Supporting Information

Chemoselective N-tert-butyloxycarbonylation of amines in glycerol

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CONTENTS

<table>
<thead>
<tr>
<th>CONTENTS</th>
<th>PAGE</th>
</tr>
</thead>
<tbody>
<tr>
<td>General Information</td>
<td>S1</td>
</tr>
<tr>
<td>Experimental Section</td>
<td>S2</td>
</tr>
<tr>
<td>Characterization Data of the Products</td>
<td>S3-S11</td>
</tr>
<tr>
<td>Scans of $^1$H NMR and $^{13}$C NMR Spectra</td>
<td>S12-S73</td>
</tr>
</tbody>
</table>
EXPERIMENTAL SECTION

Materials and method

All chemicals were reagent grade, purchased from Aldrich and Alfa Aesar and were used without purification. Melting points were determined by open glass capillary method and were uncorrected. IR spectra were recorded on a Shimadzu FTIR Affinity-1 Fourier Transform Infrared spectrophotometer. NMR spectra were recorded on BRUKER AVANCE II 400 FT spectrometer (400 for $^1$H NMR, 100 MHz for $^{13}$C NMR) using tetramethylsilane (TMS) as the internal standard and CDCl$_3$ and DMSO-d$_6$ as solvent. The chirality was determined on a JASCO P-2000 polarimeter. All the reactions were monitored by TLC using precoated sheets of silica gel G/UV-254 plates. All the products are known compounds, and were characterized by melting points in comparison with the literature values, FTIR and $^1$H NMR and $^{13}$C NMR spectroscopy were used for structural identification.

General procedure for N-Boc protection of amines:-

A mixture of amine (1 mmol) and (Boc)$_2$O (1mmol) in glycerol (2.0 ml) was vigorously stirred at room temperature for appropriate time (Table 2 and 3) until complete disappearance of amines was observed in the TLC monitoring. After the completion of reactions, the reaction mixture was extracted with mixture of pet ether/ethyl acetate (9:1). The combined organic layer was dried over anhydrous sodium sulfate and concentrated under reduced pressure to give a crude product. The glycerol phase was dried under vacuum and reused without loss of activity. After removal of the solvent, the pure products were obtained and no recrystallization or column chromatography is needed. All the compounds were characterized by comparison with mp and IR, $^1$H NMR, $^{13}$C NMR spectra with literature.
Characterization Data of the Products:

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\text{Tert-butyl phenylcarbamate (Table 2, entry 1): White solid; m.p. 132-133^\circ\text{C}; Yield 98%; }^1\text{H NMR (400MHz, CDCl}_3\text{, TMS, ppm): } \delta 7.36-7.26(\text{m, 4H}), 7.05-7.03(\text{m, 1H}), 6.46(\text{bs, 1H}), 1.52(\text{s, 9H); }^{13}\text{C NMR (100 MHz, CDCl}_3\text{, ppm): } \delta 152.73, 138.30, 123.97, 123.01, 118.49, 80.49, 28.33.\]

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\text{Tert-butyl o-tolyldicarbamate (Table 2, entry 2): White solid; m.p.84-85^\circ\text{C; Yield 93%; }^1\text{H NMR (400 MHz, CDCl}_3\text{, TMS, ppm): } \delta 7.83(\text{d, J = 8Hz, 1H}), 7.22(\text{t, J = 8 Hz, 1H}), 7.17(\text{d, J = 8Hz, 1H}), 7.03(\text{t, J = 8Hz, 1H}), 6.37(\text{bs, 1H}), 2.28(\text{s, 3H}), 1.57(\text{s, 9H); }^{13}\text{C NMR (100 MHz, CDCl}_3\text{, ppm): } \delta 153.00, 136.20, 132.80, 130.20, 126.60, 123.60, 121.00, 80.20, 28.20, 17.60.\]

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\text{Tert-butyl m-tolyldicarbamate (Table 2, entry 3): White solid; Yield 95%; }^1\text{H NMR (400 MHz, CDCl}_3\text{, } \delta \text{ ppm) 7.28(s, 1H), 7.18(d, J = 8Hz, 1H), 7.13(d, J = 8Hz, 1H), 6.88(d, J = 8Hz, 1H), 6.53(bs, 1H), 2.35(s, 3H), 1.55(s, 9H); }^{13}\text{C NMR (100 MHz, CDCl}_3\text{, ppm): } \delta 152.80, 138.80, 138.20, 128.70, 123.80, 119.10, 115.60, 80.30, 28.30, 21.40.\]
**Tert-butyl p-tolylcarbamate (Table 2, entry 4):** White solid; m.p. 81-82°C; Yield 98%; $^1$H NMR (400 MHz, CDCl$_3$, TMS, ppm): $\delta$ 7.25(d, $J$ = 8Hz, 2H), 7.08(d, $J$ = 8Hz, 2H), 6.42(bs, 1H), 2.29(s, 3H), 1.51(s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): $\delta$ 152.90, 135.71, 132.53, 129.66, 80.30, 28.35, 20.72.

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\text{NHBoc} \\
\text{OMe}
\]

**Tert-butyl (4-methoxyphenyl)carbamate (Table 2, entry 5):** White solid; m.p. 94-96°C; Yield 98%; $^1$H NMR (400 MHz, CDCl$_3$, ppm): $\delta$ 7.26(d, $J$ = 8Hz, 2H), 6.82(d, $J$ = 8Hz, 2H), 6.44(bs, 1H), 3.77(s, 3H), 1.50(s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): $\delta$ 155.60, 153.20, 131.40, 120.50, 114.10, 80.10, 55.40, 28.30.

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\text{NHBoc} \\
\text{OH}
\]

**Tert-butyl (4-hydroxyphenyl)carbamate (Table 2, entry 6):** White solid; m.p. 145-147°C; Yield 96%; $^1$H NMR (400 MHz, CDCl$_3$, TMS, ppm): $\delta$ 7.19(d, $J$ = 8Hz, 2H), 6.75(d, $J$ = 8Hz, 2H), 6.40(bs, 1H), 4.90(bs, 1H), 1.51(s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): $\delta$ 153.63, 152.12, 130.84, 121.71, 115.75, 80.46, 28.38.

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\text{NHBoc} \\
\text{NH}_2
\]

**Tert-butyl (4-aminophenyl)carbamate (Table 2, entry 8):** White solid; m.p. 110-113°C; Yield 97%; $^1$H NMR (400 MHz, CDCl$_3$, TMS, ppm): $\delta$ 7.13(d, $J$ = 8Hz, 2H), 6.64(d, $J$ = 8Hz, 2H),

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6.30(bs, 1H), 3.20(bs, 2H), 1.50(s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): δ 153.34, 142.38, 129.71, 120.91, 115.62, 80.01, 28.40.

Tert-butyl (2-aminophenyl)carbamate (Table 2, entry 9): White solid; m.p. 109-111°C; Yield 93%; $^1$H NMR (400 MHz, CDCl$_3$, ppm): δ 7.25(d, $J = 8$Hz, 1H), 7.01(m, 1H), 6.80(m, 2H), 6.22(bs, 1H), 3.72(bs, 2H), 1.51(s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): δ 153.94, 139.18, 126.20, 125.09, 124.79, 120.13, 117.98, 80.67, 28.34.

Tert-butyl (4-chlorophenyl)carbamate (Table 2, entry 10): White solid; m.p. 102-103°C; Yield $^1$H NMR (400 MHz, CDCl$_3$, ppm): δ 7.29-7.32(d, $J = 9$Hz, 2H), 7.24(d, $J = 8$Hz, 2H), 6.52(bs, 1H), 1.51(s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): δ 151.80, 144.40, 142.70, 125.10, 117.40, 81.90, 28.10.

Tert-butyl (4-bromophenyl)carbamate (Table 2, entry 11): White solid; m.p. 101-103°C; Yield; $^1$H NMR (400 MHz, CDCl$_3$, TMS, ppm): δ 7.39(d, $J = 8$Hz, 2H), 7.25(d, $J = 8$Hz, 2H), 6.47(bs, 1H), 1.51(s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): δ 152.50, 137.46, 131.89, 120.02, 115.43, 80.92, 28.31.
Tert-butyl (4-nitrophenyl)carbamate (Table 2, entry 12): Yellowish Oil, b.p. 308-312°C; Yield 84%; $^1$H NMR (300 MHz, CDCl$_3$, TMS, ppm): $\delta$ 8.19-8.16(d, J =8Hz, 2H), 7.54-7.51(d, J =8Hz, 2H), 6.89(bs, 1H), 1.47(s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): $\delta$ 152.70, 137.60, 126.30, 125.3, 113.00, 51.50, 29.10.

![NHBoc](image1)

Tert-butyl (2-nitrophenyl)carbamate (Table 2, entry 13): Yellow solid; m.p. 89-90°C; Yield 80% $^1$H NMR (400 MHz, CDCl$_3$, TMS, ppm): $\delta$ 9.65(bs, 1H), 8.55(d, J = 8Hz, 1H), 8.18(d, J = 8Hz, 1H), 7.60(t, J = 8Hz, 1H), 7.08(t, J = 8Hz, 1H), 1.54 (s, 9H); $^{13}$C NMR (100MHz, CDCl$_3$, ppm): $\delta$ 152.20, 135.90, 135.70, 125.80, 121.80, 120.70, 81.80, 28.20.

![NHBoc](image2)

Tert-butyl (3-nitrophenyl)carbamate (Table 2, entry 14): Yellow solid; m.p. 180-182 °C; Yield 87%; $^1$H NMR (300 MHz, CDCl$_3$, TMS, ppm): $\delta$ 8.30(s, 1H), 7.88(d, J = 9Hz, 1H), 7.68 (d, J = 3Hz, 1H), 7.44(t, J = 9Hz, 1H), 6.72(bs, 1H), 1.54(s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): $\delta$ 152.40, 149.20, 139.90, 129.90, 124.10, 117.80, 113.40, 81.80, 28.50.

![NHBoc](image3)

Tert-butyl naphthalen-1-ylcarbamate (Table 2, entry 15): White solid; m.p. 96-99°C; Yield 93%; $^1$H NMR (400 MHz, CDCl$_3$, TMS, ppm): $\delta$ 7.86(m, 3H), 7.61(m, 1H), 7.50-7.44(m, 3H), 6.86(bs, 1H), 1.56(S, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): $\delta$ 153.52, 146.76, 134.08, 132.94, 126.04, 124.46, 120.44, 118.64, 80.69, 28.39.

![NHBoc](image4)
Tert-butyl naphthalen-2-ylcarbamate (Table 2, entry 16): Yellow solid; m.p. 85-86°C; Yield 94%; ¹H NMR (400 MHz, CDCl₃, TMS, ppm): δ 8.00(s, 1H), 7.77-7.75(m, 3H), 7.46-7.43(t, J = 8Hz, 1H), 7.39-7.33(m, 2H), 6.66(bs, 1H), 1.57(s, 9H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 153.1, 136.1, 134.3, 130.3, 128.9, 127.7, 127.6, 126.6, 124.7, 119.4, 114.8, 80.8, 28.6.

Tert-butyl pyridin-2-ylcarbamate (Table 2, entry 17): ¹H NMR (400 MHz, CDCl₃, TMS, ppm): δ 11.23(s, 1H), 7.91(d, J = 8Hz, 1H), 7.81(d, J = 8Hz, 1H), 7.39(d, J = 8Hz, 1H), 7.28(dd, J = 8 & 4Hz, 1 H), 1.59(s, 9H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 161.37, 152.85, 152.62, 147.52, 138.34, 121.00, 80.87, 28.37

Tert-butyl thiazol-2-ylcarbamate (Table 2, entry 18): White solid; m.p. 137-145°C; Yield 96%; ¹H NMR (400 MHz, CDCl₃, TMS, ppm): δ 7.32(d, J = 4Hz, 1H), 6.82(d, J = 4Hz, 1H), 1.52 (s, 9H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 161.80, 152.90, 136.70, 112.10, 82.00, 28.30.

Tert-butyl benzo[d]thiazol-2-ylcarbamate (Table 2, entry 19): White solid; m.p. 98-100°C; 93%; ¹H NMR (400 MHz, d₆-DMSO, TMS, ppm): δ 11.70(bs, 1H), 7.91(d, J = 8Hz, 1H), 7.79(d, J = 8Hz, 1H), 7.39(d, J = 8Hz, 1H), 7.28(dd, J = 8 & 4Hz, 1H), 1.59(s, 9H); ¹³C NMR (100 MHz, d₆-DMSO, ppm): δ 161.56, 152.83, 148.67, 131.50, 125.68, 123.37, 121.04, 120.86, 83.25, 28.35.
**Tert-butyl 1H-benzo[d]imidazol-2-ylcarbamate (Table 2, entry 20):** White solid; m.p. 208-210°C; Yield 91%; $^1$H NMR (400 MHz, CDCl$_3$, TMS, ppm): $\delta$ 7.61(d, J = 8Hz, 1H), 7.3(d, J = 8Hz, 1H), 7.20(t, J = 8Hz, 1H), 7.05(t, J = 8Hz, 1H), 6.54 (s, 1H), 1.71(s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): $\delta$ 153.90, 150.70, 142.10, 130.30, 124.4, 120.70, 116.30, 113.90, 85.90, 28.10.

**Tert-butyl butylcarbamate (Table 3, entry 1):** Colorless oil; Yield 98%; $^1$H NMR (400 MHz, CDCl$_3$, TMS, ppm): $\delta$ 4.49(bs, 1H), 3.10(t, J = 8Hz, 2H), 1.48(s, 9H), 1.48-1.35(m, 2H), 1.34-1.28(m, 2H), 0.91(t, J = 8Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): $\delta$ 156.00, 79.00, 40.40, 32.20, 28.40, 19.90, 13.70.

**Tert-butyl (2-hydroxyethyl)carbamate (Table 3, entry 2):** Colorless oil; b.p. 90-92°C; Yield 97%; $^1$H NMR (400 MHz, CDCl$_3$, TMS, ppm): $\delta$ 5.23(bs, 1H), 3.66(t, 2H), $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): $\delta$ 156.84, 79.62, 62.09, 43.00, 28.35.

**Tert-butyl (2-aminoethyl)carbamate (Table 3, entry 3):** Colorless oil; b.p. 70-73°C; Yield 97%; $^1$H NMR (400 MHz, CDCl$_3$, TMS, ppm): $\delta$ 4.99(bs, 1H), 3.20(t, J = 4Hz, 2H), 2.82(t, J = 4Hz, 2H), 2.05(s, 2H), 1.43(s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): $\delta$ 156.02, 79.07,41.63, 40.37, 28.40.

**Tert-butyl (3-aminopropyl)carbamate (Table 3, entry 4):** Colorless oil; b.p. 200-202°C; Yield 96%; $^1$H NMR (400 MHz, CDCl$_3$, TMS, ppm): $\delta$ 4.90(bs, 1H), 3.21(t, J = 4Hz, 2H), 2.76(t, J =
Tert-butyl cyclohexylcarbamate (Table 3, entry 5): White solid; m.p. 78-81°C; Yield 98%; \(^1\)H NMR (400 MHz, CDCl\(_3\), TMS, ppm): δ 4.40(bs, 1H), 3.41(bs, 1H), 1.92-1.89 (m, 2H), 1.70-1.59(m, 3H), 1.43(s, 9H), 1.33-1.27 (m, 2H), 1.18-1.04 (m, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\), ppm): δ 155.50, 79.20, 49.70, 33.70, 28.60, 25.80, 25.10.

Tert-butyl benzylcarbamate (Table 3, entry 6): White solid; m.p. 54-56°C; Yield 98%; \(^1\)H NMR (400 MHz, CDCl\(_3\), TMS, ppm): 7.34-7.24(m, 5H), 4.90(bs, 1H), 4.31(s, 2H), 1.46(s, 9H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\), ppm): 155.90, 146.74, 138.90, 128.58, 127.47, 127.30, 79.45, 44.66, 28.40.

Tert-butyl (pyridin-3-ylmethyl)carbamate (Table 3, entry 7): Colorless oil; Yield 96%; \(^1\)H NMR (400 MHz, TMS, CDCl\(_3\), ppm): δ 8.52-8.49(m, 2H), 7.64(d, J = 8Hz, 1H), 7.28-7.25(m, 1H), 5.18(bs, 1H), 4.33(s, 2H) 1.45(s, 9H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\), ppm): δ 155.90, 148.80, 148.50, 135.30, 134.70, 123.50, 79.80, 42.10, 28.30.

Tert-butyl (pyridin-2-ylmethyl)carbamate (Table 3, entry 8): Colorless oil; Yield 95%; \(^1\)H NMR (400 MHz, CDCl\(_3\), TMS, ppm): δ 8.52(s, 1H), 7.65(d, J = 8Hz, 1H), 7.27(d, J = 4Hz, 1H),
7.17(d, J = 4Hz, 1H), 5.89(bs, 1H), 4.45(S, 2H), 1.46(S, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): δ 157.65, 156.05, 148.95, 136.66, 122.10, 121.56, 79.29, 45.68, 28.34.

Tert-butyl piperidine-1-carboxylate (Table 3, entry 9): Colorless oil; Yield 91%; $^1$H NMR (400 MHz, $d_6$-DMSO, TMS, ppm): δ 3.35(m, 4H), 1.56-1.45(m, 15H); $^{13}$C NMR (100 MHz, $d_6$-DMSO, ppm): δ 154.89, 79.03, 44.31, 28.42, 25.69, 24.45.

Tert-butyl morpholine-4-carboxylate (Table 3, entry 10): White solid; m.p. 65-66°C; Yield 95%; $^1$H NMR (400 MHz, CDCl$_3$, TMS, ppm): δ 3.64(t, J = 4 Hz, 4H), 3.42(t, J = 4 Hz, 4H), 1.47(s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): δ 154.77, 79.92, 66.67, 43.48, 28.37.

N-(tert-Butyloxycarbonyl)-(R)-(+-)1-phenylethylamine (Table 3, entry 11): White solid; Yield 97%; $^1$H NMR (400 MHz, TMS, CDCl$_3$, ppm): δ 7.28-7.15(m, 5H), 4.86(s, 1H), 4.74(bs, 1H), 1.39(s, 3H), 1.37(s, 9H); $^{13}$C NMR (100 MHz, CDCl$_3$, ppm): δ 155.00, 144.00, 128.40, 127.00, 125.80, 79.20, 50.00, 28.30, 22.50.

N-(tert-Butyloxycarbonyl)-L-alanine ethyl ester (Table 3, entry 12): Colorless oil; Yield 93%; $^1$H NMR (400 MHz, CDCl$_3$, TMS ppm): δ 5.04(bs, 1H), 4.30-4.27(m, 1H), 4.19(q, J =
8Hz, 2H), 1.44(s, 9H), 1.37(d, J = 8Hz, 3H), 1.27(t, J = 8Hz, 3H); $^{13}$C NMR (10MHz, CDCl$_3$, ppm): δ 173.30, 155.10, 79.70, 61.30, 49.20, 28.30, 18.70, 14.10.

![N-(tert-Butyloxy carbonyl)-L-phenyl alanine methyl ester](image)

N-(tert-Butyloxy carbonyl)-L-phenyl alanine methyl ester (Table 3, entry 13): Colorless oil; Yield 92%; $^1$H NMR (400 MHz, CDCl$_3$, TMS, ppm): δ 7.31-7.22(m, 3H), 7.12(d, J = 8Hz, 2H), 4.98(bs, 1H), 4.59 (d, J = 8Hz, 1H), 3.71(s, 3H), 3.13-3.02(m, 2H), 1.41(s, 9H); $^{13}$C NMR (125 MHz, CDCl$_3$, ppm): δ 172.30, 155.00, 136.00, 129.20, 128.50, 127.0, 79.90, 54.4, 52.10, 38.30, 28.30.

![NHBoc COOEt](image)

(2R,3R)-ethyl-3-((tert-butoxycarbonyl)amino)-2-hydroxy-3-phenylpropanoate (Table3, entry 14): White solid; mp 115-117°C; Yield 92% $^1$H NMR (400 MHz, CDCl$_3$, TMS, ppm): δ 7.27-7.26(m, 5H), 5.64-5.62(d, J = 7.6Hz, 1H), 5.11-5.10(d, J = 7.2Hz, 1H), 4.57(bs, 1H), 4.16-4.08 (m, 2H), 2.94-2.93(d, J = 6.4 Hz), 1.42(s, 9H), 1.25-1.22(t, J = 7.2Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): δ 171.84, 154.94, 128.34, 128.07, 127.40, 79.84, 73.15, 61.99, 56.53, 28.29, 14.03.
$^1$H NMR Spectra (Table 2, entry 1)
$^{13}$C NMR Spectra (Table 2, entry 1)
$^1$H NMR Spectra (Table 2, entry 2)
$^{13}$C NMR Spectra (Table 2, entry 2)
$^1$H NMR Spectra (Table 2, entry 3)
$^{13}$C NMR Spectra (Table 2, entry 3)
$^1$H NMR Spectra (Table 2, entry 4)
$^{13}$C NMR Spectra (Table 2, entry 4)
$^1$H NMR Spectra (Table 2, entry 5)
$^{13}$C NMR Spectra (Table 2, entry 5)
\(^1\)H NMR Spectra (Table 2, entry 6)
$^{13}$C NMR Spectra (Table 2, entry 6)
$^1$H NMR Spectra (Table 2, entry 8)
$^{13}$C NMR Spectra (Table 2, entry 8)
$^1$H NMR Spectra (Table 2, entry 9)
$^{13}$C NMR Spectra (Table 2, entry 9)
$^1$H NMR Spectra (Table 2, entry 10)
$^{13}$C NMR Spectra (Table 2, entry 10)
$^1$H NMR Spectra (Table 2, entry 11)
$^{13}$C NMR Spectra (Table 2, entry 11)
$^1$H NMR Spectra (Table 2, entry 12)
$^1$H NMR Spectra (Table 2, entry 13)
$^1$H NMR Spectra (Table 2, entry 14)
$^{13}$C NMR Spectra (Table 2, entry 14)
$^1$H NMR Spectra (Table 2, entry 15)
$^{13}$C NMR Spectra (Table 2, entry 15)
$^1$H NMR Spectra (Table 2, entry 16)
$^1$H NMR Spectra (Table 2, entry 17)
$^{13}$C NMR Spectra (Table 2, entry 17)
\(^1\)H NMR Spectra (Table 2, entry 18)
$^{13}$C NMR Spectra (Table 2, entry 18)
$^1$H NMR Spectra (Table 2, entry 19)
$^{13}$C NMR Spectra (Table 2, entry 19)
$^1$H NMR Spectra (Table 2, entry 20)
$^{13}$C NMR Spectra (Table 2, entry 20)
$^1$H NMR Spectra (Table 3, entry 1)
$^1$H NMR Spectra (Table 3, entry 2)
$^{13}$C NMR Spectra (Table 3, entry 2)
$^1$H NMR Spectra (Table 3, entry 3)
$^{13}$C NMR Spectra (Table 3, entry 3)
$^1$H NMR Spectra (Table 3, entry 4)
$^{13}$C NMR Spectra (Table 3, entry 4)
$^1$H NMR Spectra (Table 3, entry 5)
$^{13}$C NMR Spectra (Table 3, entry 5)
$^1$H NMR Spectra (Table 3, entry 6)
$^{13}$C NMR Spectra (Table 3, entry 6)
$^1$H NMR Spectra (Table 3, entry 7)
$^{13}$C NMR Spectra (Table 3, entry 7)
$^1$H NMR Spectra (Table 3, entry 8)
$^{13}\text{C}$ NMR Spectra (Table 3, entry 8)
$^1$H NMR Spectra (Table 3, entry 9)
$^{13}\text{C}$ NMR Spectra (Table 3, entry 9)
$^1$H NMR Spectra (Table 3, entry 10)
$^{13}$C NMR Spectra (Table 3, entry 10)
$^1$H NMR Spectra (Table 3, entry 11)
$^{13}$C NMR Spectra (Table 3, entry 11)
$^1$H NMR Spectra (Table 3, entry 12)
$^{13}$C NMR Spectra (Table 3, entry 12)
$^1$H NMR Spectra (Table 3, entry 13)
$^{13}$C NMR Spectra (Table 3, entry 13)

![NMR Spectrum Image]

Peptide structure:

- Me-O-\(\text{Ph}\)
- NH\text{Boc}

Chemical shifts:

- 172.3 ppm
- 155.0 ppm
- 136.0 ppm
- 126.2 ppm
- 125.9 ppm
- 123.0 ppm
- 90.9 ppm
- 84.4 ppm
- 52.1 ppm
- 36.3 ppm
- 26.3 ppm
NMR Spectra (Table 3, entry 14)
$^{13}$C NMR Spectra (Table 3, entry 14)