

## Supplementary Materials

# Preparation and Use of a General Solid-Phase Intermediate to Biomimetic Scaffolds and Peptide Condensations

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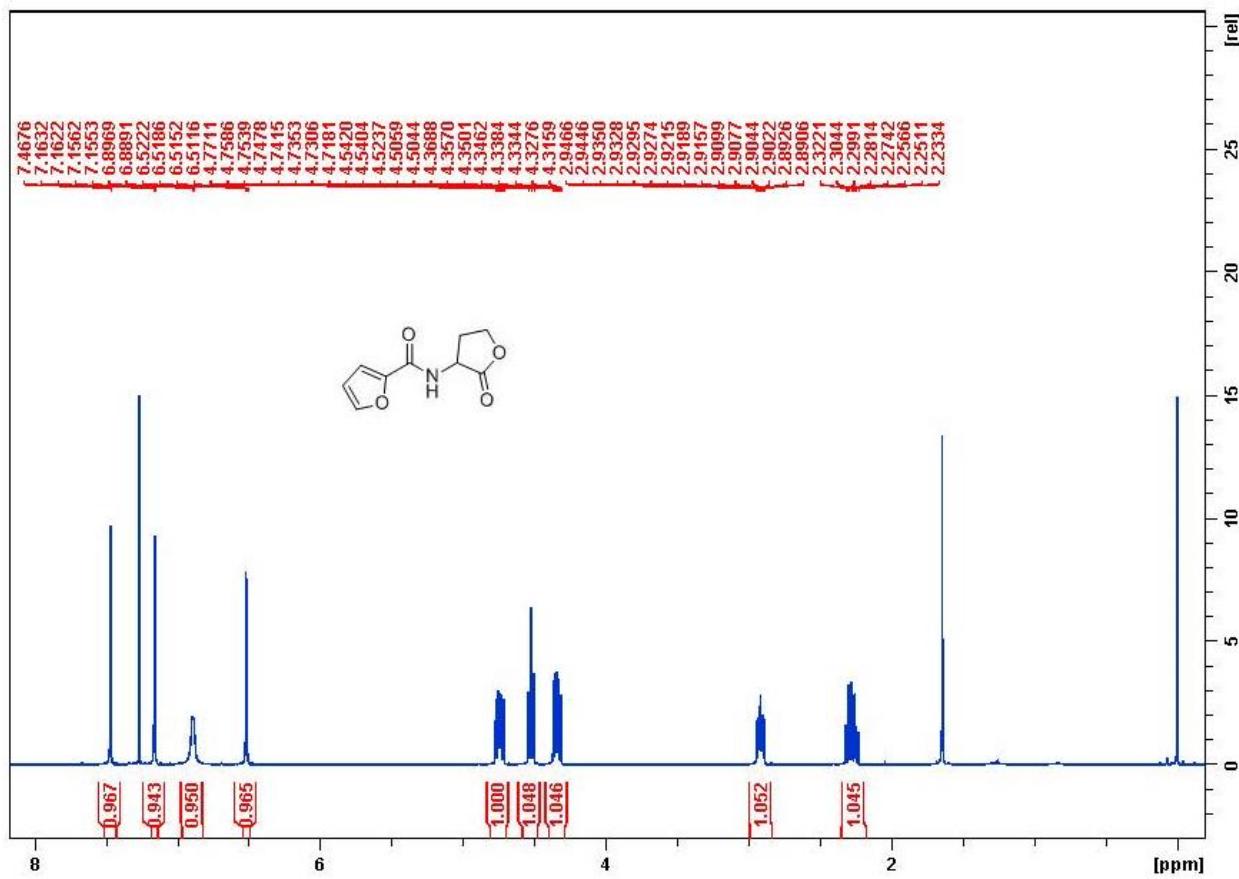
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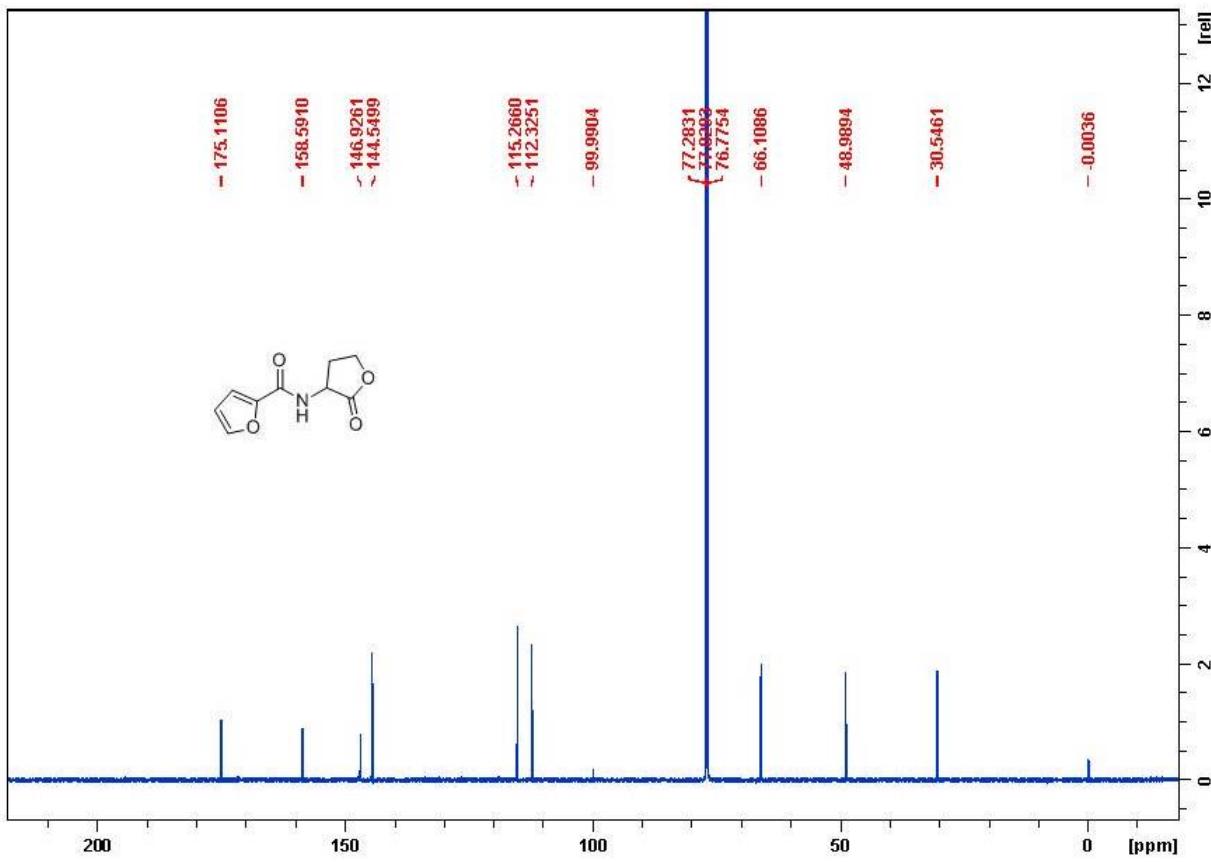
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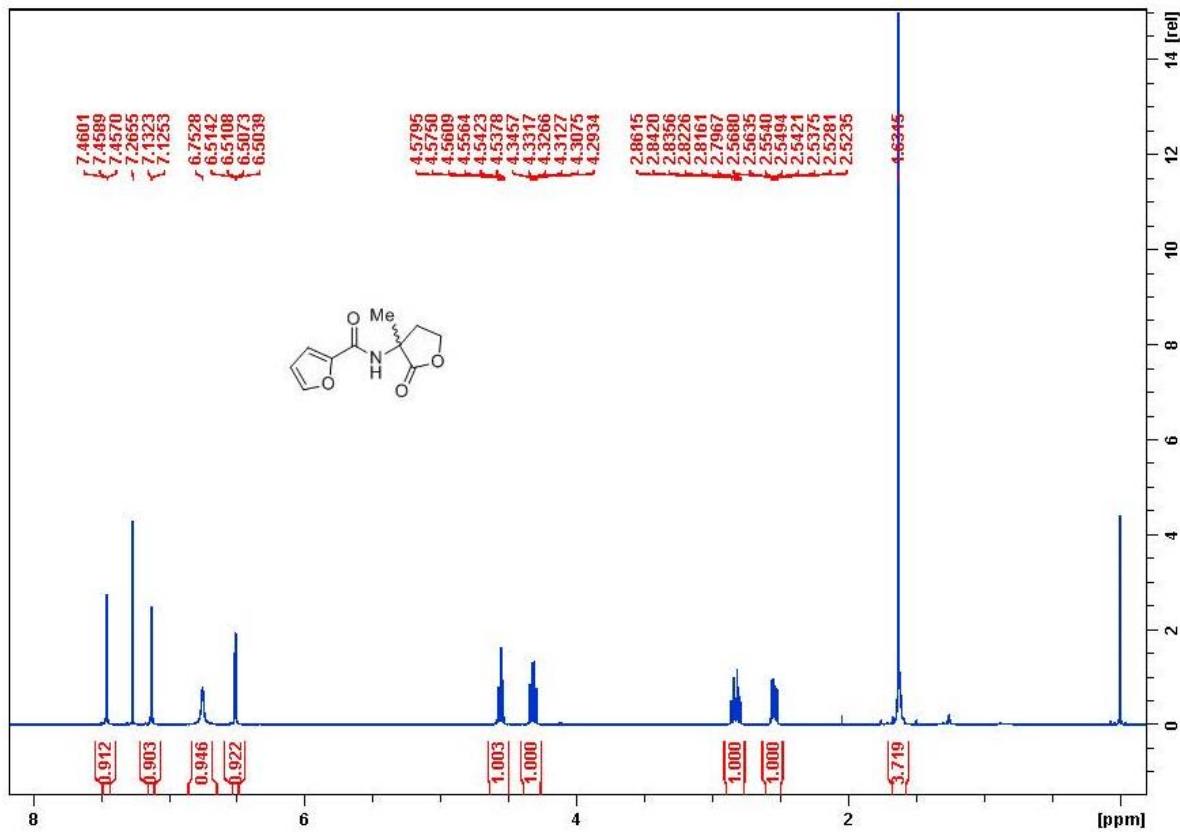
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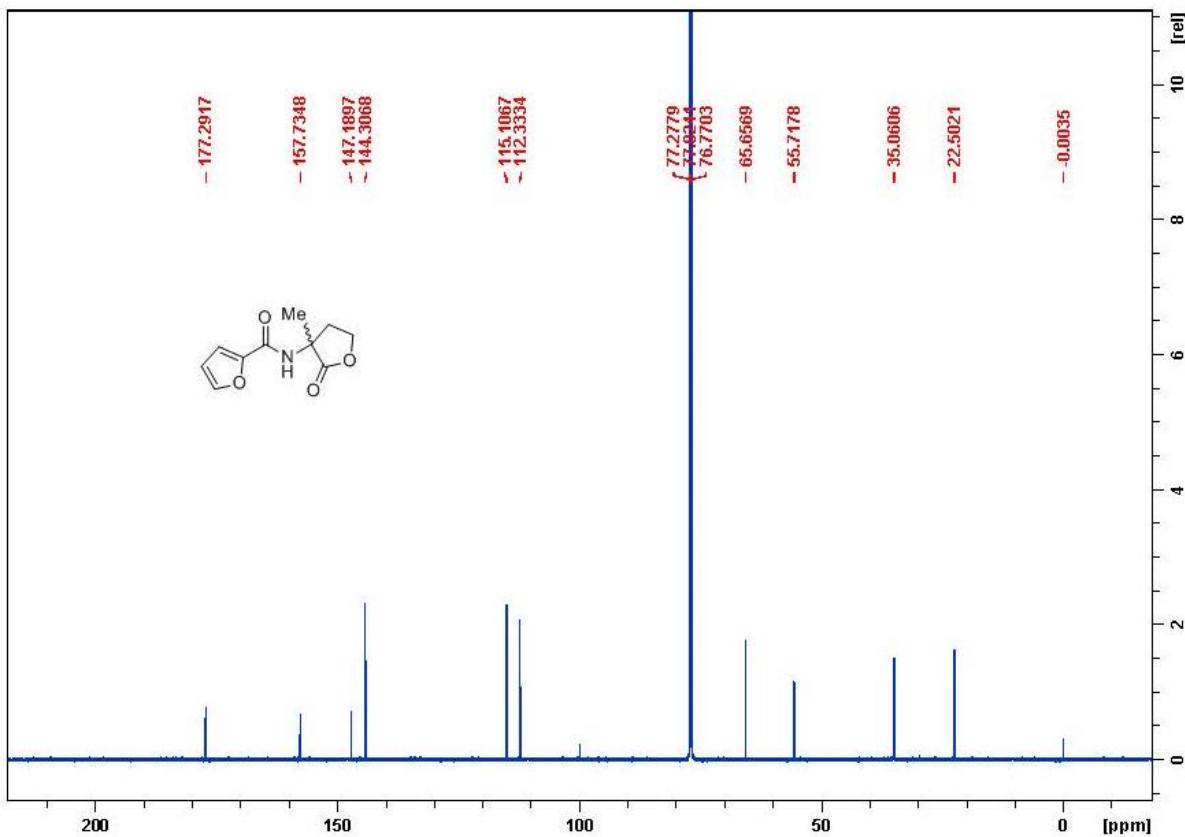
**Figure S1.** Proton NMR Spectrum of **19a** in  $\text{CDCl}_3$



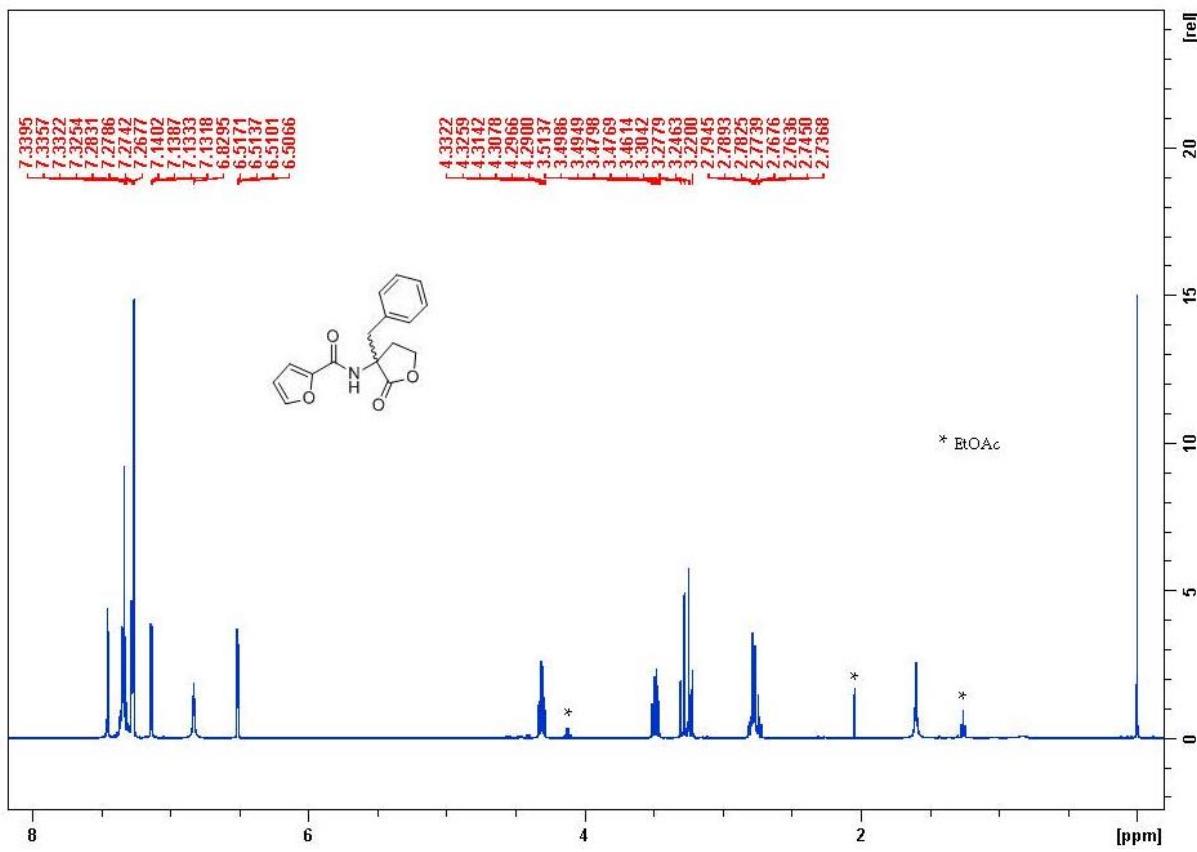
**Figure S2.** Carbon-13 NMR Spectrum of **19a** in  $\text{CDCl}_3$



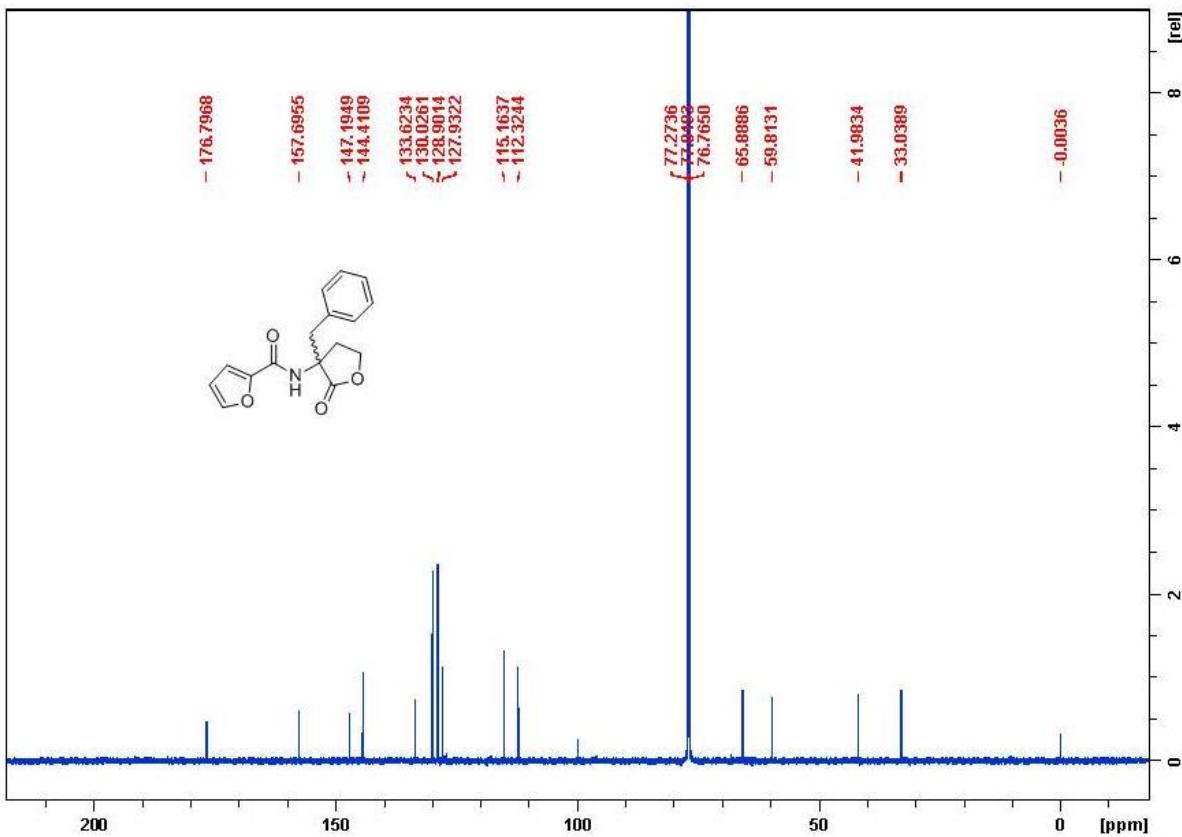
**Figure S3.** Proton NMR Spectrum of **19b** in  $\text{CDCl}_3$



**Figure S4.** Carbon-13 NMR Spectrum of **19b** in  $\text{CDCl}_3$



**Figure S5.** Proton NMR Spectrum of **19c** in  $\text{CDCl}_3$



**Figure S6.** Carbon-13 NMR Spectrum of **19c** in  $\text{CDCl}_3$

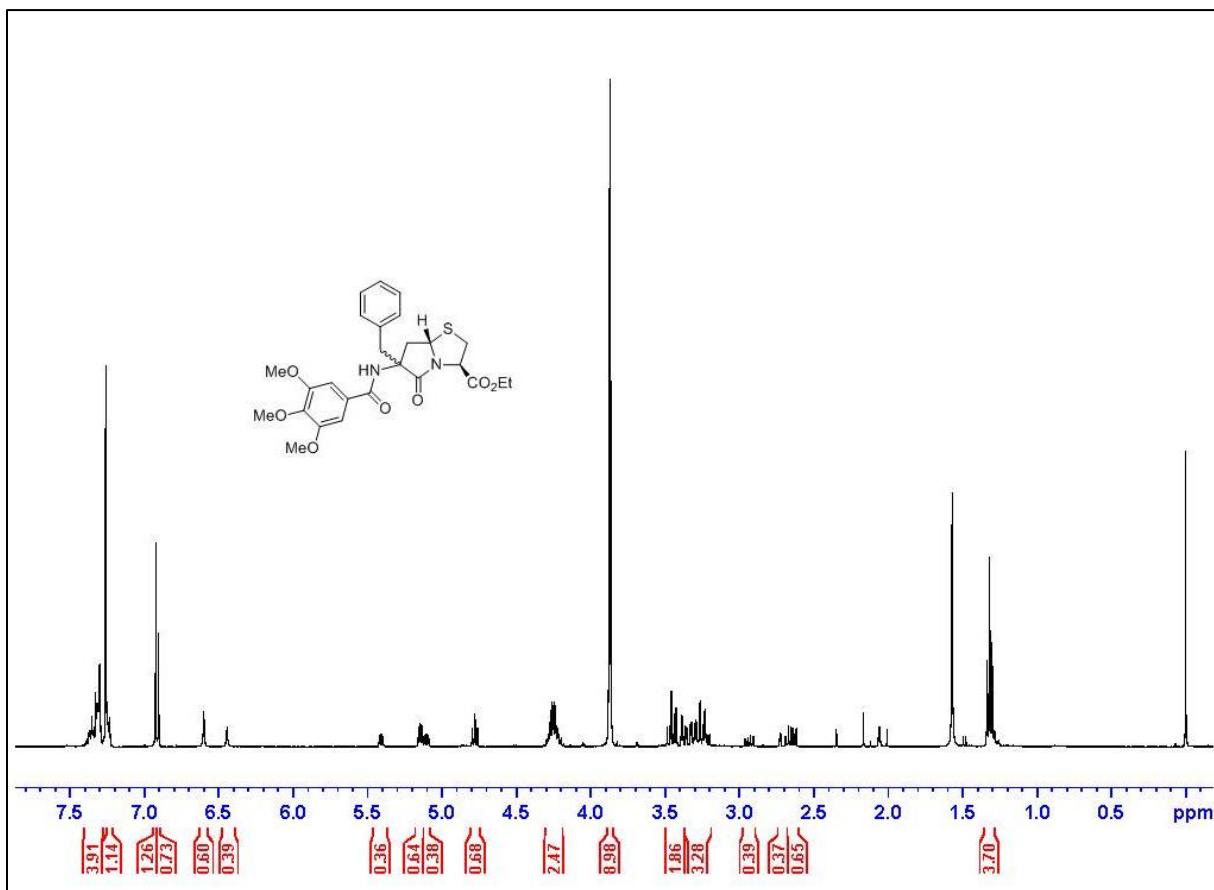


Figure S7. Proton NMR Spectrum of  $\alpha$ -21c and  $\beta$ -21c in  $\text{CDCl}_3$

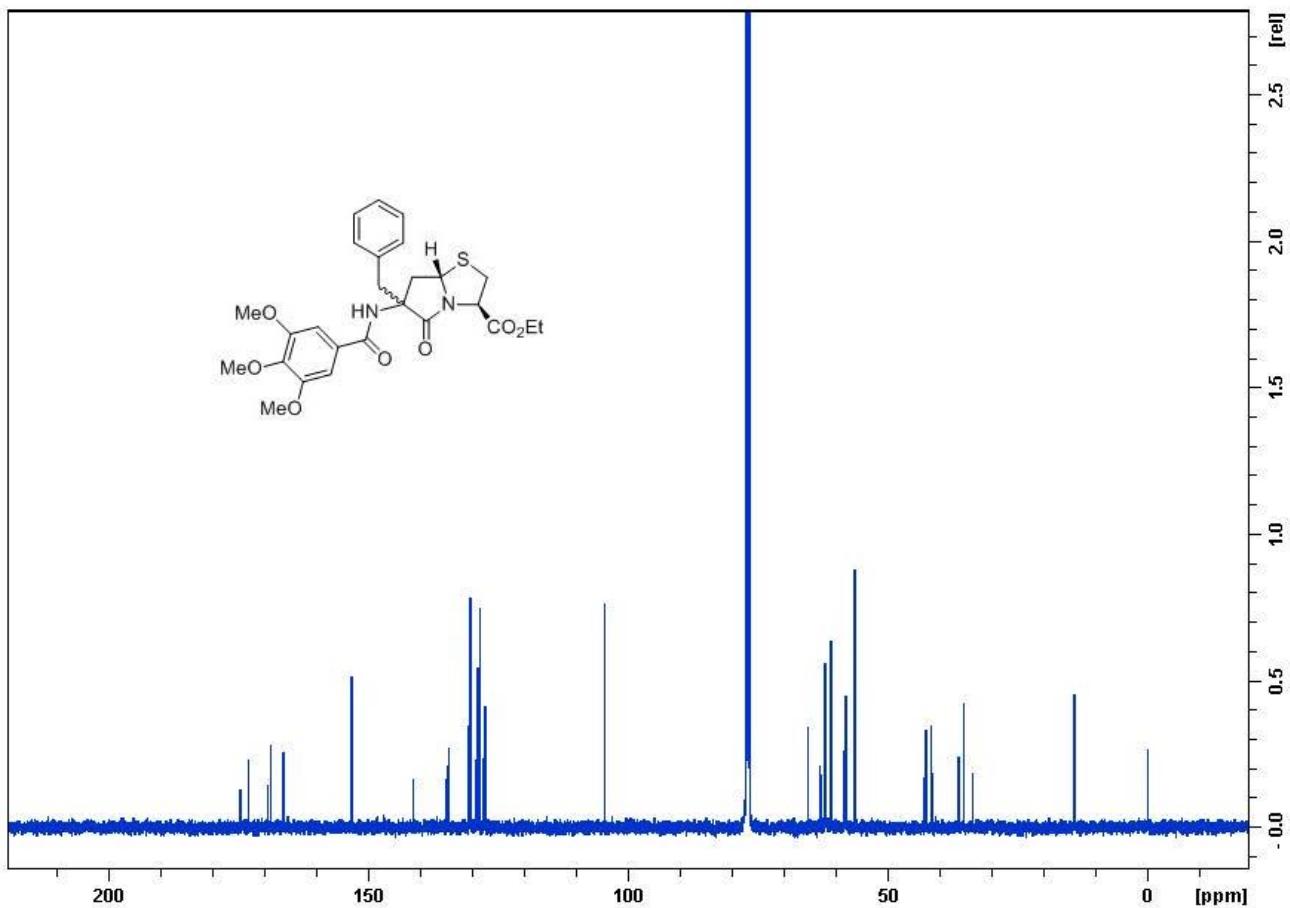
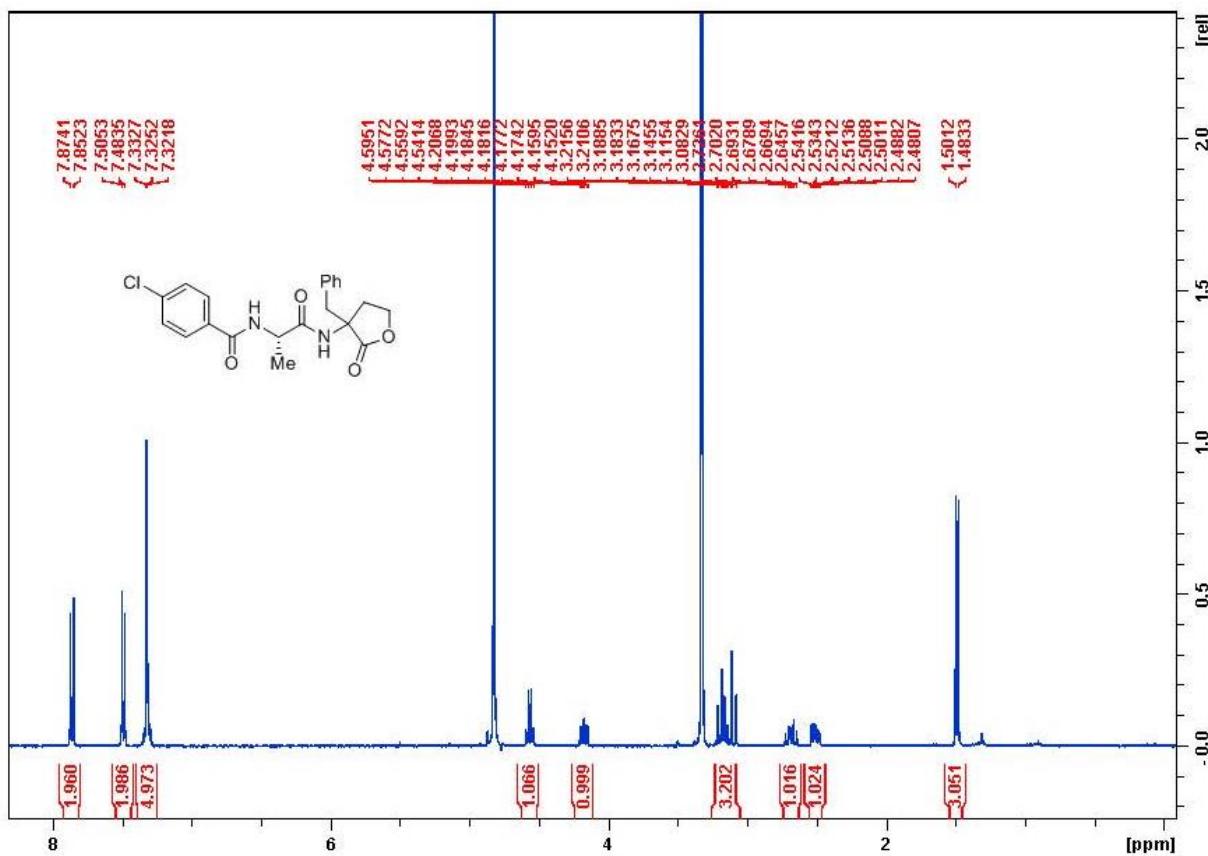
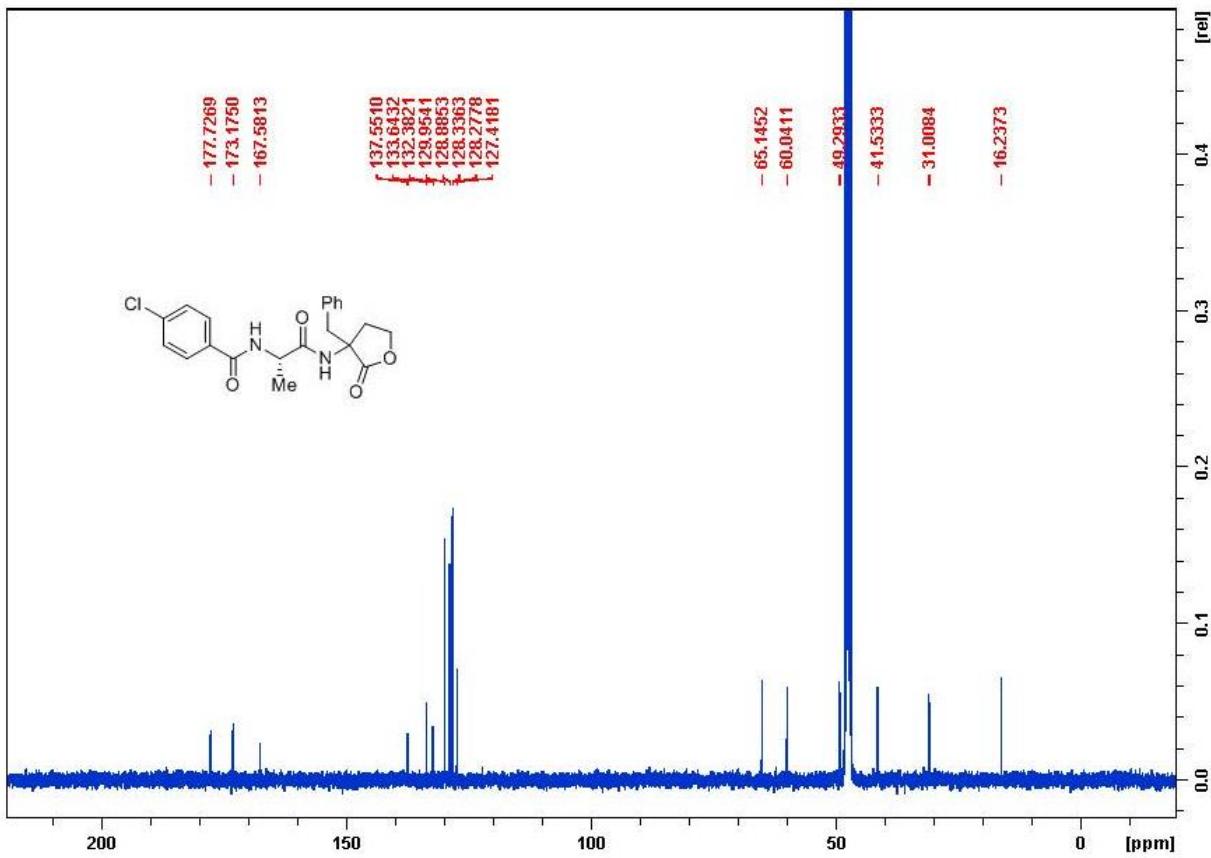


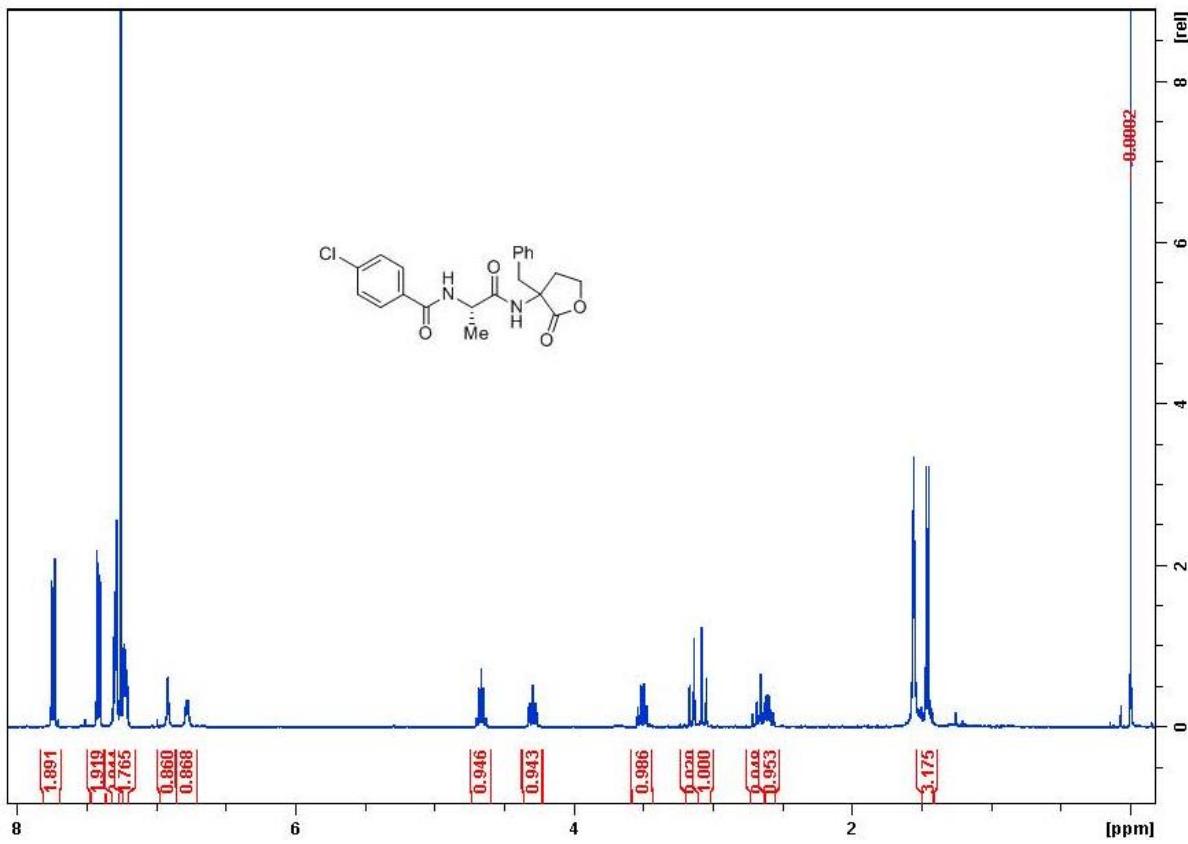
Figure S8. Carbon-13 NMR Spectrum of  $\alpha$ -21c and  $\beta$ -21c in  $\text{CDCl}_3$



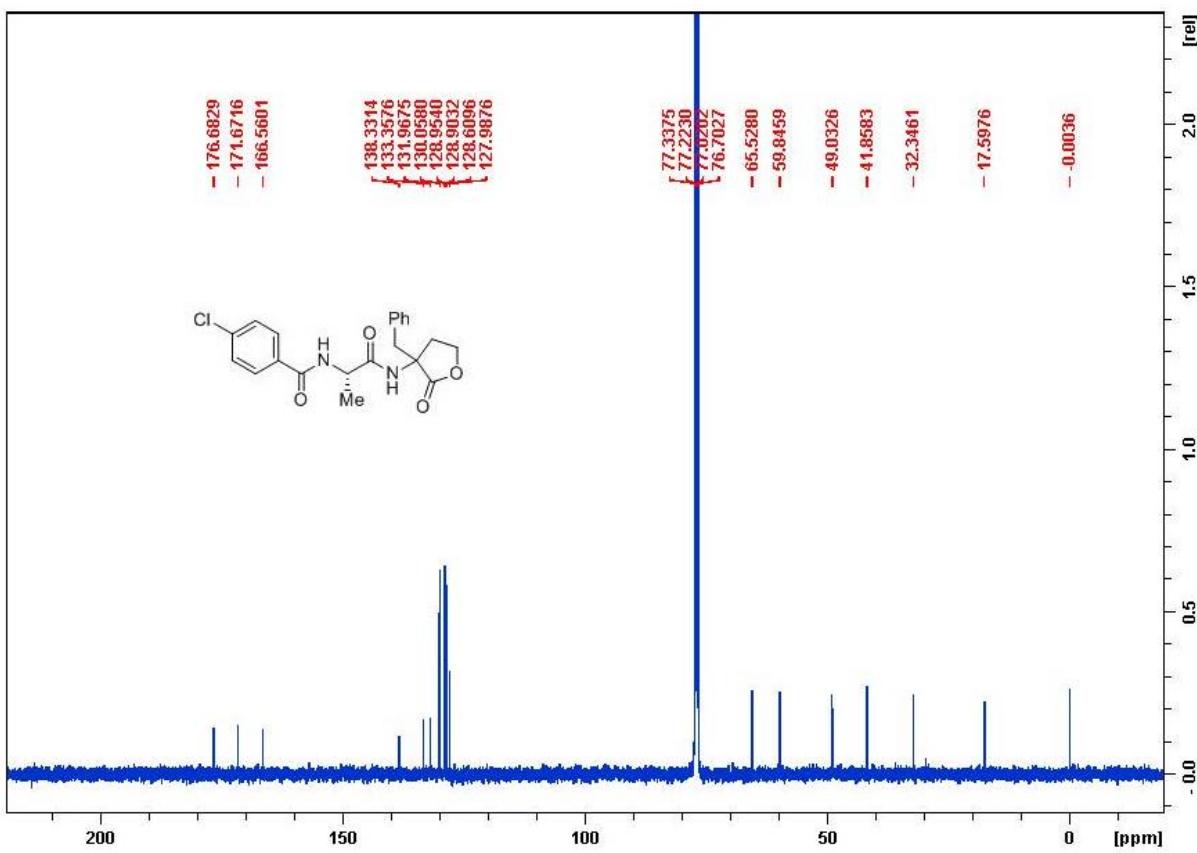
**Figure S9.** Proton NMR Spectrum of **24c** (earlier  $R_t$ ) in  $\text{CD}_5\text{OD}$



**Figure S10.** Carbon-13 NMR Spectrum of **24c** (earlier  $R_t$ ) in  $\text{CD}_3\text{OD}$



**Figure S11.** Proton NMR Spectrum of **24c** (later  $R_t$ ) in  $\text{CDCl}_3$



**Figure S12.** Carbon-13 NMR Spectrum of **24c** (later R<sub>t</sub>) in CDCl<sub>3</sub>

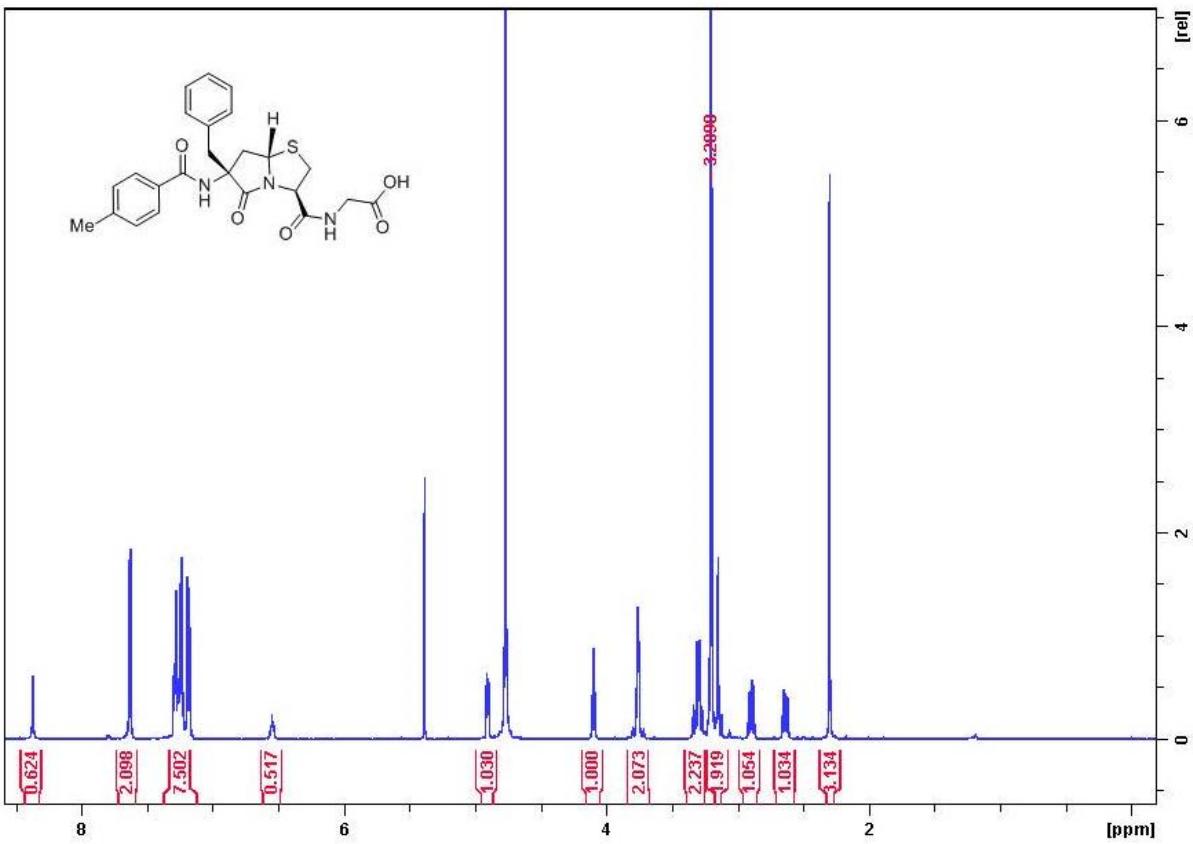
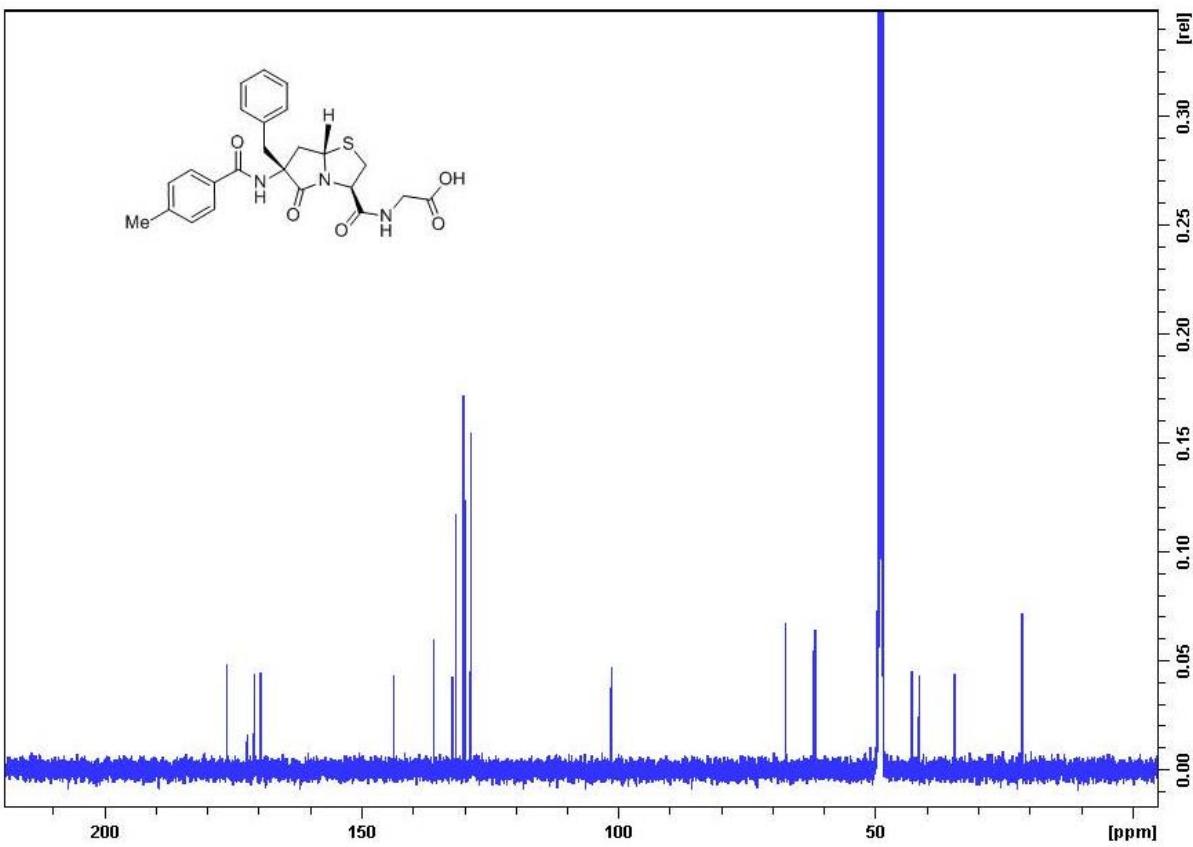
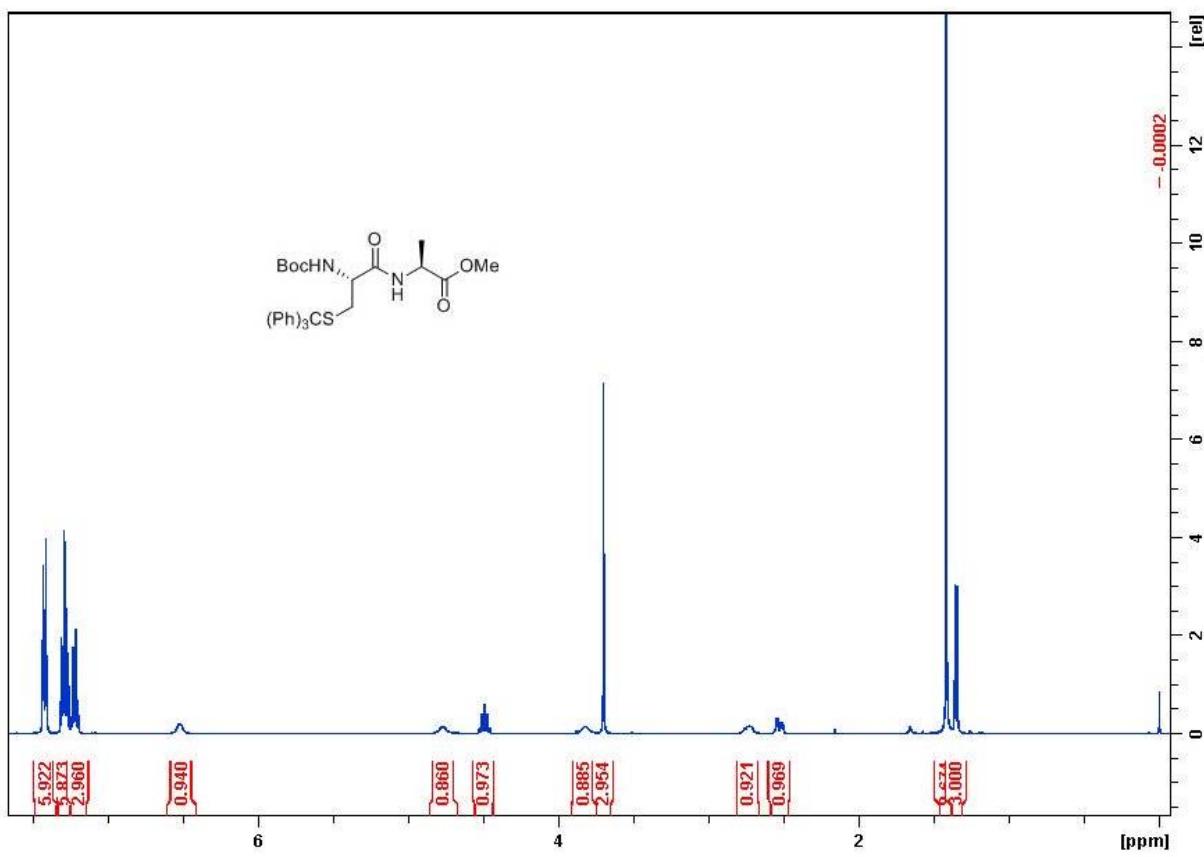


Figure S13. Proton NMR Spectrum of  $\beta$ -26c in  $\text{CD}_3\text{OD}$



**Figure S14.** Carbon-13 NMR Spectrum of  $\beta\text{-26c}$  in  $\text{CD}_3\text{OD}$



**Figure S15.** Proton NMR Spectrum of 31 in  $\text{CDCl}_3$

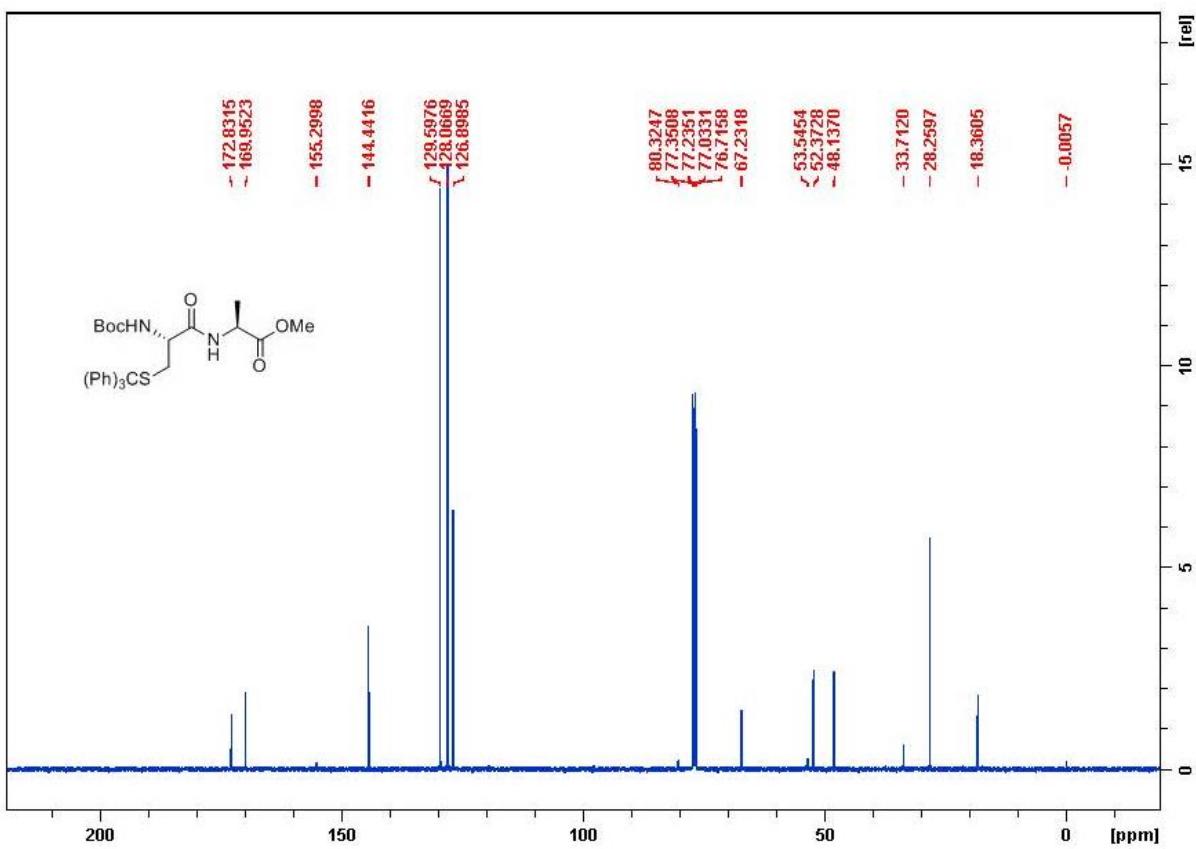


Figure S16. Carbon-13 NMR Spectrum of **31** in  $\text{CDCl}_3$

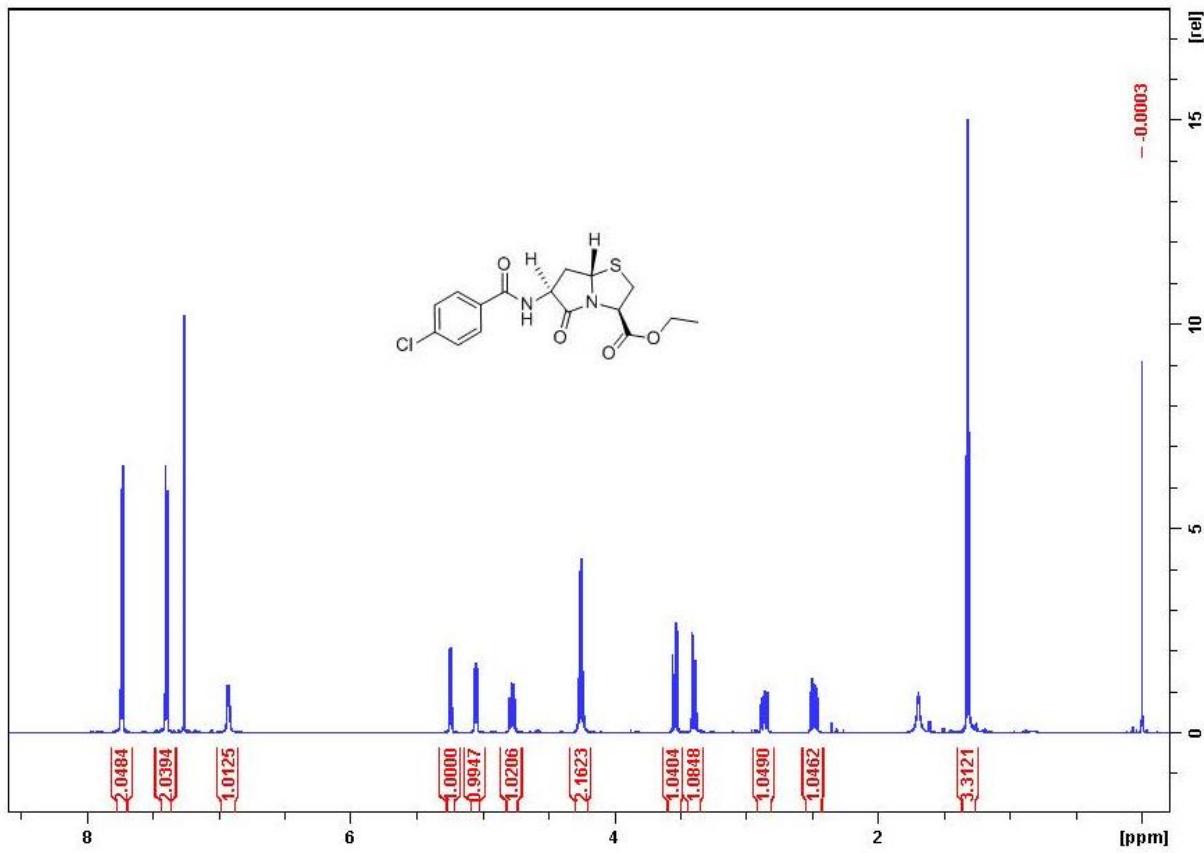
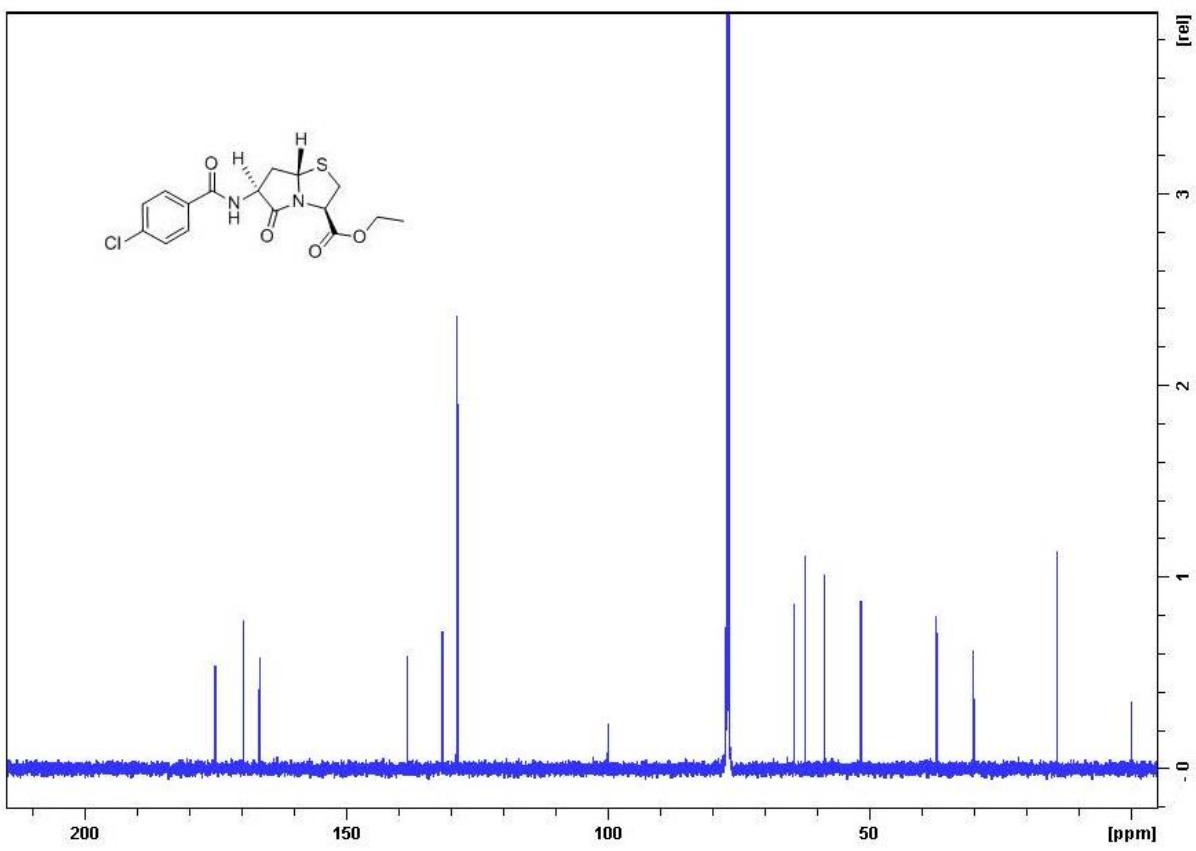


Figure S17. Proton NMR Spectrum of  $\alpha$ -33a in  $\text{CDCl}_3$



**Figure S18.** Carbon-13 NMR Spectrum of  $\alpha$ -33a in  $\text{CDCl}_3$

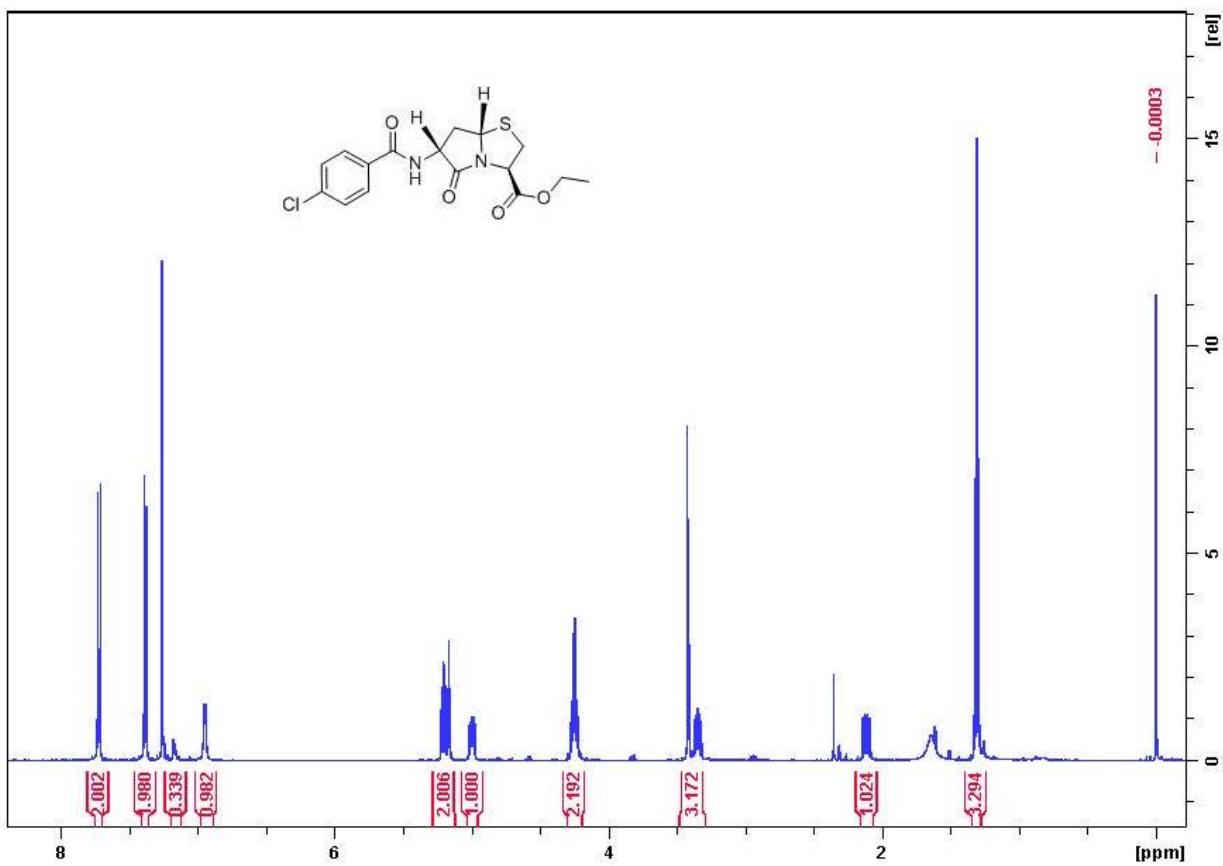


Figure S19. Proton NMR Spectrum of  $\beta$ -33a in  $\text{CDCl}_3$

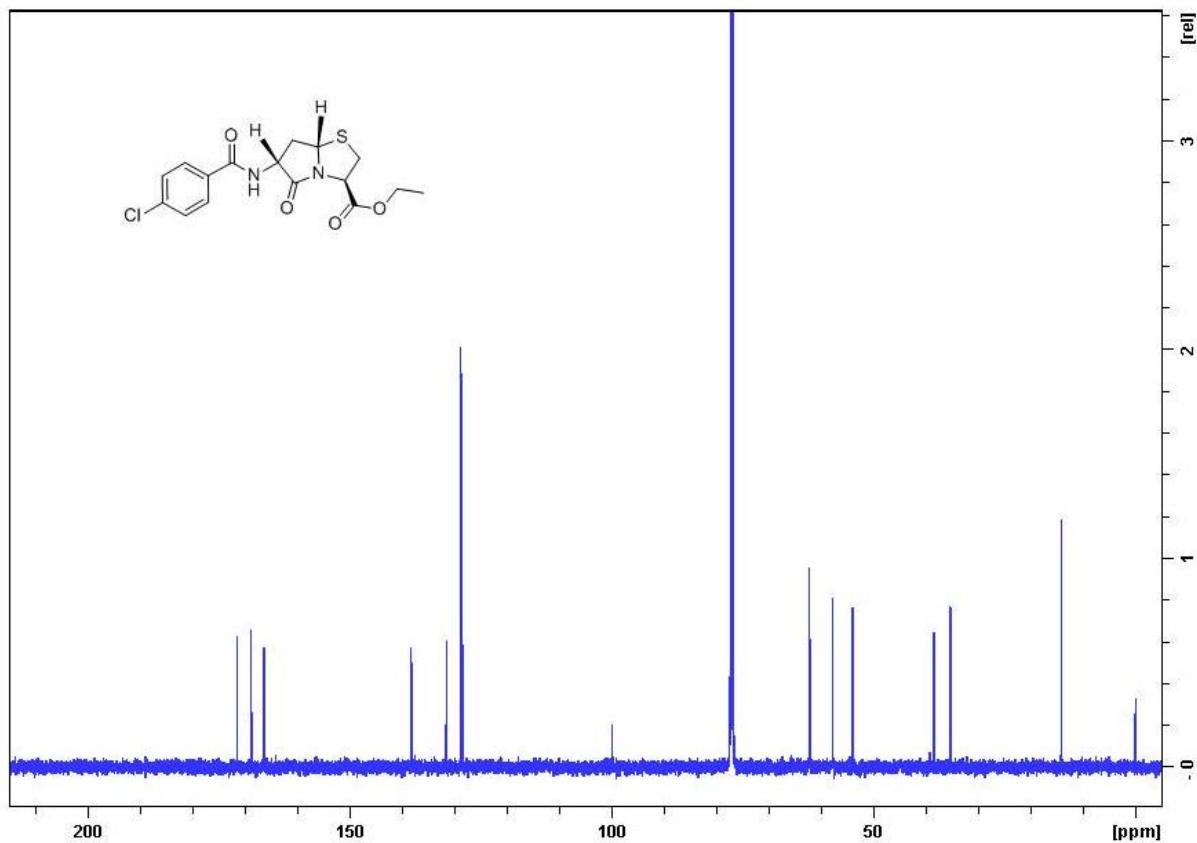
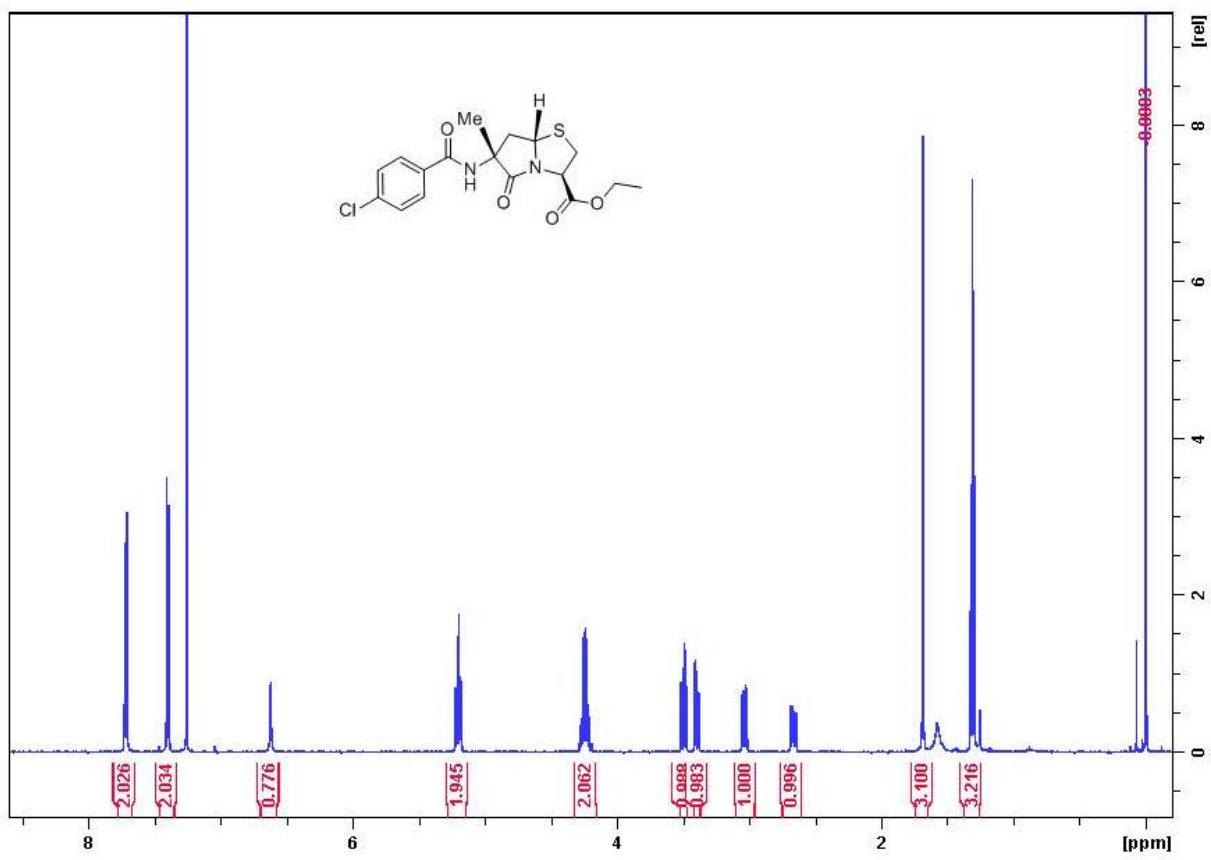
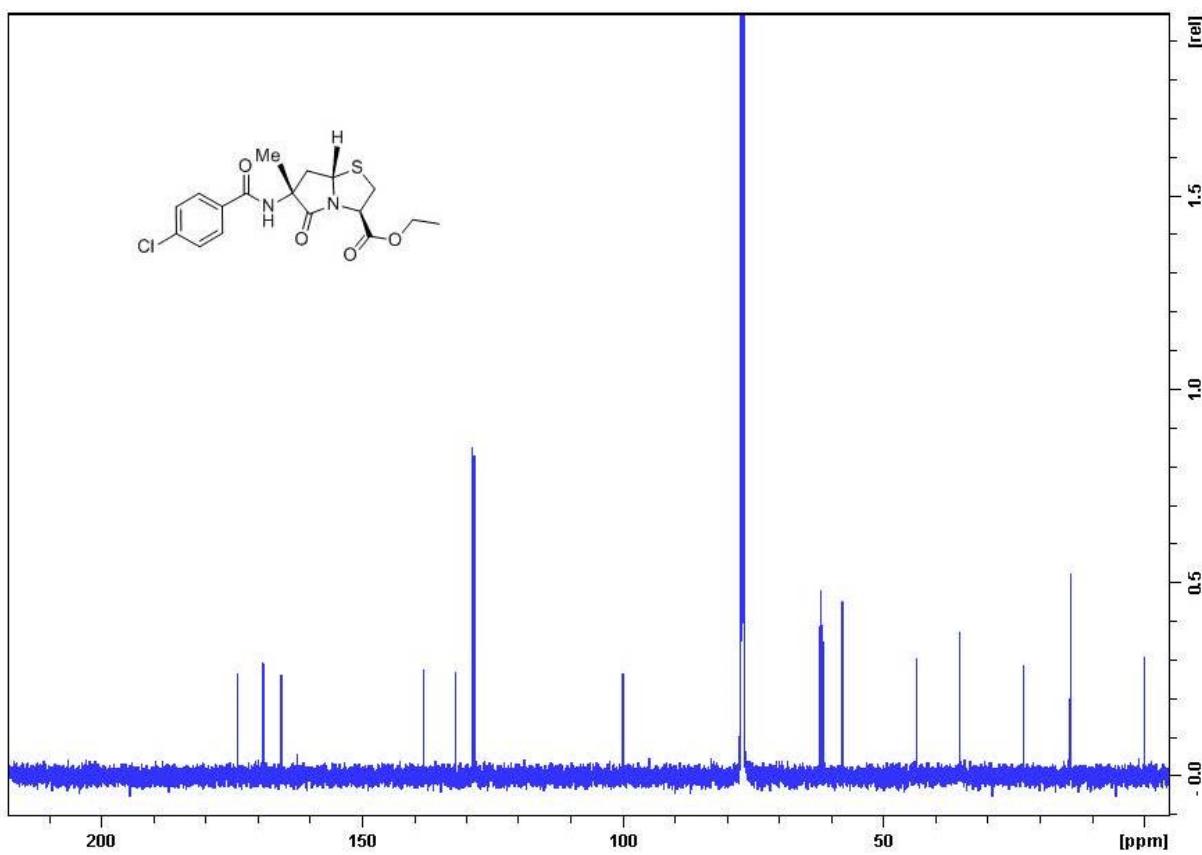


Figure S20. Carbon-13 NMR Spectrum of  $\beta$ -33a in CDCl<sub>3</sub>



**Figure S21.** Proton NMR Spectrum of  $\beta$ -33b in  $\text{CDCl}_3$



**Figure S22.** Carbon-13 NMR Spectrum of  $\beta\text{-}33\text{b}$  in  $\text{CDCl}_3$

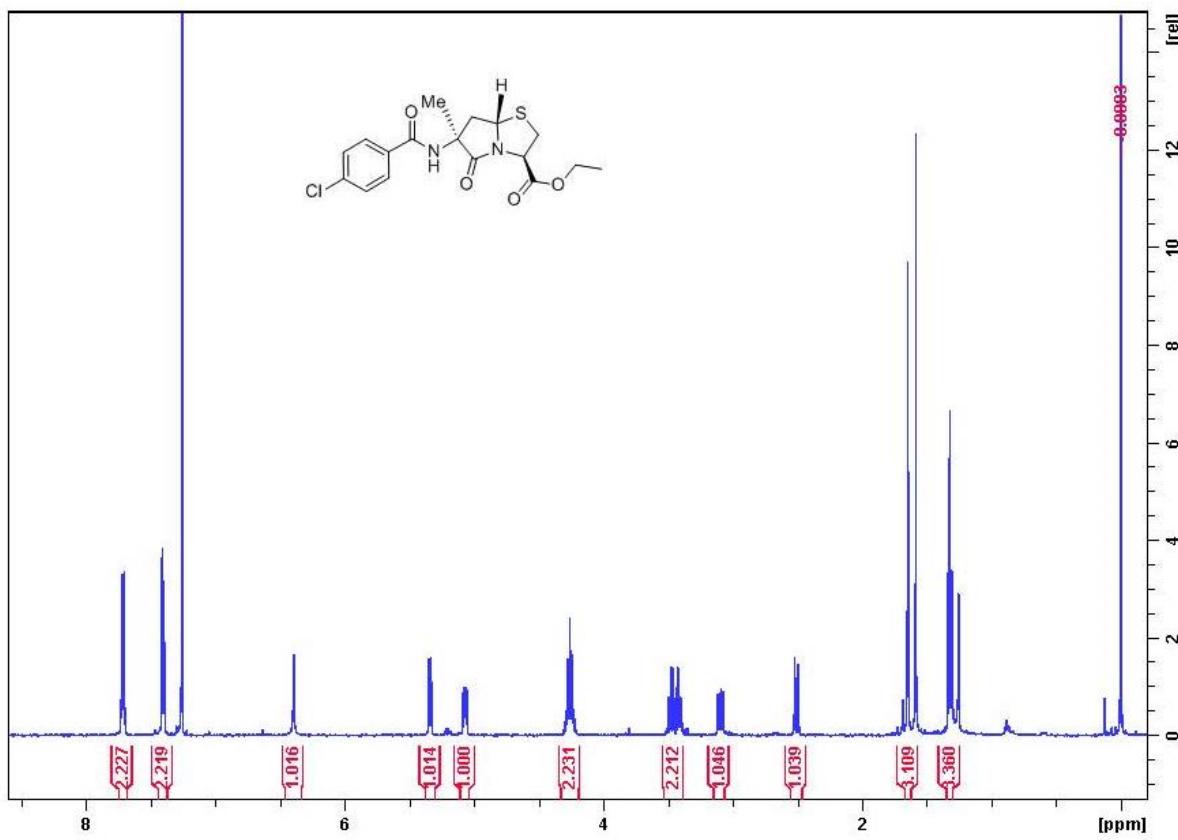


Figure S23. Proton NMR Spectrum of  $\alpha$ -33b in  $\text{CDCl}_3$

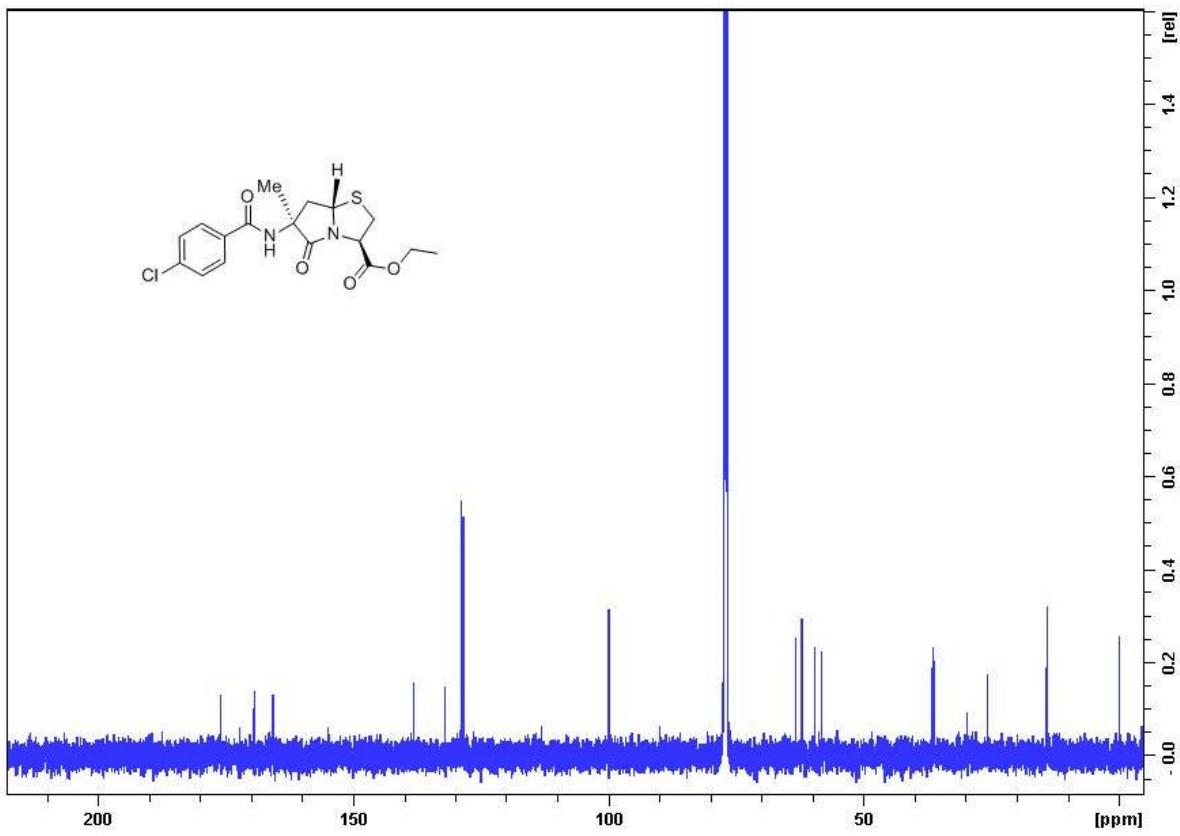


Figure S24. Carbon-13 NMR Spectrum of  $\alpha\text{-}33\text{b}$  in  $\text{CDCl}_3$

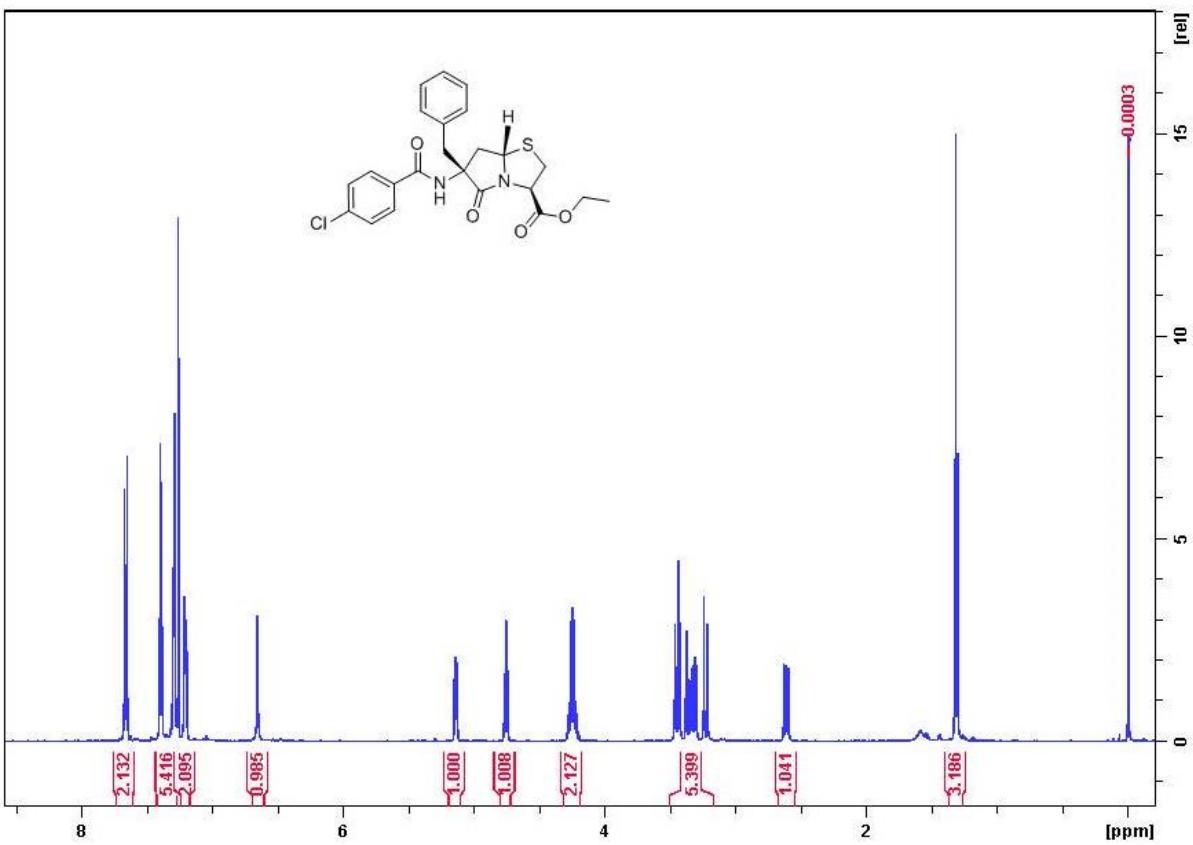


Figure S25. Proton NMR Spectrum of  $\beta$ -33c in  $\text{CDCl}_3$

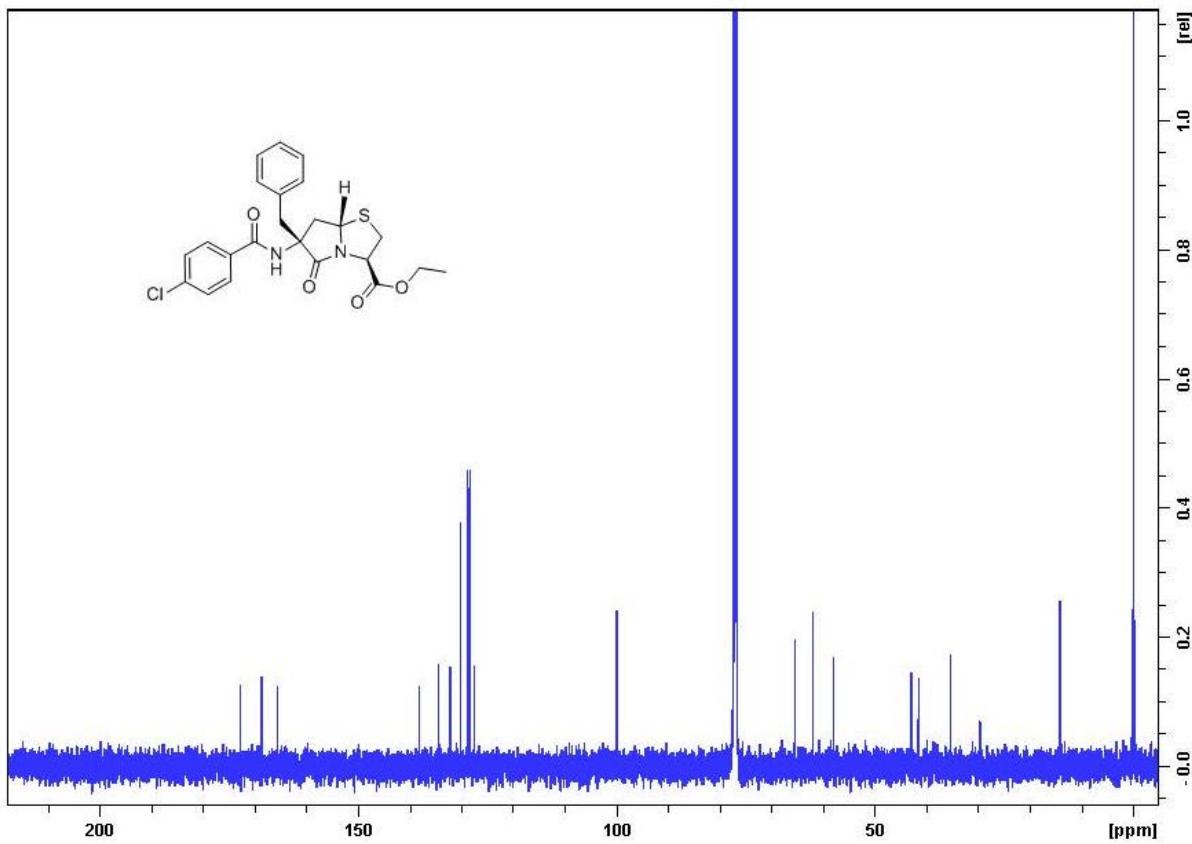


Figure S26. Carbon-13 NMR Spectrum of  $\beta$ -33c in  $\text{CDCl}_3$

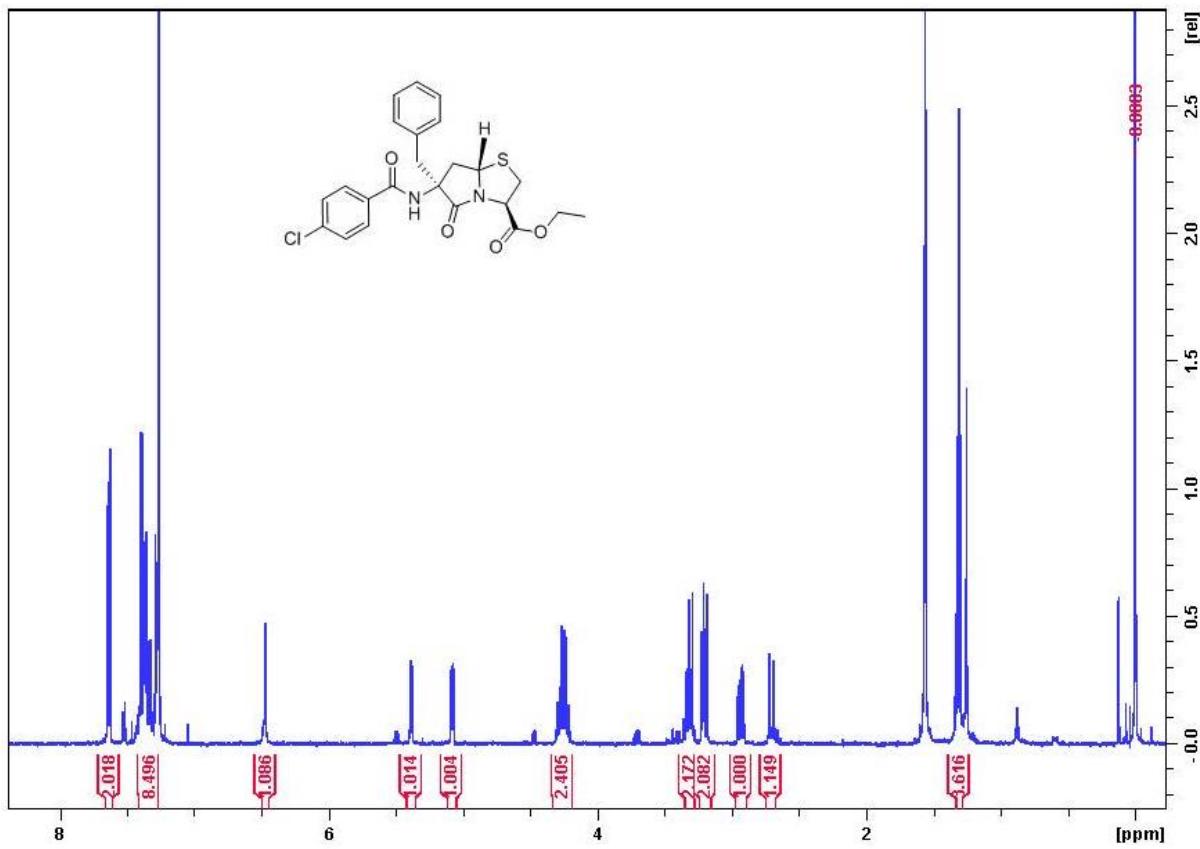


Figure S27. Proton NMR Spectrum of  $\alpha$ -33c in  $\text{CDCl}_3$

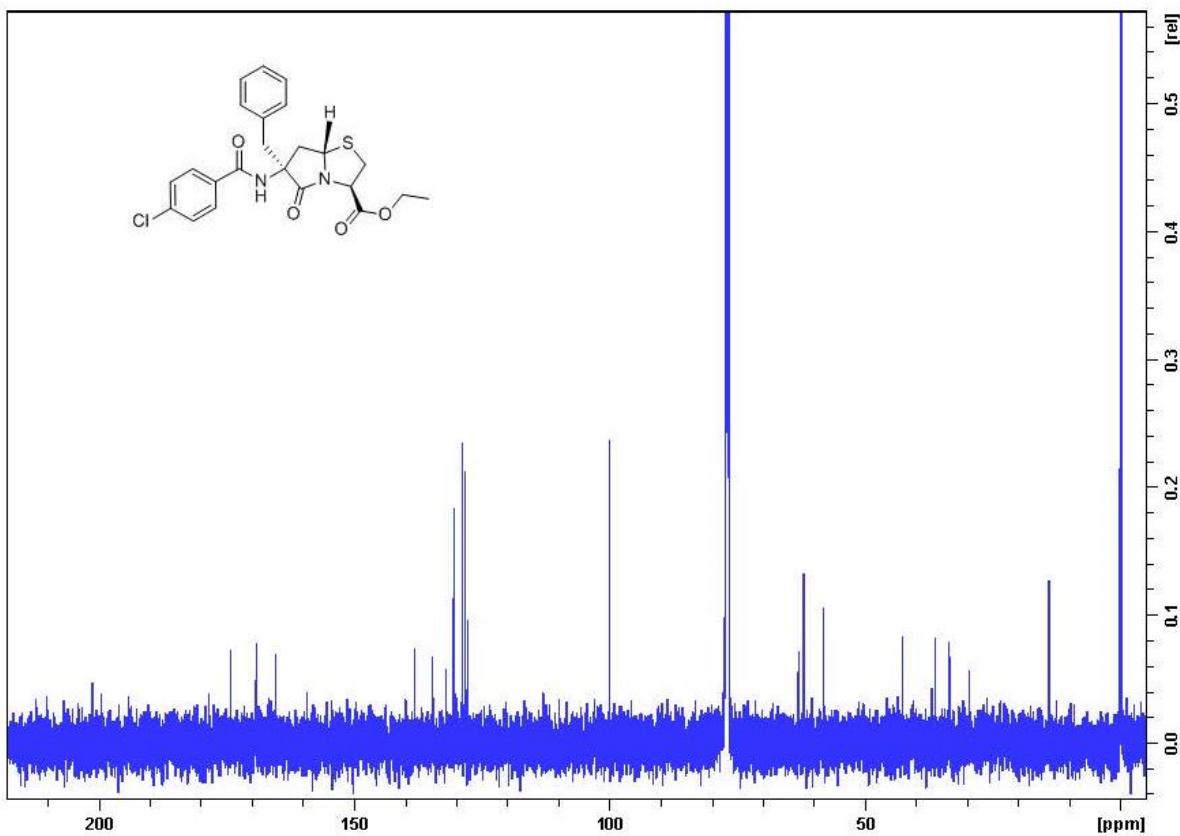


Figure S28. Carbon-13 NMR Spectrum of  $\alpha$ -33c in  $\text{CDCl}_3$

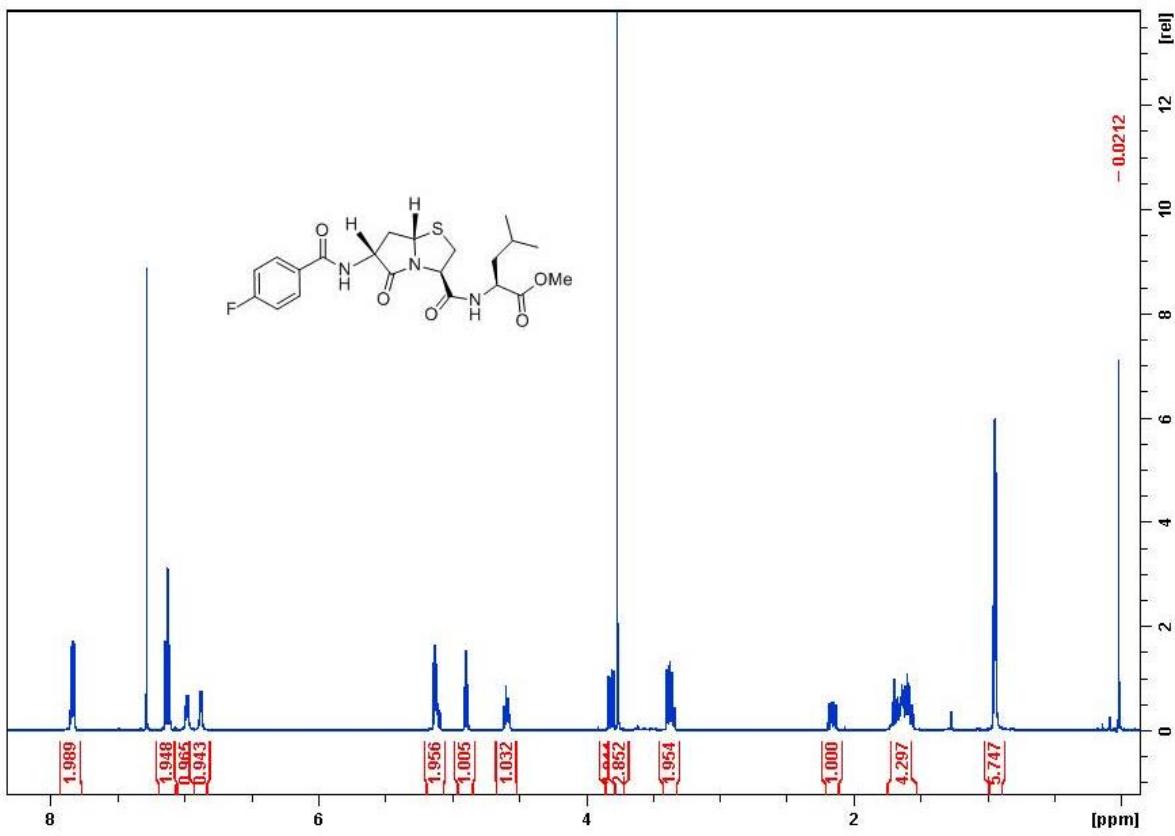
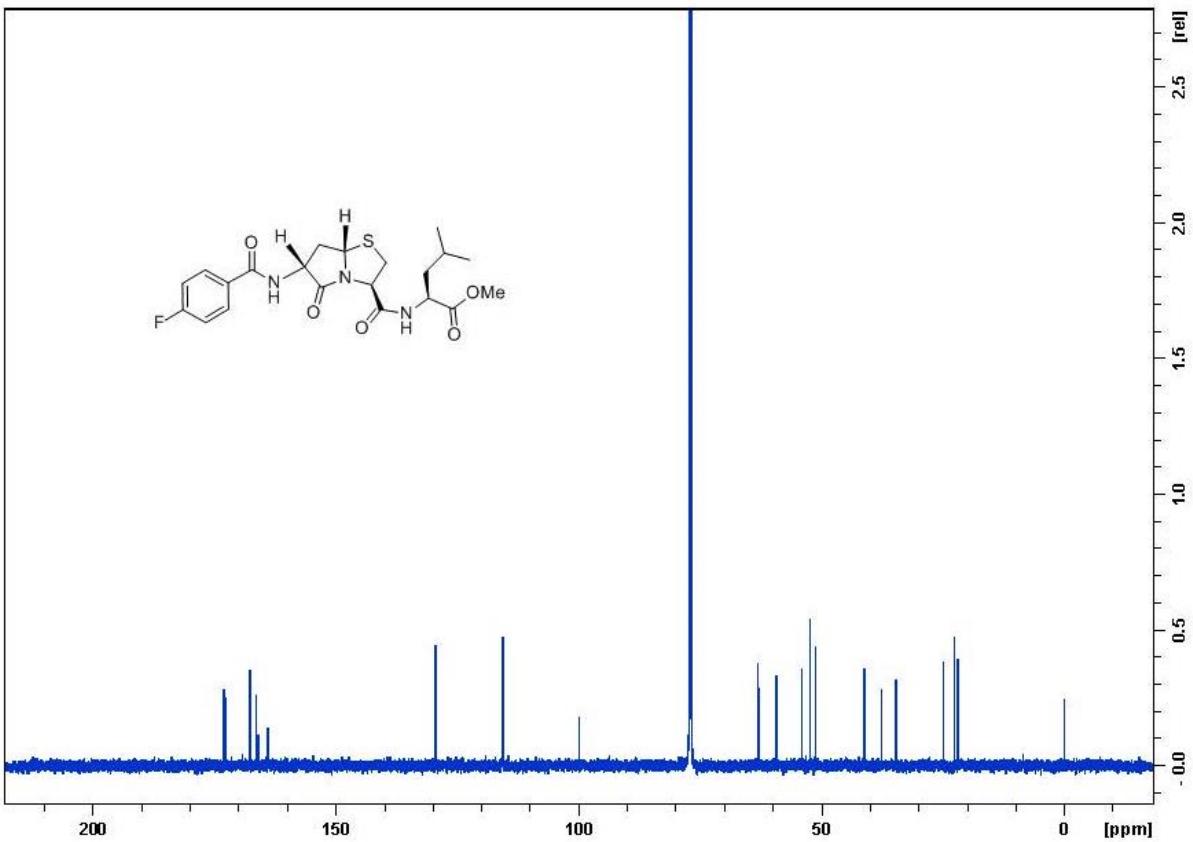


Figure S29. Proton NMR Spectrum of  $\beta$ -34a in  $\text{CDCl}_3$



**Figure S30.** Carbon-13 NMR Spectrum of  $\beta$ -34a in  $\text{CDCl}_3$

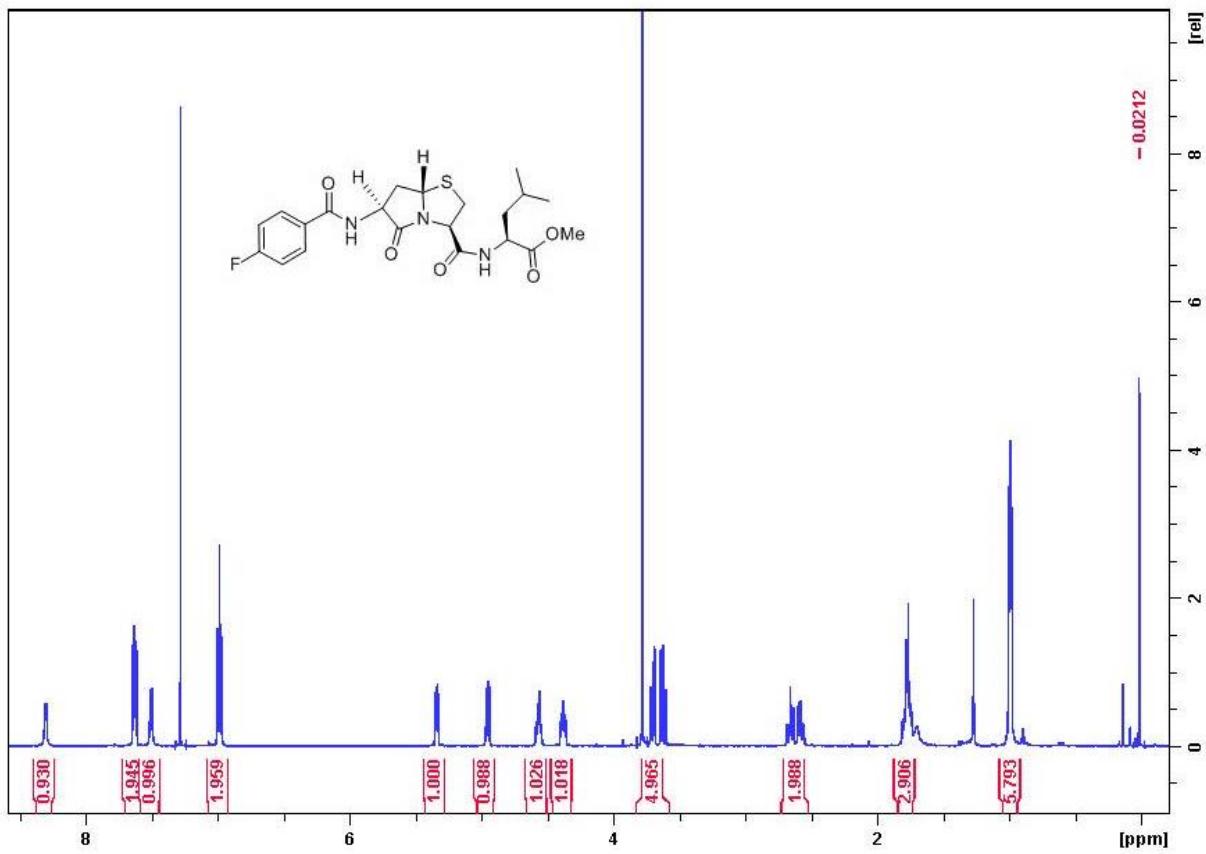
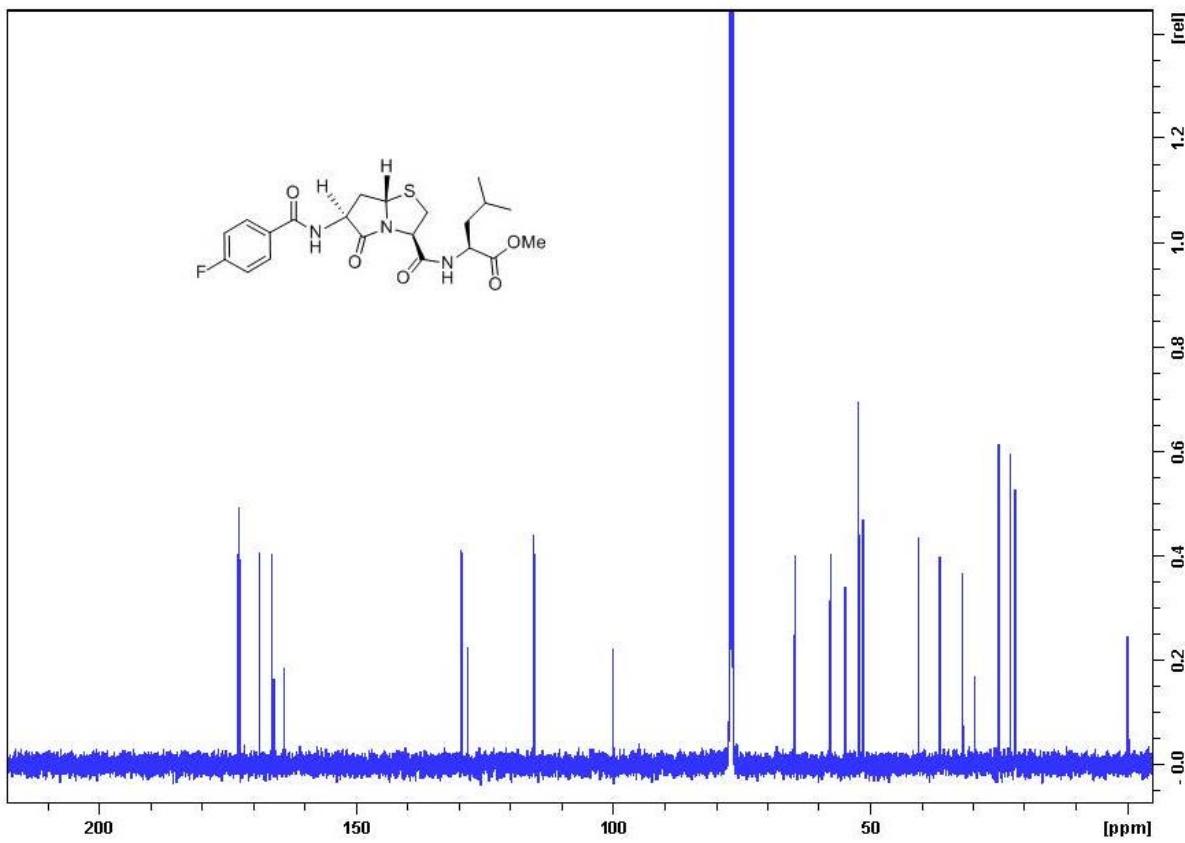


Figure S31. Proton NMR Spectrum of  $\alpha$ -34a in  $\text{CDCl}_3$



**Figure S32.** Carbon-13 NMR Spectrum of  $\alpha\text{-}34\text{a}$  in  $\text{CDCl}_3$

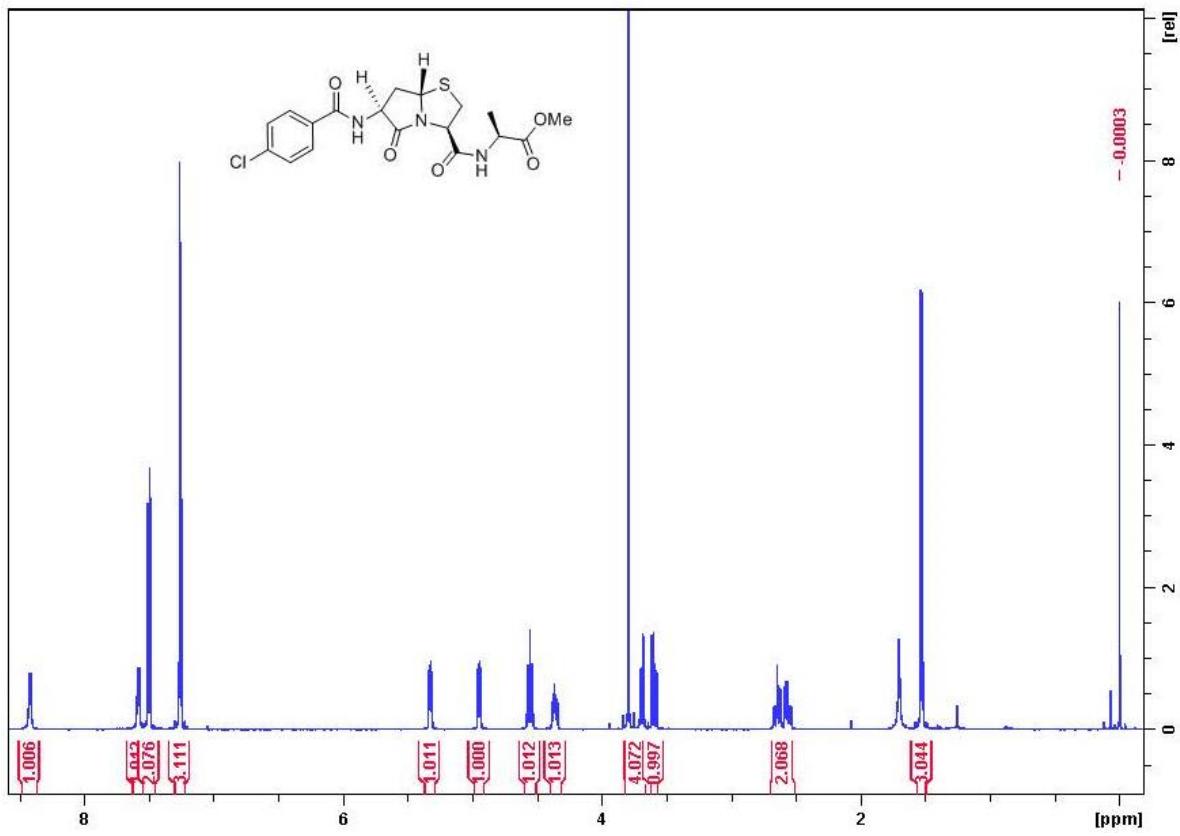


Figure S33. Proton NMR Spectrum of  $\alpha$ -35a in  $\text{CDCl}_3$

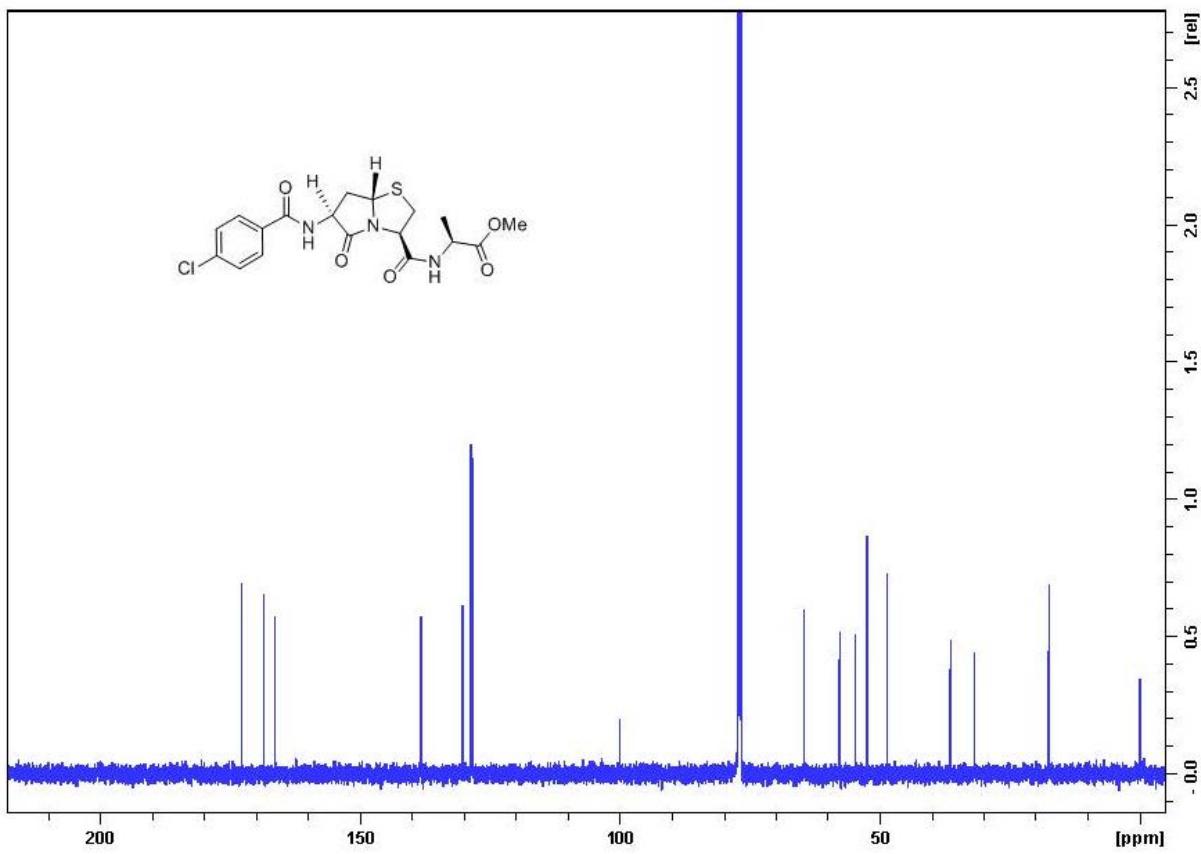


Figure S34. Carbon-13 NMR Spectrum of  $\alpha$ -35a in  $\text{CDCl}_3$

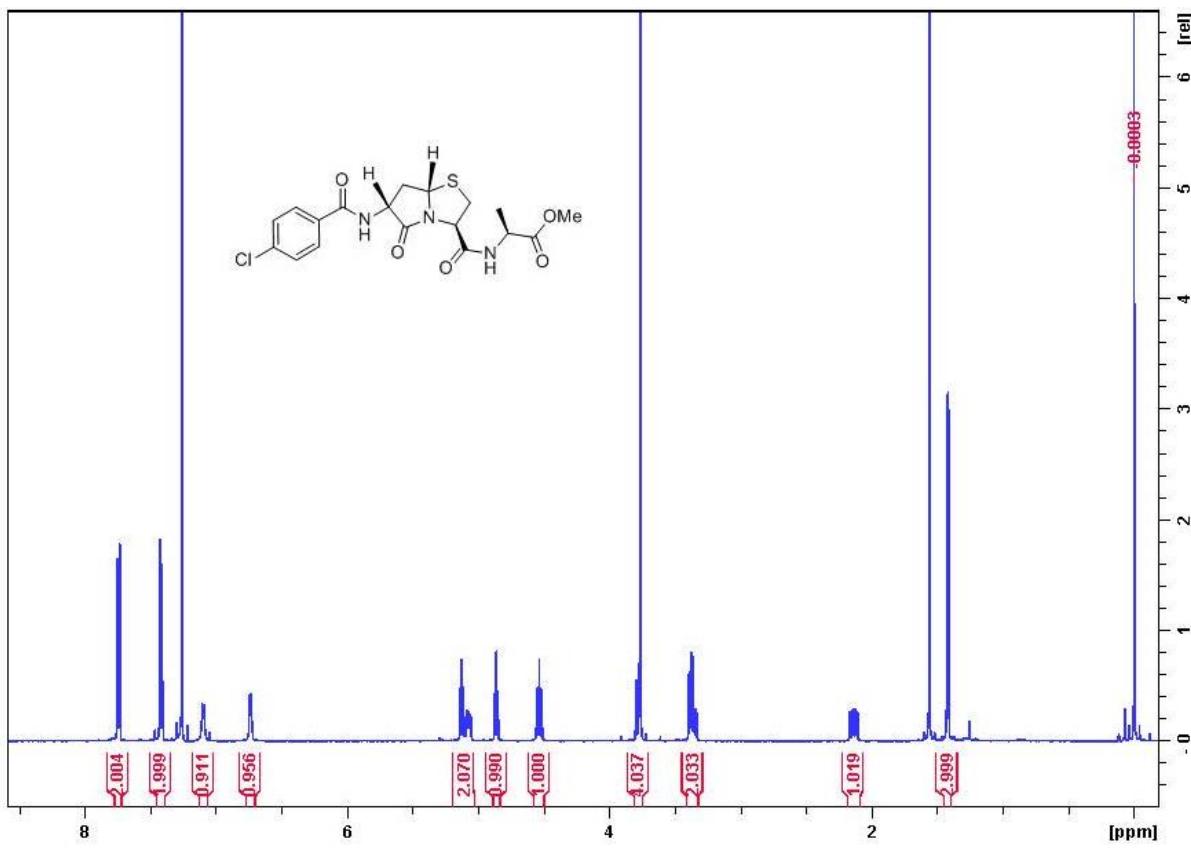
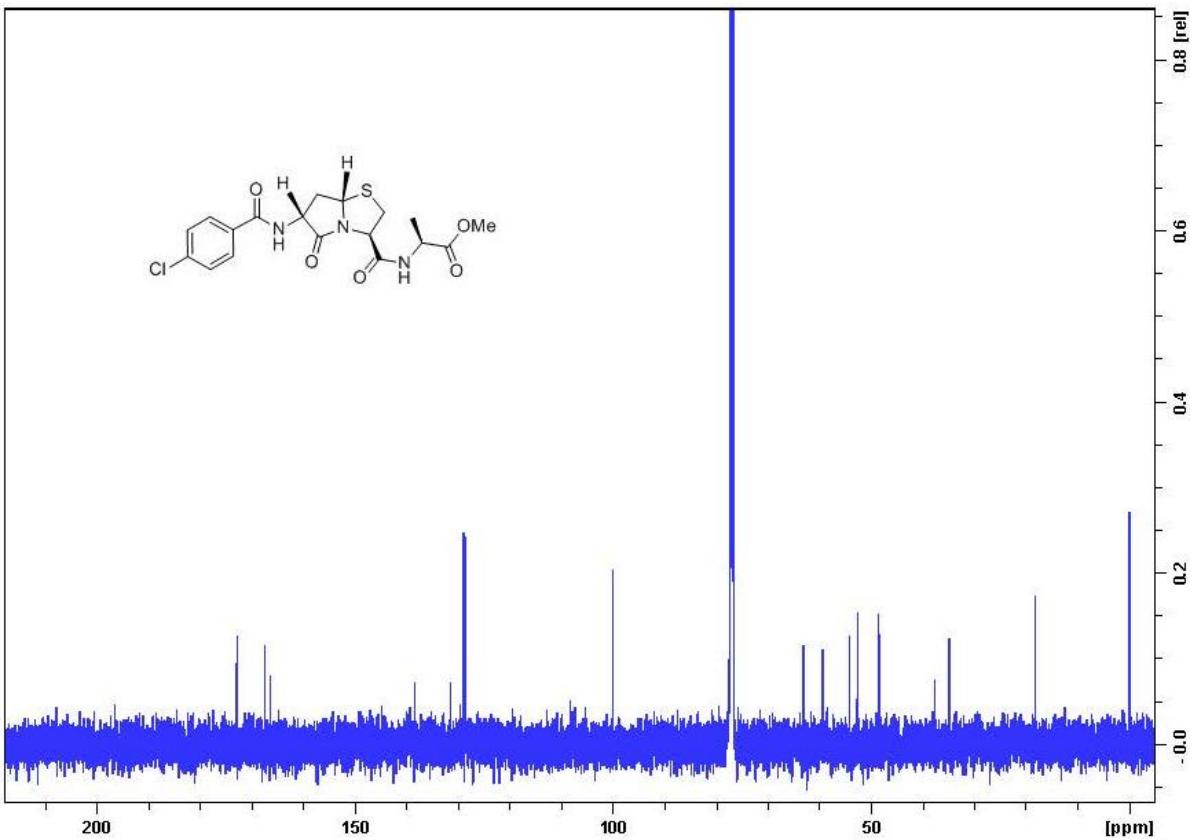


Figure S35. Proton NMR Spectrum of  $\beta$ -35a in  $\text{CDCl}_3$



**Figure S36.** Carbon-13 NMR Spectrum of  $\beta$ -35a in  $\text{CDCl}_3$

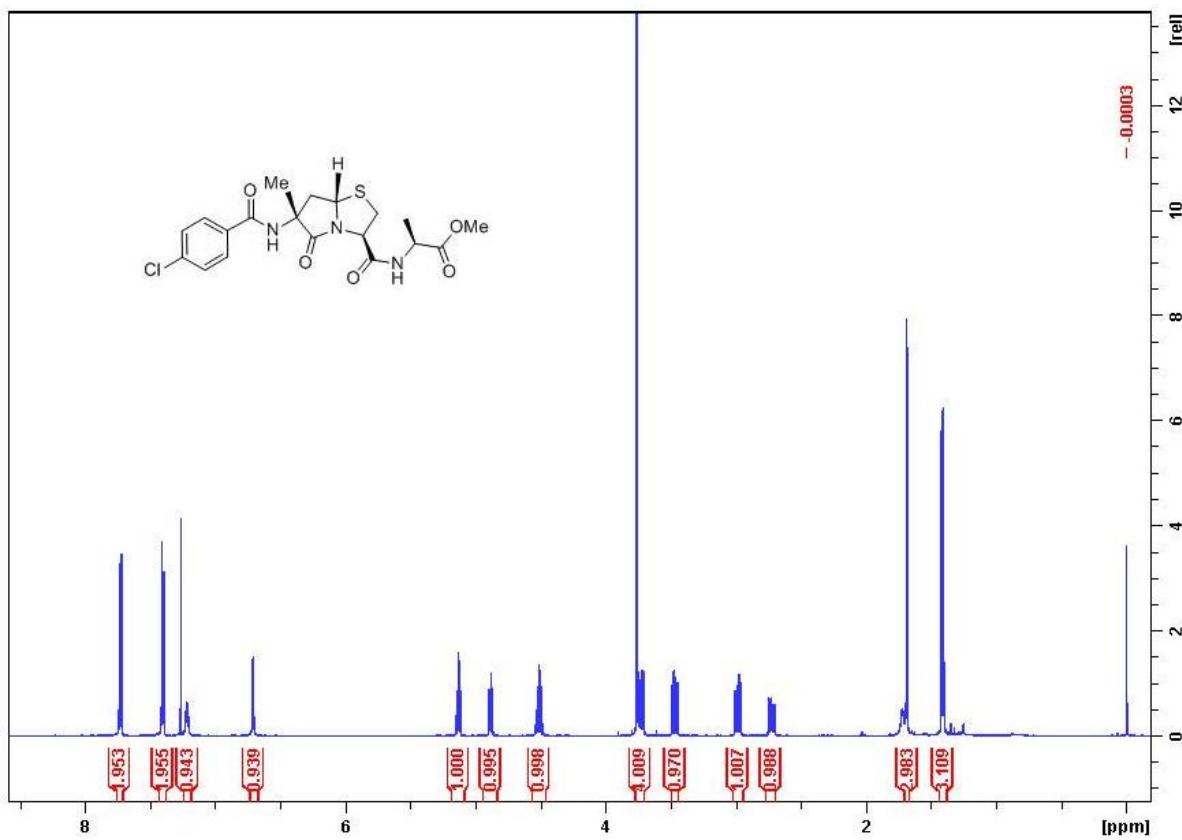


Figure S37. Proton NMR Spectrum of  $\beta$ -35b in  $\text{CDCl}_3$

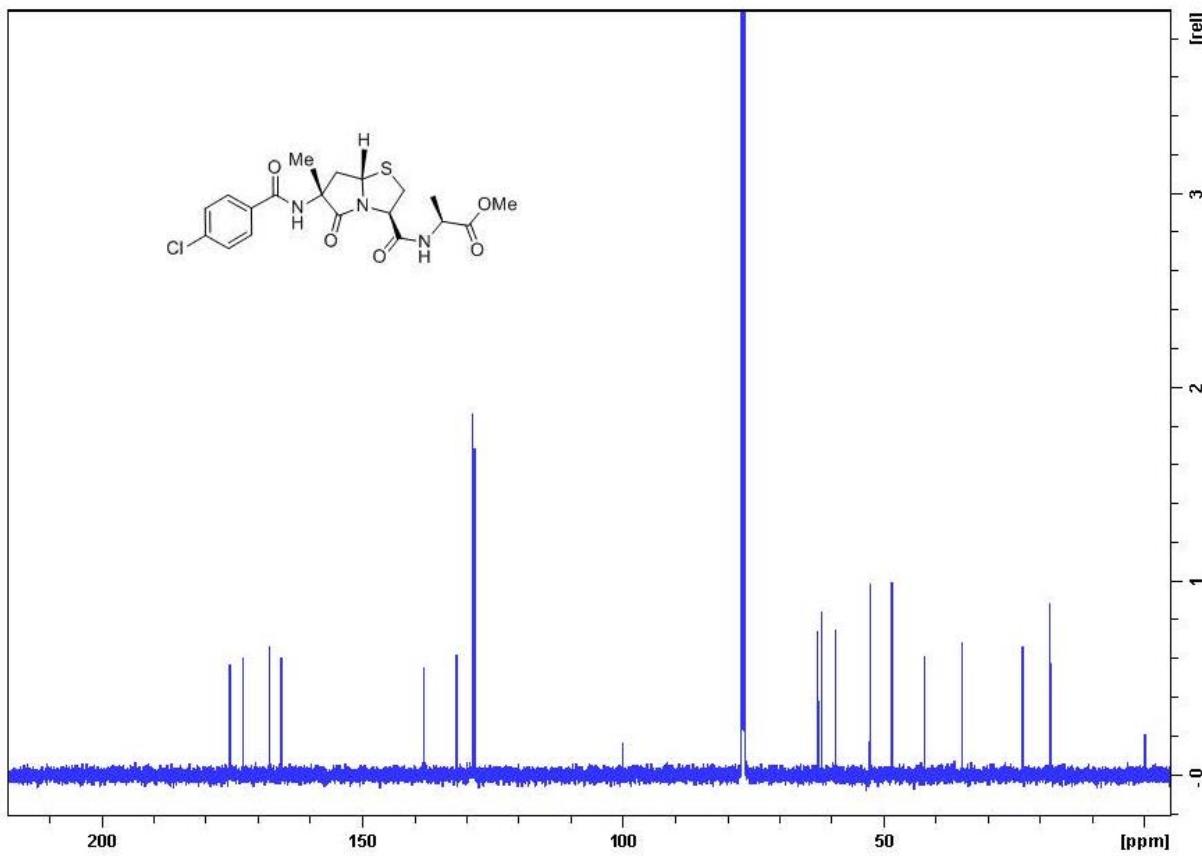


Figure S38. Carbon-13 NMR Spectrum of  $\beta\text{-}35\text{b}$  in  $\text{CDCl}_3$

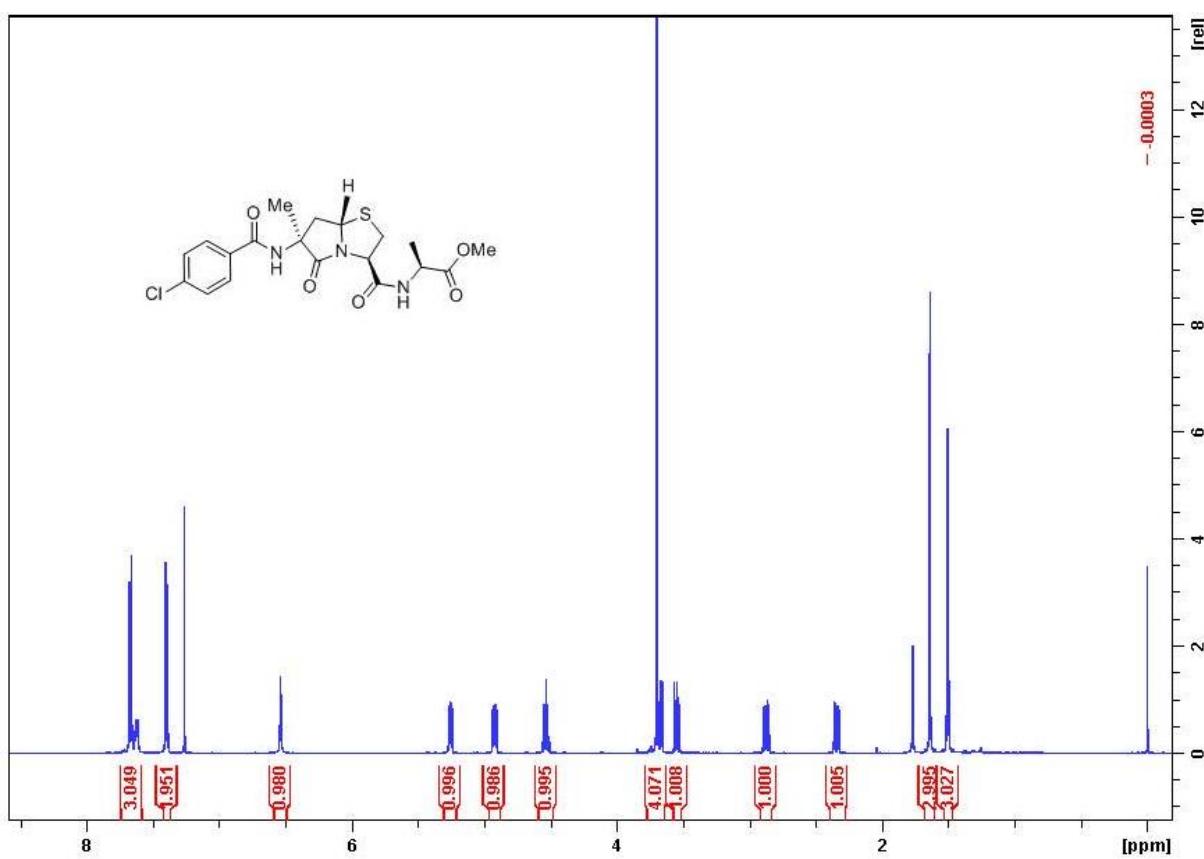


Figure S39. Proton NMR Spectrum of  $\alpha$ -35b in  $\text{CDCl}_3$

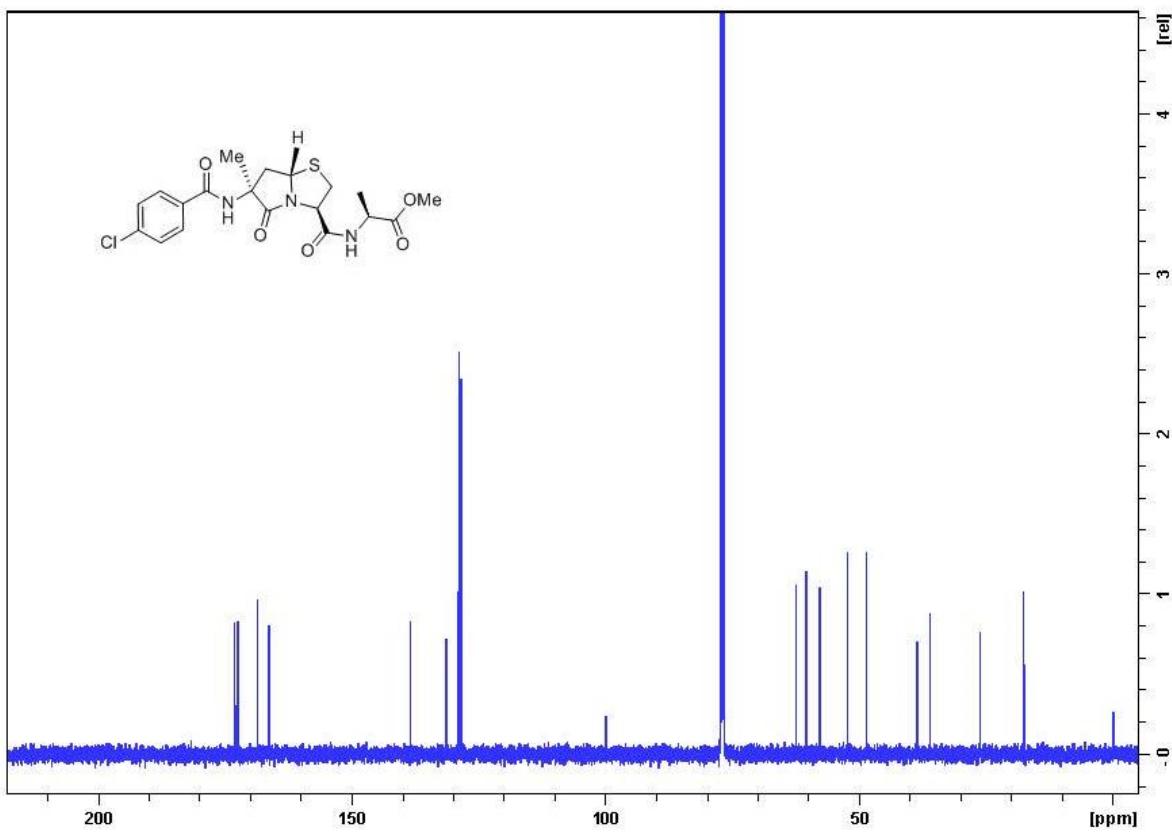
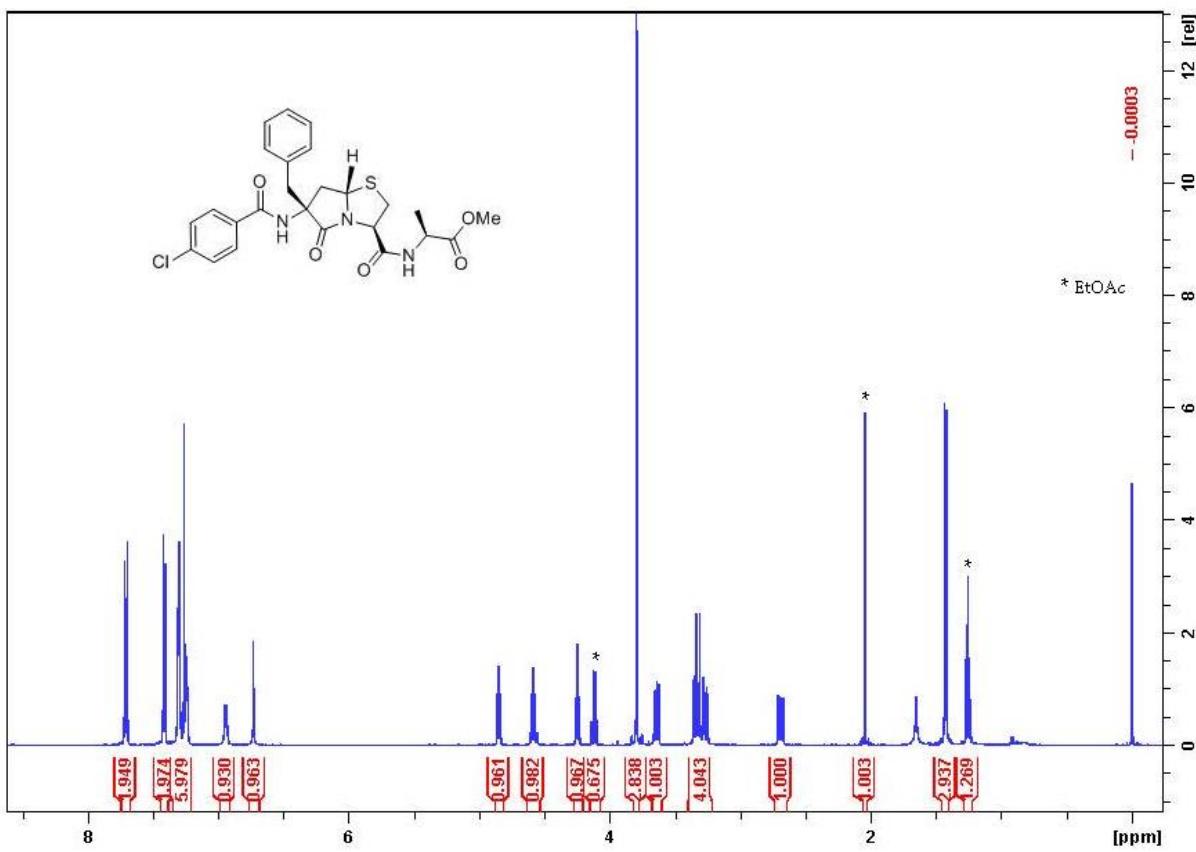
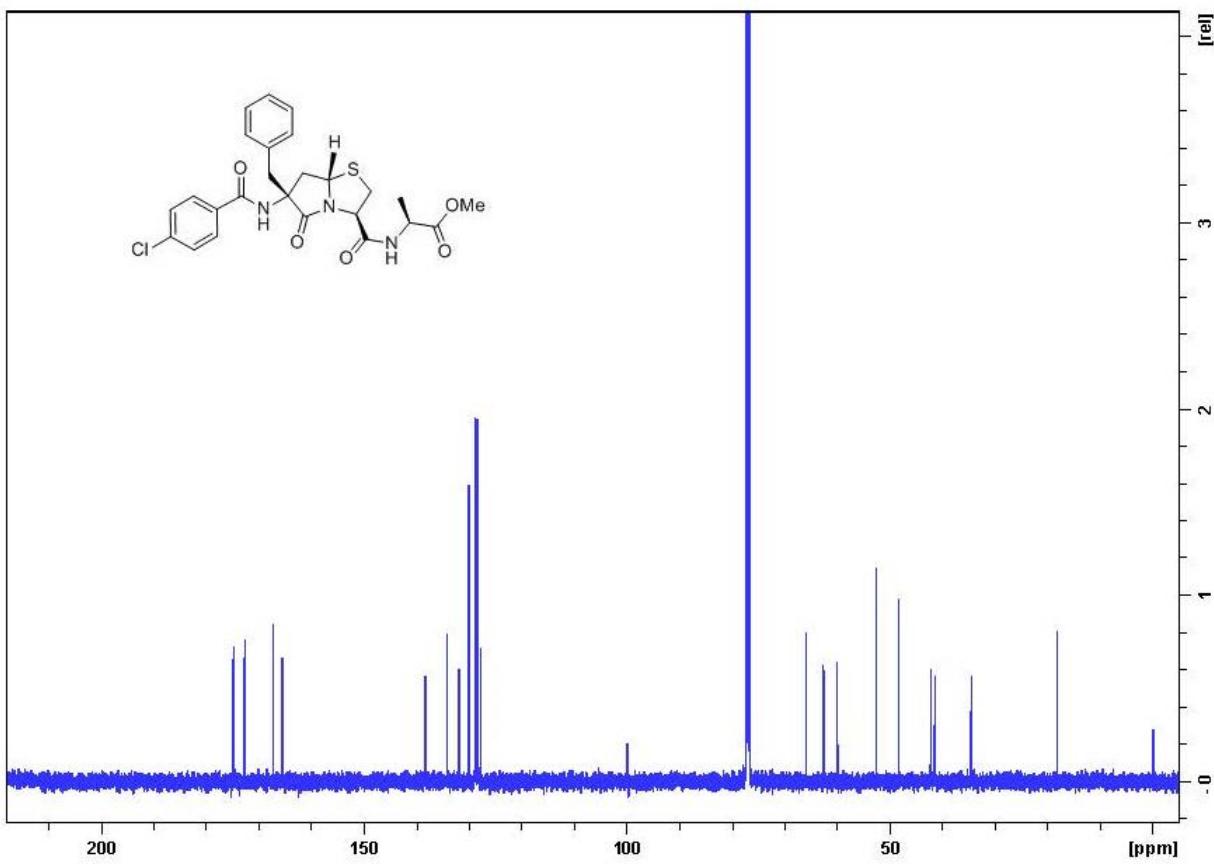


Figure S40. Carbon-13 NMR Spectrum of  $\alpha$ -35b in CDCl<sub>3</sub>



**Figure S41.** Proton NMR Spectrum of  $\beta$ -35c in  $\text{CDCl}_3$



**Figure S42.** Carbon-13 NMR Spectrum of  $\beta\text{-}35\text{c}$  in  $\text{CDCl}_3$

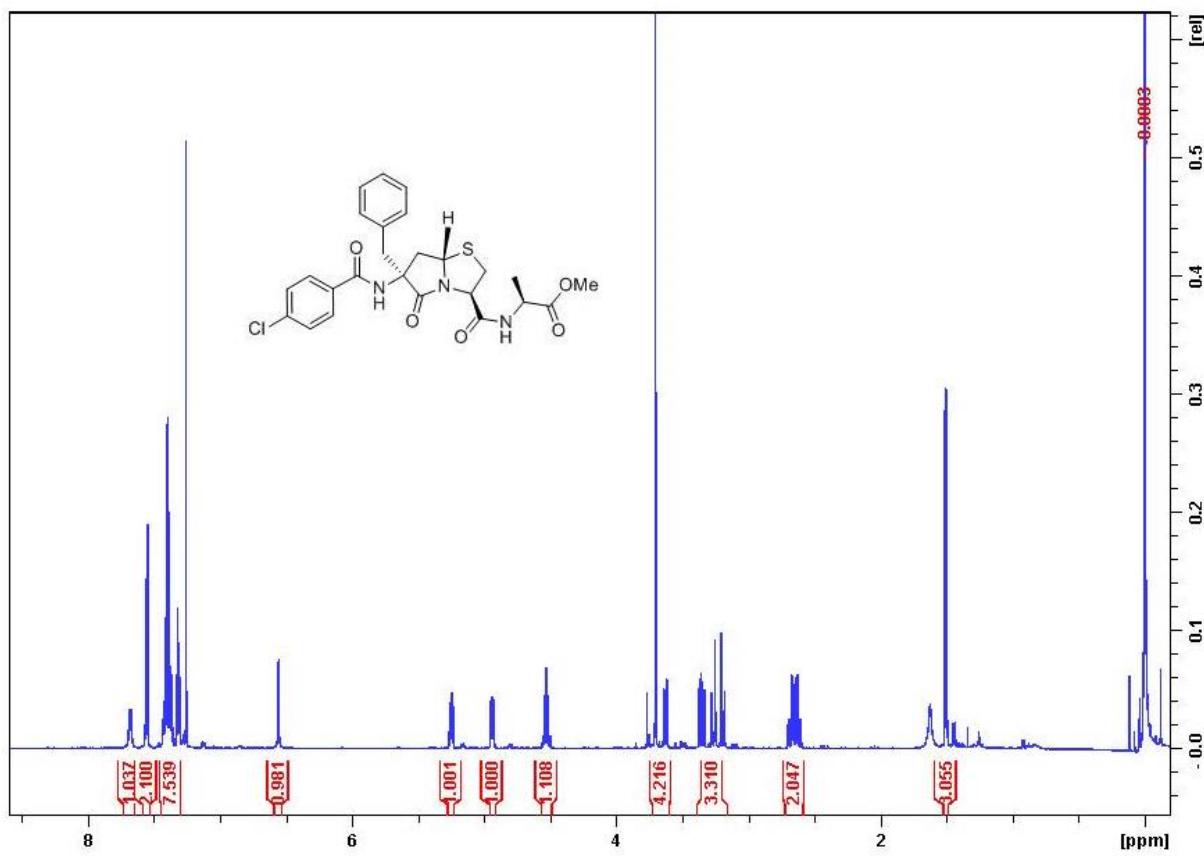
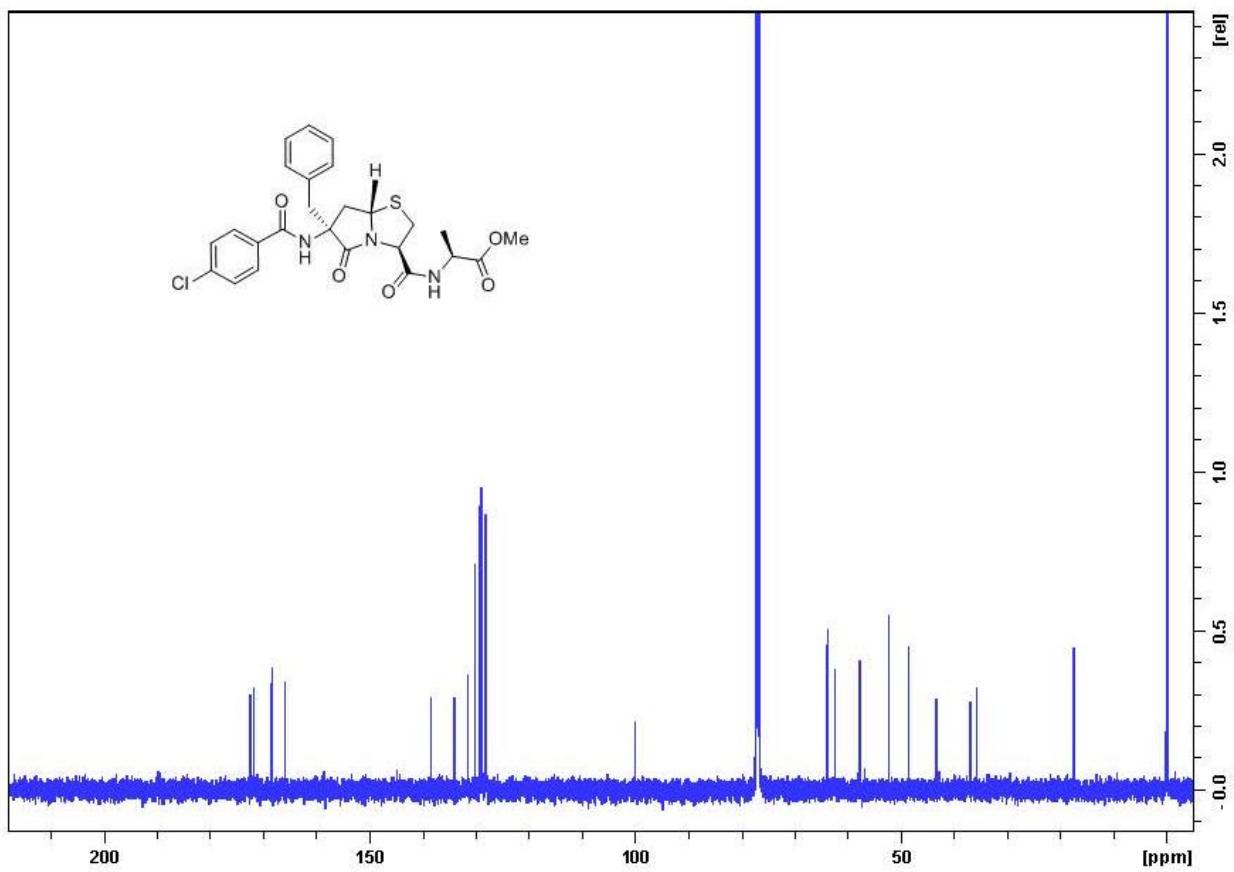
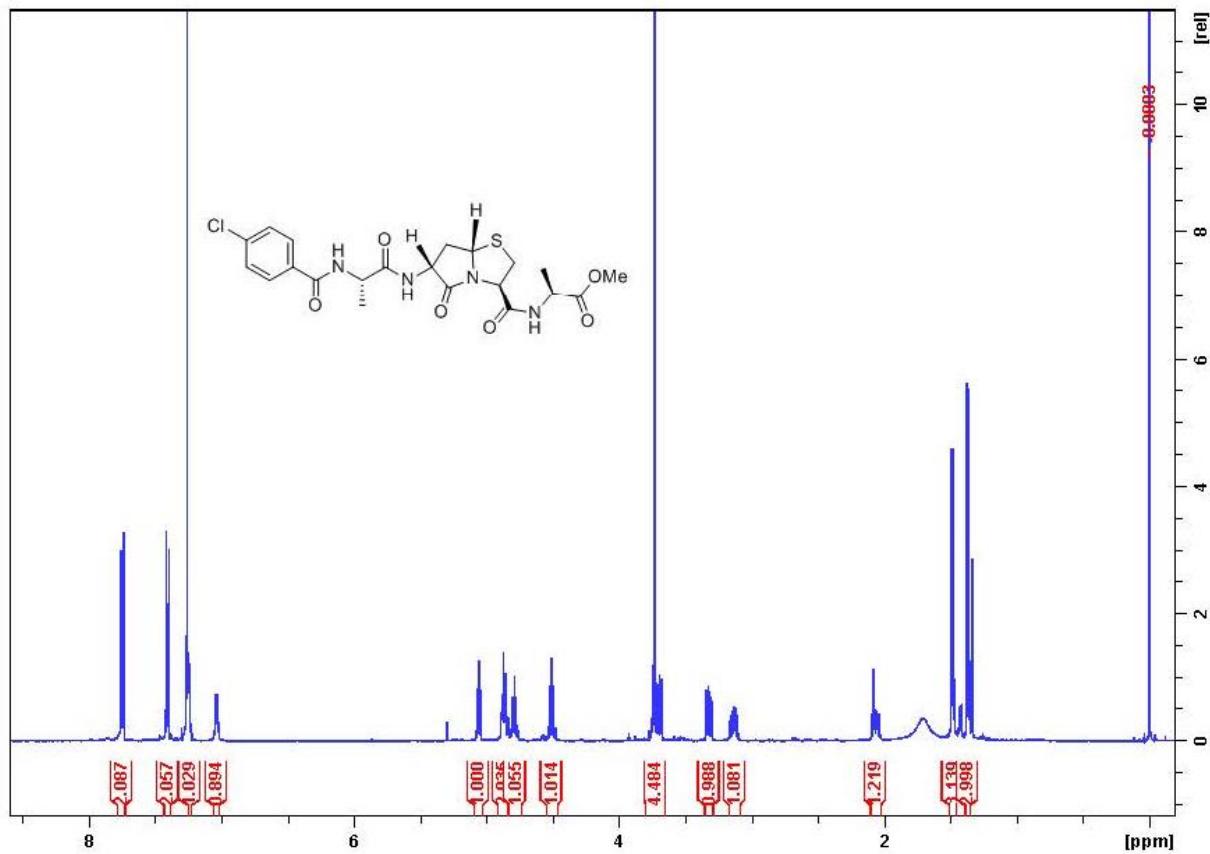


Figure S43. Proton NMR Spectrum of  $\alpha$ -35c in  $\text{CDCl}_3$



**Figure S44.** Carbon-13 NMR Spectrum of  $\alpha\text{-}35\text{c}$  in  $\text{CDCl}_3$



**Figure S45.** Proton NMR Spectrum of **β-36a** in  $\text{CDCl}_3$

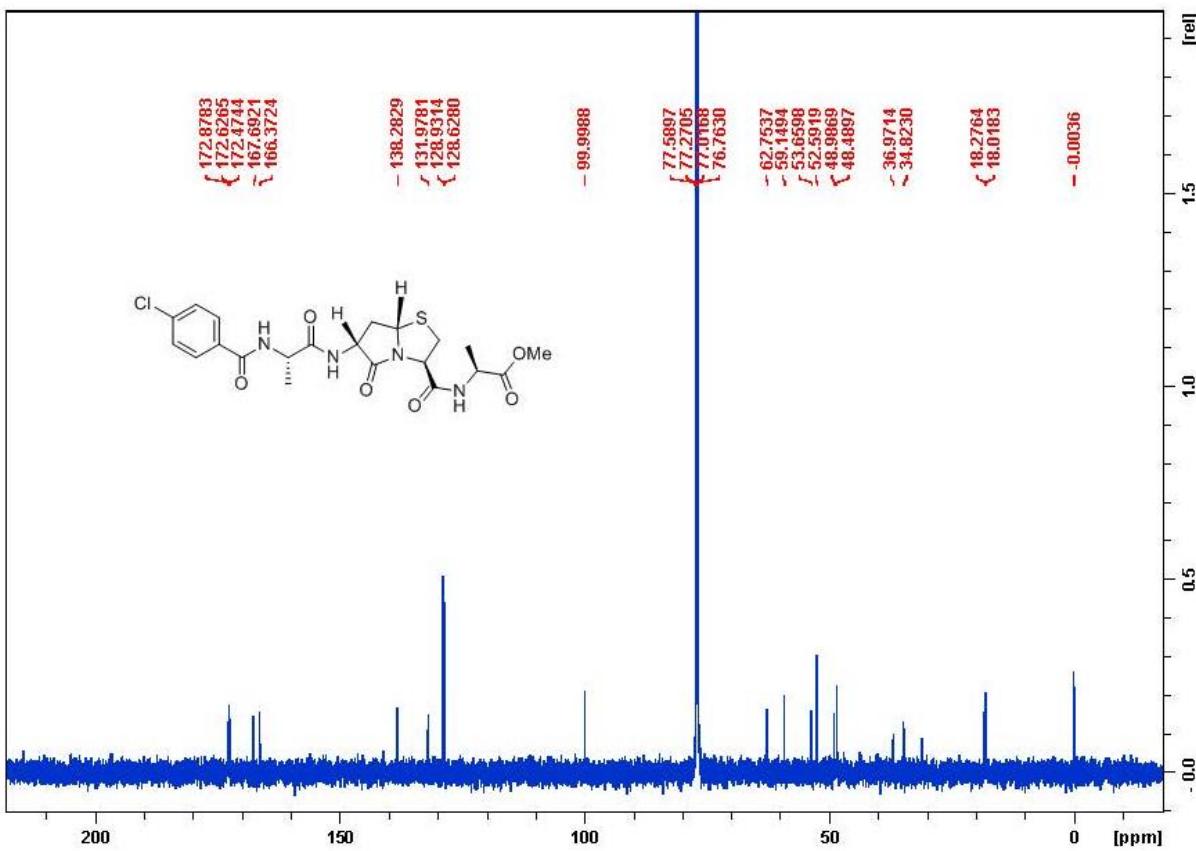


Figure S46. Carbon-13 NMR Spectrum of  $\beta$ -36a in  $\text{CDCl}_3$

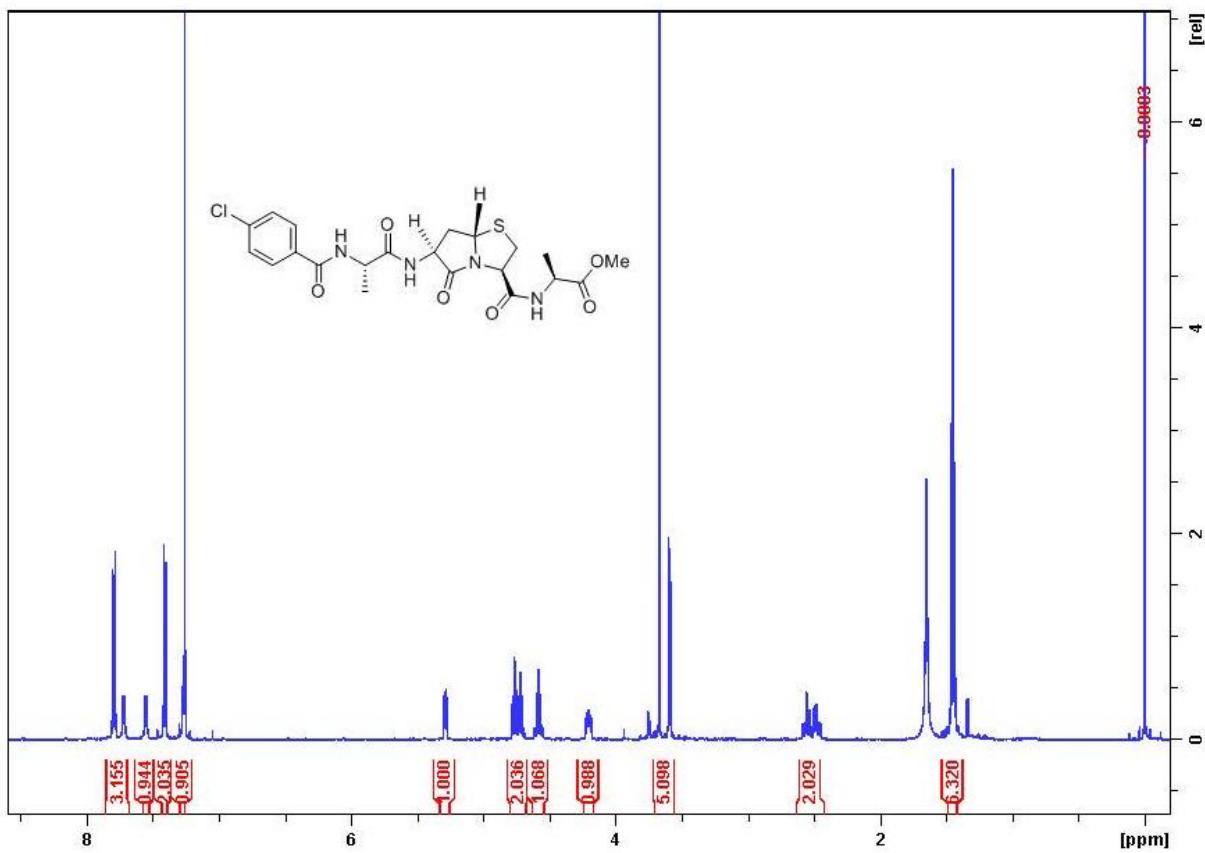


Figure S47. Proton NMR Spectrum of  $\alpha$ -36a in  $\text{CDCl}_3$

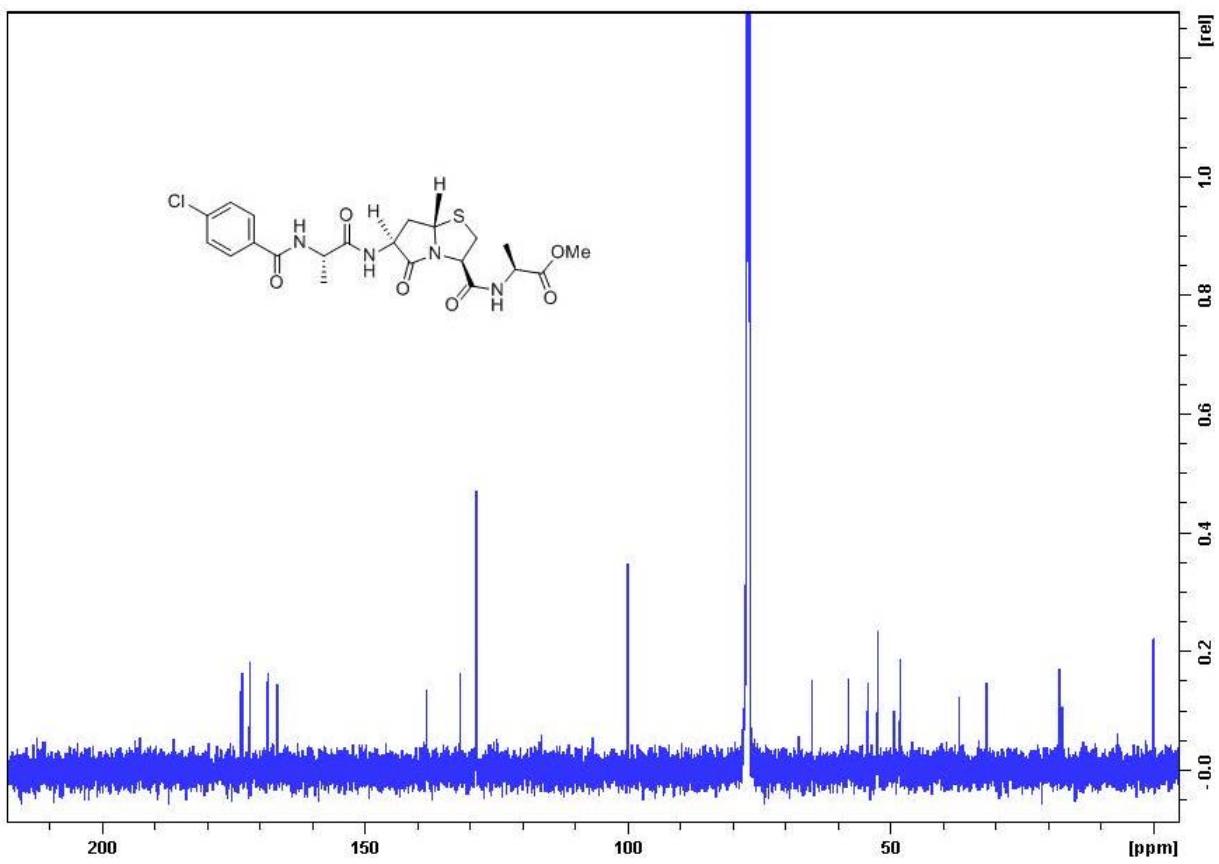


Figure S48. Carbon-13 NMR Spectrum of  $\alpha$ -36a in CDCl<sub>3</sub>

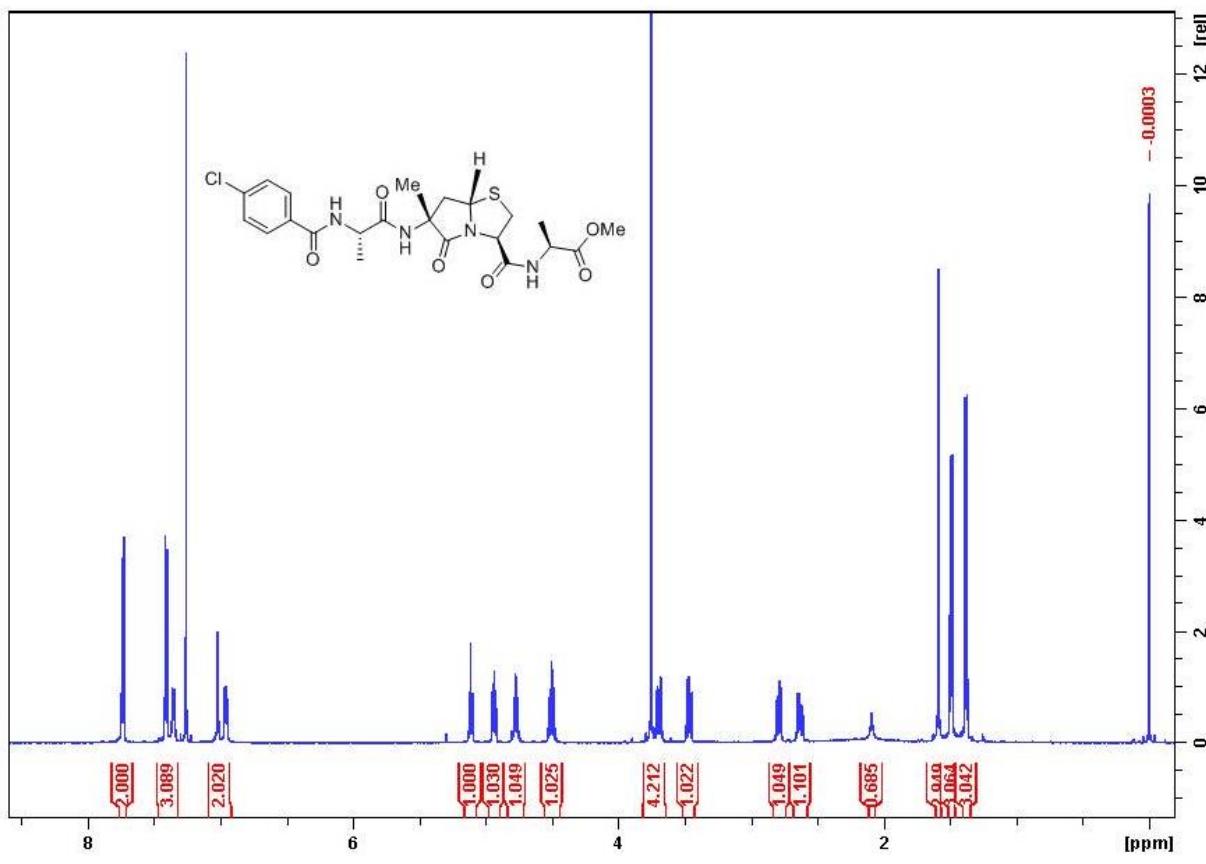


Figure S49. Proton NMR Spectrum of  $\beta$ -36b in  $\text{CDCl}_3$

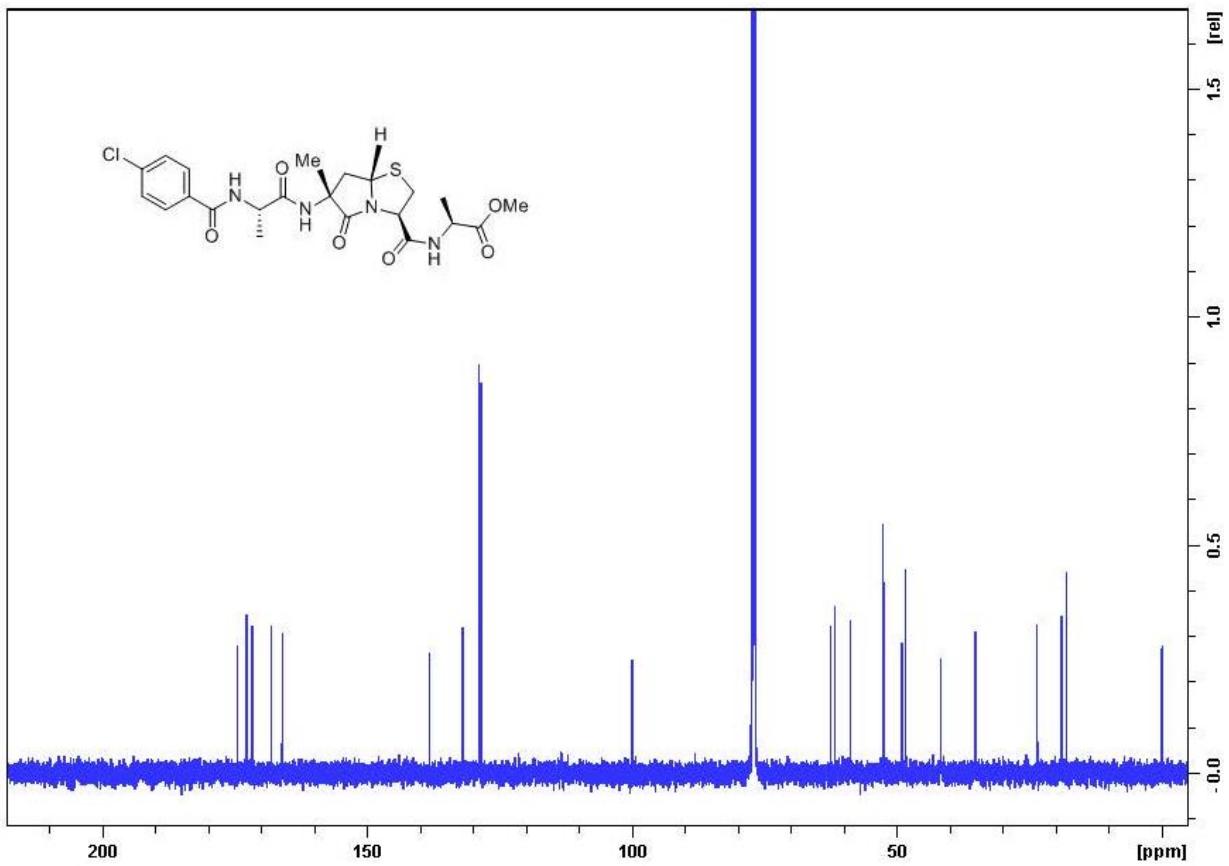


Figure S50. Carbon-13 NMR Spectrum of  $\beta$ -36b in  $\text{CDCl}_3$

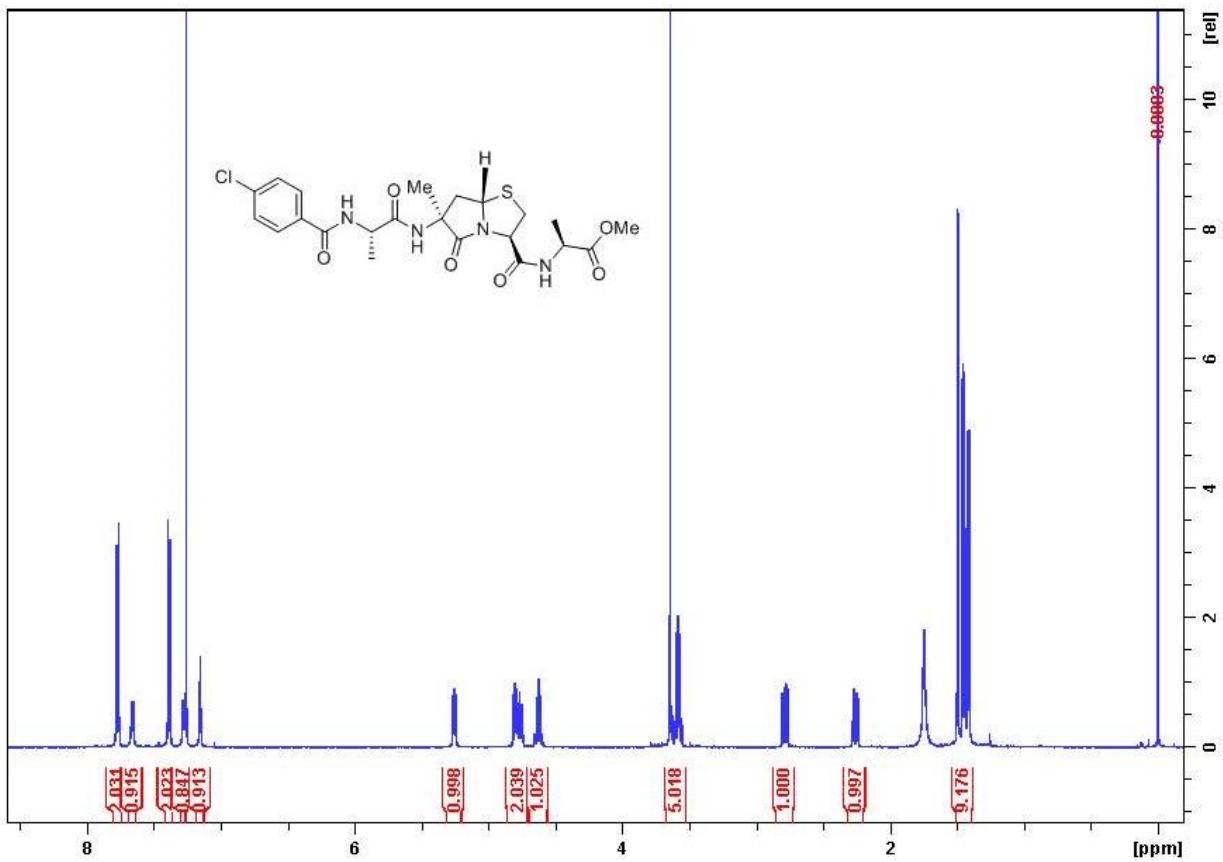


Figure S51. Proton NMR Spectrum of  $\alpha$ -36b in  $\text{CDCl}_3$

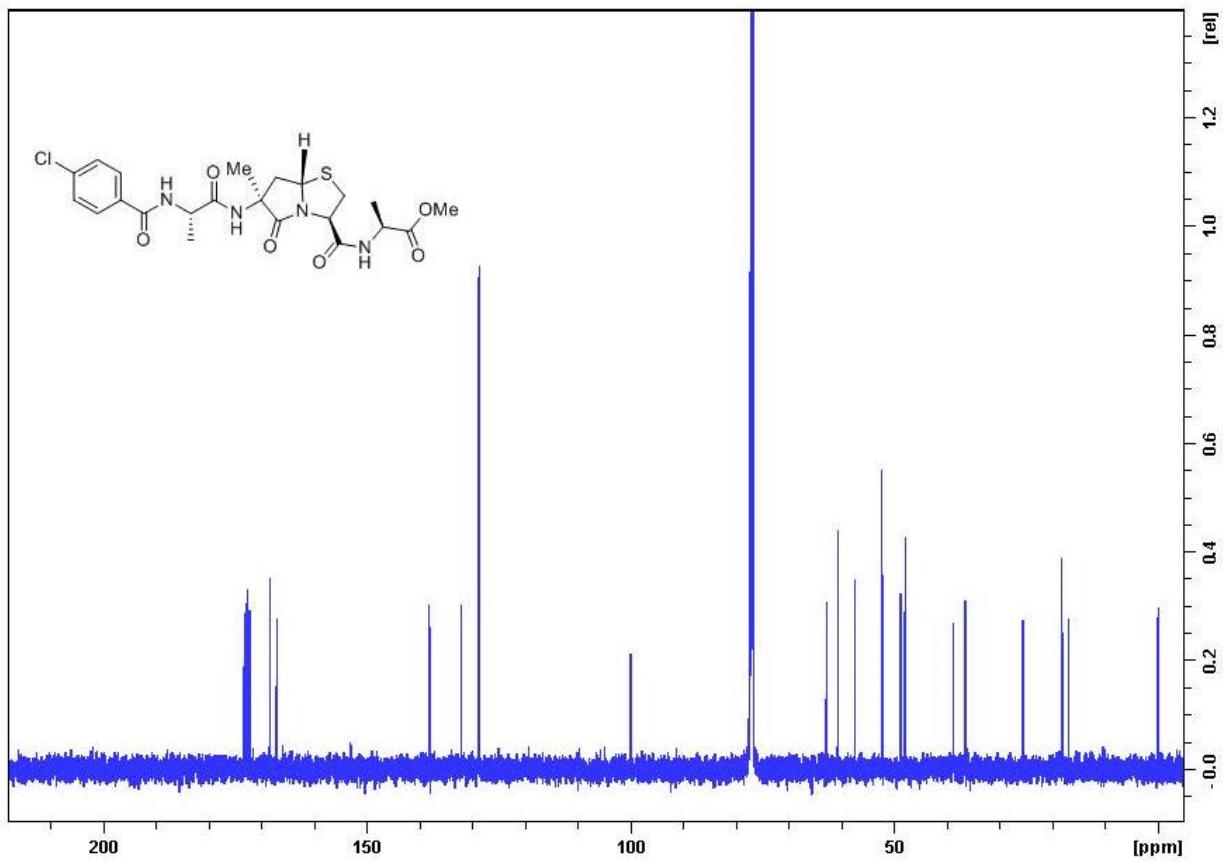


Figure S52. Carbon-13 NMR Spectrum of  $\alpha\text{-}36\text{b}$  in  $\text{CDCl}_3$

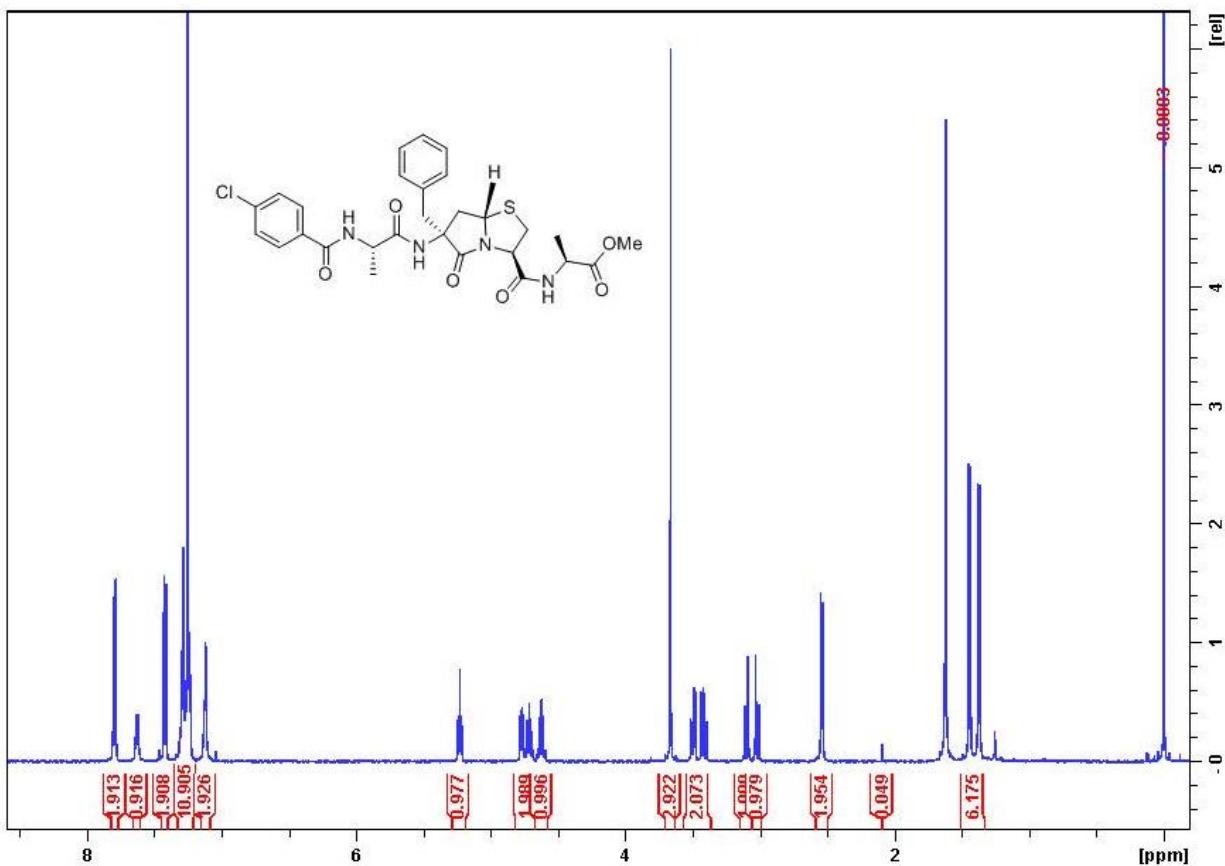


Figure S53. Proton NMR Spectrum of  $\alpha$ -36c in  $\text{CDCl}_3$

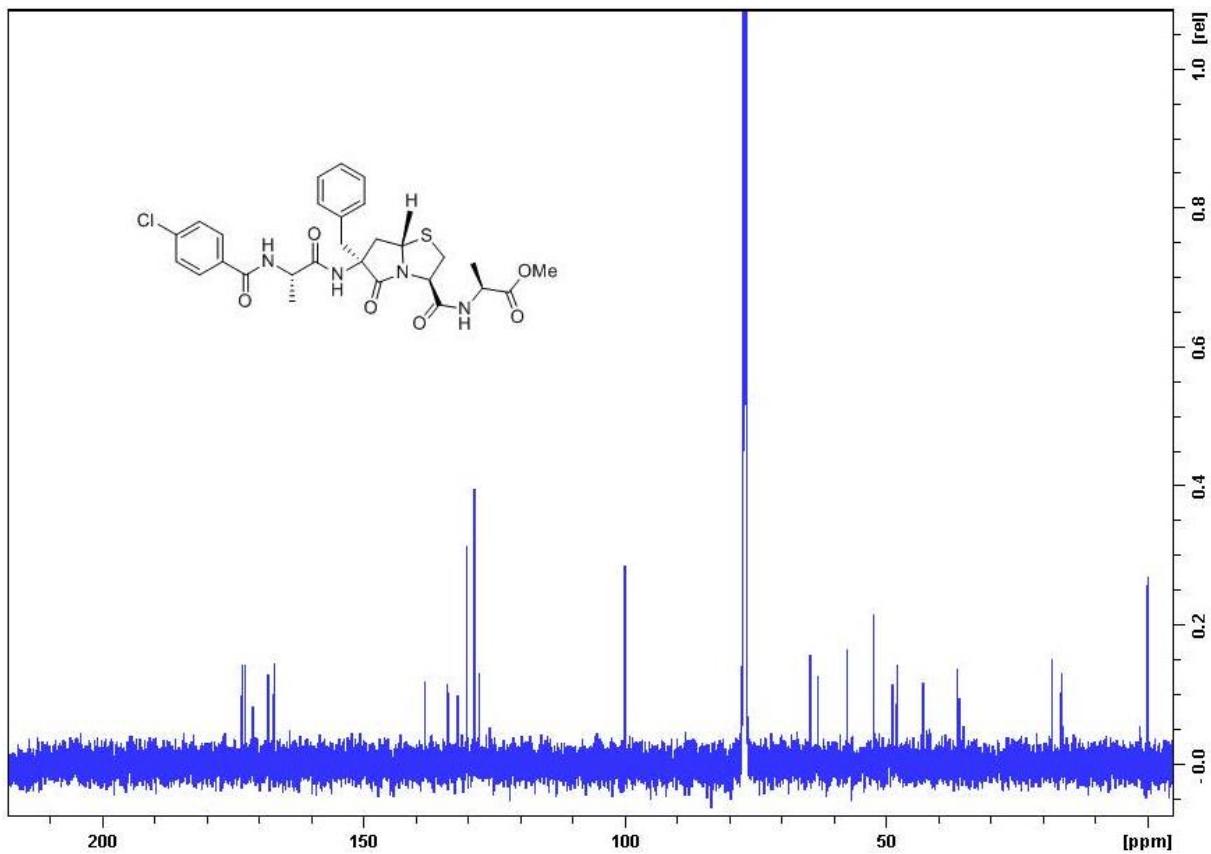


Figure S54. Carbon-13 NMR Spectrum of  $\alpha$ -36c in  $\text{CDCl}_3$

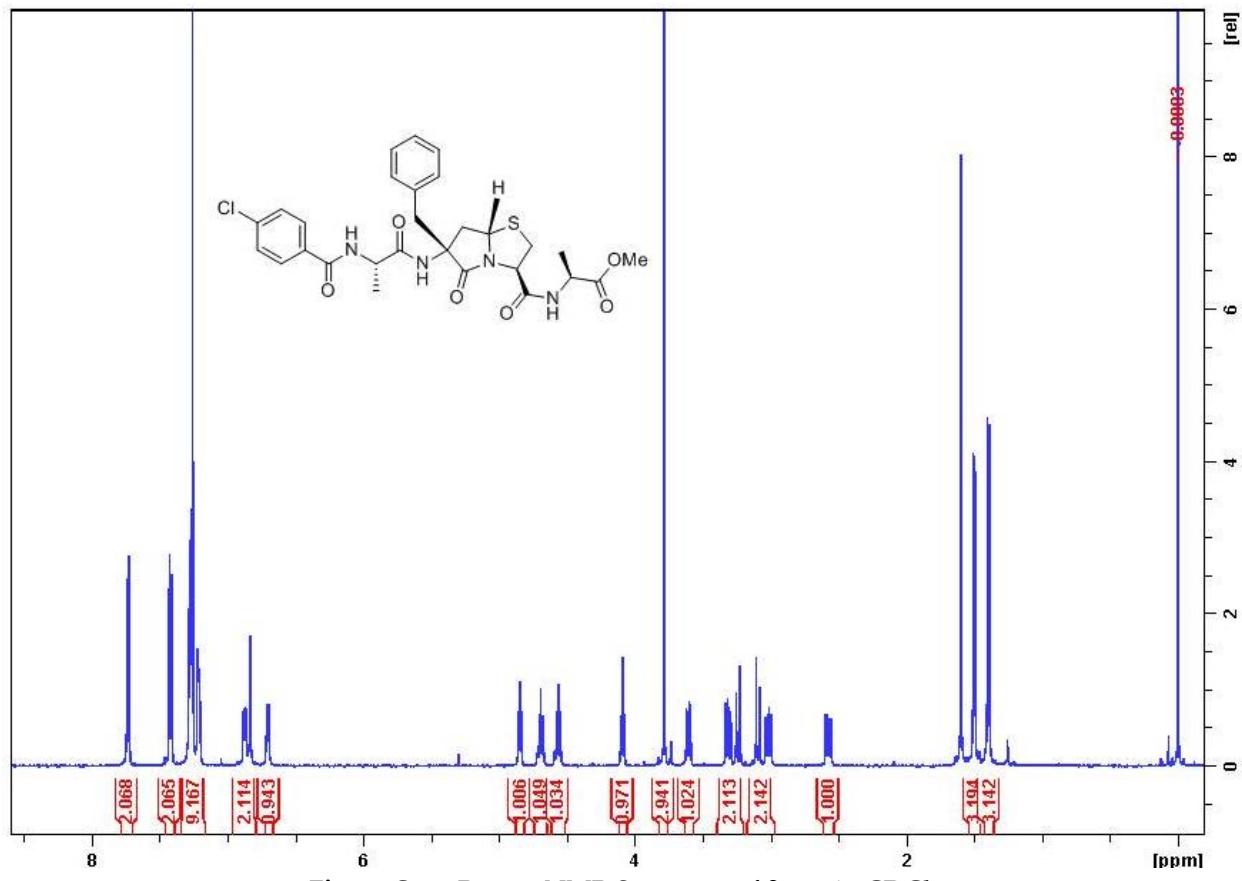


Figure S55. Proton NMR Spectrum of  $\beta$ -36c in  $\text{CDCl}_3$

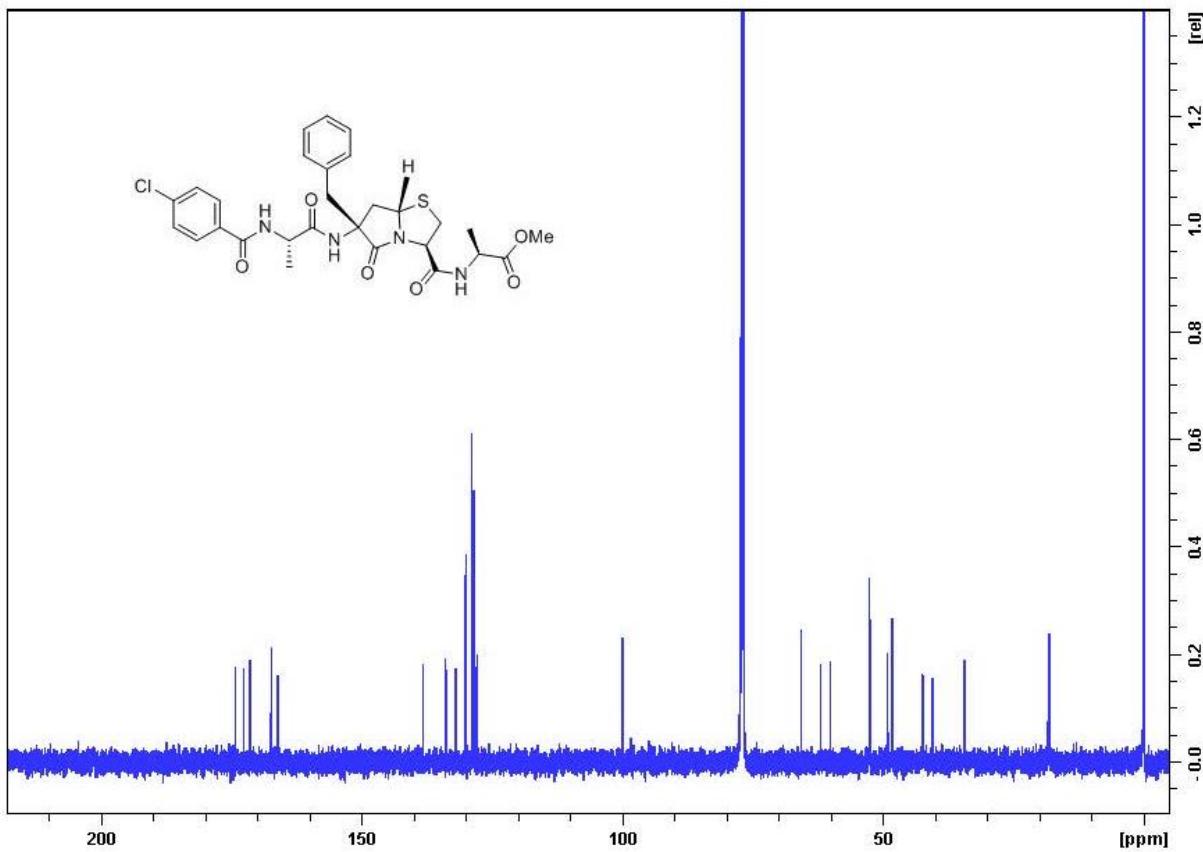


Figure S56. Carbon-13 NMR Spectrum of  $\beta$ -36c in  $\text{CDCl}_3$

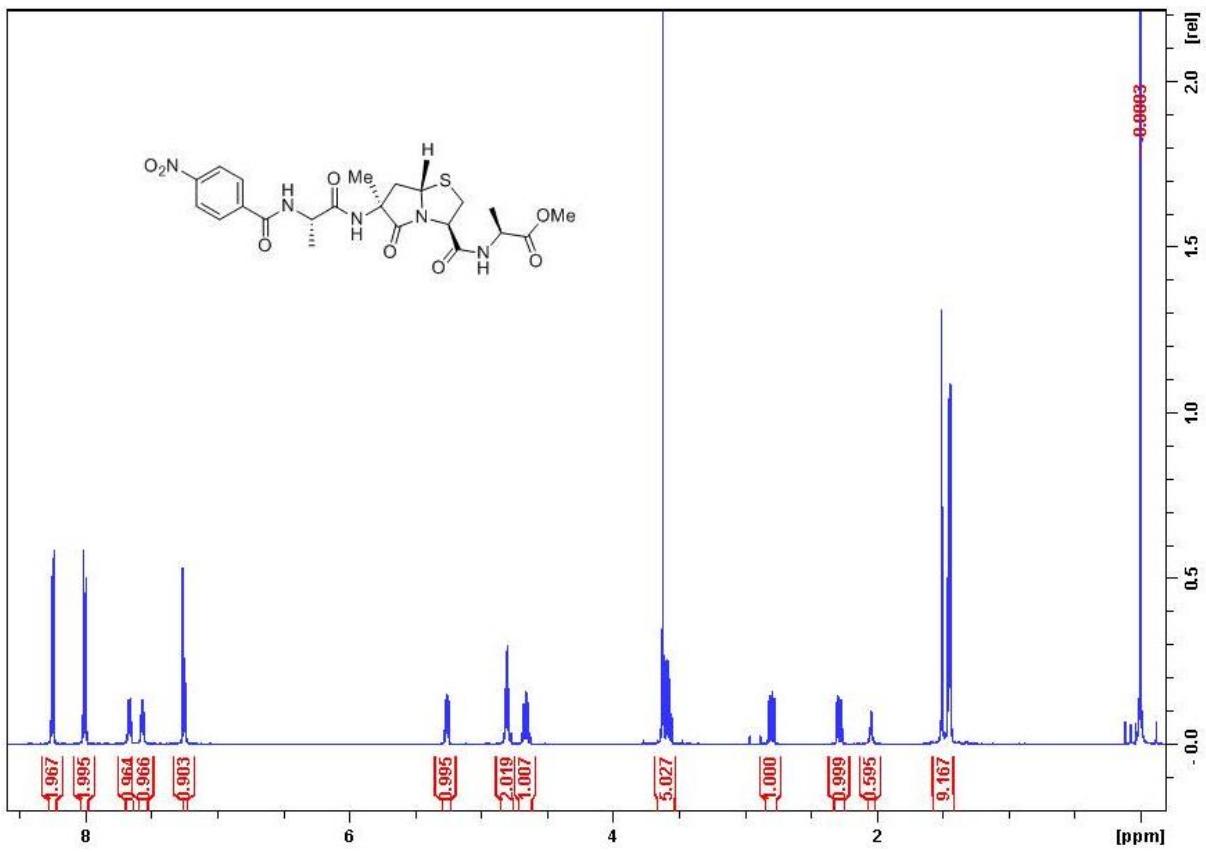


Figure S57. Proton NMR Spectrum of  $\alpha$ -30b in  $\text{CDCl}_3$

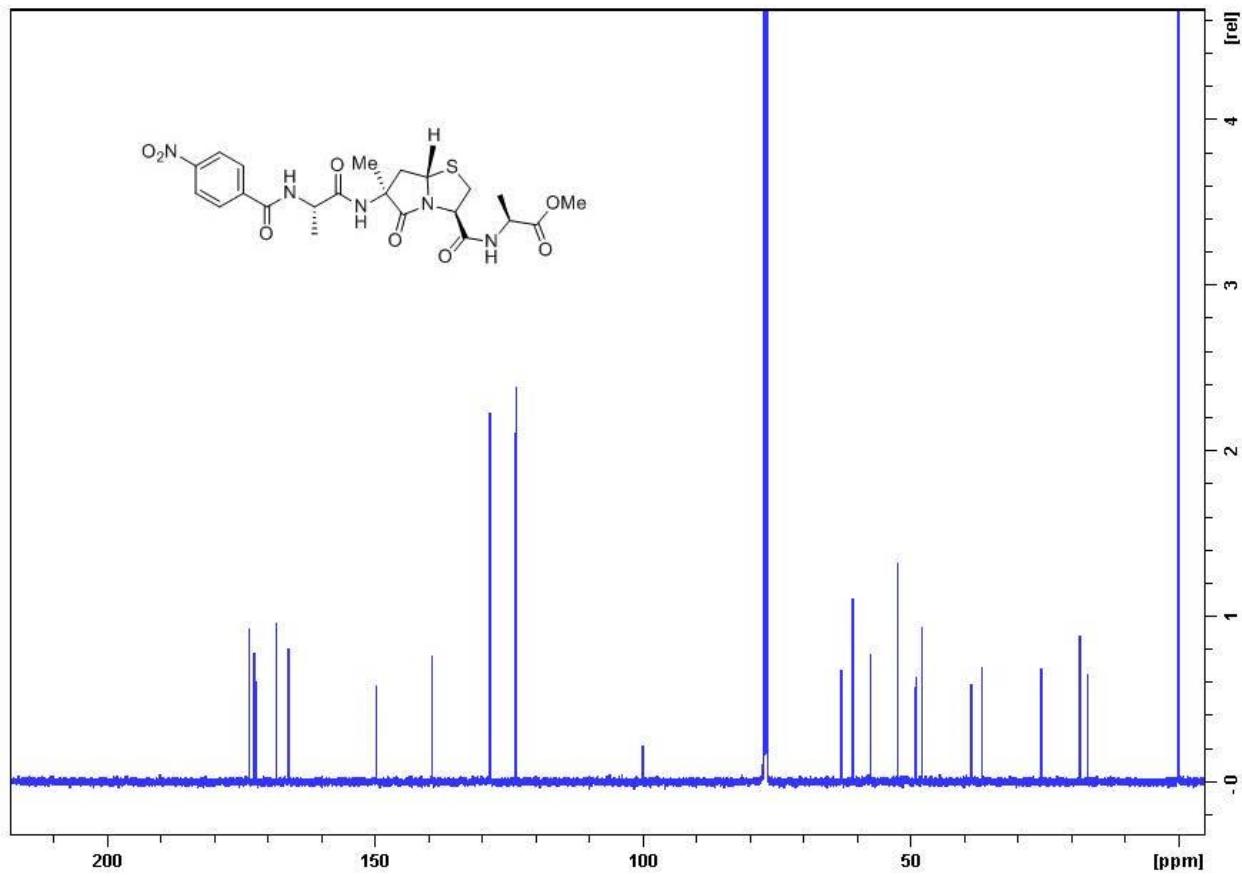


Figure S58. Carbon-13 NMR Spectrum of  $\alpha\text{-}30\text{b}$  in  $\text{CDCl}_3$

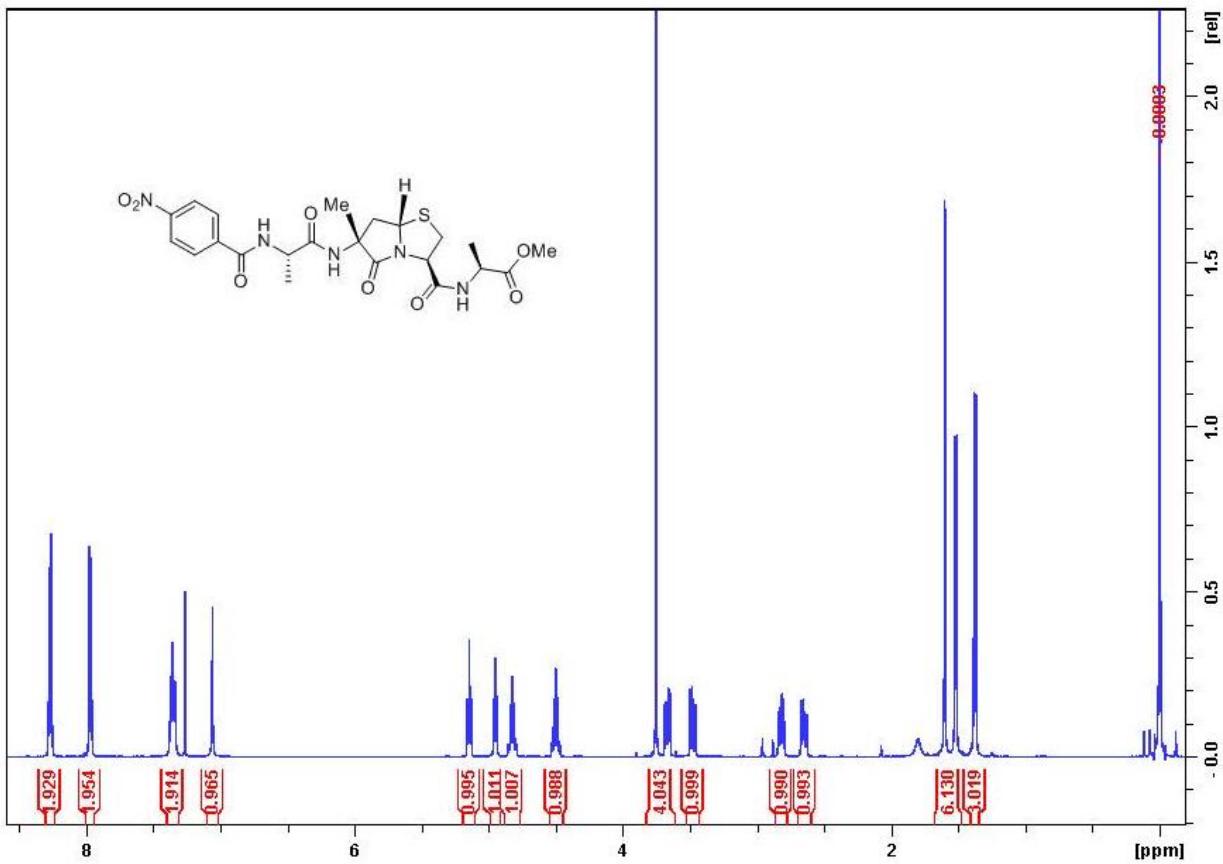


Figure S59. Proton NMR Spectrum of  $\beta$ -30b in  $\text{CDCl}_3$

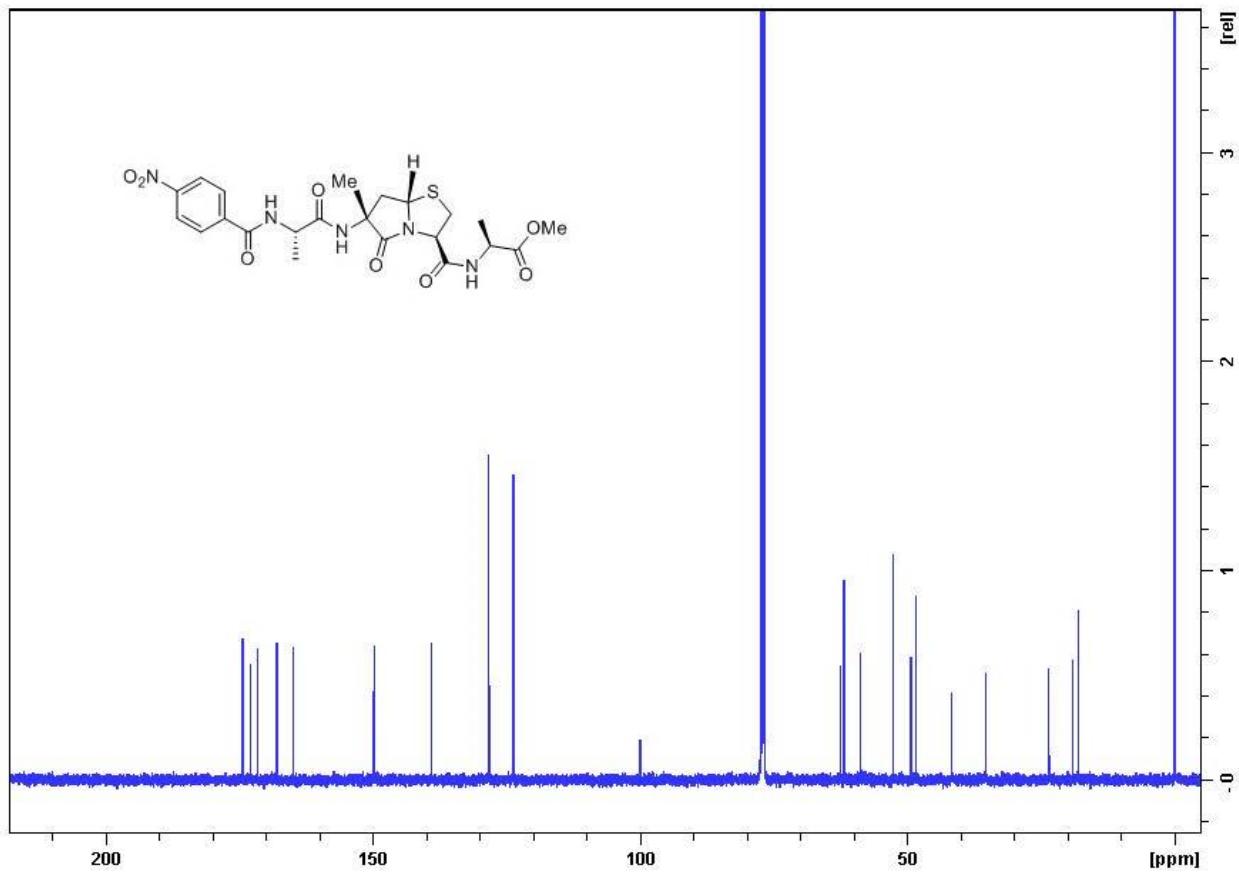


Figure S60. Carbon-13 NMR Spectrum of  $\beta$ -30b in  $\text{CDCl}_3$

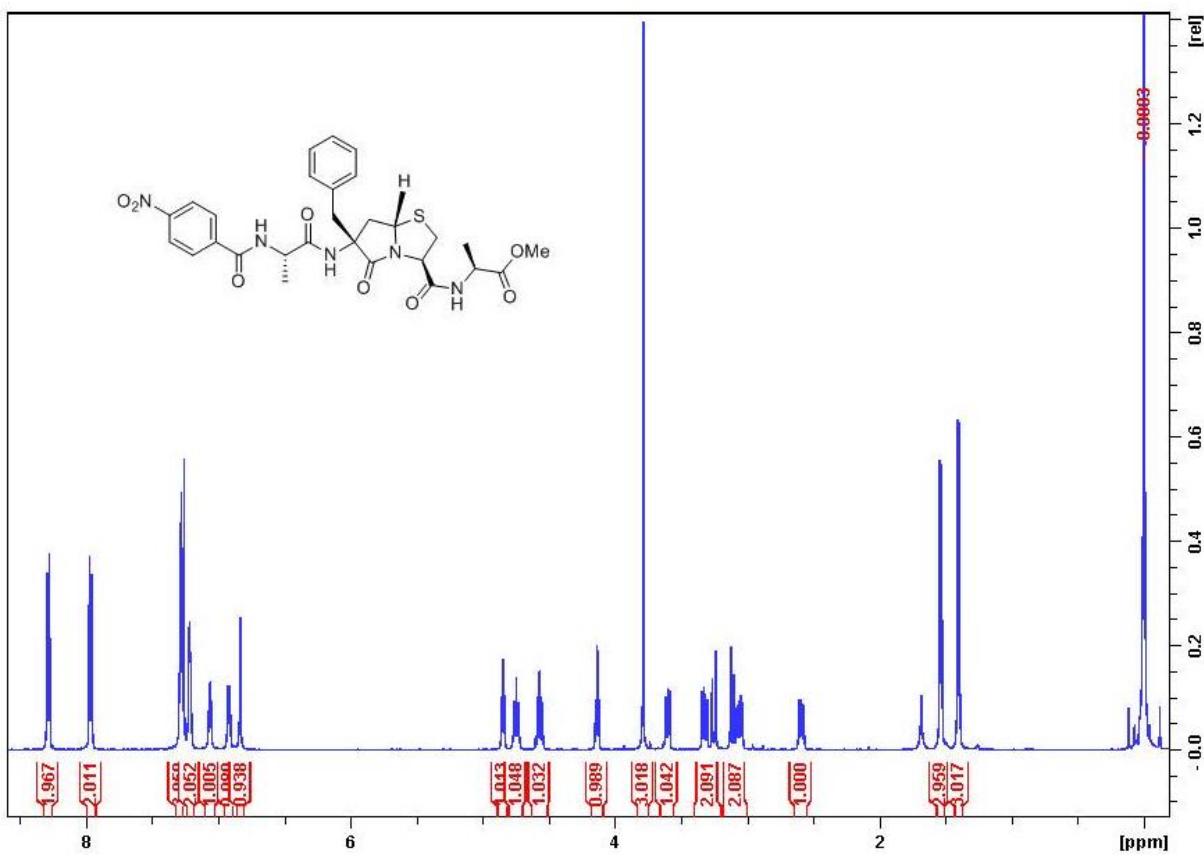


Figure S61. Proton NMR Spectrum of  $\beta$ -30c in  $\text{CDCl}_3$

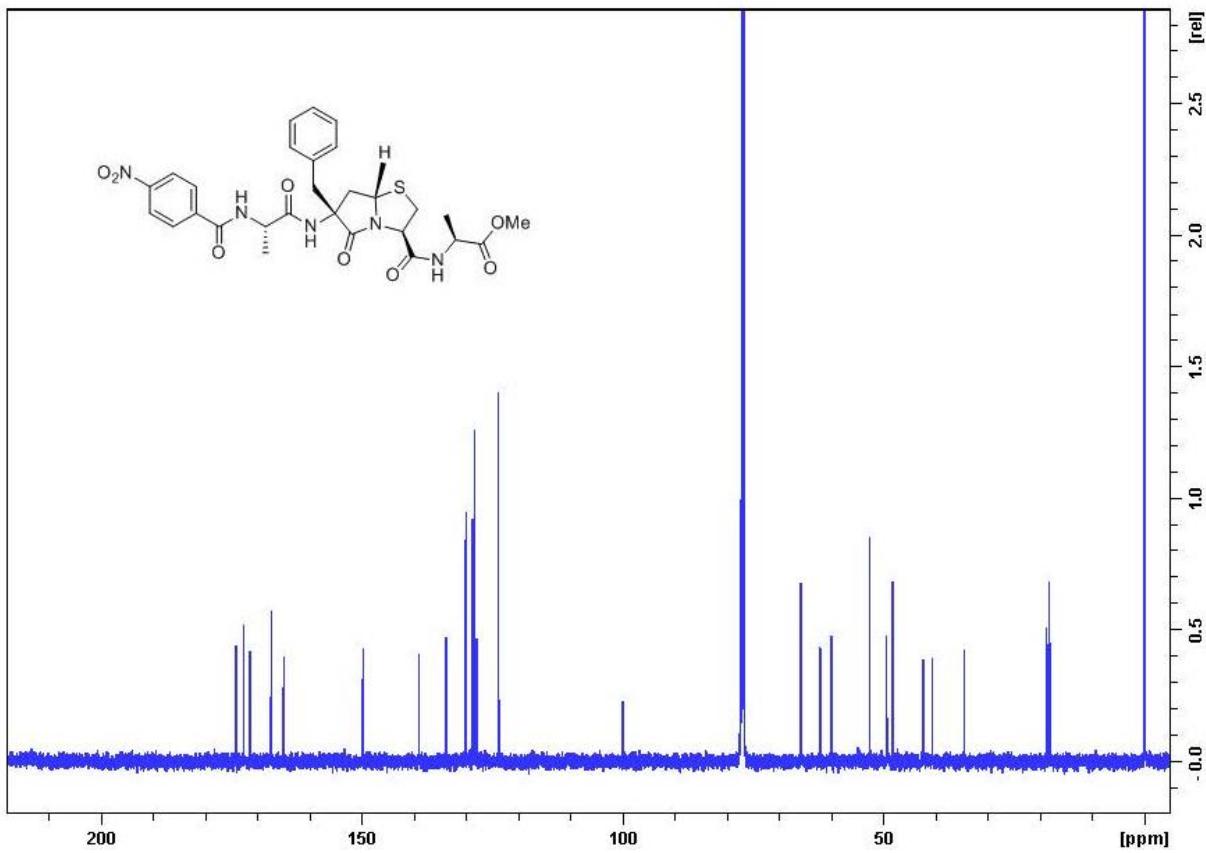


Figure S62. Carbon-13 NMR Spectrum of  $\beta$ -30c in  $\text{CDCl}_3$

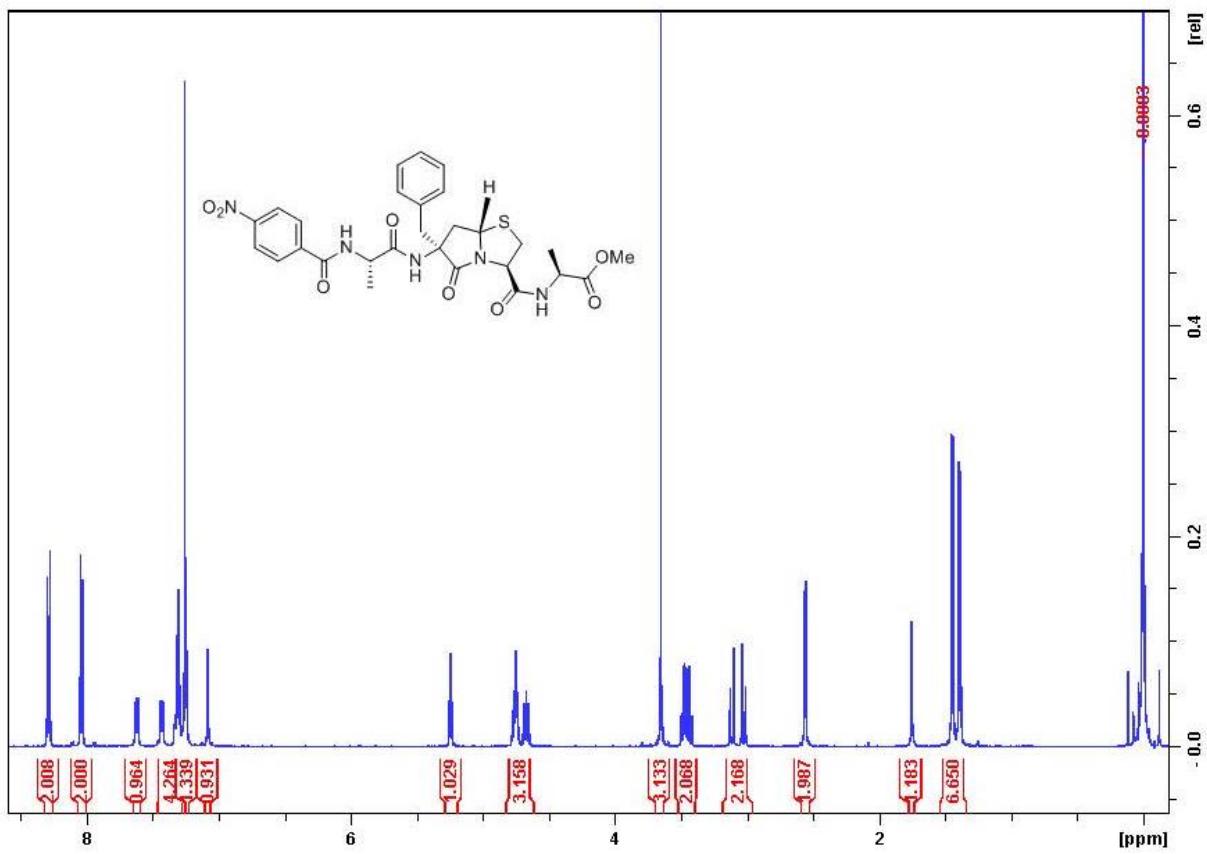


Figure S63. Proton NMR Spectrum of  $\alpha$ -30c in  $\text{CDCl}_3$

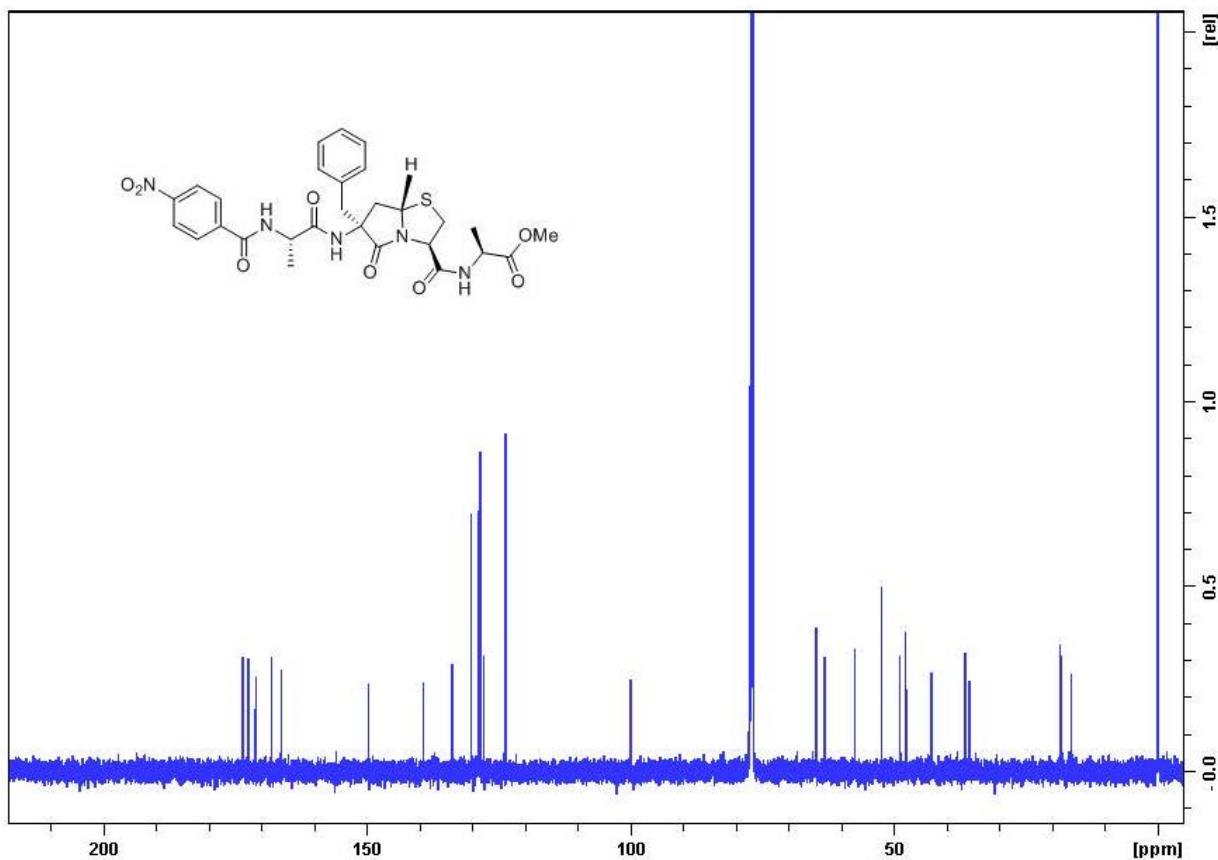
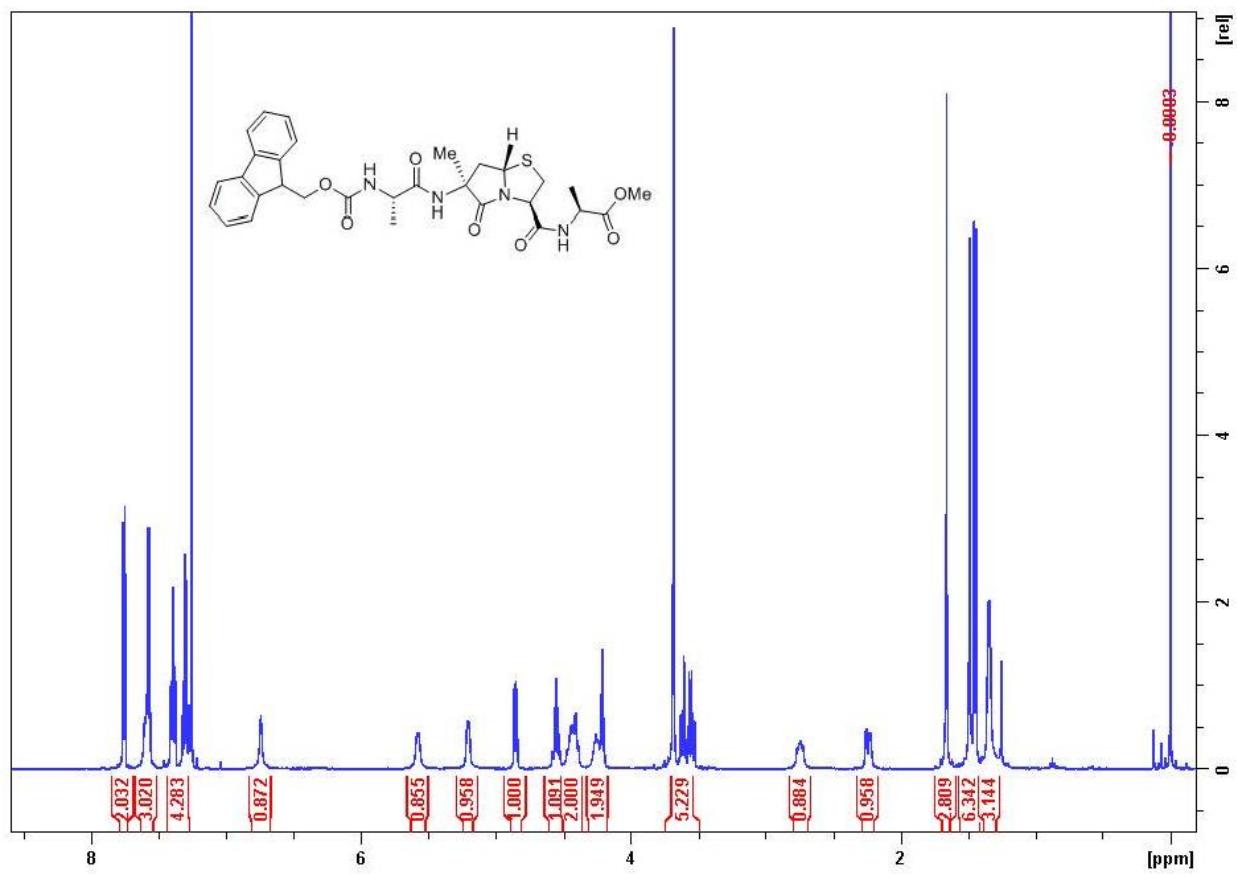


Figure S64. Carbon-13 NMR Spectrum of  $\alpha$ -30c in  $\text{CDCl}_3$



**Figure S65.** Proton NMR Spectrum of  $\alpha$ -37b in  $\text{CDCl}_3$

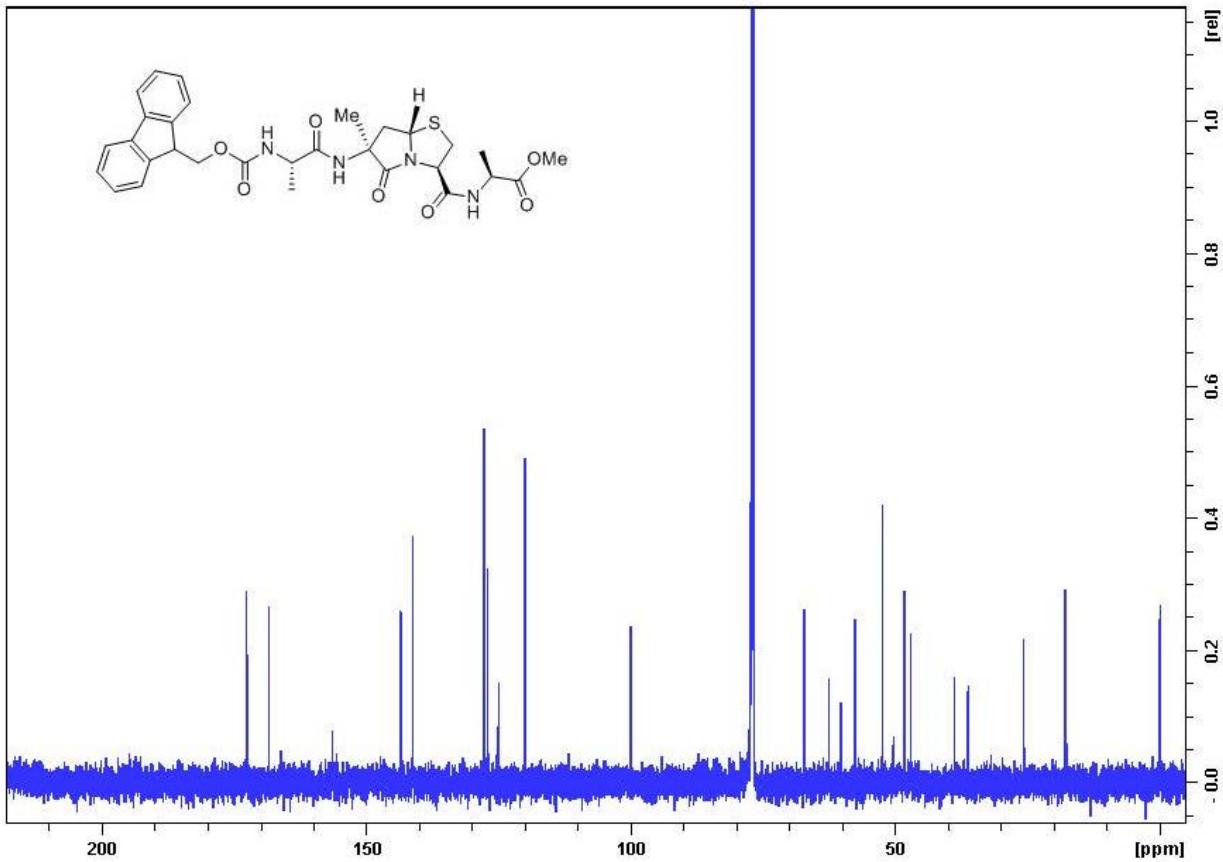


Figure S66. Carbon-13 NMR Spectrum of  $\alpha$ -37b in  $\text{CDCl}_3$

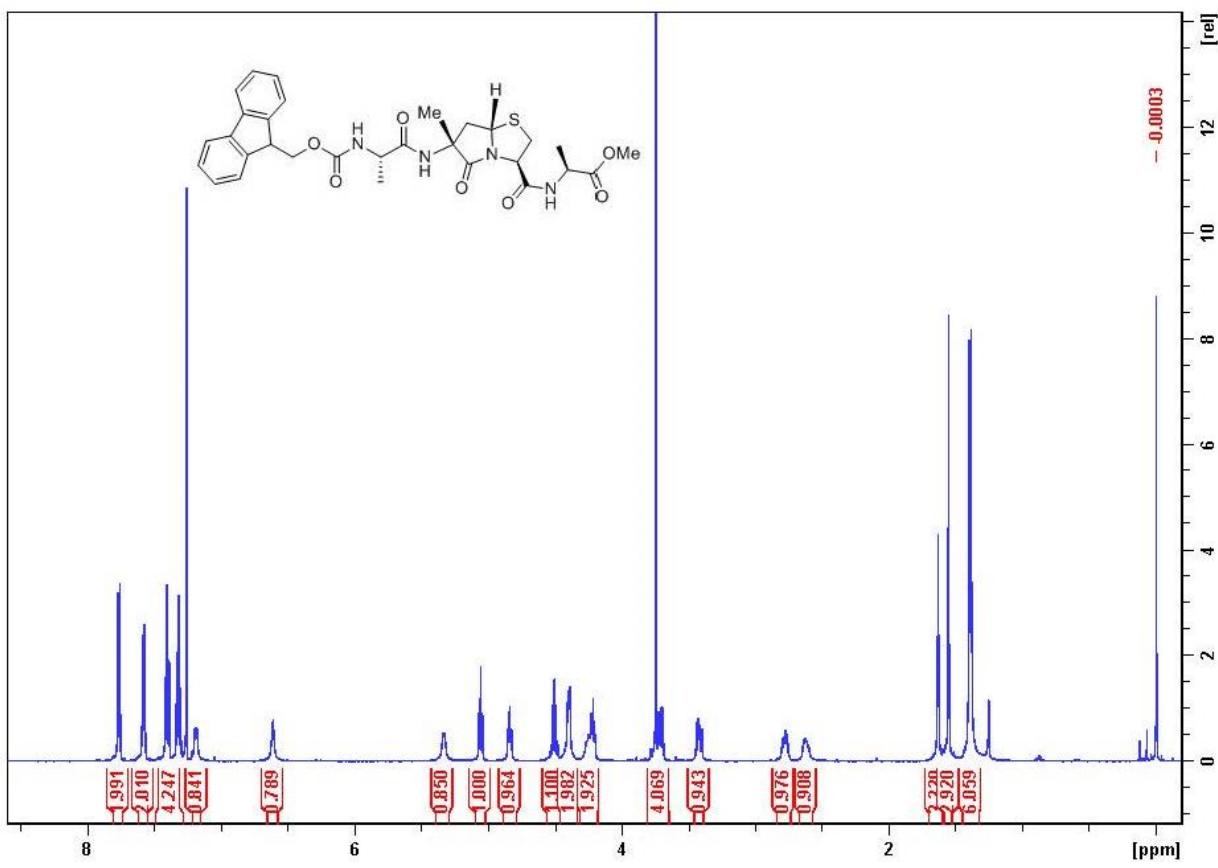


Figure S67. Proton NMR Spectrum of  $\beta$ -37b in  $\text{CDCl}_3$

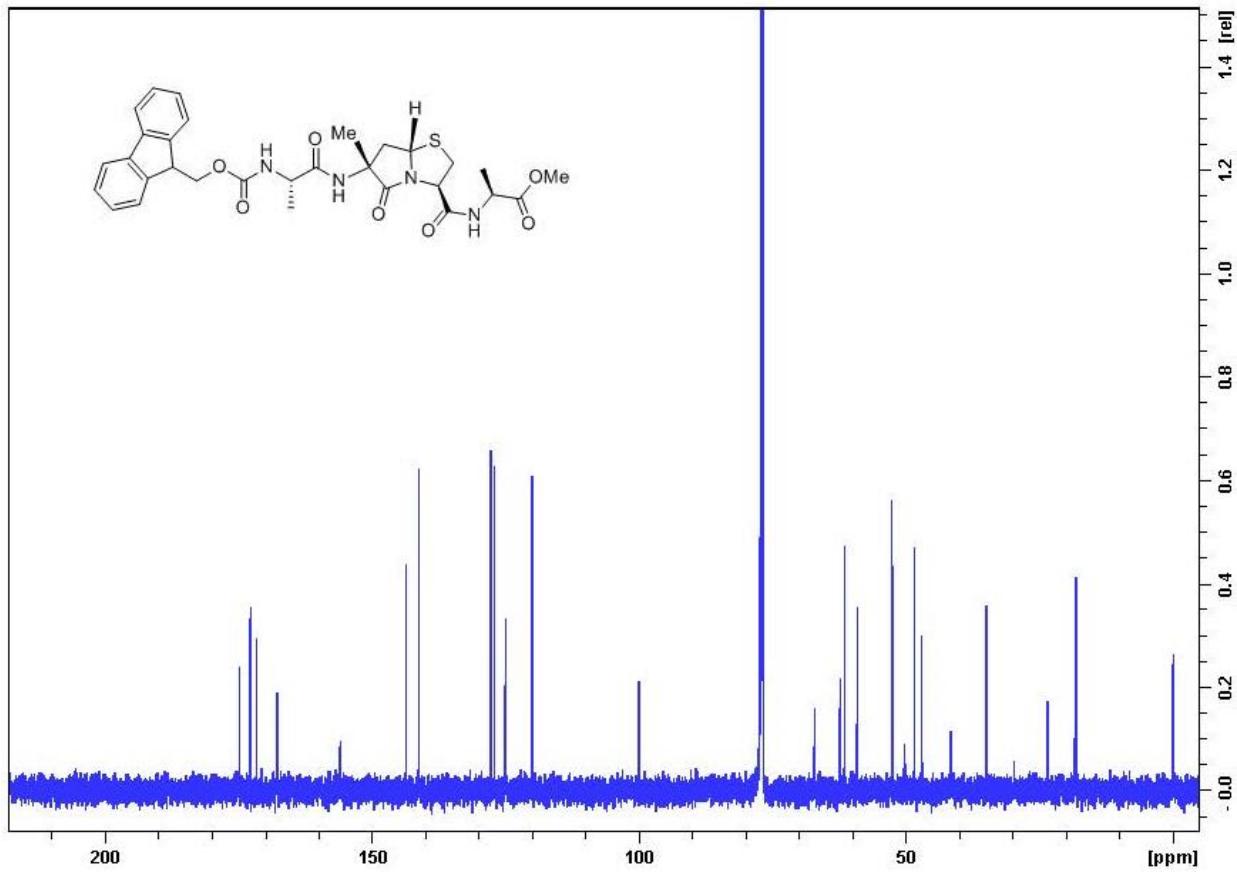
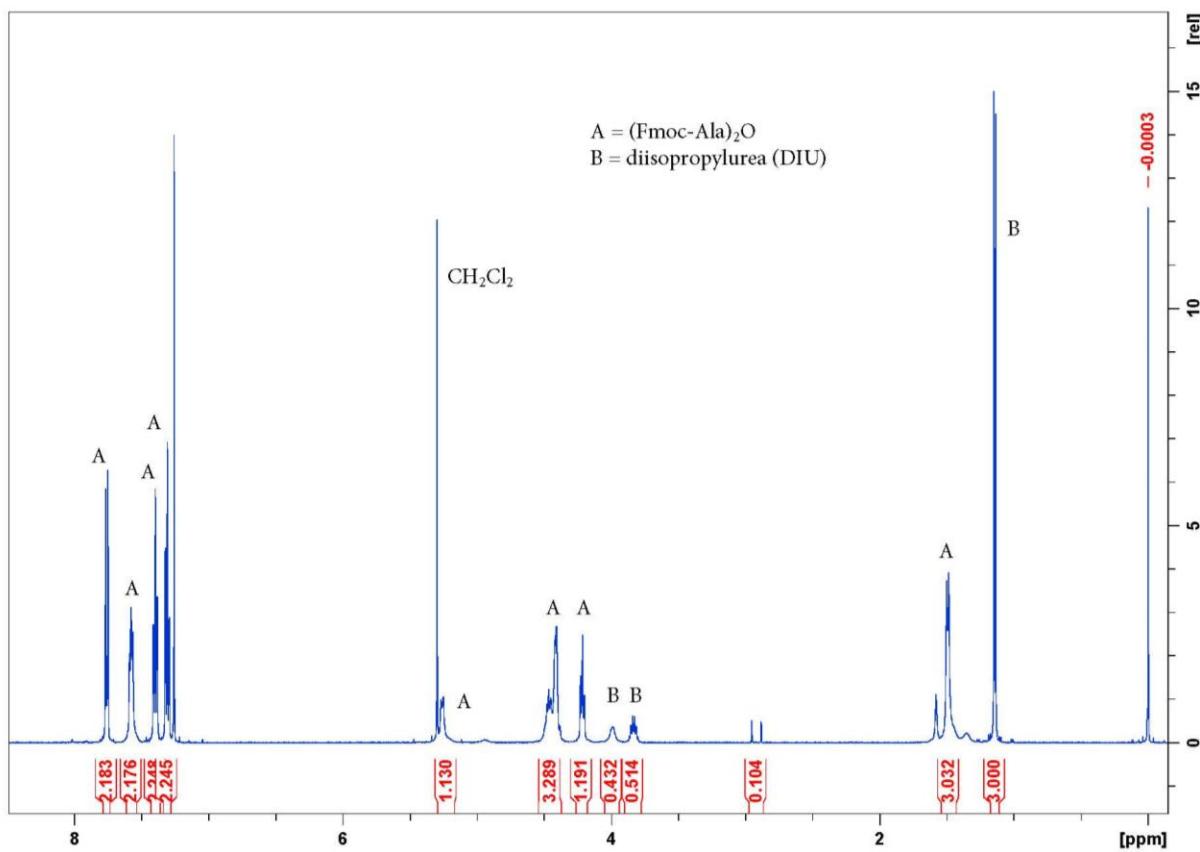


Figure S68. Carbon-13 NMR Spectrum of  $\beta$ -37b in  $\text{CDCl}_3$



**Figure S69.** Proton NMR Spectrum of (Fmoc-Ala)<sub>2</sub>O in CDCl<sub>3</sub> with Diisopropylurea (DIU)

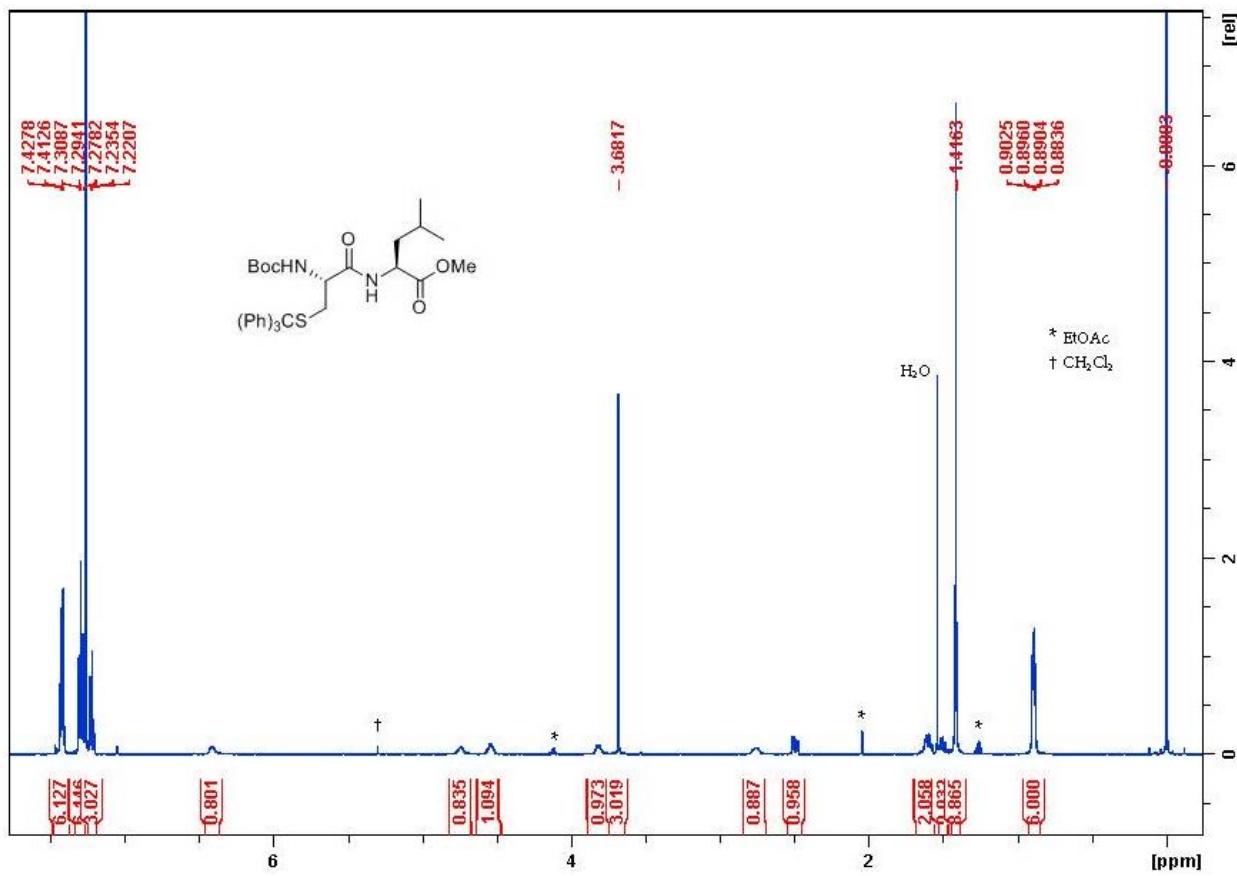
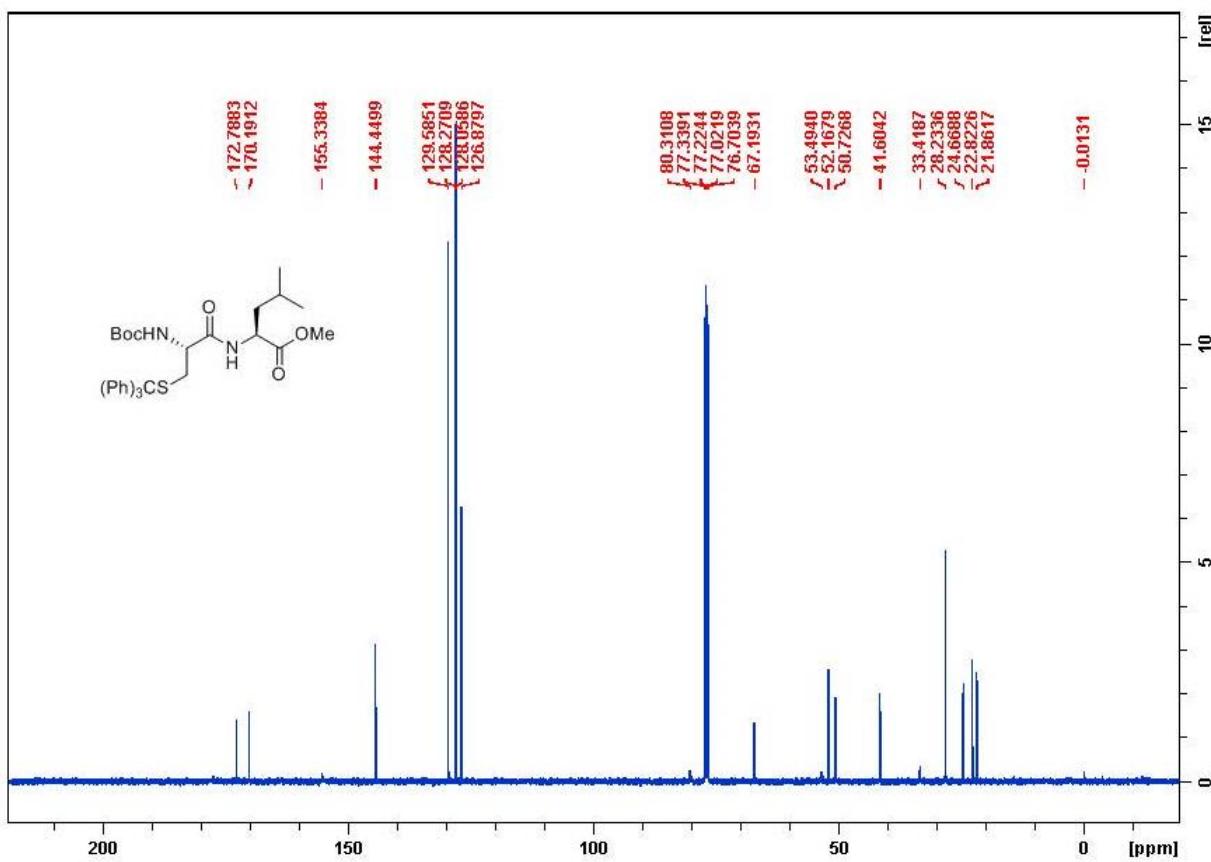


Figure S70. Proton NMR Spectrum of Boc-Cys(Trt)-Leu-OMe in CDCl<sub>3</sub>



**Figure S71.** Carbon-13 NMR Spectrum of Boc-Cys(Trt)-Leu-OMe in  $\text{CDCl}_3$