Supporting Information

Efficient Microwave-Assisted Synthesis of Aminoxy Acid Conjugates

Bogdan Draghici,[‡a] *Finn K. Hansen,*[‡a] *Ana-Maria Buciumas,*[a] *Bahaa El-Dien M. El-Gendy,*[a,b] *Ekaterina Todadze,*[a] *Alan R. Katritzky*[a*]

[a]Center for Heterocyclic Compounds, Department of Chemistry, University of Florida, Gainesville, FL 32611-7200, E-mail: Katritzky@chem.ufl.edu

[b]Department of Chemistry, Faculty of Science, Benha University, Benha, Egypt.

General procedure for the synthesis of *N*-Cbz-protected α-aminoxy acids 3a-d, (3c+3c'). NaH (2 equiv) was added portionwise to a stirring solution of benzylhydroxycarbamate (1 equiv) in THF under argon atmosphere. The resulting reaction mixture was stirred at -20 C for 15 min. A solution of the respective α-bromo carboxylic acid (1 equiv) in THF (3 mL) was added dropwise. The resulting reaction mixture was allowed to warm up to rt and stirred for additional 8h. The solvent was removed under reduced pressure. The resulting residue was washed with hexanes (2 x 20 mL) followed by hexanes:diethyl ether (1:1, 2 x 20 mL) The resulting residue was dissolved in water, acidified to pH = 3 (4N HCl) and extracted with diethyl ether (3 x 30 mL). The combined organic layers were dried over $MgSO_4$, the organic solvent was removed under reduced pressure to give the corresponding *N*-Cbz-protected α-aminoxy acids **3a-d, (3c+3c')**, which were used without further purification.

Compound **2a** was purchased from Sigma Aldrich Inc. and was used without any further purification. Compounds **2b-d, (2c+2c')** were prepared from commercially available α-amino acids according to literature procedure (*2*).

2-((((Benzyloxy)carbonyl)amino)oxy)acetic acid (3a). White solid, mp 68.0 - 69.0 °C, lit. mp 67.0 – 67.8 °C (*1*), 53% yield. ^{1}H-NMR (300 MHz, $CDCl_3$) δ, 8.37 (br s, 1H), 7.36 (br s, 5H), 5.21 (s, 2H), 4.49 (s, 2H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ, 172.8, 158.7, 135.0, 129.0, 128.9, 128.7, 73.7, 68.7.

(*R*)-2-((((Benzyloxy)carbonyl)amino)oxy)propanoic acid (3b). White solid, mp 82.0 – 85.0 °C, 59% yield. ^{1}H-NMR (300 MHz, $CDCl_3$) δ, 8.95 (br s, 1H), 8.52 (br s, 1H), 7.43 – 7.28 (m, 5H), 5.18 (s, 2H), 4.50 (q, J = 7.1 Hz, 1H), 1.48 (d, J = 7.1 Hz, 3H). ^{13}C-NMR

(75 MHz, $CDCl_3$) δ, 176.2, 158.4, 135.2, 128.8, 128.5, 80.3, 68.3, 16.4. Anal. Calcd for. $C_{11}H_{13}NO_5$: C 55.23; H 5.48; N 5.85. Found C 55.16; H 5.46; N 5.71.

(*R*)-2-((((Benzyloxy)carbonyl)amino)oxy)-3-phenylpropanoic acid (3c). Yellow oil, 47% yield. ^{1}H-NMR (300 MHz, $CDCl_3$) δ, 10.22 (br s, 1H), 8.64 (br s, 1H), 7.31 – 7.28 (m, 3H), 7.20 – 7.17 (m, 7H), 5.05 (s, 2H), 4.60 (dd, J = 8.5, 4.1 Hz, 1H), 3.16 (dd, J = 14.9, 4.1 Hz, 1H), 3.00 (dd, J = 14.9, 8.5 Hz, 1H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ, 174.2, 158.3, 135.7, 134.8, 129.1, 128.4, 128.2, 128.1, 126.8, 84.8, 68.0, 36.9.

(*R*,*S*)-2-((((Benzyloxy)carbonyl)amino)oxy)-3-phenylpropanoic acid (3c+3c'). Yellow oil, 53% yield. ^{1}H-NMR (300 MHz, $CDCl_3$) δ, 8.18 (br s, 1H), 7.41 – 7.20 (m, 10H), 5.13 (s, 2H), 4.61 (dd, J = 8.9, 3.7 Hz, 1H), 3.26 (dd, J = 14.7, 3.8 Hz, 1H), 3.04 (dd, J = 14.7, 8.9 Hz, 1H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ, 173.9, 158.8, 147.6, 129.5, 129.2, 128.9, 128.7, 128.6, 127.2, 86.1, 68.7, 37.4.

(*R*)-2-((((Benzyloxy)carbonyl)amino)oxy)-4-methylpentanoic acid (3d). Yellow oil, 61% yield. ^{1}H-NMR (300 MHz, $CDCl_3$) δ, 8.66 (s, 1H), 7.36 – 7.34 (m, 5H), 7.05 (br s, 2H), 5.16 – 5.10 (m, 2H), 4.42 (dd, J = 9.9, 3.8 Hz, 1H), 1.90 – 1.67 (m, 1H), 1.61 – 1.51 (m, 1H), 0.94 – 0.87 (m, 6H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ, 176.1, 158.1, 135.2, 128.7, 128.5, 123.3, 82.8, 68.1, 68.0, 39.8, 24.6, 23.2, 21.5. Anal. Calcd. for $C_{14}H_{19}NO_5$: C 59.78; H 6.81; N 4.98. Found C 59.47; H 7.22; N 5.10.

General procedure for *N*-Cbz-protected (α-aminoxyacyl)benzotriazoles 4a-d, (4c+4c'). Thionyl chloride (1 equiv) was added dropwise to a stirred solution of benzotriazole (3 equiv) in THF (20 mL). The resulting mixture was stirred at rt. for 30 min under argon. The *N*-Cbz-protected α-aminoxy acids **3a-d, (3c+3c')** (1 equiv) were each added portionwise and the resulting reaction mixtures were stirred at rt for additional 4h. Each precipitate was filtered off and the solvent removed under reduced presure to give oils that were redissolved in diethylether (10 mL), washed with water (20 mL) and Na_2CO_3 (10% solution, 2 x 20 mL), and the solvent evaporated to give a colorless oils which were crystallized from diethylether:hexane (1:2) to give *N*-Cbz-protected (α-aminoxyacyl)benzotriazoles **4a-d, (4c+4c')**.

Benzyl 2-(1*H*-benzotriazol-1-yl)-2-oxoethoxycarbamate (4a). White solid, mp 88.0 - 90.0 °C, lit. mp 86.0 – 87.0 °C (*1*), 76% yield. ^{1}H-NMR (300 MHz, $CDCl_3$) δ, 8.25 (ddd, J = 8.2, 0.9, 0.9 Hz, 1H), 8.14 (ddd, J = 8.2, 0.9, 0.9 Hz, 1H), 7.69 (ddd, J = 8.3, 7.2, 1.0 Hz, 1H), 7.69 (ddd, J = 8.3, 7.4, 1.0 Hz, 1H), 7.54 (ddd, J = 8.3, 7.1, 1.0 Hz, 1H), 7.38 – 7.35 (m, 5H), 5.55 (s, 2H), 5.22 (s, 2H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ, 168.1, 157.1, 146.0, 135.3, 131.1, 130.9, 128.8, 128.7, 128.5, 126.8, 120.6, 114.1, 74.7,68.1.

(*R*)-Benzyl (1-(*1H*-benzotriazol-1-yl)-1-oxopropan-2-yl)oxycarbamate (4b). White prisms, 88.5 – 91.0 ^{o}C, 77% yield. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.23 (dd, J = 8.3, 1.0 Hz, 1H), 8.15 (dd, J = 8.3, 1.0 Hz, 1H), 7.91 (s, 1H), 7.70 (ddd, J = 8.2, 7.1, 1.0 Hz, 1H), 7.55 (ddd, J = 8.2, 7.1, 1.0 Hz, 1H), 7.26 – 7.36 (m, 5H), 5.94 (q, J = 6.9 Hz, 1H), 5.19 (s, 2H), 1.77 (d, J = 7.0 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.2, 157.3, 146.0, 135.4, 131.2, 131.0, 128.8, 128.7, 128.5, 126.8, 120.6, 114.4, 80.8, 68.0, 17.2., Anal. Calcd. for $C_{17}H_{16}N_4O_4$, C, 60.00; H, 4.74; N, 16.46. found C, 59.93; H, 4.54; N, 16.36.

(*R*)-Benzyl(1-(1*H*-benzotriazol-1-yl)-1-oxo-3-phenylpropan-2-yl)oxycarbamate (4c). White prisms, mp 90.0 – 91.0 °C, 88% yield. ^{1}H-NMR (300 MHz, $CDCl_3$) δ, 8.28 (ddd, J = 8.2, 0.8, 0.8 Hz, 1H), 8.14 (ddd, J = 8.2, 0.8, 0.8 Hz, 1H), 7.79 (s, 1H), 7.68 (ddd, J = 8.2, 7.5, 0.8 Hz, 1H), 7.54 (ddd, J = 8.2, 7.5, 0.8 Hz, 1H), 7.32 – 7.19 (m, 10H), 6.14 (dd, J = 7.8, 4.3 Hz, 1H), 5.11 (s, 2H), 3.47 (dd, J = 14.7, 4.3 Hz, 1H), 3.36 (dd, J = 14.7, 7.9 Hz, 1H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ, 169.7, 157.2, 146.0, 135.3, 135.3, 131.0, 129.5, 128.7, 128.6, 128.6, 128.4, 127.3, 126.8, 120.5, 114.3, 85.1, 68.0, 37.8. Anal. Calcd. for $C_{23}H_{20}N_4O_4$: C 66.34; H 4.84; N 13.45. Found C 66.44; H 5.06; N 12.98.

(*R*,*S*)-Benzyl(1-(1*H*-benzotriazol-1-yl)-1-oxo-3-phenylpropan-2-yl)oxycarbamate (4c+4c'). White prisms, mp 114.0 – 116.0 °C, 82% yield. ^{1}H-NMR (300 MHz, $CDCl_3$) δ, 8.43 (ddd, J = 8.2, 1.0, 1.0 Hz, 0.2H), 8.24 (ddd, J = 8.2, 1.0, 1.0 Hz, 0.8H), 7.76 (s, 2H), 7.72 – 7.70 (m, 2H), 7.52 – 7.48 (m, 2H), 7.40 – 7.20 (m, 10H), 6.14 (dd, J = 7.8, 4.3 Hz, 1H), 5.11 (s, 2H), 3.43 (dd, J = 14.7, 4.3 Hz, 1H), 3.36 (dd, J = 14.7, 7.8 Hz, 1H).^{13}C-NMR (75 MHz, $CDCl_3$) δ, 157.2, 148.9, 135.3, 131.6, 131.0, 130.4, 129.5, 129.5, 128.6, 128.4, 127.3, 126.8, 126.4, 120.5, 116.2, 115.0, 114.4, 85.1, 68.0, 37.8. Anal. Calcd. for $C_{23}H_{20}N_4O$: C 66.34; H 4.84; N 13.45. Found C 66.50; H 4.88; N 13.67.

(*R*)-Benzyl(1-(1*H*-benzotriazol-1-yl)-4-methyl-1-oxopentan-2-yl)oxycarbamate (4d). Colorless oil, 86% yield. ^{1}H-NMR (300 MHz, $CDCl_3$) δ, 8.28 (dd, J = 8.2, 1.0 Hz, 1H), 8.13 (dd, J = 8.1, 1.1 Hz, 1H), 8.10 (s, 1H), 7.66 (ddd, J = 8.2, 7.2, 1.1 Hz, 1H), 7.53 (ddd, J = 8.2, 7.2, 1.1 Hz, 1H), 7.50 – 7.30 (m, 5H), 5.94 (dd, J = 9.8, 3.1 Hz, 1H), 5.18 (s, 2H), 2.18 – 2.08 (m, 1H), 1.94 – 1.80 (m, 2H), 1.10 (d, J = 6.7 Hz, 3H), 0.97 (d, J = 6.7 Hz, 3H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ, 171.3, 157.4, 146.0, 135.3, 131.2, 131.0,

128.7, 128.7, 128.6, 126.6, 120.4, 114.5, 83.5, 68.0, 40.5, 25.0, 23.4, 21.4. Anal. Calcd. for $C_{20}H_{22}N_4O_4$: C 62.82; H 5.80; N 14.65. Found C 62.63 H 5.90; N 14.18.

(*R*)-((3a*R*,5*R*,5a*S*,8a*S*,8b*R*)-2,2,7,7-Tetramethyltetrahydro-3a*H*-bis[1,3]dioxolo[4,5-*b*:4',5'-*d*]pyran-5-yl)methyl 2-(benzyl-oxycarbonylaminooxy)propanoate (6b). Colorless oil, 71% yield, $[\alpha]_D^{21}$ = +20.2 (c 0.14 in CH_2Cl_2). Anal. Calcd. for $C_{23}H_{31}NO_{10}$: C 57.37; H 6.49; N 2.91. Found C 57.48; H 6.76; N 2.81. ^{1}H-NMR (300 MHz, $CDCl_3$) δ, 8.20 (s, 1H), 7.37-7.32 (m, 5H), 5.54 (d, *J* = 5.0 Hz, 1H), 5.21-5.12 (m, 2H), 4.62 (dd, *J* = 7.7, 2.5 Hz, 1H), 4.54 (q, *J* = 7.0 Hz, 1H), 4.47 (dd, *J* = 11.4, 4.4 Hz, 1H), 4.33 (dd, *J* = 5.0, 2.5 Hz, 1H), 4.27-4.17 (m, 2H), 4.06-4.00 (m, 1H), 1.48 (d, *J* = 7.0 Hz, 3H), 1.45 (s, 3H), 1.43 (s, 3H), 1.32 (s, 3H), 1.26 (s, 3H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ, 172.0, 157.1, 135.7, 128.8, 128.6, 128.5, 110.1, 109.2, 96.5, 80.5, 71.1, 71.0, 70.5, 67.8, 65.9, 63.9, 26.1, 26.0, 25.0, 24.6, 16.4.

(*R*)-((3a*R*,5*R*,6*S*,6a*R*)-5-((*R*)-2,2-Dimethyl-1,3-dioxolan-4-yl)-2,2dimethyltetra-hydrofuro[2,3-*d*][1,3]dioxol-6-yl) 2-(benzyloxycarbonylaminooxy)-4-methyl-pentanoate (6c). Colorless oil, 60% yield, $[\alpha]_D^{21}$ = +32.5 (c 0.24 in CH_2Cl_2). Anal. Calcd for $C_{26}H_{37}NO_{10}$: C 59.64; H 7.12; N 2.68. Found: C 59.43; H 7.33; N 2.59. ^{1}H-NMR (300 MHz, $CDCl_3$) δ, 8.81 (s, 1H), 7.41-7.29 (m, 5H), 5.89 (d, *J* = 3.8 Hz, 1H), 5.23-5.05 (m, 3H), 4.56-4.50 (m, 2H), 4.35-4.29 (m, 1H), 4.22 (dd, *J* = 9.0, 3.6 Hz, 1H), 4.14 (dd, *J* = 9.0, 5.7 Hz, 1H), 4.07 (dd, *J* = 9.0, 3.3 Hz, 1H), 2.03-1.63 (m, 2H), 1.53 (s, 3H), 1.51-1.42 (m, 1H), 1.34 (s, 3H), 1.32 (s, 3H), 1.23 (s, 3H), 1.01-0.91 (m, 6H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ, 171.4, 156.7, 135.6, 128.7, 112.6, 110.2, 105.4, 84.0, 83.7, 80.3, 77.7, 73.0, 68.0, 67.8, 39.6, 27.0, 26.5, 24.9, 24.8, 23.2, 21.9.

(*R*)-((3a*R*,5*R*,6*S*,6a*R*)-5-((*R*)-2,2-Dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyltetra-hydrofuro[2,3-*d*][1,3]dioxol-6-yl) 2-(benzyloxycarbonylaminooxy)-3-phenyl-propanoate (6d). Colorless oil, 68% yield, $[\alpha]_D^{21}$ = +13.1 (c 0.18 in CH_2Cl_2). Anal.

Calcd for $C_{29}H_{35}NO_{10}$: C 62.47; H 6.33; N 2.51. Found: C 62.51; H 6.43; N 2.48. ^{1}H-NMR (300 MHz, $CDCl_3$) δ, 8.86 (s, 1H), 7.37-7.23 (m, 10H), 5.62 (d, J = 3.5 Hz, 1H), 5.16 (d, J = 12.0 Hz, 1H), 5.08 (d, J = 12.0 Hz, 1H), 5.02 (d, J = 3.4 Hz, 1H), 4.80 (dd, J = 6.7, 5.2 Hz, 1H), 4.29-4.02 (m, 5H), 3.24-3.06 (m, 2H), 1.49 (s, 3H), 1.32 (s, 3H), 1.28 (s, 3H), 1.21 (s, 3H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ, 169.8, 156.7, 135.5, 135.3, 129.7, 128.7, 128.7, 128.5, 127.3, 112.5, 110.2, 105.3, 85.4, 83.3, 80.2, 77.8, 73.2, 67.9, 67.8, 37.0, 27.0, 26.4, 24.7.

(*R*)-((3a*R*,5*R*,5a*S*,8a*S*,8b*R*)-2,2,7,7-Tetramethyltetrahydro-3a*H*-bis[1,3]dioxolo[4,5-*b*:4',5'-*d*]pyran-5-yl)methyl 2-(benzyl-oxycarbonylaminooxy)-3-phenylpropanoate (6e). Colorless oil, 84% yield, $[\alpha]_D^{21}$ = +12.3 (c 0.20 in CH_2Cl_2). Anal. Calcd for $C_{29}H_{35}NO_{10}$: C 62.47; H 6.33; N 2.51. Found: C 62.52; H 6.36; N 2.38. ^{1}H-NMR (300 MHz, $CDCl_3$) δ, 8.18 (s, 1H), 7.39-7.18 (m, 10H), 5.53 (d, J = 5.1 Hz, 1H), 5.17-5.02 (m, 2H), 4.69 (t, J = 6.4 Hz, 1H), 4.60 (dd, J = 7.8, 2.6 Hz, 1H), 4.39 (dd, J = 11.3, 5.1 Hz, 1H), 4.32 (ddd, J = 5.0, 2.6, 0.6 Hz, 1H), 4.25-4.12 (m, 2H), 4.03-3.94 (m, 1H), 3.19-3.06 (m, 2H), 1.45 (s, 3H), 1.43 (s, 3H), 1.31 (s, 3H),1.25 (s, 3H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ, 170.7, 157.0, 135.9, 135.6, 129.5, 128.7, 128.6, 127.1, 110.0, 109.2, 96.5, 85.2, 71.0, 70.9, 70.5, 67.7, 65.7, 63.8, 37.2, 26.1, 26.0, 25.0, 24.7.

((3a*R*,5*R*,5a*S*,8a*S*,8b*R*)-2,2,7,7-Tetramethyltetrahydro-3a*H*-bis[1,3]dioxolo[4,5-*b*:4',5'-*d*]pyran-5-yl)methyl 2-(benzyloxy-carbonylaminooxy)-3-phenylpropanoate (6e + 6e'). Colorless oil, 65% yield, $[\alpha]_D^{21}$ = -14.1 (c 0.18 in CH_2Cl_2). Anal. Calcd for $C_{29}H_{35}NO_{10}$: C 62.47; H 6.33; N 2.51. Found: C 62.47; H 6.73; N 2.31. ^{1}H-NMR (300 MHz, $CDCl_3$) δ, 8.21 (s, 0.6H), 8.14 (s, 0.4H), 7.32-7.24 (m, 10H), 5.58-5.48 (m, 1H), 5.18-5.04 (m, 2H), 4.75-4.55 (m, 2H), 4.48-3.96 (m, 5H), 3.18-3.10 (m, 2H), 1.50-1.40

(m, 6H), 1.32-1.24 (m, 6H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ, 170.7, 170.7, 157.0, 135.9, 135.7, 135.6, 129.5, 128.7, 128.5, 128.3, 127.0, 110.0, 109.2, 109.0, 96.5, 85.1, 84.7, 70.9, 70.9, 70.6, 70.4, 67.7, 67.6, 66.0, 65.6, 64.0, 63.7, 37.1, 37.0, 26.1, 25.9, 25.0, 24.6.

(*R*)-((*R*)-2-Hydroxy-2-((3a*R*,5*R*,6*S*,6a*R*)-6-hydroxy-2,2-dimethyltetrahydro-furo[3,2-*d*][1,3]dioxol-5-yl)ethyl) 2-(benzyloxycarbonylaminooxy)propanoate (6f). White microcrystals, mp 112.0-115.0 °C, 62% yield, $[\alpha]_D^{21}$ = +43.3 (c 0.13 in CH_2Cl_2). Anal. Calcd for $C_{20}H_{27}NO_{10}$: C 54.42; H 6.16; N 3.17. Found: C 54.46; H 6.22; N 3.00. ^{1}H-NMR (300 MHz, DMSO-d_6) δ, 10.50 (s, 1H), 7.40-7.30 (m, 5H), 5.79 (d, J = 3.5 Hz, 1H), 5.27 (d, J = 4.5 Hz, 1H), 5.14-5.04 (m, 3H), 4.43-4.26 (m, 3H), 4.07-3.84 (m, 4H), 1.36 (s, 3H), 1.33 (d, J = 6.9 Hz, 3H), 1.22 (s, 3H). ^{13}C-NMR (75 MHz, DMSO-d_6) δ, 171.1, 157.1, 136.2, 128.4, 128.0, 127.9, 110.6, 104.5, 84.6, 80.2, 79.1, 72.8, 67.3, 66.1, 65.1, 26.7, 26.2, 16.3.

(*R*,*Z*)-3,7-Dimethylocta-2,6-dien-1-yl 2-((((benzyloxy)-carbonyl)amino)oxy)-propanoate (8a). Colorless oil, 60% yield, $[\alpha]_D^{21}$ = +91.0 (c 0.22 in CH_2Cl_2). HRMS calcd. for $[C_{21}H_{29}NO_5+Na]^+$= 398.1938, found = 398.1949. ^{1}H-NMR (300 MHz, $CDCl_3$) δ, 8.11 (s, 1H), 7.35-7.33 (m, 5H), 5.34 (t, *J* = 7.7 Hz, 1H), 5.58 (d, *J* = 12.3 Hz, 1H), 5.13 (d, *J* = 12.3 Hz, 1H), 5.10-5.06 (m, 1H), 4.64 (d, *J* = 7.6 Hz, 2H), 4.50 (q, *J* = 7.0 Hz, 1H), 2.10-2.05 (m, 4H), 1.76 (s, 3H), 1.67 (s, 3H), 1.59 (s, 3H), 1.45 (d, *J* = 7.0 Hz, 3H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ, 172.2, 157.1, 143.4, 135.6, 132.3, 128.6, 128.4, 128.3, 123.5, 118.6, 79.8, 67.5, 61.9, 32.2, 26.6, 25.7, 23.5, 17.7, 16.3.

(*R*,*S*)-(*E*)-3,7-Dimethylocta-2,6-dien-1-yl 2-((((benzyloxy) carbonyl)amino)oxy)-3-phenylpropanoate (8b+8b'). Colorless oil, 54% yield, $[\alpha]_D^{21}$ = +0.01 (c 1.96 in CH_2Cl_2). Anal. Calcd for $C_{27}H_{33}NO_5$: C 71.82; H 7.37; N 3.10. Found: C 72.15; H 7.75; N 3.25. ^{1}H-NMR (300 MHz, $CDCl_3$) δ, 7.78 (s, 1H), 7.37-7.28 (m, 5H), 7.28-7.18 (m, 5H), 5.24 (t, *J* = 7.2 Hz, 1H), 5.15 (d, *J* = 12.1 Hz. 1H), 5.10 (d, *J* = 12.1 Hz, 1H), 5.10-5.04 (m, 1H), 4.67 (dd, *J* = 7.0, 5.2 Hz, 1H), 4.62 (d, *J* = 7.2 Hz, 2H), 3.16 (dd, *J* = 12.1, 4.9 Hz, 1H), 3.09 (dd, *J* = 12.5, 5.1 Hz, 1H), 2.10-2.03 (m, 4H), 1.68 (s, 3H), 1.67 (s, 3H), 1.60 (s, 3H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ, 170.9, 157.0, 143.3, 135.8, 135.5, 132.1, 129.5, 128.7, 128.5, 128.4, 127.0, 123.7, 117.6, 84.5, 67.7, 62.3, 39.7, 37.3, 26.4, 25.9, 17.9, 16.7.

(*R*,*E*)-2,7-Dimethyloctal-2,6-dien-1-yl-2-(((benzyloxy)-carbonyl)amino)oxy)-4-metylpentanoate (8c). Colorless oil, 50% yield, $[\alpha]_D^{21}$ = +56.8 (c 0.29 in CH_2Cl_2). Anal. Calcd for $C_{24}H_{35}NO_5$: C 69.04; H 8.45; N 3.35. Found: C 68.70; H 8.87; N 3.22. ^{1}H-NMR (300 MHz, $CDCl_3$) δ, 8.24 (br s, 1H), 7.32 (s, 5H), 5.34 (t, *J* = 7.2 Hz, 1H), 5.18 (d, *J* = 12.1 Hz, 1H), 5.12 (d, *J* = 12.3 Hz, 1H), 5.09-5.08 (m, 1H), 4.63 (d, *J* = 7.3 Hz, 2H),

4.44 (dd, J = 9.8, 3.8 Hz, 1H), 2.11-2.10 (m, 6H), 1.99-1.88 (m, 1H), 1.76 (s, 3H), 1.67 (s, 3H), 1.59 (s, 3H), 0.93 (d, J = 5.7 Hz, 3H), 0.91 (d, J = 5.1 Hz, 3H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ, 172.3, 157.3, 143.2, 135.7, 132.2, 128.6, 128.4, 123.6, 118.7, 82.7, 67.6, 61.8, 61.2, 39.9, 32.2, 26.7, 25.8, 24.6, 23.6, 23.2, 21.6, 17.7.

(2*R*)-(10*R*,13*R*)-17-((2*R*,5*R*)-5-Ethyl-6-methylheptan-2-yl)-10,13-dimethyl-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradeca-hydro-1*H*-cyclopenta[*a*]phenanthren-3-yl 2-((((benzyloxy)-carbonyl)amino)oxy)propanoate (10a). White microcrystals, mp 102.0-103.0 °C, 22% yield, $[\alpha]_D^{21}$ = +25.3 (c 0.23 in CH_2Cl_2). Anal. Calcd for $C_{40}H_{61}NO_5$: C 75.55; H 9.67; N 2.20. Found: C 75.36; H 10.04; N 2.13. ^{1}H-NMR (300 MHz, $CDCl_3$) δ, 7.88 (s, 1H), 7.37-7.35 (m, 5H), 5.38 (d, J = 3.8 Hz, 1H), 5.20 (d, J = 12.1 Hz, 1H), 5.15 (d, J = 12.1 Hz, 1H), 4.70-4.67 (m, 1H), 4.47 (q, J = 7.0 Hz, 1H), 2.33 (d, J = 7.7 Hz, 2H), 2.02 - 1.95 (m, 2H), 1.95-1.61 (m, 2H), 1.61-0.76 (m, 41H), 0.67 (s, 3H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ, 171.6, 157.0, 139.4, 135.6, 128.7, 128.6, 128.4, 123.2, 79.2, 75.2, 67.7, 56.8, 56.2, 50.1, 46.0, 42.5, 39.9, 39.0, 38.4, 37.0, 36.7, 36.3, 36.0, 34.1, 33.9, 32.6, 32.1, 32.0, 30.4, 29.3, 28.4, 27.9, 26.2, 24.4, 23.2, 21.2, 20.4, 20.0, 19.5, 19.2, 18.9, 18.9, 18.4, 16.4, 15.5, 12.2, 12.0.

(2*R*)-(10*R*,13*R*)-10,13-Dimethyl-17-((*R*)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclo-penta[*a*]phenanthren-3-yl 2-((((benzyloxy)carbonyl)amino)-oxy)-3-phenylpropanoate (10b). White microcrystals, mp 114.0-115.0 °C, 25% yield, $[\alpha]_D^{21}$ = +20.4 (c 0.21 in CH_2Cl_2). Anal. Calcd for $C_{44}H_{61}NO_5$: C 77.27; H 8.99; N 2.05. Found: C 76.92; H 9.33; N 1.96. ^{1}H-NMR (300 MHz, $CDCl_3$) δ, 7.79 (s, 1H), 7.36-7.31 (m, 5H), 7.28-7.21 (m, 5H), 5.36 (d, J = 4.7 Hz, 1H), 5.16 (d, J = 12.1 Hz, 1H), 5.10 (d, J = 12.1 Hz, 1H), 4.64 (dd, J = 6.8, 5.4 Hz, 1H), 4.59 (dd, J = 8.4, 4.8 Hz, 1H), 3.13 (s, 1H), 3.10 (d, J = 1.6 Hz, 1H), 2.25 (d, J = 7.7 Hz, 2H), 2.07-1.90 (m, 2H), 1.90-1.69 (m, 3H), 1.62-1.10 (m, 17H), 1.00-0.82 (m, 13H), 0.67 (s, 3H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ, 170.4, 157.1, 139.4, 135.9, 135.6, 129.6, 128.7, 128.6, 128.4, 128.4, 127.0, 123.1, 84.5, 75.3, 67.7, 56.8, 56.3, 50.1, 42.4,

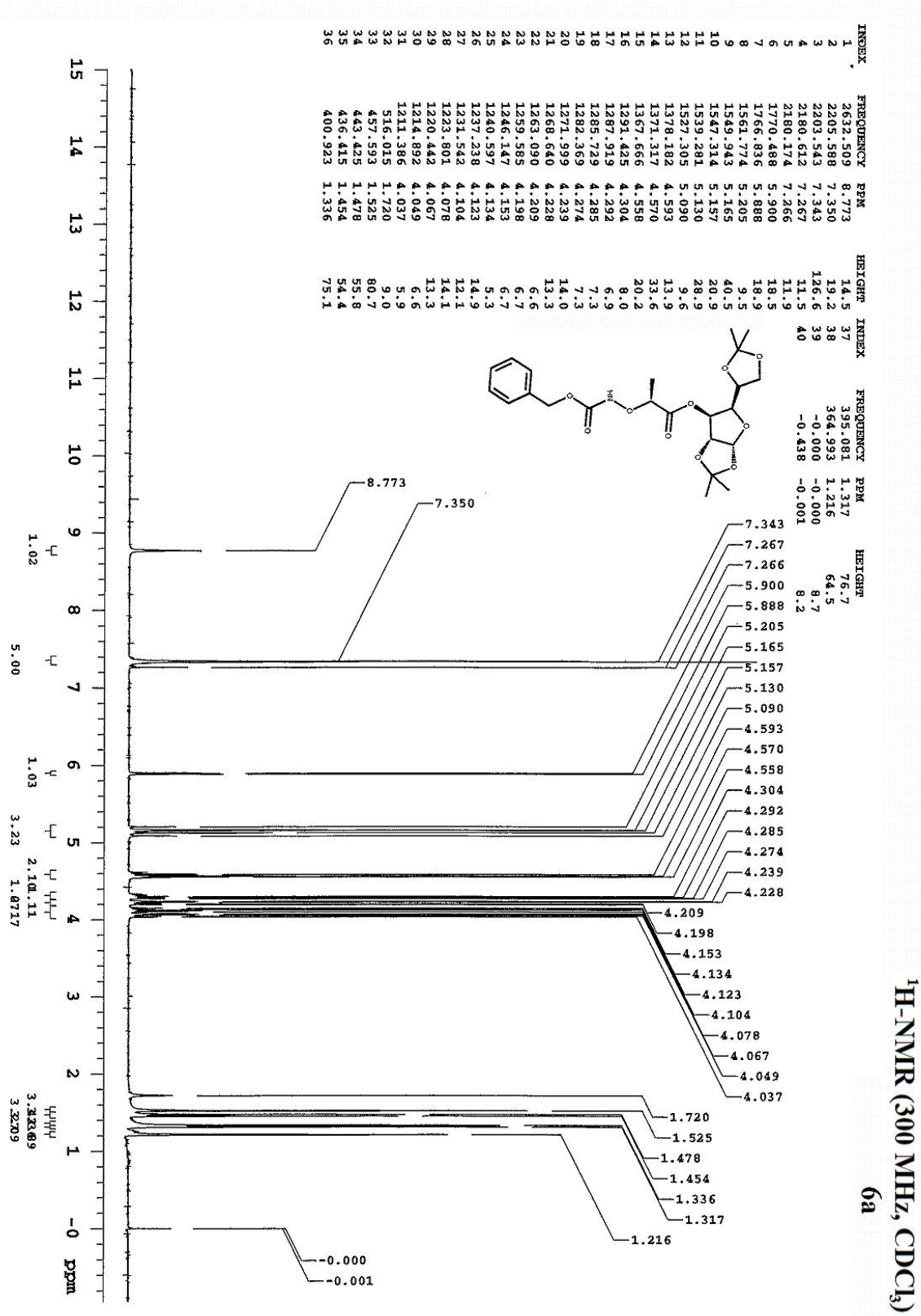
39.8, 39.7, 38.1, 37.3, 37.0, 36.7, 36.3, 35.9, 32.0, 32.0, 28.4, 28.2, 27.7, 24.4, 24.0, 23.0, 22.7, 21.2, 19.4, 18.9, 12.0.

(10*R*,13*R*)-10,13-Dimethyl-17-((*R*)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclo-penta[*a*]phenanthren-3-yl 2-((((benzyloxy)carbonyl)amino)-oxy)-3-phenylpropanoate (10b+10b'). White microcrystals, mp 112.0-114.0 °C, 25% yield, $[\alpha]_D^{21}$ = -14.6 (c 0.19 in CH_2Cl_2). Anal. Calcd for $C_{44}H_{61}NO_5$: C 77.27; H 8.99; N 2.05. Found: C 77.04; H 9.33; N 2.13. ^{1}H-NMR (300 MHz, $CDCl_3$) δ, 7.76 (s, 0.5H), 7.76 (s, 0.5H), 7.37-7.30 (m, 5H), 7.28-7.21 (m, 5H), 5.36-5.34 (m, 1H), 5.16 (d, J = 12.1 Hz, 1H), 5.10 (d, J = 12.2 Hz, 1H), 4.63 (t, J = 6.2 Hz, 1H), 4.60-4.57 (m, 1H), 3.12 (d, J = 6.5 Hz, 2H), 2.28-1.67 (m, 7H), 1.57-0.85 (m, 33H), 0.67 (s, 3H). ^{13}C-NMR (75 MHz, $CDCl_3$) δ, 170.4, 157.1, 139.4, 135.9, 135.6, 129.6, 128.6, 128.4, 128.4, 127.0, 123.1, 84.5, 75.4, 67.7, 56.8, 56.3, 50.1, 42.5, 39.9, 39.7, 38.1, 38.0, 37.3, 37.0, 36.7, 36.3, 35.9, 32.0, 31.2, 28.4, 28.2, 27.8, 27.7, 24.4, 24.0, 23.0, 22.7, 21.2, 19.4, 18.9, 12.0.

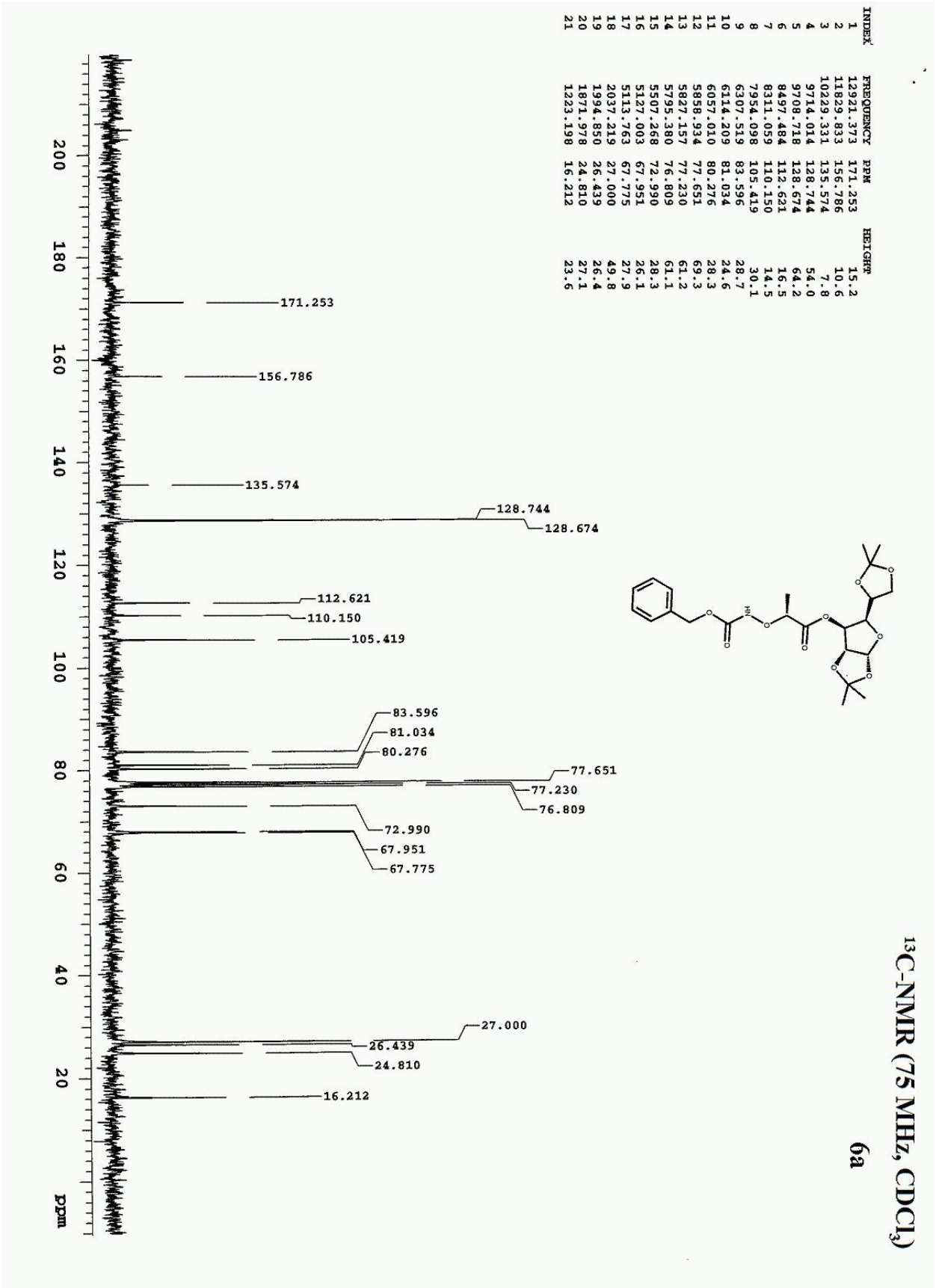
(2*R*)-(10*R*,13*R*)-17-((2*R*,5*S*,*E*)-5-Ethyl-6-methylhept-3-en-2-yl)-10,13-dimethyl-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetrade-cahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl 2-((((benzyloxy)-carbonyl)amino)oxy)-4-methylpentanoate (10d). White microcrystals, mp 120.0-122.0 °C, 20% yield, $[\alpha]_D^{21}$ = +25.9 (c 0.10 in CH_2Cl_2). HRMS calcd. for $[C_{43}H_{65}NO_5+Na]^+$= 698.4755, found = 698.4774. ^{1}H-NMR (300 MHz, $CDCl_3$) δ, 7.79 (s, 1H), 7.36-7.34 (m, 5H), 5.38 (d, J = 5.0 Hz, 1H), 5.20 (d, J = 12.1 Hz, 1H), 5.14 (d, J = 12.1 Hz, 1H), 5.01 (dd, J = 15.2, 8.5 Hz, 1H), 4.69-4.67 (m, 1H), 4.40 (dd, J = 9.8, 3.9 Hz, 1H), 2.33 (d, J = 7.7 Hz, 2H), 2.11-1.82 (m, 6H), 1.78-1.38 (m, 14H), 1.38-1.08 (m, 5H), 1.03-0.98 (m, 8H), 0.95-0.93 (m, 6H), 0.88-0.78 (m, 9H), 0.70 (s, 3H). ^{13}C-

NMR (75 MHz, $CDCl_3$) δ, 171.7, 157.0, 139.4, 138.4, 135.6, 129.4, 128.7, 128.6, 128.5, 123.1, 82.8, 75.1, 67.8, 64.3, 56.9, 56.1, 51.4, 50.2, 42.4, 40.7, 39.9, 39.8, 38.2, 37.1, 36.7, 32.0, 29.1, 27.9, 25.6, 24.7, 24.5, 23.3, 21.7, 21.4, 21.3, 19.5, 19.2, 12.4, 12.2.

Benzyl ((*R*)-1-((9-((2*R*,3*R*,4*S*,5*R*)-3,4-dihydroxy-5-(hydroxy-methyl)tetrahydro-furan-2-yl)-9*H*-purin-6-yl)amino)-1-oxo-3-phenylpropan-2-yl)oxycarbamate (12b). White microcrystals, mp 110.0-113.0°C, 54% yield, $[\alpha]_D^{21}$ = -9.6 (c 0.5 in CH_3OH). HRMS calcd. for $[C_{27}H_{28}N_6O_8+Na]^+$= 587.1861, found = 587.1859. ^{1}H-NMR (500 MHz, DMSO-d_6) δ, 10.52 (brs, 1H), 8.38 (s, 1H), 8.35 (s, 1H), 7.46 (br s, 1H), 7.38-7.20 (m, 10H), 5.94 (d, J = 7.5 Hz, 2H), 5.36 (d, J = 5.6 Hz, 1H), 5.09 (d, J = 12.8 Hz, 1H), 5.06 (d, J = 12.8 Hz, 1H), 4.72 (dd, J = 7.5, 4.5 Hz, 1H), 4.10 (br s, 1H), 3.68 (dd, J = 12.4, 3.4 Hz, 1H), 3.59 (dd, J = 12.2, 3.0 Hz, 1H), 3.20 (dd, J = 14.9, 4.5 Hz, 1H), 3.04 (dd, J = 14.8, 7.4 Hz, 1H). ^{13}C-NMR (125 MHz, DMSO-d_6) δ, 169.4, 157.0, 152.3, 149.0, 140.0, 136.2, 129.2, 129.0, 128.4, 128.1, 128.0, 127.8, 126.5, 119.4, 87.5, 83.5, 83.2, 74.3, 71.6, 66.1, 61.5, 36.5.

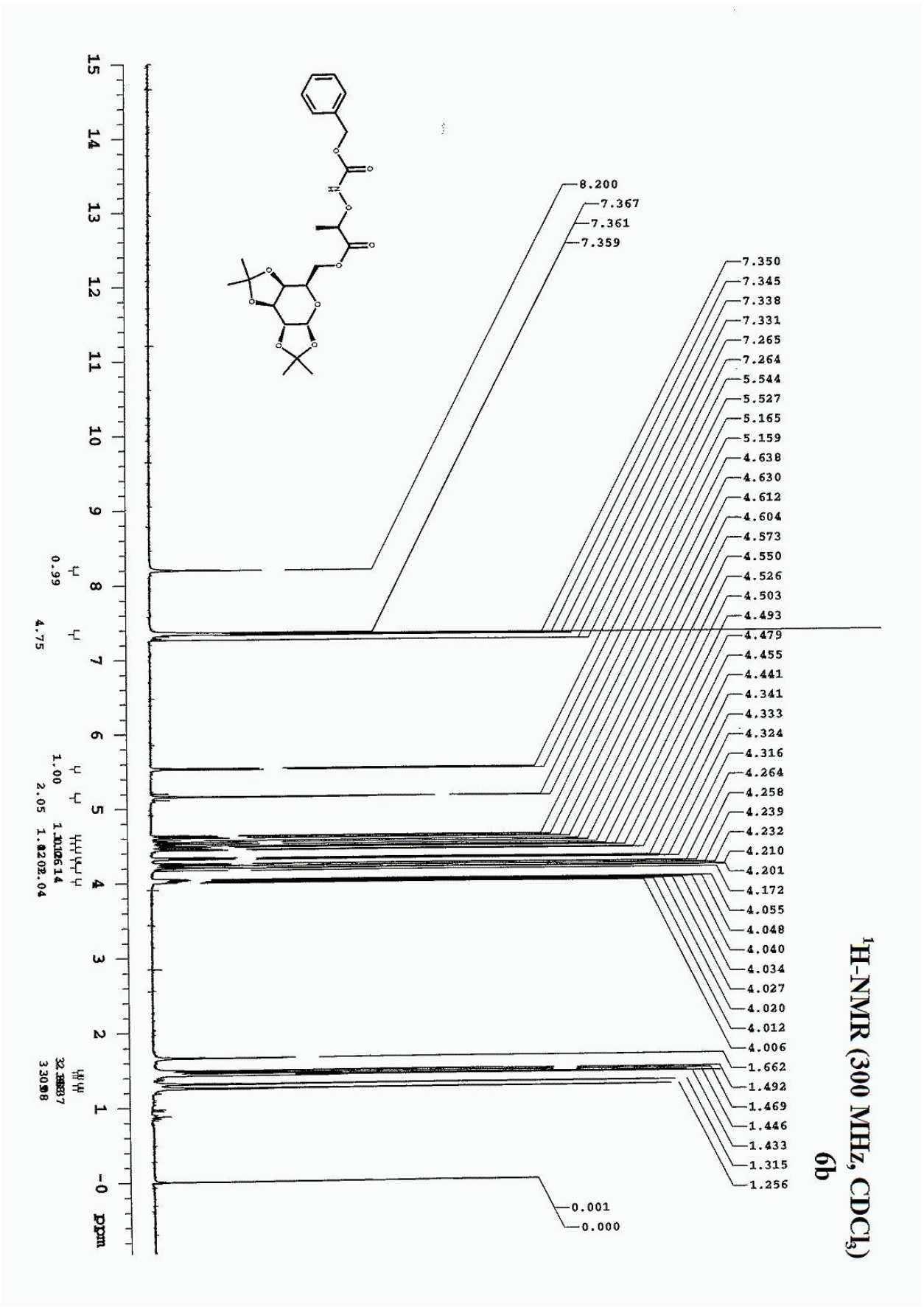


^{1}H-NMR (300 MHz, $CDCl_3$) 6a



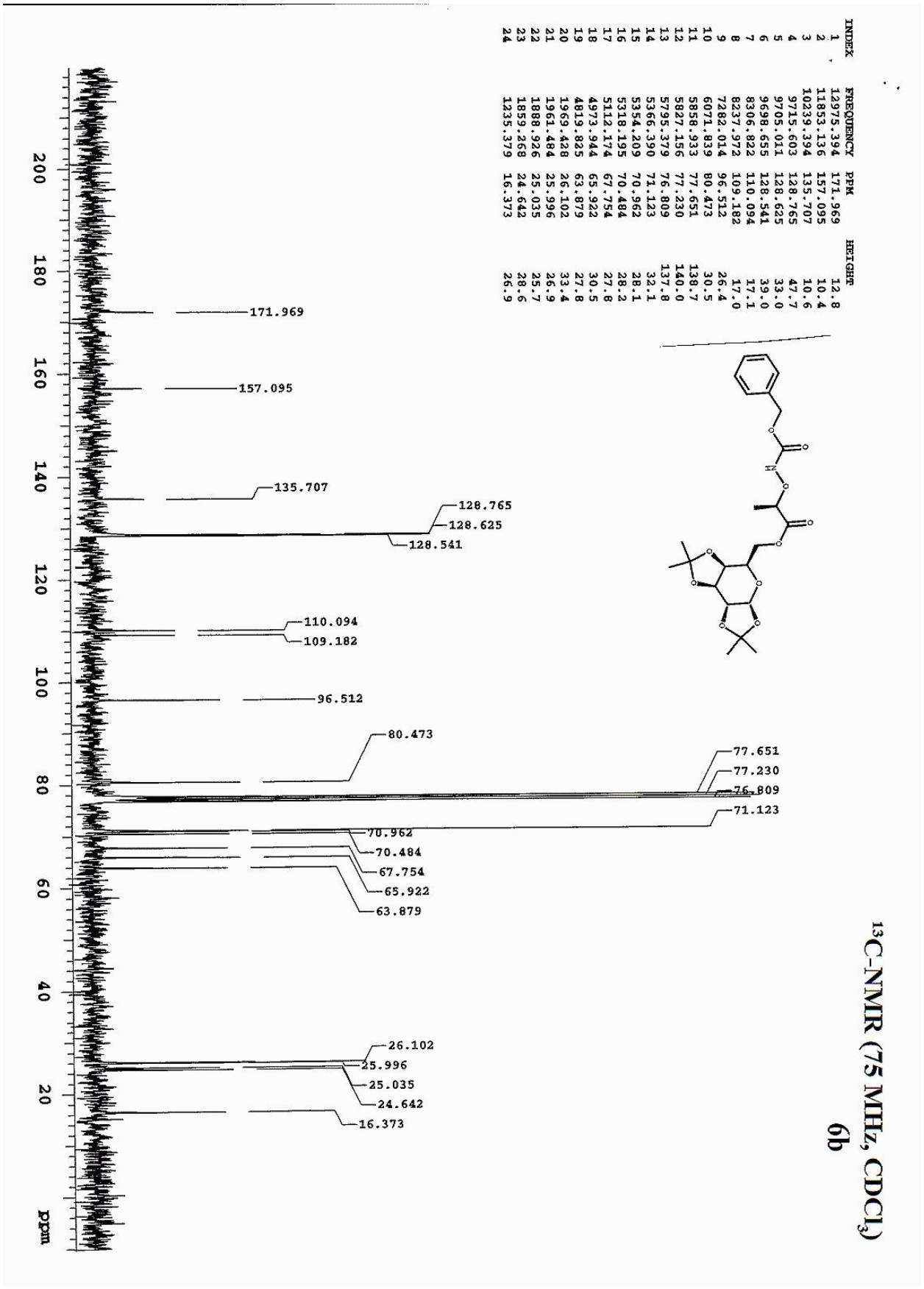
^{13}C–NMR (75 MHz, $CDCl_3$)

6a



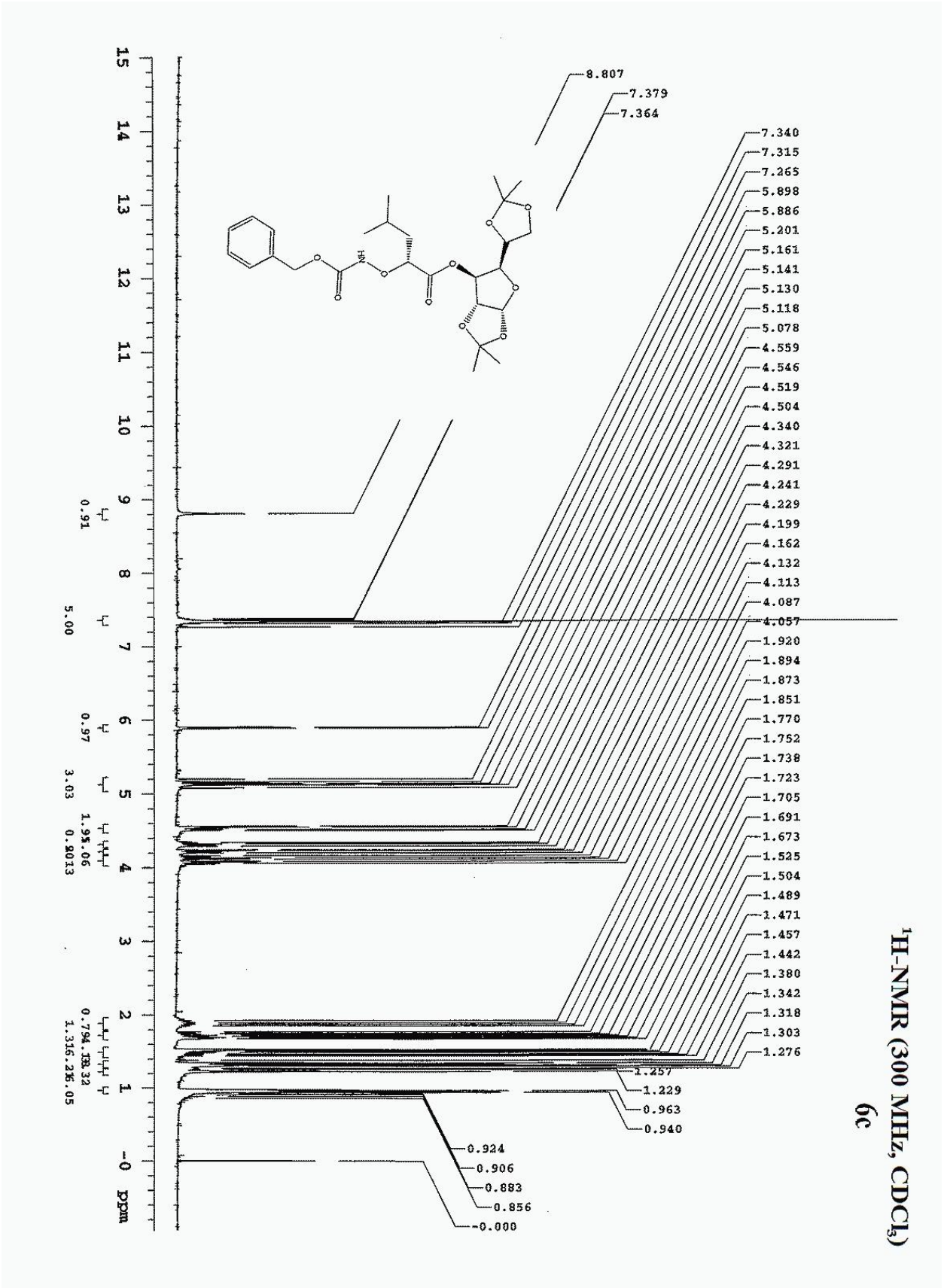
^{1}H-NMR (300 MHz, $CDCl_3$)

6b



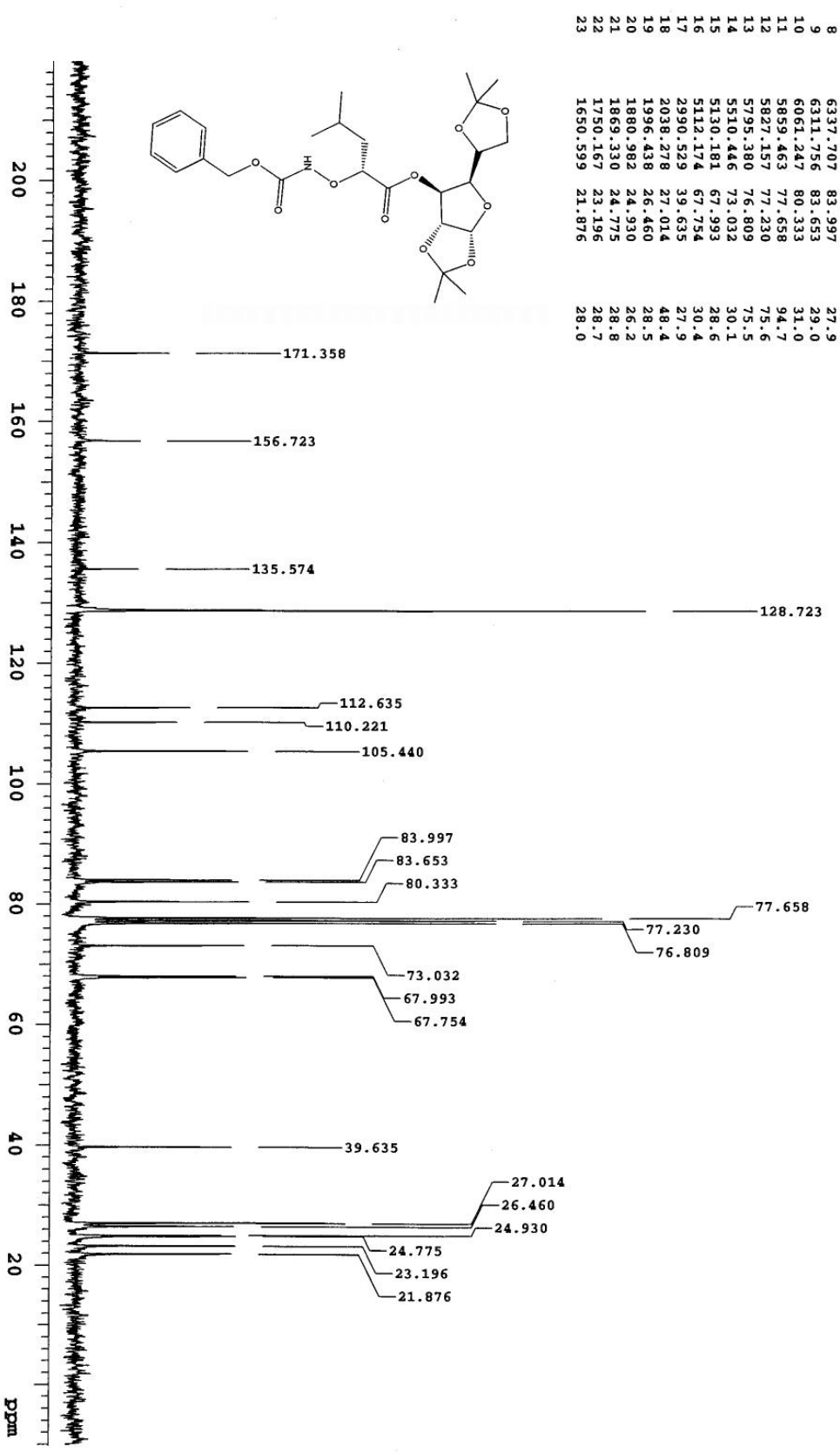
^{13}C-NMR (75 MHz, $CDCl_3$)

6b



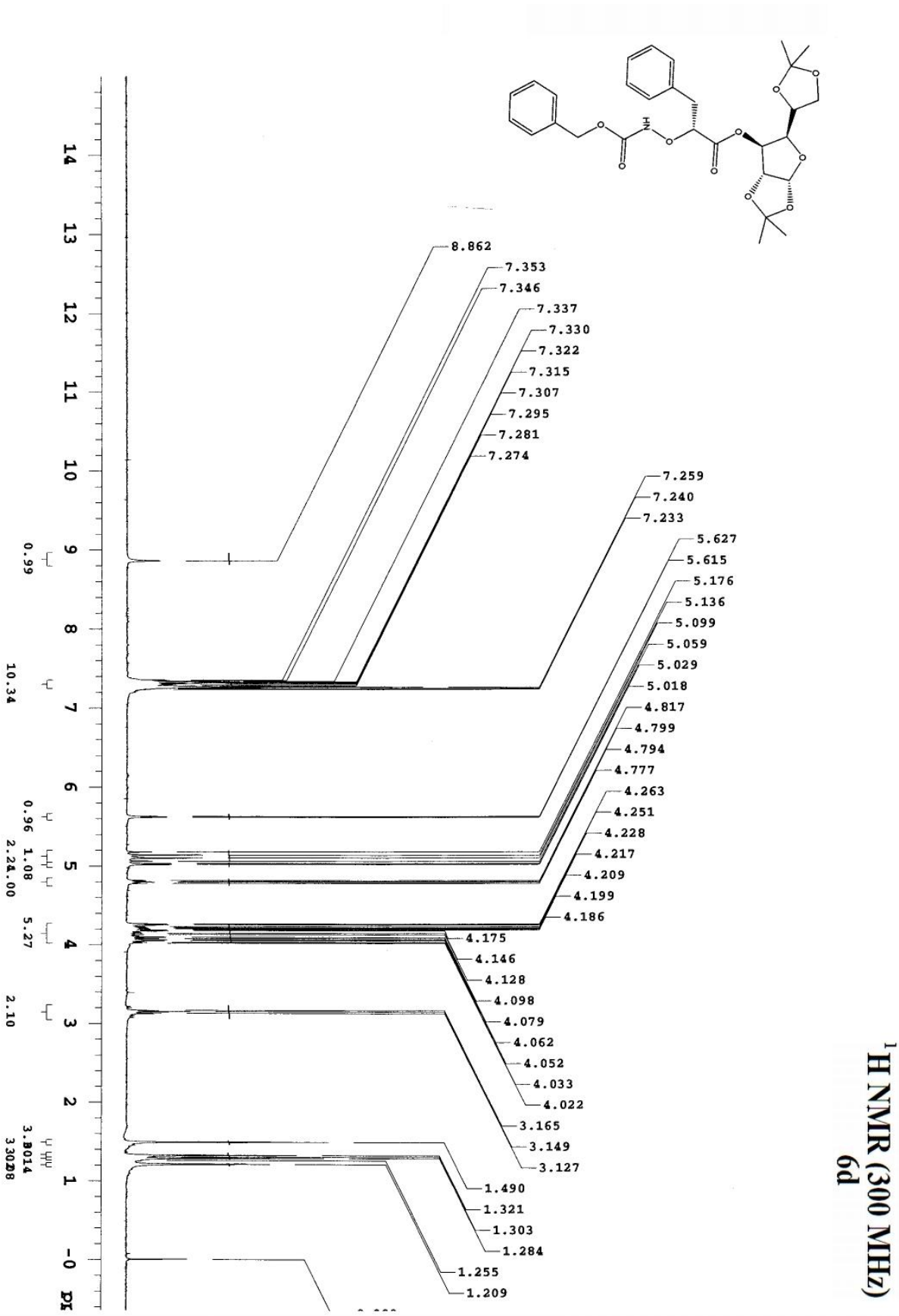
^{1}H-NMR (300 MHz, $CDCl_3$)

39c

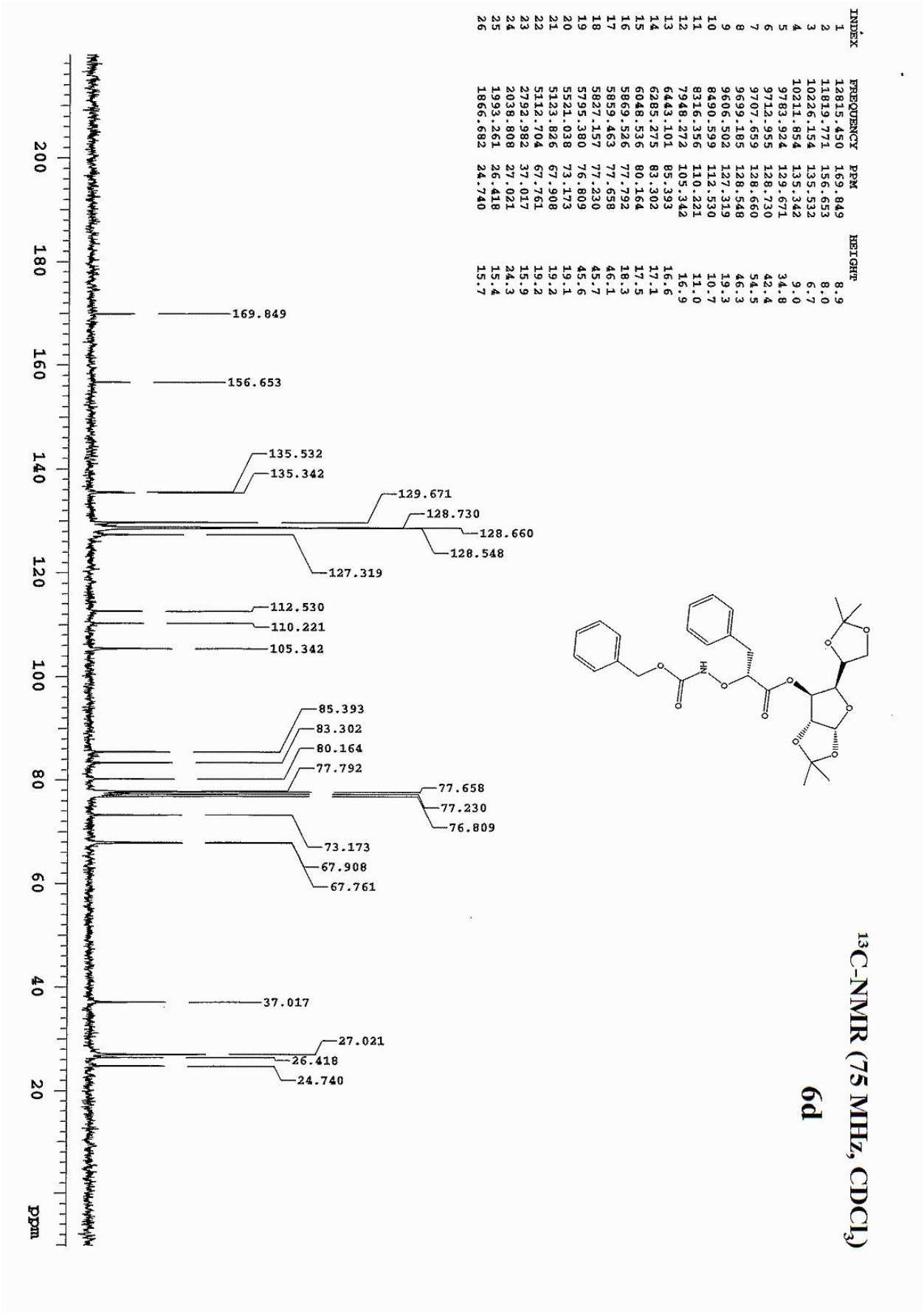


^{13}C-NMR (75 MHz, $CDCl_3$)

6c

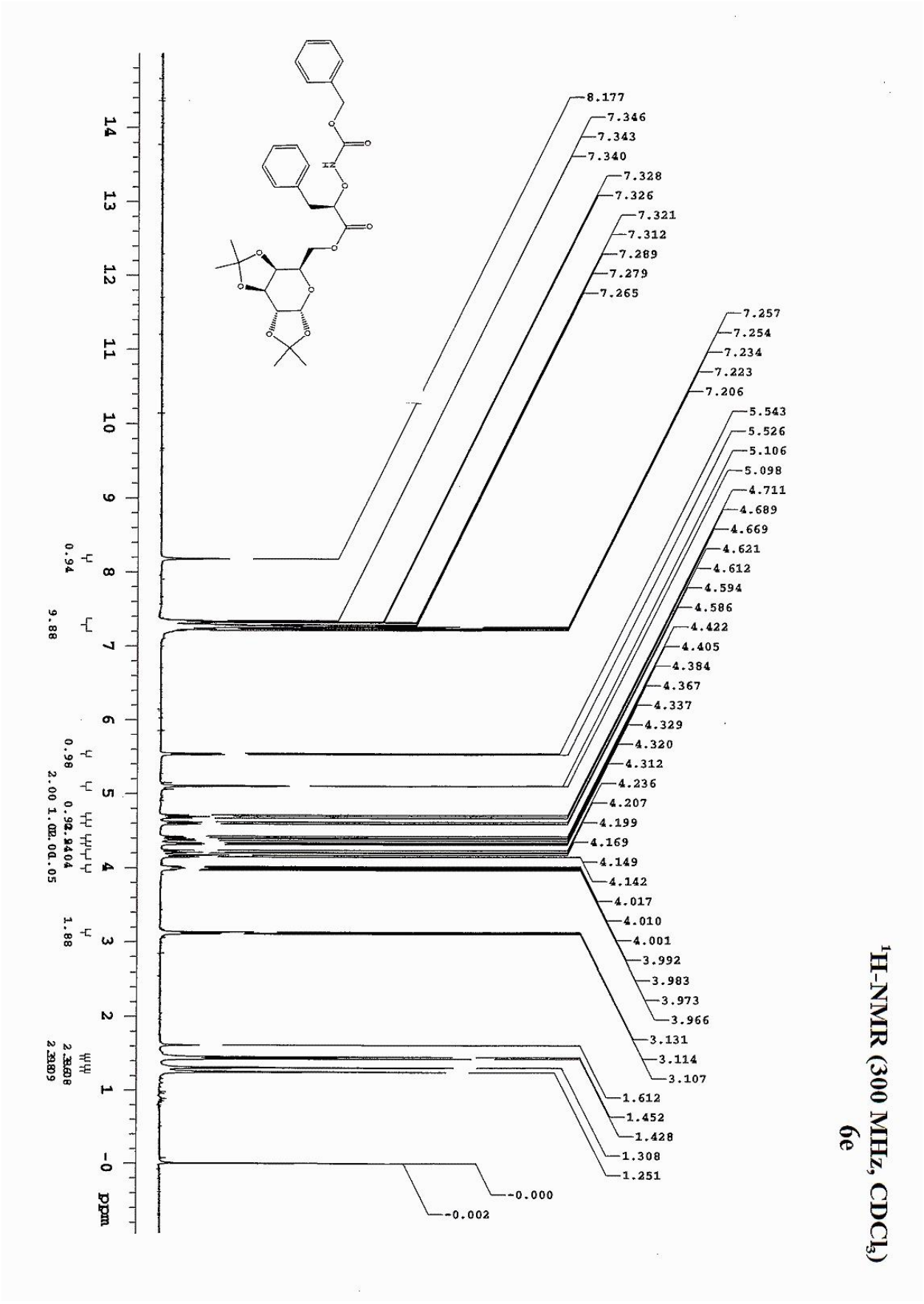


^{1}H NMR (300 MHz)
6d

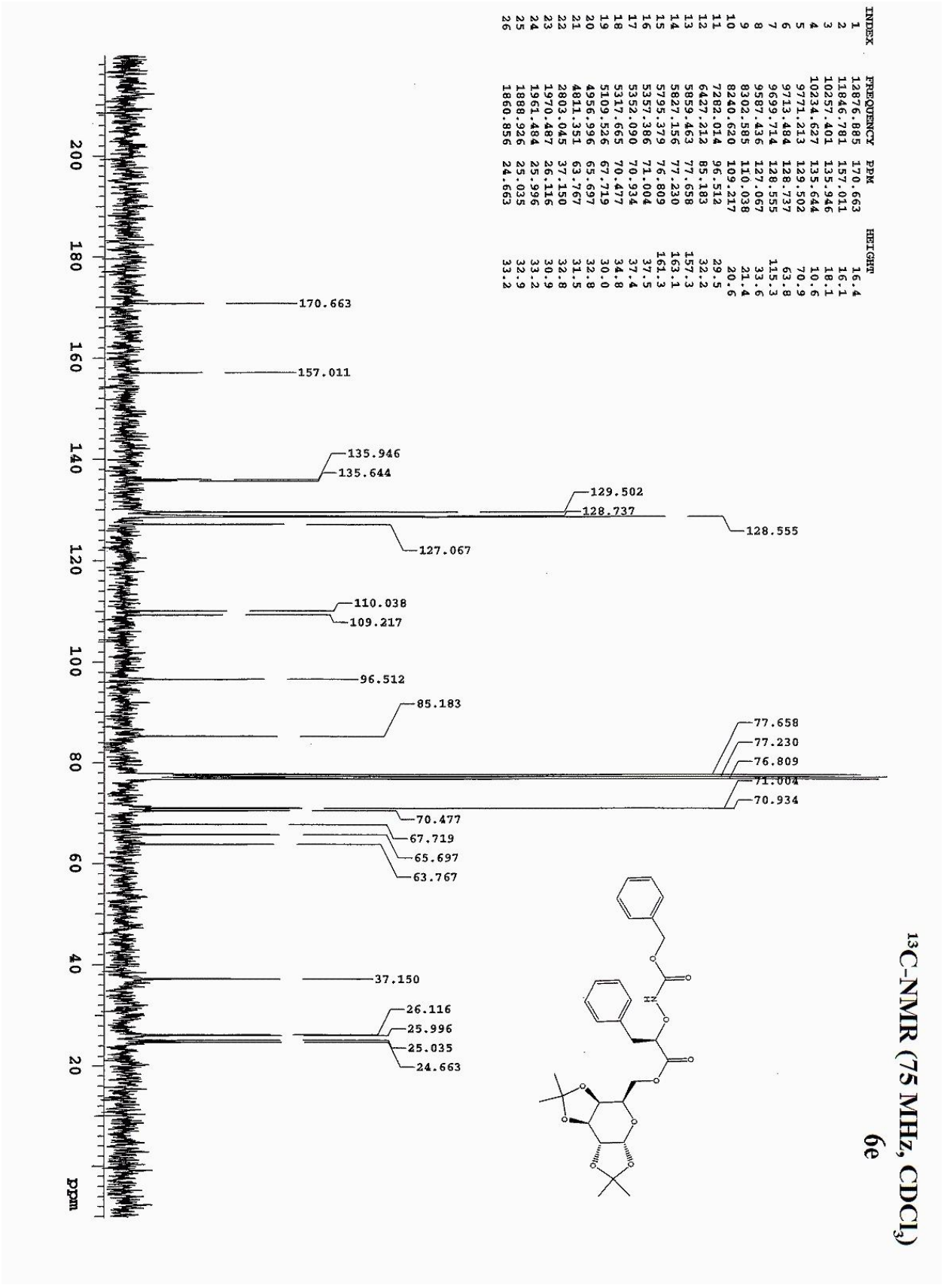


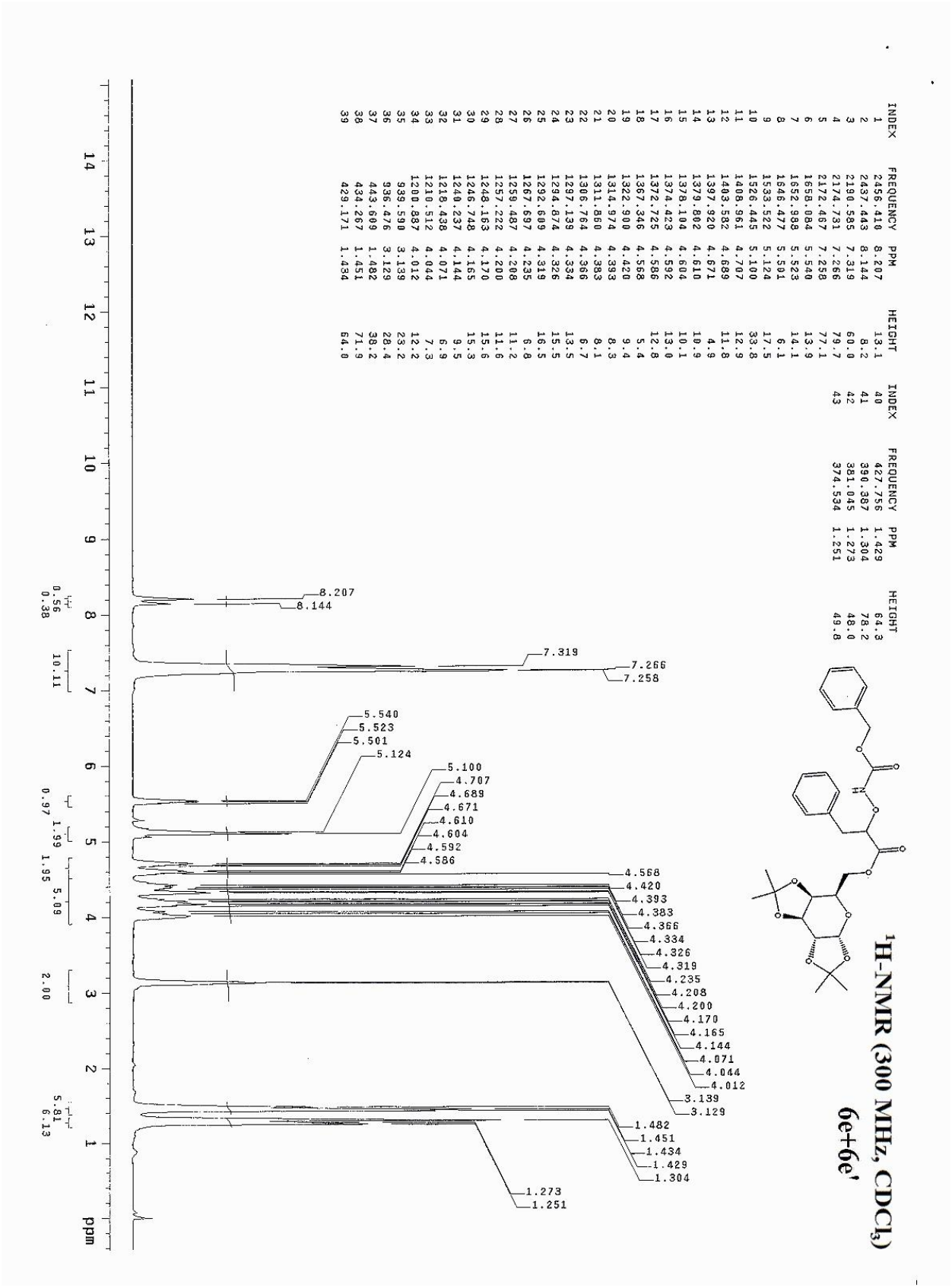
^{13}C-NMR (75 MHz, $CDCl_3$)

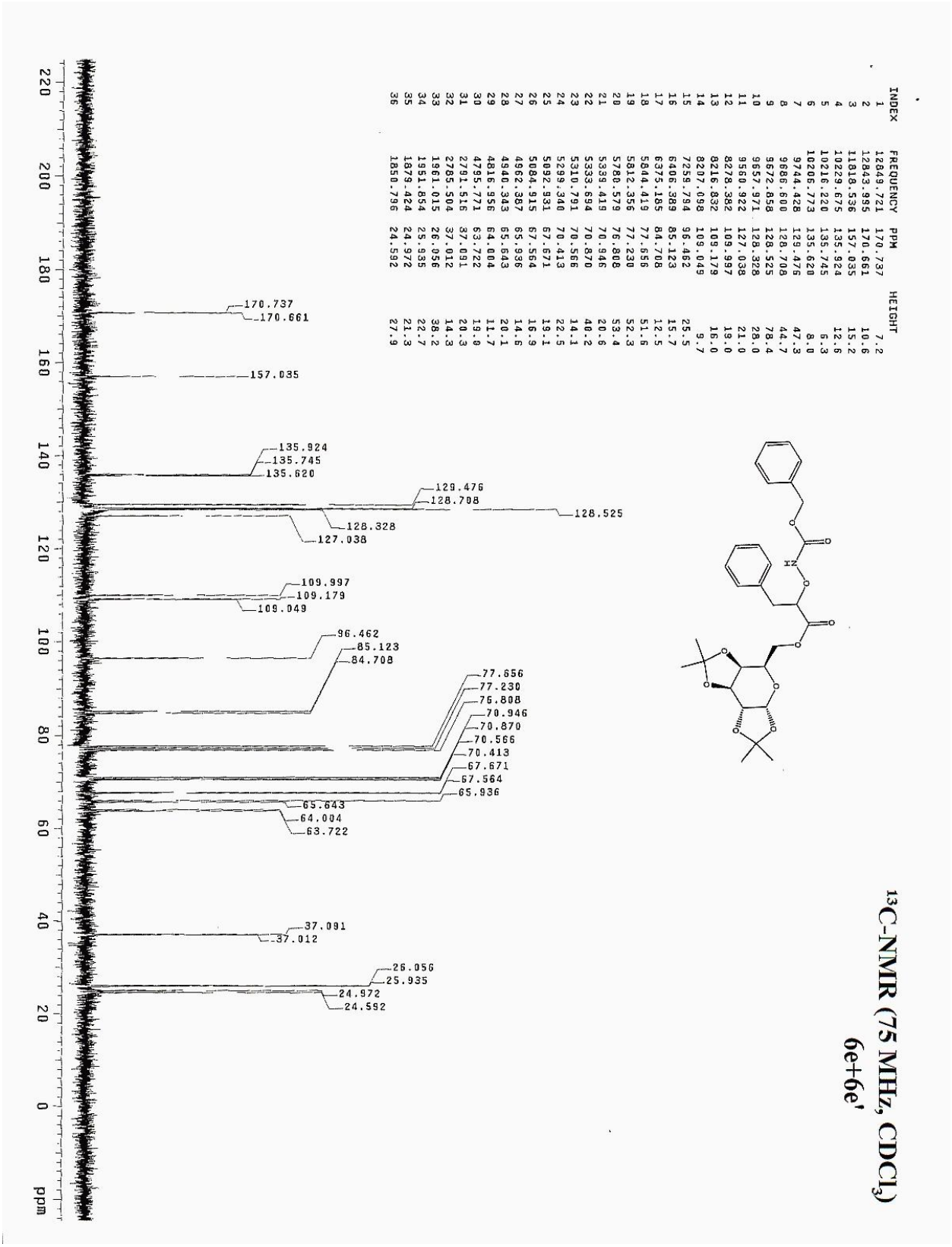
6d

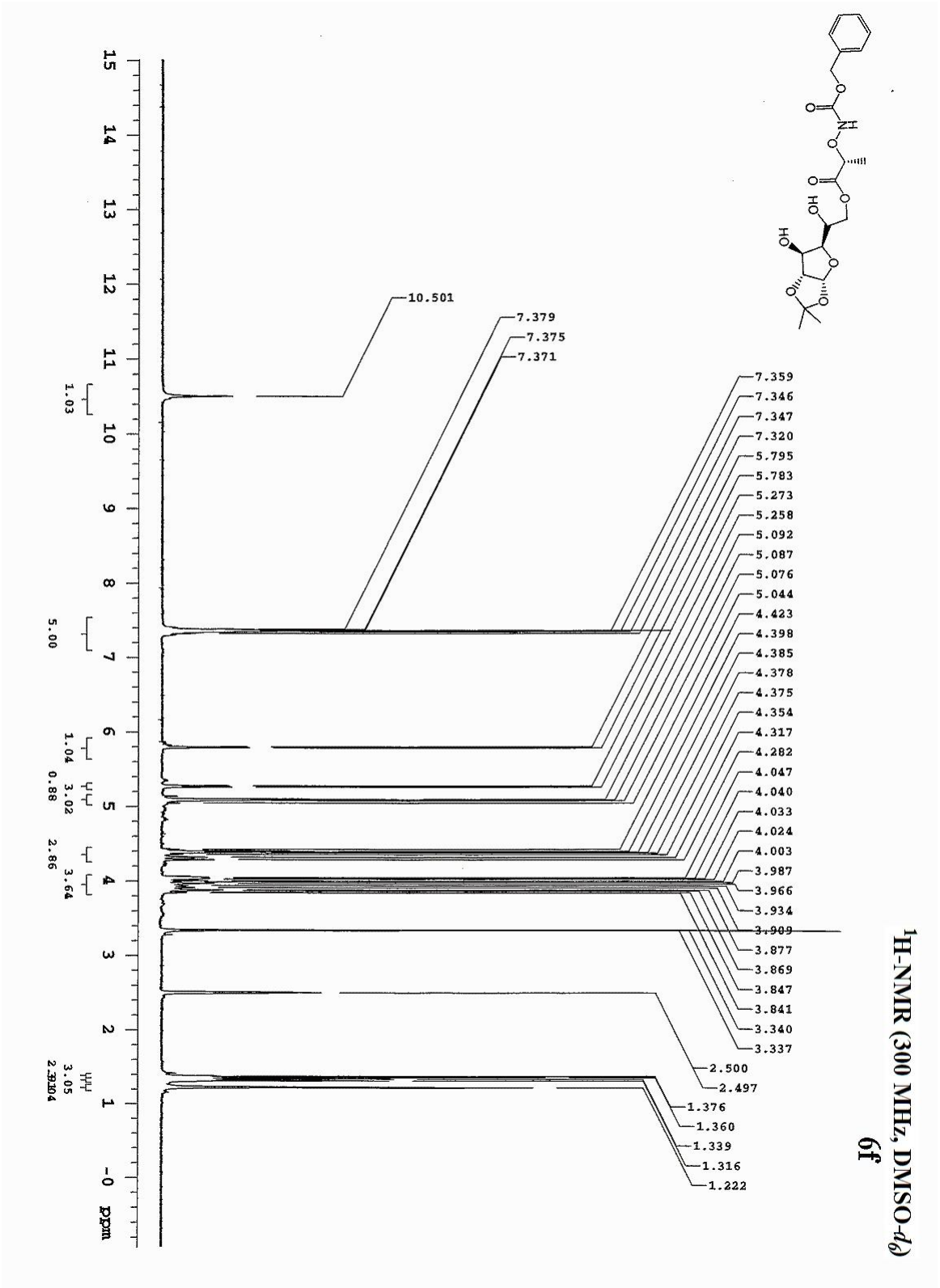


^{1}H-NMR (300 MHz, $CDCl_3$)
6e


INDEX FREQUENCY PPM HEIGHT
1 12876.885 170.663 16.4
2 11846.781 157.011 16.1
3 10257.401 135.946 18.1
4 10234.627 135.644 10.6
5 9771.213 129.502 70.9
6 9713.484 128.737 63.8
7 9699.714 128.555 115.3
8 9587.436 127.067 33.6
9 8302.585 110.038 21.4
10 8240.620 109.217 20.6
11 7282.014 96.512 29.5
12 6427.212 85.183 32.2
13 5859.463 77.658 157.3
14 5827.156 77.230 163.1
15 5795.379 76.809 161.3
16 5357.386 71.004 37.5
17 5352.090 70.934 37.4
18 5317.665 70.477 34.8
19 5109.526 67.719 30.0
20 4956.996 65.697 32.8
21 4811.351 63.767 31.5
22 2803.045 37.150 32.8
23 1970.487 26.116 30.9
24 1961.484 25.996 33.2
25 1888.926 25.035 32.9
26 1860.856 24.663 33.2
200
180
160
140
120
100
80
60
40
20
ppm
170.663
157.011
135.946
135.644
129.502
128.737
128.555
127.067
110.038
109.217
96.512
85.183
77.658
77.230
76.809
71.004
70.934
70.477
67.719
65.697
63.767
37.150
26.116
25.996
25.035
24.663
^{13}C-NMR (75 MHz, $CDCl_3$)
6e

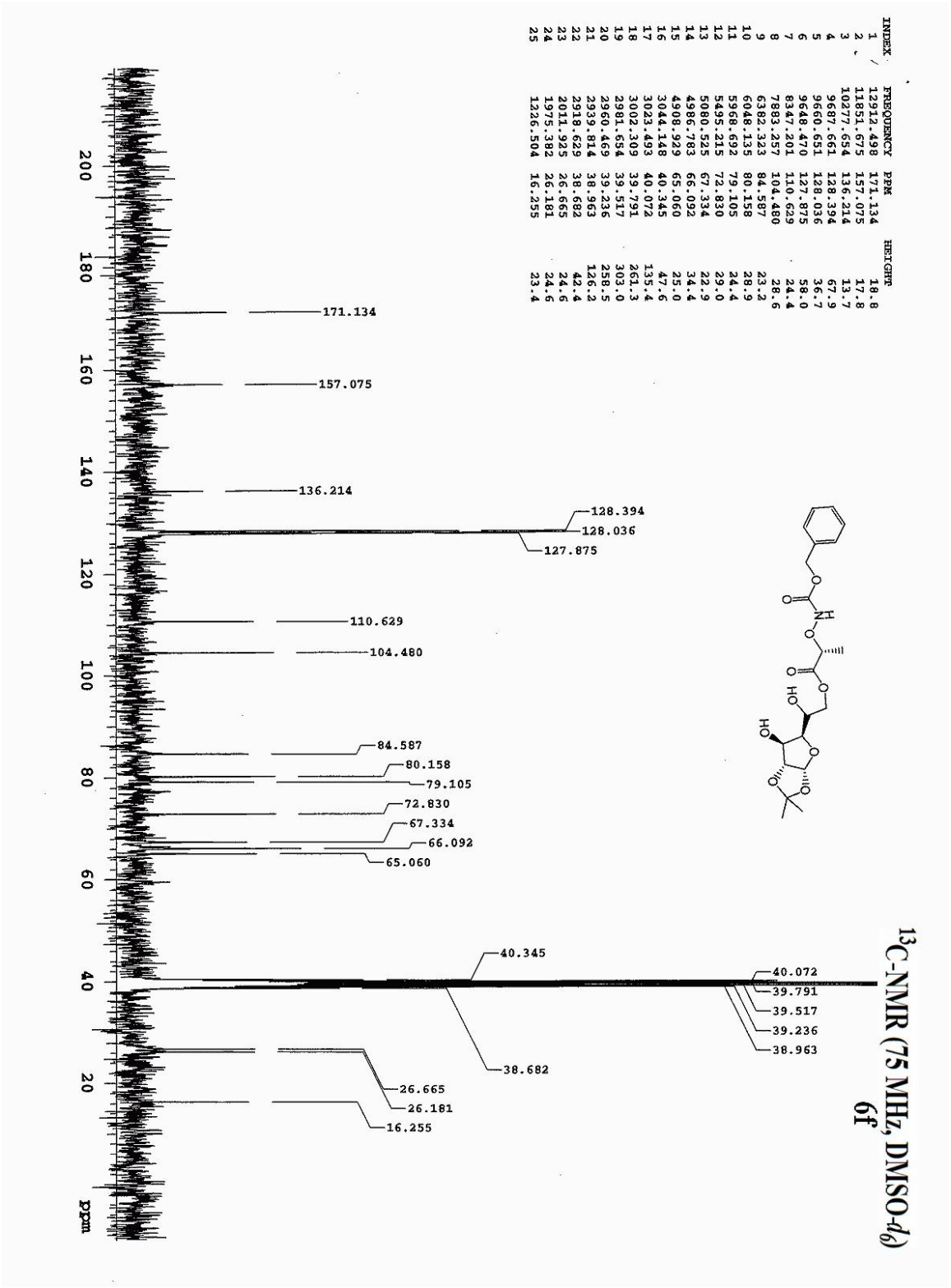

INDEX FREQUENCY PPM HEIGHT
1 2456.410 8.207 13.1
2 2437.443 8.144 8.2
3 2190.585 7.319 60.0
4 2174.731 7.266 79.7
5 2172.467 7.258 77.1
6 1658.084 5.540 13.9
7 1652.988 5.523 14.1
8 1646.477 5.501 6.1
9 1533.522 5.124 17.5
10 1526.445 5.100 33.8
11 1408.961 4.707 12.9
12 1403.582 4.689 11.8
13 1397.920 4.671 4.9
14 1379.802 4.610 10.9
15 1378.104 4.604 10.1
16 1374.423 4.592 13.0
17 1372.725 4.586 12.8
18 1367.346 4.568 5.4
19 1322.900 4.420 9.4
20 1314.974 4.393 8.3
21 1311.860 4.383 8.1
22 1306.764 4.366 6.7
23 1297.139 4.334 13.5
24 1294.874 4.326 15.5
25 1292.609 4.319 16.5
26 1267.697 4.235 6.8
27 1259.487 4.208 11.2
28 1257.222 4.200 11.6
29 1248.163 4.170 15.6
30 1246.748 4.165 15.3
31 1240.237 4.144 9.5
32 1218.438 4.071 6.9
33 1210.512 4.044 7.3
34 1200.837 4.012 12.2
35 939.590 3.139 23.2
36 936.476 3.129 28.4
37 443.609 1.482 38.2
38 434.267 1.451 71.9
39 429.171 1.434 64.0
INDEX FREQUENCY PPM HEIGHT
40 427.756 1.429 64.3
41 380.387 1.304 78.2
42 381.045 1.273 48.0
43 374.534 1.251 49.8
8.207
8.144
7.319
7.266
7.258
5.540
5.523
5.501
5.124
5.100
4.707
4.689
4.671
4.610
4.604
4.592
4.586
4.568
4.420
4.393
4.383
4.366
4.334
4.326
4.319
4.235
4.208
4.200
4.170
4.165
4.144
4.071
4.044
4.012
3.139
3.129
1.482
1.451
1.434
1.429
1.304
1.273
1.251
0.56
0.38
10.11
0.97
1.99
1.95
5.09
2.00
5.81
6.13
ppm
^{1}H-NMR (300 MHz, $CDCl_3$)
6e+6e'





^{1}H-NMR (300 MHz, DMSO-d_6)

6f



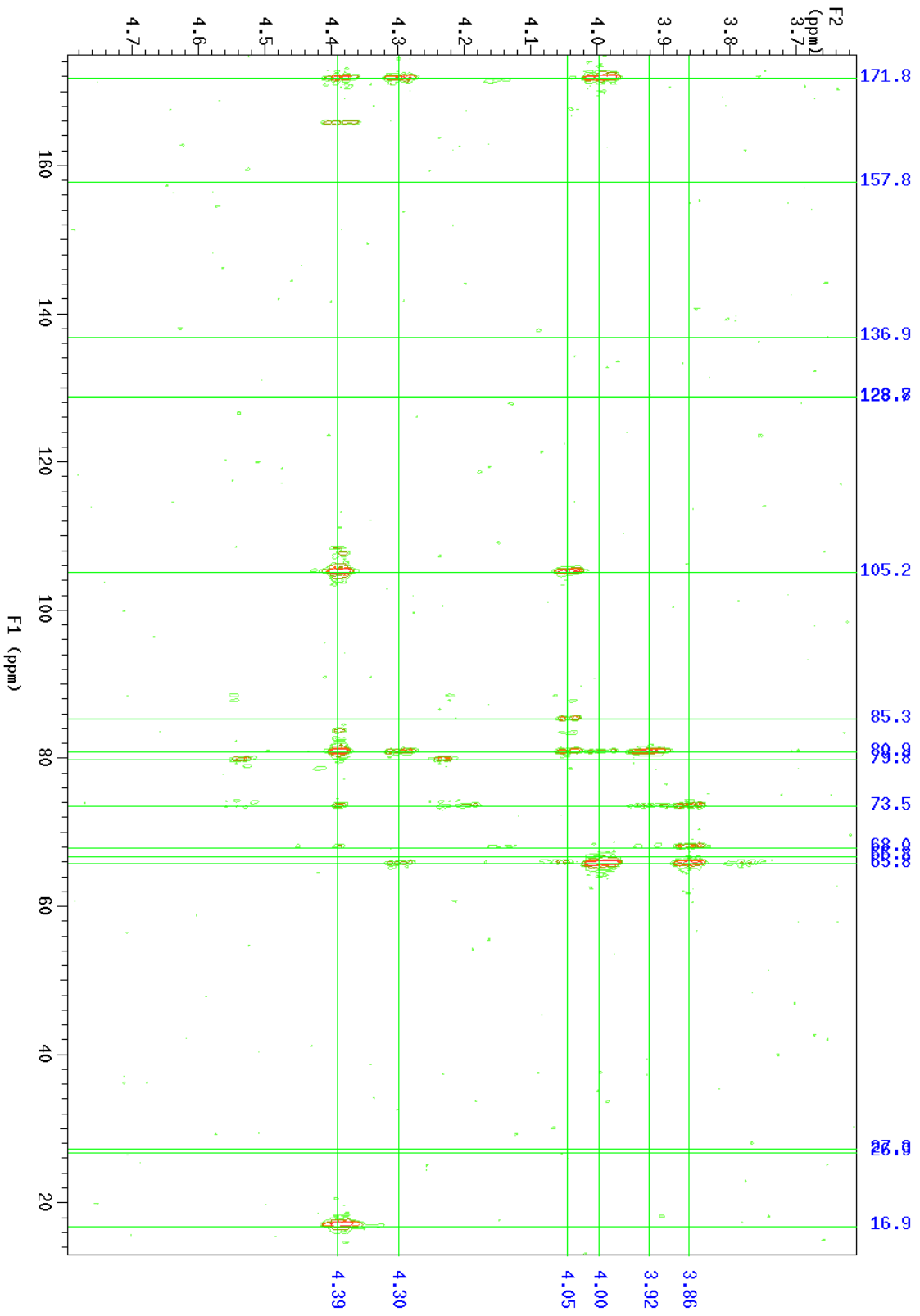


Figure S1. ^{1}H-^{13}C gHMBC experiment for **6f**

Proton conectivity for sugar part of compound **6f** – determined by ^{1}H-^{1}H gDQCOSY experiment, expansion is provided in **Figure S2-S3.**

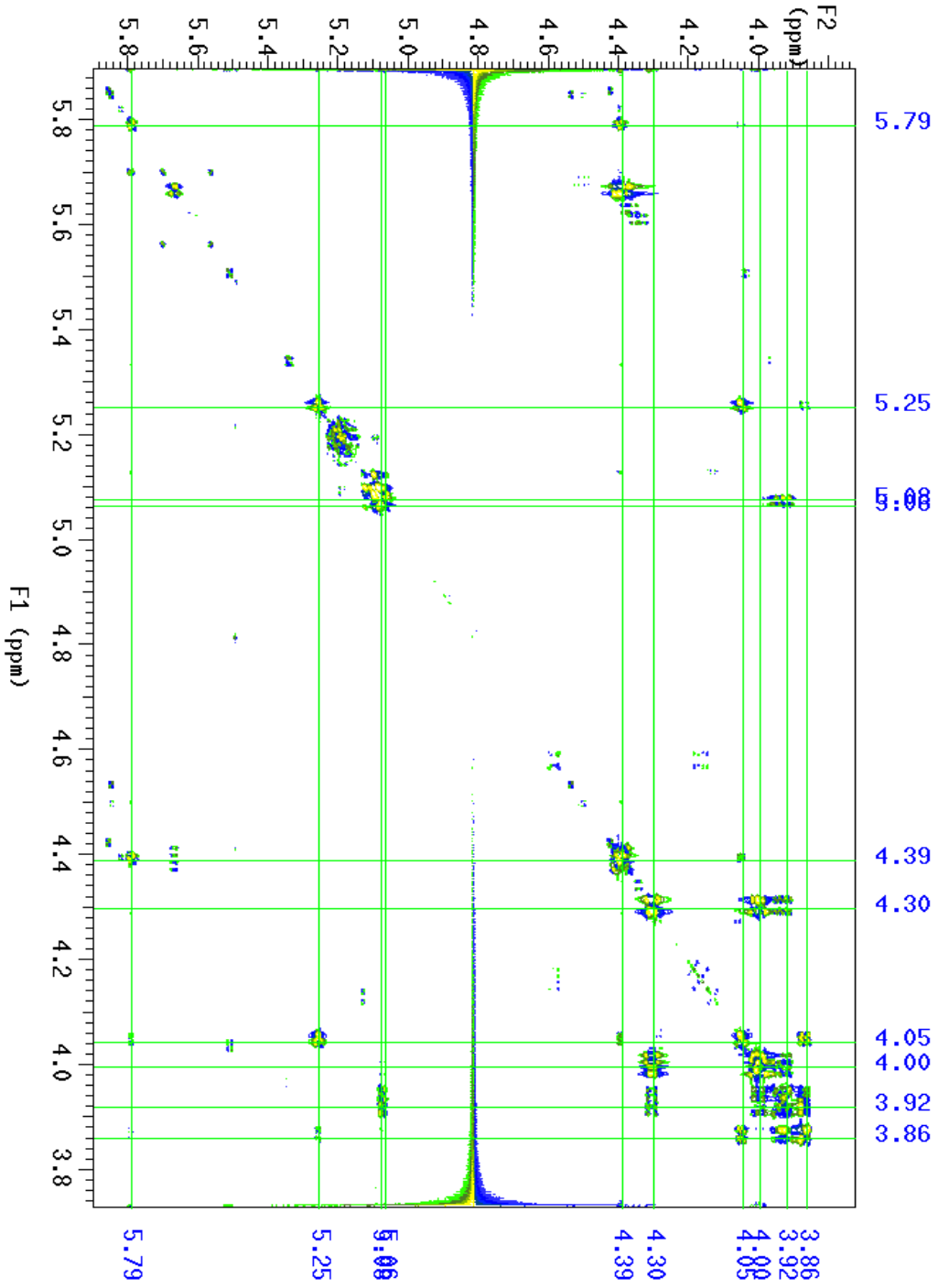


Figure S2. ^{1}H-^{1}H DQCOSY of **6f**.

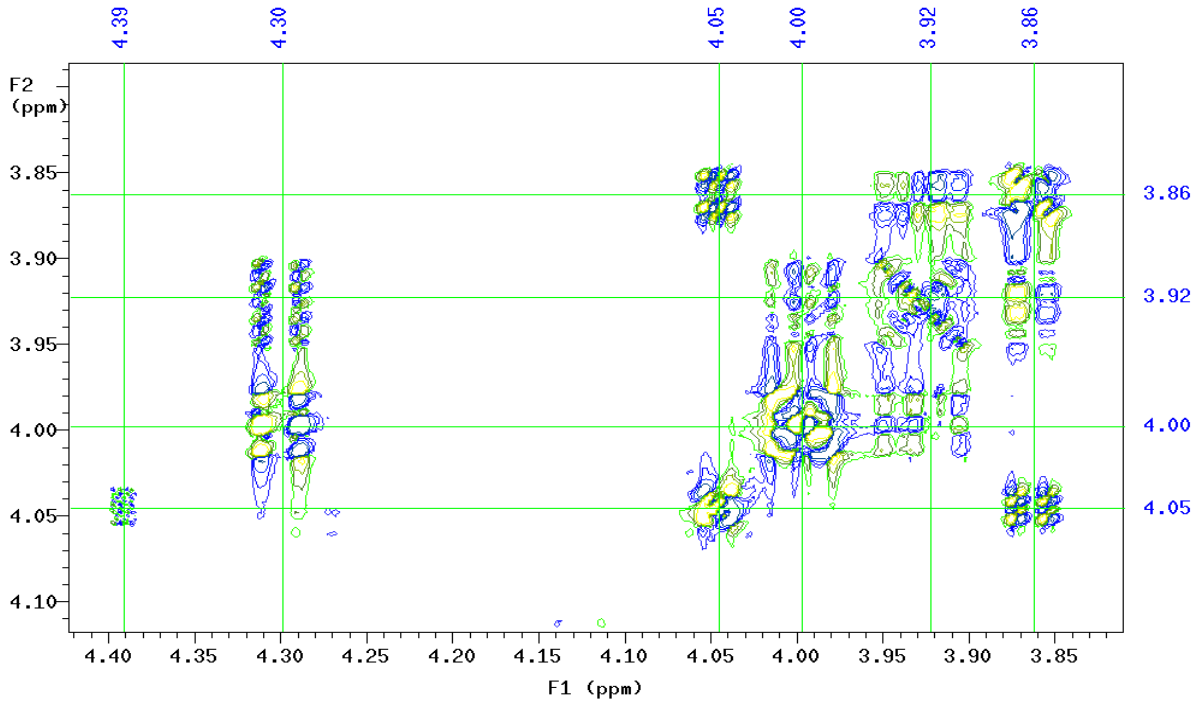
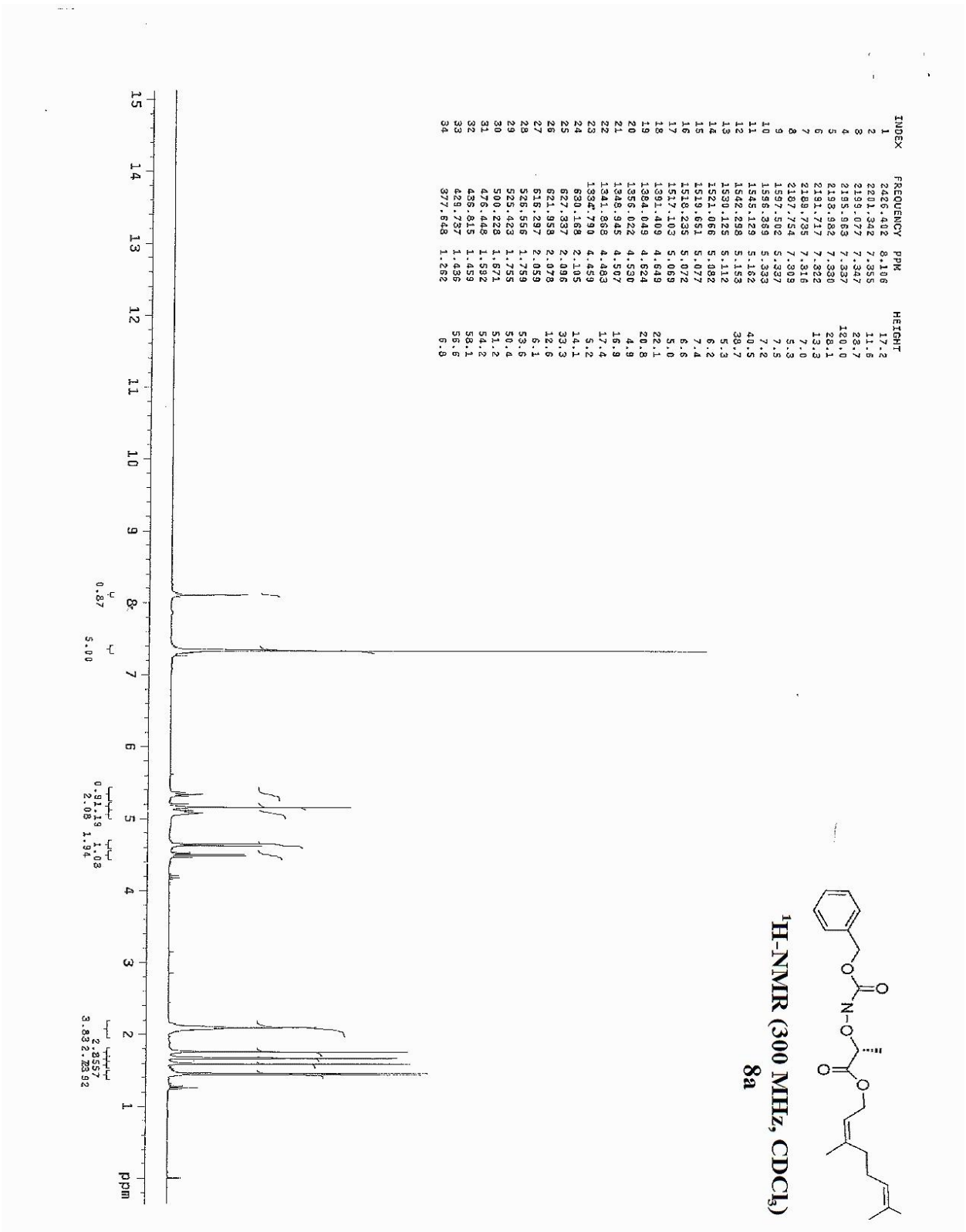
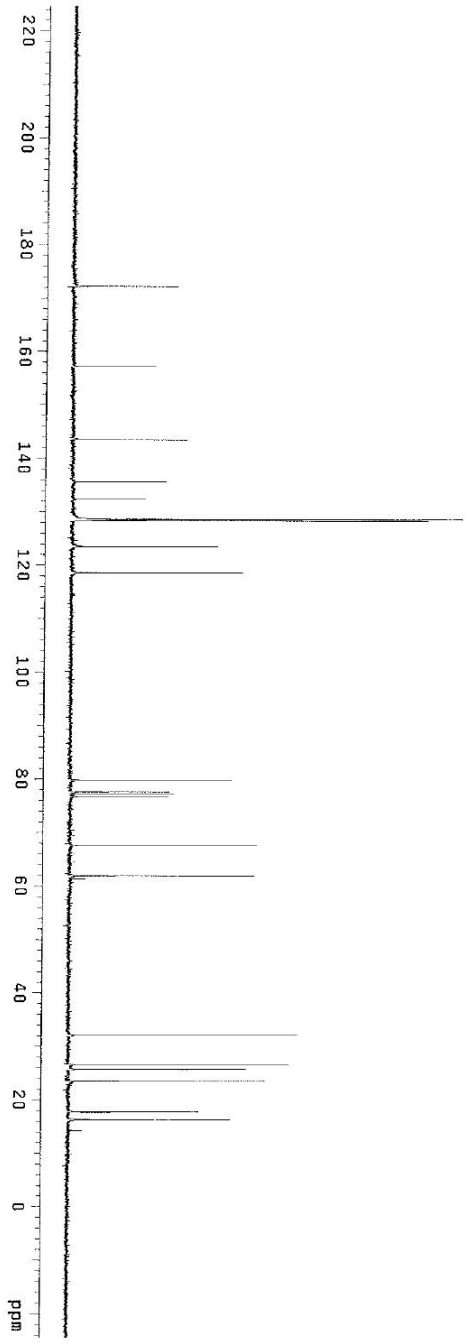
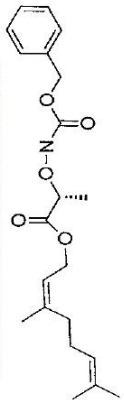


Figure S3. ^{1}H-^{1}H gDQCOSY expansion for sugar part



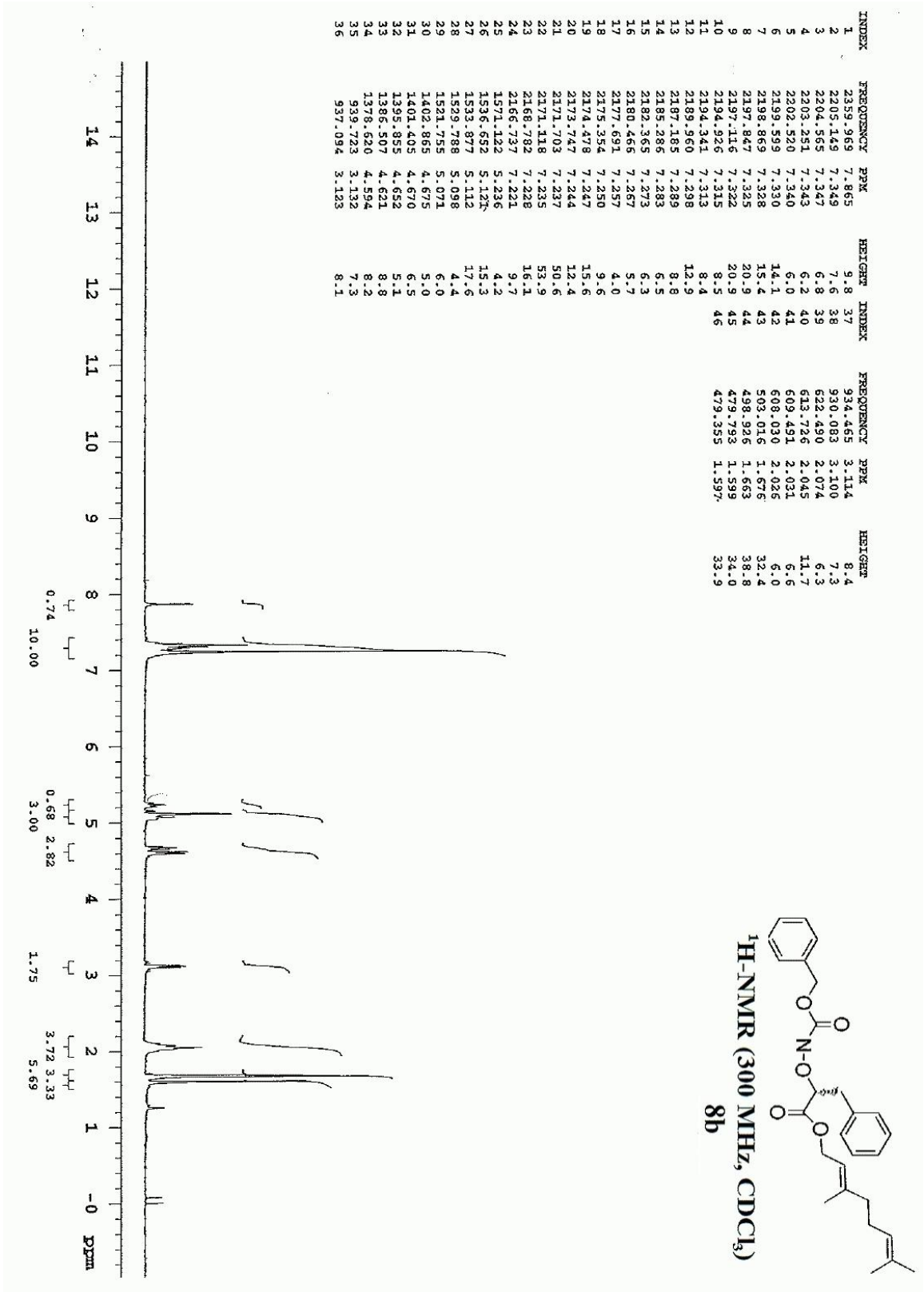


INDEX	FREQUENCY	PPM	HEIGHT
1	12956.642	172.150	19.7
2	11824.114	157.103	15.6
3	10792.356	143.394	21.8
4	10204.048	135.577	17.7
5	9956.701	132.291	13.9
6	9678.150	128.590	73.2
7	9665.839	128.426	43.2
8	9653.243	128.259	66.6
9	9291.956	123.459	27.6
10	8922.653	118.552	32.2
11	6006.307	79.804	30.3
12	5839.118	77.582	18.8
13	5807.341	77.160	19.6
14	5775.278	76.734	18.5
15	5083.050	67.537	35.3
16	4656.777	61.873	34.8
17	2421.779	32.177	43.0
18	2003.809	26.624	41.5
19	1934.523	25.703	33.5
20	1770.490	23.524	37.1
21	1330.762	17.681	24.8
22	1223.120	16.251	30.7

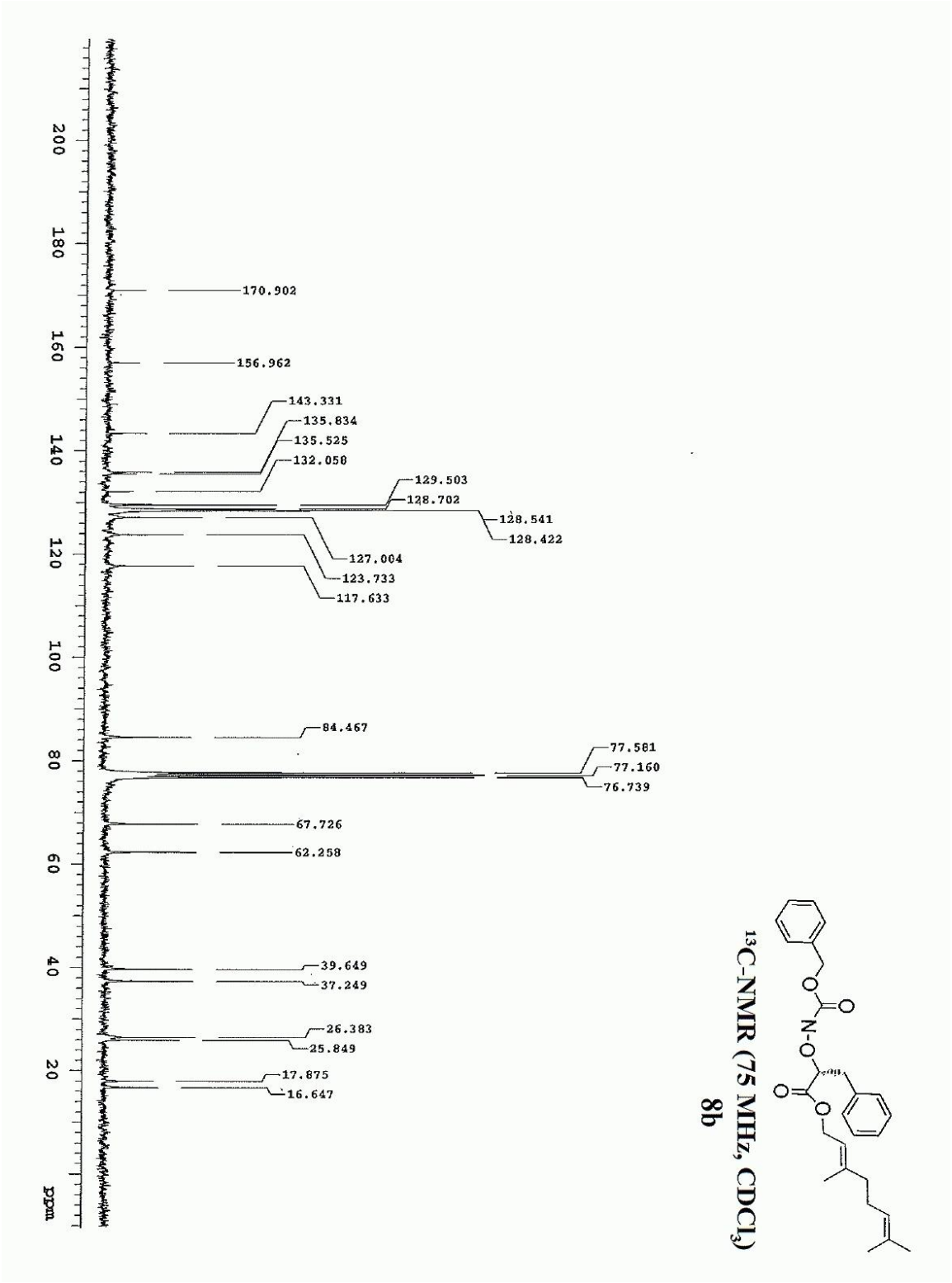


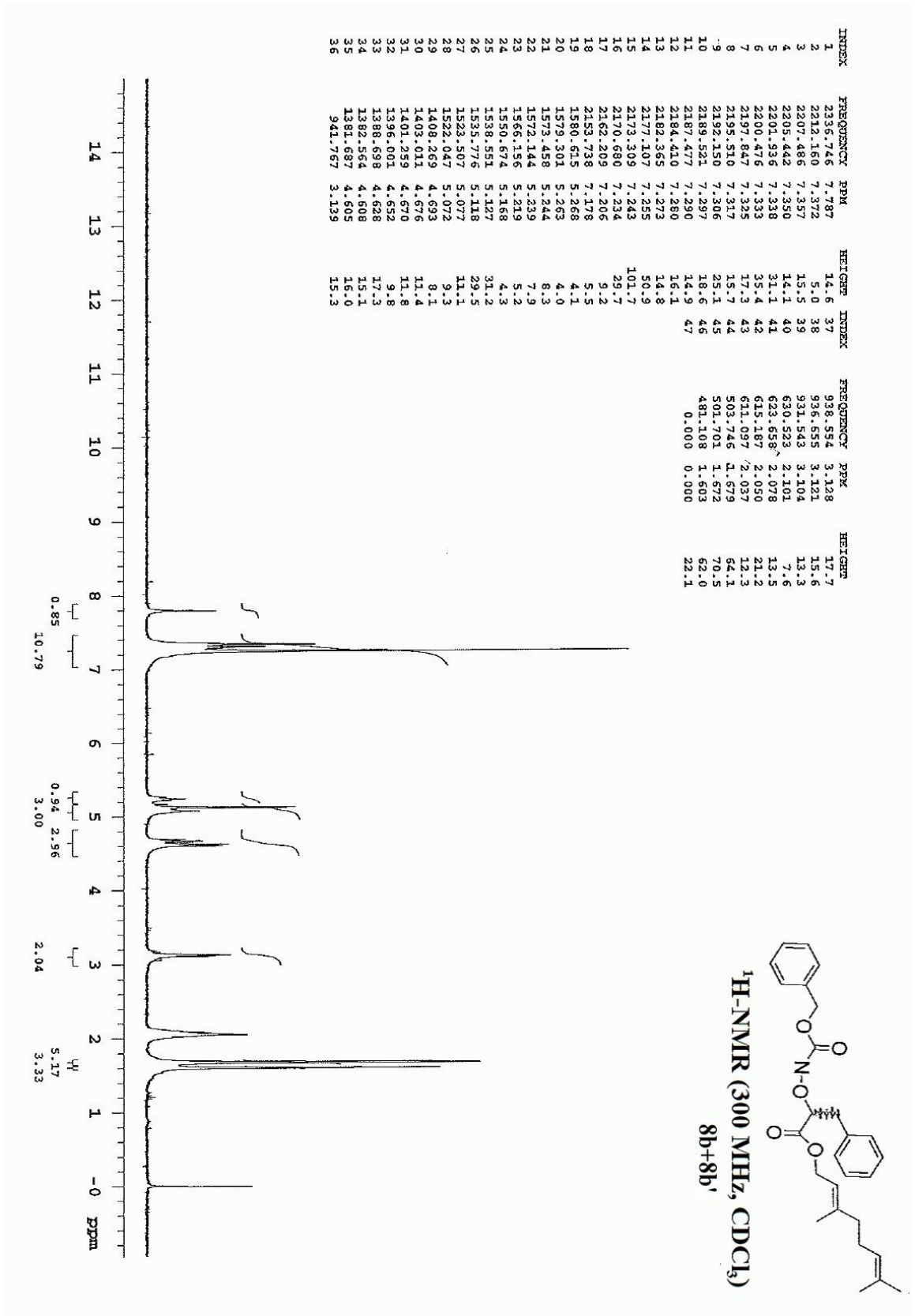
^{13}C-NMR (75 MHz, $CDCl_3$)

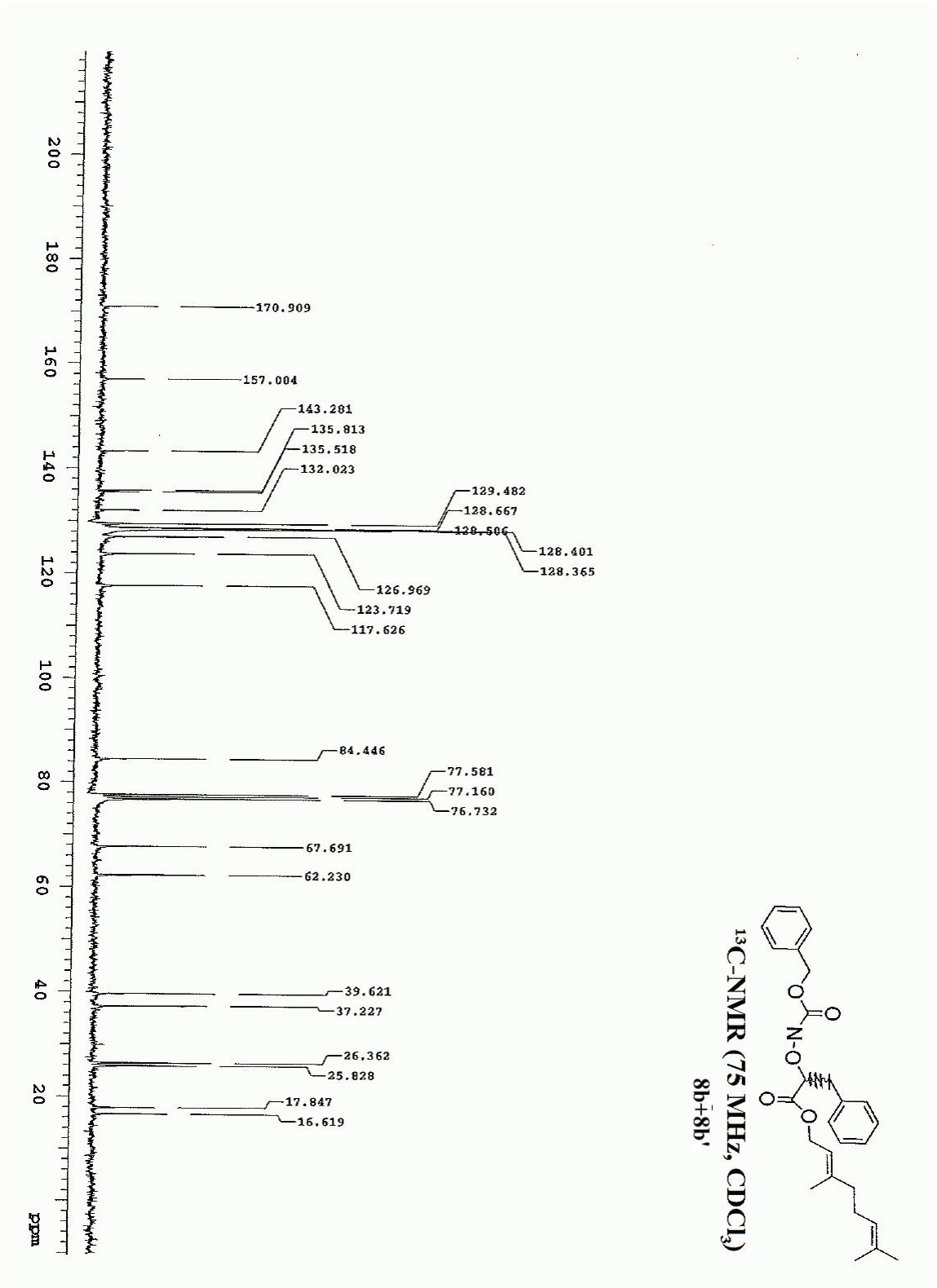
8a



INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	2359.969	7.865	9.8	37	934.465	3.114	8.4
2	2205.149	7.349	7.6	38	930.083	3.100	7.3
3	2204.565	7.347	6.8	39	622.490	2.074	6.3
4	2203.251	7.343	6.2	40	613.726	2.045	11.7
5	2202.520	7.340	6.0	41	609.491	2.031	6.6
6	2199.599	7.330	14.1	42	608.030	2.026	6.0
7	2198.869	7.328	15.4	43	503.016	1.676	32.4
8	2197.847	7.325	20.9	44	498.926	1.663	38.8
9	2197.116	7.322	20.9	45	479.793	1.599	34.0
10	2194.926	7.315	8.5	46	479.355	1.597	33.9
11	2194.341	7.313	8.4				
12	2189.960	7.298	12.9				
13	2187.185	7.289	8.8				
14	2185.286	7.283	6.5				
15	2182.365	7.273	6.3				
16	2180.466	7.267	5.7				
17	2177.691	7.257	4.0				
18	2175.354	7.250	9.6				
19	2174.478	7.247	15.6				
20	2173.747	7.244	12.4				
21	2171.703	7.237	50.6				
22	2171.118	7.235	53.9				
23	2168.782	7.228	16.1				
24	2166.737	7.221	9.7				
25	1571.122	5.236	4.2				
26	1536.652	5.121	15.3				
27	1533.877	5.112	17.6				
28	1529.788	5.098	4.4				
29	1521.755	5.071	6.0				
30	1402.865	4.675	5.0				
31	1401.405	4.670	6.5				
32	1395.855	4.652	5.1				
33	1386.507	4.621	8.8				
34	1378.620	4.594	8.2				
35	939.723	3.132	7.3				
36	937.094	3.123	8.1				

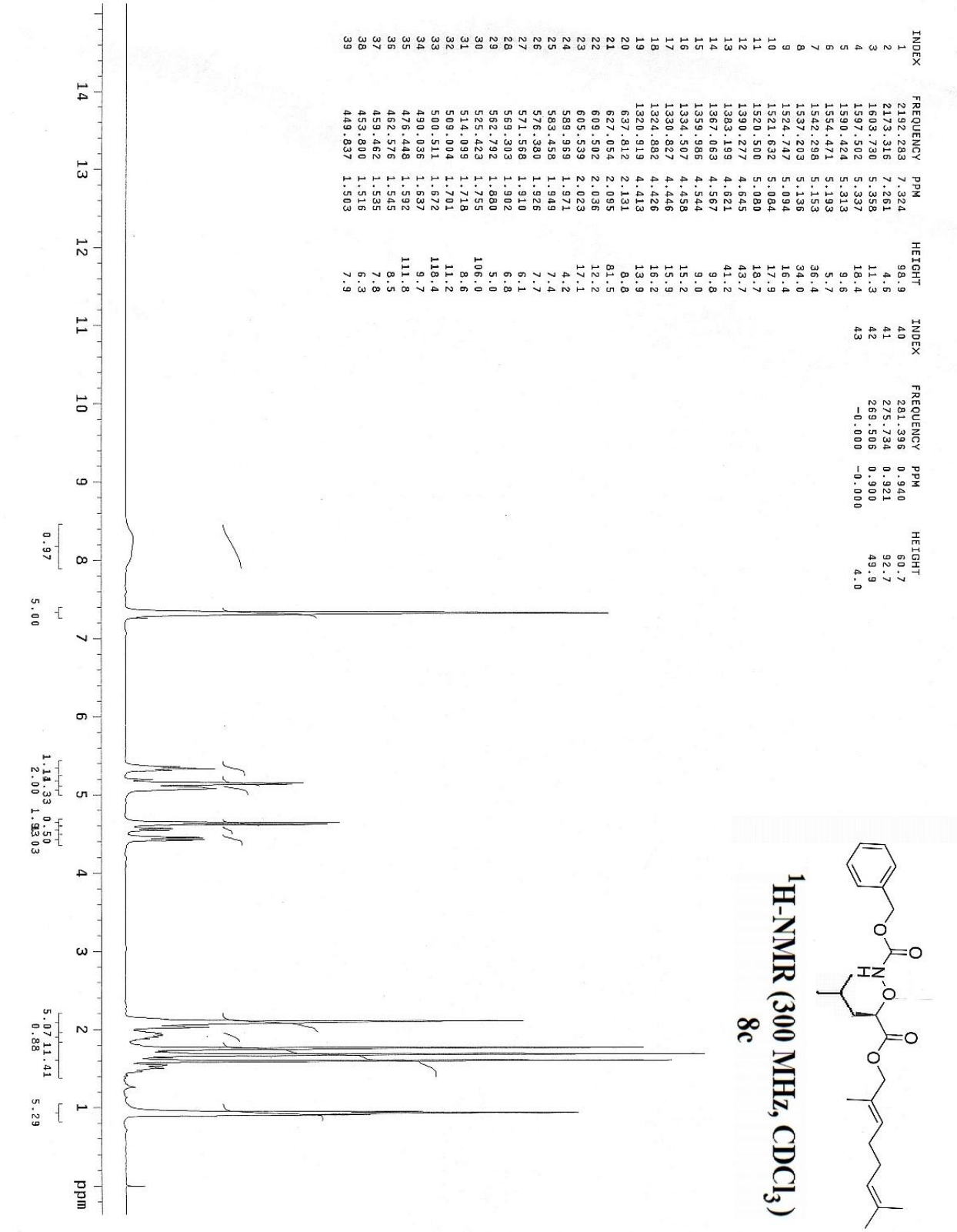

170.902
156.962
143.331
135.834
135.525
132.058
129.503
128.702
128.541
128.422
127.004
123.733
117.633
84.467
77.581
77.160
76.739
67.726
62.258
39.649
37.249
26.383
25.849
17.875
16.647
200
180
160
140
120
100
80
60
40
20
ppm
^{13}C-NMR (75 MHz, CDCl$_3$)
8b


INDEX FREQUENCY PPM HEIGHT INDEX FREQUENCY PPM HEIGHT
1 2336.746 7.787 14.6 37 938.554 3.128 17.7
2 2212.160 7.372 5.0 38 936.555 3.121 15.6
3 2207.486 7.357 15.5 39 931.543 3.104 13.3
4 2205.442 7.350 14.1 40 630.523 2.101 7.6
5 2201.936 7.338 31.1 41 623.658 2.078 13.5
6 2200.476 7.333 35.4 42 615.187 2.050 21.2
7 2197.847 7.325 17.3 43 611.097 2.037 12.3
8 2195.510 7.317 15.7 44 503.746 1.679 64.1
9 2192.150 7.306 25.1 45 501.701 1.672 70.5
10 2189.521 7.297 18.6 46 481.108 1.603 62.0
11 2187.477 7.290 14.9 47 0.000 0.000 22.1
12 2184.410 7.280 16.1
13 2182.365 7.273 14.8
14 2177.107 7.255 50.9
15 2173.309 7.243 101.7
16 2170.680 7.234 29.7
17 2162.209 7.206 9.2
18 2153.738 7.178 5.5
19 2580.615 5.268 4.1
20 1579.301 5.263 4.0
21 1573.458 5.244 8.3
22 1572.144 5.239 7.9
23 1566.156 5.219 5.2
24 1550.674 5.168 4.3
25 1538.551 5.127 31.2
26 1535.776 5.118 29.5
27 1523.507 5.077 11.1
28 1522.047 5.072 9.3
29 1408.269 4.693 8.1
30 1403.011 4.676 11.4
31 1401.259 4.670 11.8
32 1396.001 4.652 9.8
33 1388.698 4.628 17.3
34 1382.564 4.608 15.1
35 1381.687 4.605 16.0
36 941.767 3.139 15.3
14 13 12 11 10 9 8 7 6 5 4 3 2 1 -0 ppm
0.85 0.94 2.96 5.17
10.79 3.00 2.04 3.33
O
N-O
O
O
O
^{1}H-NMR (300 MHz, CDCl$_3$)
8b+8b'



^{13}C-NMR (75 MHz, $CDCl_3$)

8b+8b'

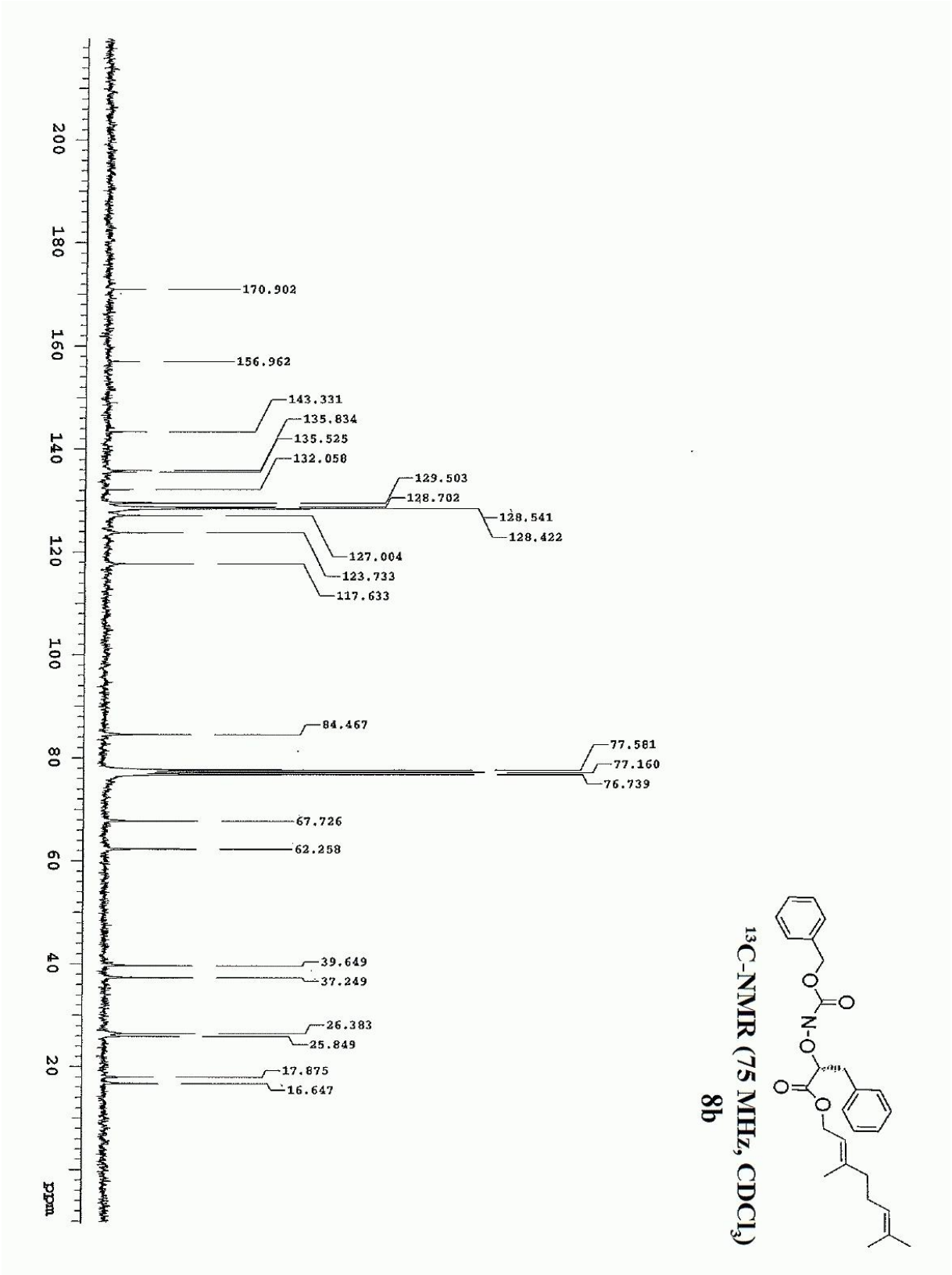


INDEX	FREQUENCY	PPM	HEIGHT
1	2192.283	7.324	98.9
2	2173.316	7.261	4.6
3	1603.730	5.358	11.3
4	1597.502	5.337	18.4
5	1590.424	5.313	9.6
6	1554.471	5.193	5.7
7	1542.298	5.153	36.4
8	1537.203	5.136	34.0
9	1524.747	5.094	16.4
10	1521.632	5.084	17.9
11	1520.500	5.080	18.7
12	1390.277	4.645	43.7
13	1383.199	4.621	41.2
14	1367.063	4.567	9.8
15	1359.986	4.544	9.0
16	1334.507	4.458	15.2
17	1330.827	4.446	15.9
18	1324.882	4.426	16.2
19	1320.919	4.413	13.9
20	1637.812	2.131	8.8
21	627.054	2.095	81.5
22	609.502	2.036	12.2
23	605.539	2.023	17.1
24	589.969	1.971	4.2
25	583.458	1.949	7.4
26	576.380	1.926	7.7
27	571.568	1.910	6.1
28	569.303	1.902	6.8
29	562.792	1.880	5.0
30	525.423	1.755	106.0
31	514.099	1.718	8.6
32	509.004	1.701	11.2
33	500.511	1.672	118.4
34	490.036	1.637	9.7
35	476.448	1.592	111.8
36	462.576	1.545	8.5
37	459.462	1.535	7.8
38	453.800	1.516	6.3
39	449.837	1.503	7.9

INDEX	FREQUENCY	PPM	HEIGHT
40	281.396	0.940	60.7
41	275.734	0.921	92.7
42	269.506	0.900	49.9
43	-0.000	-0.000	4.0

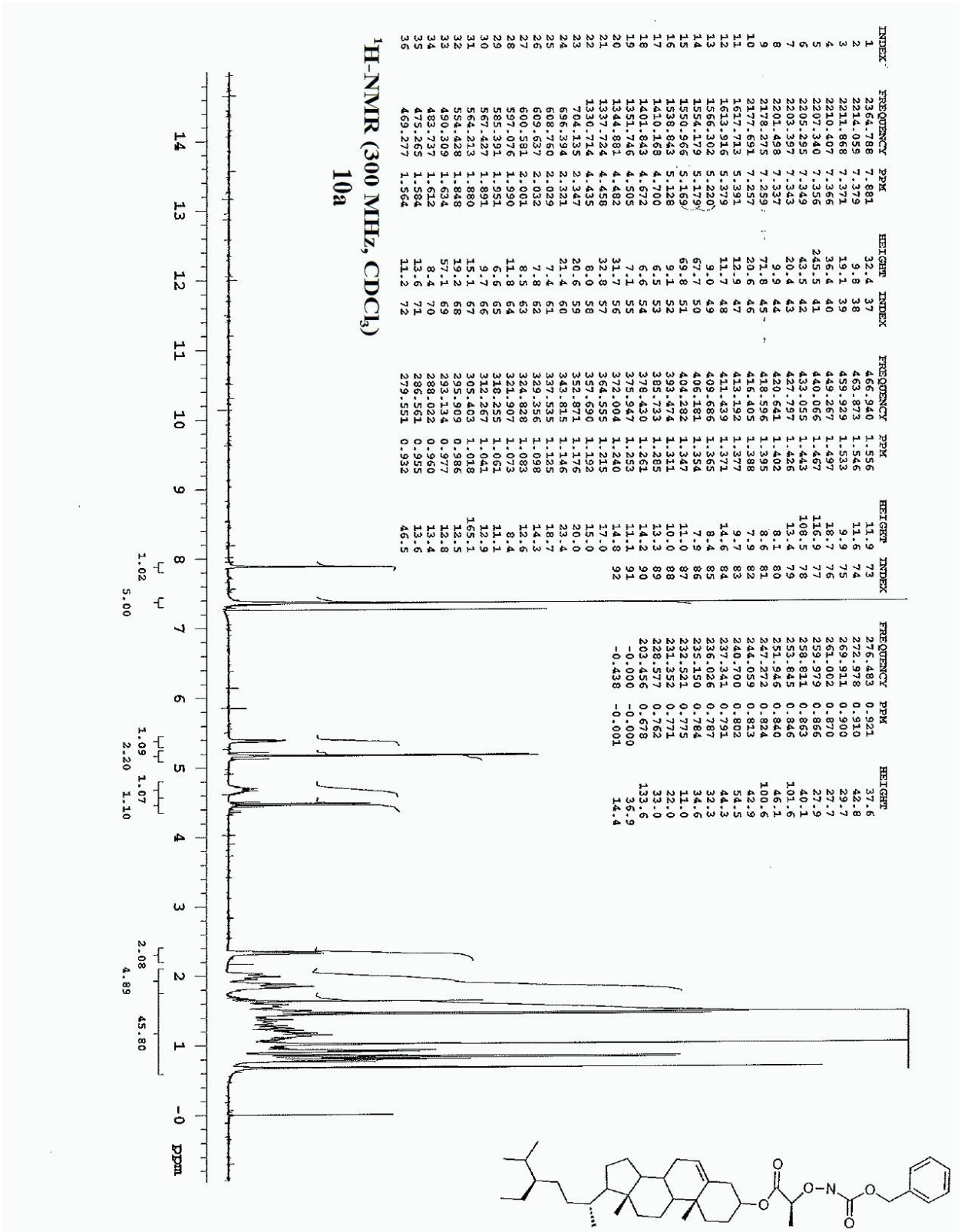
^{1}H-NMR (300 MHz, $CDCl_3$)

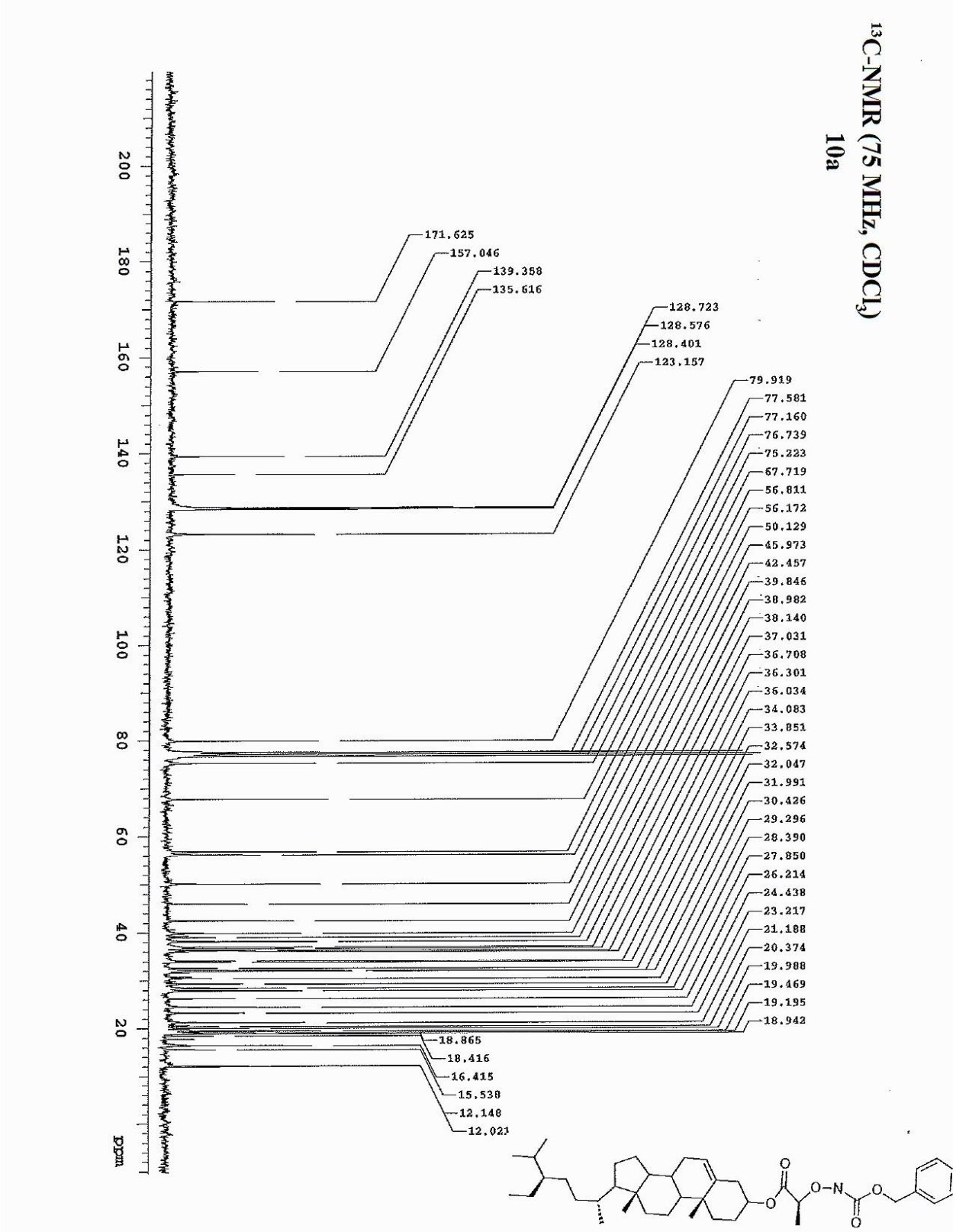
8c

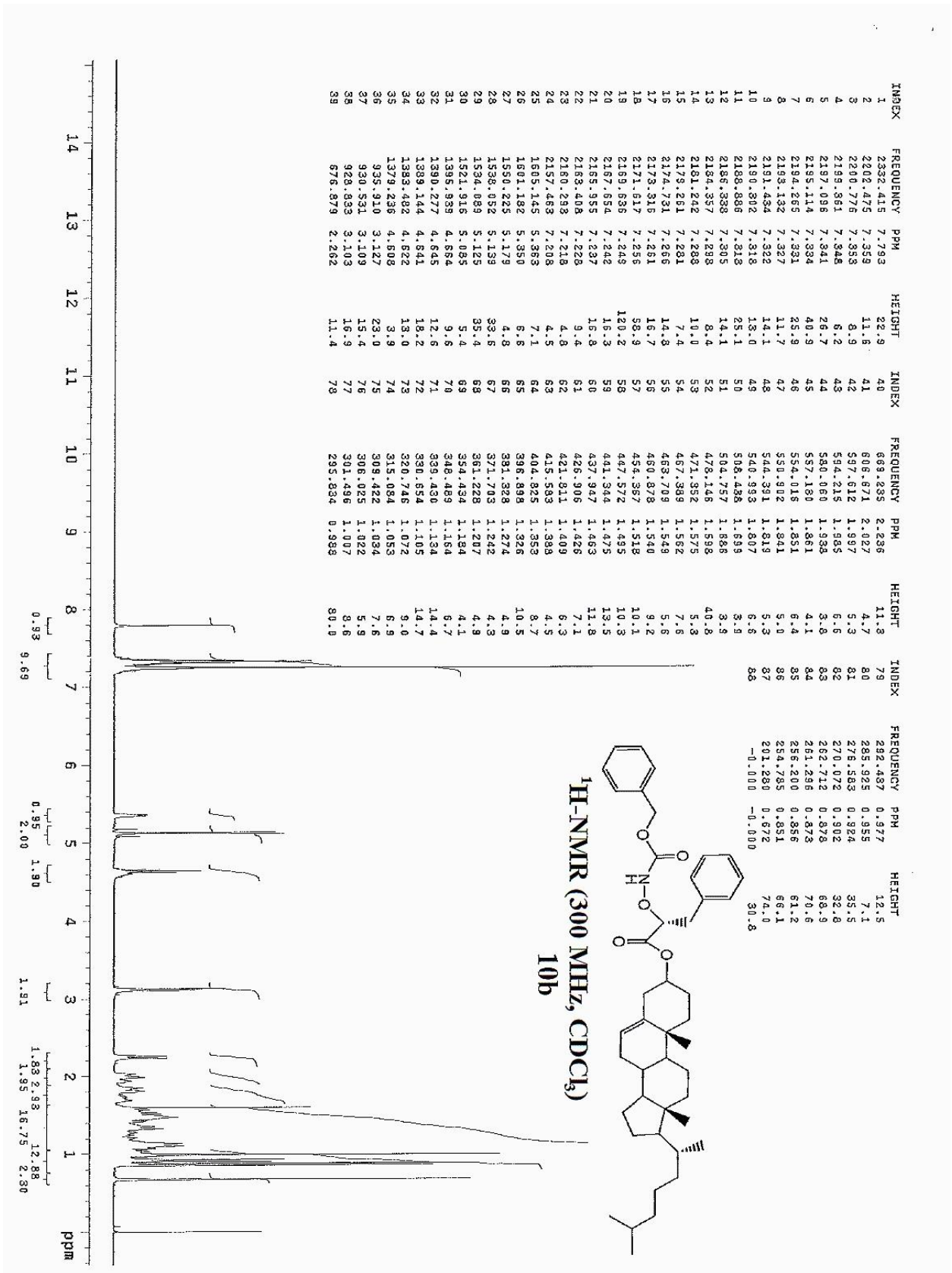


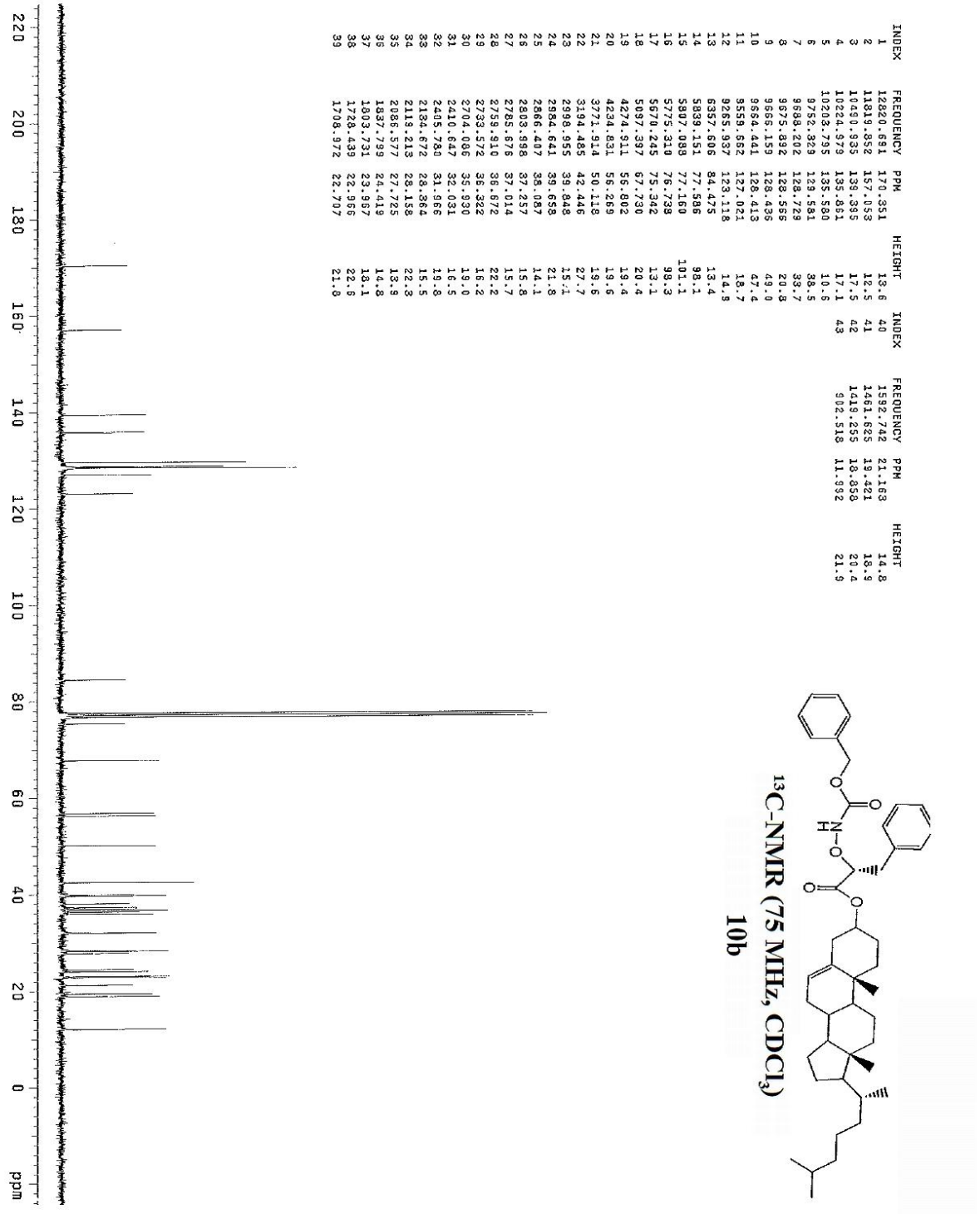
^{13}C-NMR (75 MHz, $CDCl_3$)

8b


INDEX FREQUENCY PPM HEIGHT INDEX FREQUENCY PPM HEIGHT INDEX FREQUENCY PPM HEIGHT
1 2364.788 7.881 32.4 37 466.940 1.556 11.9 73 276.483 0.921 37.6
2 2214.059 7.379 9.8 38 463.873 1.546 11.6 74 272.978 0.910 42.8
3 2211.868 7.371 19.1 39 459.929 1.533 9.9 75 269.911 0.900 29.7
4 2210.407 7.366 36.4 40 449.267 1.497 18.7 76 261.002 0.870 27.7
5 2207.340 7.356 245.5 41 440.066 1.467 116.9 77 259.979 0.866 27.9
6 2205.295 7.349 43.5 42 433.055 1.443 108.5 78 258.811 0.863 40.1
7 2203.397 7.343 20.4 43 427.797 1.426 13.4 79 253.845 0.846 101.6
8 2201.498 7.337 9.9 44 420.641 1.402 8.1 80 251.946 0.840 46.1
9 2178.275 7.259 71.8 45 418.596 1.395 8.6 81 247.272 0.824 100.6
10 2177.691 7.257 20.6 46 416.405 1.388 7.9 82 244.059 0.813 42.9
11 1617.713 5.391 12.9 47 413.192 1.377 9.7 83 240.700 0.802 54.5
12 1613.916 5.379 11.7 48 411.439 1.371 14.6 84 237.341 0.791 44.3
13 1556.302 5.220 9.0 49 409.686 1.365 8.4 85 236.026 0.787 32.3
14 1554.179 5.179 67.7 50 406.181 1.354 7.9 86 235.150 0.784 34.6
15 1550.966 5.169 69.8 51 404.282 1.347 11.0 87 232.521 0.775 11.0
16 1538.843 5.128 9.1 52 393.474 1.311 10.0 88 231.352 0.771 22.0
17 1410.168 4.700 6.5 53 385.733 1.285 13.3 89 228.577 0.762 33.0
18 1401.843 4.672 6.6 54 378.430 1.261 14.2 90 203.456 0.678 133.6
19 1351.746 4.505 7.1 55 375.947 1.253 11.1 91 -0.000 -0.000 36.9
20 1344.881 4.482 31.7 56 372.004 1.240 14.8 92 -0.438 -0.001 14.4
21 1337.724 4.458 32.8 57 364.555 1.215 17.0
22 1330.714 4.435 8.0 58 357.690 1.192 15.0
23 704.135 2.347 20.6 59 352.871 1.176 20.0
24 696.394 2.321 21.4 60 343.815 1.146 23.4
25 608.760 2.029 7.4 61 337.535 1.125 18.7
26 603.637 2.032 7.8 62 329.356 1.098 14.3
27 600.581 2.001 8.5 63 324.828 1.083 12.6
28 597.076 1.990 11.8 64 321.907 1.073 8.4
29 585.391 1.951 6.6 65 318.255 1.061 11.1
30 567.427 1.891 9.7 66 312.267 1.041 12.9
31 564.213 1.880 15.1 67 305.403 1.018 165.1
32 554.428 1.848 19.2 68 295.909 0.986 12.5
33 490.309 1.634 57.1 69 293.134 0.977 12.8
34 483.737 1.612 8.4 70 288.022 0.960 13.4
35 475.265 1.584 13.6 71 285.561 0.955 13.6
36 469.277 1.564 11.2 72 279.551 0.932 46.5
1H-NMR (300 MHz, CDCl3)
10a
14 13 12 11 10 9 8 7 6 5 4 3 2 1 -0 ppm
1.02 5.00 1.09 1.07 2.20 1.10 2.08 45.80 4.89
O-N
O
O
O
O




INDEX FREQUENCY PPM HEIGHT INDEX FREQUENCY PPM HEIGHT INDEX FREQUENCY PPM HEIGHT
1 2332.415 7.793 22.9 40 669.235 2.236 11.8 79 282.487 0.977 12.5
2 2202.475 7.359 11.6 41 606.871 2.027 4.7 80 285.925 0.955 7.1
3 2200.776 7.353 8.9 42 597.612 1.997 5.3 81 276.583 0.924 35.5
4 2195.361 7.348 6.2 43 594.215 1.985 6.6 82 270.072 0.902 32.8
5 2197.096 7.341 26.7 44 580.060 1.938 3.8 83 262.712 0.878 68.9
6 2195.114 7.334 40.9 45 557.180 1.861 4.1 84 261.296 0.873 70.6
7 2194.265 7.331 25.9 46 554.016 1.851 6.4 85 256.200 0.856 61.2
8 2193.132 7.327 11.7 47 550.902 1.841 5.0 86 254.785 0.851 66.1
9 2191.434 7.322 14.1 48 544.391 1.819 5.3 87 201.280 0.672 74.0
10 2190.302 7.318 13.0 49 540.993 1.807 6.6 88 -0.000 -0.000 30.8
11 2188.886 7.313 25.1 50 508.486 1.699 3.9
12 2186.338 7.305 14.1 51 504.757 1.686 3.9
13 2184.357 7.298 8.4 52 478.146 1.598 40.8
14 2181.242 7.288 10.0 53 471.352 1.575 5.3
15 2179.261 7.281 7.4 54 467.389 1.562 7.6
16 2174.731 7.266 14.8 55 463.709 1.549 5.6
17 2173.316 7.261 16.7 56 460.878 1.540 9.2
18 2171.617 7.256 58.9 57 454.367 1.518 10.1
19 2169.636 7.249 120.2 58 447.572 1.495 10.3
20 2167.654 7.242 16.3 59 441.344 1.475 13.5
21 2165.955 7.237 16.8 60 437.947 1.463 11.8
22 2163.408 7.228 9.4 61 426.906 1.426 7.1
23 2160.293 7.218 4.8 62 421.611 1.409 6.3
24 2157.463 7.208 4.5 63 415.583 1.388 4.5
25 2605.145 5.363 7.1 64 404.825 1.353 8.7
26 2601.182 5.350 6.6 65 396.898 1.326 10.5
27 2550.225 5.179 4.8 66 391.328 1.274 4.9
28 2538.052 5.139 33.5 67 371.703 1.242 4.3
29 2534.089 5.125 35.4 68 361.228 1.207 4.9
30 2521.916 5.085 5.4 69 354.434 1.184 4.1
31 2395.939 4.664 9.6 70 348.489 1.164 6.7
32 2390.277 4.645 12.6 71 339.430 1.134 14.4
33 2389.144 4.641 18.2 72 330.654 1.105 14.7
34 2383.482 4.622 13.0 73 320.746 1.072 9.0
35 2379.236 4.608 3.9 74 315.084 1.053 6.9
36 935.910 3.127 23.0 75 309.422 1.034 7.6
37 930.531 3.109 15.4 76 306.025 1.022 5.9
38 928.833 3.103 16.9 77 301.496 1.007 8.6
39 676.879 2.262 11.4 78 295.834 0.988 80.0
^{1}H-NMR (300 MHz, CDCl$_3$)
10b
0.93 9.69
0.95 2.00 1.90
1.91
1.83 2.93 12.88
1.95 16.75 2.30
14 13 12 11 10 9 8 7 6 5 4 3 2 1 ppm

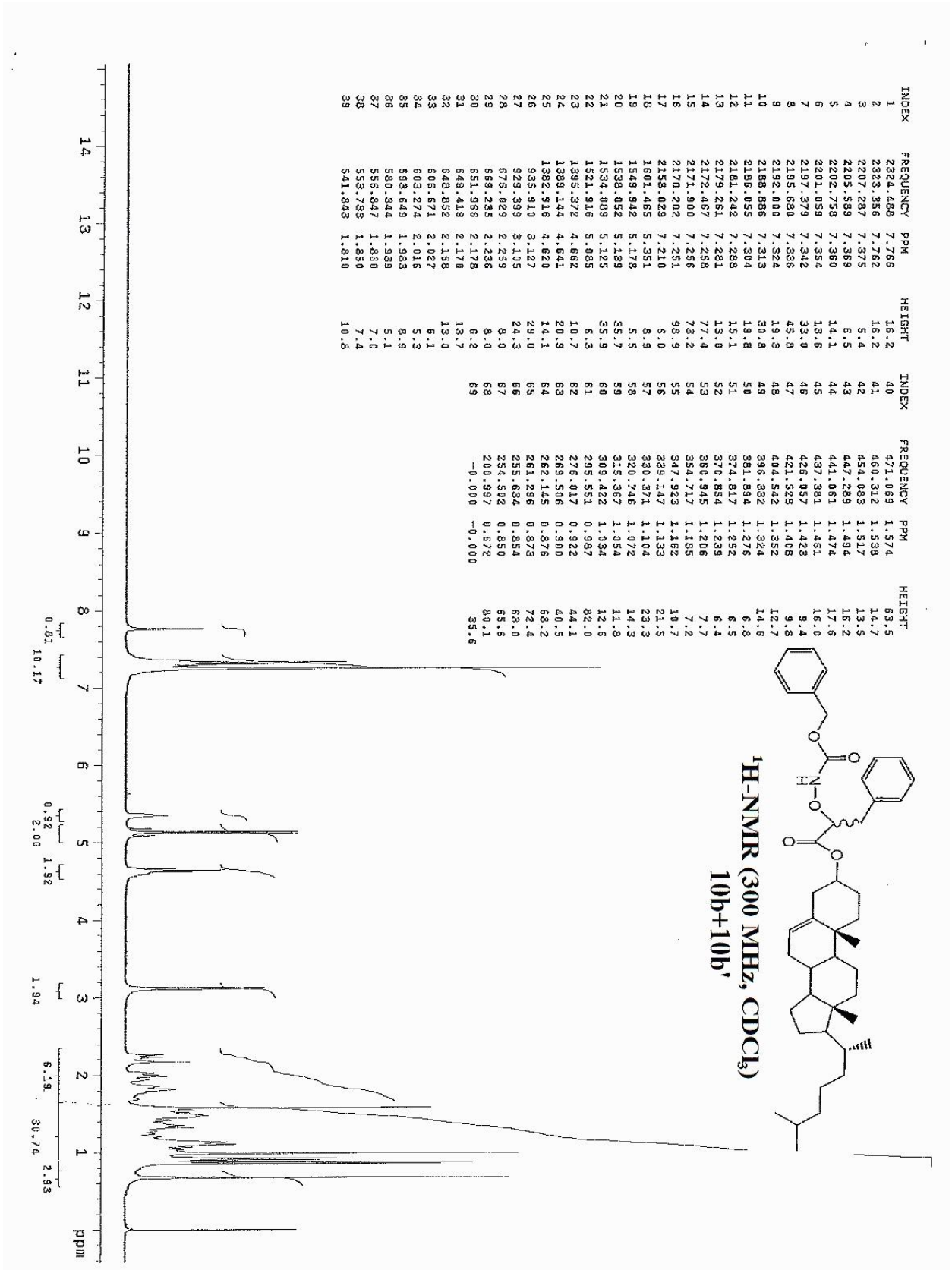


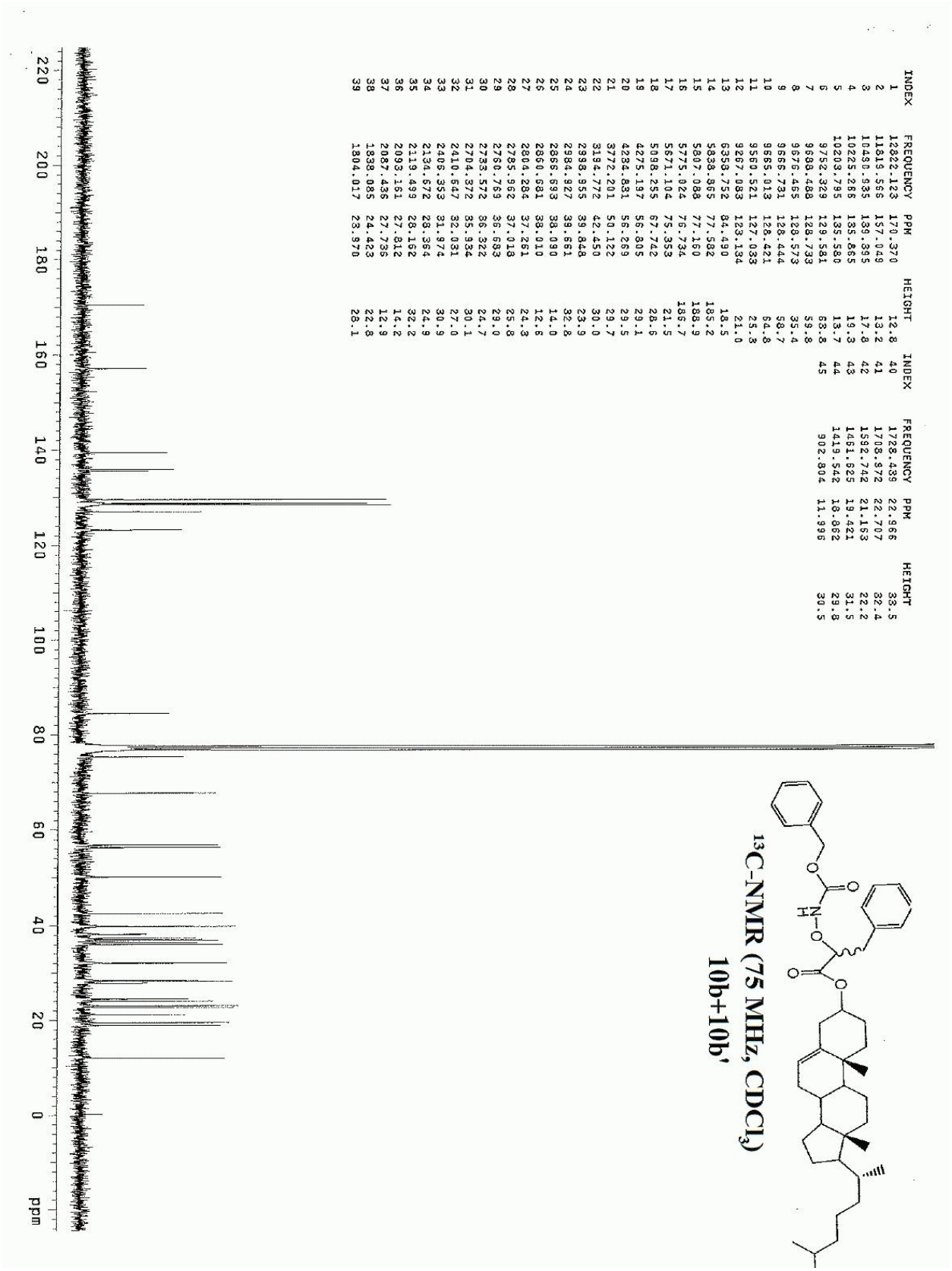
INDEX	FREQUENCY	PPM	HEIGHT
1	12820.681	170.351	13.6
2	11819.852	157.053	12.5
3	10490.935	139.395	17.5
4	10224.979	135.861	17.1
5	10208.795	135.580	10.6
6	9752.329	129.581	38.5
7	9688.202	128.729	33.7
8	9675.892	128.565	20.8
9	9666.159	128.436	49.0
10	9664.441	128.413	47.4
11	9559.862	127.021	18.7
12	9265.937	123.118	14.8
13	6357.606	84.475	13.4
14	5839.151	77.586	98.1
15	5807.988	77.160	101.1
16	5775.310	76.738	98.3
17	5670.245	75.342	13.1
18	5097.397	67.730	20.4
19	4274.911	56.802	19.4
20	4234.831	56.269	19.6
21	3771.914	50.118	19.6
22	3194.485	42.446	27.7
23	2998.955	39.848	15.1
24	2984.641	39.658	21.8
25	2866.407	38.087	14.1
26	2803.998	37.257	15.8
27	2785.676	37.014	15.7
28	2759.910	36.672	22.2
29	2733.572	36.322	16.2
30	2704.086	35.930	19.0
31	2410.647	32.031	16.5
32	2405.758	31.966	19.8
33	2134.672	28.364	15.5
34	2119.213	28.158	22.3
35	2086.577	27.725	13.9
36	1837.799	24.419	14.8
37	1803.731	23.967	18.1
38	1728.439	22.966	22.6
39	1708.972	22.707	21.8

INDEX	FREQUENCY	PPM	HEIGHT
40	1582.742	21.163	14.8
41	1461.625	19.421	18.9
42	1419.255	18.858	20.4
43	902.518	11.992	21.9

^{13}C-NMR (75 MHz, $CDCl_3$)

10b


INDEX FREQUENCY PPM HEIGHT
1 2324.488 7.766 16.2
2 2323.356 7.762 16.2
3 2207.287 7.375 5.4
4 2205.599 7.369 6.5
5 2202.758 7.360 14.1
6 2201.059 7.354 13.6
7 2197.379 7.342 33.0
8 2195.638 7.336 45.8
9 2192.100 7.324 19.3
10 2188.886 7.313 30.8
11 2186.055 7.304 19.8
12 2181.242 7.288 15.1
13 2179.261 7.281 13.0
14 2172.467 7.258 77.4
15 2171.000 7.256 73.2
16 2170.202 7.251 98.9
17 2158.028 7.210 6.0
18 1601.465 5.351 8.9
19 1548.942 5.173 5.5
20 1538.052 5.139 35.7
21 1534.089 5.125 35.9
22 1521.916 5.085 6.3
23 1395.372 4.662 10.7
24 1389.144 4.641 20.9
25 1382.916 4.620 14.1
26 935.910 3.127 29.0
27 929.399 3.105 24.3
28 675.029 2.259 8.0
29 669.235 2.236 8.0
30 651.966 2.178 6.2
31 648.418 2.170 13.7
32 648.852 2.168 13.0
33 606.671 2.027 6.1
34 603.274 2.016 5.3
35 593.649 1.983 8.9
36 580.844 1.939 5.1
37 556.847 1.860 7.0
38 553.733 1.850 7.4
39 541.843 1.810 10.8
INDEX FREQUENCY PPM HEIGHT
40 471.069 1.574 63.5
41 460.312 1.538 14.7
42 454.083 1.517 13.3
43 447.289 1.494 16.2
44 441.061 1.474 17.6
45 437.381 1.461 16.0
46 426.057 1.423 9.4
47 421.528 1.408 9.8
48 404.542 1.352 12.7
49 396.332 1.324 14.6
50 381.894 1.276 6.8
51 374.817 1.252 6.5
52 370.854 1.239 6.4
53 360.945 1.206 7.7
54 354.717 1.185 7.2
55 347.923 1.162 10.7
56 339.147 1.133 21.5
57 330.371 1.104 23.3
58 320.746 1.072 14.3
59 315.367 1.054 11.8
60 309.422 1.034 12.6
61 295.551 0.987 82.0
62 276.017 0.922 44.1
63 269.506 0.900 40.5
64 262.145 0.876 68.2
65 261.296 0.873 72.4
66 255.634 0.854 68.0
67 254.592 0.850 65.6
68 200.997 0.672 80.1
69 -0.000 -0.000 35.6
0.81
10.17
0.92
2.00
1.92
1.94
6.19
30.74
2.93
1
2
3
4
5
6
7
8
9
10
11
12
13
14
ppm
^{1}H-NMR (300 MHz, CDCl$_3$)
10b+10b'

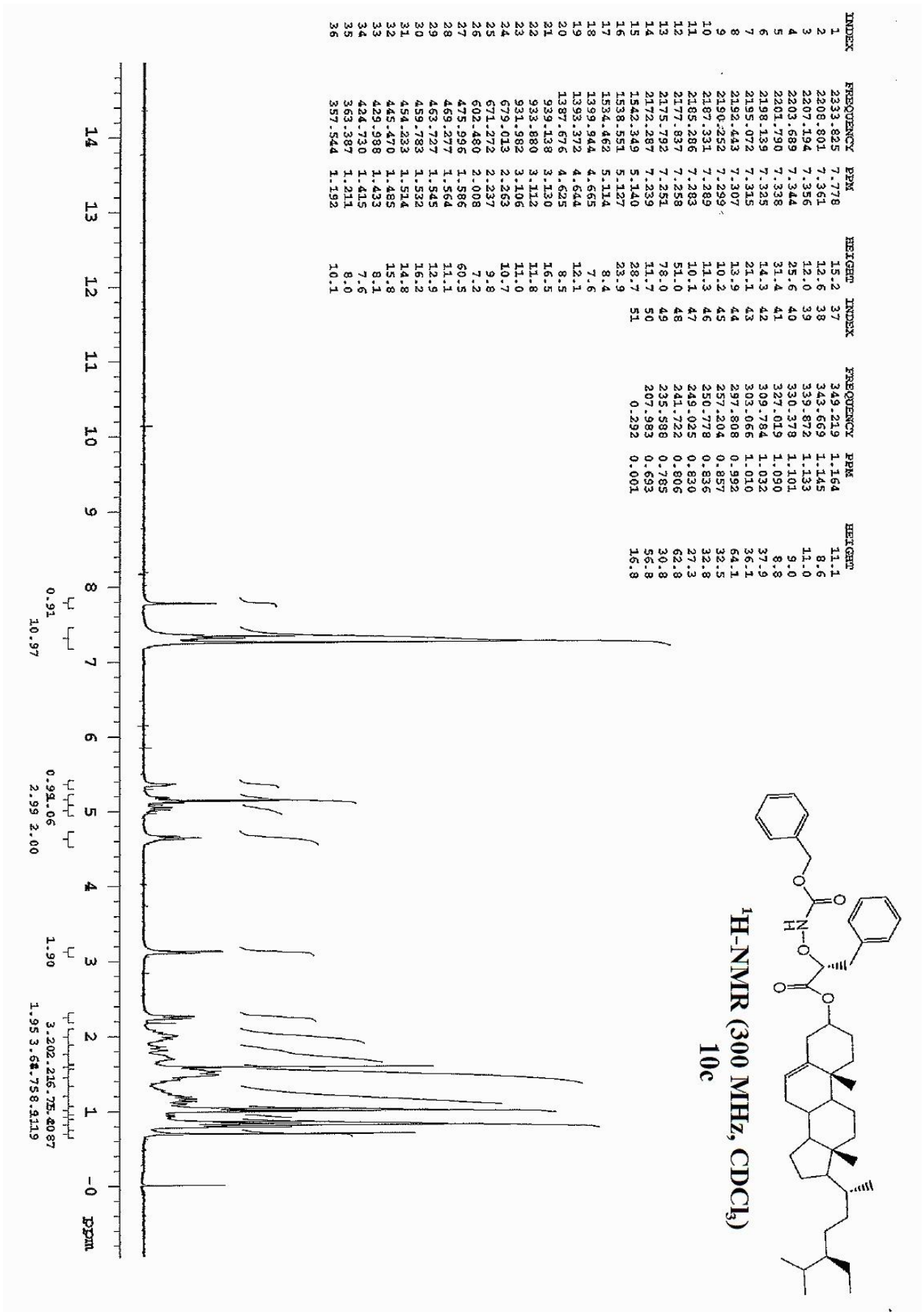


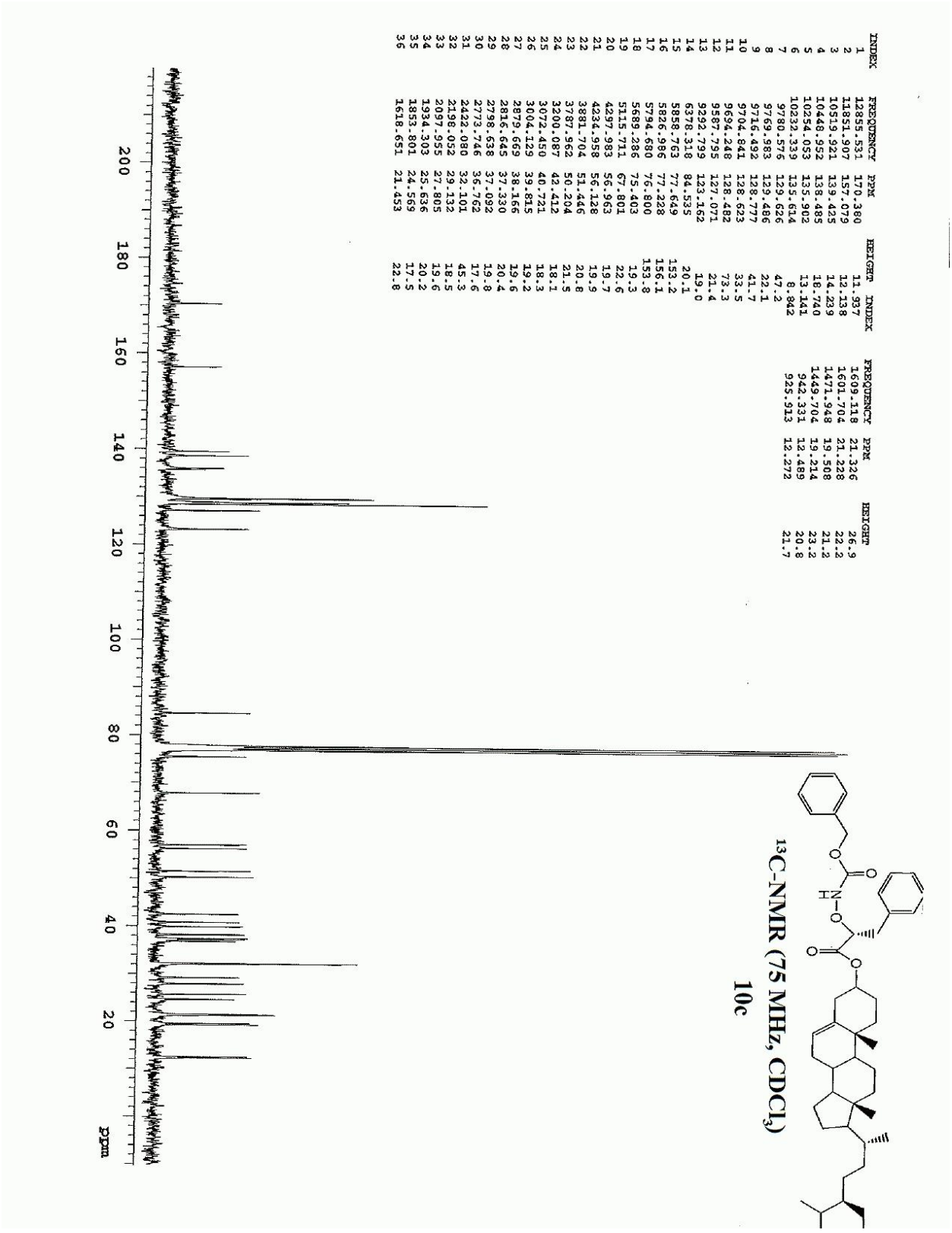
INDEX	FREQUENCY	PPM	HEIGHT
1	12822.123	170.370	12.8
2	11819.566	157.049	13.2
3	10480.935	139.295	17.8
4	10225.265	135.865	19.3
5	10203.795	135.580	13.7
6	9752.329	129.581	63.8
7	9688.488	128.733	59.8
8	9676.465	128.573	35.4
9	9666.731	128.444	58.7
10	9565.013	128.421	64.8
11	9560.521	127.033	25.3
12	9267.083	123.134	21.0
13	6358.752	84.490	18.5
14	5838.865	77.582	185.2
15	5807.088	77.160	188.9
16	5775.024	76.734	186.7
17	5671.104	75.353	21.5
18	5098.255	67.742	28.6
19	4275.197	56.805	29.1
20	4234.831	56.269	29.5
21	3772.201	50.122	29.7
22	3184.772	42.450	30.0
23	2998.955	39.848	23.9
24	2984.927	39.661	32.8
25	2866.693	38.090	14.0
26	2860.681	38.010	12.6
27	2804.284	37.261	24.3
28	2785.962	37.013	25.8
29	2760.769	36.683	29.0
30	2733.572	36.322	24.7
31	2704.372	35.934	30.1
32	2410.647	32.031	27.0
33	2406.353	31.974	30.9
34	2134.672	28.364	24.9
35	2119.499	28.162	32.2
36	2093.161	27.812	14.2
37	2087.436	27.735	12.9
38	1838.085	24.423	22.8
39	1804.017	23.970	28.1

INDEX	FREQUENCY	PPM	HEIGHT
40	1728.439	22.966	33.5
41	1708.972	22.707	32.4
42	1592.742	21.163	22.2
43	1461.625	19.421	31.5
44	1419.542	18.862	29.8
45	902.804	11.996	30.5

^{13}C-NMR (75 MHz, $CDCl_3$)

10b+10b'

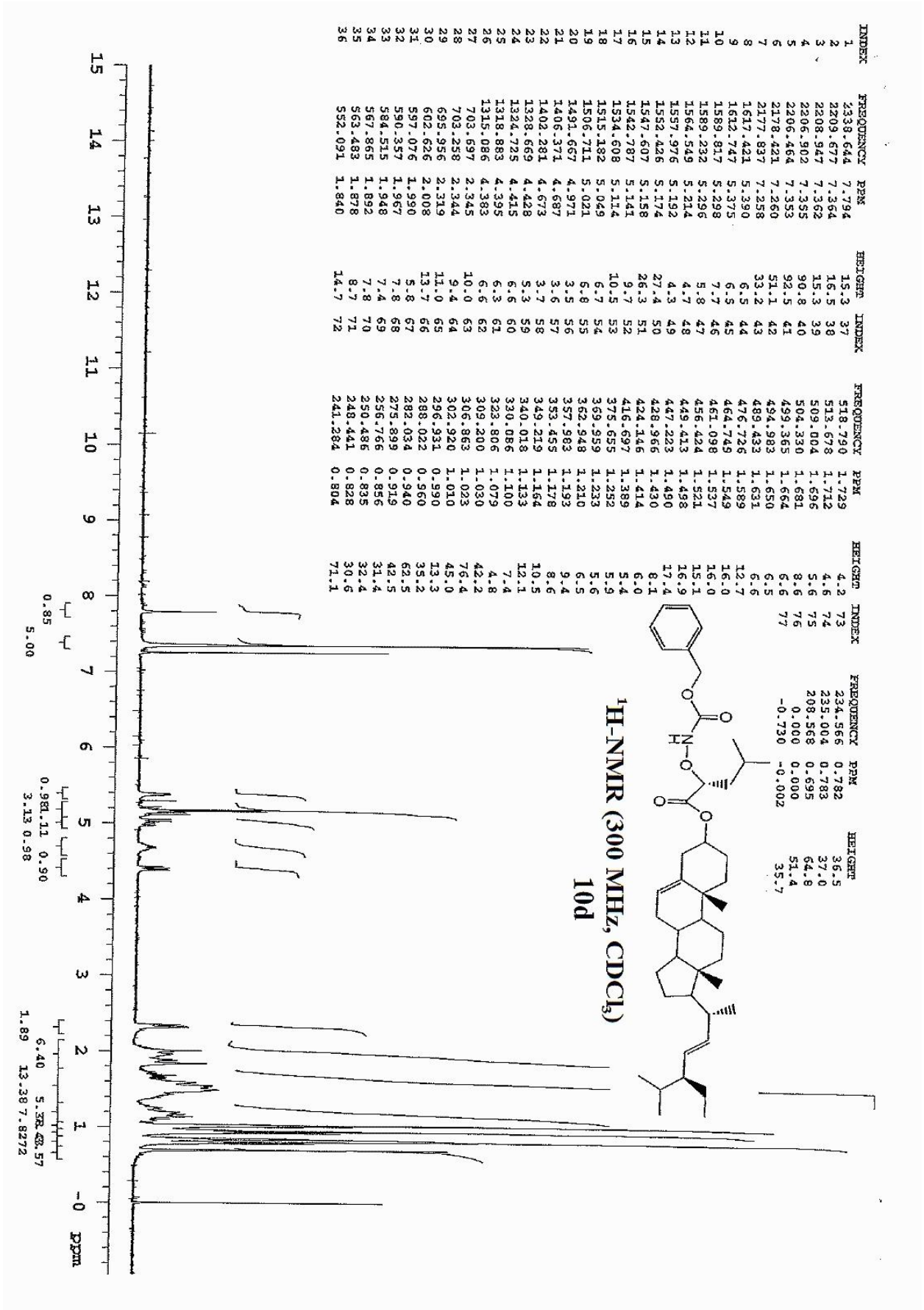

INDEX FREQUENCY PPM HEIGHT INDEX FREQUENCY PPM HEIGHT
1 2333.625 7.778 15.2 37 349.219 1.164 11.1
2 2208.801 7.361 12.6 38 343.669 1.145 8.6
3 2207.194 7.356 12.0 39 339.872 1.133 11.0
4 2203.689 7.344 25.6 40 330.378 1.101 9.0
5 2201.790 7.338 31.4 41 327.019 1.090 8.8
6 2198.139 7.325 14.3 42 309.784 1.032 37.9
7 2195.072 7.315 21.1 43 303.066 1.010 36.1
8 2192.443 7.307 13.9 44 297.808 0.992 64.1
9 2190.252 7.299 10.2 45 257.204 0.857 32.5
10 2187.331 7.289 11.3 46 250.778 0.836 32.8
11 2185.286 7.283 10.1 47 249.025 0.830 27.3
12 2177.837 7.258 51.0 48 241.722 0.806 62.8
13 2175.792 7.251 78.0 49 235.586 0.785 30.8
14 2172.287 7.239 11.7 50 207.983 0.693 56.8
15 1542.349 5.140 28.7 51 0.292 0.001 16.8
16 1538.551 5.127 23.9
17 1534.462 5.114 8.4
18 1399.944 4.665 7.6
19 1393.372 4.644 12.1
20 1387.676 4.625 8.5
21 939.138 3.130 16.5
22 933.880 3.112 11.8
23 931.982 3.106 11.0
24 679.013 2.263 10.7
25 671.272 2.237 9.8
26 602.480 2.008 7.2
27 475.996 1.586 60.5
28 469.277 1.564 11.1
29 463.727 1.545 12.9
30 459.783 1.532 16.2
31 454.233 1.514 14.8
32 445.470 1.485 15.8
33 429.988 1.433 8.1
34 424.730 1.415 7.6
35 363.387 1.211 8.0
36 357.544 1.192 10.1
^{1}H-NMR (300 MHz, CDCl$_3$)
10c
14 13 12 11 10 9 8 7 6 5 4 3 2 1 -0 ppm
10.97 0.91 2.99 2.00 0.99 1.06 1.90 1.95 3.68 3.02 2.21 6.75 8.75 8.40 7.87 9.11 9

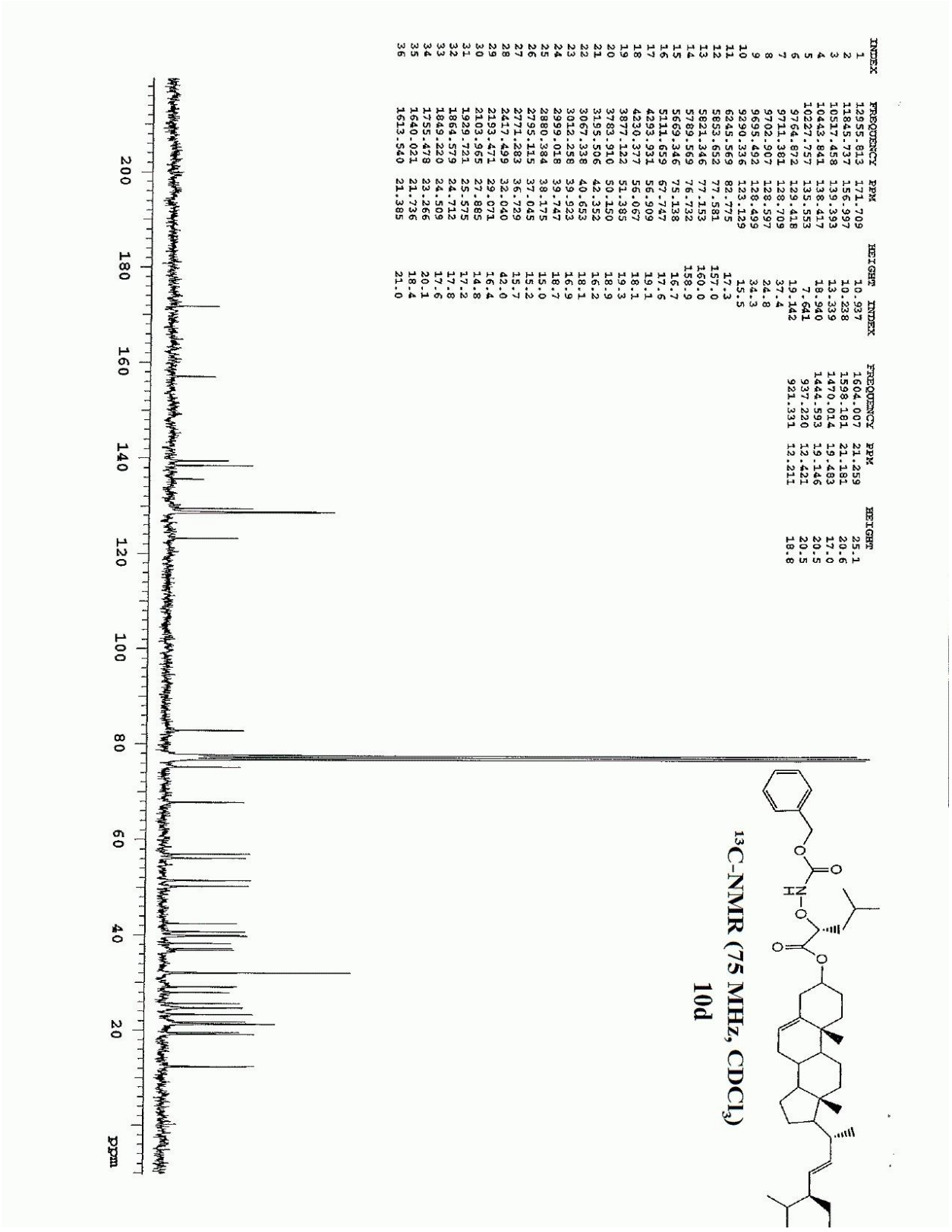


INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	12855.531	170.380	11.937		1609.118	21.326	26.9
2	11851.907	157.079	12.138		1601.704	21.228	22.2
3	10519.921	139.425	14.239		1471.948	19.508	21.2
4	10448.952	138.485	18.740		1449.704	19.214	23.2
5	10234.053	135.902	13.141		942.331	12.489	20.8
6	10232.339	135.614	8.842		925.913	12.272	21.7
7	9780.576	129.626	47.2				
8	9769.983	129.486	22.1				
9	9716.492	128.777	41.7				
10	9704.841	128.623	33.5				
11	9694.248	128.482	73.3				
12	9587.795	127.071	21.4				
13	9292.799	123.162	19.0				
14	6378.318	84.535	20.1				
15	5858.763	77.649	153.2				
16	5826.986	77.228	156.1				
17	5794.680	76.800	153.8				
18	5689.286	75.403	19.3				
19	5115.711	67.801	22.6				
20	4297.983	56.963	19.7				
21	4234.958	56.128	19.9				
22	3881.704	51.446	20.8				
23	3787.962	50.204	21.5				
24	3200.087	42.412	18.1				
25	3072.450	40.721	18.3				
26	3004.129	39.815	19.2				
27	2879.669	38.166	19.6				
28	2816.645	37.330	20.4				
29	2798.638	37.092	19.8				
30	2773.746	36.762	17.6				
31	2422.080	32.101	45.3				
32	2198.052	29.132	18.5				
33	2097.955	27.805	19.6				
34	1934.303	25.636	20.2				
35	1853.801	24.569	17.5				
36	1618.651	21.453	22.8				

^{13}C-NMR (75 MHz, $CDCl_3$)

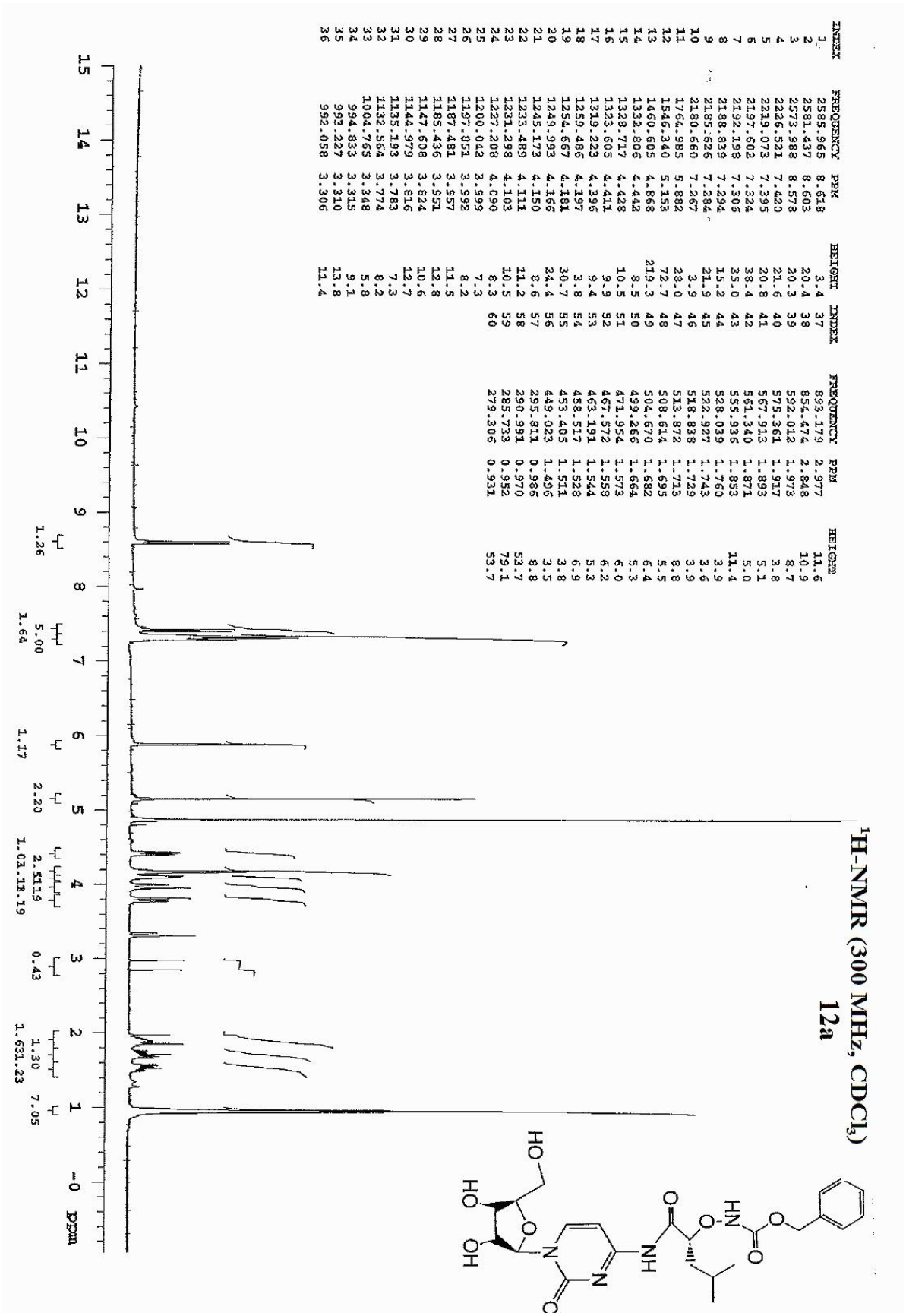
10c

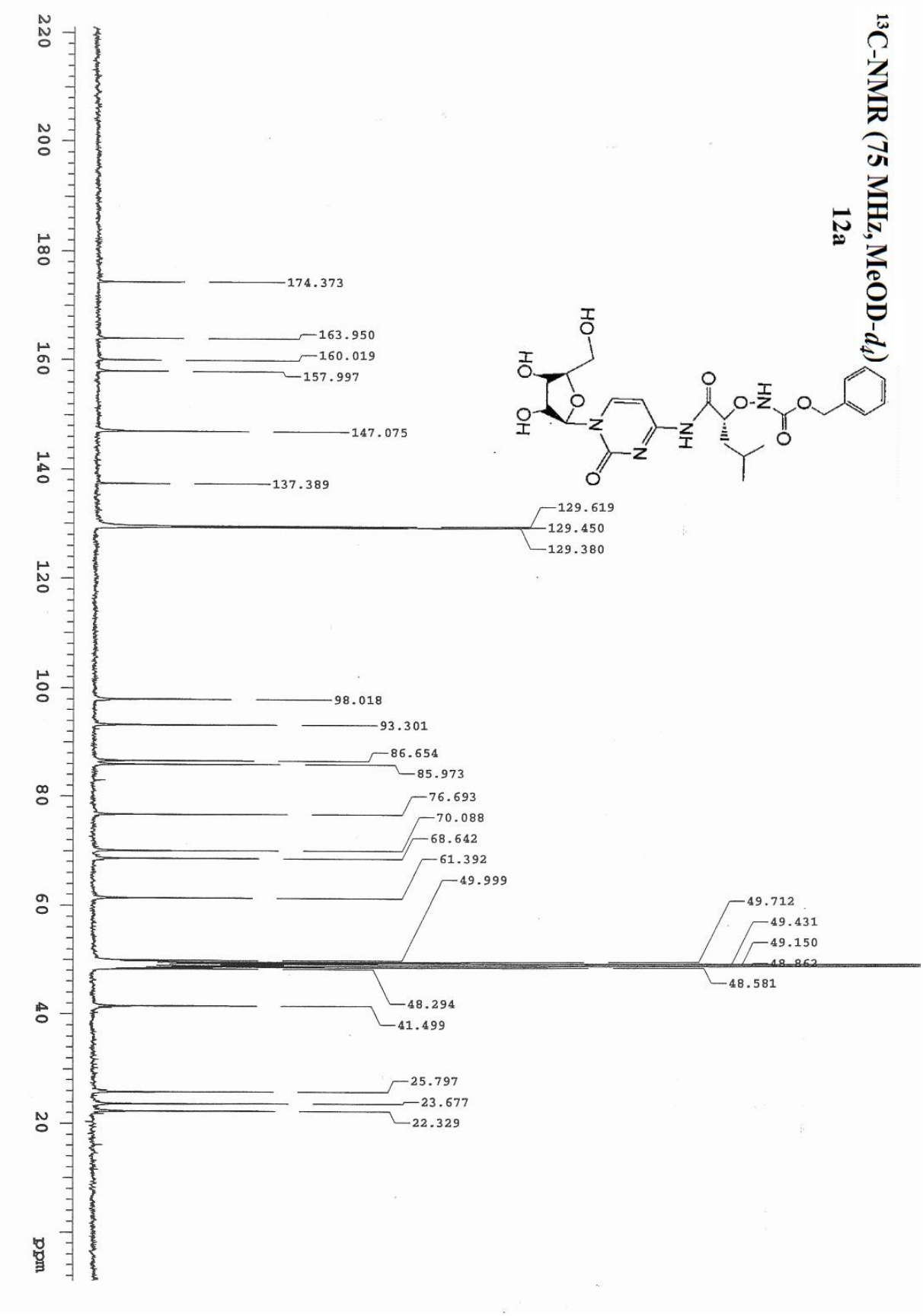

INDEX FREQUENCY PPM HEIGHT INDEX FREQUENCY PPM HEIGHT INDEX FREQUENCY PPM HEIGHT
1 2338.644 7.794 15.3 37 518.790 1.729 4.2 73 234.566 0.782 36.5
2 2209.677 7.364 16.5 38 513.678 1.712 4.6 74 235.004 0.783 37.0
3 2208.947 7.362 15.3 39 509.004 1.696 5.6 75 208.568 0.695 64.8
4 2206.902 7.355 90.8 40 504.330 1.681 8.6 76 0.000 0.000 51.4
5 2206.464 7.353 92.5 41 499.365 1.664 6.6 77 -0.730 -0.002 35.7
6 2178.421 7.260 51.1 42 494.983 1.650 6.5
7 2177.837 7.258 33.2 43 489.432 1.631 6.6
8 1617.421 5.390 6.5 44 476.726 1.589 12.7
9 1612.747 5.375 6.5 45 464.749 1.549 16.0
10 1589.817 5.298 7.7 46 461.098 1.537 16.0
11 1589.232 5.296 5.8 47 456.424 1.521 15.1
12 1564.549 5.214 4.7 48 449.413 1.498 16.9
13 1557.976 5.192 4.3 49 447.223 1.490 17.4
14 1552.426 5.174 27.4 50 428.966 1.430 8.1
15 1547.607 5.158 26.3 51 424.146 1.414 6.0
16 1542.787 5.141 9.7 52 416.697 1.389 5.4
17 1534.608 5.114 10.5 53 375.655 1.252 5.9
18 1515.182 5.049 6.7 54 369.959 1.233 5.6
19 1506.711 5.021 6.8 55 362.948 1.210 6.5
20 1491.667 4.971 3.5 56 357.983 1.193 9.4
21 1406.371 4.687 3.6 57 353.455 1.178 8.6
22 1402.281 4.673 3.7 58 349.219 1.164 10.5
23 1328.669 4.428 5.3 59 340.018 1.133 12.1
24 1324.725 4.415 6.6 60 330.086 1.100 7.4
25 1318.883 4.395 6.3 61 323.806 1.079 4.8
26 1315.086 4.383 6.6 62 309.200 1.030 42.2
27 703.697 2.345 10.0 63 306.863 1.023 76.4
28 703.258 2.344 9.4 64 302.920 1.010 45.0
29 695.956 2.319 11.0 65 296.931 0.990 13.3
30 602-626 2.008 13.7 66 288.022 0.960 35.2
31 597.076 1.990 5.8 67 282.034 0.940 62.5
32 590.357 1.967 7.8 68 275.899 0.919 42.5
33 584.515 1.948 7.4 69 256.766 0.856 31.4
34 567.865 1.892 7.8 70 250.486 0.835 32.4
35 563.483 1.878 8.7 71 248.441 0.828 30.6
36 552.091 1.840 14.7 72 241.284 0.804 71.1
^{1}H-NMR (300 MHz, CDCl$_3$)
10d
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 -0 ppm
0.85 5.00
0.98 3.13 0.90 1.11 0.98
1.89 13.38 7.8272

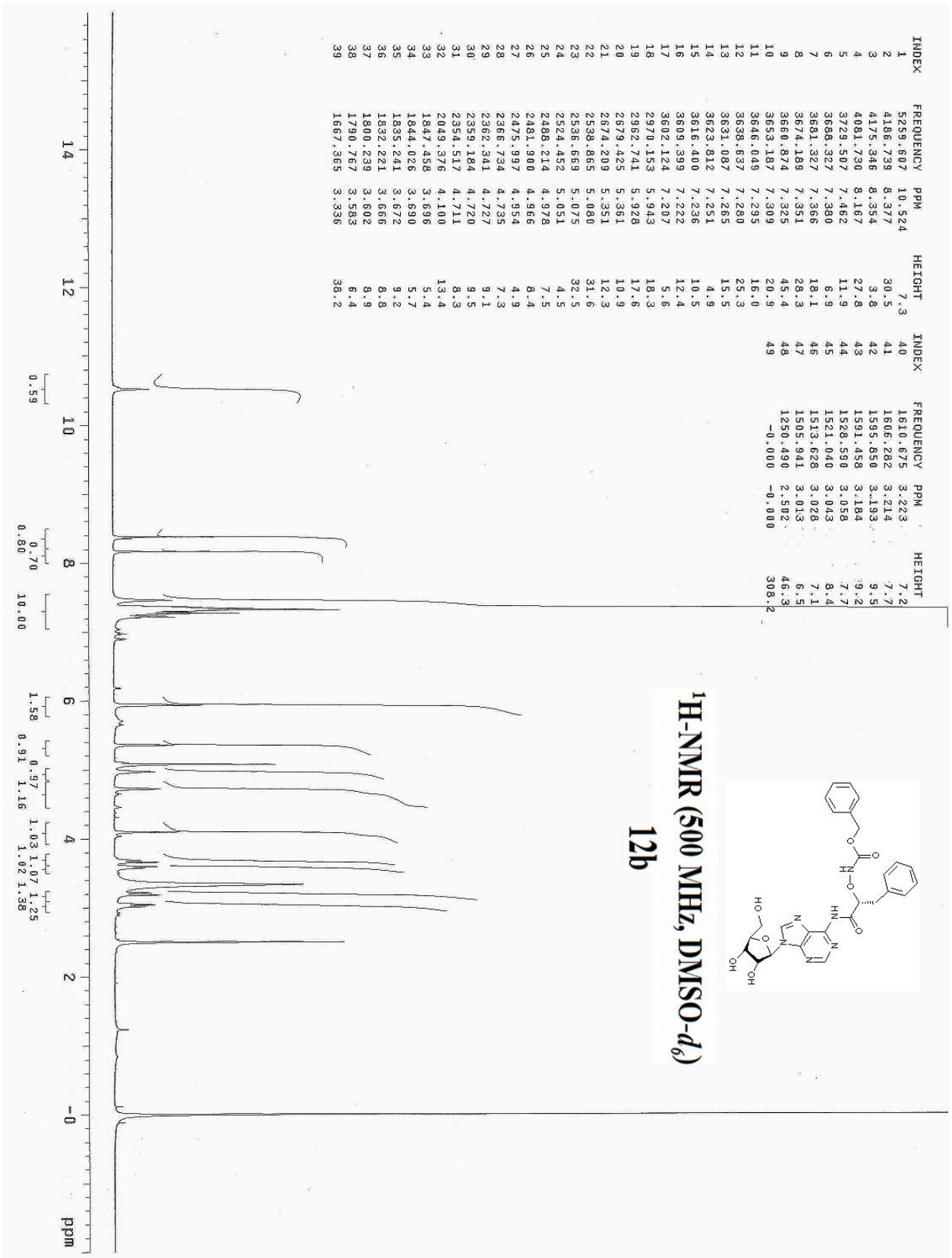


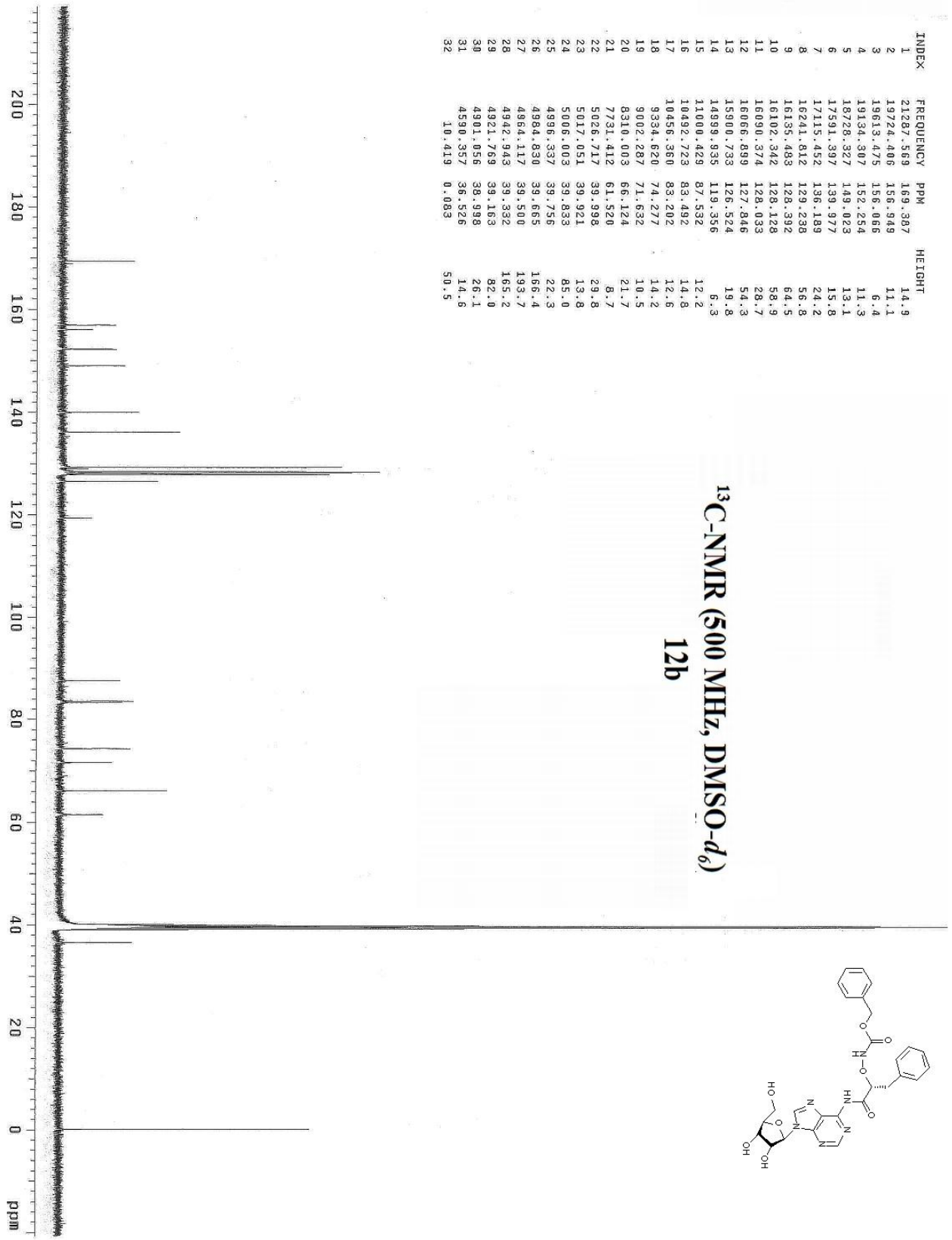
^{13}C–NMR (75 MHz, $CDCl_3$)

10d


^{1}H-NMR (300 MHz, CDCl$_3$)
12a
INDEX FREQUENCY PPM HEIGHT INDEX FREQUENCY PPM HEIGHT
1 2585.965 8.618 3.4 37 893.179 2.977 11.6
2 2581.437 8.603 20.4 38 854.474 2.848 10.9
3 2573.988 8.578 20.3 39 592.012 1.973 8.7
4 2226.521 7.420 21.6 40 575.361 1.917 3.8
5 2219.073 7.395 20.8 41 567.913 1.893 5.1
6 2197.602 7.324 38.4 42 561.340 1.871 5.0
7 2192.198 7.306 35.0 43 555.936 1.853 11.4
8 2188.839 7.294 15.2 44 528.039 1.760 3.9
9 2185.626 7.284 21.9 45 522.927 1.743 3.6
10 2180.660 7.267 3.9 46 518.838 1.729 3.9
11 1764.985 5.882 28.0 47 513.872 1.713 8.8
12 1546.340 5.153 72.7 48 508.614 1.695 5.5
13 1460.605 4.868 219.3 49 504.670 1.682 6.4
14 1332.806 4.442 8.5 50 499.266 1.664 5.3
15 1328.717 4.428 10.5 51 471.954 1.573 6.0
16 1323.605 4.411 9.9 52 467.572 1.558 6.2
17 1319.223 4.396 9.4 53 463.191 1.544 5.3
18 1259.486 4.197 3.8 54 458.517 1.528 6.9
19 1254.667 4.181 30.7 55 453.405 1.511 3.8
20 1249.993 4.166 24.4 56 449.023 1.496 3.5
21 1245.173 4.150 8.6 57 295.811 0.986 8.8
22 1233.489 4.111 11.2 58 290.991 0.970 53.7
23 1231.298 4.103 10.5 59 285.733 0.952 79.1
24 1227.208 4.090 8.3 60 279.306 0.931 53.7
25 1200.042 3.999 7.3
26 1197.851 3.992 8.2
27 1187.481 3.957 11.5
28 1185.436 3.951 12.8
29 1147.608 3.824 10.6
30 1144.979 3.816 12.7
31 1135.193 3.783 7.3
32 1132.564 3.774 8.2
33 1004.765 3.348 5.8
34 994.833 3.315 9.1
35 993.227 3.310 13.8
36 992.058 3.306 11.4
1.26
5.00
1.64
1.17
2.20
2.5119
1.03.18.19
0.43
1.30
7.05
1.631.23
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 -0 ppm


^{13}C-NMR (75 MHz, MeOD-d_4)
12a
174.373
163.950
160.019
157.997
147.075
137.389
129.619
129.450
129.380
98.018
93.301
86.654
85.973
76.693
70.088
68.642
61.392
49.999
49.712
49.431
49.150
48.581
48.294
41.499
25.797
23.677
22.329
ppm





INDEX	FREQUENCY	PPM	HEIGHT
1	21287.569	169.387	14.9
2	19724.406	156.949	11.1
3	19613.475	156.066	6.4
4	19134.307	152.254	11.3
5	18728.327	149.023	13.1
6	17591.397	139.977	15.8
7	17115.452	136.189	24.2
8	16241.812	129.238	56.8
9	16135.483	128.392	64.5
10	16102.342	128.128	58.9
11	16090.374	128.033	28.7
12	16066.899	127.846	54.3
13	15900.733	126.524	19.8
14	14999.935	119.356	6.3
15	11000.429	87.532	12.2
16	10492.723	83.492	14.8
17	10456.360	83.202	12.6
18	9334.620	74.277	14.2
19	9002.287	71.632	10.5
20	8310.003	66.124	21.7
21	7731.412	61.520	8.7
22	5026.717	39.998	29.8
23	5017.051	39.921	13.8
24	5006.003	39.833	85.0
25	4996.337	39.756	22.3
26	4984.830	39.665	166.4
27	4964.117	39.500	193.7
28	4942.943	39.332	165.2
29	4921.769	39.163	82.0
30	4901.056	38.998	26.1
31	4590.357	36.526	14.6
32	10.419	0.083	50.5

^{13}C-NMR (500 MHz, DMSO-d_6)

12b

References:

(1) Katritzky, A. R., Avan, I., and Tala, S. R. (2009) Efficient Preparation of Aminoxyacyl Amides, Aminoxy Hybrid Peptides, and α-Aminoxy Peptides. *J. Org. Chem.*, 74, 8690-8694.

(2) Tanasova, M., Yang, Q., Olmsted, C., C.; Vasileiou, C., Li, X., Anyika, M., and Borhan, B. (2009) An Unusual Conformation of α-Haloamides Due to Cooperative Binding with Zincated Porphyrins. *Eur. J. Org. Chem.,* 25, 4242-4253.