Supplementary File

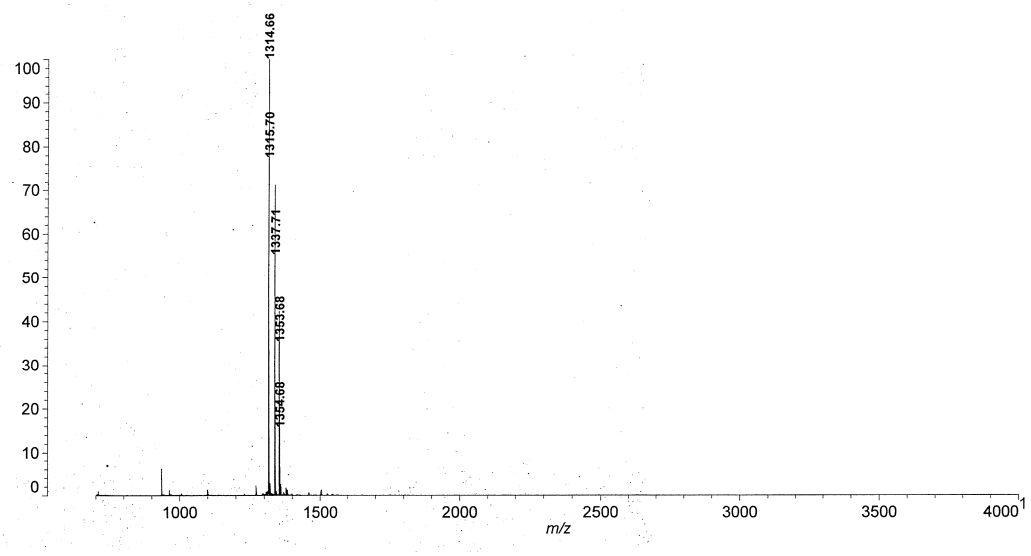

Figure S1. MALDI-TOF mass spectra of **D-Aib1**. An α-CHCA was used as a matrix. **D-Aib1**; calcd. $[M+H]^+$ = 1315.67, obsd. $[M+H]^+$ = 1314.66.

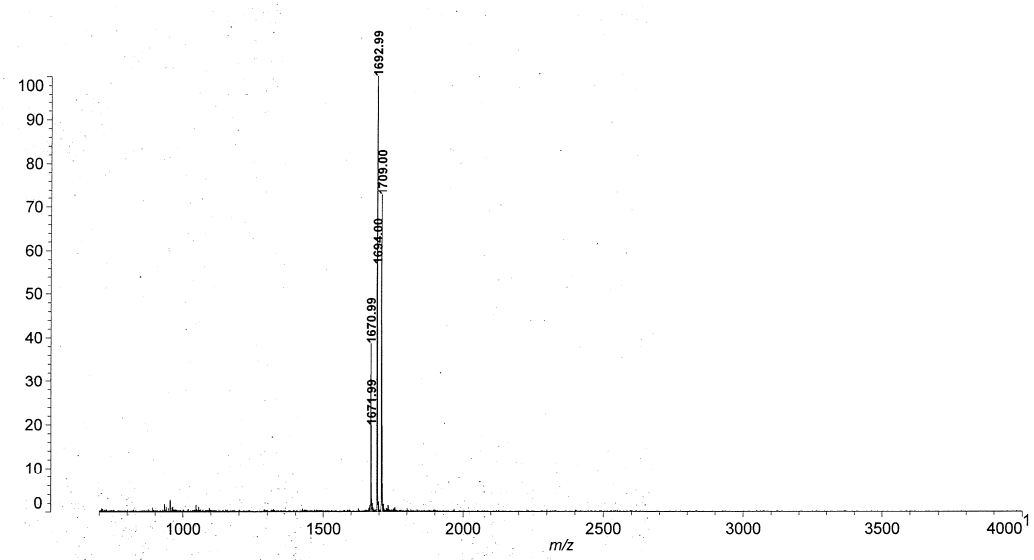

Figure S2. MALDI-TOF mass spectra of **D-Aib2**. An α-CHCA was used as a matrix. **D-Aib2**; calcd. $[M+H]^+$ = 1671.82, obsd. $[M+H]^+$ = 1670.99.

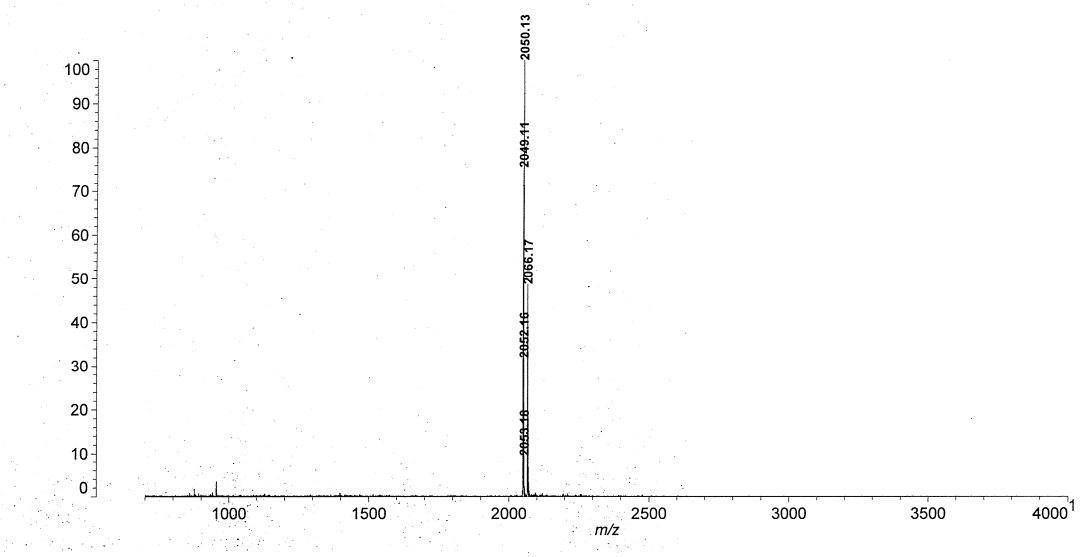

Figure S3. MALDI-TOF mass spectra of **D-Aib3**. An α-CHCA was used as a matrix. **D-Aib3**; calcd. $[M+Na]^+ = 2049.97$, obsd. $[M+Na]^+ = 2050.13$.

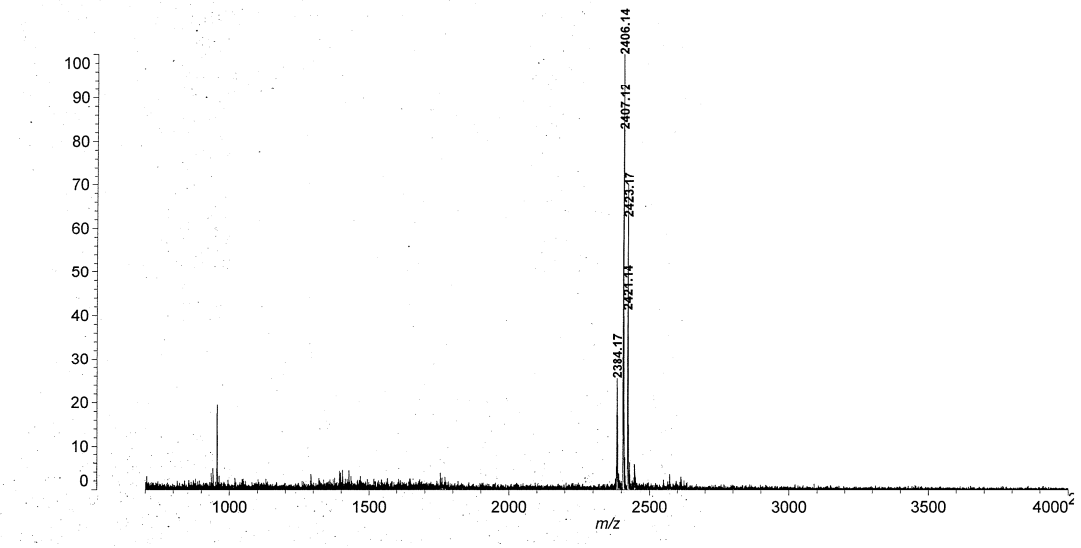

Figure S4. MALDI-TOF mass spectra of **D-Aib4**. An α-CHCA was used as a matrix. **D-Aib4**; calcd. $[M+Na]^+ = 2406.12$, obsd. $[M+Na]^+ = 2406.14$.

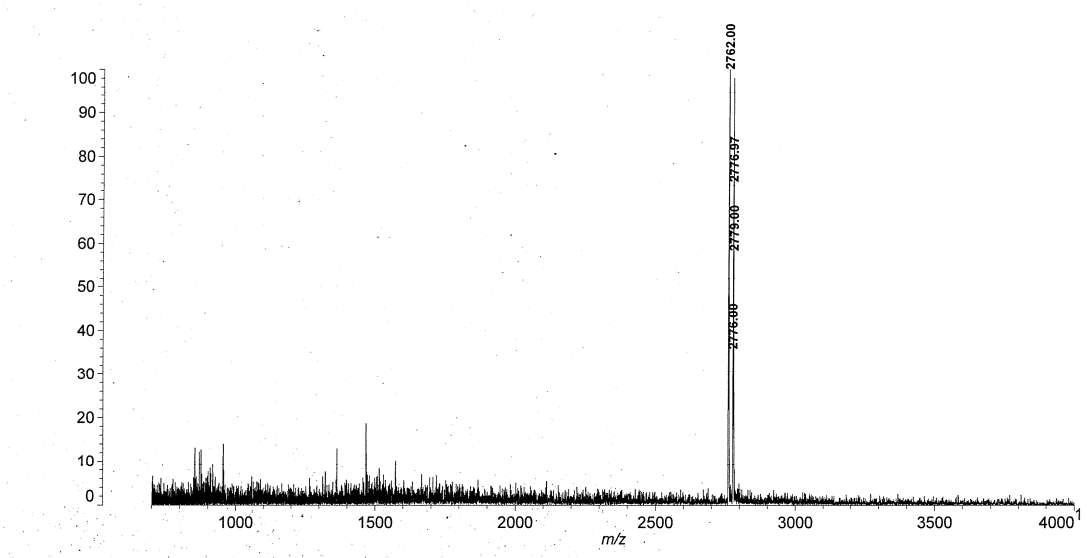

Figure S5. MALDI-TOF mass spectra of **D-Aib5**. An α-CHCA was used as a matrix. **D-Aib5**; calcd. $[M+Na]^+$ = 2762.28, obsd. $[M+Na]^+$ = 2762.00.

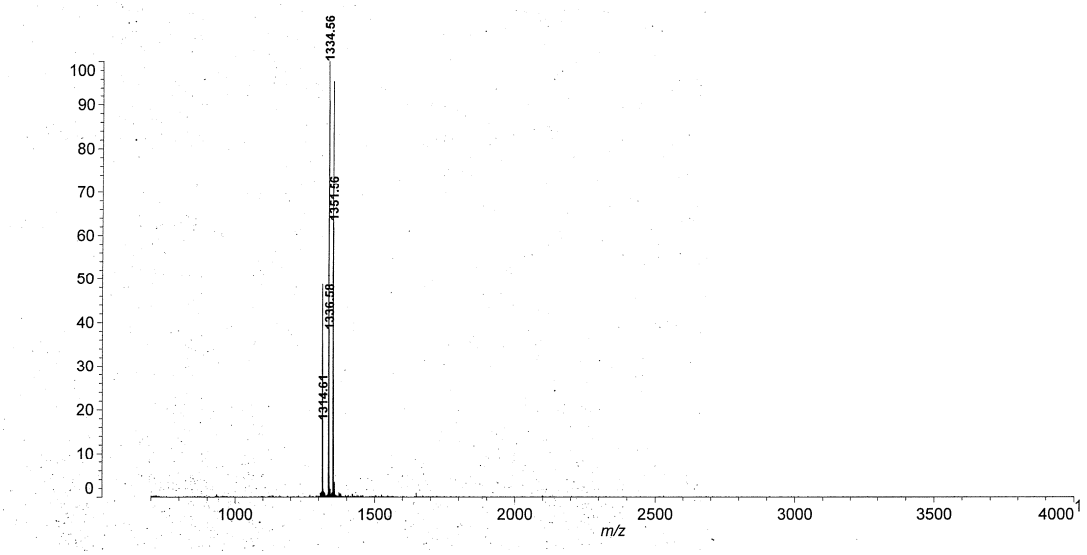

Figure S6. MALDI-TOF mass spectra of **L-Aib1**. An α-CHCA was used as a matrix. **L-Aib1**; calcd. $[M+H]^+$ = 1315.67, obsd. $[M+H]^+$ = 1314.61.

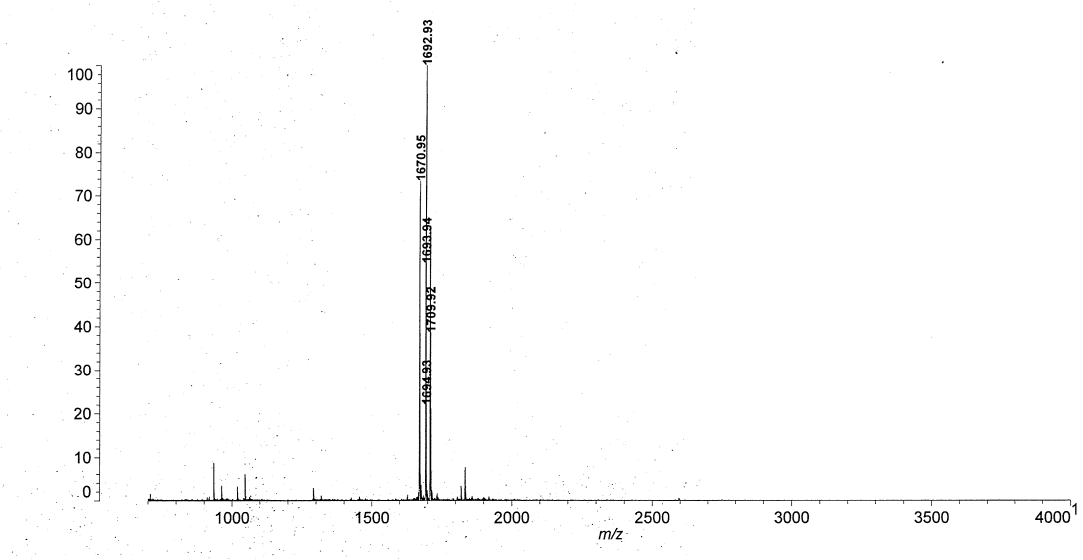

Figure S7. MALDI-TOF mass spectra of **L-Aib2**. An α-CHCA was used as a matrix. **L-Aib2**; calcd. $[M+H]^+ = 1671.82$, obsd. $[M+H]^+ = 1670.95$.

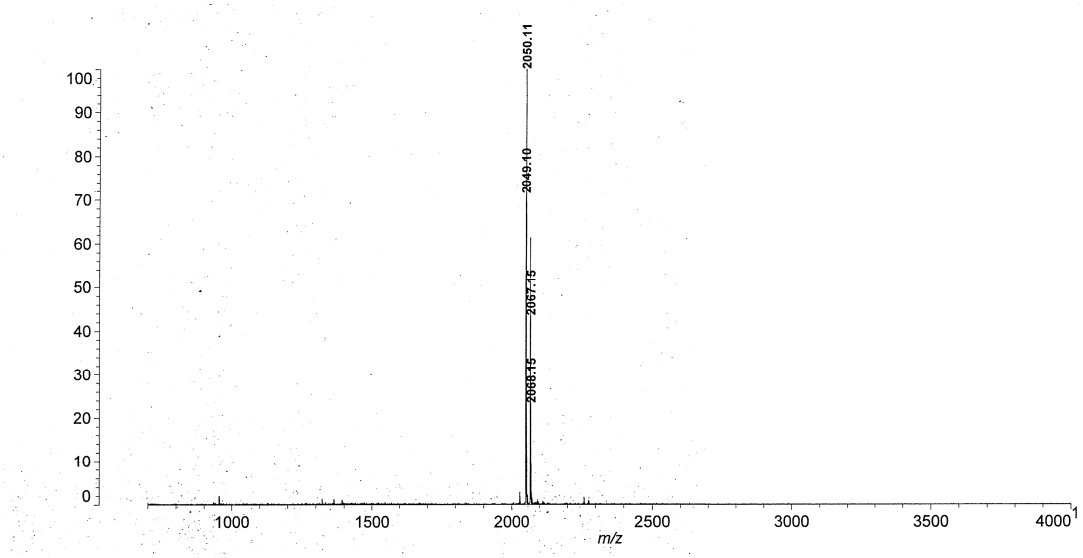

Figure S8. MALDI-TOF mass spectra of **L-Aib3**. An α-CHCA was used as a matrix. **L-Aib3**; calcd. $[M+Na]^+ = 2050.97$, obsd. $[M+Na]^+ = 2050.11$.

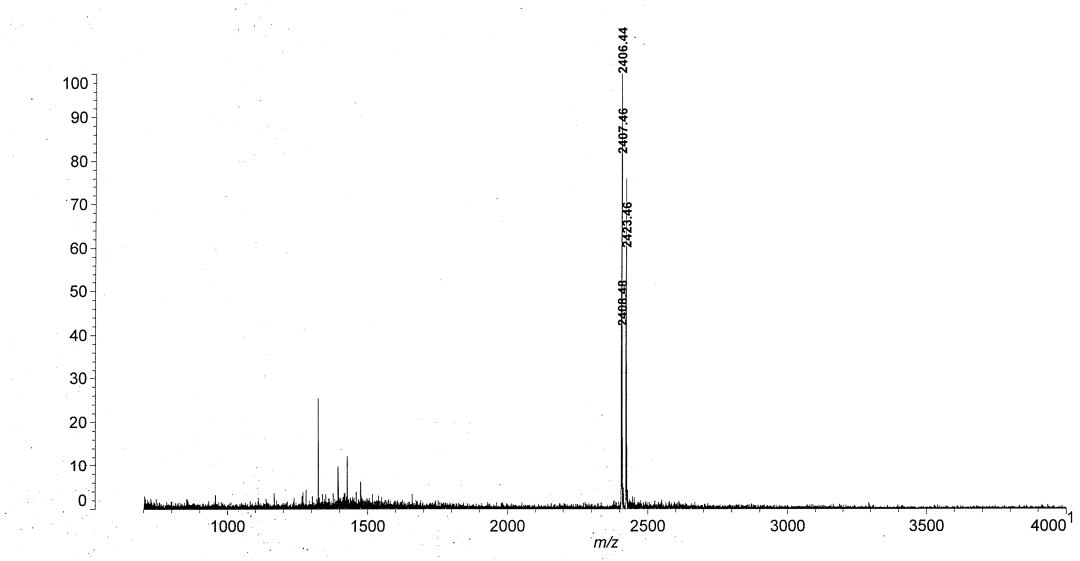

Figure S9. MALDI-TOF mass spectra of **L-Aib4**. An α-CHCA was used as a matrix. **L-Aib4**; calcd. $[M+Na]^+ = 2406.12$, obsd. $[M+Na]^+ = 2406.44$.

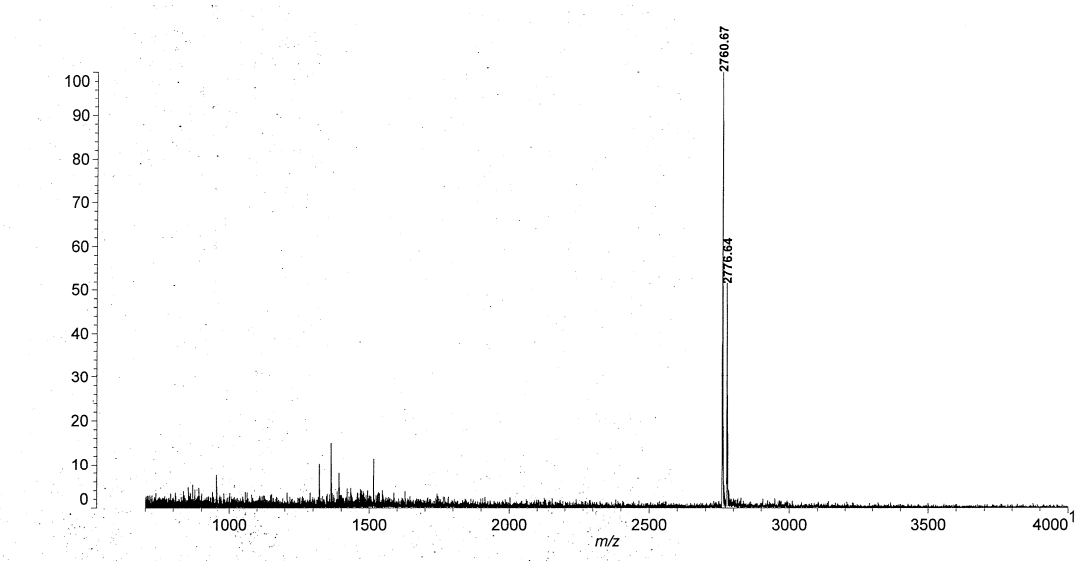

Figure S10. MALDI-TOF mass spectra of **L-Aib5**. An α-CHCA was used as a matrix. **L-Aib5**; calcd. $[M+Na]^+ = 2762.28$, obsd. $[M+Na]^+ = 2760.67$.

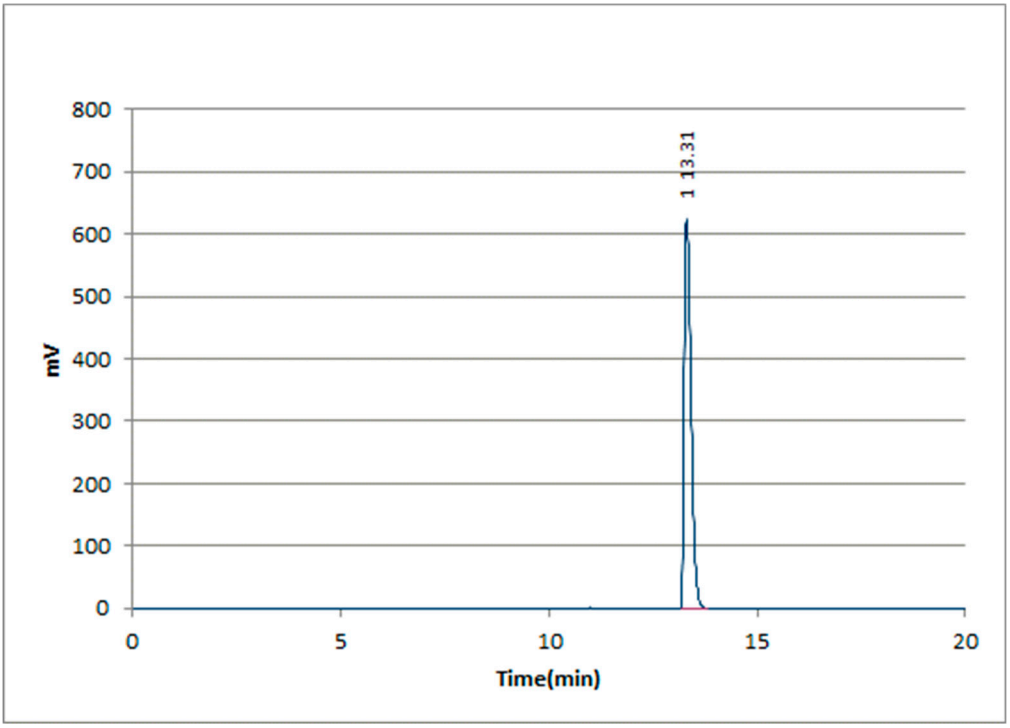

Figure S11. RP-HPLC chart of **D-Aib1**. Buffer A, 0.1% TFA in water; buffer B, acetonitrile, and monitoring at 340 nm with a gradient of 20-100% for 20 min.

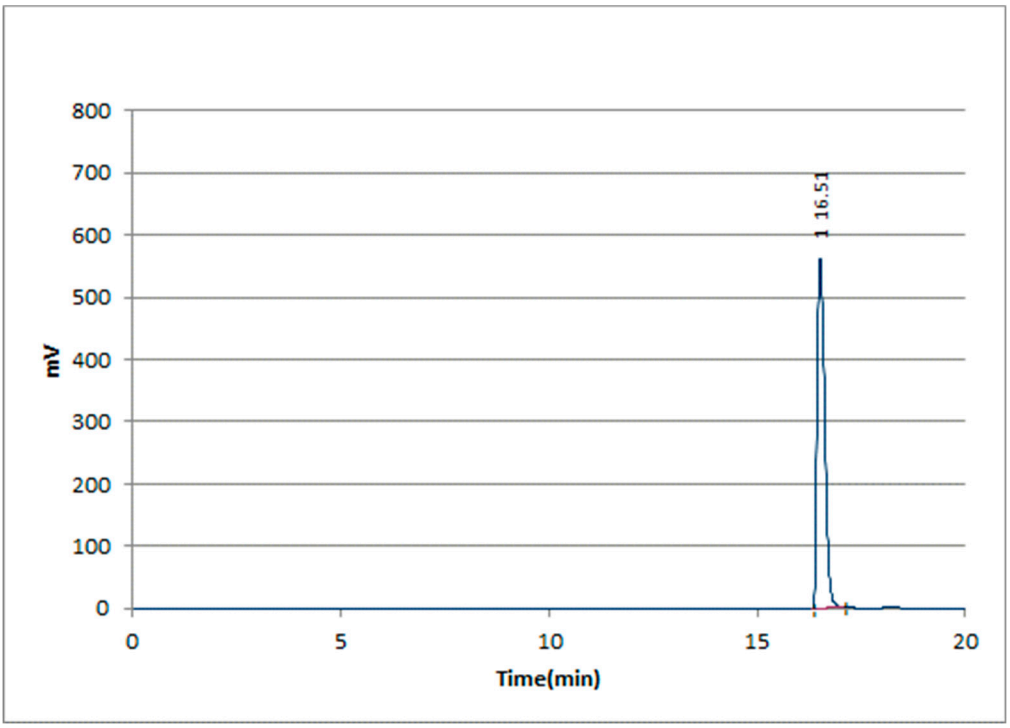

Figure S12. RP-HPLC chart of **D-Aib2**. Buffer A, 0.1% TFA in water; buffer B, acetonitrile, and monitoring at 340 nm with a gradient of 20-100% for 20 min.

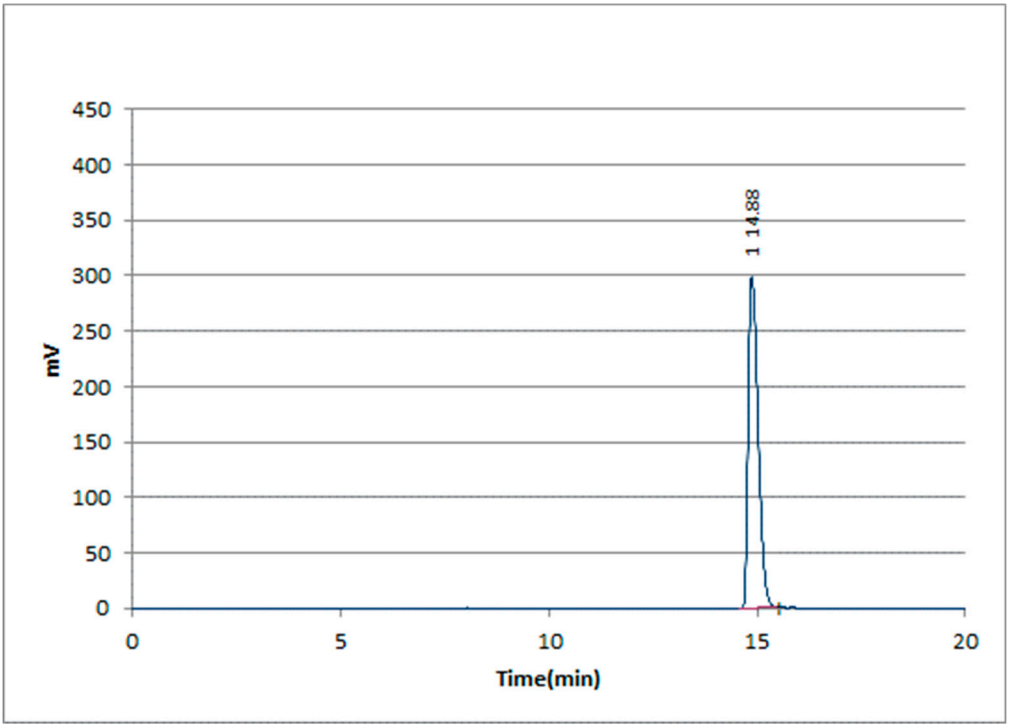

Figure S13. RP-HPLC chart of **D-Aib3**. Buffer A, 0.1% TFA in water; buffer B, acetonitrile, and monitoring at 340 nm with a gradient of 50-100% for 20 min.

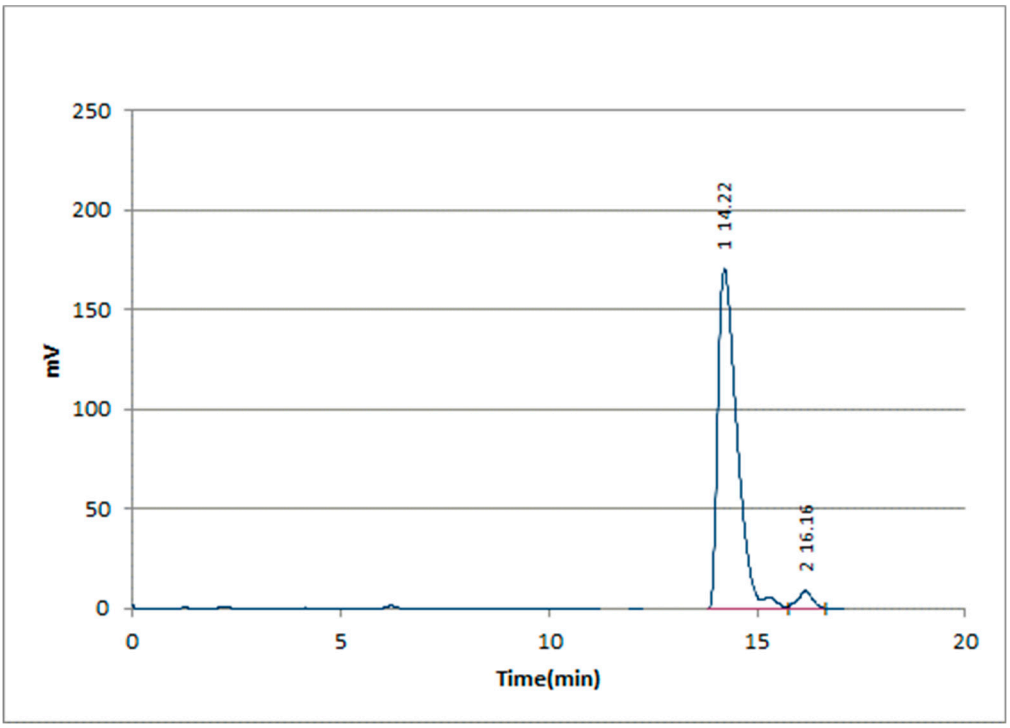

Figure S14. RP-HPLC chart of **D-Aib4**. Buffer A, 0.1% TFA in water; buffer B, acetonitrile, and monitoring at 340 nm with a gradient of 70-100% for 20 min.

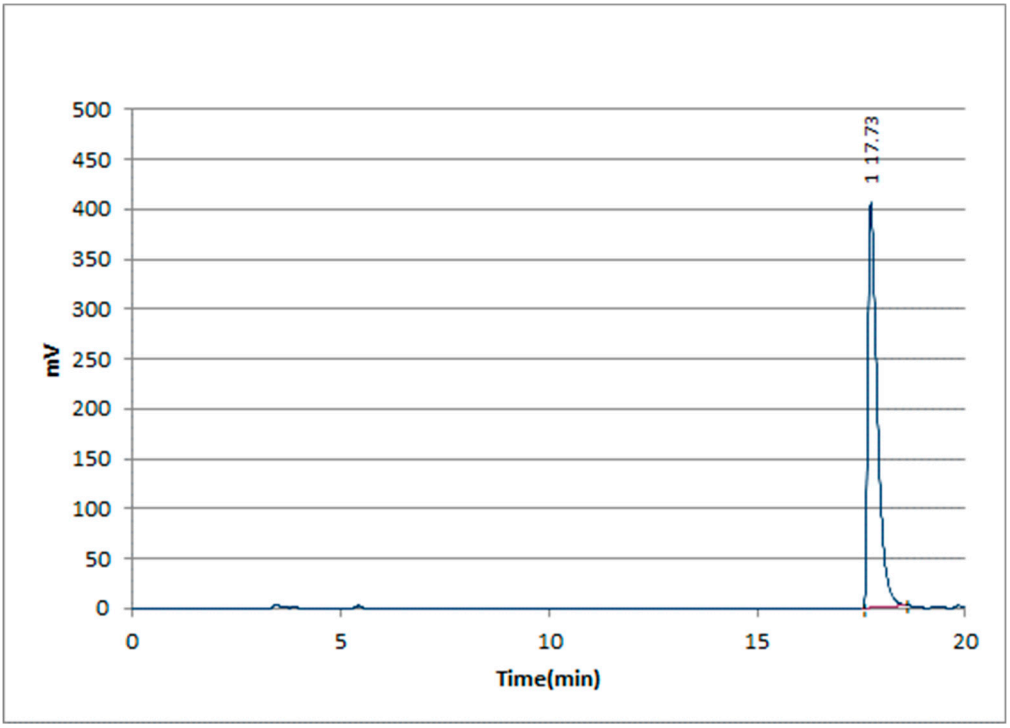

Figure S15. RP-HPLC chart of **D-Aib5**. Buffer A, 0.1% TFA in water; buffer B, acetonitrile, and monitoring at 340 nm with a gradient of 80-100% for 20 min.

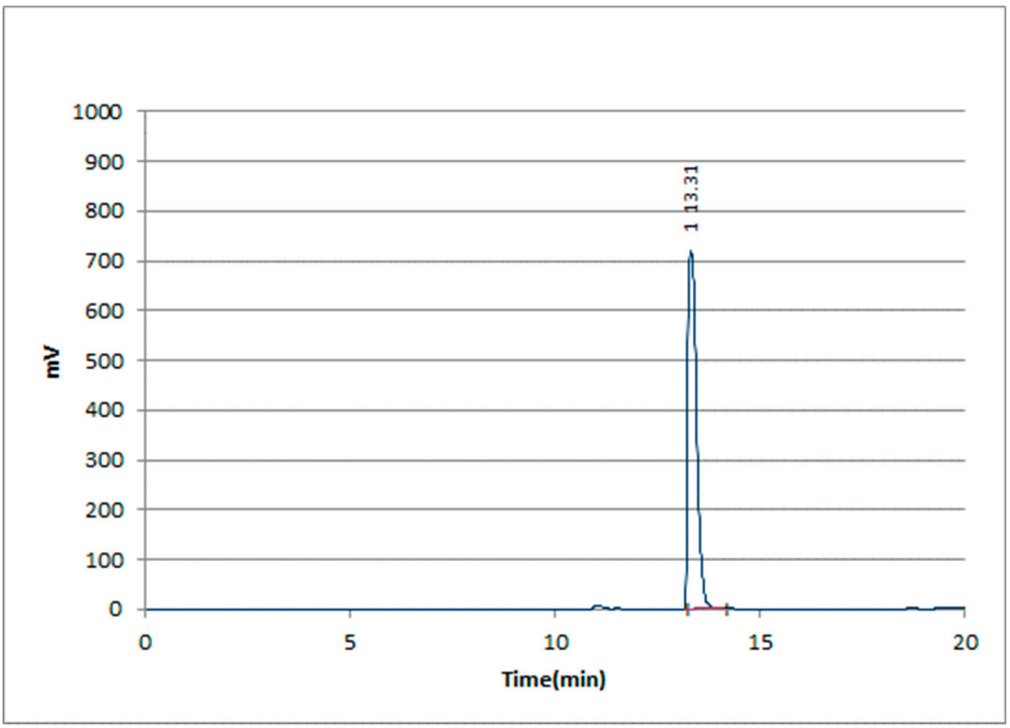

Figure S16. RP-HPLC chart of **L-Aib1**. Buffer A, 0.1% TFA in water; buffer B, acetonitrile, and monitoring at 340 nm with a gradient of 20-100% for 20 min.

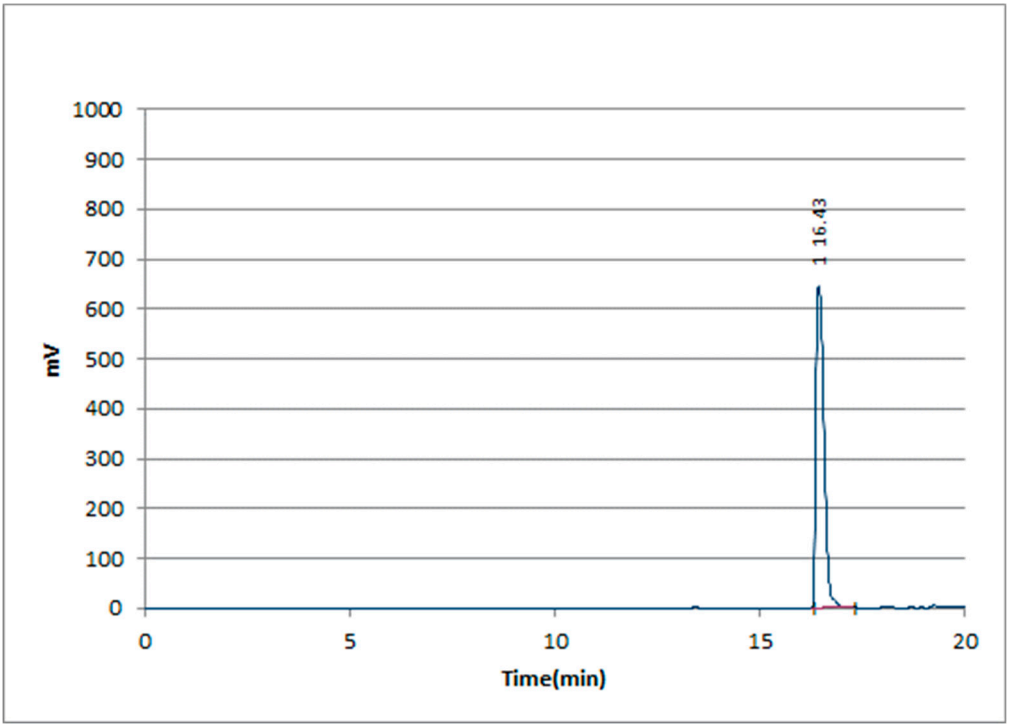

Figure S17. RP-HPLC chart of **L-Aib2**. Buffer A, 0.1% TFA in water; buffer B, acetonitrile, and monitoring at 340 nm with a gradient of 20-100% for 20 min.

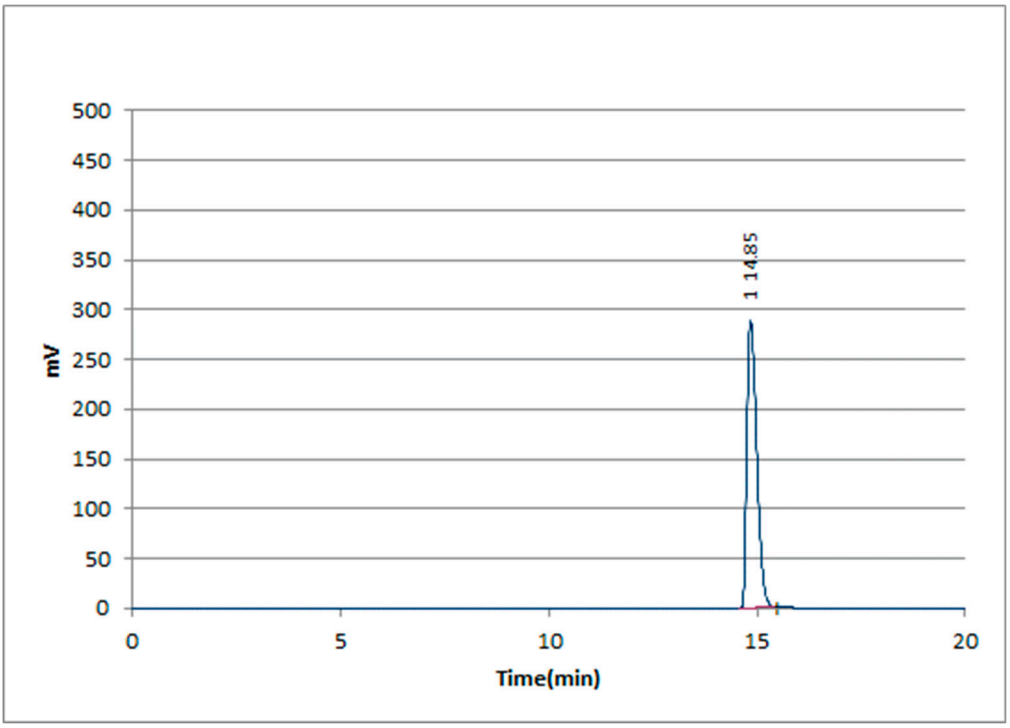

Figure S18. RP-HPLC chart of **L-Aib3**. Buffer A, 0.1% TFA in water; buffer B, acetonitrile, and monitoring at 340 nm with a gradient of 50-100% for 20 min.

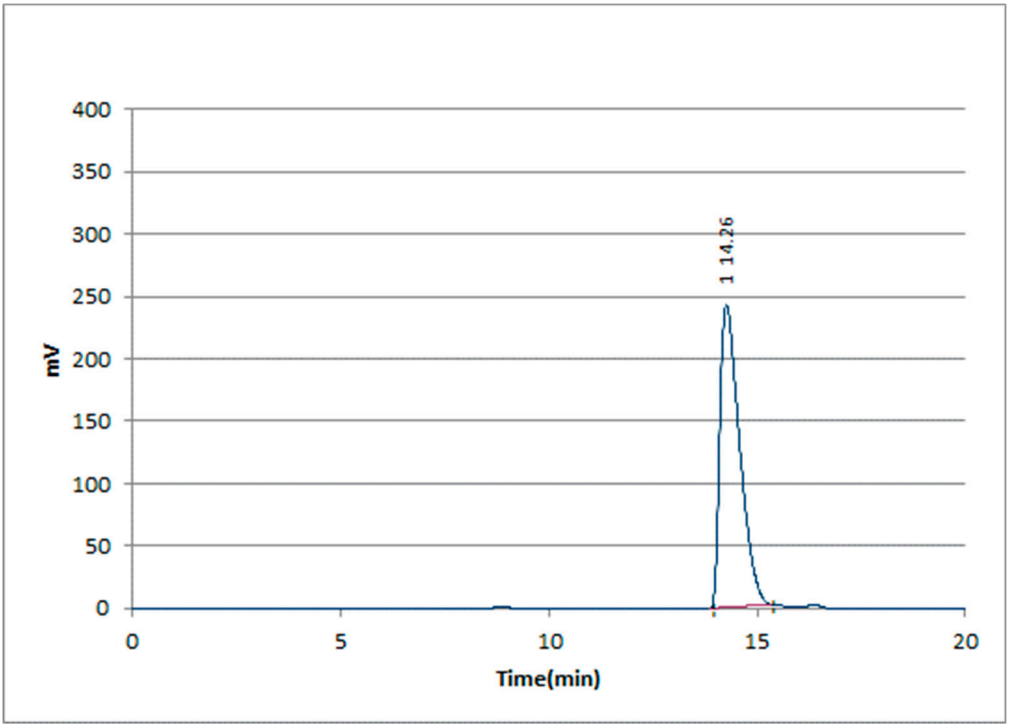

Figure S19. RP-HPLC chart of **L-Aib4**. Buffer A, 0.1% TFA in water; buffer B, acetonitrile, and monitoring at 340 nm with a gradient of 70-100% for 20 min.

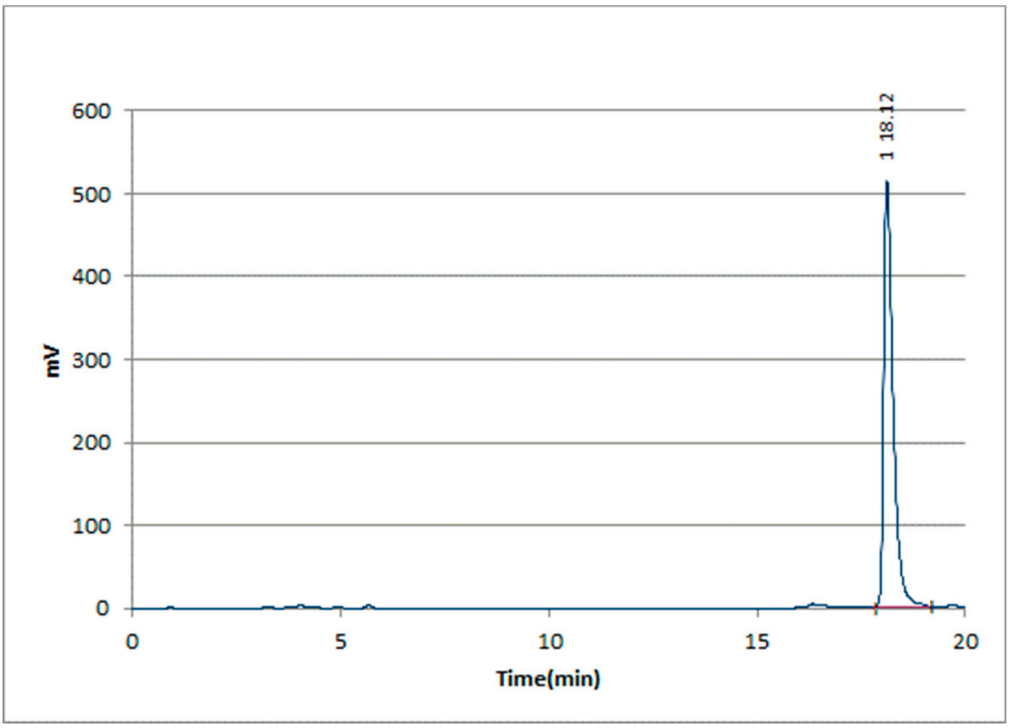

Figure S20. RP-HPLC chart of **L-Aib5**. Buffer A, 0.1% TFA in water; buffer B, acetonitrile, and monitoring at 340 nm with a gradient of 80-100% for 20 min.

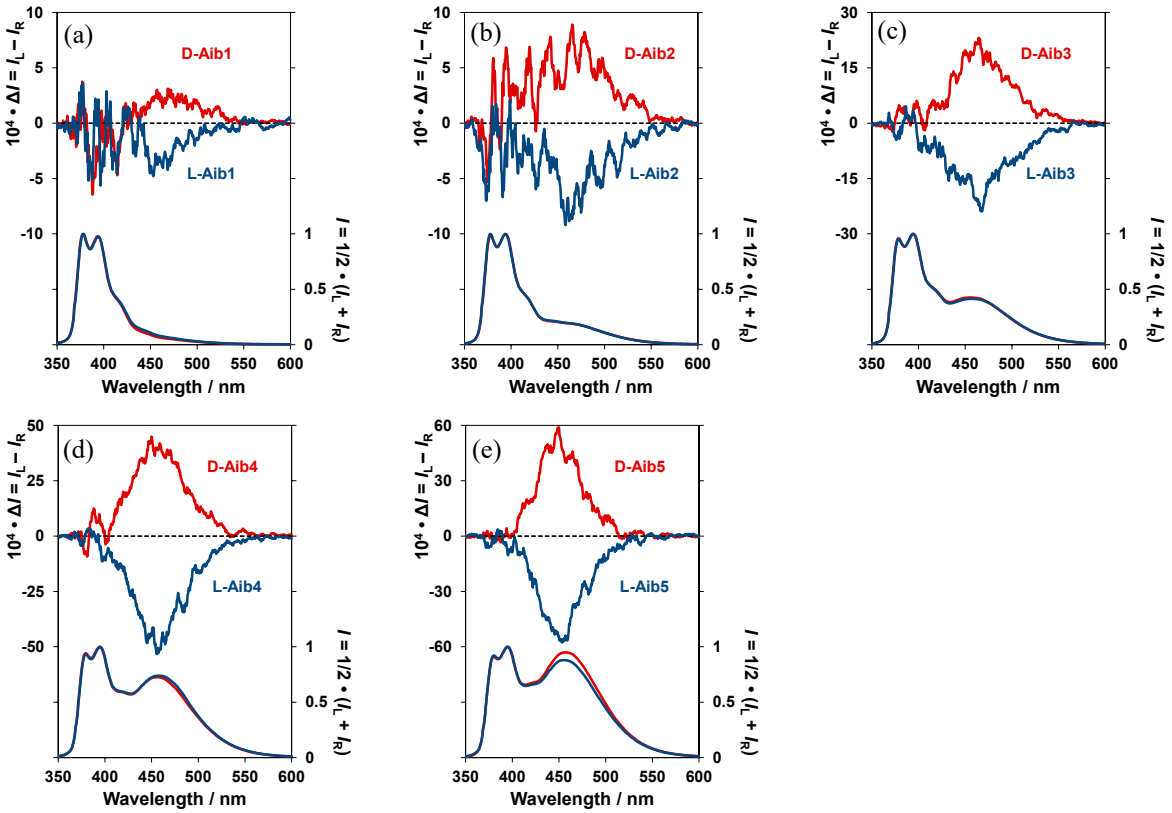

Figure S21. CPL (upper panel) and PL (lower panel) spectra of (**a**) **D-Aib1**/**L-Aib1**; (**b**) **D-Aib2**/**L-Aib2**; (**c**) **D-Aib3**/**L-Aib3**; (**d**) **D-Aib4**/**L-Aib4**; and (**e**) **D-Aib5**/**L-Aib5** in ethanol (1.0×10^{-4} M). λ_{ex} = 300 nm D-isomer and L-isomer spectra are shown in red and blue, respectively. Path length = 2 mm.

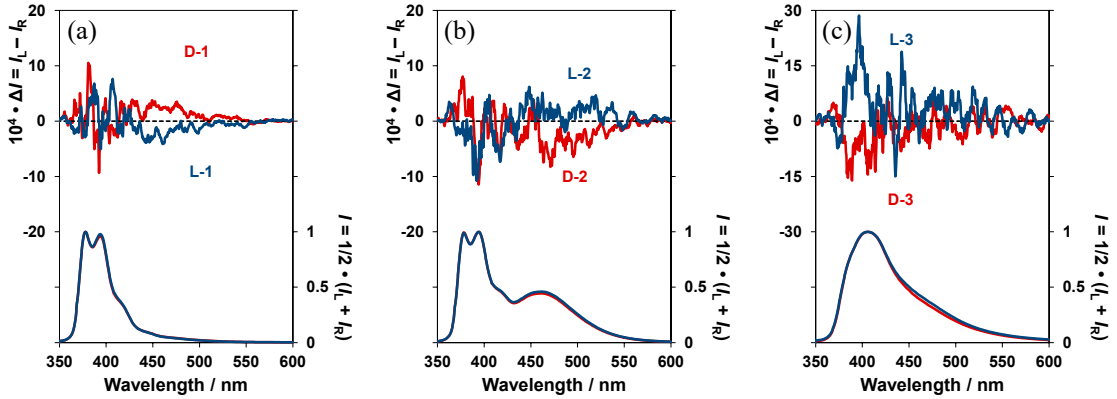

Figure S22. CPL (upper panel) and PL (lower panel) spectra of (a) **D-1** / **L-1**; (b) **D-2** / **L-2**; and (c) **D-3** / **L-3** in ethanol (1.0×10^{-4} M). λ_{ex} = 300 nm D-isomer and L-isomer spectra are shown in red and blue, respectively. Path length = 2 mm.

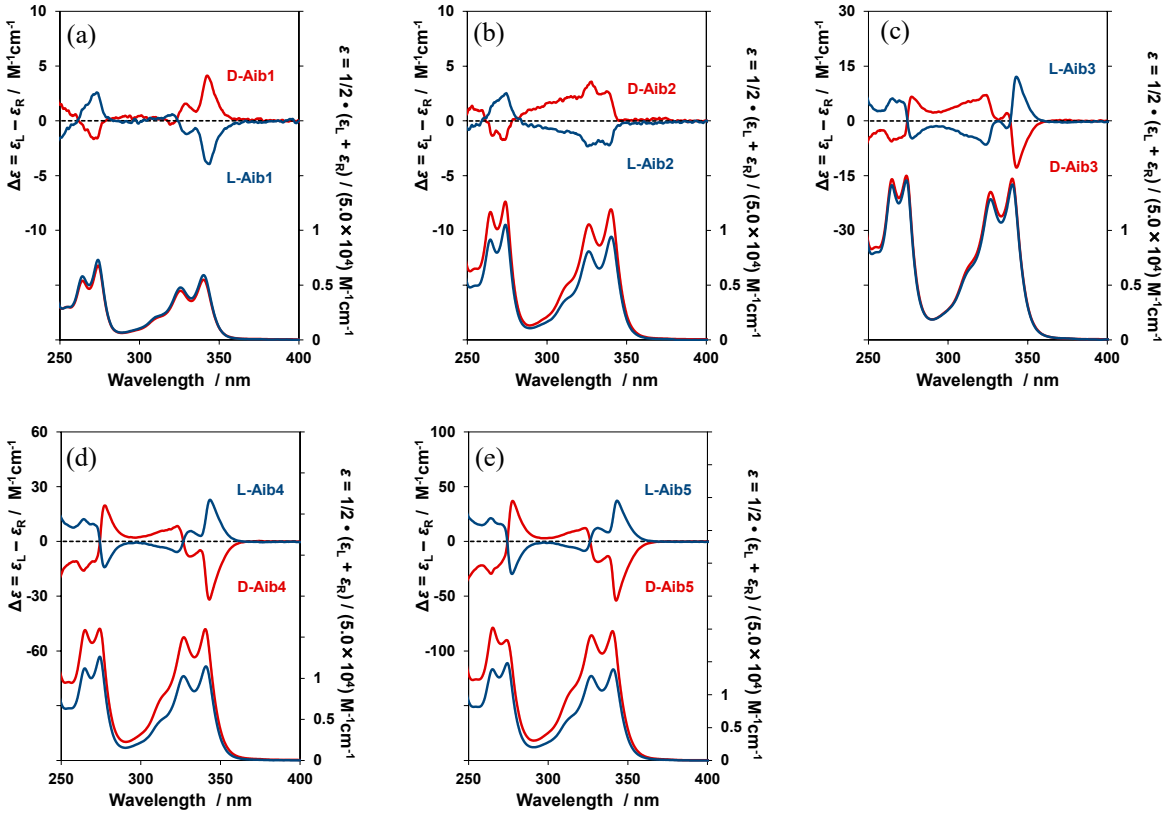

Figure S23. CD (upper panel) and UV–Vis (lower panel) spectra of (**a**) **D-Aib1**/**L-Aib1**; (**b**) **D-Aib2**/**L-Aib2**; (**c**) **D-Aib3**/**L-Aib3**; (**d**) **D-Aib4**/**L-Aib4**; and (**e**) **D-Aib5**/**L-Aib5** in ethanol (1.0×10^{-4} M). D-isomer and L-isomer spectra are shown in red and blue, respectively. Path length = 2 mm.

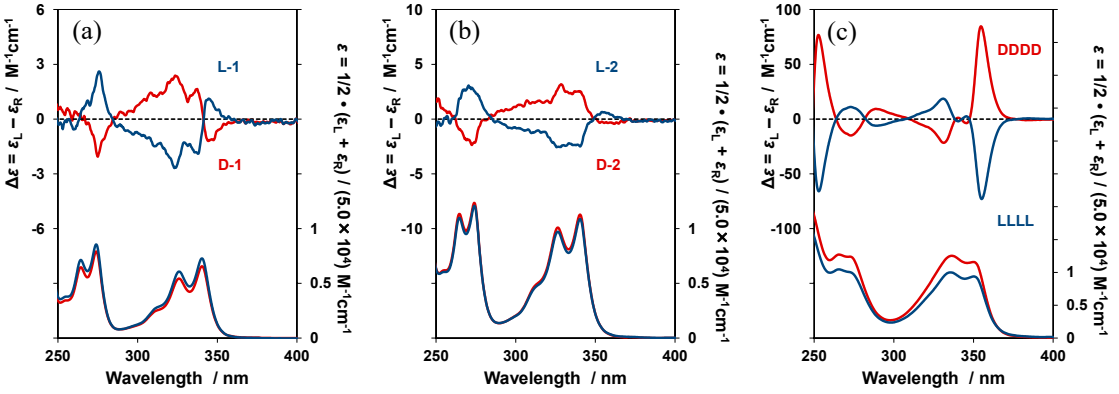

Figure S24. CD (upper panel) and UV–Vis (lower panel) spectra of (a) **D-1** / **L-1**; (b) **D-2** / **L-2**; and (c) **D-3** / **L-3** in ethanol (1.0×10^{-4} M). D-isomer and L-isomer spectra are shown in red and blue, respectively. Path length = 2 mm.

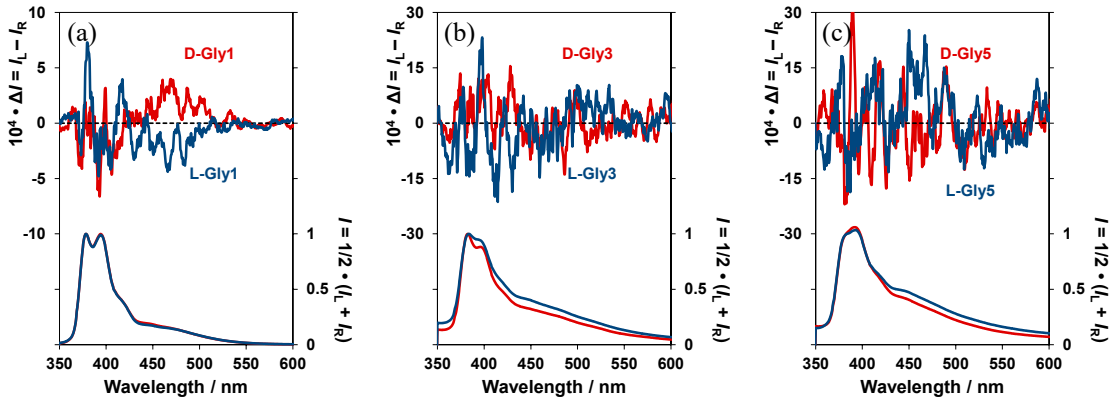

Figure S25. CPL (upper panel) and PL (lower panel) spectra of (**a**) **D-Gly1**/**L-Gly1**; (**b**) **D-Gly3**/**L-Gly3**; (**c**) **D-Gly5**/**L-Gly5** in ethanol (1.0×10^{-4} M). λ_{ex} = 300 nm D-isomer and L-isomer spectra are shown in red and blue, respectively. Path length = 1 mm.

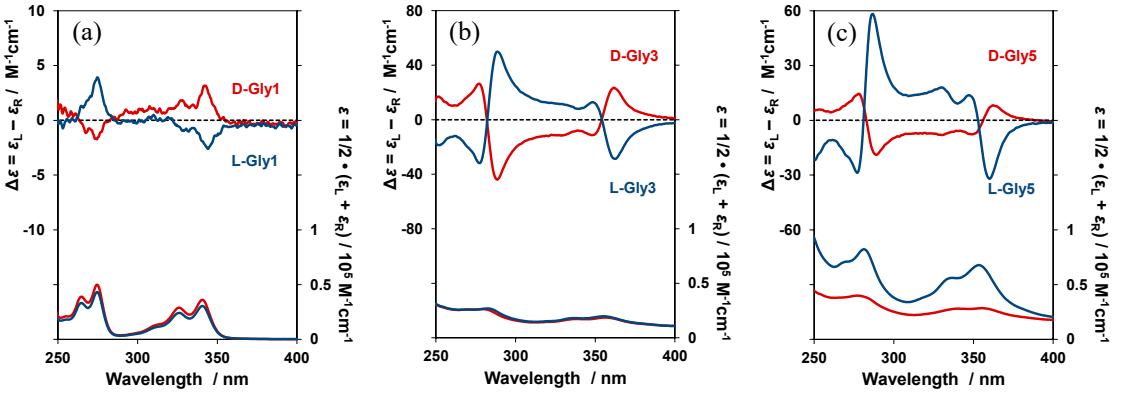

Figure S26. CD (upper panel) and UV–Vis (lower panel) spectra of (**a**) **D-Gly1**/**L-Gly1**; (**b**) **D-Gly3**/**L-Gly3**; (**c**) **D-Gly5**/**L-Gly5** in ethanol (1.0×10^{-4} M). D-isomer and L-isomer spectra are shown in red and blue, respectively. Path length = 1 mm.

Table S1. CPL, PL, CD, and UV–Vis properties of peptide–pyrene luminophores in ethanol.

Name	Monomer PL (nm)	Excimer PL (nm)	Φ_F	g_{CPL} ($\times 10^{-3}$)	λ_{CD} (nm)	g_{CD} ($\times 10^{-4}$)	Note
D-Aib1	378	468.5	0.06	+5.7	343	+1.7	
L-Aib1	378	453.5		-5.0		-1.9	
D-Aib2	394	465.5	0.10	+4.7	338	+0.51	
L-Aib2	378	458.5		-4.7		-4.9	
D-Aib3	394	456.5	0.13	+5.5	343	-2.2	
L-Aib3	394.5	457		-6.0		+2.1	
D-Aib4	394	456.5	0.16	+6.3	343	-4.8	
L-Aib4	395	458.5		-7.2		+4.8	
D-Aib5	395	456.5	0.18	+2.3	343	-6.2	
L-Aib5	395	456.5		-2.9		+6.1	
D-Gly1	379	469	0.09	+2.7	341	+0.95	
L-Gly1	379	466		-2.9		-1.1	
D-Gly3	ND	ND	0.05	ND	362	+13	Aggregated
L-Gly3	ND	ND		ND		-16	Aggregated
D-Gly5	ND	ND	0.9	ND	361	+3.1	Aggregated
L-Gly5	ND	ND		ND		-5.5	Aggregated
D-1	378	479	0.06	+7.3	348	-1.2	
L-1	378	462.5		-7.8		+0.48	
D-2	394	461.5	0.13	ND	361	-6.4	
L-2	394	462		ND		+2.3	
D-3	406.5	ND	0.10	ND	355	+18	Aggregated
L-3	406	ND		ND		-19	Aggregated