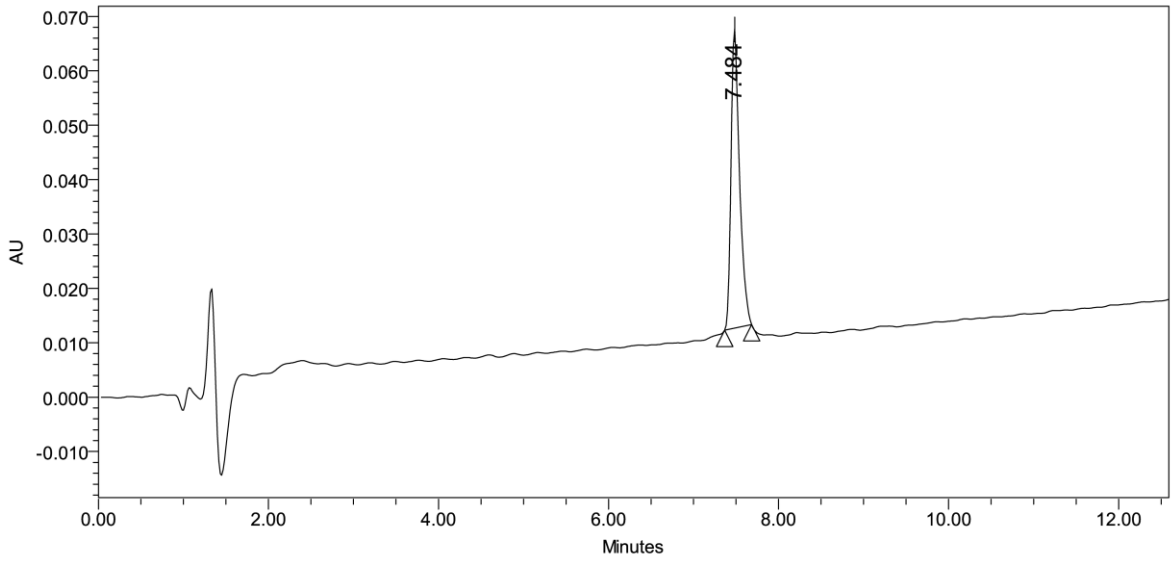


Figure S16. (a) UPLC profile and (b) MALDI-TOF MS of **Compound 23.**



(a) UPLC profile



(b) MALDI-TOF MS

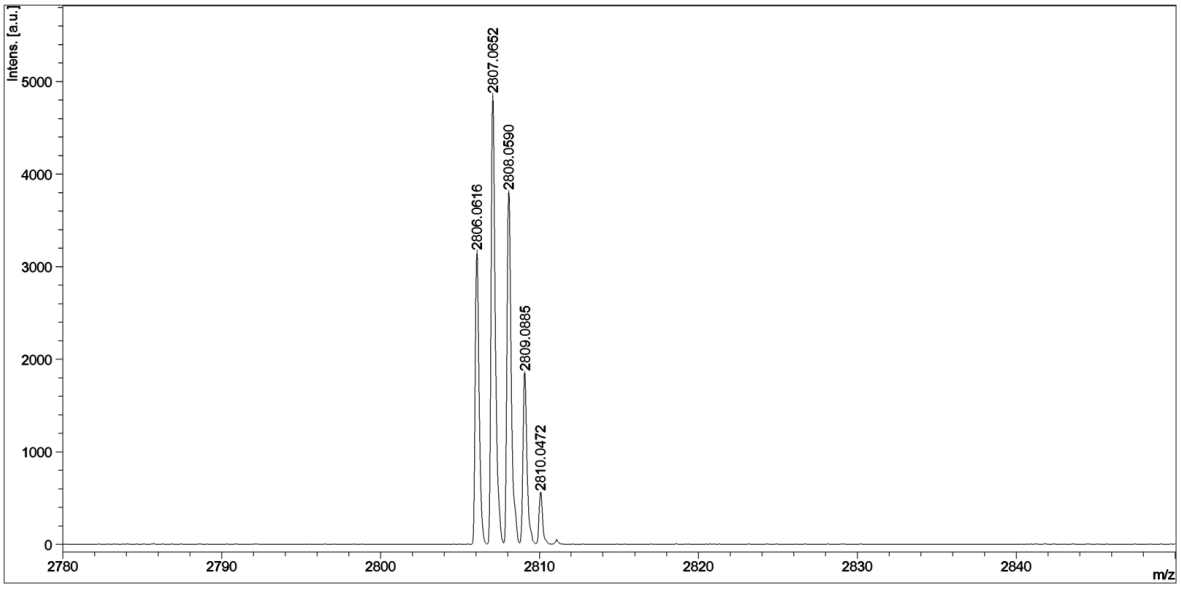
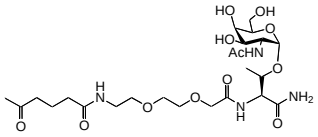
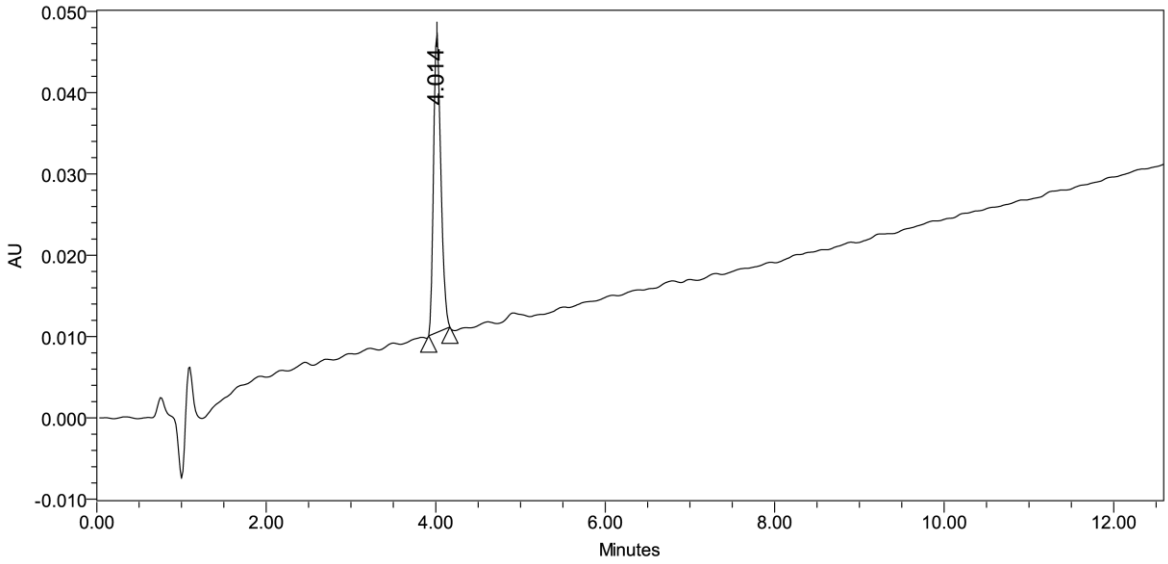


Figure S17. (a) UPLC profile and (b) MALDI-TOF MS of **Compound 26.**



(a) UPLC profile



(b) MALDI-TOF MS

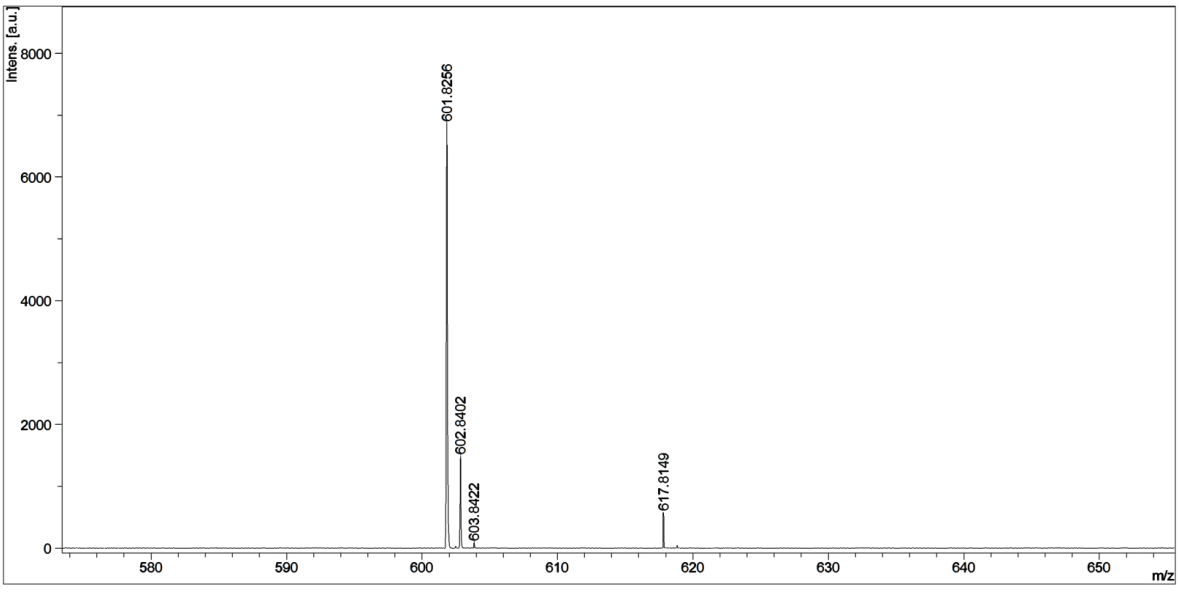
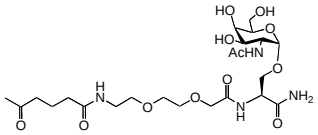
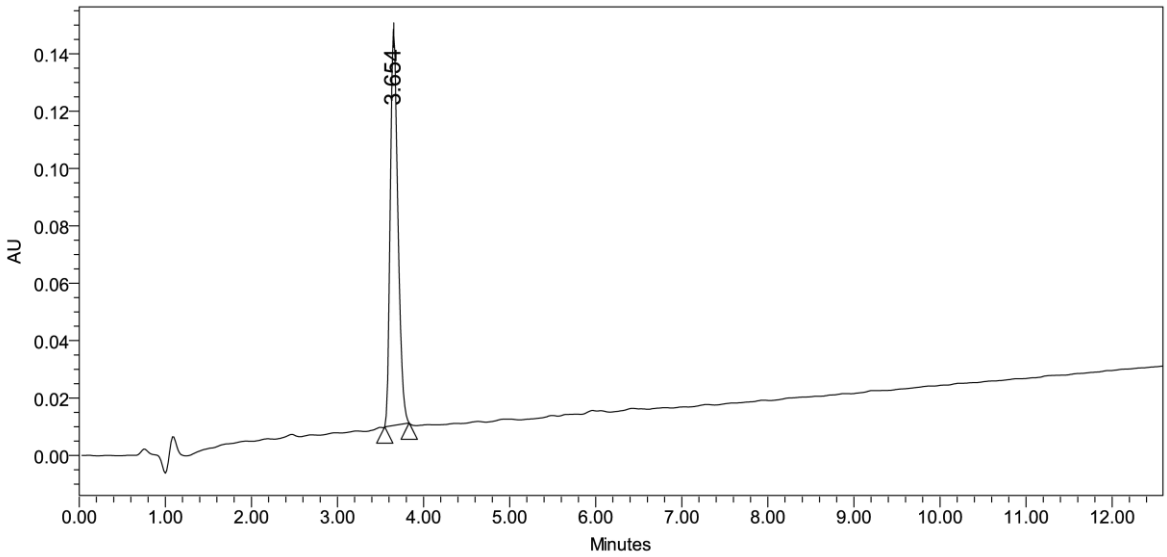


Figure S18. (a) UPLC profile and (b) MALDI-TOF MS of **Compound 27.**



(a) UPLC profile



(b) MALDI-TOF MS

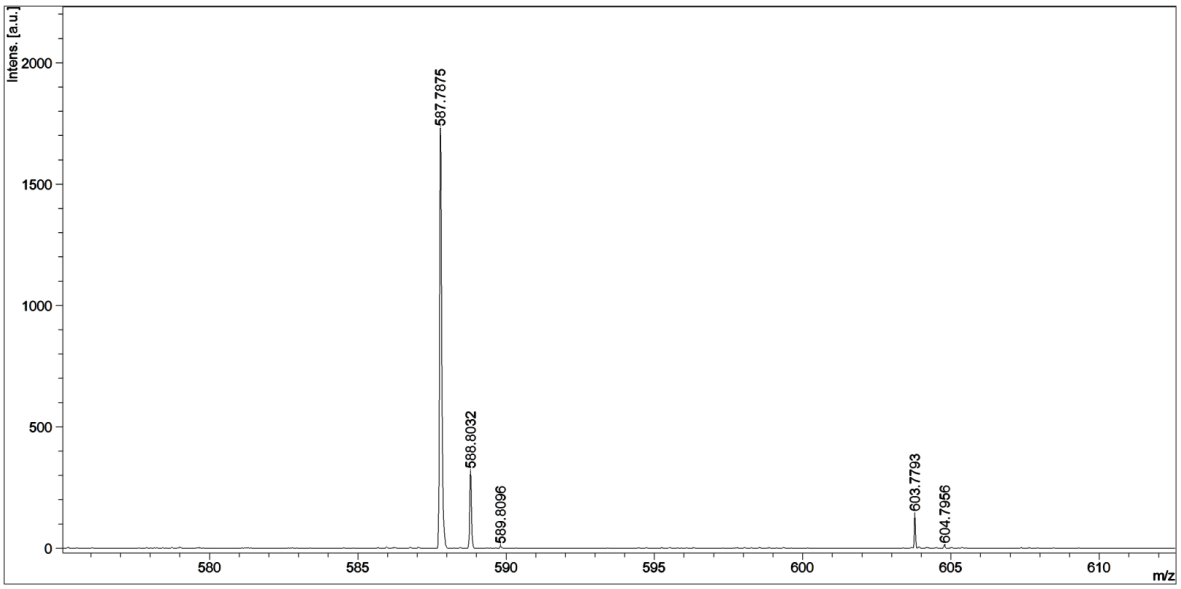
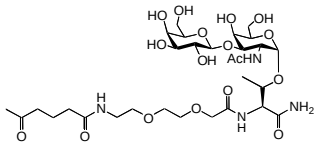
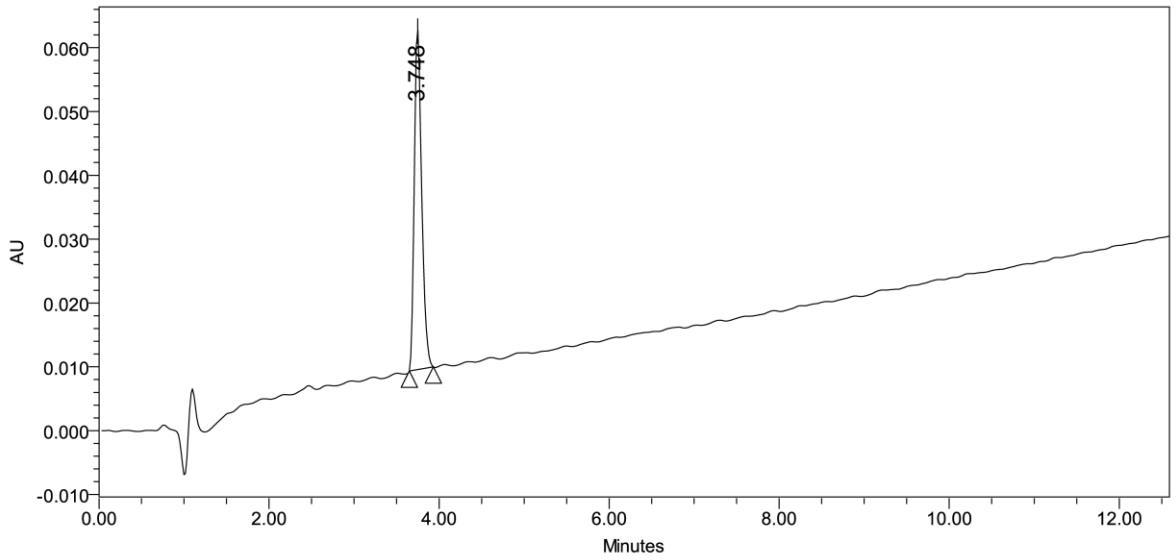


Figure S19. (a) UPLC profile and (b) MALDI-TOF MS of **Compound 28.**



(a) UPLC profile



(b) MALDI-TOF MS

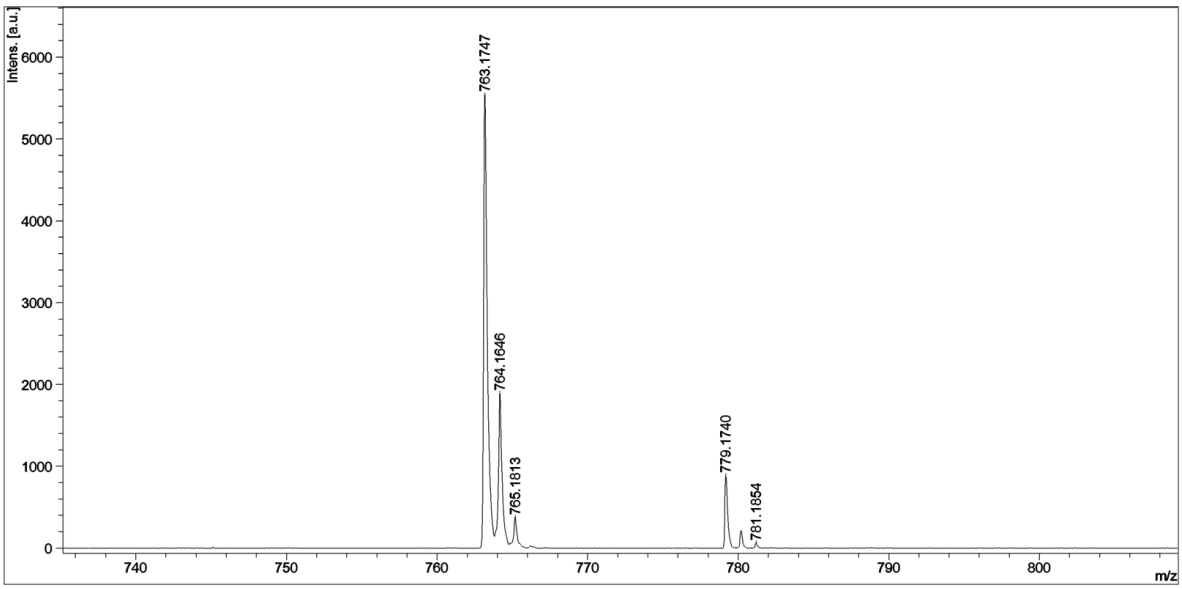
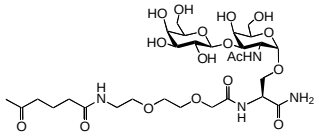
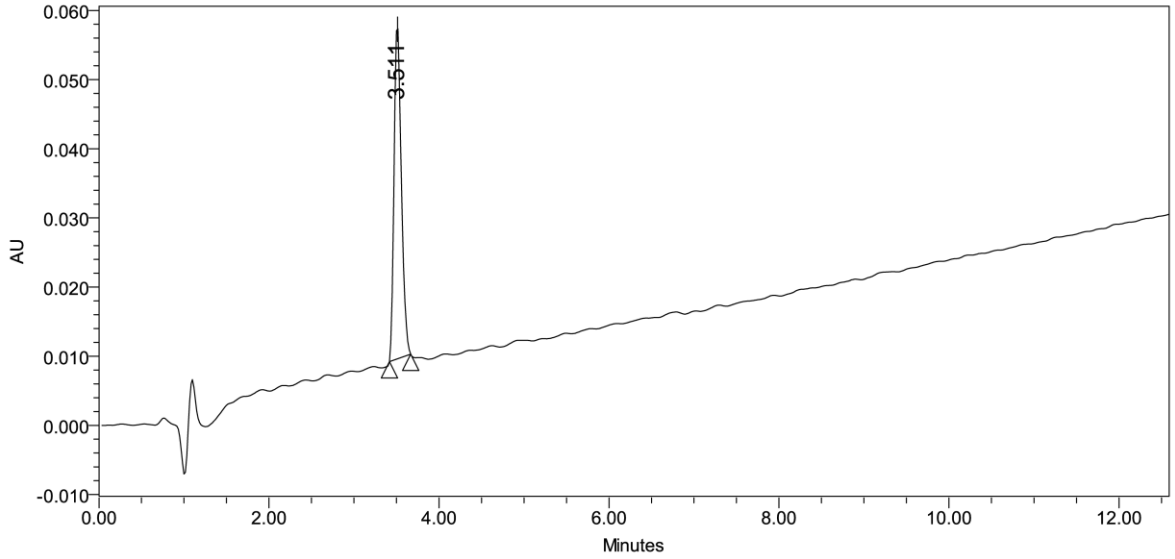


Figure S20. (a) UPLC profile and (b) MALDI-TOF MS of **Compound 29.**



(a) UPLC profile



(b) MALDI-TOF MS

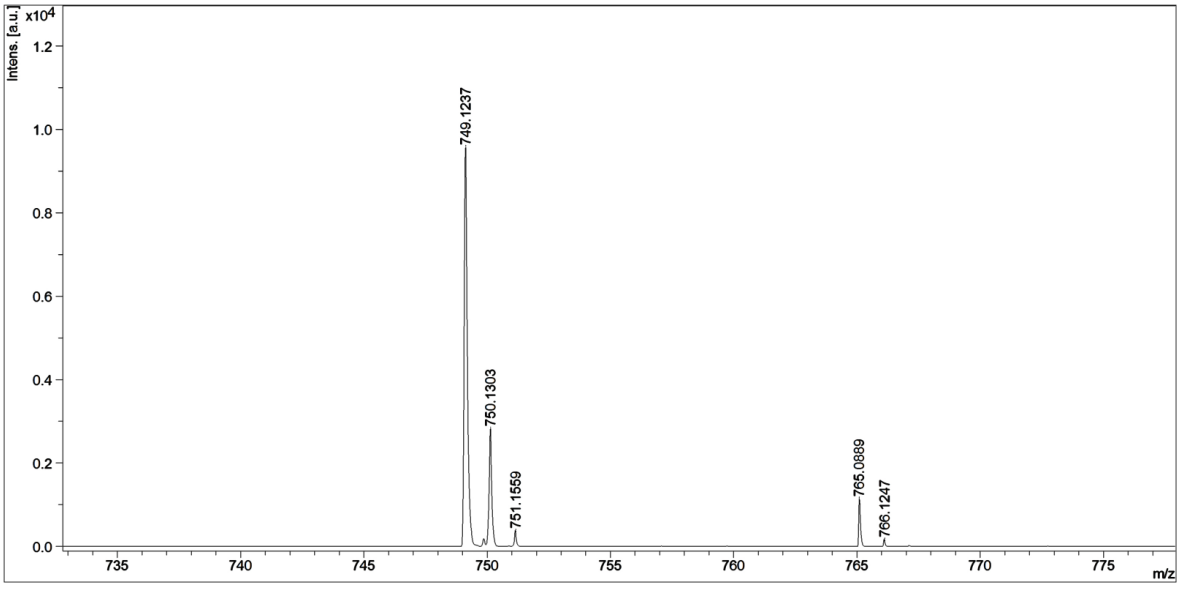
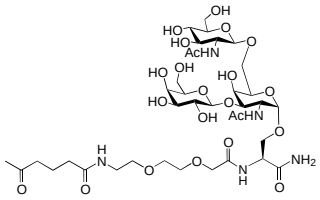
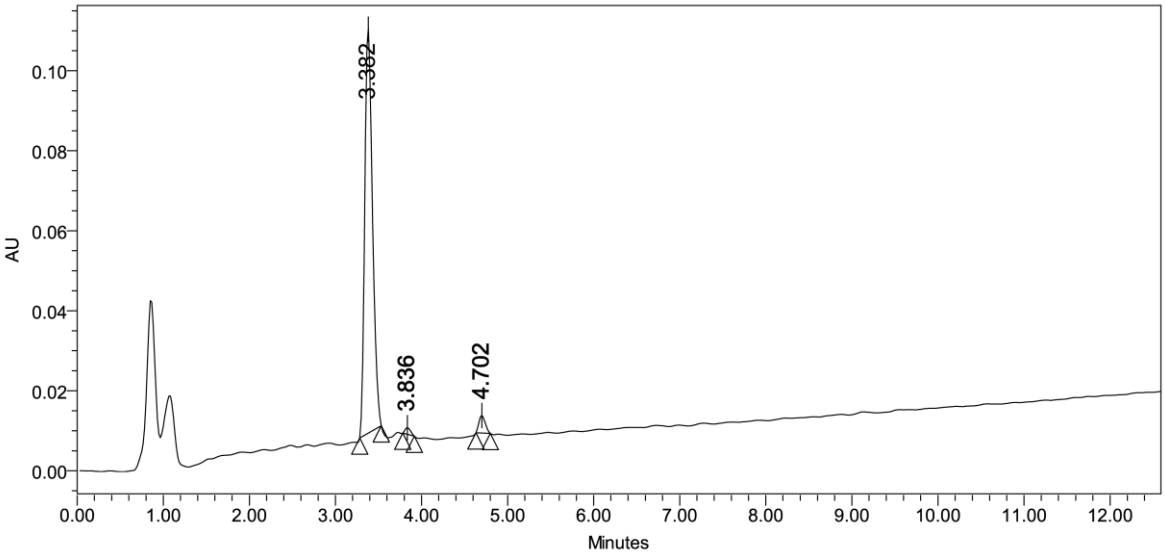


Figure S21. (a) UPLC profile and (b) MALDI-TOF MS of **Compound 30.**



(a) UPLC profile



(b) MALDI-TOF MS

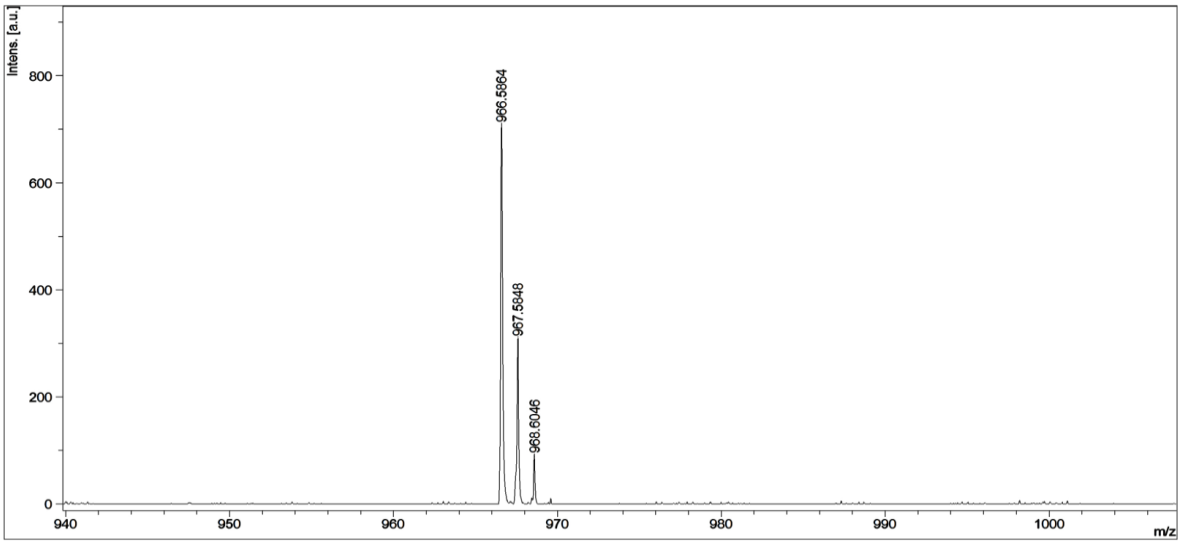
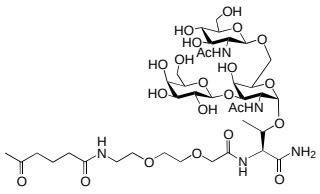
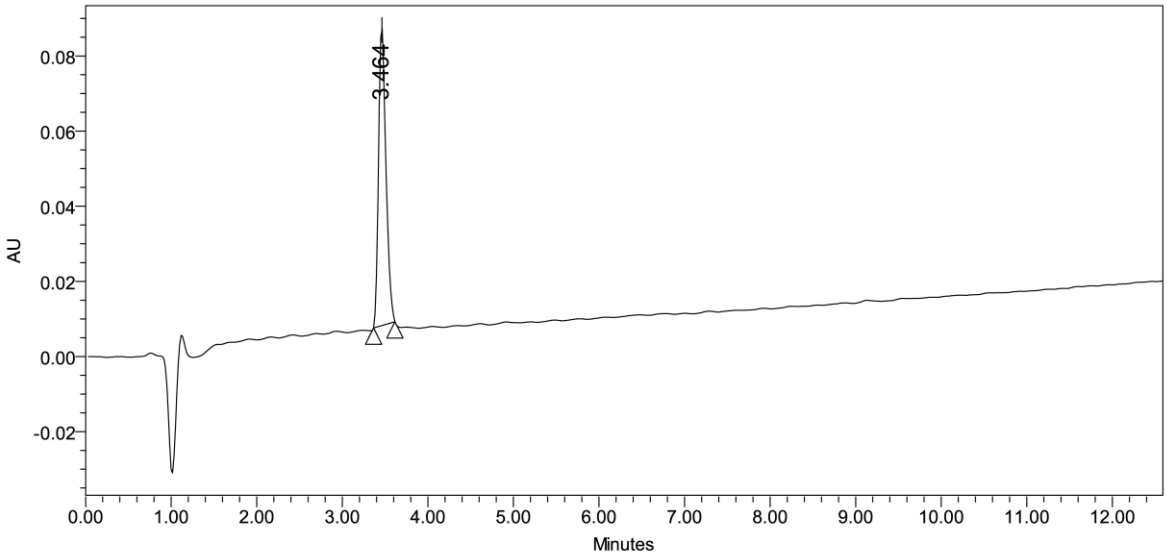


Figure S22. (a) UPLC profile and (b) MALDI-TOF MS of **Compound 31.**



(a) UPLC profile



(b) MALDI-TOF MS

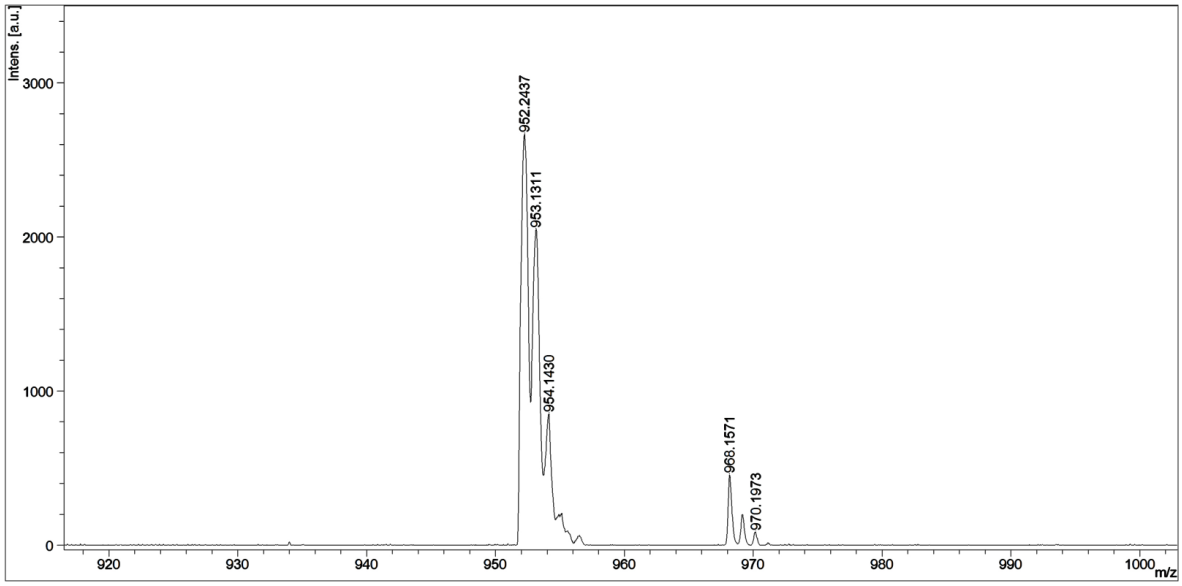


Figure S23. Relative fluorescence units (RFU) with its standard deviation for compound **1**~**35** after treatment with Gal-3CRD, galectin-1, and C-GRP.