

Supporting Information

Synthesis and Assignment of Stereochemistry of the Antibacterial Cyclic Peptide Xenematide

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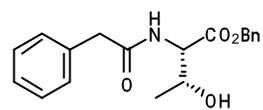
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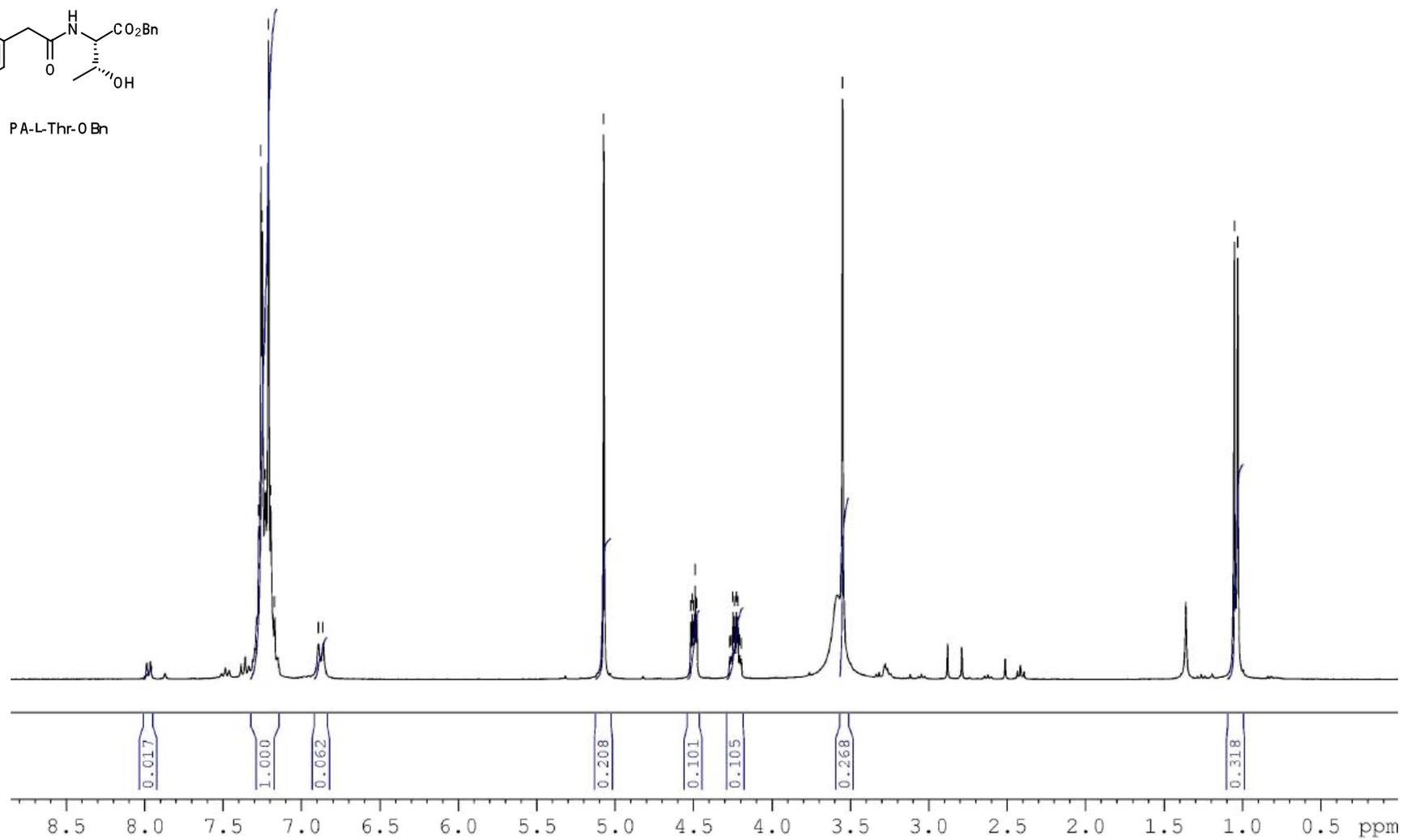
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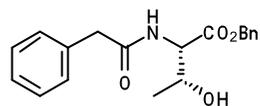
^1H NMR spectrum of PA-L-Thr-OBn (300 MHz; CDCl_3)



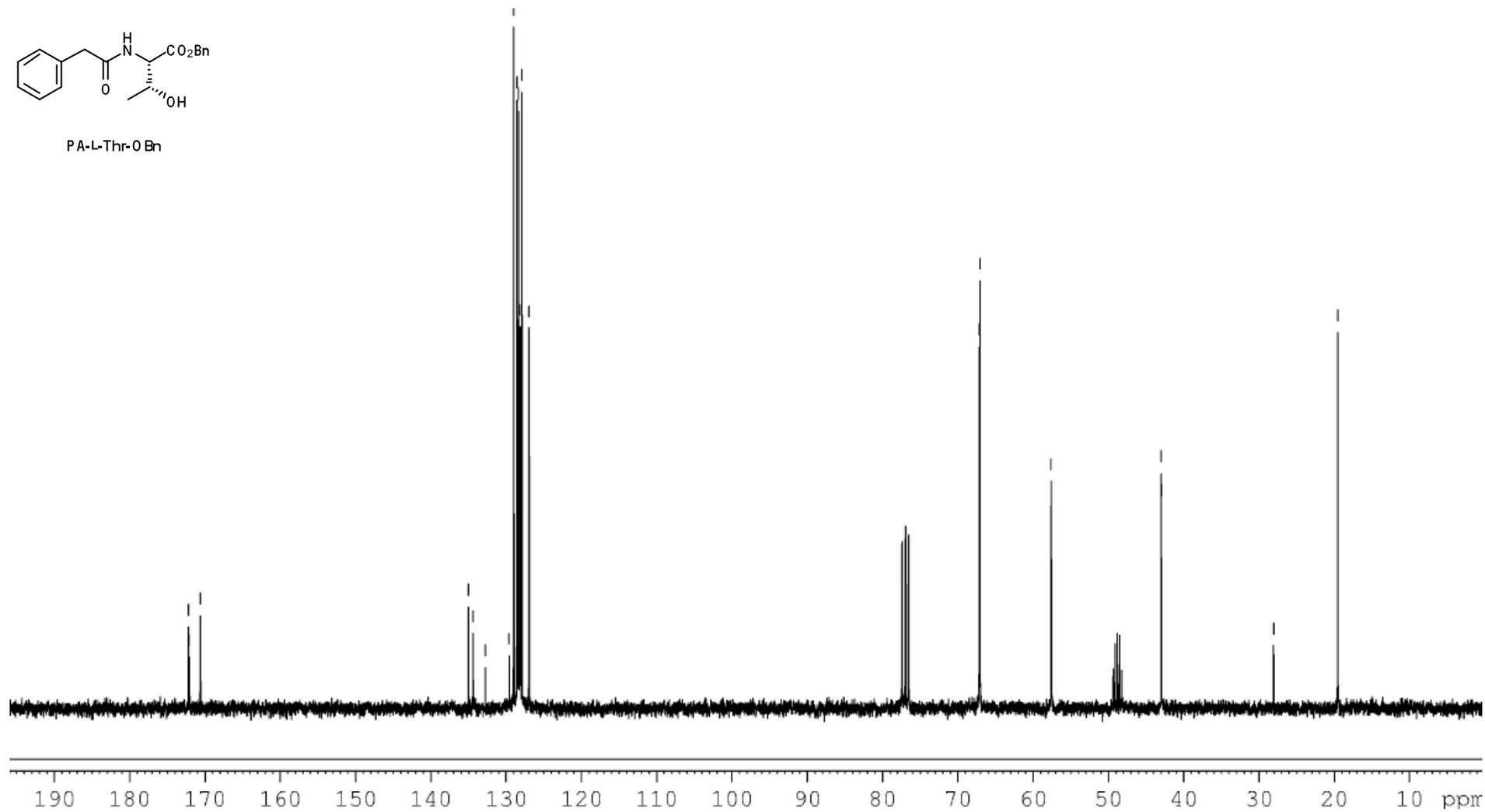
PA-L-Thr-OBn



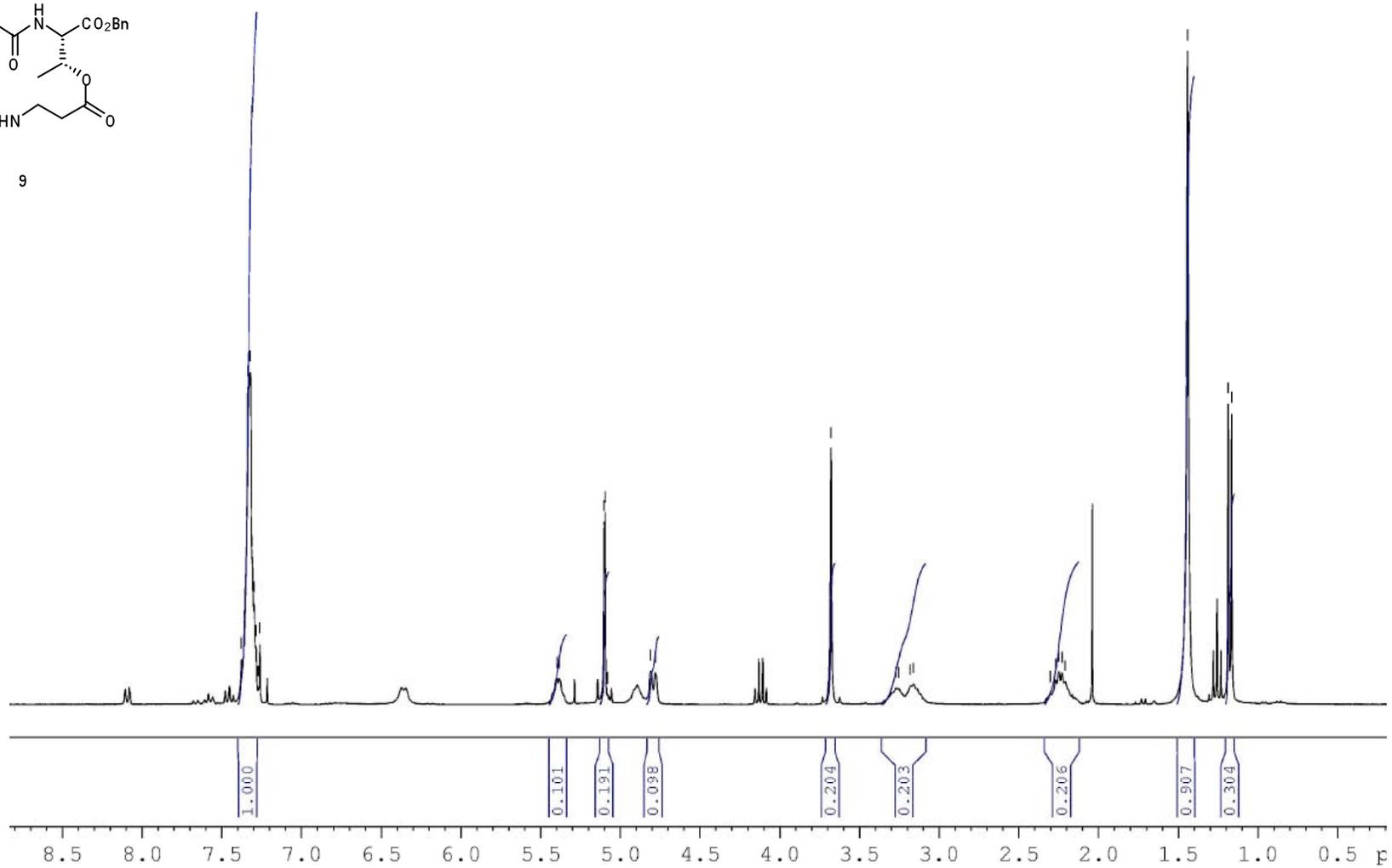
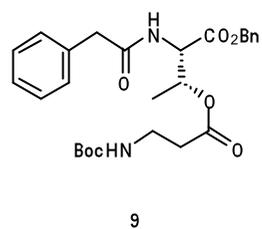
^{13}C NMR spectrum of PA-L-Thr-OBn (75 MHz; CDCl_3)



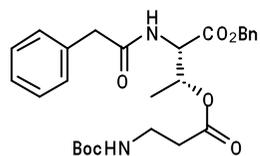
PA-L-Thr-OBn



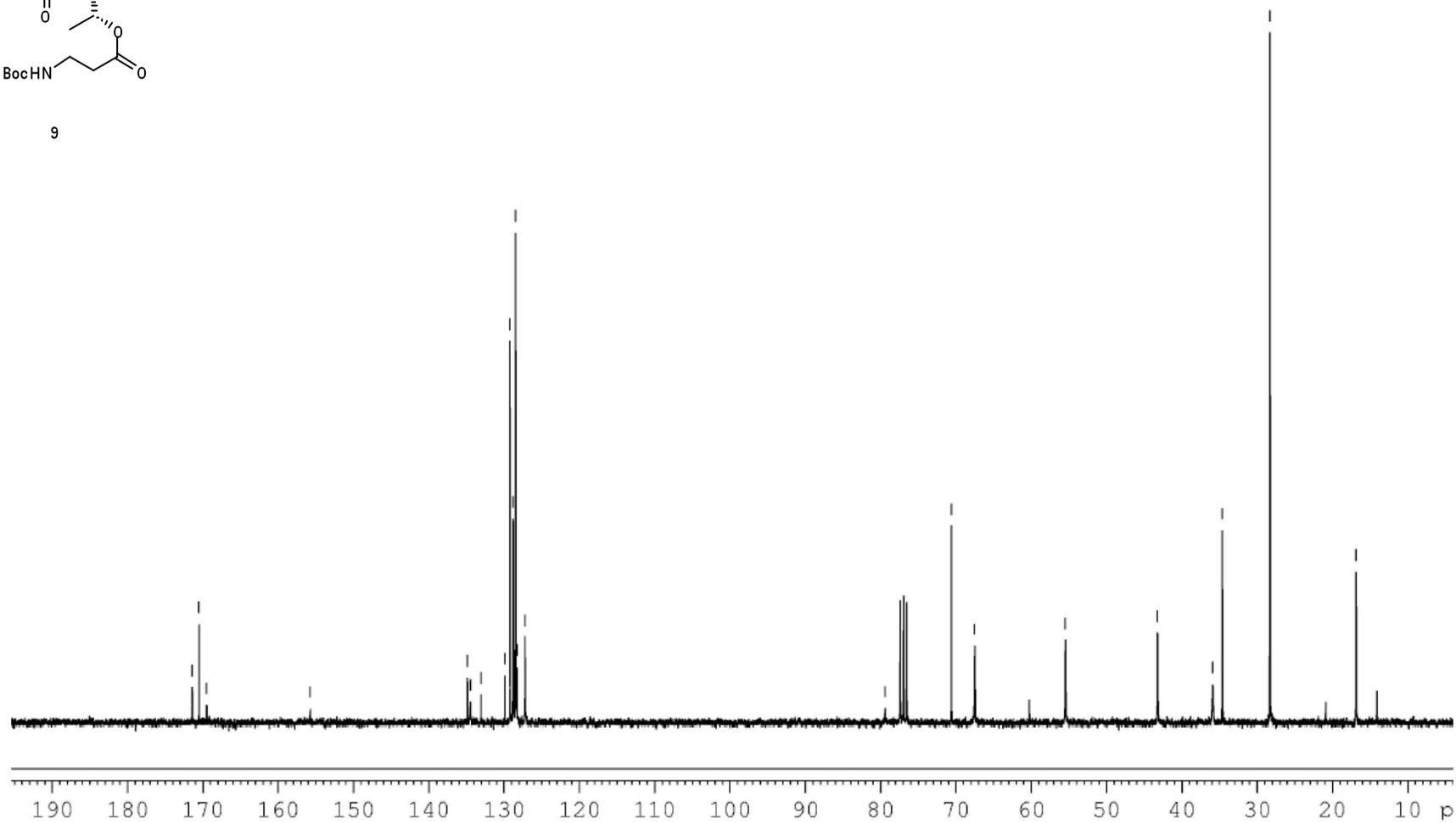
¹H NMR spectrum of compound 9 (300 MHz; CDCl₃)



^{13}C NMR spectrum of compound 9 (75 MHz; CDCl_3)



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HPLC Chromatograms of peptides **8** and **10** after cleavage from resin:

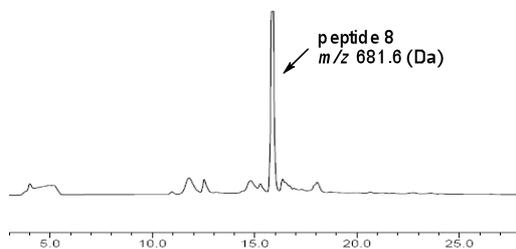


Figure 1. Analytical HPLC profile ($\lambda = 210$ nm) together with ESI-MS data of crude peptide **8** mixture.

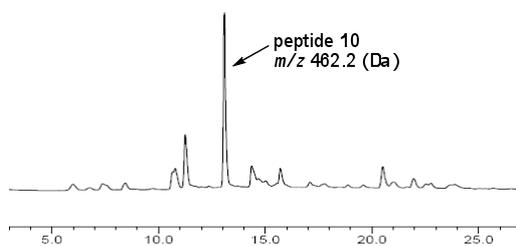


Figure 2. Analytical HPLC profile ($\lambda = 210$ nm) together with ESI-MS data of crude peptide **10** mixture.

HPLC Chromatograms of purified peptides 11, 12 and unnatural diastereomers of xenematide:

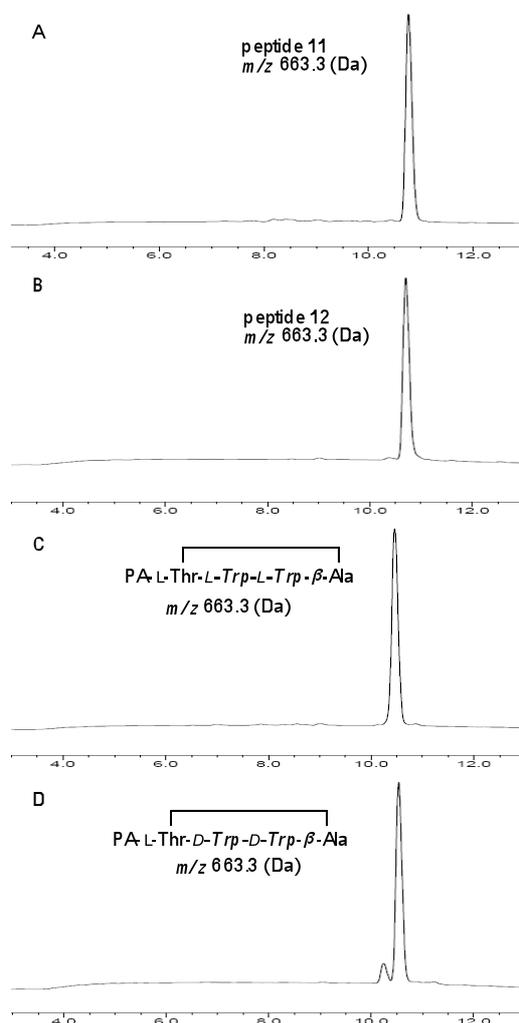
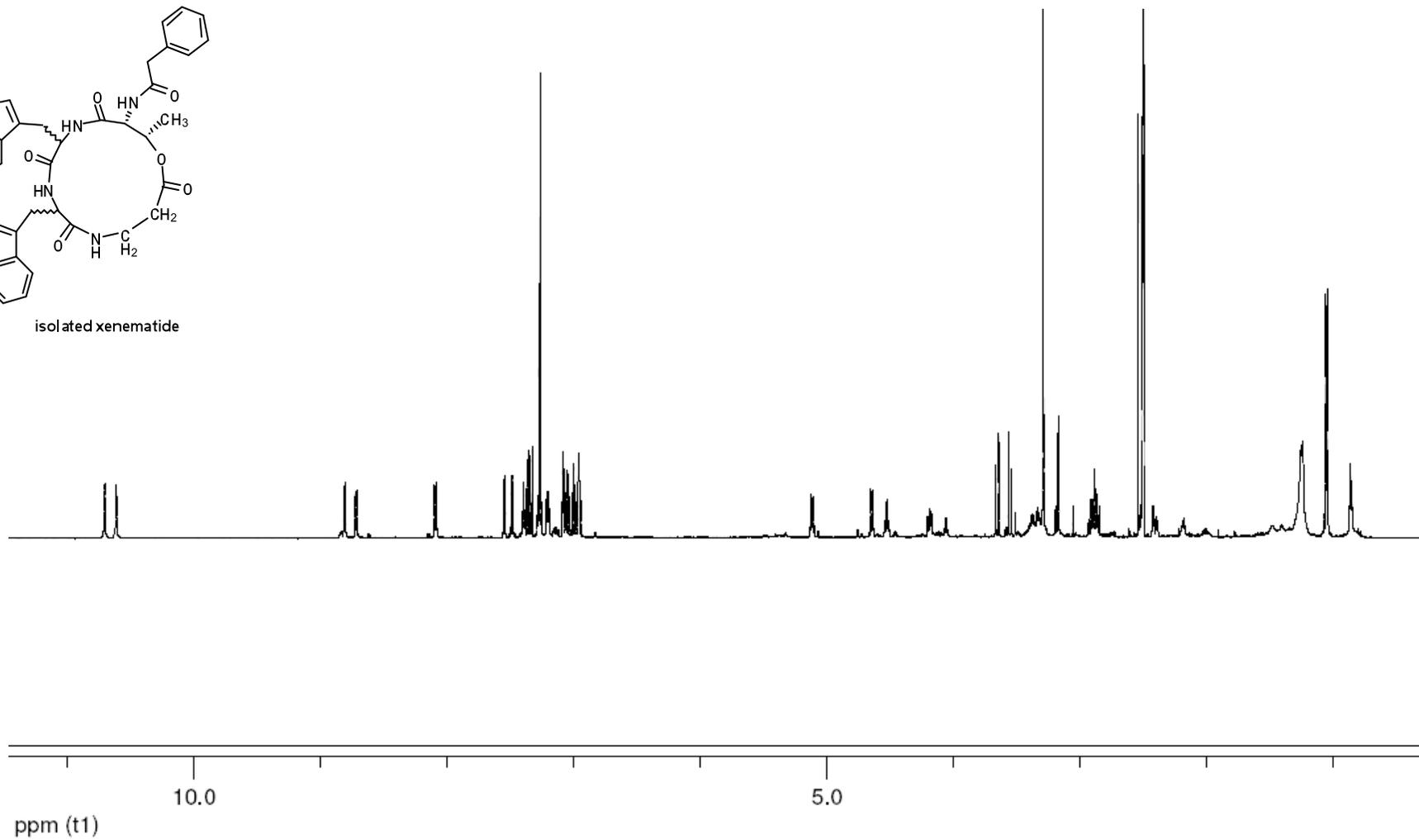
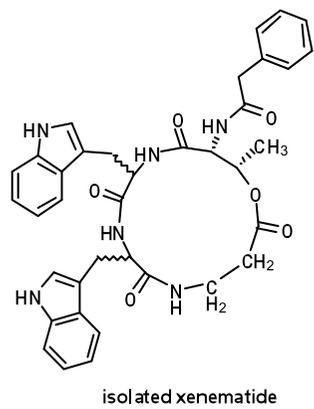
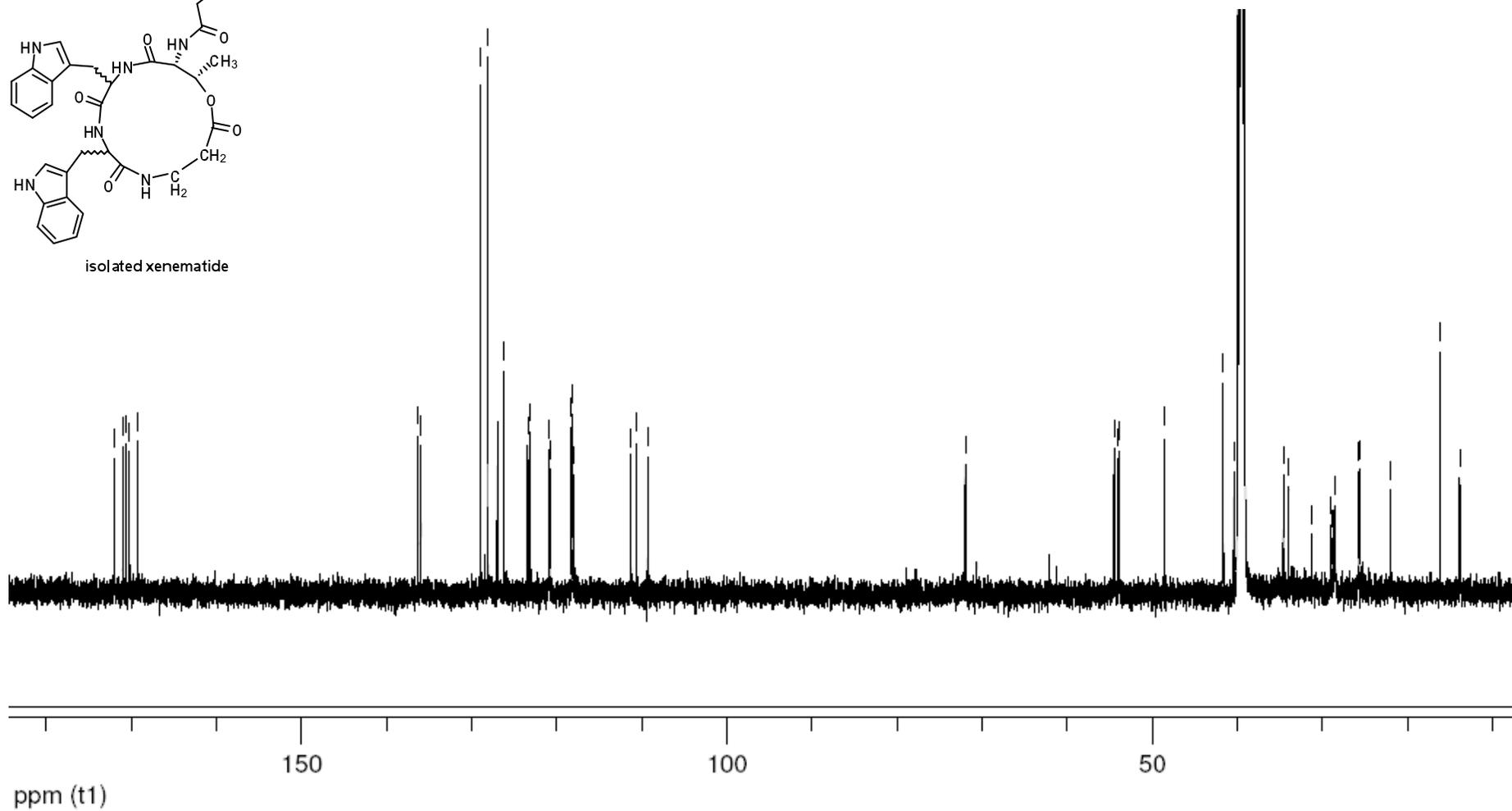
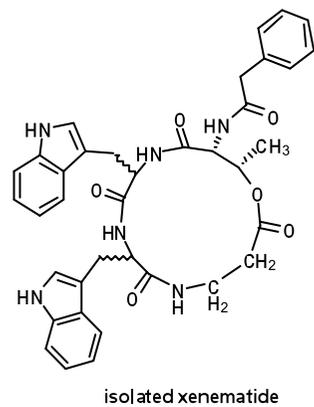


Figure 3. Analytical HPLC profile ($\lambda = 210$ nm) together with ESI-MS data of: (A) PA-L-Thr-D-Trp-L-Trp- β -Ala **11**; (B) PA-L-Thr-L-Trp-D-Trp- β -Ala **12**; (C) PA-L-Thr-L-Trp-L-Trp- β -Ala and (D) PA-L-Thr-D-Trp-D-Trp- β -Ala (>90% purity).

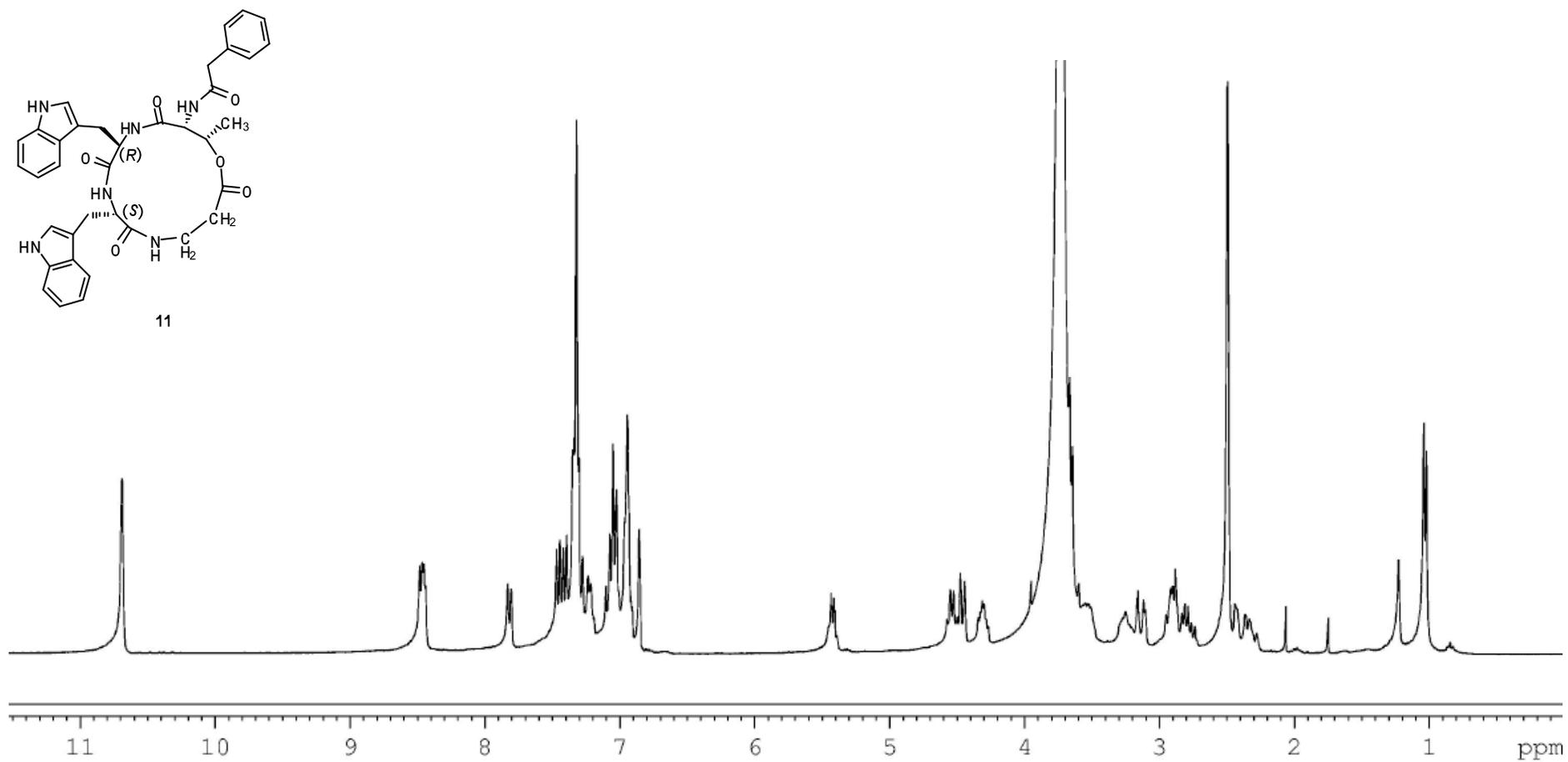
^1H NMR spectrum of isolated xenematide (600 MHz; $\text{DMSO-}d_6$)¹



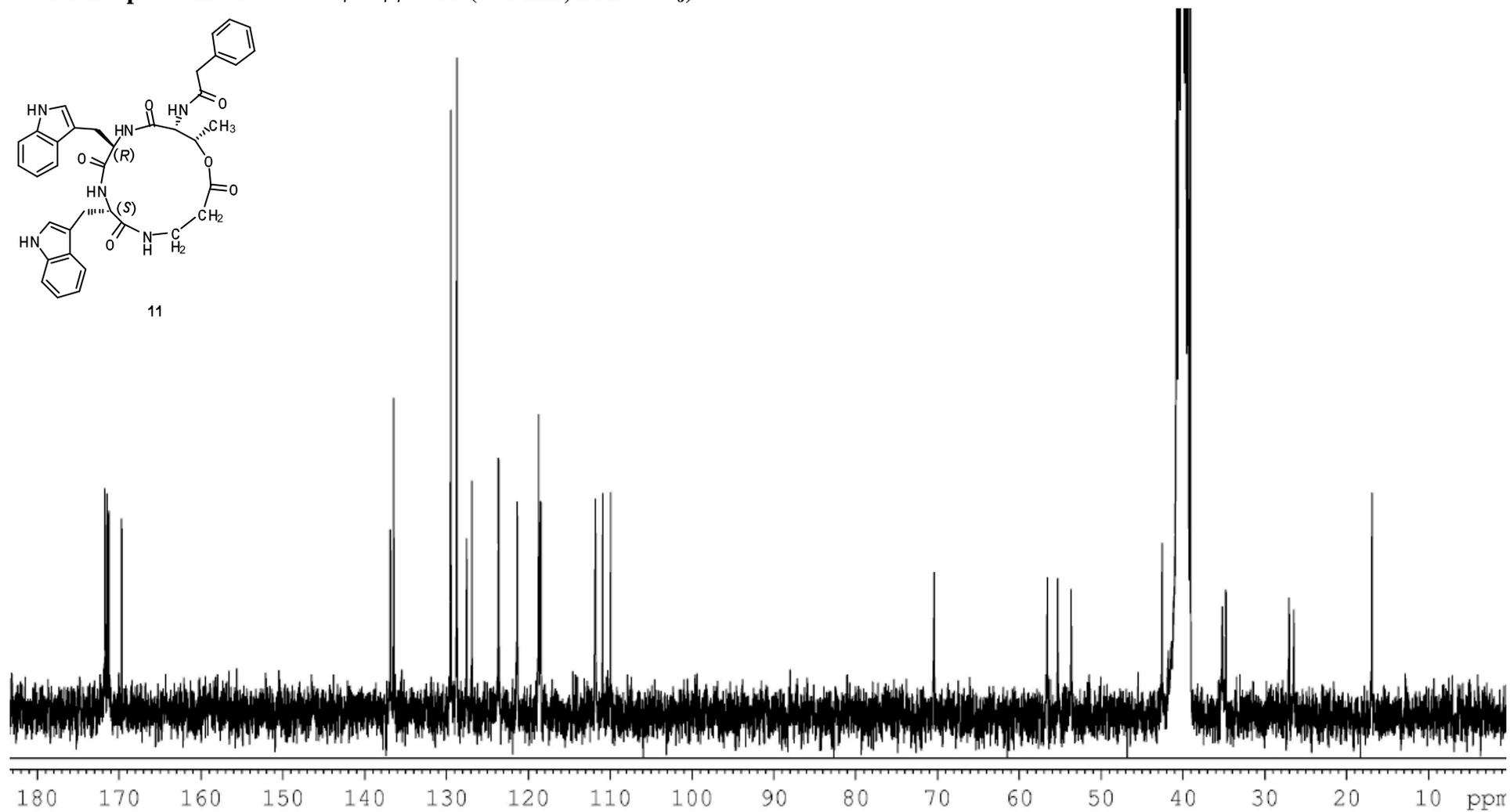
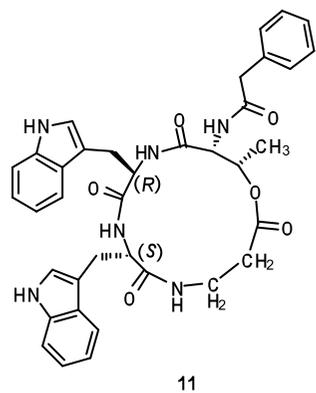
^{13}C NMR spectrum of isolated xenematide (600 MHz; $\text{DMSO-}d_6$)¹



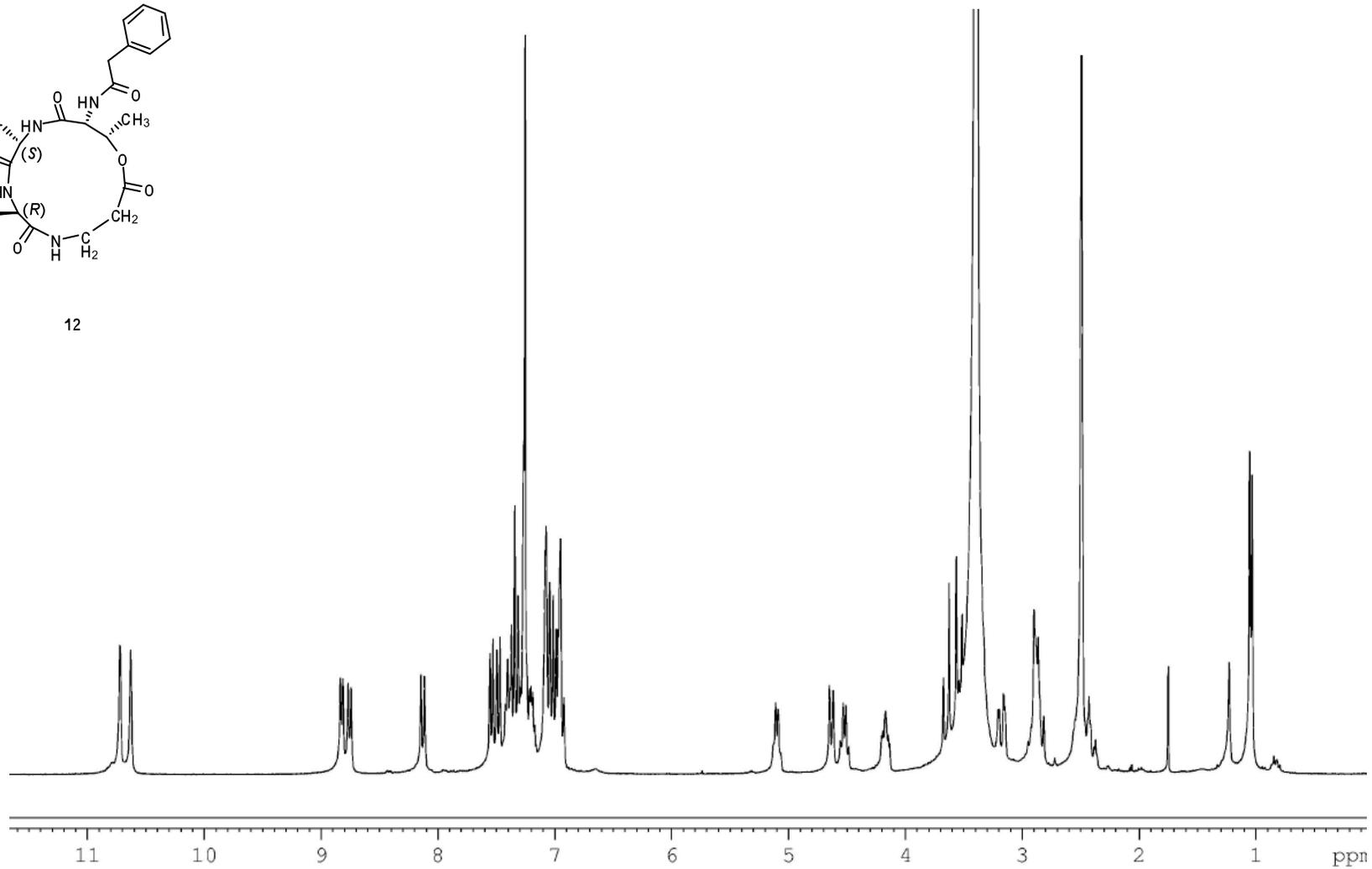
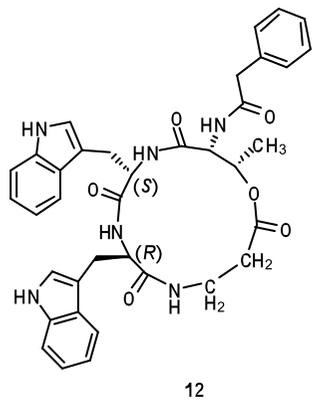
¹H NMR spectrum of P A-L-Thr-D-Trp-L-Trp-β-Ala **11** (300 MHz; DMSO-*d*₆)



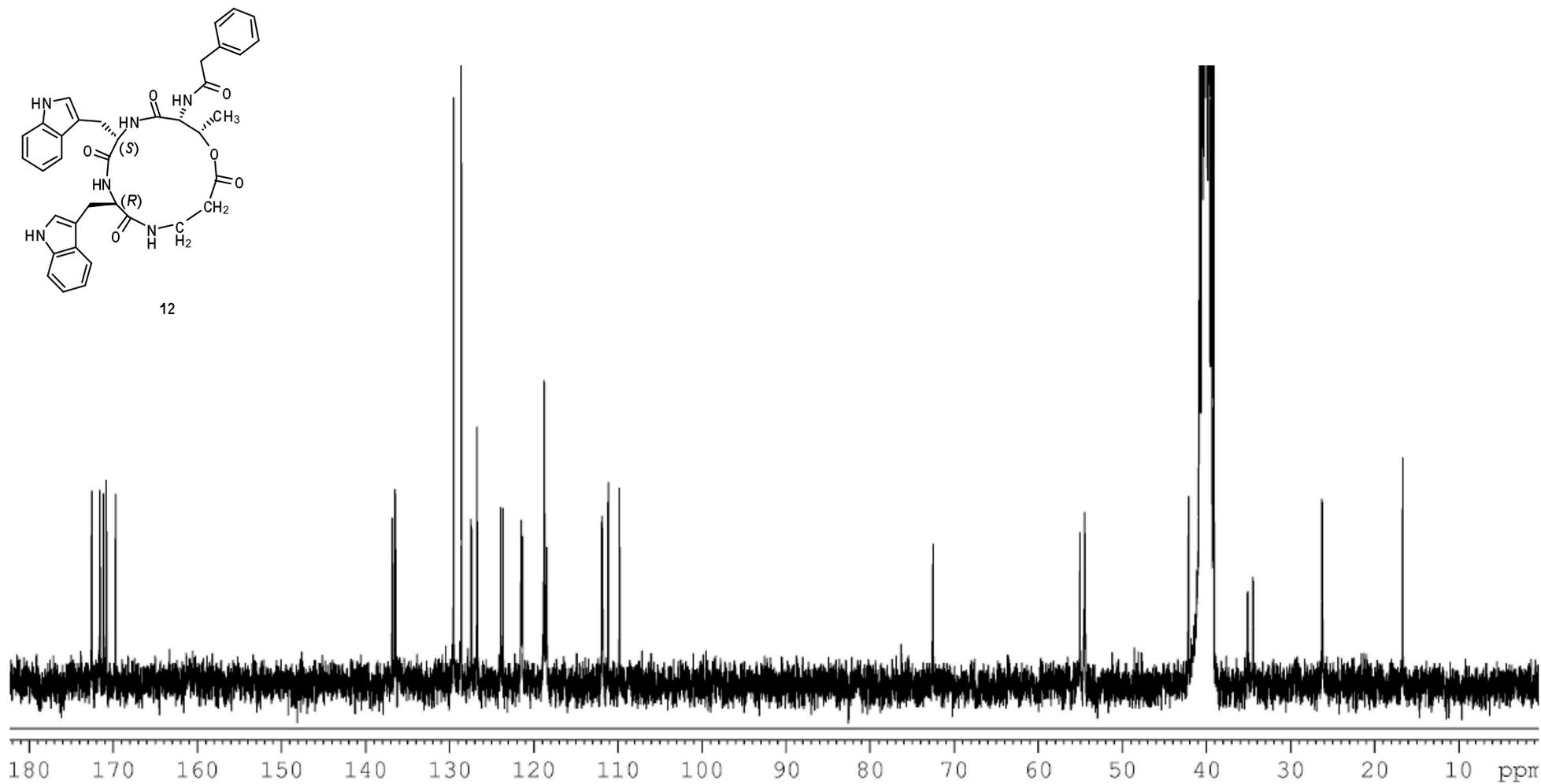
¹³C NMR spectrum of $\text{P}_A\text{-L-Thr-D-Trp-L-Trp-}\beta\text{-Ala}$ **11** (75 MHz; DMSO-*d*₆)



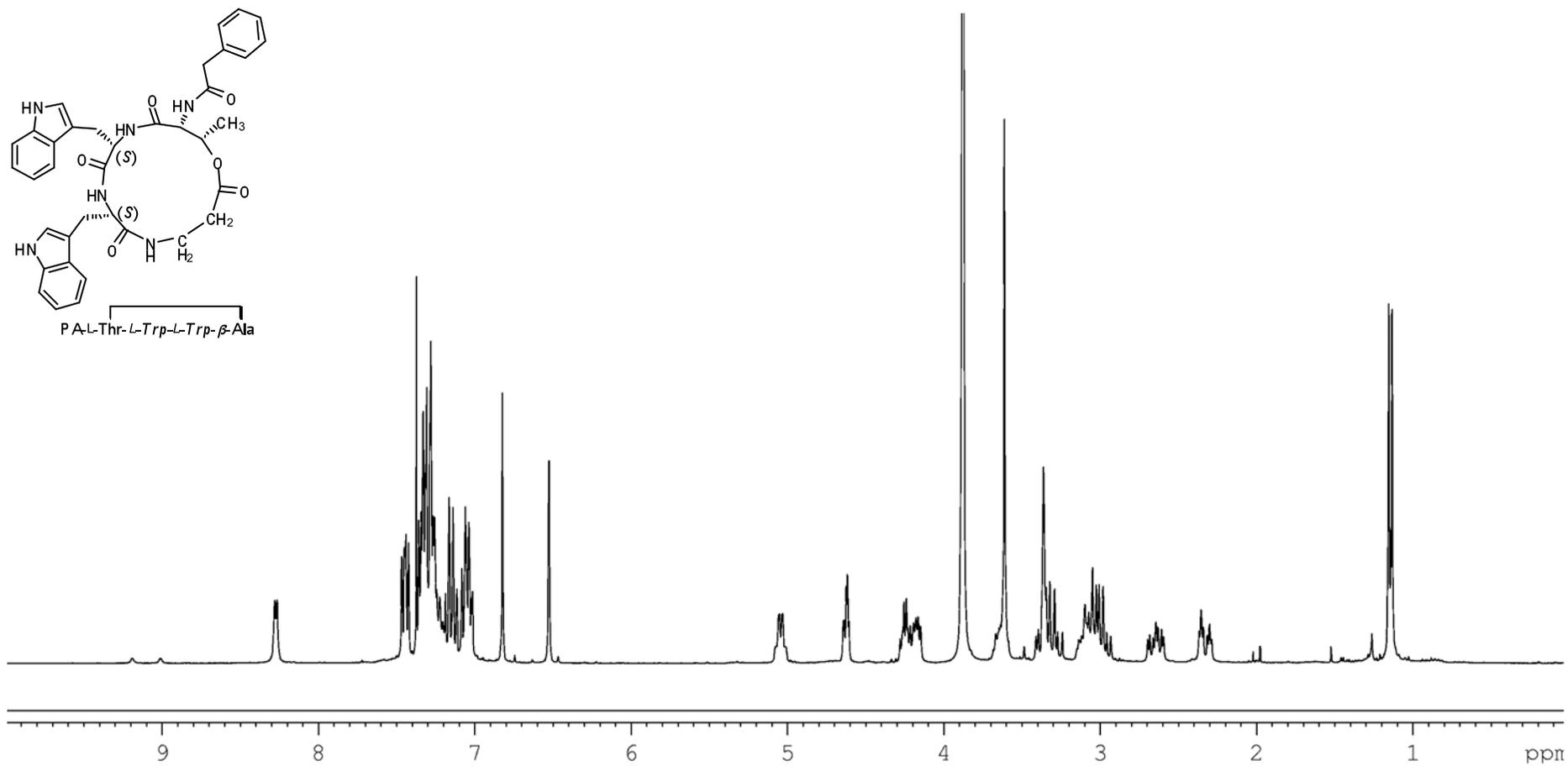
¹H NMR spectrum of P_A-L-Thr-L-Trp-D-Trp-β-Ala **12** (300 MHz; DMSO-*d*₆)



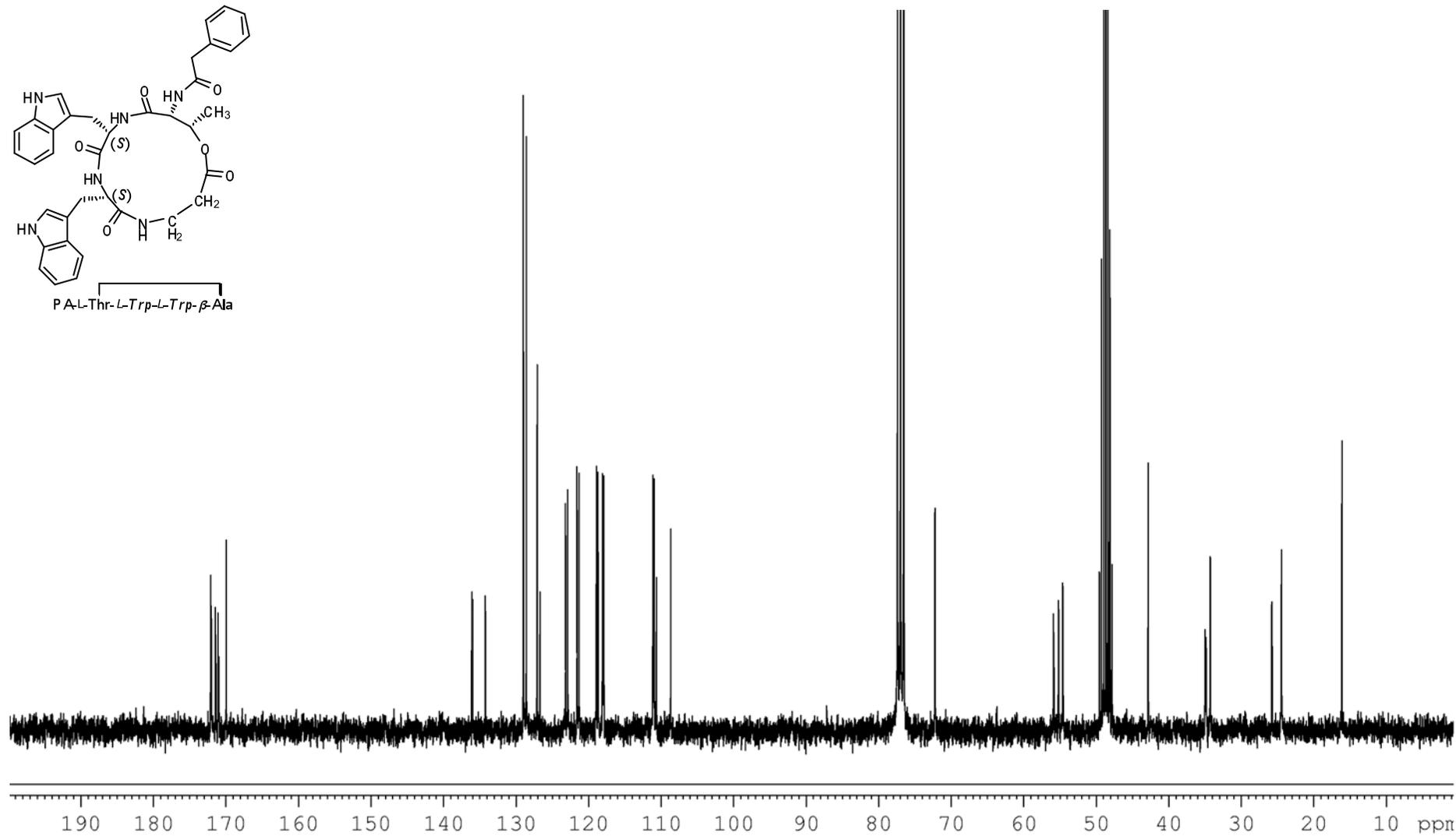
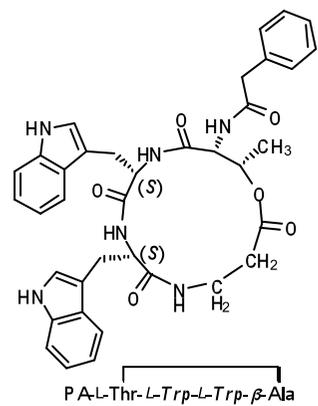
^{13}C NMR spectrum of $\text{P}_{\text{A-L-Thr-L-Trp-D-Trp-}\beta\text{-Ala}}$ **12** (75 MHz; $\text{DMSO-}d_6$)



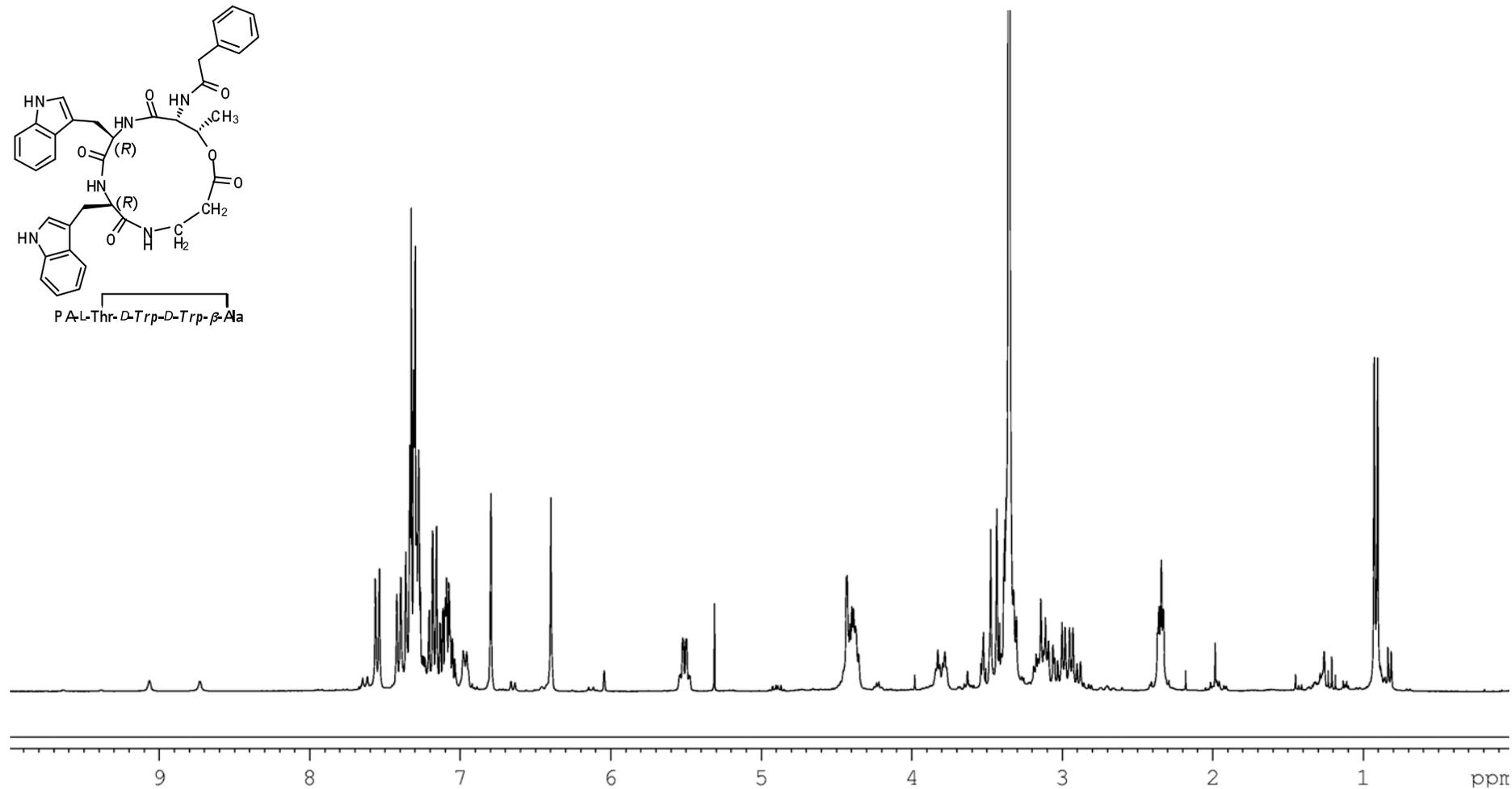
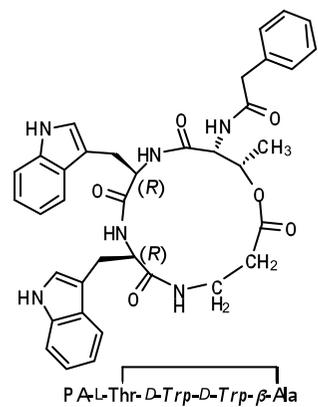
^1H NMR spectrum of $\text{P A-L-Thr-L-Trp-L-Trp-}\beta\text{-Ala}$ (300 MHz; CDCl_3)



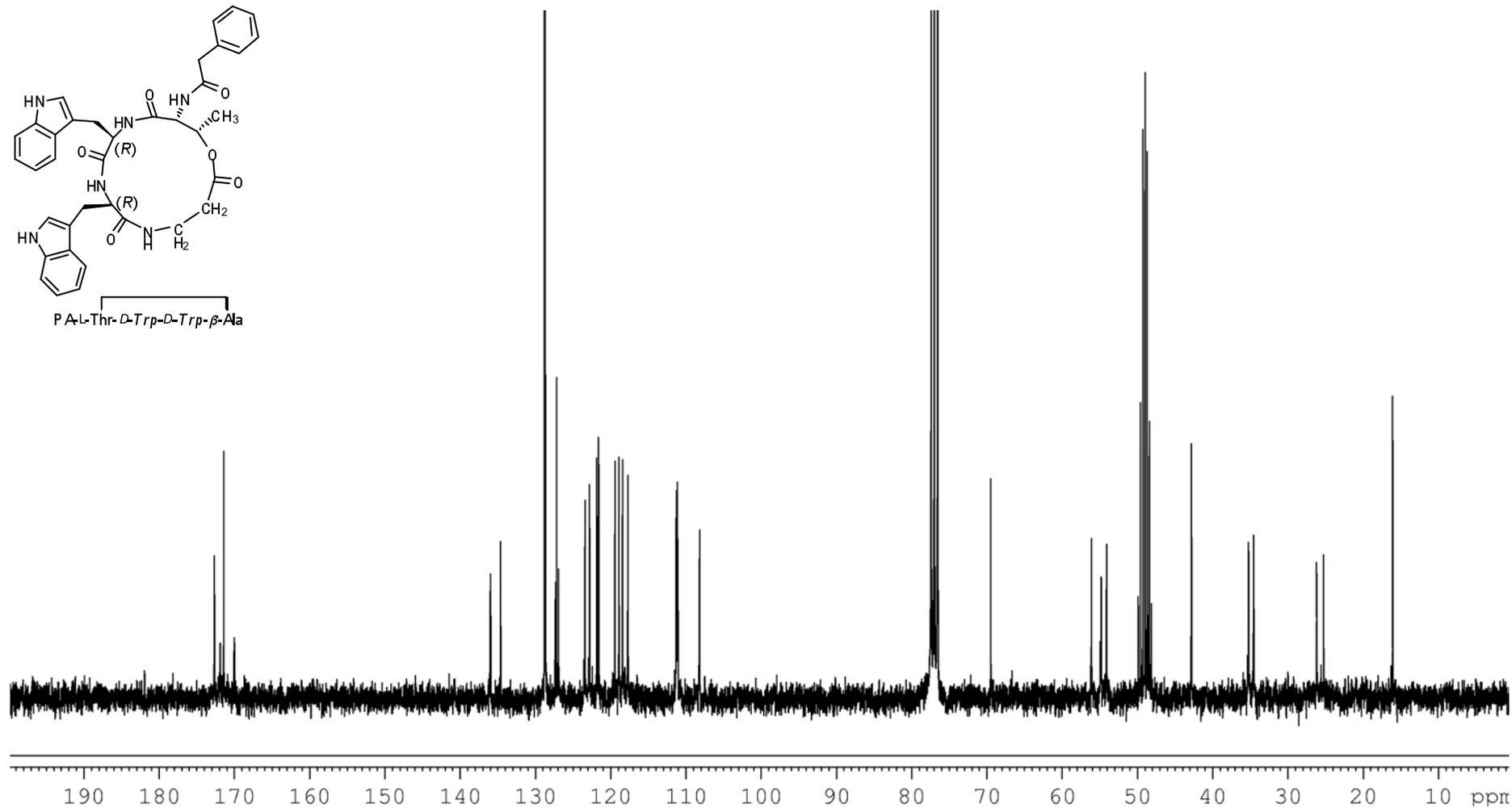
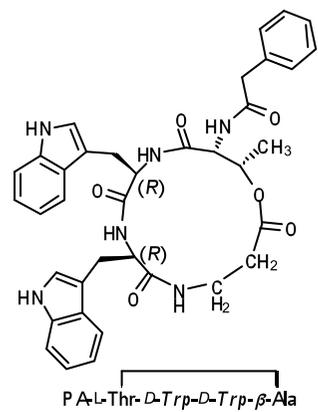
^{13}C NMR spectrum of $\text{P A-L-Thr-L-Trp-L-Trp-}\beta\text{-Ala}$ (75 MHz; CDCl_3)



^1H NMR spectrum of $\text{P Ala-L-Thr-D-Trp-D-Trp-}\beta\text{-Ala}$ (300 MHz; CDCl_3)



^{13}C NMR spectrum of $\text{P A-L-Thr-D-Trp-D-Trp-}\beta\text{-Ala}$ (75 MHz; CDCl_3)



References

1. G. Lang, T. Kalvelage, A. Peters, J. Wiese and J. F. Imhoff, *J. Nat. Prod.*, 2008, **71**, 1074–1077.