Electronic Supplementary Material (ESI) for RSC Advances.

SUPPORTING INFORMATION

An efficient mechanochemical synthesis of amides and dipeptides using 2,4,6-trichloro-1,3,5-triazine and PPh_3

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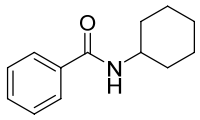
Contents

Materials and method

All compounds were used as received from the suppliers. The reaction was monitored by thin-layer chromatography carried out on silica gel plates ($60F_{254}$, MERCK, Germany) and visualized under UV light (245 nm). Column chromatography was performed over silica gel 60 (70–230 mesh, MERCK, Germany). Melting points were determined using SANYO, Gallenkamp apparatus at a heating rate of 10 ^{o}C/min and were uncorrected. Optical rotations were recorded on an automatic polarimeter–AUTOPOL I. NMR measurements were conducted on a Bruker AVANCE^{TM} (400 MHz for ^{1}H-NMR) using chloroform-*d* ($CDCl_3$) and dimethylsulfoxide (DMSO-d_6) as the solvent and tetramethylsilane (TMS) as an internal standard. Chemical shifts were reported in parts per million (ppm, δ) downfield from TMS. Splitting patterns are described as singlet (s), doublet (d), triplet (t), quartet (q), multiplet (m), broad (br), doublet of doublet (dd), and triplet of doublet (td). High Resolution Mass Spectrometry (HRMS) was performed with a MicroTOF$_{LC}$, Bruker Daltonics. Chiral HPLC analysis was performed on a Agilent 1100 series HPLC system using Daicel IB (150 x 4.6 mm) chiral column with *n*-hexane : 2-propanol (IPA) as the mobile phase.

Solvent-free syntheses of amides and dipeptides using TCT/PPh_3

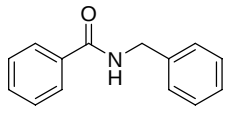
In a glove bag filled with N_2, TCT (0.0497 g, 0.27 mmol) and PPh_3 (0.0071 g, 0.027 mmol) were ground together with mortar and pestle, followed by adding carboxylic acid (0.27 mmol) and K_2CO_3 (0.1759 g, 0.54 mmol). The mixture was ground for a specified time during which a few drops of CH_2Cl_2 (*ca.* 1.5 μL/mg of solids) was added to facilitate homogeneous mixing. Amine (0.30 mmol) was then added with further grinding at room temperature. Progress of the reaction was monitored by TLC using a glass capillary tube filled with ethanol inside. After completion of the reaction, the crude mixture was purified by short column chromatography to afford pure compound. Dipeptides were synthesized according to the above described procedure from *N*-protected α-amino acid and amino methyl ester hydrochloride. In these cases, the amount of K_2CO_3 was increased to 0.2638 g (0.81 mmol).



Chemical Formula: $C_{13}H_{17}NO$
Molecular Weight: 203.29

***N*-Cyclohexylbenzamide[1] (Table 2, entry 1)**

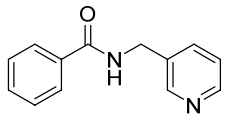
Following the general procedure with benzoic acid (0.0331 g, 0.271 mmol) and cyclohexylamine (0.0296 g, 0.298 mmol), the product was purified by column chromatography using 20% EtOAc/hexane as the eluent; white solid (0.0548 g, 99% yield); mp 143-144 °C; R_f 0.50 (30% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.78-7.76 (m, 2H), 7.49-7.38 (m. 3H), 6.20 (s, 1H), 4.02-3.93 (m, 1H), 2.04-2.00 (m, 2H), 1.78-1.73 (m, 2H), 1.67-1.62 (m, 1H), 1.46-1.36 (m, 2H), 1.29-1.17 (m, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.7, 135.1, 131.2, 128.5, 126.9, 48.7, 33.2, 25.6, 24.9.



Chemical Formula: $C_{14}H_{13}NO$
Molecular Weight: 211.26

***N*-Benzylbenzamide[2] (Table 2, entry 2)**

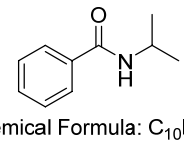
Following the general procedure with benzoic acid (0.0331 g, 0.271 mmol) and benzylamine (0.0319 g, 0.298 mmol), the product was purified by column chromatography using 20% EtOAc/hexane as the eluent; white solid (0.0569 g, 99% yield); mp 104-105 °C; R_f 0.37 (30% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.78 (d, J = 7.2 Hz, 2H); 7.47 (t, J = 7.2 Hz, 1H), 7.38 (t, J = 7.2 Hz, 2H), 7.32-7.25 (m, 5H), 7.03 (br s, 1H), 4.58 (d, J = 5.6 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.8, 138.2, 134.3, 131.6, 128.7, 128.5, 127.8, 127.5, 127.0, 44.0.



Chemical Formula: $C_{13}H_{12}N_2O$
Molecular Weight: 212.25

***N*-(Pyridin-3-ylmethyl)benzamide[3] (Table 2, entry 3)**

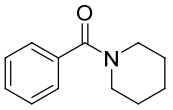
Following the general procedure with benzoic acid (0.0331 g, 0.271 mmol) and 3-picolylamine (0.0322 g, 0.298 mmol), the product was purified by column chromatography using 20% EtOAc/hexane as the eluent; yellow oil (0.0518 g, 90% yield); R_f 0.25 (20% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.90-7.90 (m, 2H), 7.45-7.39 (m, 3H), 7.12-6.86 (m, 4H), 4.19 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 171.8, 151.9, 151.3, 139.7, 138.3, 137.2, 135.1, 131.9, 130.6, 127.3, 44.5.



Chemical Formula: $C_{10}H_{13}NO$
Molecular Weight: 163.22

***N*-Isopropylbenzamide[4] (Table 2, entry 4)**

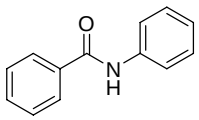
Following the general procedure with benzoic acid (0.0331 g, 0.271 mmol) and propan-2-amine (0.0176 g, 0.298 mmol), the product was purified by column chromatography using 20% EtOAc/hexane as the eluent; white crystal (0.0262 g, 59% yield); mp 103-104 °C; R_f 0.40 (30% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.76 (d, J = 7.2 Hz, 2H), 7.47 (t, J =7.2 Hz, 1H), 7.39 (t, J = 7.2 Hz, 2H), 6.23 (br s, 1H), 4.32-4.24 (m, 1H), 1.25 (d, J = 6.8 Hz, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.8, 135.0, 131.2, 128.5, 126.9, 41.9, 22.8.



Chemical Formula: $C_{12}H_{15}NO$
Molecular Weight: 189.26

Phenyl(1-piperidinyl)methanone[5] (Table 2, entry 5)

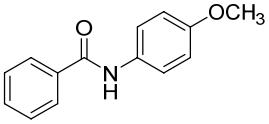
Following the general procedure with benzoic acid (0.0331 g, 0.271 mmol) and piperidine (0.0254g, 0.298 mmol), the product was purified by column chromatography using 20% EtOAc/hexane as the eluent; light yellow liquid (0.0510 g, 99% yield); R_f 0.22 (20% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.39 (br m, 5H), 3.71 (br s, 2H), 3.33 (br s, 2H), 1.67 (br s, 4H), 1.51 (br, s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 170.3, 136.5, 129.4, 128.4, 126.8, 48.8, 43.1, 26.5, 25.6, 24.6.



Chemical Formula: $C_{13}H_{11}NO$
Molecular Weight: 197.24

***N*-Phenylbenzamide[6] (Table 2, entry 6)**

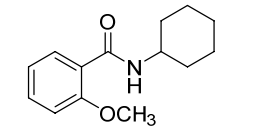
Following the general procedure with benzoic acid (0.0331 g, 0.271 mmol) and aniline (0.0278 g, 0.298 mmol), the product was purified by column chromatography using 20% EtOAc/hexane as the eluent; white solid (0.0320 g, 60% yield); mp 162-163 °C; R_f 0.55 (30% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.87 (d, J= 7.6 Hz, 2H), 7.65 (d, J= 7.6 Hz, 2H), 7.45-7.56 (m, 3H), 7.36 (t, J = 7.6 Hz, 2H), 7.15 (t, J = 7.6 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.8, 137.9, 135.0, 131.8, 129.0, 128.7, 127.1, 124.6,120.4



Chemical Formula: $C_{14}H_{13}NO_2$
Molecular Weight: 227.26

***N*-(4-methoxyphenyl)benzamide[7] (Table 2, entry 7)**

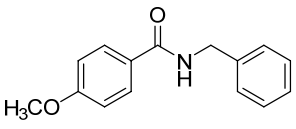
Following the general procedure with benzoic acid (0.0331 g, 0.271 mmol) and 4-methoxyaniline (0.0367g, 0.298 mmol), the product was purified by column chromatography using 20% EtOAc/hexane as the eluent; white solid (0.0472 g, 77% yield); mp 153-154 °C; R_f 0.44 (30% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$+CD_3OD 3 drops) δ 7.82 (d, J = 8.8 Hz, 2H), 7.52-7.46 (m, 3H), 7.40 (t, J = 7.2 Hz, 2H), 6.84 (d, J = 8.8 Hz, 2H), 3.76 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$+CD_3OD 3 drops) δ 166.5, 156.5, 134.9, 131.6, 129.8, 128.5, 127.1, 122.4, 114.1, 55.4.



Chemical Formula: $C_{14}H_{19}NO_2$
Molecular Weight: 233.31

***N*-cyclohexyl-2-methoxybenzamide[8] (Table 2, entry 8)**

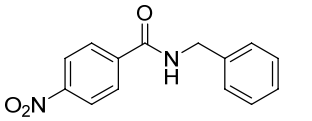
Following the general procedure with 2-methoxybenzoic acid (0.0412 g, 0.271 mmol) and cyclohexylamine (0.0296 g, 0.298 mmol), the product was purified by column chromatography using 20% EtOAc/hexane as the eluent; yellow oil (0.0582 g, 92% yield); R_f 0.35 (20% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.20 (dd, J = 7.8, 1.6 Hz, 1H), 7.82 (br d, J = 5.2 Hz, 1H), 7.43 (td, J = 7.8, 1.6 Hz, 1H), 7.08 (t, J = 7.8, 1H), 6.97 (d, J = 7.8, 1H), 4.07-3.99 (m, 1H), 3.96 (s, 3H), 2.02-1.98 (m, 2H), 1.75-1.70 (m, 2H), 1.65-1.60 (m, 1H), 1.51-1.40 (m, 2H), 1.35-1.25 (m, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 164.2, 157.4, 132.5, 132.2, 122.0, 121.3, 111.3, 56.0, 48.0, 33.0, 25.7, 24.7.



Chemical Formula: $C_{15}H_{15}NO_2$
Molecular Weight: 241.29

***N*-Benzyl-4-methoxybenzamide[9] (Table 2, entry 9)**

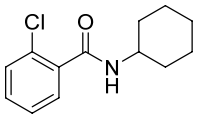
Following the general procedure with 4-methoxybenzoic acid (0.041 g, 0.271 mmol) benzylamine (0.0319 g, 0.298 mmol), the product was purified by column chromatography using 20% EtOAc/hexane as the eluent; white crystal (0.0649 g, 99% yield); mp 128-129 °C; R_f 0.30 (30% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75 (d, J = 6.8 Hz, 2H), 7.32-7.27 (m, 5H), 6.88 (d, J = 6.8 Hz, 2H), 4.57 (s, 2H), 3.81 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.3, 162.2, 153.4, 138.4, 128.8, 128.7, 127.8, 127.4, 133.7, 55.4, 44.0.



Chemical Formula: $C_{14}H_{12}N_2O_3$
Molecular Weight: 256.26

_N_-Benzyl-4-nitrobenzamide[10] (Table 2, entry 10)

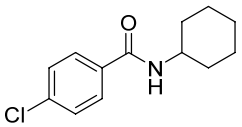
Following the general procedure with 4-nitrobebzoic acid (0.0453 g, 0.271 mmol) and benzylamine (0.0319 g, 0.298 mmol), the product was purified by column chromatography using 20% EtOAc/hexane as the eluent; yellow solid (0.0578 g, 83% yield); mp 131-132 °C; R_f 0.39 (30% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.20 (d, J = 6.8 Hz, 2H), 7.94 (d, J = 6.8 Hz, 2H), 7.30-7.22 (m, 5H), 4.56 (s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.9, 149.5, 139.9, 137.7, 128.7, 128.4, 127.7, 127.6, 123.6, 44.0**.**



Chemical Formula: $C_{13}H_{16}ClNO$
Molecular Weight: 237.73

2-Chloro-_N_-cyclohexylbenzamide[11] (Table 2, entry 11)

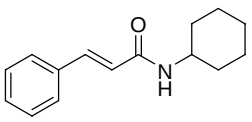
Following the general procedure with 2-chlorobenzoic acid (0.0424 g, 0.271 mmol) cyclohexylamine (0.0296 g, 0.298 mmol), the product was purified by column chromatography using 20% EtOAc/hexane as the eluent; white solid (0.0601 g, 93% yield); mp 128-129 °C; R_f 0.47 (30% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.62 (dd, J = 7.2, 2.4 Hz, 1H), 7.40-7.28 (m, 3H), 6.12 (s, 1H), 4.03-3.99 (m, 1H), 2.06-2.02 (m, 2H), 1.78-1.73 (m, 2H), 1.67-1.63 (m, 1H), 1.48-1.39 (m, 2H), 1.32-1.20 (m, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.6, 135.6, 131.0, 130.5, 130.1, 130.0, 127.0, 48.9, 32.9, 25.5, 24.7.



Chemical Formula: $C_{13}H_{16}ClNO$
Molecular Weight: 237.73

4-Chloro-_N_-cyclohexylbenzamide[12] (Table 2, entry 12)

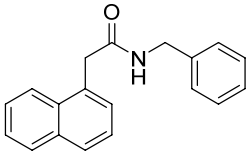
Following the general procedure with 4-chlorobebzoic acid (0.0424 g, 0.271 mmol) and cyclohexylamine (0.0296 g, 0.298 mmol), the product was purified by column chromatography using 20% EtOAc/hexane as the eluent; white solid (0.0639 g, 99% yield); mp 186-187 °C; R_f 0.31 (20% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.69 (d, J = 8.80 Hz, 2H), 7.36 (d, J = 8.80 Hz, 2H), 6.21 (s, 1H), 3.98-3.89 (m, 1H), 2.02-1.98 (m, 2H), 1.77-1.72 (m, 2H), 1.67-1.62 (m, 1H), 1.42-1.39 (m, 2H), 1.28-1.19 (m, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.6, 137.4, 133.4, 128.7, 128.4, 48.9, 33.1, 25.5, 24.9.



Chemical Formula: $C_{15}H_{19}NO$
Molecular Weight: 229.32

***N*-Cyclohexylcinnamamide[13] (Table 2, entry 13)**

Following the general procedure with cinnamic acid (0.0402 g, 0.271 mmol) and cyclohexylamine (0.0296 g, 0.298 mmol), the product was purified by column chromatography using 20% EtOAc/hexane as the eluent; white solid (0.0592 g, 95% yield); mp 178-179 °C; R_f 0.47 (30% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.63 (d, J = 15.6 Hz, 1H), 7.50-7.48 (m, 2H), 7.35-7.33 (m. 3H), 6.44 (dd, J = 15.6, 2.4 Hz, 1H), 5.90 (s, 1H), 3.98-3.88 (m, 1H), 2.02-1.95 (m, 2H), 1.77-1.72 (m, 2H), 1.67-1.62 (m, 1H), 1.45-1.35 (m, 2H), 1.26-1.17 (m, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 165.0, 140.6, 135.0, 129.5, 128.8, 127.7, 121.3, 48.4, 33.2, 25.6, 24.9.

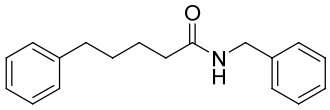


Chemical Formula: $C_{19}H_{17}NO$
Molecular Weight: 275.35

***N*-Benzyl-2-(naphthalen-1-yl)acetamide[14] (Table 2, entry 14)**

Following the general procedure with 2-(naphthalene-1-yl)acetic acid (0.0505 g, 0.271 mmol) and benzylamine (0.0319 g, 0.298 mmol), the product was purified by column chromatography using 20% EtOAc/hexane as the eluent; white solid (0.0684 g, 92% yield); mp 155-156 °C; R_f 0.30 (30% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.03 (dd, J

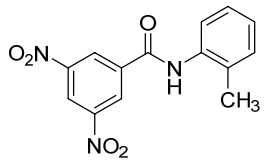
=8.0, 2.0 Hz, 1H), 7.90 (dd, *J* =8.0, 2.0 Hz, 1H), 7.84 (d, *J* =8.0 Hz, 1H), 7.60-7.54 (m, 2H), 7.48-7.42 (m, 2H), 7.22-7.21 (m, 3H), 7.04 (dd, *J* =7.2, 3.2 Hz, 2H), 5.77 (br s, 1H), 4.37 (d, *J* =6.0 Hz, 2H), 4.10 (s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 171.0, 138.1, 134.0, 132.1, 131.0, 128.8, 128.6, 128.5, 128.4, 127.3, 127.26, 126.8, 126.3, 125.7, 123.9, 43.4, 41.8.



Chemical Formula: $C_{18}H_{21}NO$
Molecular Weight: 267.37

_N_-Benzyl-5-phenylpentanamide[15] (Table 2, entry 15)

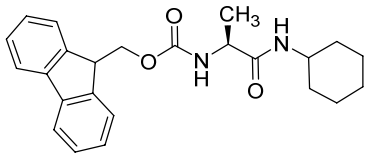
Following the general procedure with 5-phenylpentanoic acid (0.0483 g, 0.271 mmol) and benzylamine (0.0319 g, 0.298 mmol), the product was purified by column chromatography using 20% EtOAc/hexane as the eluent; white solid (0.0650 g, 90% yield); mp 75-76 °C; R_f 0.41 (30% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.37-7.29 (m, 7H), 7.23-7.18 (m, 3H), 6.00 (br s, 1H), 4.43 (d, *J* = 6.0 Hz, 2H), 2.65 (t, *J* = 7.2 Hz, 2H), 2.24 (t, *J* = 7.2 Hz, 2H), 1.75-1.67 (m, 4H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 172.9, 142.2, 138.4, 128.7, 128.4, 128.3, 127.8, 127.5, 125.8, 43.6, 36.6, 35.7, 31.1, 25.4.



Chemical Formula: $C_{14}H_{11}N_3O_5$
Molecular Weight: 301.26

3,5-Dinitro-_N_-(o-tolyl)benzamide[16] (Table 2, entry 16)

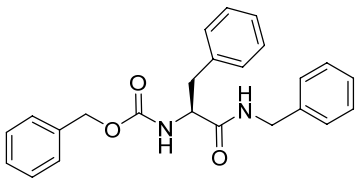
Following the general procedure with 3,5-dinitrobenzoic acid (0.0575 g, 0.271 mmol) and *o*-toluidine (0.0319 g, 0.298 mmol), the product was purified by column chromatography using 20% EtOAc/hexane as the eluent; yellow solid (0.0418 g, 51% yield); mp 246-247 °C; R_f 0.39 (20% EtOAc/hexanes); **^{1}H NMR** (400 MHz, DMSO-d_6) δ 10.64 (s, 1H), 9.22 (s, 2H), 9.06 (s, 1H), 7.40-7.27 (m, 4H), 2.30 (s, 3H); **^{13}C NMR** (100 MHz, DMSO-d_6) δ 161.8, 148.7, 137.6, 136.0, 134.4, 131.0, 128.4, 127.2, 126.7, 121.6, 18.3.



Chemical Formula: $C_{24}H_{28}N_2O_3$
Molecular Weight: 392.50

(9*H*-Fluoren-9-yl)methyl 1-(cyclohexylamino)-1-oxopropan-2-ylcarbamate (Table 2, entry 17)

Following the general procedure with Fmoc-L-alanine (0.0844 g, 0.271 mmol) and cyclohexylamine (0.0296 g, 0.298 mmol), the product was purified by column chromatography using 40% EtOAc/hexane as the eluent; white solid (0.0964 g, 78% yield); mp 192-193 °C; R_f 0.40 (40% EtOAc/hexanes); $[\alpha]_D^{26}$ -15.2 (c 0.98, $CHCl_3$); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 7.2 Hz, 2H), 7.57 (d, *J* = 7.2 Hz, 2H), 7.39 (t, *J* = 7.2 Hz, 2H), 7.29 (t, *J* = 7.2 Hz, 2H), 6.18 (br d, *J* = 6.4 Hz, 1H), 5.68 (br d, *J* = 7.2 Hz, 1H), 4.36 (d, *J* = 7.2 Hz, 2H), 4.20 (q, *J* = 7.2 Hz, 1H), 3.76-3.69 (m, 1H), 1.88-1.85 (m, 2H), 1.69-1.65 (m, 2H), 1.60-1.56 (m, 1H), 1.37 (d, *J* = 7.2 Hz, 3H), 1.35 –1.26 (m, 3H), 1.14-1.08 (m, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 171.3, 156.0, 143.8, 141.3, 127.8, 127.1, 125.1, 120.0, 67.1, 48.3, 48.2, 47.1, 32.9, 25.4, 24.8, 19.1; **HRMS (ESI)**: MNa+ found 415.2001. $[C_{24}H_{28}N_2O_3Na]^+$ requires 415.1998. Chiral HPLC: Daicel IB; homogeneous single peak, retention time 4.71 min (*n*-Hexane/IPA 90:10); %ee > 99%.

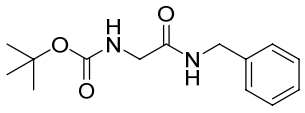


Chemical Formula: $C_{24}H_{24}N_2O_3$
Molecular Weight: 388.47

Benzyl (*S*)-(1-(benzylamino)-1-oxo-3-phenylpropan-2-yl)carbamate[17] (Table 2, entry 18)

Following the general procedure with Cbz-L-phenylalanine (0.0811 g, 0.271 mmol) and benzylamine (0.0319 g, 0.298 mmol), the product was purified by column chromatography using 30% EtOAc/hexane as the eluent; white solid (0.0881 g, 84% yield); mp 160-161 °C; R_f 0.47 (40% EtOAc/hexane); $[\alpha]_D^{26}$ +10.2 (c 1.03, $CHCl_3$)

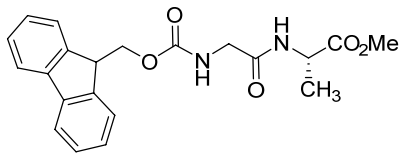
Ref[17]: $[\alpha]_D^{22}$ +8.0 (c 0.90, $CHCl_3$); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.34-7.03 (m, 15H), 6.98 (br s, 1H), 5.94 (br d, J = 8.0 Hz, 1H), 5.03-4.95 (m, 2H), 4.47-4.21 (m, 3H), 3.04 (d, J = 6.8 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 171.2, 156.2, 137.5, 136.4, 129.4, 128.6, 128.5, 128.4, 128.2, 127.9, 127.6, 127.4, 126.9, 67.0, 56.3, 43.3, 38.8; Chiral HPLC: Daicel IB; homogeneous single peak, retention time 4.441 min (*n*-Hexane/ IPA 90:10); %ee >99%.



Chemical Formula: $C_{14}H_{20}N_2O_3$
Molecular Weight: 264.32

***tert*-Butyl (2-(benzylamino)-2-oxoethyl)carbamate[18] (Table 2, entry 19)**

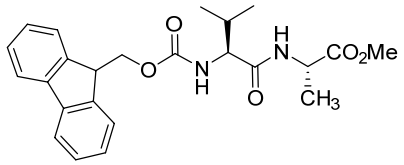
Following the general procedure with Boc-glycine (0.0475 g, 0.271 mmol) and benzylamine (0.0319 g, 0.298 mmol), the product was purified by column chromatography using 30% EtOAc/hexane as the eluent; yellow liquid (0.0638 g, 89% yield); R_f 0.47 (70% EtOAc/hexane); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.30-7.26 (br m, 5H), 6.87 (br s, 1H), 5.45 (br s, 1H), 4.42 (d, J = 3.2 Hz, 2H), 3.80 (s, 2H), 1.42 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.6, 156.2, 138.0, 128.7, 127.6, 127.5, 80.2, 44.4, 44.3, 28.3.



Chemical Formula: $C_{21}H_{22}N_2O_5$
Molecular Weight: 382.42

Methyl 2-(2-((9H-fluoren-9-yl)methoxy)carbonylamino)aceta-mido) propanoate[19] (Table 3, entry 1)

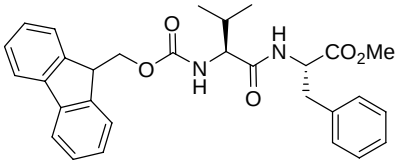
Following the general procedure with Fmoc-glycine (0.0806 g, 0.271 mmol) and L-alanine methyl ester hydrochloride (0.0416 g, 0.298 mmol), the product was purified by column chromatography using 60% EtOAc/hexane as the eluent; colorless oil (0.0836 g, 81% yield); R_f 0.36 (60% EtOAc/hexanes); $[\alpha]_D^{26}$ +11.5 (c 1.04, $CHCl_3$); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75 (d, J = 7.2 Hz, 2H), 7.58 (d, J = 7.2 Hz, 2H), 7.38 (t, J = 7.2 Hz, 2H), 7.29 (t, J = 7.2 Hz, 2H), 6.81 (br, d, J = 6.4 Hz, 1H), 5.74 (t, J = 5.2 Hz, 1H), 4.59 (t, J = 7.2 Hz, 1H), 4.40 (d, J = 6.8 Hz, 2H), 4.21 (t, J = 7.2 Hz, 1H), 4.11 (q, J = 7.2 Hz, 1H), 3.91 (t, J = 6.8 Hz, 1H), 3.72 (s, 3H), 1.40 (d, J = 7.2 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.3, 168.8, 156.7, 143.7, 141.3, 127.8, 127.10, 125.1, 120.0, 67.3, 52.6, 48.1, 47.1, 44.3, 18.2; Chiral HPLC: Daicel IB; homogeneous single peak, retention time 8.223 min (*n*-Hexane/IPA 90:10); %ee > 99%.



Chemical Formula: $C_{24}H_{28}N_2O_5$
Molecular Weight: 424.50

Methyl (((9H-fluoren-9-yl)methoxy)carbonyl)-*L*-valyl-*L*-alaninate[20] (Table 3, entry 2)

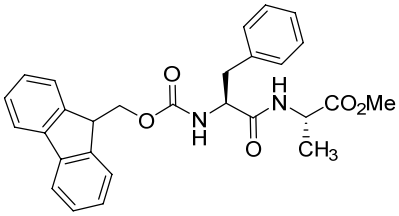
Following the general procedure with Fmoc-L-valine (0.0475 g, 0.271 mmol) and L-alanine methyl ester hydrochloride (0.0416 g, 0.298 mmol), the product was purified by column chromatography using 40% EtOAc/hexane as the eluent; white solid (0.0872 g, 76% yield); R_f 0.36 (40% EtOAc/hexanes); mp = 205- 207 °C; $[\alpha]_D^{26}$ -18.8 (c 1.00, $CHCl_3$) Ref[20]: $[\alpha]_D^{25}$ -18.1 (c 1.05, $CHCl_3$); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.77 (d, J = 6.8 Hz, 2H), 7.60 (d, J = 6.8 Hz, 2H), 7.40 (t, J = 6.8 Hz, 2H) , 7.30 (t, J = 6.8 Hz, 2H), 6.93 (br s, 1H), 5.92 (d, 1H, J = 8.8 Hz), 4.61 (t, J = 6.8 Hz, 1H), 4.43-4.32 (m, 2H), 4.23 (t, J = 6.8 Hz, 1H), 4.16-4.11 (m, 1H), 3.73 (s, 3H), 2.15-2.10 (m, 1H), 1.40 (d, J = 6.8 Hz, 3H), 1.01 (d, J = 7.6 Hz, 3H), 0.99 (d, J = 7.6 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.1, 171.4, 156.6, 143.9, 141.3, 127.7, 127.1, 125.1, 120.0, 67.2, 60.3, 52.5, 48.1, 47.1, 31.4, 19.1, 18.1.



Chemical Formula: $C_{30}H_{32}N_2O_5$
Molecular Weight: 500.60

Methyl (((9H-fluoren-9-yl)methoxy)carbonyl)-*L*-valyl-*L*-phenylalaninate[21] (Table 3, entry 3)

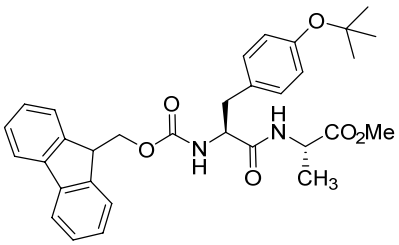
Following the general procedure with Fmoc-L-valine (0.0475 g, 0.271 mmol) and L-phenylalanine methyl ester hydrochloride (0.0643 g, 0.298 mmol), the product was purified by column chromatography using 30% EtOAc/hexane as the eluent; a white solid (0.1067 g, 79% yield); R_f 0.31 (30% EtOAc/hexanes); mp = 182-183 °C; C $[\alpha]_D^{26}$ +15.9 (c 1.02, $CHCl_3$) Ref[22]: $[\alpha]_D^{25}$ +15.5 (c 0.50, $CHCl_3$) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.79 (d, J = 7.2 Hz, 2H), 7.62 (d, J = 7.2 Hz, 2H), 7.42 (t, J = 7.2 Hz, 2H), 7.34 (t, J = 7.2 Hz, 2H), 7.28-7.20 (m, 3H), 7.10 (d, J = 3.6 Hz, 2H), 6.46 (br s, 1H), 5.47 (d, J = 6.8 Hz, 1H), 4.46 (t, J = 8.0 Hz, 1H), 4.32 (t, J = 6.8 Hz, 1H), 4.23 (t, J = 6.8 Hz, 1H), 4.06 (br, s, 1H), 3.72 (s, 3H), 3.18-3.01 (m, 2H), 2.11-2.07 (m, 1H), 0.96 (d, J = 6.0 Hz, 3H), 0.93 (d, J = 6.0 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 171.7, 170.9, 156.3, 143.9, 141.3, 135.6, 129.2, 128.6, 127.7, 127.2, 127.1, 125.1, 120.0, 67.1, 60.2, 53.1, 52.3, 47.2, 37.9, 31.2, 19.1, 17.8.



Chemical Formula: $C_{28}H_{28}N_2O_5$
Molecular Weight: 472.54

Methyl (((9H-fluoren-9-yl)methoxy)carbonyl)-*L*-phenylalanyl-*L*-alaninate[23] (Table 3, entry 4)

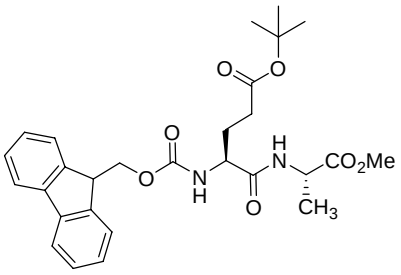
Following the general procedure with Fmoc-L-phenylalanine (0.0475 g, 0.271 mmol) and L-alanine methyl ester hydrochloride (0.0416 g, 0.298 mmol), the product was purified by column chromatography using 30% EtOAc/hexane as the eluent; white solid (0.1090 g, 85% yield); mp 182.0-183.0 °C; R_f 0.32 (30% EtOAc/hexanes); $[\alpha]_D^{26}$ -17.8 (c 0.94, $CHCl_3$) Ref[23]: $[\alpha]_D^{25}$ -18 (c 1.00, $CHCl_3$); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.73 (d, J = 7.6 Hz, 2H), 7.51 (t, J = 8.0 Hz, 2H), 7.37 (t, J = 7.6 Hz, 2H), 7.29-7.20 (m, 7H), 6.71 (br s, 1H), 5.70 (br s, 1H), 4.53-4.50 (m, 2H), 4.41-4.37 (m, 1H), 4.27 (br s, 1H), 4.17-4.14 (m, 1H), 3.67 (s, 3H), 3.08 (d, 2H, J = 3.6 Hz), 1.32 (d, J = 6.4 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 172.9, 170.7, 156.0, 143.8, 141.8, 136.4, 132.0, 129.4, 128.6, 128.5, 127.7, 125.1, 120.0, 67.1, 56.0, 52.5, 48.2, 47.1, 38.6, 18.2.



Chemical Formula: $C_{32}H_{36}N_2O_6$
Molecular Weight: 544.65

Methyl ((S)-2-((((9H-fluoren-9-yl)methoxy)carbonyl)amino)-3-(4-(tert-butoxy)phenyl)propanoyl)-*L*-alaninate[24] (Table 3, entry 5)

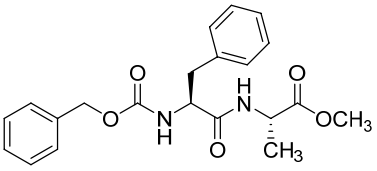
Following the general procedure with Fmoc-L-Tyr(O^{t}Bu)-OH (0.0475 g, 0.271 mmol) and L-alanine methyl ester hydrochloride (0.0416 g, 0.298 mmol), the product was purified by column chromatography using 30% EtOAc/hexane as the eluent; white solid (0.1364 g, 92% yield); mp 163.0-164.0 °C; R_f 0.36 (30% EtOAc/hexanes); $[\alpha]_D^{26}$ -9.1 (c 0.85, DMF) Ref[25]: $[\alpha]_D^{20}$ -6.6 (c 0.60, DMF); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75 (d, J = 7.2 Hz, 2H), 7.55 (br s, 2H), 7.39 (t, J = 7.2 Hz, 2H), 7.30 (t, J = 7.2 Hz, 2H), 7.08 (br s, 2H), 6.90 (d, J = 7.2 Hz, 2H), 6.36 (br s, 1H), 5.46 (br s, 1H), 4.50-4.34 (m, 4H), 4.18 (t, J = 6.8 Hz, 1H), 3.70 (s, 3H), 3.11-2.96 (m, 2H), 1.32 (d, J = 10.8 Hz, 3H), 1.31 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 172.8, 170.7, 156.0, 154.4, 143.8, 141.3, 132.1, 129.9, 128.6, 127.7, 127.1, 125.1, 124.2, 120.0, 78.4, 67.1, 56.1, 52.4, 48.2, 47.1, 38.1, 28.8, 18.2.



Chemical Formula: $C_{28}H_{34}N_2O_7$
Molecular Weight: 510.59

tert-Butyl (S)-4-((((9H-fluoren-9-yl)methoxy)carbonyl)amino)-5-(((S)-1-methoxy-1-oxopropan-2-yl)amino)-5-oxopentanoate[24] (Table 3, entry 6)

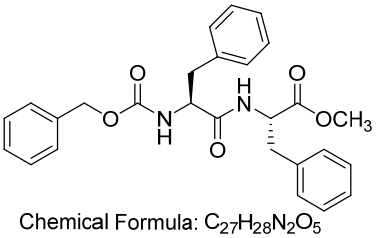
Following the general procedure with Fmoc-L-Glu(O^{t}Bu)-OH (0.0475 g, 0.271 mmol) and L-alanine methyl ester hydrochloride (0.0416 g, 0.298 mmol), the product was purified by column chromatography using 40% EtOAc/hexane as the eluent; white solid (0.1096 g, 79% yield); mp 109.0-110.0 °C; R_f 0.46 (40% EtOAc/hexanes); $[\alpha]_D^{26}$ -15.7 (c 0.90, $CHCl_3$); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75 (d, J = 7.2 Hz, 2H), 7.59 (d, J = 7.2 Hz, 2H), 7.39 (t, J = 7.2 Hz, 2H), 7.30 (t, J = 7.2 Hz, 2H), 6.91 (br s, 1H), 5.77 (br s, 1H), 4.57 (t, J = 7.2 Hz, 1H), 4.38-4.19 (m, 4H), 3.74 (s, 3H), 2.42 (d, J = 5.2 Hz, 2H), 2.10-1.93 (m, 2H), 1.46 (s, 9H), 1.32 (d, J = 7.2 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.0, 172.8, 171.2, 156.3, 143.9, 141.3, 127.7, 127.1, 125.1, 120.0, 80.9, 67.1, 54.0, 52.4, 48.2, 47.1, 31.3, 28.4, 28.1, 17.9.



Chemical Formula: $C_{21}H_{24}N_2O_5$
Molecular Weight: 384.43

Methyl ((benzyloxy)carbonyl)-*L*-phenylalanyl-*L*-alaninate[26] (Table 3, entry 7)

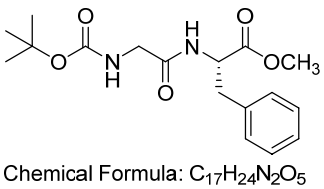
Following the general procedure with Cbz-L-phenylalanine (0.0475 g, 0.271 mmol) and L-alanine methyl ester hydrochloride (0.0416 g, 0.298 mmol), the product was purified by column chromatography using 30% EtOAc/hexane as the eluent; white solid (0.0868 g, 83% yield); mp 126.0-127.0 °C; R_f 0.35 (30% EtOAc/hexanes); $[\alpha]_D^{26}$ -24.1 (c 1.10, EtOH) Ref[26]: $[\alpha]_D^{25}$ -23.3 (c 1.20, EtOH); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.33-7.19 (m, 10H), 6.73 (br s, 1H), 5.63 (d, J = 8.0 Hz, 1H), 5.07 (m, 2H), 4.54-4.51 (m, 2H), 3.71 (s, 3H), 3.09 (d, J = 5.2 Hz, 2H), 1.33 (d, J = 6.4 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 172.9, 170.7, 156.0, 136.4, 129.4, 128.6, 128.5, 128.2, 128.0, 127.0, 67.0, 56.0, 52.4, 48.1, 38.6, 18.1.



Chemical Formula: $C_{27}H_{28}N_2O_5$
Molecular Weight: 460.53

Methyl 2-(2-(benzyloxycarbonylamino)-3-phenylpropanamido)-3-phenylpropanoate[27] (Table 3, entry 8)

Following the general procedure with Cbz-L-phenylalanine (0.0475 g, 0.271 mmol) and L-phenylalanine methyl ester hydrochloride (0.0643 g, 0.298 mmol), the product was purified by column chromatography using 30% EtOAc/hexane as the eluent; white solid (0.0887 g, 71% yield); mp 132.0-133.0 °C; R_f 0.46 (30% EtOAc/hexanes); $[\alpha]_D^{26}$ -23.7 (c 0.940, $CHCl_3$) Ref[27]: $[\alpha]_D^{25}$ -21.79 (c 1.00, $CHCl_3$); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.39 – 7.18 (m, 13H), 7.00 (d, J = 4.4 Hz, 2H), 6.37 (d, J = 6.8 Hz, 1H), 5.36(d, J = 7.2 Hz, 1H), 5.09 (s 2H), 4.81 (dd, J = 13.6, 6.0 Hz, 1H), 4.45 (br d, J = 6.8 Hz, 1H), 3.69 (s, 3H), 3.12 – 3.00 (m, 4H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 171.4, 170.5, 155.9, 136.2, 135.6, 129.4, 129.2, 128.7, 128.6, 128.2, 128.0, 127.2, 127.0, 67.1, 56.0, 53.3, 52.3, 37.9.

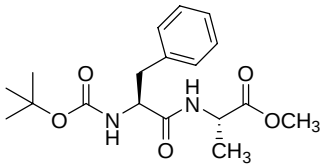


Chemical Formula: $C_{17}H_{24}N_2O_5$
Molecular Weight: 336.39

Methyl (*tert*-butoxycarbonyl)glycyl-*L*-phenylalaninate[28] (Table 3, entry 9)

Following the general procedure with Boc-glycine (0.0475 g, 0.271 mmol) L-phenylalanine methyl ester hydrochloride (0.0643 g, 0.298 mmol), the product was purified by column chromatography using 30% EtOAc/hexane as the eluent; colorless oil (0.0780 g, 88% yield); R_f 0.32 (40% EtOAc/hexanes); $[\alpha]_D^{26}$ -11.3 (c 0.85, MeOH); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.30-7.08 (5H, m), 7.00 (br d, J =7.2 Hz, 1H), 5.62 (br s, 1H), 4.84 (br d, J =6.8 Hz, 1H), 3.77-3.69 (m, 3H), 3.65 (3H, s), 3.13-3.02 (m, 2H), 1.41 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 171.8, 169.4, 156.1,

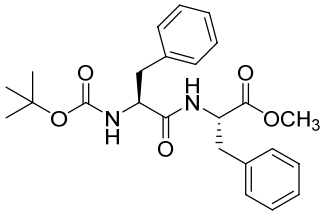
135.9, 129.2, 128.6, 127.0, 80.0, 55.8, 53.2, 52.2, 37.8, 28.3; Chiral HPLC: Daicel IB; homogeneous single peak, retention time 4.936 min (*n*-Hexane/IPA 90:10); %ee > 99%.



Chemical Formula: $C_{18}H_{26}N_2O_5$
Molecular Weight: 350.41

Methyl (*tert*-butoxycarbonyl)-*L*-phenylalanyl-*L*-alaninate[29] (Table 3, entry 10)

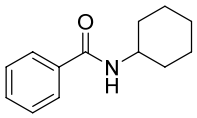
Following the general procedure with Boc-L-phenylalanine (0.0475 g, 0.271 mmol) and L-alanine methyl ester hydrochloride (0.0416 g, 0.298 mmol), the product was purified by column chromatography using 30% EtOAc/hexane as the eluent; colorless oil (0.0733 g, 77% yield); $[\alpha]_D^{26}$ -20.1 (c 0.94, MeOH) Ref[29]: $[\alpha]_D^{22}$ -18.5 (c 0.50, MeOH); R_f 0.34 (30% EtOAc/hexanes); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.29-7.22 (m, 5H), 6.66 (br s, 1H) , 5.20 (br s, 1H), 4.53 (br s, 1H), 4.41 (br s, 1H), 3.72 (s, 3H), 3.07 (br s, 2H) , 1.41 (s, 9H), 1.36 (d, 3H, J = 7.2 Hz); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 172.9, 171.0, 155.4, 136.5, 129.4, 128.6, 126.9, 80.5, 55.6, 52.5, 48.1, 38.4, 28.2, 18.3.



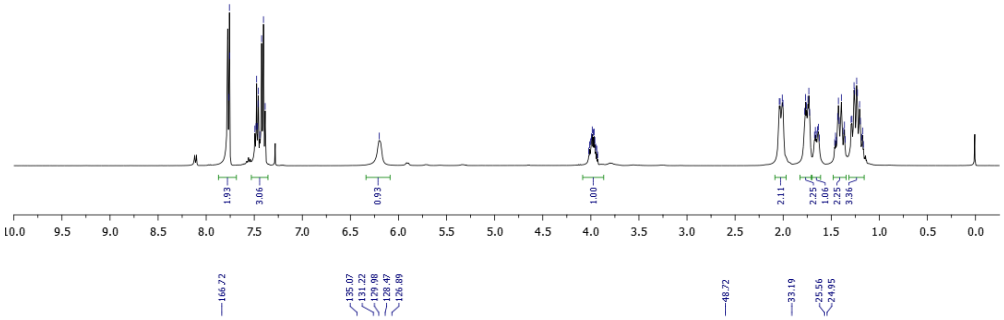
Chemical Formula: $C_{24}H_{30}N_2O_5$
Molecular Weight: 426.51

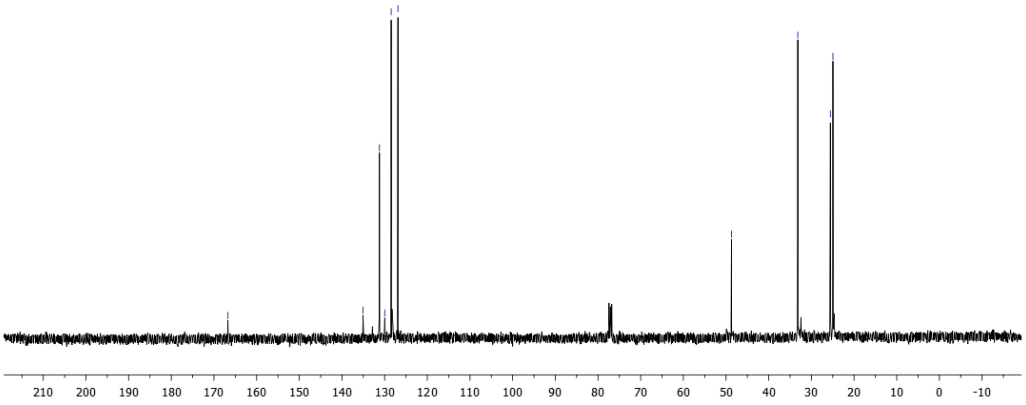
Methyl (tert-butoxycarbonyl)-L-phenylalanyl-L-phenylalaninate[30]

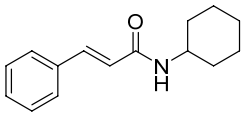
(Table 3, entry 11) Following the general procedure with Boc-L-phenylalanine (0.0475 g, 0.271 mmol) and L-phenylalanine methyl ester hydrochloride (0.0643 g, 0.298 mmol), the product was purified by column chromatography using 30% EtOAc/hexane as the eluent;white solid (0.0893 g, 77% yield); mp 110.0-111.0 °C; R_f 0.46 (30% EtOAc/hexanes); $[\alpha]_D^{26}$ +26.7 (c 0.95, $CHCl_3$) Ref[30]: $[\alpha]_D^{21}$ +23.2 (c 1.00, $CHCl_3$); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.30-7.20 (m, 8H), 7.01 (d, J = 5.6 Hz, 2H), 6.39 (br d, J = 6.0 Hz, 1H) , 5.03 (br s, 1H), 4.81 (br d, J = 5.6 Hz, 1H), 4.36 (br s, 1H), 3.68 (s, 3H), 3.06 (t, J = 6.0 Hz, 2H) , 1.42 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 171.4, 170.8, 155.3, 136.6, 135.7, 129.4, 129.2, 128.6, 128.54, 127.1, 127.0, 80.1, 53.3, 52.2, 38.0, 28.2.



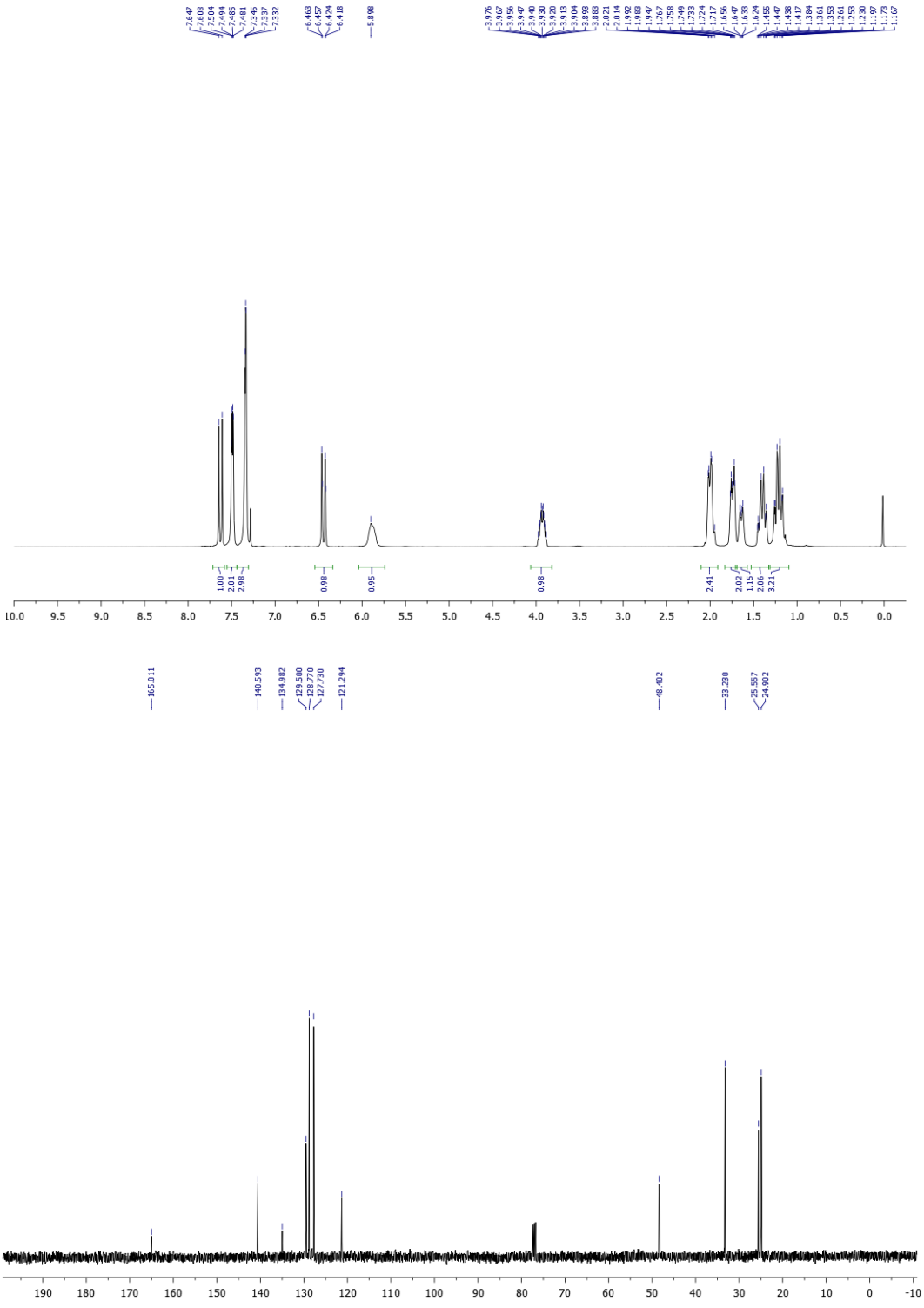
^{1}H NMR (400 MHz, $CDCl_3$); ^{13}C NMR (100 MHz, $CDCl_3$)

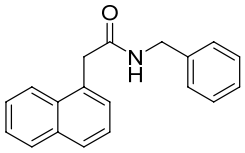




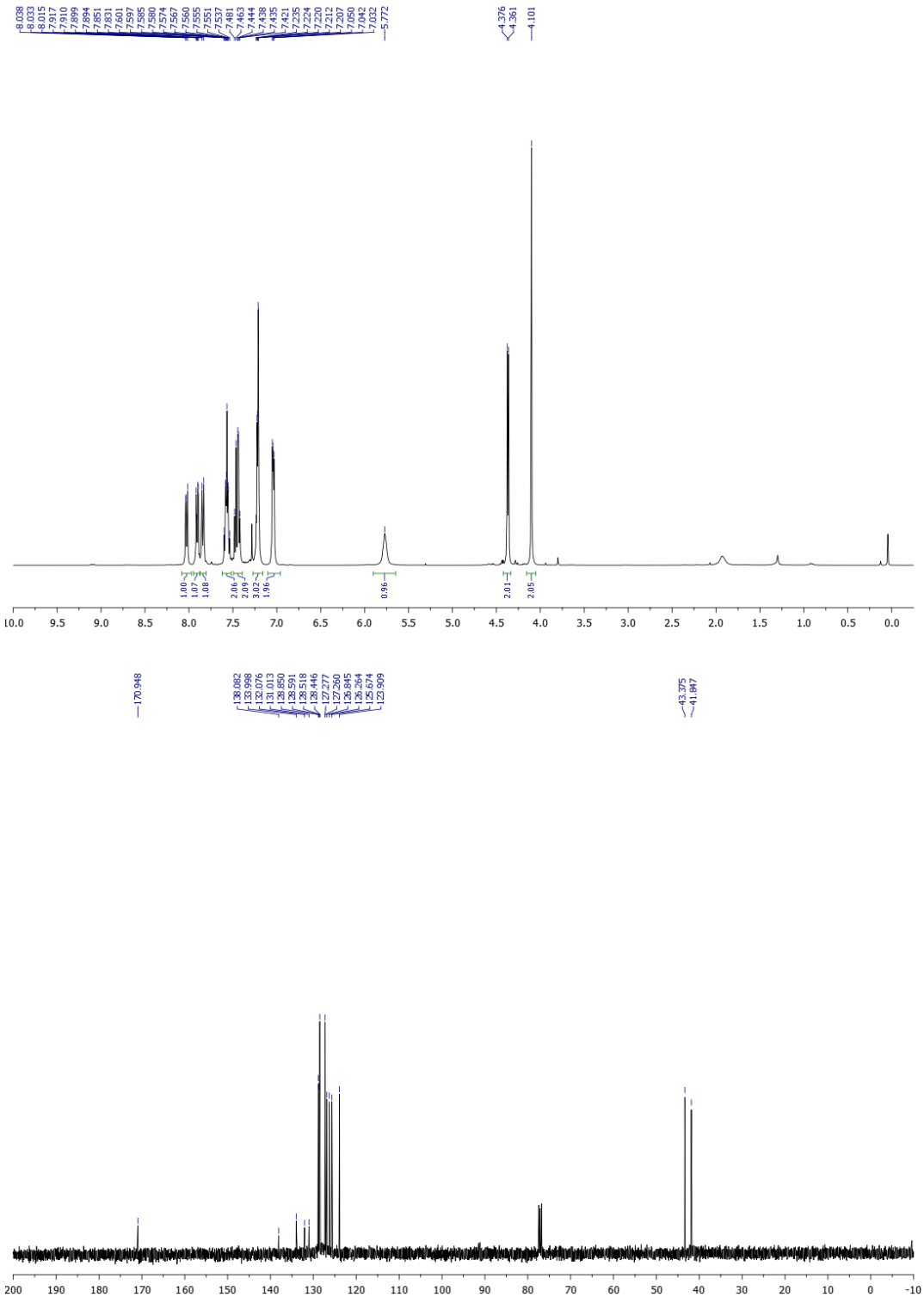


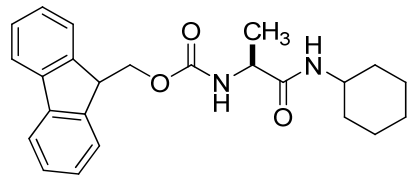
^{1}H NMR (400 MHz, $CDCl_3$); ^{13}C NMR (100 MHz, $CDCl_3$)



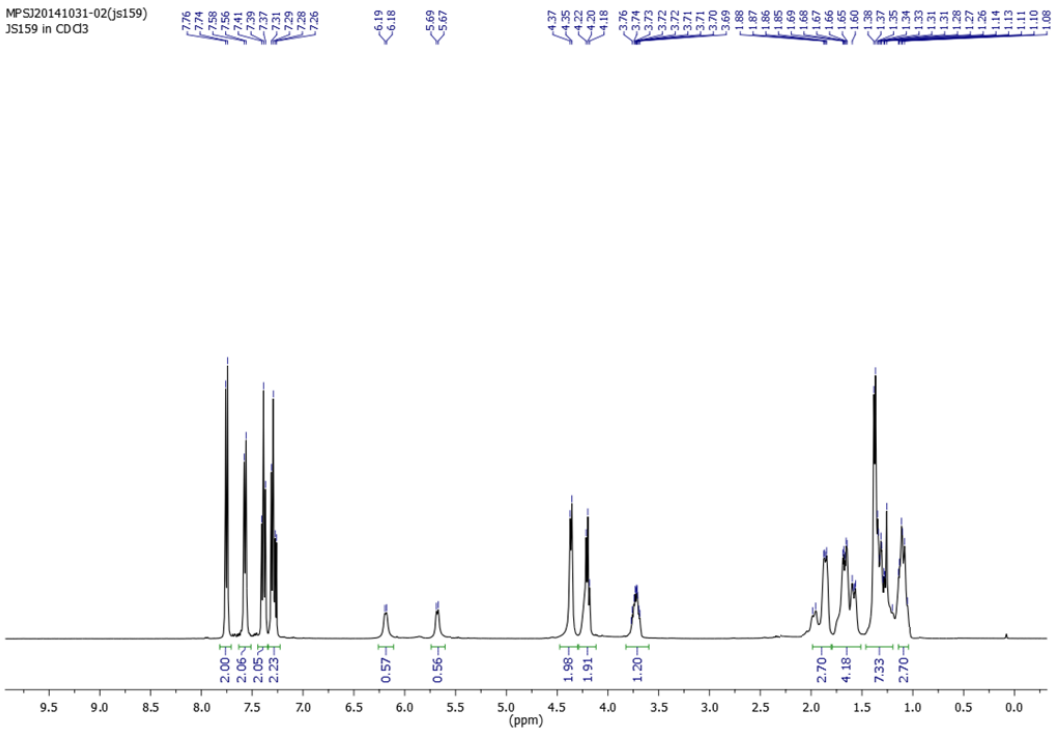


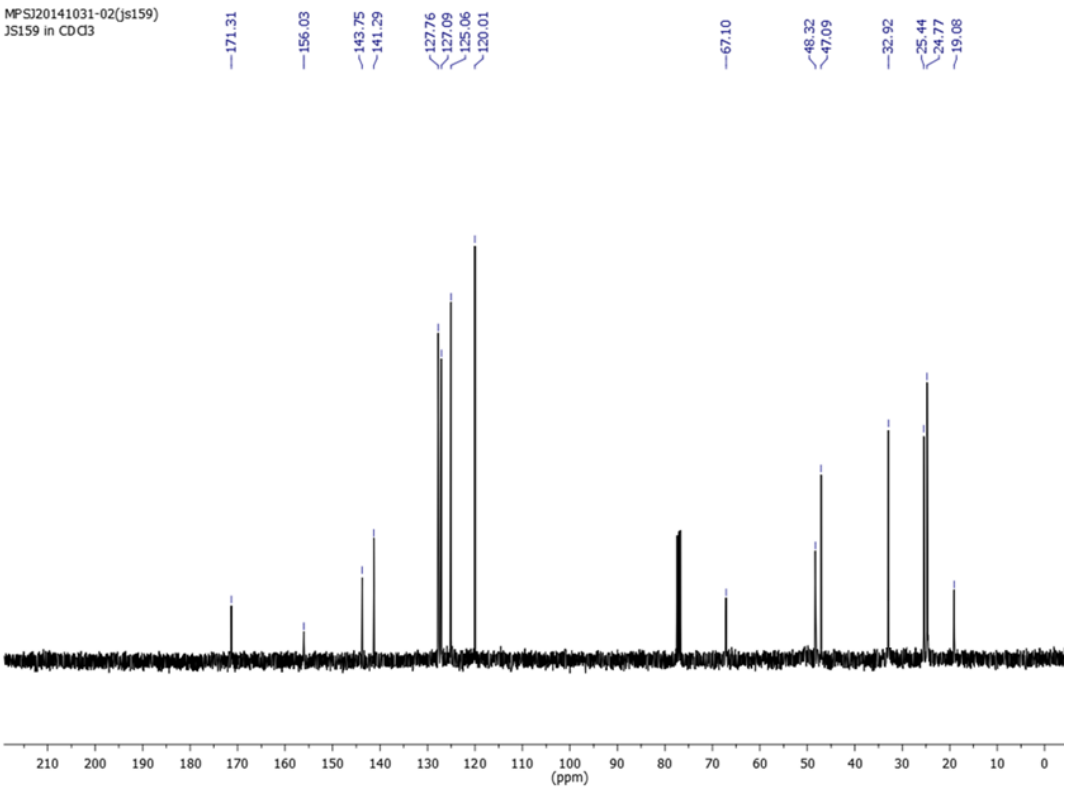
^{1}H NMR (400 MHz, $CDCl_3$); ^{13}C NMR (100 MHz, $CDCl_3$)

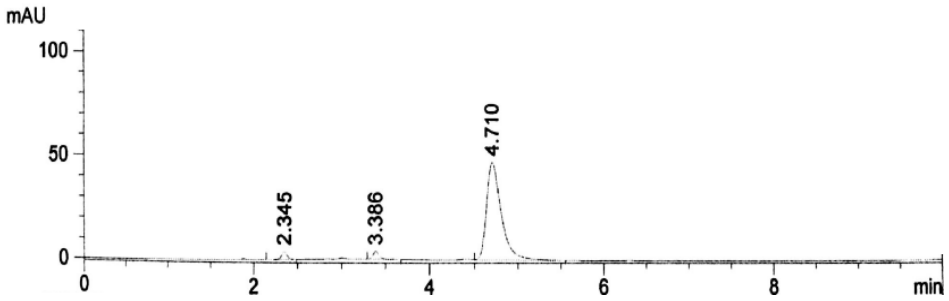




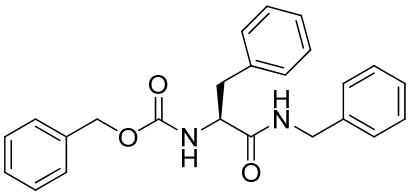
^{1}H NMR (400 MHz, $CDCl_3$); ^{13}C NMR (100 MHz, $CDCl_3$)



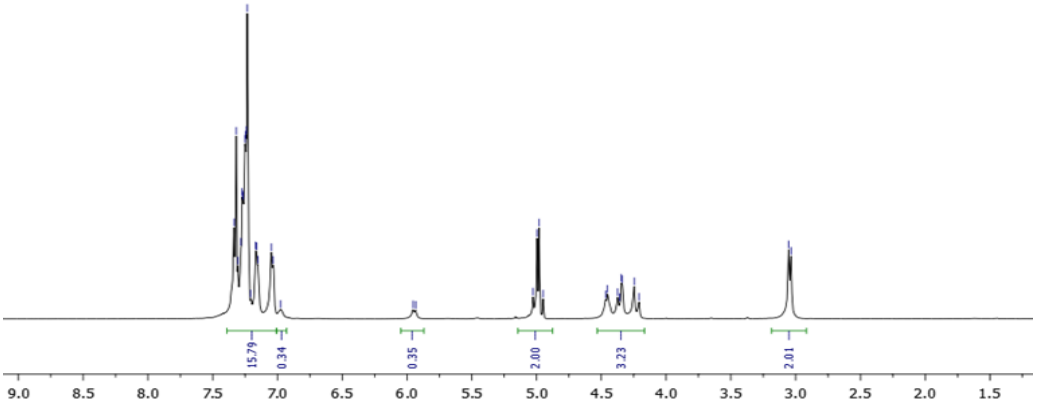


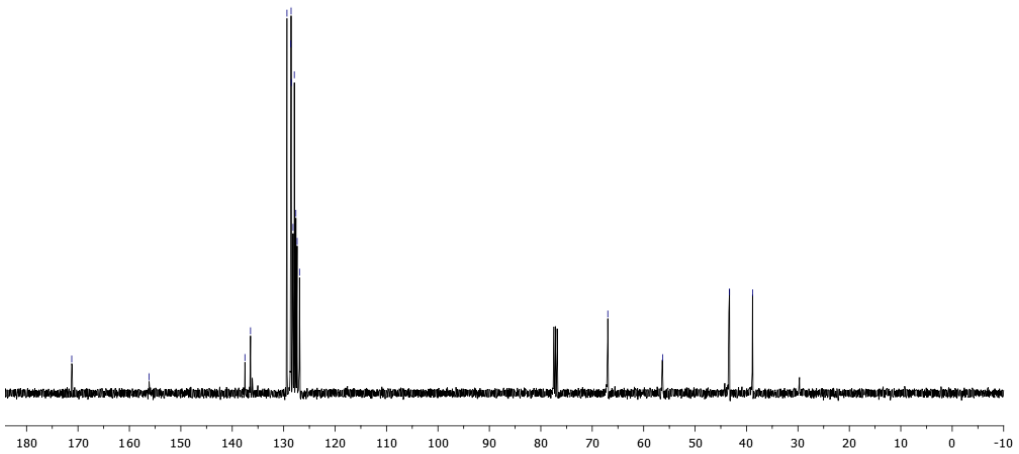


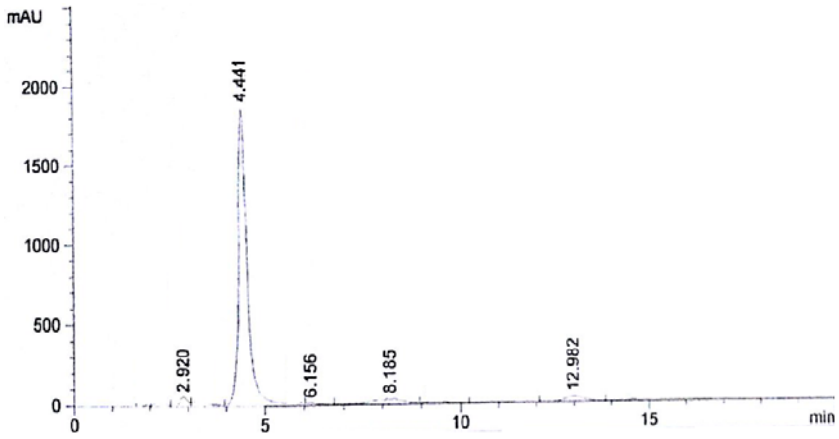
Chiral HPLC chromatogram of (9H-fluoren-9-yl)methyl 1-(cyclohexylamino)-1-oxopropan-2-ylcarbamate (Table 2, entry 17)



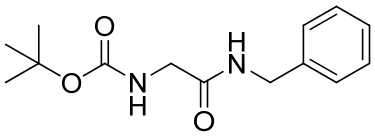
^{1}H NMR (400 MHz, $CDCl_3$); ^{13}C NMR (100 MHz, $CDCl_3$)



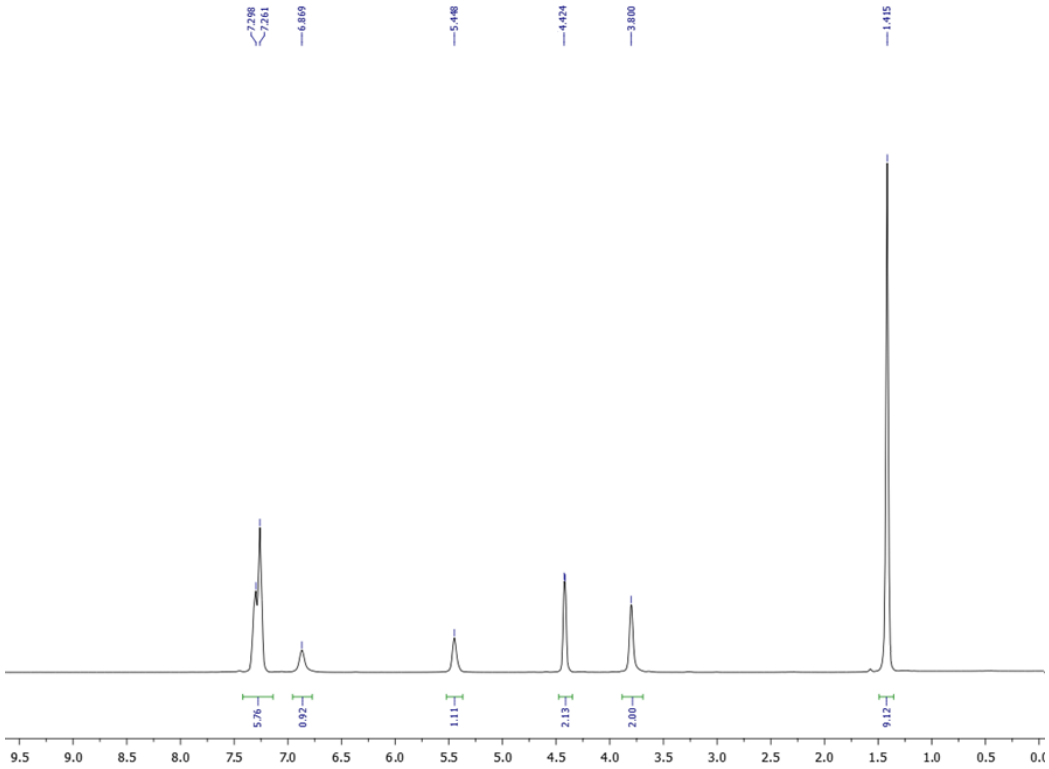


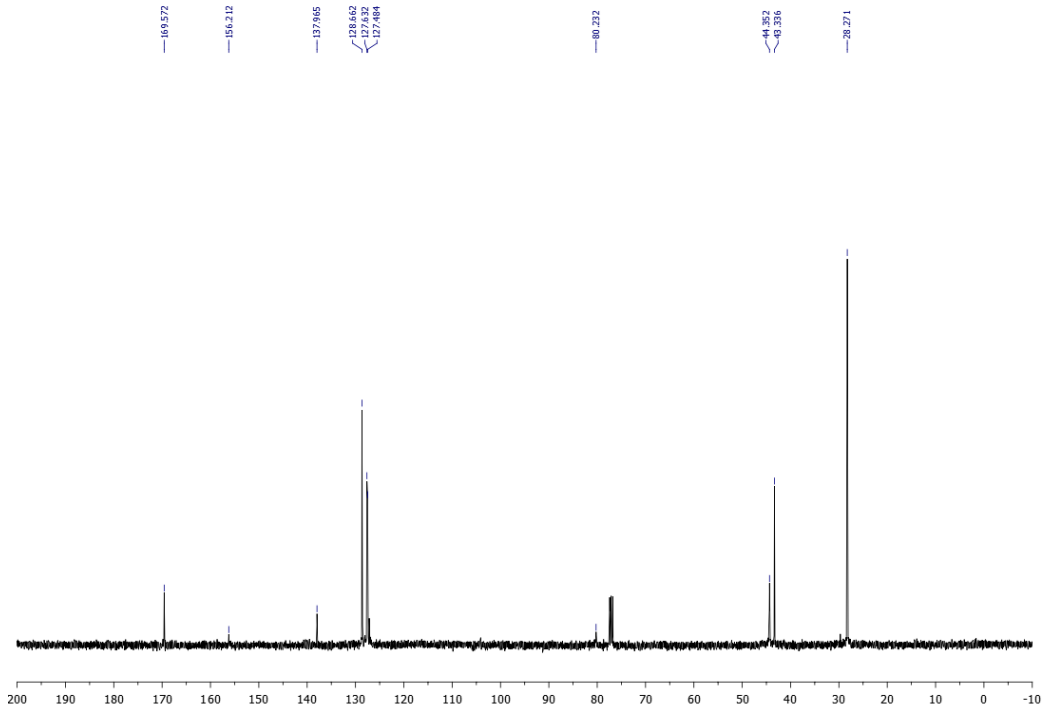


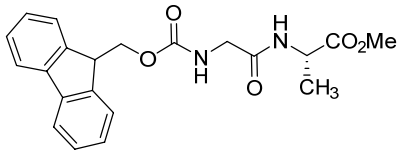
Chiral HPLC chromatogram of benzyl (*S*)-(1-(benzylamino)-1-oxo-3-phenylpropan-2-yl)carbamate (Table 2, entry 18)



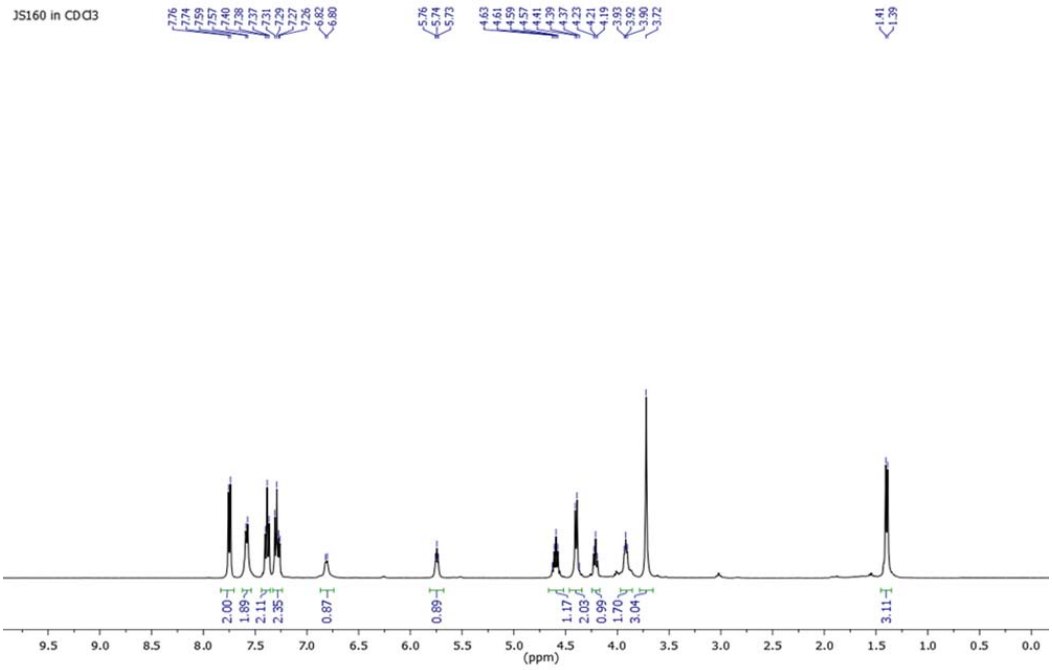
^{1}H NMR (400 MHz, $CDCl_3$); ^{13}C NMR (100 MHz, $CDCl_3$)

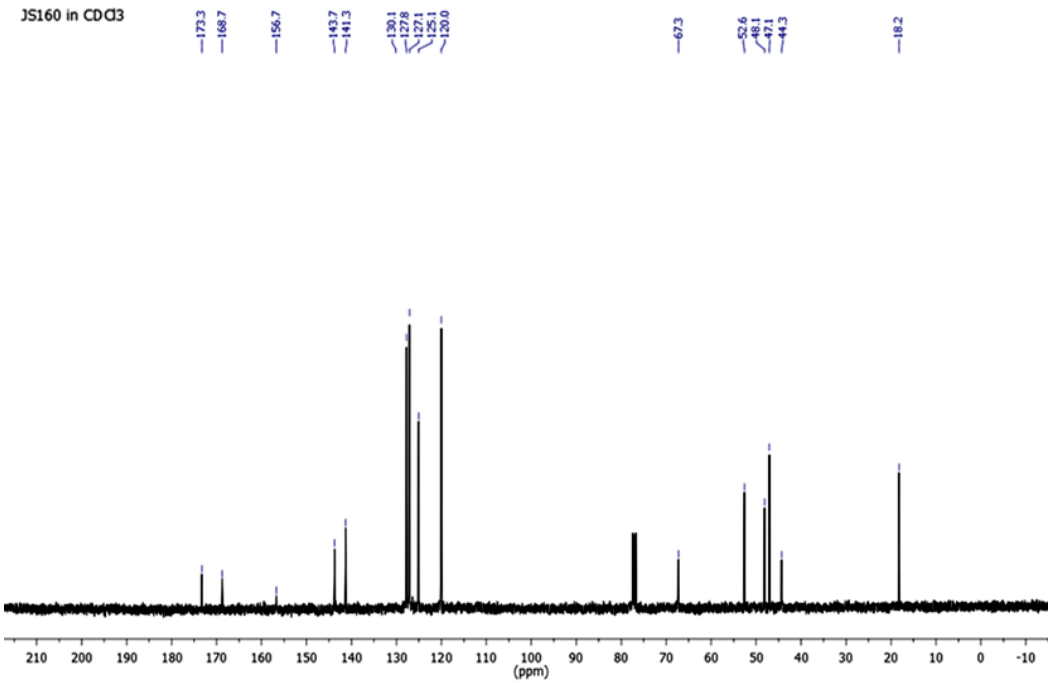


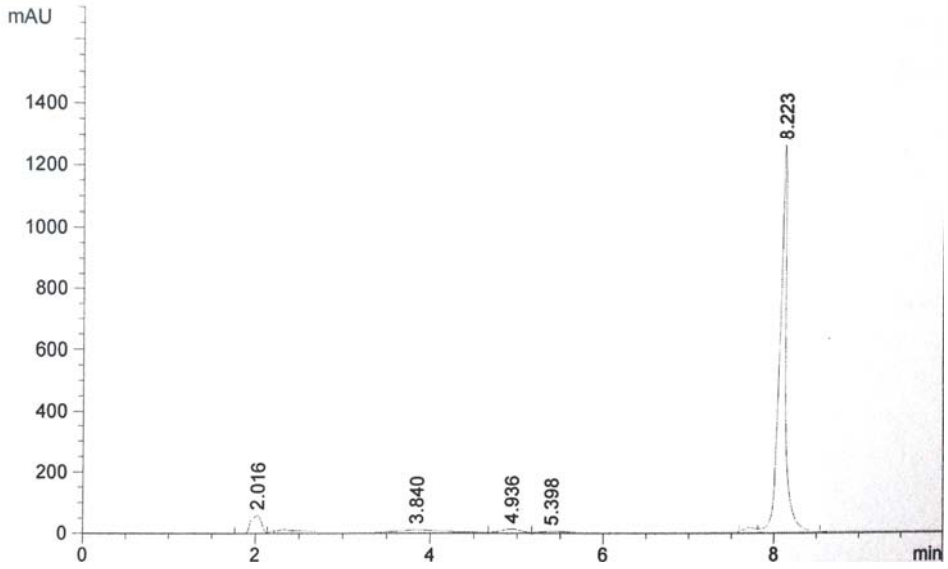




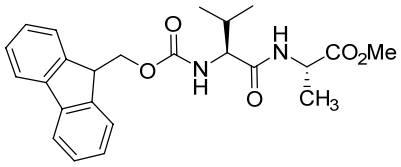
^{1}H NMR (400 MHz, $CDCl_3$); ^{13}C NMR (100 MHz, $CDCl_3$)



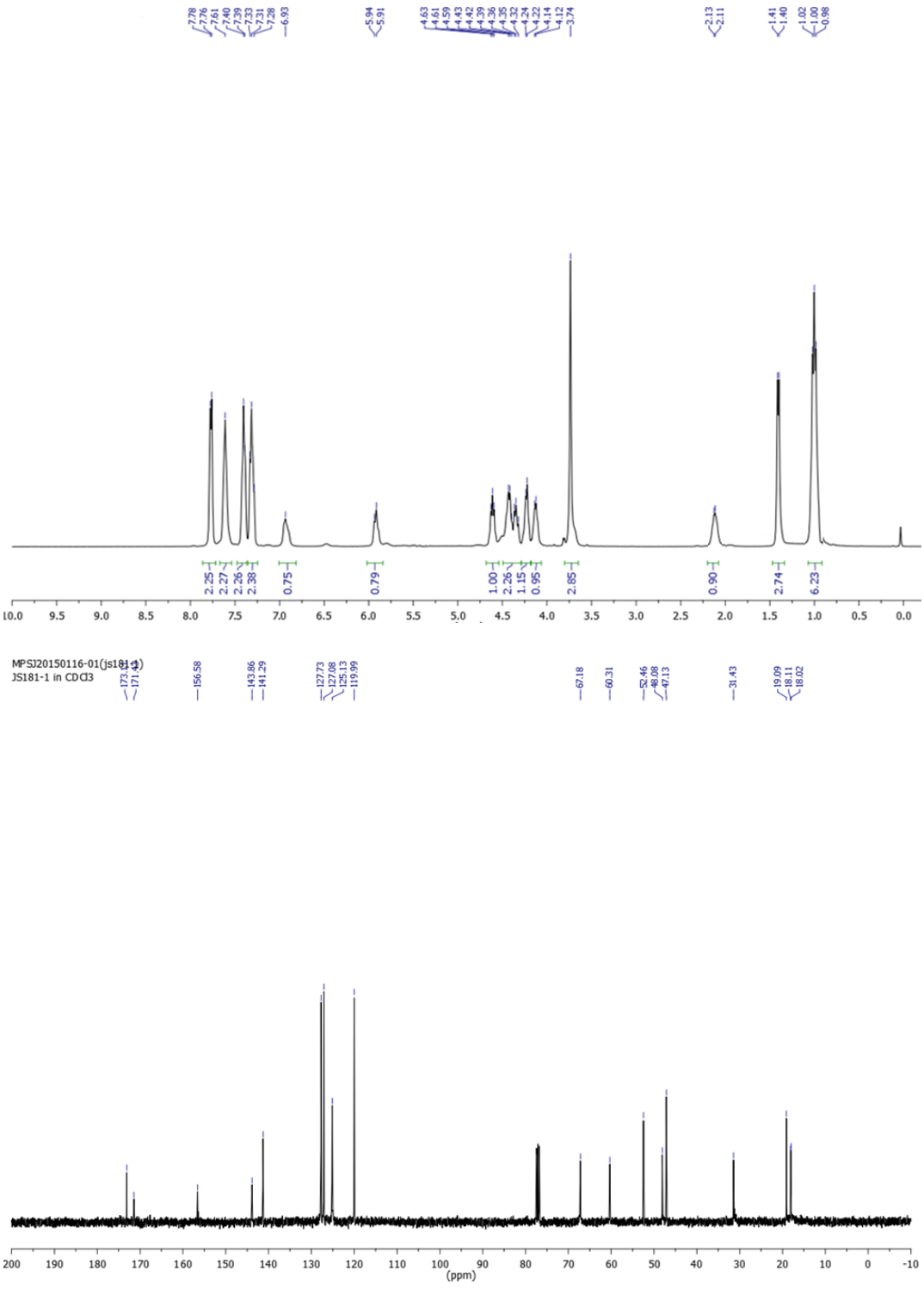


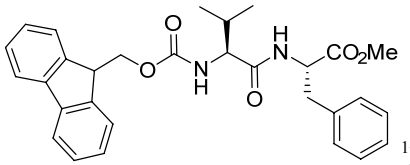


Chiral HPLC chromatogram of Fmoc-L-Gly-L-Ala-OMe (Table 3, entry 1)

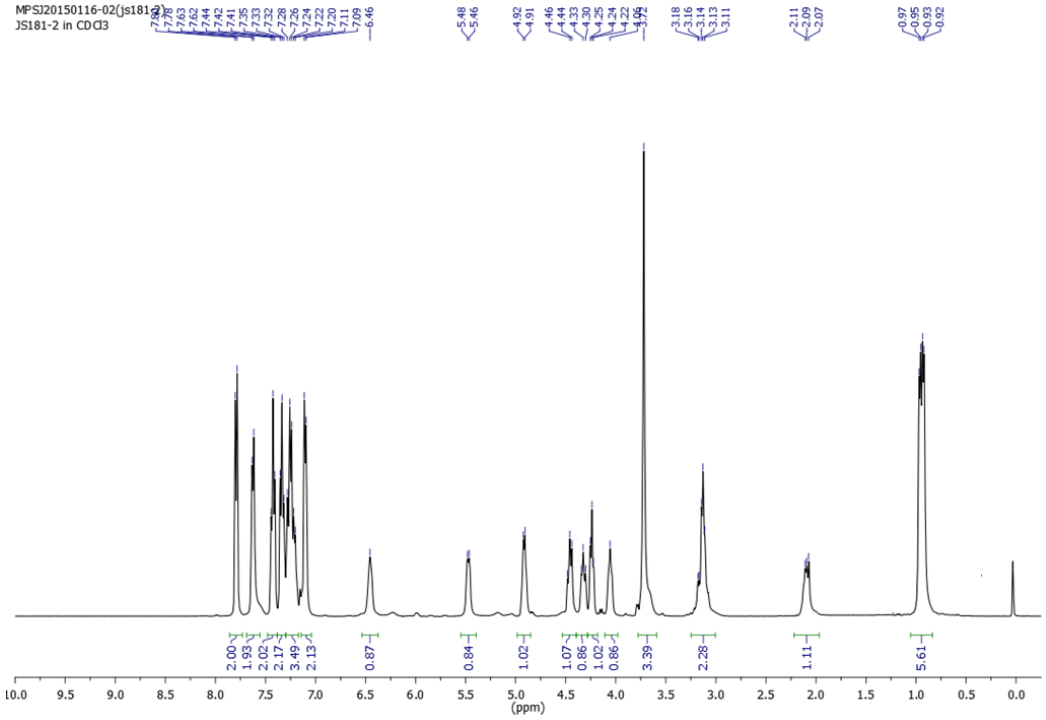


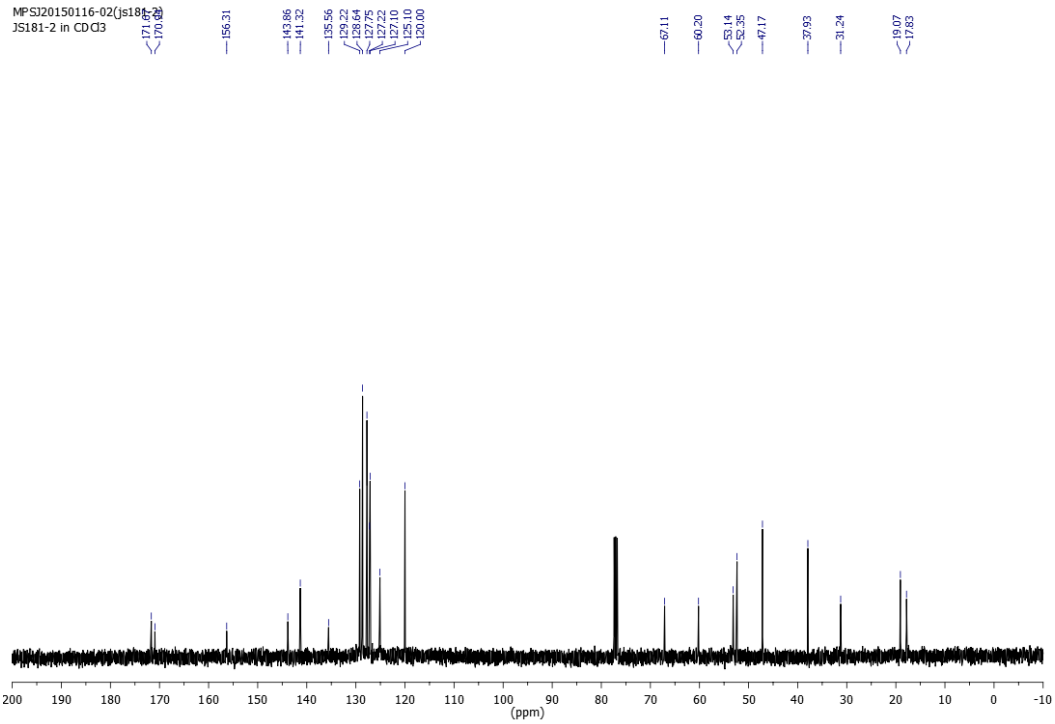
^{1}H NMR (400 MHz, $CDCl_3$); ^{13}C NMR (100 MHz, $CDCl_3$)

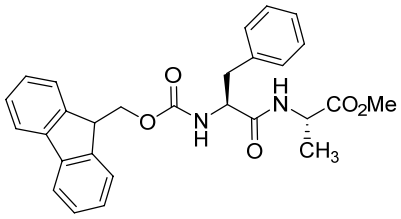




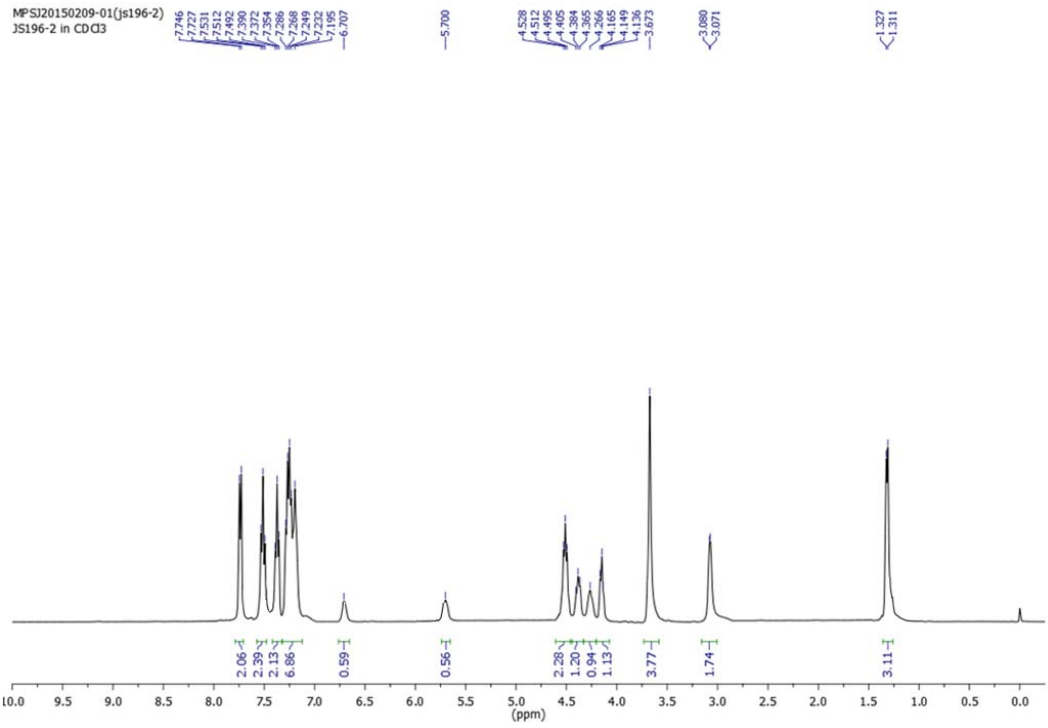
^{1}H NMR (400 MHz, $CDCl_3$); ^{13}C NMR (100 MHz, $CDCl_3$)

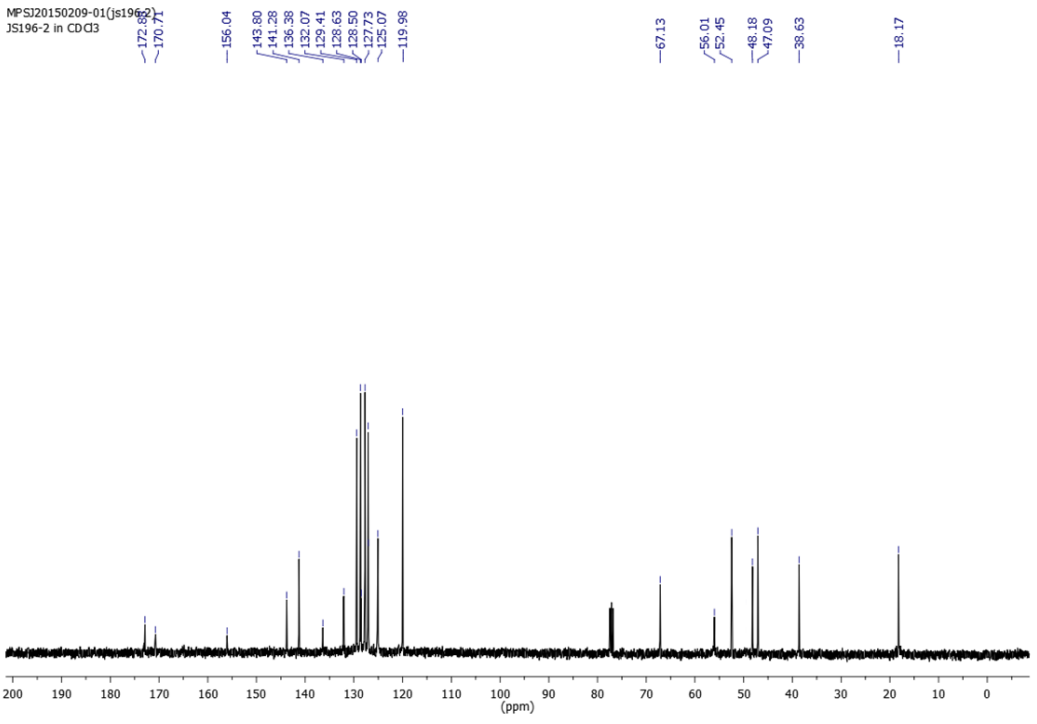


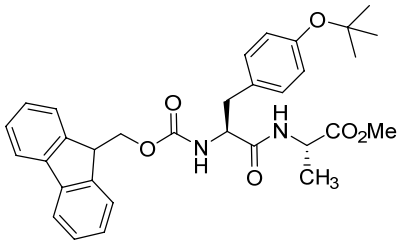




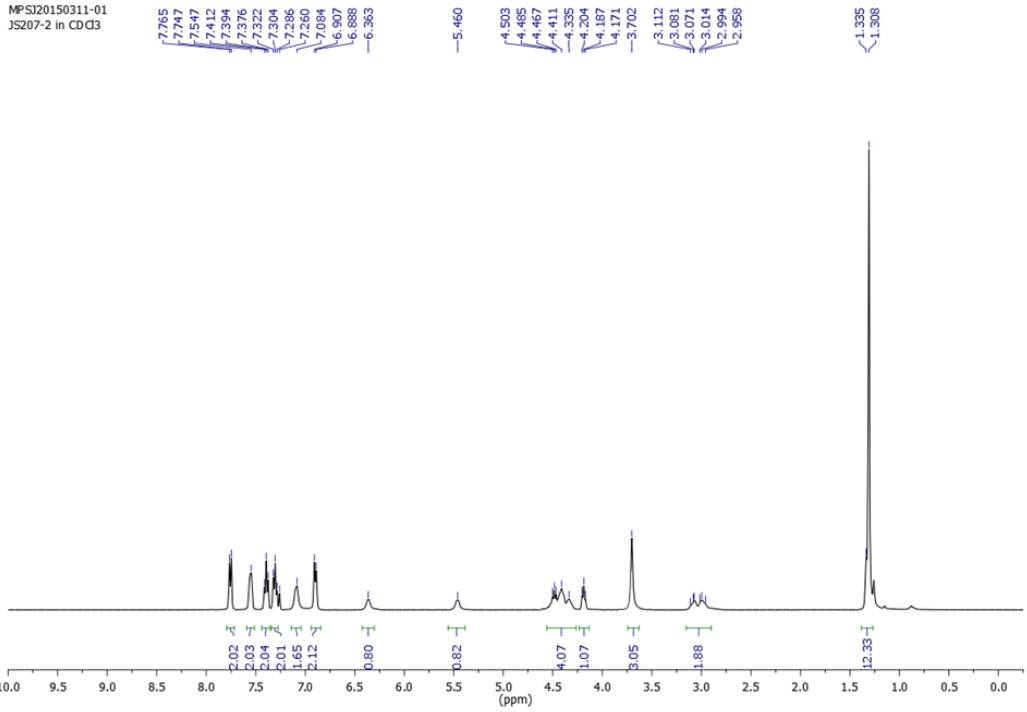
^{1}H NMR (400 MHz, $CDCl_3$); ^{13}C NMR (100 MHz, $CDCl_3$)

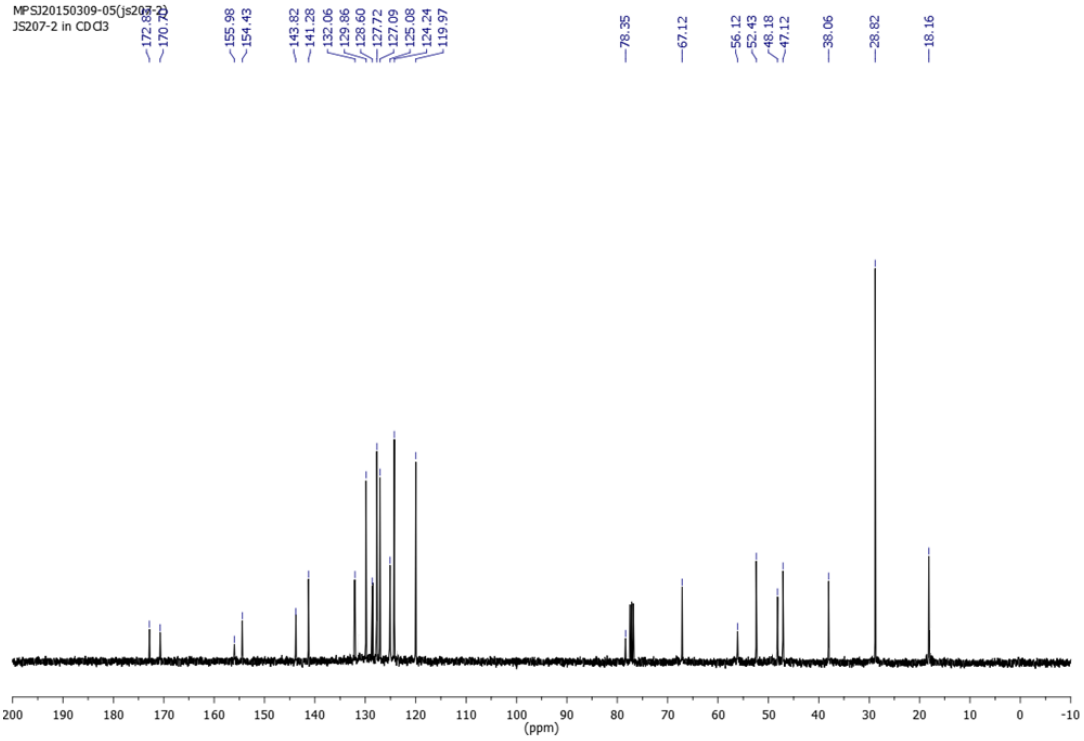


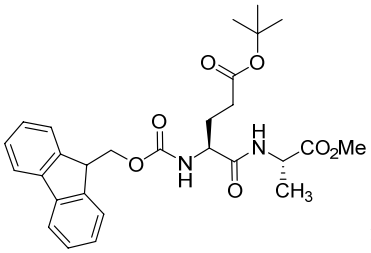




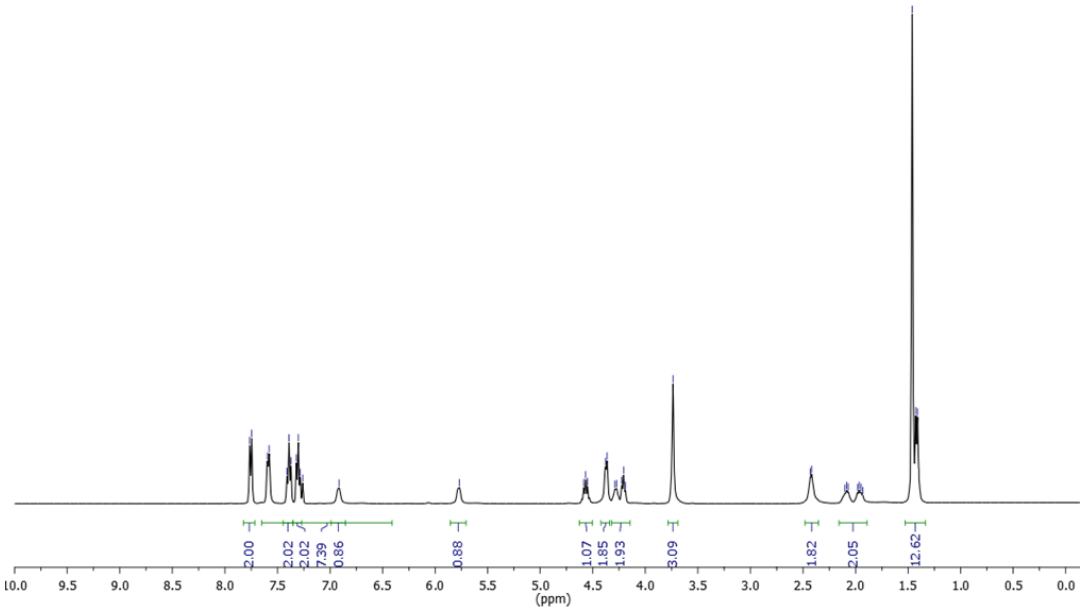
^{1}H NMR (400 MHz, $CDCl_3$); ^{13}C NMR (100 MHz, $CDCl_3$)

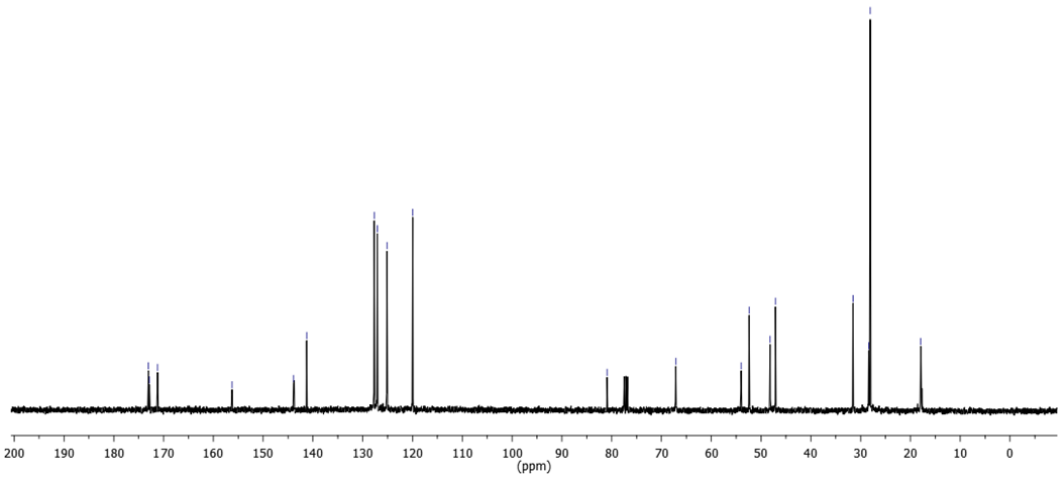


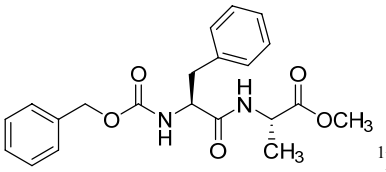




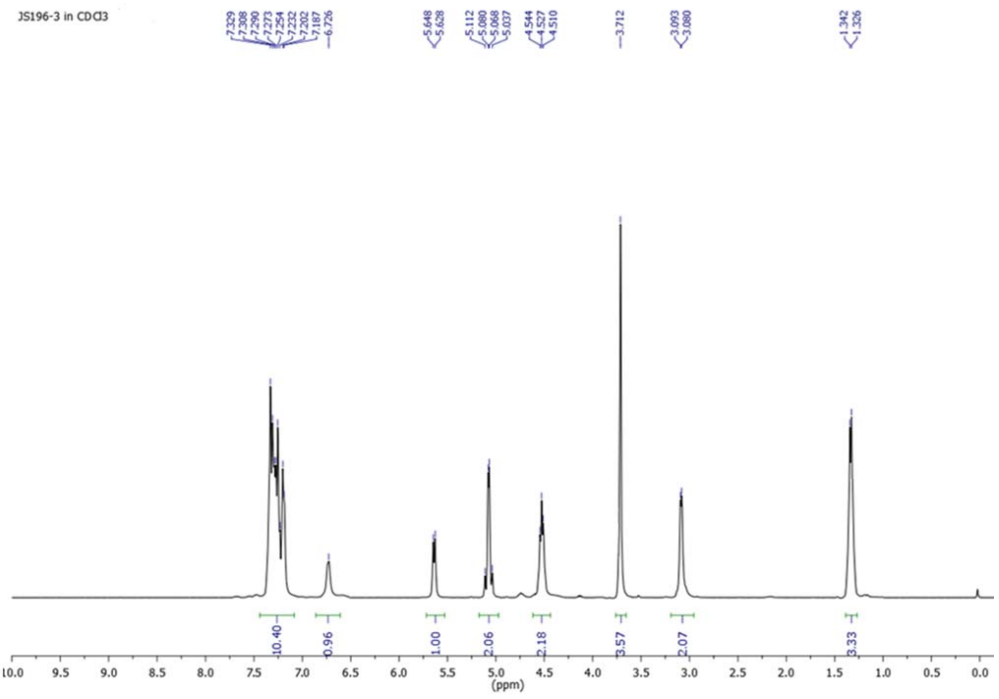
^{1}H NMR (400 MHz, $CDCl_3$); ^{13}C NMR (100 MHz, $CDCl_3$)

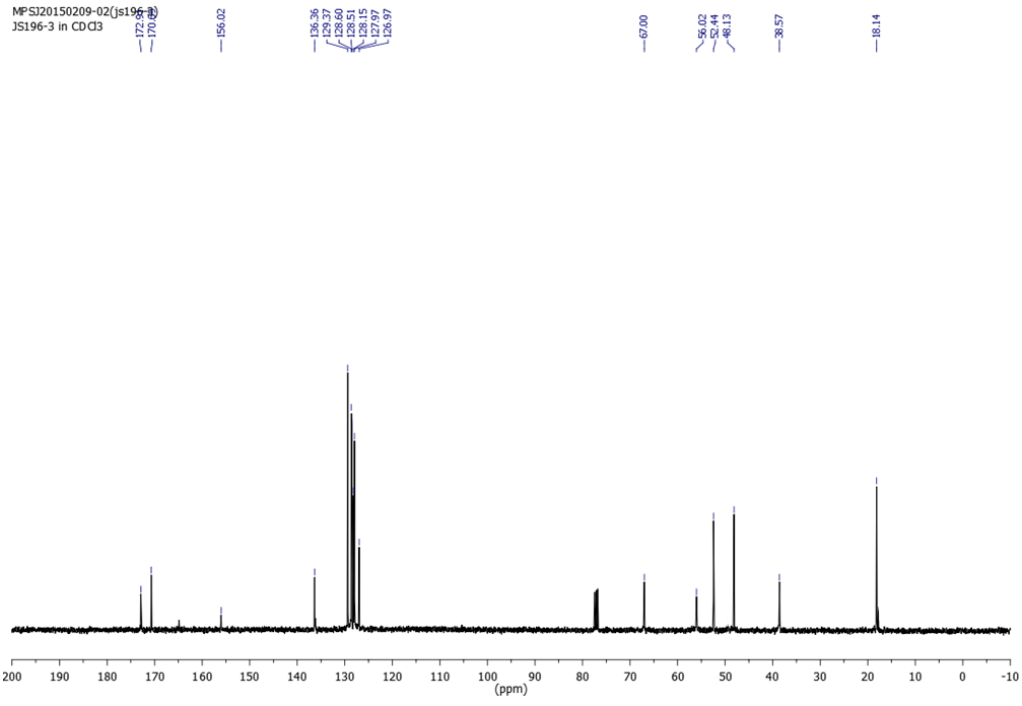


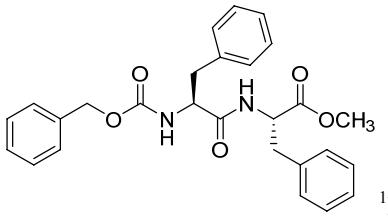




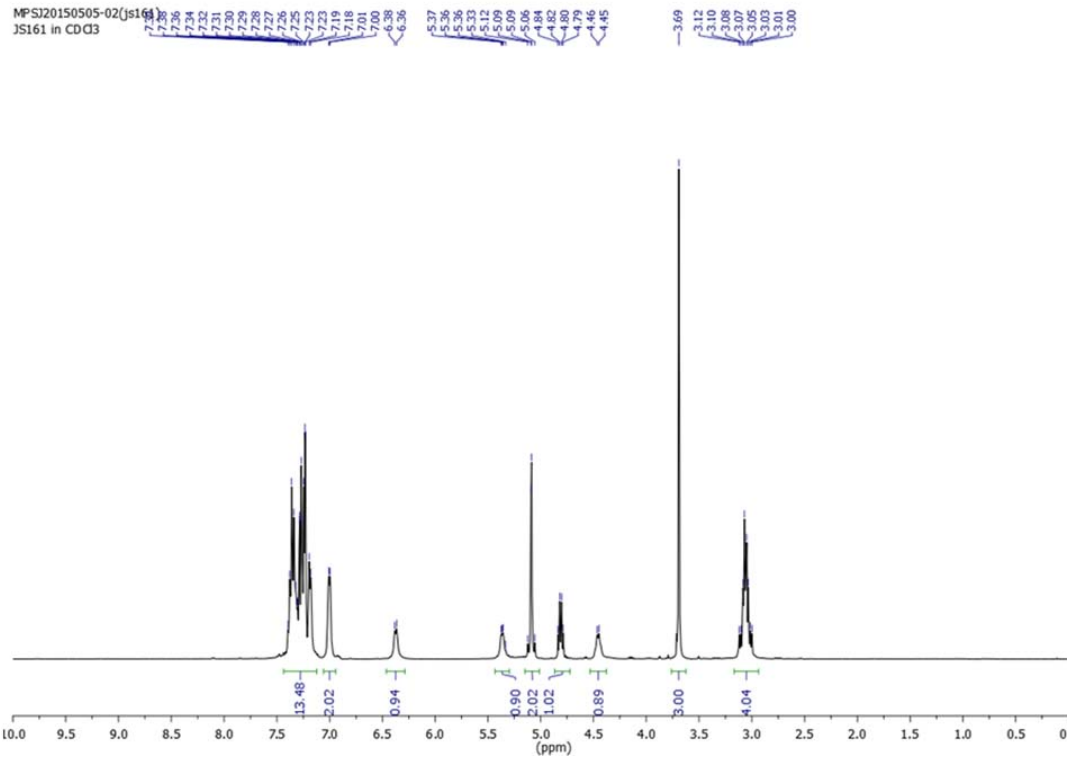
^{1}H NMR (400 MHz, $CDCl_3$); ^{13}C NMR (100 MHz, $CDCl_3$)

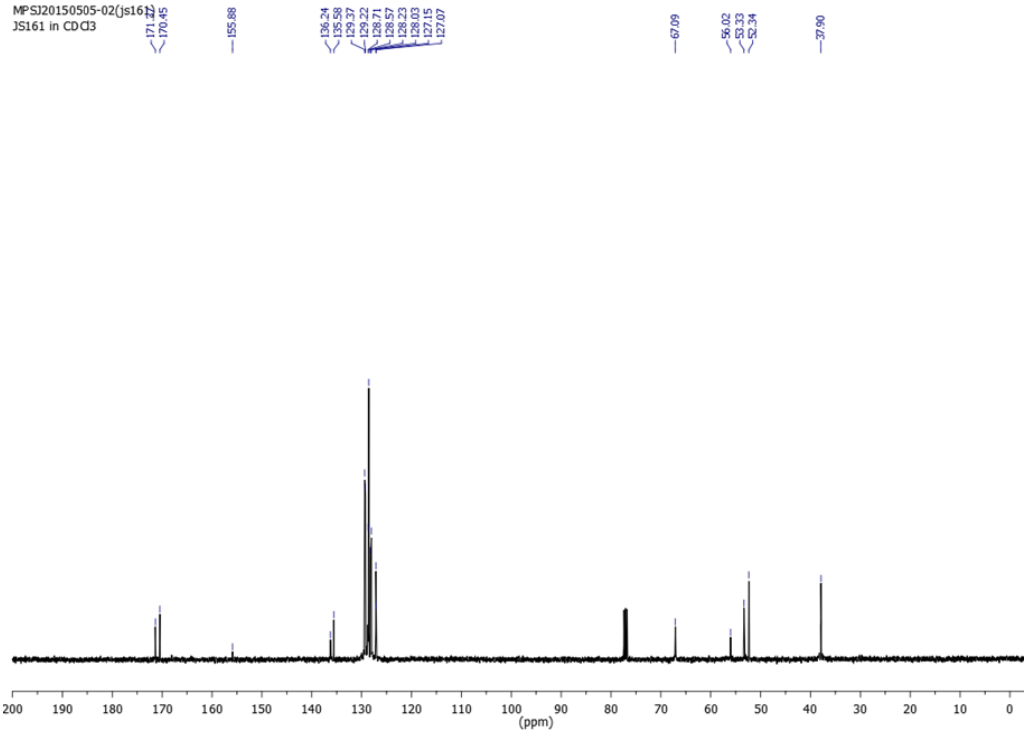


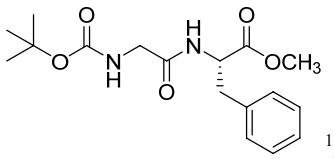




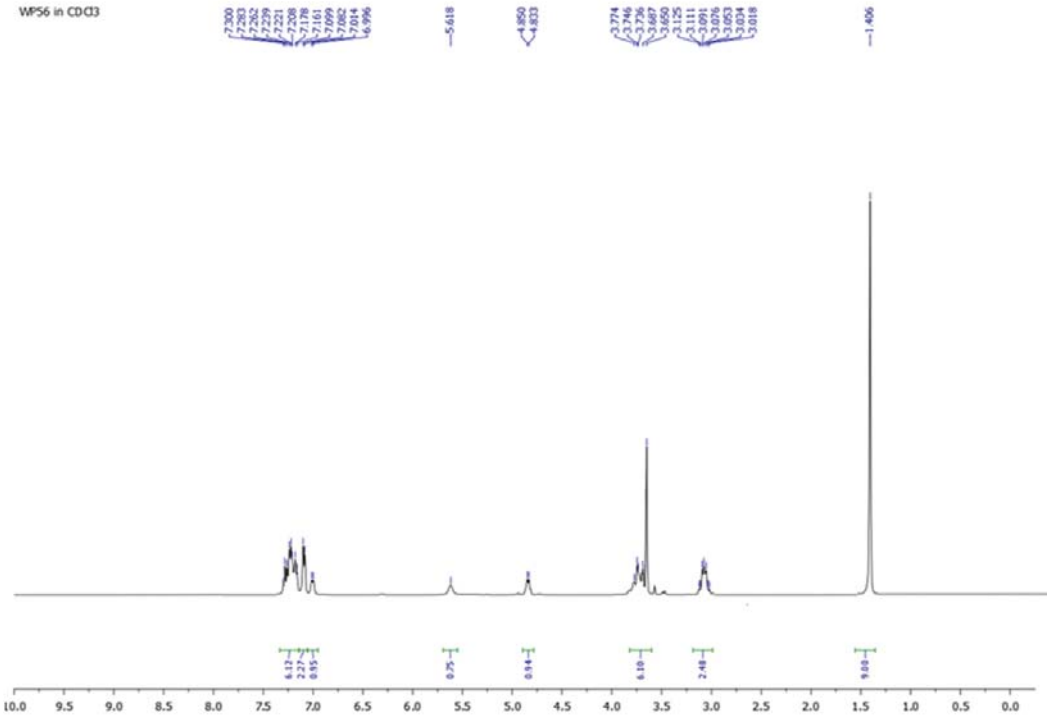
^{1}H NMR (400 MHz, $CDCl_3$); ^{13}C NMR (100 MHz, $CDCl_3$)

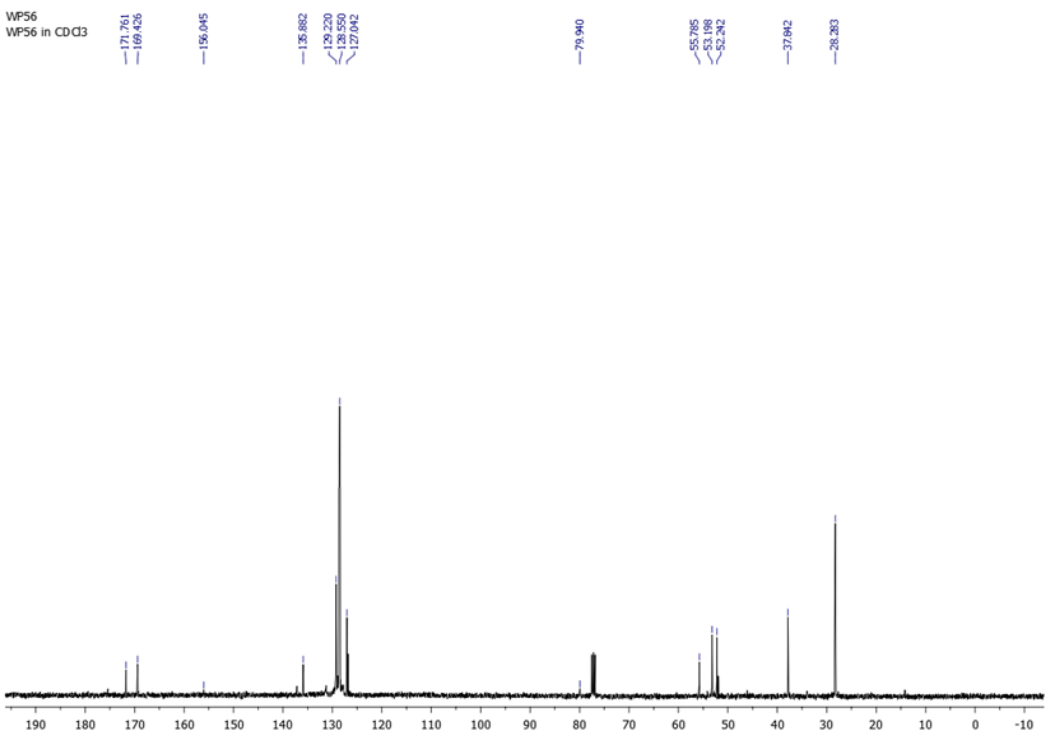


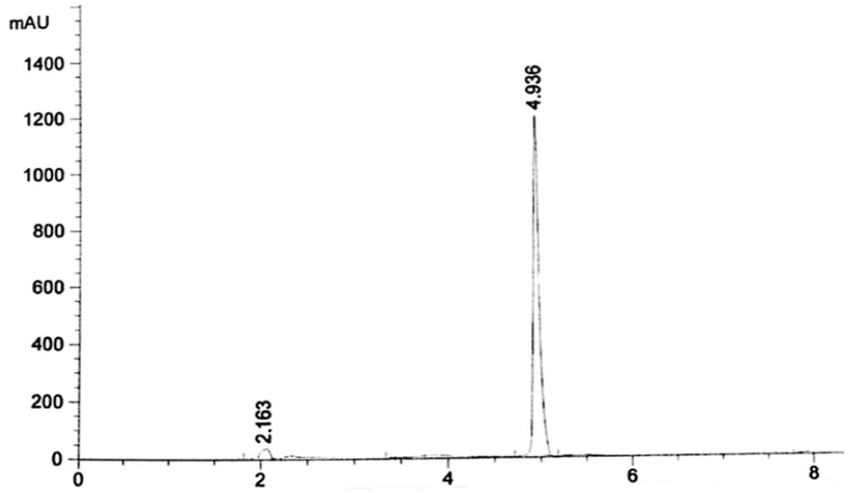




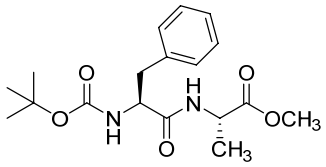
^{1}H NMR (400 MHz, $CDCl_3$); ^{13}C NMR (100 MHz, $CDCl_3$)



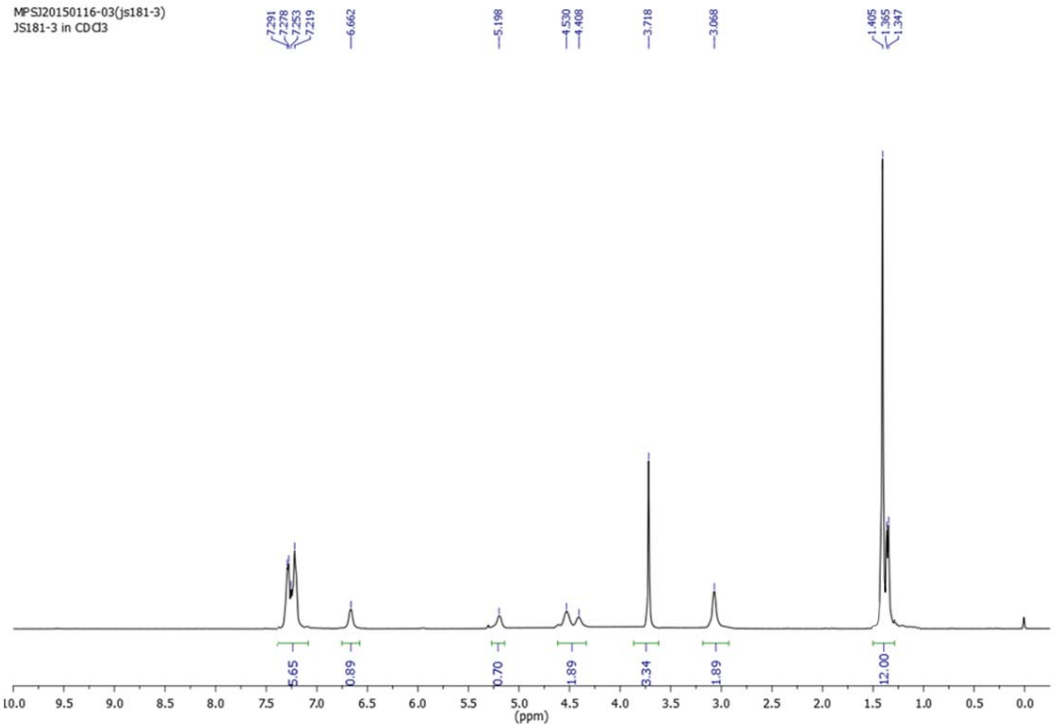


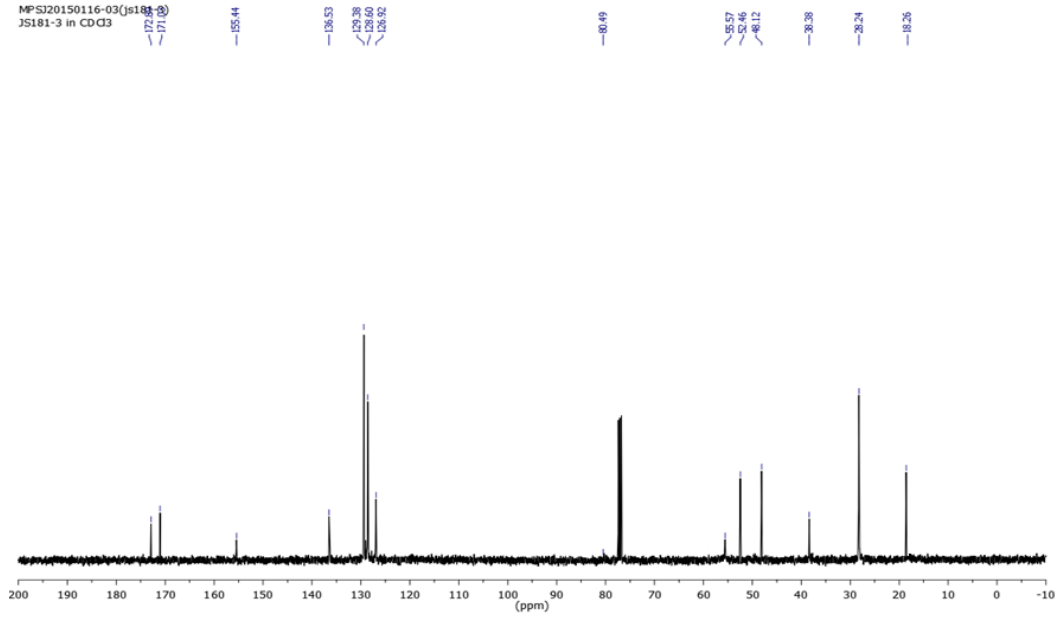


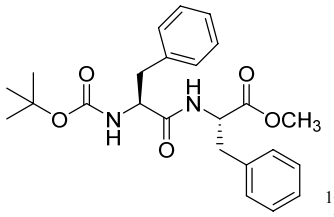
Chiral HPLC chromatogram of Boc-L-Gly-L-Phe-OMe (Table 3, entry 9)



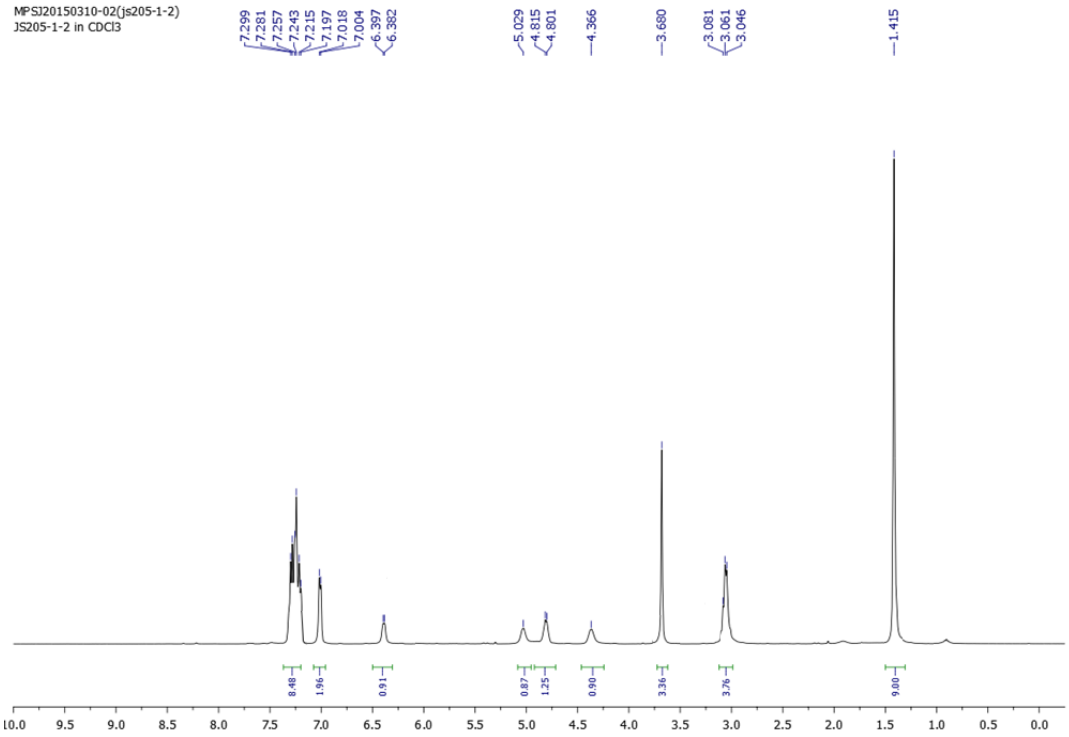
^{1}H NMR (400 MHz, $CDCl_3$); ^{13}C NMR (100 MHz, $CDCl_3$)

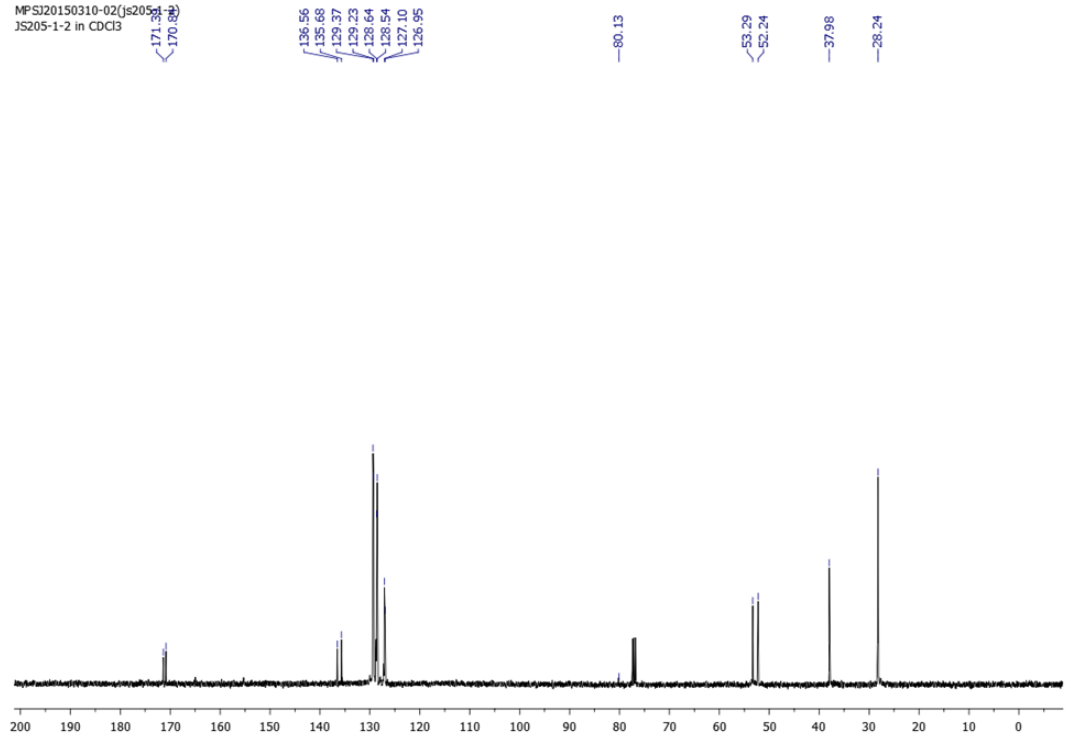






^{1}H NMR (400 MHz, $CDCl_3$); ^{13}C NMR (100 MHz, $CDCl_3$)





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2. I. Greiner, F. D. Sypaseuth, A. Grun, E. Karsai and G. Keglevich, *Lett. Org. Chem.* , 2009, **6**, 529-534.
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