**Supplementary Materials**

**Novel peptide-drug conjugates with dual anticancer activity**

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ESI-MS and RP-HPLC for L-K6 peptide amide.

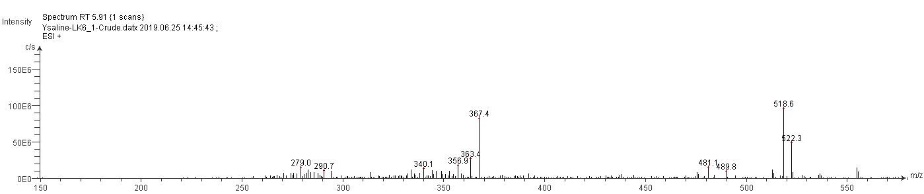


Figure S1 ESI-MS (m/z): Calcd for C75H146N20O14 1551.13, Found: 518.6 [M+3H]3+.

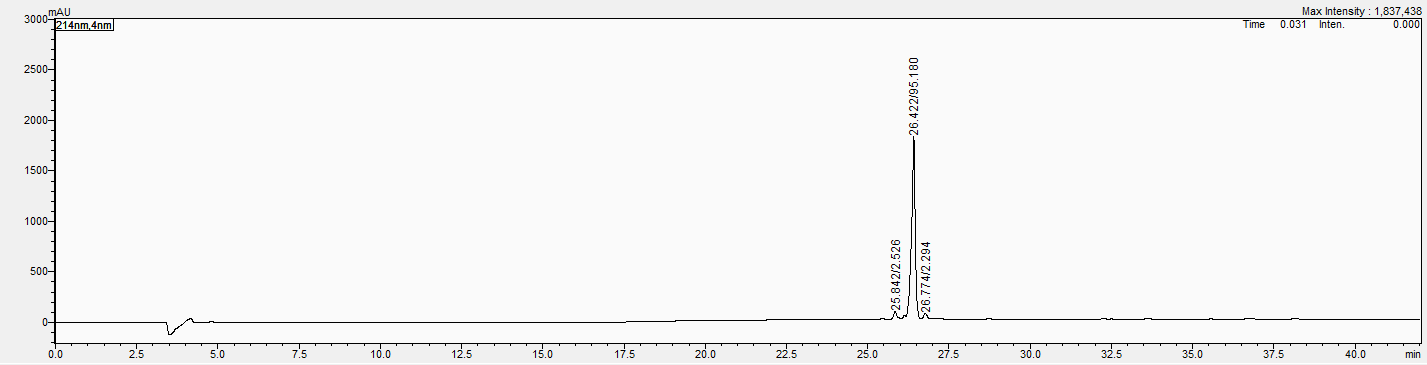


Figure S2 RP-HPLC: tR = 26.422 min, 95% purity.

ESI-MS, RP-HPLC, and MALDI-TOF MS of hydrazinoacetylated L-K6 peptide, **6**

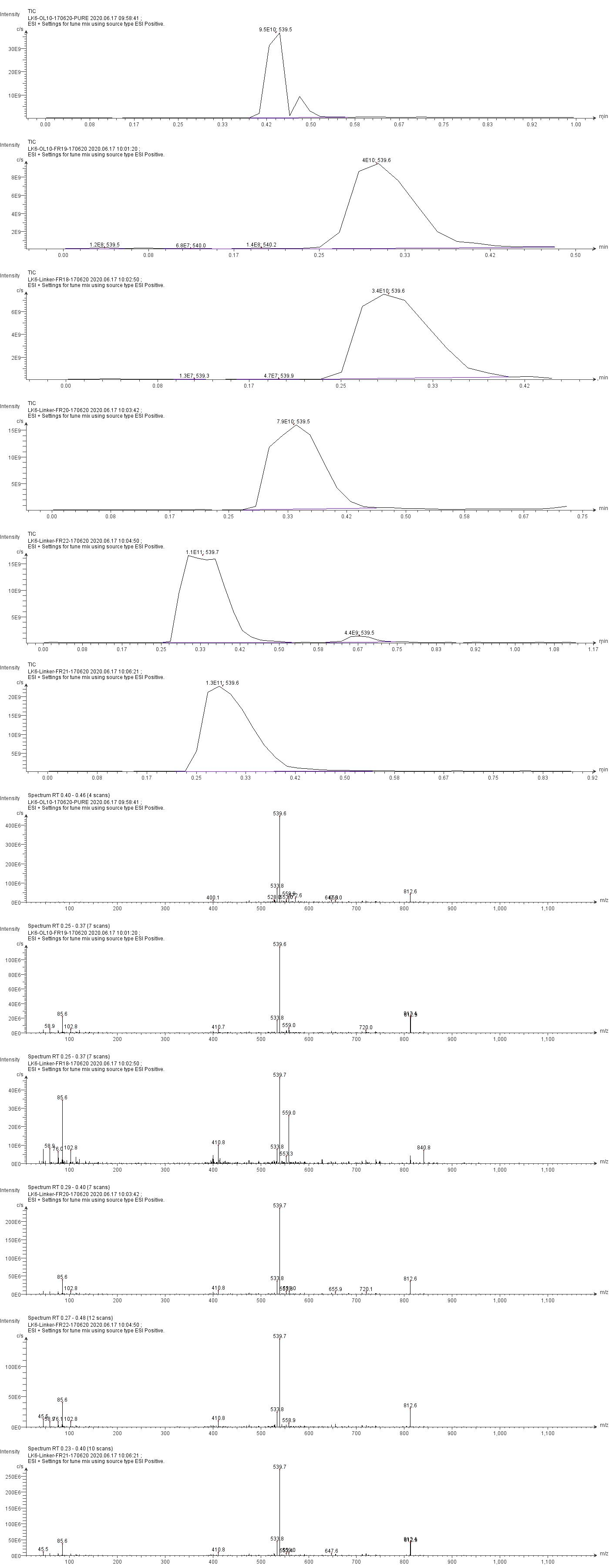


Figure S3 ESI-MS (m/z): Calcd for C77H150N22O15 1624.15, Found: 812.6 [M+2H]2+.

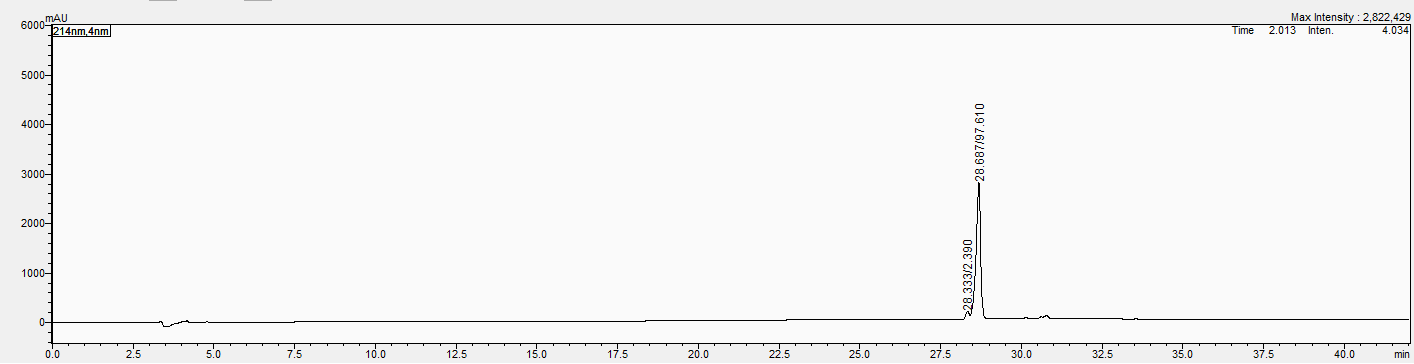


Figure S4 RP-HPLC: tR = 28.687 min, 97% purity.

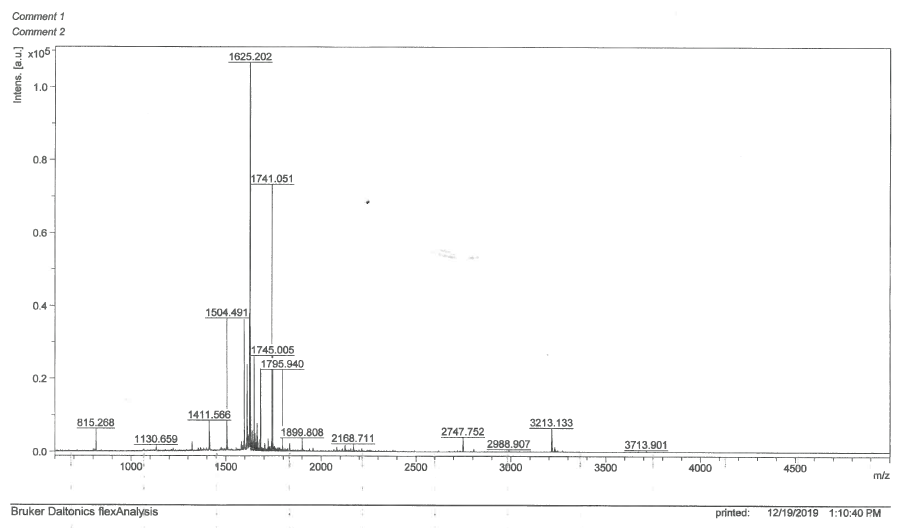


Figure S5 MALDI-TOF MS (m/z): Calcd for C77H150N22O15 1624.15, Found: 1625.202 [M+H]+.

ESI-MS, RP-HPLC, and MALDI-TOF MS for the conjugate **7** from the hydrazinothiazole-based usnic acid derivative **4** and the hydrazinoacetylated peptide **6**.

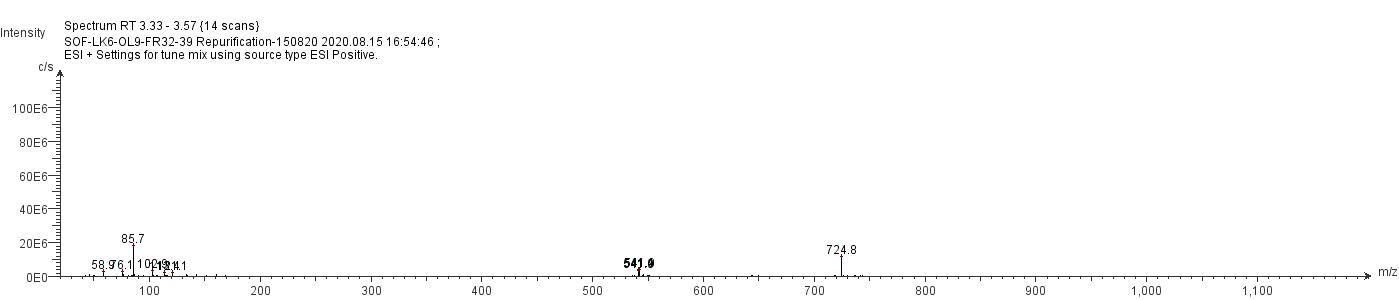


Figure S6 ESI-MS (m/z): Calcd for C103H166BrN25O19S 2170.58, Found: 724.4 [M+3H]3+.

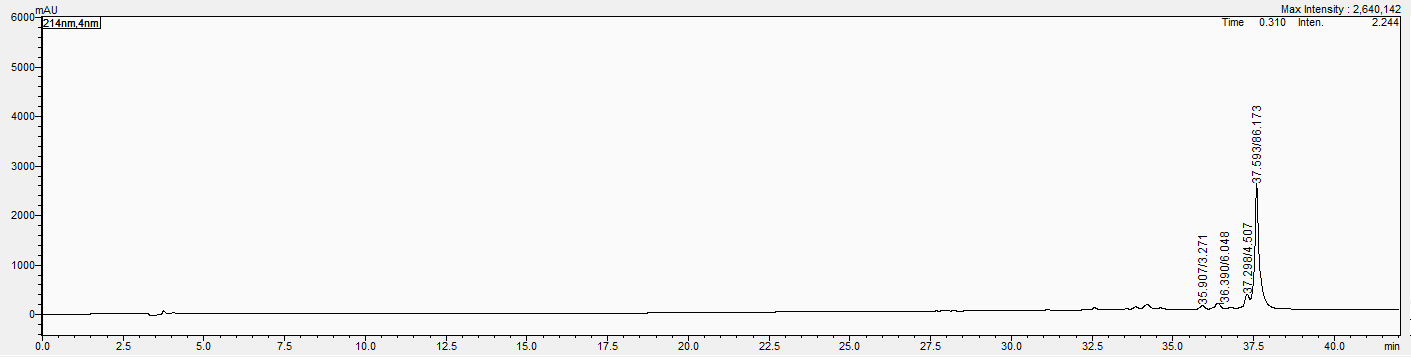


Figure S7 RP-HPLC: tR = 37.593 min, 86% purity.

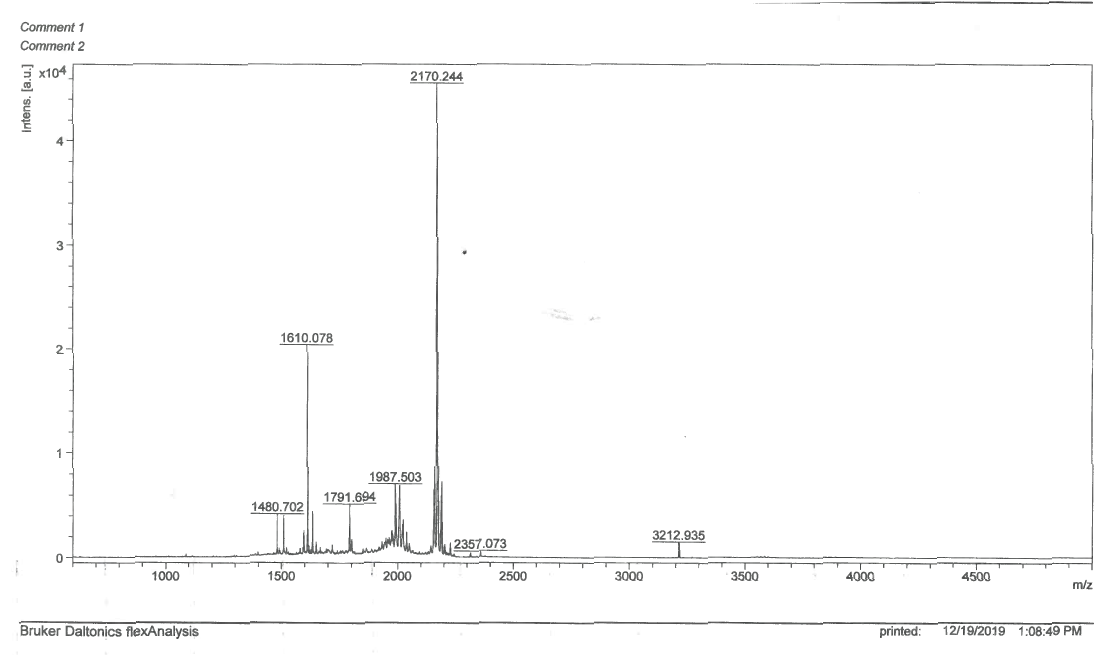


Figure S8 MALDI-TOF MS (m/z): Calcd for C103H166BrN25O19S: 2170.58, Found: 2170.244 [M+].

ESI-MS, RP-HPLC, and MALDI-TOF MS for conjugate **8** from the benzylidenefuranone-based usnic acid derivative **5** and the hydrazinoacetylated peptide **6**.

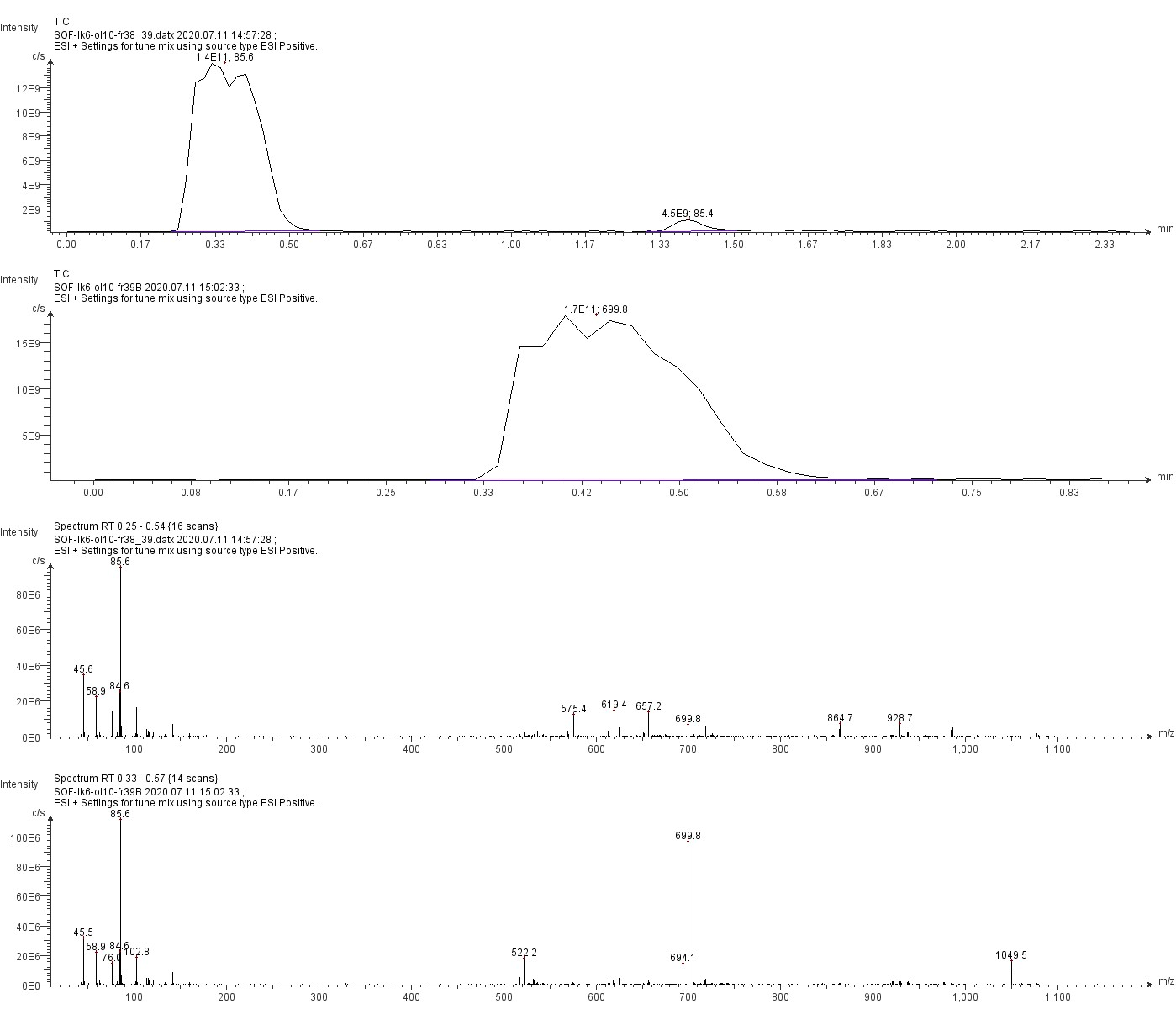


Figure S9 ESI-MS (m/z): Calcd for C102H163BrN22O20 2095.16, Found: 699.8 [M+3H]3+, 1049.5 [M+2H]2+.

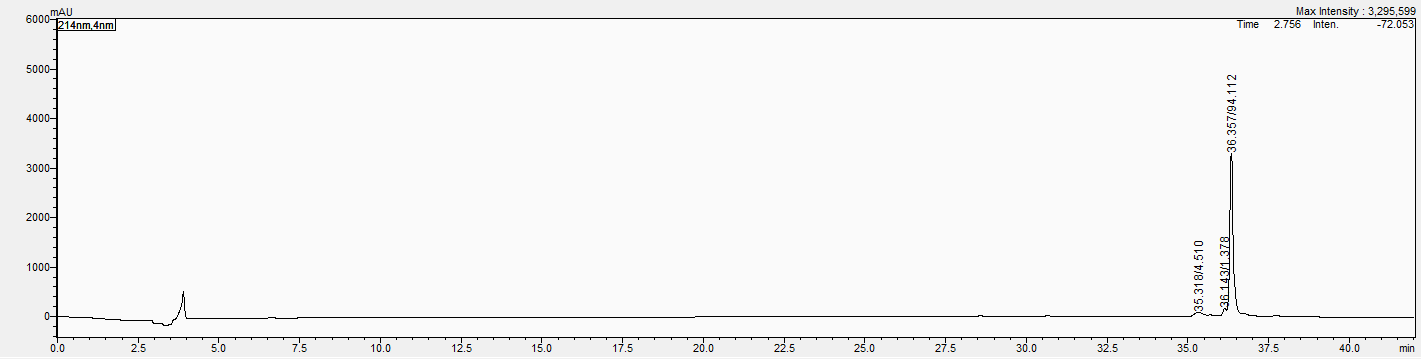


Figure S10 RP-HPLC: tR = 36.357 min, 94% purity.

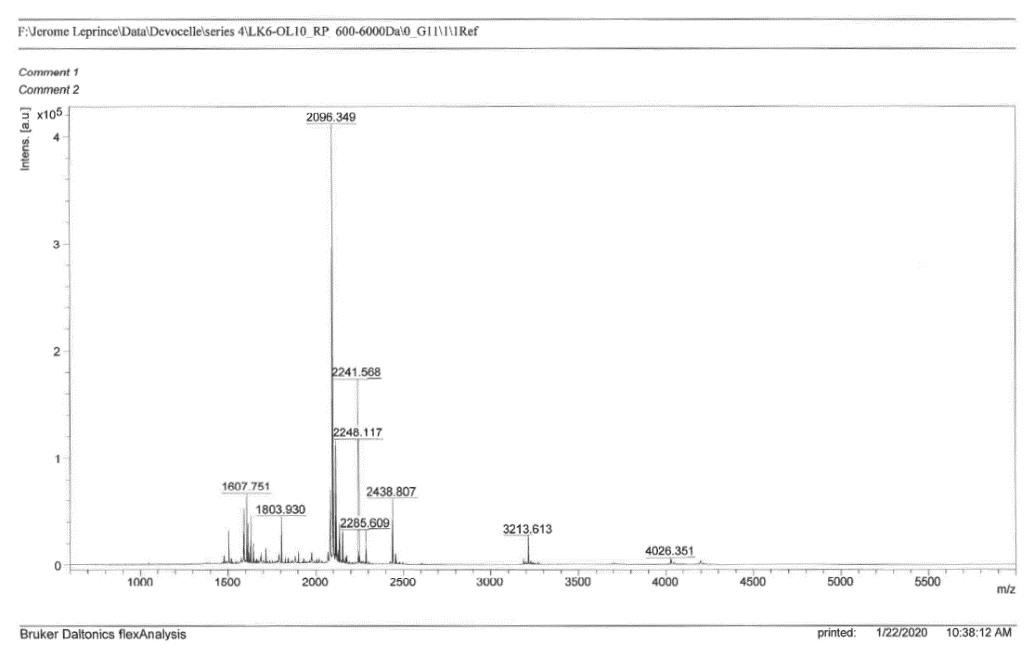


Figure S11 MALDI-TOF MS (m/z): Calcd for C102H163BrN22O20 2095.16, Found: 2096.349 [M+H]+.