

Supporting Information

A Rink-Amide Soluble Support: High Purity Conotoxins and Other Peptides Accessed with Minimal Reagents

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NMR spectra of compounds

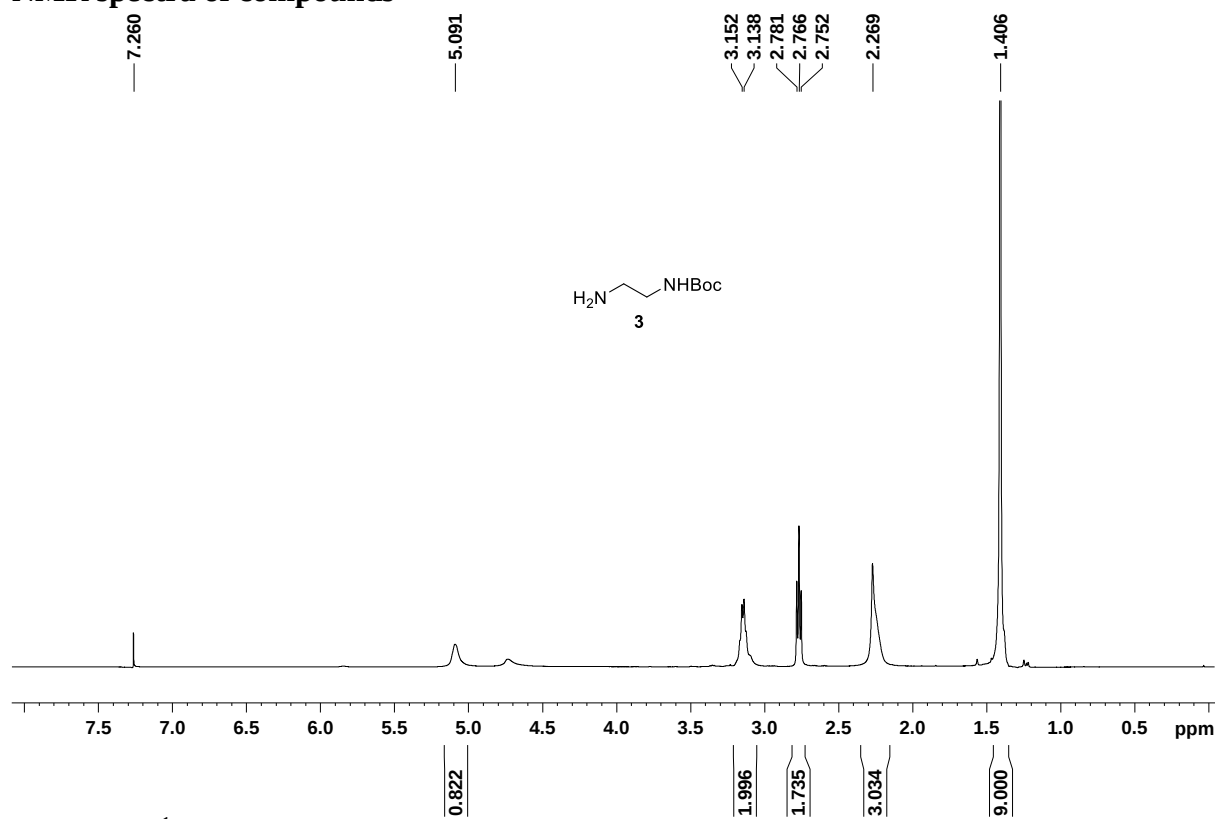


Figure S1. ¹H NMR spectrum of compound 3.

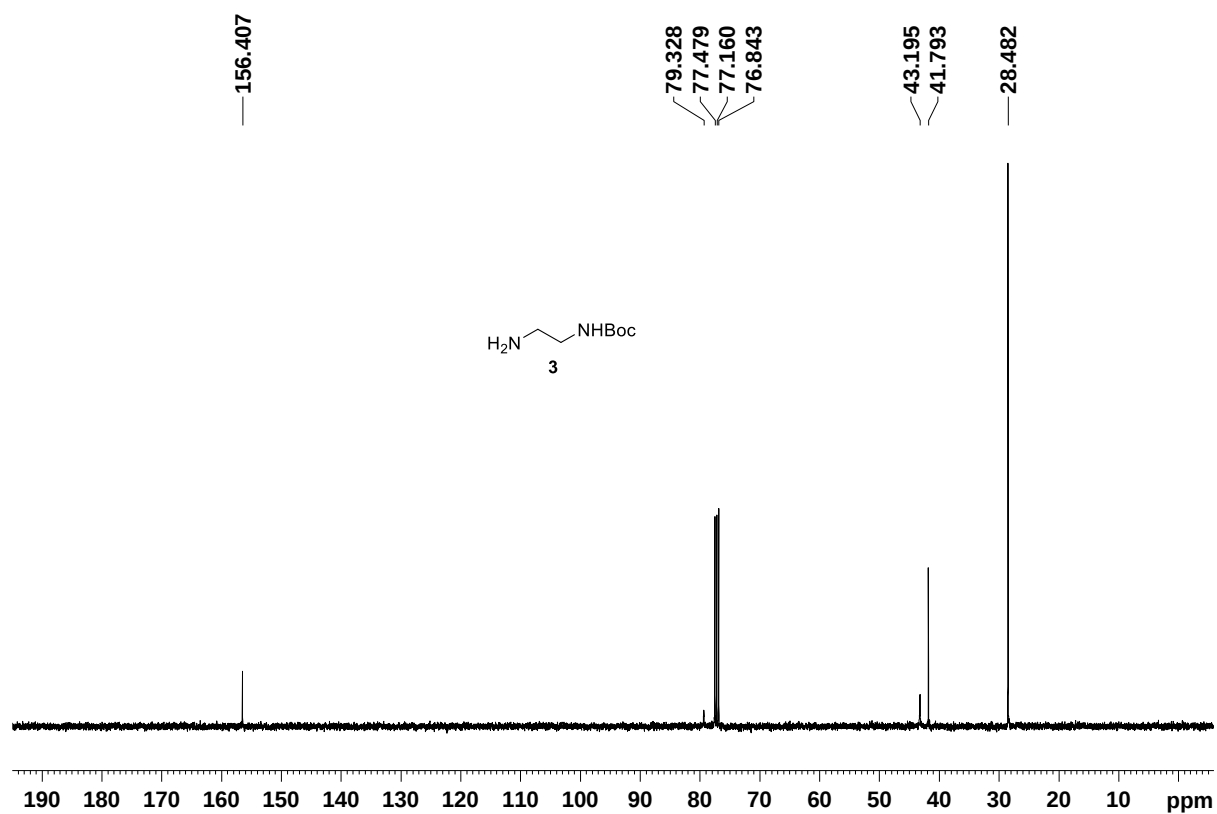


Figure S2. ¹³C NMR spectrum of compound 3.

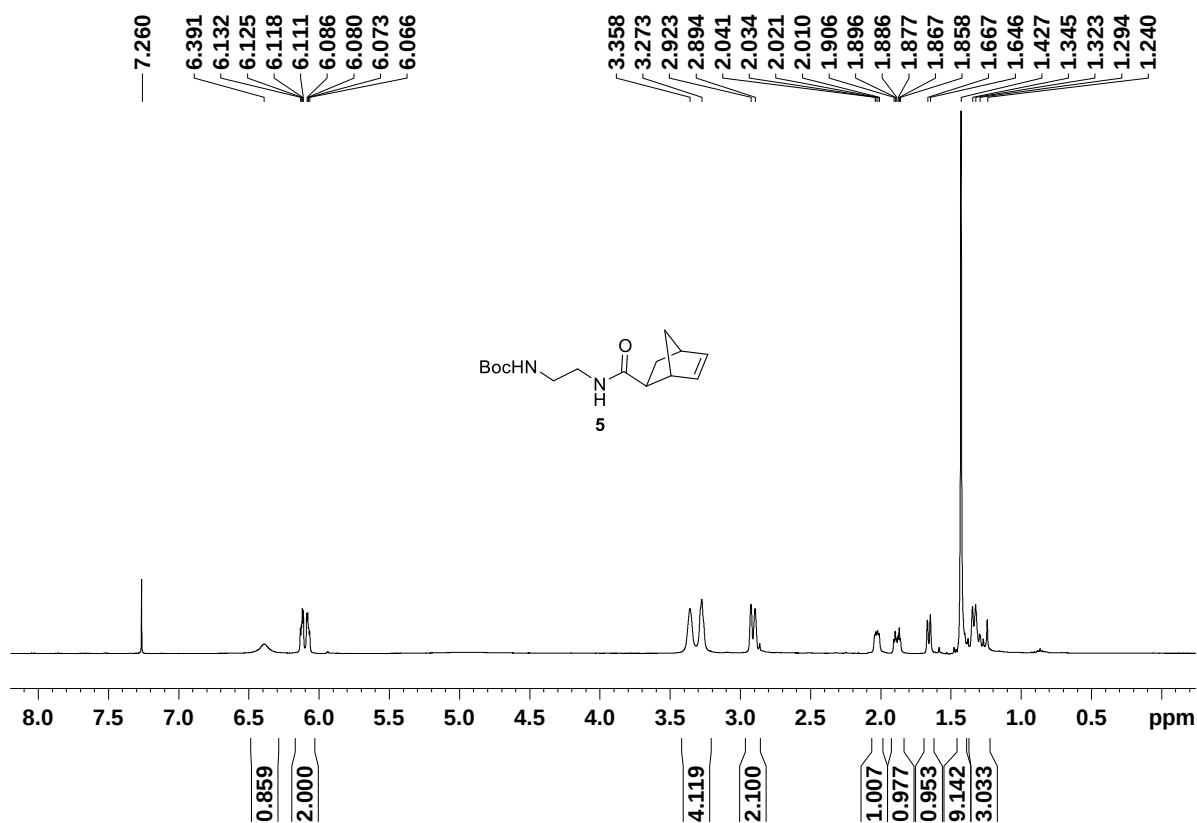


Figure S3. ¹H NMR spectrum of compound 5.

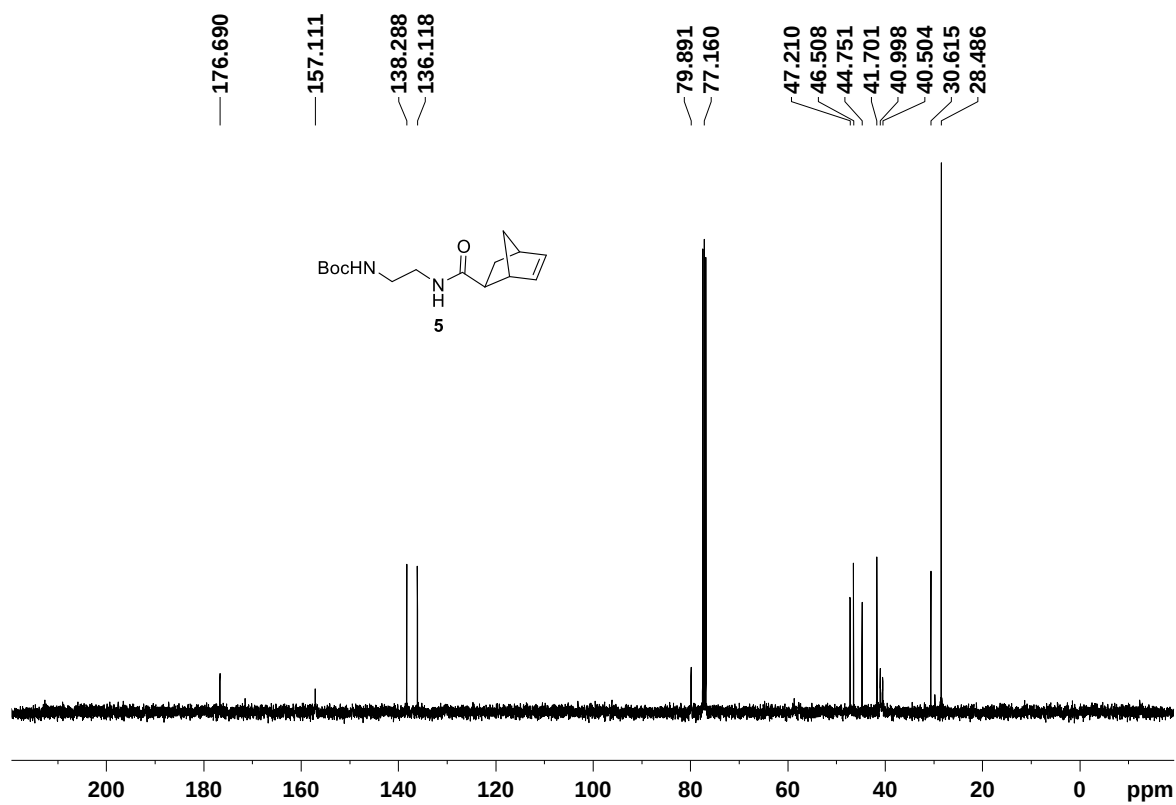


Figure S4. ¹³C NMR spectrum of compound 5.

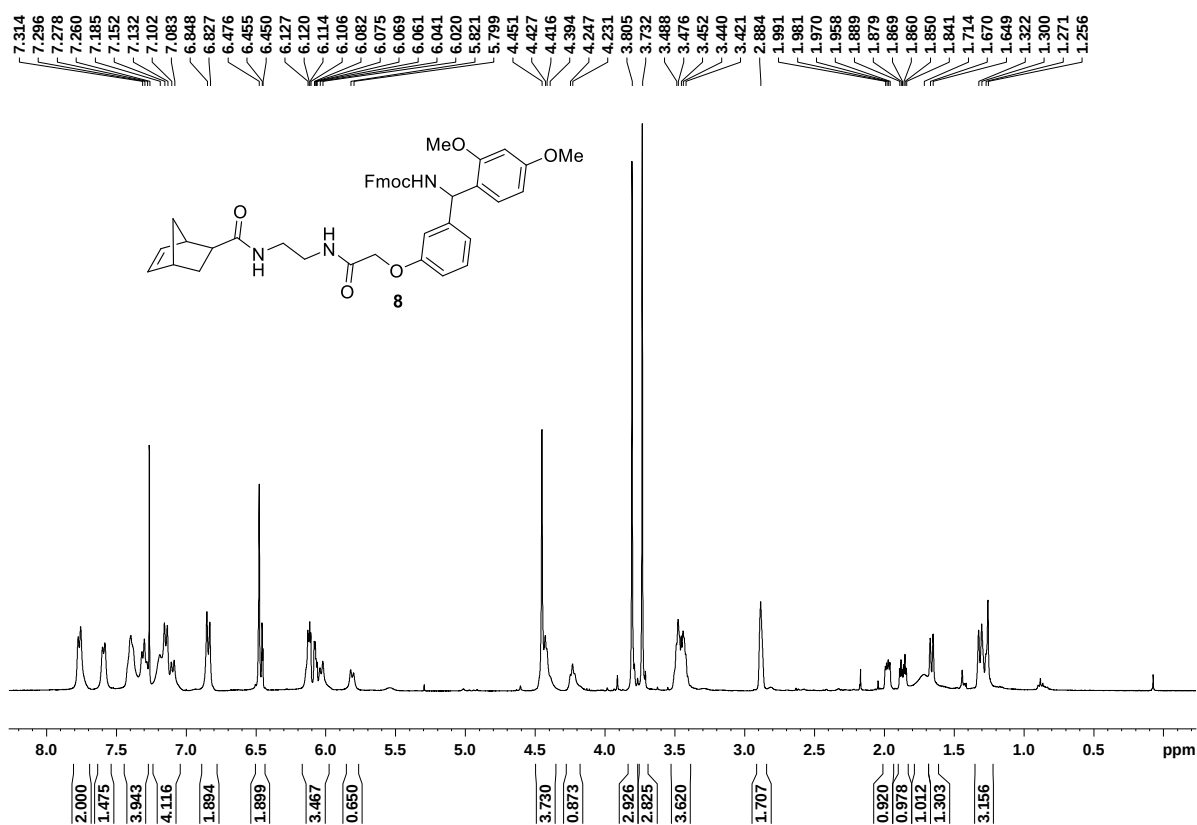


Figure S5. ¹H NMR spectrum of compound 8.

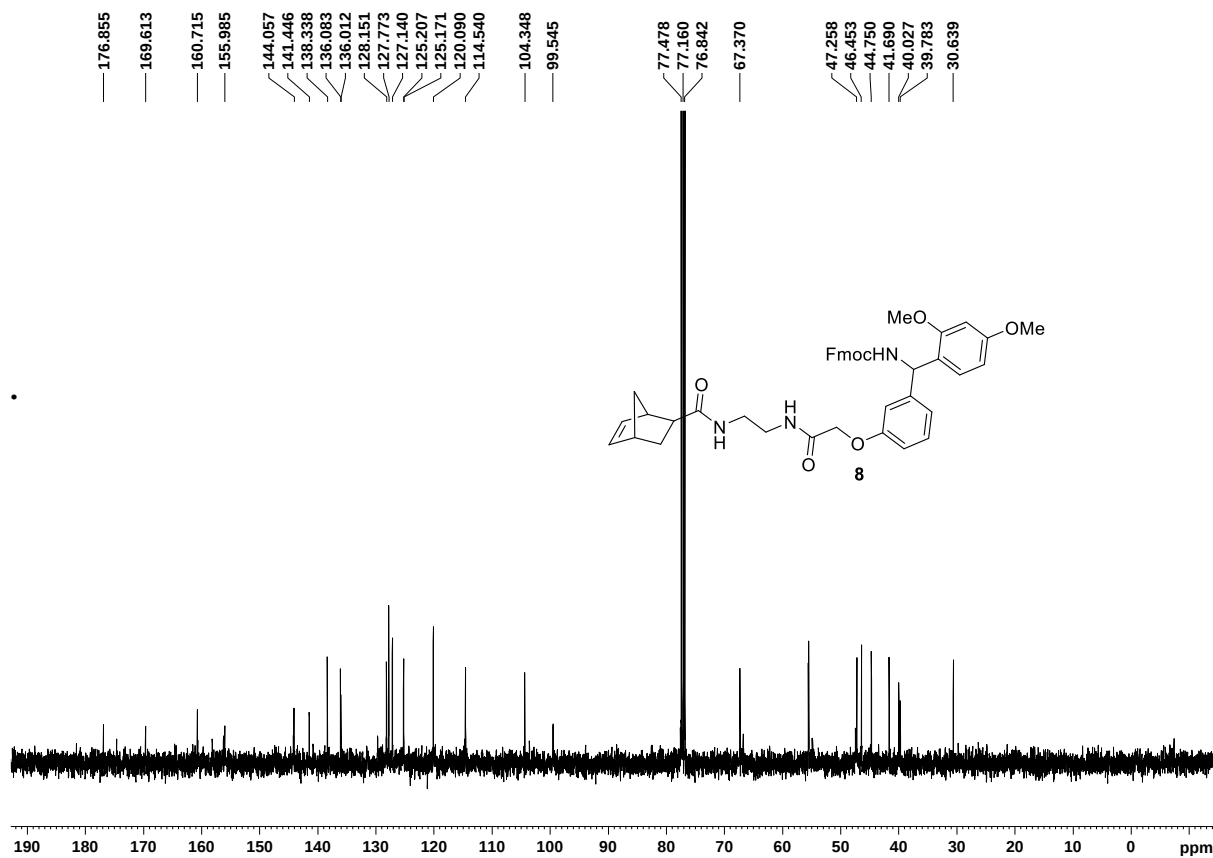


Figure S6. ¹³C NMR spectrum of compound 8.

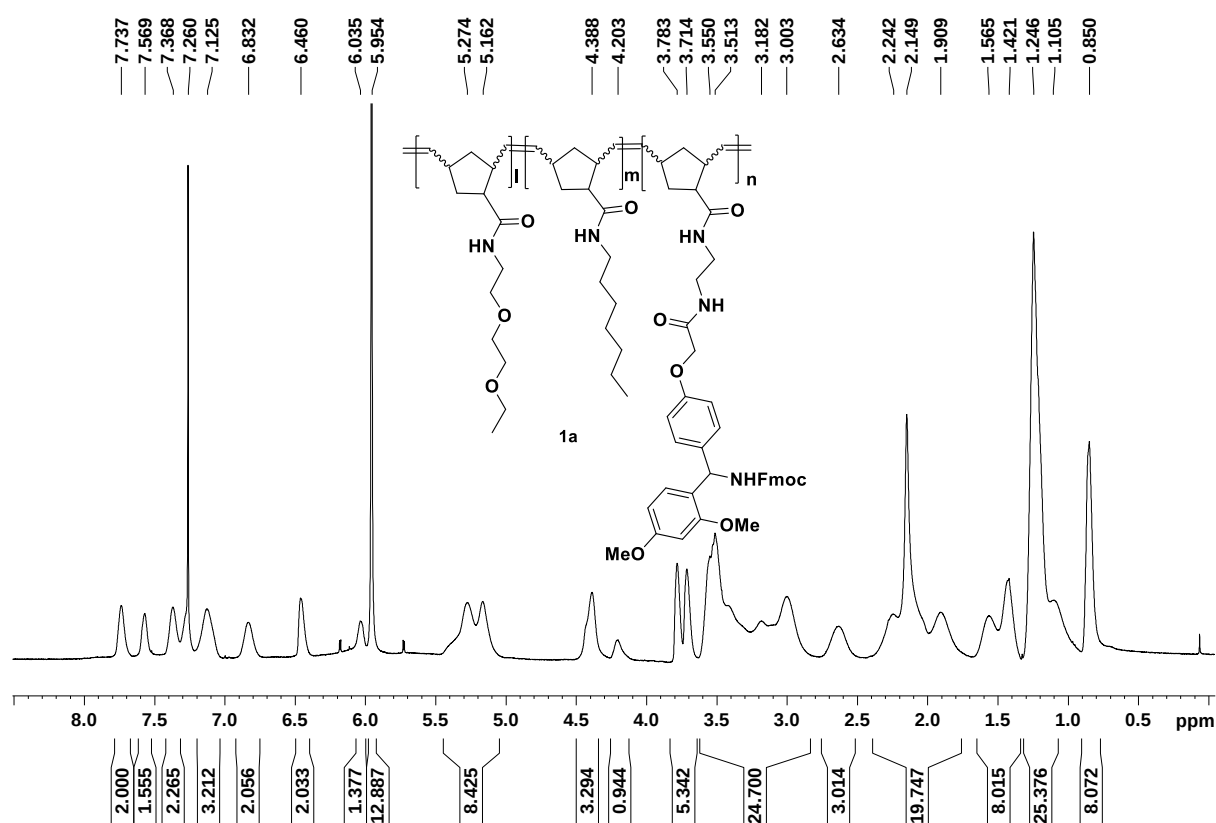


Figure S7. ¹H NMR spectrum of compound 1a.

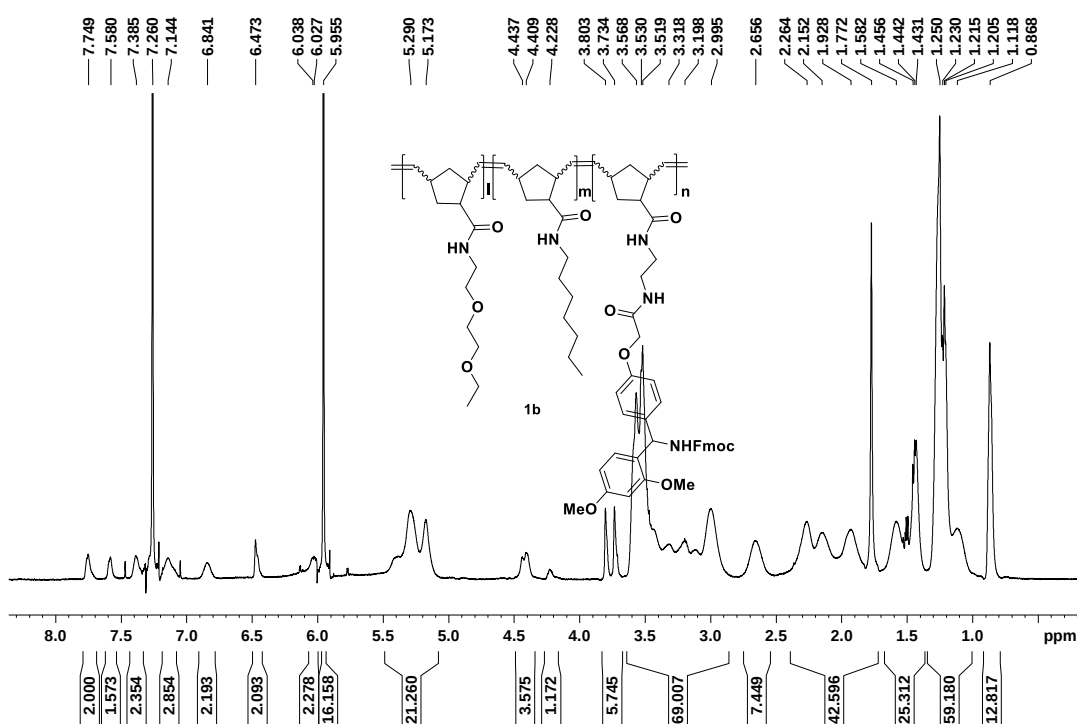


Figure S8. ¹H NMR spectrum of compound 1b.

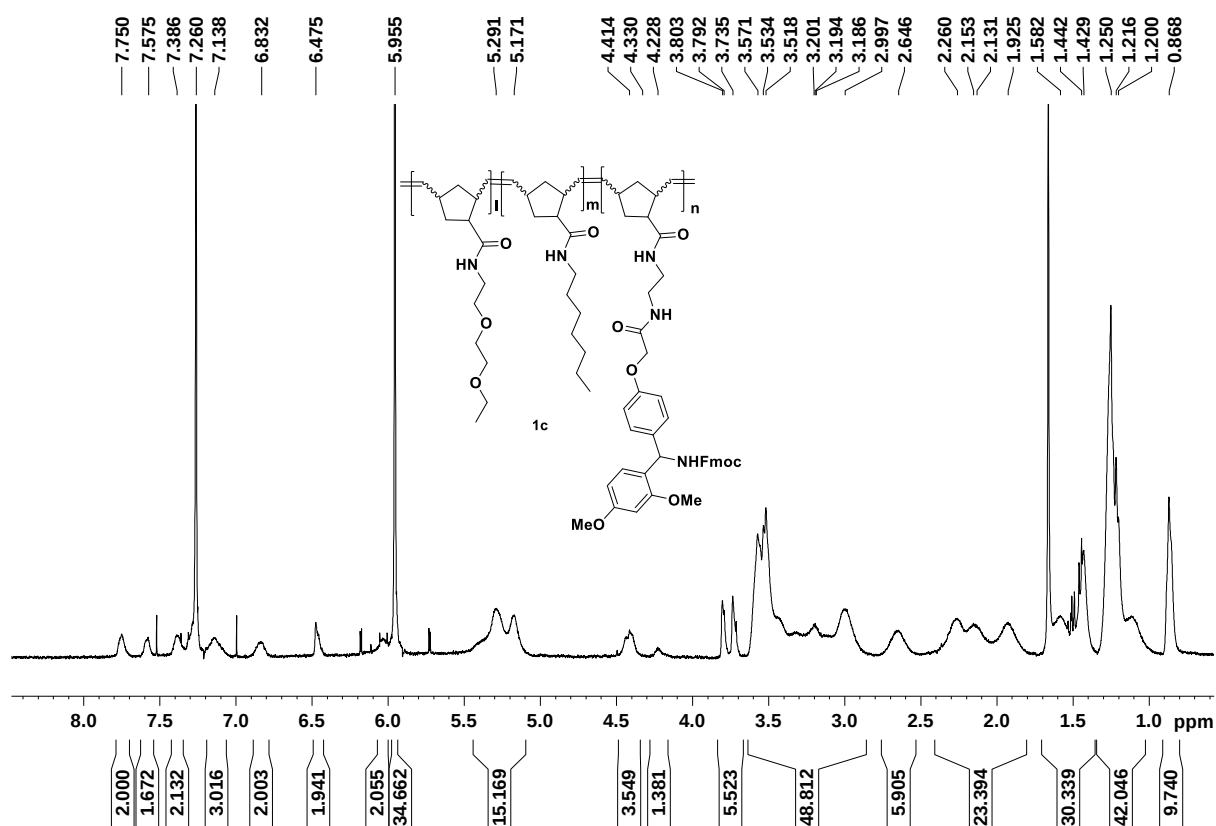


Figure S9. ¹H NMR spectrum of compound 1c.

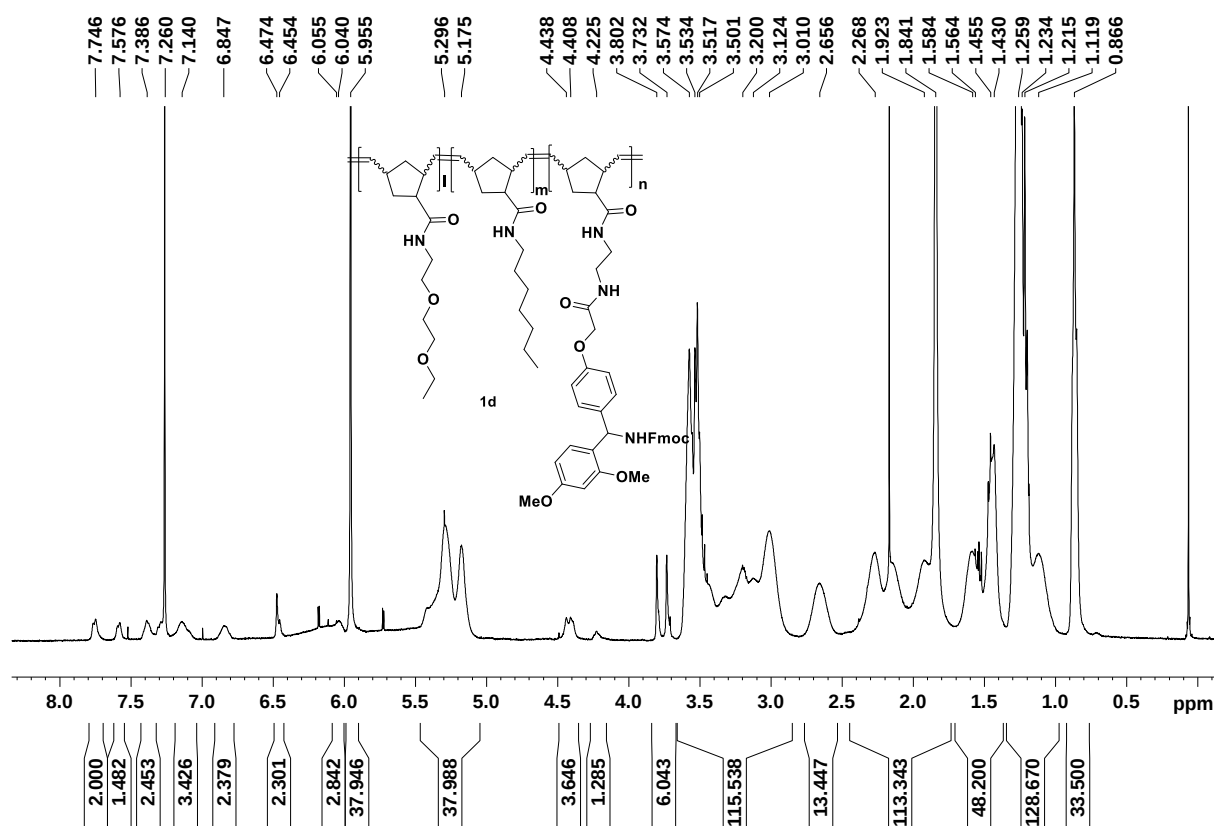


Figure S10. ¹H NMR spectrum of compound 1d.

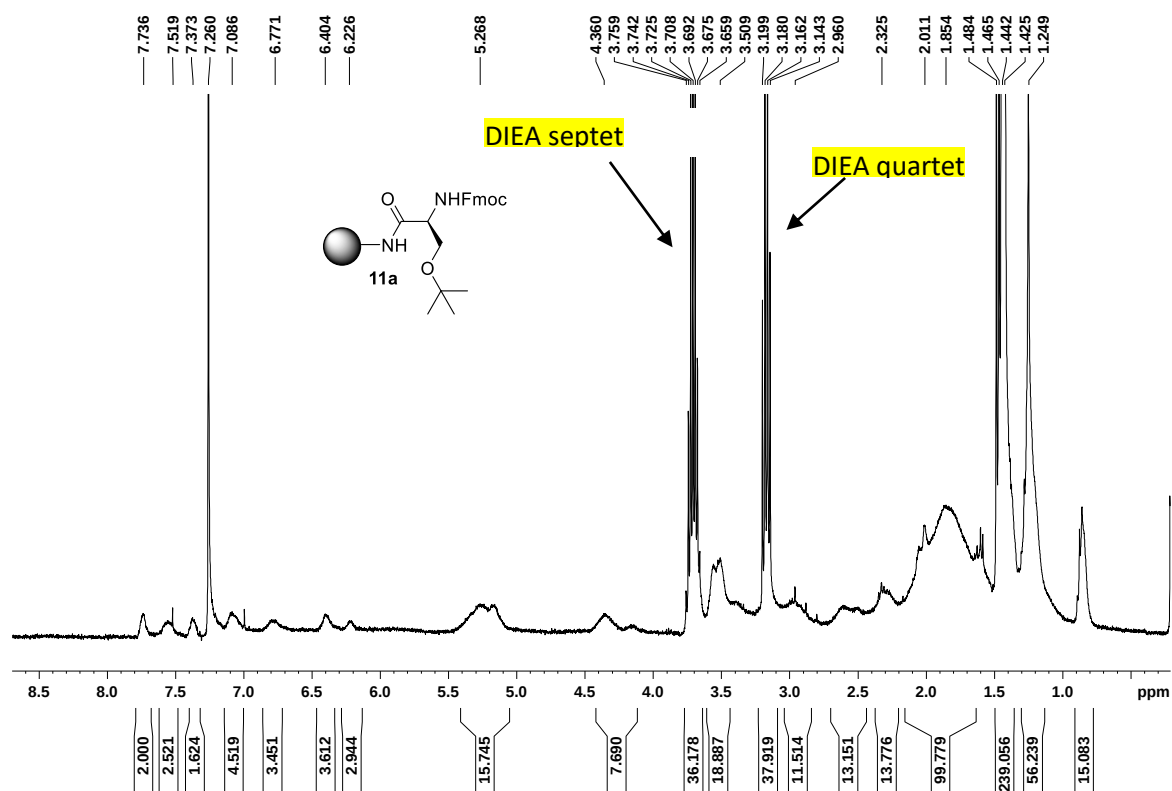


Figure S11. ^1H NMR spectrum of compound **11a**.

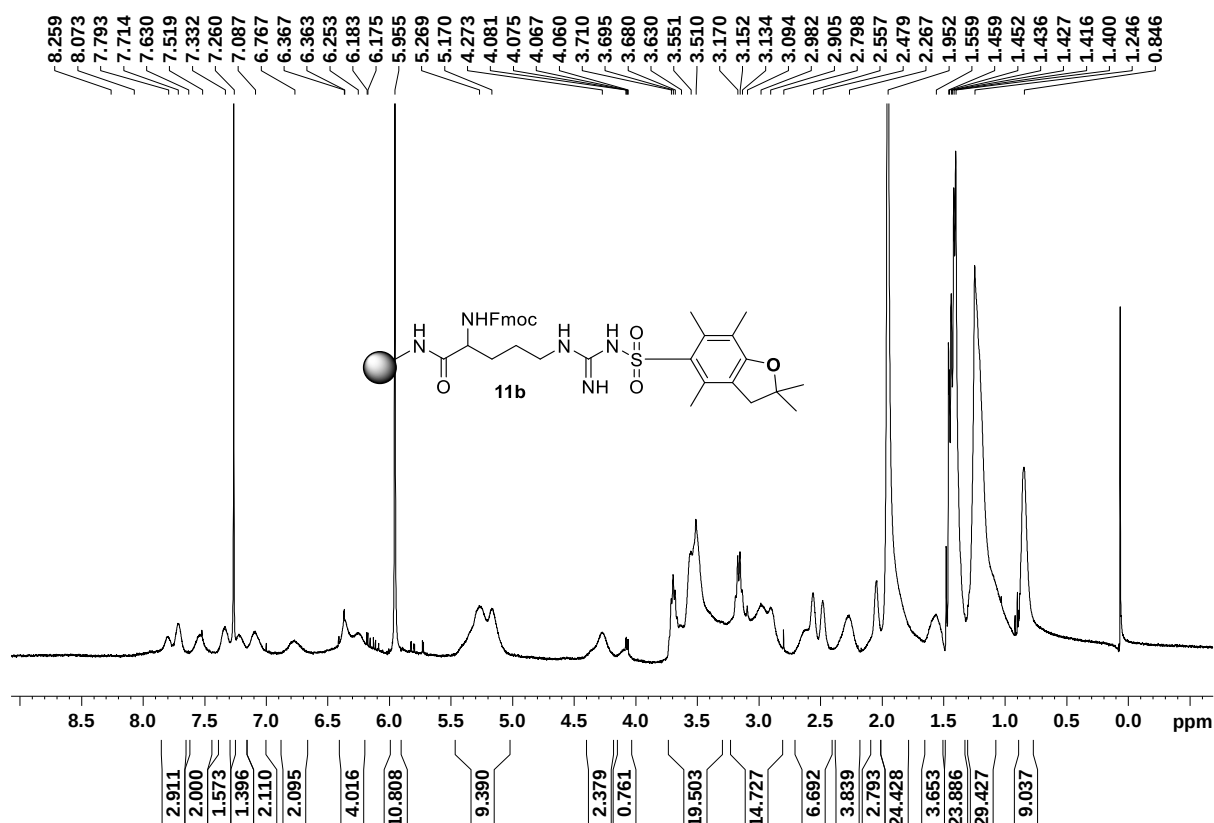


Figure S12. ^1H NMR spectrum of compound **11b**.

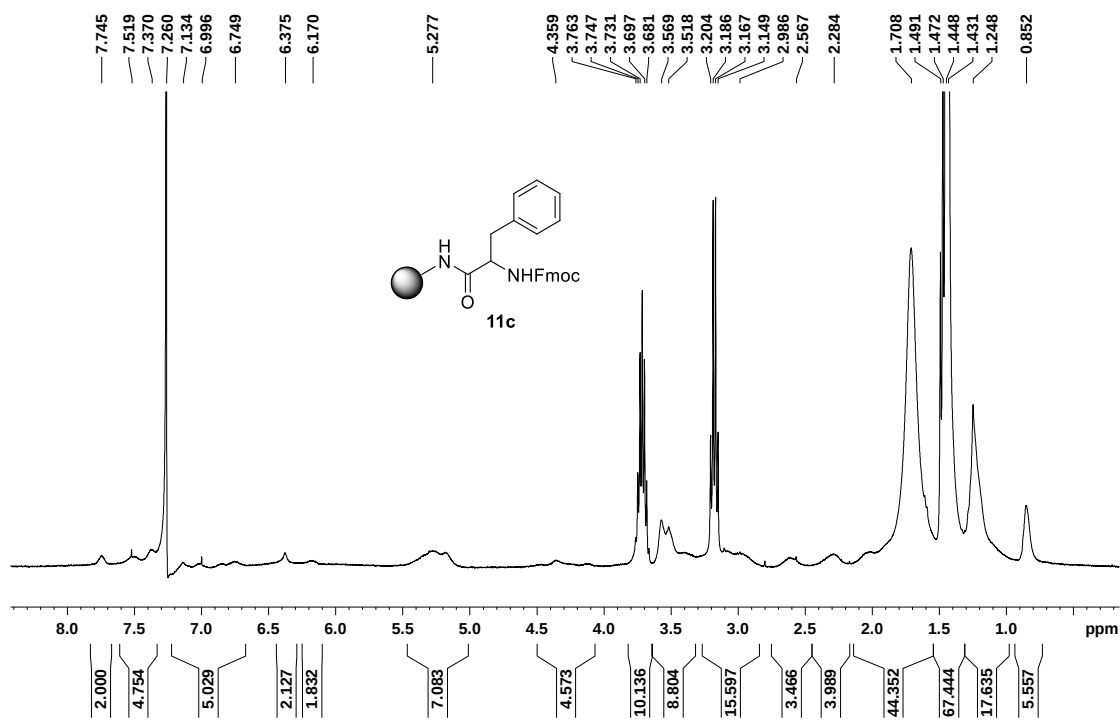


Figure S13. ¹H NMR spectrum of compound 11c.

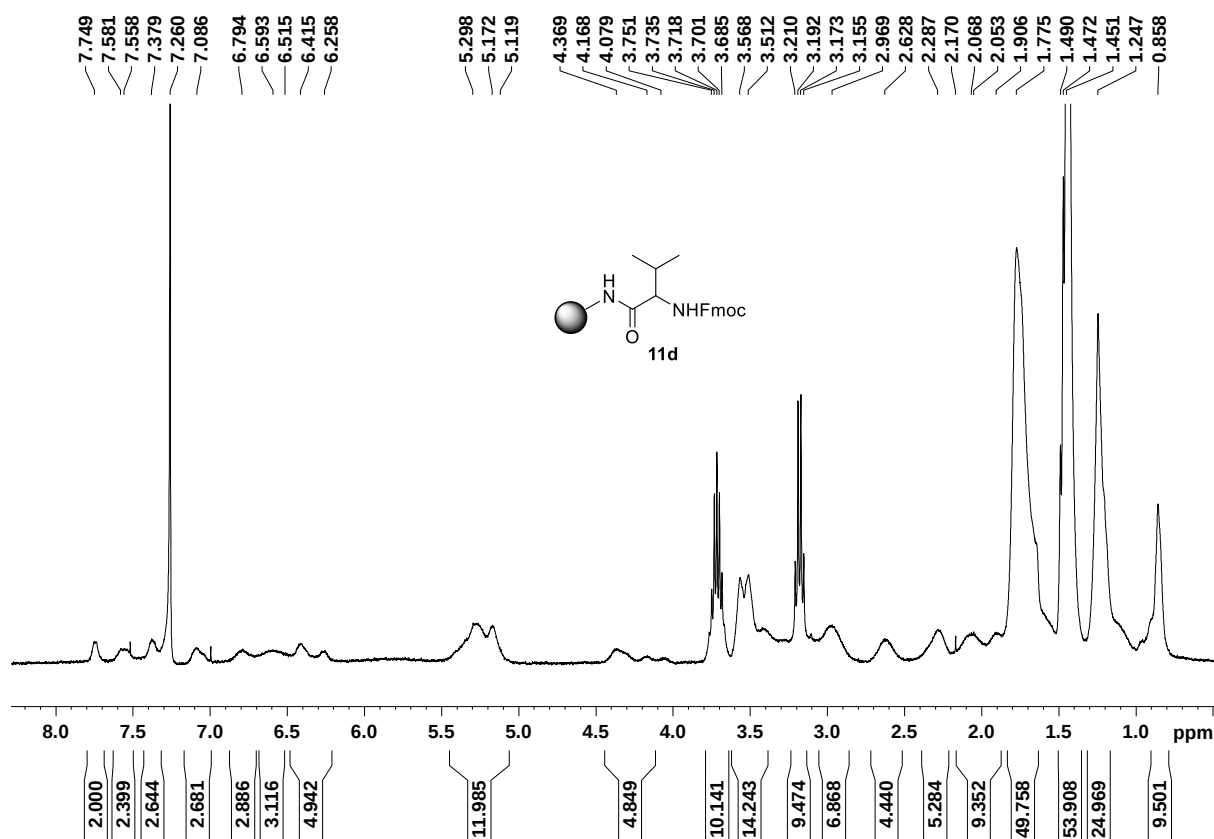


Figure S14. ¹H NMR spectrum of compound 11d.

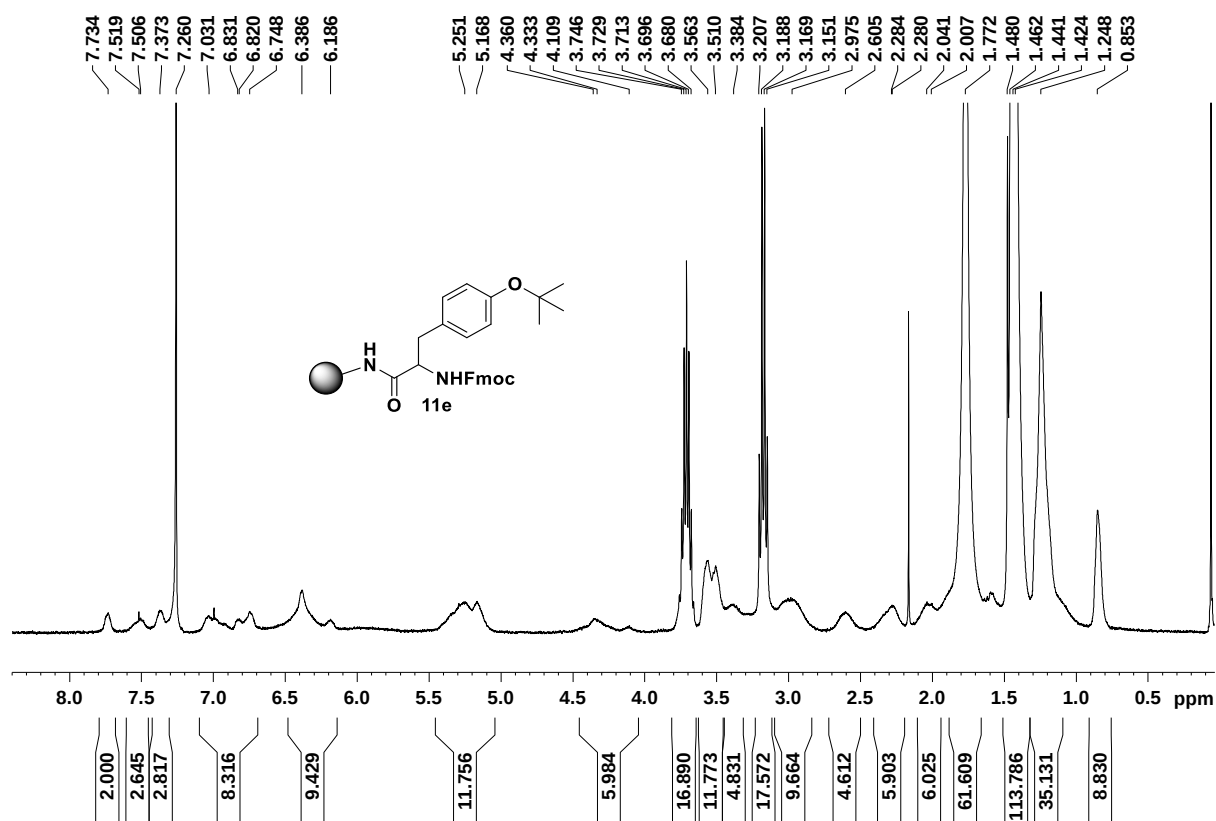


Figure S15. ¹H NMR spectrum of compound 11e.

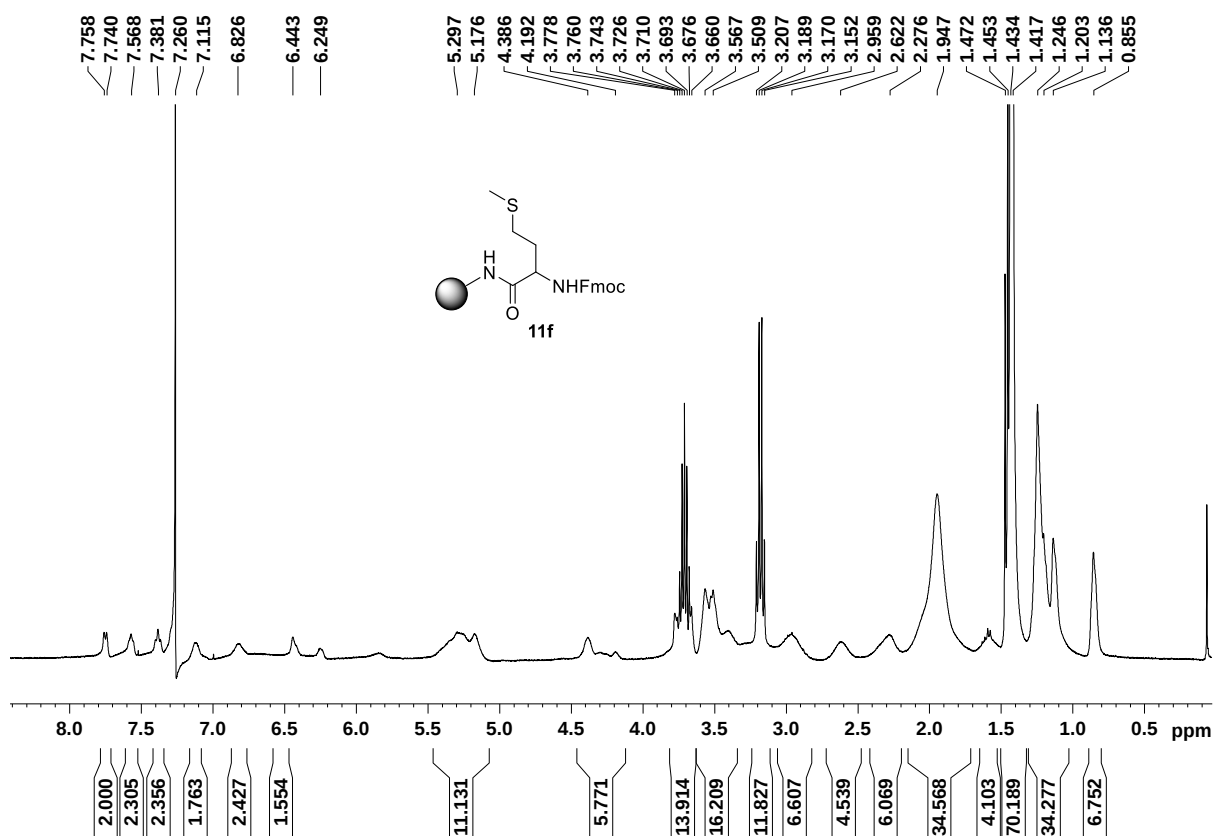


Figure S16. ¹H NMR spectrum of compound 11f.

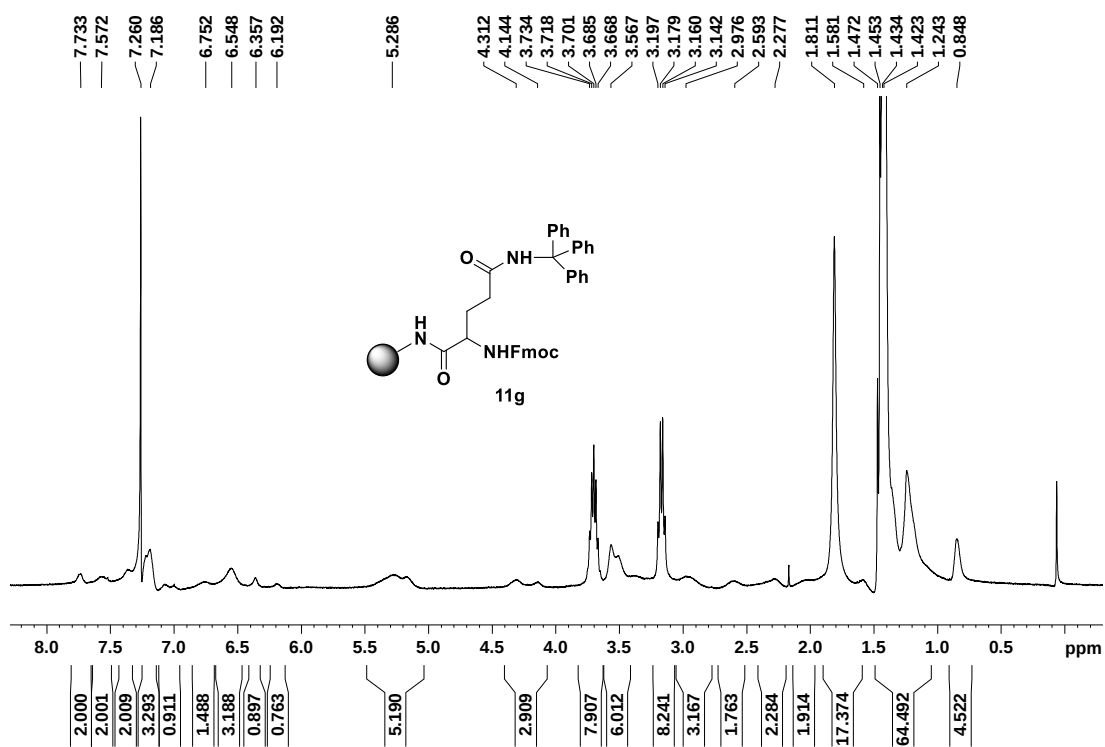


Figure S17. ¹H NMR spectrum of compound **11g**.

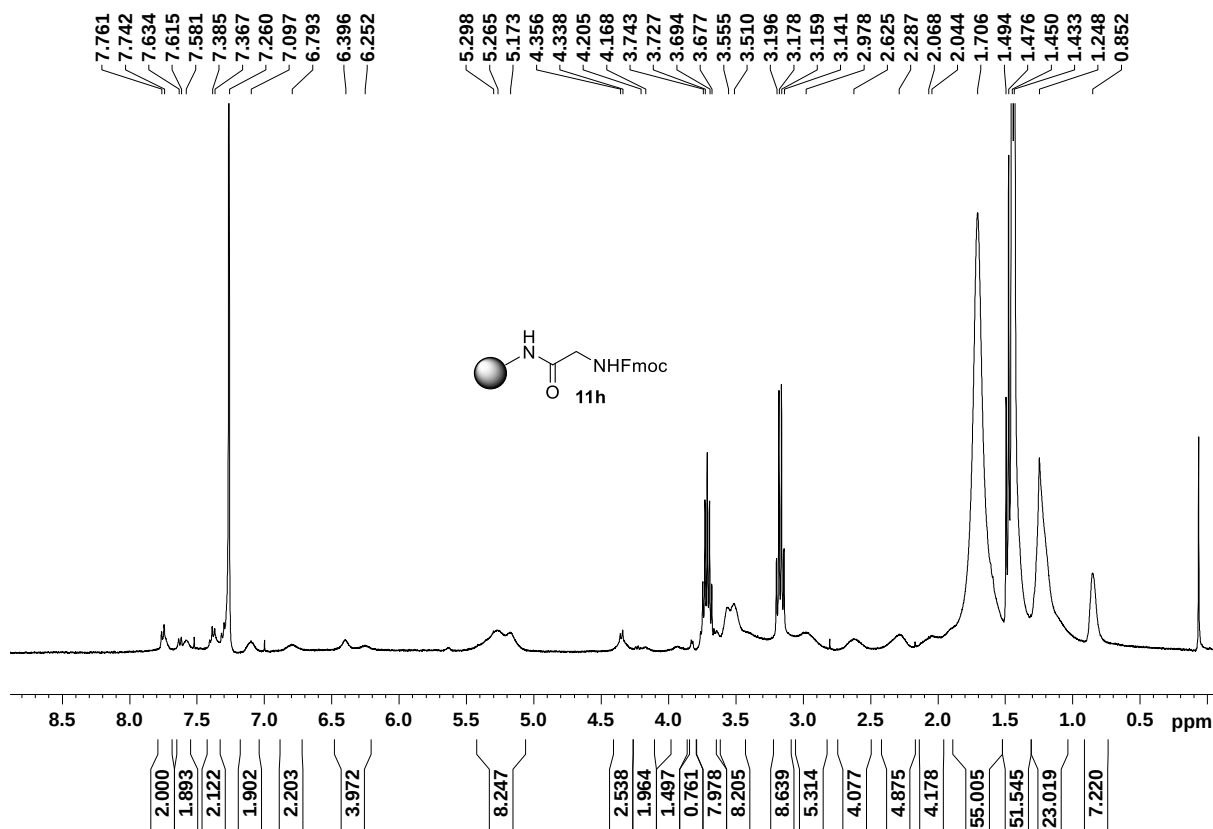


Figure S18. ¹H NMR spectrum of compound **11h**.

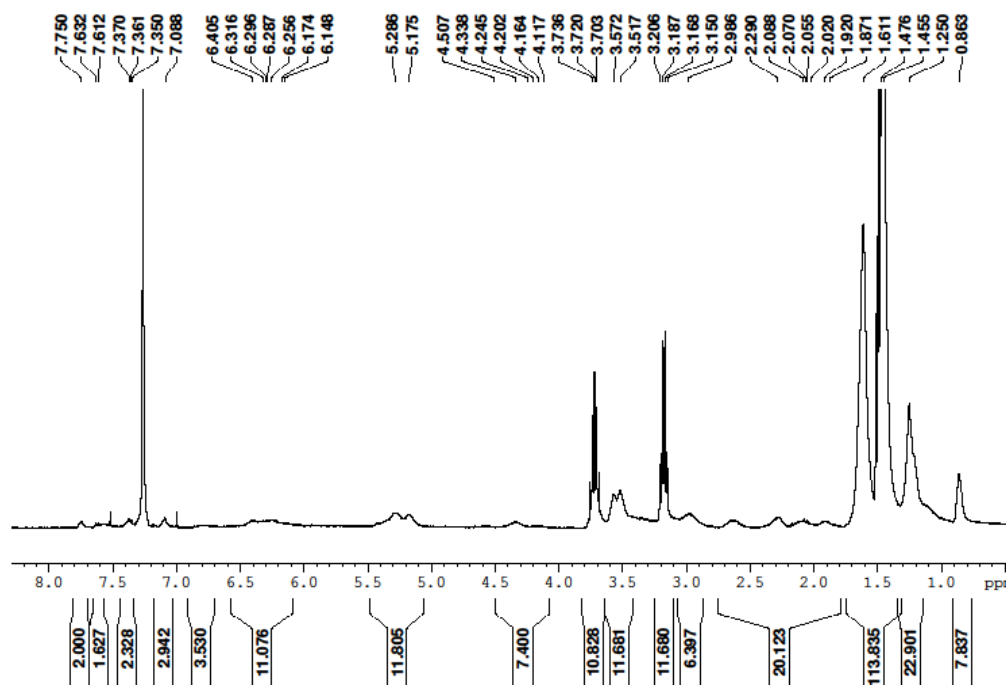
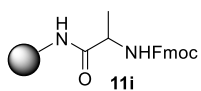


Figure S19. ^1H NMR spectrum of compound **11i**

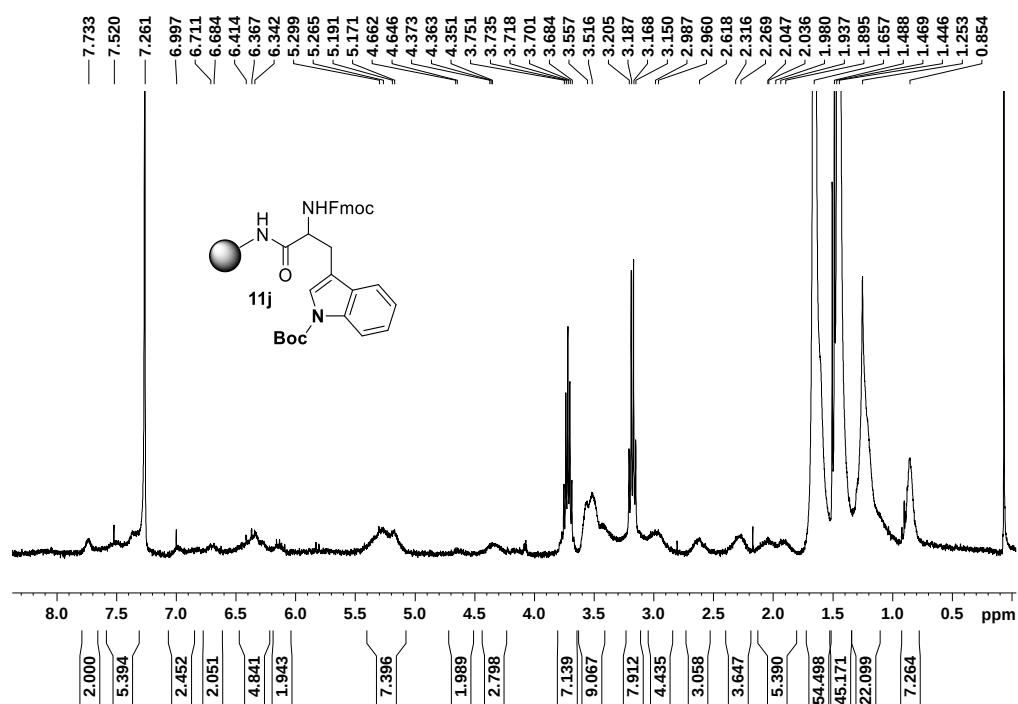


Figure S20. ^1H NMR spectrum of compound **11j**.

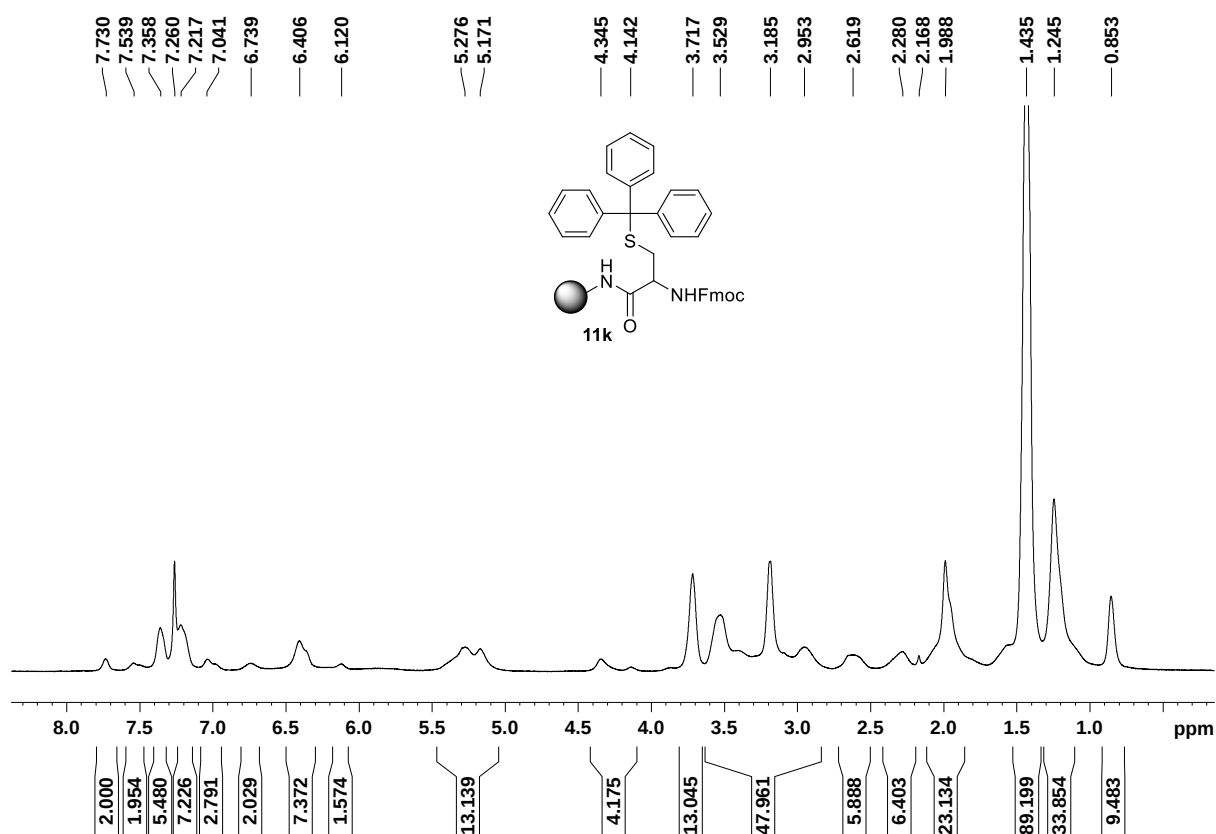


Figure S21. ¹H NMR spectrum of compound **11k**.

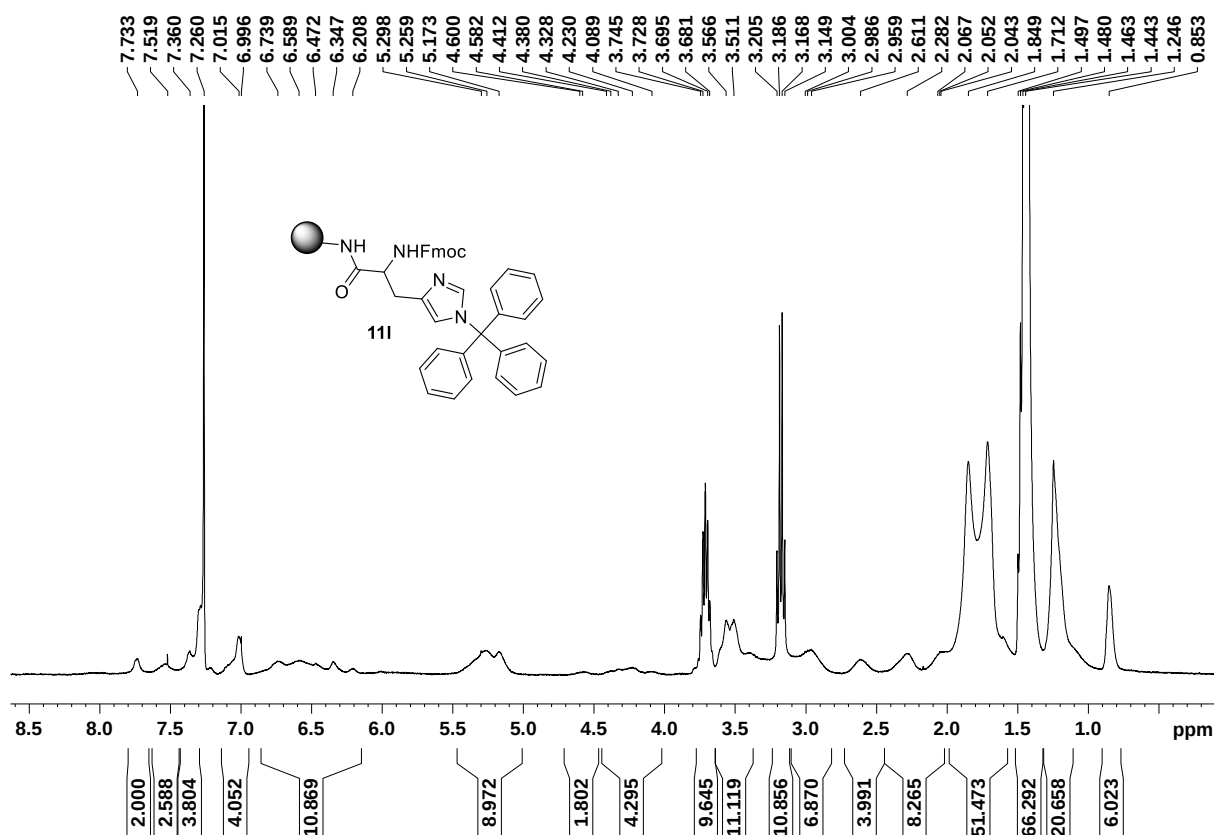


Figure S22. ¹H NMR spectrum of compound **11l**.

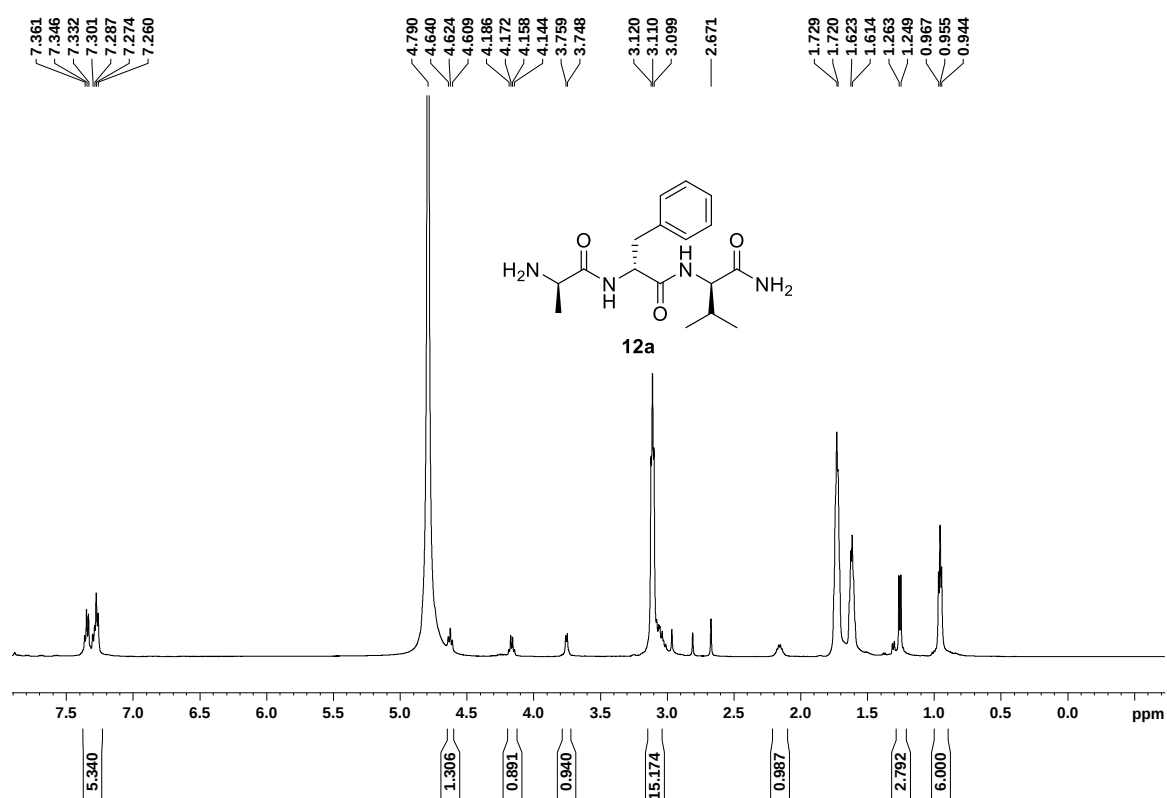


Figure S23. ¹H NMR spectrum of compound **12a**

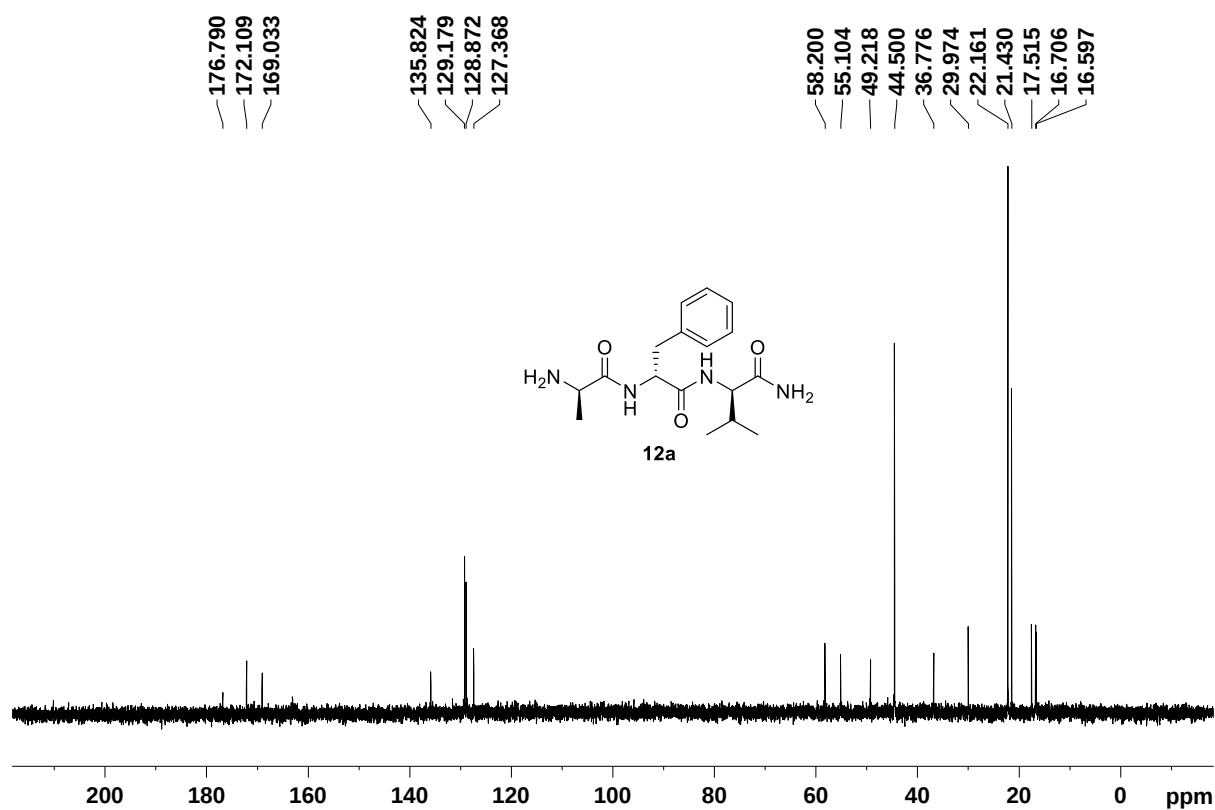


Figure S24. ¹³C NMR spectrum of compound **12a**

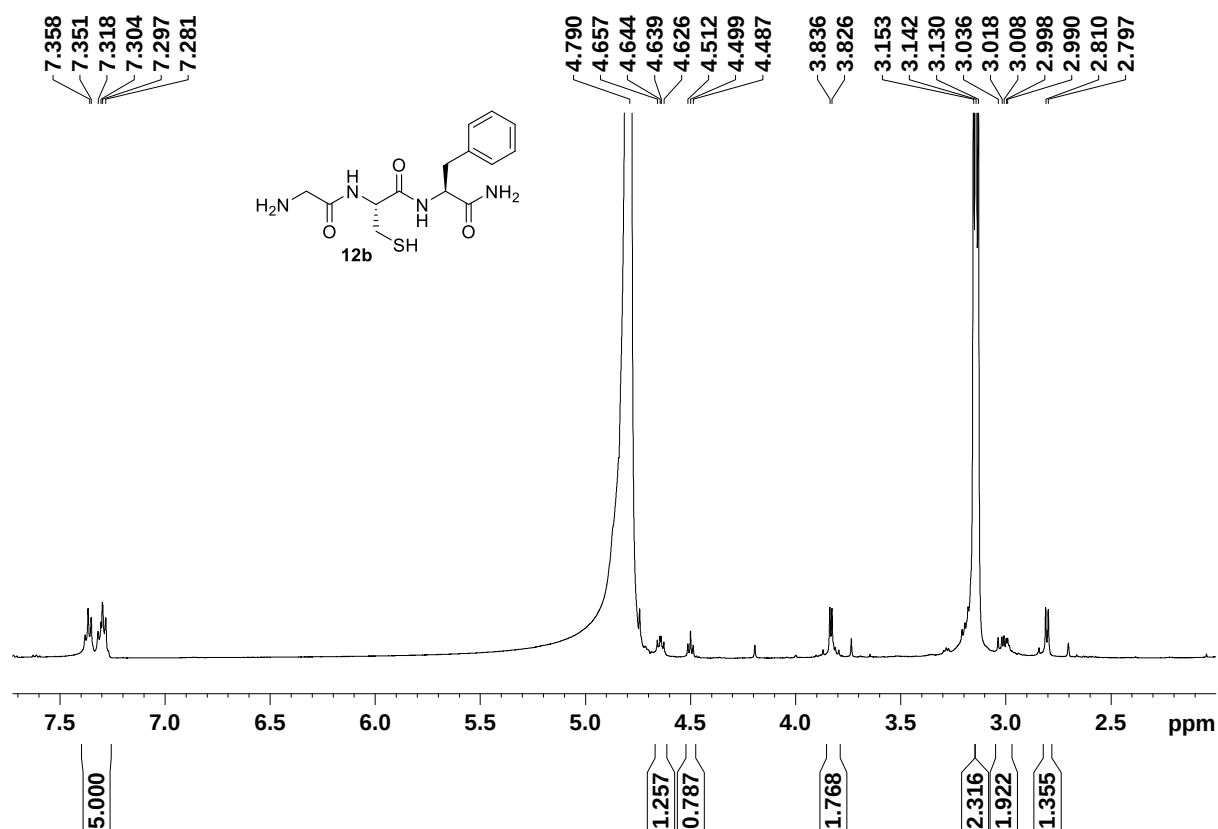


Figure S25. ¹H NMR spectrum of compound 12b.

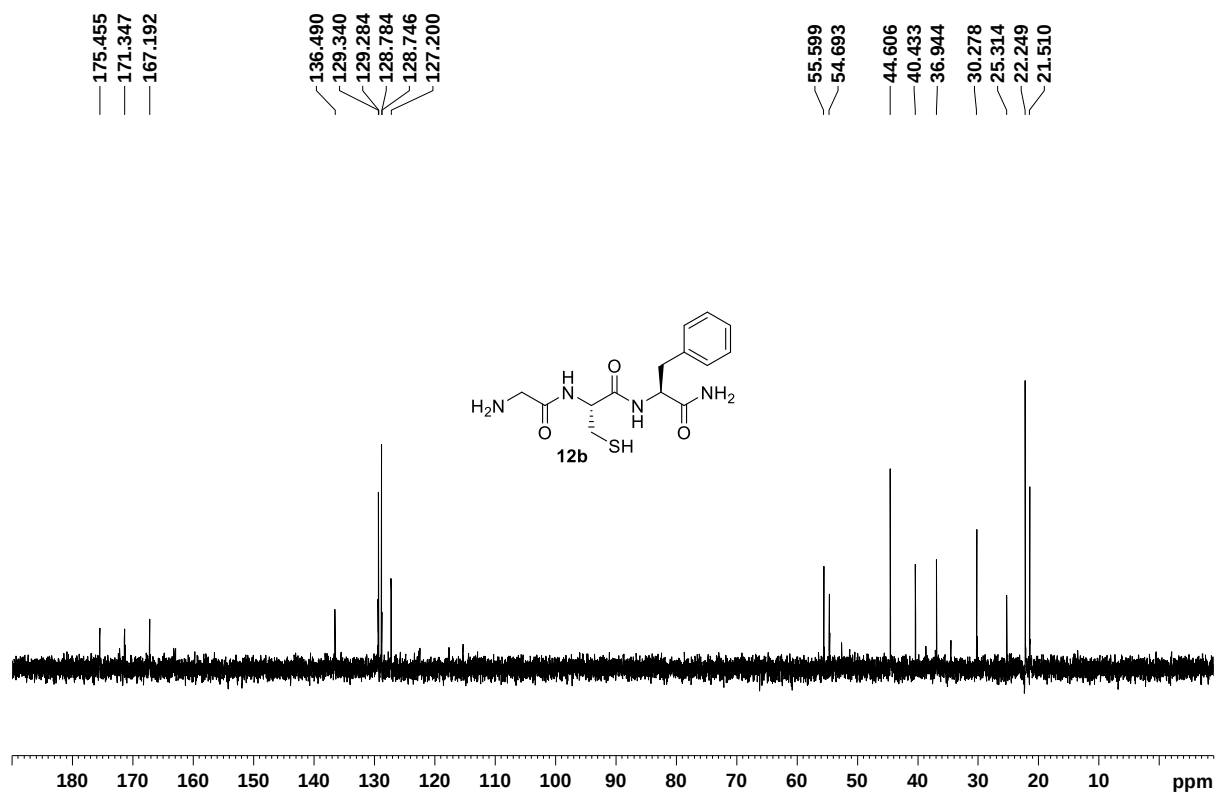


Figure S26. ¹³C NMR spectrum of compound 12b.

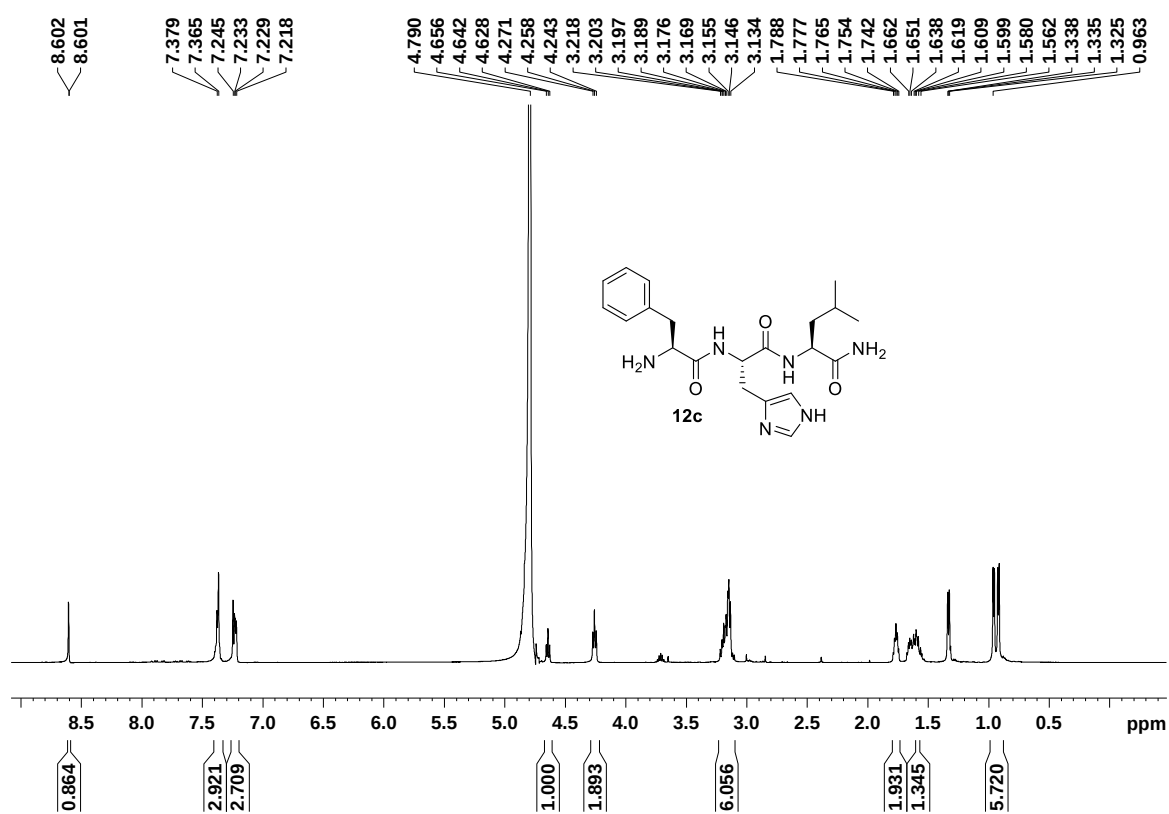


Figure S27. ¹H NMR spectrum of compound 12c.

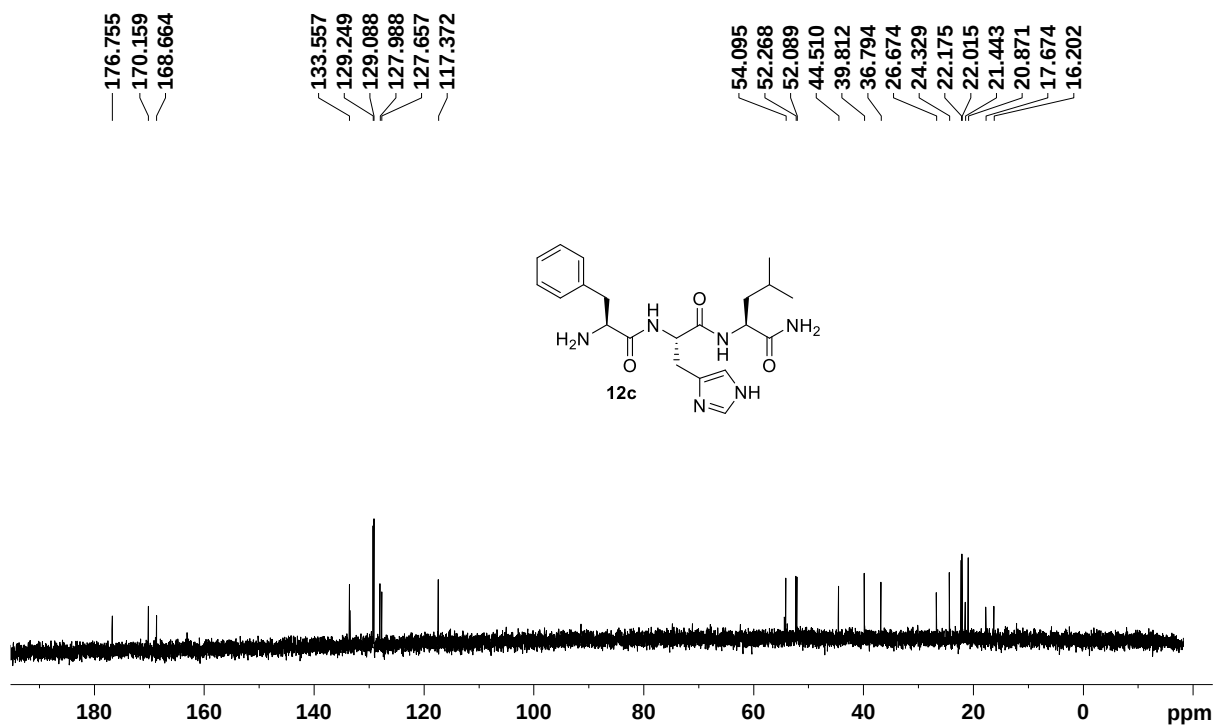


Figure S28 ¹³C NMR spectrum of compound 12c.

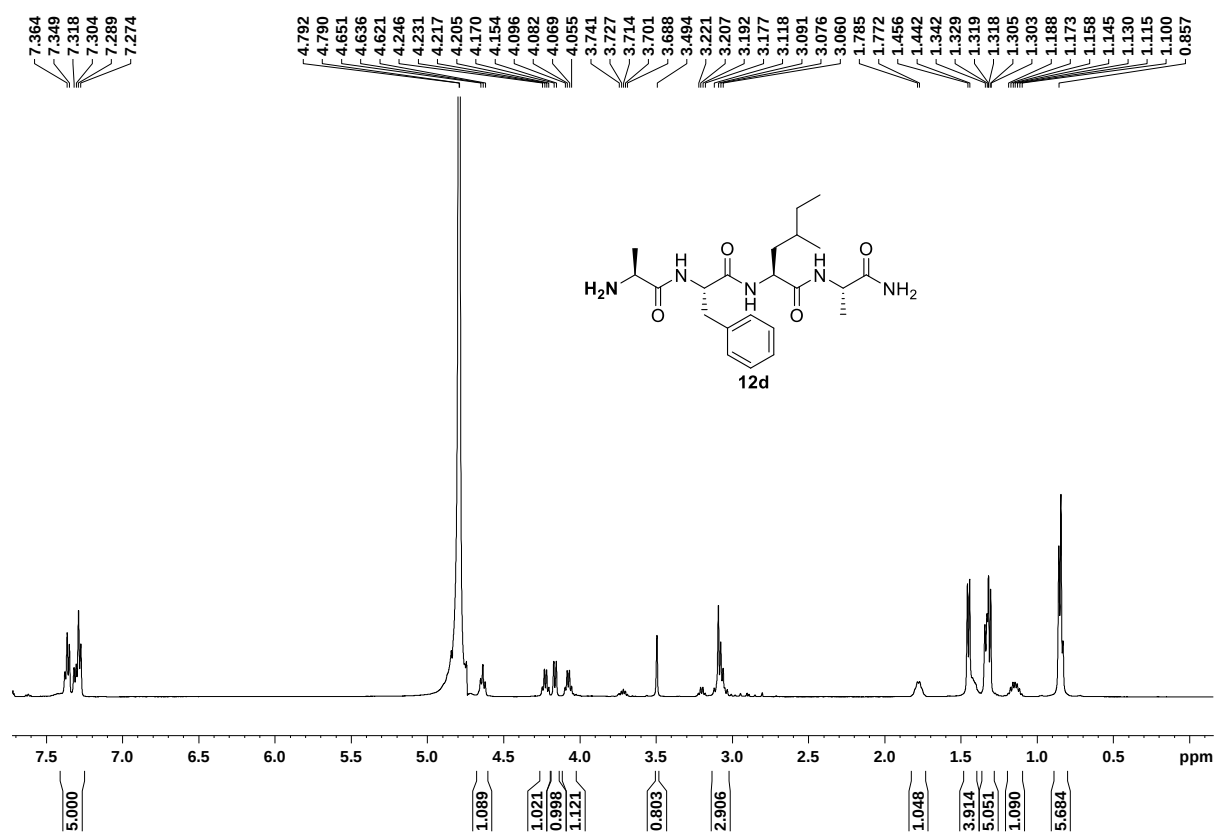


Figure S29 ¹H NMR spectrum of compound 12d.

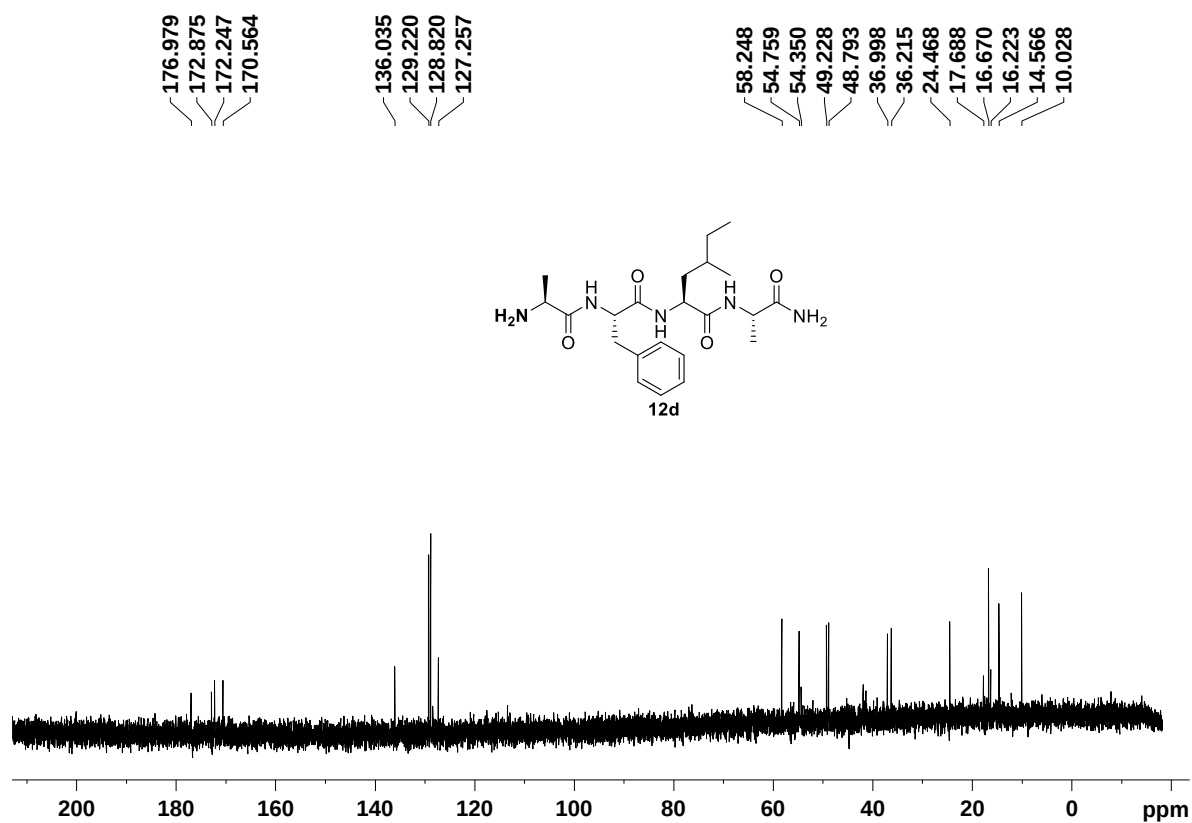


Figure S30 ¹³C NMR spectrum of compound 12d.

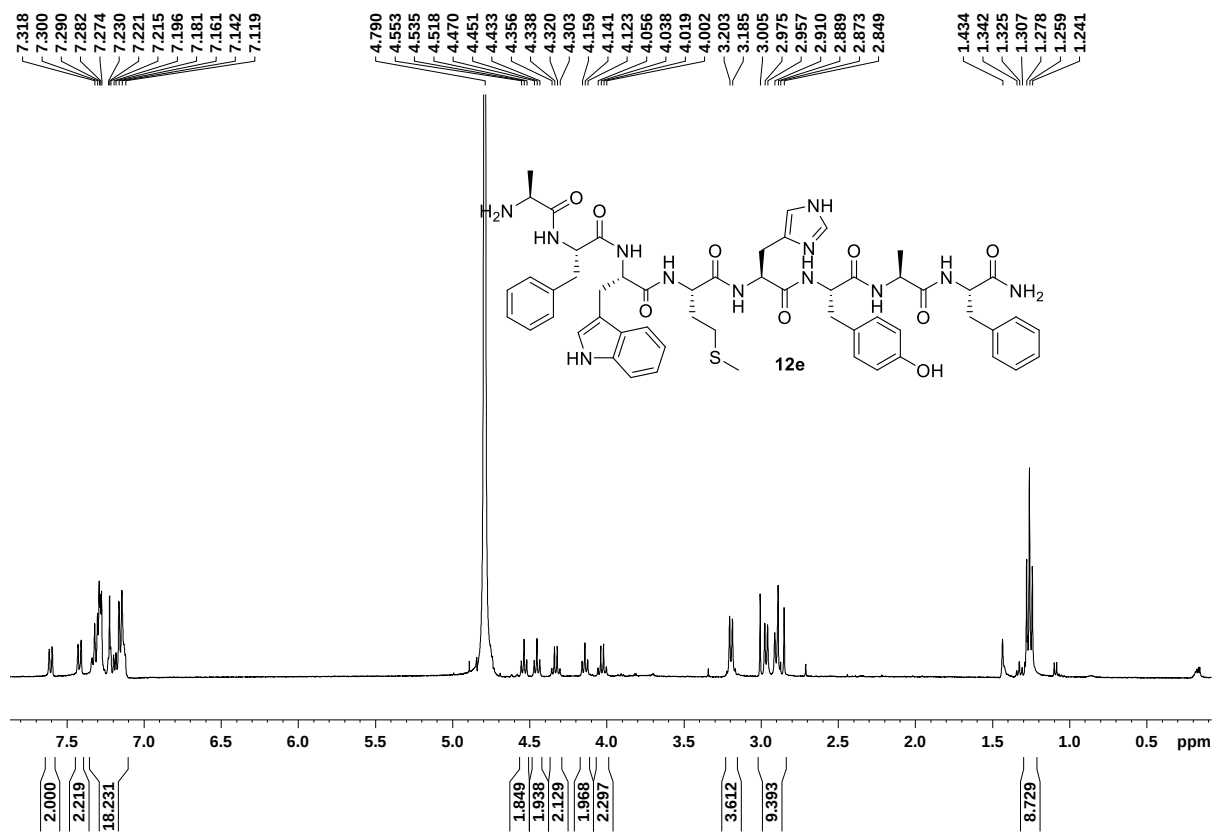


Figure S31 ¹H NMR spectrum of compound 12e.

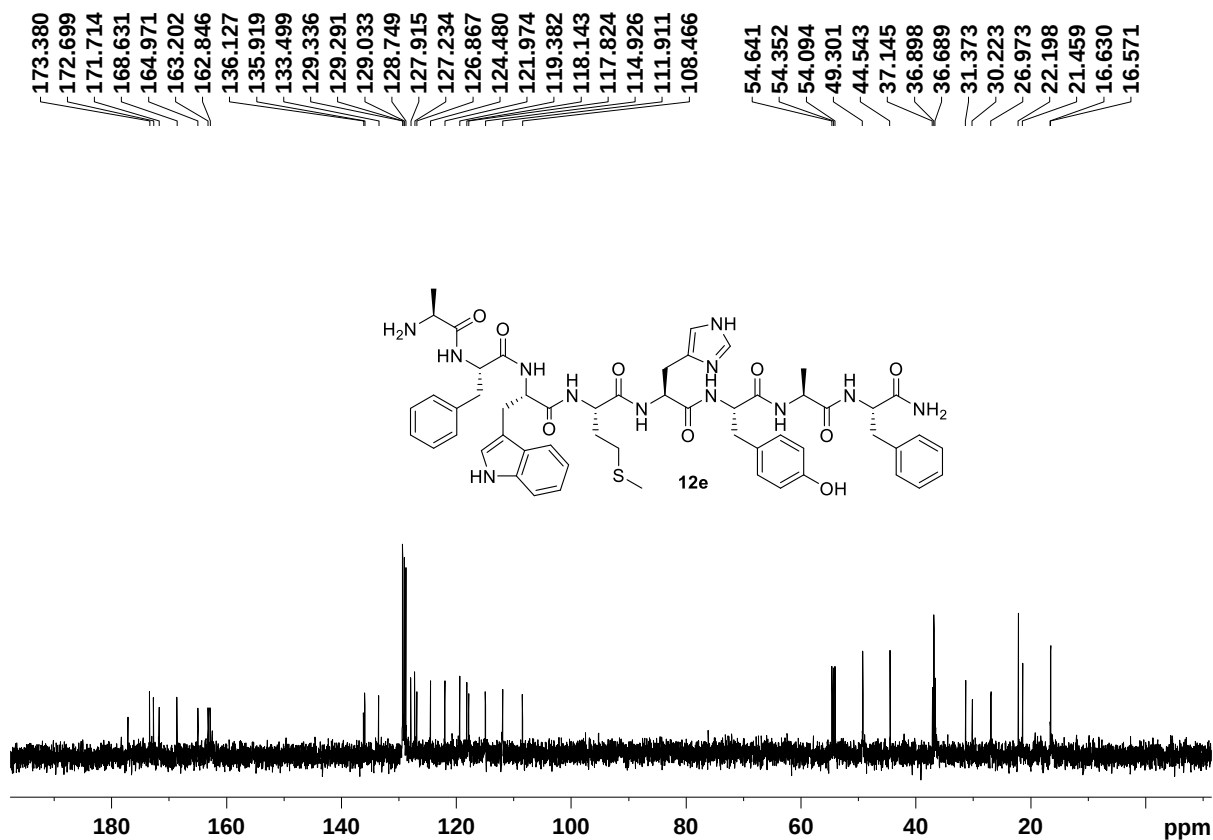


Figure S32 ¹³C NMR spectrum of compound 12e.

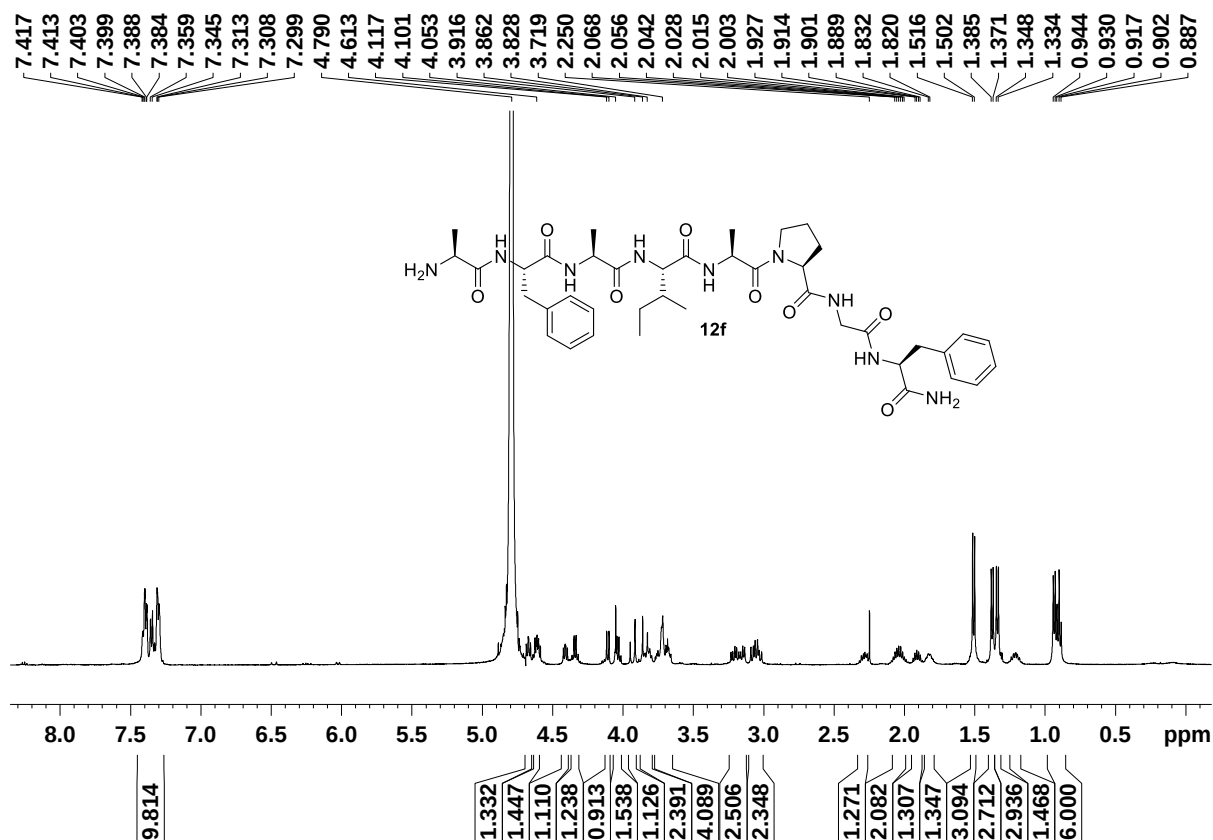


Figure S33 ^1H NMR spectrum of compound 12f.

Representative example illustrating removal of amino acids during washes after peptide synthesis

The general procedure for attachment of the first amino acid was followed. The combined supernatants obtained after washing the support with ether were collected. The TLC of the supernatants showed the presence of the amino acid starting material (Figure S34)

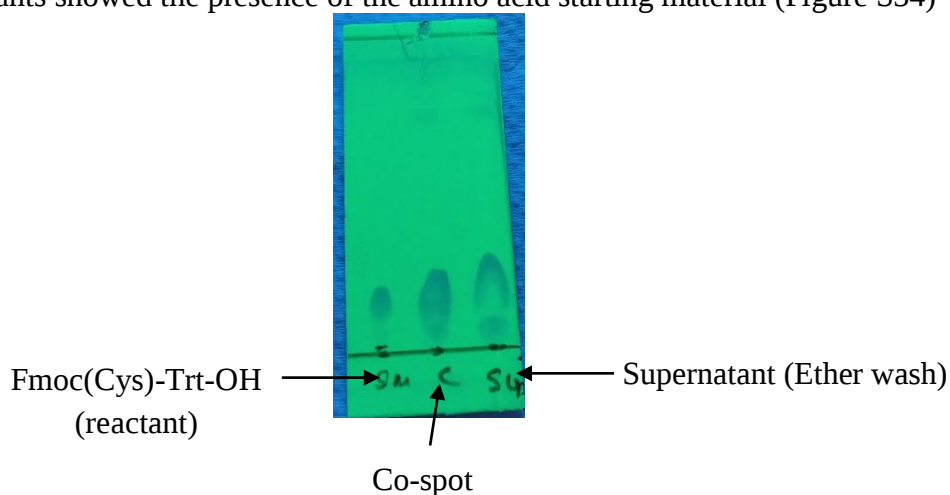


Figure S34 TLC comparing supernatant after coupling reaction with FmocCys-Trt-OH starting material (Solvent system: 50% ethylacetate/hexane).

General procedure for HPLC purification and analysis of larger oligopeptides

All peptides were purified by semi preparative RP-HPLC (Reversed Phase High Performance Liquid Chromatography) on a Shimadzu system with water (0.1% TFA) and acetonitrile (0.1% TFA) as mobile phase solvents. The flow rate used for analytical RP-HPLC & semi preparative RP-HPLC were 1 mL/min and 4.5 mL/min respectively. Peptide was injected at a concentration of 1.0 mg/mL for analytical HPLC and 10 mg/mL for semi-preparative HPLC. Peptide elution was monitored at 254 nm and 220 nm. The gradient system shown in Table S1 was used to separate the larger oligopeptides and conotoxins. Hexadapeptide **12k** was separated using an isocratic method (40% acetonitrile/water) Note: All the LCMS spectra were acquired in Thermo Fisher Dionex ultimate 3000 UHPLC system using C-18, 5 μ m, 10 $\times$ 250 mm column with the flow rate of 0.5mL/min.

Table S1. Gradient method used for HPLC of peptides **12e –12-j**.

S. No.	Time (min)	Acetonitrile (%)	Water (%)
1	0	10	90
2	10	20	80
3	15	30	70
4	20	40	60
5	25	50	50
6	30	60	40
7	35	70	30

HPLC traces of peptides 12e-12k

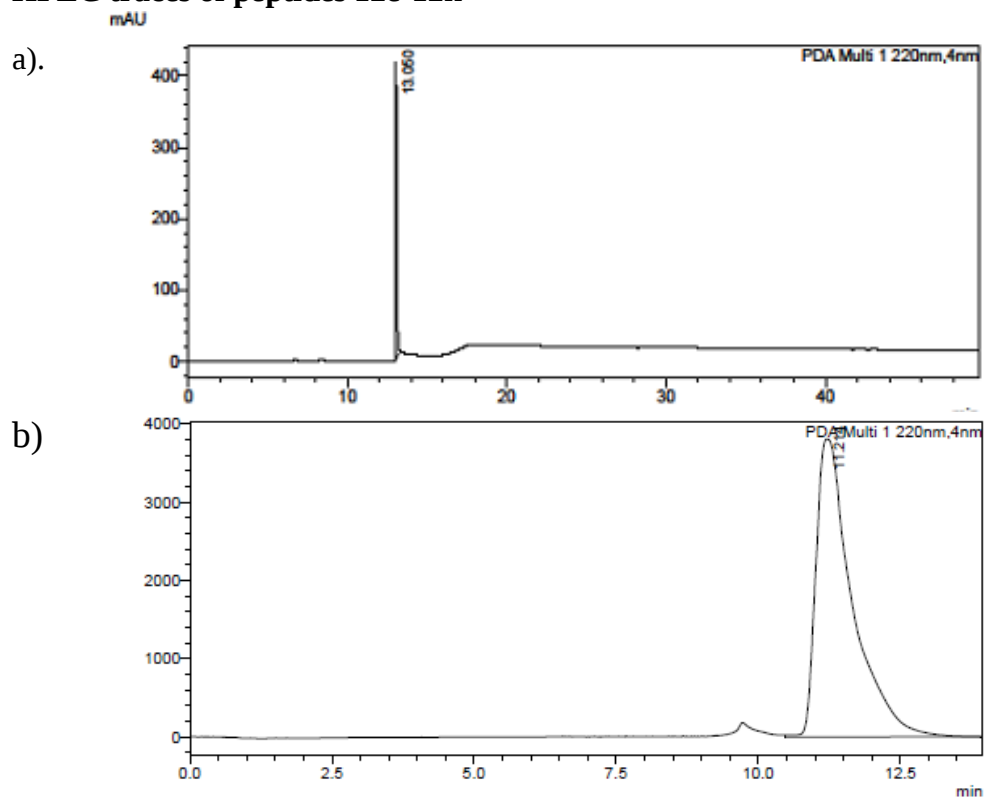


Figure S35. HPLC traces of a) crude; b) pure peptide AFWMHYAF **12e**

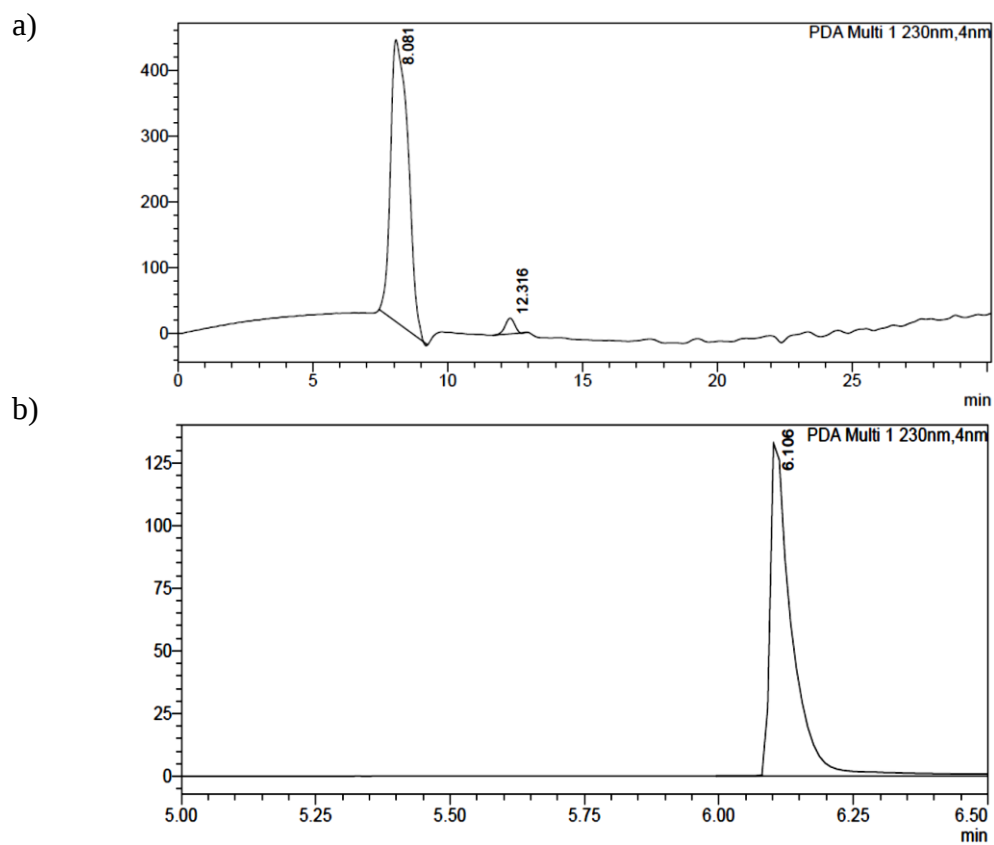


Figure S36. HPLC traces of a) crude; b) pure peptide AFAIAPGF **12f**.

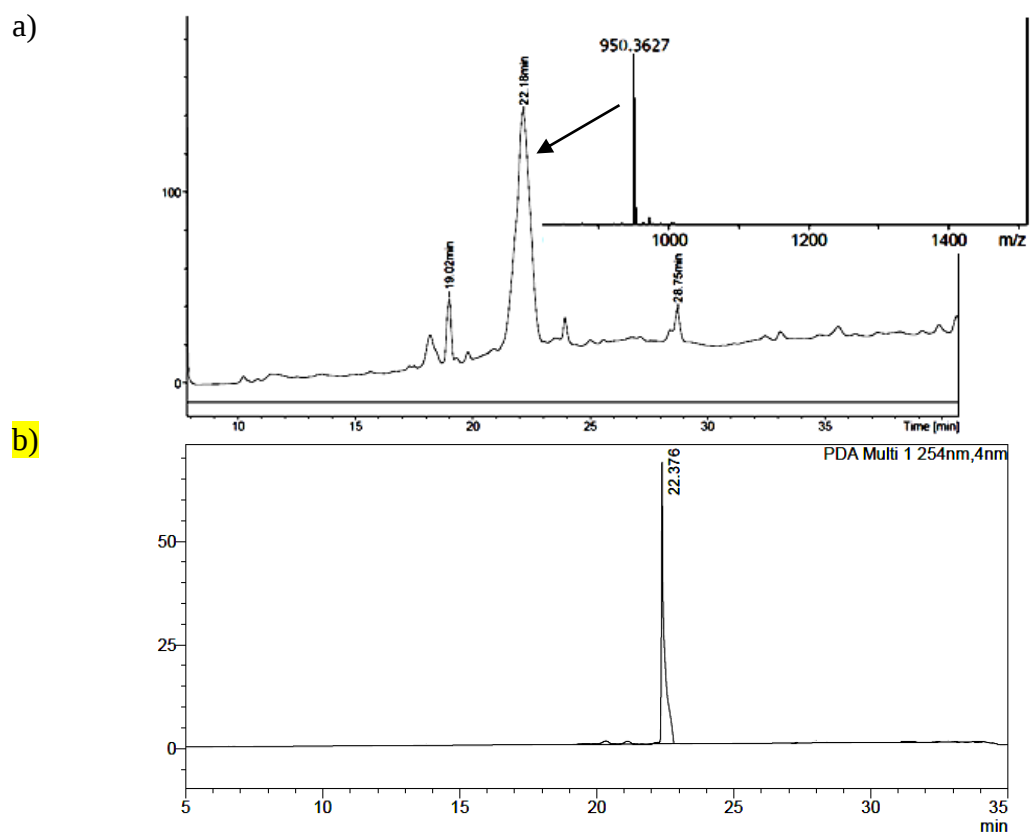


Figure S37. HPLC traces of a) crude; b) pure peptide GCPWQPYC 12g.

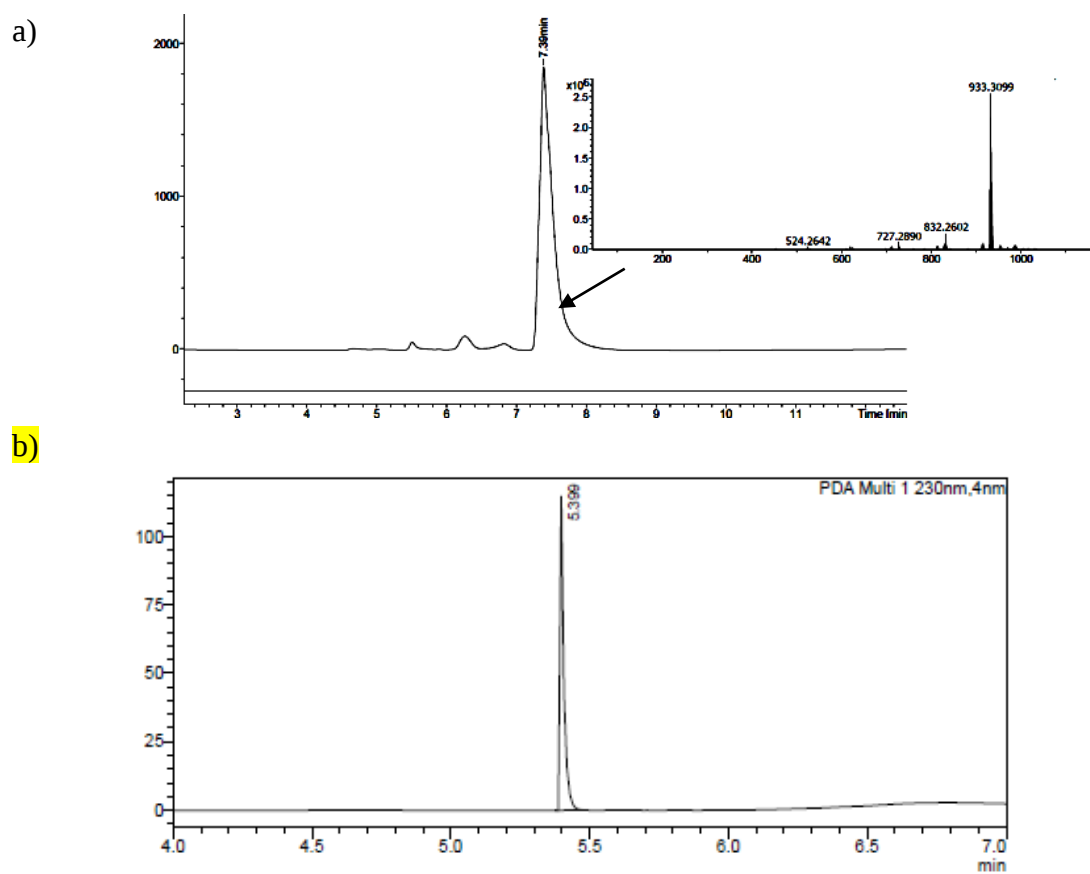


Figure S38. HPLC traces of a) crude; b) pure peptide TCFGCTPCC 12h

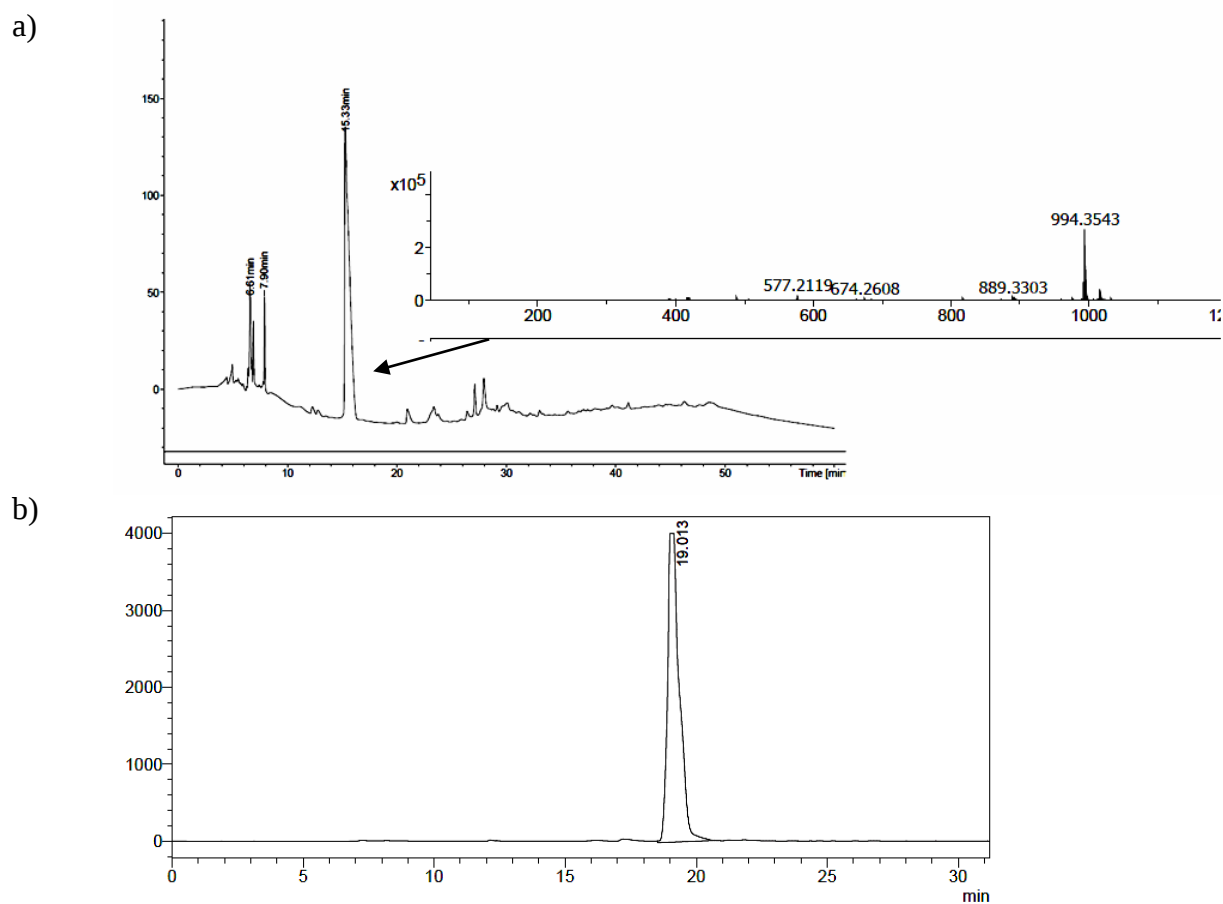


Figure S39. HPLC traces of a) crude; b) pure peptide CCPPALWCC **12i**.

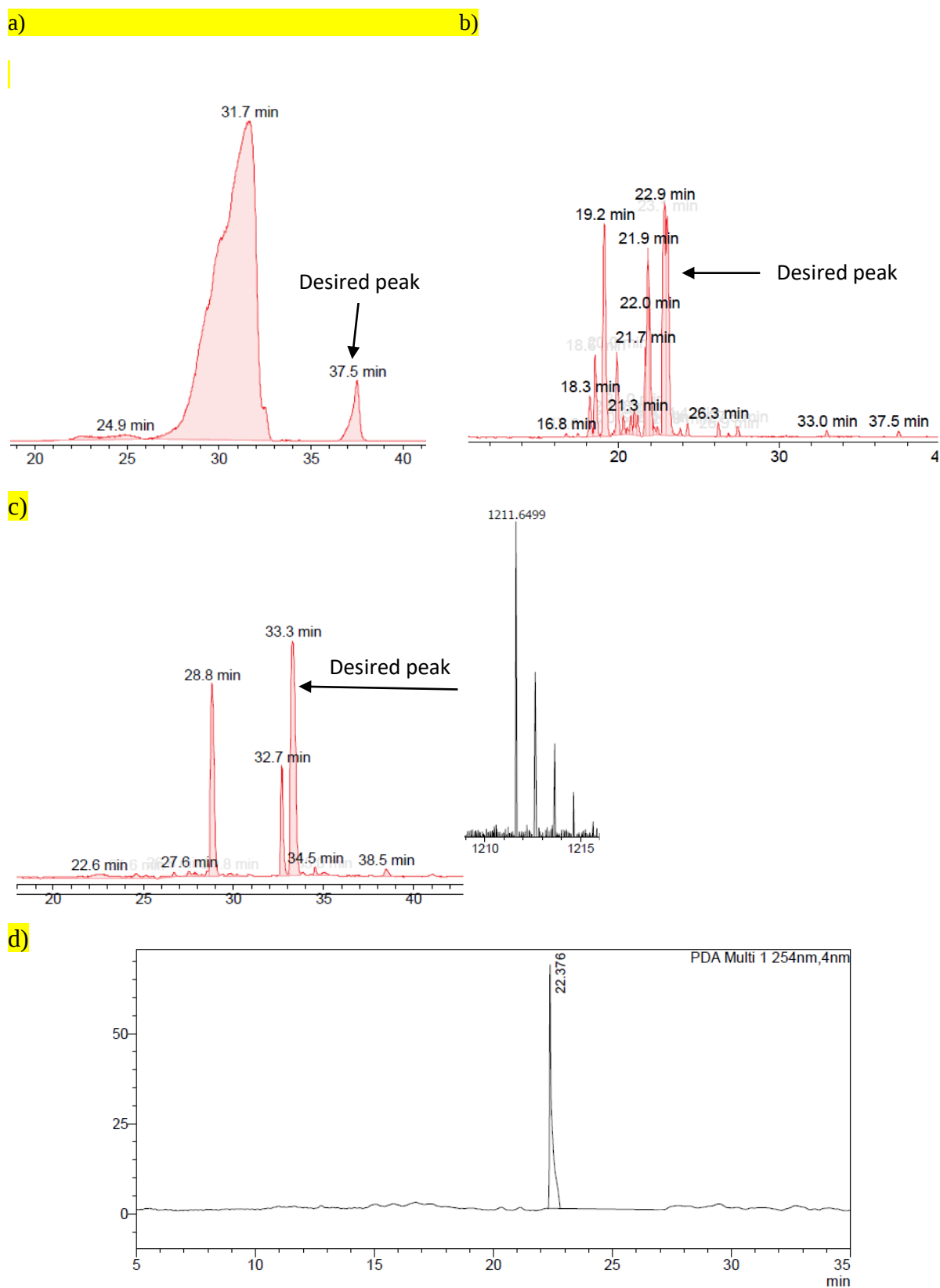


Figure S40. HPLC traces of peptide WFTTLISTIM 12j a) crude synthesized on 1a; b) crude synthesized on 1b; c) crude synthesized on 1d; d) pure peptide synthesized on 1d.

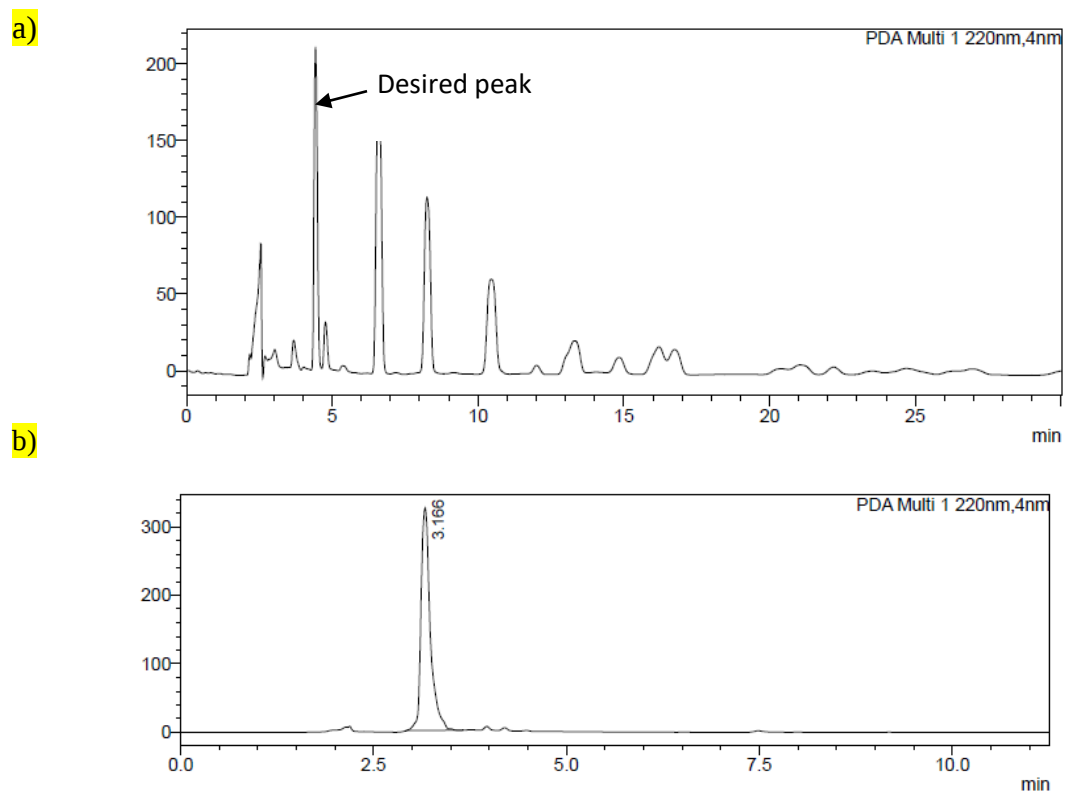


Figure S41. HPLC trace of a) crude; b) pure peptide GCGAFACRFGCTPCC 12k.

Mass spectra of peptides 12a-12k.

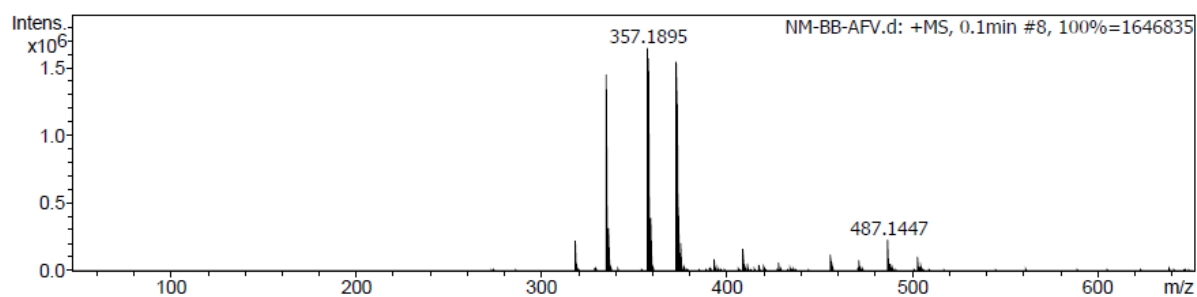


Figure S42. ESI of 12a

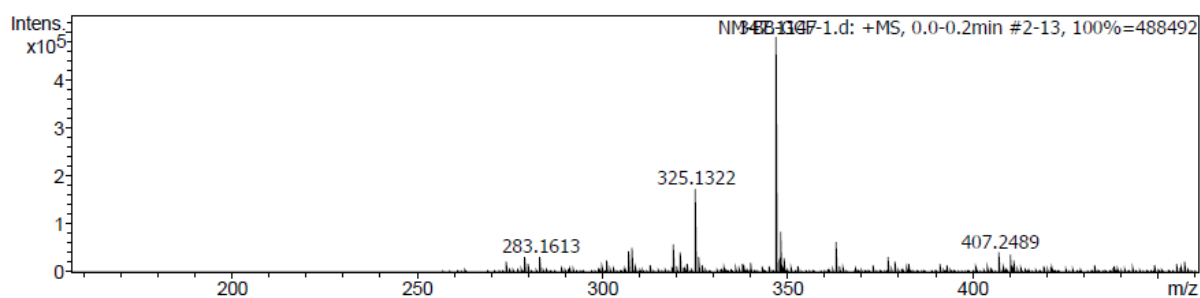


Figure S43. ESI of 12b.

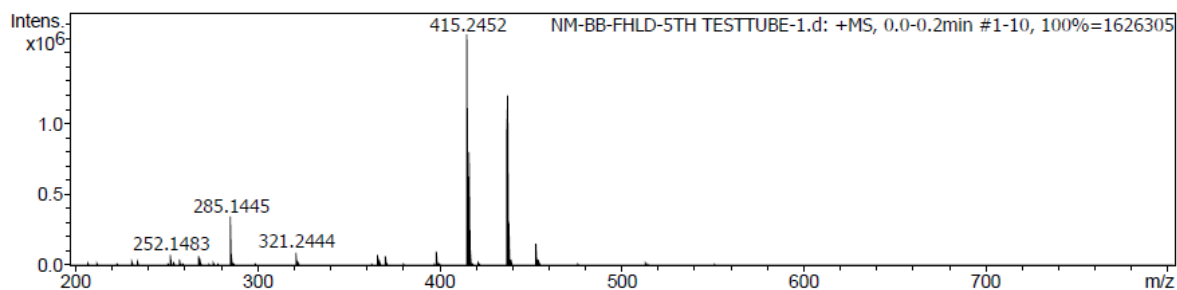


Figure S44. ESI of 12c.

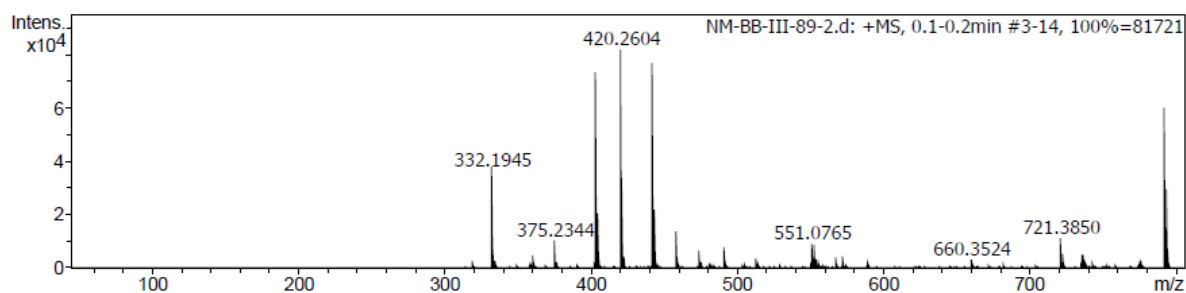


Figure S45. ESI of 12d.

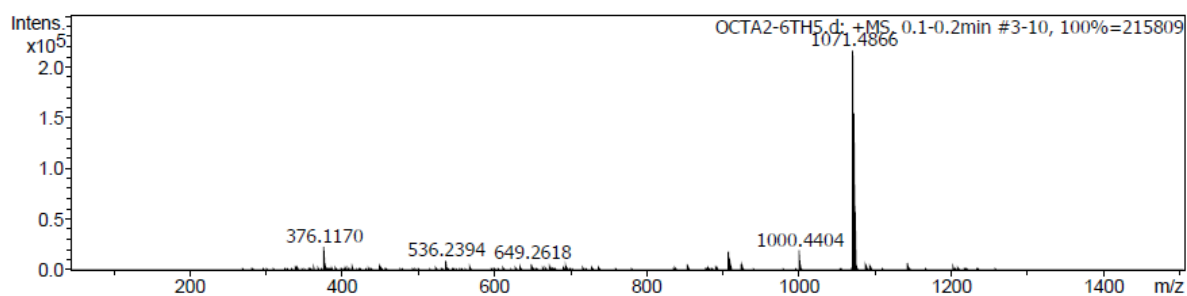


Figure S46. ESI of 12e

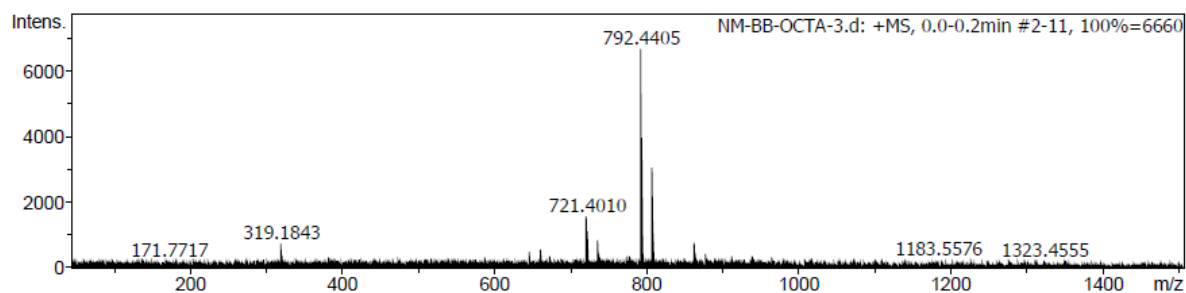


Figure S47. ESI of 12f

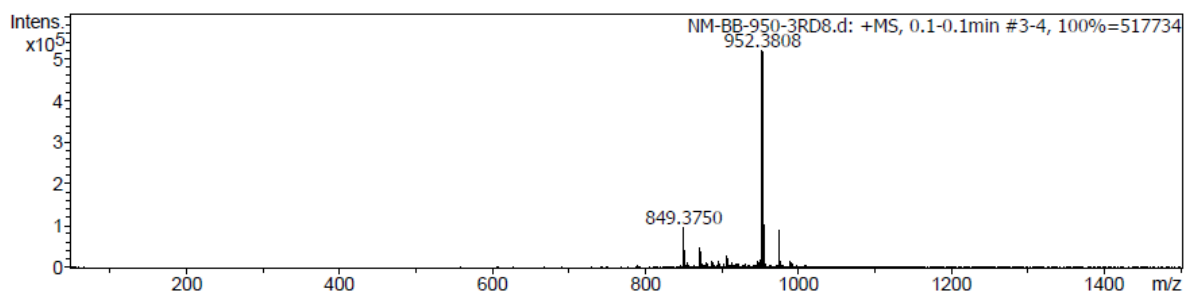


Figure S48. ESI of 12g

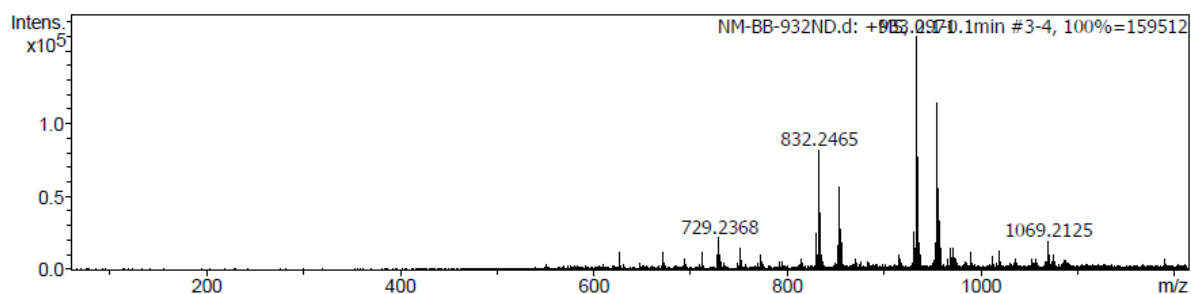


Figure S49. ESI of 12h

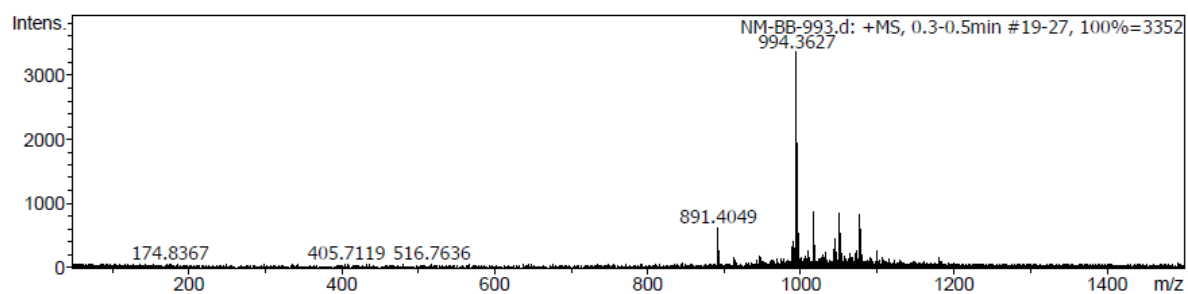


Figure S50. ESI of 12i

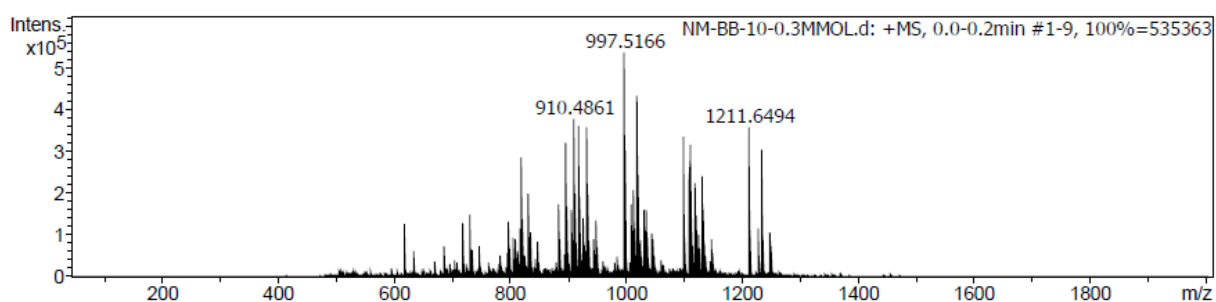


Figure S51. ESI of 12j

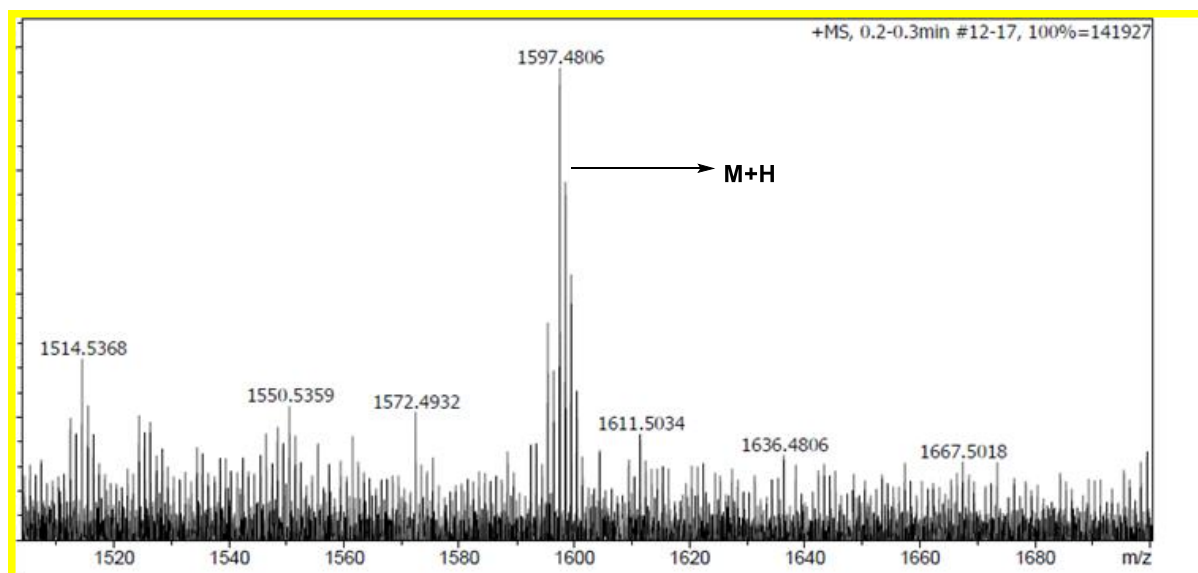


Figure S52. ESI of 12k

DLS data for support 1b

The support **1b** was dissolved in chloroform (1.5 mg/mL)

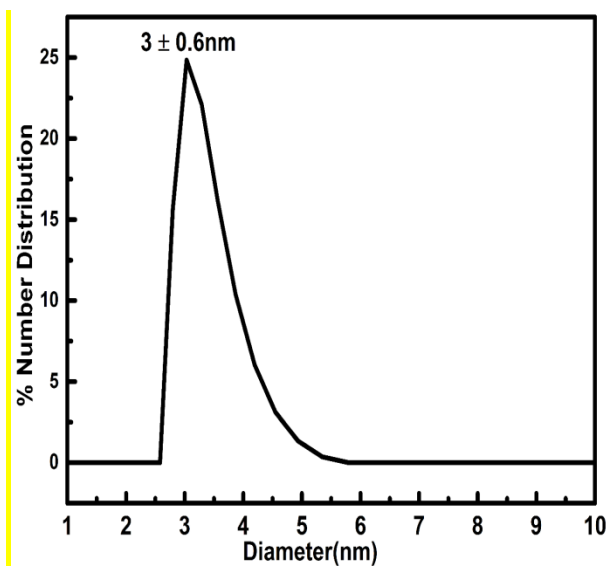


Figure S53. DLS of support **1b**