Supporting Information for

Boiling Water-Catalyzed Neutral and Selective *N*-Boc Deprotection

*Jia Wang, Yan-Liang Liang, Jin Qu**

The State Key Laboratory of Elemento-organic Chemistry,

Nankai University, Tianjin 300071, P. R. China

Fax: +86-(022)-23499247 and E-mail:qujin@nankai.edu.cn

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General

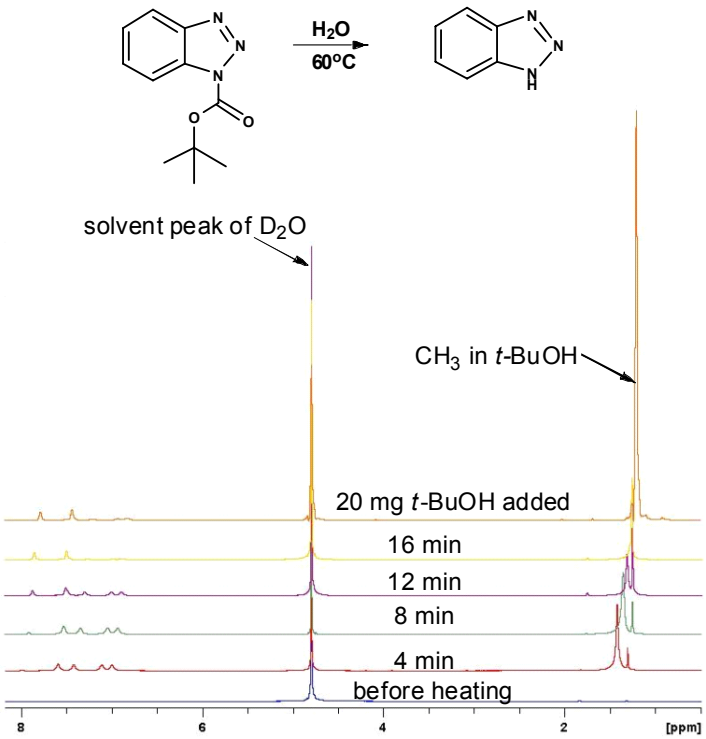
All reactions were carried out in aerial atmosphere, unless otherwise mentioned. Water was purchased from Watson water or from Milli-Q® Ultrapure Water Purification System. Flash chromatography was performed on silica gel columns (200 – 300 mesh). All of the compounds were characterized by ^{1}H NMR and ^{13}C NMR. ^{1}H NMR spectra were recorded at 300 MHz, 400 MHz or 600 MHz; ^{13}C NMR were recorded at 75 MHz, 100 MHz or 150 MHz. Peaks recorded are relative to internal standards: TMS (δ = 0.00) for ^{1}H and $CDCl_3$ (δ = 77.00) for ^{13}C spectra.

General Procedure for *N*-Boc Deprotection Reaction in Boiling Water

In a 50 mL round bottle flask filled with 20 mL of water, *tert*-butyl 3–(hydroxymethyl) phenylcarbamate (223 mg, 1 mmol) was added. Then the flask topped with a condenser was dipped in a 110℃ oil bath. TLC was used to monitor the progress of the reaction. The reaction mixture was cooled down after 1.5 h and was extracted with ethyl acetate (40 mL×3). The extract was washed with brine, dried over anhydrous Na_2SO_4, and then concentrated in vacuum. The residue was purified by column chromatography on silica gel with ethyl acetate/petroleum ether (1:1, v/v) to afford the free amine as a colorless solid (120 mg, yield 98%, for most of cases, no further purification is needed). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (s, 1H), 7.21–7.24 (m, 2H), 7.03 (d, J = 6.8 Hz, 1H), 6.56 (br s, 1H, NH), 4.65 (s, 2H), 1.95 (br s, 1H, OH), 1.51 (s, 9H)

Most of the substrate was insoluble in water at room temperature, but it became partially soluble or completely miscible when temperature was raised above 60°C. For very sticky N^{α},N^{ind}-diBoc-tryptamine, the substrate was firstly dissolved in 1 mL of 1,4-dioxane and then 19 mL of water was added. We note that most organic compounds containing one or more hydrogen-bond-forming functional groups should have good solubility in hot water (60–100°C).

^{1}H NMR Studies of the Deprotection of *N*-Boc-benzotriazole in D_2O at 60^{o}C



The white solid of *N*-Boc-benzotriazole (100 mg) and 500 μL of D_2O were placed in an NMR tube and were sonicated for 1 minute at room temperature. There is no signal in the ^{1}H NMR spectrum recorded because *N*-Boc-benzotriazole is not soluble in pure water at room temperature. The NMR tube was then immersed into a 60^{o}C water bath for 4 min (shake the NMR tube occasionally) and then was cooled down in an ice bath. The peaks of *N*-Boc-benzotriazole appear in the ^{1}H NMR spectrum were recorded because *N*-Boc-benzotriazole is partially soluble in water after heating (in order to insure that the signals in ^{1}H NMR spectrum belong to the starting material, a ^{13}C NMR spectrum of the 4 min sample was also taken and compared with the ^{13}C NMR spectrum of *N*-Boc-benzotriazole taken in $CDCl_3$). The high-field singlet attributes to the *t*-butanol formed in the reaction. Similarly, the NMR tube was heated for another 4 minutes and was cooled down before the ^{1}H NMR spectrum was recorded. On the ^{1}H NMR spectrum, the signals of the product rise while the signals of the starting material fall down. After the reaction finished (16 min), 20 mg of authentic *t*-butanol was added in the NMR tube and it is found that the high field singlet do not split which means it is the signal from *t*-butanol.

Analytical Data for Compounds in Table 1

tert–Butyl 1H–imidazole–1–carboxylate [1]

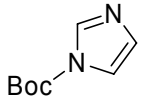


Table 1 - Entry 1

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (d, *J* = 2.4 Hz, 1H), 7.38 (s, 1H), 7.03 (d, *J* = 2.4 Hz, 1H), 1.63 (s, 9H)

tert–Butyl 1H–pyrazole–1–carboxylate [2]

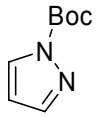


Table 1 - Entry 2

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (d, *J* = 2.4 Hz, 1H), 7.72 (s, 1H), 6.39 (t, *J* = 2.8 Hz, 1H), 1.66 (s, 9H)

tert–Butyl 1H–benzo[d]imidazole–1–carboxylate [3]

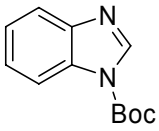


Table 1 - Entry 3

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.43 (s, 1H), 7.99 (d, *J* = 7.8 Hz, 1H), 7.79 (d, *J* = 7.8 Hz, 1H), 7.34–7.39 (m, 2H), 1.70 (s, 9H)

tert–Butyl 1H–benzo[d][1,2,3]triazole–1–carboxylate [4]

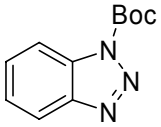


Table 1 - Entry 4

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.12 (d, *J* = 8.4 Hz, 1H), 8.07 (d, *J* = 8.4 Hz, 1H), 7.63 (t, *J* = 7.8 Hz, 1H), 7.48 (t, *J* = 7.8 Hz, 1H), 1.77 (s, 9H)

tert–Butyl 1H–indole–1–carboxylate [5]

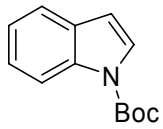


Table 1 - Entry 5

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.15 (m, 1H), 7.59 (s, 1H), 7.55 (d, *J* = 7.8 Hz, 1H), 7.30 (d, *J* = 7.8 Hz, 1H), 7.22 (m, 1H), 6.56 (s, 1H), 1.67 (s, 9H)

tert–Butyl 1H–pyrrolo[2,3–b]pyridine–1–carboxylate [6]

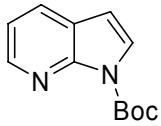


Table 1 - Entry 6

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (dd, *J* = 1.6, 4.8 Hz, 1H), 7.87 (dd, *J* = 1.6, 7.6 Hz, 1H), 7.64 (d, *J* = 4.0 Hz, 1H), 7.18 (dd, *J* = 4.8, 7.6 Hz, 1H), 6.50 (d, *J* = 4.0 Hz, 1H), 1.67 (s, 9H)

tert–Butyl 3–acetyl–1H–indole–1–carboxylate [7]

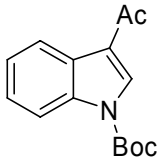


Table 1 - Entry 7

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.37 (m, 1H), 8.23 (s, 1H), 8.12 (m, 1H), 7.37 (m, 2H), 2.57 (s, 3H, CH_3), 1.72 (s, 9H)

Analytical Data for Compounds in Table 2

tert–Butyl phenylcarbamate [8]

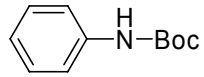


Table 2 - Entry 1

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39 (d, *J* = 8.0 Hz, 2H), 7.32 (t, *J* = 8.0 Hz, 2H), 7.03 (t, *J* = 7.2 Hz, 1H), 6.48 (s, 1H, NH), 1.52 (s, 9H)

tert–Butyl *p*–tolylcarbamate [9]

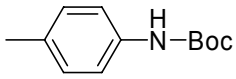


Table 2 - Entry 2

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 (d, *J* = 8.0 Hz, 2H), 7.09 (d, *J* = 8.4 Hz, 2H), 6.42 (br s, 1H, NH), 2.29 (s, 3H), 1.51 (s, 9H)

tert–Butyl 4–chlorophenylcarbamate [10]

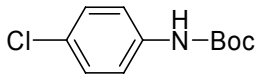


Table 2 - Entry 3

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (d, *J* = 8.8 Hz, 2H), 7.24 (d, *J* = 9.2 Hz, 2H), 6.53 (br s, 1H, NH), 1.51 (s, 9H)

tert–Butyl 4–bromophenylcarbamate [9]

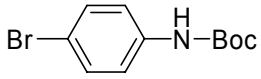


Table 2 - Entry 4

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.38 (d, *J* = 8.4 Hz, 2H), 7.25 (d, *J* = 8.4 Hz, 2H), 6.48 (br s, 1H, NH), 1.53 (s, 9H)

tert–Butyl 4–hydroxyphenylcarbamate [9]

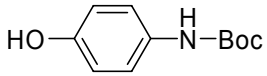


Table 2 - Entry 5

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.17 (d, *J* = 7.2 Hz, 2H), 6.73 (d, *J* = 7.2 Hz, 2H), 6.34 (br s, 1H, NH), 5.27 (br s, 1H, OH), 1.51 (s, 9H)

tert–Butyl 3–hydroxyphenylcarbamate [11]

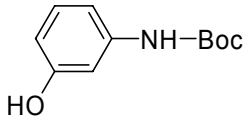


Table 2 - Entry 6

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.11 (m, 2H), 6.72 (d, *J* = 7.6 Hz, 1H), 6.52 (t, *J* = 8.0 Hz, 2H), 5.39 (br s, 1H), 1.52 (s, 9H)

tert–Butyl 4–methoxyphenylcarbamate [1]

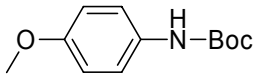


Table 2 - Entry 7

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.25 (d, *J* = 9.0 Hz, 2H), 6.83 (d, *J* = 9.0 Hz, 2H), 6.35 (br s, 1H, NH), 3.78 (s, 3H), 1.51 (s, 9H)

tert–Butyl 4–acetoxyphenylcarbamate [12]

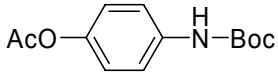


Table 2 - Entry 8

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36 (d, *J* = 9.0 Hz, 2H), 7.01 (d, *J* = 8.7 Hz, 2H), 6.49 (br s, 1H, NH), 2.28 (s, 3H), 1.51 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 188.62, 152.69, 146.04, 136.01, 121.93, 119.42, 80.65, 28.31, 21.04

tert–Butyl 3–(hydroxymethyl)phenylcarbamate [13]

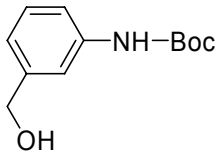


Table 2 - Entry 9

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (s, 1H), 7.21–7.24 (m, 2H), 7.03 (d, *J* = 6.8 Hz, 1H), 6.56 (br s, 1H, NH), 4.65 (s, 2H), 1.95 (br s, 1H, OH), 1.51 (s, 9H)

tert–Butyl 4–acetylphenylcarbamate [14]

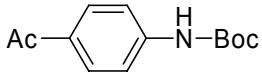


Table 2 - Entry 10

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.7 Hz, 2H), 7.43 (d, *J* = 8.1 Hz, 2H), 6.55 (s, 1H, NH), 2.47 (s, 3H), 1.41 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.12, 152.31, 143.26, 131.36, 129.67, 117.34, 80.88, 28.09, 26.20

tert–Butyl 4–nitrophenylcarbamate [1]

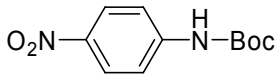


Table 2 - Entry 11

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (d, *J* = 9.2 Hz, 2H), 7.54 (d, *J* = 9.2 Hz, 2H), 6.96 (br s, 1H, NH), 1.54 (s, 9H)

tert–Butyl 4–(dimethylamino)phenylcarbamate [15]

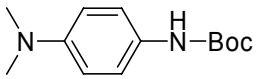


Table 2 - Entry 12

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 (d, *J* = 6.4 Hz, 2H), 6.70 (t, *J* = 6.4 Hz, 2H), 6.25 (br s, 1H, NH), 2.89 (s, 6H), 1.50 (s, 9H)

Analytical Data for Compounds in Table 3

tert–Butyl Butylcarbamate [3]

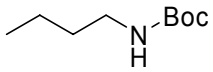


Table 3 - Entry 1 - Substrate

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.52 (s, 1H, NH), 2.74-2.75 (m, 2H), 1.08 (s, 9H), 1.07-1.11 (m, 2H), 0.95-1.01 (m, 2H), 0.55 (t, J = 0.8 Hz, 3H)

tert–Butyl 5–hydroxypentylcarbamate [16]

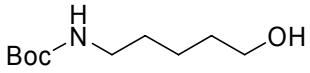
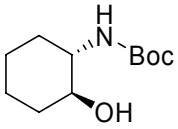


Table 3 - Entry 2 - Substrate

^{1}H NMR (600 MHz, $CDCl_3$) δ 4.68 (s, 1H, NH), 3.64 (t, J = 6.0 Hz, 3H), 3.12 (br, 2H), 2.08–2.19 (m, 1H), 1.57–1.60 (m, 2H), 1.50–1.52 (m, 2H), 1.44 (s, 9H), 1.38–1.41 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 156.08, 79.06, 62.53, 40.36, 32.19, 29.79, 28.37, 22.87

tert–Butyl 2–hydroxycyclohexylcarbamate [17]



trans

Table 3 - Entry 3 - Substrate

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.61 (br s, 1H), 3.30 (br s, 3H), 1.95–2.05 (m, 2H), 1.68–1.70 (m, 2H), 1.45 (s, 9H), 1.12–1.30 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.12, 79.85, 75.13, 56.52, 34.08, 31.72, 28.31, 24.64, 24.00

tert–Butyl 4–hydroxypiperidine–1–carboxylate [18]

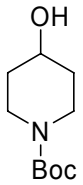


Table 3 - Entry 4 - Substrate

^{1}H NMR (600 MHz, $CDCl_3$) δ 3.80–3.88 (m, 3H), 3.03 (m, 2H), 1.83–1.88 (m, 2H), 1.67 (br s, 1H, OH), 1.46 (s, 9H), 1.39–1.54 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 154.80, 79.52, 67.71, 41.23, 34.14, 28.40

tert-Butyl 2-oxopiperidine-1-carboxylate [19]

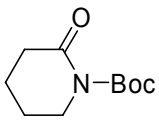


Table 3 - Entry 5 - Substrate

^{1}H NMR (600 MHz, $CDCl_3$) δ 3.52–3.58 (m, 2H), 2.35–2.46 (m, 2H), 1.67–1.77 (m, 4H), 1.42 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 171.15, 152.45, 82.54, 46.12, 34.68, 27.79, 25.57, 20.28

tert–Butyl (S)–1–(methoxycarbonyl)ethylcarbamate [20]

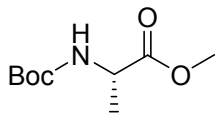


Table 3 - Entry 7 - Substrate

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.05 (br, 1H), 4.32 (m, 1H), 3.75 (s, 3H), 1.45 (s, 9H), 1.38 (d, J = 6.4 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 173.80, 155.06, 79.77, 52.24, 49.10, 28.26, 18.59

tert–Butyl (S)–1–(cyclohexylcarbamoyl)–2–hydroxyethylcarbamate [21]

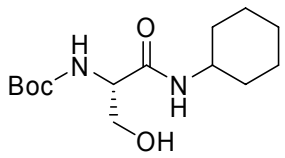


Table 3 - Entry 8 - Substrate

^{1}H NMR (300 MHz, $CDCl_3$) δ 6.72 (s, 1H, NH), 5.70 (s, 1H), 4.04–4.13 (m, 2H), 3.70–3.76 (m, 2H), 3.64 (s, 1H), 1.46 (s, 9H), 1.17–2.07 (m, 10H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.45, 156.33, 80.47, 62.85, 54.81, 54.76, 48.13, 32.68, 28.26, 25.43, 25.51

(S)–2–amino–*N*–cyclohexyl–3–hydroxypropanamide [22]

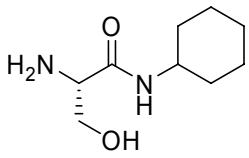


Table 3 - Entry 8 - Product

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37–7.39 (br s, 1H), 3.72–3.82 (br, 3H), 3.43 (t, *J* = 4.8 Hz, 1H), 2.62 (br s, 4H), 1.13–2.05 (m, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.63, 65.30, 55.86, 47.81, 32.98, 32.87, 25.46, 24.72

tert–Butyl 2–(1–(*tert*–butoxycarbonyl)–1H–imidazol–5–yl)ethylcarbamate [23]

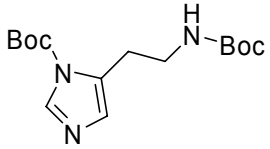


Table 3 - Entry 9 - Substrate

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (s, 1H), 7.14 (s, 1H), 5.02 (br s, 1H, NH), 3.43 (t, *J* = 6.3 Hz, 2H), 2.74 (t, *J* = 6.6 Hz, 2H), 1.61 (s, 9H), 1.44 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.89, 146.99, 141.20, 136.78, 113.65, 85.36, 79.04, 39.80, 28.37, 28.29, 27.86

tert–Butyl 2–(1H–imidazol–5–yl)ethylcarbamate [24]

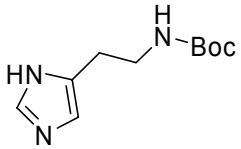


Table 3 - Entry 9 - Product

^{1}H NMR (400 MHz, $CDCl_3$) δ 11.07 (s, 1H), 7.53 (s, 1H), 6.74 (s, 1H), 5.35 (s, 1H, NH), 3.30 (s, 2H), 2.72 (s, 2H), 1.33 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 156.06, 134.72, 116.77, 99.80, 78.92, 40.20, 28.16, 27.24

tert–Butyl 2–(1–(*tert*–butoxycarbonyl)–1H–indol–3–yl)ethylcarbamate [25]

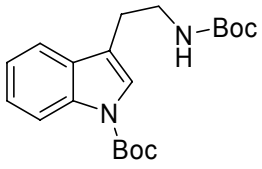


Table 3 - Entry 10 - Substrate

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.14 (d, *J* = 8.0 Hz, 1H), 7.52 (d, *J* = 7.8 Hz, 1H), 7.41 (s, 1H), 7.30 (t, *J* = 7.2 Hz, 1H), 7.21 (t, *J* = 7.2 Hz, 1H), 4.85 (br s, 1H, NH), 3.44 (s, 2H), 2.87 (s, 2H), 1.65 (s, 9H), 1.43 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 155.77, 149.49, 135.39, 130.29, 124.23, 122.92, 122.28, 118.77, 117.65, 115.08, 83.22, 78.90, 40.07, 29.50, 28.22, 27.99

tert–Butyl 2–(1H–indol–3–yl)ethylcarbamate [26]

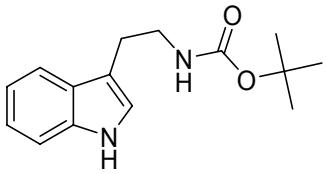


Table 3 - Entry 10 - Product

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.08 (s, 1H), 7.61 (d, *J* = 7.8 Hz, 1H), 7.37 (d, *J* = 7.8 Hz, 1H), 7.20 (t, *J* = 7.2 Hz, 1H), 7.12 (t, *J* = 7.8 Hz, 1H), 7.03 (s, 1H), 4.62 (s, 1H), 3.47 (d, *J* = 6.0 Hz, 2H), 2.96 (t, *J* = 6.0 Hz, 2H), 1.44 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 156.06, 136.43, 127.37, 122.01, 119.30, 118.72, 112.99, 112.93, 111.19, 79.24, 41.07, 28.41, 25.81

(S)-*tert*-Butyl 2-(butyl-*tert*-Butoxycarbonylcarbamoyl)pyrrolidine-1-carboxylate

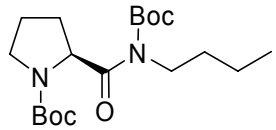


Table 3 - Entry 11 - Substrate

^{1}H NMR (600 MHz, $CDCl_3$) δ 5.18-5.22 (m, 1H), 3.58-3.75 (m, 3H), 3.42-3.50 (m, 1H), 2.32 (s, 1H), 1.86-1.95 (m, 3H), 1.56 (s, 5H), 1.54 (s, 4H), 1.47 (s, 4H), 1.40 (s, 5H), 1.27-1.36 (m, 4H), 0.91-0.96 (m, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 175.73, 175.39, 154.10, 153.42, 152.79, 152.59, 82.73, 82.48, 78.90, 78.87, 61.01, 60.54, 46.65, 46.35, 44.26, 44.21, 30.91, 30.57, 30.40, 30.25, 28.18, 28.00, 27.69, 23.28, 27.72, 19.78, 19.73, 13.53, 13.50 (*cis/trans*=1:1); LRMS (ESI) $[M+H]^+$ m/z 371.0; HRMS (ESI) for $C_{19}H_{34}N_2O_5$: Calcd for $[M+H]^+$ m/z 371.2573, found 371.2579.

(S)-*tert*-Butyl 2-(butylcarbamoyl)pyrrolidine-1-carboxylate [27]

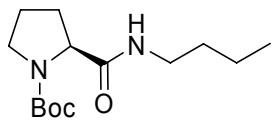
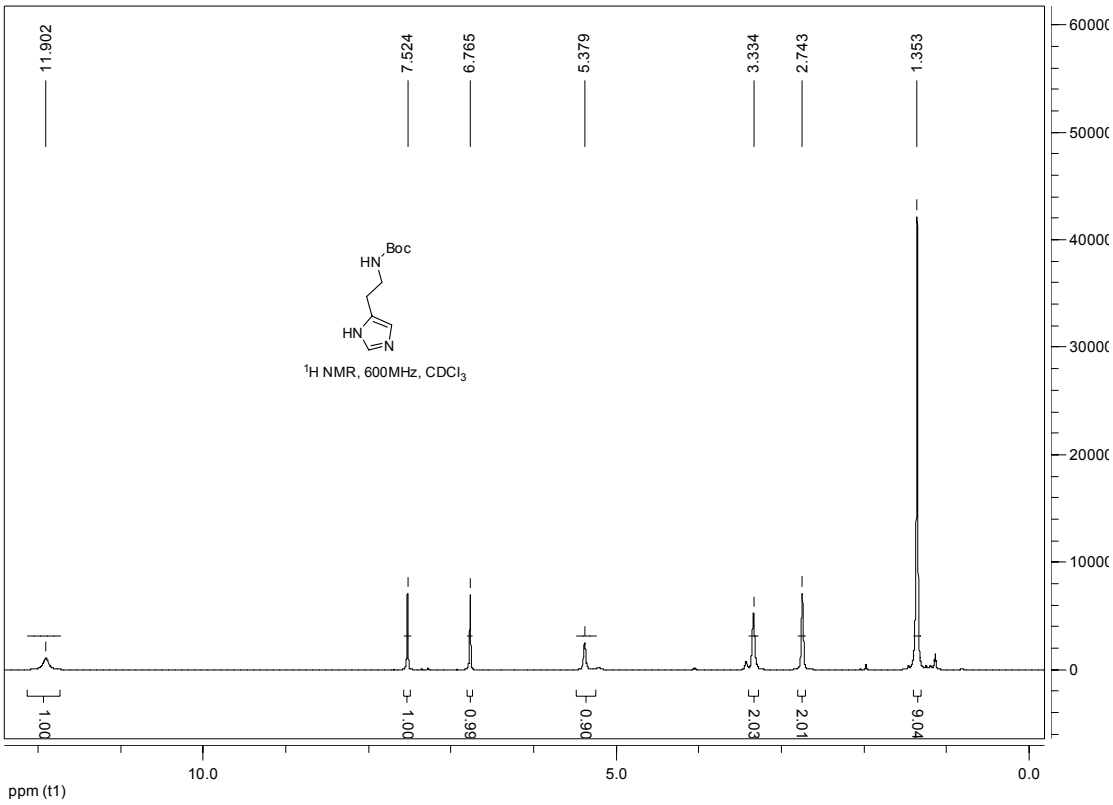


Table 3 - Entry 11 - Product

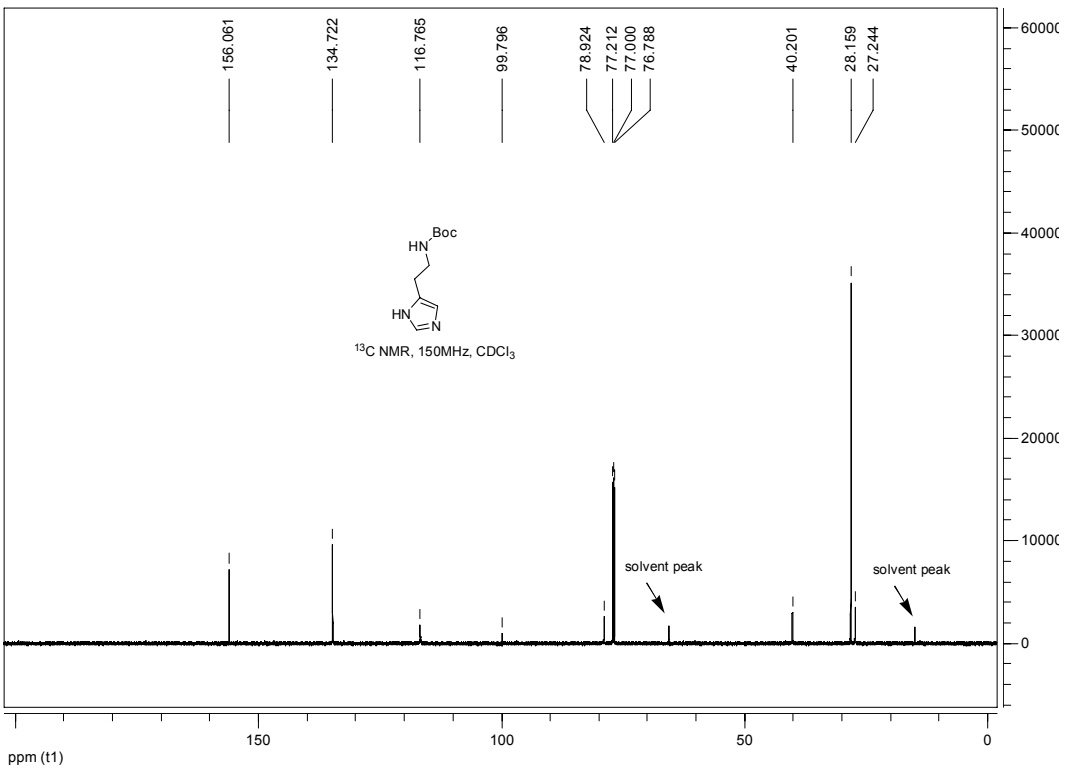
^{1}H NMR (600 MHz, $CDCl_3$) δ 6.85 (s, 0.5H), 6.42 (s, 0.5H), 3.93-4.00 (m, 1H), 2.93-3.19 (m, 4H), 1.60-1.99 (m, 4H), 1.19 (s, 9H), 1.08-1.09 (m, 4H), 0.65 (m, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 171.58, 171.55, 155.08, 154.11, 99.70, 79.59, 60.63, 59.77, 46.54, 38.51, 31.23, 31.19, 31.06, 30.72, 27.83, 24.02, 23.20, 19.48, 13.19 (*cis/trans*=1:1)

Selected NMR Spectra

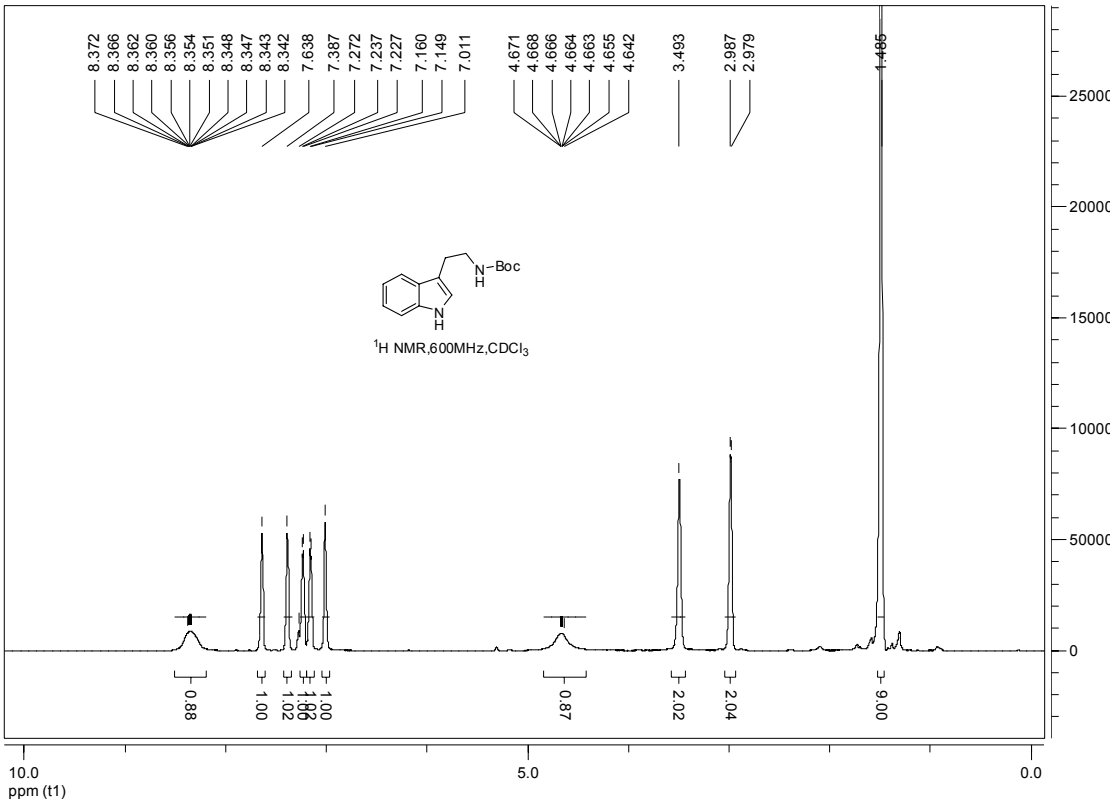
tert–Butyl 2–(1H–imidazol–5–yl)ethylcarbamate ^{1}H NMR



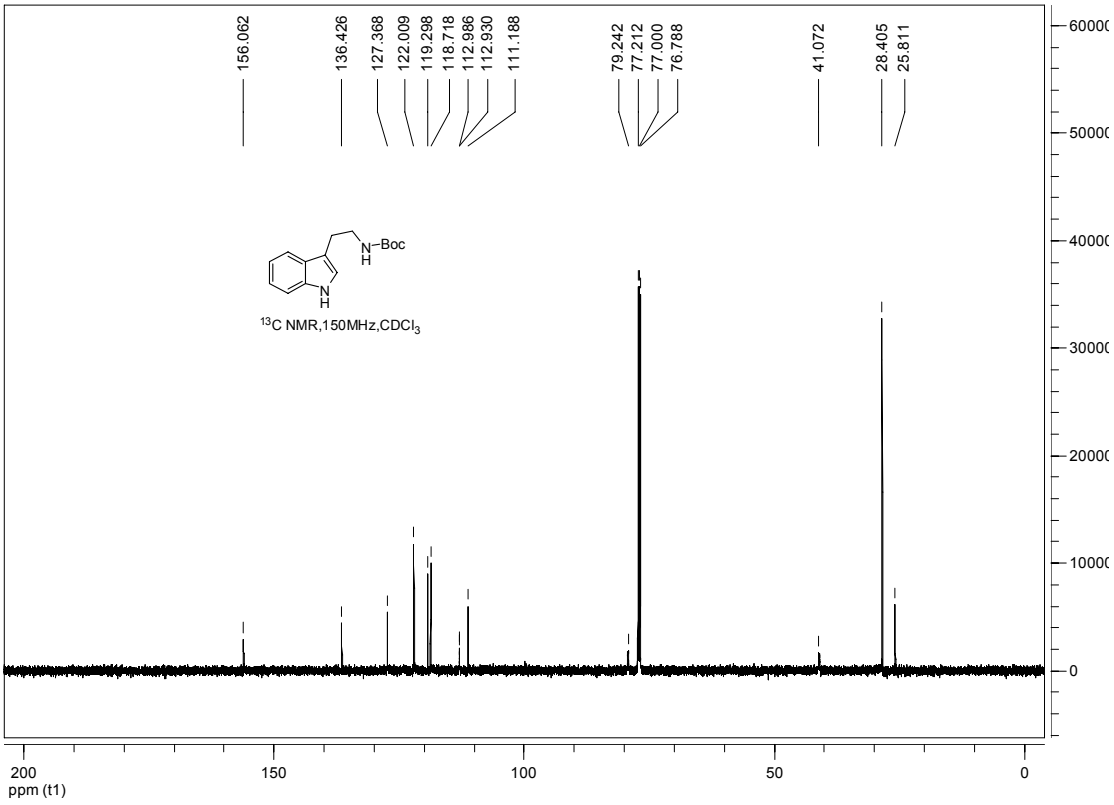
tert–Butyl 2–(1H–imidazol–5–yl)ethylcarbamate ^{13}C NMR



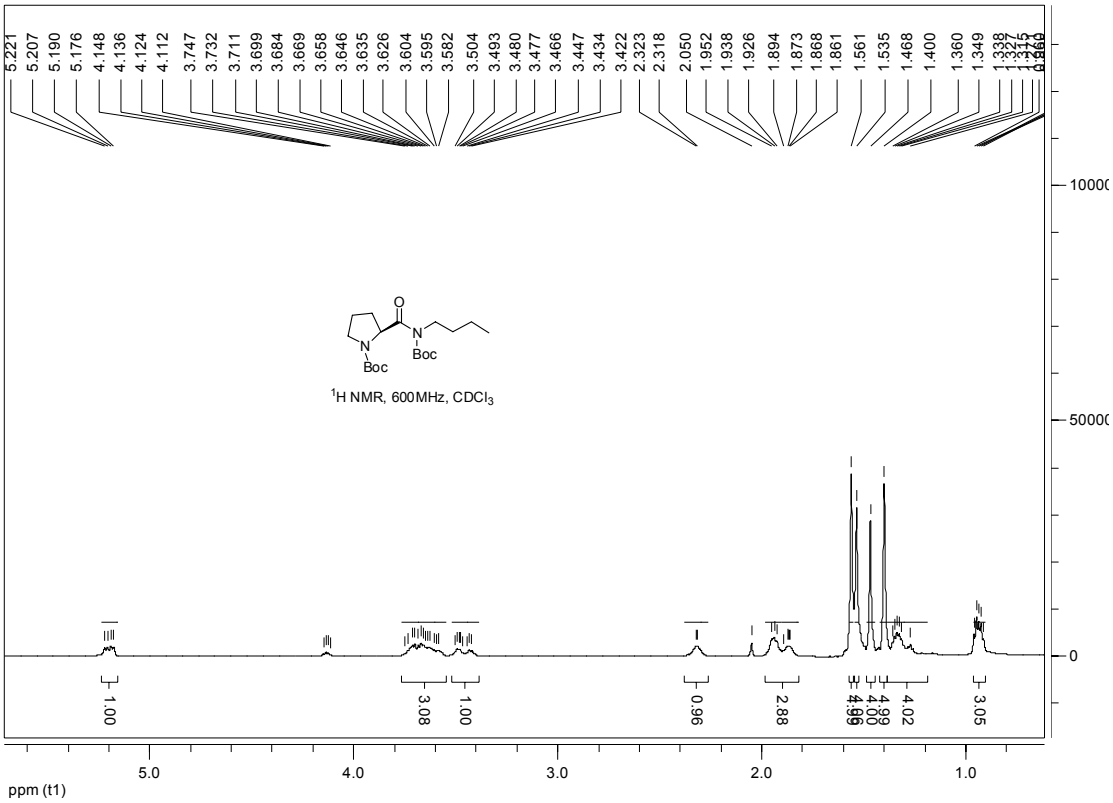
tert–Butyl 2–(1H–indol–3–yl)ethylcarbamate ^{1}H NMR



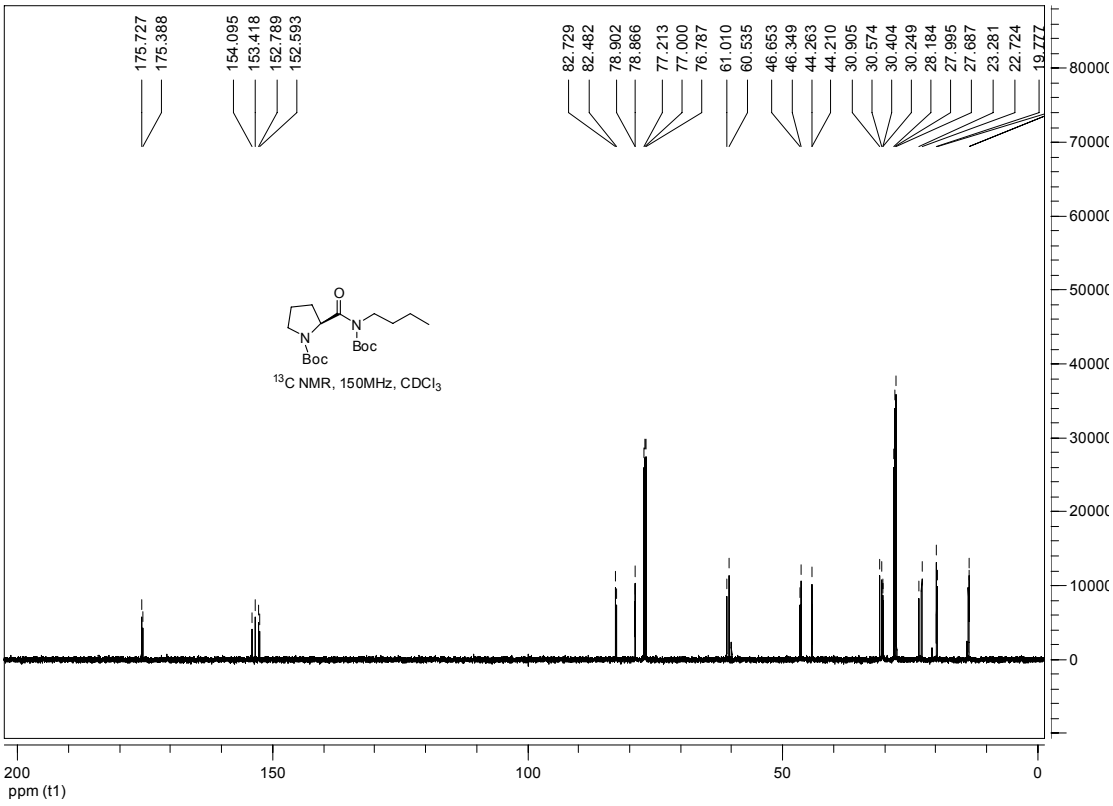
tert–Butyl 2–(1H–indol–3–yl)ethylcarbamate ^{13}C NMR



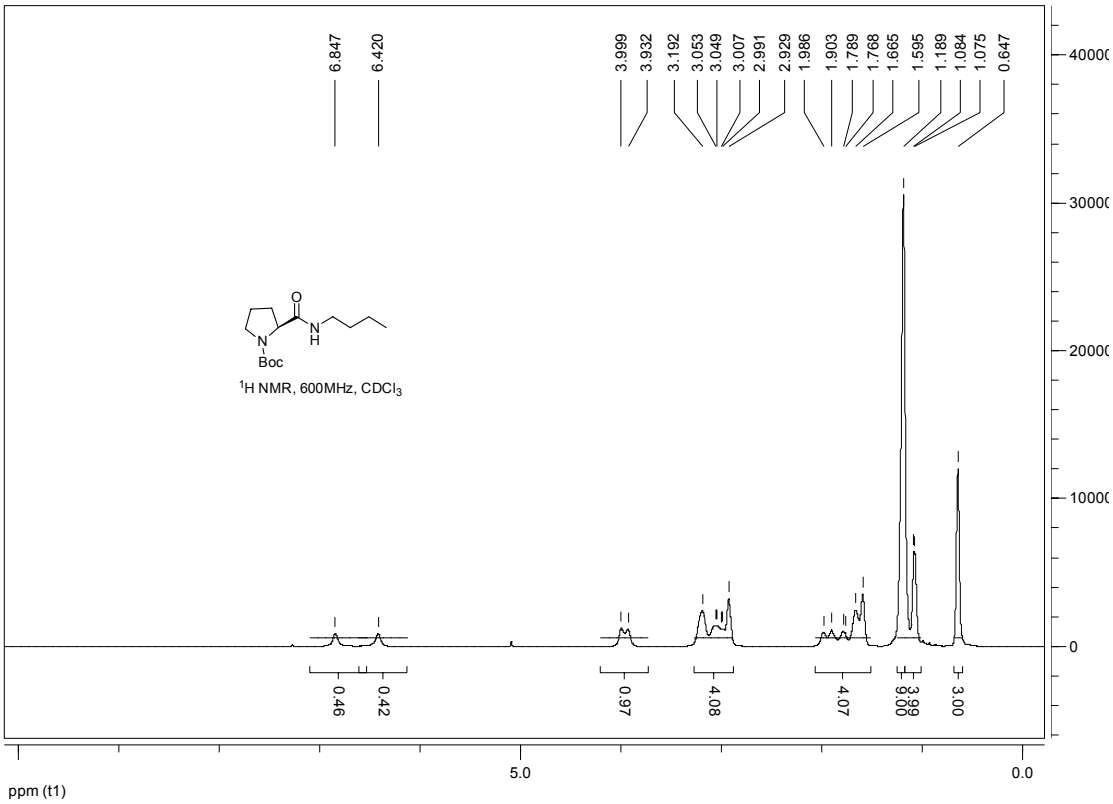
(S)-*tert*-Butyl 2-(butyl-*tert*-Butoxycarbonylcarbamoyl)pyrrolidine-1-carboxylate ^{1}H NMR



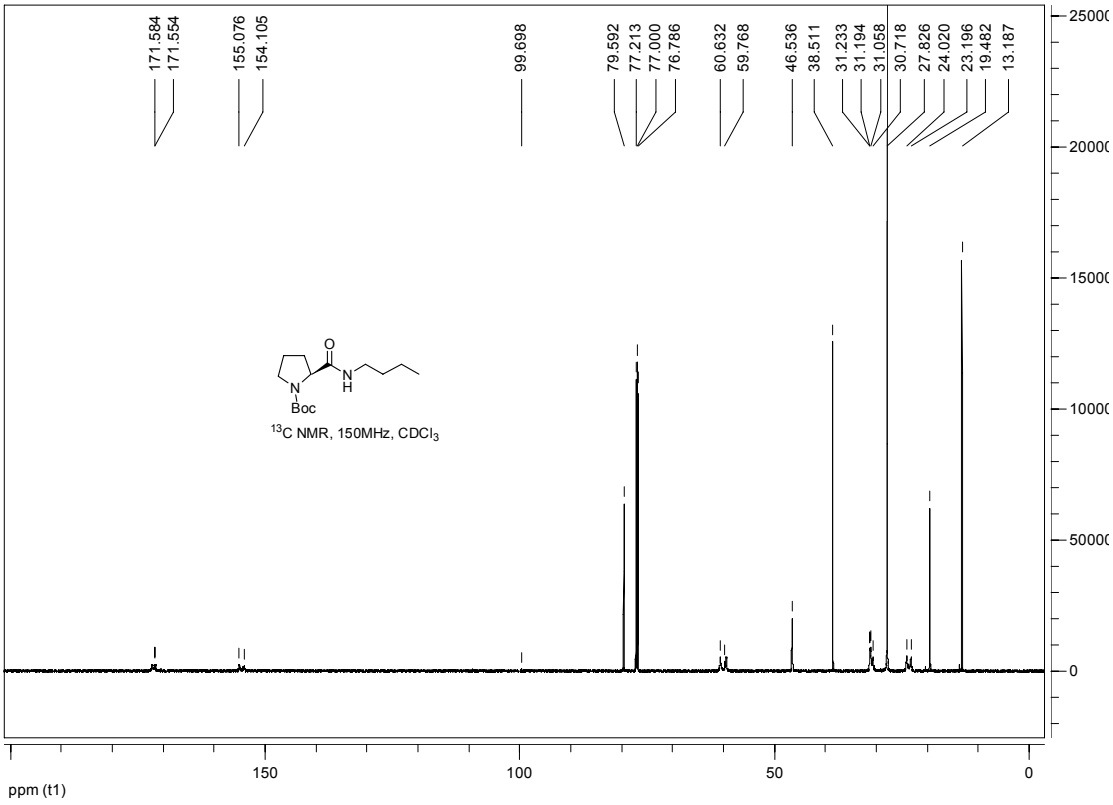
(S)-*tert*-Butyl 2-(butyl-*tert*-Butoxycarbonylcarbamoyl)pyrrolidine-1-carboxylate ^{13}C NMR



(S)-*tert*-Butyl 2-(butylcarbamoyl)pyrrolidine-1-carboxylate ^{1}H NMR



(S)-*tert*-Butyl 2-(butylcarbamoyl)pyrrolidine-1-carboxylate ^{13}C NMR



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