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Thionation of di and tripeptides employing thiourea as a sulfur transfer reagent

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(S)-methyl 2-((S)-2-(((benzyloxy)carbonyl)amino)-2-phenylethanethioamido)-3-phenylpropanoate (1.2a)

Gum, yield 95%; R_f = 0.34 (EtOAc/n-hexane, 2:8); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.95-2.98 (m, 2H), 3.74 (s, 3H), 3.76 (s, 1H), 4.93 (s, 1H), 5.09 (s, 2H), 6.11 (brs, 1H), 7.03-7.38 (m, 15H), 9.16 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 37.70, 52.76, 65.98, 67.14, 67.22, 127.12, 127.22, 127.31, 127.44, 128.27, 128.61, 128.68, 128.72, 129.20, 129.25, 129.34, 136.27, 155.60, 171.40, 206.81 ppm; ESI-MS calcd for $C_{26}H_{26}N_2O_4S$ *m/z* 461.15, found 460.98 $(M - H)^+$.

(S)-methyl 2-(2-(((benzyloxy)carbonyl)amino)propanethioamido)acetate (1.2b)

Gum, yield 92%; R_f = 0.40 (EtOAc/n-hexane, 2:8); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.24 (d, J = 8.4 Hz, 3H), 3.72 (s, 2H), 3.79 (s, 3H), 4.20-4.29 (m, 1H), 5.26 (s, 2H), 5.40 (t, J = 7.8 Hz, 1H), 7.24-7.39 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.45, 46.48, 53.66, 61.49, 67.12, 128.15, 128.52, 129.86, 138.52, 155.06, 169.21, 204.31; ESI-MS calcd for $C_{14}H_{18}N_2O_4S$ *m/z* 310.09, found 311.10 $(M + H)^+$.

(S)-methyl 2-((S)-2-((((9H-fluoren-9-yl)methoxy)carbonyl)amino)propanethioamido)-3-phenylpropanoate1 (1.2c)

Yellow solid, yield 90%; m.p. 136-138 ^{o}C; R_f = 0.38 (EtOAc/n-hexane, 2:8); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.46 (d, J = 8 Hz, 3H), 3.18 (d, J = 12Hz, 1H), 3.42 (d, J = 8 Hz, 1H), 3.74 (s, 3H), 3.78 (s, 1H), 4.22 (t, J = 7.8 Hz, 1H), 4.35 (d, J = 6.8 Hz, 2H), 5.35-5.39 (m, 1H), 5.85 (brs, 1H), 7.05-7.77 (m, 13H), 8.53 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 28.93, 32.70, 47.13, 52.43, 56.41, 58.56, 67.43, 120.12, 123.42, 125.36, 127.20, 127.23, 129.32, 135.47, 141.41, 143.94, 144.23, 155.82, 171.05, 205.42; HRMS calcd for $C_{28}H_{28}N_2NaO_4S$ *m/z* 511.1667, found 511.1667 $(M + Na)^+$.

(S)-methyl 2-((S)-2-((((9H-fluoren-9-yl)methoxy)carbonyl)amino)-3-phenylpropanethioamido)propanoate (1.2d)

Yellow solid, yield 88%; m.p. 132-134 ^{o}C; R_f = 0.37 (EtOAc/n-hexane, 2:8); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.29 (d, J = 7.8 Hz, 3H), 2.92 (d, J = 11.4 Hz, 1H), 3.14 (d, J = 7.8 Hz, 1H), 3.68 (s, 3H), 4.03 (s, 1H), 4.38 (t, J = 8 Hz, 1H), 4.48 (m, 1H), 4.66 (d, J = 10.4 Hz, 2H), 5.80 (brs, 1H), 7.11-7.78 (m, 13H), 8.46 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 29.80, 35.03, 47.21, 53.63, 58.31, 61.45, 66.43, 120.50, 125.49, 125.81, 127.47, 127.60, 128.42, 128.89, 139.19, 141.03, 143.29, 155.21, 172.21, 206.07; HRMS calcd for $C_{28}H_{28}N_2O_4S$ *m/z* 488.1770, found 488.1767 $(M)^+$.

(S)-methyl 2-((S)-2-((((9H-fluoren-9-yl)methoxy)carbonyl)amino)-3-phenylpropanethioamido)-4-methylpentanoate (1.2e)

Yello solid, yield 86%; m.p. 136-138 ^{o}C; R_f = 0.39 (EtOAc/n-hexane, 2:8); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.84-0.93 (m, 6H), 1.57-1.83 (m, 3H), 3.12-3.16 (m, 2H), 3.65 (s, 3H), 4.21 (d, J = 7.8 Hz, 1H), 4.38 (t, J = 8.0 Hz, 2H), 4.66 (brs, 1H), 5.02-5.07 (m, 1H), 6.16 (brs, 1H), 7.17-7.89 (m, 13H), 9.03 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.91, 27.32, 39.93, 40.94, 46.42, 51.83, 64.09, 65.73, 67.03, 119.5, 126.34, 126.76, 127.38, 127.93, 128.65, 129.13, 136.18, 143.22, 155.66, 171.26, 204.14; HRMS calcd for $C_{31}H_{34}N_2NaO_4S$ *m/z* 553.2137, found 553.2136 $(M + Na)^+$.

(S)-methyl 4-methyl-2-phenylthioamidopentanoate, (1.2f)

Yellow solid, yield 91%; m.p. 116-118 ^{o}C; R_f = 0.36 (EtOAc/n-hexane, 2:8); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.99-1.01 (m, 6H), 1.20-1.26 (m, 1H), 1.74-1.93 (m, 2H), 3.80 (s, 3H), 5.36-5.41 (m, 1H), 7.26-7.49 (m, 3H), 7.76 (d, J = 7.2 Hz, 2H) 7.94 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.02, 22.63, 24.12, 40.16, 54.55, 56.71, 126.94, 128.65, 131.21, 141.04, 171.42, 201.12; HRMS calcd for $C_{14}H_{19}NO_2S$ *m/z* 265.1135, found 266.1232 $(M + H)^+$.

(S)-methyl 2-ethanethioamido-3-phenylpropanoate (1.2g)

Yellow solid, yield 87%; m.p. 108-110 ^{o}C; R_f = 0.34 (EtOAc/n-hexane, 2:8); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.05 (s, 3H), 3.18-3.23 (m, 2H), 3.76 (s, 3H), 4.86-4.92 (m, 1H), 6.02 (brs, 1H) 7.06-7.30 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 32.97, 37.79, 52.25,

53.18, 127.05, 128.52, 129.19, 135.95, 169.79, 192.20; HRMS calcd for $C_{12}H_{15}NO_2S$ *m/z* 237.1710, found 237.1712 $(M)^+$.

(5S,8S,11S)-methyl 5-benzyl-11-((R)-sec-butyl)-8-isobutyl-3,6-dioxo-1-phenyl-9-thioxo-2-oxa-4,7,10-triazadodecan-12-oate (1.7a)

Yellow solid, yield 87%; m.p. 115-117 ^oC; R_f = 0.40 (EtOAc/n-hexane, 2:8); ^1H NMR (400 MHz, DMSO-d_6) δ 0.84-0.96 (m, 12H), 1.19-1.22 (m, 4H), 1.85-1.86 (m, 1H), 3.04 (d, J = 6.4 Hz, 2H), 3.70 (s, 3H), 4.39-4.42 (m, 3H), 5.04 (s, 2H), 5.12-5.22 (m, 1H), 6.22 (brs, 1H), 6.76 (brs, 1H), 7.14-7.33 (m, 10H), 8.40 (brs, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 9.08, 14.50, 20.81, 22.87, 23.26, 35.93, 36.36, 41.99, 49.82, 54.17, 54.59, 55.33, 64.00, 78.03, 124.63, 125.91, 126.10, 126.43, 126.67, 127.71, 135.31, 136.17, 154.20, 169.35, 171.56, 205.95; HRMS calcd for $C_{30}H_{41}N_3O_5S$ *m/z* = 555.2767, found 556.2875 $(M + H)^+$.

(5S,8S)-methyl 8-benzyl-1-(9H-fluoren-9-yl)-5-methyl-3,6-dioxo-9-thioxo-2-oxa-4,7,10-triazadodecan-12-oate (1.7b)

Yellow solid, yield 92%; m.p. 90-92 ^oC; R_f = 0.42 (EtOAc/n-hexane, 2:8); ^1H NMR (400 MHz, DMSO-d_6) δ 1.25 (d, J = 7.2 Hz, 3H), 3.16 (d, J = 6 Hz, 2H), 3.43 (s, 2H), 3.62 (s, 3H), 3.87 (t, J = 8 Hz, 1H), 4.21 (d, J = 7.8 Hz, 1H), 4.64-4.87 (m, 3H), 5.30 (brs, 1H), 6.94 (d, J = 5.2 Hz, 1H), 7.20-7.88 (m, 13H), 8.23 (brs, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 18.43, 42.56, 46.78, 47.45, 52.49, 62.70, 64.41, 73.40, 120.24, 125.45, 126.37, 127.27, 127.89, 128.23, 129.42, 137.70, 140.82, 143.88, 154.01, 169.02, 171.55, 204.93; HRMS calcd for $C_{30}H_{31}N_3NaO_5S$ *m/z* 545.1984, found 546.1975 $(M + H)^+$.

methyl 9-benzyl-12-isobutyl-2,2-dimethyl-4,7-dioxo-10-thioxo-3-oxa-5,8,11-triazatridecan-13-oate (1.7c)

Gum, yield 86%; R_f = 0.36 (EtOAc/n-hexane, 2:8); ^1H NMR (400 MHz, $CDCl_3$) δ 0.86-0.92 (m, 6H), 1.38 (s, 9H), 1.49-1.55 (m, 1H), 1.65-1.87 (m, 3H), 3.14 (d, J = 6.0 Hz, 2H), 3.68 (s, 3H), 3.82 (s, 2H), 4.03 (t, J = 7.2 Hz, 1H), 4.82 (brs, 1H), 6.88 (d, J = 6.8 Hz 1H), 7.02-7.28 (m, 5H), 8.23 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.96, 24.64, 28.45, 39.35, 41.62, 45.82, 52.34, 56.72, 68.23, 79.96, 125.62, 127.74, 128.62, 136.54, 156.32, 169.78, 172.94, 205.31; HRMS calcd for $C_{23}H_{35}N_3NaO_5S$ *m/z* 488.2189, found 488.2189 $(M + Na)^+$.

methyl 8-(sec-butyl)-11-isobutyl-5-methyl-3-oxo-1-phenyl-6,9-dithioxo-2-oxa-4,7,10-triazadodecan-12-oate (1.9a)

Gum, yield 94%; R_f = 0.38 (EtOAc/n-hexane, 2:8); ^{1}H NMR (400 MHz, DMSO-d_6) δ 0.79-0.87 (m, 12H), 1.03-1.05 (m, 1H), 1.16 (d, J = 6.8 Hz, 3H), 1.42-1.56 (m, 1H), 1.58-1.16 (m, 4H), 3.59 (s, 3H), 3.99 (t, J = 6.8 Hz, 1H), 4.17-4.19 (m, 1H), 4.99-5.08 (m, 3H), 7.28-7.34 (m, 5H), 7.98 (d, J = 7.2 Hz, 1H), 8.55 (d, J = 8.8 Hz, 1H), 8.78 (d, J = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 11.10, 14.24, 18.21, 21.62, 22.77, 24.34, 24.95, 38.36, 41.09, 57.08, 60.24, 65.59, 71.61, 74.04, 127.80, 127.89, 128.43, 137.08, 154.13, 172.21, 205.68, 206.61; HRMS calcd for $C_{24}H_{37}N_3O_4S_2$ *m/z* 495.2225, found 496.2308 $(M + H)^+$.

(5S,8S,11S)-methyl 5-((R)-sec-butyl)-11-isobutyl-8-methyl-3-oxo-1-phenyl-6,9-dithioxo-2-oxa-4,7,10-triazadodecan-12-oate (1.9b)

Gum, yield 85%; R_f = 0.41 (EtOAc/n-hexane, 2:8); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.82-0.91 (d, J = 6.8Hz, 6H), 1.33-1.37 (m, 9H), 1.42-1.57 (m, 3H), 1.70-1.86 (m, 3H), 3.67 (s, 3H), 4.03-4.06 (m, 1H), 4.32 (t, J = 6.8 Hz, 1H), 4.50 (t, J = 7.2 Hz, 1H), 5.05 (s, 2H), 6.90 (brs, 1H), 7.26-7.30 (m, 5H), 8.13 (brs, 1H), 8.78 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 11.24, 15.30, 17.94, 18.20, 21.68, 22.65, 24.63, 30.56, 33.81, 48.76, 50.82, 52.09, 59.57, 66.75, 127.80, 127.99, 128.39, 136.28, 156.32, 171.35, 202.11, 203.06; HRMS calcd for $C_{24}H_{37}N_3O_4S_2$ *m/z* 495.2225, found 496.2308 $(M + H)^+$.

(8S,11S)-methyl 11-benzyl-1-(9H-fluoren-9-yl)-8-isopropyl-3-oxo-6,9-dithioxo-2-oxa-4,7,10-triazadodecan-12-oate (1.9c)

Yellow solid, yield 91%; m.p. 128-130 ^{o}C; R_f = 0.34 (EtOAc/n-hexane, 2:8); ^{1}H NMR (400 MHz, DMSO-d_6) δ 0.87 (d, J = 6.8 Hz, 6H), 2.02-2.18 (m, 1H), 2.96 (d, J = 7.2 Hz, 1H), 3.07 (d, J = 5.6 Hz, 1H), 3.69 (s, 3H), 3.75-3.80 (m, 1H), 4.19 (t, J = 6.8 Hz, 1H), 4.37 (d, J = 6.8 Hz, 2H), 4.72 (d, J = 6.0 Hz, 2H), 4.91-4.93 (m, 1H), 7.09 (d, J = 7.2 Hz, 1H), 7.16-7.76 (m, 13H), 8.53 (brs, 1H), 8.64 (brs, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 18.70, 19.07, 32.57, 47.53, 52.32, 54.41, 67.01, 75.36, 120.41, 125.75, 127.13, 127.62, 128.20, 128.78, 129.90, 137.26, 141.55, 144.36, 157.41, 173.39, 198.32, 204.28; HRMS calcd for $C_{32}H_{35}N_3O_4S_2$ *m/z* 589.2069, found 590.2151 $(M + H)^+$.

(S)-(9H-fluoren-9-yl)methyl (4-methyl-1-thioxo-1-(p-tolylamino)pentan-2-yl)carbamate (1.11a)

Yellow solid, yield 87%; m.p. 82-84 ^{o}C; R_f = 0.36 (EtOAc/n-hexane, 2:8); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.23 (d, J = 8.0Hz, 6H), 1.55-1.72 (m, 3H), 4.07-4.19 (m, 2H), 4.38 (d, J = 7.2 Hz, 2H), 5.05 (s, 2H), 6.24 (d, J = 8.0 Hz, 1H), 6.96 (brs, 1H), 7.23-8.01 (m, 12H), 10.03 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.15, 24.96, 25.66, 41.08, 47.10, 67.47, 68.00, 120.53, 123.91, 125.01, 125.14, 127.15, 127.80, 128.69, 141.31, 143.50, 143.70, 156.04, 200.22; HRMS calcd for $C_{28}H_{30}N_2O_2S$ *m/z* 458.2106, found 459.2093 $(M + H)^+$.

(S)-(9H-fluoren-9-yl)methyl (1-(phenylamino)-1-thioxopropan-2-yl)carbamate (1.11b)

Yellow solid, yield 86%; m.p. 85-87 ^{o}C; R_f = 0.39 (EtOAc/n-hexane, 2:8); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.26 (d, J = 9.2Hz, 3H), 4.16 (t, J = 7.2 Hz, 1H), 4.31 (d, J = 6.4 Hz, 2H), 4.90 (brs, 1H), 6.22 (brs, 1H), 7.18-7.74 (m, 13H), 10.35 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.84, 46.97, 67.64, 68.03, 120.10, 123.56, 126.09, 126.20, 126.88, 127.17, 128.01, 138.52, 141.29, 143.45, 156.19, 203.55; ESI-MS calcd for $C_{24}H_{22}N_2O_2S$ *m/z* 402.14, found 403.14 $(M + H)^+$.

(S)-benzyl 3-((((9H-fluoren-9-yl)methoxy)carbonyl)amino)-4-thioxo-4-(p-tolylamino)butanoate (1.11c)

Yellow solid, yield 84%; m.p. 88-90 ^{o}C; R_f = 0.42 (EtOAc/n-hexane, 2:8); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.22 (s, 3H), 2.48-2.59 (m, 2H), 3.69-3.78 (m, 1H), 4.13 (t, J = 4.6 Hz, 1H), 4.26 (d, J = 6.4 Hz, 2H), 4.88 (brs, 1H), 5.29, (s, 2H), 6.96-7.75 (m, 17H), 10.48 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.32, 45.22, 46.96, 65.29, 67.51, 69.51, 120.73, 124.13, 125.02, 125.17, 127.16, 127.64, 127.79, 128.57, 138.45, 138.77, 141.22, 141.26, 143.41, 143.76, 156.87, 171.31, 204.26; ESI-MS calcd for $C_{33}H_{30}N_2O_4S$ *m/z* 551.39, found 550.22 $(M - H)^+$.

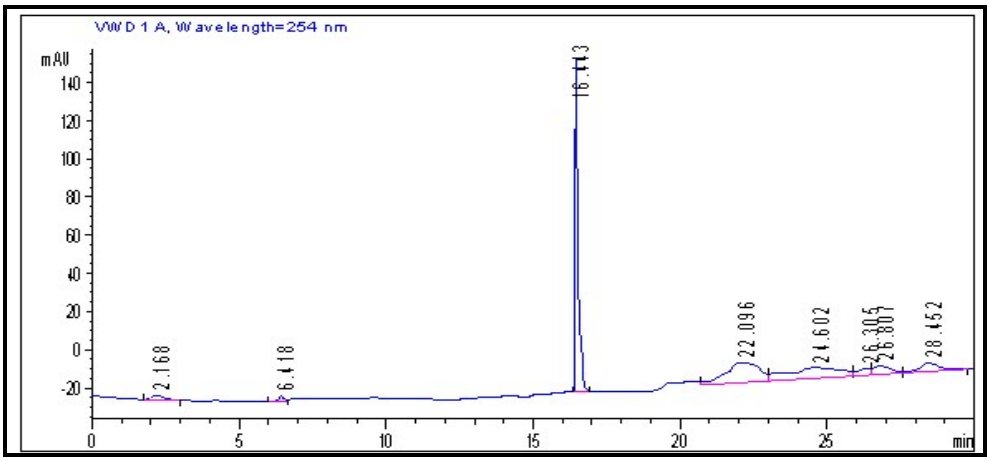
(S)-benzyl (3-phenyl-1-(phenylamino)-1-thioxopropan-2-yl)carbamate (1.11d)

Yellow solid, yield 83%; m.p. 81-83 ^{o}C; R_f = 0.34 (EtOAc/n-hexane, 2:8); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.19- 3.28 (m, 2H), 4.09-4.14 (m, 1H), 5.29 (s, 2H), 6.00 (brs, 1H), 7.20-7.37 (m, 15H), 9.52 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 42.68, 67.20, 68.00, 123.77, 126.96, 127.13, 127.77, 128.25, 128.59, 128.66, 128.74, 129.51, 135.92, 136.52, 138.09, 156.12, 202.00; HRMS calcd for $C_{23}H_{22}N_2O_2S$ *m/z* 390.1402, found 391.1505 $(M + H)^+$.

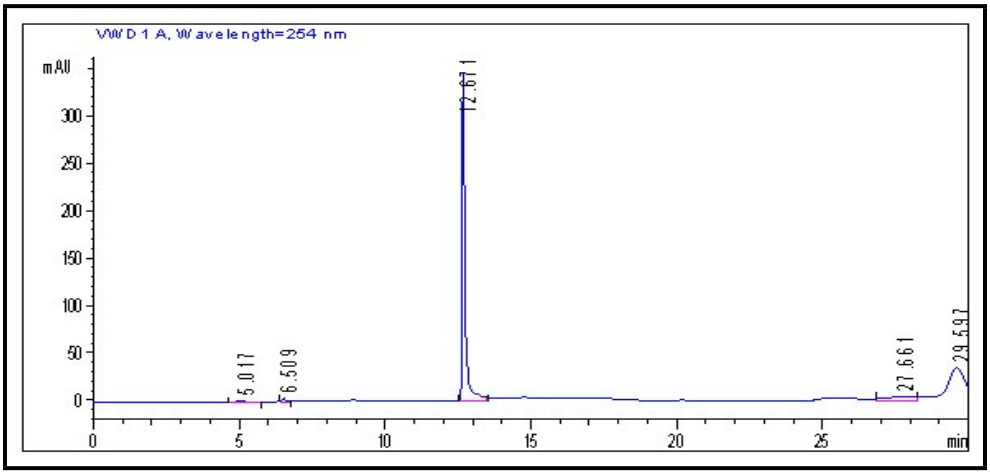
benzyl (2-(phenylamino)-2-thioxoethyl)carbamate (1.11e)

Yellow solid, yield 79%; m.p. 90-92 ^{o}C; R_f = 0.44 (EtOAc/n-hexane, 2:8); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.42 (s, 2H) 4.02 (t, J = 10.4 Hz, 1H), 5.18 (s, 2H), 7.26 – 7.50 (m, 5H), 7.64 – 7.70

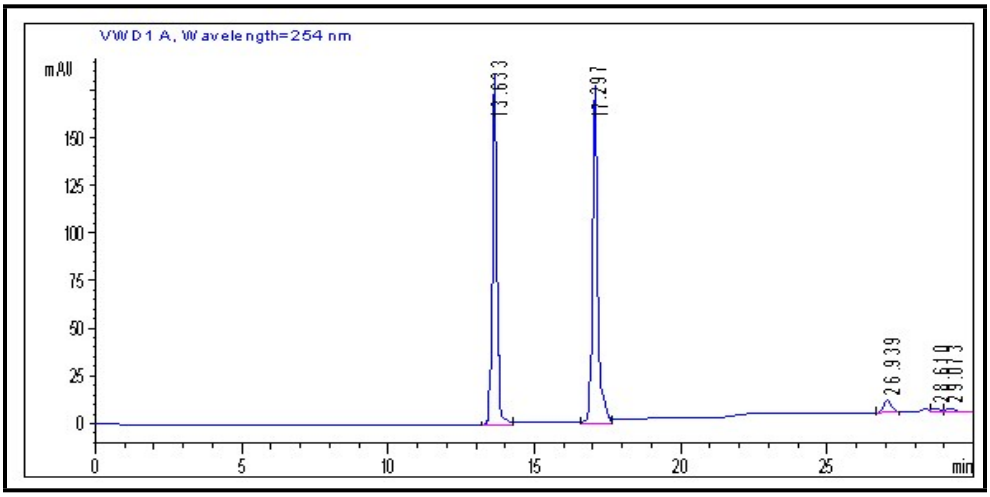
(m, 5H), 8.32 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 59.49, 67.44, 125.13, 125.20, 128.20, 1280.31, 128.44, 128.70, 128.81, 139.63, 156.33, 198.40; ESI-MS calcd for $C_{16}H_{16}N_2O_2S$ *m/z* 300.09, found 301.16 $(M + H)^+$.



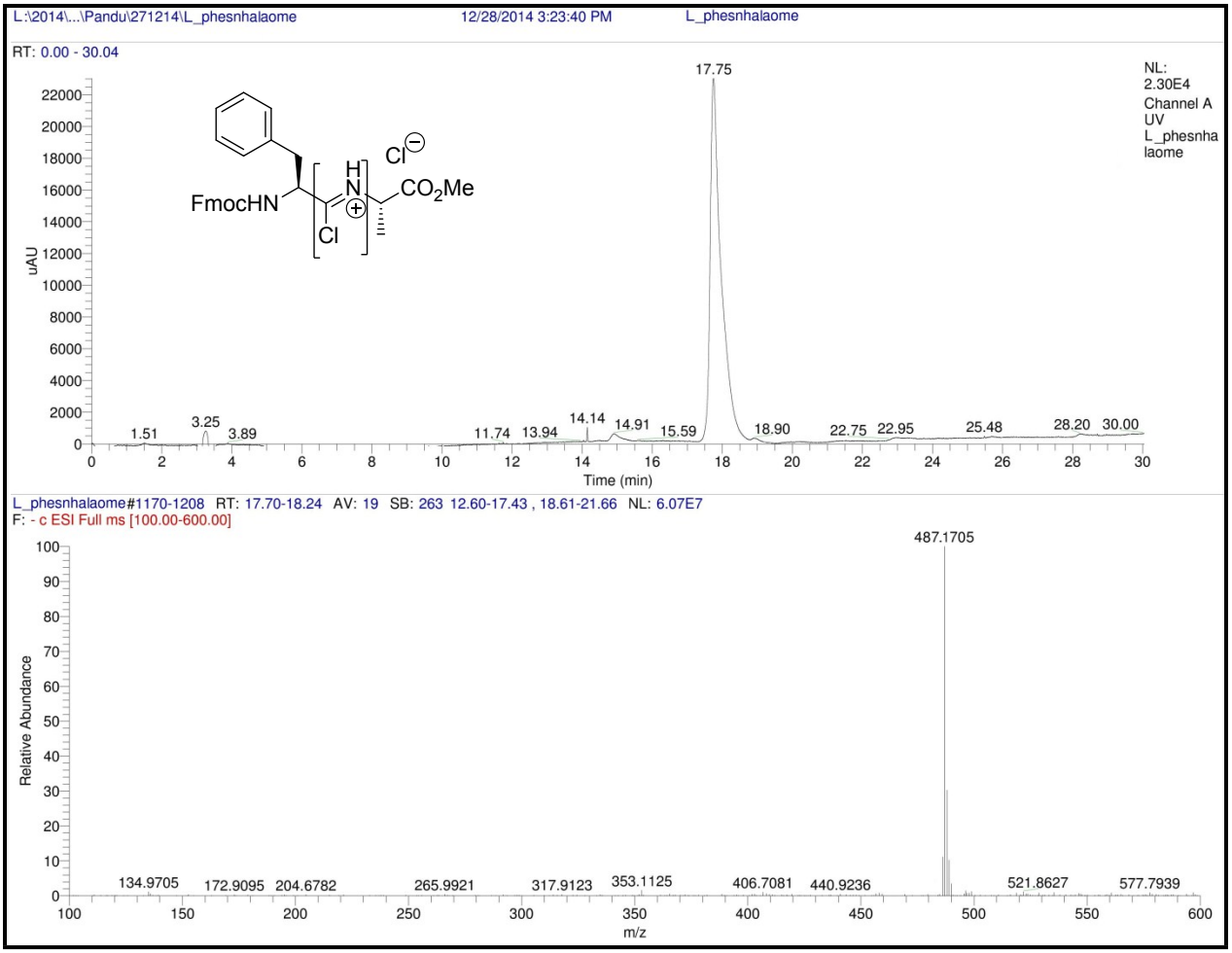
Racemization study of Cbz-L-Phg-ψ[CSNH]-Phe-COOMe



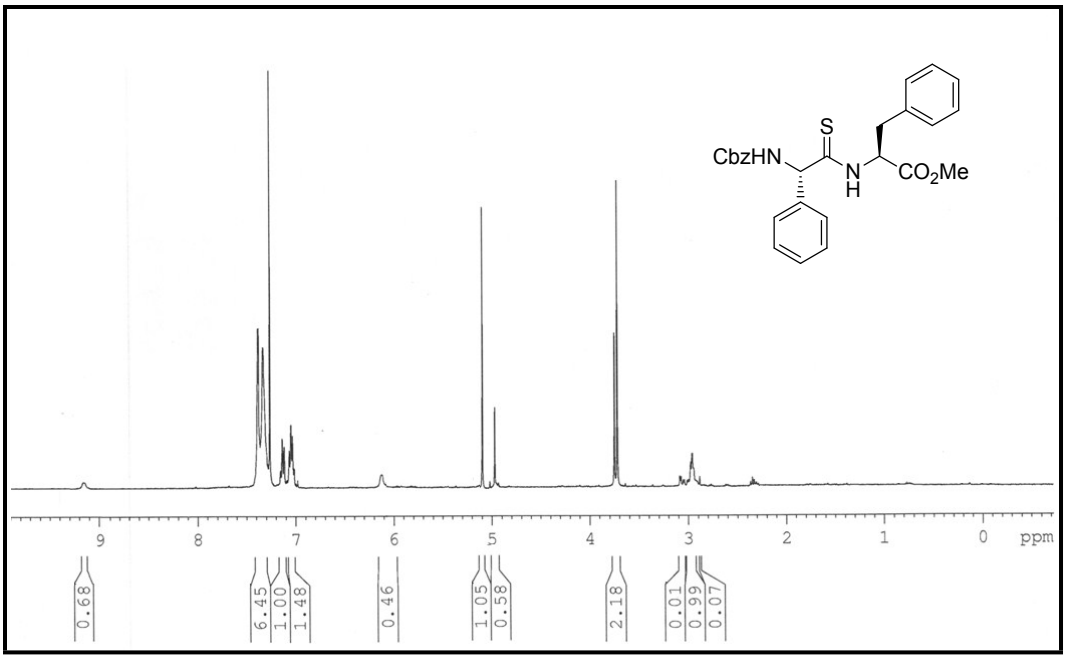
Racemization study of Cbz-D-Phg-ψ[CSNH]-Phe-COOMe



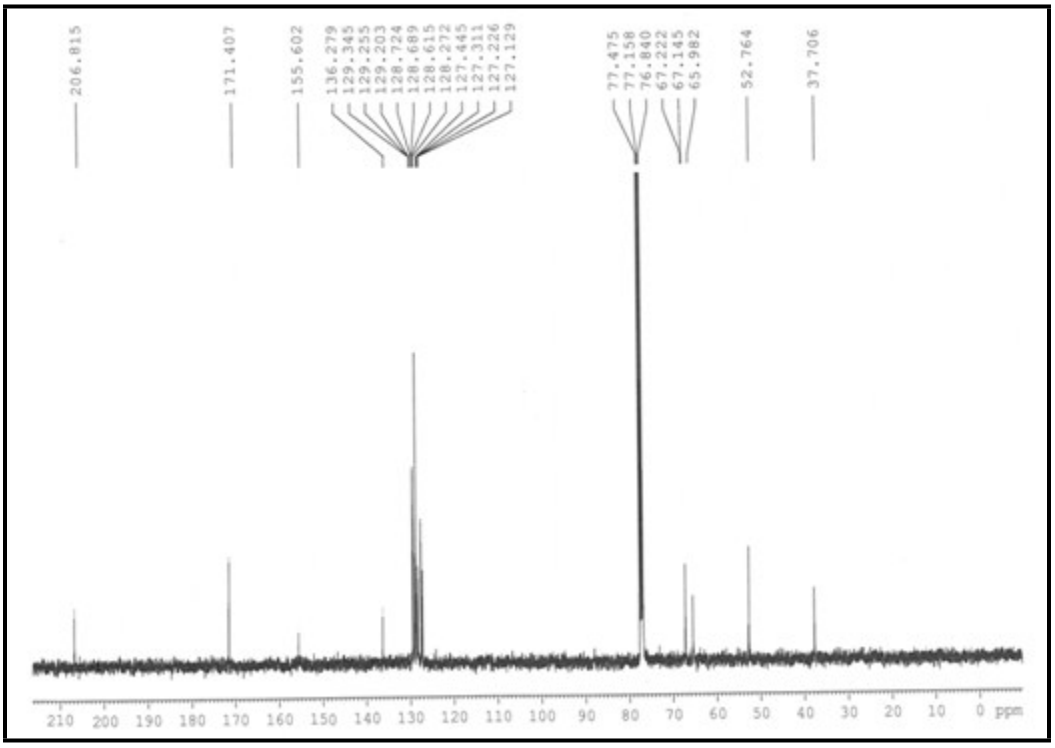
Racemization study of Cbz-L-Phg-ψ[CSNH]-Phe-COOMe and Cbz-D-Phg-ψ[CSNH]-Phe-COOMe



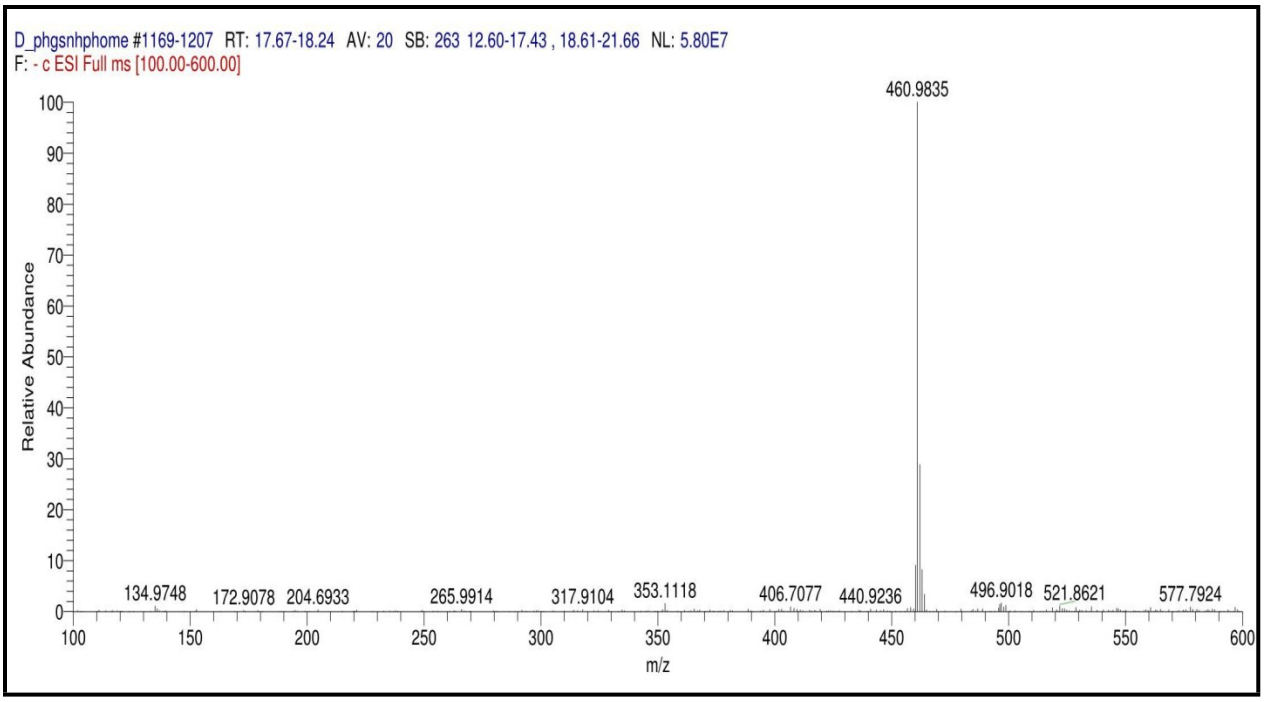
HPLC chromatogram and ESI MS of intermediate 1.4



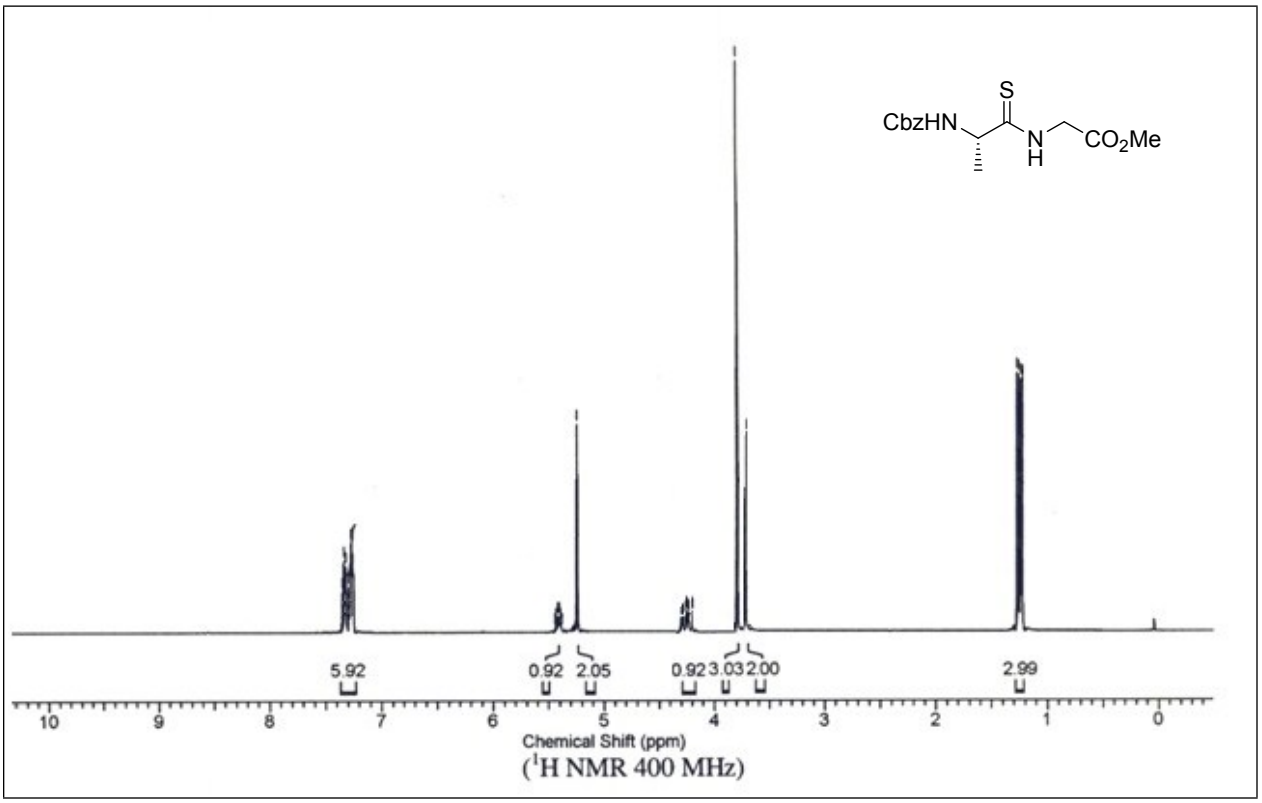
^{1}H NMR Spectrum of Cbz-Phg-ψ[CSNH]-Phe-COOMe 1.2a



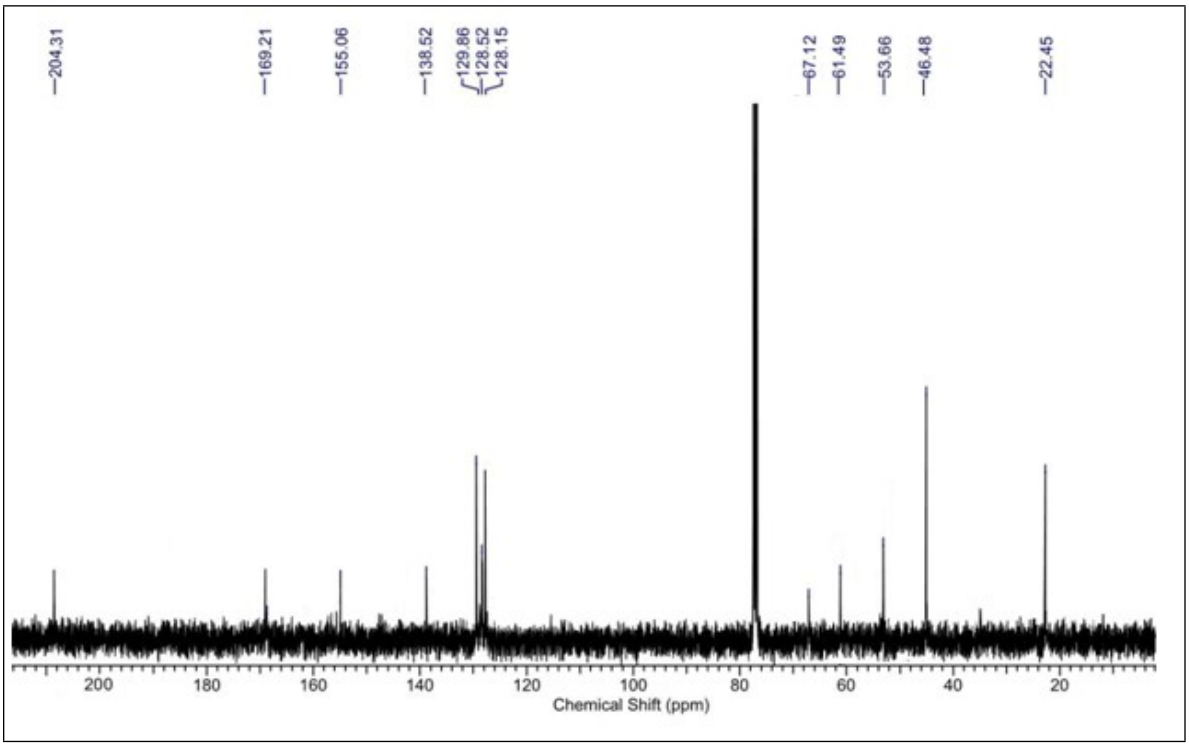
^{13}C NMR Spectrum of Cbz-Phg-ψ[CSNH]-Phe-COOMe 1.2a



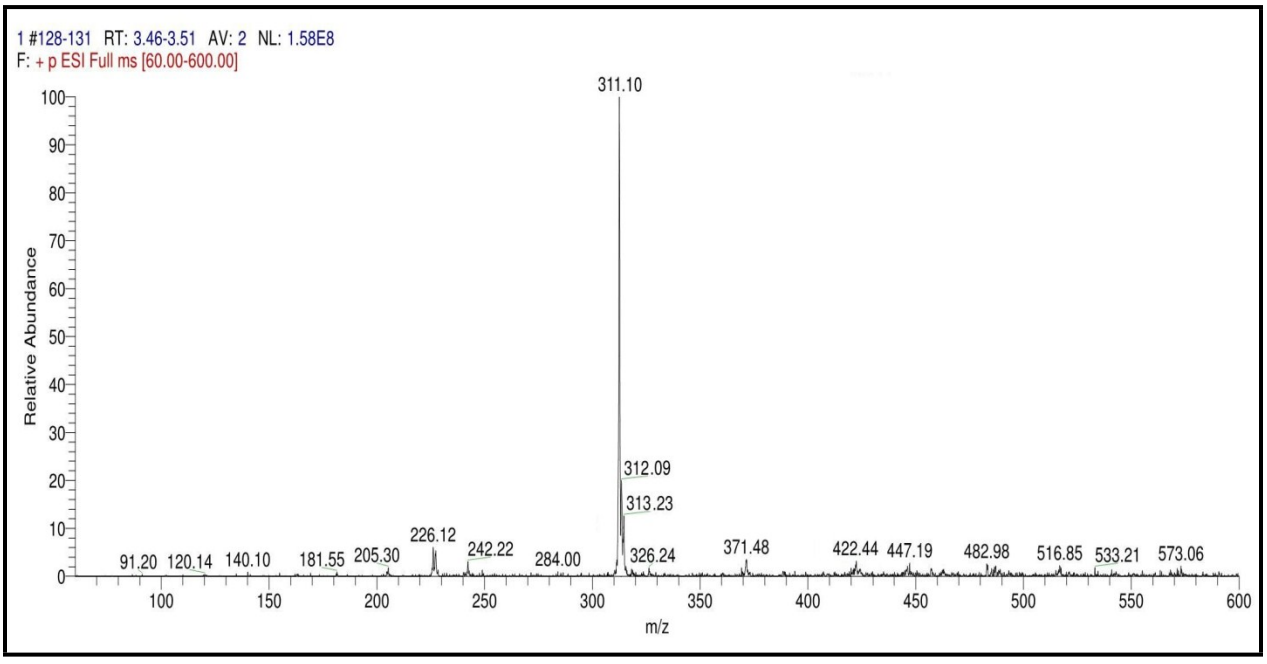
ESI MS of Cbz-Phg-ψ[CSNH]-Phe-COOMe 1.2a



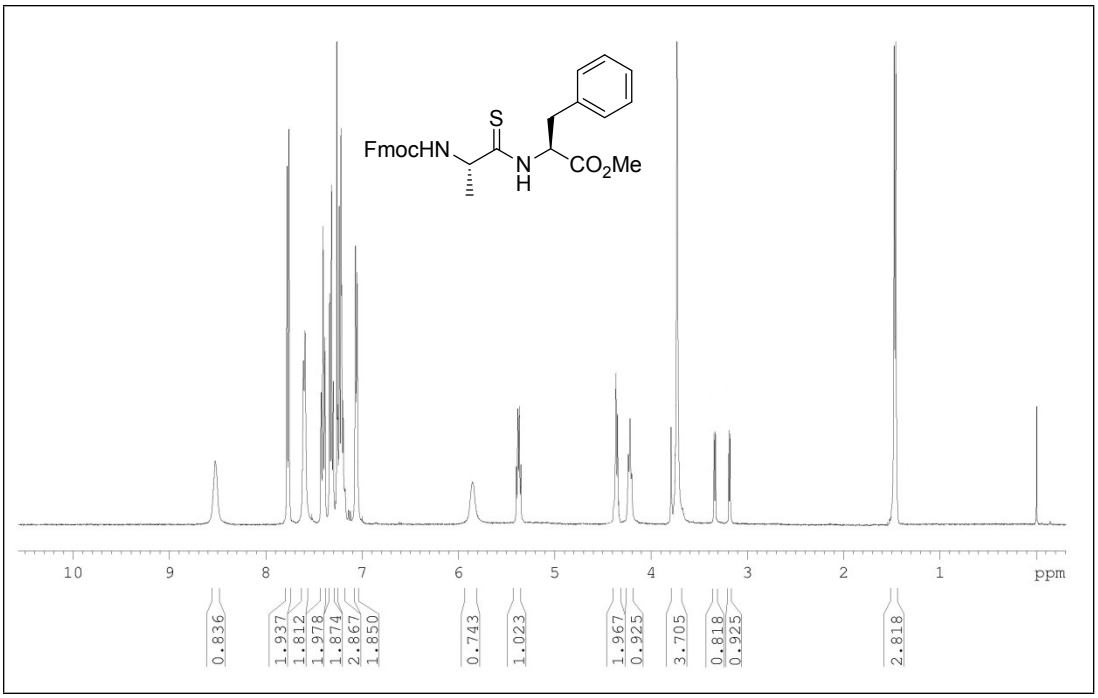
^{1}H NMR Spectrum of Cbz-Ala-ψ[CSNH]-Gly-COOMe 1.2b



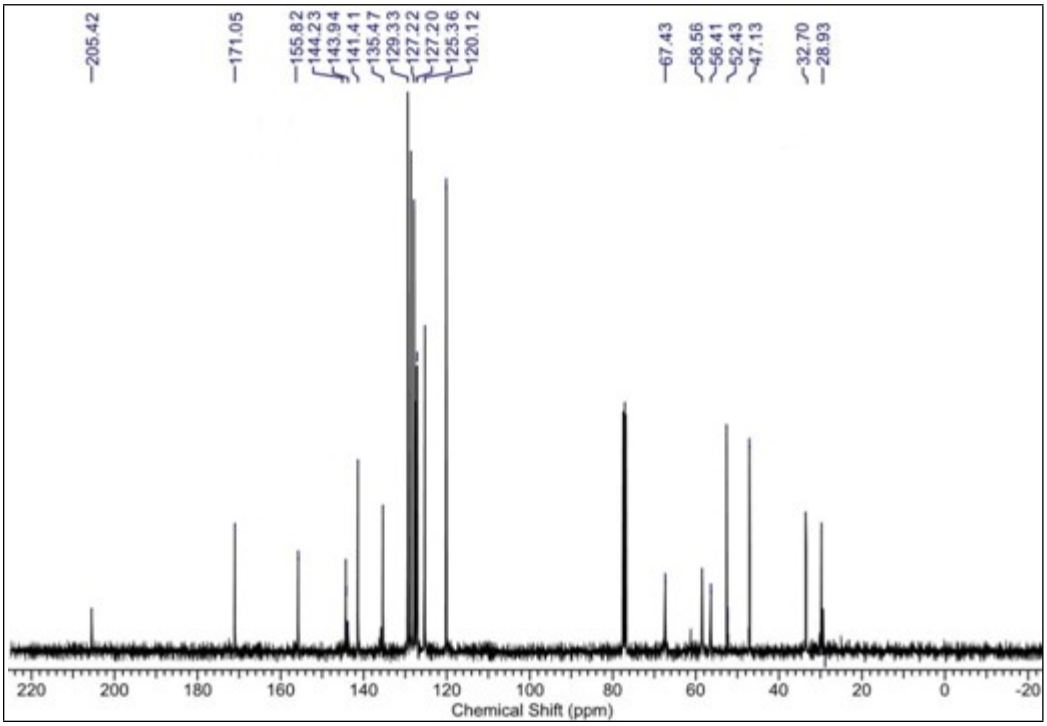
^{13}C NMR Spectrum of Cbz-Ala-ψ[CSNH]-Gly-COOMe 1.2b



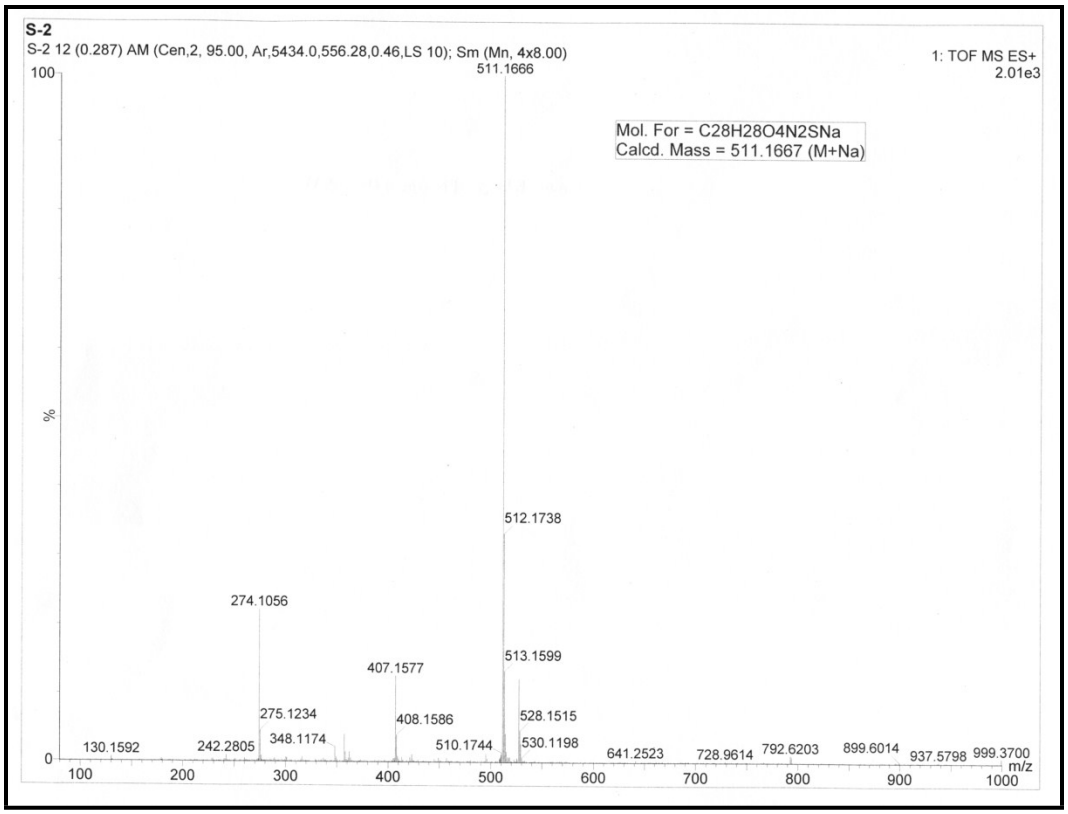
ESI MS of Cbz-Ala-ψ[CSNH]-Gly-COOMe 1.2b



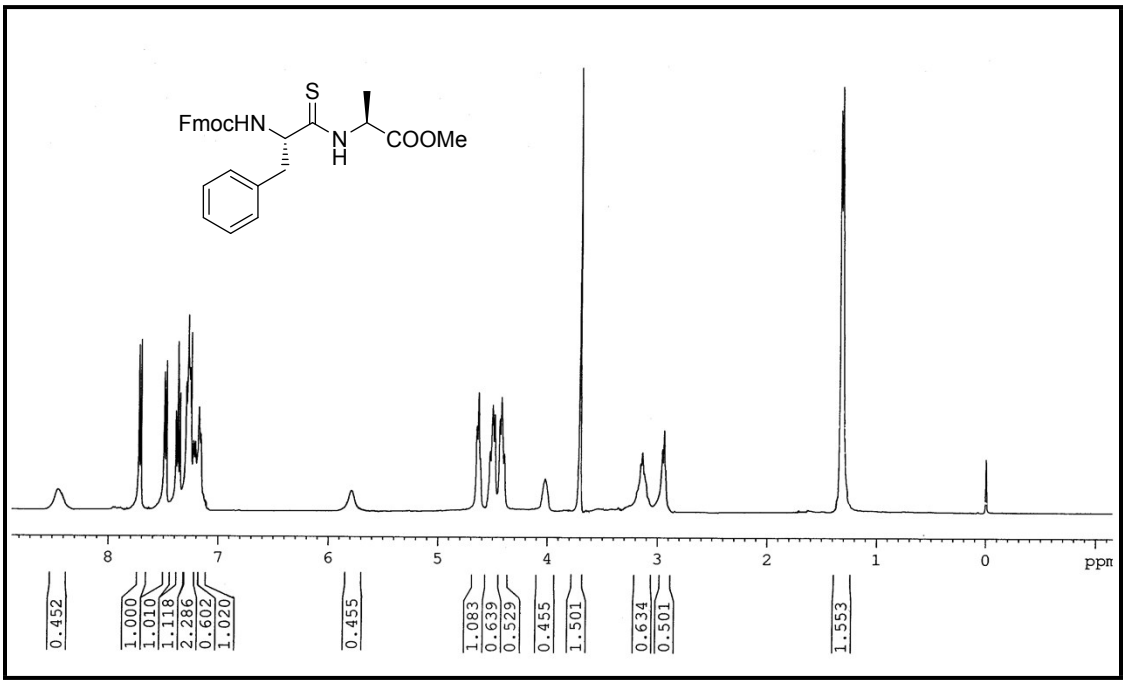
^{1}H NMR Spectrum of Fmoc-Ala-ψ[CSNH]-Phe-COOMe 1.2c



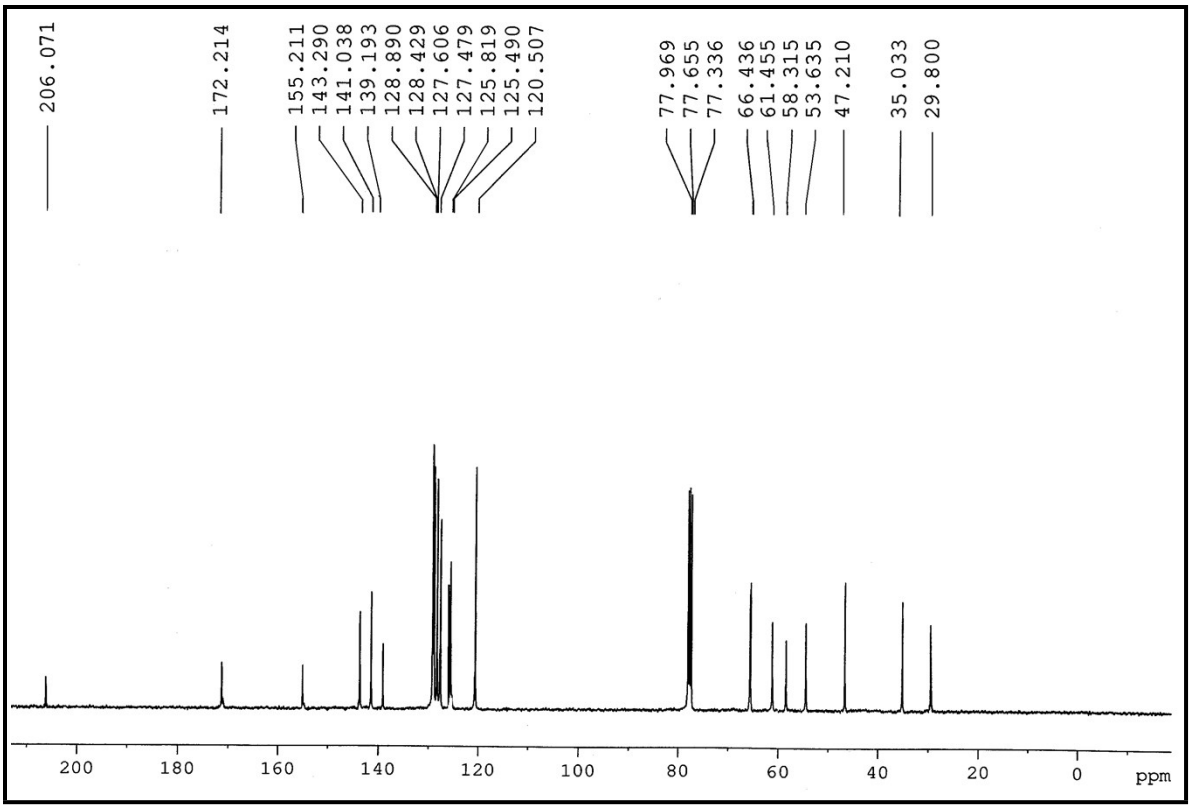
^{13}C NMR Spectrum of Fmoc-Ala-ψ[CSNH]-Phe-COOMe 1.2c



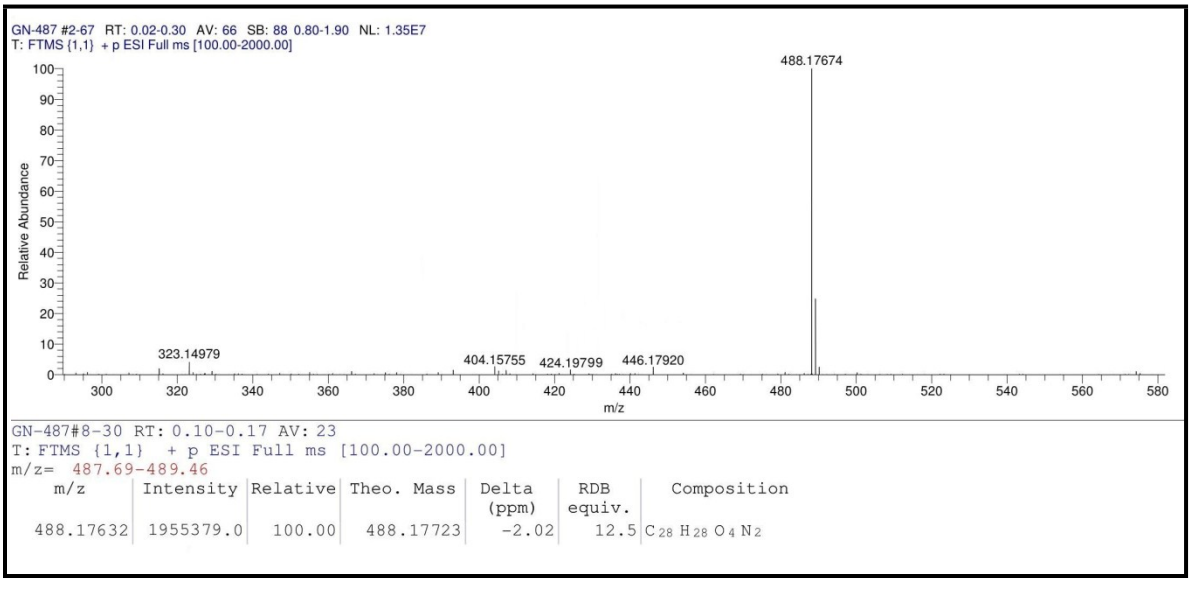
HRMS of Fmoc-Ala-ψ[CSNH]-Phe-COOMe 1.2c



^{1}H NMR Spectrum of Fmoc-Phe-ψ[CSNH]-Ala-COOMe 1.2d



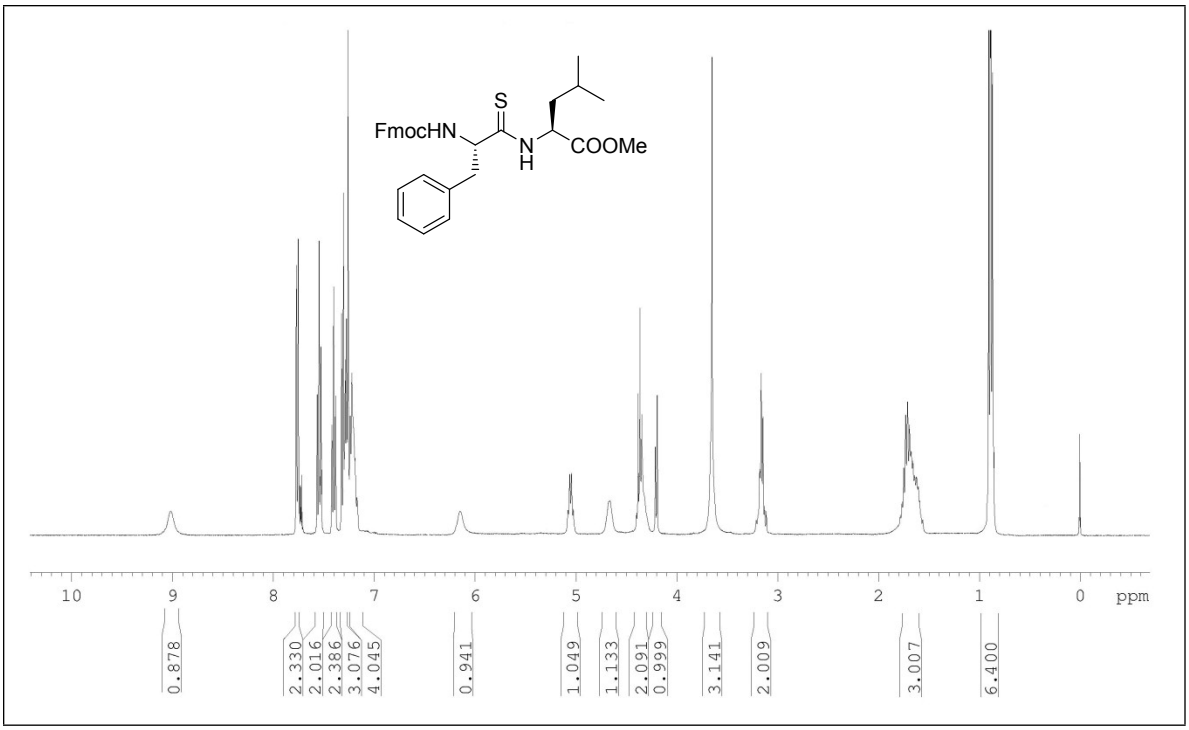
^{13}C NMR Spectrum of Fmoc-Phe-ψ[CSNH]-Ala-COOMe 1.2d



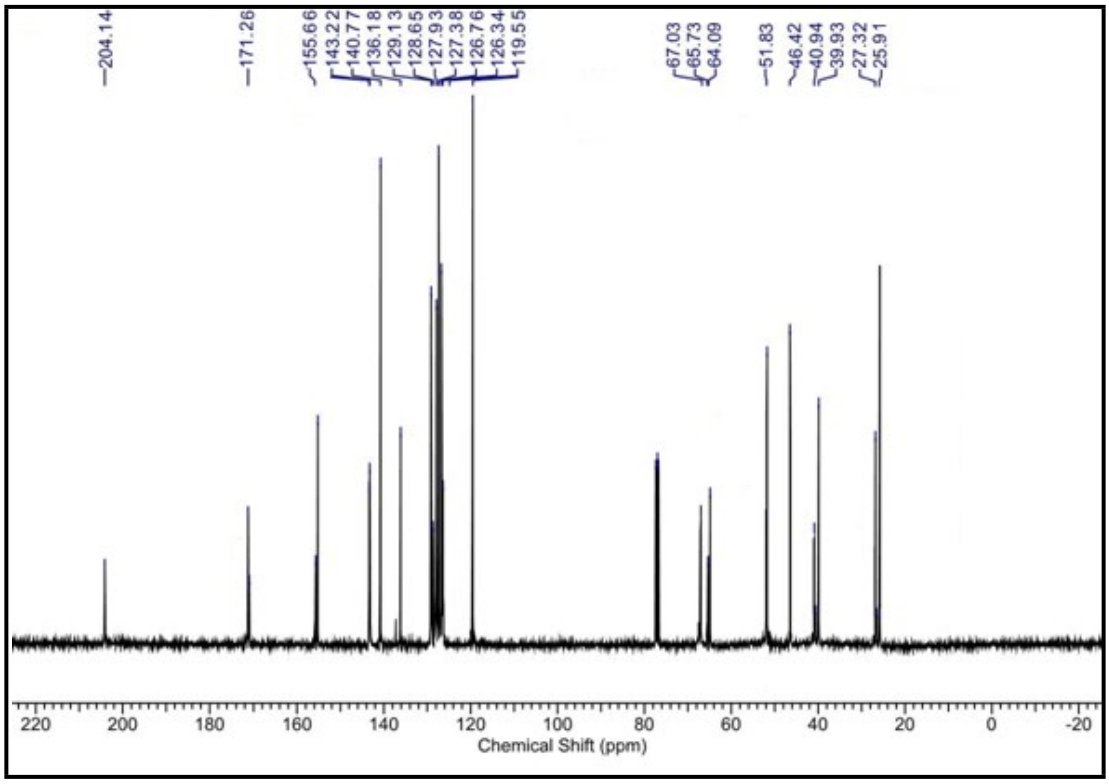
GN-487#8-30 RT: 0.10-0.17 AV: 23
T: FTMS {1,1} + p ESI Full ms [100.00-2000.00]
m/z= 487.69-489.46

m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
488.17632	1955379.0	100.00	488.17723	-2.02	12.5	$C_{28}H_{28}O_4N_2$

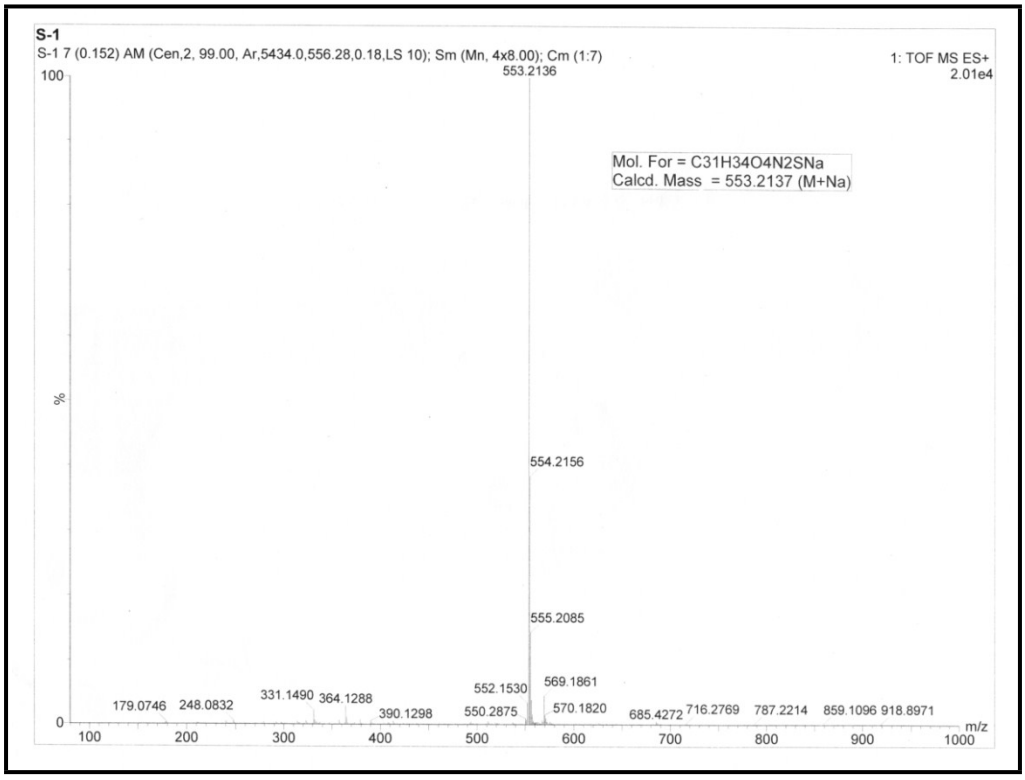
HRMS of Fmoc-Phe-ψ[CSNH]-Ala-COOMe 1.2d



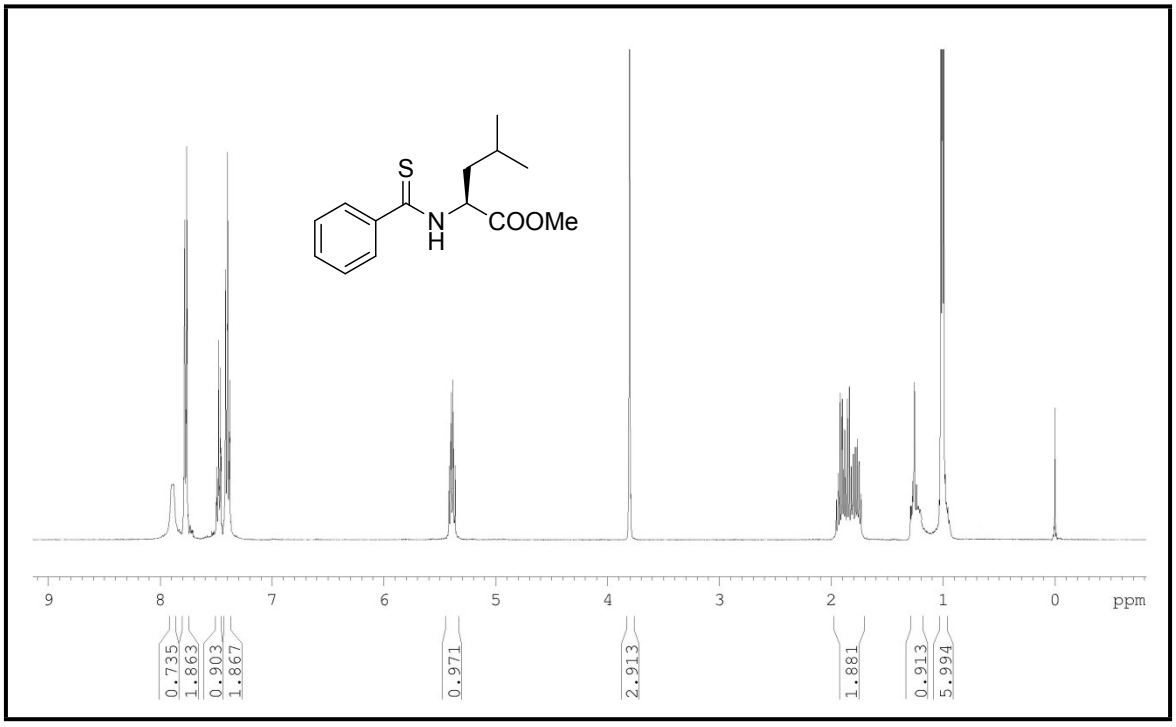
^{1}H NMR Spectrum of Fmoc-Phe-ψ[CSNH]-Leu-COOMe 1.2e



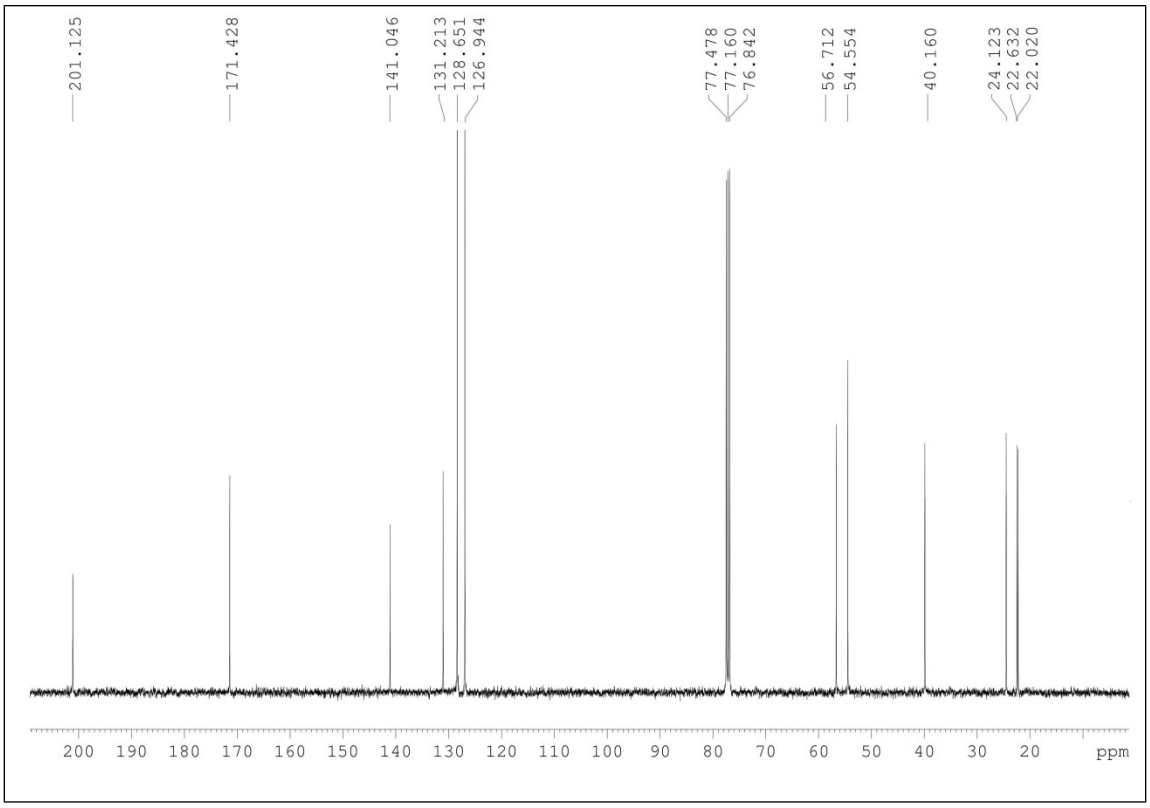
^{13}C NMR Spectrum of Fmoc-Phe-ψ[CSNH]-Leu-COOMe 1.2e



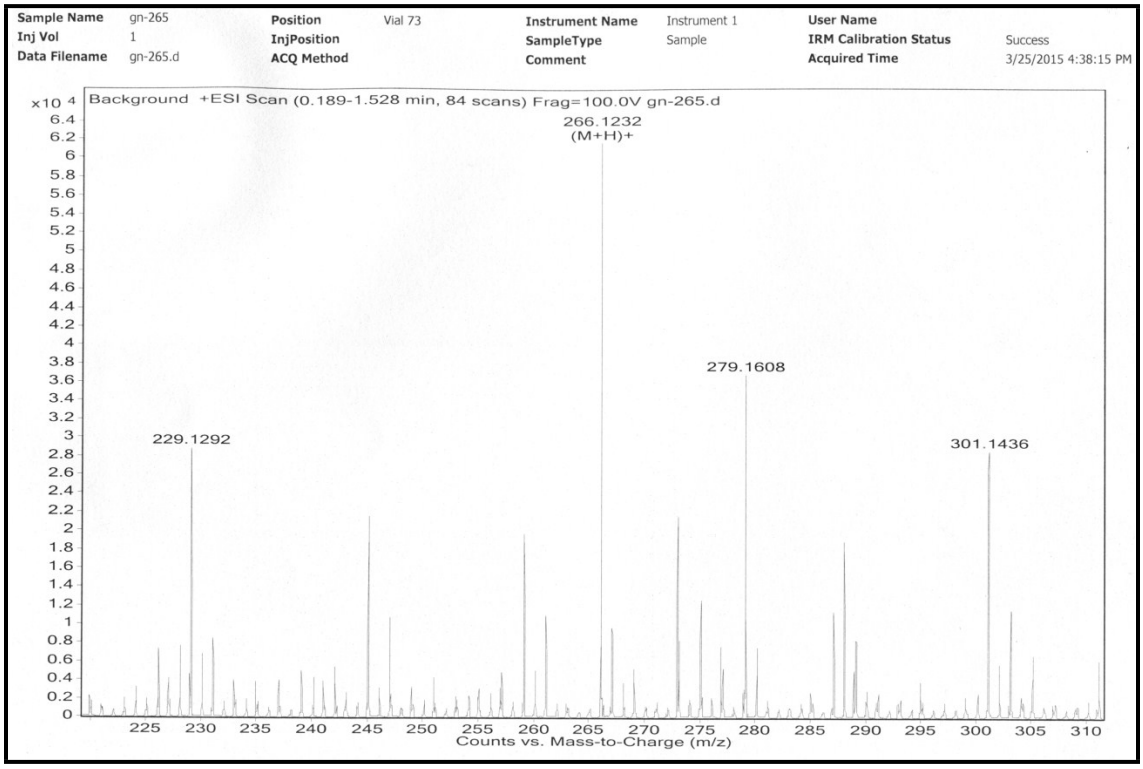
HRMS of Fmoc-Phe-ψ[CSNH]-Leu-COOMe 1.2e



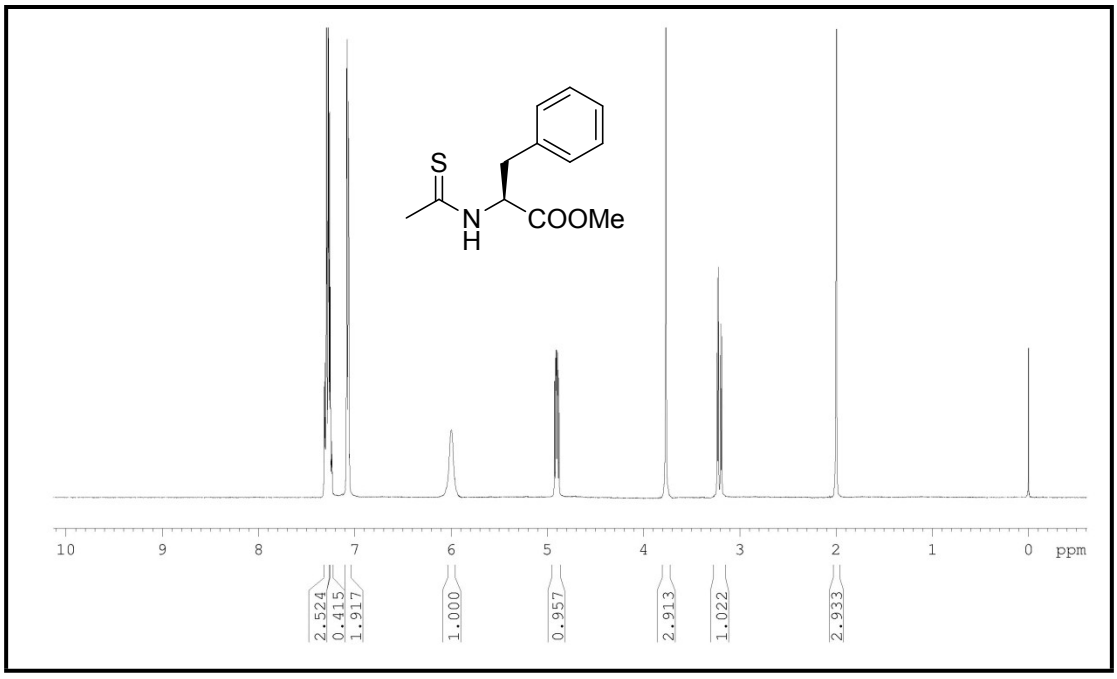
^{1}H NMR Spectrum of C_6H_5-ψ[CSNH]-Leu-COOMe 1.2f



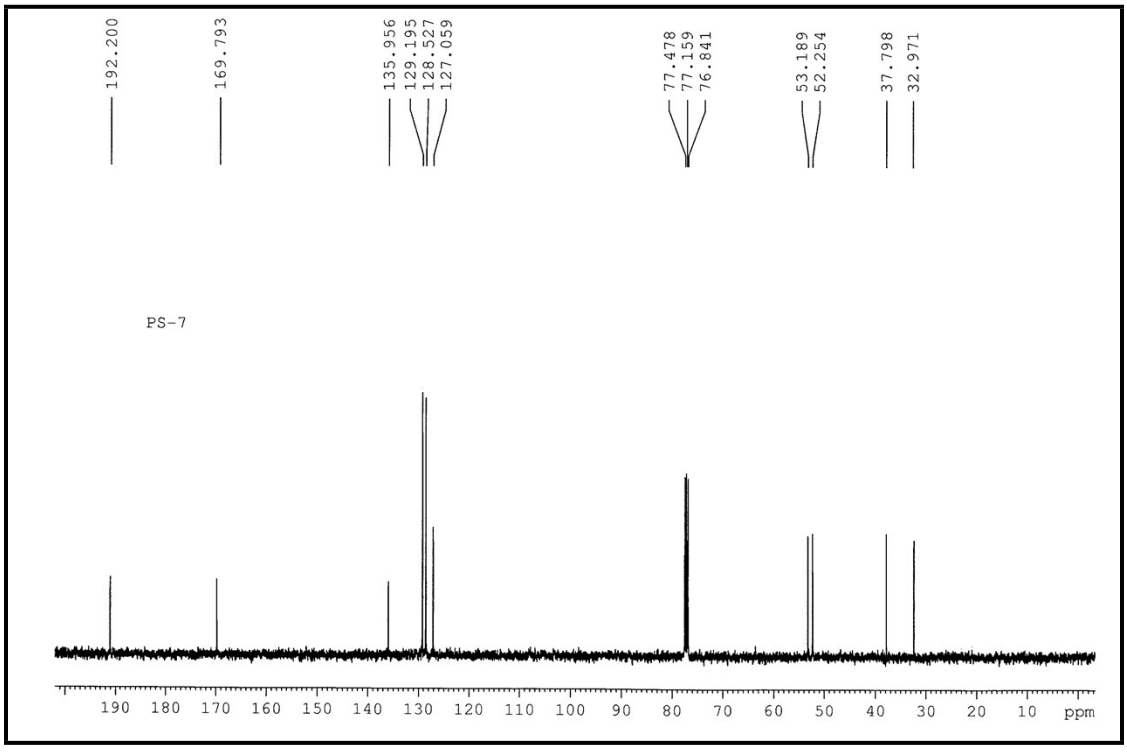
^{13}C NMR Spectrum of C_6H_5-ψ[CSNH]-Leu-COOMe 1.2f



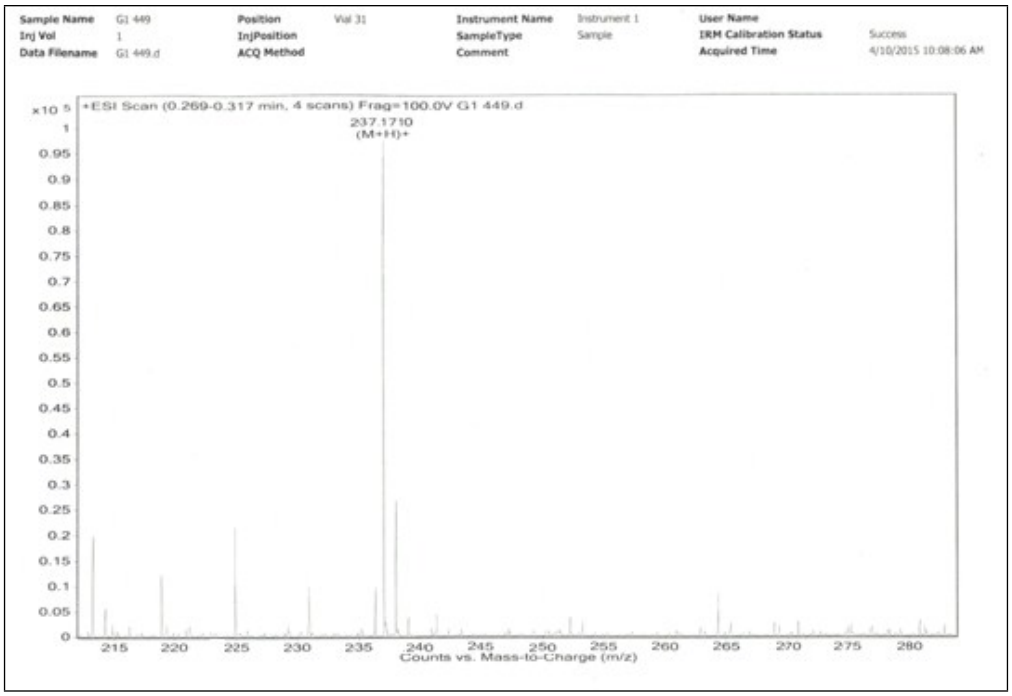
HRMS of C_6H_5-ψ[CSNH]-Leu-COOMe 1.2f



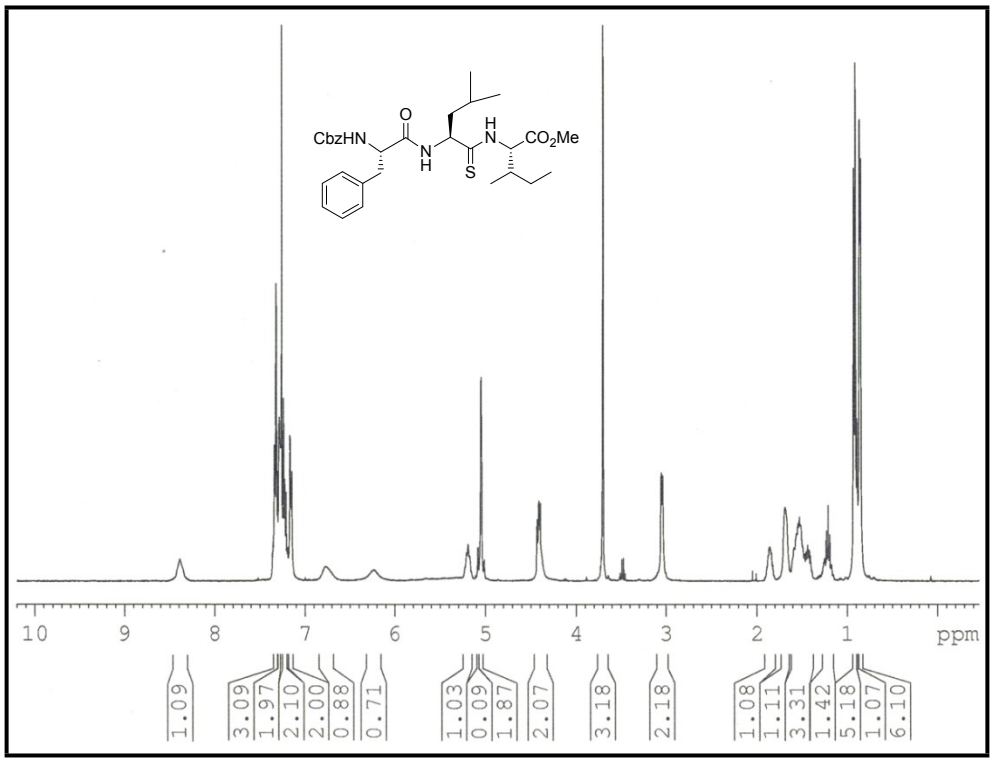
^{1}H NMR Spectrum of CH_3-ψ[CSNH]-Phe-COOMe 1.2g



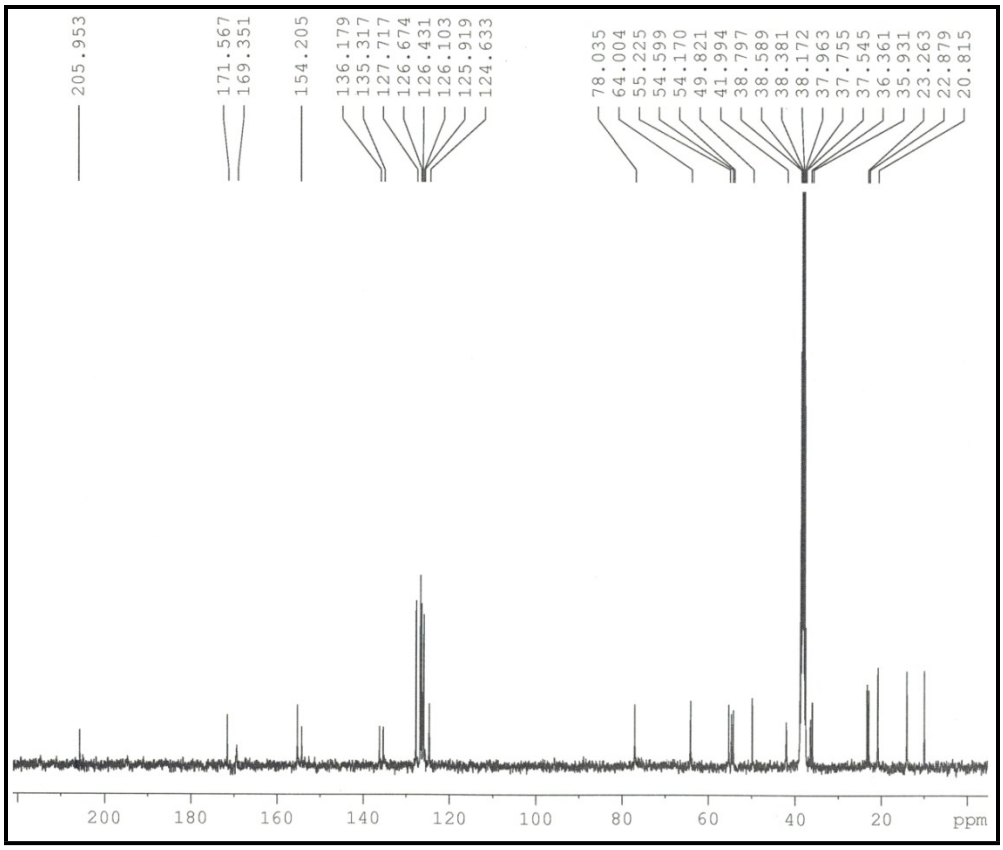
^{13}C NMR Spectrum of CH_3-ψ[CSNH]-Phe-COOMe 1.2g



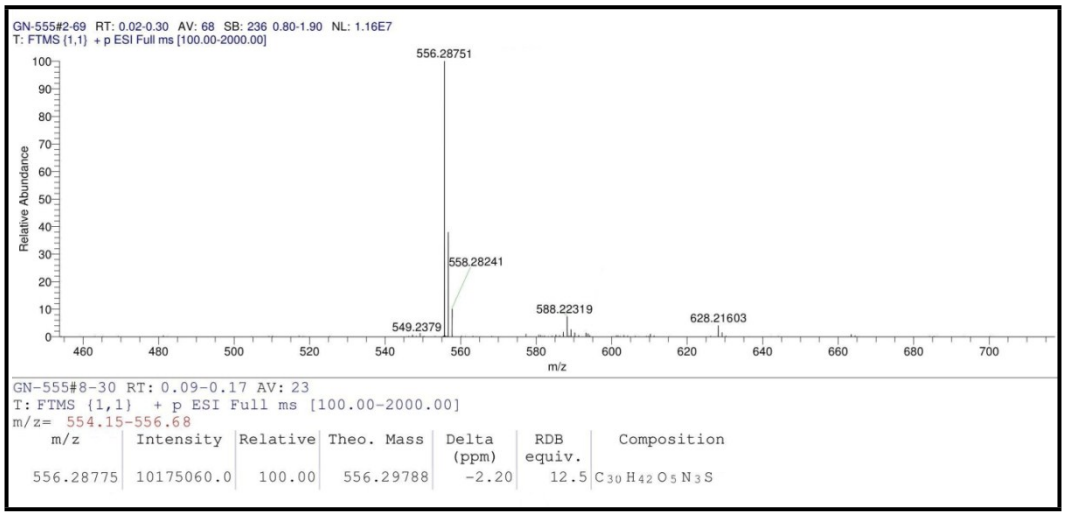
HRMS of CH_3-ψ[CSNH]-Phe-COOMe 1.2g



^{1}H NMR Spectrum of Cbz-Phe-Leu-ψ[CSNH]-ILe-COOMe 1.7a

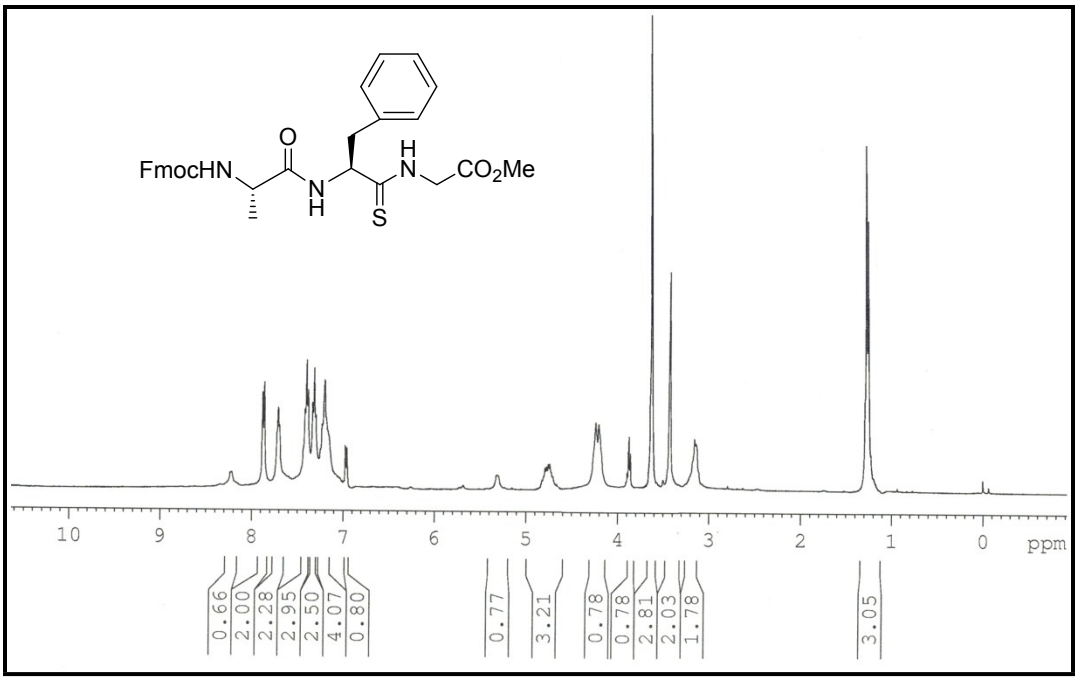


^{13}C NMR Spectrum of Cbz-Phe-Leu-ψ[CSNH]-ILe-COOMe 1.7a

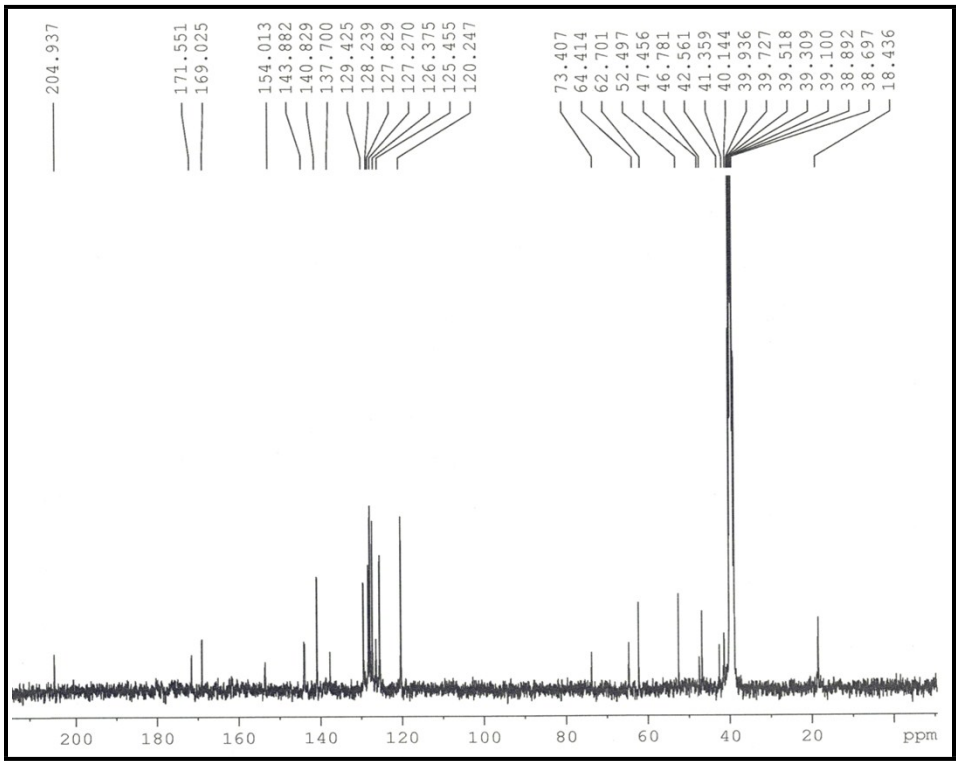


m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
556.28775	10175060.0	100.00	556.29788	-2.20	12.5	$C_{30}H_{42}O_5N_3S$

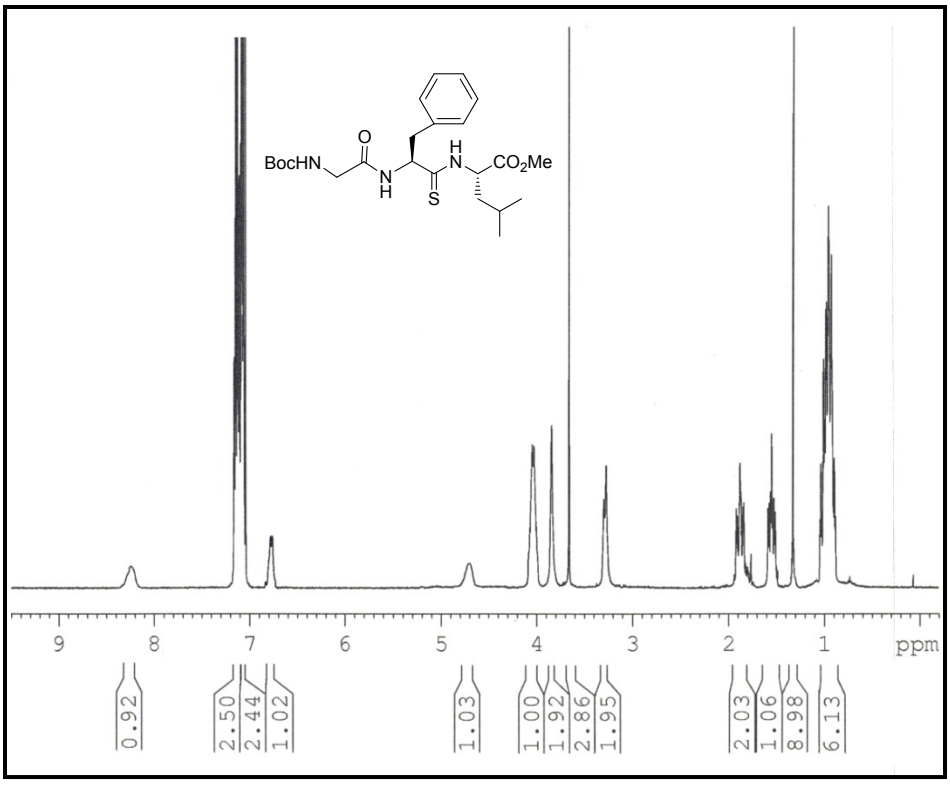
HRMS of Cbz-Phe-Leu-ψ[CSNH]-ILe-COOMe 1.7a



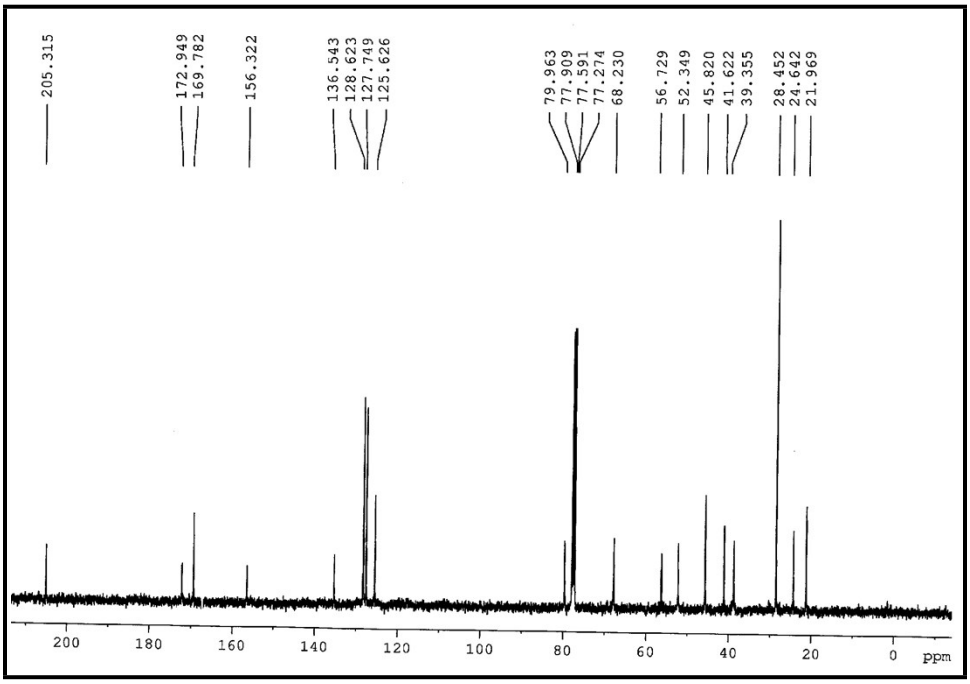
^{1}H NMR Spectrum of Fmoc-Ala-Phe-ψ[CSNH]-Gly-COOMe 1.7b



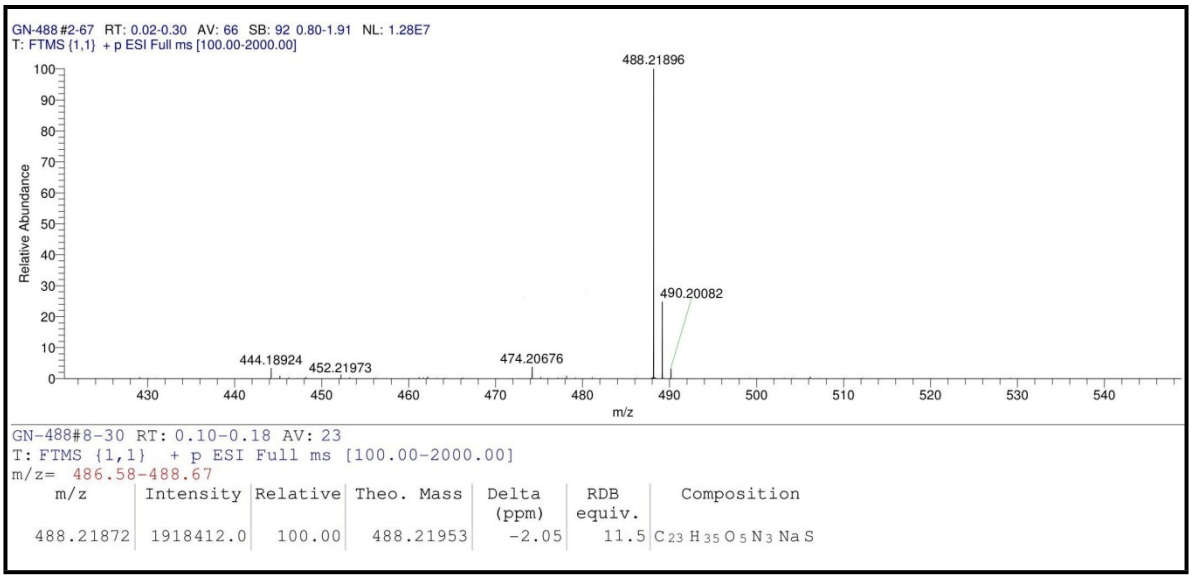
^{13}C NMR Spectrum of Fmoc-Ala-Phe-ψ[CSNH]-Gly-COOMe 1.7b



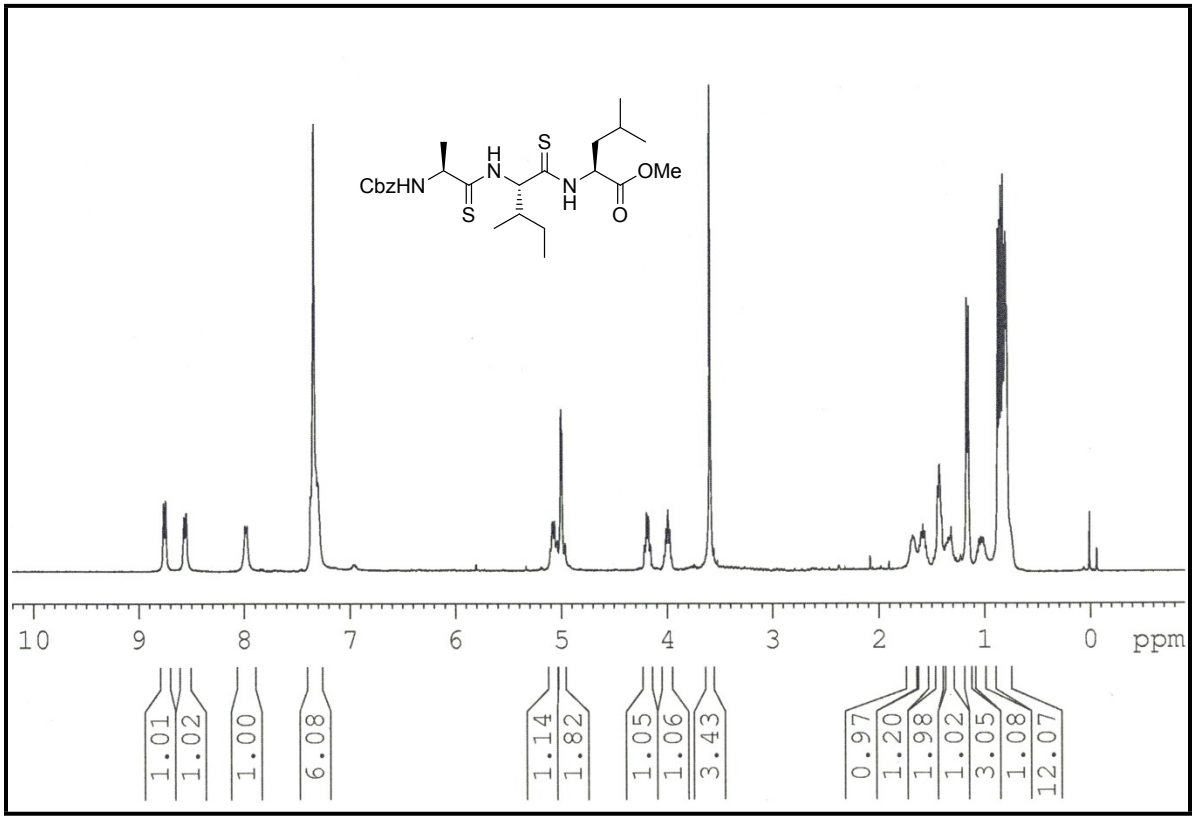
^{1}H NMR Spectrum of Boc-Gly-Phe-ψ[CSNH]-Leu-COOMe 1.7c



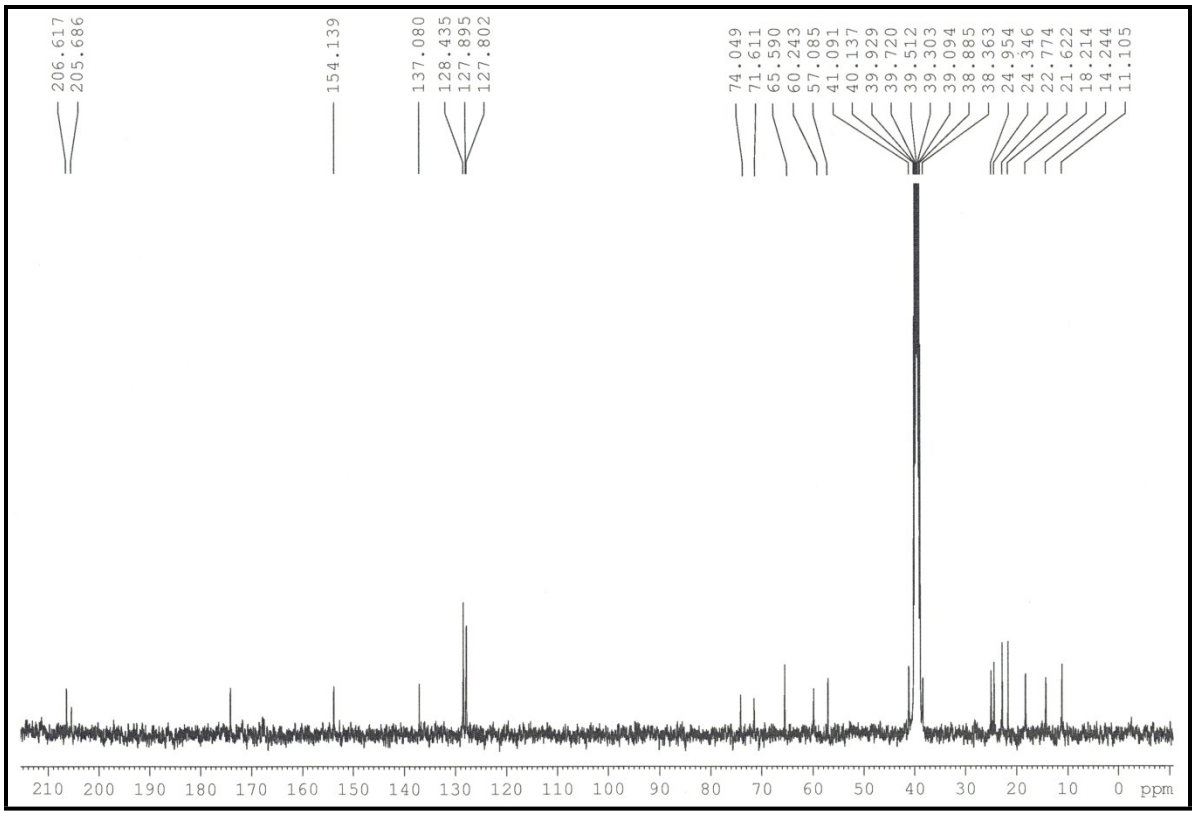
^{13}C NMR Spectrum of Boc-Gly-Phe-ψ[CSNH]-Leu-COOMe 1.7c



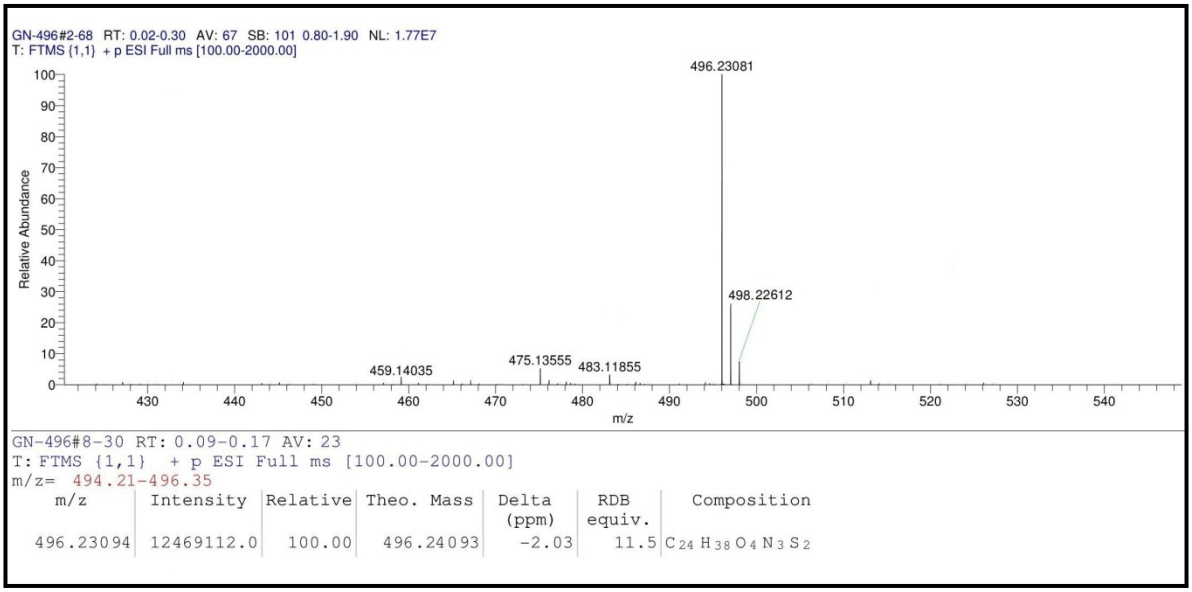
HRMS of Boc-Gly-Phe-ψ[CSNH]-Leu-COOMe 1.7c



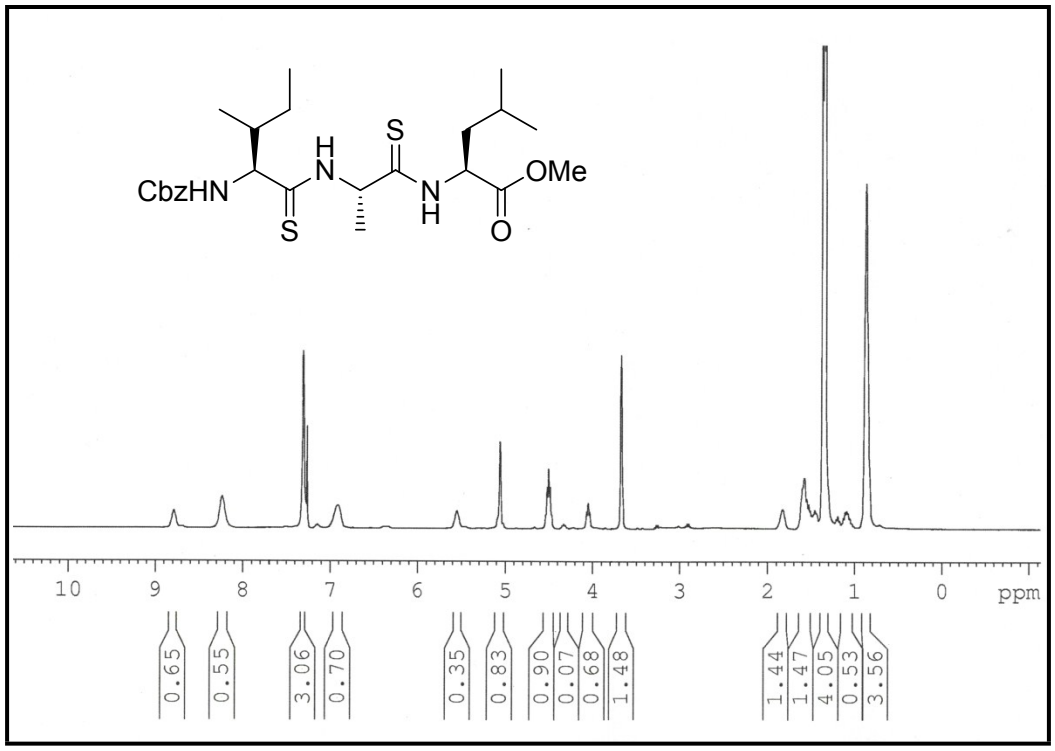
^{1}H NMR Spectrum of Cbz-Ala- ψ[CSNH]-Ile-ψ[CSNH]-Leu-COOMe 1.9a



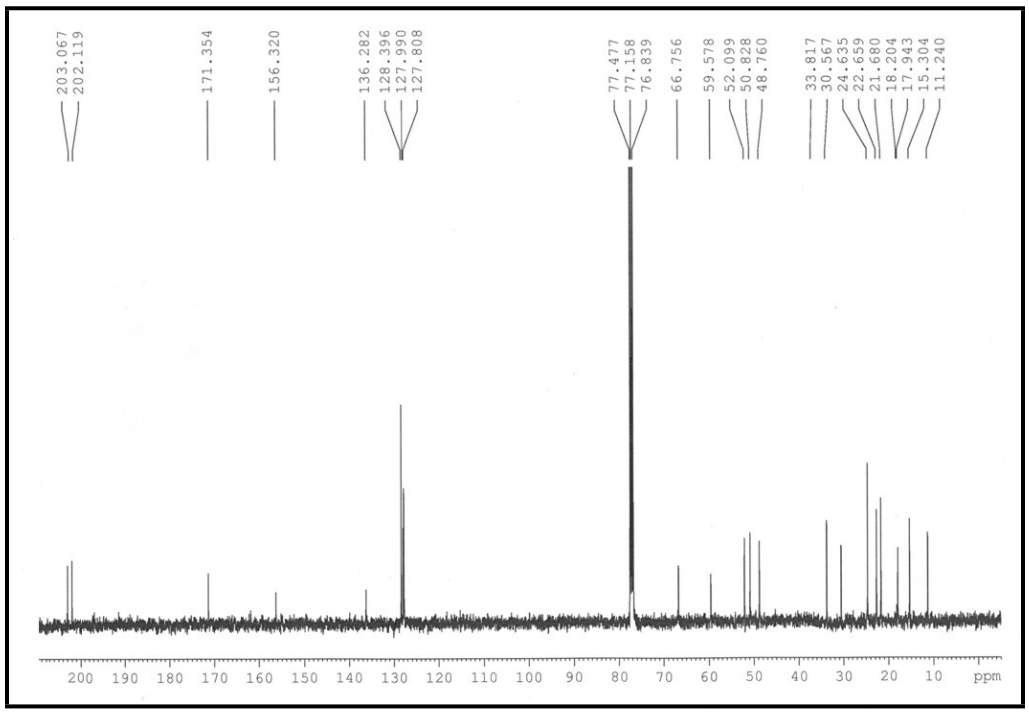
^{13}C NMR Spectrum of Cbz-Ala- ψ[CSNH]-Ile-ψ[CSNH]-Leu-COOMe 1.9a



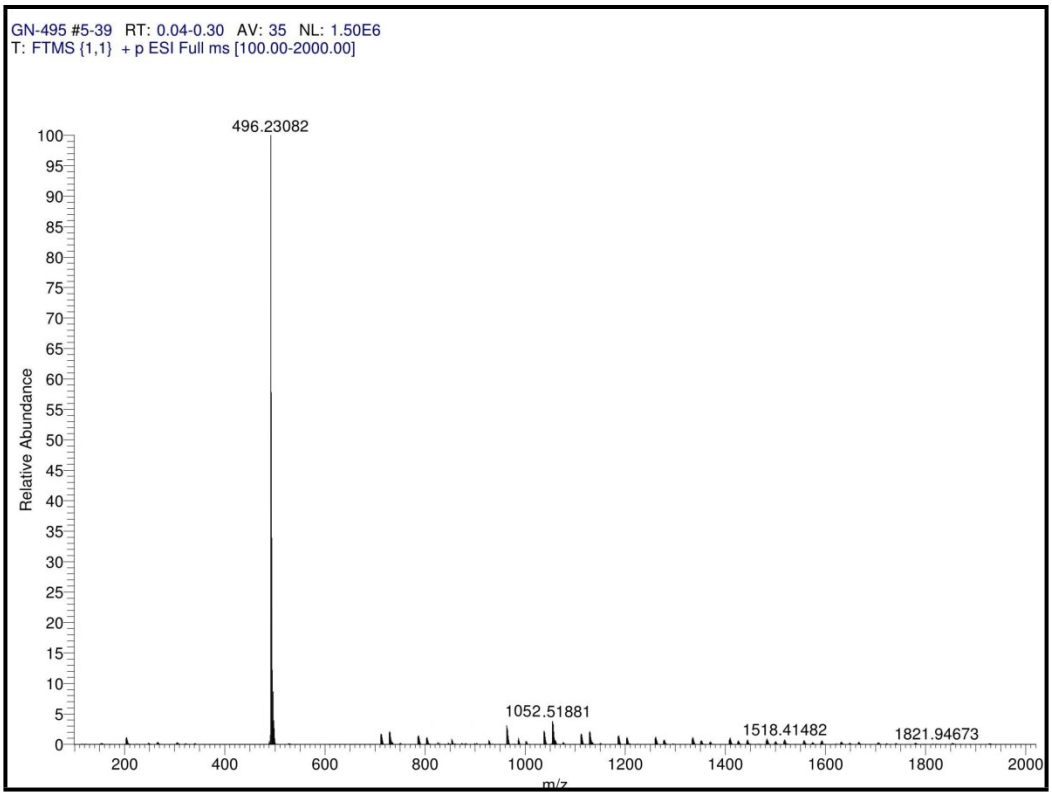
HRMS of Cbz-Ala- ψ[CSNH]-Ile-ψ[CSNH]-Leu-COOMe 1.9a



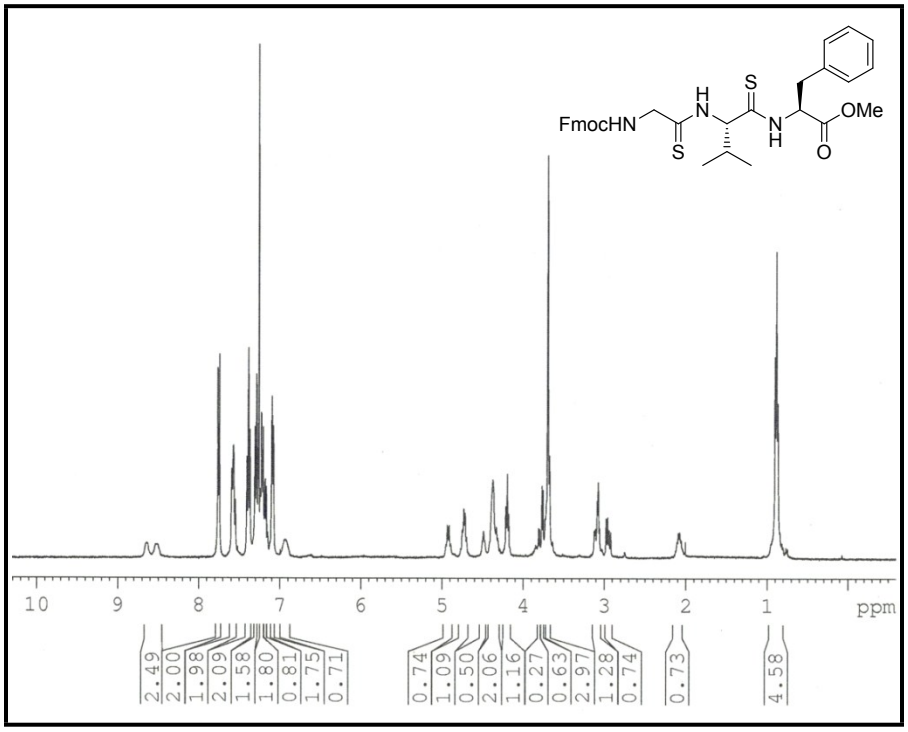
^{1}H NMR Spectrum of Cbz-Ile-ψ[CSNH]-Ala-ψ[CSNH]-Leu-COOMe 1.9b



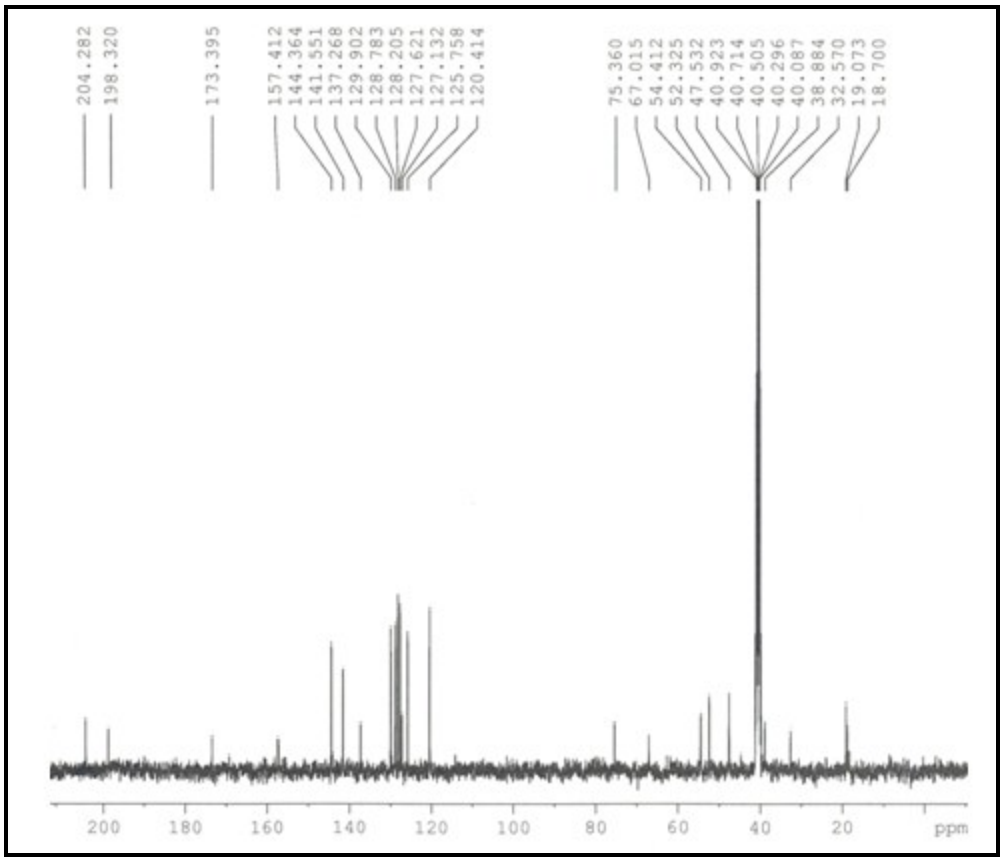
^{13}C NMR Spectrum of Cbz-Ile-ψ[CSNH]-Ala-ψ[CSNH]-Leu-COOMe 1.9b



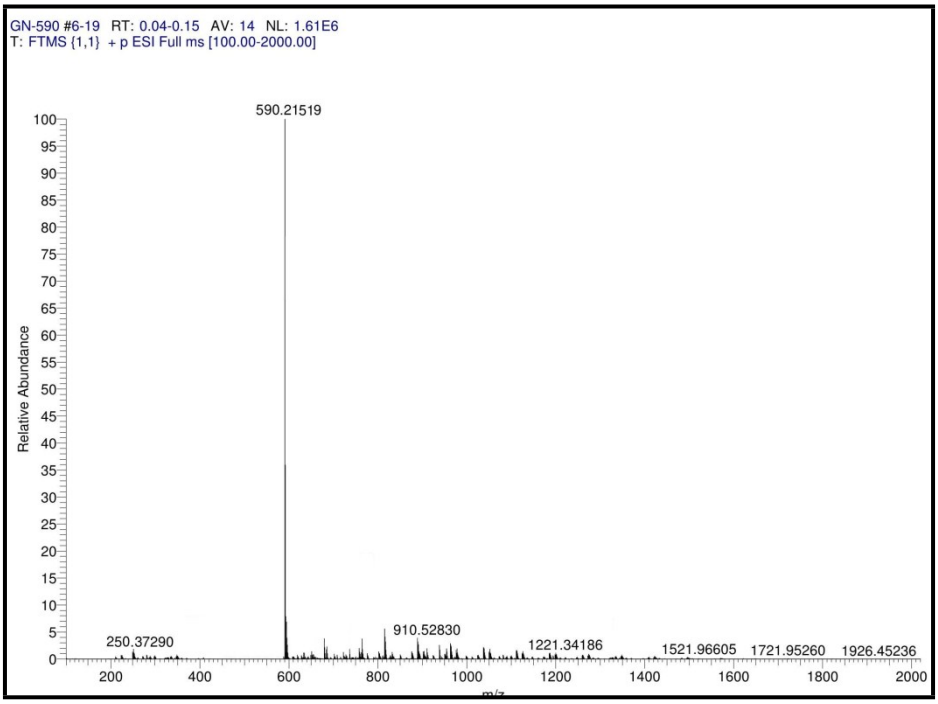
HRMS of Cbz-Ile-ψ[CSNH]-Ala-ψ[CSNH]-Leu-COOMe 1.9b



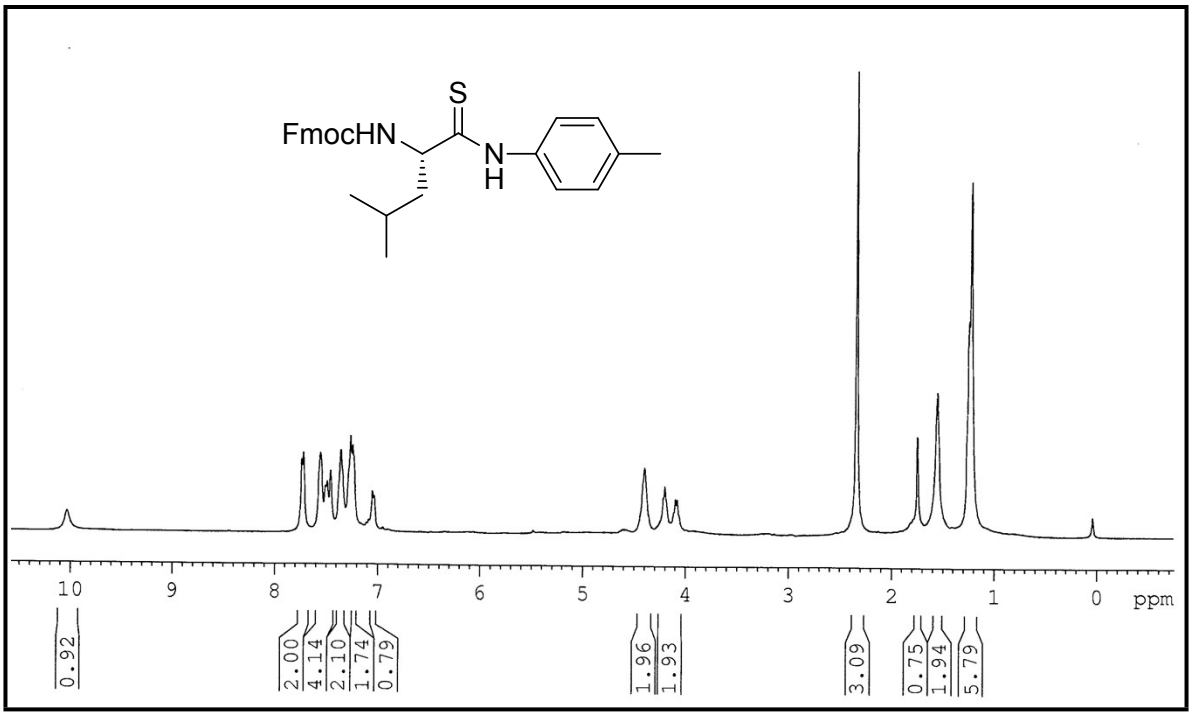
^{1}H NMR Spectrum of Fmoc-Gly-ψ[CSNH]-Val-ψ[CSNH]-Phe-COOMe 1.9c



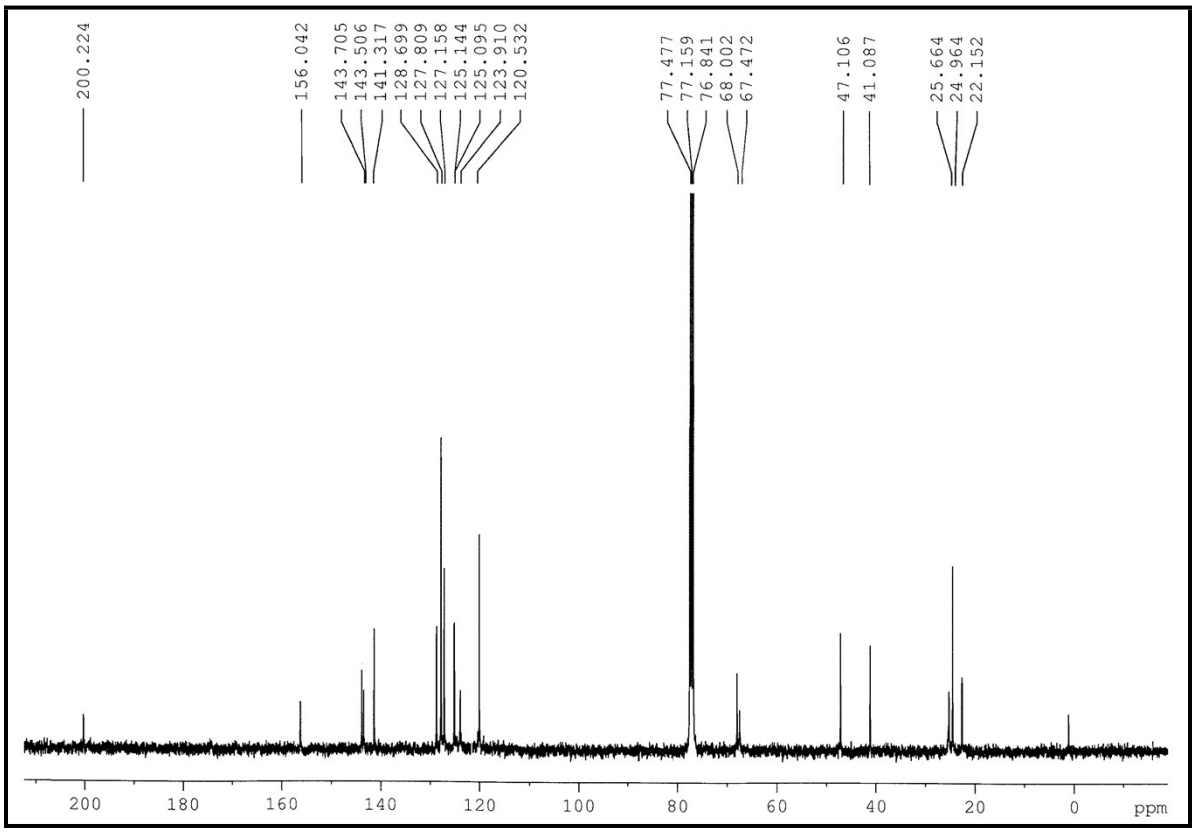
^{13}C NMR Spectrum of Fmoc-Gly-ψ[CSNH]-Val-ψ[CSNH]-Phe-COOMe 1.9c



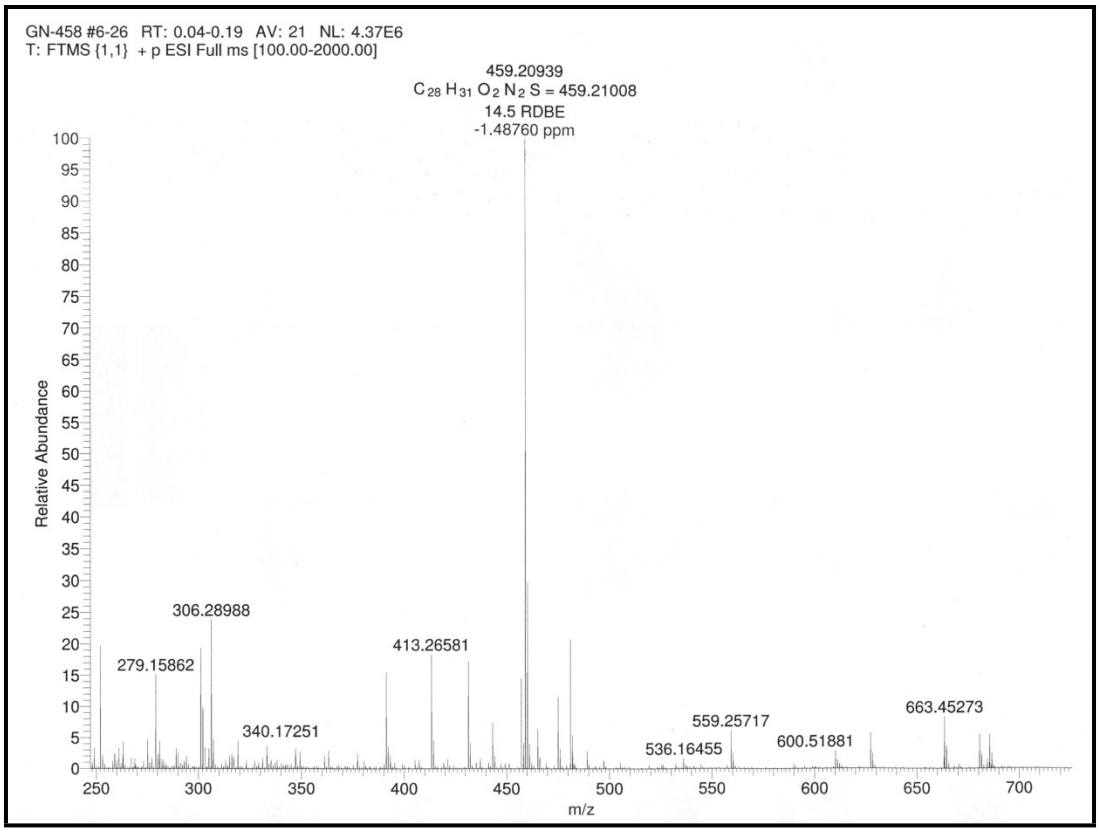
HRMS of Fmoc-Gly-ψ[CSNH]-Val-ψ[CSNH]-Phe-COOMe 1.9c



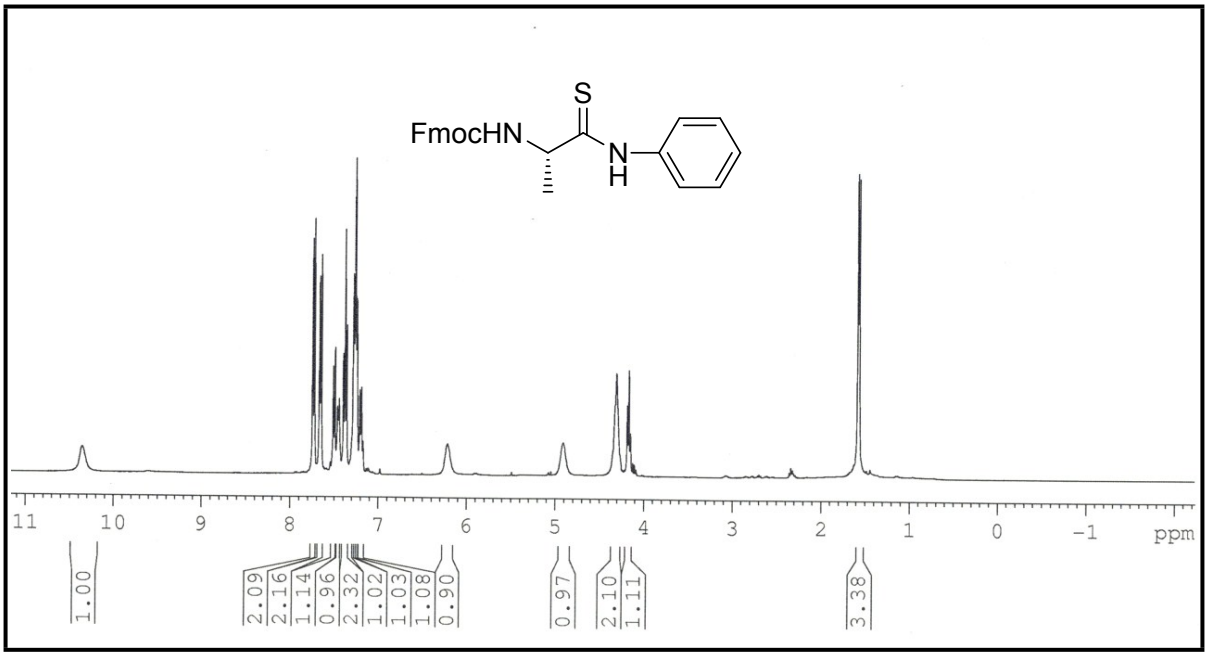
^{1}H NMR Spectrum of Compound 1.11a



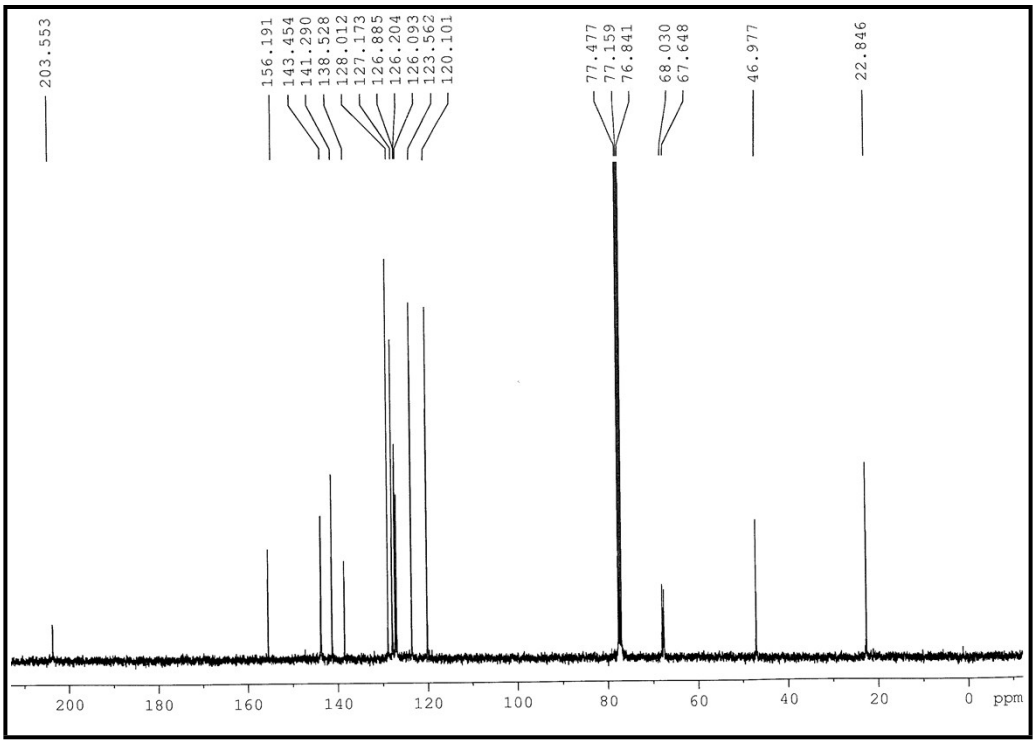
^{13}C NMR Spectrum of Compound 1.11a



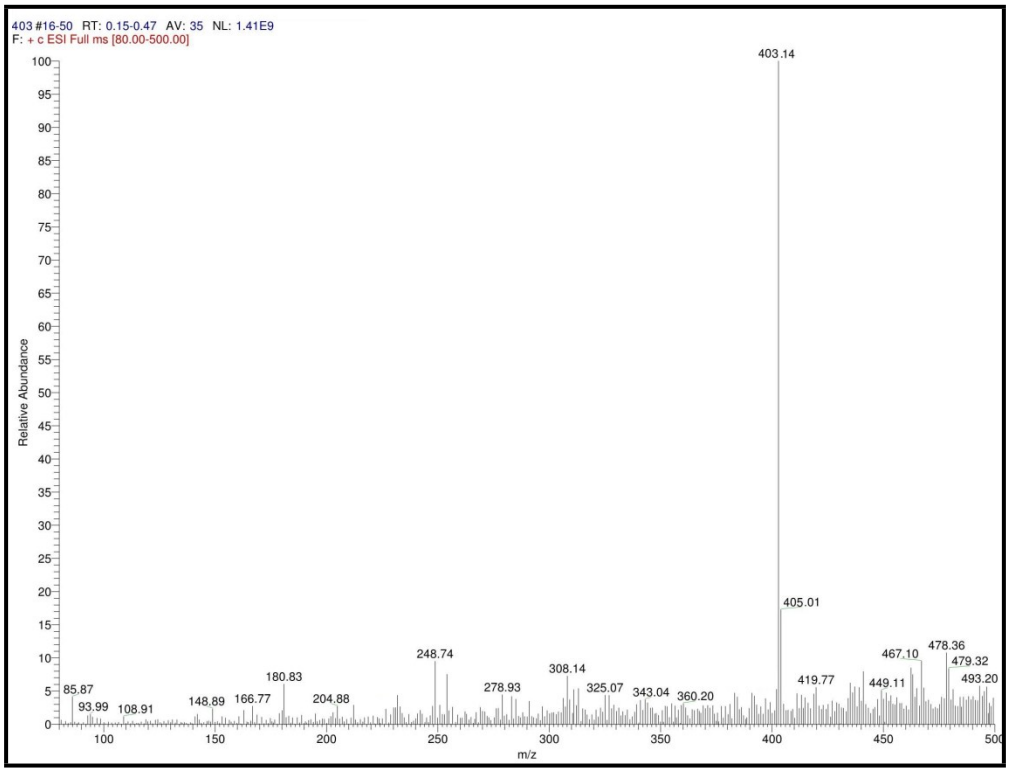
HRMS of Compound 1.11a



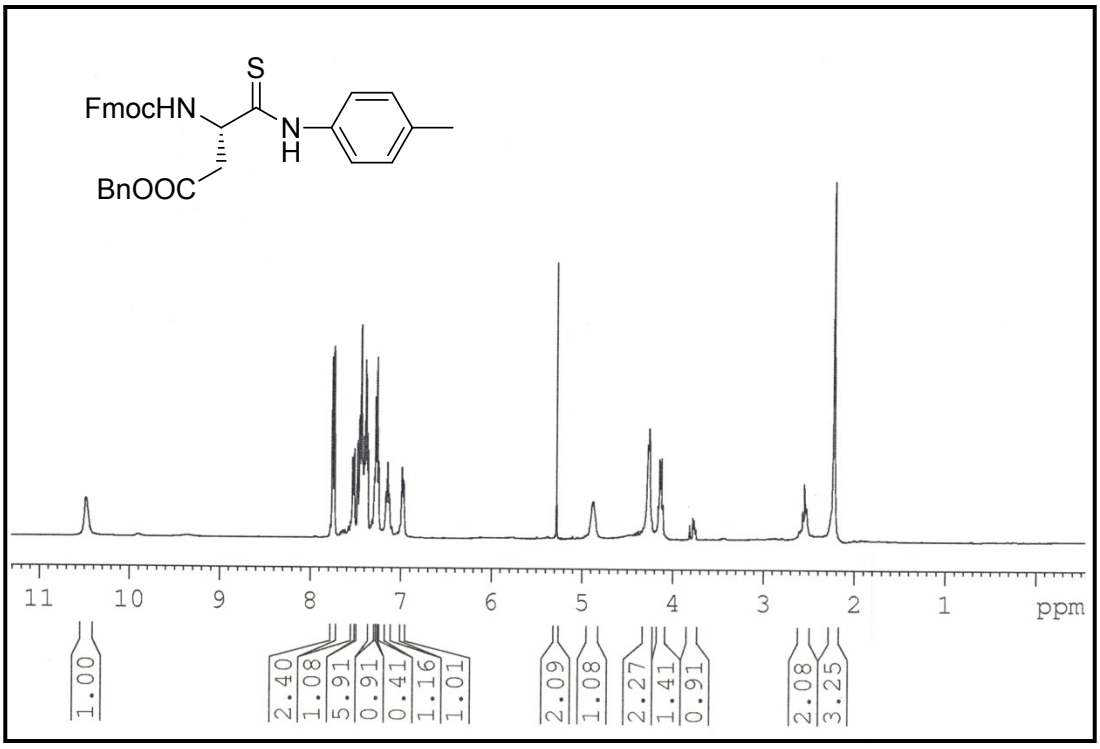
^{1}H NMR Spectrum of Compound 1.11b



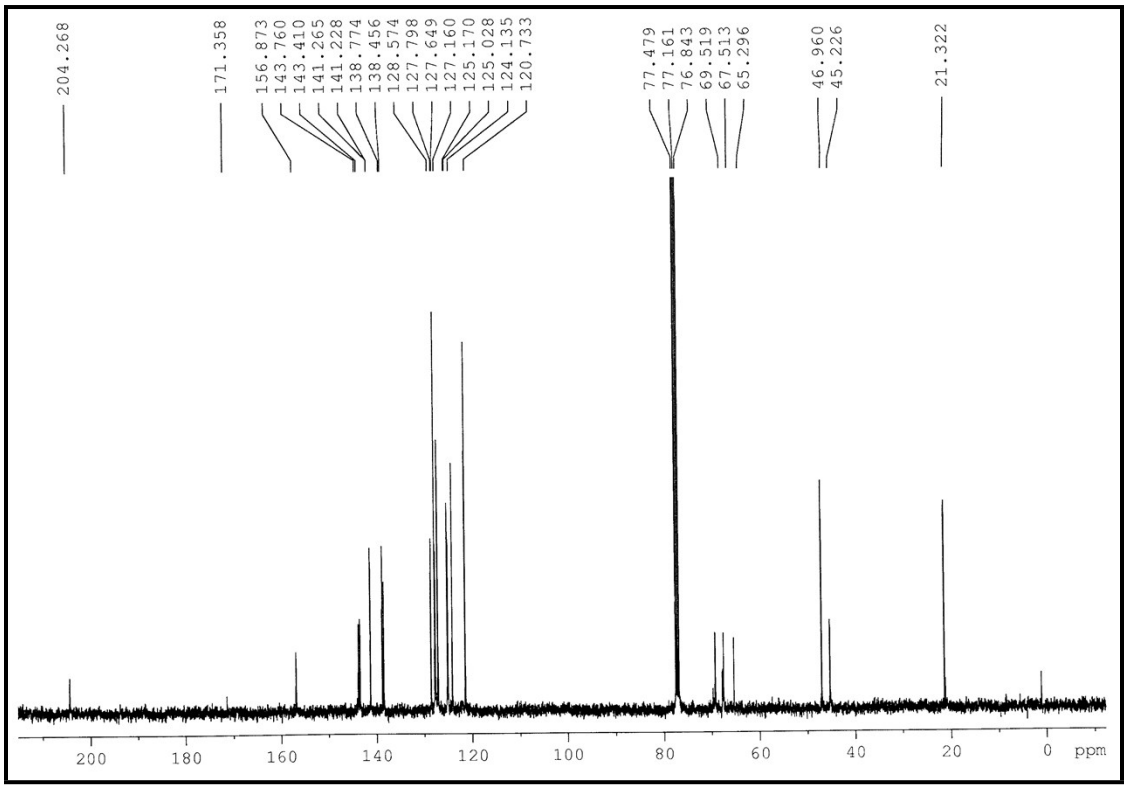
^{13}C NMR Spectrum of Compound 1.11b



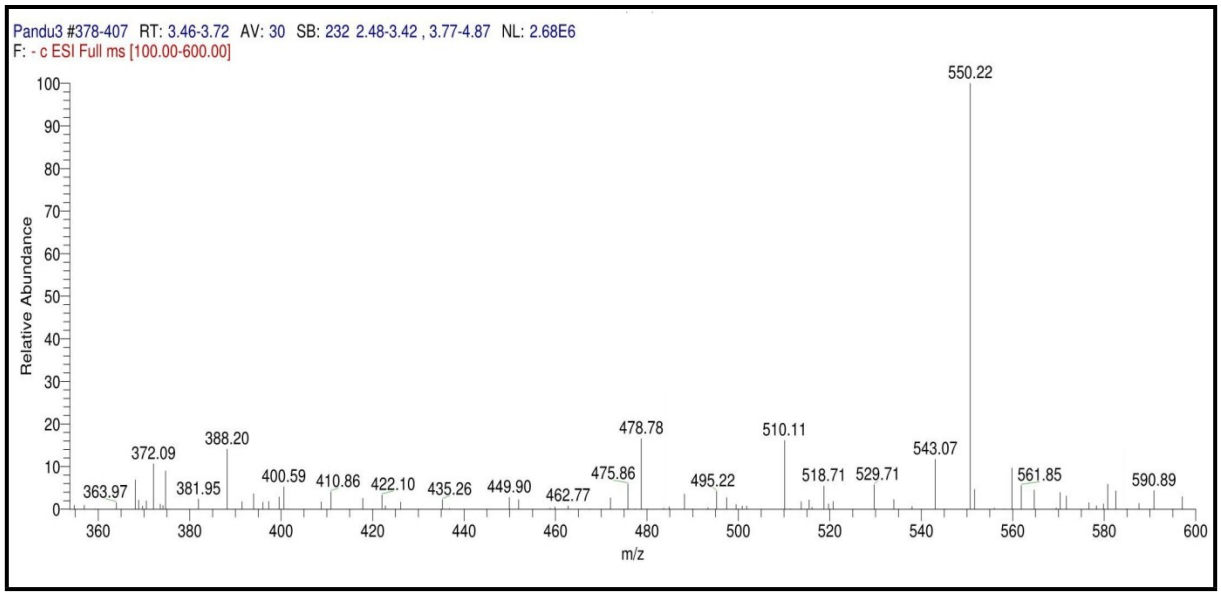
ESI MS of Compound 1.1b



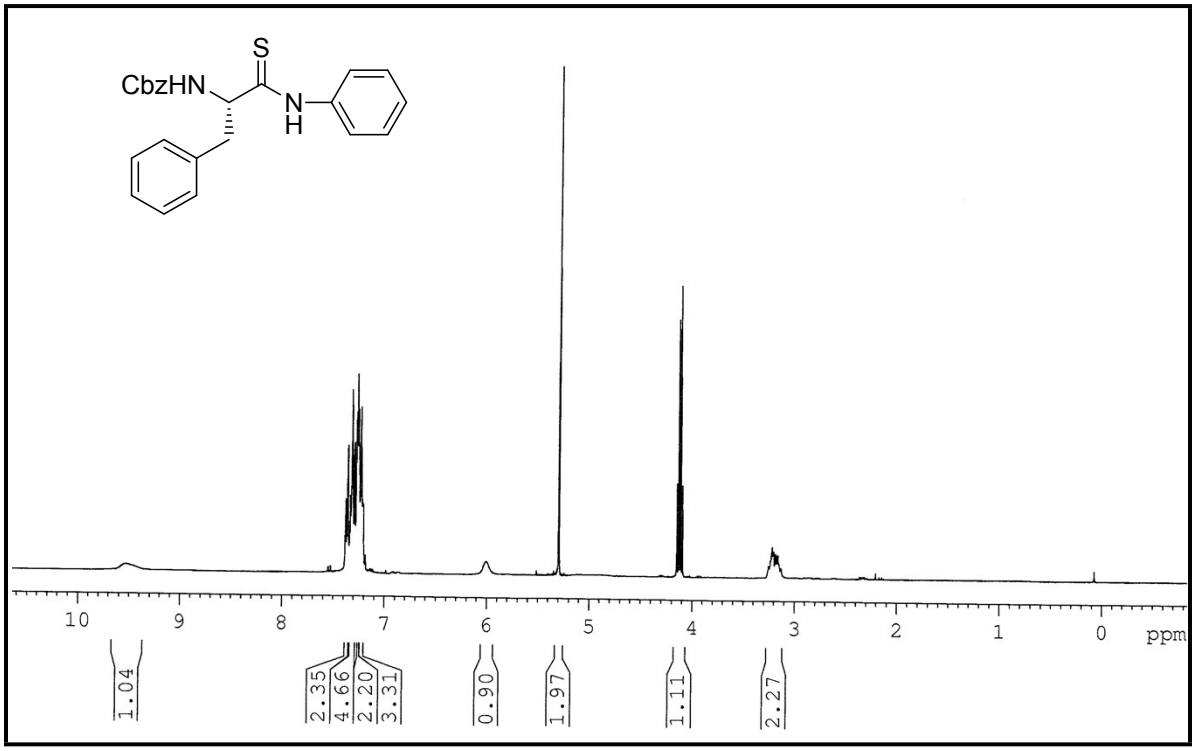
^{1}H NMR Spectrum of Compound 1.11c



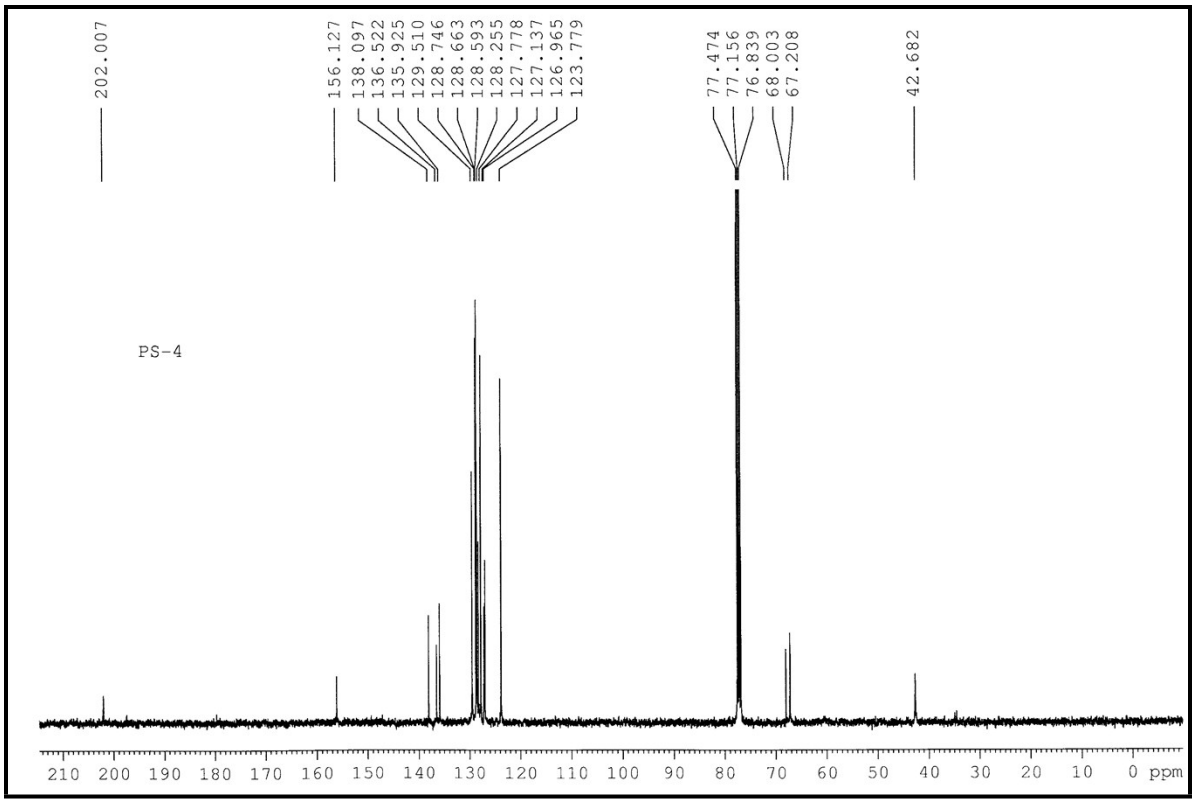
^{13}C NMR Spectrum of Compound 1.11c



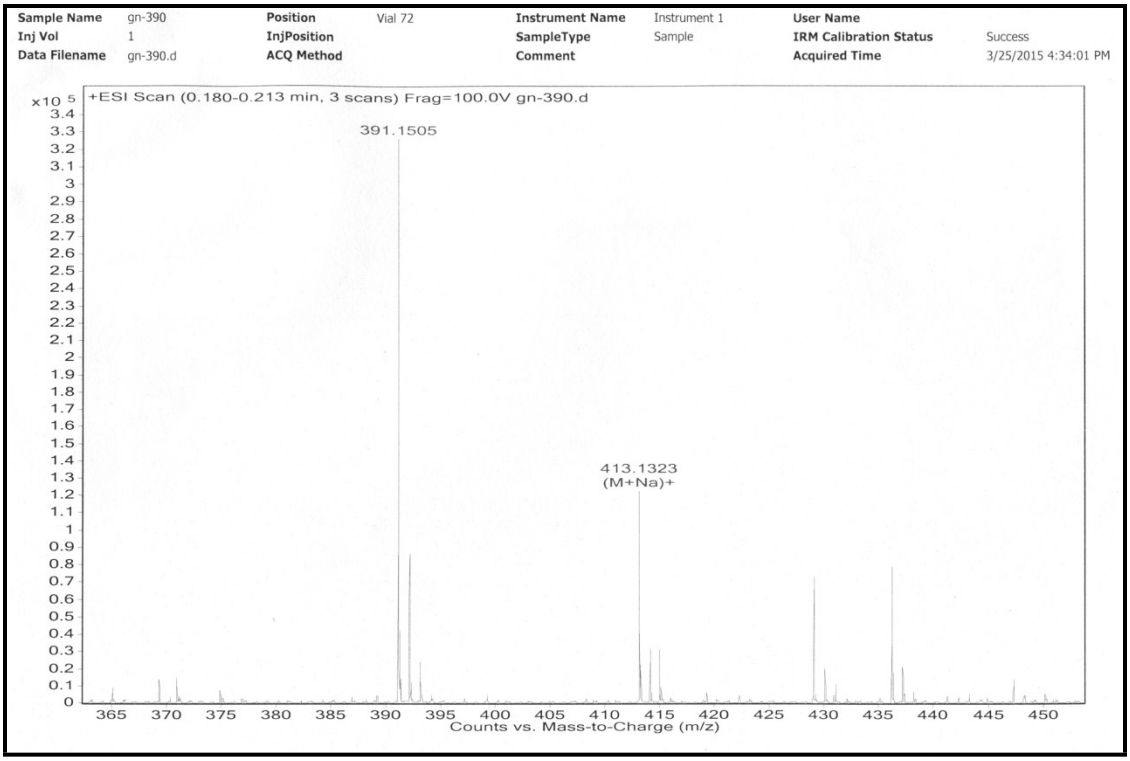
ESI MS of Compound 1.11c



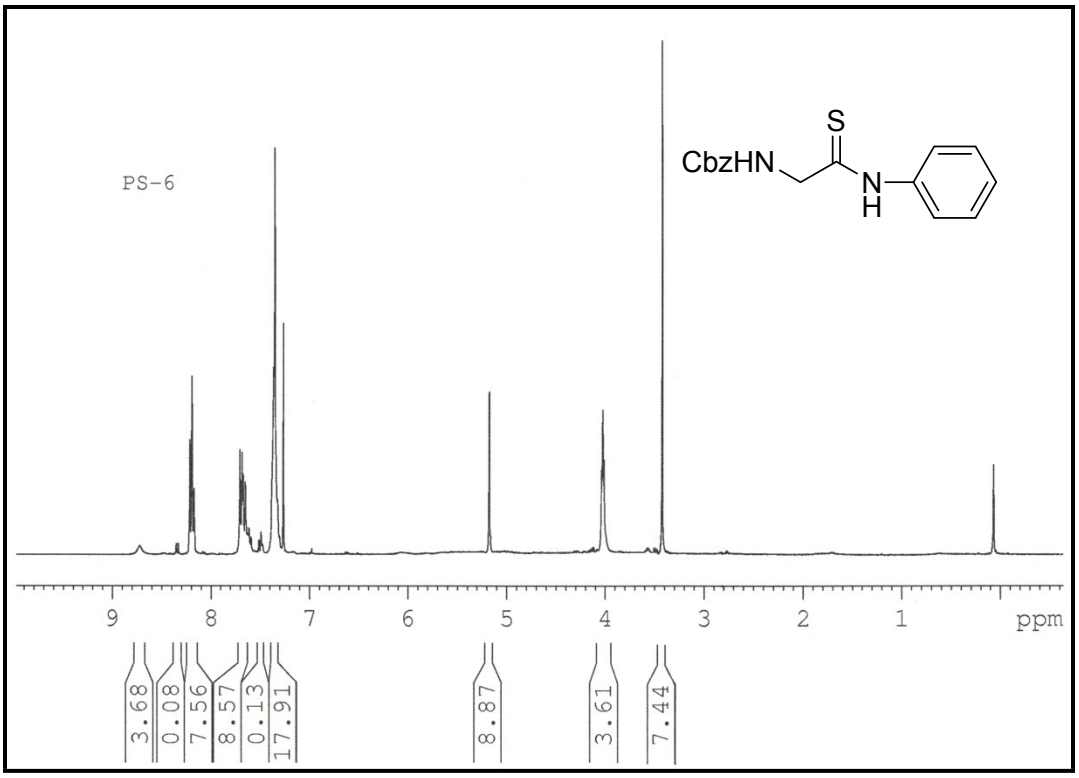
^{1}H NMR Spectrum of Compound 1.11d



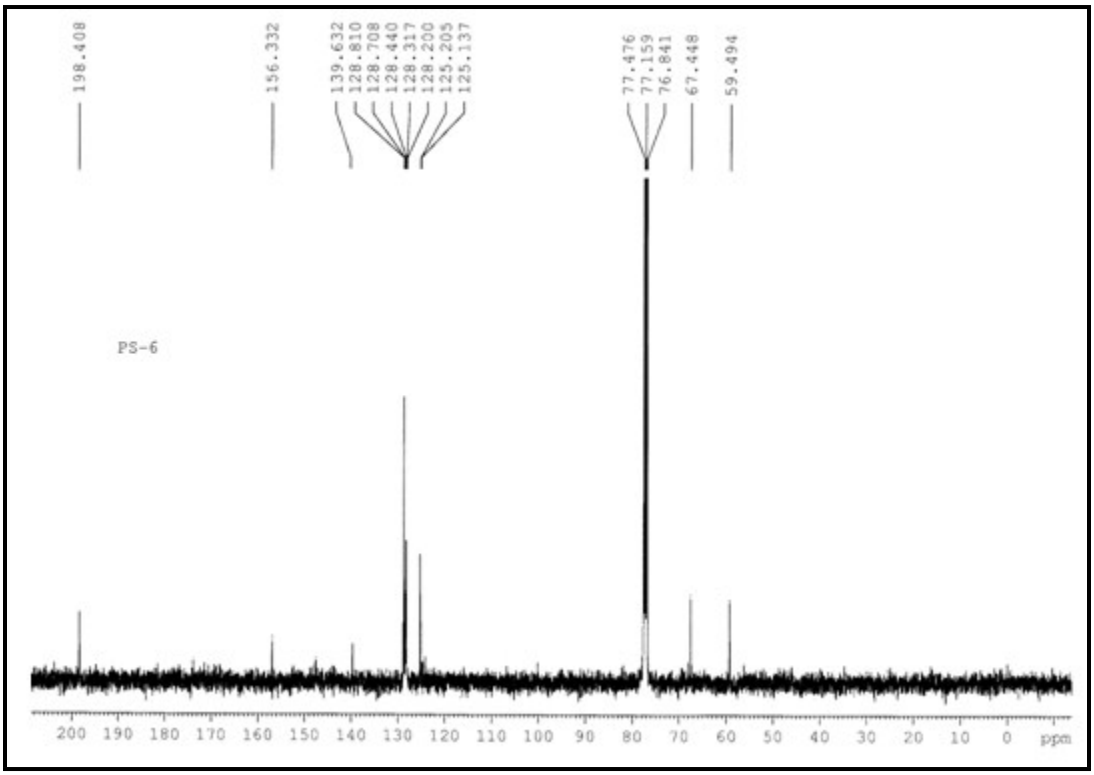
^{13}C NMR Spectrum of Compound 1.11d



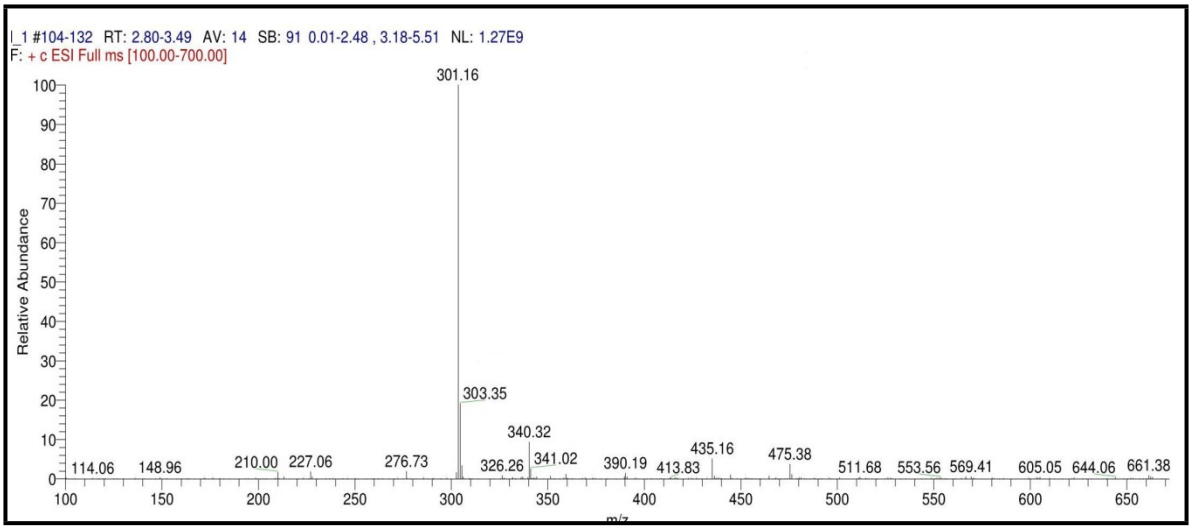
HRMS of Compound 1.11d



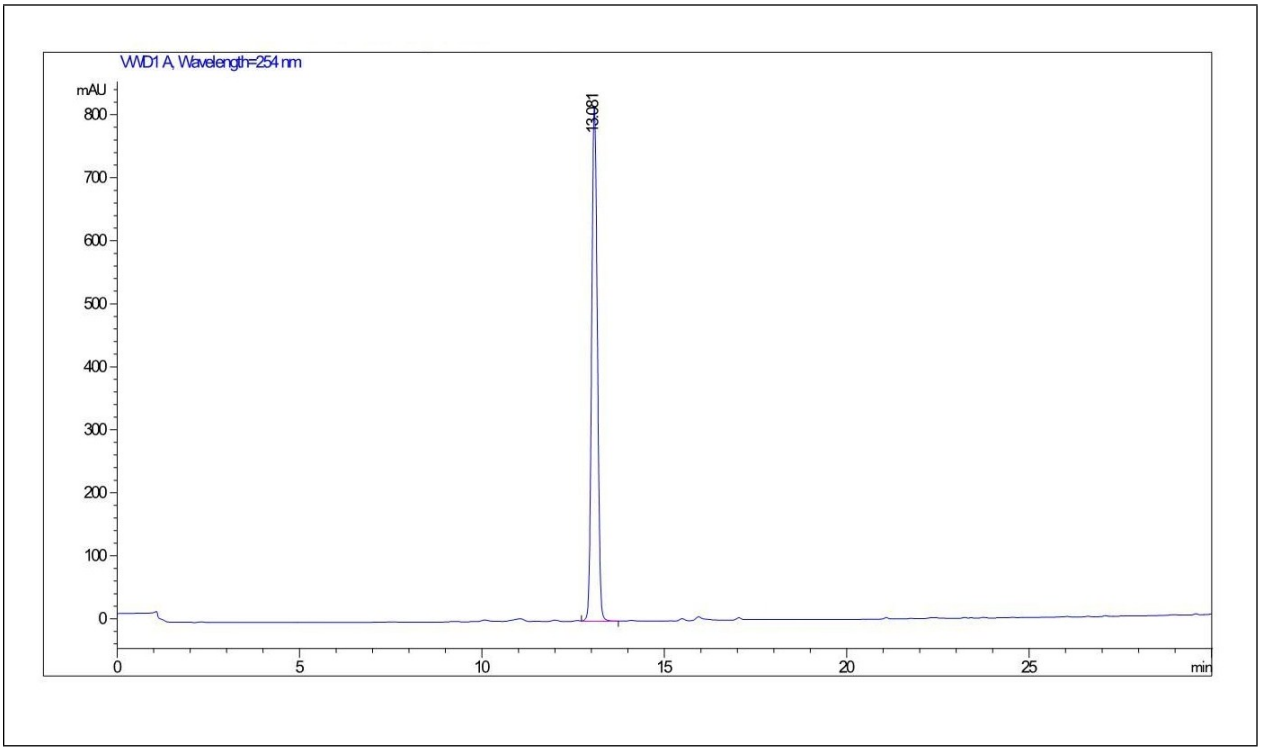
^{1}H NMR Spectrum of Compound 1.11e



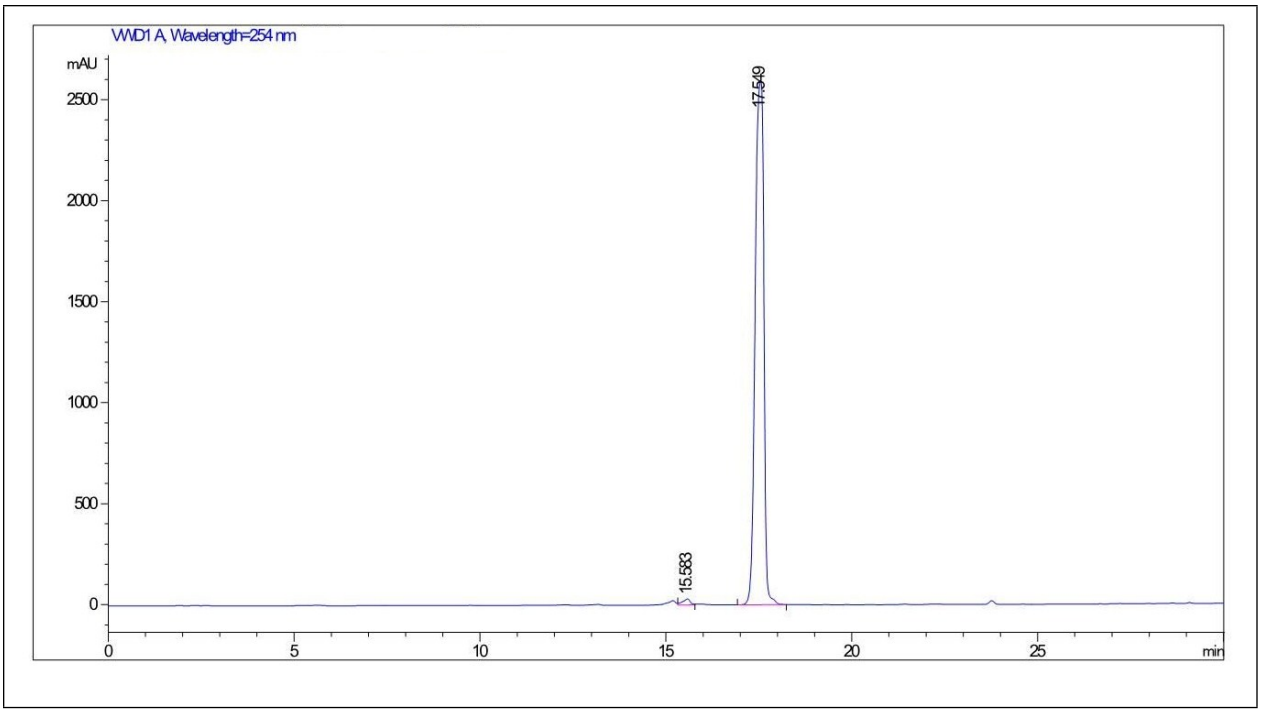
^{13}C NMR Spectrum of Compound 1.11e



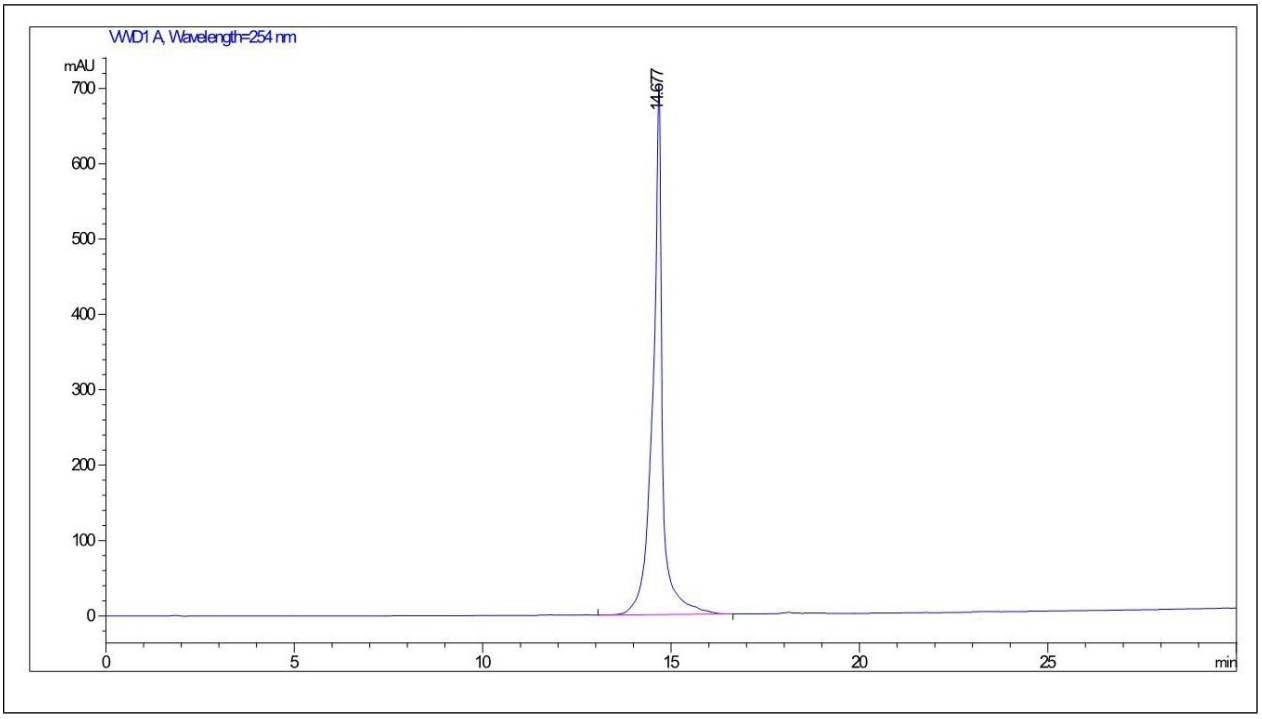
ESI MS of Compound 1.11e



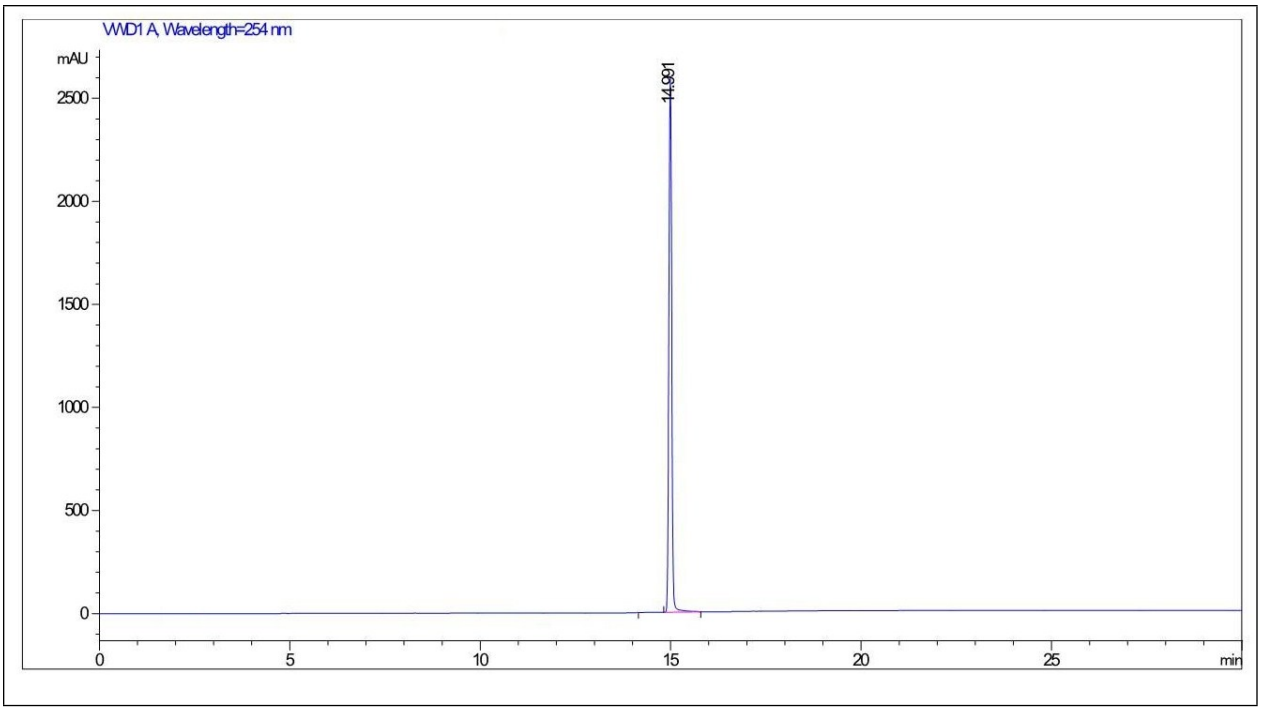
HPLC Spectrum of Fmoc-Phe-ψ[CSNH]-Ala-COOMe 1.2d



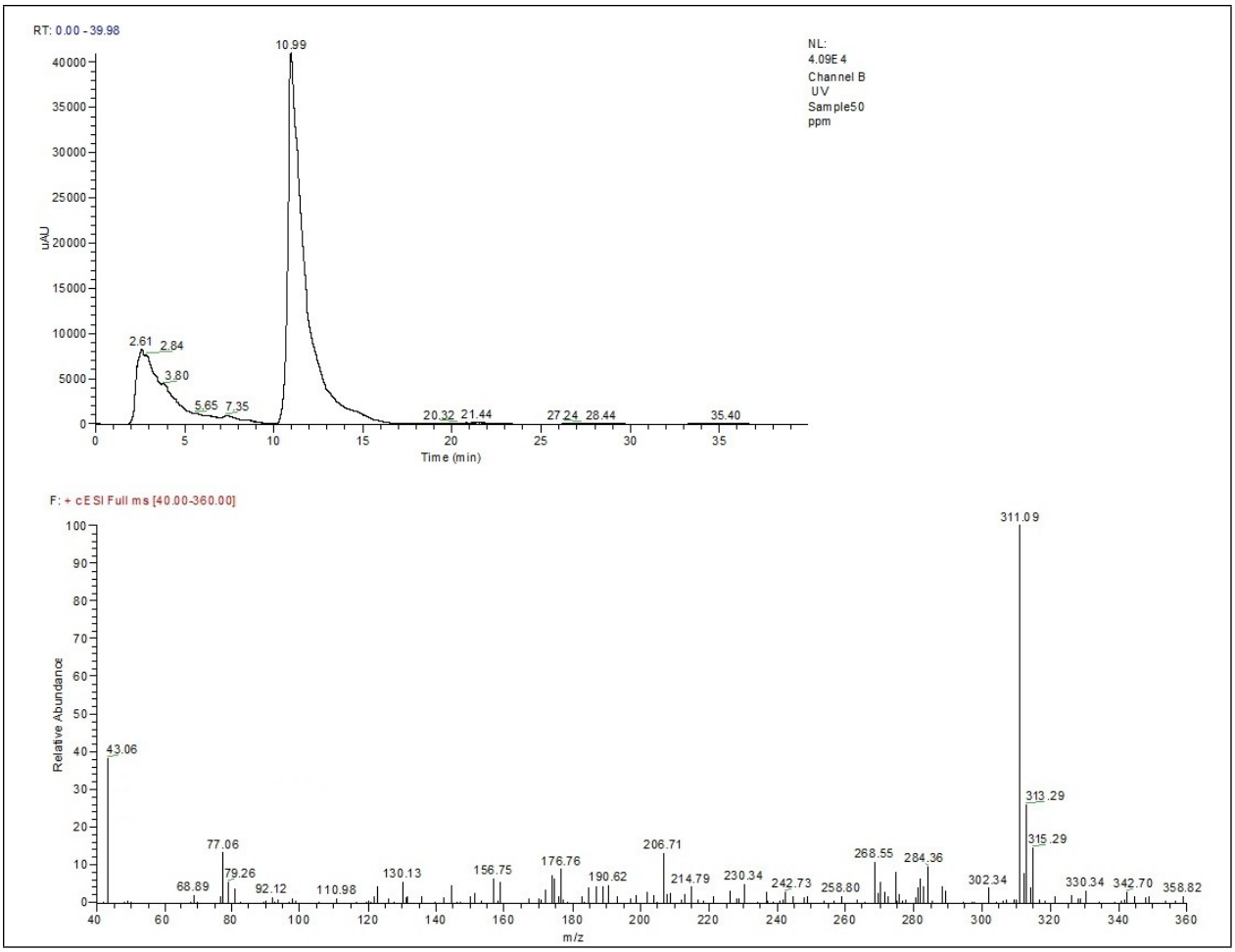
HPLC Spectrum of Fmoc-Ala-Phe-ψ[CSNH]-Gly-COOMe 1.7b



HPLC Spectrum of Cbz-Ala- ψ[CSNH]-Ile-ψ[CSNH]-Leu-COOMe 1.9a



HPLC Spectrum of Compound 1.11c



LCMS of crude Cbz-Ala-ψ[CSNH]-Gly-COOMe 1.2b and proof for the NH_2CN (peak at 43.06)