

Synthesis of *O*-Benzyl Hydroxamates Employing the Sulfonate Esters of *N*-Hydroxybenzotriazole

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Supporting Information

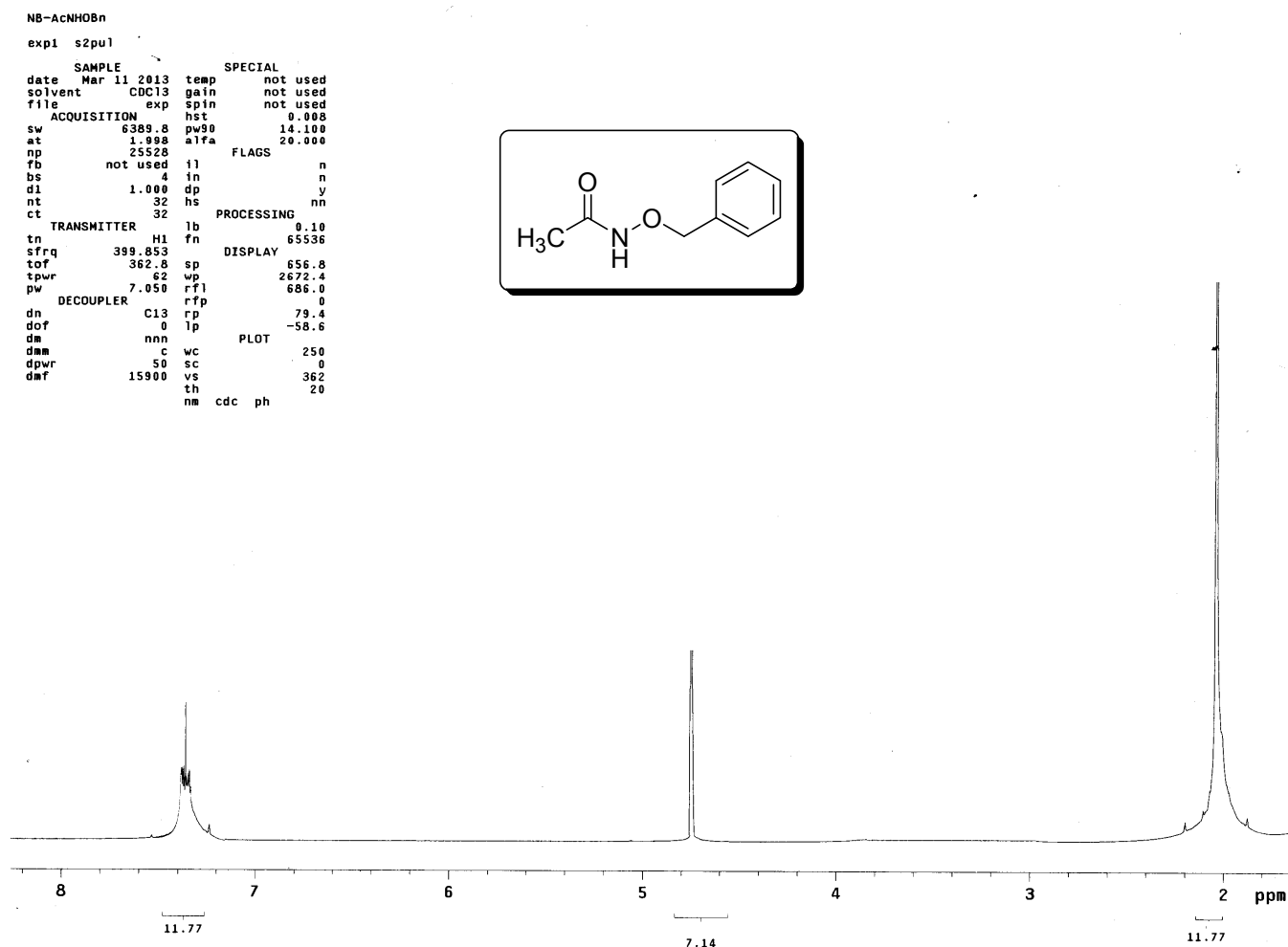


Figure S1. ¹H NMR spectra of Ac-NHOBn (entry 1, table 2)

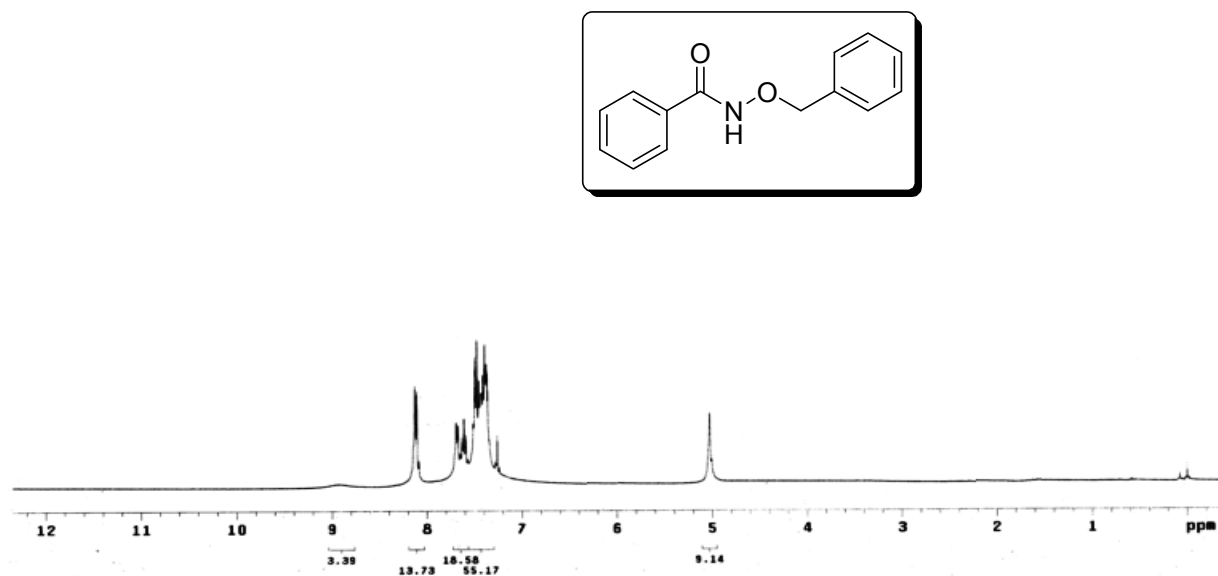


Figure S2. ¹H NMR spectra of BzCONHOBn (entry 2, table 2)

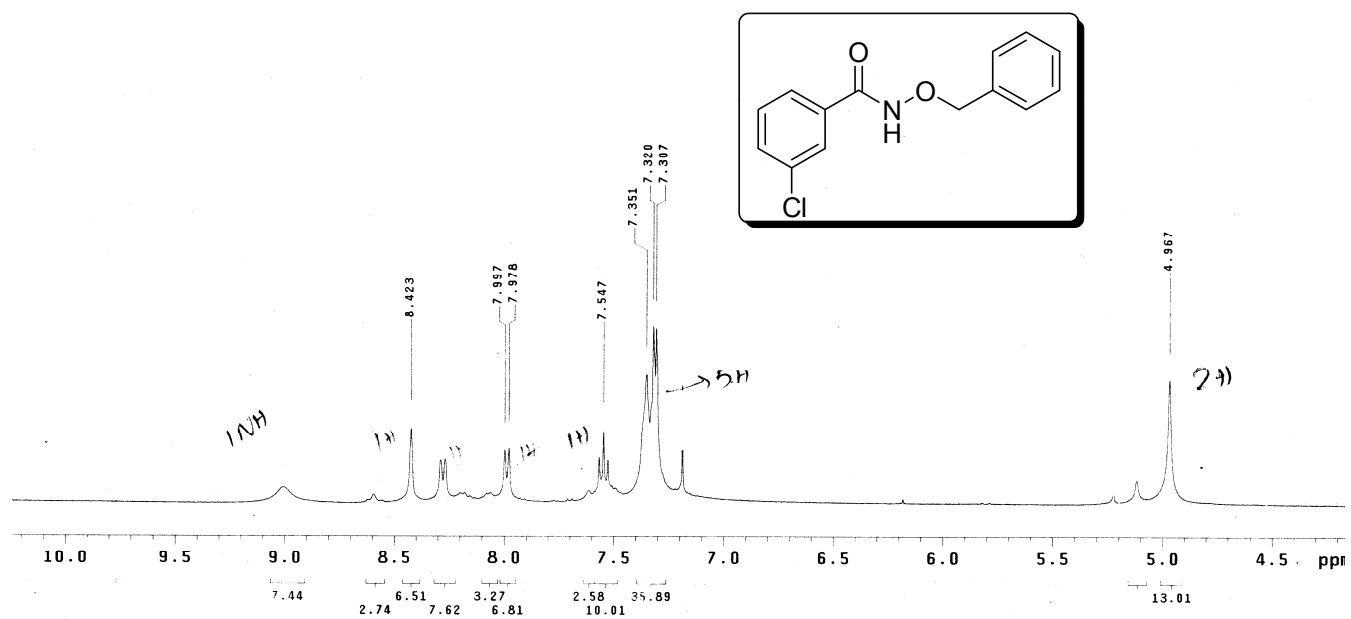


Figure S3. ¹H NMR spectra of *m*-Cl₂-C₆H₄-CONHOBn (entry 3, table 2)

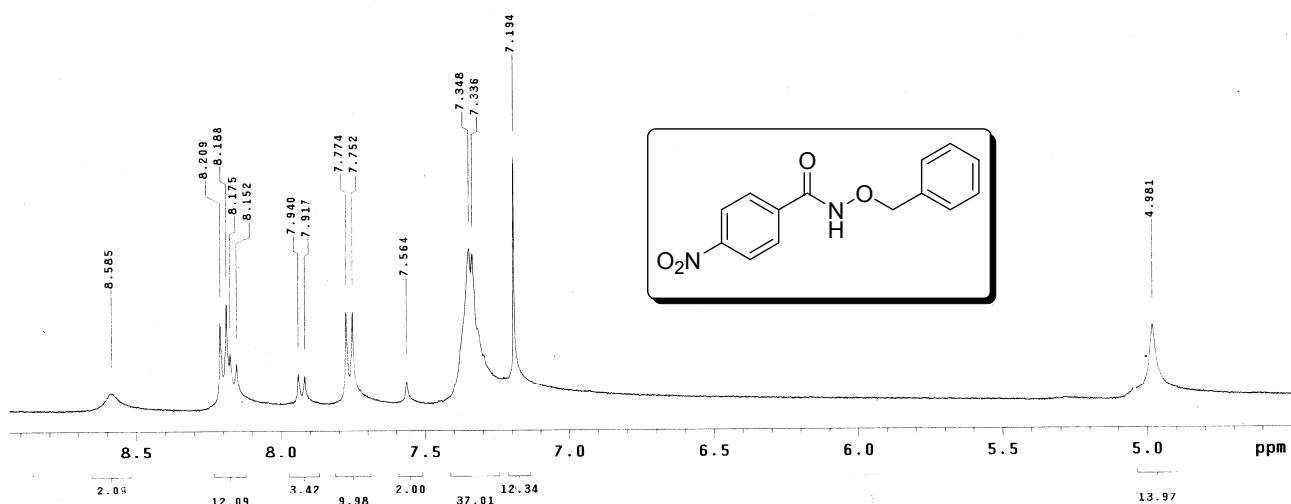


Figure S4. ¹H NMR spectra of *p*-NO₂-C₆H₄-CONHOBn (entry 4, table 2)

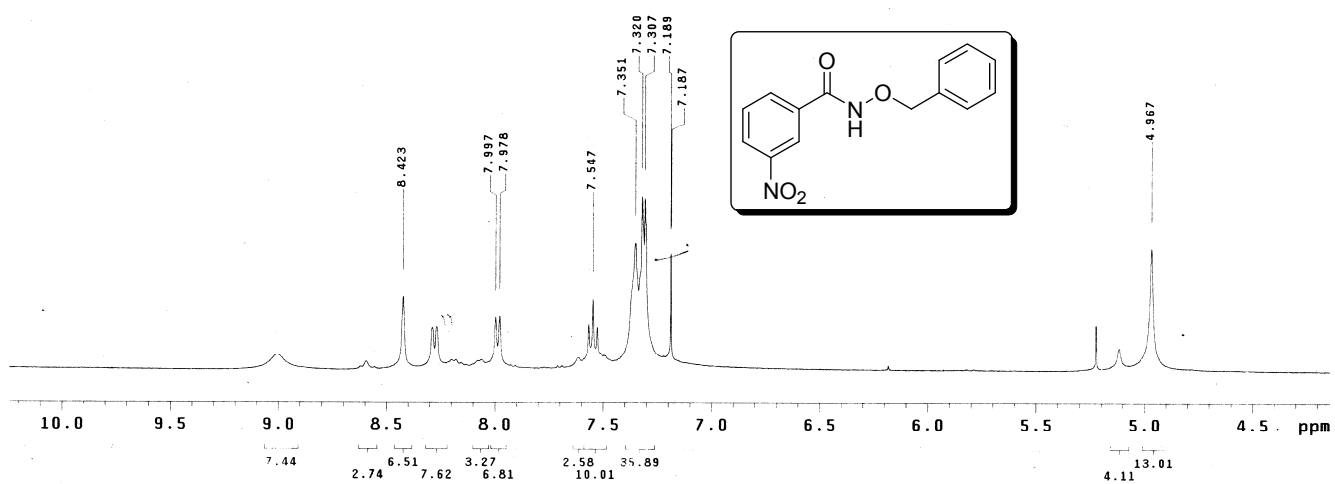


Figure S5. ¹H NMR spectra of *m*-NO₂-C₆H₄-CONHOBn (entry 5, table 2)

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d1 1.000 dp y
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tpr 57 wp 790.4
pw 9.050 rF1 0
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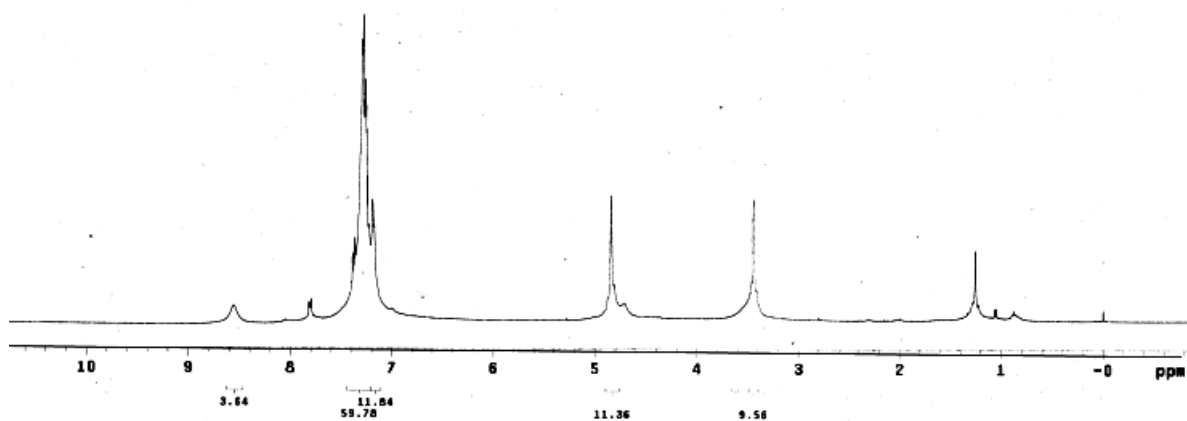
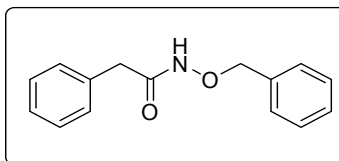


Figure S6. ^1H NMR spectra of BnCONHOBn (entry 6, table 2)

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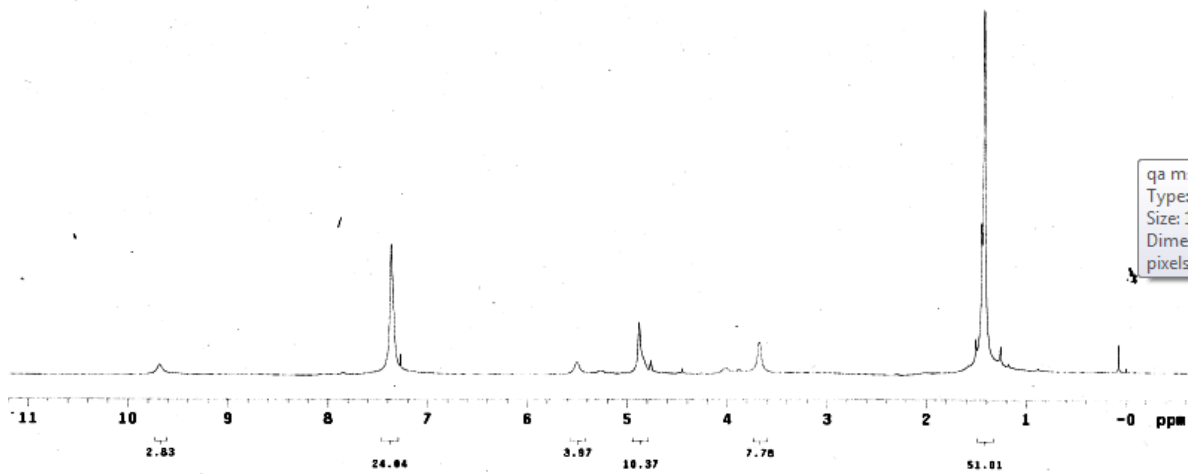
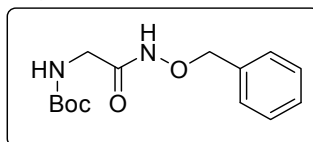


Figure S7. ^1H NMR spectra of Boc-Gly-NHOBn (entry 7, table 2)

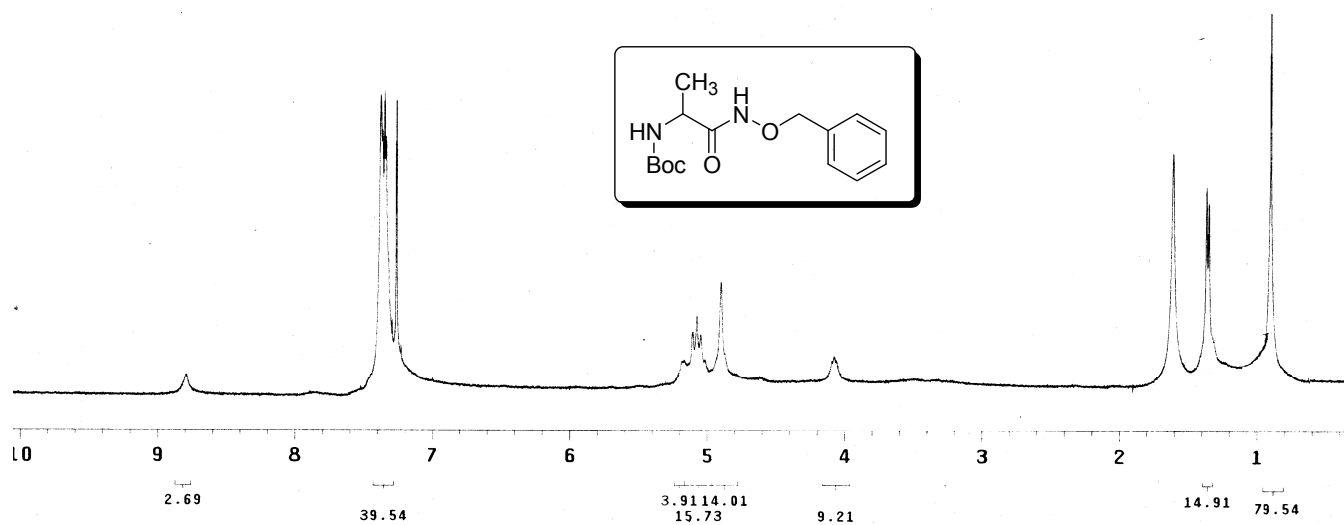


Figure S8. ¹H NMR spectra of Boc-Ala-NHOBn (entry 8, table 2)

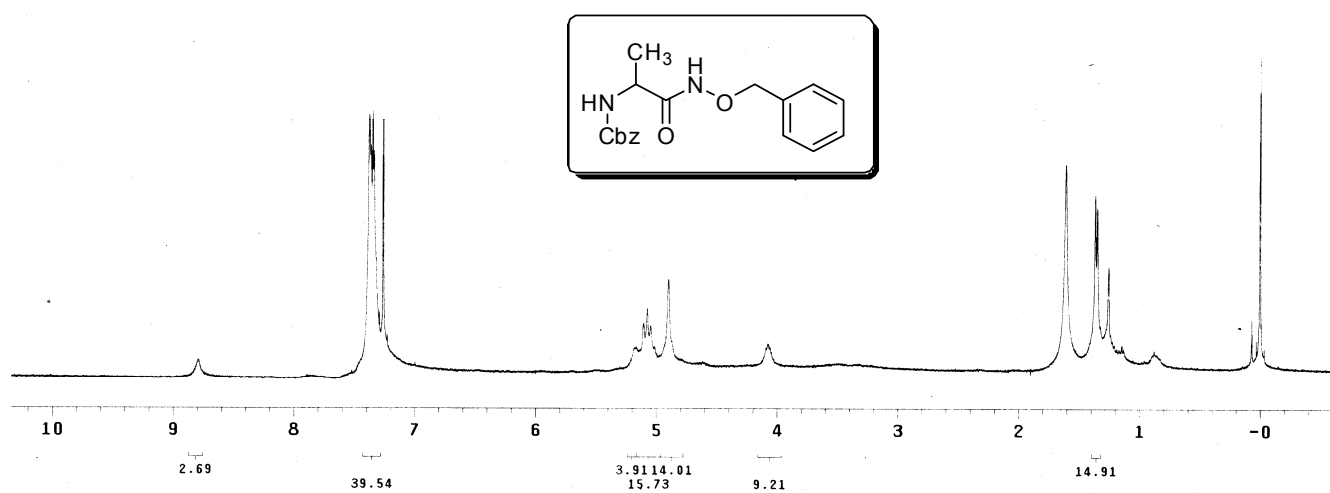


Figure S9. ¹H NMR spectra of Cbz-Ala-NHOBn (entry 9, table 2)

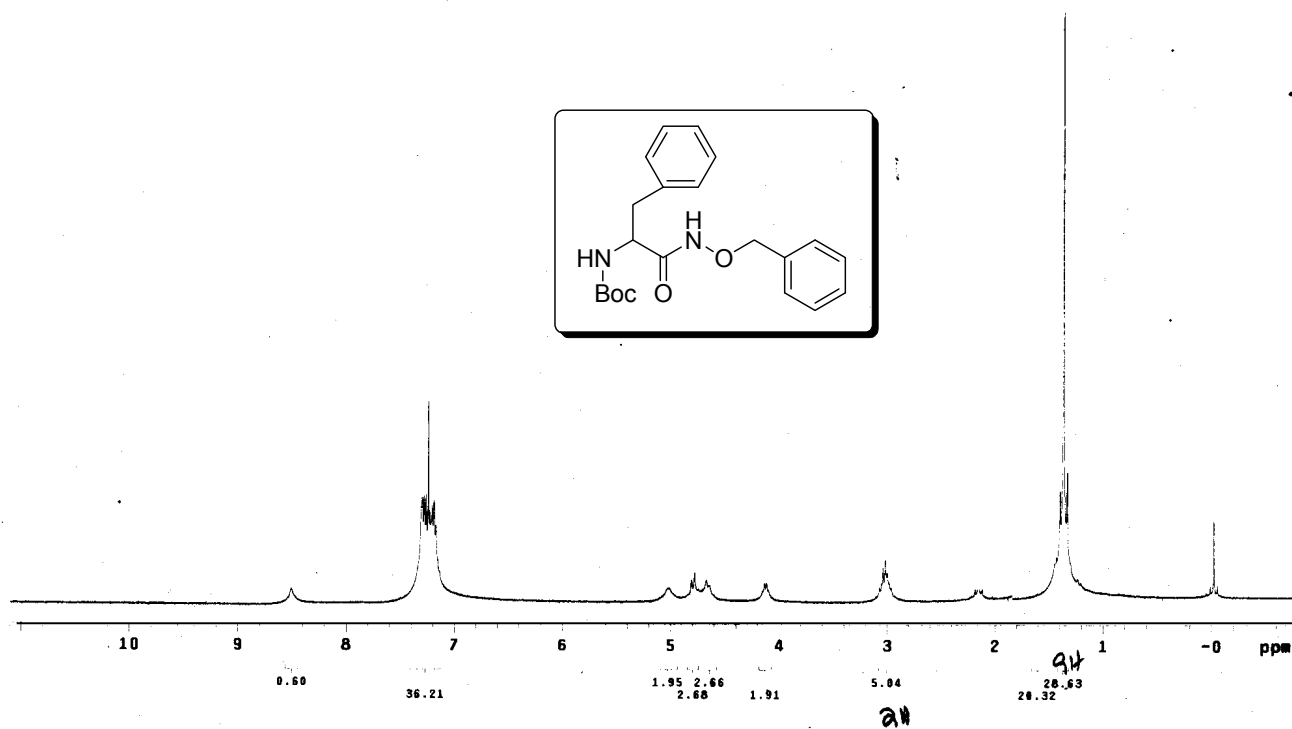


Figure S10. ¹H NMR spectra of Boc-Phe-NHOBn (entry 10, table 2)

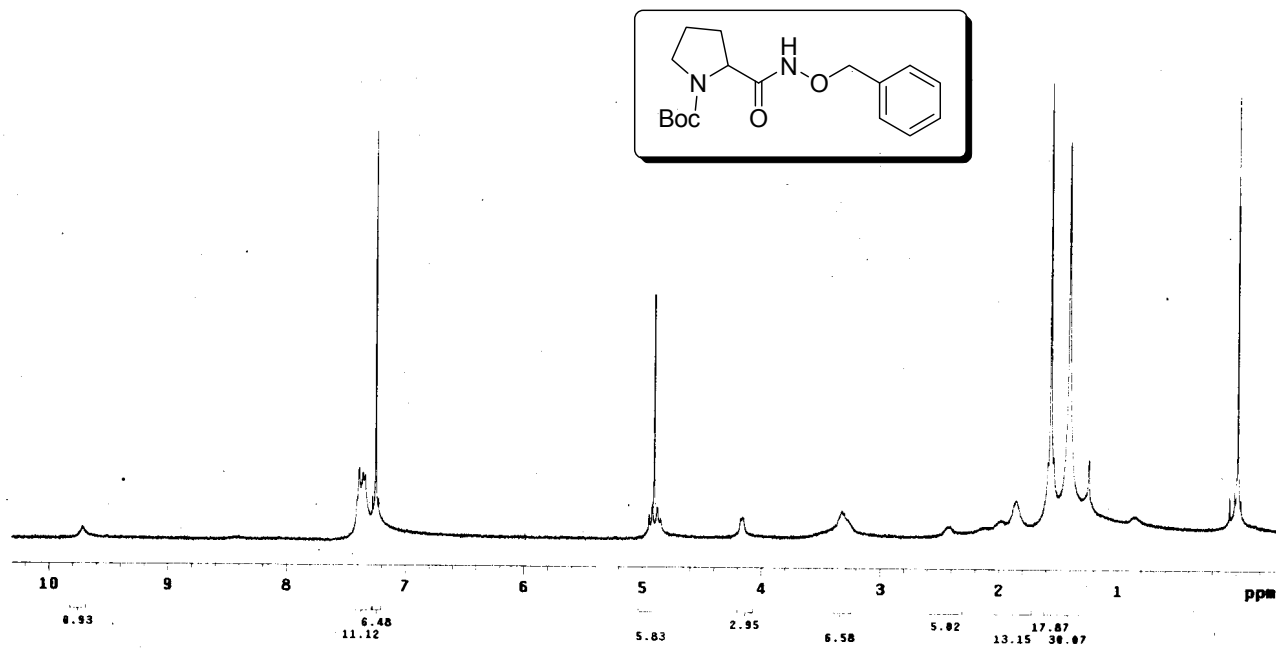


Figure S11. ¹H NMR spectra of Boc-Pro-NHOBn (entry 11, table 2)

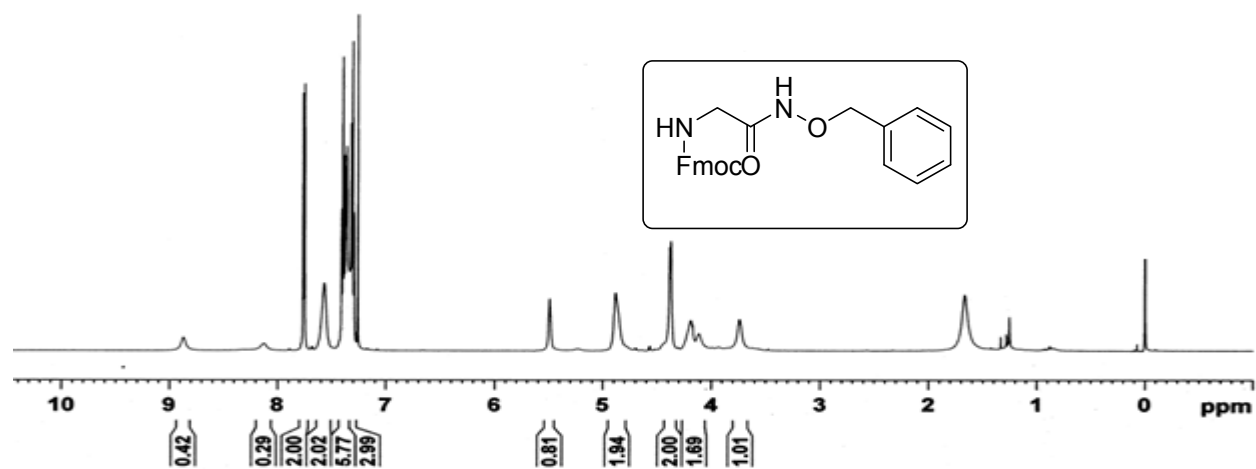


Figure S12. ¹H NMR spectra of Fmoc-Gly-NHOBn (entry 12, table 2)

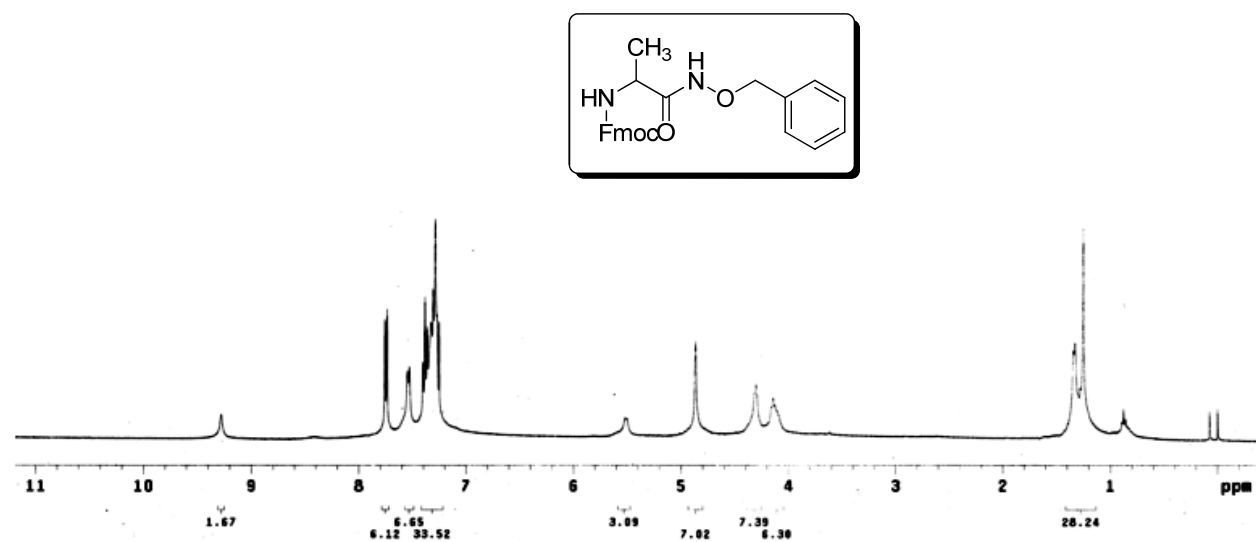


Figure S13. ¹H NMR spectra of Fmoc-Ala-NHOBn (entry 13, table 2)

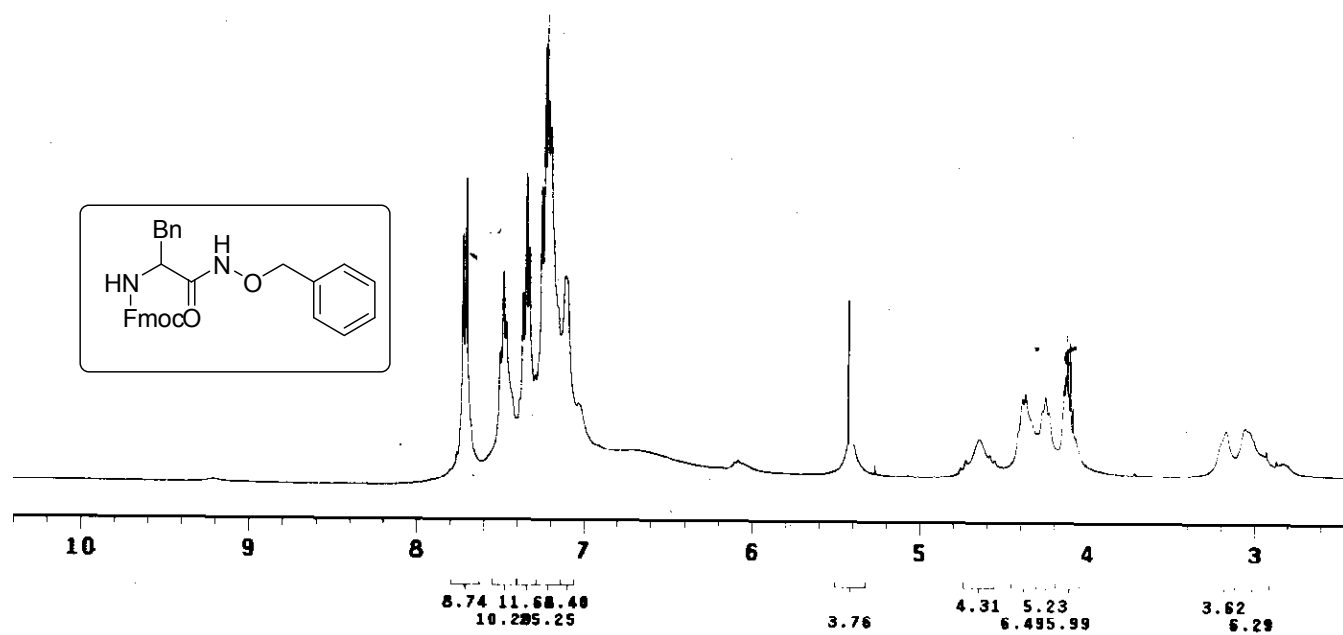


Figure S14. ¹H NMR Spectra of Fmoc-Phe-NHOBn (entry 14 table 2)

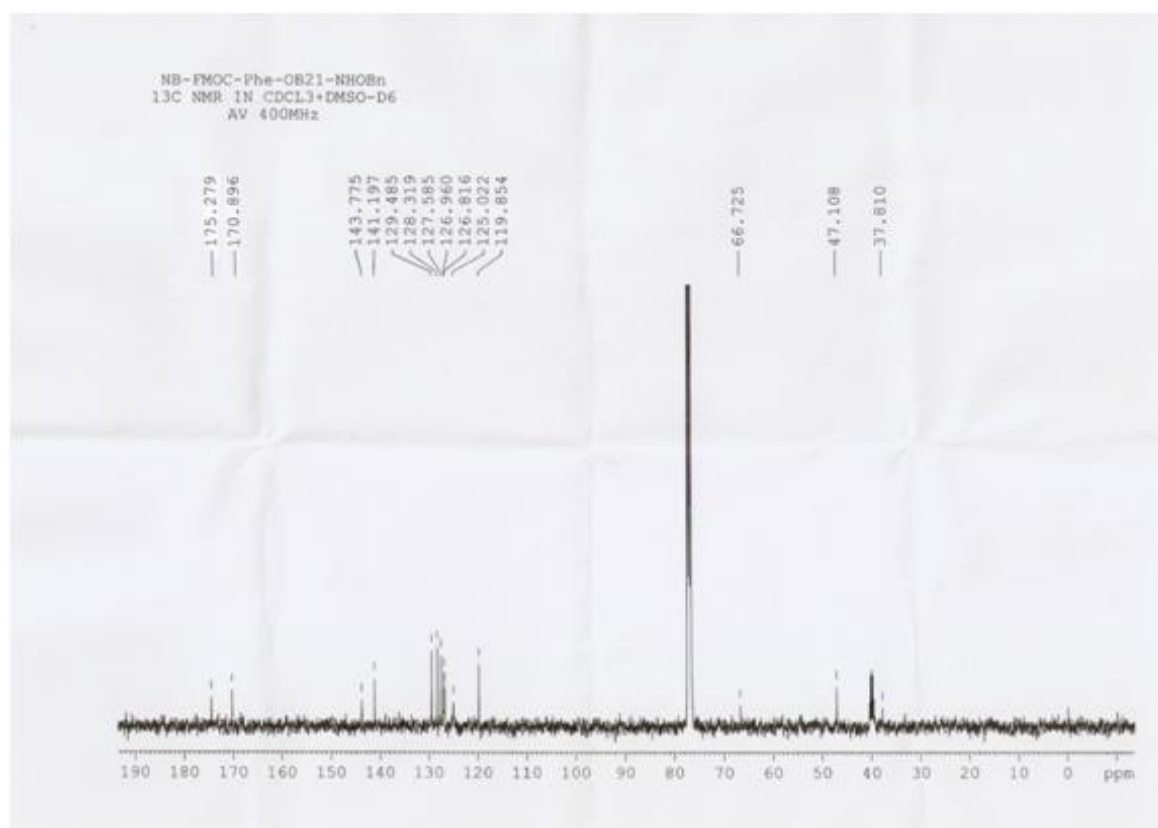


Figure S15. ¹³C NMR spectra of Fmoc-Phe-NHOBn (entry 15, table 2)

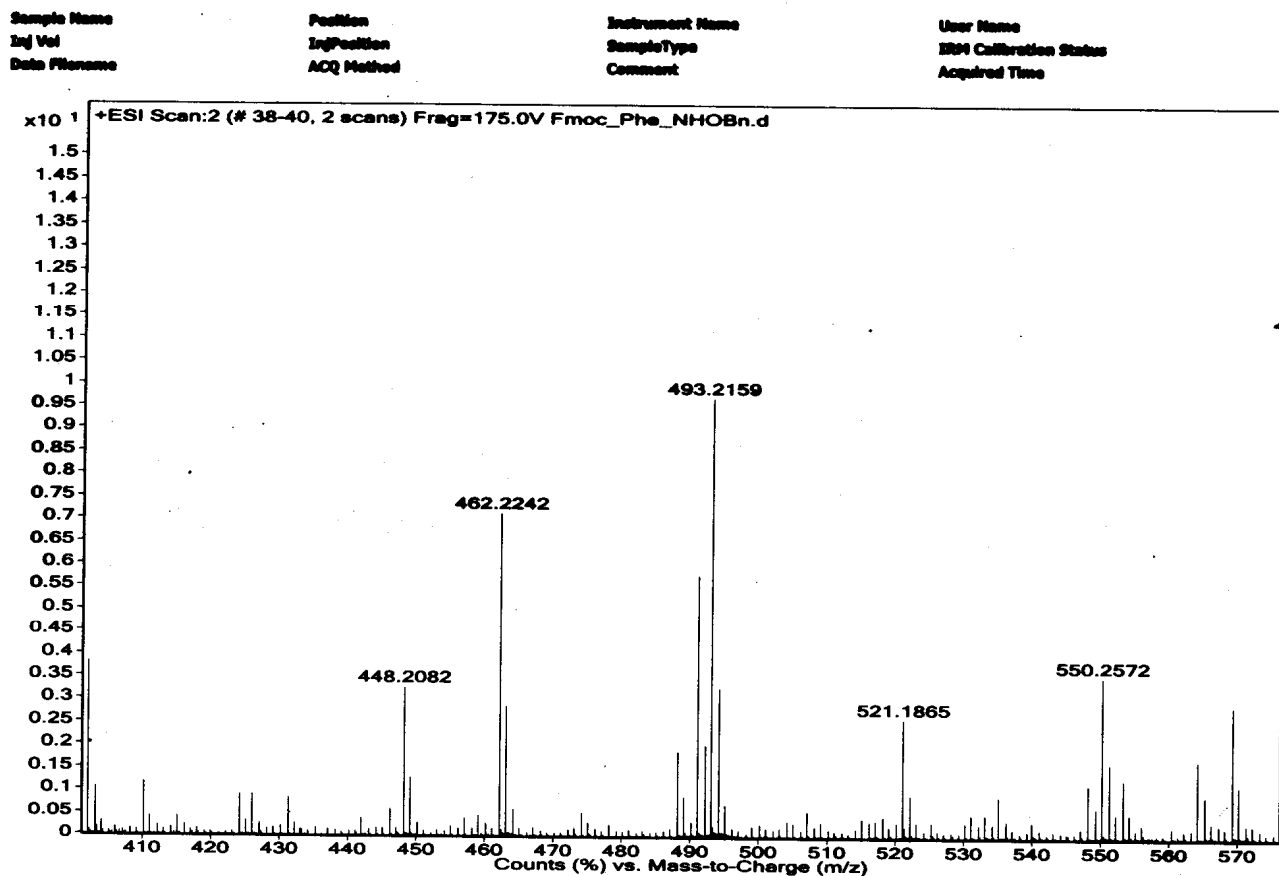


Figure S16. HRMS spectra of Fmoc-Phe-NHOBn (entry 14, table 2). Calcd. mass for $[M+H]^+$: 493.2127 found: 493.2159.

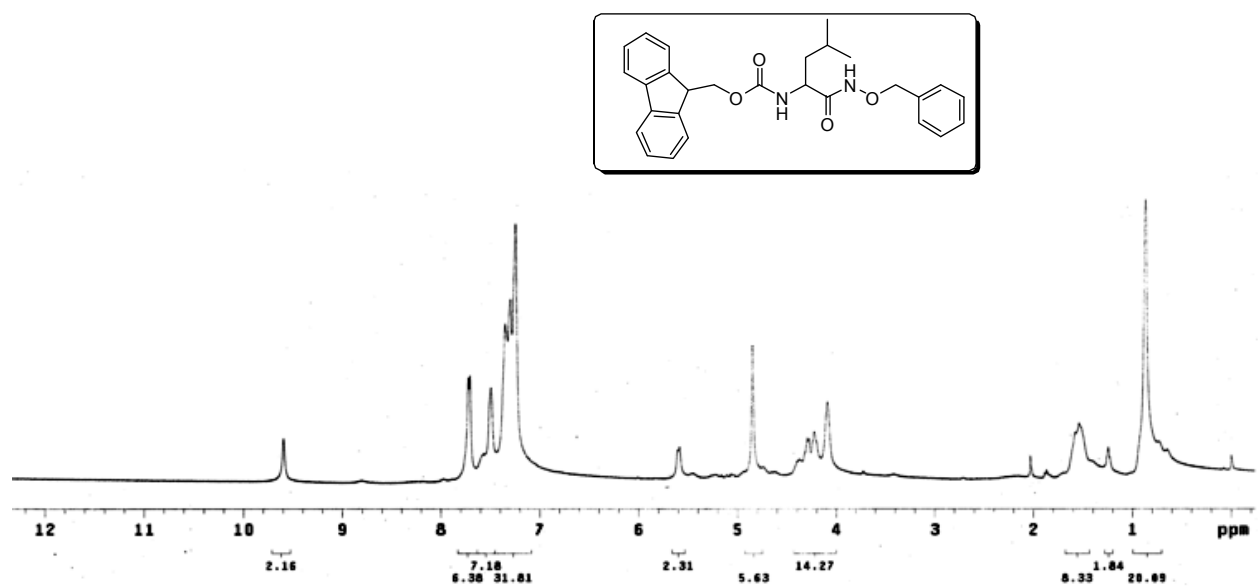


Figure S17. ^1H NMR spectra of Fmoc-Leu-NHOBn (entry 15, table 2)

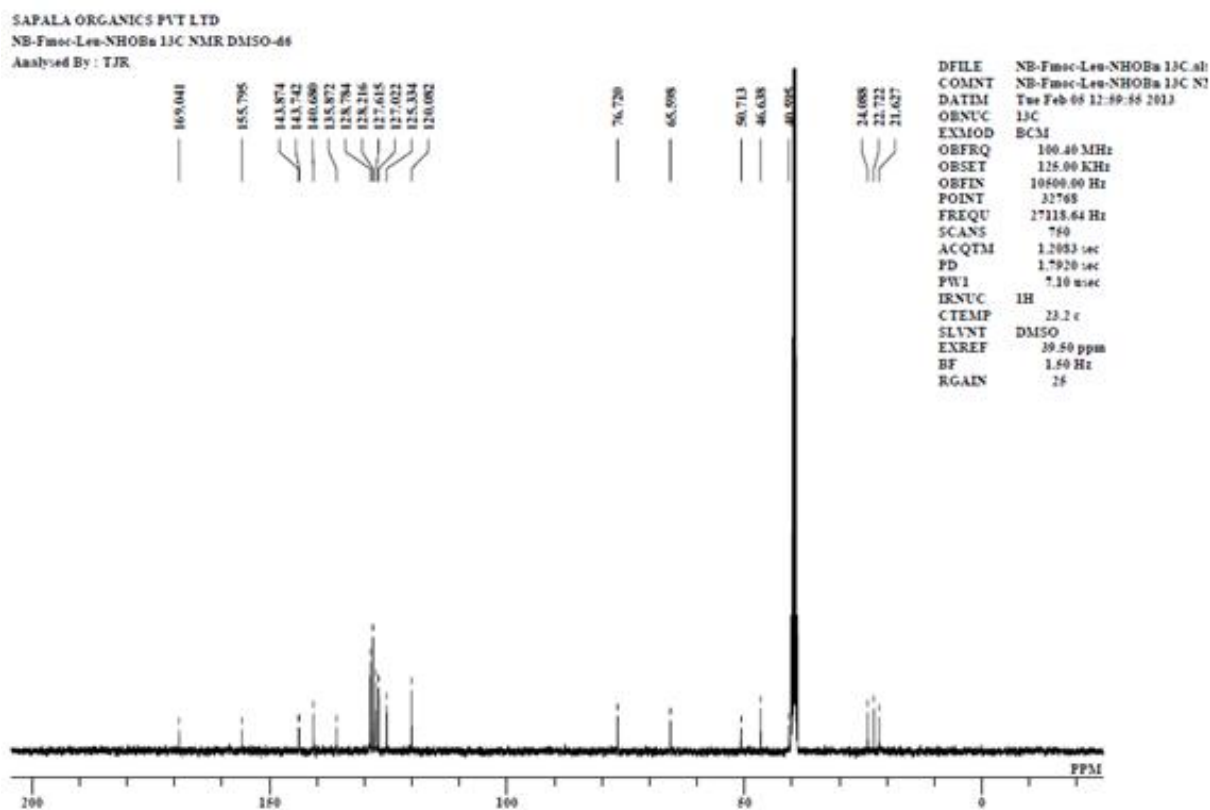


Figure S18. ¹³C NMR spectra of Fmoc-Leu-NHOBn (entry 15, table 2)

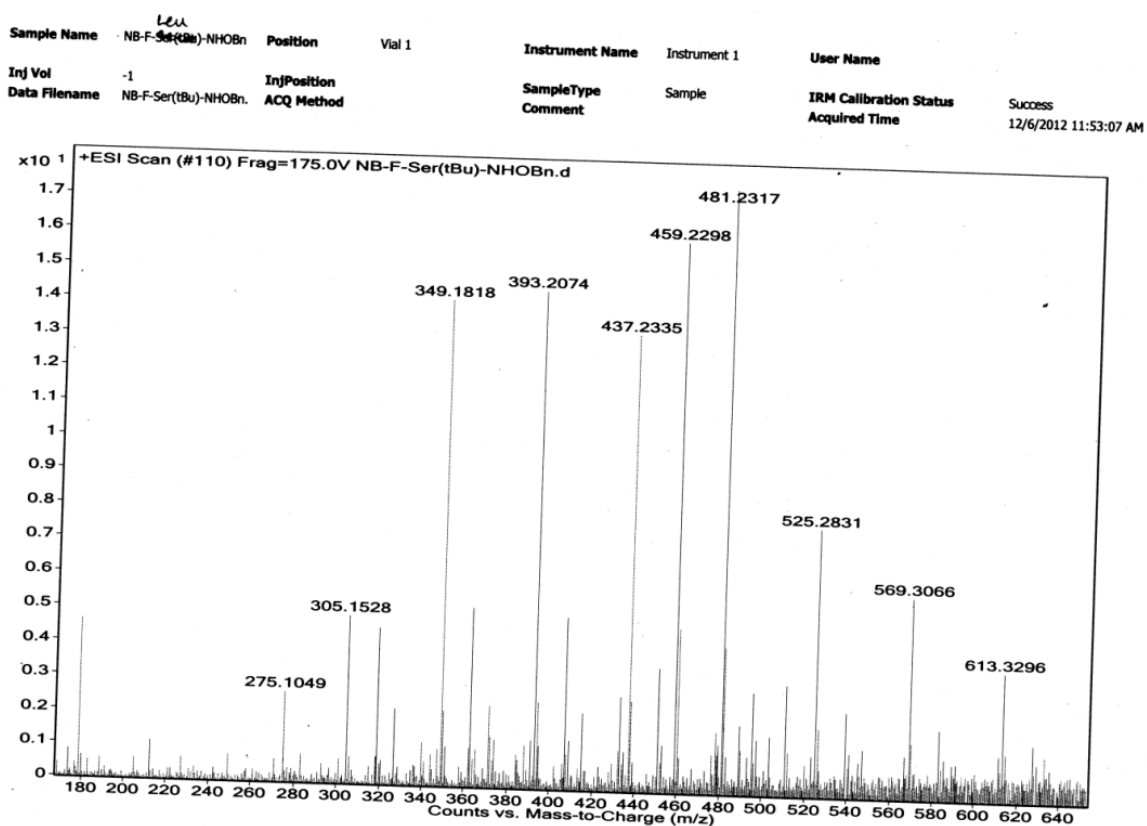


Figure S19. HRMS spectra for Fmoc-Leu-NHOBn, Calcd. mass for [M+H]⁺: 459.2284 found: 459.2298.

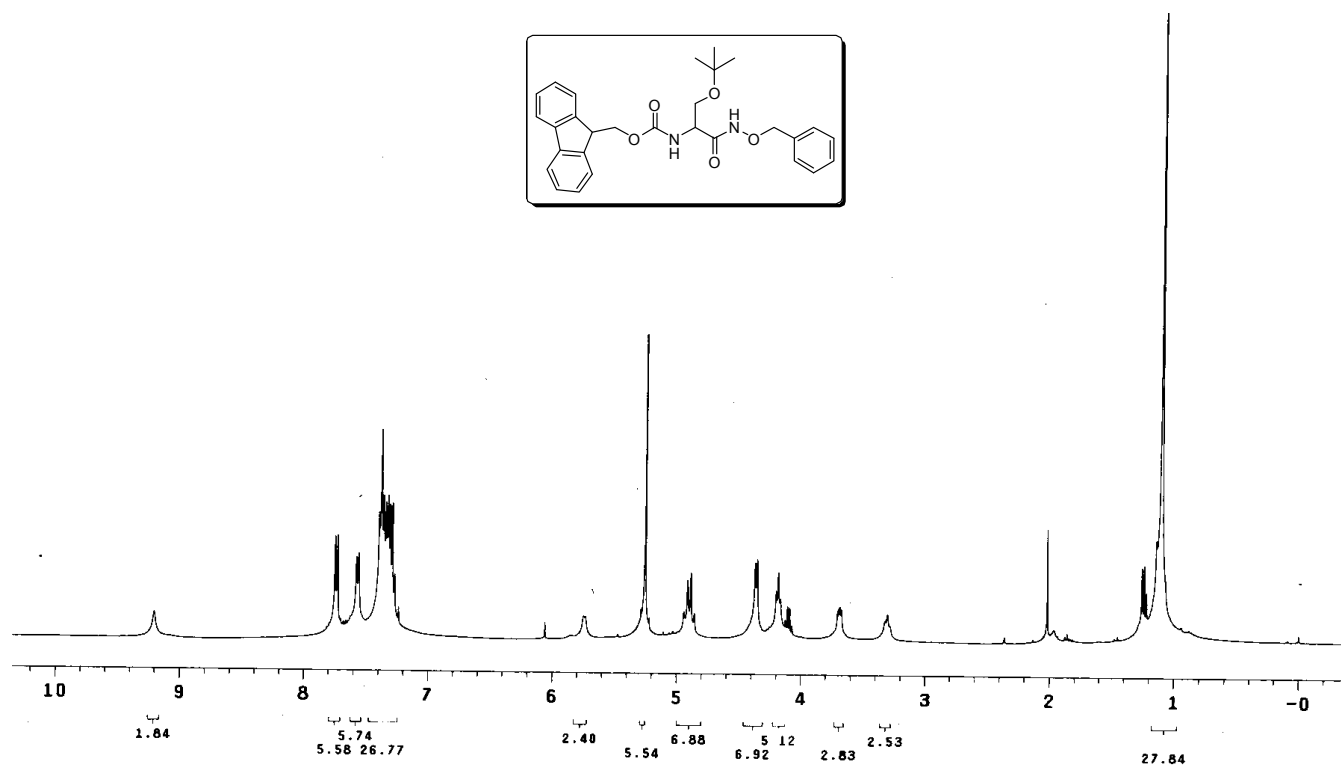


Figure S20. ¹³C NMR spectra of Fmoc-Ser(^tBu)-NHOBn (entry 16, table 2)

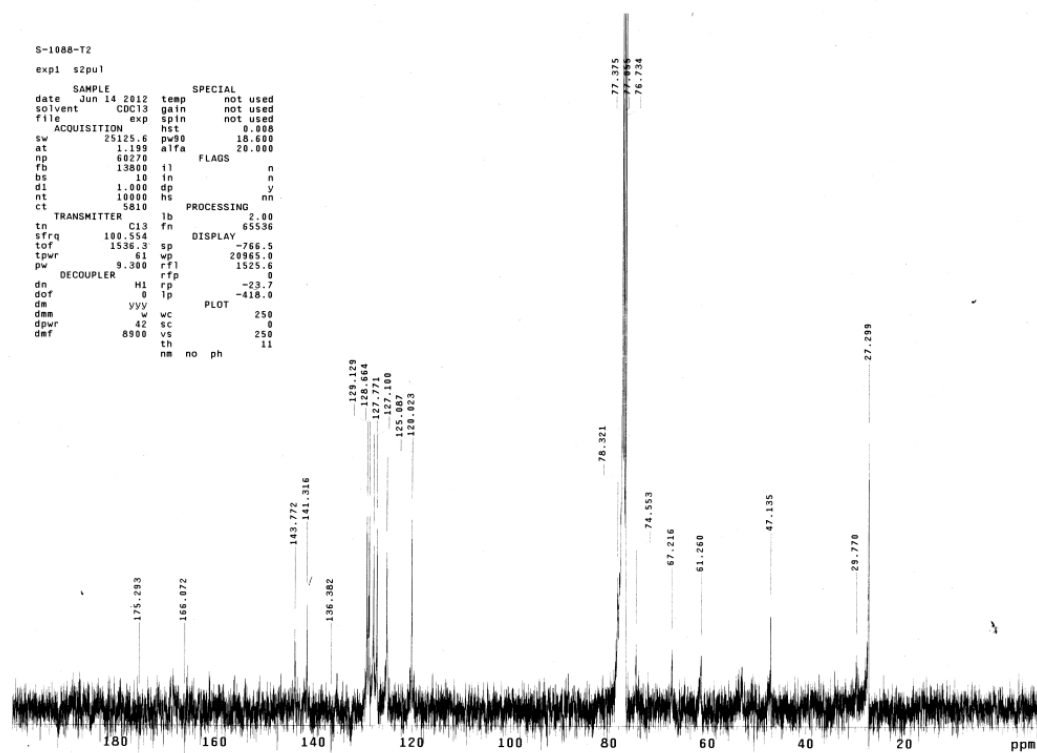


Figure S21. ¹³C NMR spectra of Fmoc-Ser(^tBu)-NHOBn (entry 16, table 2)

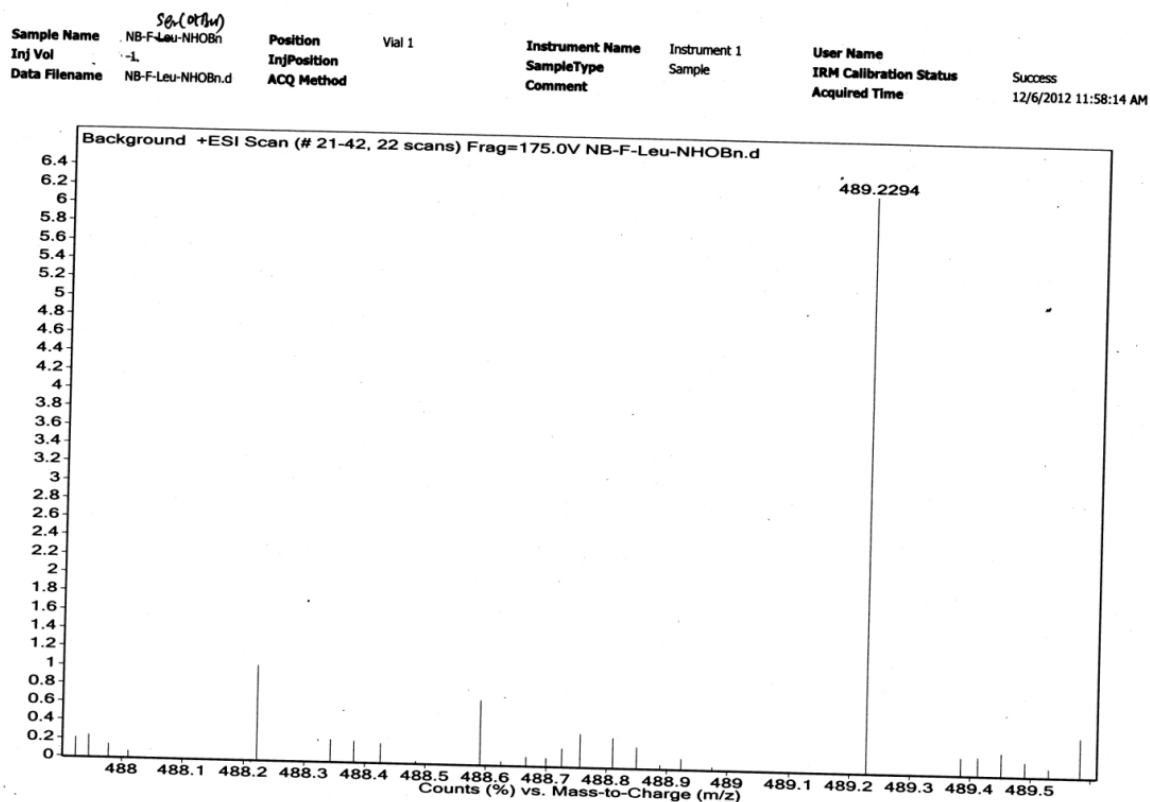


Figure S22. HRMS spectra of Fmoc-Ser(tBu)-NHOBn, Calcd. mass for $[M+H]^+$: 489.2389 found: 489.2294. (entry 16, table 2)

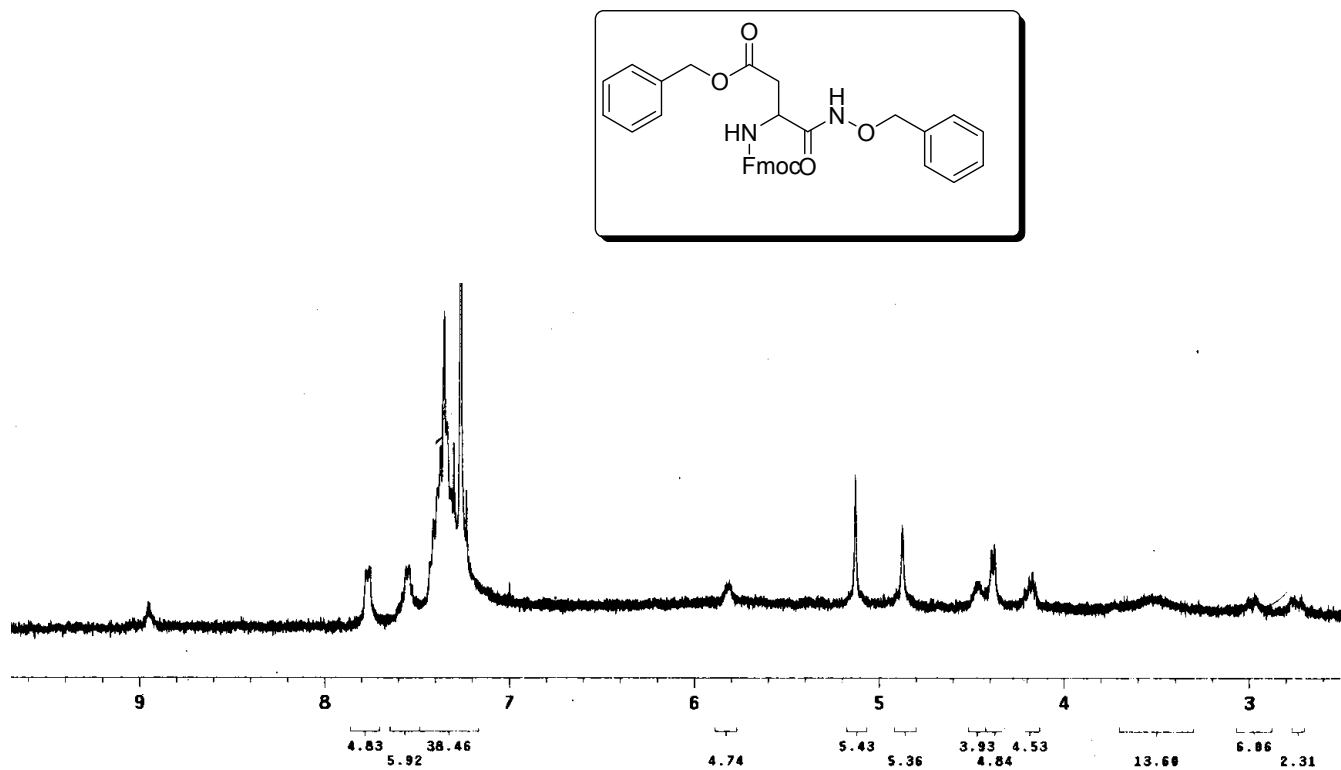


Figure S23. ^1H NMR spectra of Fmoc-Asp(OBzl)-NHOBn (entry 17, table 2)

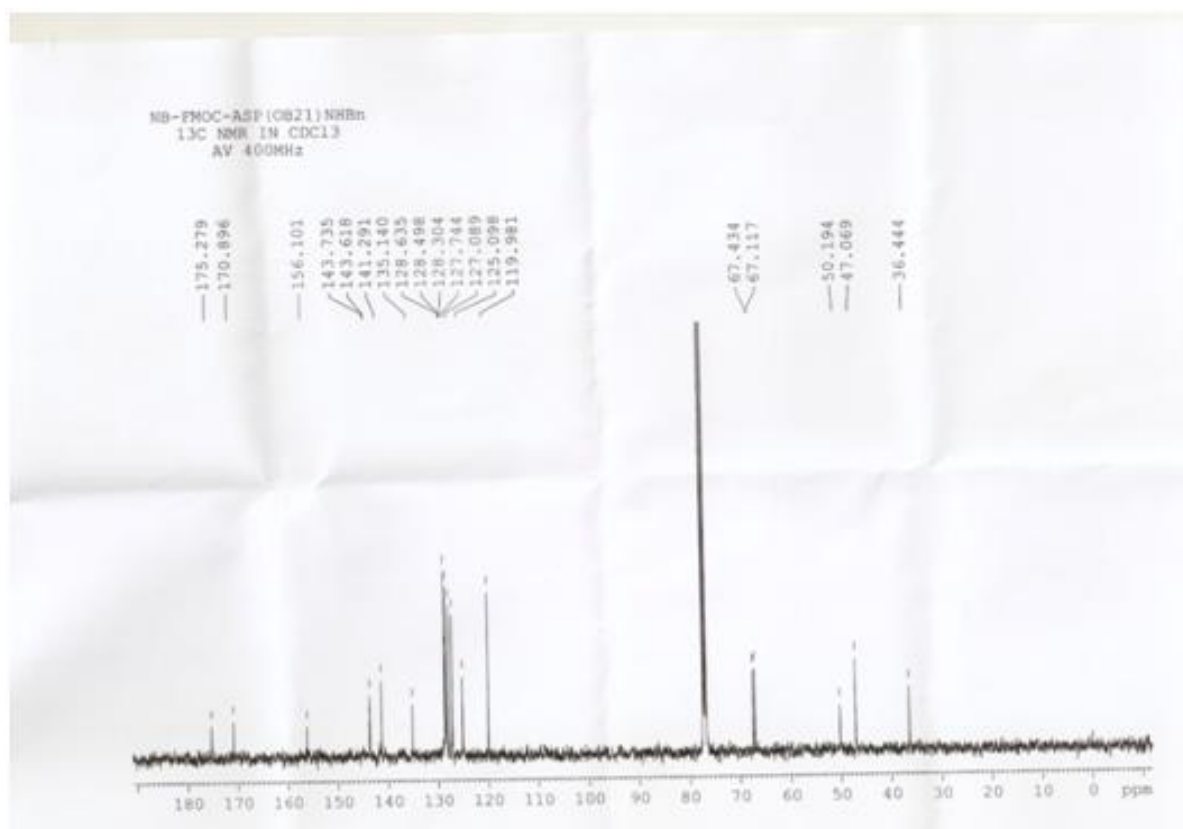


Figure S24. ¹³C NMR spectra of Fmoc-Asp(OBzl)-NHOBn (entry 17, table 2)

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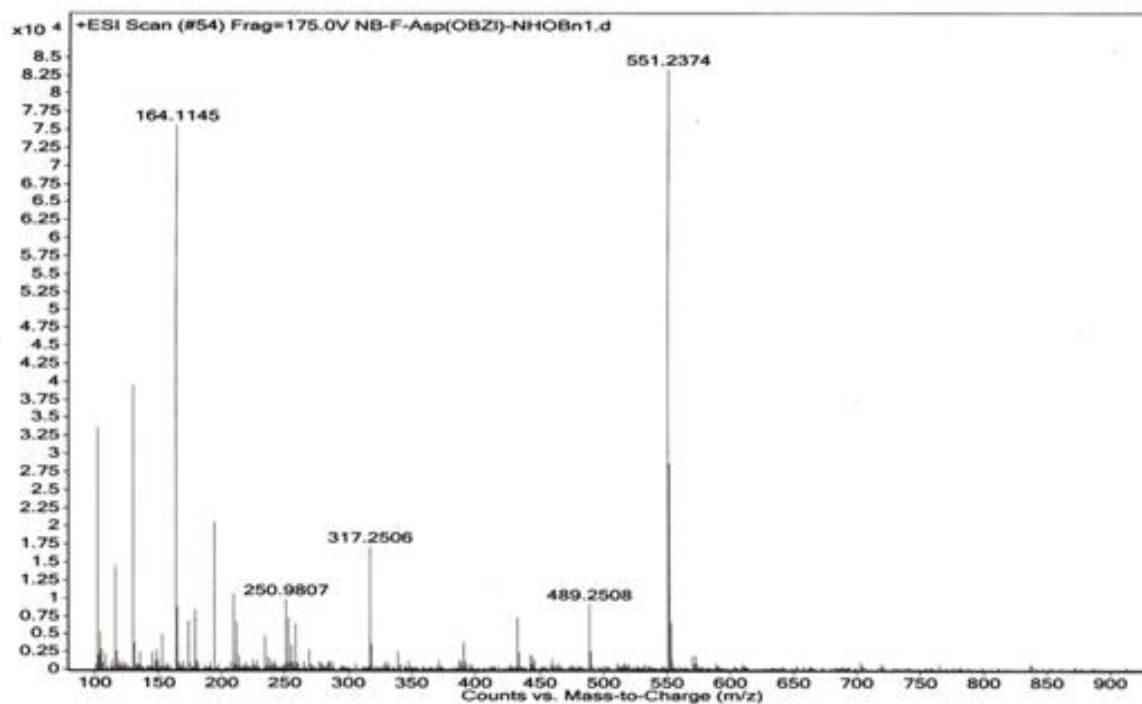


Figure S25. HRMS spectra of Fmoc-Asp(OBzl)-NHOBn Calcd. mass for $[M+H]^+$: 551.2182 found: 551.2374. (entry 17, table 2)

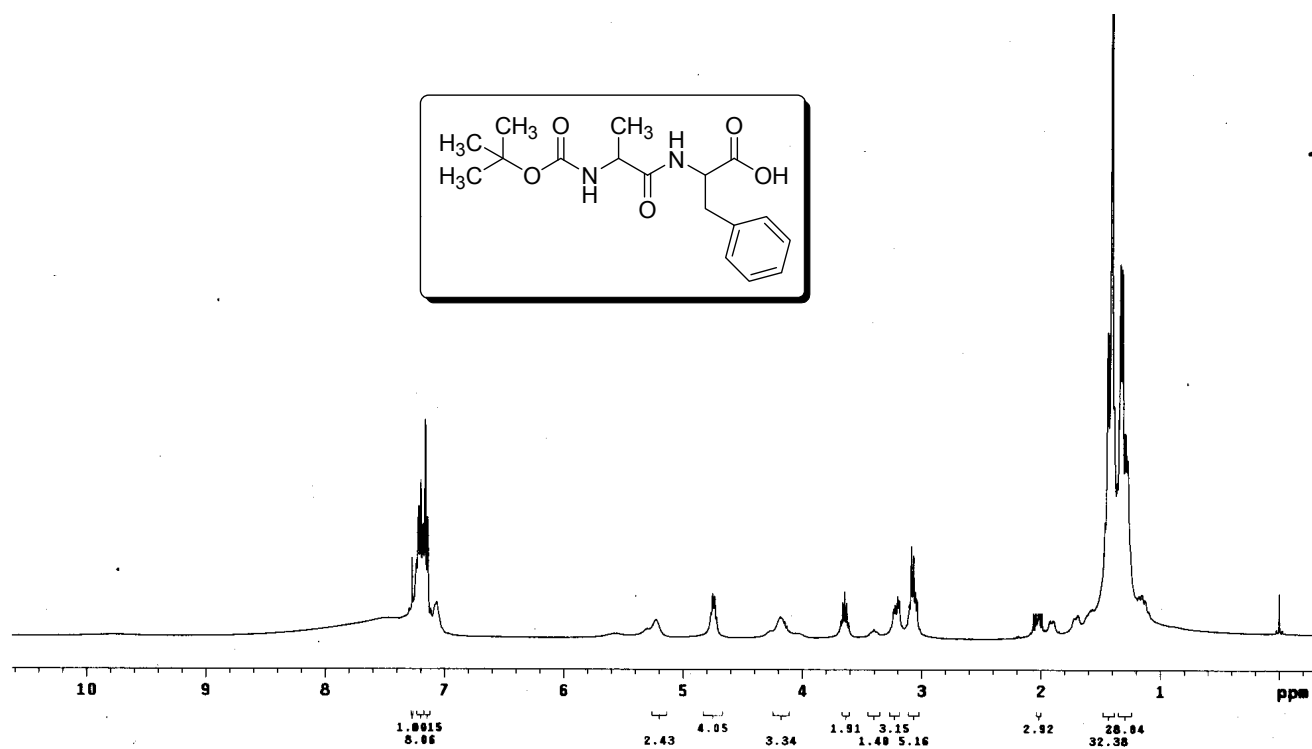


Figure S26. ¹H NMR spectra of Boc-Ala-Phe-OH

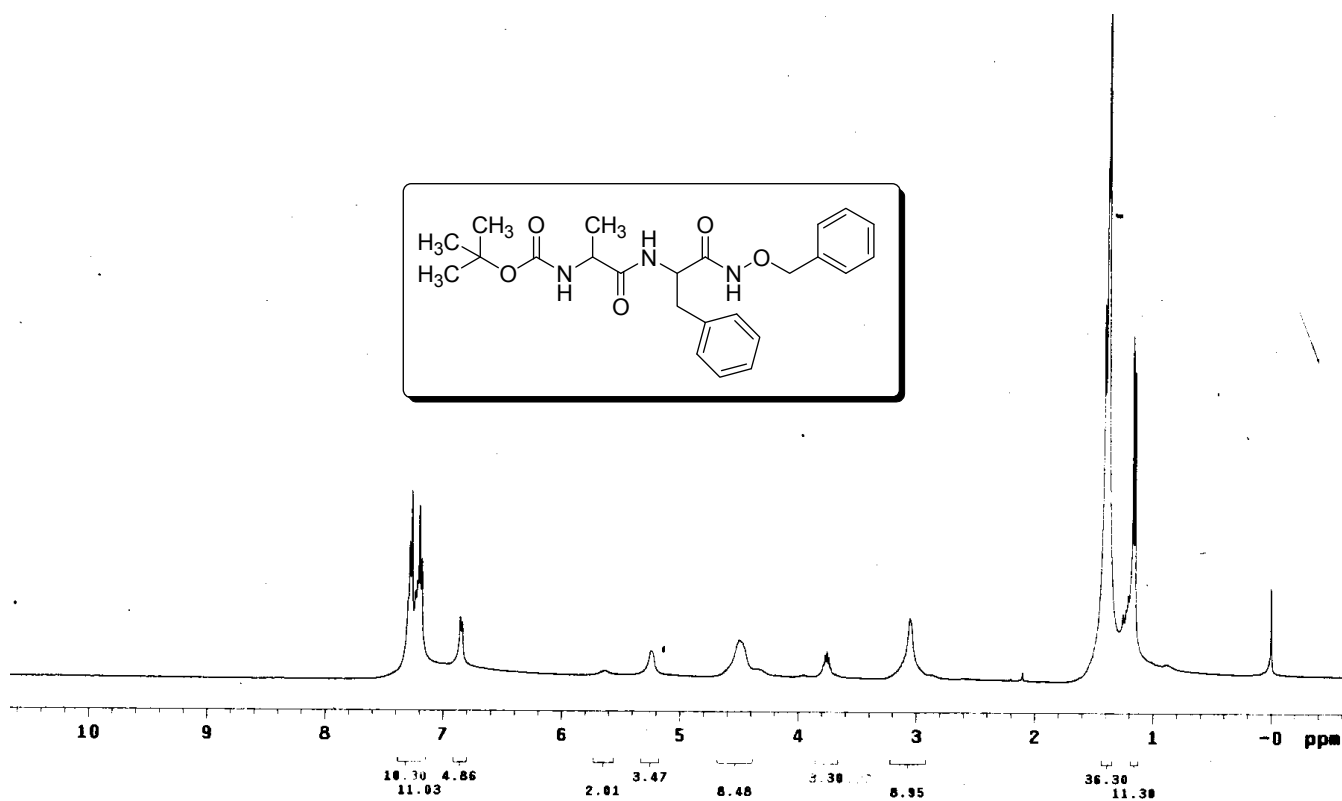


Figure S27. ¹H NMR spectra of Boc-Ala-Phe-NHOBn (entry 1, table 3)

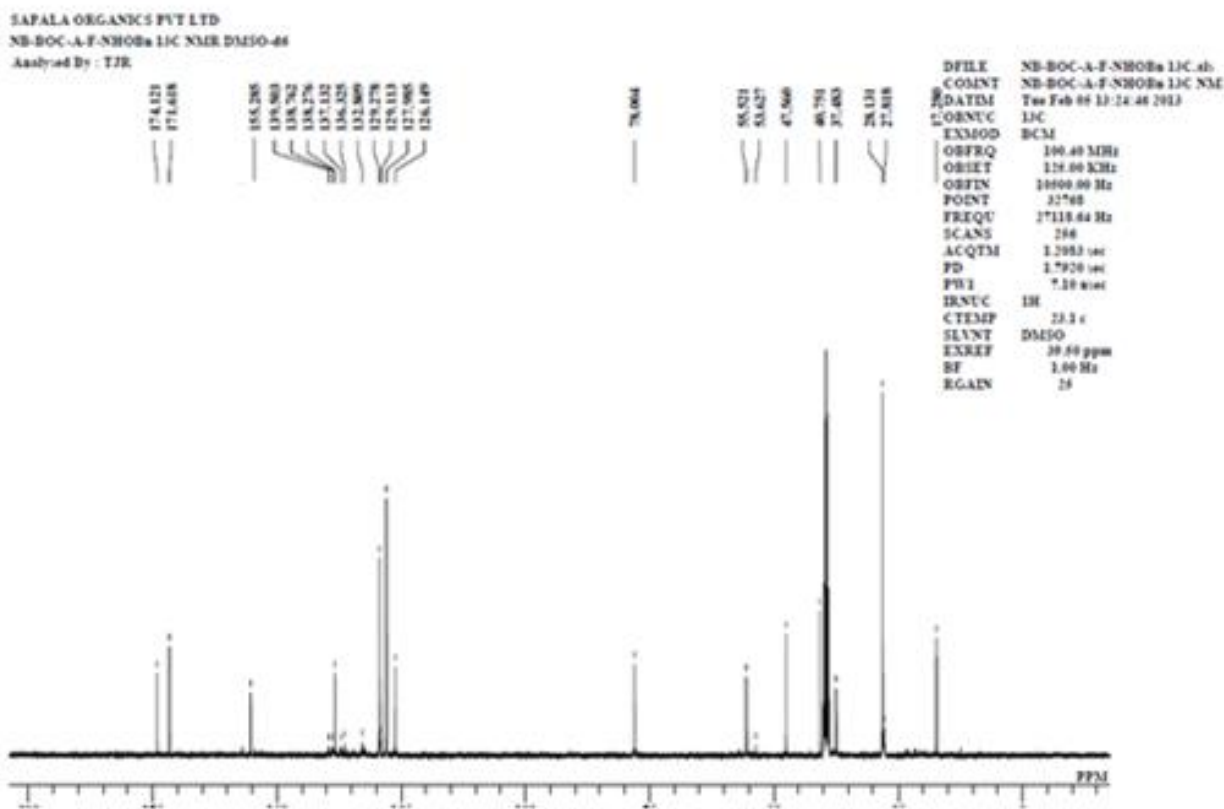


Figure S28 ¹³C NMR spectra of Boc-Ala-Phe-NHOBn (entry 1, table 3)

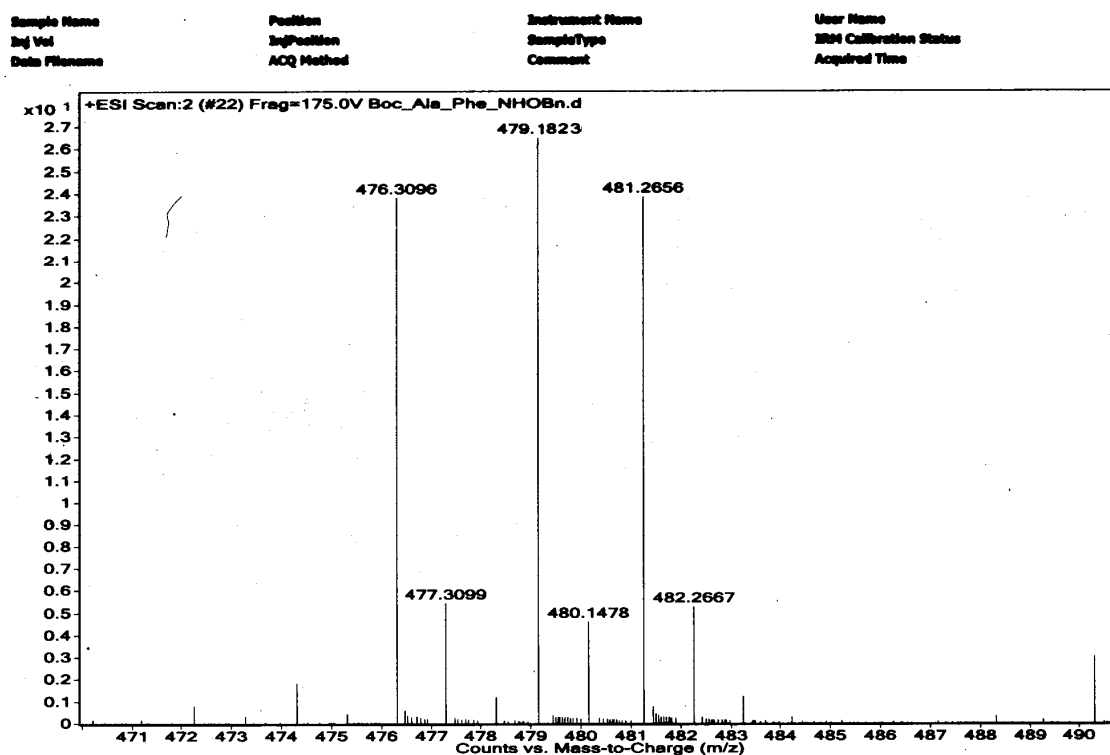


Figure S29. HRMS spectra of Boc-Ala-Phe-NHOBn Calcd. mass for $[M+K]^+$: 479.1826 found: 479.1823. (entry 1, table 3)

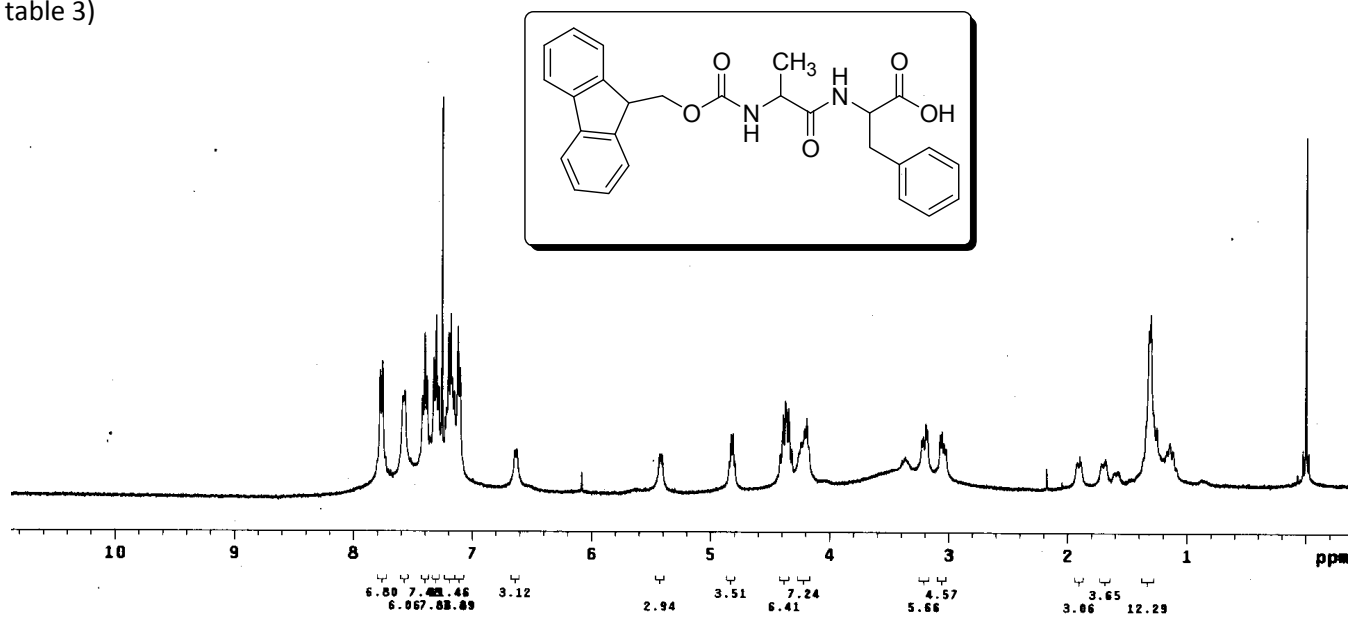


Figure S30. ¹H NMR spectra of Fmoc-Ala-Phe-OH

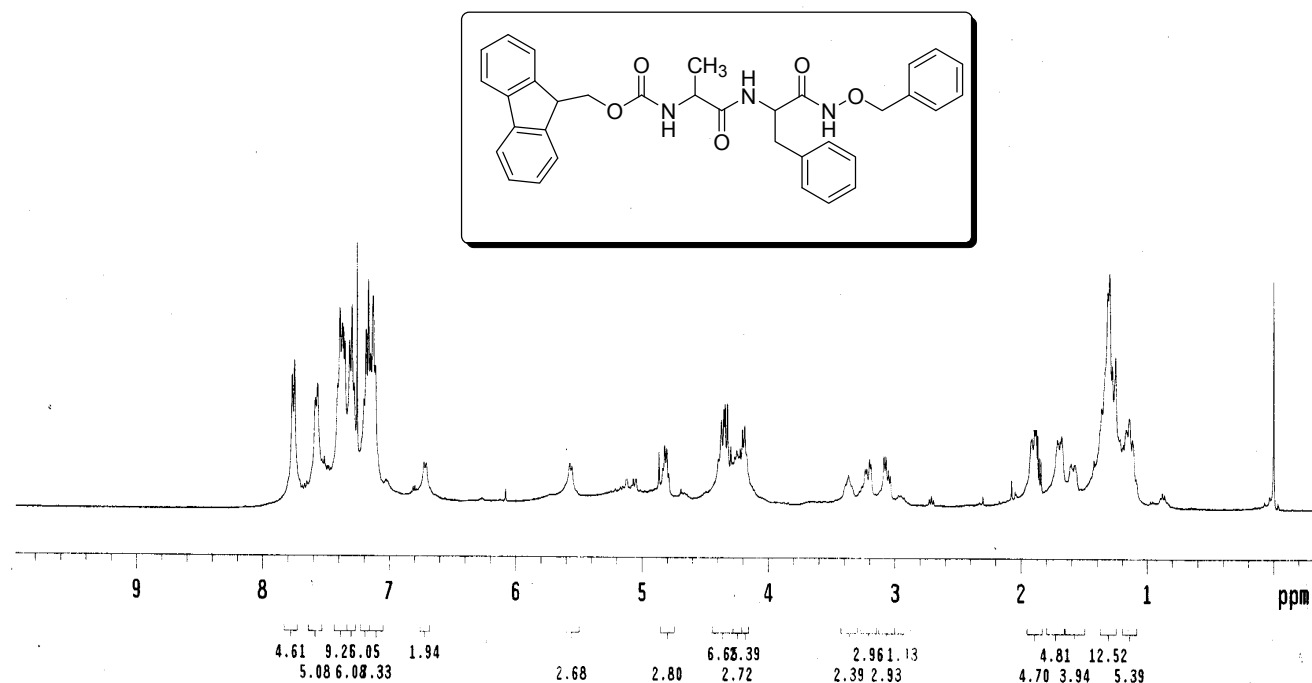


Figure S31. ¹H NMR spectra of Fmoc-Ala-Phe-NHOBn (entry 20, table 3)

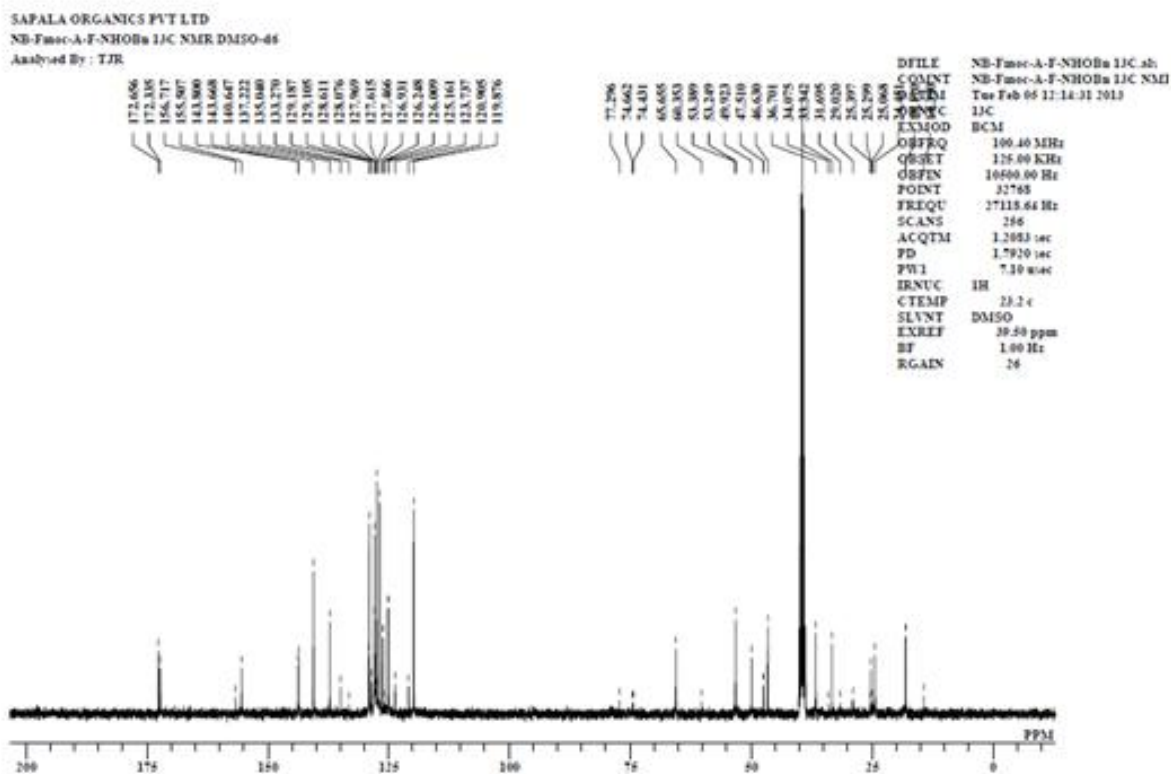


Figure S32. ¹³C NMR spectra of Fmoc-Ala-Phe-NHOBn (entry 20, table 3)

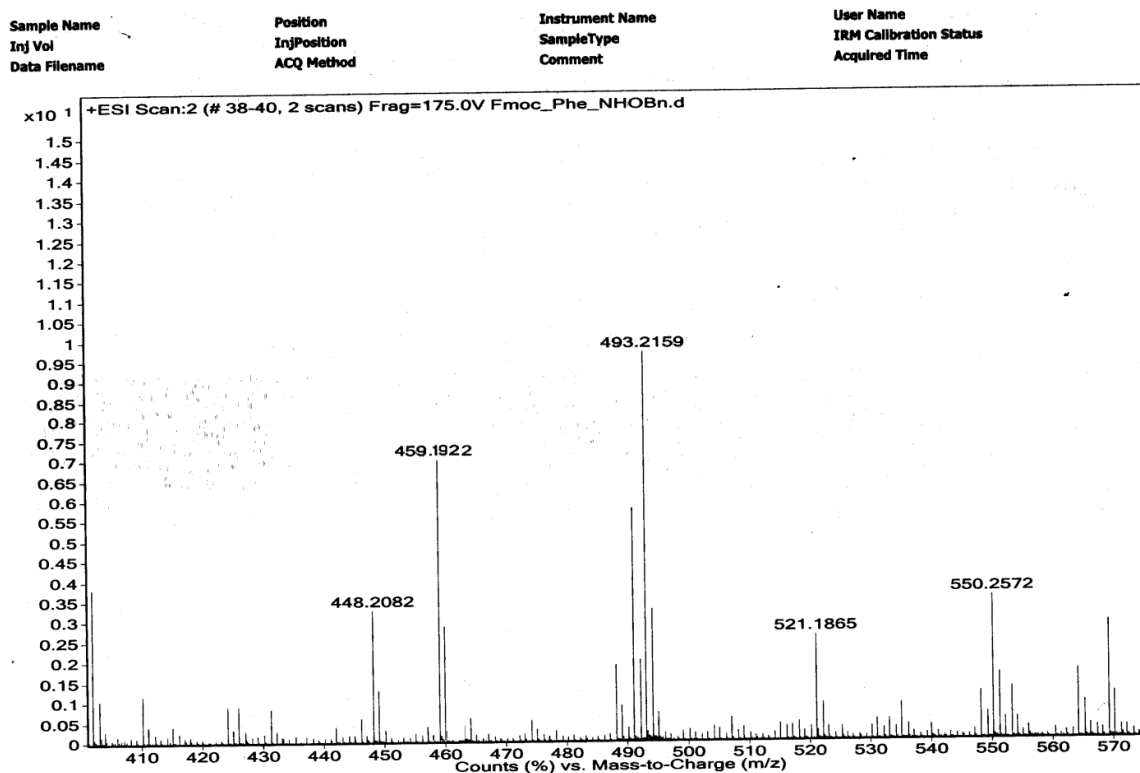


Figure S33. HRMS spectra of Fmoc-Ala-Phe-NHOBn Calcd. mass for $[M+H]^+$ 564.2498 found: 564.2456. (entry 16, table 2)

Racemization study:

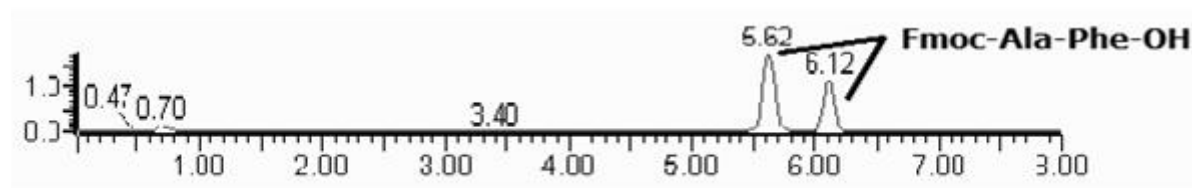


Figure S34. LC-MS Chromatograms for the compound Fmoc-Ala-Phe-OH using the acetonitrile and Millipore water as solvent with 0.1% formic acid.

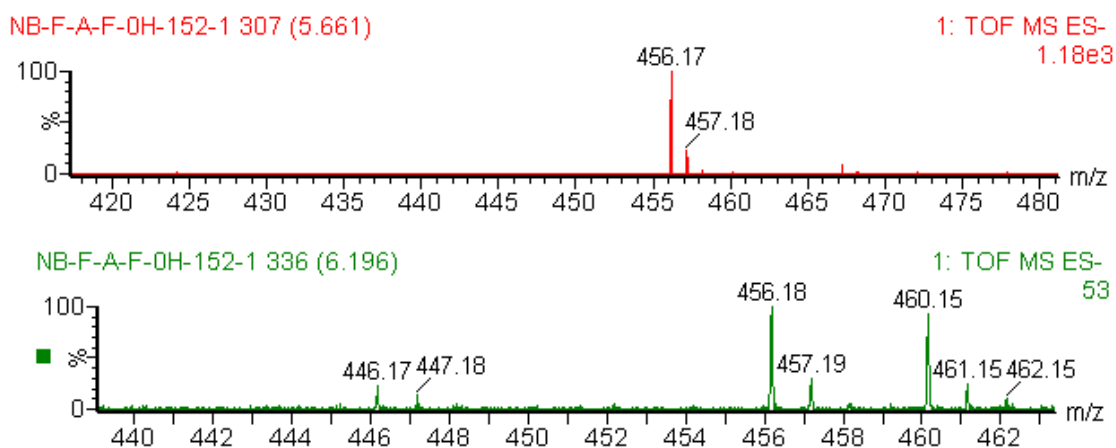


Figure S35. MS spectra for the compound Fmoc-Ala-Phe-OH at two different retention times (Rt 5.6, upper panel) and (Rt 6.1, lower panel). Calcd. mass for $[M+H]^+$ 457.17 found: 457.18.

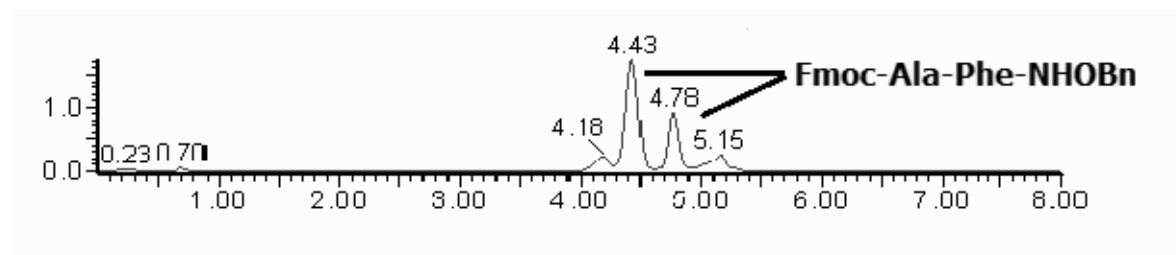


Figure S36. LC-MS Chromatograms for the compound Fmoc-Ala-Phe-NHOBn using the acetonitrile and Millipore water as solvent with 0.1% formic acid

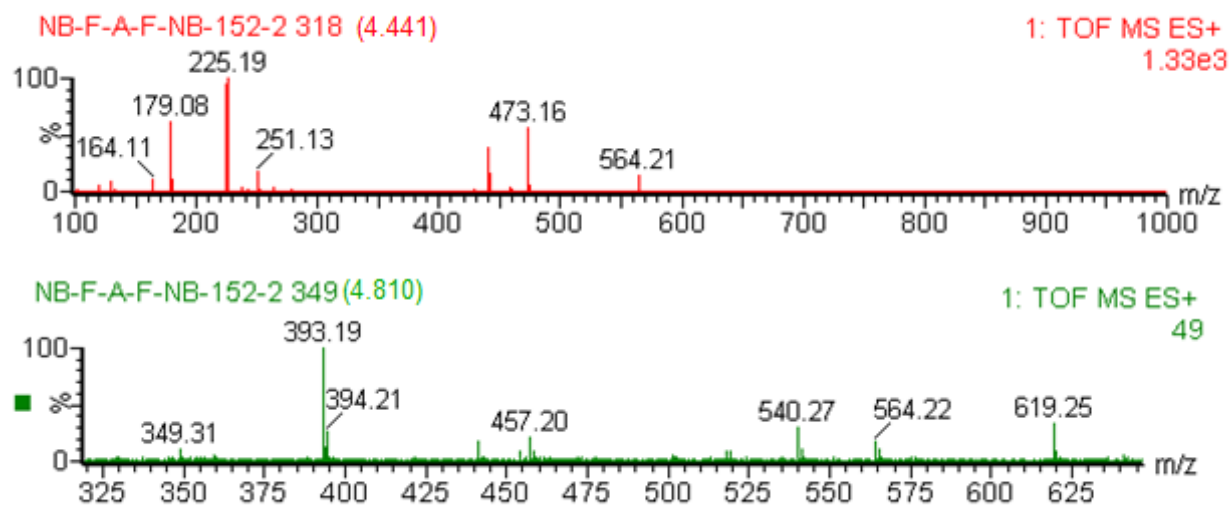


Figure S37. MS spectra for the compound Fmoc-Ala-Phe-NHOBn, at two different retention times (Rt 4.4, upper panel) and (Rt 4.8, lower panel). Calcd. mass for $[M+H]^+$ 564.24 found: 564.22.

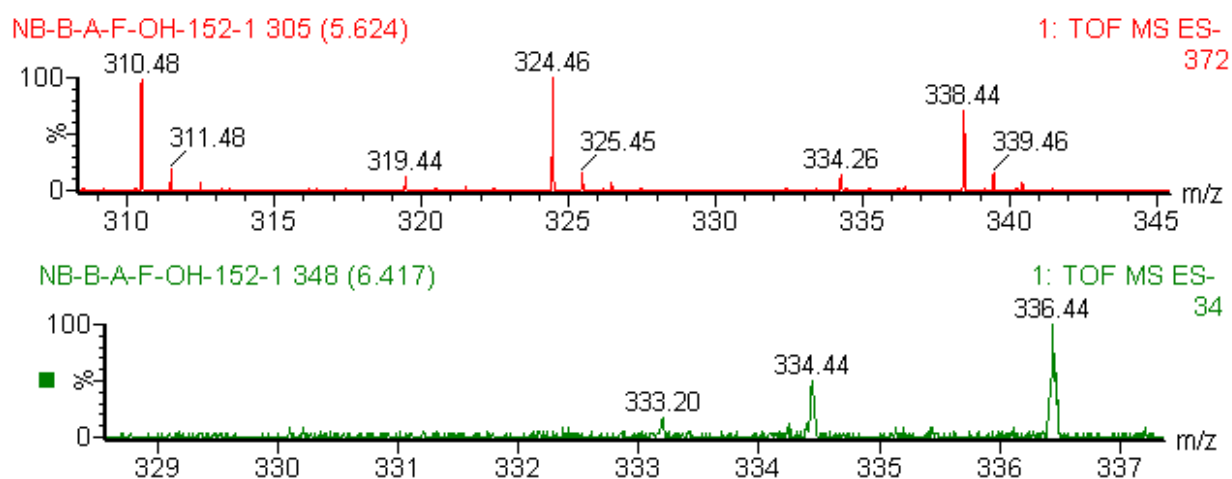


Figure S38. MS spectra for the compound Boc-Ala-Phe-OH, at two different retention times (Rt 5.6 min, upper panel) and (Rt 6.4 min, lower panel). Calcd. mass for $[M+H]^+$ 334.16 found: 334.26.

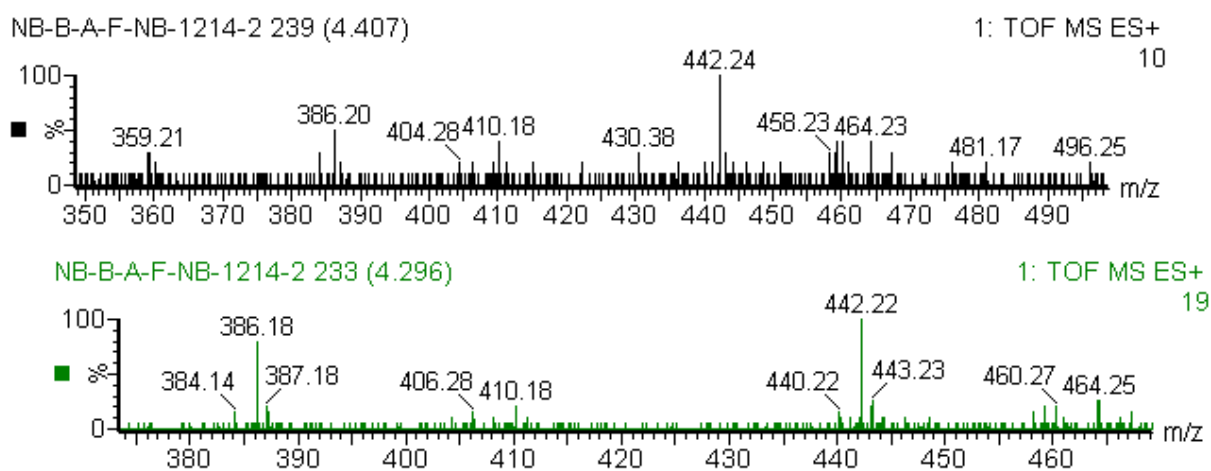


Figure S39. MS spectra for the compound Boc-Ala-Phe-NHOBn, at two different retention times. Calcd. mass for $[M+H]^+$ 442.23; found: 442.22.

Mechanism study:

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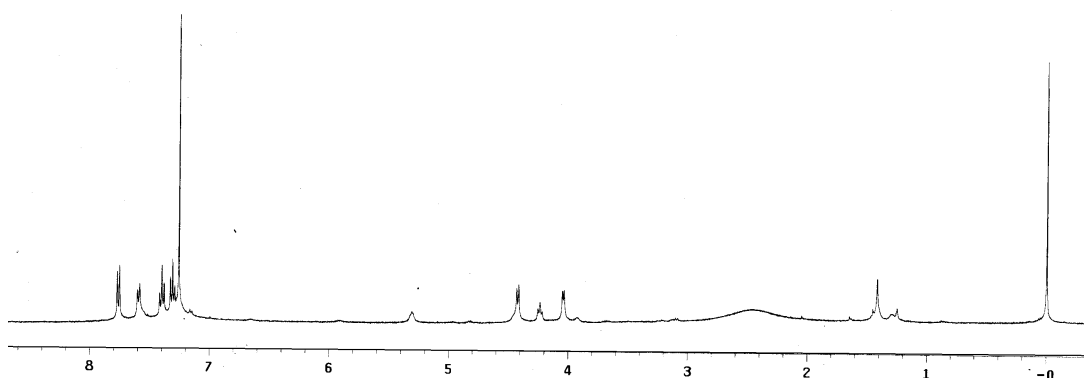


Figure S40. ¹H-NMR spectra of Fmoc-Gly-OH

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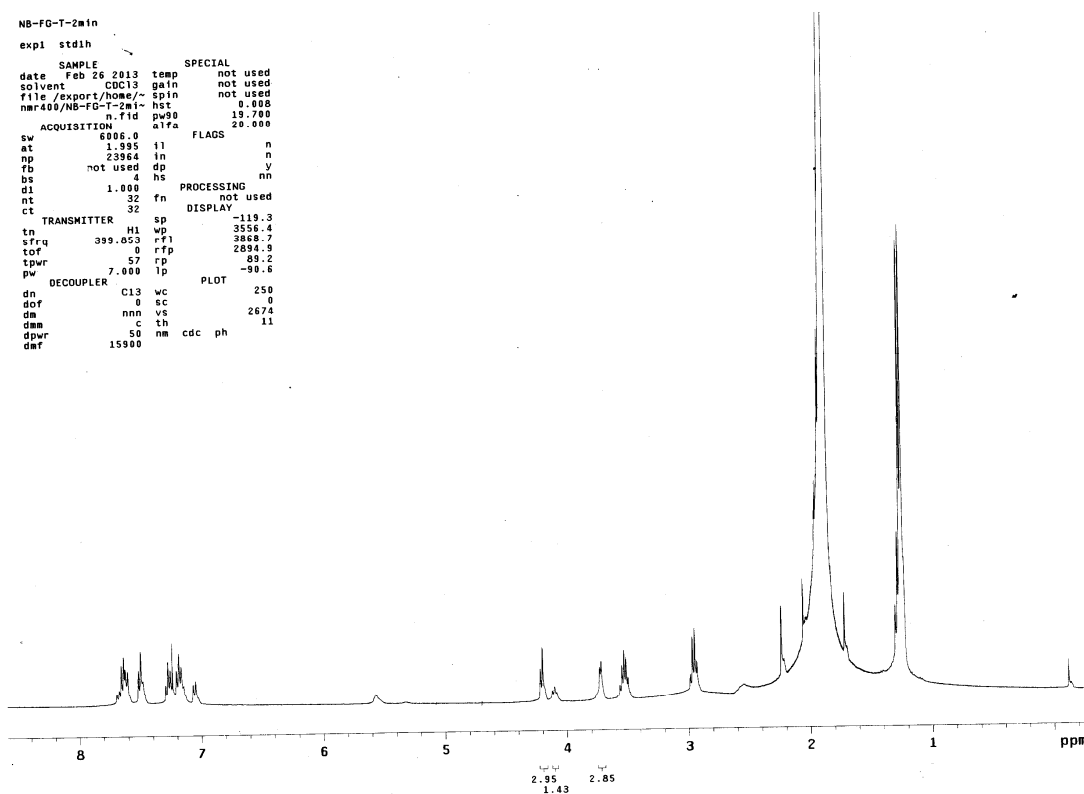


Figure S41. ¹H-NMR spectra of Fmoc-Gly-OH + TsOBt (after 2 min.) along with DIPEA (2 equiv.)

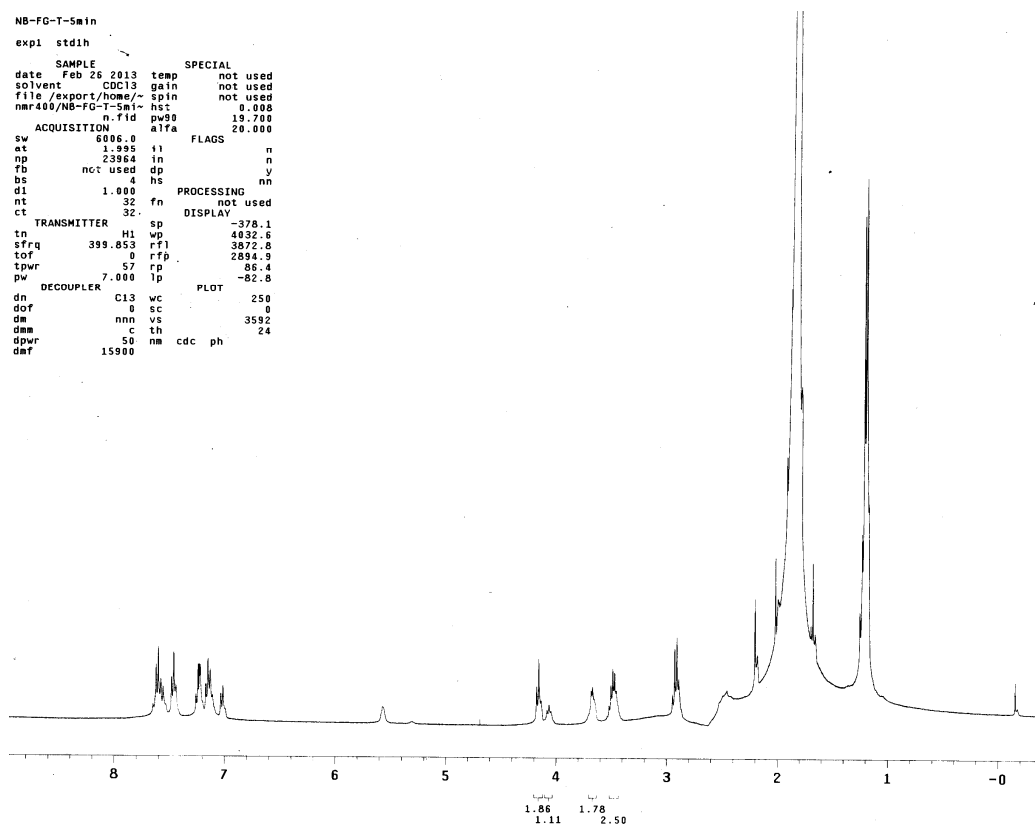


Figure S42. ¹H-NMR spectra of Fmoc-Gly-OH + TsOBt (after 5 min.) along with DIPEA (2 equiv.)

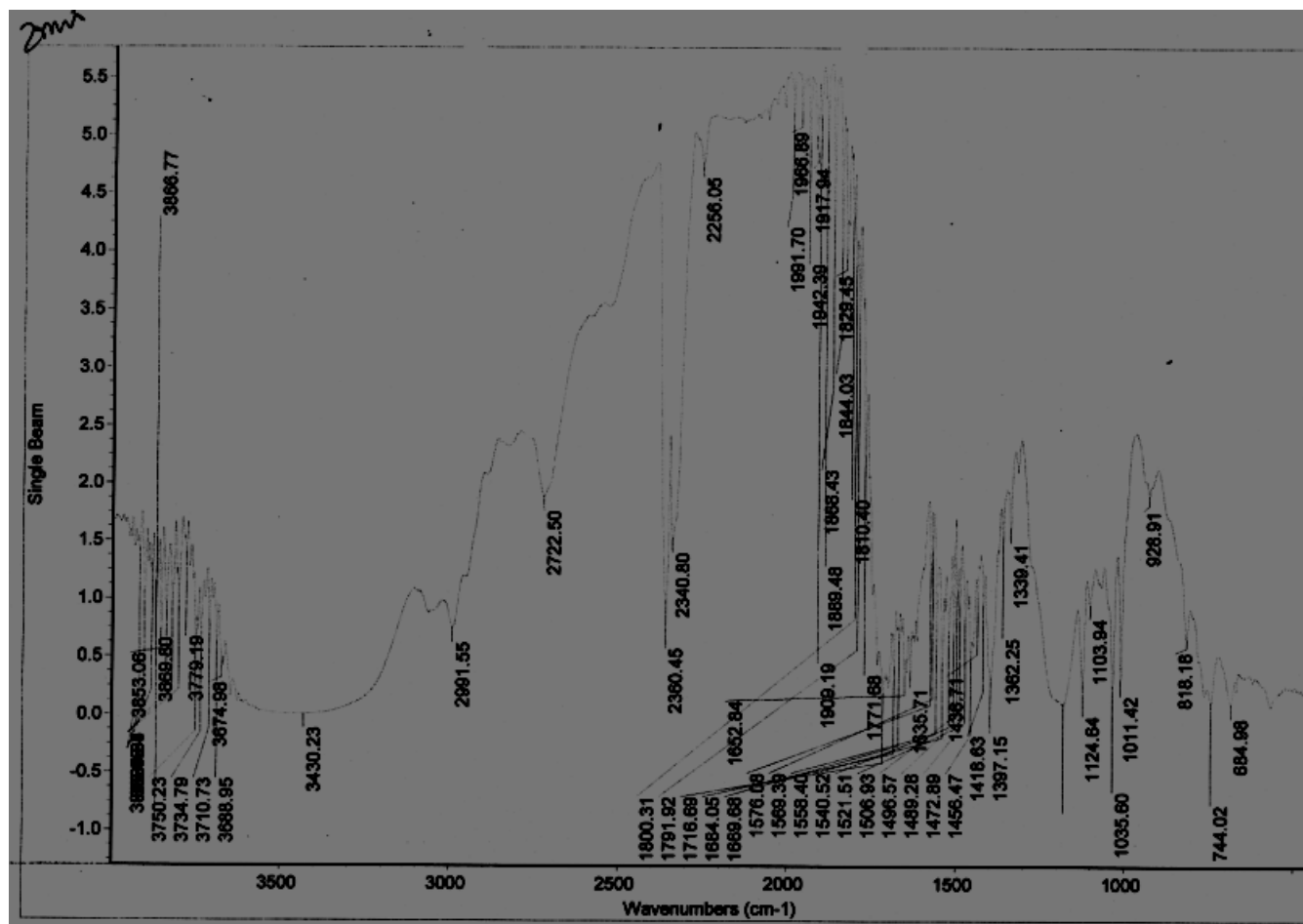


Figure S43. IR spectra of Fmoc-Gly-OH + TsOBt (after 2 min.) along with DIPEA (2 equiv.)

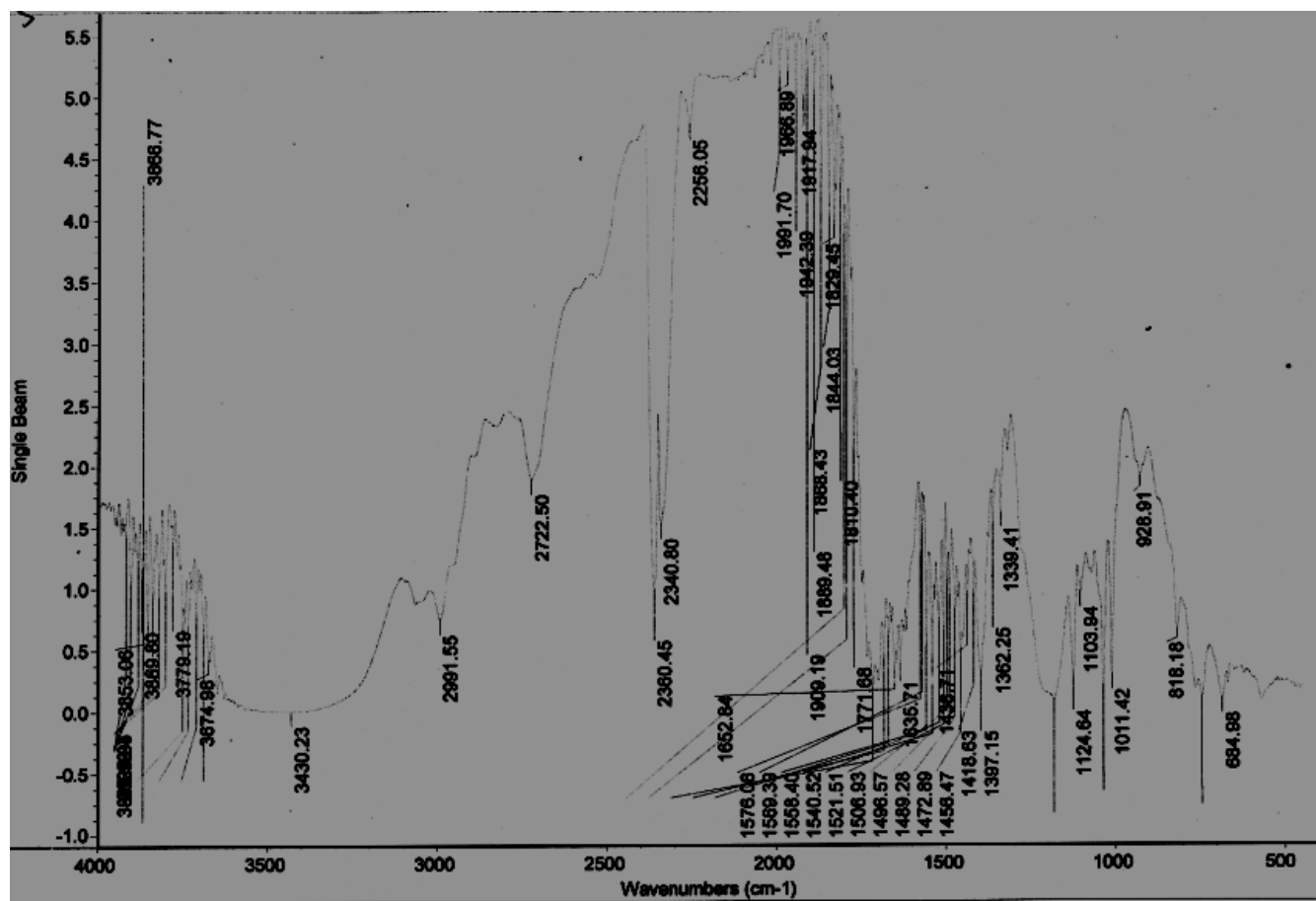


Figure S44. IR spectra of Fmoc-Gly-OH + TsOBt (after 5 min.) along with DIPEA (2 equiv.)