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

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Supporting Information

Figure S1. $^1$H NMR spectra of Ac-NHOBn (entry 1, table 2)
Figure S2. $^1$H NMR spectra of BzCONHOBn (entry 2, table 2)

Figure S3. $^1$H NMR spectra of $m$-Cl$_2$-C$_6$H$_4$-CONHOBn (entry 3, table 2)
**Figure S4.** $^1$H NMR spectra of $p$-NO$_2$-C$_6$H$_4$-CONHOBn (entry 4, table 2)

**Figure S5.** $^1$H NMR spectra of $m$-NO$_2$-C$_6$H$_4$-CONHOBn (entry 5, table 2)
Figure S6. $^1$H NMR spectra of BnCONHOBn (entry 6, table 2)

Figure S7. $^1$H NMR spectra of Boc-Gly-NHOBn (entry 7, table 2)
Figure S8. $^1$H NMR spectra of Boc-Ala-NHOBn (entry 8, table 2)

Figure S9. $^1$H NMR spectra of Cbz-Ala-NHOBn (entry 9, table 2)
**Figure S10.** $^1$H NMR spectra of Boc-Phe-NHOBn (entry 10, table 2)

**Figure S11.** $^1$H NMR spectra of Boc-Pro-NHOBn (entry 11, table 2)
Figure S12. $^1$H NMR spectra of Fmoc-Gly-NHOBn (entry 12, table 2)

Figure S13. $^1$H NMR spectra of Fmoc-Ala-NHOBn (entry 13, table 2)
Figure S14. H NMR Spectra of Fmoc-Phe-NHOBn (entry 14, table 2)

Figure S15. $^{13}$C 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.** ^1^H NMR spectra of Fmoc-Leu-NHOBn (entry 15, table 2)
Figure S18. $^{13}$C 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. $^{13}$C NMR spectra of Fmoc-Ser(tBu)-NHOBn (entry 16, table 2)

Figure S21. $^{13}$C 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. $^{13}$C NMR spectra of Fmoc-Asp(OBzl)-NHOBn (entry 17, table 2)

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. $^1$H NMR spectra of Boc-Ala-Phe-OH

Figure S27. $^1$H NMR spectra of Boc-Ala-Phe-NHOBn (entry 1, table 3)
Figure S28 $^{13}$C 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. $^{13}$C NMR spectra of Fmoc-Ala-Phe-NHBn (entry 20, table 3)

Figure S33. HRMS spectra of Fmoc-Ala-Phe-NHBn 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.
Mechanisms study:

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

Figure S41. $^1$H-NMR spectra of Fmoc-Gly-OH + TsOBt (after 2 min.) along with DIPEA (2 equiv.)
Figure S42. $^1$H-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.)