Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Supplementary Data

New and simple synthesis of acid azides, ureas and carbamates from carboxylic acids: Application of peptide coupling agents EDC and HBTU

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Experimental section

General information:

All solvents were freshly distilled prior to use. Melting points were determined using capillary method and are uncorrected. . The TLC was effected with silica gel GF_{254} pre-coated on glass plates using the following solvent systems as mobile phases : (a) Ethyl acetate : Hexane (2:8) for azides (b) Chloroform : Methanol (9:1) for ureas, (b) Ethyl acetate : Hexane (3:7) for carbamates.

CHARACTERIZATION DATA

pyridine-3-carboxylic acid azide 2a:

R_f 0.3 (n-hexane/EtOAc 8:2); IR (KBr) υ_{max} = 2130 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.78 – 7.68 (2H, m), 8.11 (1H, d, J = 7.6 Hz), 8.30 (1H, s); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 126.42, 129.31, 135.83, 151.18, 152.24, 172.35; HRMS Calc'd for $C_6H_4N_4O$ m/z: 171.0283 (M^++Na), found 171.0286

pyridine-2-carboxylic acid azide 2b:

R_f 0.3 (n-hexane/EtOAc 8:2); IR (KBr) υ_{max} = 2128 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 7.37 (1H, m), 7.71 (1H, m), 7.95 (1H, d, *J* = 7.10 Hz), 8.53 (1H, d, *J* = 6.5 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 125.00, 128.36, 137.59, 148.18, 150.19, 172.20; ES MS m/z: Calc'd for $C_6H_4N_4O$ m/z: 171.0 (M^++Na), found 171.0

furan-2-carboxylic acid azide 2c:

R_f 0.4 (n-hexane/EtOAc 9:1); IR (KBr) υ_{max} = 2132 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 6.87 (1H, m), 7.30 (1H, d, *J* = 7.0 Hz), 7.23 (1H, d, *J* = 6.9 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 112.04, 120.50, 144.30, 146.85, 178.95; HRMS Calc'd for $C_5H_3N_3O_2$ m/z: 160.0123 (M^++Na), found 160.0131

thiophene-2-carboxylic acid azide 2d:

R_f 0.5 (n-hexane/EtOAc 9:1); IR (KBr) υ_{max} = 2140 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 6.98 (1H, d, *J* = 6.8Hz), 7.21 (1H, d, *J* = 6.1 Hz), 7.25 (1H, m): ^{13}C NMR ($CDCl_3$, 100 MHz) δ 129.22, 130.15, 132.06, 142.52, 174.48; HRMS Calc'd for $C_5H_3N_3OS$ m/z: 175.9870 (M^++Na), found 175.9895

2-(1*H*-indol-3-yl) acetyl azide 2e:

R_f 0.4 (n-hexane/EtOAc 8:2); IR (KBr) υ_{max} = 2138 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 3.86 (2H, s), 7.02 (1H, d, *J* = 2.0 Hz), 7.05- 7.59 (4H, m), 8.84 (1H, s); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 31.84, 111.81, 111.93, 120.03, 122.50, 123.74, 123.89, 127.69, 136.69, 172.83; HRMS Calc'd for $C_{10}H_8N_4O$ m/z: 201.0776 (M^++H), found 201.0781

cinnamoyl azide 2f:

R_f 0.4 (n-hexane/EtOAc 8:2); IR (KBr) υ_{max} = 2137 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 6.60 (1H, d, *J* = 8.6 Hz), 7.12 (2H, d, *J* = 9.2 Hz), 7.25-7.55 (3H, m) 7.58 (1H, d, *J* = 8.8 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 125.08, 126.21, 126.52, 127.58, 133.39, 140.52, 181.83; HRMS Calc'd for $C_9H_7N_3O$ m/z: 174.0667 (M^++H), found 174.0632

phenyl acetyl azide 2g:

R_f 0.6 (n-hexane/EtOAc 9:1); IR (KBr) υ_{max} = 2135 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 3.98 (2H, s), 7.23 (1H, m), 7.28 (2H, d, *J* = 6.8 Hz), 7.34 (2H, m); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 38.18, 127.84, 129.31, 129.91, 141.33, 181.45; HRMS Calc'd for $C_8H_7N_3O$ m/z: 184.0487 (M^++Na), found 184.0472

***p*-nitro benzoyl azide 2h :**

R_f 0.4 (n-hexane/EtOAc 8:2); IR (KBr) υ_{max} = 2130 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 7.57 (2H, d, *J* = 7.3 Hz), 7.68 (2H, d, *J* = 7.1 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 120.49, 128.29, 144.27, 156.63, 179.55; HRMS Calc'd for $C_7H_4N_4O_3$ m/z: 215.0181 (M^++Na), found 215.0190

***p*-(Fmoc-amino)benzoyl azide 2i:**

R_f 0.3 (n-hexane/EtOAc 8:2); IR (KBr) υ_{max} = 2138 cm^{-1} ; ^1H NMR ($CDCl_3$, 400 MHz) δ 4.32 (1H, m), 4.51 (2H, d, *J* = 6.8 Hz), 7.25 -7.95 (12H, m); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 47.56, 67.05, 120.52, 125.20, 125.42, 127.60, 128.26, 136.27, 138.16, 141.83, 144.22, 156.08, 178.90; ES MS Calc'd for $C_{22}H_{16}N_4O_3$ m/z: 407.1 (M^++Na), found 407.1

(R)-(9H-fluoren-9-yl)methyl 1-azido-1-oxopropan-2-ylcarbamate {Fmoc-Ala-N3} 4a:

R_f 0.4 (n-hexane/EtOAc 9:1); IR (KBr) υ_{max} = 2148 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 1.45 (3H, s), 4.23-4.27 (1H, br), 4.28-4.48 (3H, br), 5.42 (1H, br), 7.26-7.79 (8H, m); ^{13}C

NMR ($CDCl_3$, 100 MHz) δ 18.64, 47.53, 51.95, 67.59, 120.54, 125.43, 127.62, 128.31, 141.82, 144.10, 150.65, 180.94; HRMS Calc'd for $C_{18}H_{16}N_4O_3$ m/z: 359.1120 (M^++Na), found 359.1126

(9H-fluoren-9-yl)methyl 1-azido-3-methyl-1-oxobutan-2-ylcarbamate{Fmoc-Val-N3} 4b:

R_f 0.4 (n-hexane/EtOAc 9:1); IR (KBr) υ_{max} = 2138 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 0.92 - 0.93 (6H, br), 2.11 (1H, m), 4.15-4.34 (2H, m), 4.35 (1H, d, *J* = 7.2 Hz), 4.41 (1H, d, *J* = 6.4 Hz), 5.15 (1H, m), 7.22-7.70 (8H, m); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 17.72, 31.36, 47.68, 61.23, 67.64, 125.44, 125.56, 127.60, 128.26, 141.84, 144.20, 156.80, 180.16; HRMS Calc'd for $C_{20}H_{20}N_4O_3$ m/z: 387.1433 (M^++Na), found 387.1423

(9H-fluoren-9-yl)methyl 1-azido-4-methyl-1-oxopentan-2-ylcarbamate{Fmoc-Leu-N3} 4c:

R_f 0.5 (n-hexane/EtOAc 9:1); IR (KBr) υ_{max} = 2137 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 0.85 (6H, br), 1.42-1.68 (3H, br), 4.11- 4.40, (4H, m), 5.05 (1H, br), 7.21-7.68 (8H, m); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 25.10, 25.30, 41.44, 45.78, 54.82, 67.54, 125.45, 125.55, 127.59, 128.25, 141.84, 144.16, 156.56, 181.13; HRMS Calc'd for $C_{21}H_{22}N_4O_3$ m/z: 401.1590 (M^++Na), found 401.1573

(9H-fluoren-9-yl)methyl 1-azido-4-methyl-1-oxopentan-2-ylcarbamate{Fmoc-Ile-N3} 4d:

R_f 0.5 (n-hexane/EtOAc 9:1); IR (KBr) υ_{max} =2150 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 0.91 (3H, m), 1.00–1.35 (5H, m), 1.92 (1H, m), 4.44 (2H, m), 4.51 (2H, d, *J* = 6.3 Hz), 7.25–7.77 (8H, m); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 11.63, 15.10, 25.70, 30.02, 47.58,

55.82, 67.09, 120.49, 125.76, 127.67, 128.29, 141.59, 144.27, 156.63, 180.09; HRMS Calc'd for $C_{21}H_{22}N_4O_3$ m/z: 401.1590 (M^++Na), found 401.1596

(9H-fluoren-9-yl)methyl 1-azido-1-oxo-3-phenylpropan-2-ylcarbamate{Fmoc-Phe-N3} 4e:

R_f 0.5 (n-hexane/EtOAc 9:1); IR (KBr) υ_{max} = 2145 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 3.75 (2H, s), 4.18-4.31 (3H, m), 4.48-4.54 (1H, br), 5.29 (1H, s), 7.28-7.78 (11 H, m); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 26.10, 47.53, 53.95, 68.45, 125.43, 125.78, 126.84, 127.63, 128.33, 129.15, 129.58, 136.70, 141.82, 144.10, 157.98, 179.18; HRMS Calc'd for $C_{24}H_{20}N_4O_3$ m/z: 451.1172 (M^++K), found 451.1615

azido(pyrrolidin-2-yl)methanone(9H-fluoren-9-yl)methyl2-azidocarbonyl)pyrrolidine-1 – carboxylate {Fmoc-Pro-N3} 4f:

R_f 0.4 (n-hexane/EtOAc 9:1); IR (KBr) υ_{max} =2142 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 1.42–1.95 (4H, m), 3.12 (2H, m), 4.26 (2H, m), 4.42 (2H, d, *J* = 5.9 Hz), 7.22–7.55 (8H, m); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 24.55, 26.12, 41.29, 43.68, 52.77, 66.25, 126.61, 127.82, 128.10, 129.35, 141.11, 144.58, 156.20, 178.95; HRMS Calc'd for $C_{20}H_{18}N_4O_3$ m/z: 385.1277 (M^++Na), found 385.1130

benzyl 1-azido-1-oxo-3-phenylpropan-2-ylcarbamate {Z-Phe-N3} 4g:

R_f 0.3 (n-hexane/EtOAc 9:1); IR (KBr) υ_{max} = 2133 cm^{-1} ; ^1H NMR ($CDCl_3$, 400 MHz) δ 3.01-3.18 (2H, m), 4.62 (1H, m), 5.09 (2H, s), 7.12-7.38 (10H, m); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 38.13, 57.22. 67.67, 128.07, 128.64, 128.79, 129.09, 129.32, 129.77, 135.93, 136.70 156.36, 179.81; HRMS Calc'd for $C_{17}H_{16}N_4O_3$ m/z: 347.1120 (M^++Na), found 347.1098

(S)-benzyl 4-(2-azido-2-oxoethyl)-5-oxooxazolidine-3-carboxylate4h {Z-Asp(oxa)-N_3} 4h:

R_f 0.4 (n-hexane/EtOAc 9:1); IR (KBr) υ_{max} = 2146.62 cm^{-1} ; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 5.14 (1H, s), 5.39-5.44 (1H, m), 5.58-5.67 (2H, m) 7.35 (m, 5H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 21.46, 55.60, 67.78, 79.02, 128.57, 128.93, 129.26, 136.92, 153.34, 172.63; ESMS Calc'd for $C_{13}H_{12}N_4O_5$ m/z: 327.0 (M^++Na), found 327.1

(S)-methyl2-(3-((S)-1-(((9H-fluoren-9-yl)methoxy)carbonyl)ethyl)ureido)-3-methylbutanoate {Fmoc-Ala-ψ[NHCONH]-Val-OMe} 5a: R_f 0.4 ($CHCl_3$/MeOH 9:1); IR (KBr) υ_{max} = 1642 cm^{-1} ; ^{1}H NMR (DMSO, 400 MHz) δ 0.79 (6H, br), 1.16-1.94 (4H, br), 3.55 (3H, s), 4.10-4.24 (2H, br), 4.51-4.82 (2H, br), 5.13 (1H, br), 6.31-6.37 (2H, br), 7.22-7.94 (8H, m); ^{13}C NMR (DMSO, 100 MHz) δ 18.34, 20.75, 31.28, 45.18, 51.35, 53.82, 59.74, 66.15, 126.53, 126.81, 127.12, 128.39, 141.12, 144.23, 156.02, 157.23, 174.12; HRMS Calc'd for $C_{24}H_{29}N_3O_5$ m/z: 462.2005 (M^++Na), found 462.2008

(S)-methyl2-(3-((S)-1-(((9H-fluoren-9-yl)methoxy)carbonyl)-2-phenylethyl)ureido)propanoate {Fmoc-Phe-ψ[NHCONH]-Ala-OMe 5b: R_f 0.4 ($CHCl_3$/MeOH 9:1); IR (KBr) υ_{max} = 1651cm^{-1} ; ^{1}H NMR (DMSO, 400 MHz) δ 0.83 (3H, s), 3.43 (2H, m), 3.49 (3H, s), 4.32 – 4.52 (2H, m), 4.61 (2H, d, J = 6.6 Hz), 5.05 (1H, br), 7.25–7.82 (13H, m) ; ^{13}C NMR (DMSO, 100 MHz) δ 19.08, 38.13, 47.54, 48.79, 52.43, 62.51, 66.31, 120.55, 125.88, 126.92, 127.71, 128.29, 128.81, 130.03, 138.42, 141.56, 144.51, 156.38, 157.10, 174.89; HRMS Calc'd for $C_{28}H_{29}N_3O_5$ m/z: 510.2005 (M^++Na), found 510.2010

(S)-methyl 2-(3-((S)-1-(tert-butoxycarbonyl)-2-methylpropyl)ureido)propanoate {Boc-Val-ψ[NHCONH]-Ala-OMe} 5c:

R_f 0.4 ($CHCl_3$/MeOH 9:1); IR (KBr) υ_{max} = 1641 cm^{-1} ; ^1H NMR (DMSO, 400 MHz) δ 0.84 (6H, d, *J* = 6.0 Hz) 1.25 (3H, d, *J* = 7.2 Hz), 1.36 (9H, s), 1.87 (1H, br), 3.64 (3H, s), 4.25 (1H, m), 4.76 (1H, br), 6.22 – 6.58 (2H, br); ^{13}C NMR (DMSO, 100 MHz) δ 17.69, 17.79, 27.66, 31.91, 47.55, 51.17, 62.53, 75.38, 154.56, 156.26, 173.66; HRMS Calc'd for $C_{14}H_{27}N_3O_5$ m/z: 340.1848 (M^++Na), found 340.1825

(S)-methyl2-(3-((S)-1-(tert-butoxycarbonyl)-3-methylbutyl)ureido)-3-methylbutanoate {Boc-Leu-*ψ*[NHCONH]-Val-OMe} 5d:

R_f 0.4 ($CHCl_3$/MeOH 9:1); IR (KBr) υ_{max} = 1649 cm^{-1} ;^1H NMR (DMSO, 400 MHz) δ 0.85 (12H, br), 1.38 (9H, s)**,** 1.55 (2H, br) 2.09 (2H, br), 3.63 (3H, s), 4.21 (1H, m), 4.32 (1H, m), 5.03 (1H, br), 6.41-6.53 (2H, m); ^{13}C NMR (DMSO, 100 MHz) δ 17.12, 17.88, 18.56, 27.15, 31.08, 48.11, 52.96, 55.14, 63.18, 76.31, 155.82, 156.33, 172.68; HRMS Calc'd for $C_{17}H_{33}N_3O_5$ m/z: 382.2318 (M^++Na), found 382.2313

Z-Ala-*ψ*[NH-CO-NH]-Gly-OMe, 5e:

IR (KBr) υ_{max} = 1641 cm^{-1} ; ^1H NMR ($CDCl_3$ 400 MHz) δ 1.38 (3H, *J* = 6.7 Hz, d) 3.55 (3H, s), 4.61 (2H, *J* = 7.0 Hz, d), 4.99 (2H, s), 5.56 (1H, m), 7.22 (m, 5H); ^{13}C NMR (DMSO, 100 MHz) δ 20.8, 43.2, 50.6, 48.9, 69.9, 126.8, 127.1, 128.5, 136.8, 155.1, 156.8, 171.9; ESMS Calc'd for $C_{14}H_{19}N_3O_5$ m/z: 332.1 (M^++Na), found 332.1

(S)-methyl2-(3-((R)-1-((S)-2-(((9H-fluoren-9-yl)methoxy)carbonyl)-3-methylbutanamido)ethyl)ureido)-4-methylpentanoate {Fmoc-Val-Ala-*ψ*[NHCONH]-Leu-OMe} 5f:

R_f 0.4 ($CHCl_3$/MeOH9:1); IR (KBr) υ_{max} = 1652 cm^{-1} ; ^1H NMR (DMSO, 400 MHz) δ 0.81 (6H, br), 1.08 (6H, br), 1.18 (2H, m), 2.52 – 2.63 (2H, br), 3.53 (3H, s), 4.23 (1H, m) 4.41–4.52 (2H, m), 4.62 (2H, d, *J* = 7.0 Hz) , 5.09 (1H, m), 6.32 (1H, br), 7.21 – 7.89 (8H,

m); ^{13}C NMR (DMSO, 100 MHz) δ 18.99, 20.20, 24.18, 25.30, 31.00, 35.00, 47.55, 55.34, 56.17, 61.03, 66.60, 126.27, 128.02, 128.60, 129.88, 141.58, 144.76, 156.30, 157.10, 171.35, 174.39; HRMS Calc'd for $C_{30}H_{40}N_4O_6$ m/z: 575.2846 (M^++Na), found 575.2853

(S)-(9H-fluoren-9-yl)methyl 2-(((R)-1-(3-((S)-1-methoxy-3-methyl-1-oxobutan-2-yl)ureido)-2-methylpropyl)carbamoyl)pyrrolidine-1-carboxylate {Fmoc-Pro-Ala-*ψ*[NHCONH]-Val-OMe 5g:

R_f 0.4 ($CHCl_3$/MeOH9:1); IR (KBr) υ_{max} = 1648 cm^{-1} ; ^{1}H NMR (DMSO, 400 MHz) δ 0.84 (6H, m), 1.33 (3H, d, *J* = 6.0 Hz), 1.85-2.00 (5H, br), 3.34 (3H, s), 3.43 (1H, m), 3.56-3.64 (2H, m), 4.17-4.28 (5H, br), 5.45 (1H, br), 6.47 (2H, m), 7.27-7.77 (8H, m); ^{13}C NMR (DMSO, 100 MHz) δ 18.39, 20.12, 24.82, 26.09, 31.93, 41.89, 46.11, 52.17, 54.98, 56.81, 64.22, 66.18, 124.39, 126.05, 126.52, 128.39, 141.23, 144.12, 156.55, 157.20, 171.58, 173.85; HRMS Calc'd for $C_{29}H_{36}N_4O_6$ m/z: 559.2533 (M^++Na), found 559.2539

1-(1*H*-indol-3-yl)methyl)-3-p-tolylurea 5h:

R_f 0.4 (Hexane/ EtOAc 5:5); IR (KBr) υ_{max} = 1644 cm^{-1}; ^{1}H NMR (DMSO, 400 MHz) δ 2.19 (3H, s), 4.75 (2H, d, *J* = 4.8Hz), 6.71-6.80 (2H, m), 6.98 (1H, d, *J* = 6.4 Hz), 7.02 – 7.65 (8H, m); ^{13}C NMR (DMSO, 100 MHz) δ 21.84, 35.93, 117.42, 117.95, 119.23, 119.46, 120.28, 121.57, 122.81, 123.61, 129.38, 136.86, 139.18, 139.48, 154.61;HRMS Calc'd for $C_{17}H_{17}N_3O$ m/z: 280.1450 (M^++H), found 280.1445

1-(3-bromophenyl)-3-(thiophen-2-yl)urea 5i:

R_f 0.6 (Hexane/ EtOAc 7:3); IR (KBr) υ_{max} =1647 cm^{-1} ; ^{1}H NMR (DMSO, 400 MHz) δ 6.05 (1H, br), 6.91 (1H, d, *J* = 6.3 Hz), 6.98 (1H, m), 7.20 (1H, d, *J* = 6.4 Hz), 7.32 (2H, d, *J* = 6.5 Hz), 7.38 (2H, d, *J* = 6.6 Hz); ^{13}C NMR (DMSO, 100 MHz) δ 114.88, 117.03,

123.67, 125.18, 125.78, 133.33, 138.52, 141.36, 156.28; HRMS Calc'd for $C_{11}H_9BrN_2OS$ m/z: 320.9435 (M^++Na), found 320.9506

3-benzyl-1,1-diethylurea 5j:

R_f 0.6 (Hexane/ EtOAc 8:2); IR (KBr) υ_{max} =1646 cm^{-1} ; ^1H NMR ($CDCl_3$, 400 MHz) δ 1.12 (6H, t, *J* = 6.8 Hz), 3.26 (4H, m), 4.41 (2H, d, *J* = 5.6Hz), 4.84 (1H, br), 7.22 – 7.32 (5H, m) ;^{13}C NMR ($CDCl_3$, 100 MHz) δ 14.38, 41.66, 45.29, 127.54, 129.00, 129.70, 140.49, 157.74; HRMS Calc'd for $C_{12}H_{18}N_2O$ m/z: 229.1317 (M^++Na), found 229.1321

1-benzyl-3-(2-chlorophenyl)urea 5k:

R_f 0.5 (Hexane/ EtOAc 7:3); IR (KBr) υ_{max} =1651 cm^{-1} ; ^1H NMR ($CDCl_3$, 400 MHz) δ 4.32 (2H, d, *J* = 5.6 Hz), 5.18 (1H, s), 7.12 – 7.30 (9H, m) ;^{13}C NMR ($CDCl_3$, 100 MHz) δ 41.93, 122.70, 123.37, 125.85, 127.56, 128.76, 129.07, 129.57, 129.77, 134.71, 138.34, 155.63; HRMS Calc'd for $C_{14}H_{13}ClN_2O$ m/z: 261.0794 (M^++H), found 261.0797

1-(3,5-bis(trifluoromethyl)phenyl)-3-benzylurea 5l:

R_f 0.4 (Hexane/ EtOAc 5:5); IR (KBr) υ_{max} =1652 cm^{-1} ; ^1H NMR (DMSO, 400 MHz) δ 4.47 (2H, d, *J* = 5.6 Hz), 6.62 (1H, br), 7.26 -7.37 (5H, m), 7.50 (1H, s), 7.86 (2H, s); ^{13}C NMR (DMSO, 100 MHz) δ 42.21, 120.09, 126.11, 127.91, 128.87, 129.12, 130.64, 133.38, 135.17, 139.32, 156.25; HRMS Calc'd for $C_{16}H_{12}F_6N_2O$ m/z: 363.0932 (M^++H), found 363.0943

1-(4-nitrophenyl)-3-*p*-tolylurea 5m:

R_f 0.4 (Hexane/ EtOAc 7:3); IR (KBr) υ_{max} =1650 cm^{-1} ; ^1H NMR (DMSO, 400 MHz) δ 2.20 (3H, s), 6.70 (1H, d, *J* = 6.0 Hz), 7.04 (2H, d, *J* = 6.8 Hz), 7.30 (2H, d, *J* = 6.8 Hz), 7.52 (2H, d, *J* = 6.4 Hz), 7.64 (2H, d, *J* = 6.8 Hz); ^{13}C NMR (DMSO, 100 MHz) δ 22.04,

123.90, 124.51, 129.20, 130.02, 138.93, 139.56, 141.70, 148.95, 153.24; HRMS Calc'd for $C_{14}H_{13}N_3O_3$ m/z 294.0855 (M^++Na), found 294.0852

1-(4-Fmoc-aminophenyl)-3-cyclohexylurea 5n:

R_f 0.4 (Hexane/ EtOAc 7:3); IR (KBr) υ_{max} = 1644 cm^{-1} ; ^{1}H NMR (DMSO, 400 MHz) δ 1.33 – 1.92 (10H, m), 3.32 (1H, m), 4.42 (1H, m), 4.63 (2H, d, *J* = 6.9 Hz), 7.21-7.86 (12H, m); ^{13}C NMR (DMSO, 100 MHz) δ 22.27, 23.44, 25.80, 47.08, 49.92, 66.80, 120.49, 125.20, 125.40, 125.53, 127.59, 128.24, 136.14, 137.21, 141.83, 144.24, 156.08, 156.19, HRMS Calc'd for $C_{28}H_{29}N_3O_3$ m/z: 478.2107 (M^++Na), found 478.2114

Boc-Phe-ψ[NHCONH]-(*R*)-1-phenylethylamine 7a:

m.p. = 145 °C; R_f 0.5 ($CHCl_3$/MeOH 9:1); IR (KBr) υ_{max} = 1645 cm^{-1} ; ^{1}H NMR (DMSO, 400 MHz) δ 1.28 (3H, d, *J* = 6.96 Hz) 1.32 (9H, s), 2.84 (2H, m), 4.70 (1H, m), 5.10 (1H, m), 6.17 (1H, br), 6.52 (1H br) 7.18-7.28 (10H, m); HRMS Calc'd for $C_{22}H_{29}N_3O_3$ m/z: 406.2107 (M^++Na), found 406.2098

Boc-Phe-ψ[NHCONH]-(*S*)-1-phenylethylamine 7b:

m.p. = 158 °C; R_f 0.5 ($CHCl_3$/MeOH 9:1); IR (KBr) υ_{max} =1646 cm^{-1} ; ^{1}H NMR (DMSO, 400 MHz) δ 1.25 (3H, d, *J* = 6.96 Hz)) 1.31 (9H, s), 2.85 (2H, m), 4.69 (1H, m), 5.07 (1H, m), 6.17 (1H, br), 6.52 (1H br) 7.17-7.31 (10H, m); ESMS calcd. for $C_{22}H_{29}N_3O_3$ m/z: 406.2 (M^++Na), found 406.1

Fmoc-Gly-ψ[NH-CO-OMe] 6a :

R_f 0.3 (n-hexane/EtOAc 6:4); IR (KBr) υ_{max} = 1742 cm^{-1} ; ^{1}H NMR (DMSO, 400 MHz) δ 3.38 (3H, s), 4.28-4.34 (3H, m), 4.44 (1H, t, *J* = 5.60 Hz), 7.28-7.83 (8H, m) ; ^{13}C NMR

(DMSO, 100 MHz) δ 42.70, 47.19, 61.56, 67.96, 120.51, 122.41, 123.00, 124.18, 136.31, 139.11, 155.51, 156.44; HRMS Calc'd for $C_{18}H_{18}N_2O_4$ m/z: 349.1164 (M^++Na), found 349.1162

Fmoc-Leu-ψ[NH-CO-OMe] 6b:

R_f 0.4 (n-hexane/EtOAc 6:4); IR (KBr) υ_{max} = 1742 cm^{-1}; ^1H NMR (DMSO, 400 MHz) δ 0.91 (6H, d, *J* = 5.6Hz), 1.57-1.64 (3H, br), 4.18 (1H, d, *J* = 6.00 Hz), 4.27-4.37 (2H, m), 5.19 (1H, br), 7.16-7.88 (8H, m); ^{13}C NMR (DMSO, 100 MHz) δ 21.59, 23.70, 42.85, 46.27, 50.68, 57.53, 64.93, 120.78, 124.40, 126.26, 126.85, 140.20, 143.13, 154.55, 155.13; HRMS Calc'd for $C_{22}H_{26}N_2O_4$ m/z: 405.7190 (M^++Na), found 405.1793

Fmoc-Phe-ψ[NH-CO-OMe] 6c:

R_f 0.5 (n-hexane/EtOAc 6:4); IR (KBr) υ_{max} = 1746 cm^{-1} ; ^1H NMR (DMSO, 400 MHz) δ 3.30 (2H, br), 3.71 (3H, s), 4.36 – 4.52 (3H, br), 5.06 (2H, br), 7.32 – 7.77 (13H, m); ^{13}C NMR (DMSO, 100 MHz) δ 46.18, 47.42, 47.56, 58.06, 66.63, 121.98, 125.33, 126.09, 126.12, 127.61, 128.80, 129.15, 136.72, 141.11, 143.27, 155.48, 156.01; HRMS Calc'd for $C_{25}H_{24}N_2O_4$ m/z: 439.1634 (M^++Na), found 439.1620

Cbz-Val-ψ[NH-CO-OMe] 6d:

R_f 0.4 (n-hexane/EtOAc 6:4); IR (KBr) υ_{max} = 1748 cm^{-1}; ^1H NMR (DMSO, 400 MHz) δ 0.93 – 1.02 (6H, m), 2.35 – 2.48 (1H, br), 3.42 (3H, s), 4.90 (1H, d, *J* = 8.4 Hz), 5.37 (2H, s), 7.31 – 7.75 (5H, m); ^{13}C NMR (DMSO, 100 MHz) δ 17.17, 32.11, 51.23, 61.17, 63.28, 124.09, 124.91, 126.21, 136.18, 153.23, 154.89; HRMS Calc'd for $C_{14}H_{20}N_2O_4$ m/z: 303.1433(M^++Na), found 303.1341

Cbz-Met-ψ[NH-CO-OMe] 6e:

R_f 0.3 (n-hexane/EtOAc 6:4); IR (KBr) υ_{max} = 1732 cm^{-1}; ^1H NMR (DMSO, 400 MHz) δ 2.04 (3H, s), 2.35 (2H, br), 2.45 (2H, t, *J* = 6.4 Hz), 3.75 (3H, s), 5.11 (2H, s), 5.17 (1H, br), 7.33 (5H, br); ^{13}C NMR (DMSO, 100 MHz) δ 10.26, 46.64, 54.17, 60.93, 63.43, 123.08, 123.54, 125.03, 132.09, 150.47, 151.06; HRMS Calc'd for $C_{14}H_{20}N_2O_4S$ m/z: 335.1041 (M^++Na), found 335.1039

ethyl thiophen-2-ylcarbamate 6f:

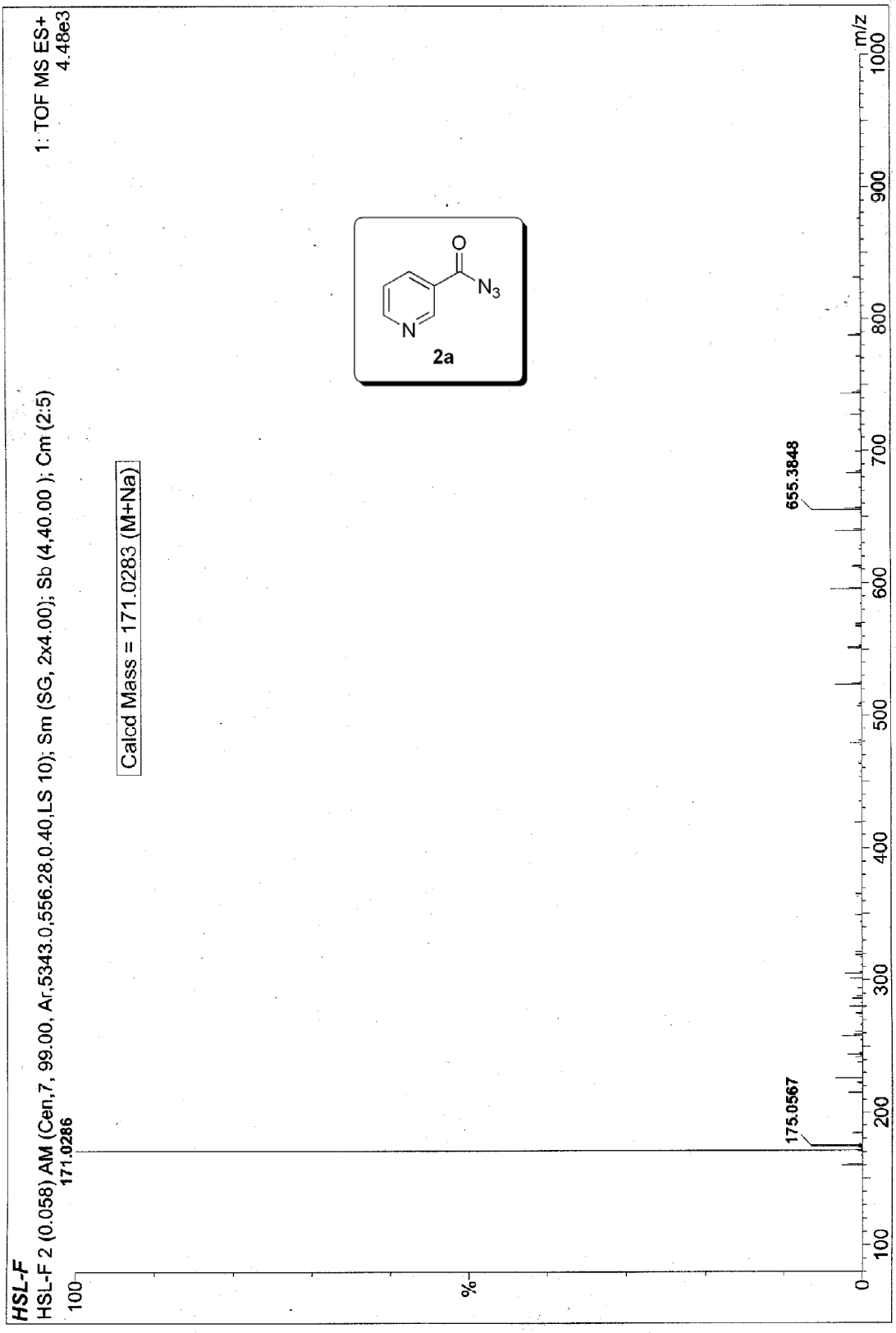
R_f 0.4 (n-hexane/EtOAc 7:3); IR (KBr) υ_{max} = 1728 cm^{-1} ; ^1H NMR (DMSO, 400 MHz) δ 1.25 (3H, t, *J* = 7.24 Hz), 4.21 (2H, q, *J* = 6.8 Hz), 6.60 – 6.78 (3H, m), 8.10 (1H, s); ^{13}C NMR (DMSO, 100 MHz) δ 14.98, 62.37, 112.80, 117.68, 125.15, 140.69, 154.59, HRMS Calc'd for $C_7H_9NO_2S$ m/z: 172.0432 (M^++H), found 172.0428

ethyl 4-nitrophenylcarbamate 6g:

R_f 0.4 (n-hexane/EtOAc 7:3); IR (KBr) υ_{max} = 1732 cm^{-1} ; ^1H NMR (DMSO, 400 MHz) δ 1.31 (3H, t, *J* = 7.2 Hz), 4.26 (2H, q, *J* = 7.2 Hz), 7.43 – 7.89 (4H, m), 8.38 (1H, s); ^{13}C NMR (DMSO, 100 MHz) δ 14.88, 62.31, 124.81, 130.25, 135.66, 139.92, 154.12; HRMS Calc'd for $C_9H_{10}N_2O_4$ m/z: 233.0538 (M^++Na), found 233.0542

References for known ureas and carbamates:

5c: V. V Sureshbabu and N. S. Sudarshan, *Synthetic Commun*., 2008, **38***,* 2168-2184; **5e**: N. Narendra, G. Chenankrishnareddy and V. V. Sureshbabu, *Org. Biomol. Chem*., 2009**,** DOI:10: 1039/b905790k; **5f**: *Indian J. Chem.,* 2008, **47B**, 910-919; **5j**: M. Shen, C. Beguin, A. Golbraikh, J. P. Stables, H. Kohn and A. Tropsha, *J. Med. Chem*., 2004**, 47**, 2356-2364; **5k**: V. Devries, J. D. Bloom, M. D. Dutia, A. S. Katocs and E. E. Largis, *J. Med. Chem.,* 1989**, 32***,* 2318-2325; **5n**: *Chem. Pharm. Bull.,* 1986, **34**, 1950-1960; **6g:** Y. Lu and R. T Taylor, *Tetrahedron Let.,* 2003, 9267, 9269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900
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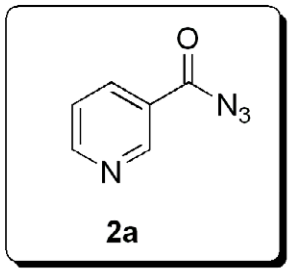
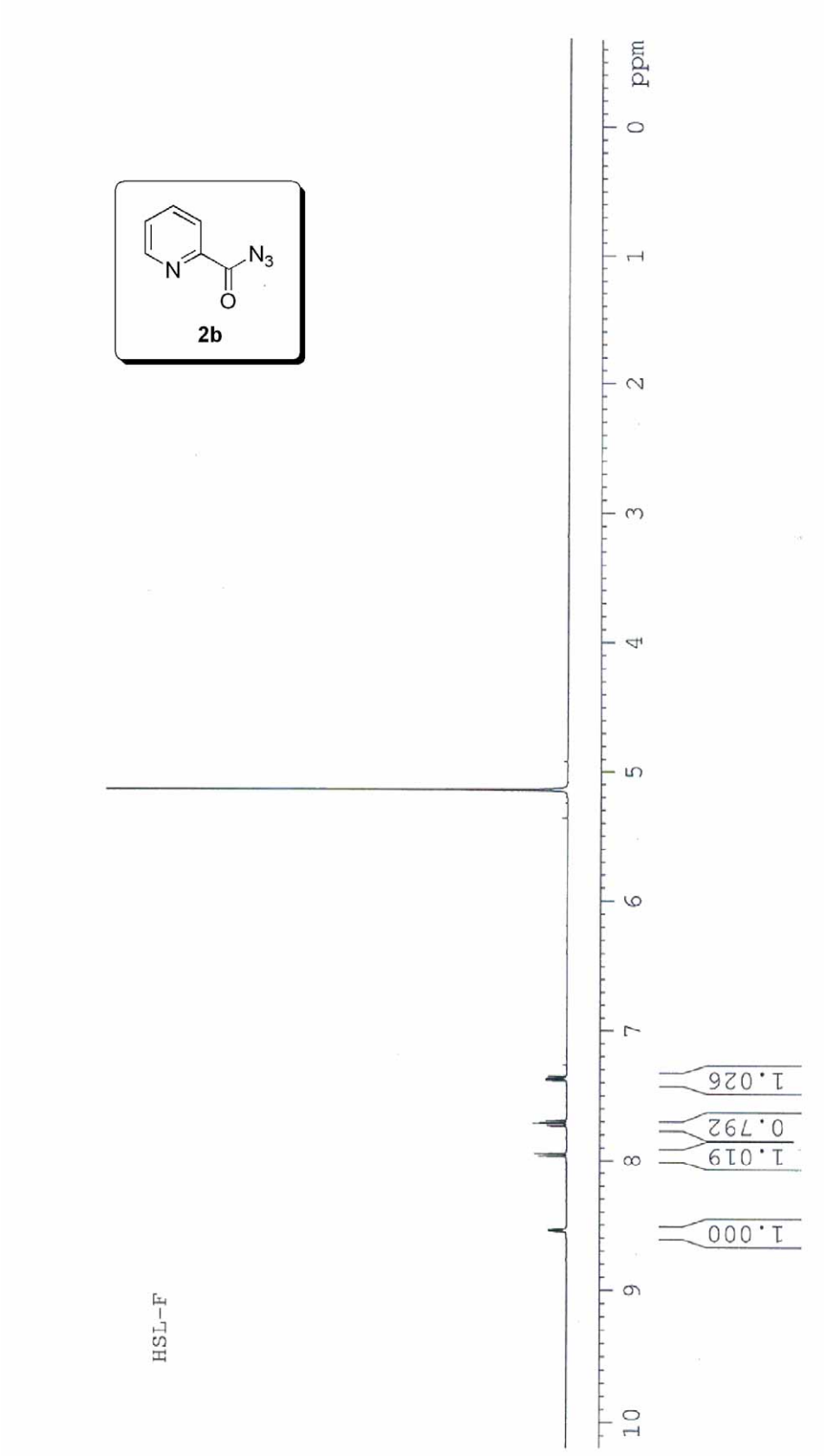


2a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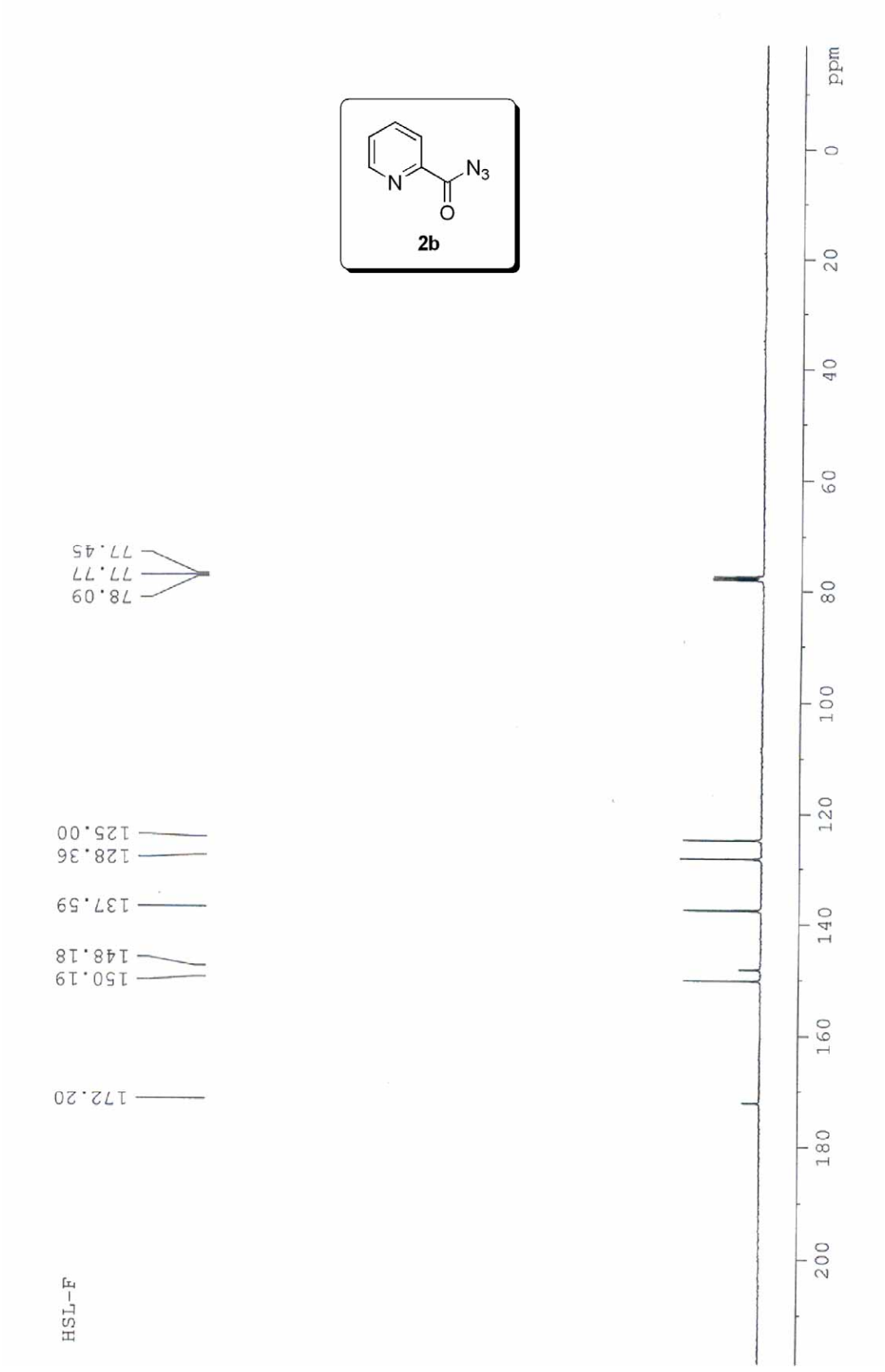
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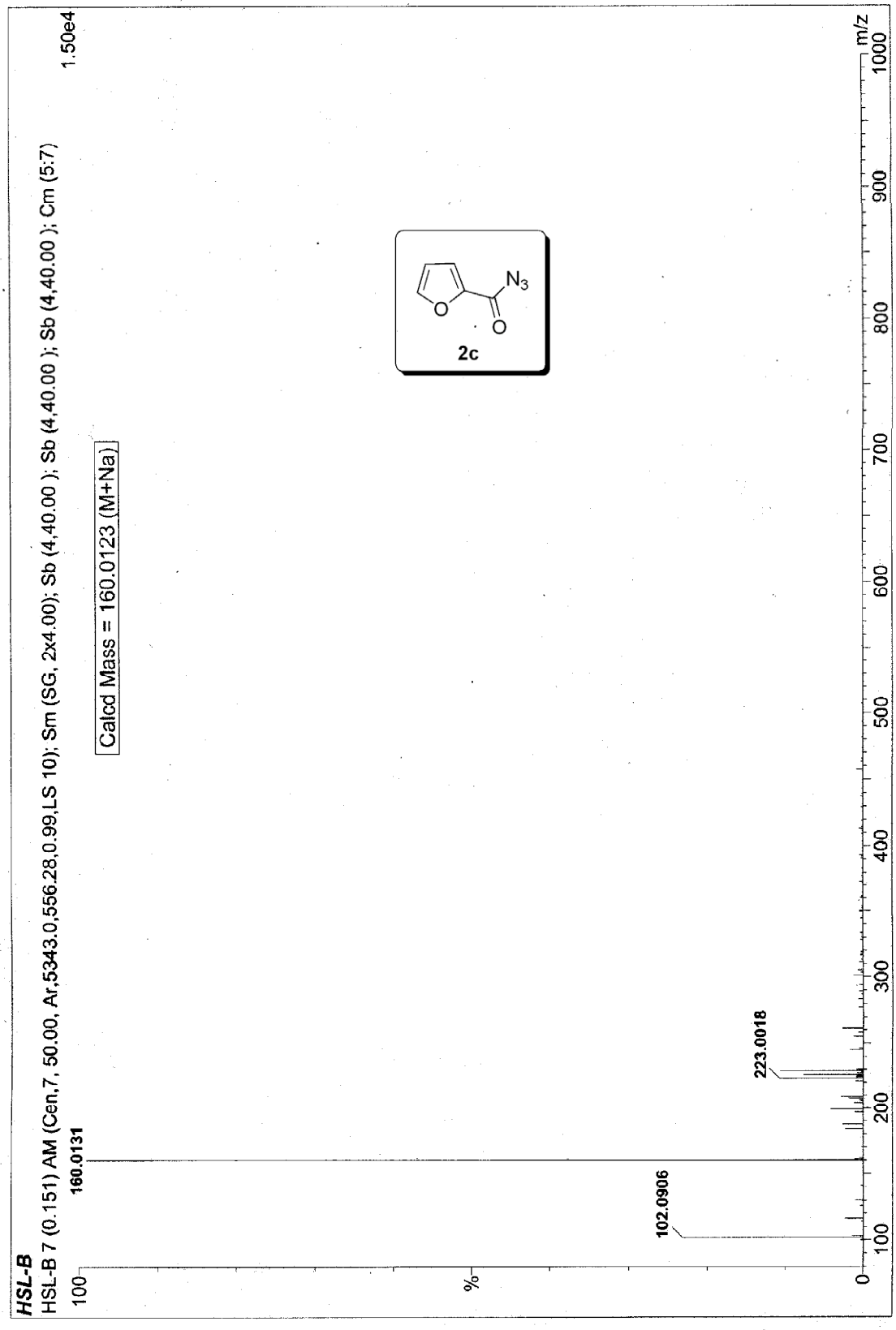
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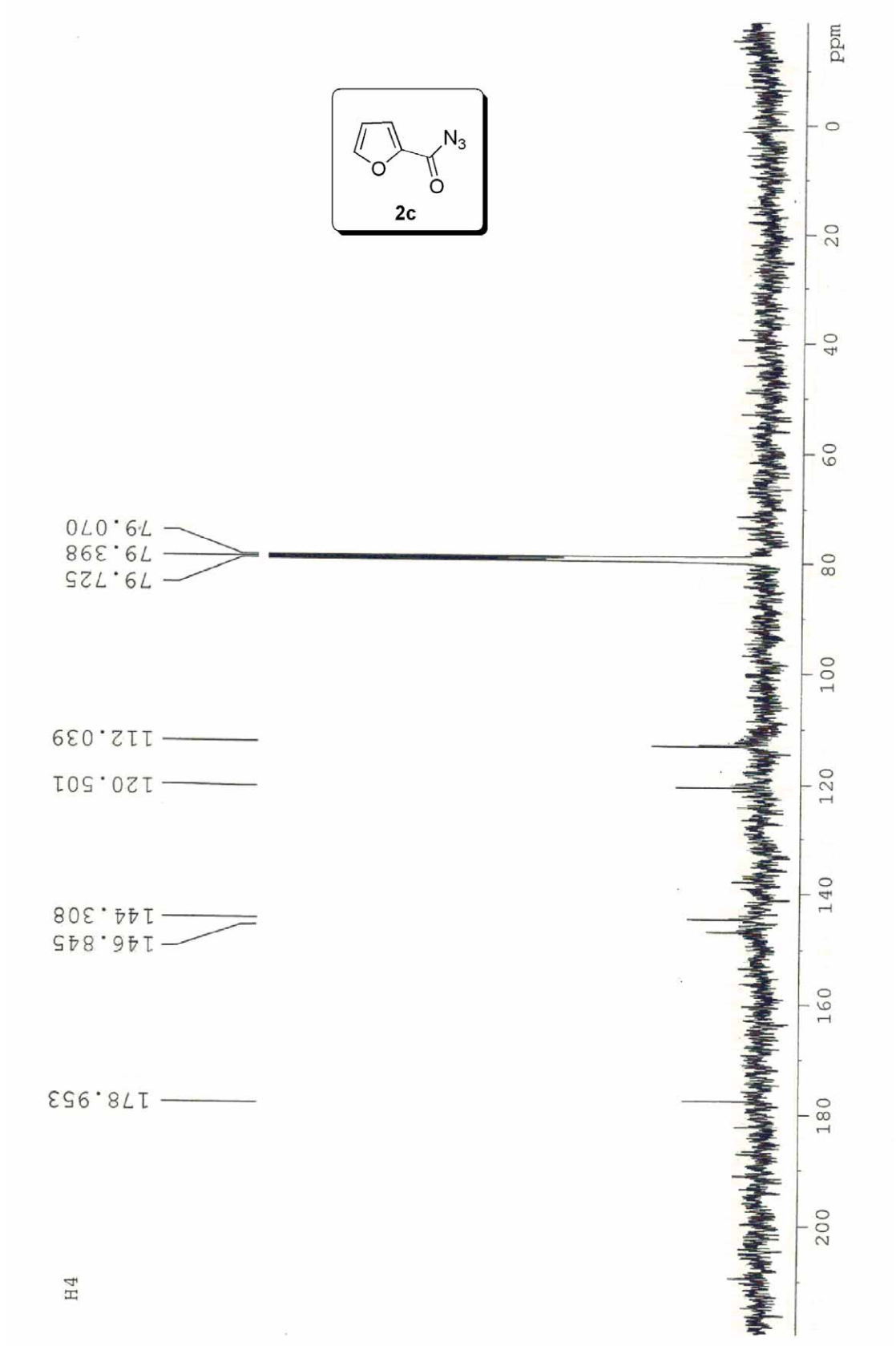
m/z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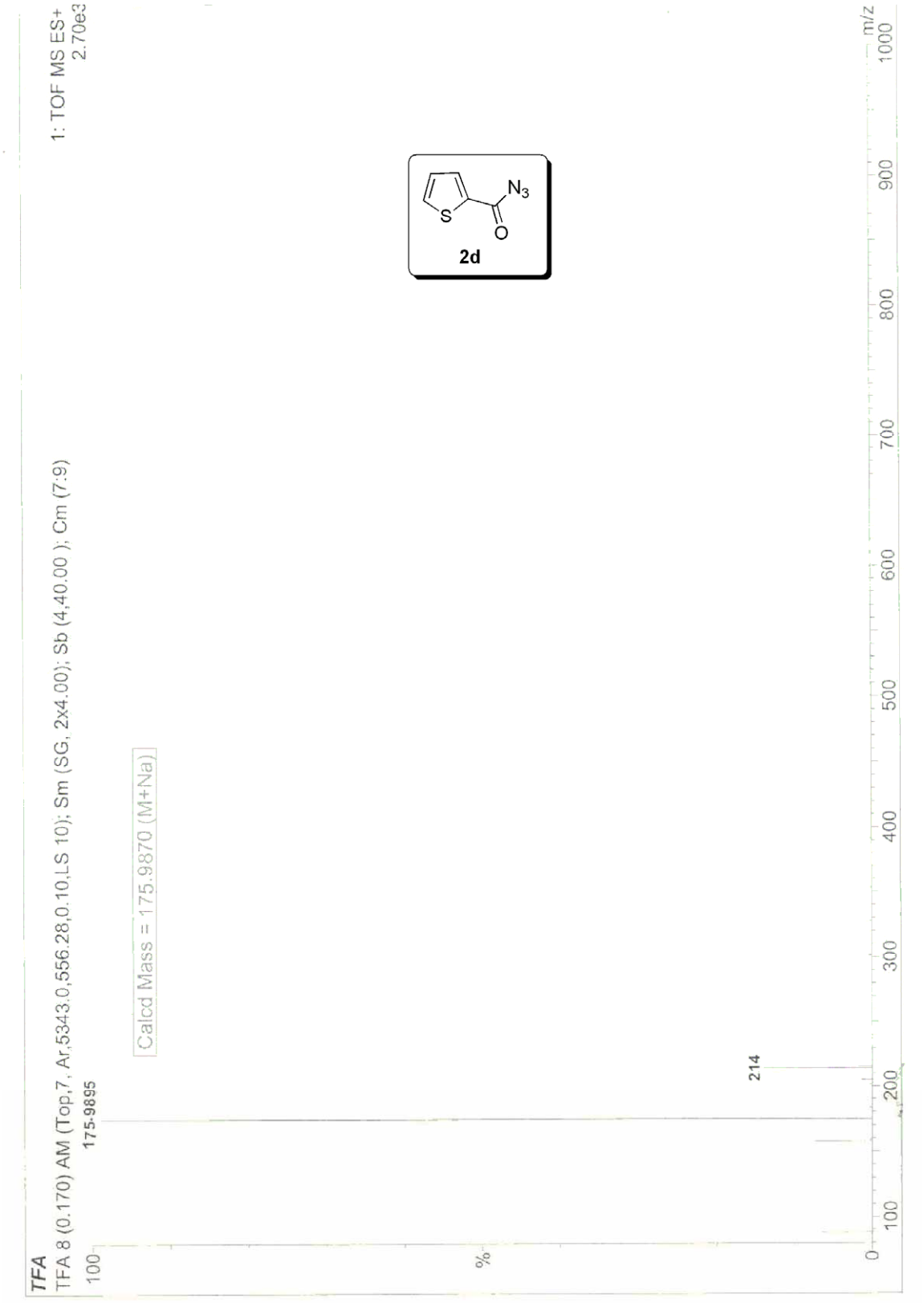
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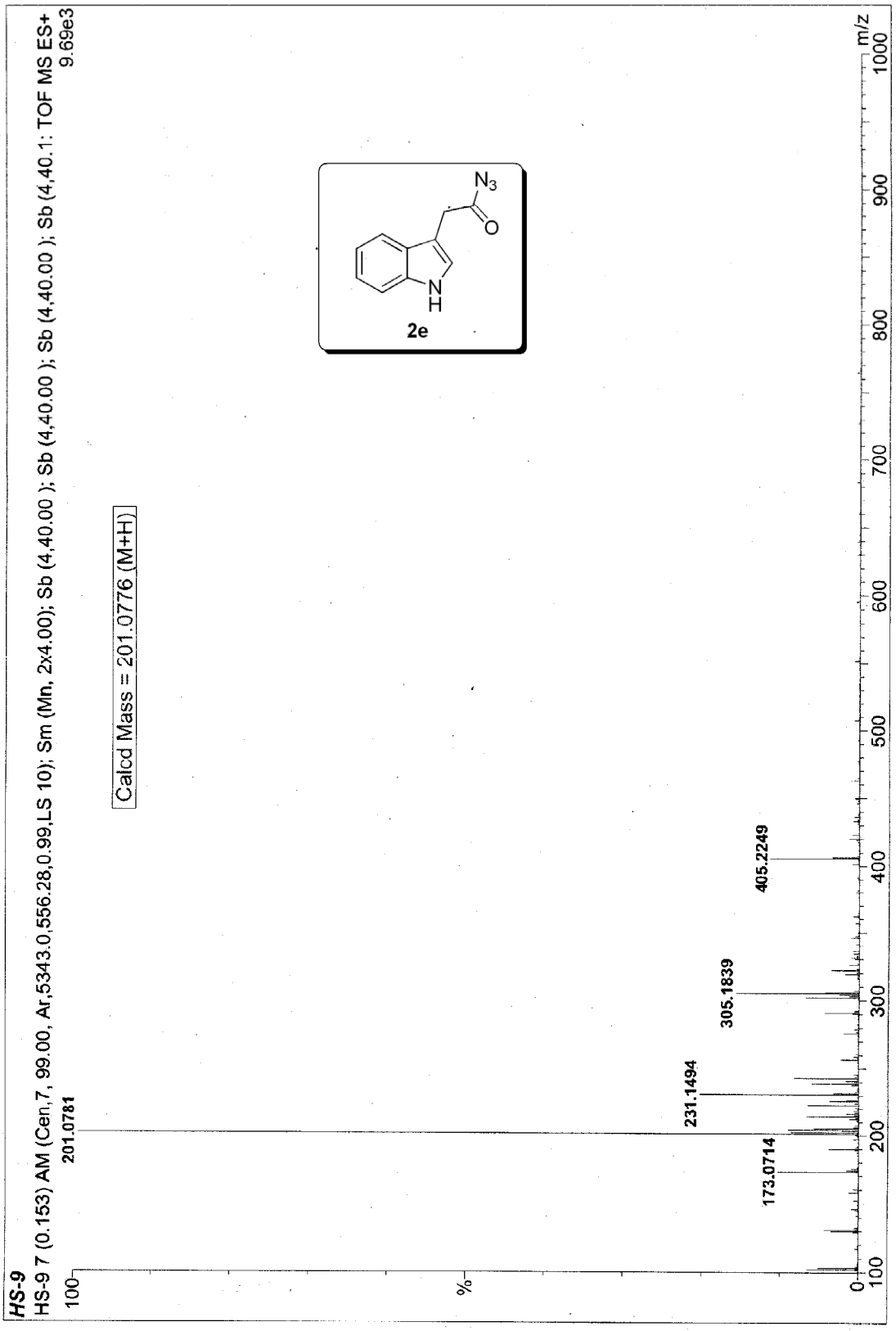
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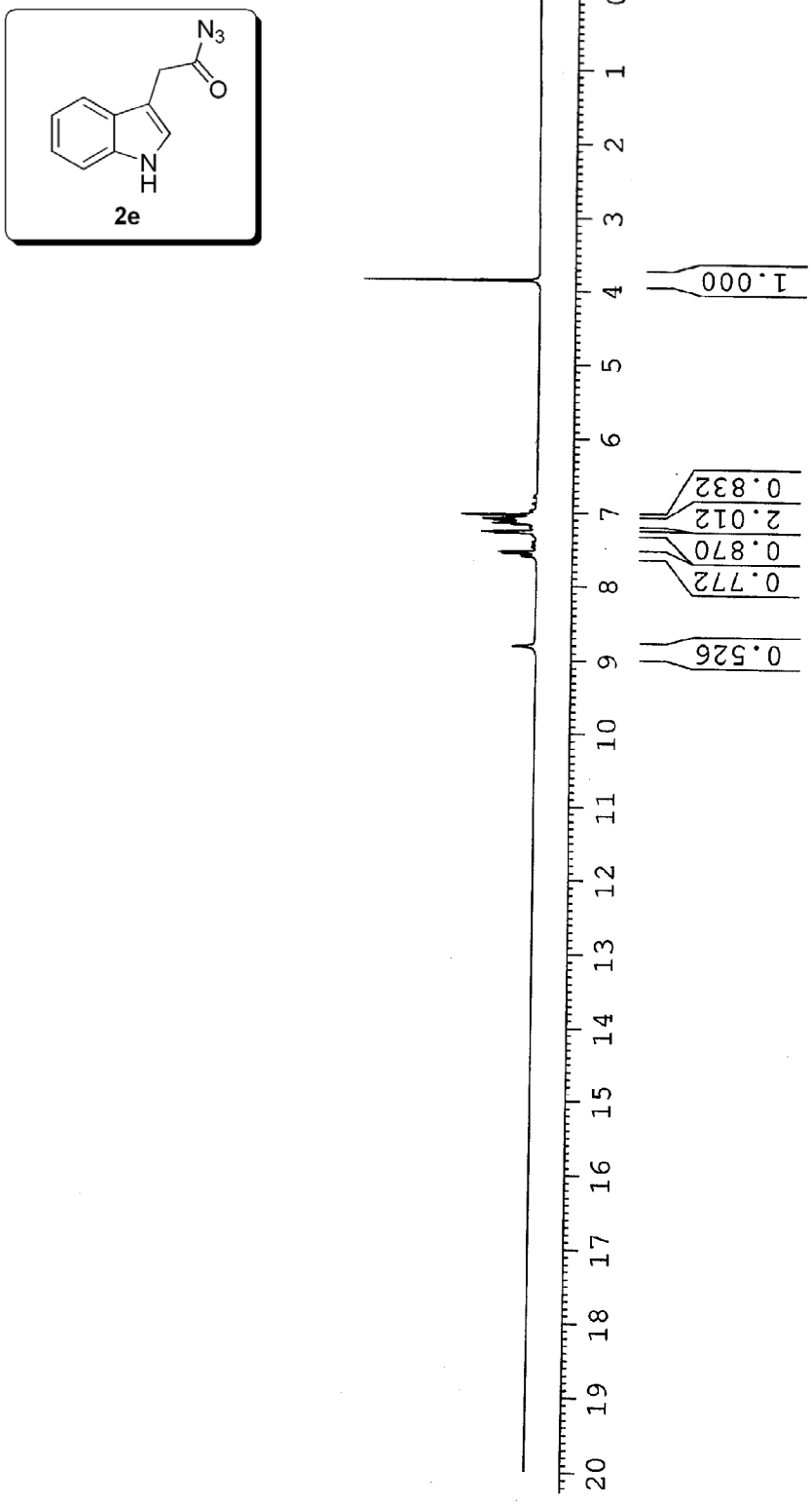
%
m/z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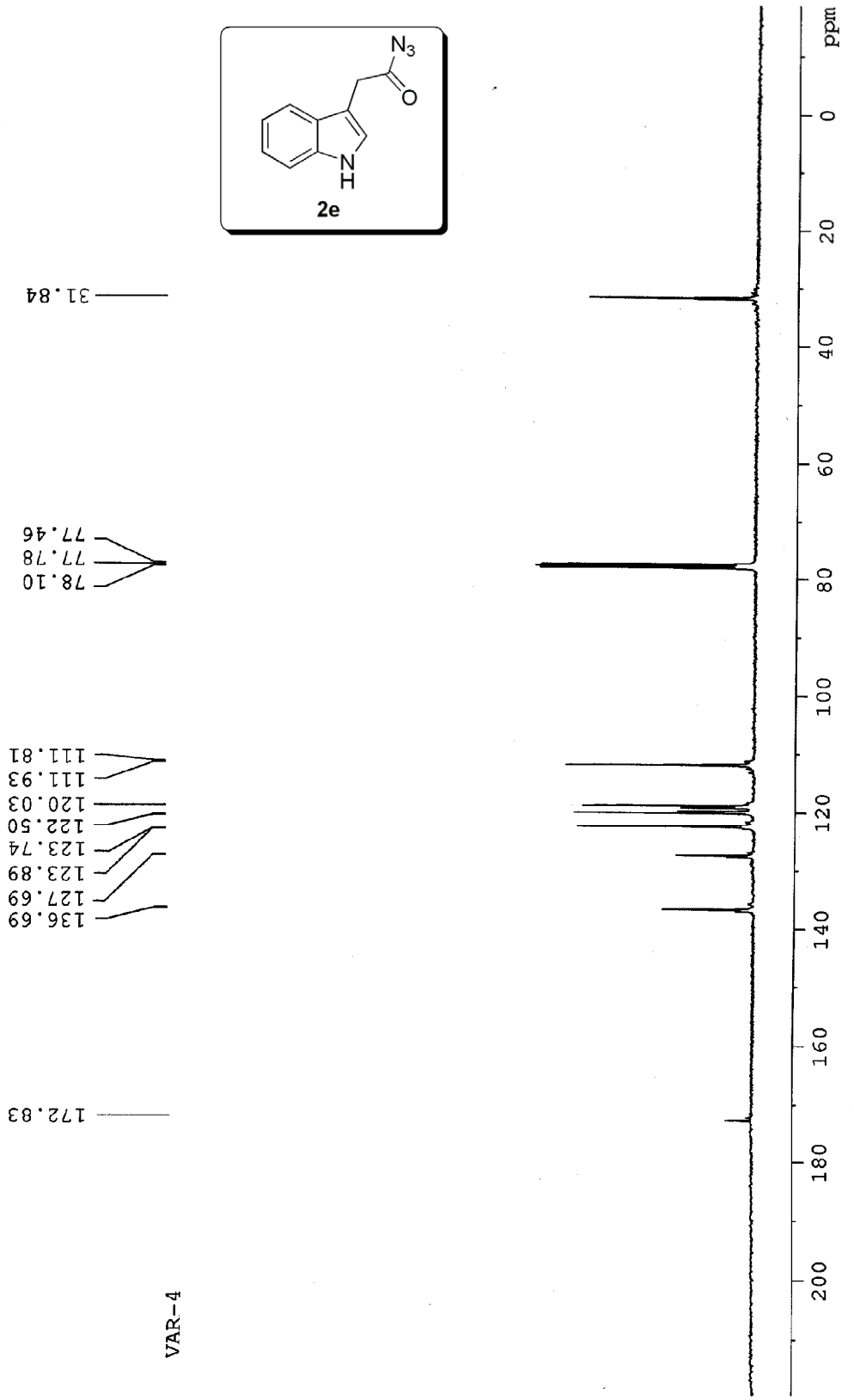
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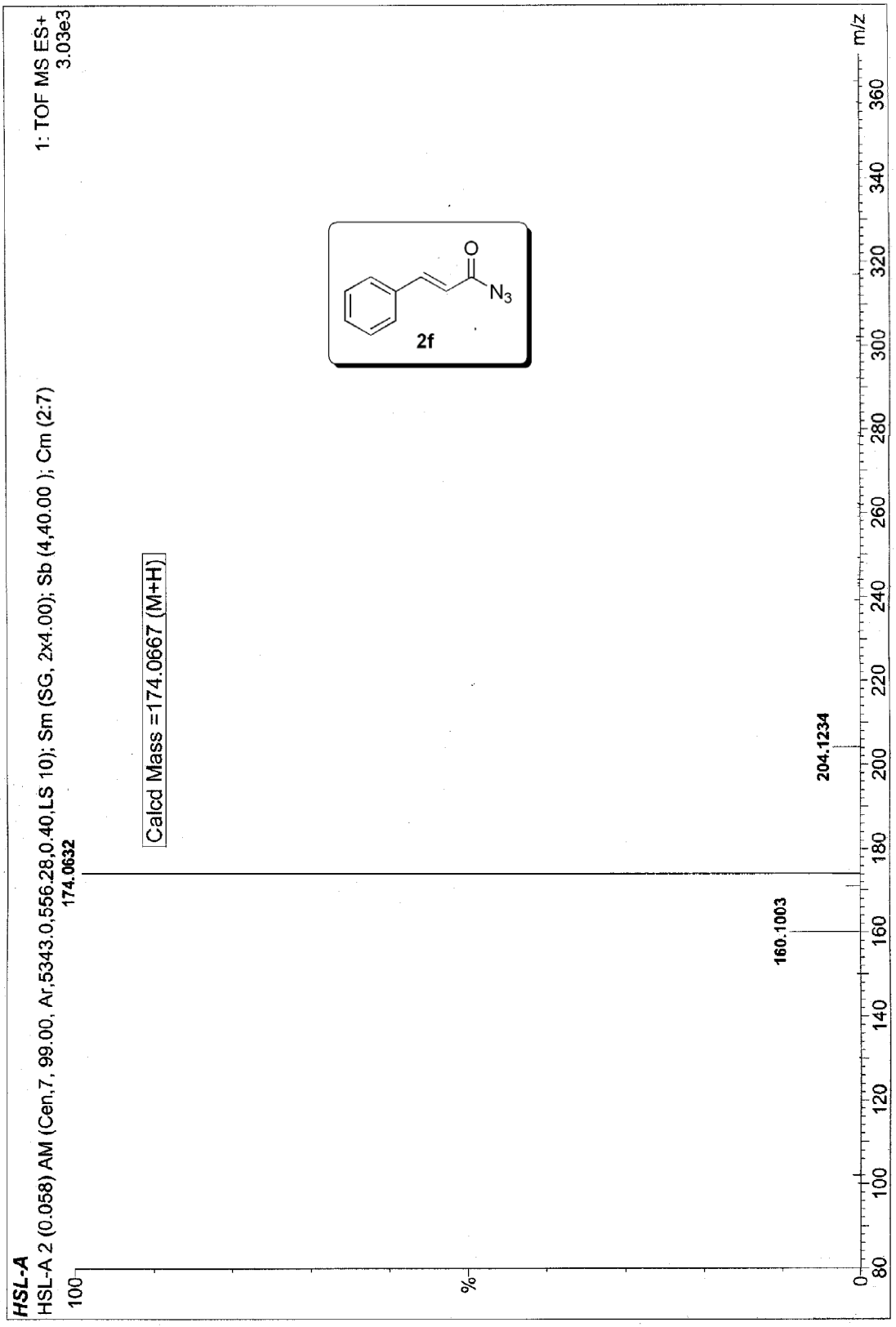
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%
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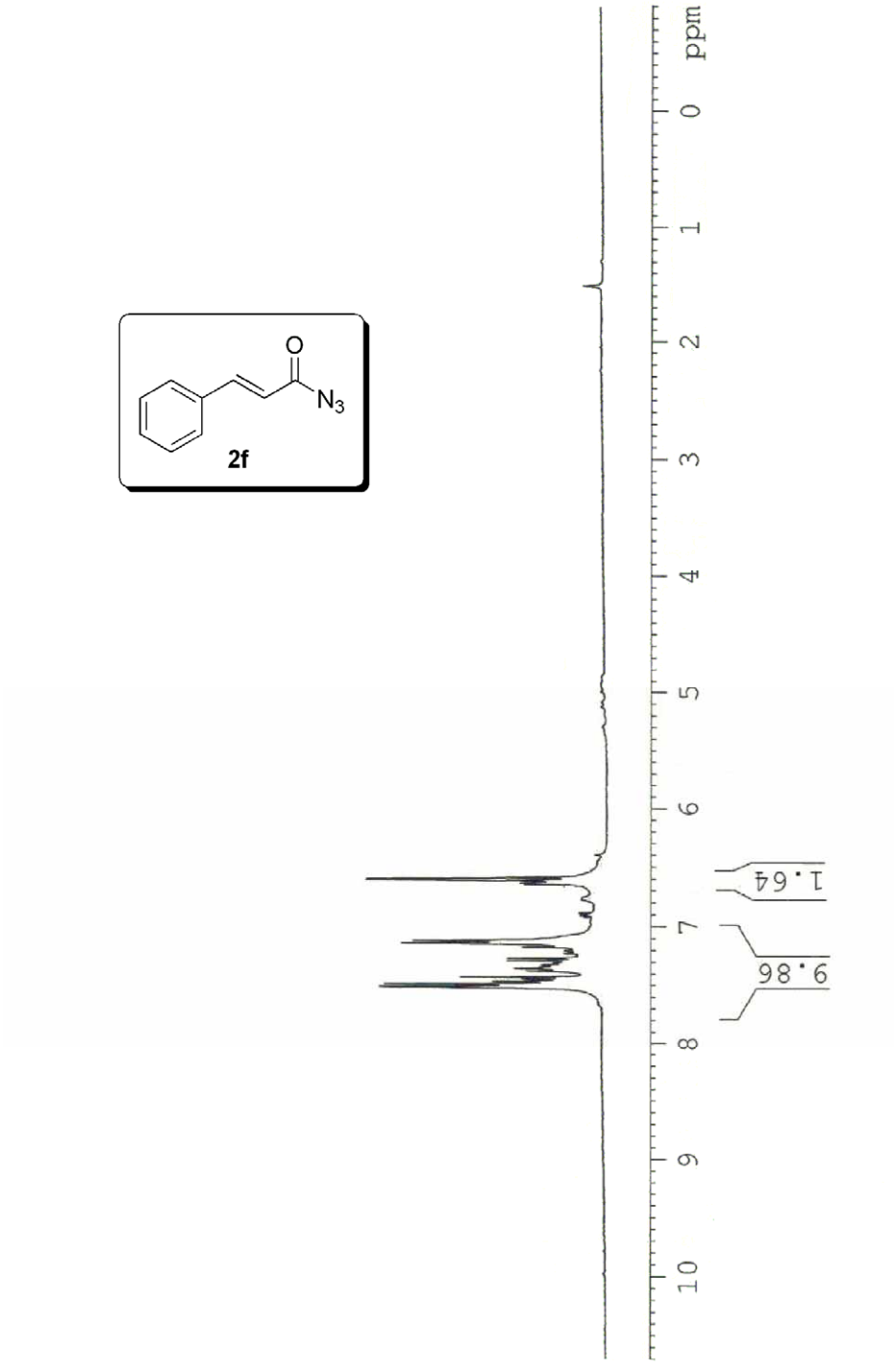
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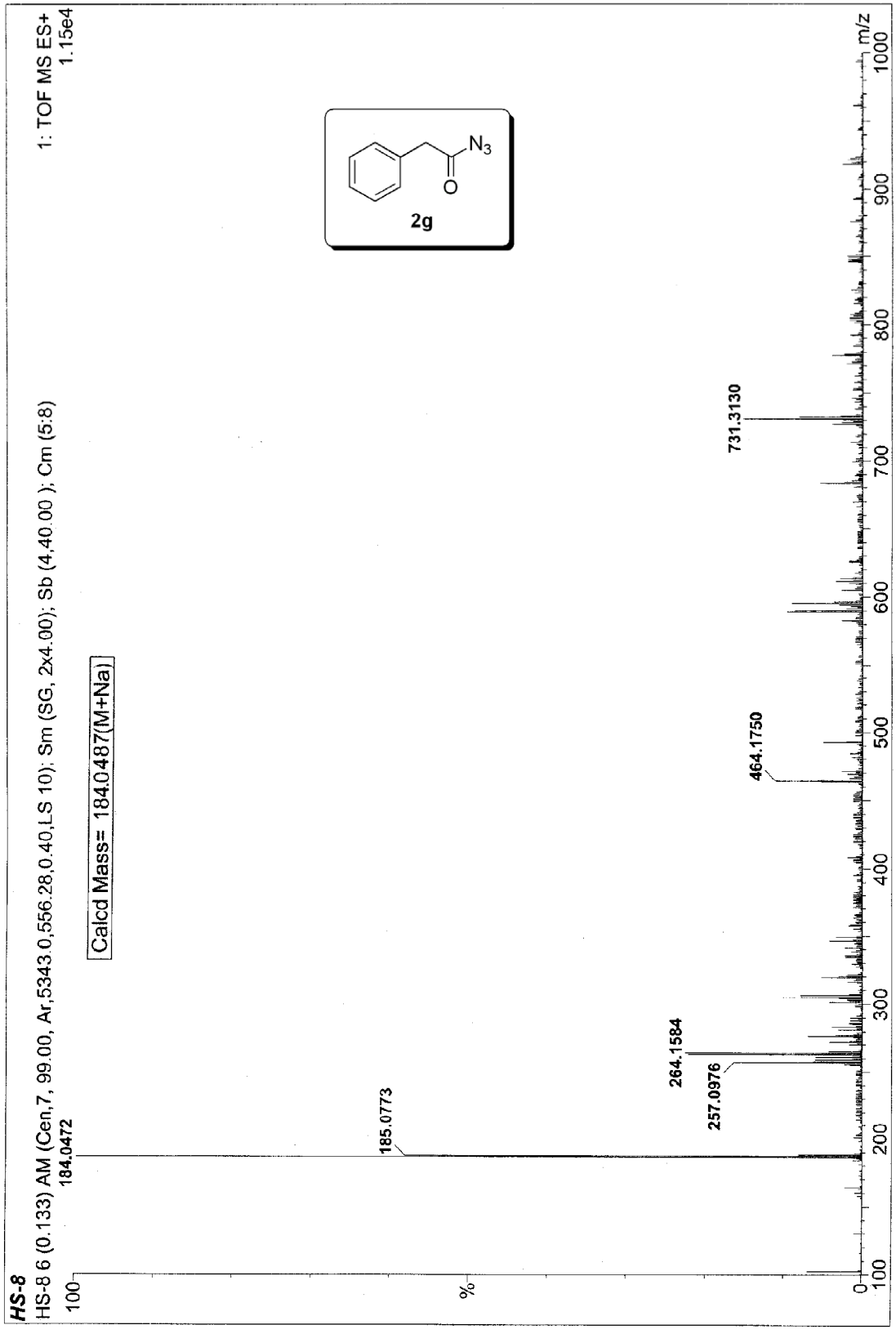
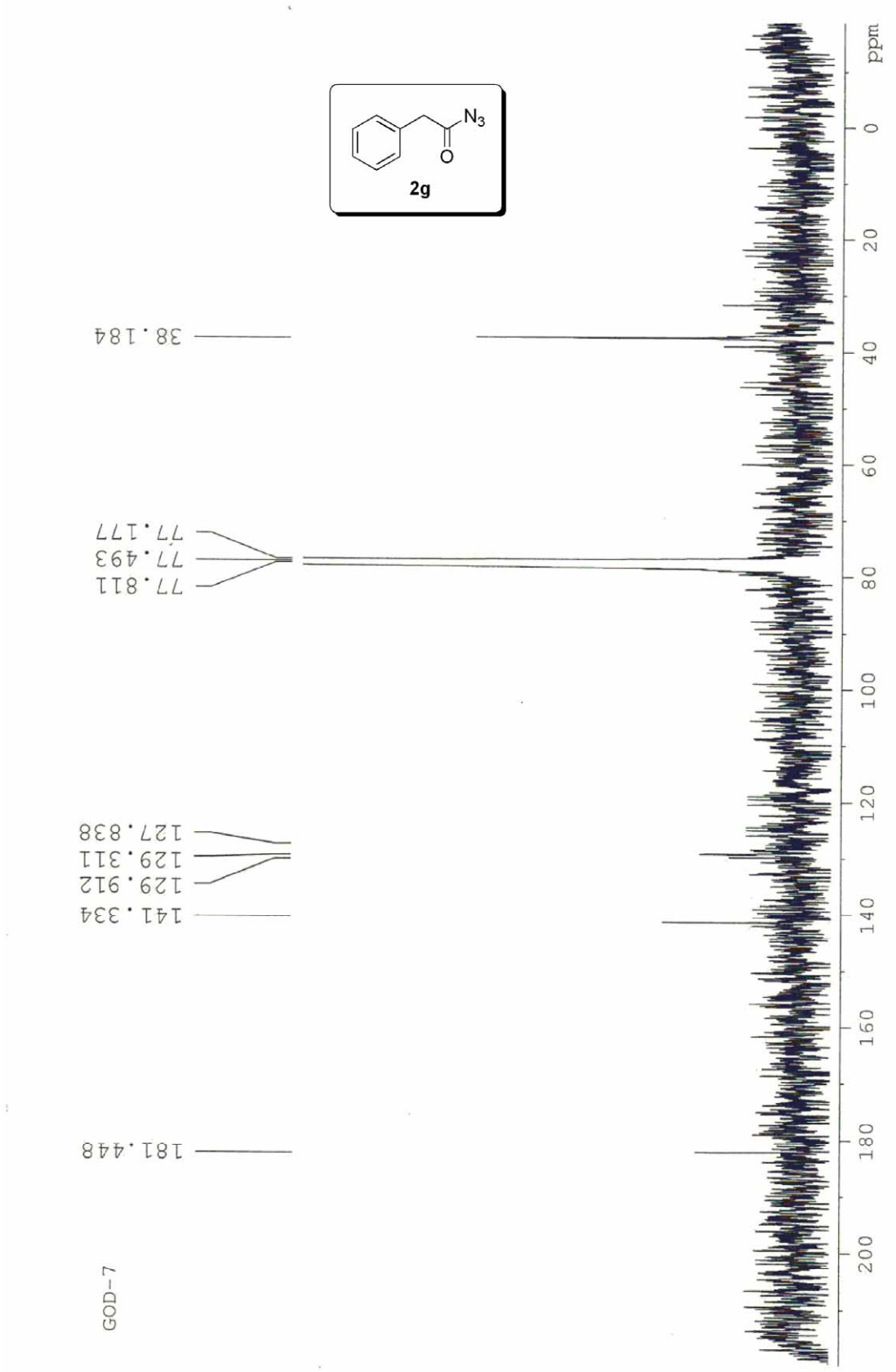
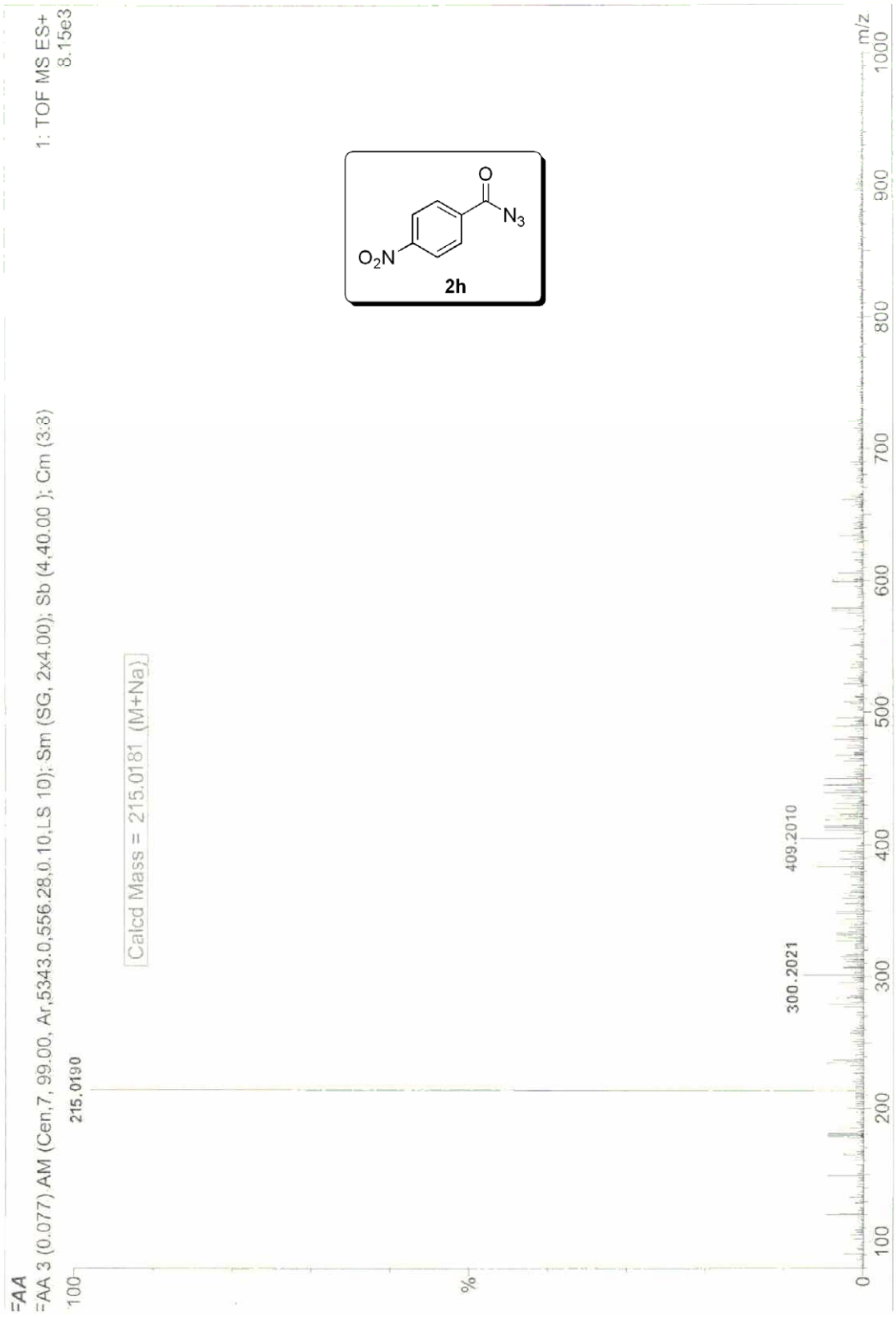
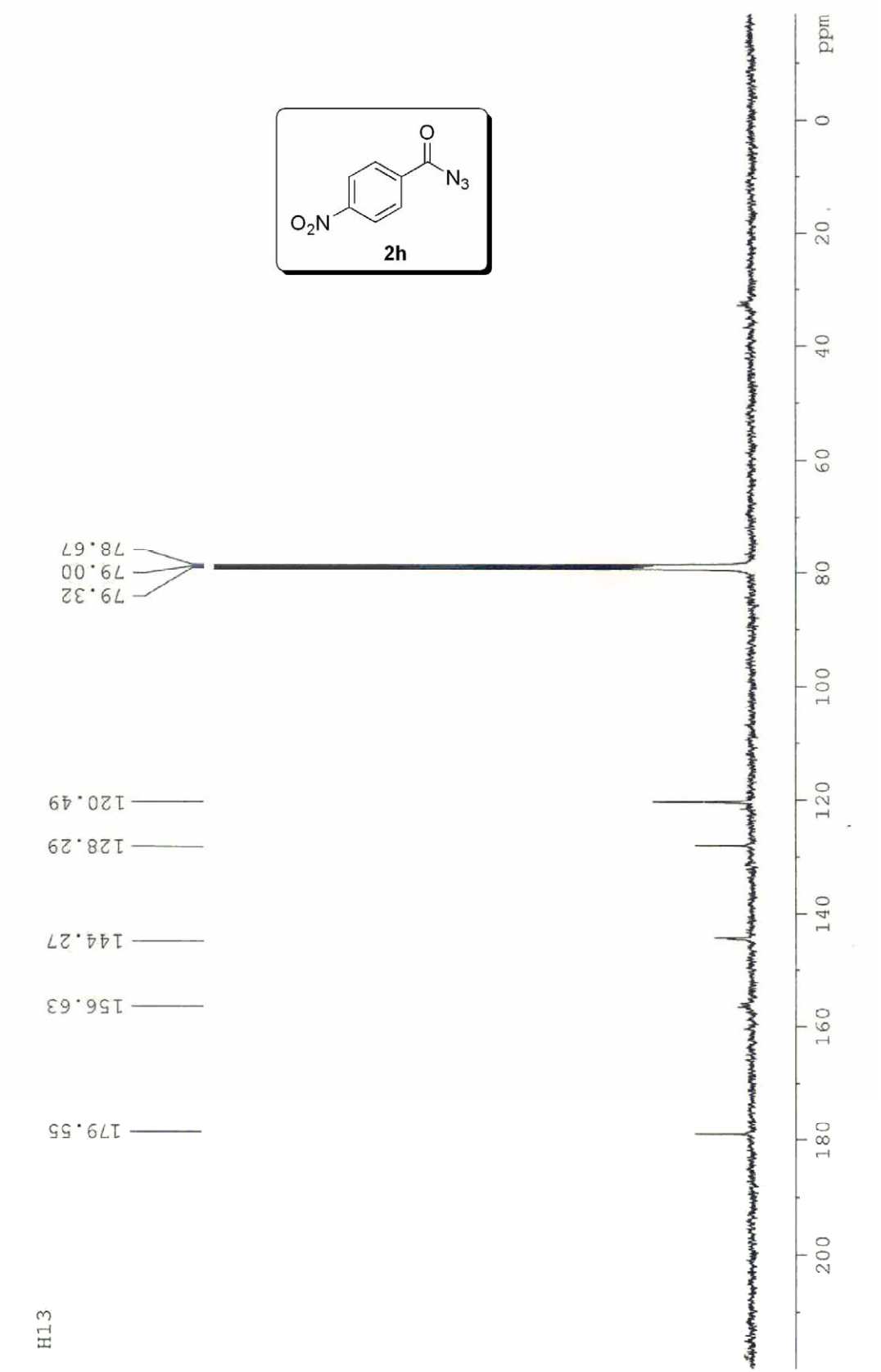
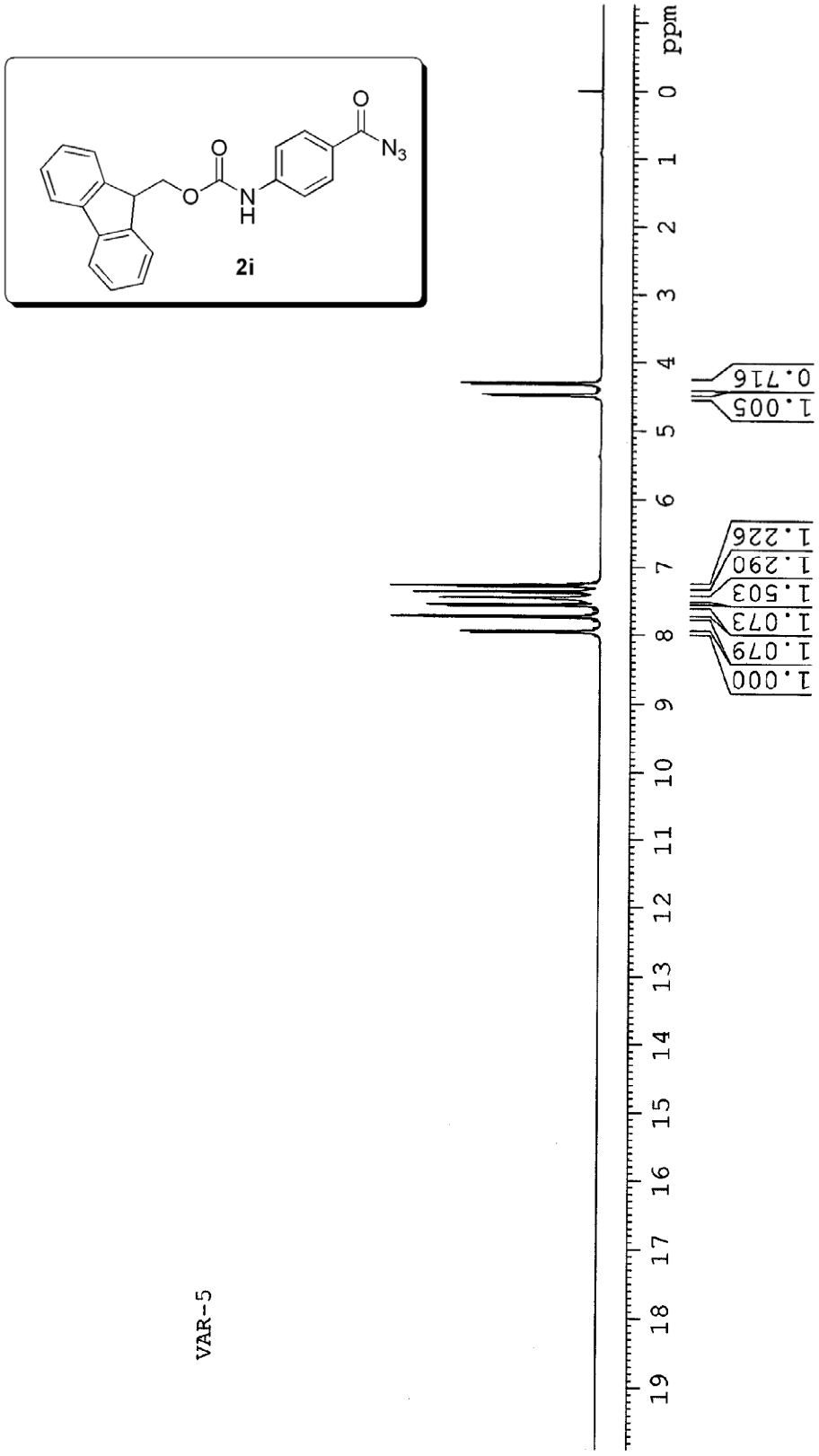
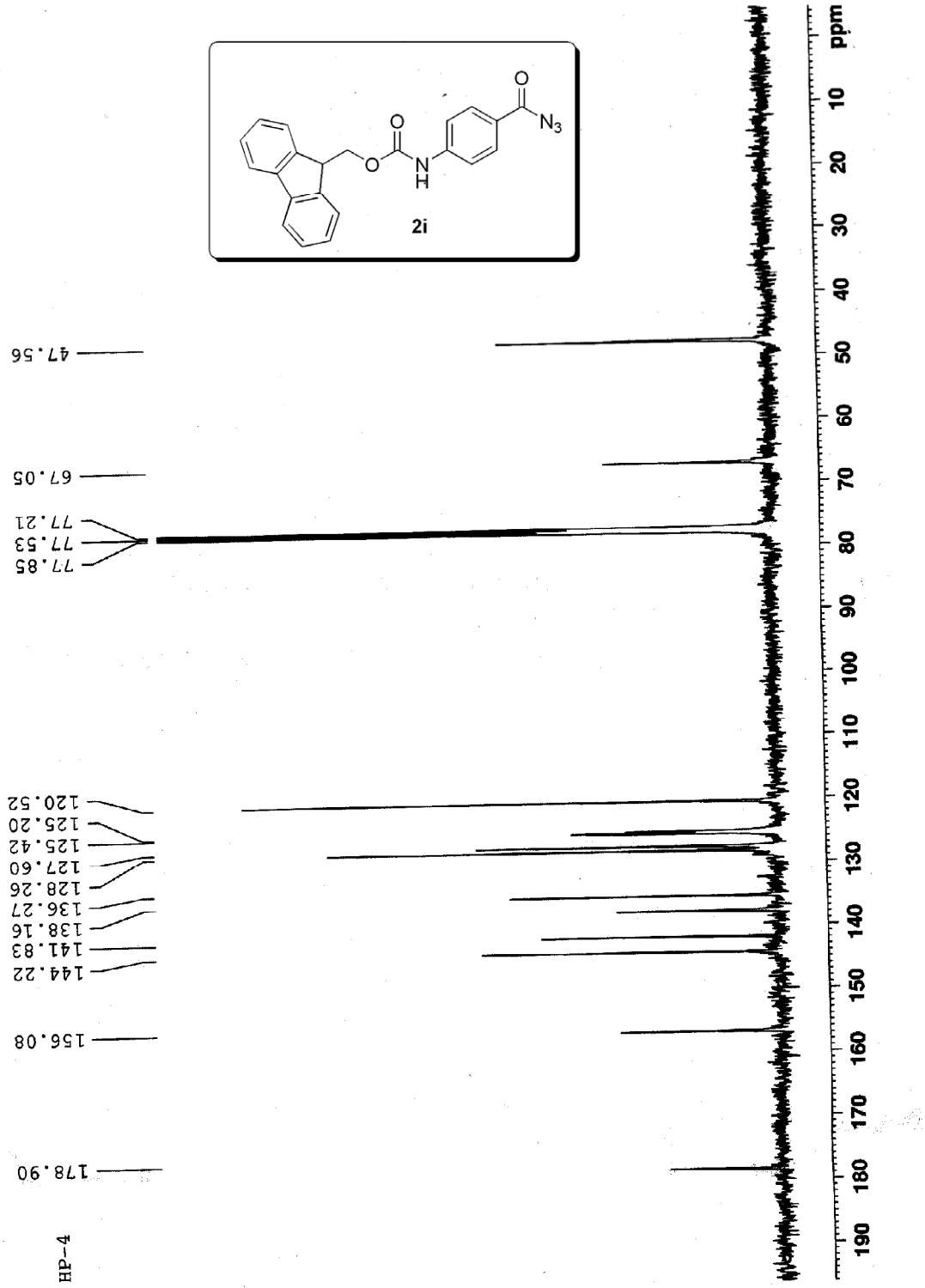

HS-8
HS-8 6 (0.133) AM (Cen,7, 99.00, Ar,5343.0,556.28,0.40,LS 10); Sm (SG, 2x4.00); Sb (4,40.00); Cm (5:8)
1: TOF MS ES+
1.15e4
Calcd Mass= 184.0487(M+Na)
184.0472
185.0773
264.1584
257.0976
464.1750
731.3130
N3
O
2g
100
%
0
100
200
300
400
500
600
700
800
900
1000
m/z

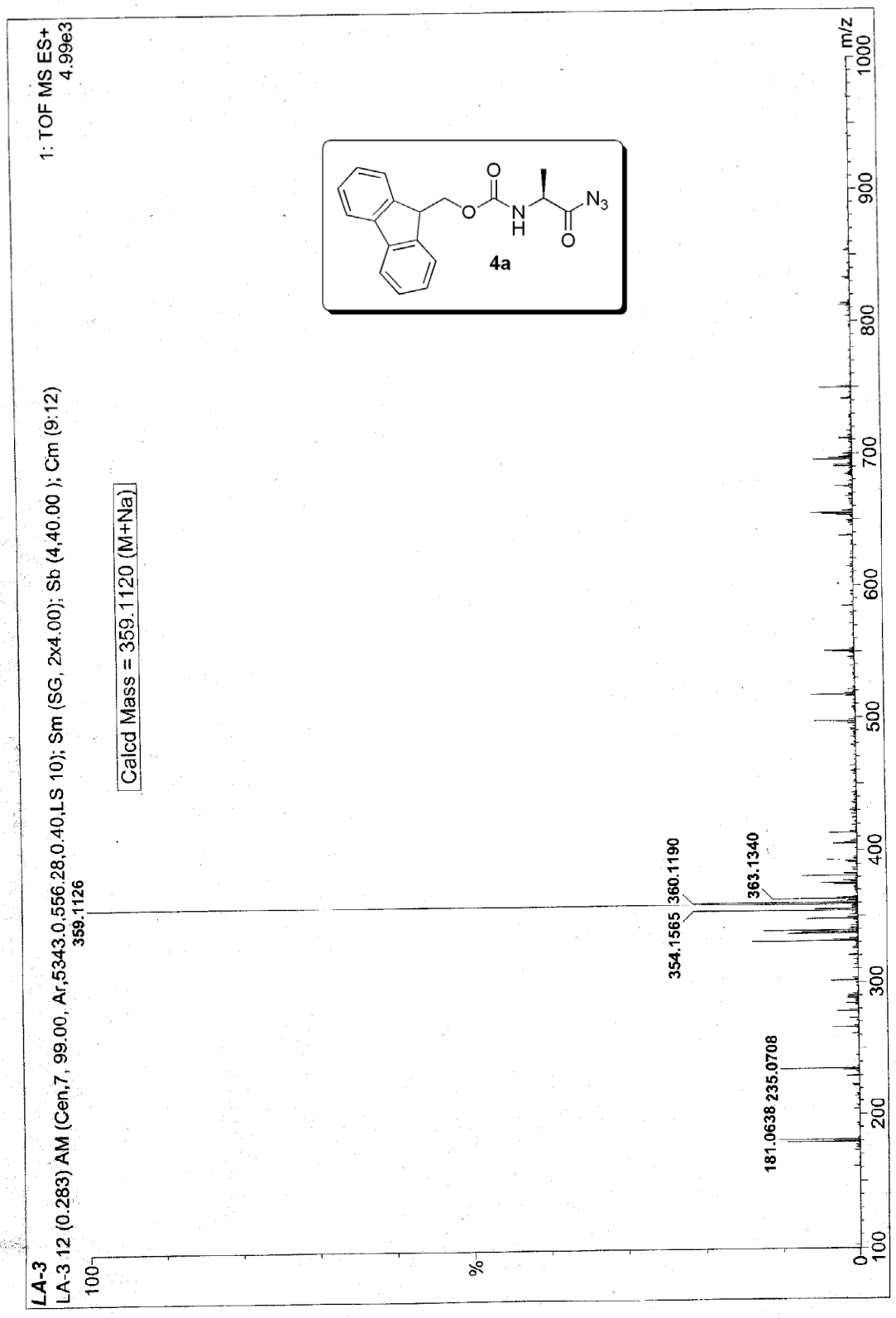

2g
38.184
77.177
77.493
77.811
127.838
129.311
129.912
141.334
181.448
GOD-7
ppm
0
20
40
60
80
100
120
140
160
180
200

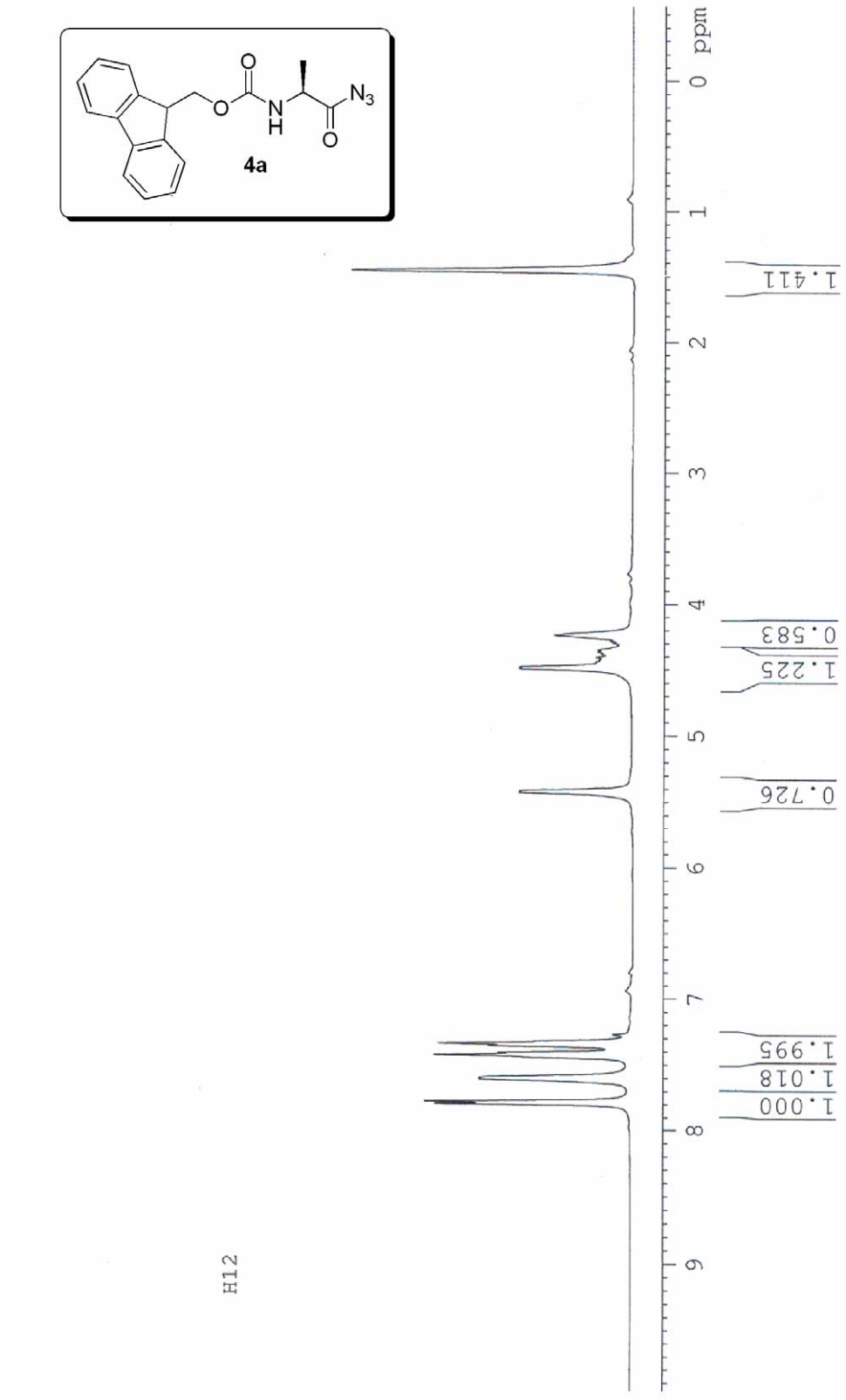

=AA
=AA 3 (0.077) AM (Cen,7, 99.00, Ar,5343.0,556.28,0.10,LS 10); Sm (SG, 2x4.00); Sb (4,40.00); Cm (3:3)
1: TOF MS ES+
8.15e3
100
%
0
215.0190
Calcd Mass = 215.0181 (M+Na)
300.2021
409.2010
100
200
300
400
500
600
700
800
900
1000
m/z
O
N3
O2N
2h

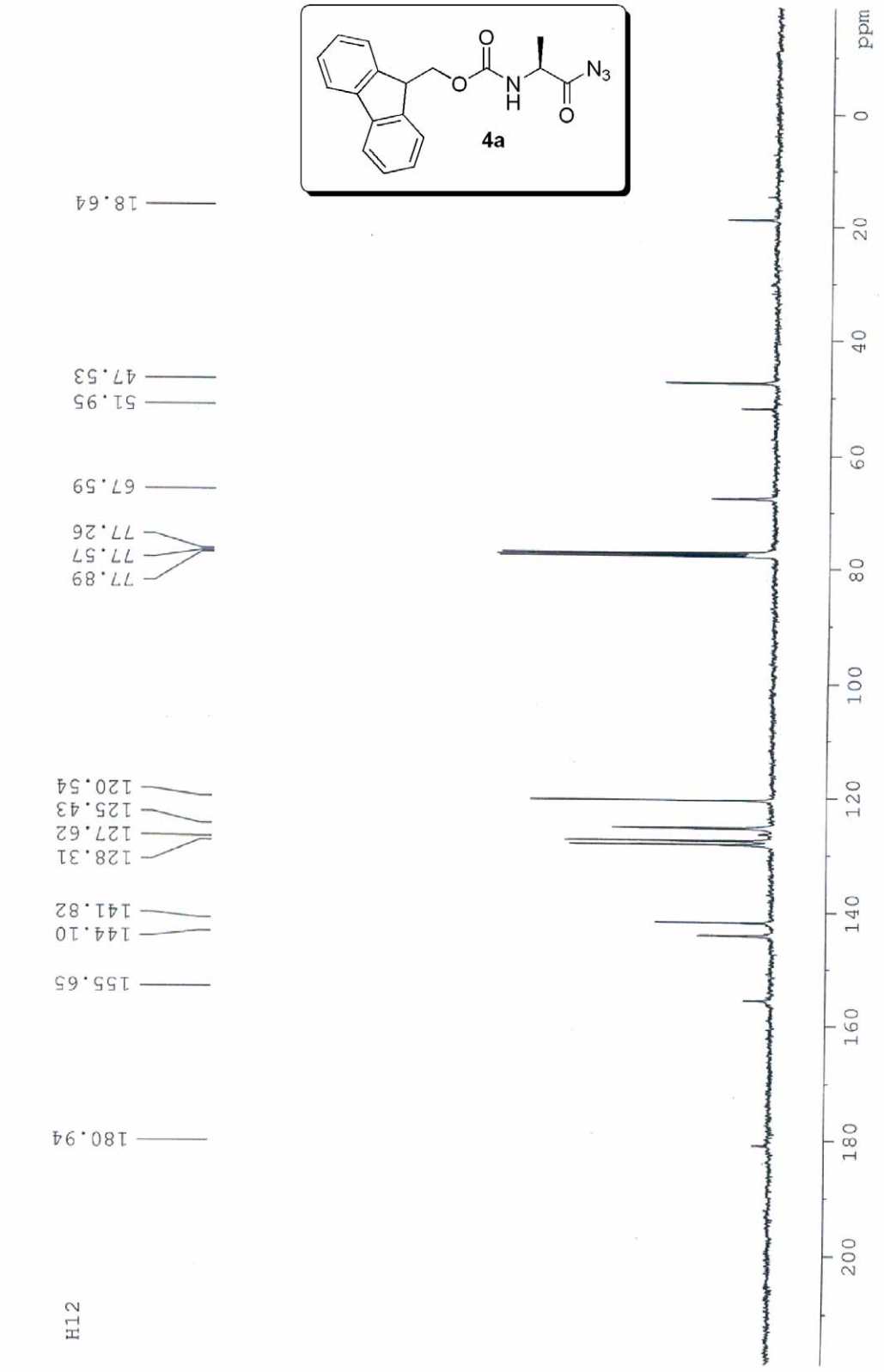

O
N3
O2N
2h
ppm
0
20
40
60
80
100
120
140
160
180
200
78.67
79.00
79.32
120.49
128.29
144.27
156.63
179.55
H13

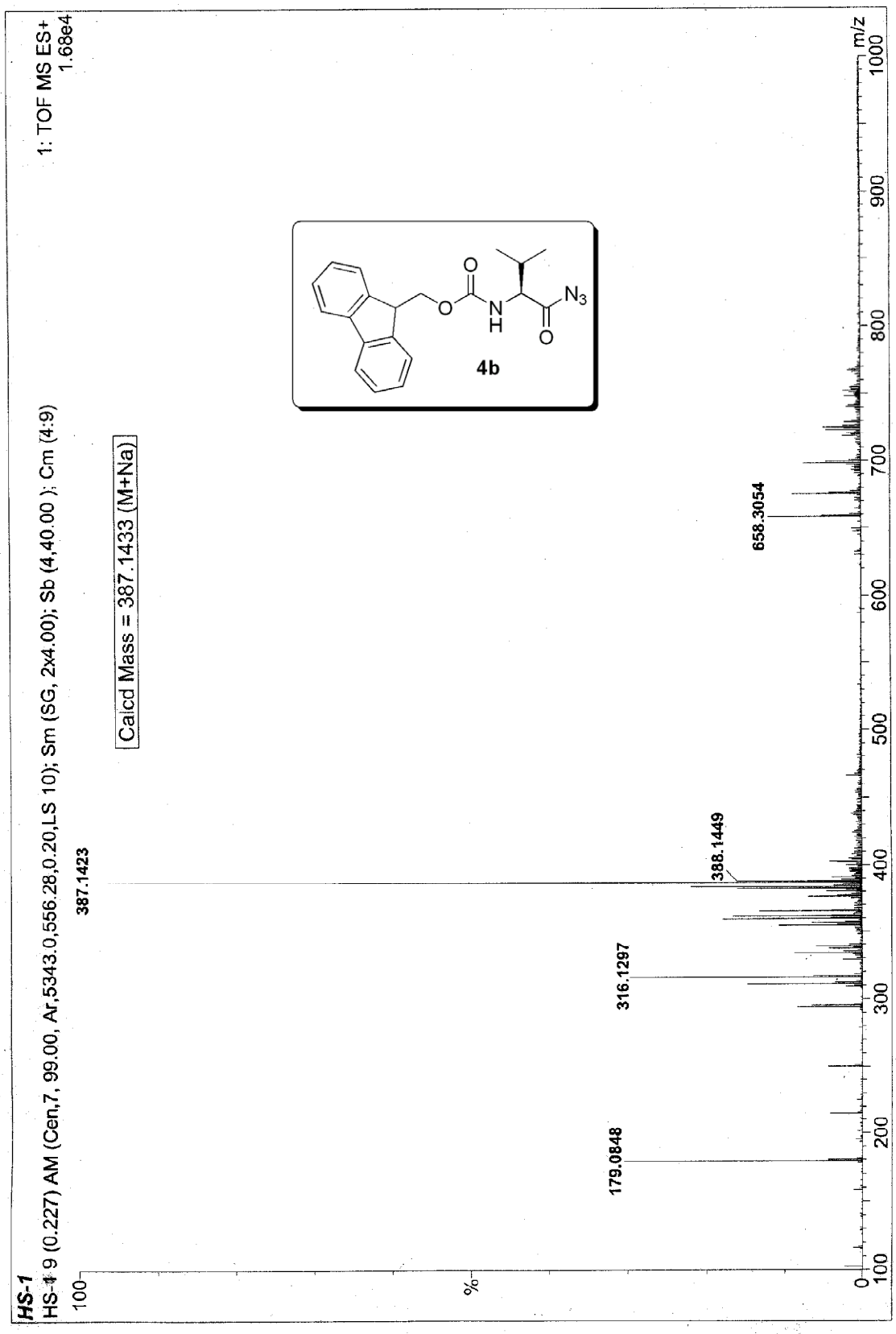

O
O
N3
O
N
H
2i
ppm
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19
1.000
1.079
1.073
1.503
1.290
1.226
1.005
0.716
VAR-5

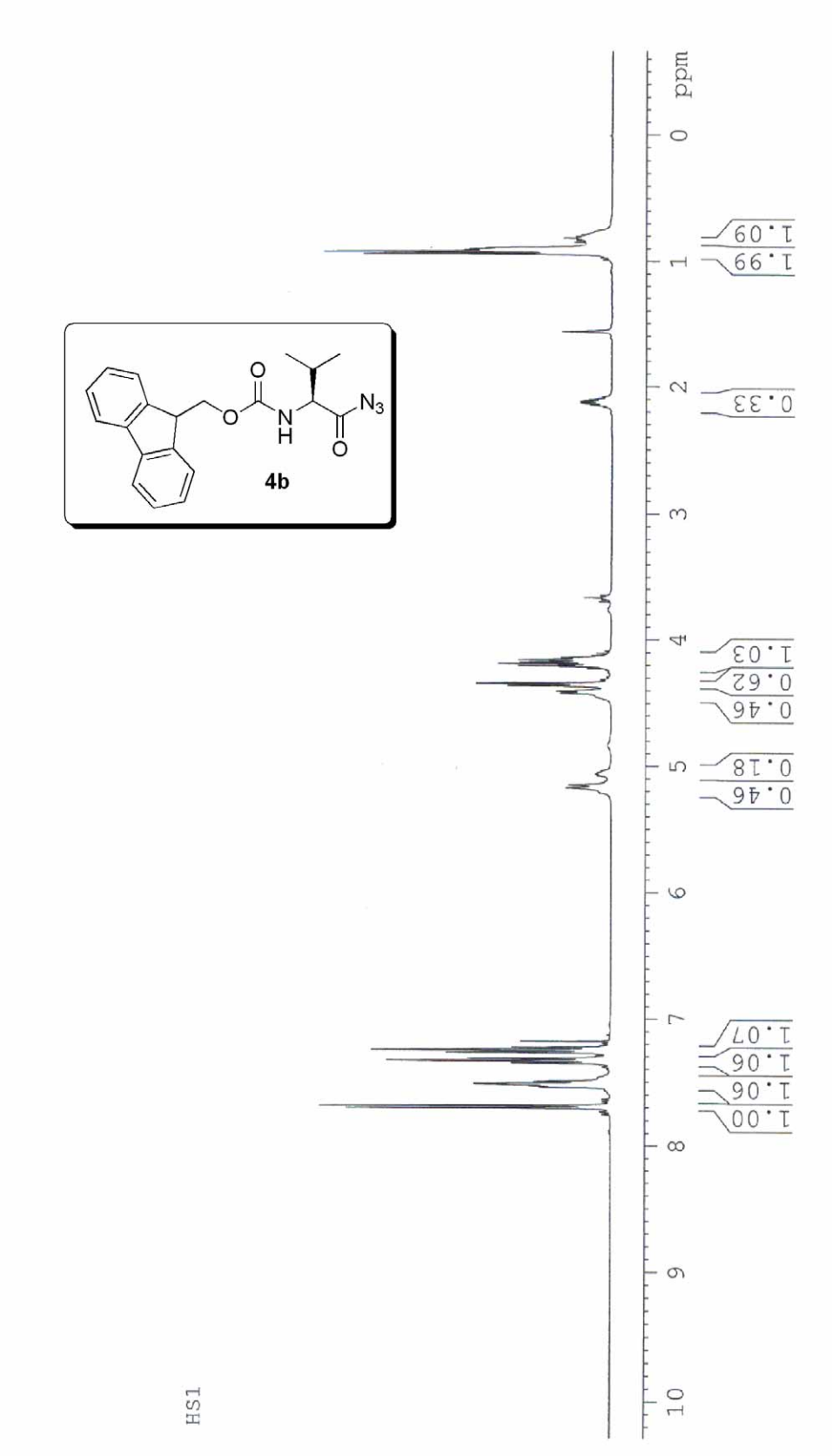

2i
47.56
67.05
77.21
77.53
77.85
120.52
125.20
125.42
127.60
128.26
136.27
138.16
141.83
144.22
156.08
178.90
HP-4
ppm

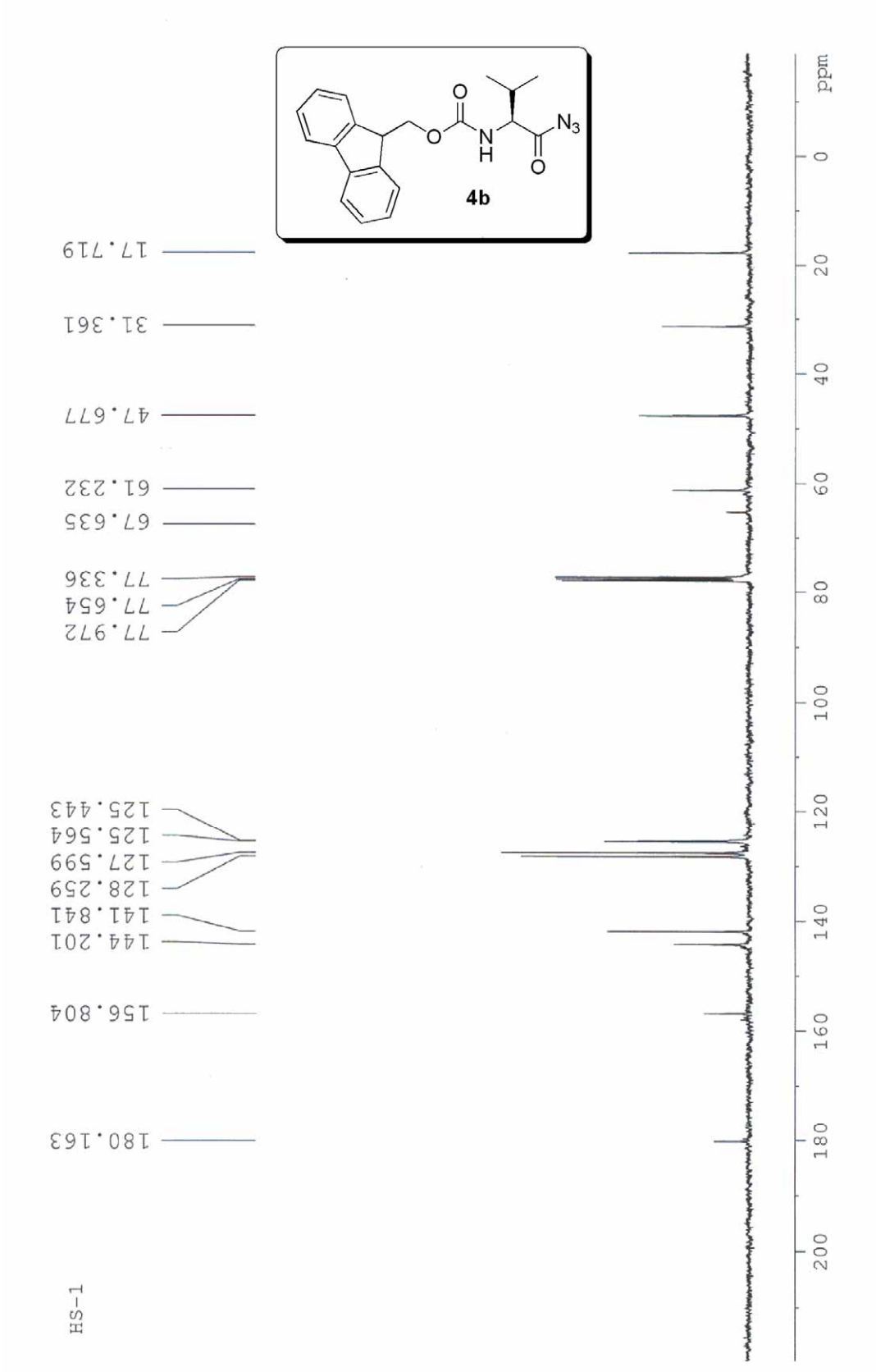

LA-3
LA-3 12 (0.283) AM (Cen,7, 99.00, Ar,5343.0,556.28,0.40,LS 10); Sm (SG, 2x4.00); Sb (4,40.00); Cm (9:12)
1: TOF MS ES+
4.99e3
Calcd Mass = 359.1120 (M+Na)
359.1126
354.1565
360.1190
363.1340
181.0638
235.0708
4a
%
m/z

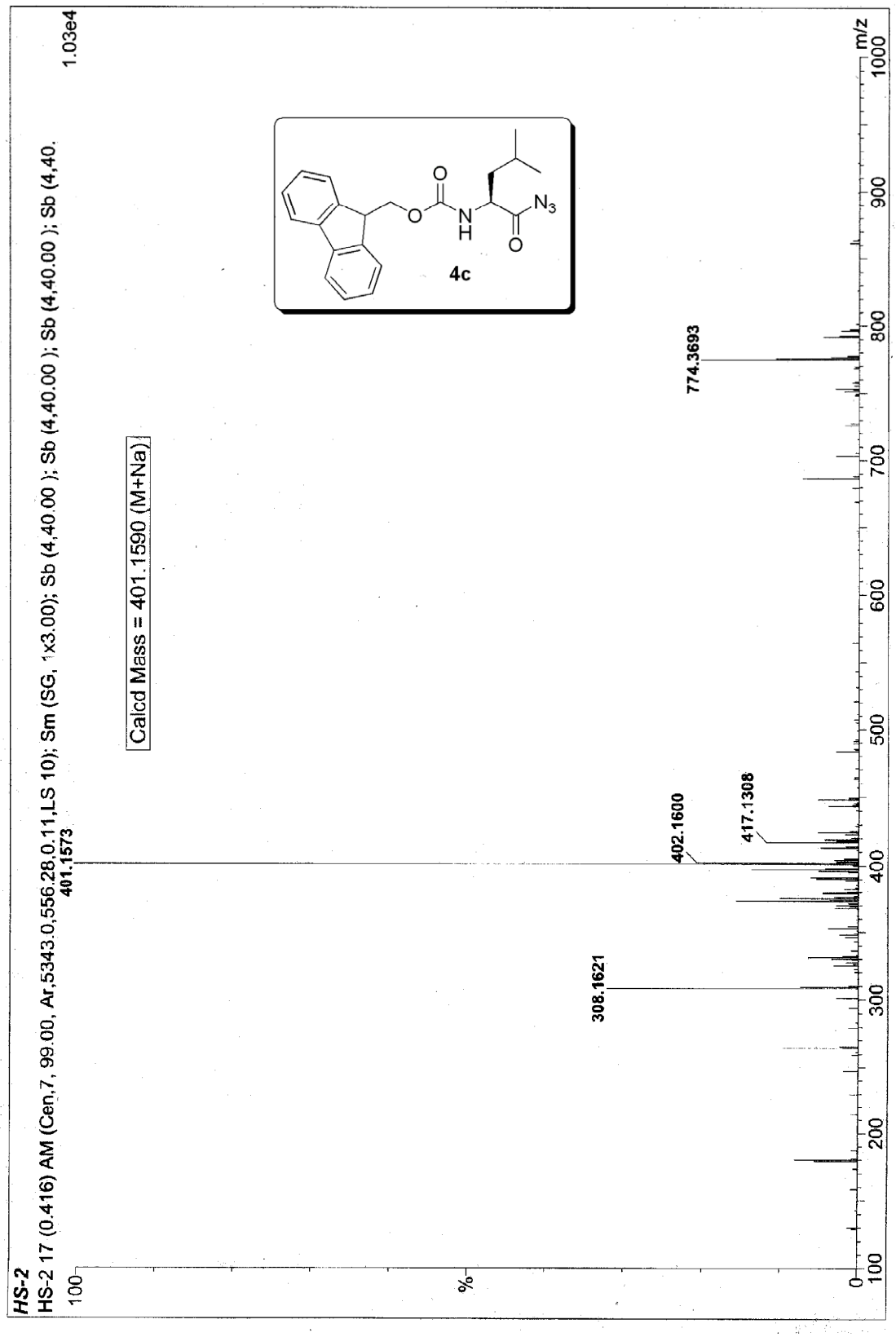

4a
O
O
N
H
O
N3
ppm
0
1
2
3
4
5
6
7
8
9
1.411
0.583
1.225
0.726
1.995
1.018
1.000
H12

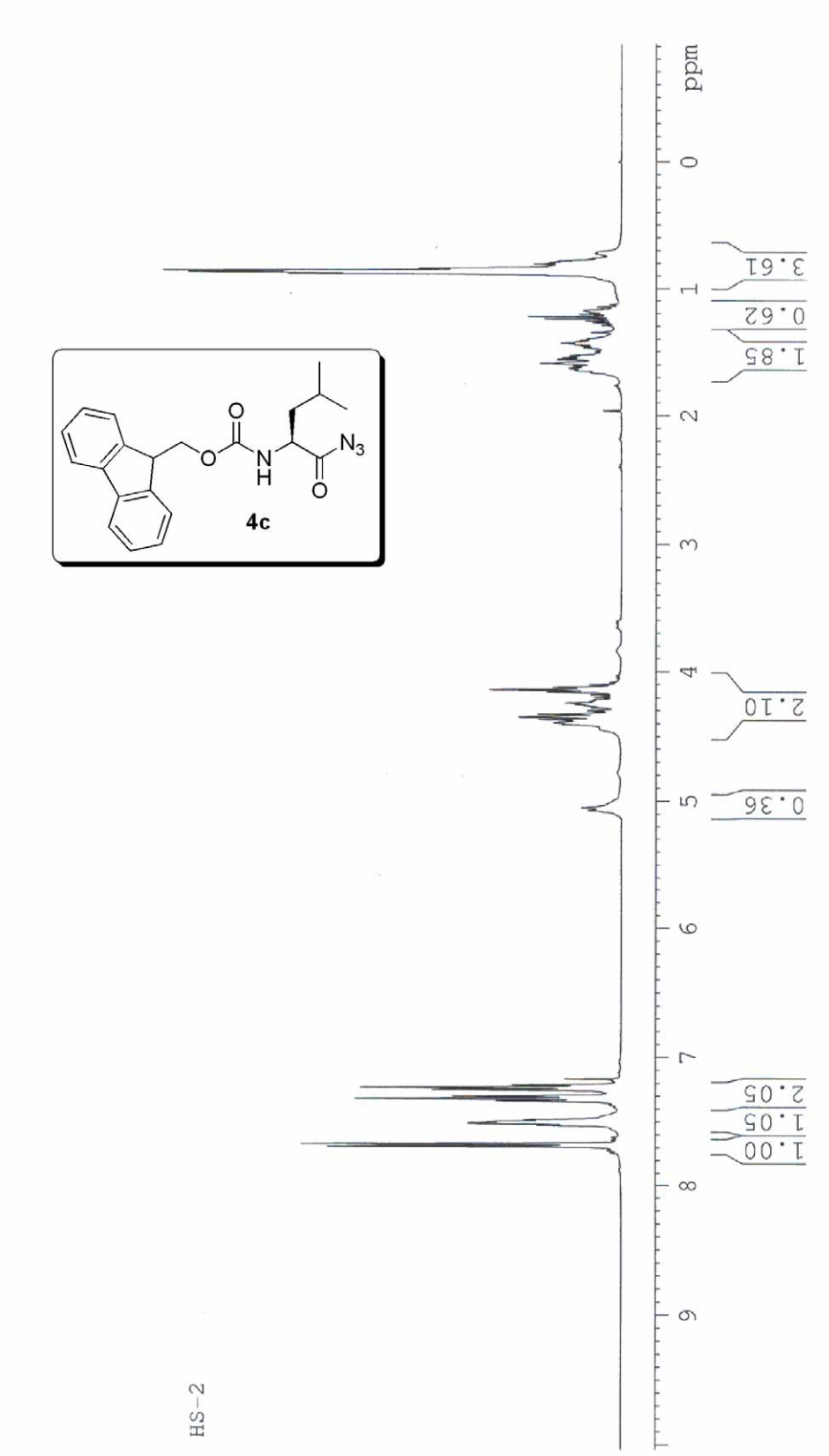

4a
18.64
47.53
51.95
67.59
77.26
77.57
77.89
120.54
125.43
127.62
128.31
141.82
144.10
155.65
180.94
H12
ppm
0
20
40
60
80
100
120
140
160
180
200

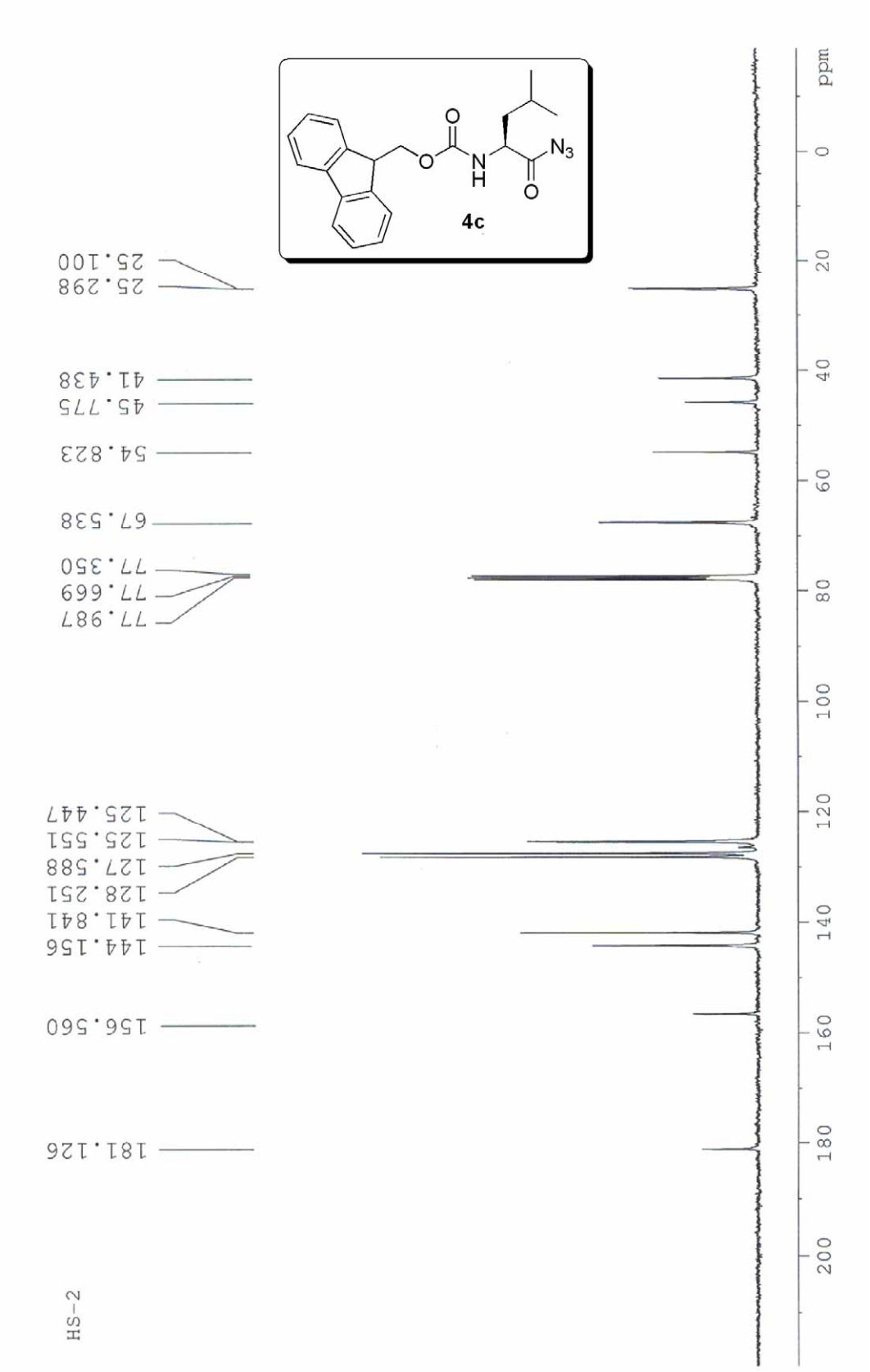

HS-1
HS-1 9 (0.227) AM (Cen,7, 99.00, Ar,5343.0,556.28,0.20,LS 10); Sm (SG, 2x4.00); Sb (4,40.00); Cm (4:9)
1: TOF MS ES+
1.68e4
Calcd Mass = 387.1433 (M+Na)
387.1423
388.1449
658.3054
316.1297
179.0848
4b
%
m/z

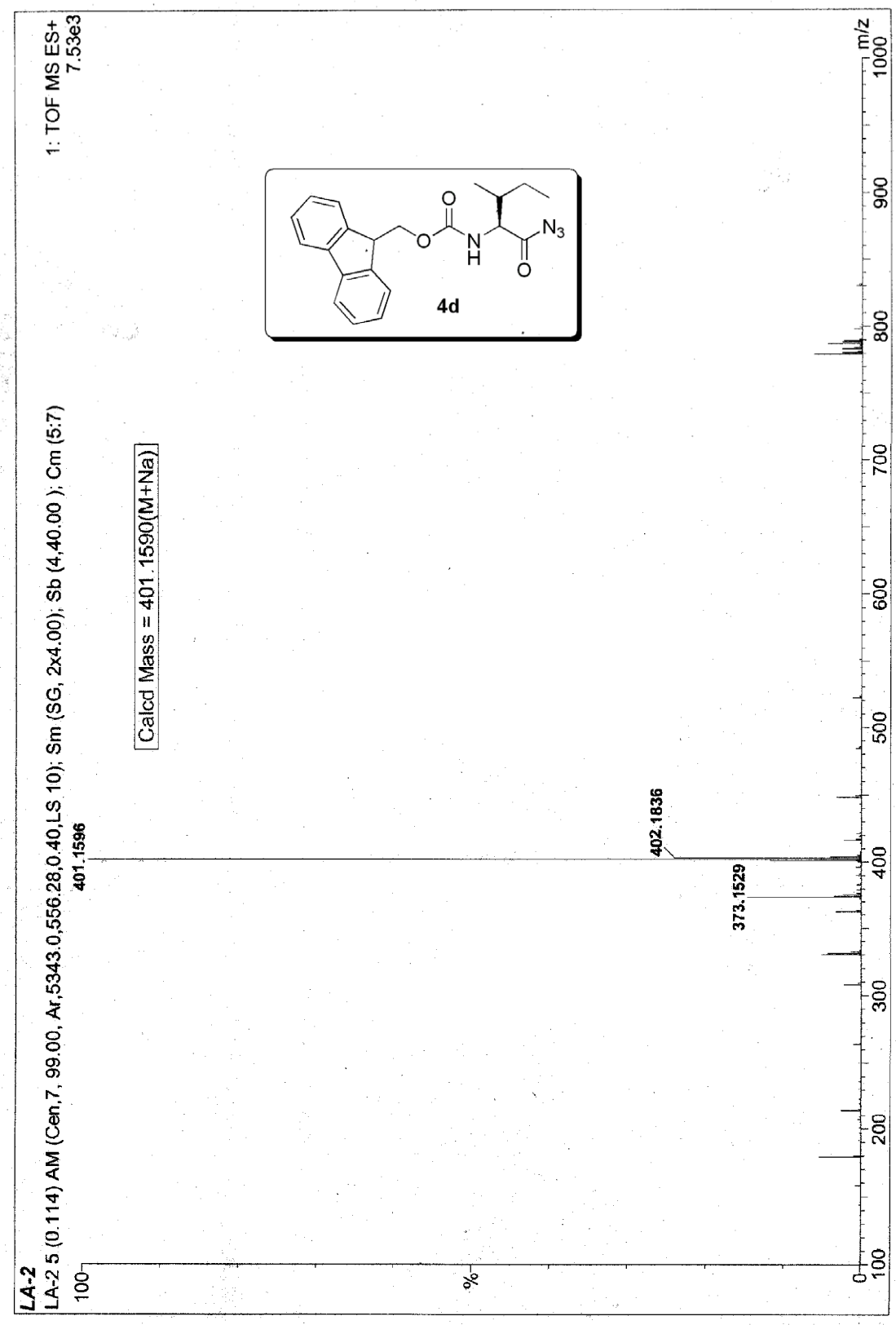

4b
ppm
0
1
2
3
4
5
6
7
8
9
10
1.09
1.99
0.33
1.03
0.62
0.46
0.18
0.46
1.07
1.06
1.06
1.00
HS1

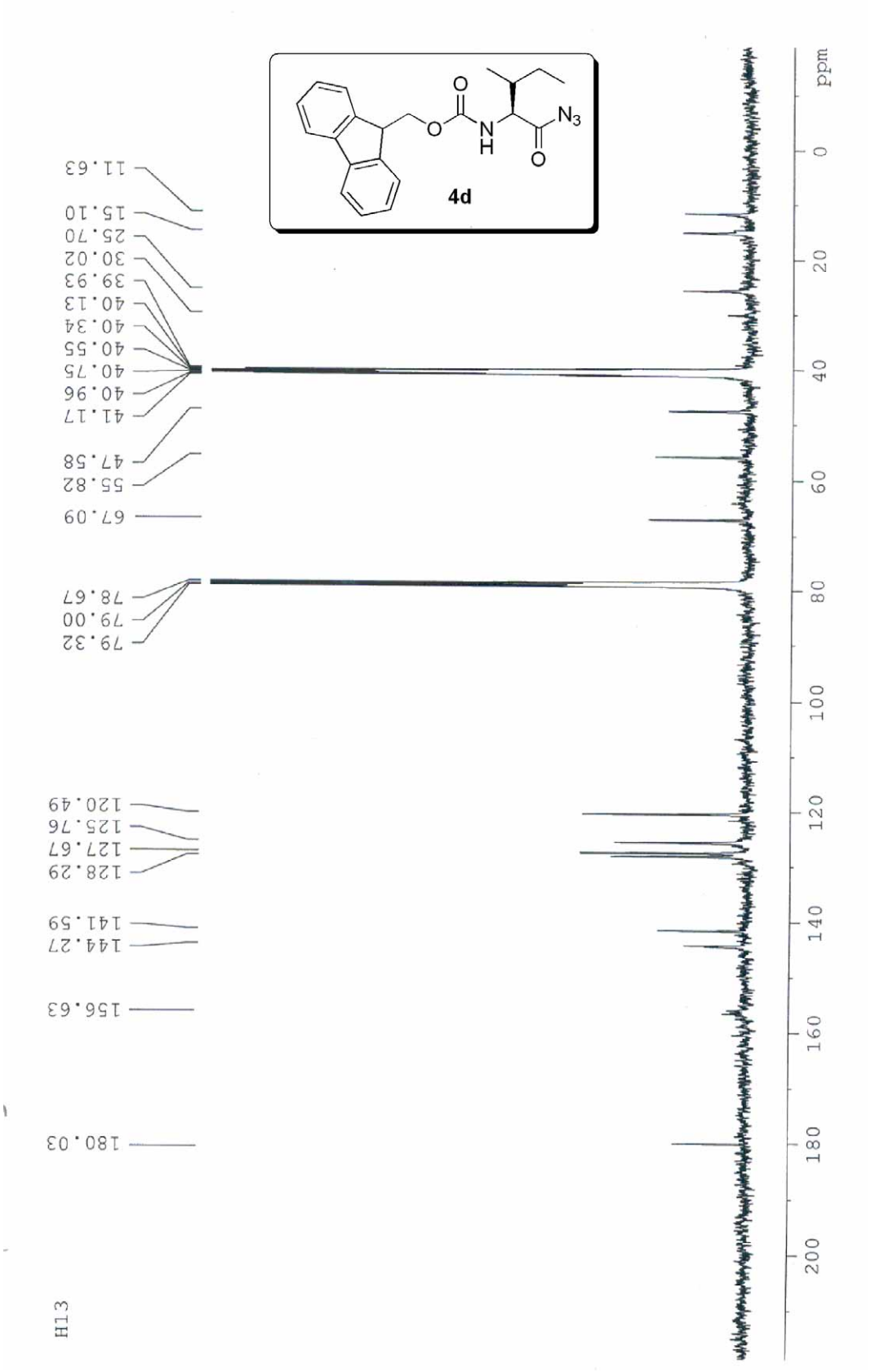

4b
17.719
31.361
47.677
61.232
67.635
77.336
77.654
77.972
125.443
125.564
127.599
128.259
141.841
144.201
156.804
180.163
HS-1
ppm
0
20
40
60
80
100
120
140
160
180
200

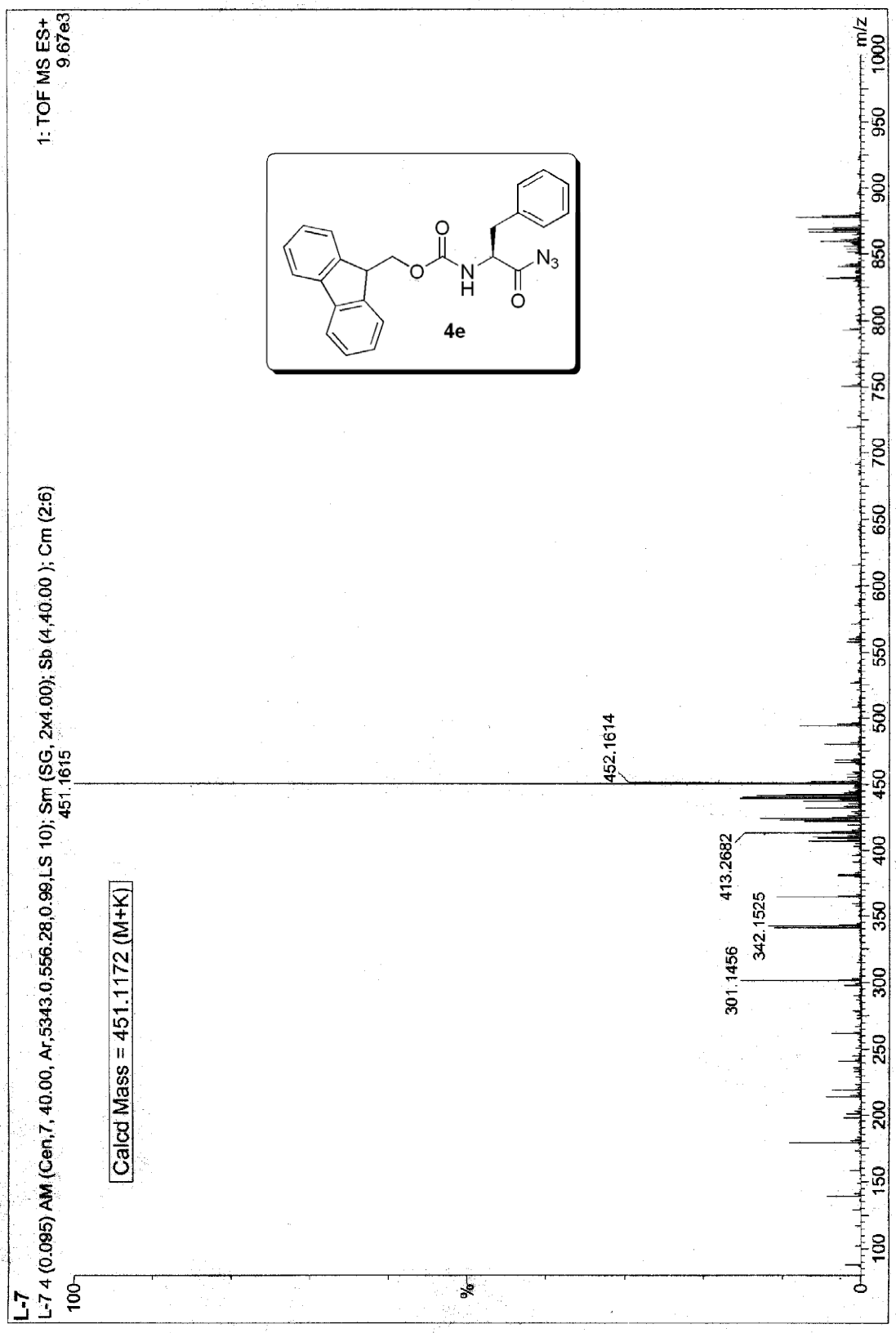

HS-2
HS-2 17 (0.416) AM (Cen,7, 99.00, Ar,5343.0,556.28,0.11,LS 10); Sm (SG, 1x3.00); Sb (4,40.00); Sb (4,40.00); Sb (4,40.00); Sb (4,40.
1.03e4
Calcd Mass = 401.1590 (M+Na)
401.1573
402.1600
417.1308
308.1621
774.3693
4c
%
m/z

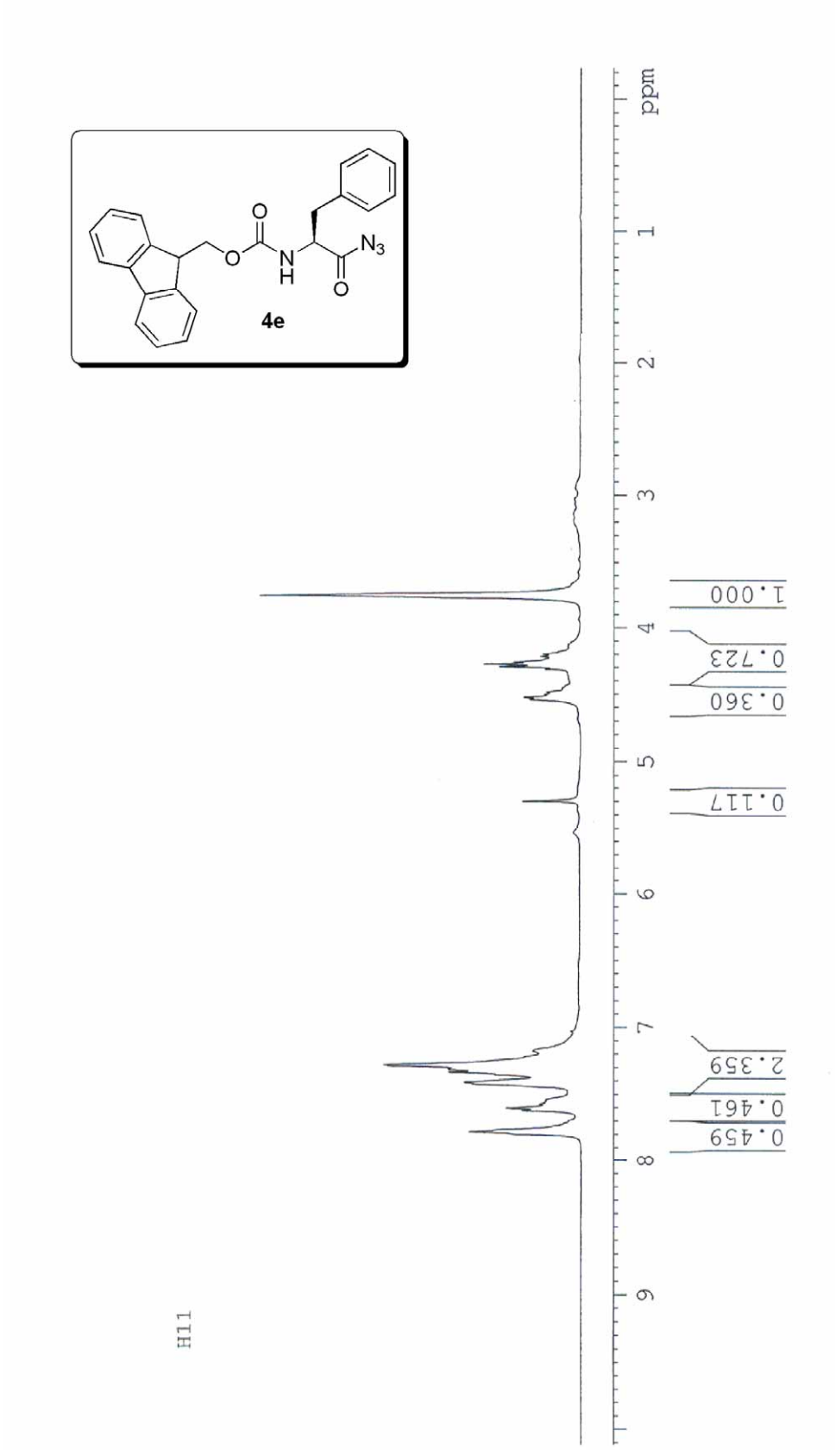

4c
ppm
3.61
0.62
1.85
2.10
0.36
2.05
1.05
1.00
HS-2

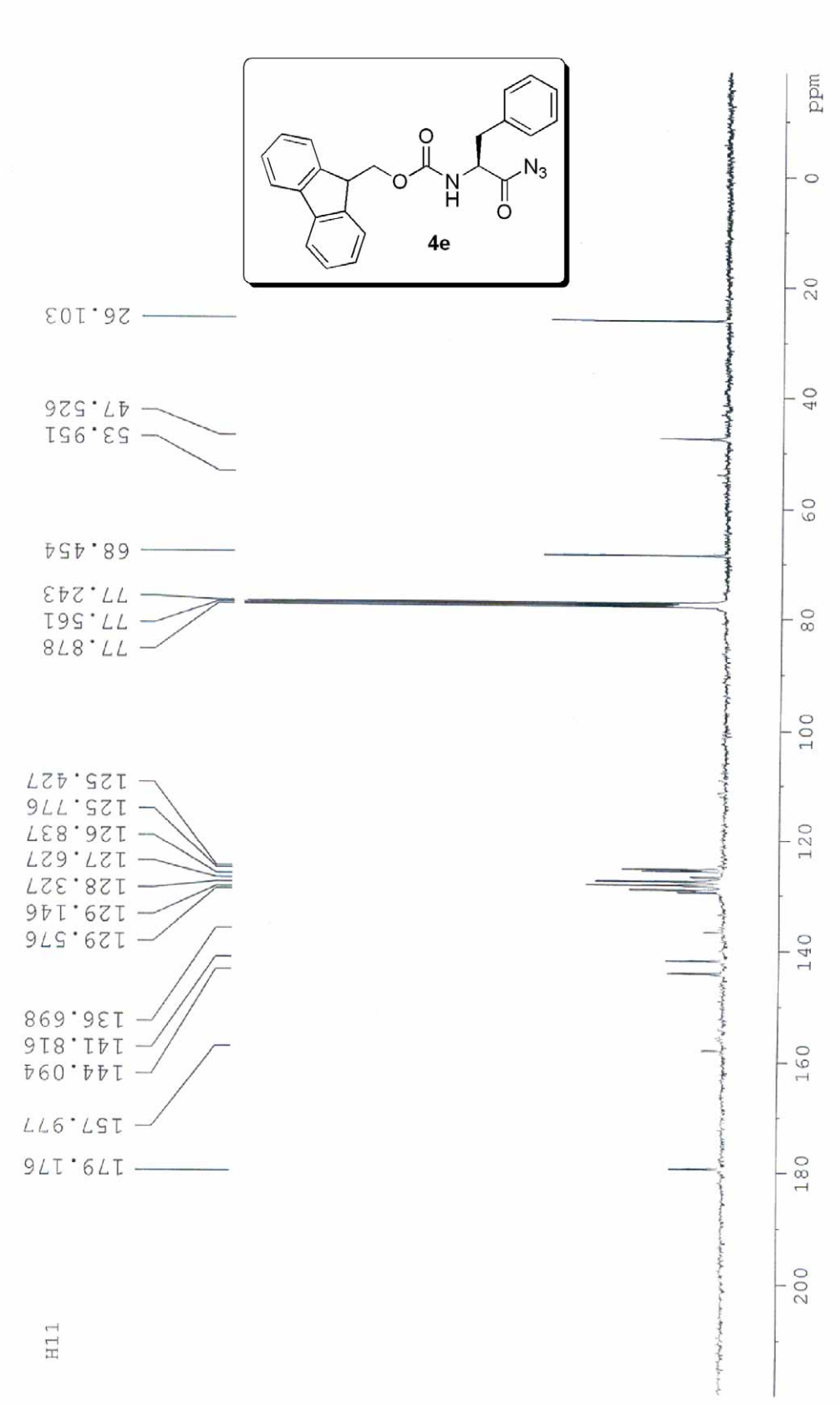

4c
25.100
25.298
41.438
45.775
54.823
67.538
77.350
77.669
77.987
125.447
125.551
127.588
128.251
141.841
144.156
156.560
181.126
HS-2
ppm
0
20
40
60
80
100
120
140
160
180
200

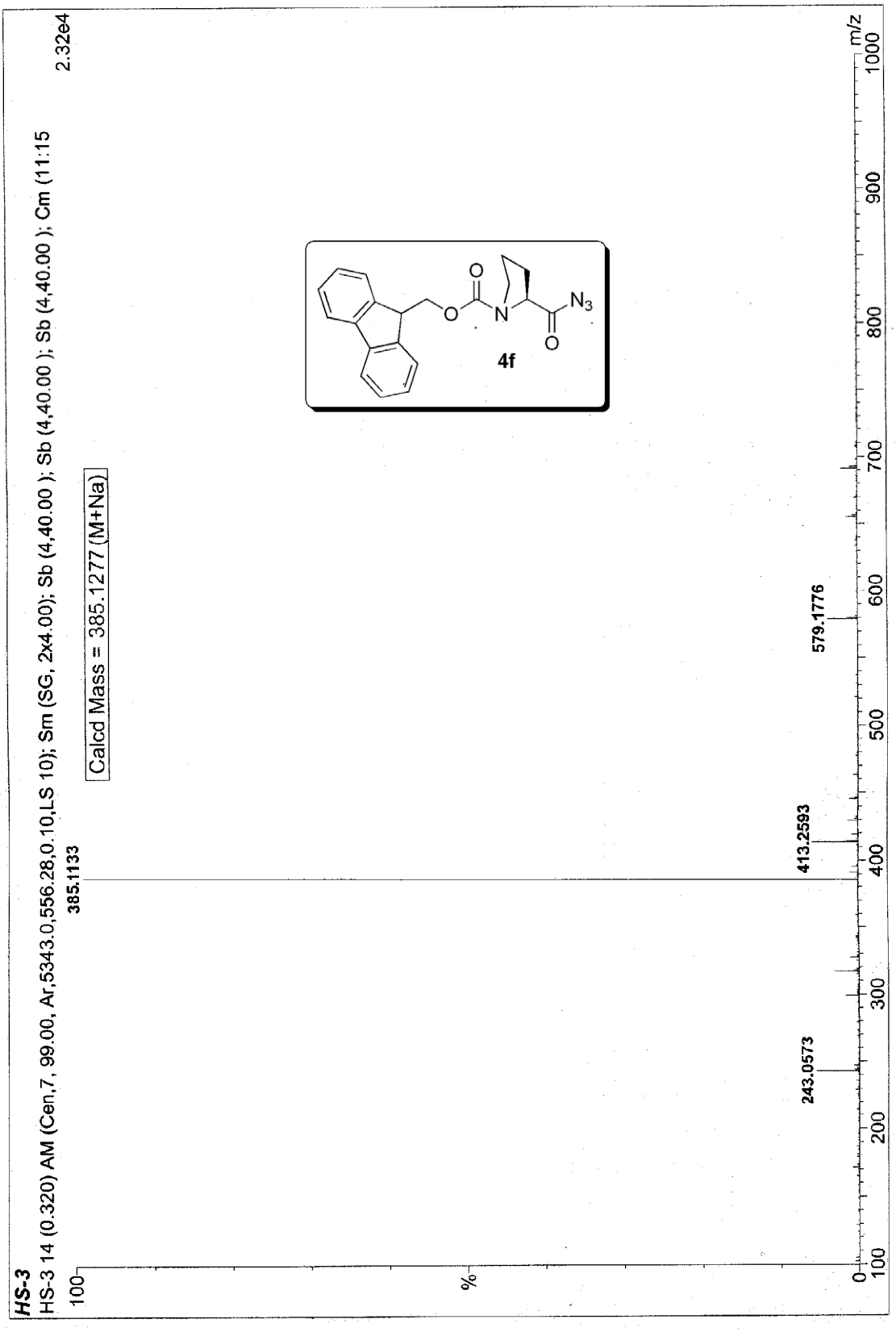

LA-2
LA-2 5 (0.114) AM (Cen,7, 99.00, Ar,5343.0,556.28,0.40,LS 10); Sm (SG, 2x4.00); Sb (4.40.00); Cm (5:7)
1: TOF MS ES+
7.53e3
Calcd Mass = 401.1590(M+Na)
401.1596
402.1836
373.1529
4d
%
m/z

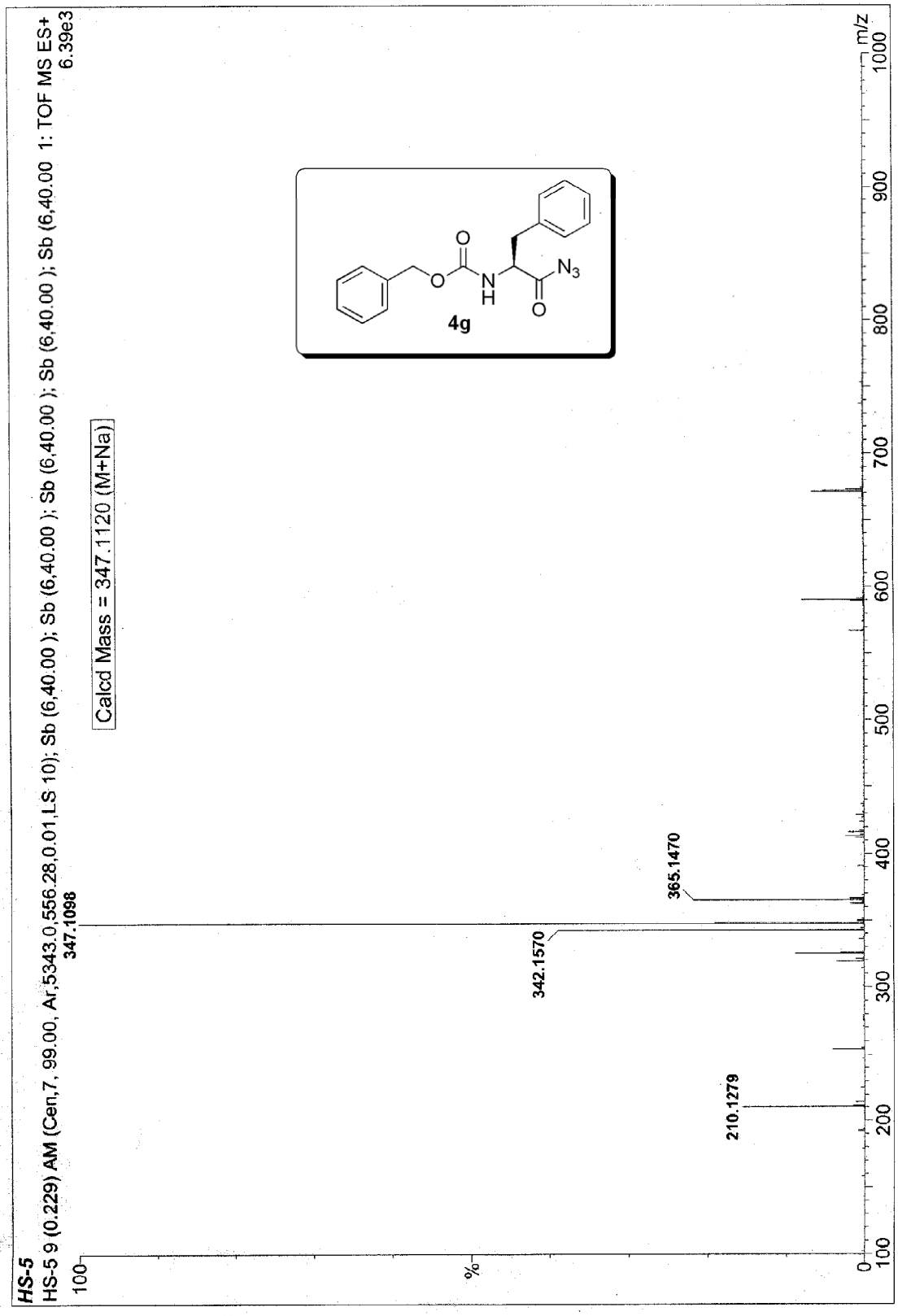

4d
O
O
N
H
N3
O
11.63
15.10
25.70
30.02
39.93
40.13
40.34
40.55
40.75
40.96
41.17
47.58
55.82
67.09
78.67
79.00
79.32
120.49
125.76
127.67
128.29
141.59
144.27
156.63
180.03
ppm
0
20
40
60
80
100
120
140
160
180
200
H13

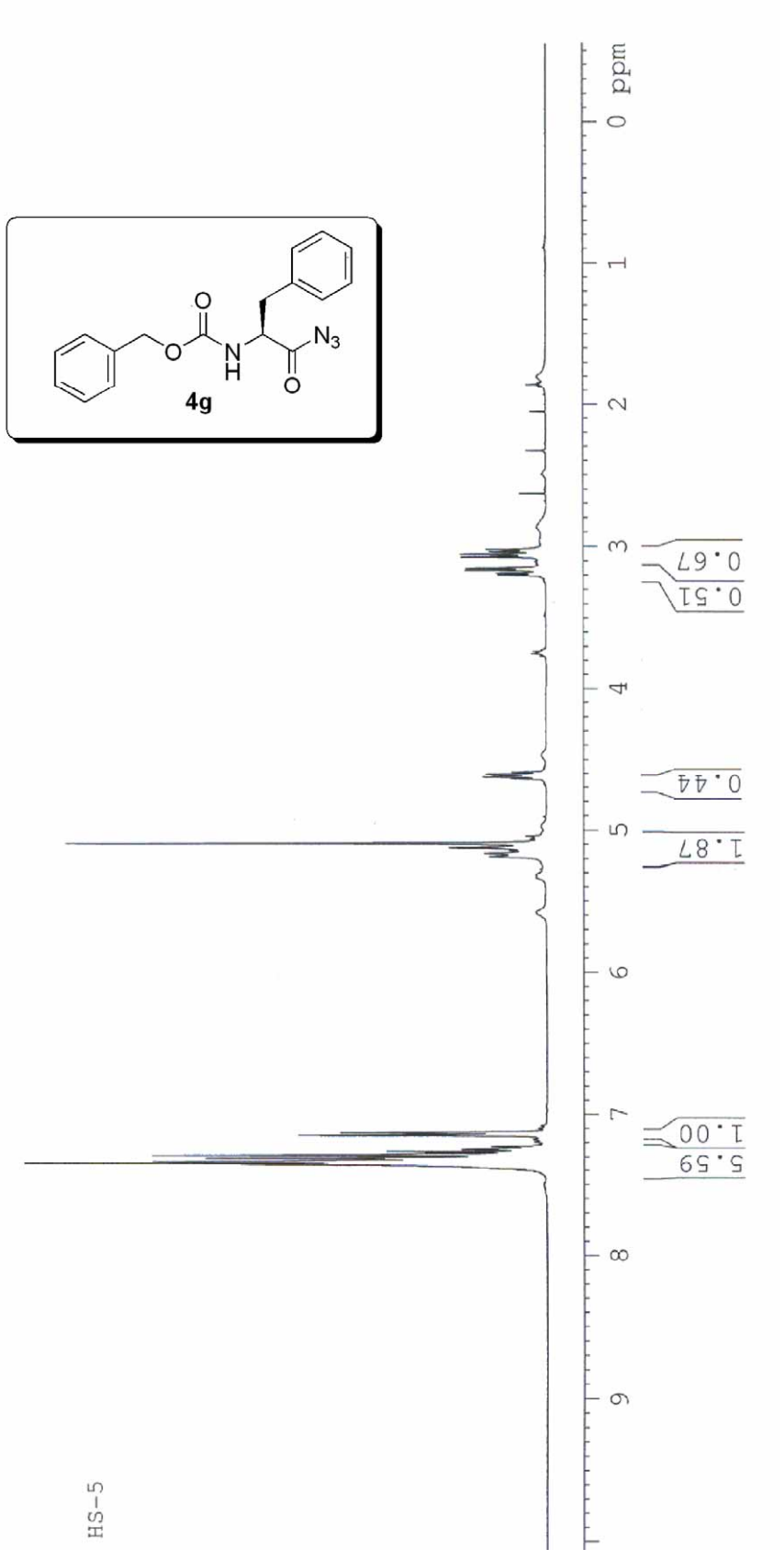

L-7
L-7 4 (0.095) AM (Cen,7, 40.00, Ar,5343.0,556.28,0.99,LS 10); Sm (SG, 2x4.00); Sb (4,40.00); Cm (2:6)
1: TOF MS ES+
9.67e3
451.1615
Calcd Mass = 451.1172 (M+K)
452.1614
413.2682
342.1525
301.1456
4e
%
m/z

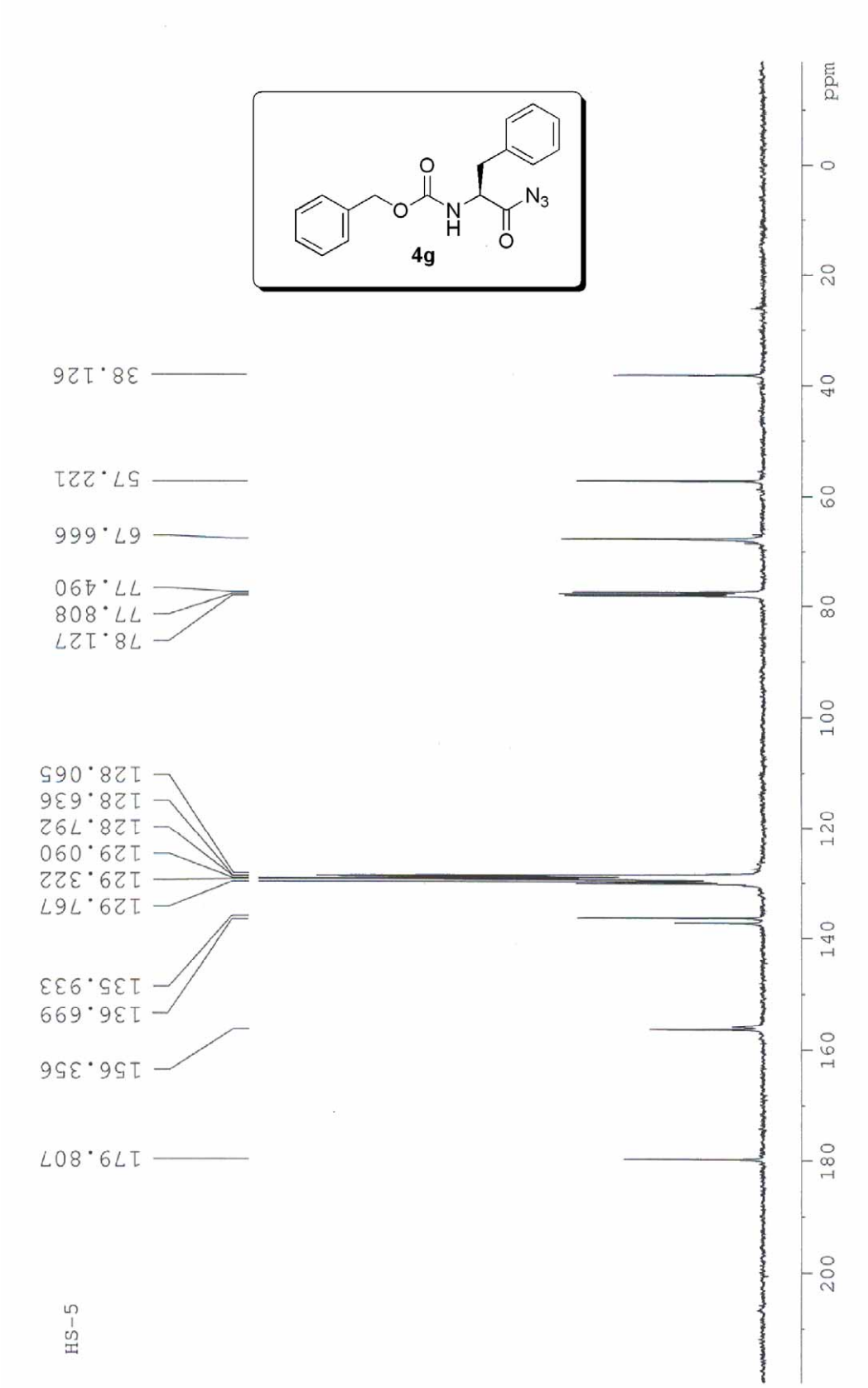

4e
ppm
1
2
3
4
5
6
7
8
9
1.000
0.723
0.360
0.117
2.359
0.461
0.459
H11

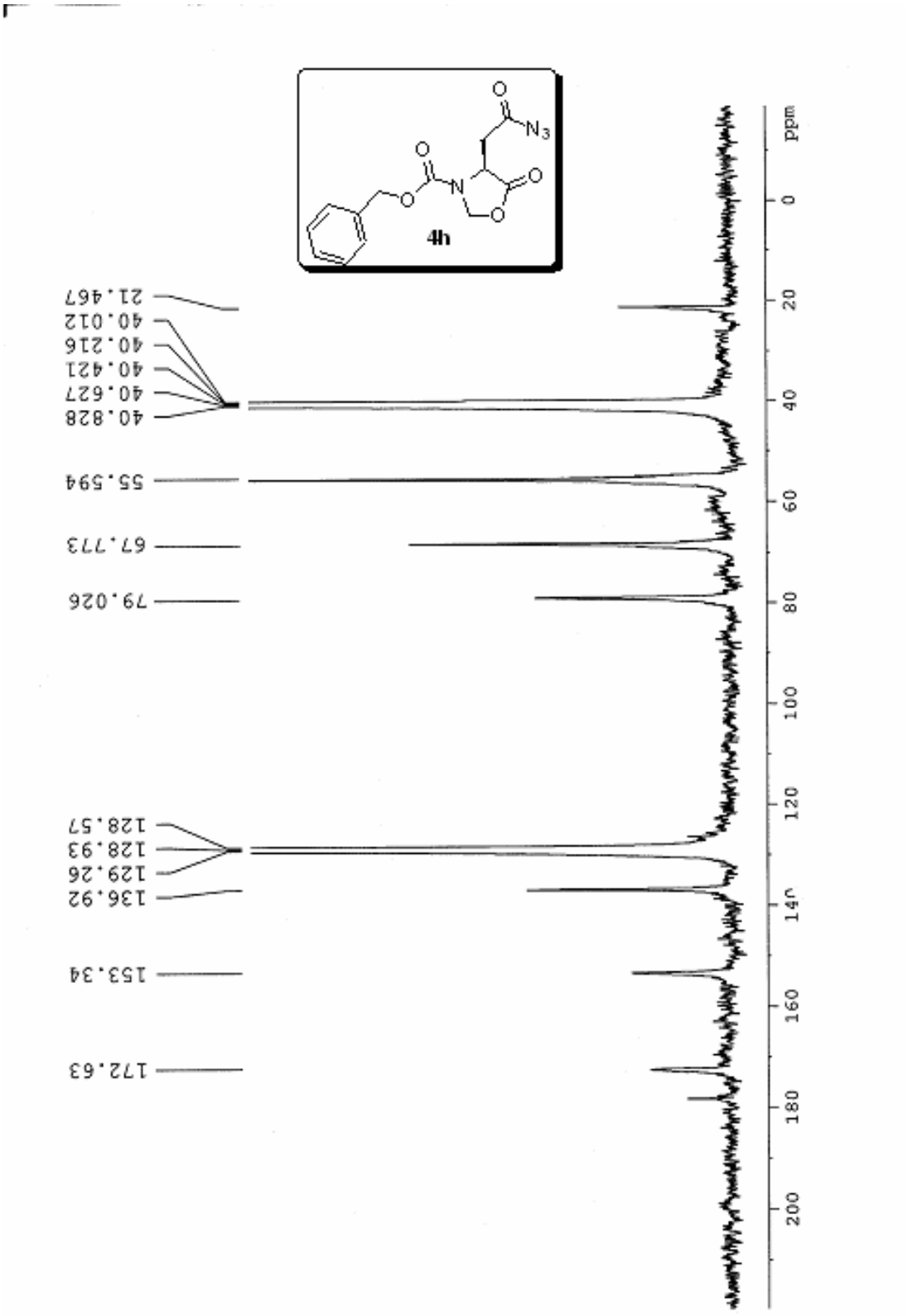

4e
26.103
47.526
53.951
68.454
77.243
77.561
77.878
125.427
125.776
126.837
127.627
128.327
129.146
129.576
136.698
141.816
144.094
157.977
179.176
H11
ppm
0
20
40
60
80
100
120
140
160
180
200

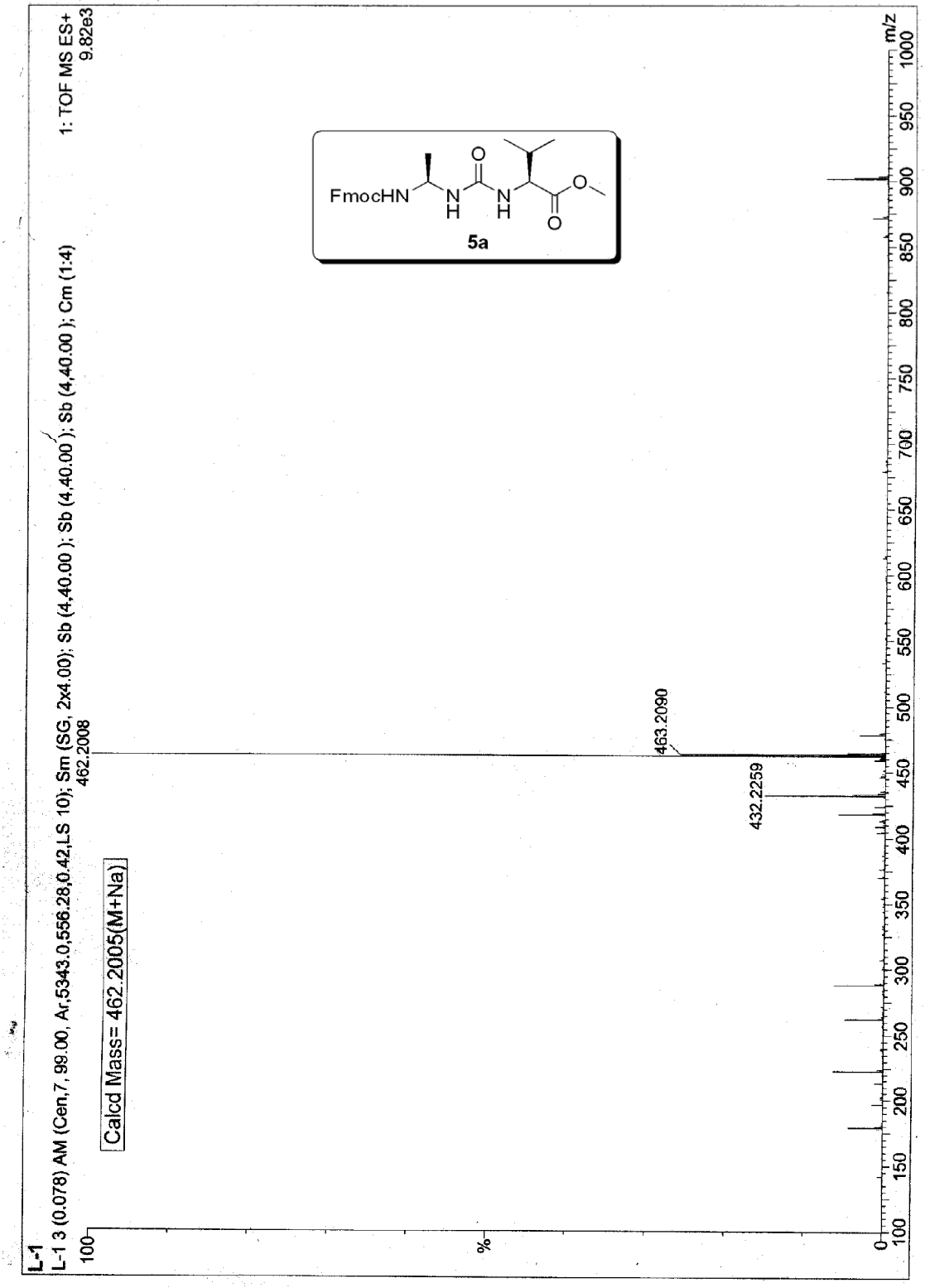

HS-3
HS-3 14 (0.320) AM (Cen,7, 99.00, Ar,5343.0,556.28,0.10,LS 10); Sm (SG, 2x4.00); Sb (4,40.00); Sb (4,40.00); Sb (4,40.00); Cm (11:15
2.32e4
Calcd Mass = 385.1277 (M+Na)
385.1133
413.2593
579.1776
243.0573
4f
%
m/z

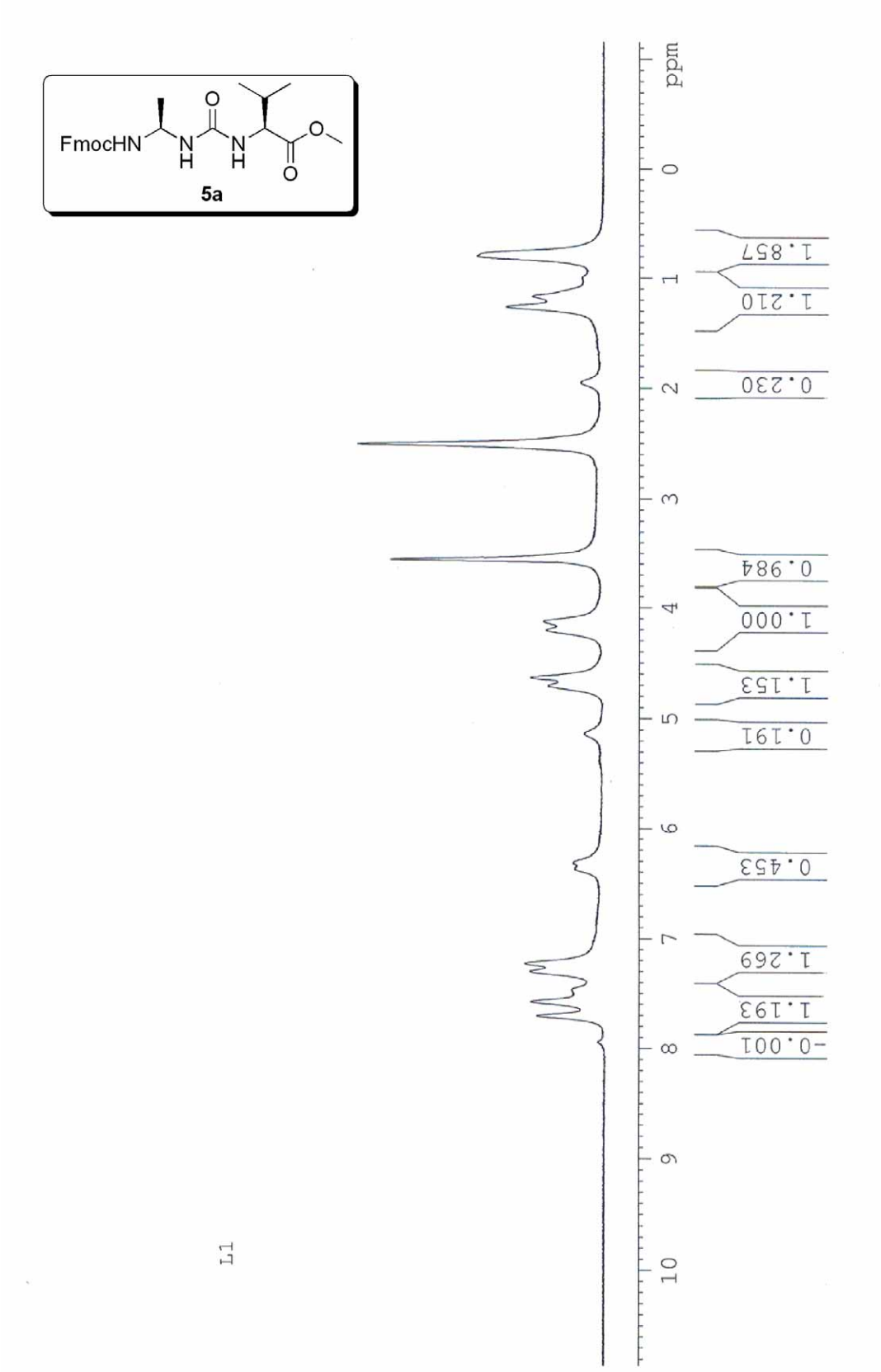

HS-5
HS-5 9 (0.229) AM (Cen,7, 99.00, Ar,5343.0,556.28,0.01,LS 10); Sb (6,40.00); Sb (6,40.00); Sb (6,40.00); Sb (6,40.00); Sb (6,40.00 1: TOF MS ES+
6.39e3
Calcd Mass = 347.1120 (M+Na)
4g
347.1098
342.1570
365.1470
210.1279
%
m/z

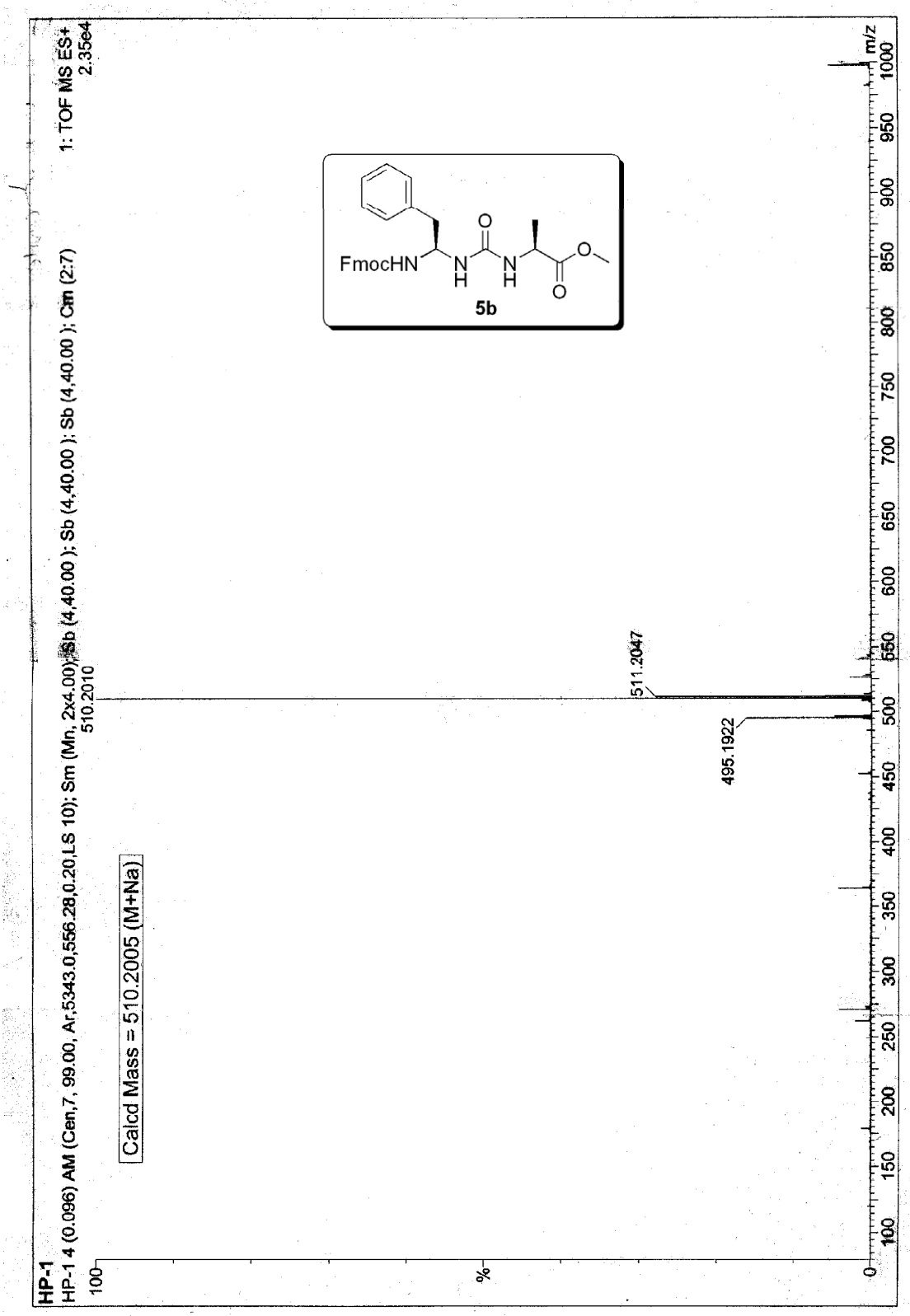

4g
0 ppm
1
2
3
4
5
6
7
8
9
0.67
0.51
0.44
1.87
1.00
5.59
HS-5

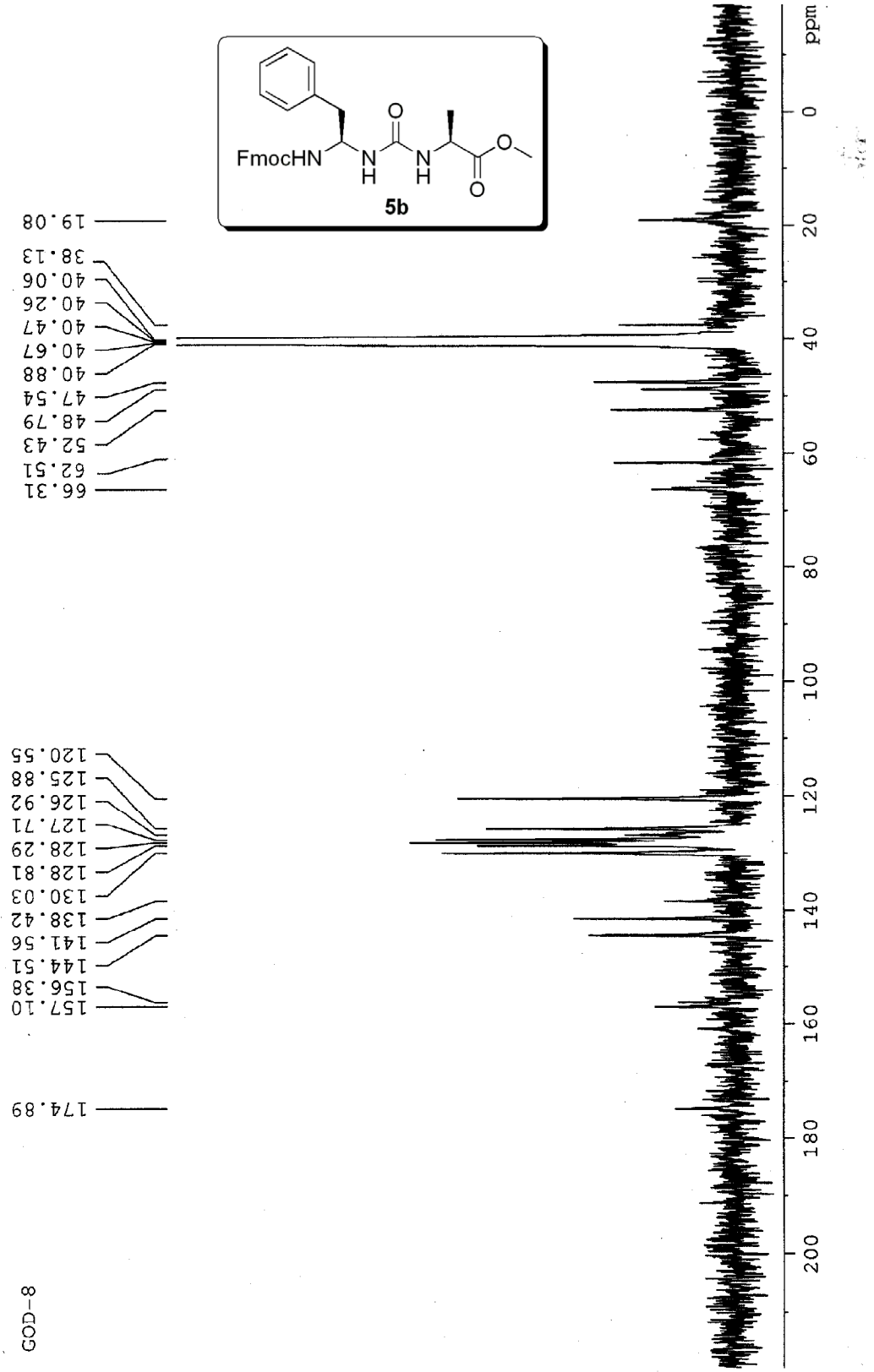

4g
O
N
H
O
O
N_3
ppm
0
20
40
60
80
100
120
140
160
180
200
38.126
57.221
67.666
77.490
77.808
78.127
128.065
128.636
128.792
129.090
129.322
129.767
135.933
136.699
156.356
179.807
HS-5

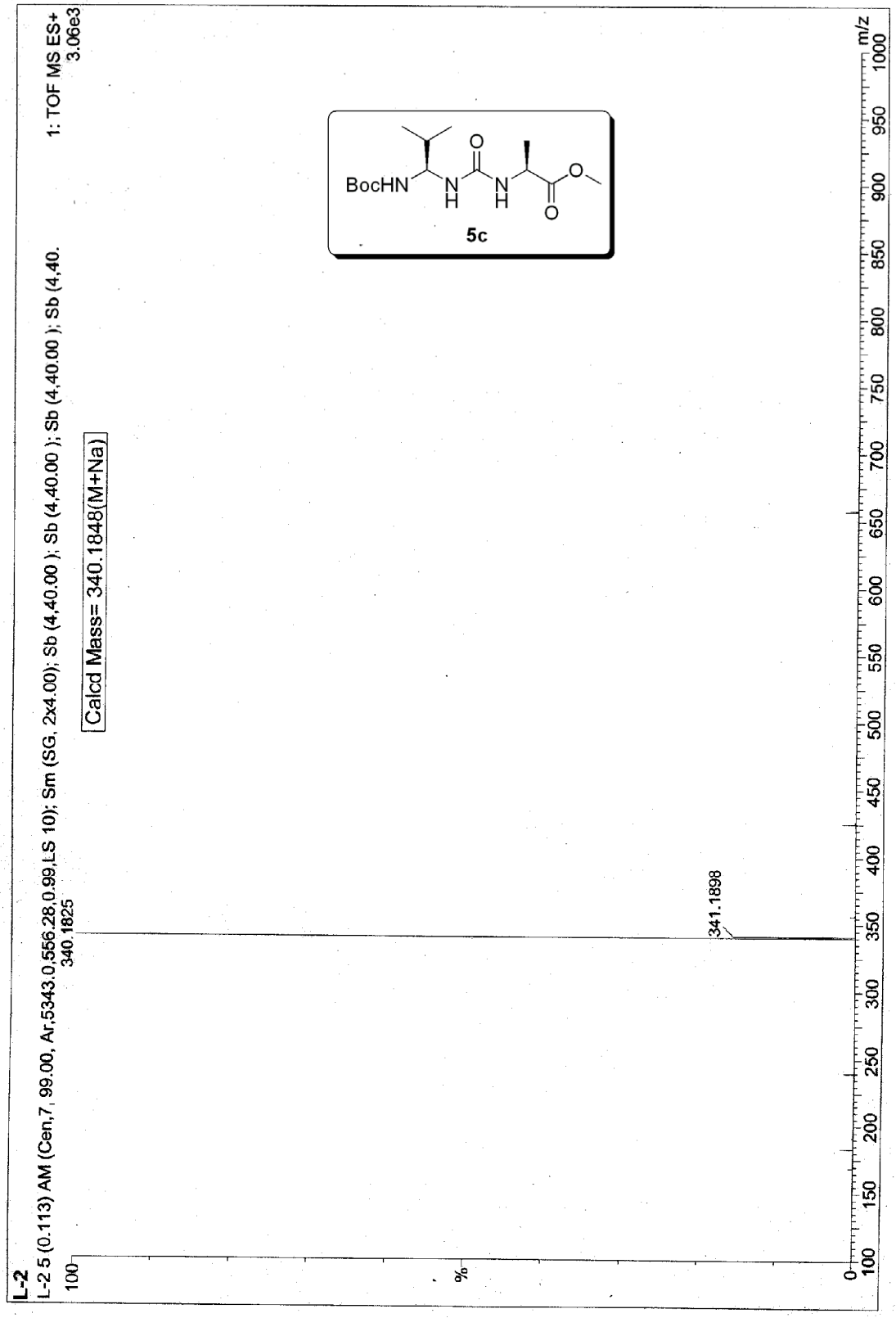

4h
21.467
40.012
40.216
40.421
40.627
40.828
55.594
67.773
79.026
128.57
128.93
129.26
136.92
153.34
172.63
ppm
0
20
40
60
80
100
120
140
160
180
200

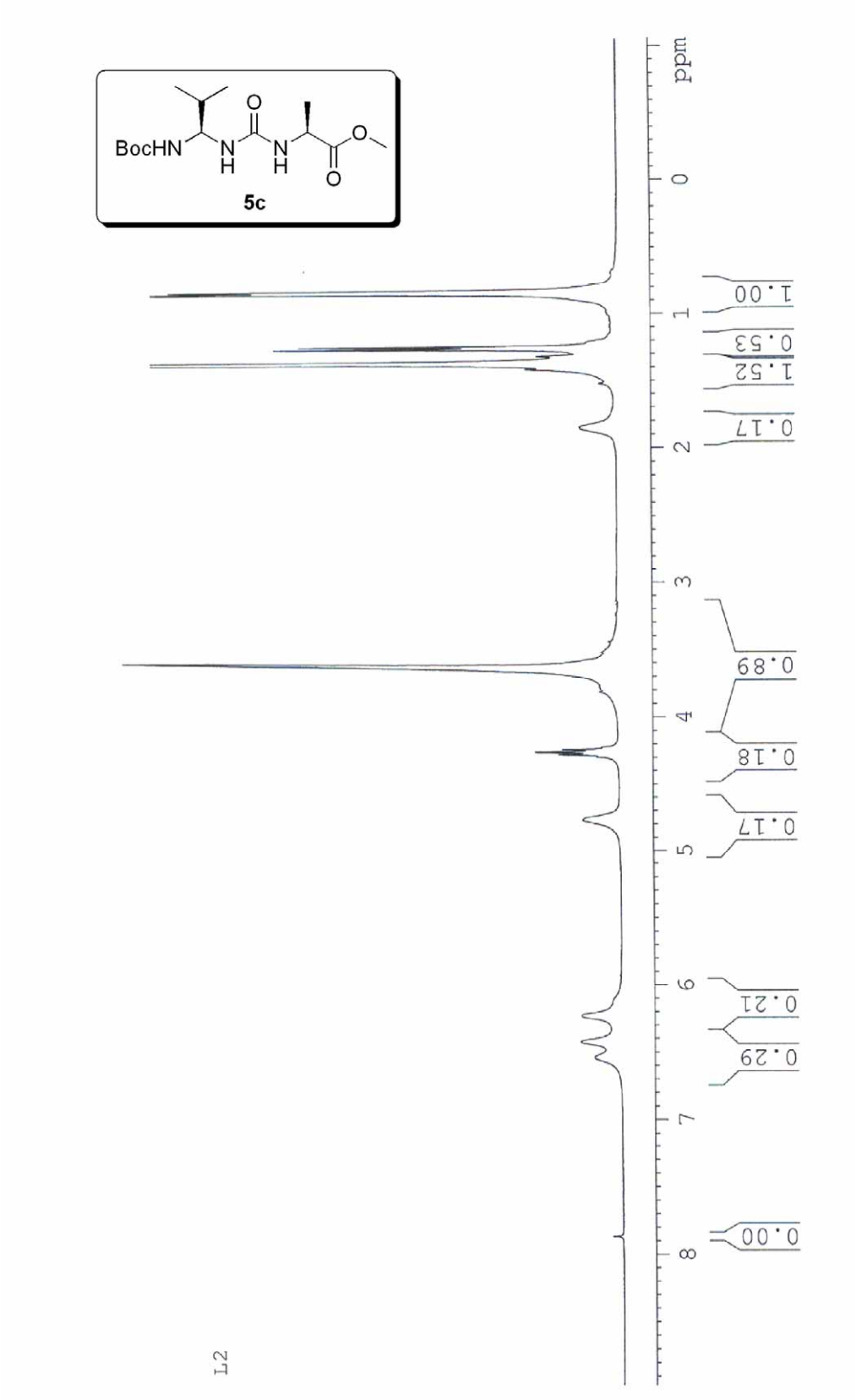

L-1
L-1 3 (0.078) AM (Cen,7, 99.00, Ar,5343.0,556.28,0.42,LS 10); Sm (SG, 2x4.00); Sb (4,40.00); Sb (4,40.00); Sb (4,40.00); Cm (1:4)
1: TOF MS ES+
9.82e3
462.2008
463.2090
432.2259
Calcd Mass= 462.2005(M+Na)
FmocHN
5a
%
m/z

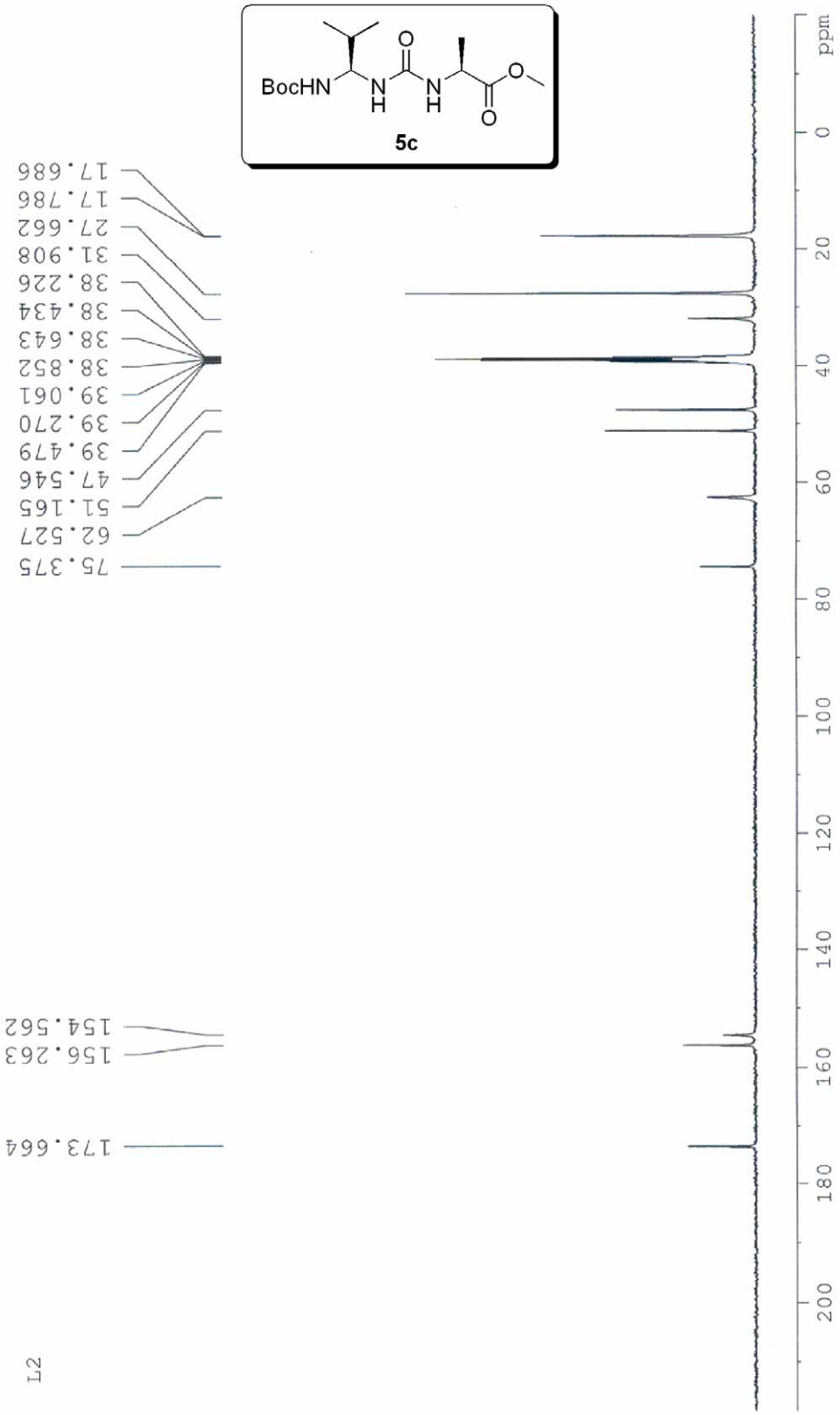

FmocHN
N
H
O
N
H
O
O
O
5a
ppm
0
1
2
3
4
5
6
7
8
9
10
1.857
1.210
0.230
0.984
1.000
1.153
0.191
0.453
1.269
1.193
-0.001

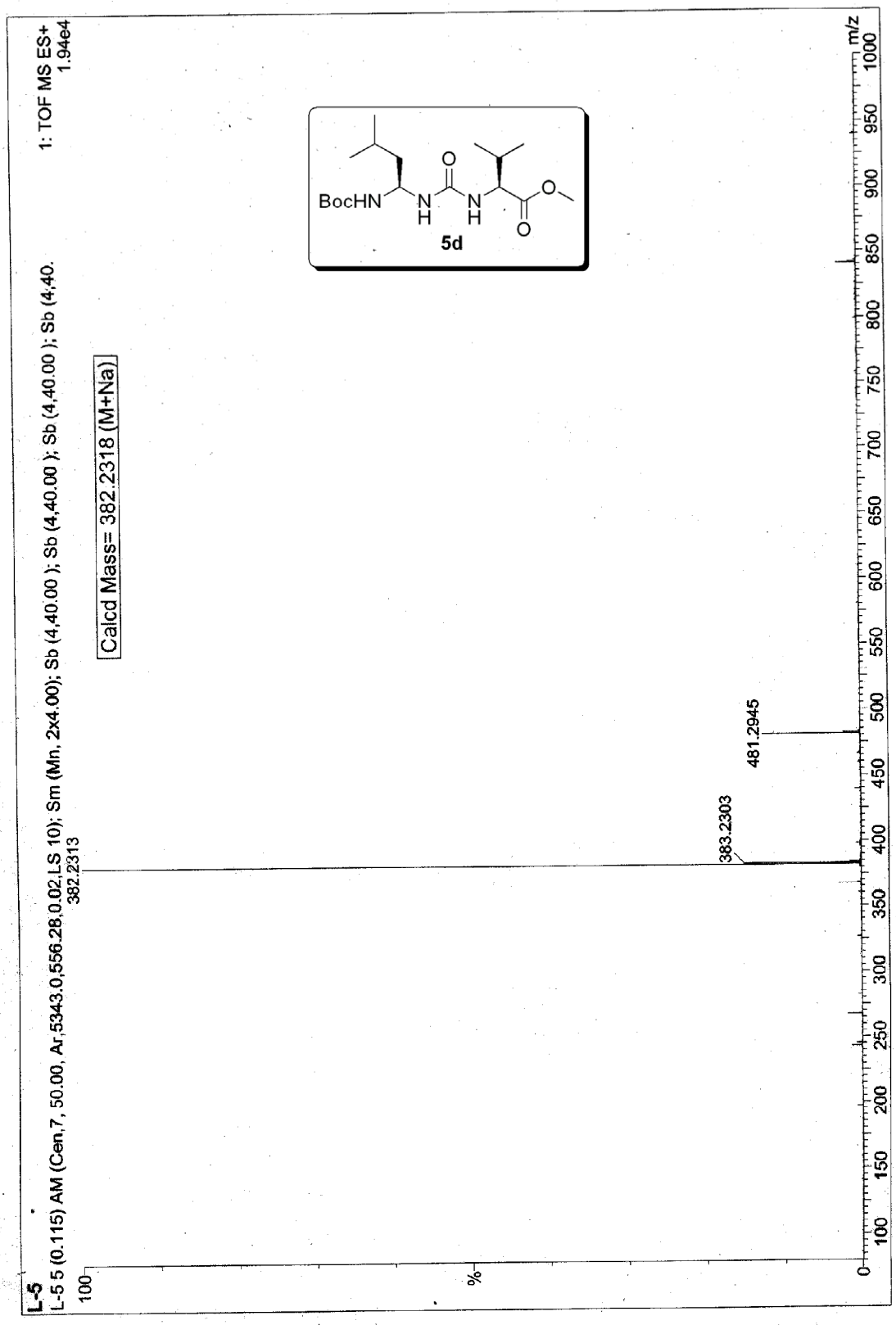

HP-1
HP-1 4 (0.096) AM (Cen,7, 99.00, Ar,5343.0,556.28,0.20,LS 10); Sm (Mn, 2x4.00); Sb (4,40.00); Sb (4,40.00); Sb (4,40.00); Cm (2:7)
1: TOF MS ES+
2.35e4
100
%
0
510.2010
511.2047
495.1922
Calcd Mass = 510.2005 (M+Na)
FmocHN
N
H
O
N
H
O
O
5b
m/z
100
150
200
250
300
350
400
450
500
550
600
650
700
750
800
850
900
950
1000

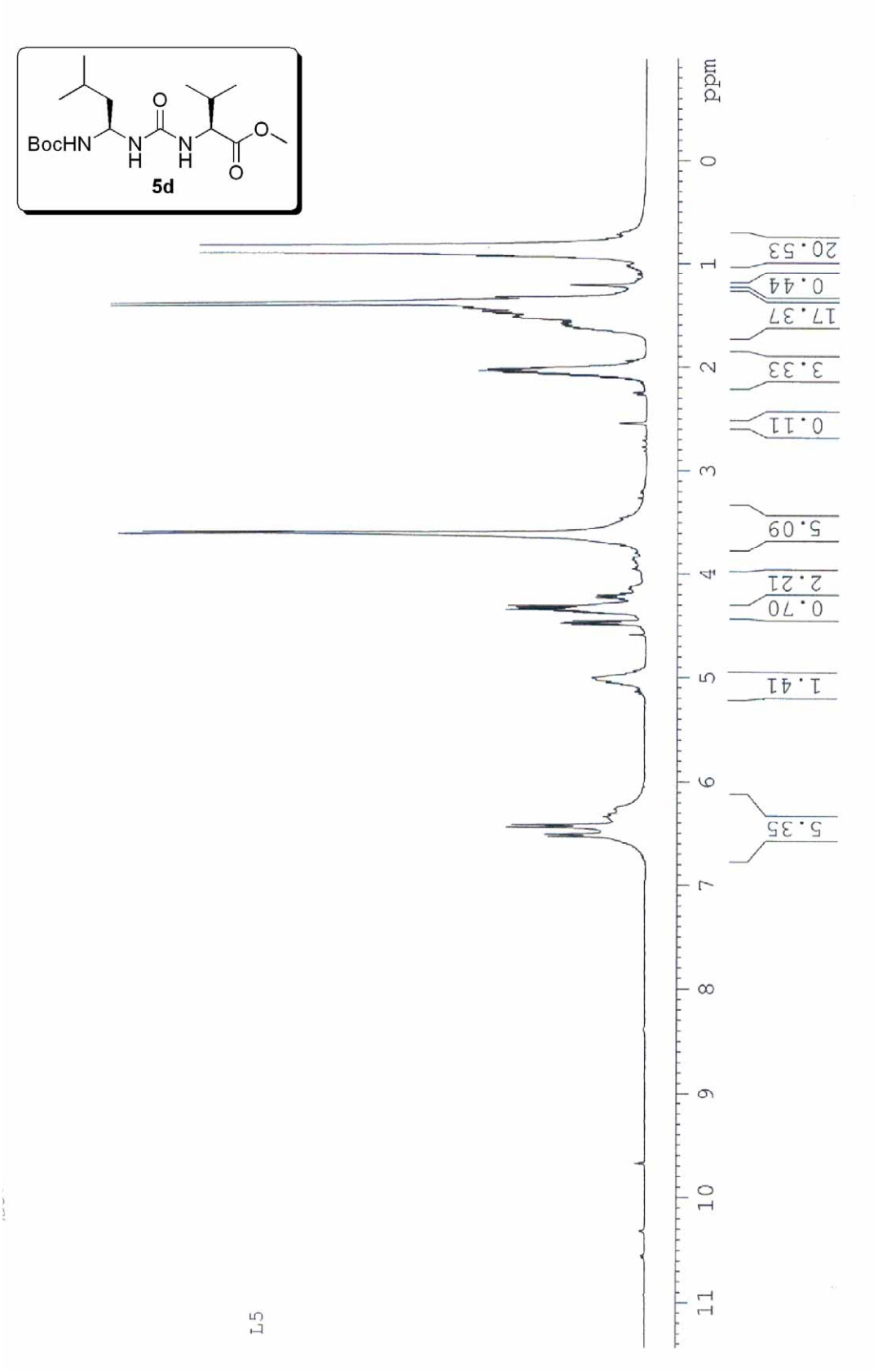

FmocHN
N
H
N
H
O
O
O
5b
ppm
0
20
40
60
80
100
120
140
160
180
200
19.08
38.13
40.06
40.26
40.47
40.67
40.88
47.54
48.79
52.43
62.51
66.31
120.55
125.88
126.92
127.71
128.29
128.81
130.03
138.42
141.56
144.51
156.38
157.10
174.89
GOD-8

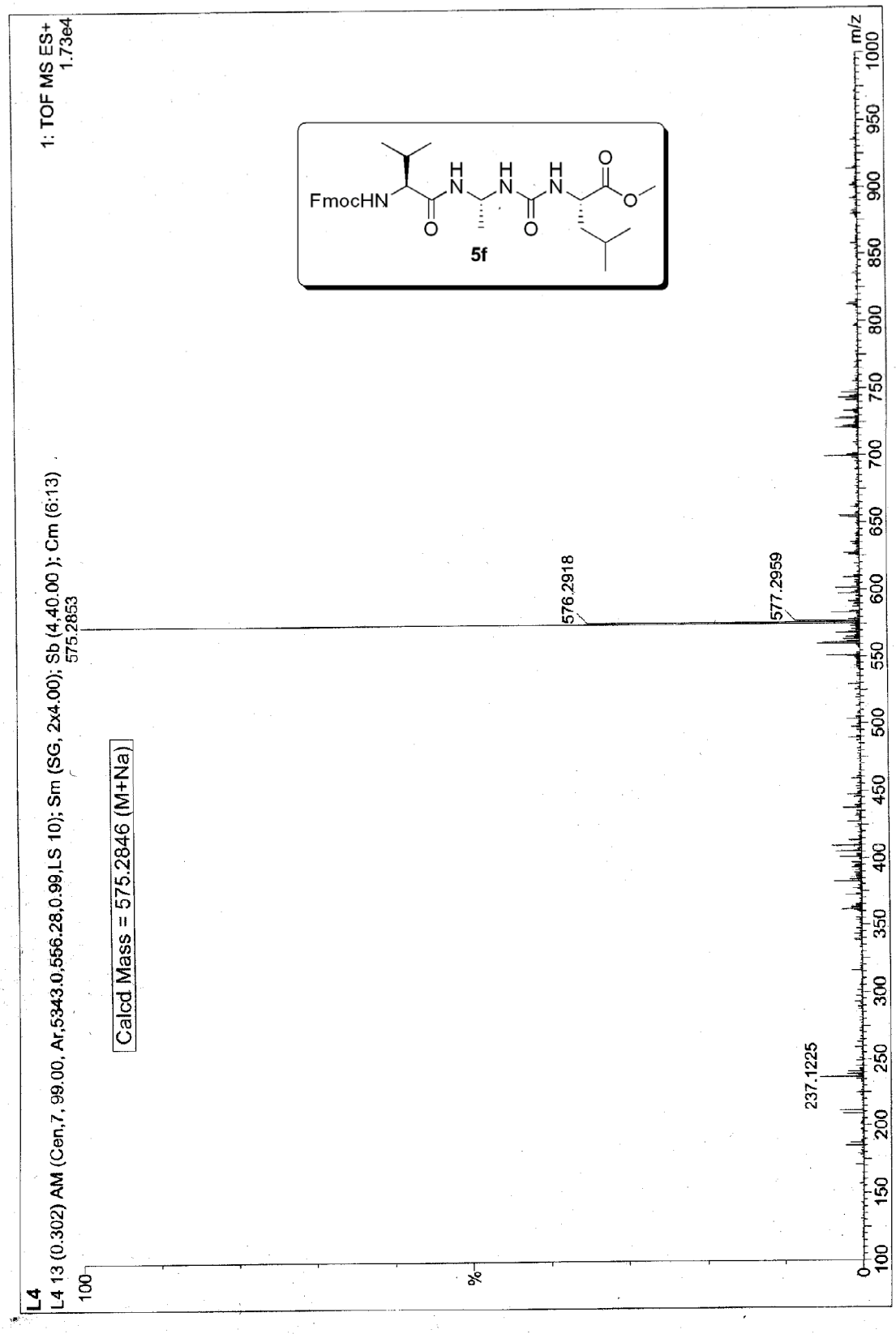

L-2
L-2 5 (0.113) AM (Cen,7, 99.00, Ar,5343.0,556.28,0.99,LS 10); Sm (SG, 2x4.00); Sb (4,40.00); Sb (4,40.00); Sb (4,40.00); Sb (4,40.
1: TOF MS ES+
3.06e3
Calcd Mass= 340.1848(M+Na)
340.1825
341.1898
BocHN
5c
%
m/z

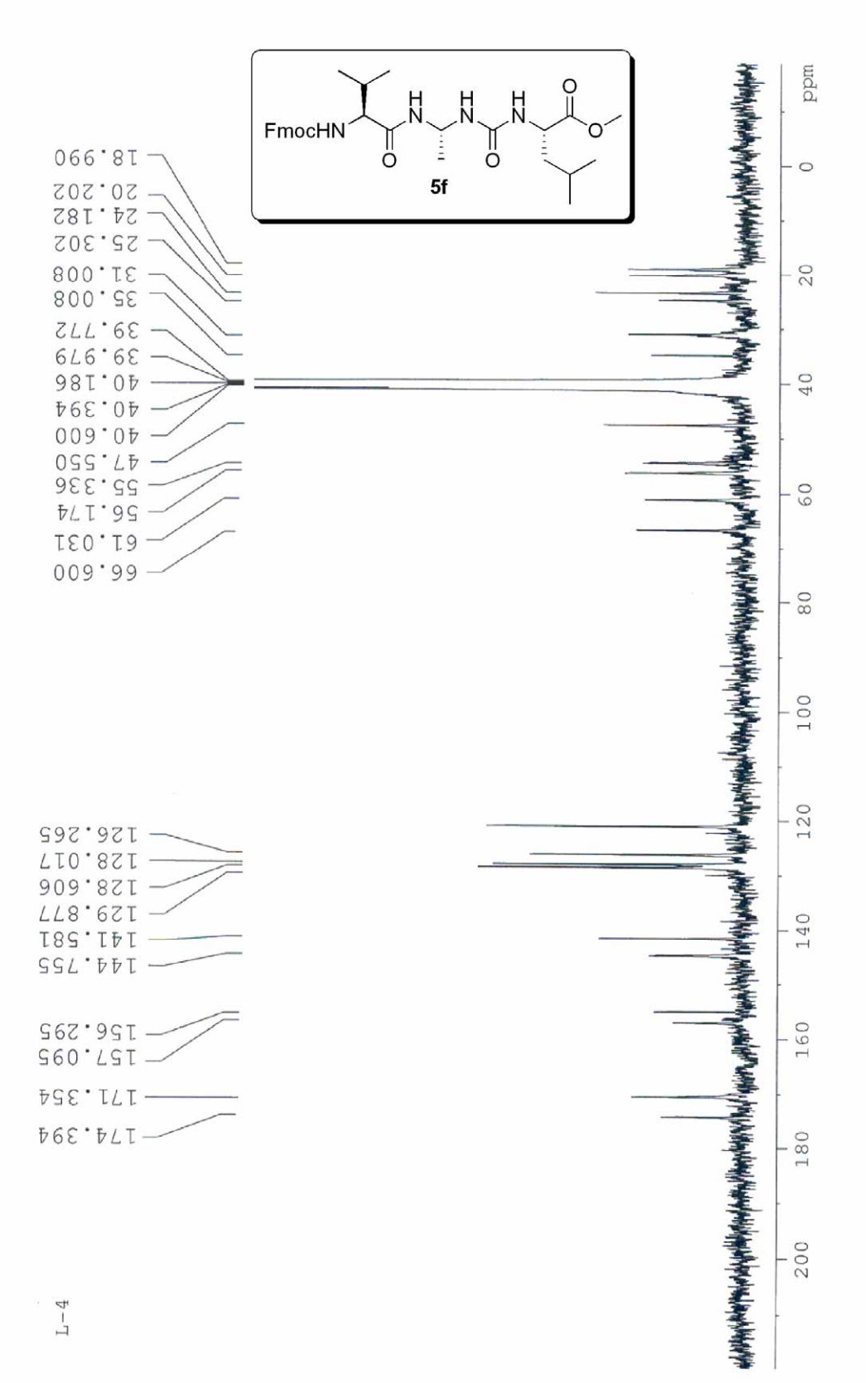

BocHN
N
H
O
N
H
O
O
5c
ppm
0
1
2
3
4
5
6
7
8
1.00
0.53
1.52
0.17
0.89
0.18
0.17
0.21
0.29
0.00
L2

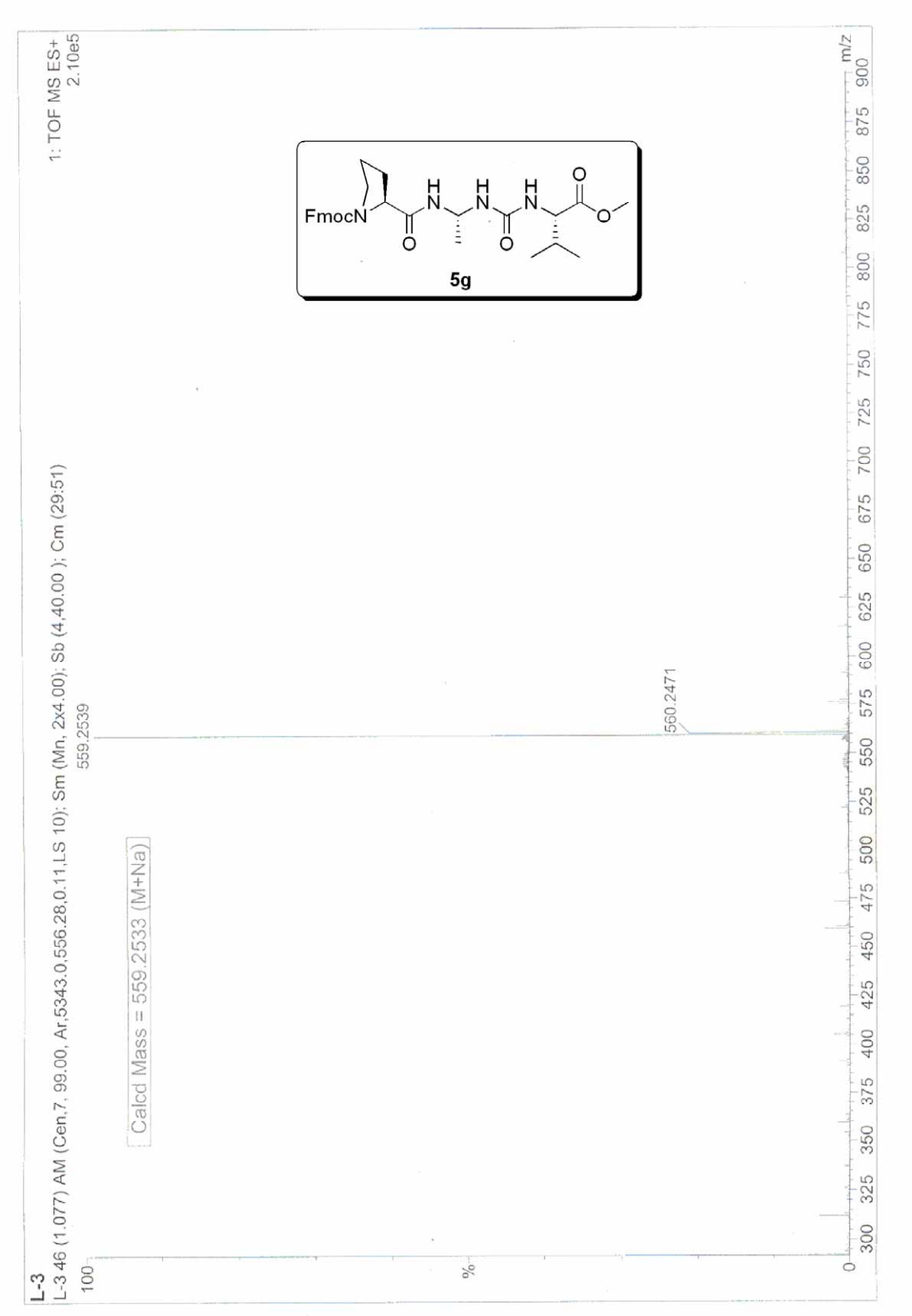

BocHN
N
H
O
N
H
O
O
5c
17.686
17.786
27.662
31.908
38.226
38.434
38.643
38.852
39.061
39.270
39.479
47.546
51.165
62.527
75.375
154.562
156.263
173.664
L2
ppm
0
20
40
60
80
100
120
140
160
180
200

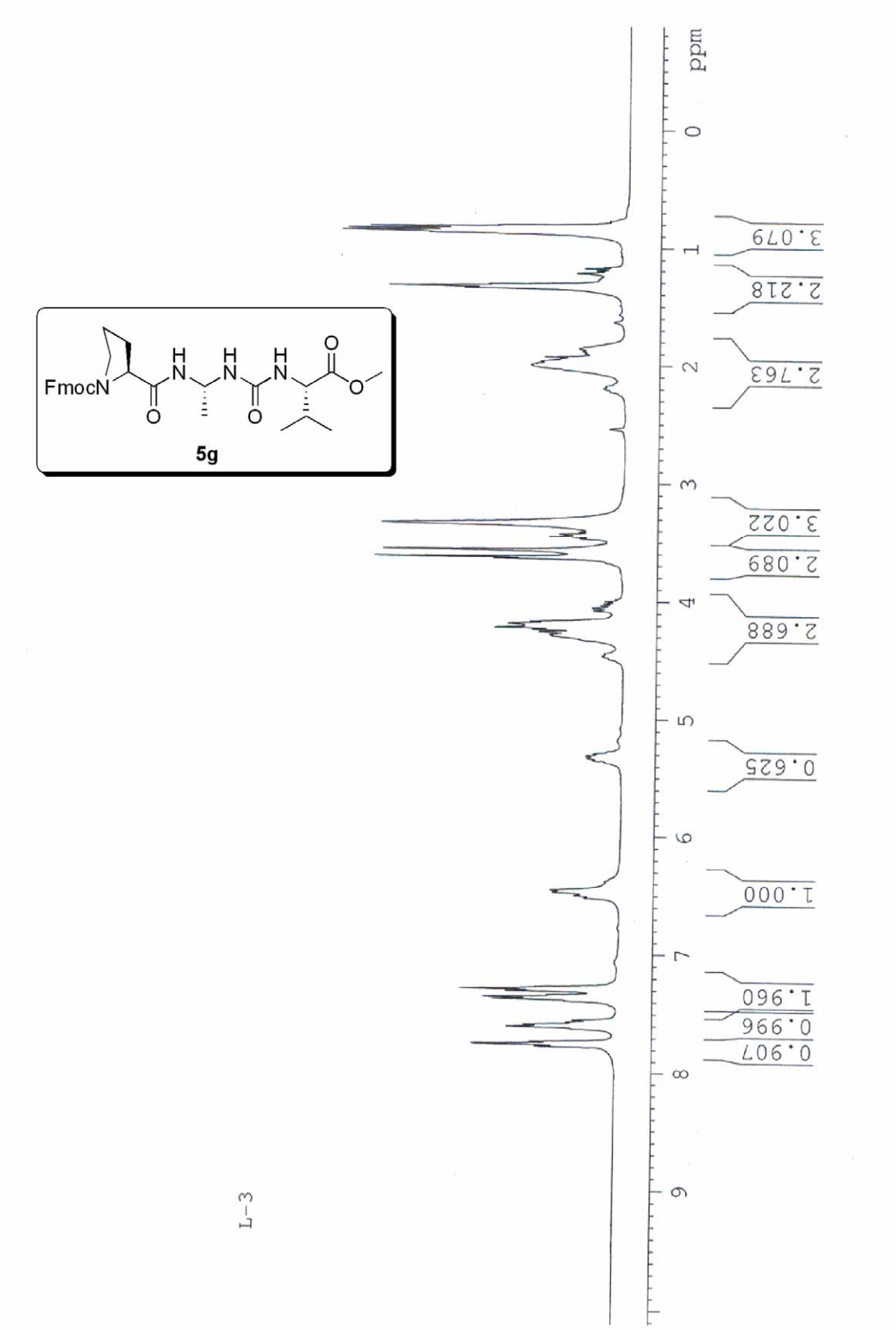

L-5
L-5 5 (0.115) AM (Cen,7, 50.00, Ar,5343.0,556.28,0.02,LS 10); Sm (Mn, 2x4.00); Sb (4,40.00); Sb (4,40.00); Sb (4,40.00); Sb (4,40.
1: TOF MS ES+
1.94e4
Calcd Mass= 382.2318 (M+Na)
382.2313
383.2303
481.2945
BocHN
5d
%
m/z

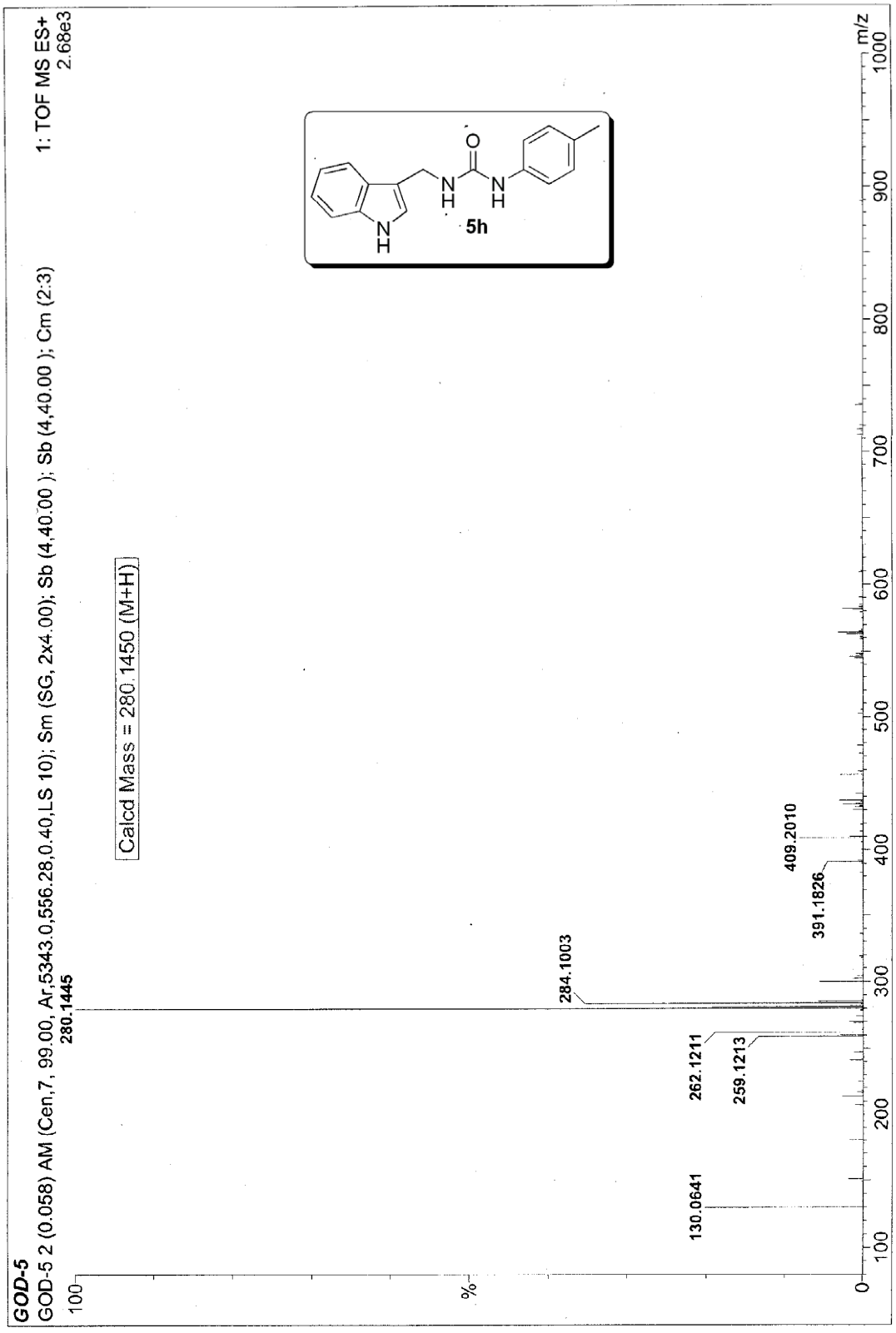

BocHN
N
H
O
N
H
O
O
5d
ppm
0
1
2
3
4
5
6
7
8
9
10
11
20.53
0.44
17.37
3.33
0.11
5.09
2.21
0.70
1.41
5.35
L5

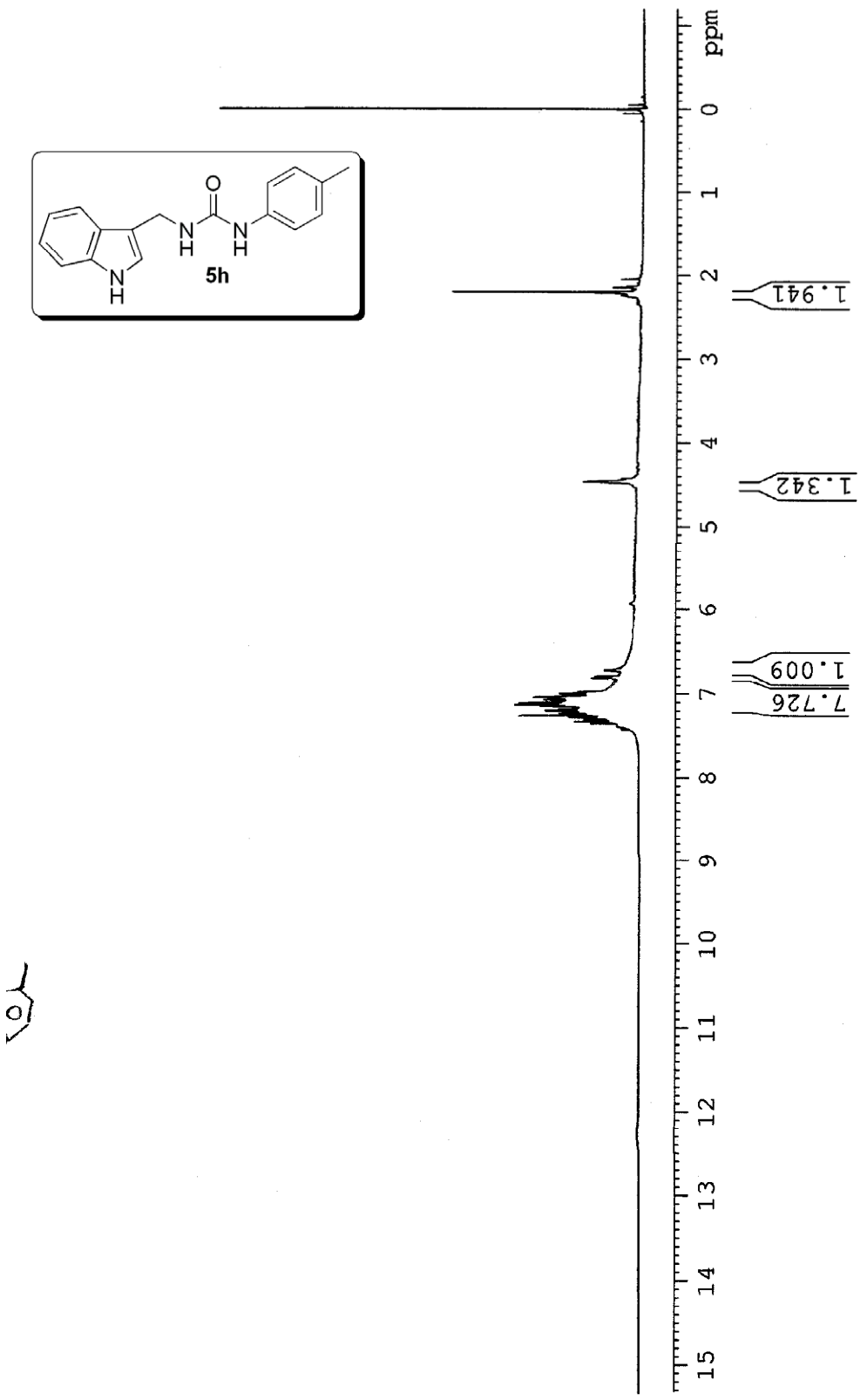

L4
L4 13 (0.302) AM (Cen,7, 99.00, Ar,5343.0,556.28,0.99,LS 10); Sm (SG, 2x4.00); Sb (4,40.00); Cm (6:13)
1: TOF MS ES+
1.73e4
FmocHN
5f
Calcd Mass = 575.2846 (M+Na)
575.2853
576.2918
577.2959
237.1225
%
m/z

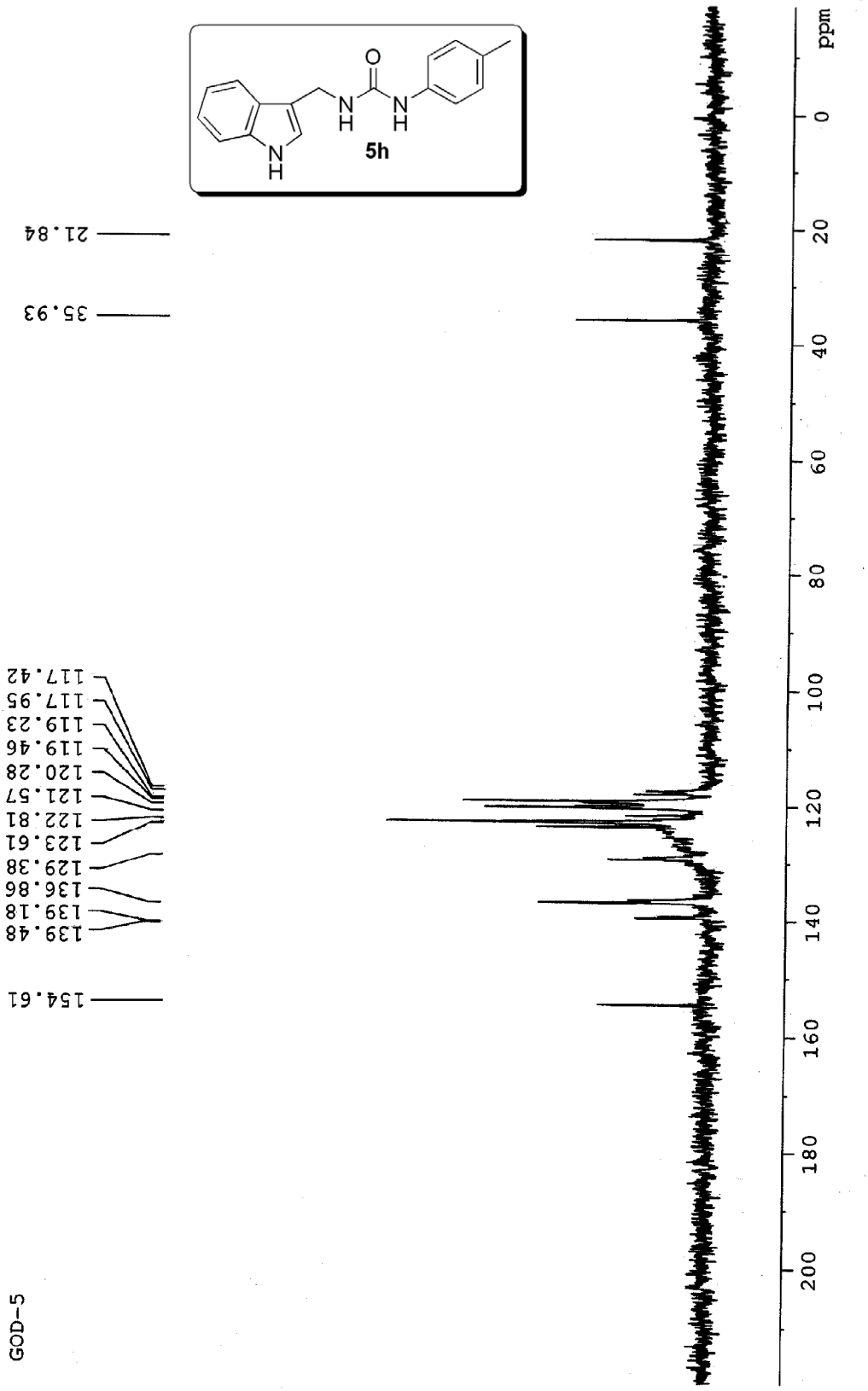

FmocHN
5f
18.990
20.202
24.182
25.302
31.008
35.008
39.772
39.979
40.186
40.394
40.600
47.550
55.336
56.174
61.031
66.600
126.265
128.017
128.606
129.877
141.581
144.755
156.295
157.095
171.354
174.394
ppm
0
20
40
60
80
100
120
140
160
180
200
L-4

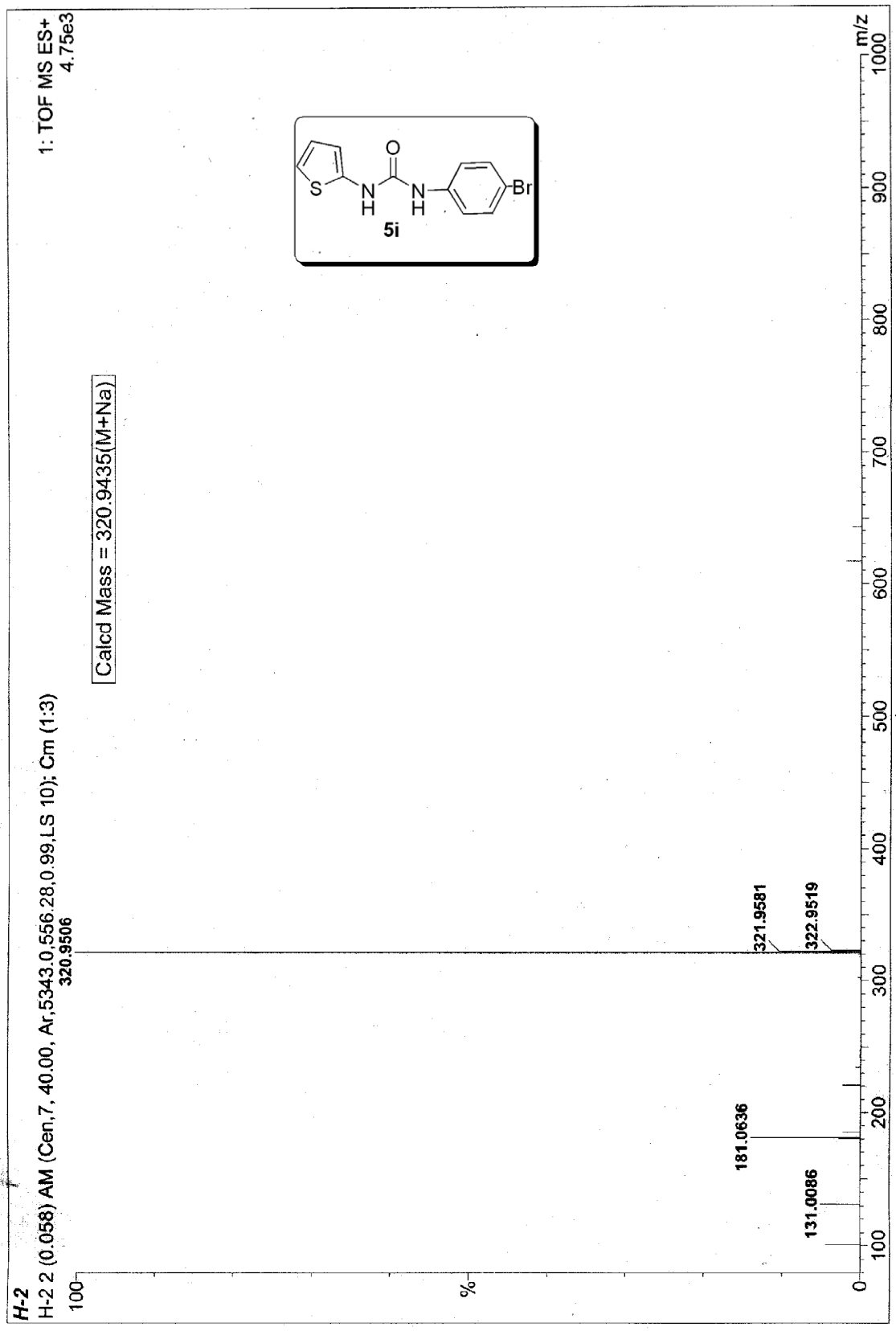

L-3
L-3 46 (1.077) AM (Cen,7, 99.00, Ar,5343.0,556.28,0.11,LS 10); Sm (Mn, 2x4.00); Sb (4,40.00); Cm (29:51)
1: TOF MS ES+
2.10e5
100
%
0
559.2539
560.2471
Calcd Mass = 559.2533 (M+Na)
FmocN
H
N
O
5g
m/z
300 325 350 375 400 425 450 475 500 525 550 575 600 625 650 675 700 725 750 775 800 825 850 875 900

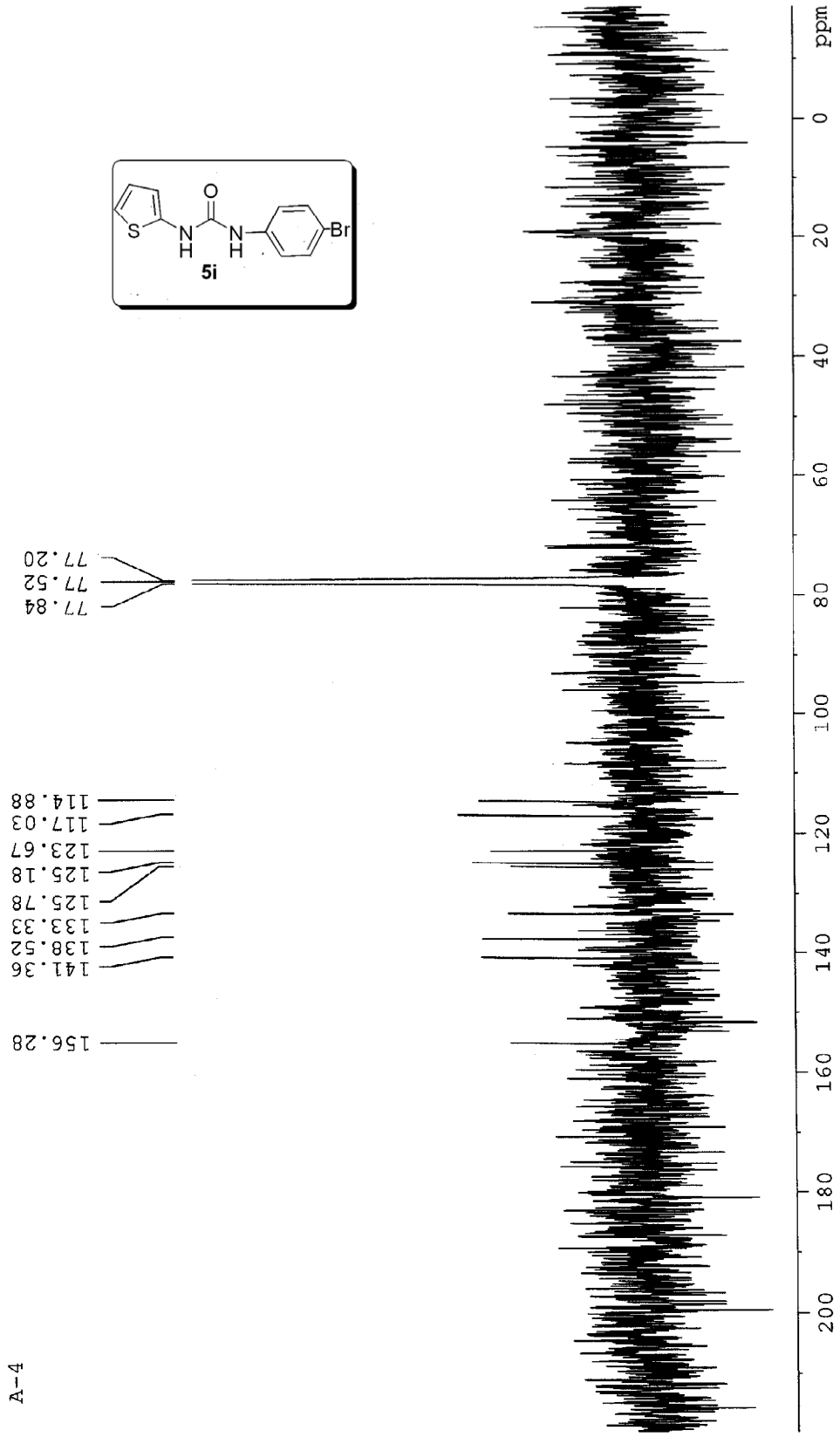

FmocN
H
N
O
5g
ppm
0
1
2
3
4
5
6
7
8
9
3.079
2.218
2.763
3.022
2.089
2.688
0.625
1.000
1.960
0.996
0.907
L-3

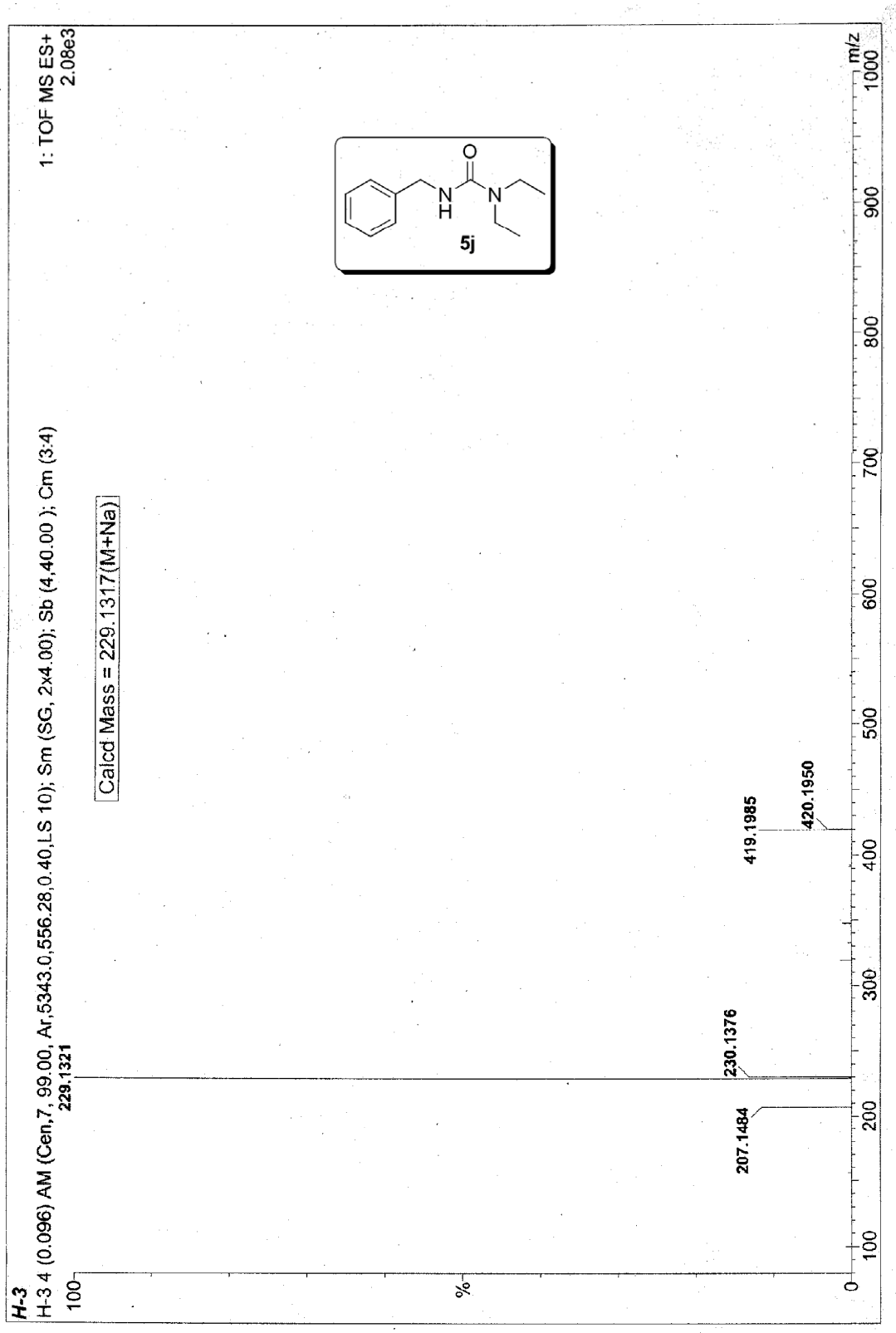

GOD-5
GOD-5 2 (0.058) AM (Cen,7, 99.00, Ar,5343.0,556.28,0.40,LS 10); Sm (SG, 2x4.00); Sb (4,40.00); Sb (4,40.00); Cm (2:3)
1: TOF MS ES+
2.68e3
Calcd Mass = 280.1450 (M+H)
5h
280.1445
284.1003
391.1826
409.2010
262.1211
259.1213
130.0641
%
m/z

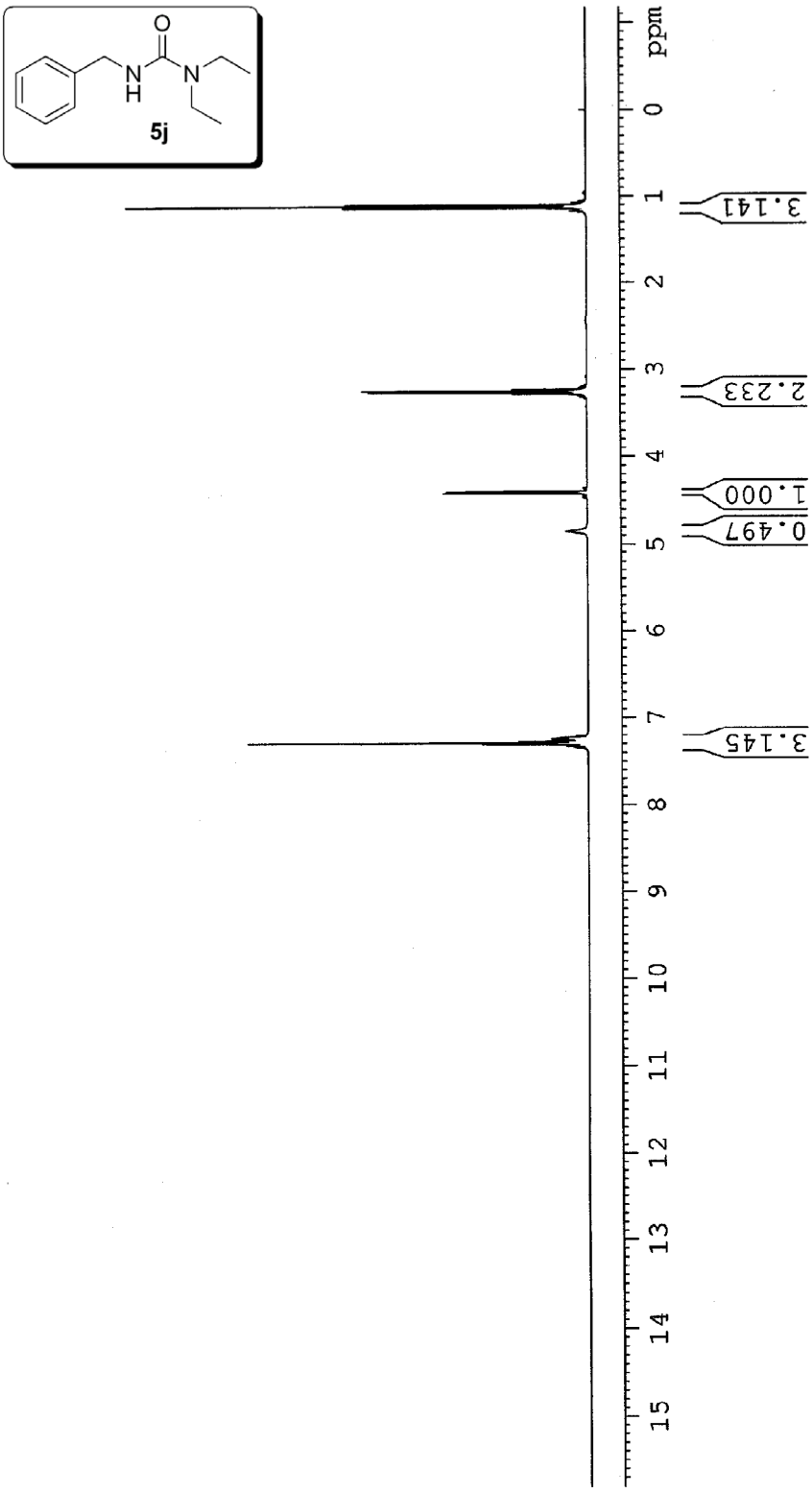

5h
ppm
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
1.941
1.342
1.009
7.726

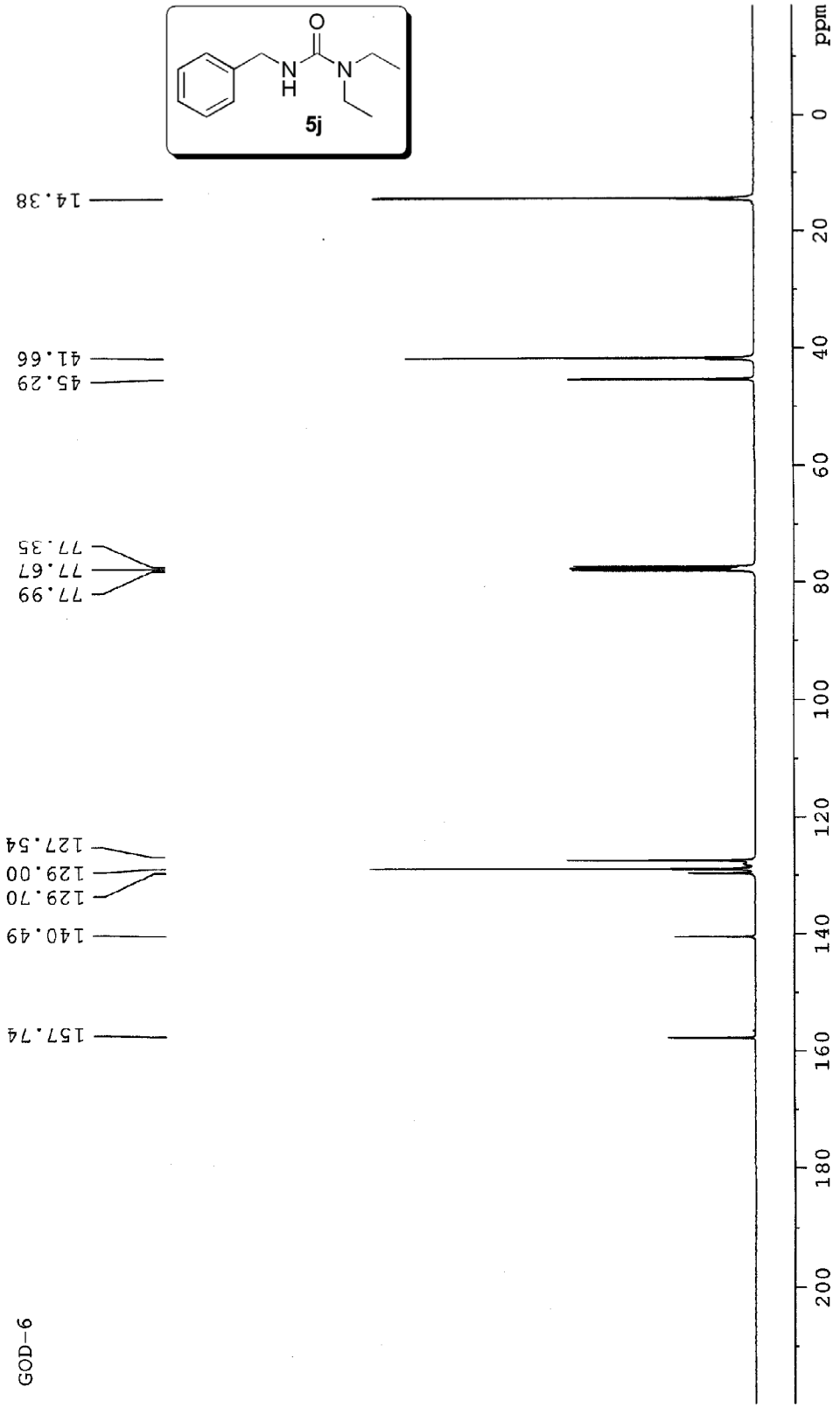

5h
21.84
35.93
117.42
117.95
119.23
119.46
120.28
121.57
122.81
123.61
129.38
136.86
139.18
139.48
154.61
GOD-5
ppm
0
20
40
60
80
100
120
140
160
180
200

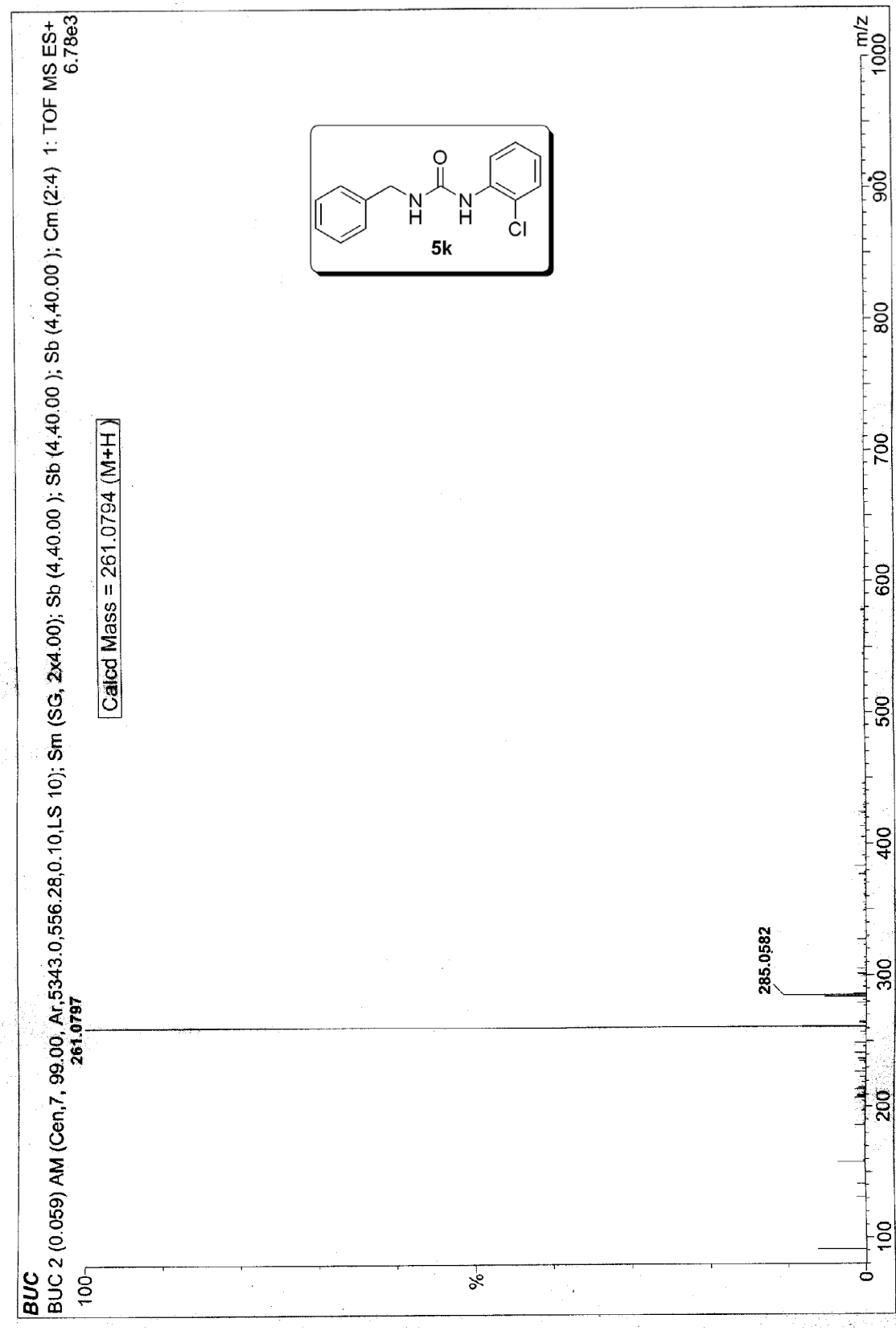

H-2
H-2 2 (0.058) AM (Cen,7, 40.00, Ar,5343.0,556.28,0.99,LS 10); Cm (1:3)
1: TOF MS ES+
4.75e3
Calcd Mass = 320.9435(M+Na)
5i
320.9506
321.9581
322.9519
181.0636
131.0086
%
m/z

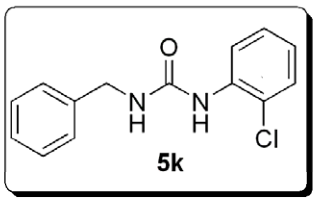

5i
S
N
H
O
N
H
Br
ppm
0
20
40
60
80
100
120
140
160
180
200
77.20
77.52
77.84
114.88
117.03
123.67
125.18
125.78
133.33
138.52
141.36
156.28
A-4

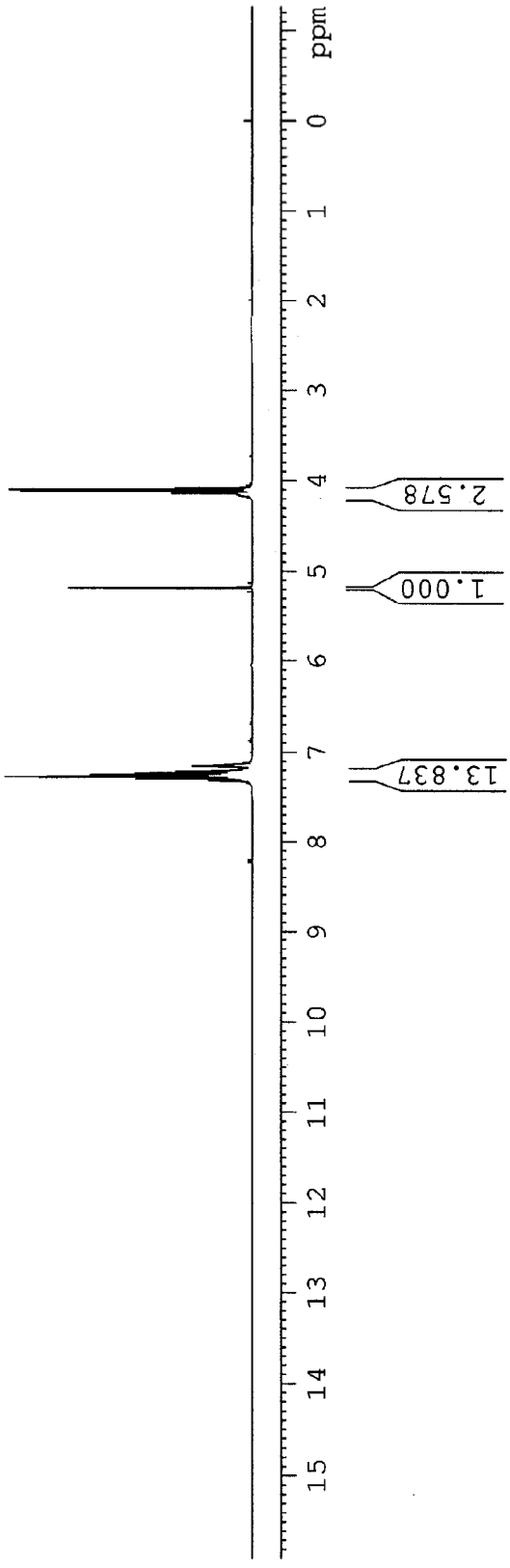

H-3
H-3 4 (0.096) AM (Cen,7, 99.00, Ar,5343.0,556.28,0.40,LS 10); Sm (SG, 2x4.00); Sb (4,40.00); Cm (3:4)
1: TOF MS ES+
2.08e3
Calcd Mass = 229.1317(M+Na)
5j
229.1321
230.1376
207.1484
419.1985
420.1950
%
m/z

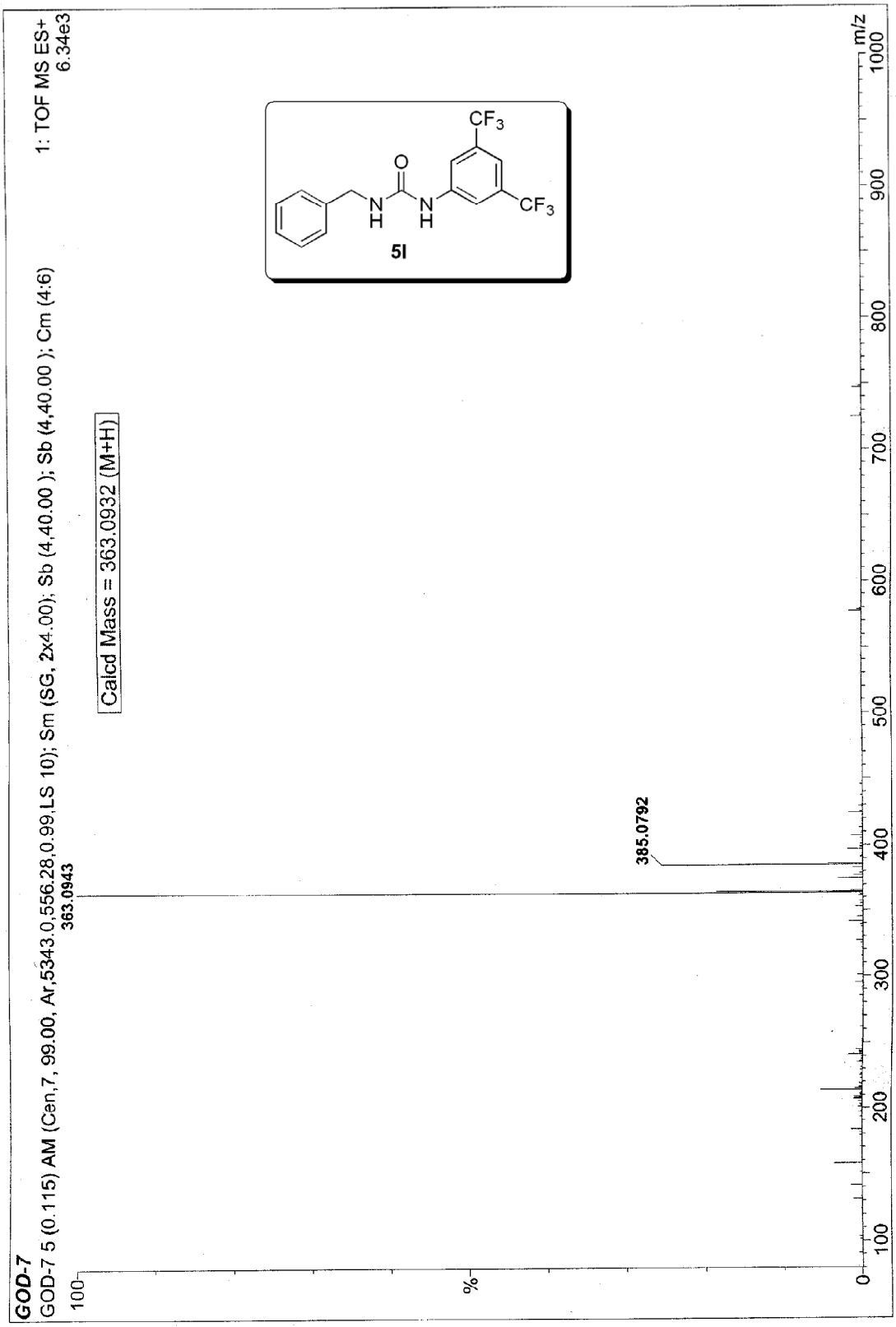

O
N
H
N
5j
ppm
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
3.141
2.233
1.000
0.497
3.145

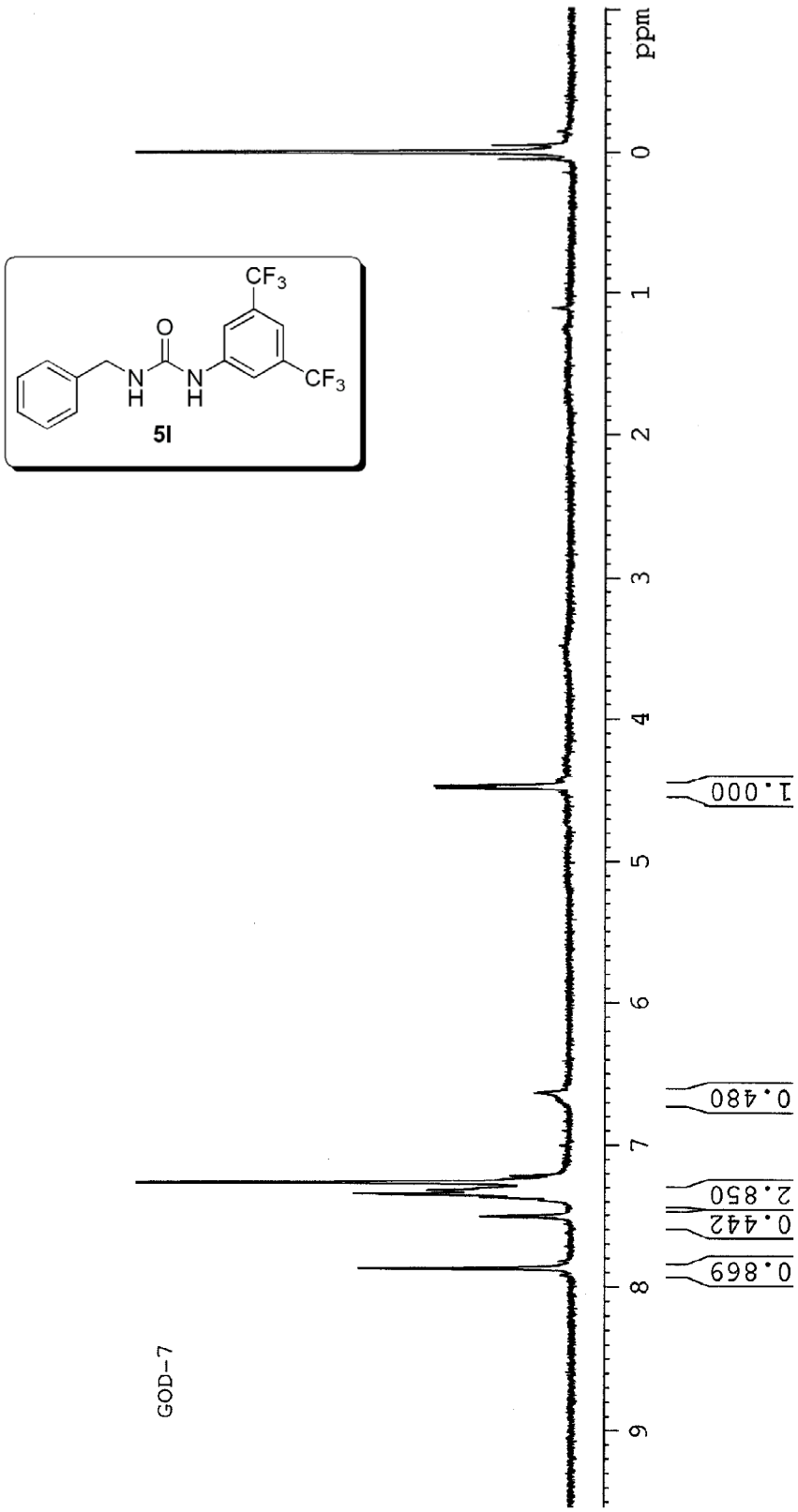

5j
14.38
41.66
45.29
77.35
77.67
77.99
127.54
129.00
129.70
140.49
157.74
GOD-6
ppm
0
20
40
60
80
100
120
140
160
180
200

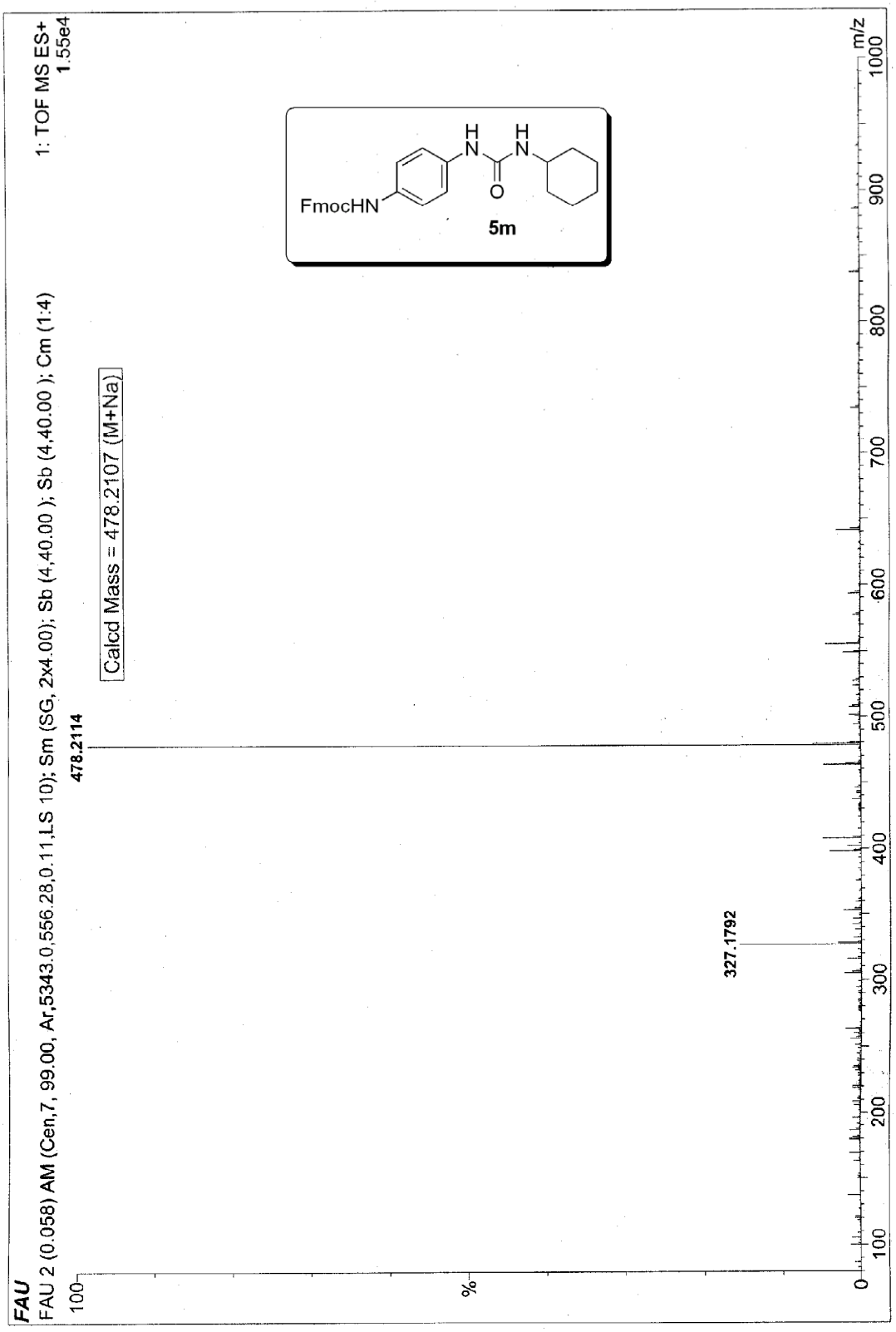

BUC
BUC 2 (0.059) AM (Cen,7, 99.00, Ar,5343.0,556.28,0.10,LS 10); Sm (SG, 2x4.00); Sb (4,40.00); Sb (4,40.00); Sb (4,40.00); Cm (2:4) 1: TOF MS ES+
6.78e3
Calcd Mass = 261.0794 (M+H)
261.0797
285.0582
5k
Cl
%
m/z

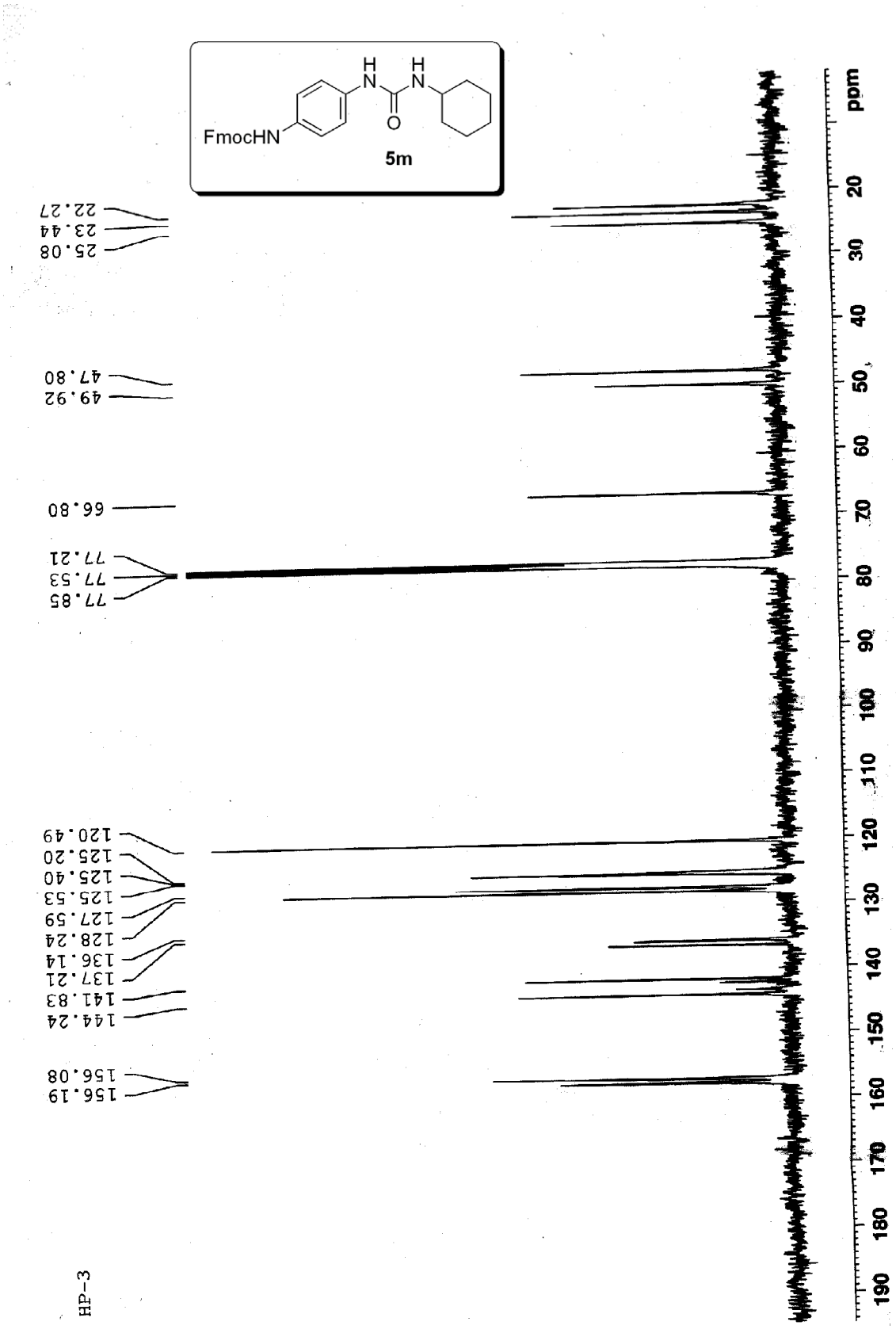

O
N
H
N
H
Cl
5k

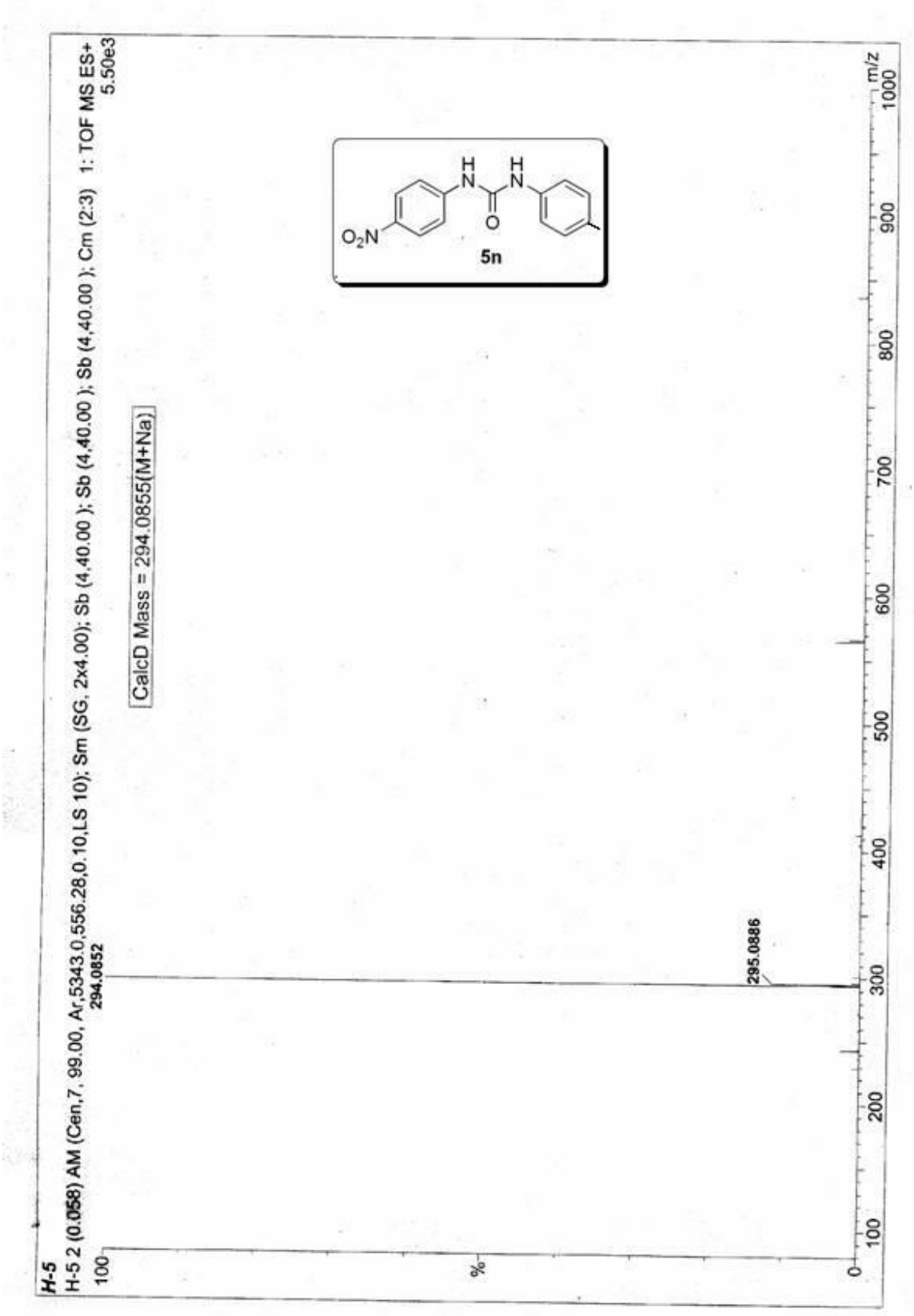

ppm
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
2.578
1.000
13.837

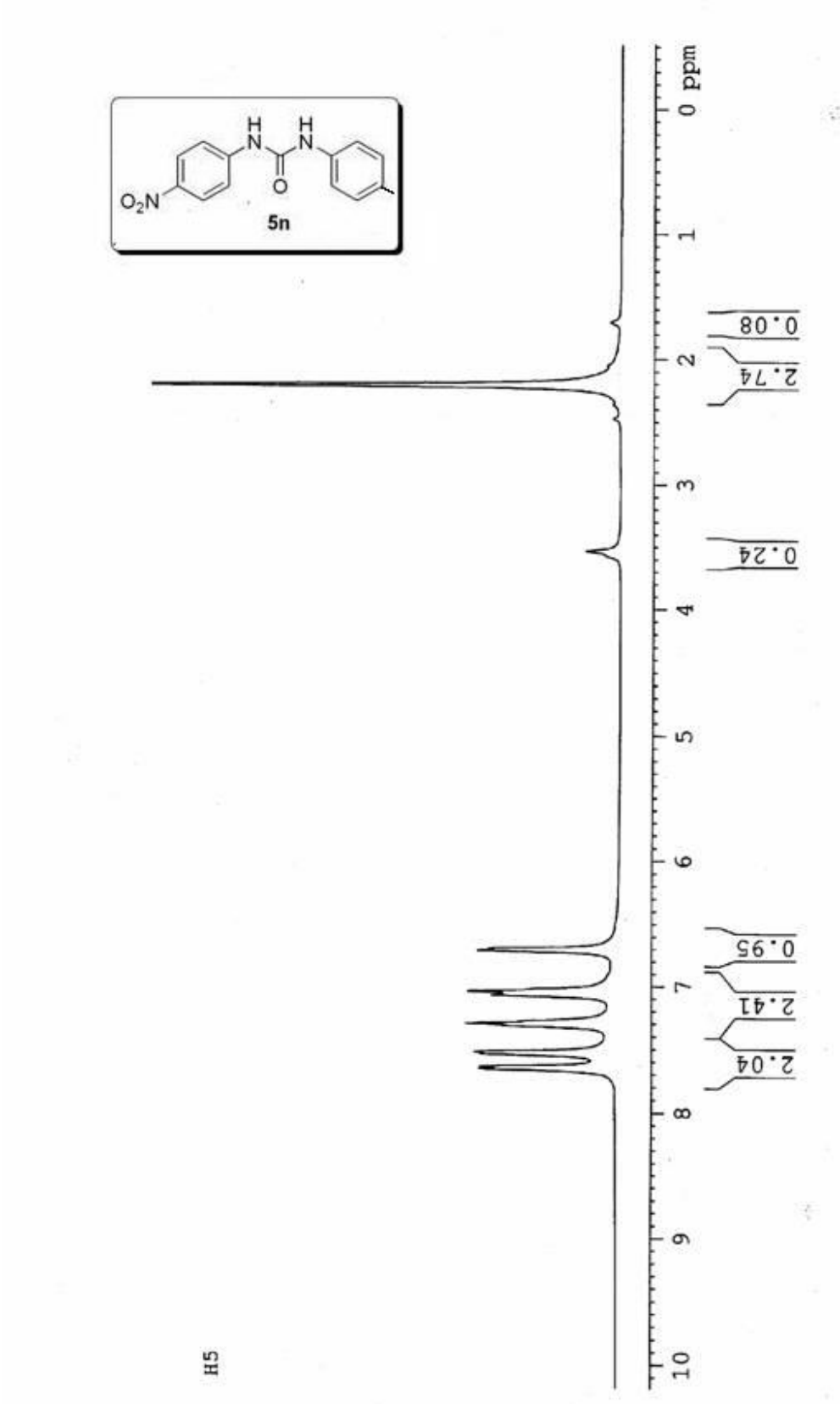

GOD-7
GOD-7 5 (0.115) AM (Cen,7, 99.00, Ar,5343.0,556.28,0.99,LS 10); Sm (SG, 2x4.00); Sb (4,40.00); Sb (4,40.00); Cm (4:6)
1: TOF MS ES+
6.34e3
363.0943
Calcd Mass = 363.0932 (M+H)
385.0792
5I
CF3
CF3
O
N
H
N
H
100
%
0
100
200
300
400
500
600
700
800
900
1000
m/z

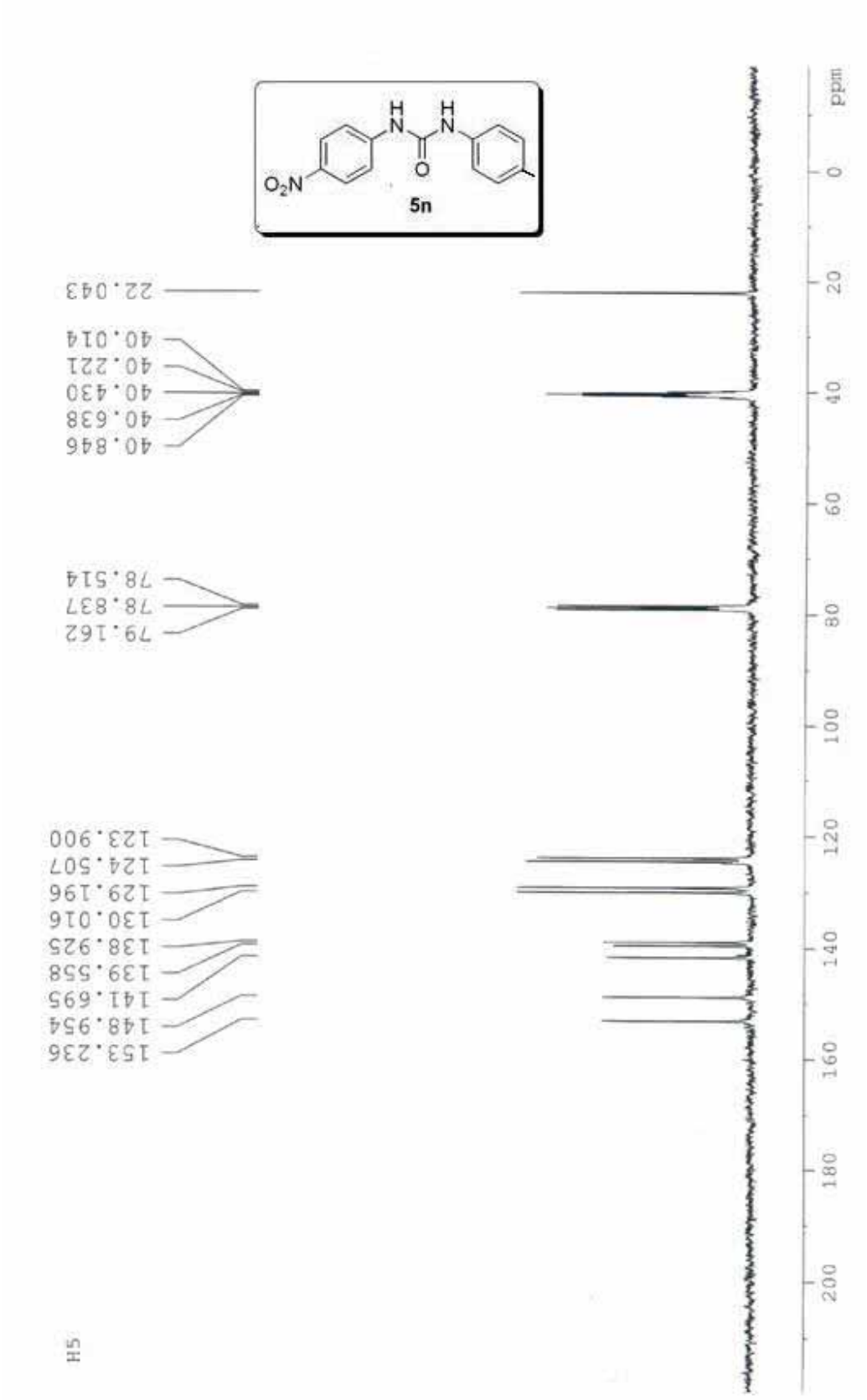

CF3
O
N
H
N
H
CF3
5l
ppm
0
1
2
3
4
5
6
7
8
9
1.000
0.480
2.850
0.442
0.869
GOD-7

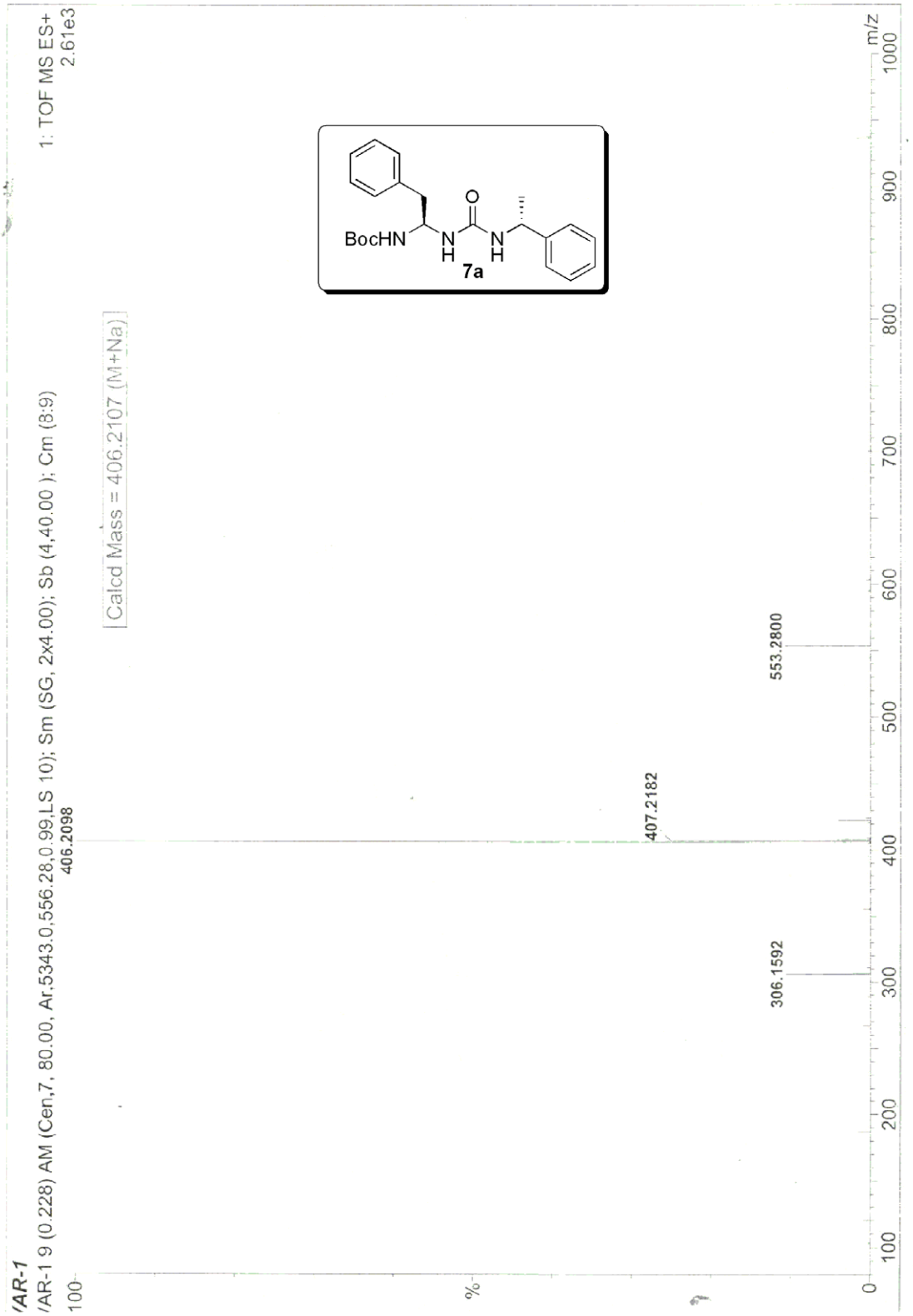

FAU
FAU 2 (0.058) AM (Cen,7, 99.00, Ar,5343.0,556.28,0.11,LS 10); Sm (SG, 2x4.00); Sb (4,40.00); Sb (4,40.00); Cm (1:4)
1: TOF MS ES+
1.55e4
Calcd Mass = 478.2107 (M+Na)
478.2114
327.1792
FmocHN
5m
%
m/z

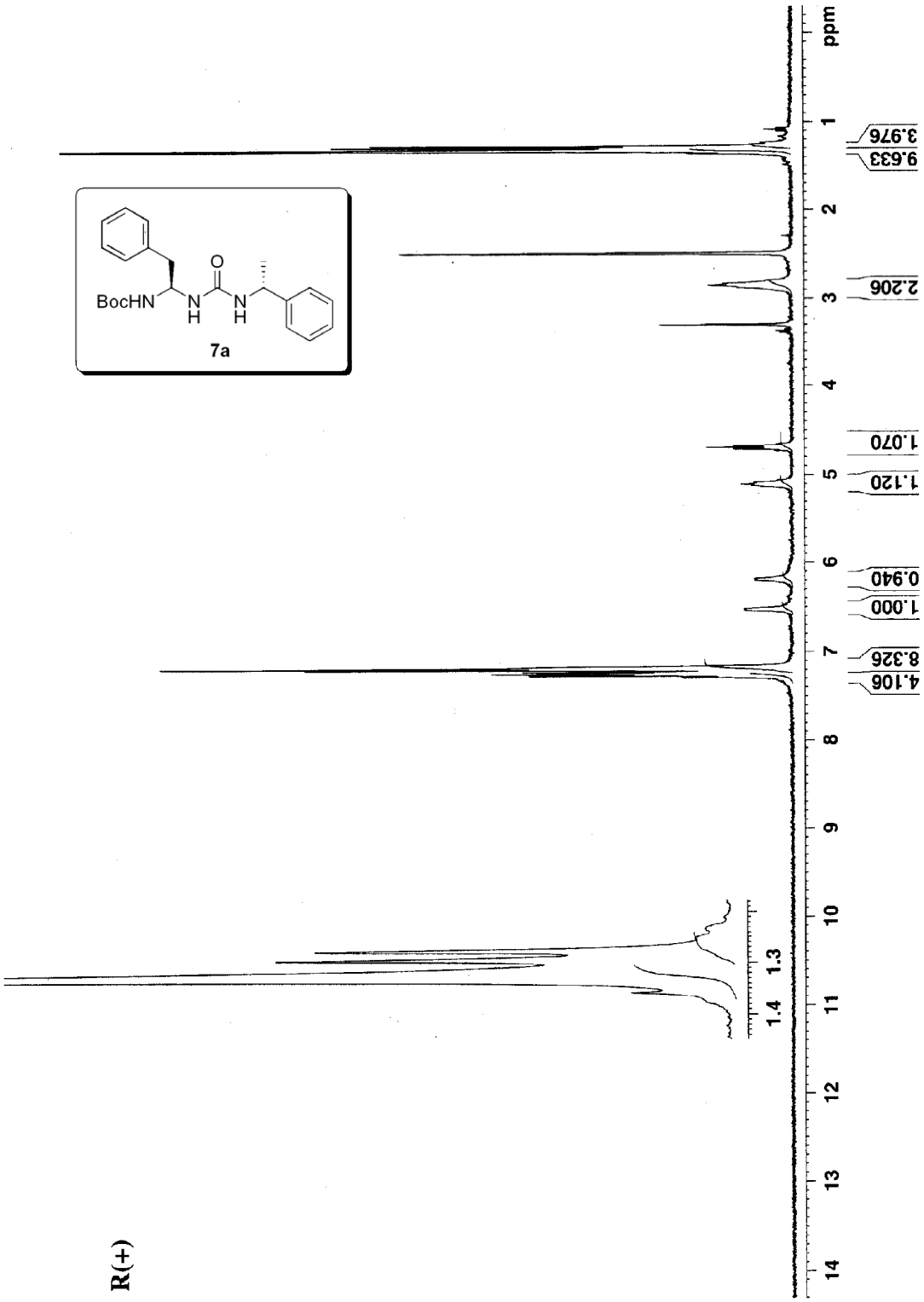

FmocHN
5m
22.27
23.44
25.08
47.80
49.92
66.80
77.21
77.53
77.85
120.49
125.20
125.40
125.53
127.59
128.24
136.14
137.21
141.83
144.24
156.08
156.19
HP-3
ppm

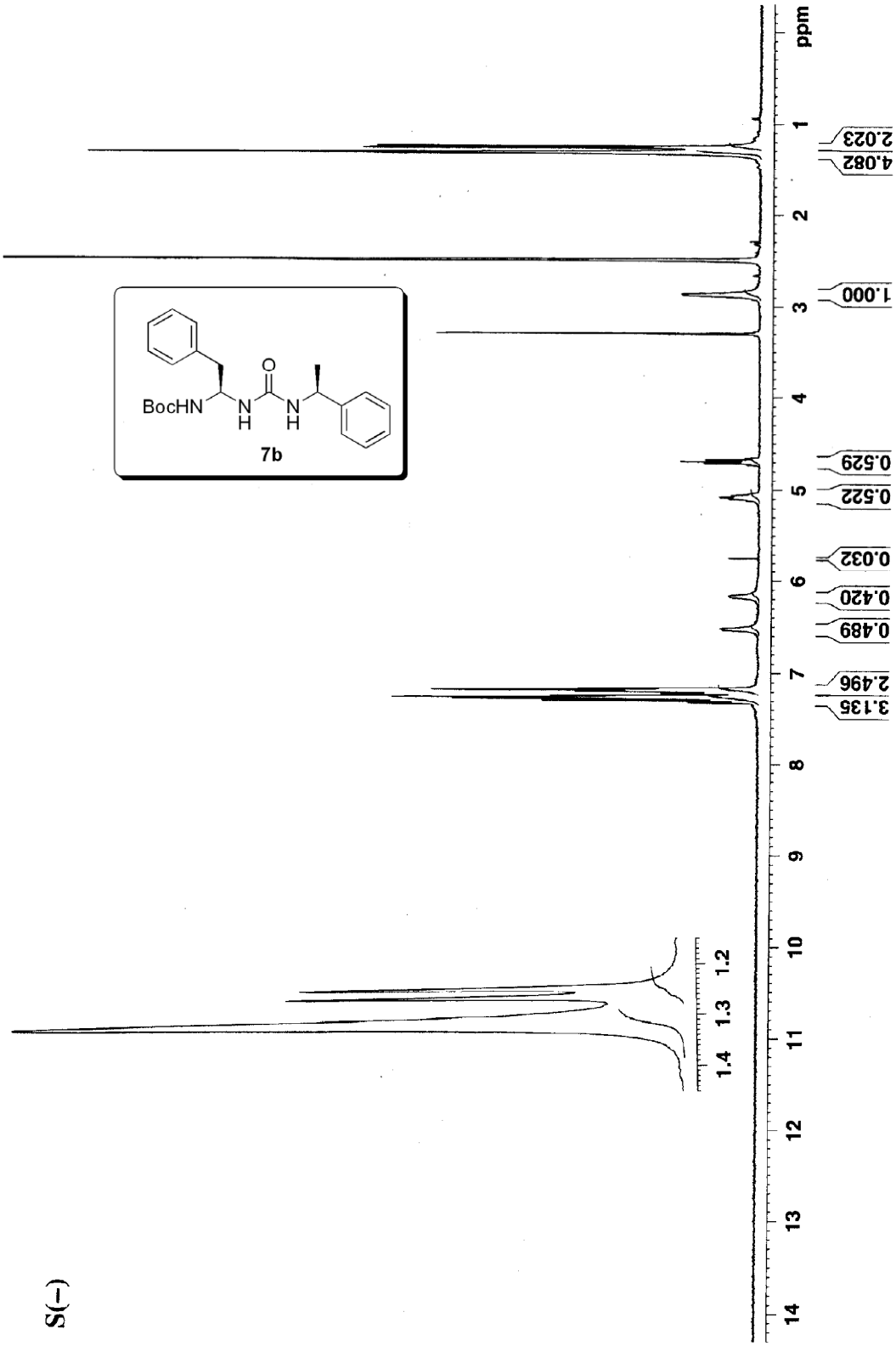

H-5
H-5 2 (0.058) AM (Cen,7, 99.00, Ar,5343.0,556.28,0.10,LS 10); Sm (SG, 2x4.00); Sb (4,40.00); Sb (4,40.00); Sb (4,40.00); Cm (2:3) 1: TOF MS ES+
5.50e3
294.0852
295.0886
CalcD Mass = 294.0855(M+Na)
5n
100
%
0
100
200
300
400
500
600
700
800
900
1000
m/z

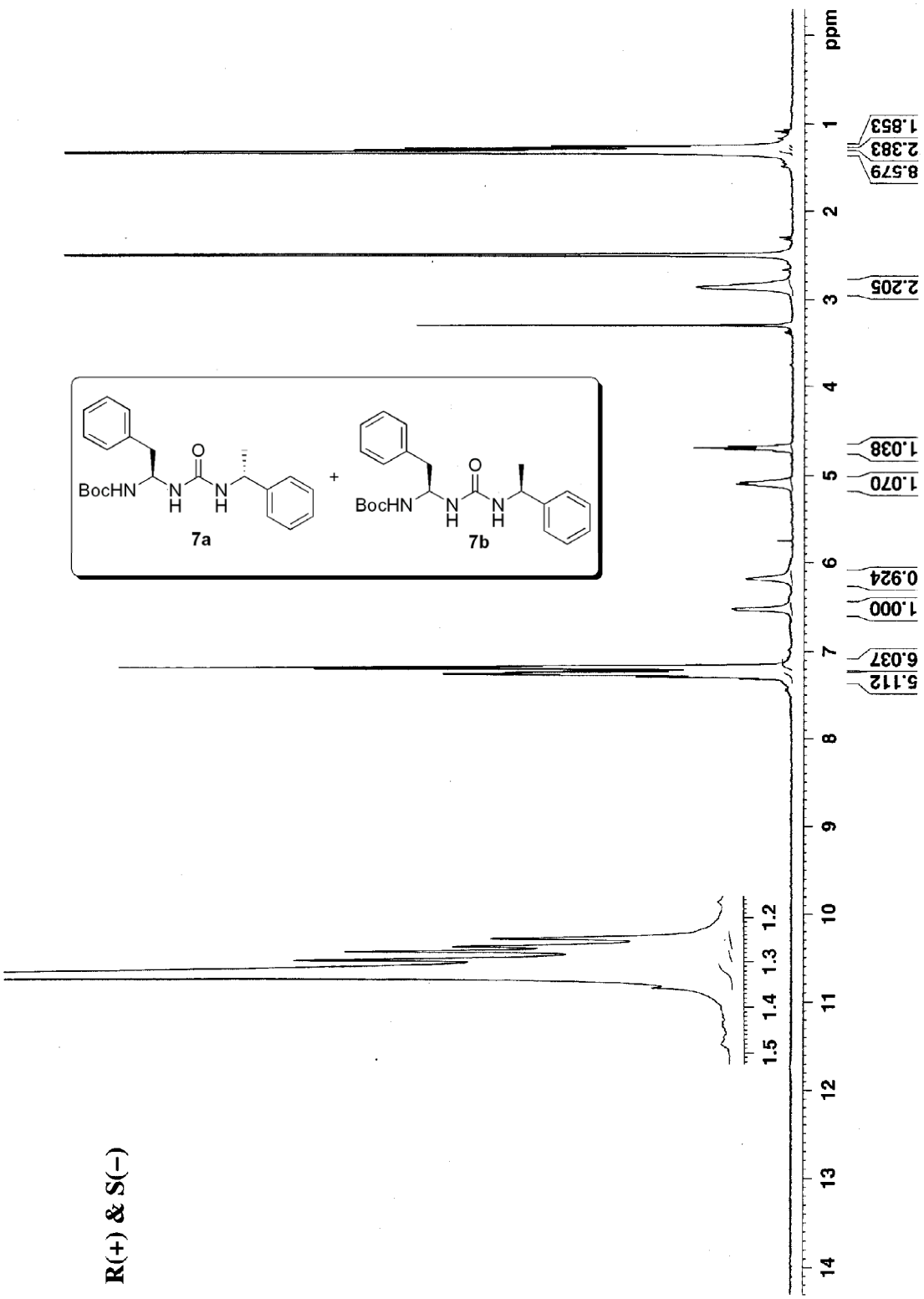

O2N
5n
0 ppm
1
2
3
4
5
6
7
8
9
10
0.08
2.74
0.24
0.95
2.41
2.04
H5

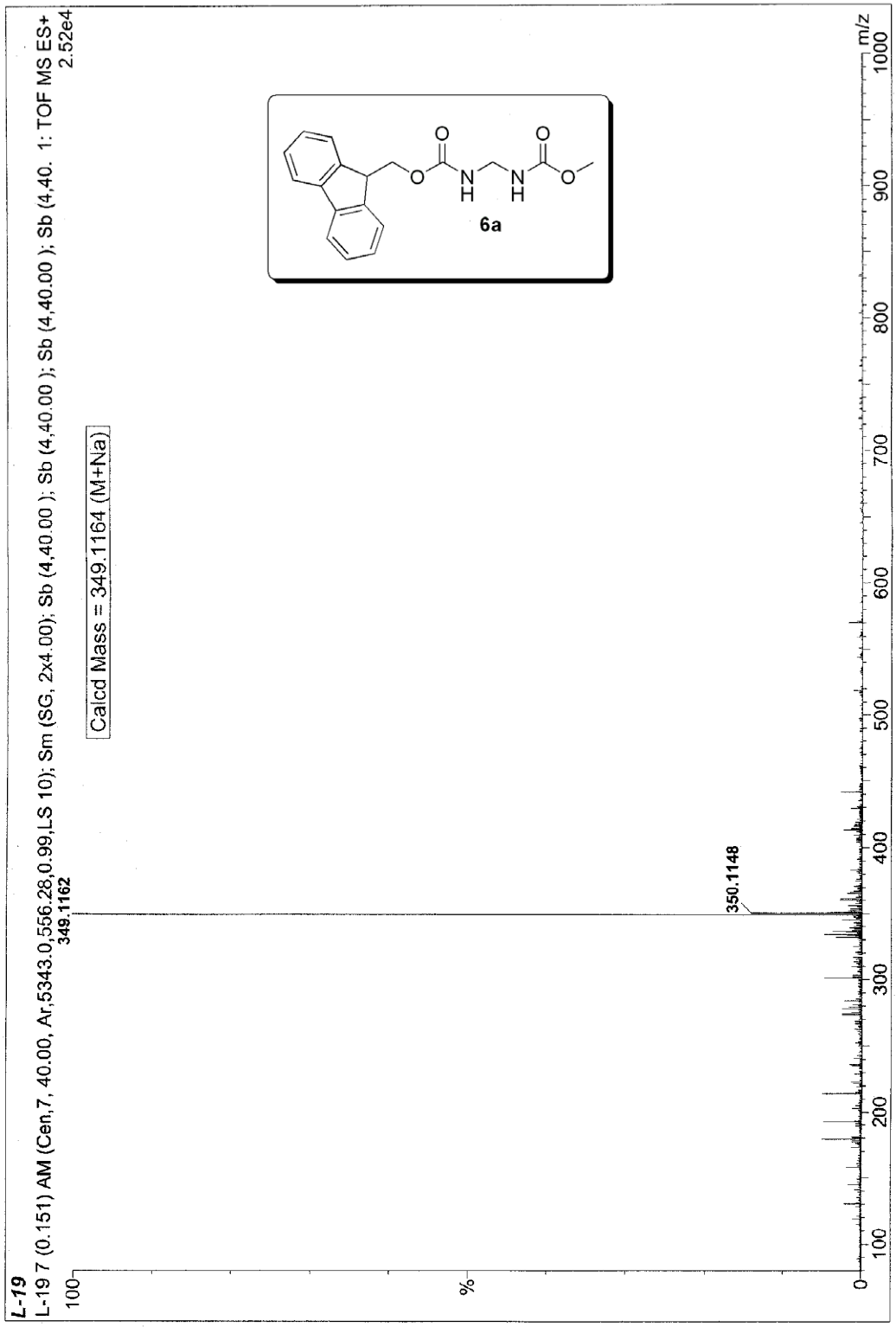

5n
22.043
40.014
40.221
40.430
40.638
40.846
78.514
78.837
79.162
123.900
124.507
129.196
130.016
138.925
139.558
141.695
148.954
153.236
H5
ppm
0
20
40
60
80
100
120
140
160
180
200

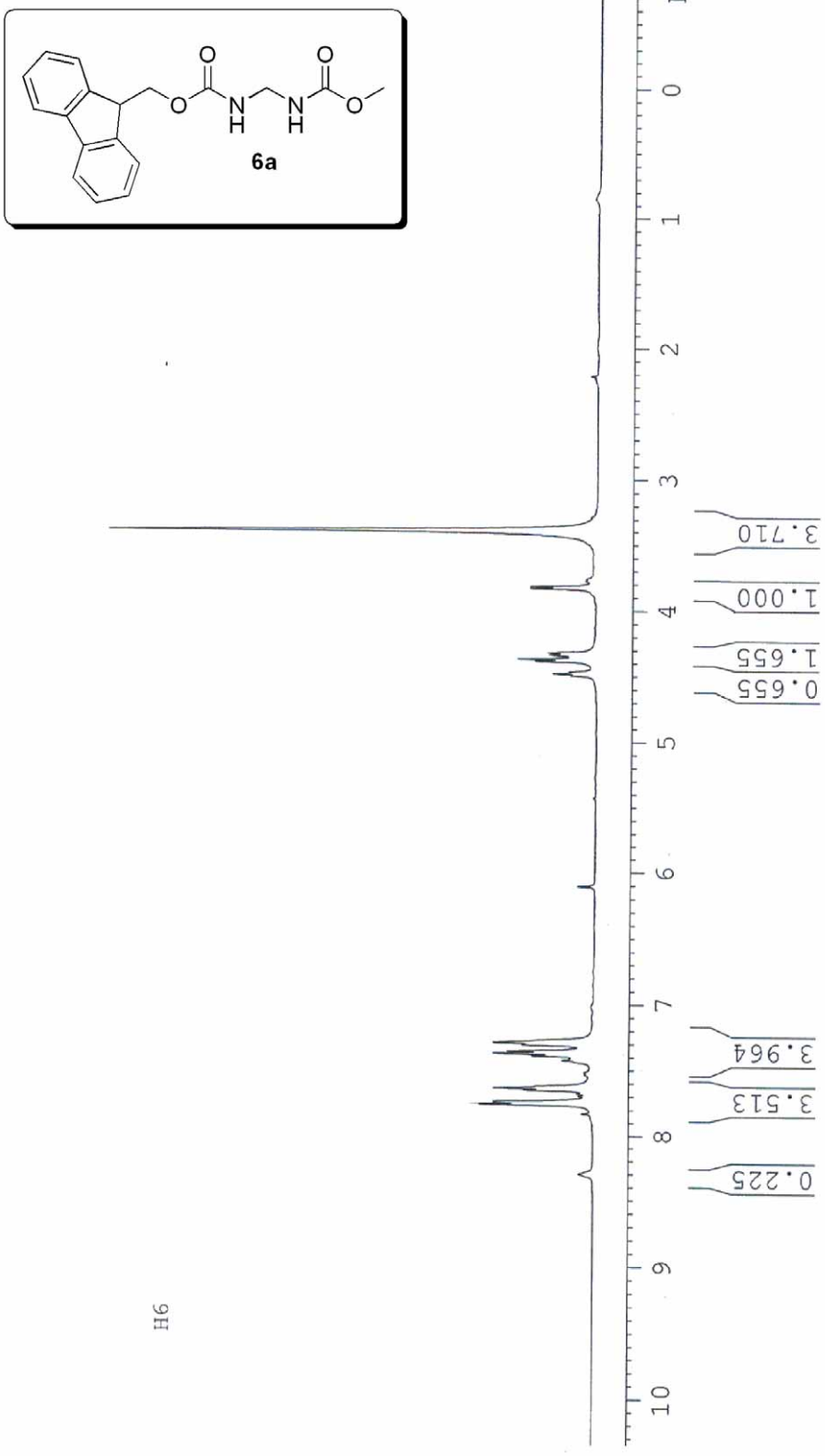

/AR-1
/AR-1 9 (0.228) AM (Cen,7, 80.00, Ar.5343.0,556.28,0.99,LS 10); Sm (SG, 2x4.00); Sb (4,40.00); Cm (8:9)
1: TOF MS ES+
2.61e3
100
%
0
Calcd Mass = 406.2107 (M+Na)
406.2098
407.2182
553.2800
306.1592
BocHN
N
H
O
N
H
7a
100
200
300
400
500
600
700
800
900
1000
m/z

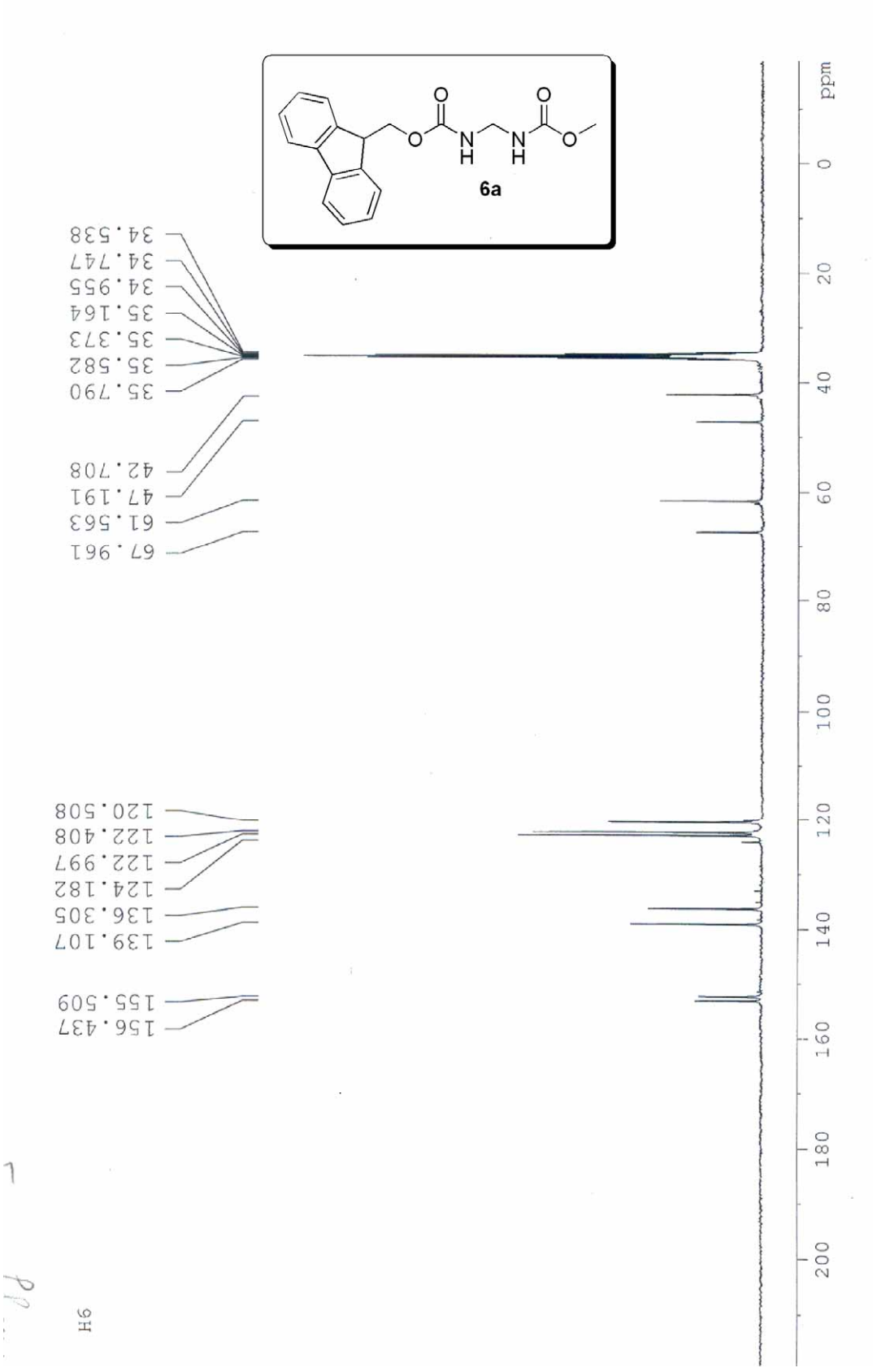

BocHN
N
H
O
N
H
7a
R(+)
ppm
3.976
9.633
2.206
1.070
1.120
0.940
1.000
8.326
4.106
1.4
1.3

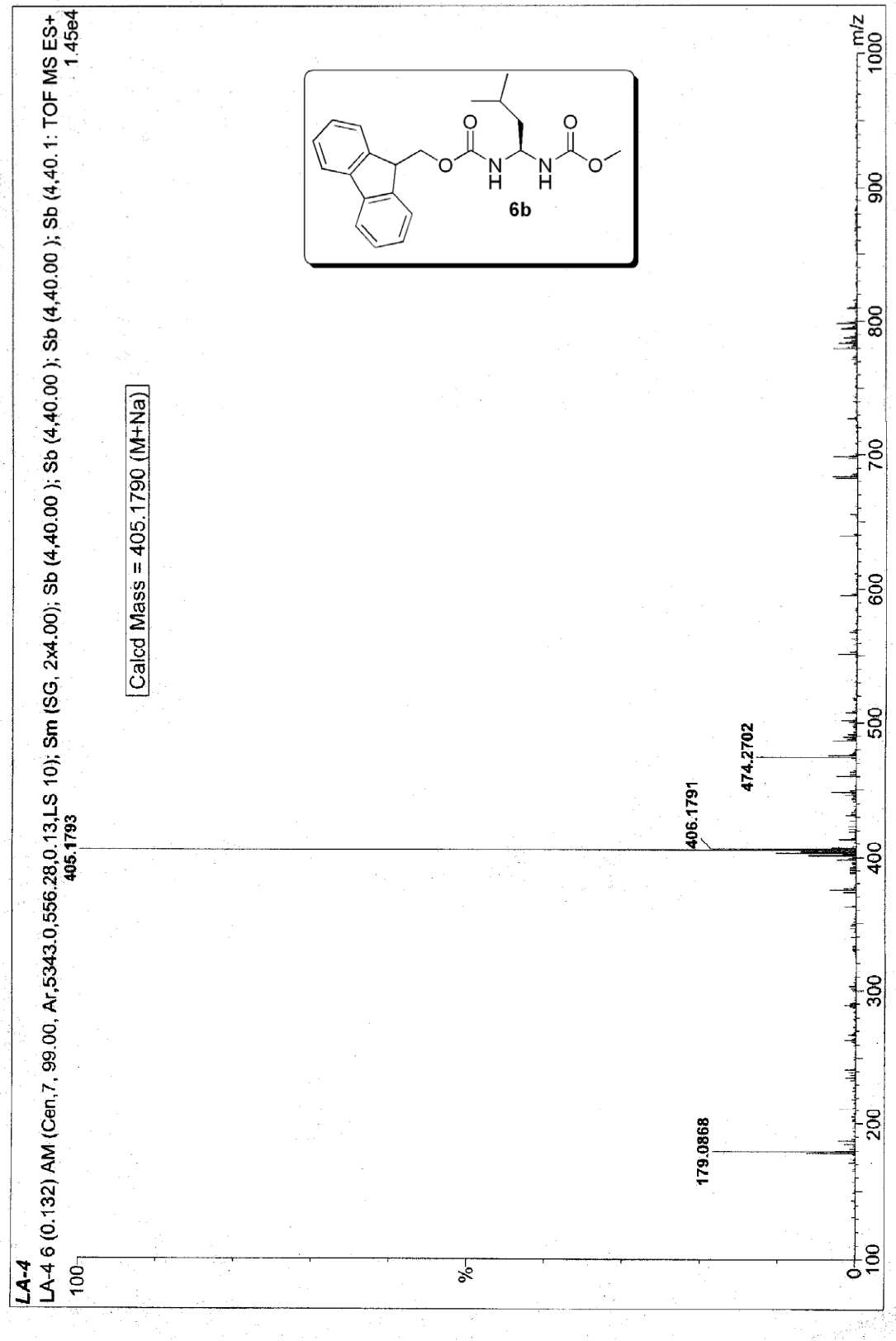

BocHN
N
H
O
N
H
7b
S(-)
ppm
2.023
4.082
1.000
0.529
0.522
0.032
0.420
0.489
2.496
3.135
1.4
1.3
1.2

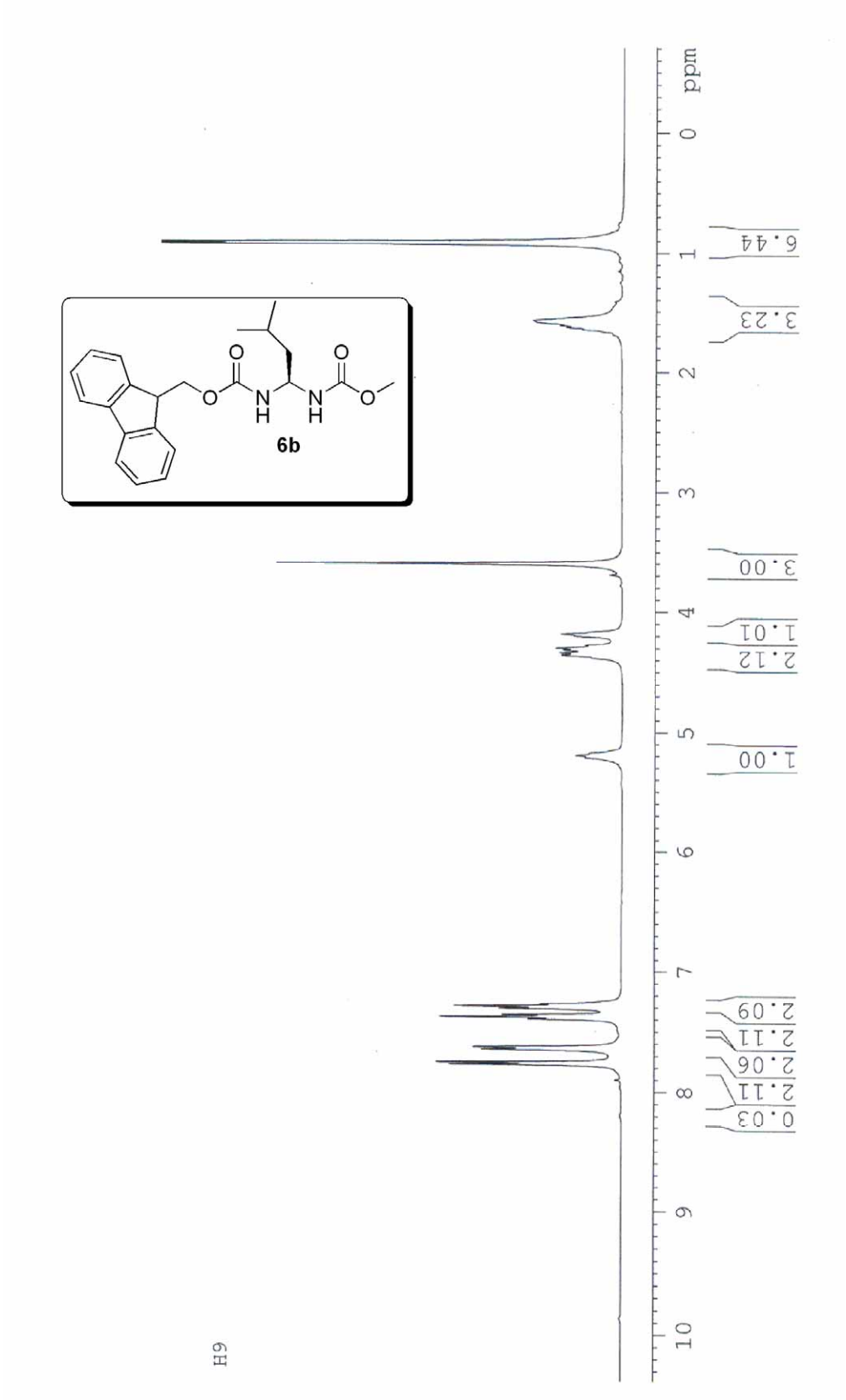

ppm
1.853
2.383
8.579
2.205
1.038
1.070
0.924
1.000
6.037
5.112
BocHN
7a
BocHN
7b
R(+) & S(−)

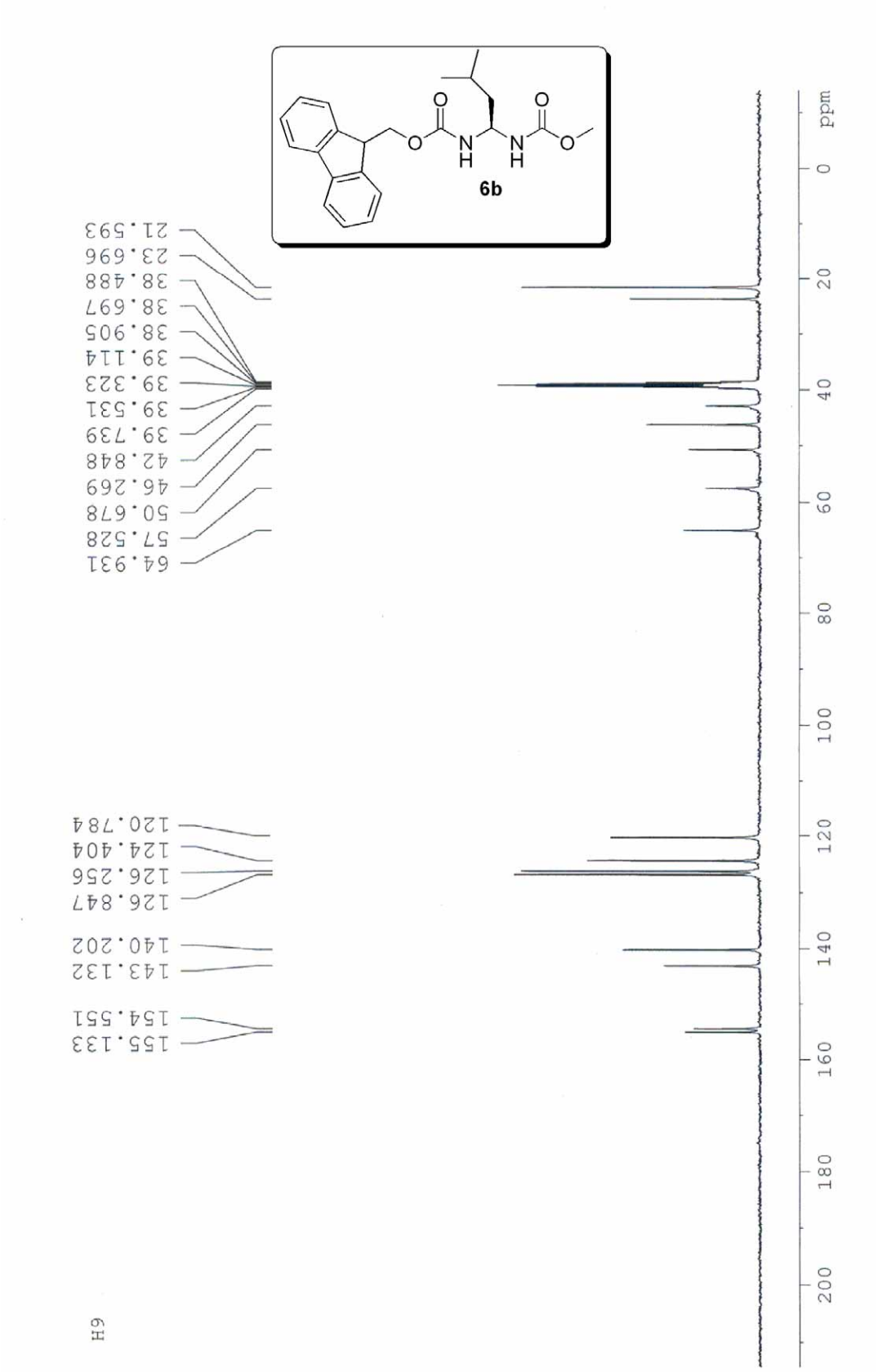

L-19
L-19 7 (0.151) AM (Cen,7, 40.00, Ar,5343.0,556.28,0.99,LS 10); Sm (SG, 2x4.00); Sb (4,40.00); Sb (4,40.00); Sb (4,40.00); Sb (4,40. 1: TOF MS ES+
2.52e4
100
%
0
Calcd Mass = 349.1164 (M+Na)
349.1162
350.1148
100
200
300
400
500
600
700
800
900
1000
m/z
O
O
O
N
H
N
H
O
6a

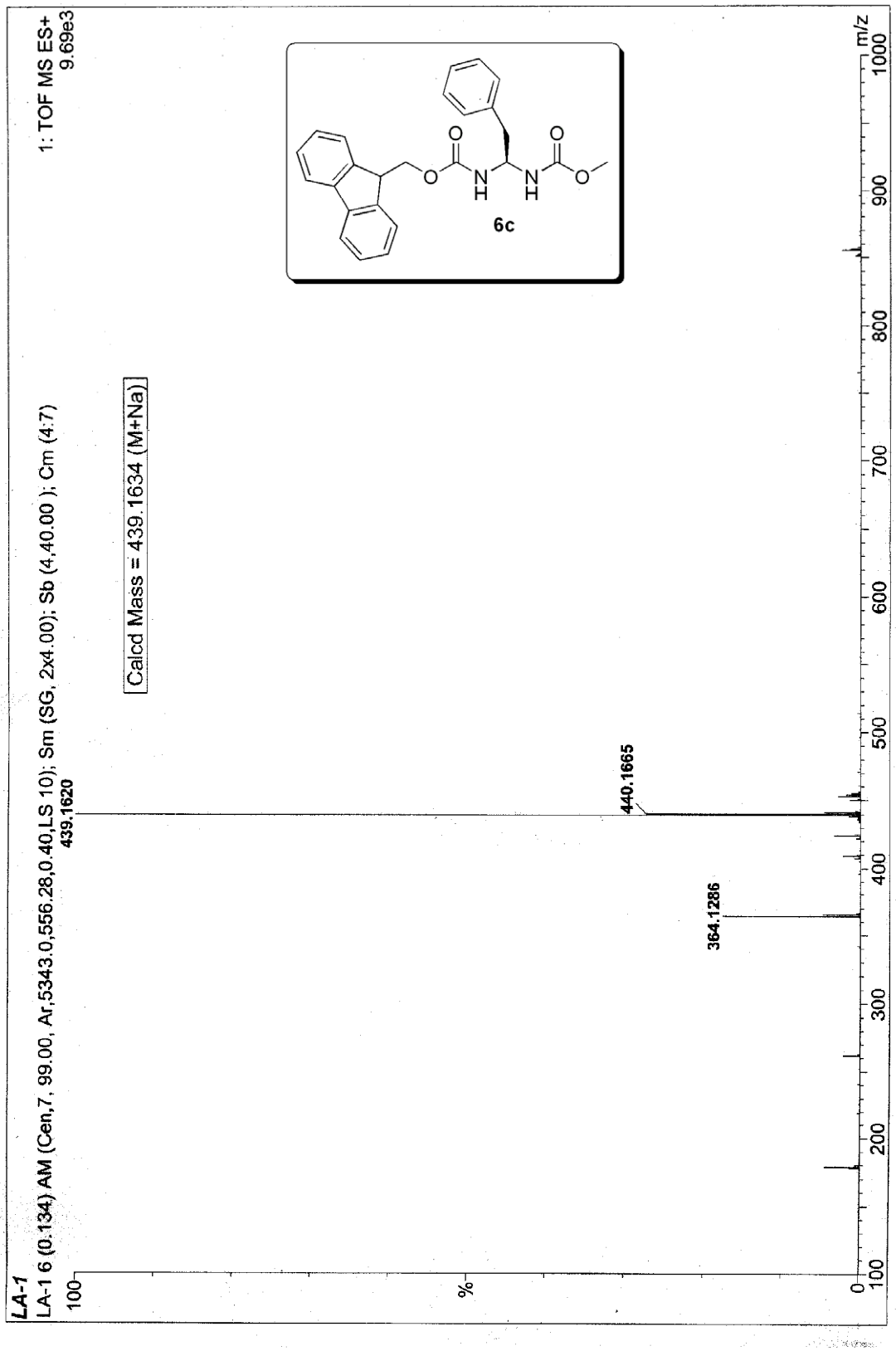

6a
ppm
0
1
2
3
4
5
6
7
8
9
10
3.710
1.000
1.655
0.655
3.964
3.513
0.225
9H

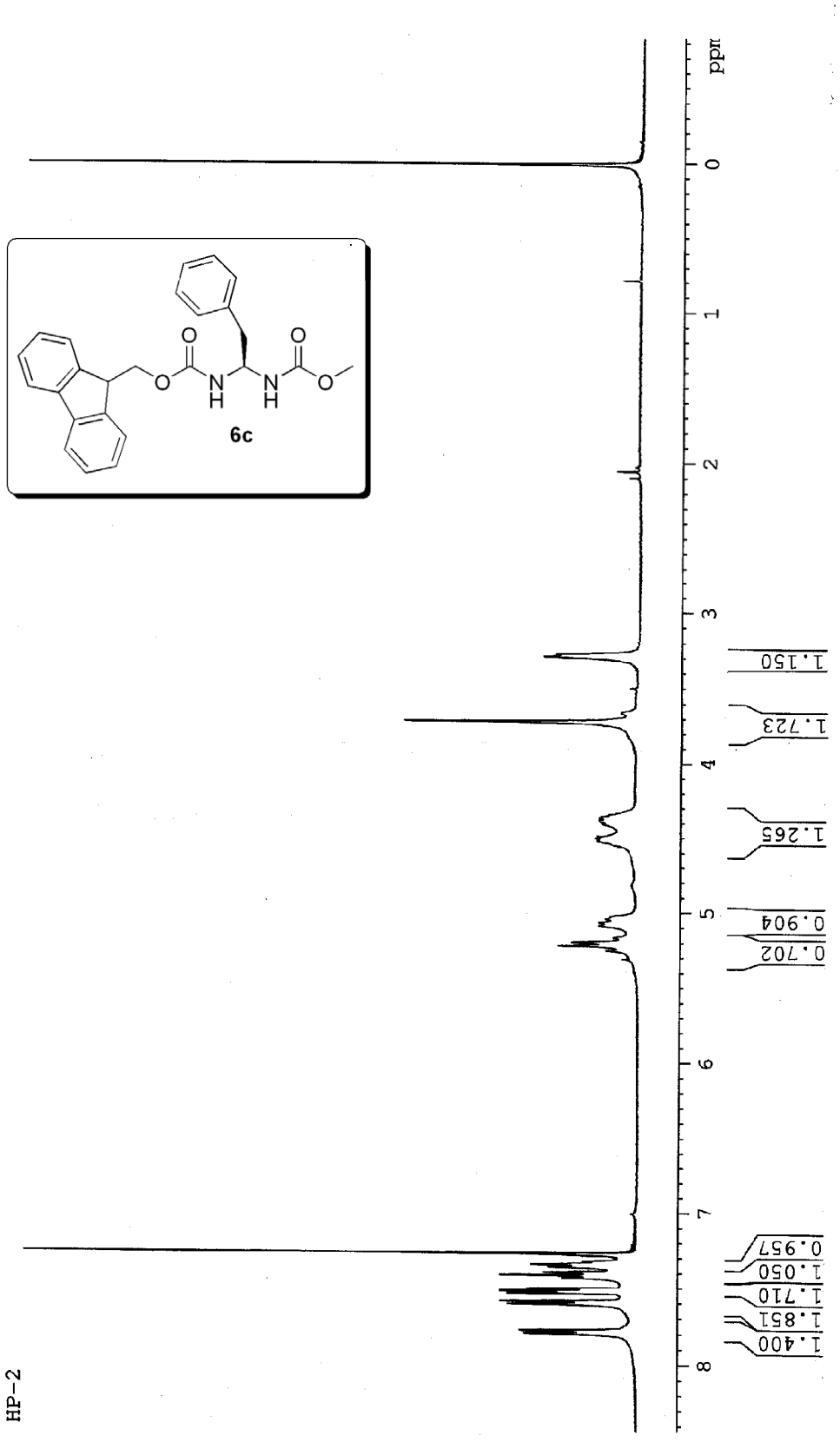

6a
34.538
34.747
34.955
35.164
35.373
35.582
35.790
42.708
47.191
61.563
67.961
120.508
122.408
122.997
124.182
136.305
139.107
155.509
156.437
ppm
0
20
40
60
80
100
120
140
160
180
200

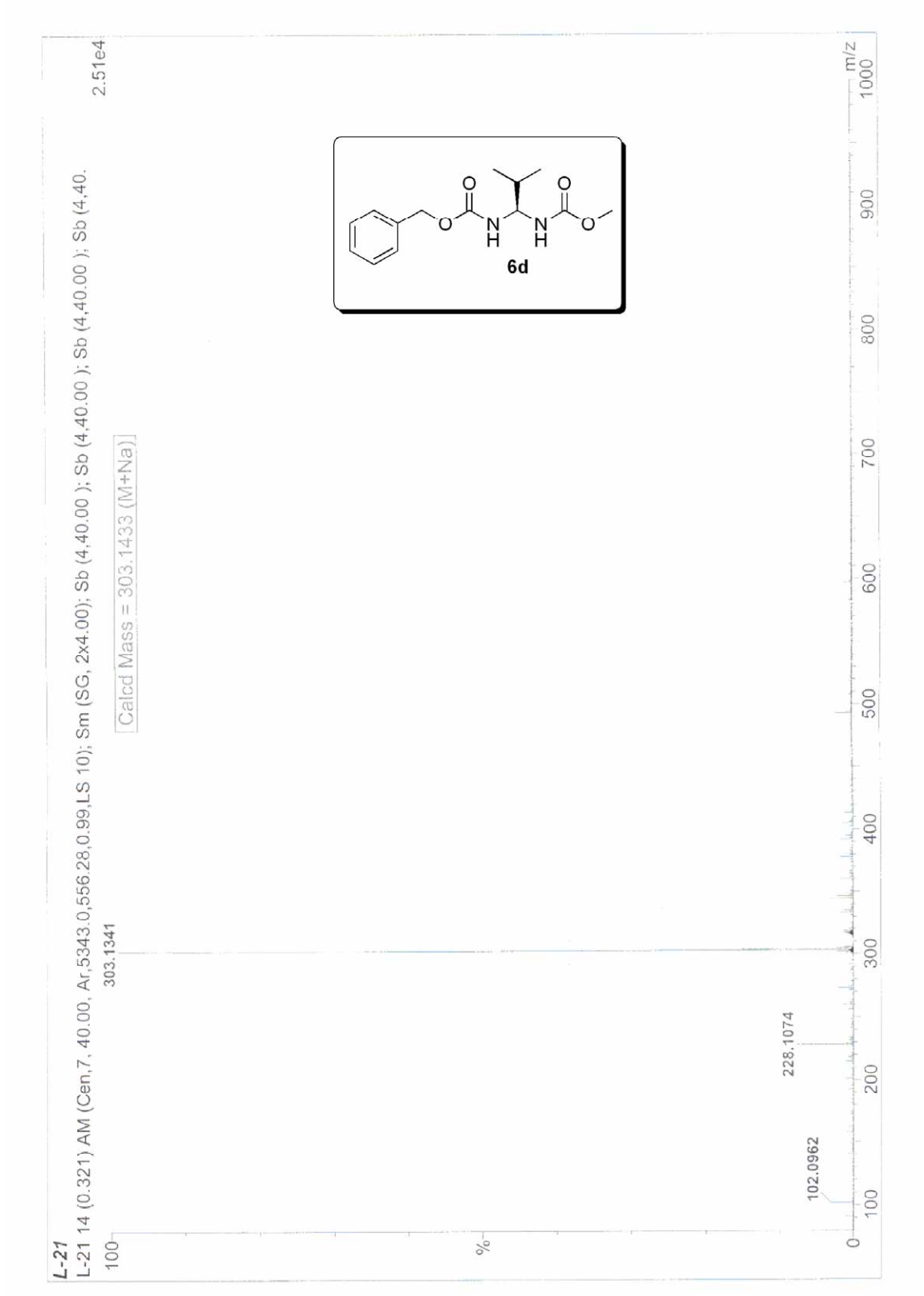

LA-4
LA-4 6 (0.132) AM (Cen,7, 99.00, Ar,5343.0,556.28,0.13,LS 10); Sm (SG, 2x4.00); Sb (4,40.00); Sb (4,40.00); Sb (4,40.00); Sb (4,40.1: TOF MS ES+
1.45e4
Calcd Mass = 405.1790 (M+Na)
6b
405.1793
406.1791
474.2702
179.0868
100
%
0
100
200
300
400
500
600
700
800
900
1000
m/z

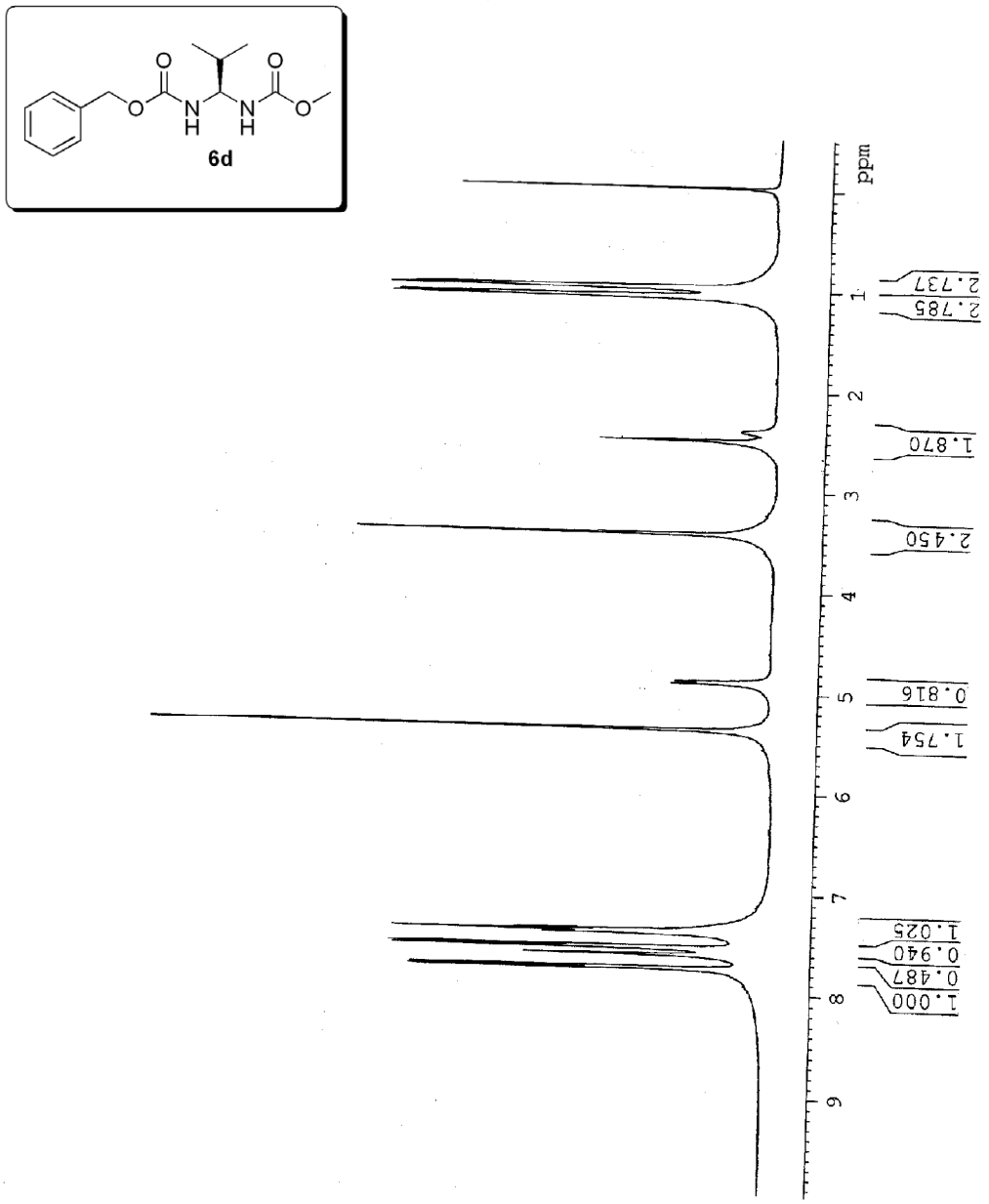

6b
ppm
0
1
2
3
4
5
6
7
8
9
10
6.44
3.23
3.00
1.01
2.12
1.00
2.09
2.11
2.06
2.11
0.03
H9

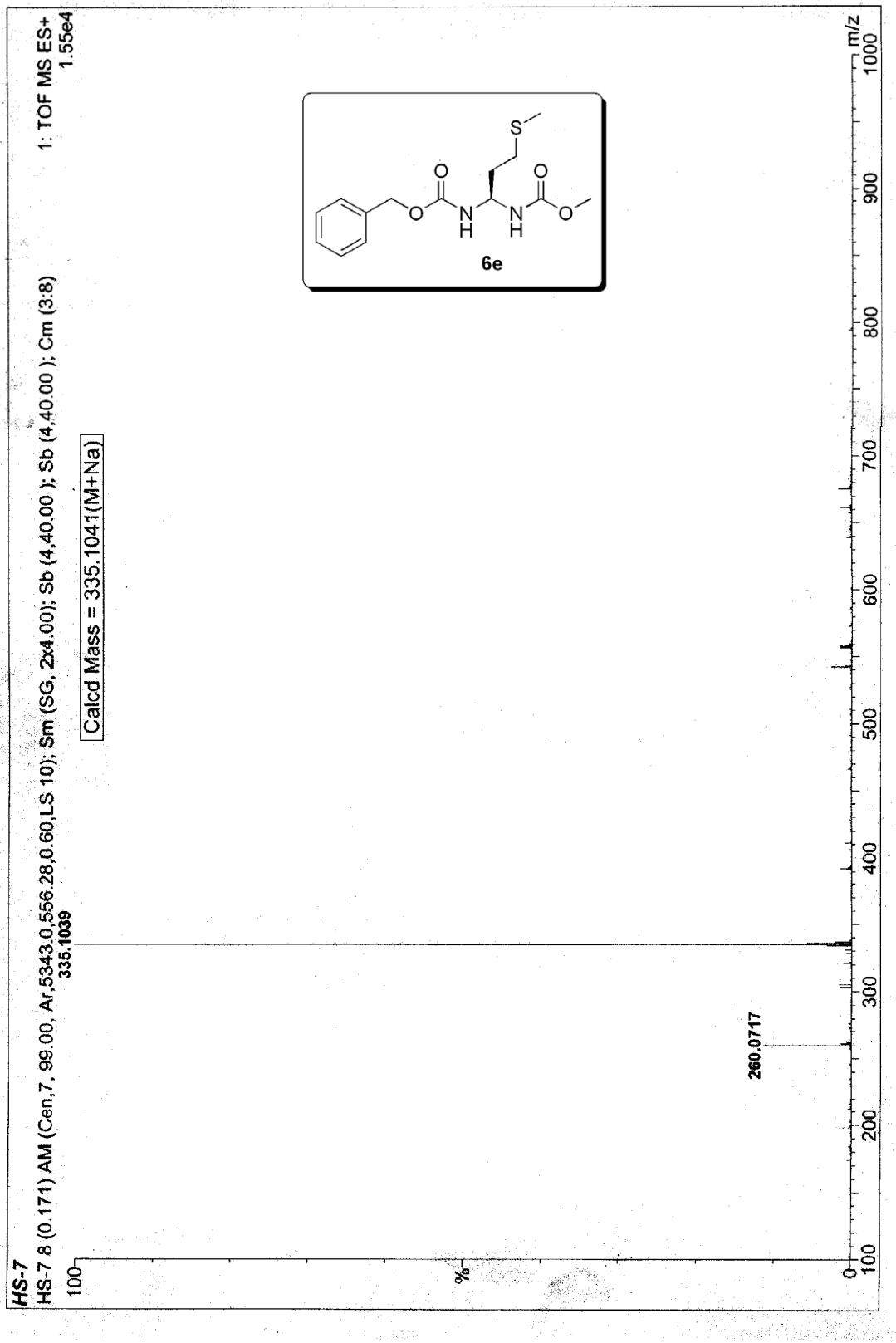

6b
21.593
23.696
38.488
38.697
38.905
39.114
39.323
39.531
39.739
42.848
46.269
50.678
57.528
64.931
120.784
124.404
126.256
126.847
140.202
143.132
154.551
155.133
6H
ppm
0
20
40
60
80
100
120
140
160
180
200

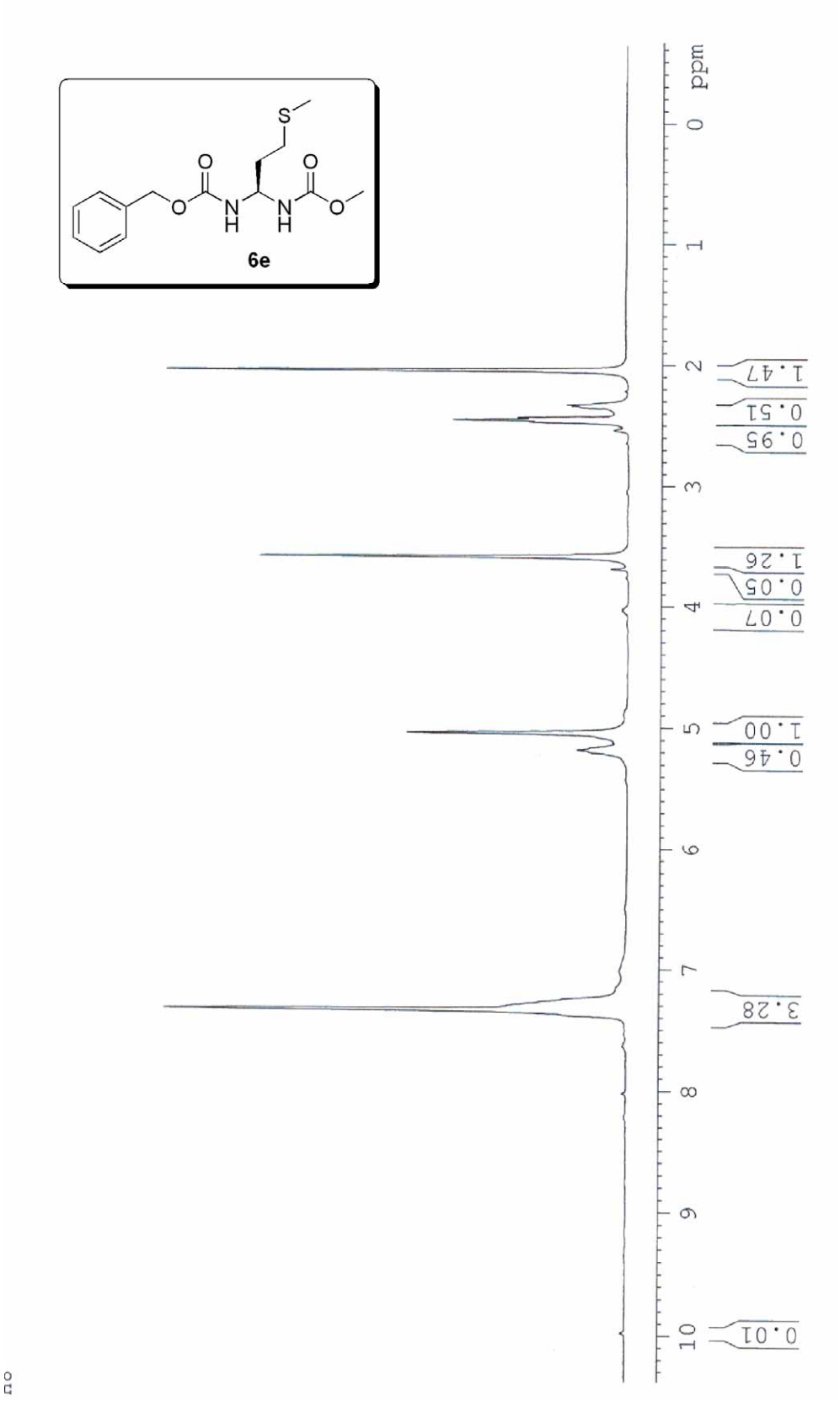

LA-1
LA-1 6 (0.134) AM (Cen,7, 99.00, Ar,5343.0,556.28,0.40,LS 10); Sm (SG, 2x4.00); Sb (4,40.00); Cm (4:7)
1: TOF MS ES+
9.69e3
Calcd Mass = 439.1634 (M+Na)
439.1620
440.1665
364.1286
6c
%
m/z

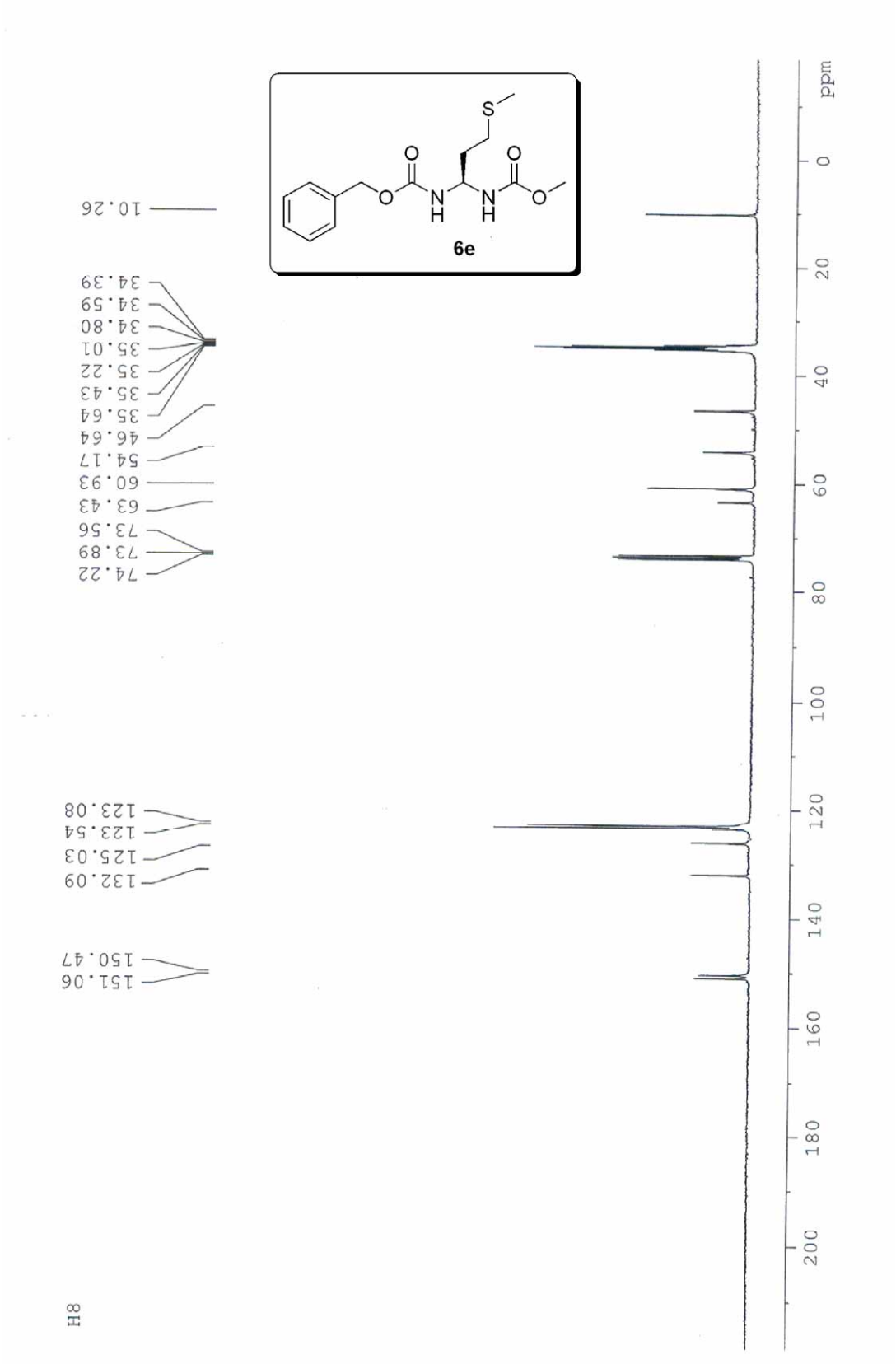

6c
HP-2
ppm
0
1
2
3
4
5
6
7
8
1.150
1.723
1.265
0.904
0.702
1.400
1.851
1.710
1.050
0.957

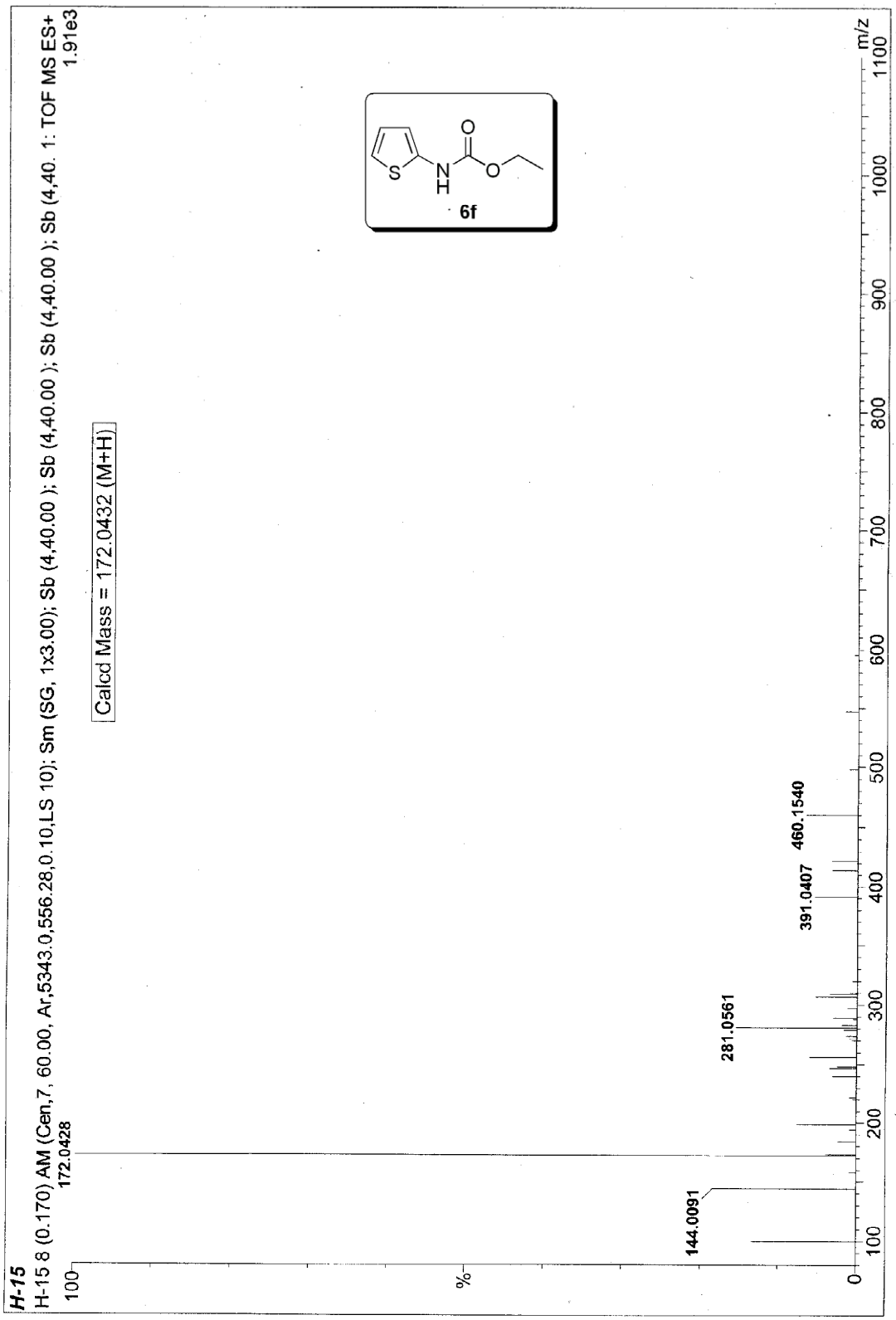

L-21
L-21 14 (0.321) AM (Cen,7, 40.00, Ar,5343.0,556.28,0.99,LS 10); Sm (SG, 2x4.00); Sb (4,40.00); Sb (4,40.00); Sb (4,40.00); Sb (4,40.
2.51e4
Calcd Mass = 303.1433 (M+Na)
303.1341
228.1074
102.0962
100
%
0
100
200
300
400
500
600
700
800
900
1000
m/z
6d

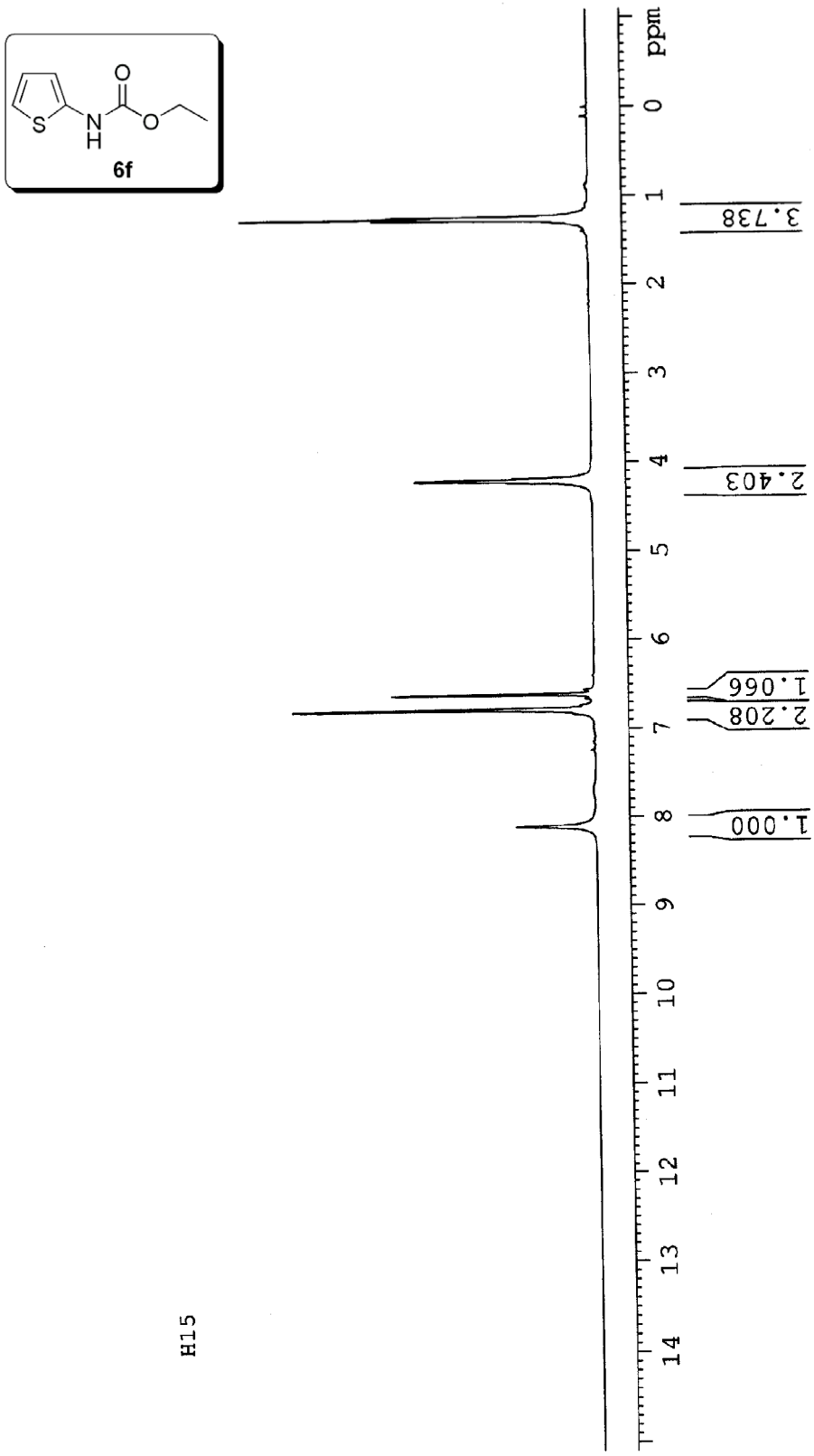

6d
ppm
1
2
3
4
5
6
7
8
9
2.737
2.785
1.870
2.450
0.816
1.754
1.025
0.940
0.487
1.000

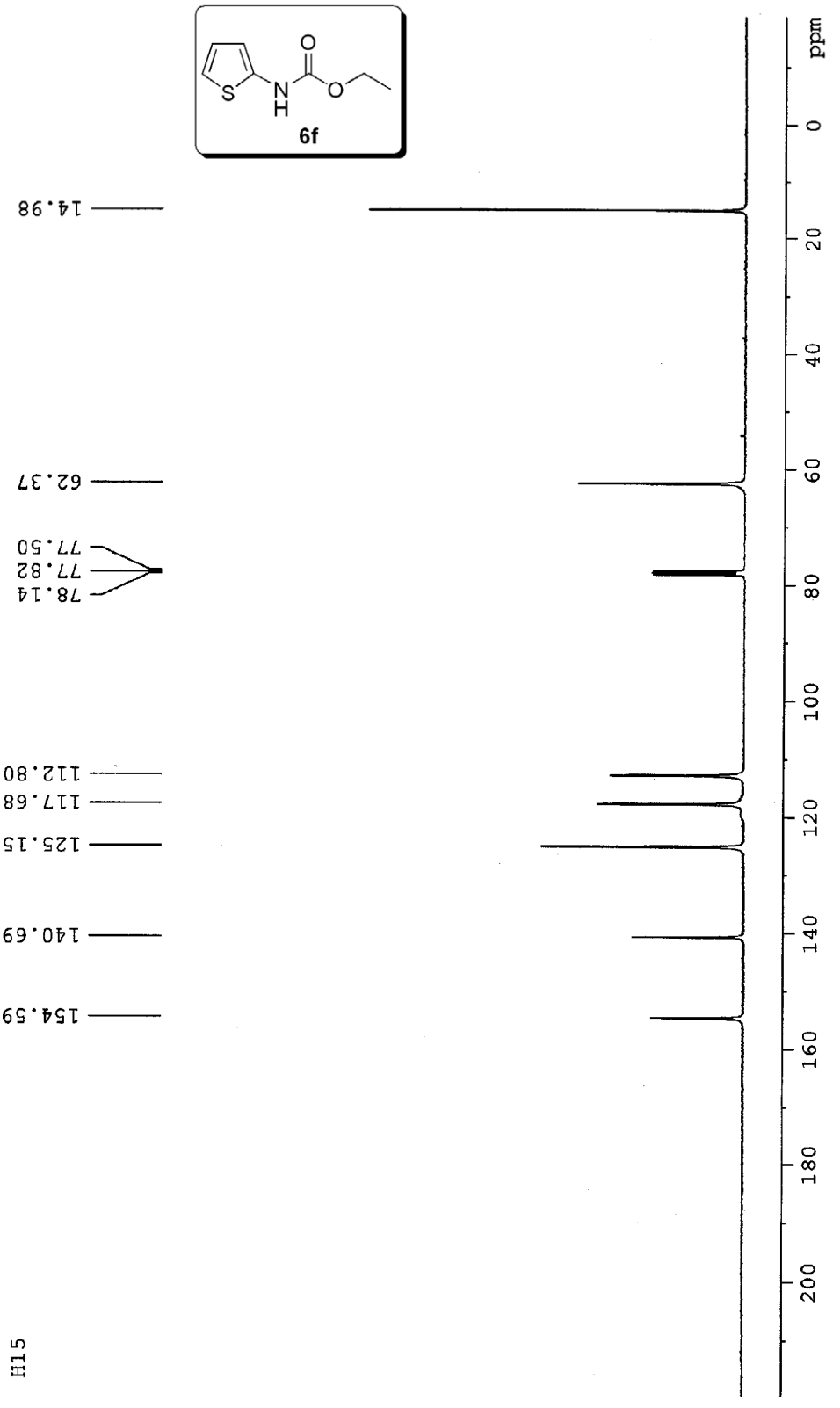

HS-7
HS-7 8 (0.171) AM (Cen,7, 99.00, Ar,5343.0,556.28,0.60,LS 10); Sm (SG, 2x4.00); Sb (4,40.00); Sb (4,40.00); Cm (3:8)
1: TOF MS ES+
1.55e4
Calcd Mass = 335.1041(M+Na)
335.1039
260.0717
6e
100
%
0
100
200
300
400
500
600
700
800
900
1000
m/z

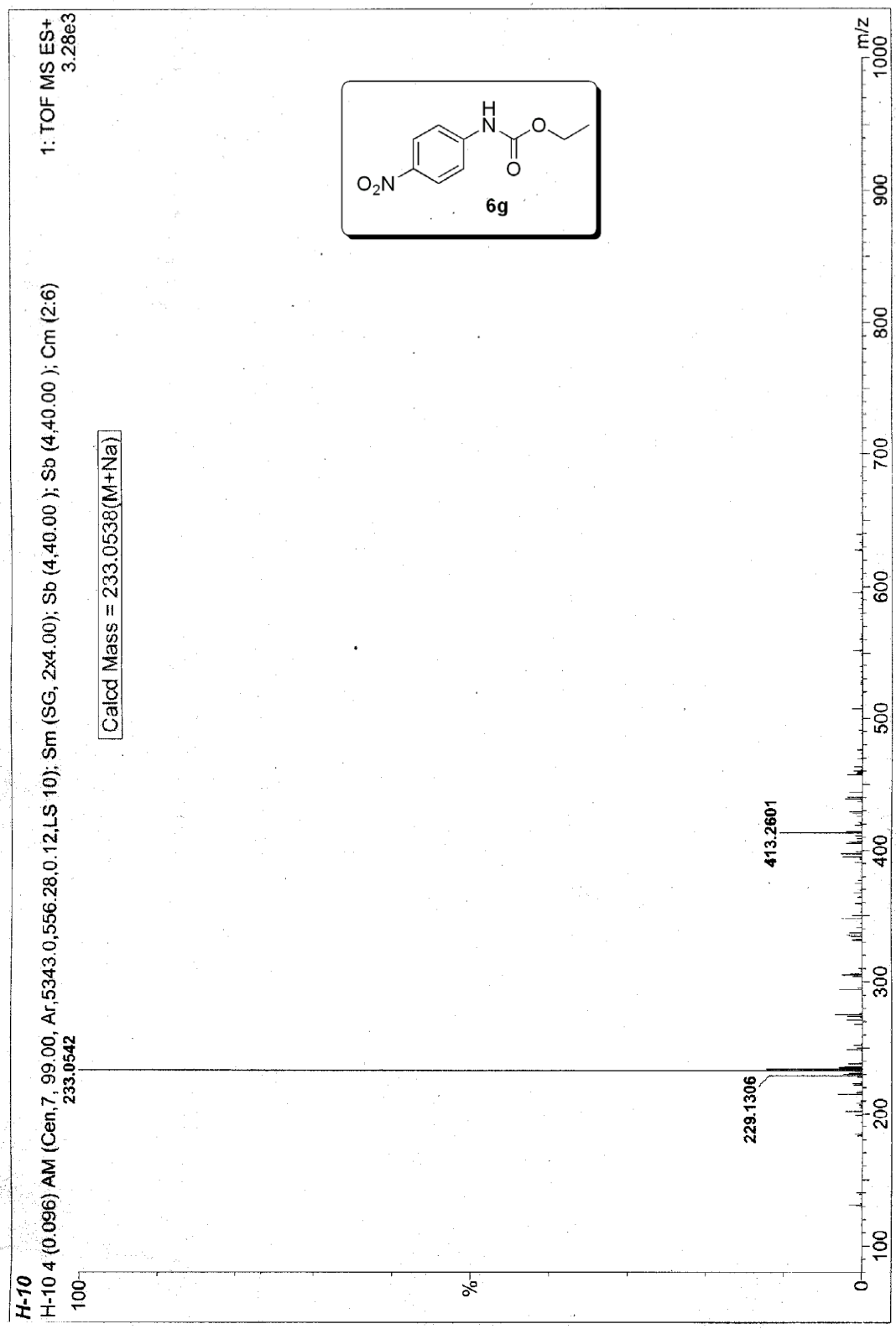

6e
ppm
0
1
2
3
4
5
6
7
8
9
10
1.47
0.51
0.95
1.26
0.05
0.07
1.00
0.46
3.28
0.01

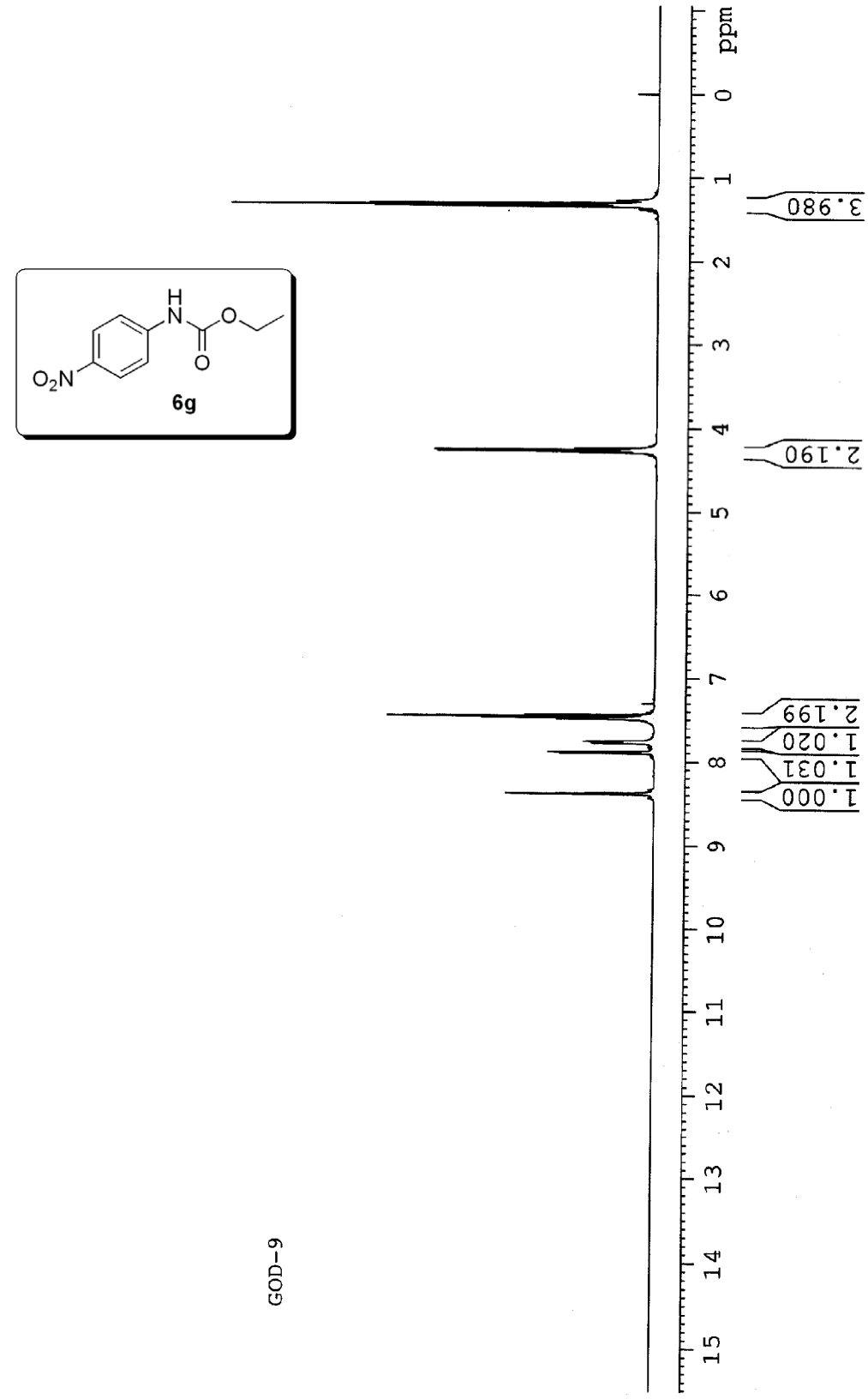

6e
10.26
34.39
34.59
34.80
35.01
35.22
35.43
35.64
46.64
54.17
60.93
63.43
73.56
73.89
74.22
123.08
123.54
125.03
132.09
150.47
151.06
8H
ppm
0
20
40
60
80
100
120
140
160
180
200

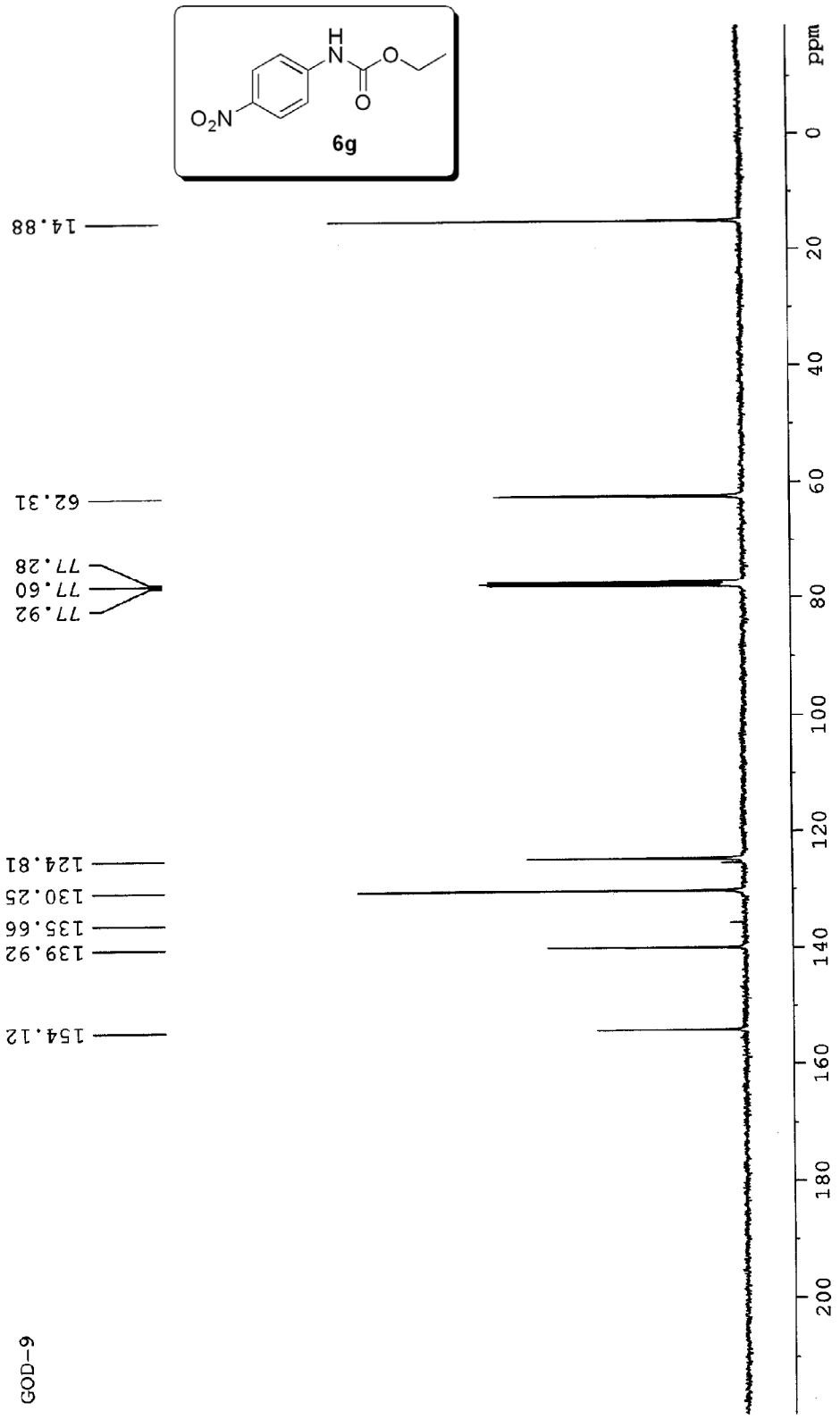

H-15
H-15 8 (0.170) AM (Cen,7, 60.00, Ar,5343.0,556.28,0.10,LS 10); Sm (SG, 1x3.00); Sb (4,40.00); Sb (4,40.00); Sb (4,40.00); Sb (4,40. 1: TOF MS ES+
1.91e3
Calcd Mass = 172.0432 (M+H)
6f
172.0428
144.0091
281.0561
391.0407
460.1540
%
m/z

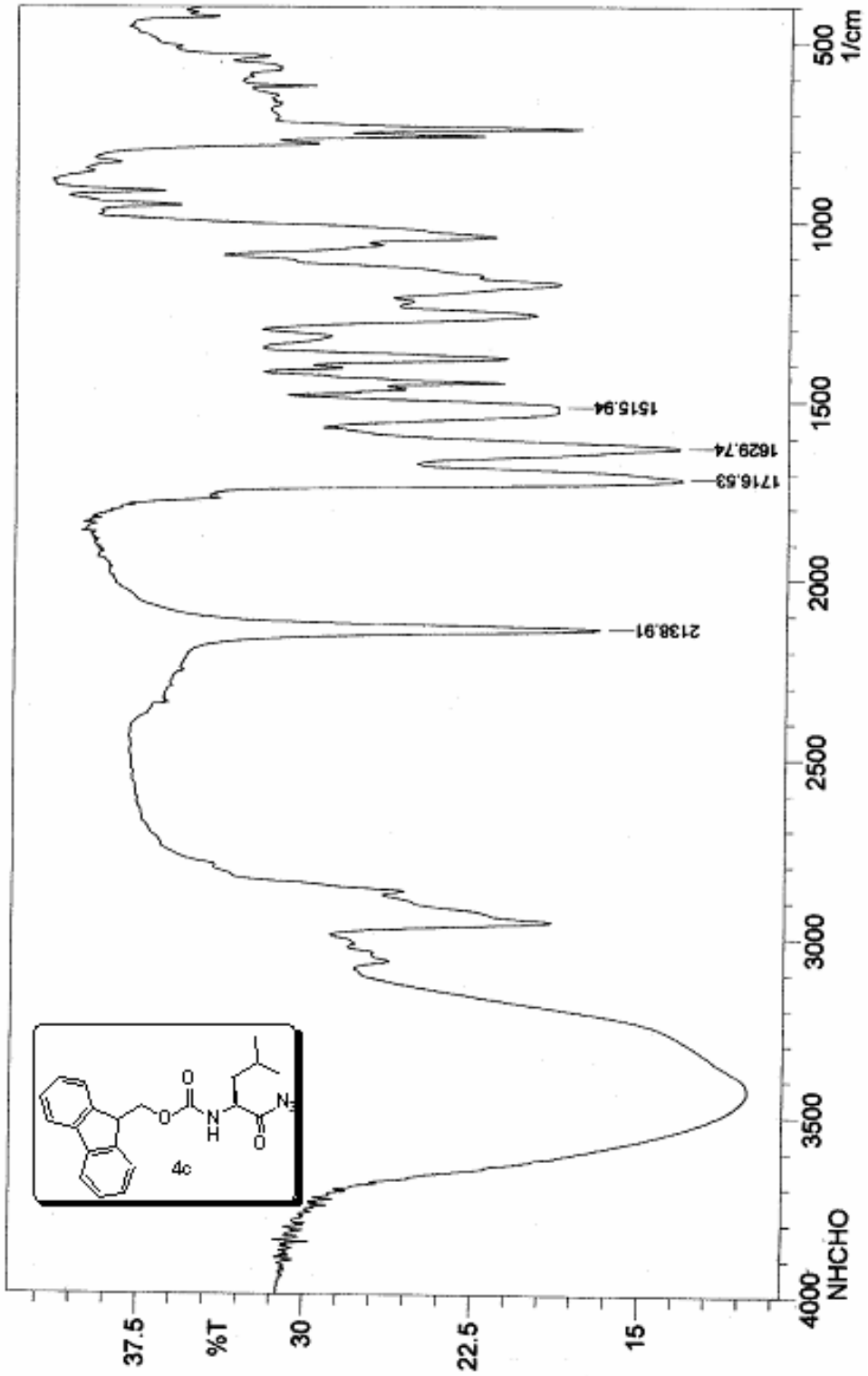

6f
ppm
3.738
2.403
1.066
2.208
1.000
H15


6f
14.98
62.37
77.50
77.82
78.14
112.80
117.68
125.15
140.69
154.59
H15
ppm
0
20
40
60
80
100
120
140
160
180
200

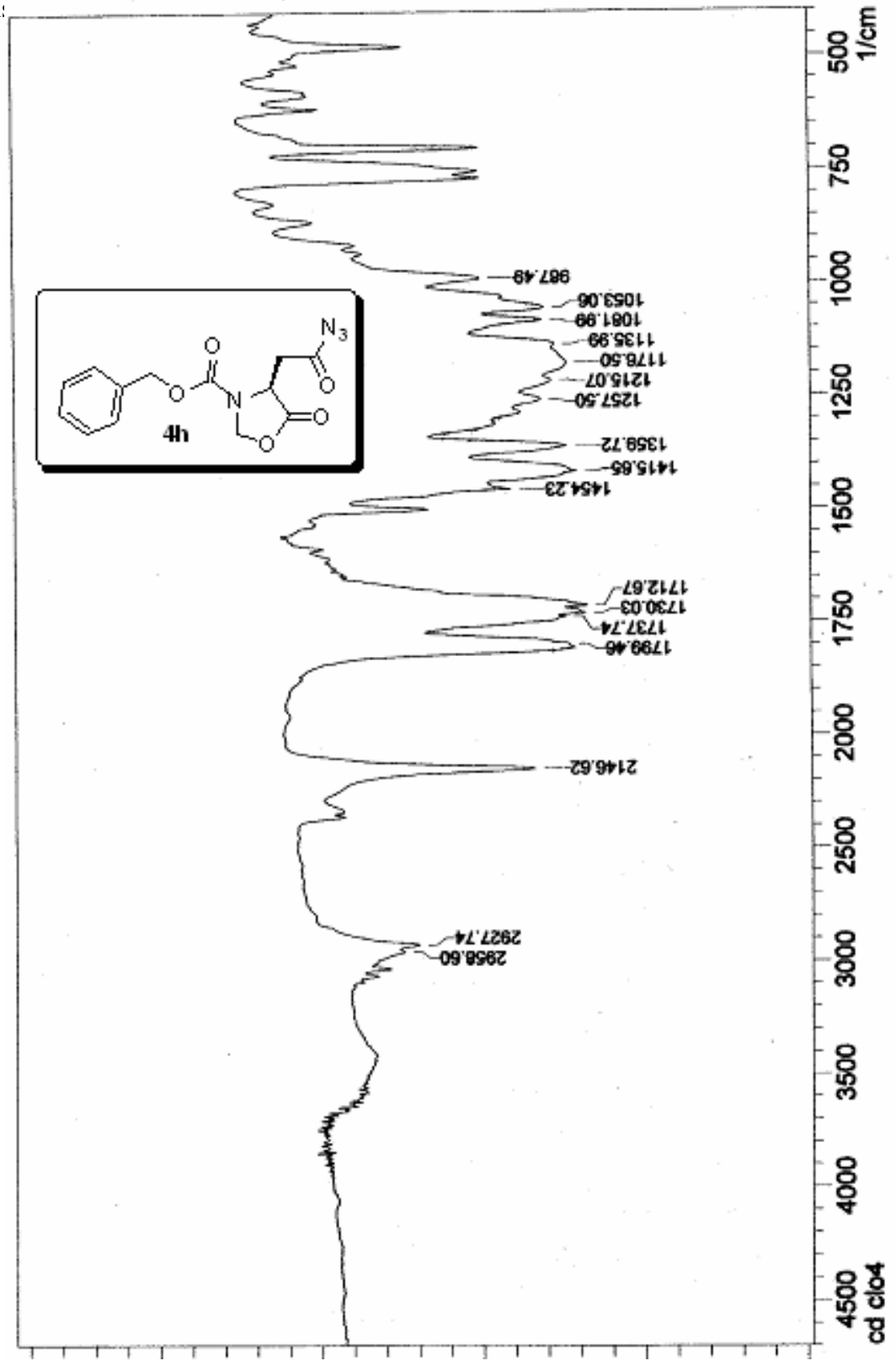

H-10
H-10 4 (0.096) AM (Cen,7, 99.00, Ar,5343.0,556.28,0.12,LS 10); Sm (SG, 2x4.00); Sb (4,40.00); Sb (4,40.00); Cm (2:6)
1: TOF MS ES+
3.28e3
Calcd Mass = 233.0538(M+Na)
233.0542
229.1306
413.2601
6g
%
m/z


6g
GOD-9
ppm
3.980
2.190
2.199
1.020
1.031
1.000


6g
14.88
62.31
77.28
77.60
77.92
124.81
130.25
135.66
139.92
154.12
GOD-9
ppm
0
20
40
60
80
100
120
140
160
180
200


4c
NHCHO
2138.91
1716.53
1629.74
1515.94
%T
37.5
30
22.5
15
4000
3500
3000
2500
2000
1500
1000
500
1/cm

Sup
This


4h
N3
O
O
O
N
O
O
987.49
1053.06
1081.99
1135.99
1178.50
1215.07
1257.50
1359.72
1415.85
1454.23
1712.67
1730.03
1737.74
1799.46
2146.62
2927.74
2958.60
500
750
1000
1250
1500
1750
2000
2500
3000
3500
4000
4500
1/cm
cd clo4