Design, Synthesis and in *vitro* Evaluation of D-Glucose-Based Cationic Glycolipids for Gene Delivery

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Contents

General experimental procedures. Chemicals used were reagent grade as supplied except where noted. Analytical thin-layer chromatography was performed using silica gel 60 F254 glass plates; Compound spots were visualized by UV light (254 nm), or by charring with 30% sulfuric acid-alcohol, or by staining with iodine in silica gel. Flash column chromatography was performed on columns (16×240 mm, 18×300 mm, 35×400 mm) of silica gel 60 (200-300 Mesh) with EtOAc-petroleum ether (60-90°C) as the eluent. Solutions were concentrated at <60°C under reduced pressure. NMR spectra were referenced using Me_4Si (0 ppm), residual $CHCl_3$ (^{1}H-NMR 7.26 ppm, ^{13}C NMR 77.0 ppm) for $CDCl_3$, or using residual DOH (1H-NMR 4.79 ppm) for D_2O, or using residual CD_3OH (1H-NMR 4.87 ppm, ^{13}C NMR 49.0 ppm) for CD_3OD. Peak assignments are based on ^{1}H NMR, ^{1}H-^{1}H gCOSY, ^{13}C NMR and (or) 1H-^{13}C gHSQC and ^{1}H-^{13}C gHMBC experiments. NMR experiments were conducted at 500, and 125 MHz for ^{1}H, ^{13}C, respectively, using Bruker Avance 500 MHz NMR Spectrometer equipped with a switchable QNP (1H, ^{13}C) probe enabling back-to-back data acquisition for the different nuclei without the need to remove sample or tune the probe. Electrospray ionization mass spectrometry (ESI-MS) in positive and negative mode was performed on a Finnigan LCQ Advantage (Thermo Finnigan LCQ) equipped with an atmospheric pressure ionization (API) source.

Synthesis of lipid 2

3'-Azidopropyl 2,3-di-*O*-tetradecyl-4,6-*O*-isopropylidene-β-D-glucopyranoside (15b)

NaH (3.3 g, 138.6 mmol) was added slowly to the solution of compound **14** (7.0 g, 23.1 mmol) in dry DMF (100.0 mL) and then myristyl bromide (27.5 mL, 92.4 mmol) was added dropwise. The reaction mixture was stirring until TLC (petroleum ether/ethyl acetate 2:1) showed the starting material was disappeared. The mixture was diluted with DCM (200.0 mL), washed with water for three times. The organic layer was dried by anhydrous Na_2SO_4 and concentrated to dryness. The residue was purified by silica gel column chromatography with petroleum ether : EtOAc = 16 : 1 as the eluent to give compound **15b** (6.5 g, 40.3 %) as a syrup. ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) 4.30 (d, 1 H, $J_{1,2}$ = 7.5 Hz, H-1), 3.94-3.87 (m, 2 H, $OCH_2CH_2CHHN_3$, H-6a), 3.76-3.70 (m, 3 H, $OCH_2(CH_2)_{12}CH_3$, H-6b), 3.66-3.58 (m, 3 H, $OCH_2CH_2CHHN_3$, $OCH_2(CH_2)_{12}CH_3$), 3.53 (dd, 1 H, $J_{4,3}$ = $J_{4,5}$ = 9.0 Hz, H-4), 3.41-3.3.39 (m, 2 H, $OCH_2CH_2CH_2N_3$), 3.25 (dd, 1 H, $J_{3,2}$ = $J_{3,4}$ = 9.0 Hz, H-3), 3.16 (ddd, 1 H, $J_{5,4}$ = 9.0 Hz, $J_{5,6a}$ = 5.5 Hz, $J_{5,6b}$ = 5.0 Hz, H-5), 3.16 (dd, 1 H, $J_{2,1}$ = 7.5 Hz, $J_{2,3}$ = 9.0 Hz, H-2), 1.87-1.85 (m, 2 H, $OCH_2CH_2CH_2N_3$), 1.56-1.46 (m, 4 H, 2 $OCH_2CH_2(CH_2)_{11}CH_3$), 1.46 (s, 3 H, $C(CH_3)_2$), 1.39 (s, 3 H, $C(CH_3)_2$), 1.38-1.20 (m, 44 H, 2 $OCH_2CH_2(CH_2)_{11}CH_3$), 0.87 (t, 6 H, J = 6.5 Hz, 2

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$OCH_2CH_2(CH_2)_{11}CH_3$); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 104.0 (1 C, C-1), 99.2 (1 C, $C(CH_3)_2$), 82.2 (1 C, C-2), 81.7 (1 C, C-3), 73.9 (1 C, C-4), 73.6 (1 C, $OCH_2(CH_2)_{12}CH_3$), 73.2 (1 C, $OCH_2(CH_2)_{12}CH_3$), 66.9 (1 C, C-5), 66.8 (1 C, $OCH_2CH_2CH_2N_3$), 62.2 (1 C, C-6), 48.2 (1 C, $OCH_2CH_2CH_2N_3$), 31.9, 30.3, 30.2, 29.7, 29.6, 29.5, 29.3, 29.2, 29.1, 26.1, 26.0, 22.7, 19.0 (25 C some signals were overlapped, 2 $OCH_2(CH_2)_{12}CH_3$, $OCH_2CH_2CH_2N_3$), 22.7 (1 C, $C(CH_3)_2$), 19.0 (1 C, $C(CH_3)_2$), 14.1, 14.1 (2 C, 2 $OCH_2(CH_2)_{12}CH_3$).

3'-Azidopropyl 2,3-di-*O*-tetradecyl-β-D-glucopyranoside (16b)

The mixture of compound **15b** (6.5 g, 9.3 mmol) and dry methanol (100.0 ml) was cooled to 0°C under stirring, and then the acetyl chloride (2.0 mL, 28.2 mmol) was added dropwise. The reaction mixture was stirring until TLC (petroleum ether : ethyl acetate = 2 : 1) showed the starting material was disappeared, during which time the temperature was gradually raised to ambient temperature. The mixture was evaporated to dryness and the residue was purified by silica gel column chromatography with petroleum ether : EtOAc = 2 : 1 as the eluent to give compound **16b** (5.0 g, 82.0%) as a white oil. ^1H NMR (500 MHz, $CDCl_3$): δ (ppm) 4.30 (d, 1 H, $J_{1,2}$ = 7.5 Hz, H-1), 3.97-3.93 (m, 1 H, $OCH_2CH_2CHHN_3$), 3.91-3.87 (m, 2 H, $OCHH(CH_2)_{12}CH_3$, H-6a), 3.81-3.74 (m, 2 H, $OCHH$ $(CH_2)_{12}CH_3$, H-6b), 3.64-3.56 (m, 3 H, $OCH_2(CH_2)_{12}CH_3$, $OCH_2CH_2CHHN_3$), 3.48-3.41 (m, 3 H, $OCH_2CH_2CH_2N_3$, H-4), 3.33 (ddd, 1 H, $J_{5,4}$ = 9.0 Hz, $J_{5,6a}$ = 4.5 Hz, $J_{5,6b}$ = 4.0 Hz, H-5), 3.19 (dd,1 H, $J_{3,2} = J_{3,4}$ = 9.0 Hz, H-3), 3.06 (dd, 1 H, $J_{2,1}$ = 7.5 Hz, $J_{2,3}$ = 9.0 Hz, H-2), 1.90-1.85 (m, 2 H, $OCH_2CH_2CH_2N_3$), 1.61-1.52 (m, 4 H, 2 $OCH_2CH_2(CH_2)_{11}CH_3$), 1.37-1.20 (m, 44 H, 2 OCH_2 $CH_2(CH_2)_{11}CH_3$), 0.87 (t, 6 H, J = 3.0 Hz, 2 $OCH_2(CH_2)_{12}CH_3$); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 103.7 (1 C, C-1), 84.2 (1 C, C-3), 82.1 (1 C, C-2), 74.8 (1 C, C-5), 73.6 (1 C, $OCH_2(CH_2)_{12}CH_3$), 73.0 (1 C, $OCH_2(CH_2)_{12}$ CH_3), 70.4 (1 C, C-4), 66.5 (1 C, $OCH_2CH_2CH_2N_3$), 62.8 (1 C, C-6), 48.2 (1 C, $OCH_2CH_2CH_2N_3$), 31.9, 30.4, 30.3, 29.7, 29.6, 29.5, 29.3, 29.2, 26.2, 26.1, 22.7 (25 C some signals were overlapped, 2 $OCH_2(CH_2)_{12}CH_3$, $OCH_2CH_2CH_2N_3$), 14.1, 14.1 (2 C, 2 $OCH_2(CH_2)_{12}CH_3$).

3'- (*N,N*-Dimethylamino)propyl 2,3-di-*O*-tetradecyl-β-D-glucopyranoside (17b)

The mixture of compound **16b** (1.0 g, 1.5 mmol) and formaldehyde (36%, 1.0 mL, 12.0 mmol), Pd/C (5%, 300 mg), methanol (60.0 mL) was stirred at the room temperature under H_2 atmosphere. The reaction mixture was stirring until TLC (ethyl acetate : methanol = 5 : 2) showed the starting material was disappeared. The mixture was filtered and the filtrate was evaporated to dryness. The residue was purified by silica gel column chromatography with EtOAc : methanol = 5 : 1 as the eluent to give compound **17b** (0.5 g, 40.6%) as a white oil.

3'- (*N,N,N*-Trimethylaminonium iodine)propyl 2,3-di-*O*-tetradecyl-β-D-glucopyranoside (18b, lipid **2)**

The mixture of compound **17b** (300.0 mg, 0.46 mmol) and iodomethane (112.0 μL, 1.8 mmol), THF (3.0 ml) was stirring at the room temperature until TLC (ethyl acetate : methanol = 2 : 1) showed the starting material was disappeared. The reaction mixture was cooled with ice bath and a solid was precipitated. The mixture was filtered, and the filter cake was washed with acetone (5.0 mL × 3) and dried by vacuum to give white solid **18b** (220.0 mg, 59.5%).^1H NMR (500 MHz, $CDCl_3$): δ (ppm) 4.31 (d, 1 H, $J_{1,2}$ = 7.5 Hz, H-1), 3.91-3.78 (m, 5 H, $OCH_2CH_2CHHN(CH_3)_3$, $OCH_2CH_2CH_2N(CH_3)_3$, $OCHH(CH_2)_{12}CH_3$, H-6a), 3.77-3.65 (m, 4 H, $OCH_2(CH_2)_{12}CH_3$, $OCH_2CH_2CHHN(CH_3)_3$, H-6b), 3.58-3.54 (m, 1 H, $OCHH(CH_2)_{12}CH_3$), 3.46-3.32 (m, 11 H, $OCH_2CH_2CH_2N(CH_3)_3$, H-4, H-5), 3.19 (dd, 1 H, $J_{3,2} = J_{3,4}$ = 9.0 Hz, H-3), 3.10 (s, 1 H, OH), 3.01 (dd, 1 H, $J_{2,1}$ = 7.5 Hz, $J_{2,3}$ = 9.0 Hz, H-2), 2.22-2.20 (m, 1 H, OCH_2 $CHHCH_2N(CH_3)_3$), 2.06-2.00 (m, 1 H, $OCH_2CHHCH_2N(CH_3)_3$), 1.60-1.50 (m, 4 H, 2 OCH_2 CH_2 $(CH_2)_{11}CH_3$), 1.37-1.20 (m, 44 H, 2 $OCH_2CH_2(CH_2)_{11}CH_3$), 0.87 (t, 6 H, J = 7.0 Hz, 2 OCH_2 $CH_2(CH_2)_{11}CH_3$) ; ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 103.9 (1 C, C-1), 84.2 (1 C, C-3), 81.9 (1 C, C-2), 75.6 (1 C, C-5), 73.6 (1 C, $OCH_2(CH_2)_{12}CH_3$), 73.0 (1 C, $OCH_2(CH_2)_{12}$ CH_3), 70.0 (1 C, C-4), 66.5 (1 C, $OCH_2CH_2CH_2N(CH_3)_3$), 64.8 (1 C, $OCH_2CH_2CH_2N(CH_3)_3$), 61.6 (1 C, C-6), 53.9, 53.9, 53.9 (3 C, $OCH_2CH_2CH_2N(CH_3)_3$), 31.8, 30.4, 29.7, 29.6, 29.3, 26.2, 26.1, 24.4, 22.7 (25 C some signals were overlapped, 2 $OCH_2(CH_2)_{12}CH_3$, $OCH_2CH_2CH_2N(CH_3)_3$), 14.0, 14.0 (2 C, 2 $OCH_2CH_2(CH_2)_{11}CH_3$). ESI-MS: m/z = 673.0, in agreement with the calculated mass for $[M]^+ = C_{40}H_{82}NO_6^+$.

Synthesis of lipid 3

3'-Azidopropyl 2,3-di-*O*-hexadecyl-4,6-*O*-isopropylidene-β-D-glucopyranoside (15c)

NaH (4.0 g, 99.6 mmol) was added slowly to the solution of compound **14** (5.0 g, 16.6 mmol) in dry DMF (200.0 mL) and then 1-bromohexadecane (20.3 mL, 66.4 mmol) was added dropwise. The reaction mixture was stirring at 50°C until TLC (petroleum ether/ethyl acetate 2:1) showed the starting material was disappeared. The mixture was diluted with DCM (200.0 mL), washed with water for three times. The organic layer was dried by anhydrous Na_2SO_4 and concentrated to dryness. The residue was purified by silica gel column chromatography with petroleum ether : EtOAc = 16 : 1 as the eluent to give compound **15c** (10.0 g, 80.1 %) as a syrup. ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) 4.30 (d, 1 H, $J_{1,2}$ = 8.0 Hz, H-1), 3.94-3.87 (m, 2 H, $OCH_2CH_2CHHN_3$, H-6a), 3.75-3.70 (m, 3 H, $OCH_2(CH_2)_{14}CH_3$, H-6b), 3.66-3.60 (m, 3 H, $OCH_2CH_2CHHN_3$, $OCH_2(CH_2)_{14}CH_3$), 3.53 (dd, 1 H, $J_{4,3}$ = $J_{4,5}$ = 9.0 Hz, H-4), 3.41-3.3.39 (m, 2 H, $OCH_2CH_2CH_2N_3$), 3.25 (dd, 1 H, $J_{3,2}$ = $J_{3,4}$ = 9.0 Hz, H-3), 3.16 (ddd, 1 H, $J_{5,4}$ = 9.0 Hz, $J_{5,6a}$ = $J_{5,6b}$ = 5.5 Hz, H-5), 3.06 (dd, 1 H, $J_{2,1}$ = 8.0 Hz, $J_{2,3}$ = 9.0 Hz, H-2), 1.87-1.85 (m, 2 H, $OCH_2CH_2CH_2N_3$), 1.56-1.52 (m, 4 H, 2 $OCH_2CH_2(CH_2)_{13}CH_3$), 1.46 (s, 3 H, $C(CH_3)_2$), 1.39 (s, 3 H, $C(CH_3)_2$), 1.38-1.20 (m, 52 H, 2 $OCH_2CH_2(CH_2)_{13}CH_3$), 0.87 (t, 6 H, J = 6.5 Hz, 2 $OCH_2CH_2(CH_2)_{13}CH_3$); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 104.0 (1 C, C-1), 99.2 (1 C, $C(CH_3)_2$), 82.2 (1 C, C-2), 81.7 (1 C, C-3), 73.9 (1 C, C-4), 73.6 (1 C, $OCH_2(CH_2)_{14}CH_3$), 73.1 (1 C, $OCH_2(CH_2)_{14}CH_3$), 67.0 (1 C, C-5), 66.8 (1 C, $OCH_2CH_2CH_2N_3$), 62.3 (1 C, C-6), 48.2 (1 C, $OCH_2CH_2CH_2N_3$), 31.9, 30.3, 30.2, 29.7, 29.6, 29.5, 29.3, 29.2, 29.1, 26.1, 22.7, 19.0 (29 C some signals were overlapped, 2 $OCH_2(CH_2)_{14}CH_3$, $OCH_2CH_2CH_2N_3$), 26.0 (1 C, $C(CH_3)_2$), 19.1 (1 C, $C(CH_3)_2$), 14.1, 14.1 (2 C, 2 $OCH_2(CH_2)_{14}CH_3$).

3'-Azidopropyl 2,3-di-*O*-hexadecyl-β-D-glucopyranoside (16c)

The mixture of compound **15c** (10.0 g, 13.3 mmol) and dry methanol (100.0 ml) was cooled to 0°C under stirring, and then the acetyl chloride (4.2 mL, 53.2 mmol) was added dropwise. The reaction mixture was stirring until TLC (petroleum ether : ethyl acetate = 2 : 1) showed the starting material was disappeared, during which time the temperature was gradually raised to ambient temperature. The mixture was evaporated to dryness and the residue was purified by silica gel column chromatography with petroleum ether : EtOAc = 2 : 1 as the eluent to give compound **16c** (9.0 g, 95.0%) as a white oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) 4.31 (d, 1 H, $J_{1,2}$ = 7.5 Hz, H-1), 3.97-3.93 (m, 1 H, $OCH_2CH_2CHHN_3$), 3.90-3.88 (m, 2 H, $OCHH(CH_2)_{14}CH_3$, H-6a), 3.81-3.73 (m, 2 H, $OCHH$ $(CH_2)_{14}CH_3$, H-6b), 3.64-3.56 (m, 3 H, $OCH_2(CH_2)_{14}CH_3$, $OCH_2CH_2CHHN_3$), 3.48-3.40 (m, 3 H, $OCH_2CH_2CH_2N_3$, H-4), 3.38 (ddd, 1 H, $J_{5,4}$ = 9.0 Hz, $J_{5,6a}$ = 4.5 Hz, $J_{5,6b}$ = 4.0 Hz, H-5), 3.19 (dd,1 H, $J_{3,2}$ = $J_{3,4}$ = 9.0 Hz, H-3), 3.06 (dd, 1 H, $J_{2,1}$ = 7.5 Hz, $J_{2,3}$ = 9.0 Hz, H-2), 2.53(s, 1 H, O*H*), 2.20(s, 1 H, O*H*), 1.90-1.85 (m, 2 H, $OCH_2CH_2CH_2N_3$), 1.61-1.52 (m, 4 H, 2 OCH_2 $CH_2(CH_2)_{13}CH_3$), 1.37-1.20 (m, 52 H, 2 OCH_2 $CH_2(CH_2)_{13}CH_3$), 0.87 (t, 6 H, J = 7.0 Hz, 2 $OCH_2(CH_2)_{14}CH_3$); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 103.8 (1 C, C-1), 84.2 (1 C, C-3), 82.1 (1 C, C-2), 74.8 (1 C, C-5), 73.6 (1 C, $OCH_2(CH_2)_{14}CH_3$), 73.0 (1 C, $OCH_2(CH_2)_{14}CH_3$), 70.5 (1 C, C-4), 66.5 (1 C, $OCH_2CH_2CH_2N_3$), 62.7 (1 C, C-6), 48.2 (1 C, $OCH_2CH_2CH_2N_3$), 31.9, 30.4, 30.3, 29.7, 29.6, 29.5, 29.3, 29.2, 26.2, 26.1, 22.7 (29 C some signals were overlapped, 2 $OCH_2(CH_2)_{14}CH_3$, $OCH_2CH_2CH_2N_3$), 14.1, 14.1 (2 C, 2 $OCH_2(CH_2)_{14}CH_3$).

3'- (*N,N*-Dimethylamino)propyl 2,3-di-*O*-hexadecyl-β-D-glucopyranoside (17c)

The mixture of compound **16c** (1.5 g, 2.1 mmol) and formaldehyde (36%, 1.3 mL, 16.8 mmol), Pd/C (5%, 450 mg), methanol (60.0 mL) was stirred at the room temperature under H_2 atmosphere. The reaction mixture was stirring until TLC (ethyl acetate : methanol = 5 : 2) showed the starting material was disappeared. The mixture was filtered and the filtrate was evaporated to dryness. The residue was purified by silica gel column chromatography with EtOAc : methanol = 5 : 1 as the eluent to give compound **17c** (0.6 g, 40.0%) as a white oil.

3'-(*N,N,N*-Trimethylaminonium iodine)propyl 2,3-di-*O*-hexadecyl-β-D-glucopyranoside (18c, lipid **3)**

The mixture of compound **17c** (250.0 mg, 0.35 mmol) and iodomethane (86.8 μL, 1.4 mmol), THF (3.0 ml) was stirring at the room temperature until TLC (ethyl acetate : methanol = 2 : 1)

showed the starting material was disappeared. The reaction mixture was cooled with ice bath and a solid was precipitated. The mixture was filtered, and the filter cake was washed with acetone (5.0 mL × 3) and dried by vacuum to give white solid **18c** (170.0 mg, 56.7%).^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) 4.31 (d, 1 H, $J_{1,2}$ = 7.5 Hz, H-1), 3.91-3.82 (m, 3 H, $OCH_2CH_2CH_2N(CH_3)_3$, H-6a), 3.80-3.77 (m, 2 H, $OCH_2CH_2CHN(CH_3)_3$, $OCH(CH_2)_{14}CH_3$), 3.75-3.66 (m, 4 H, $OCH_2(CH_2)_{14}CH_3$, $OCH_2CH_2CHN(CH_3)_3$, H-6b), 3.58-3.54 (m, 1 H, $OCH(CH_2)_{14}CH_3$), 3.45-3.32 (m, 11 H, $OCH_2CH_2CH_2N(CH_3)_3$, H-4, H-5), 3.19 (dd, 1 H, $J_{3,2} = J_{3,4}$ = 9.0 Hz, H-3), 3.03 (dd, 1 H, $J_{2,1}$ = 7.5 Hz, $J_{2,3}$ = 9.0 Hz, H-2), 2.28-2.18 (m, 1 H, $OCH_2CHCH_2N(CH_3)_3$), 2.09-2.00 (m, 1 H, $OCH_2CHCH_2N(CH_3)_3$), 1.60-1.50 (m, 4 H, 2 $OCH_2CH_2(CH_2)_{13}CH_3$), 1.37-1.20 (m, 52 H, 2 $OCH_2CH_2(CH_2)_{13}CH_3$), 0.87 (t, 6 H, J = 7.0 Hz, 2 $OCH_2CH_2(CH_2)_{13}CH_3$) ; ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 104.0 (1 C, C-1), 84.2 (1 C, C-3), 82.0 (1 C, C-2), 75.7 (1 C, C-5), 73.6 (1 C, $OCH_2(CH_2)_{14}CH_3$), 73.0 (1 C, $OCH_2(CH_2)_{14}CH_3$), 70.1 (1 C, C-4), 66.6 (1 C, $OCH_2CH_2CH_2N(CH_3)_3$), 64.9 (1 C, $OCH_2CH_2CH_2N(CH_3)_3$), 61.6 (1 C, C-6), 53.9, 53.9, 53.9 (3 C, $OCH_2CH_2CH_2N(CH_3)_3$), 31.9, 30.4, 29.7, 29.6, 29.3, 26.2, 26.1, 24.5, 22.7 (29 C some signals were overlapped, 2 $OCH_2(CH_2)_{14}CH_3$, $OCH_2CH_2CH_2N(CH_3)_3$), 14.1, 14.1 (2 C, 2 $OCH_2CH_2(CH_2)_{13}CH_3$). ESI-MS: m/z = 729.0, in agreement with the calculated mass for $[M]^+$ = $C_{44}H_{90}NO_6^+$.

Synthesis of lipid 5

3'-[(*N*,*N*-di-tetradecylamino]-propyl-β-D-glucopyranoside (20b)

The mixture of compound **19** (1.9 g , 8.0 mmol), myristyl bromide (8.9 g, 32.0 mmol), anhydrous K_2CO_3 (2.2 g, 16.0 mmol), CH_3OH (20.0 mL), CH_3CH_2OH (20.0 mL) was refluxed at 75°C until TLC (methanol) showed the starting material was disappeared.The mixture was diluted with DCM (30.0 mL), washed with water for two times. The organic layer was dried by anhydrous Na_2SO_4 and concentrated to dryness. The residue was purified by silica gel column chromatography with EtOAc : methanol = 4 : 1 as the eluent to give compound **20b** (1.6 g, 32.9 %) as a syrup. ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm): 4.30 (d, 1 H, $J_{1,2}$ = 7.5 Hz, H-1), 3.93-3.87 (m, 1 H, $OCHCH_2CH_2N(CH_2CH_2(C_{11}H_{22})CH_3)_2$), 3.82-3.77 (m, 2 H, H-6), 3.64-3.50 (m, 3 H, $OCHCH_2CH_2N(CH_2CH_2(C_{11}H_{22})CH_3)_2$), H-3, H-4), 3.35 (dd, 1 H, $J_{2,1}$ = 7.5 Hz, $J_{2,3}$ = 3.5 Hz, H-2), 3.30-3.25 (m, 1 H, H-5), 2.78-2.65 (m, 2 H, $OCH_2CH_2CH_2N(CH_2CH_2(C_{11}H_{22})CH_3)_2$), 2.57 (t, J = 6.5 Hz, 4 H, $N(CH_2CH_2(C_{11}H_{22})CH_3)_2$), 1.85-1.80 (m, 2 H, $OCH_2CH_2CH_2N(CH_2CH_2(C_{11}H_{22})CH_3)_2$), 1.50-1.40 (m, 4 H, $N(CH_2CH_2(C_{11}H_{22})CH_3)_2$), 1.37-1.21 (m, 44 H, $N(CH_2CH_2(C_{11}H_{22})CH_3)_2$), 0.89 (t, 6 H, J = 7.0 Hz, $N(CH_2CH_2(C_{11}H_{22})CH_3)_2$); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 103.1 (1 C, C-1), 76.6 (1 C, C-3), 76.0 (1 C, C-5), 73.5 (1 C, C-2), 70.0 (1 C, C-4), 68.2 (1 C, $OCH_2CH_2CH_2N(CH_2CH_2(C_{11}H_{22})CH_3)_2$), 61.7 (1 C, C-6), 52.8, 52.8 (2 C, $N(CH_2CH_2(C_{11}H_{22})CH_3)_2$), 50.7 (1 C, $OCH_2CH_2CH_2N(CH_2CH_2(C_{11}H_{22})CH_3)_2$), 31.9, 29.7, 29.6, 29.5, 29.3, 27.4, 26.3, 24.9, 22.6 (25 C, some signals were overlapped, $N(CH_2(C_{12}H_{24})CH_3)_2$), $OCH_2CH_2CH_2N(CH_2CH_2(C_{11}H_{22})CH_3)_2$), 14.1, 14.1 (2 C, $N(CH_2(C_{12}H_{24})CH_3)_2$).

3'-[(*N*,*N*-di-tetradecyl-*N*-methyl)aminonium iodine]-propyl-β-D-glucopyranoside(21b, lipid**5**)

The mixture of compound **20b** (220.0 mg, 0.32 mmol) and iodomethane (180.0 mg, 1.28 mmol, 79.0 μL), THF (5.0 ml) was stirring at the room temperature until TLC (ethyl acetate : methanol = 5 : 1) showed the starting material was disappeared. The reaction mixture was evaporated to dryness and a solid was precipitated when the acetone (10.0 mL) was drop to the syrup. The mixture was filtered, and the filter cake was washed with acetone (5.0 mL × 3) and dried by vacuum to give white solid **21b** (160.0 mg, 59.2%).^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm): 5.11 (s, 1 H, O*H*), 4.89 (s, 2 H, 2 O*H*), 4.44 (d, 1 H, $J_{1,2}$ = 7.5 Hz, H-1), 4.10-4.00 (m, 2 H, O*H*, $OCHCH_2CH_2N(CH_3)(C_{14}H_{29})_2$), 3.84-3.73 (m, 3 H, H-6, $OCHCH_2CH_2N(CH_3)(C_{14}H_{29})_2$), 3.74-3.50 (m, 4 H, H-3, H-4, $OCH_2CH_2CH_2N(CH_3)(C_{14}H_{29})_2$), 3.44-3.28 (m, 6 H, H-2, H-5, $(CH_3)N(CH_2CH_2(C_{11}H_{22})CH_3)_2$), 3.20 (s, 3 H, $(CH_3)N(CH_2CH_2(C_{11}H_{22})CH_3)_2$), 2.20-2.10 (m, 2 H, $OCH_2CH_2CH_2N(CH_3)(C_{14}H_{29})_2$), 1.72-1.60 (m, 2 H, $(CH_3)N(CH_2CH_2(C_{11}H_{22})CH_3)_2$), 1.40-1.20 (m, 44 H, $(CH_3)N(CH_2CH_2(C_{11}H_{22})CH_3)_2$), 0.85 (t, 6 H, J = 6.5 Hz, $(CH_3)N(CH_2CH_2(C_{11}H_{22})CH_3)_2$); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 102.7 (1 C, C-1), 76.2 (1 C, C-3), 75.9 (1 C, C-5), 73.2 (1 C, C-2), 69.8 (1 C, C-4), 66.2 (1 C, $OCH_2CH_2CH_2N(CH_3)(C_{14}H_{29})_2$), 61.2, 61.2, 61.2 (3 C, $OCH_2CH_2CH_2N(CH_3)(CH_2CH_2(C_{11}H_{22})$

$CH_3)_2$), 60.8 (1 C, C-6), 49.4 (1 C, (CH_3)N($CH_2CH_2(C_{11}H_{22})CH_3)_2$), 31.8, 29.6, 29.5, 29.4, 29.3, 29.1, 26.3, 23.5, 22.5 (25 C, some signals were overlapped, (CH_3)N($CH_2(C_{12}H_{24})CH_3)_2$), $OCH_2CH_2CH_2N(CH_3)(C_{14}H_{29})_2$), 14.0, 14.0 (2 C, (CH_3)N($CH_2(C_{12}H_{24})CH_3)_2$). ESI-MS: *m*/*z* = 644.8, in agreement with the calculated mass for $[M]^+ = C_{38}H_{78}NO_6^+$.

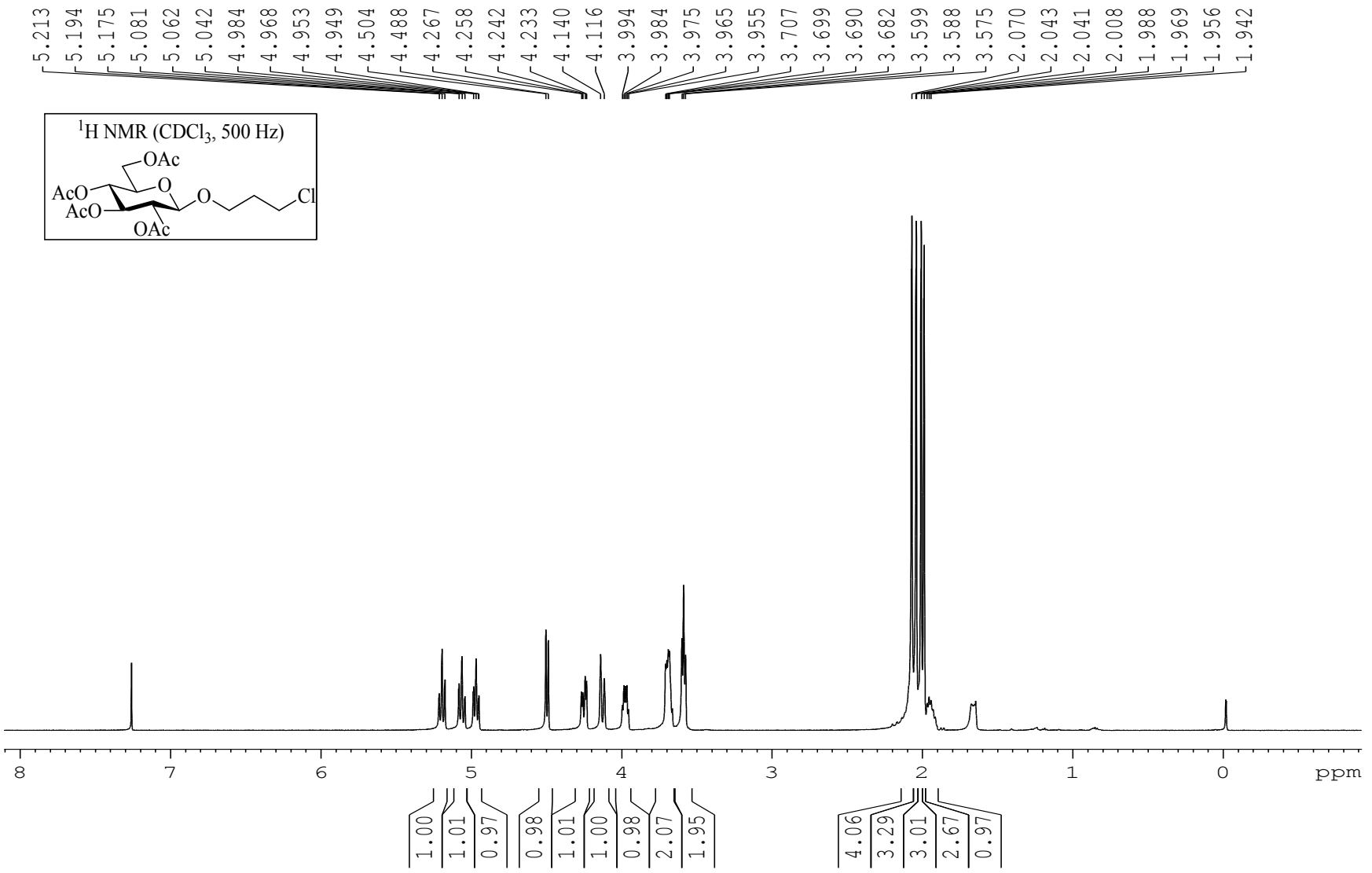
Synthesis of lipid **6**

3'-[(*N*,*N*-di-hexadecylamino]-propyl-β-D-glucopyranoside (20c)

The mixture of compound **20b** (1.2 g , 5.1 mmol), 1-bromohexadecane (6.2 g, 20.4 mmol), anhydrous K_2CO_3 (1.4 g, 10.2 mmol), CH_3OH (20.0 mL), CH_3CH_2OH (20.0 mL) was refluxed at 75°C until TLC (methanol) showed the starting material was disappeared.The mixture was diluted with DCM (30.0 mL), washed with water for two times. The organic layer was dried by anhydrous Na_2SO_4 and concentrated to dryness. The residue was purified by silica gel column chromatography with EtOAc : methanol = 4 : 1 as the eluent to give compound **20c** (1.1 g, 32.4 %) as a syrup. ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm): 4.30 (d, 1 H, $J_{1,2}$ = 7.0 Hz, H-1), 3.93-3.87 (m, 1 H, OC*H*$HCH_2CH_2N(CH_2CH_2(C_{13}H_{26})CH_3)_2$), 3.86-3.78 (m, 2 H, H-6), 3.60-3.51 (m, 3 H, OCH*H*$CH_2CH_2N(CH_2CH_2(C_{13}H_{26})CH_3)_2$), H-3, H-4), 3.36 (dd, 1 H, $J_{2,1}$ = 7.0 Hz, $J_{2,3}$ = 3.5 Hz, H-2), 3.30-3.25 (m, 1 H, H-5), 2.78-2.57 (m, 2 H, $OCH_2CH_2CH_2N(CH_2CH_2(C_{13}H_{26})CH_3)_2$), 2.57 (t, *J* = 6.5 Hz, 4 H, $N(CH_2CH_2(C_{13}H_{26})CH_3)_2$), 1.83-1.73 (m, 2 H, $OCH_2CH_2CH_2N(CH_2$ $CH_2(C_{13}H_{26})CH_3)_2$), 1.50-1.40 (m, 4 H, $N(CH_2CH_2(C_{13}H_{26})CH_3)_2$), 1.31-1.21 (m, 52 H, $N(CH_2$ $CH_2(C_{13}H_{26})CH_3)_2$), 0.87 (t, 6 H, *J* = 7.0 Hz, $N(CH_2CH_2(C_{13}H_{26})CH_3)_2$); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 103.1 (1 C, C-1), 76.5 (1 C, C-3), 76.0 (1 C, C-5), 73.5 (1 C, C-2), 70.0 (1 C, C-4), 68.4 (1 C, $OCH_2CH_2CH_2N(CH_2CH_2(C_{13}H_{26})CH_3)_2$), 61.6 (1 C, C-6), 53.1, 53.1 (2 C, $N(CH_2CH_2(C_{13}H_{26})CH_3)_2$), 50.8 (1 C, $OCH_2CH_2CH_2N(CH_2CH_2(C_{13}H_{26})CH_3)_2$), 31.9, 29.7, 29.6, 29.5, 29.3, 27.6, 26.7, 25.3, 22.7 (29 C, some signals were overlapped, $N(CH_2(C_{14}H_{24})CH_3)_2$, $OCH_2CH_2CH_2N(CH_2CH_2(C_{13}H_{26})CH_3)_2$), 14.1, 14.1 (2 C, $N(CH_2(C_{14}H_{28})CH_3)_2$).

3'-[(*N*,*N*-di-hexadecyl-*N*-methyl)aminonium iodine]-propyl-β-D-glucopyranoside(21c, lipid**6)**

The mixture of compound **20c** (200.0 mg , 0.3 mmol) and iodomethane (170.0 mg, 1.2 mmol, 75.0 μL), THF (5.0 ml) was stirring at the room temperature until TLC (ethyl acetate : methanol = 5 : 1) showed the starting material was disappeared. The reaction mixture was evaporated to dryness and a solid was precipitated when the acetone (10.0 mL) was drop to the syrup. The mixture was filtered, and the filter cake was washed with acetone (5.0 mL × 3) and dried by vacuum to give white solid **21c** (150.0 mg, 60.4%).^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm): 4.33 (d, 1 H, $J_{1,2}$ = 7.5 Hz, H-1), 4.03-3.97 (m, 1 H, OC*H*$HCH_2CH_2N(CH_3)(C_{16}H_{33})_2$), 3.93-3.90 (m, 1 H, H-6a), 3.82- 3.75 (m, 1 H, OCH*H*CH_2CH_2N $(CH_3)(C_{16}H_{33})_2)_2$), 3.73-3.66 (m, 1 H, H-6b), 3.35-3.49 (m, 2 H, $OCH_2CH_2CH_2N(CH_3)(C_{16}H_{33})_2$), 3.43-3.26 (m, 7 H, $OCH_2CH_2CH_2N(CH_3)CH_2CH_2(C_{13}H_{26})_2CH_3$, H-3, H-4, H-5), 3.22 (dd, 1 H, $J_{2,1}$ = 7.5 Hz, $J_{2,3}$ = 4.0 Hz, H-2), 3.09 (s, 3 H, $(CH_3)N(CH_2CH_2(C_{13}H_{26})CH_3)_2$), 2.14-2.05 (m, 2 H, $OCH_2CH_2CH_2N(CH_3)(C_{16}H_{33})_2$), 1.82-1.75 (m, 4 H, $(CH_3)N(CH_2CH_2(C_{13}H_{26})CH_3)_2$), 1.50-1.20 (m, 52 H, $(CH_3)N(CH_2CH_2(C_{13}H_{26})CH_3)_2$), 0.93 (t, 6 H, *J* = 6.5 Hz, $(CH_3)N(CH_2CH_2$ $(C_{13}H_{26})CH_3)_2$); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 103.0 (1 C, C-1), 76.6, 76.6 (2 C, C-3, C-5), 73.6 (1 C, C-2), 70.0 (1 C, C-4), 65.9 (1 C, $OCH_2CH_2CH_2N(CH_3)(C_{16}H_{33})_2$), 61.4, 61.4, (2 C, $N(CH_3)(CH_2CH_2(C_{13}H_{26})CH_3)_2$), 61.3 (1 C, C-6), 59.4 (1 C, $OCH_2CH_2CH_2N(CH_3)(C_{16}H_{33})_2$), 48.1 (1 C, $(CH_3)N(CH_2CH_2(C_{13}H_{26})CH_3)_2$), 31.6, 29.3, 29.2, 29.1, 28.7, 26.0, 22.8, 22.3, 21.7 (29 C, some signals were overlapped, $(CH_3)N(CH_2(C_{14}H_{28})CH_3)_2$), $OCH_2CH_2CH_2N(CH_3)(C_{16}H_{33})_2$), 13.0, 13.0 (2 C, $(CH_3)N(CH_2$ $(C_{14}H_{28})CH_3)_2$). ESI-MS: *m*/*z* = 700.9, in agreement with the calculated mass for $[M]^+ = C_{42}H_{86}NO_6^+$.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4.258
4.242
4.233
4.140
4.116
3.994
3.984
3.975
3.965
3.955
3.707
3.699
3.690
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3.599
3.588
3.575
2.070
2.043
2.041
2.008
1.988
1.969
1.956
1.942
1H NMR (CDCl3, 500 Hz)
OAc
AcO
O
O
Cl
AcO
OAc
8
7
6
5
4
3
2
1
0
ppm
1.00
1.01
0.97
0.98
1.01
1.00
0.98
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1.95
4.06
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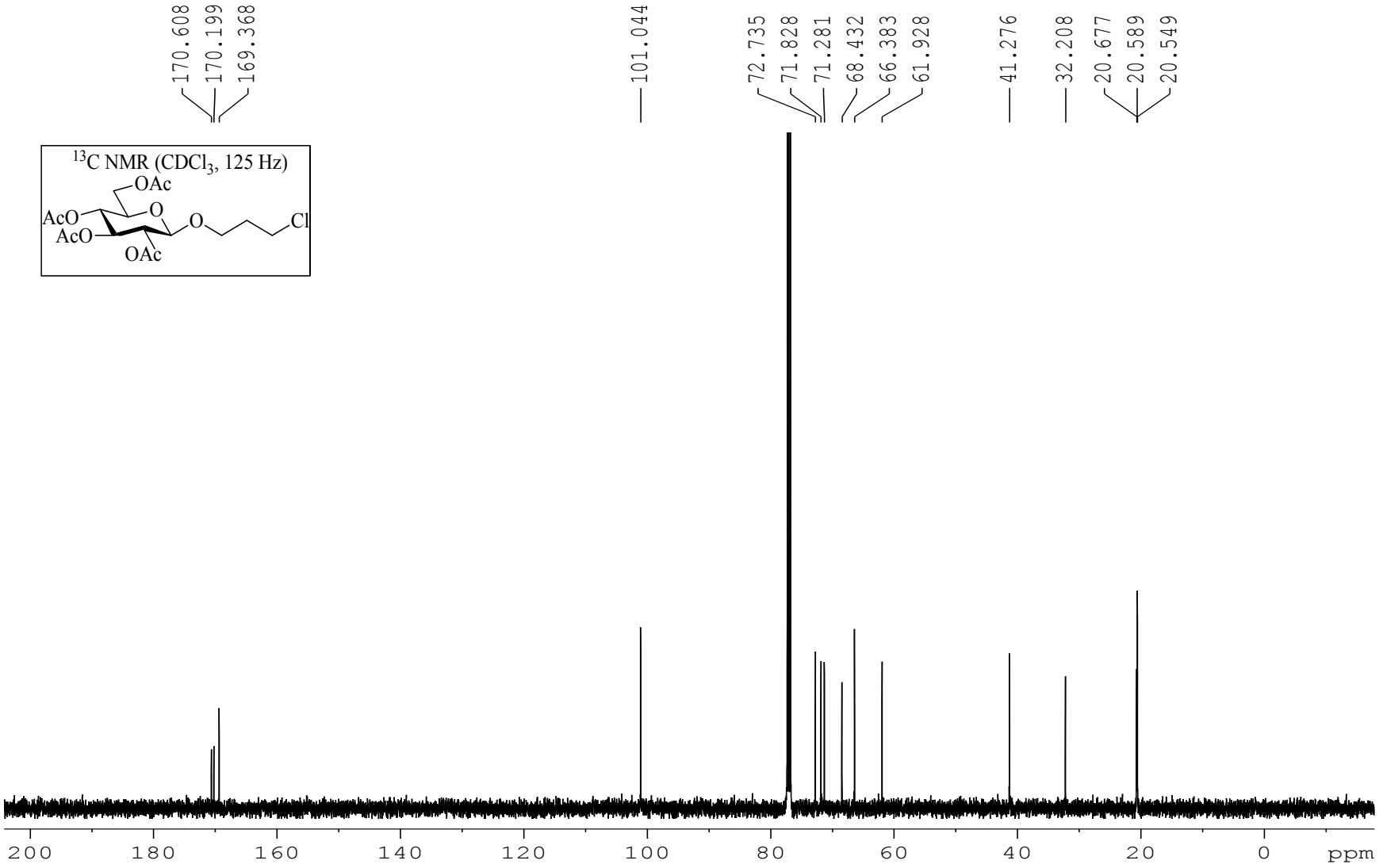
3.01
2.67
0.97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169.368
101.044
72.735
71.828
71.281
68.432
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61.928
41.276
32.208
20.677
20.589
20.549
^{13}C NMR ($CDCl_3$, 125 Hz)
OAc
AcO
AcO
O
O
Cl
OAc
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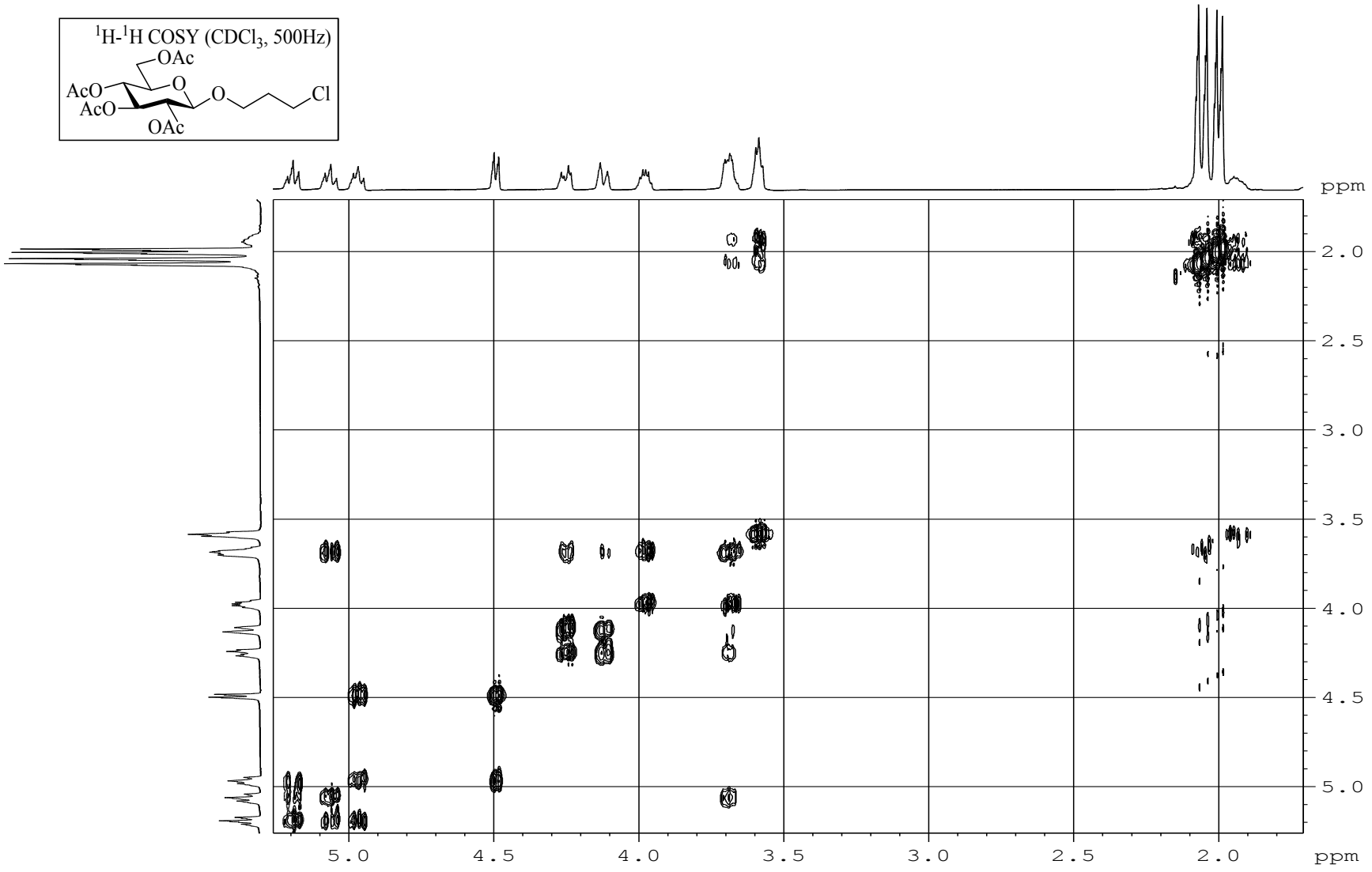
180
160
140
120
100
80
60
40
20
0
ppm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OAc
AcO
O
O
Cl
AcO
OAc
ppm
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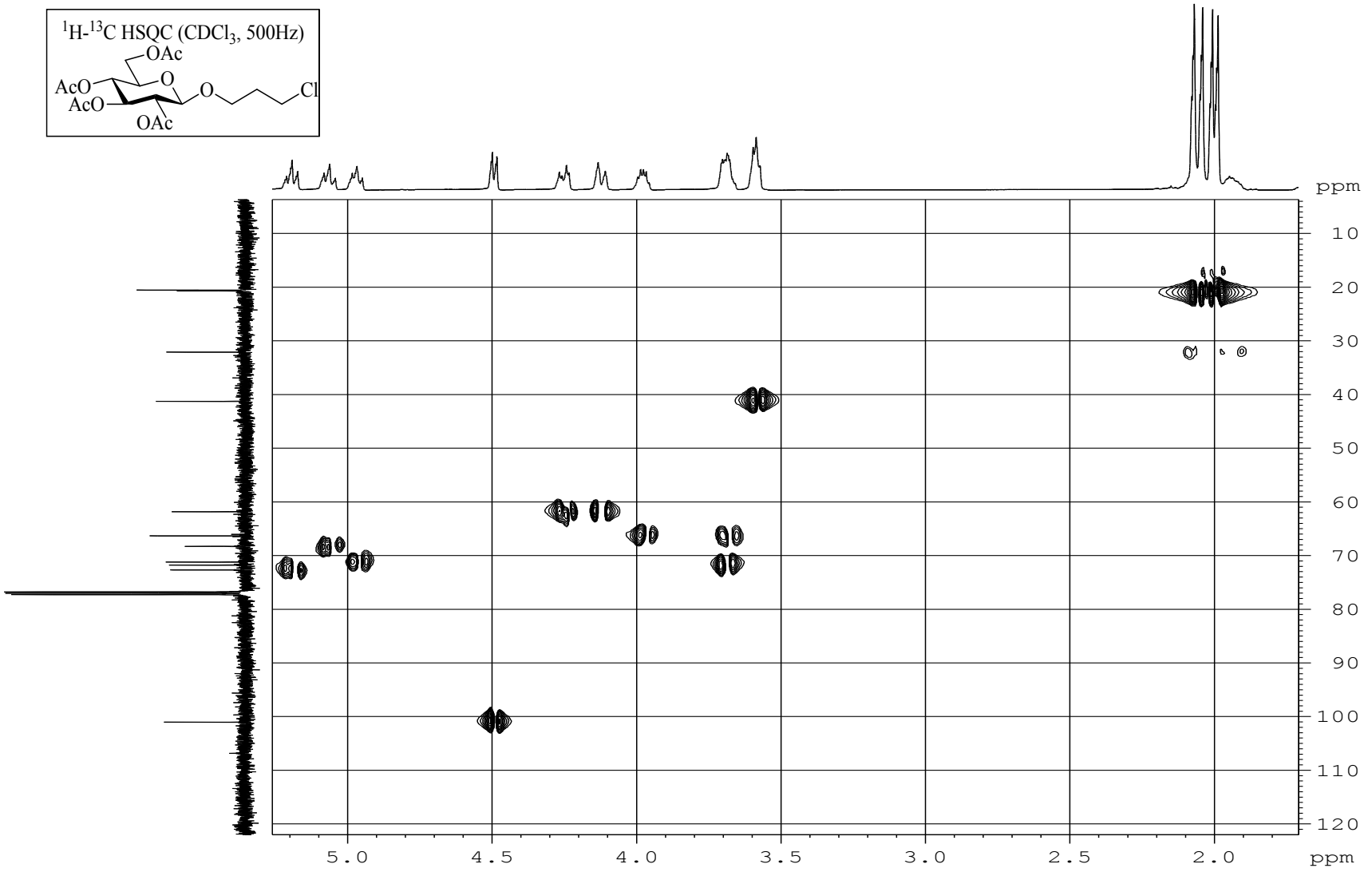
2.5
3.0
3.5
4.0
4.5
5.0
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm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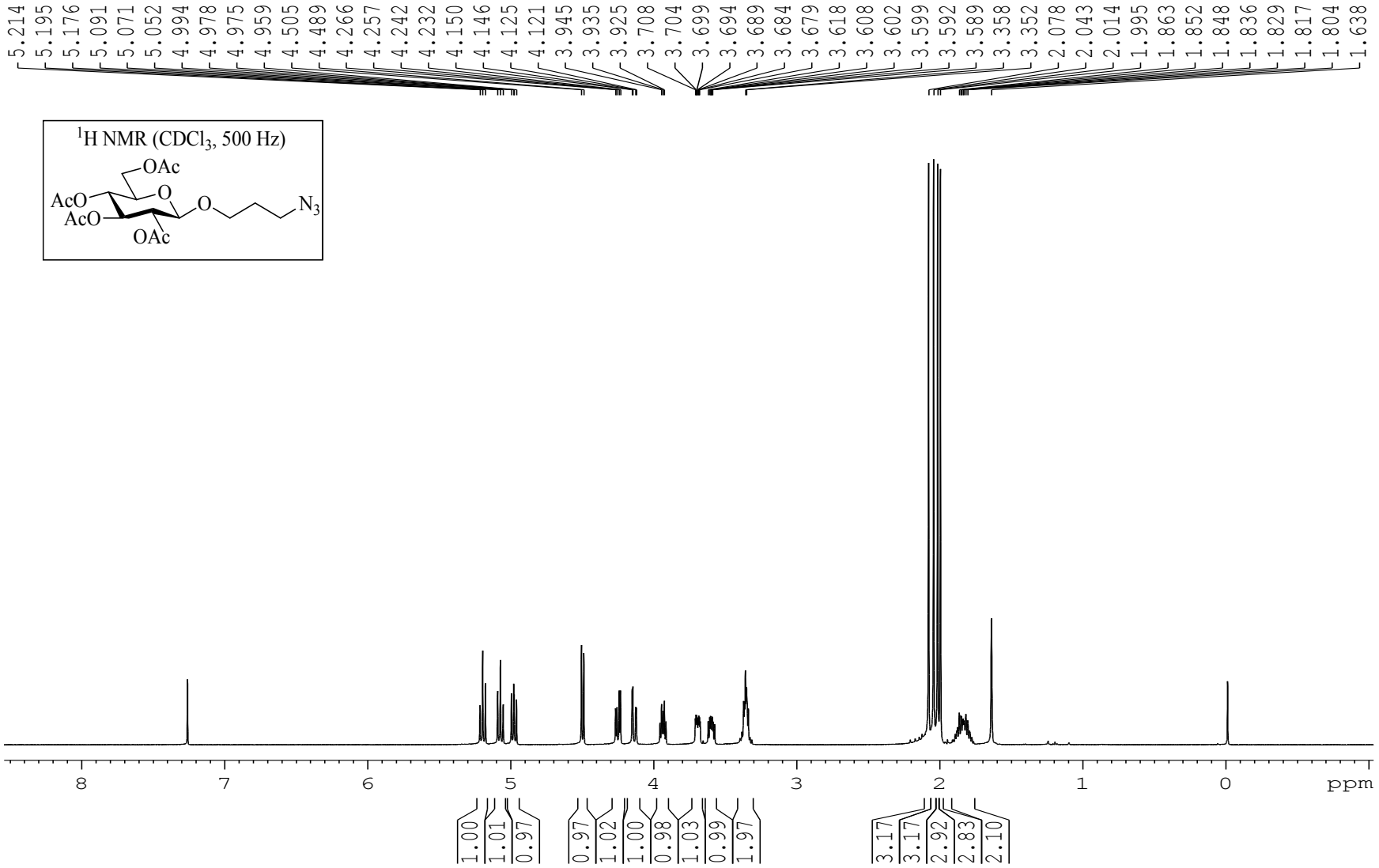
AcO
O
O
Cl
AcO
OAc
ppm
10
20
30
40
50
60
70
80
90
100
110
120
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm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5.176
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5.071
5.052
4.994
4.978
4.975
4.959
4.505
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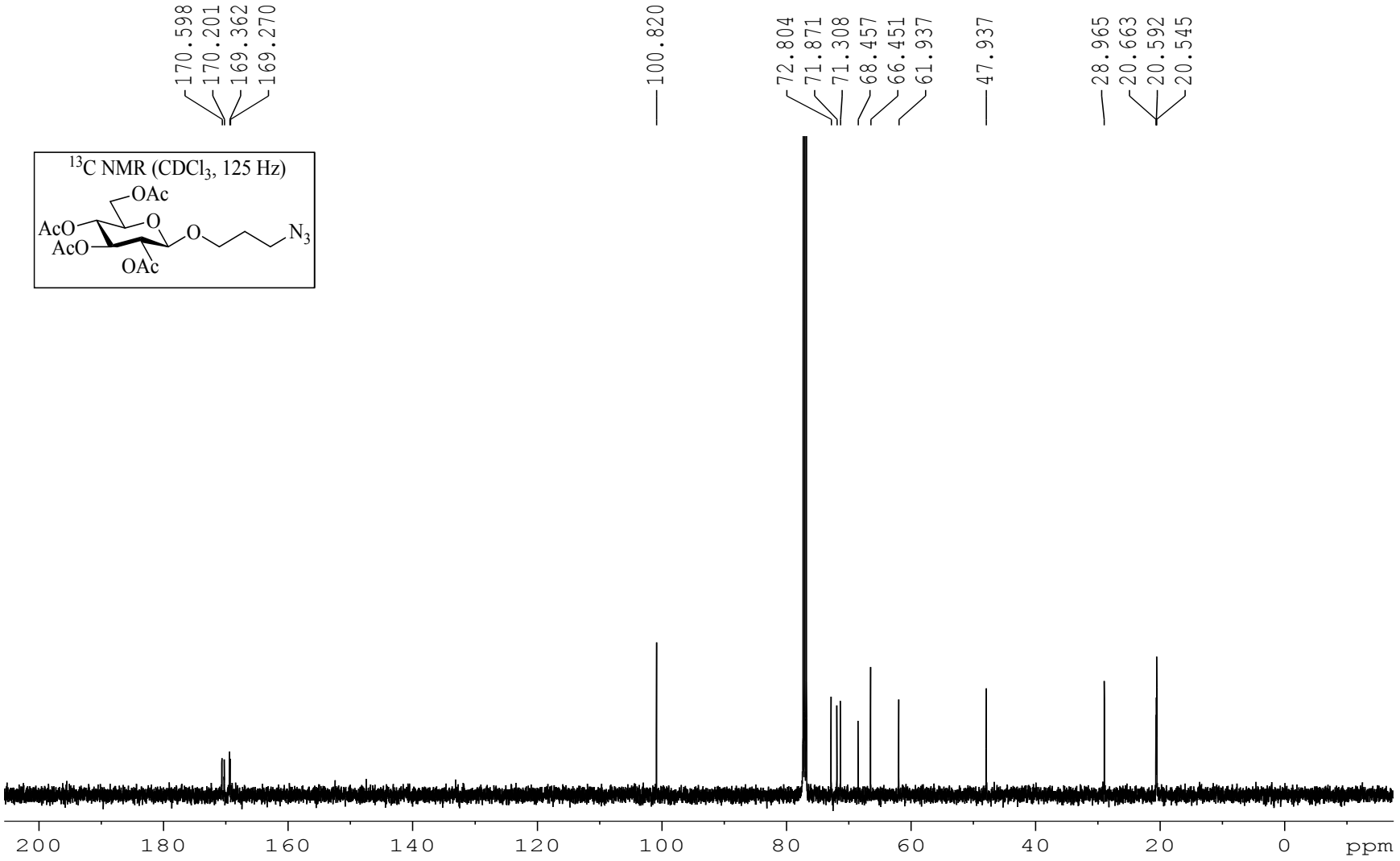
4.257
4.242
4.232
4.150
4.146
4.125
4.121
3.945
3.935
3.925
3.708
3.704
3.699
3.694
3.689
3.684
3.679
3.618
3.608
3.602
3.599
3.592
3.589
3.358
3.352
2.078
2.043
2.014
1.995
1.863
1.852
1.848
1.836
1.829
1.817
1.804
1.638
1H NMR (CDCl3, 500 Hz)
OAc
AcO
O
O
N3
AcO
OAc
8
7
6
5
4
3
2
1
0
ppm
1.00
1.01
0.97
0.97
1.02
1.00
0.98
1.03
0.99
1.97
3.17
3.17
2.92
2.83
2.10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28.965
20.663
20.592
20.545
13C NMR (CDCl3, 125 Hz)
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AcO
AcO
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O
N3
OAc
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160
140
120
100
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60
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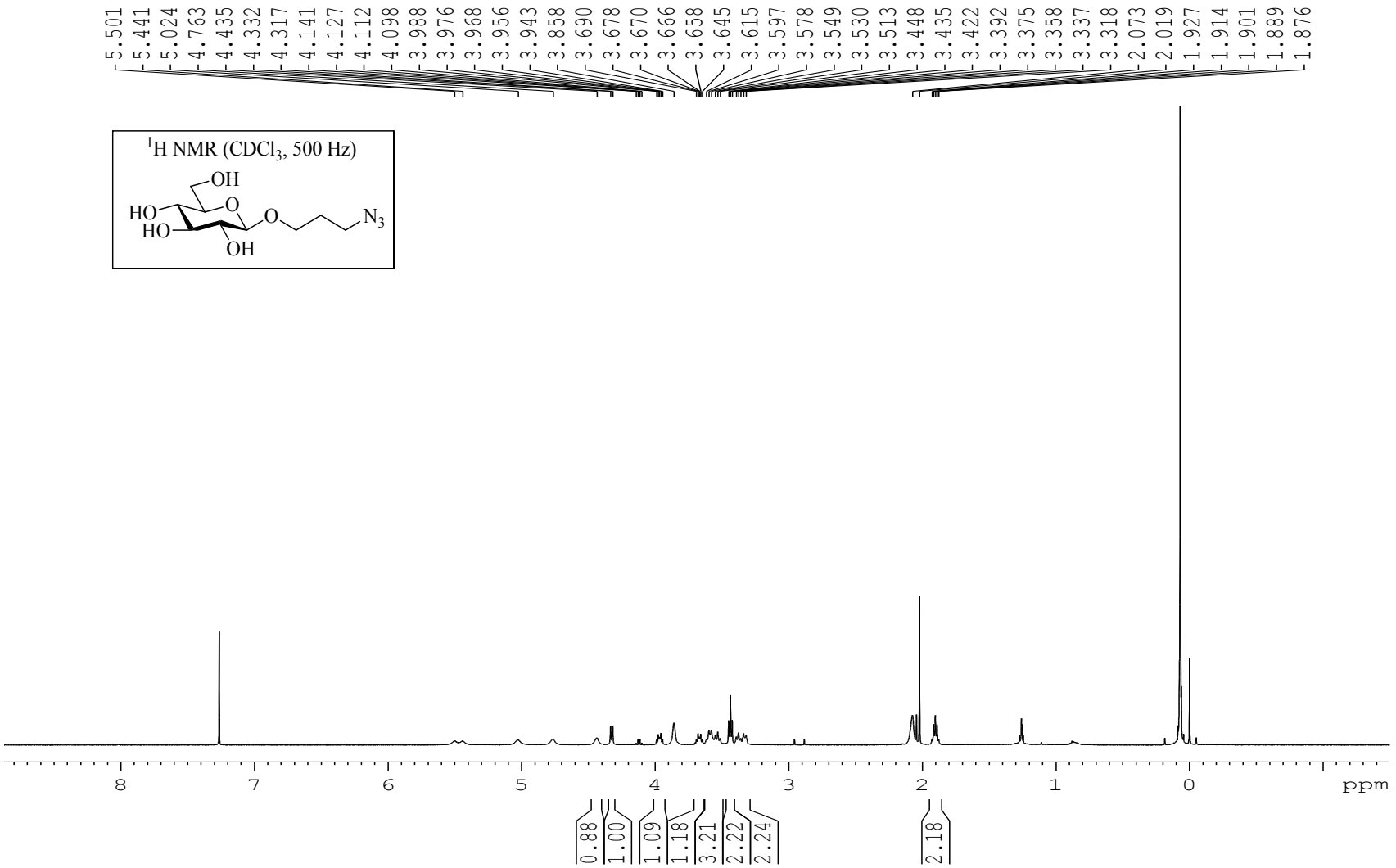
20
0
ppm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4.112
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3.968
3.956
3.943
3.858
3.690
3.678
3.670
3.666
3.658
3.645
3.615
3.597
3.578
3.549
3.530
3.513
3.448
3.435
3.422
3.392
3.375
3.358
3.337
3.318
2.073
2.019
1.927
1.914
1.901
1.889
1.876
1H NMR (CDCl3, 500 Hz)
OH
HO
O
O
N3
HO
OH
8
7
6
5
4
3
2
1
0
ppm
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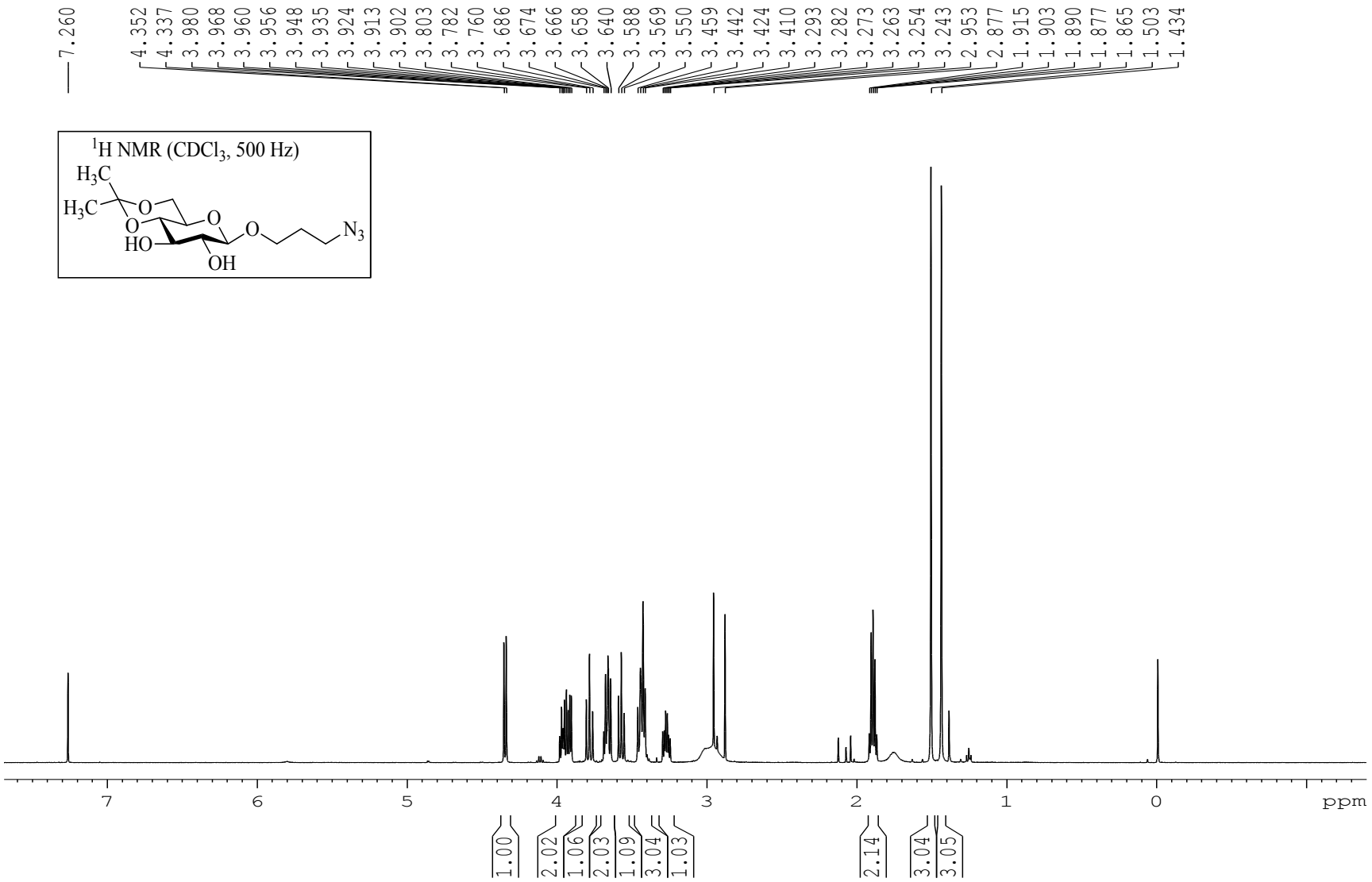
1.00
1.09
1.18
3.21
2.22
2.24
2.18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3.924
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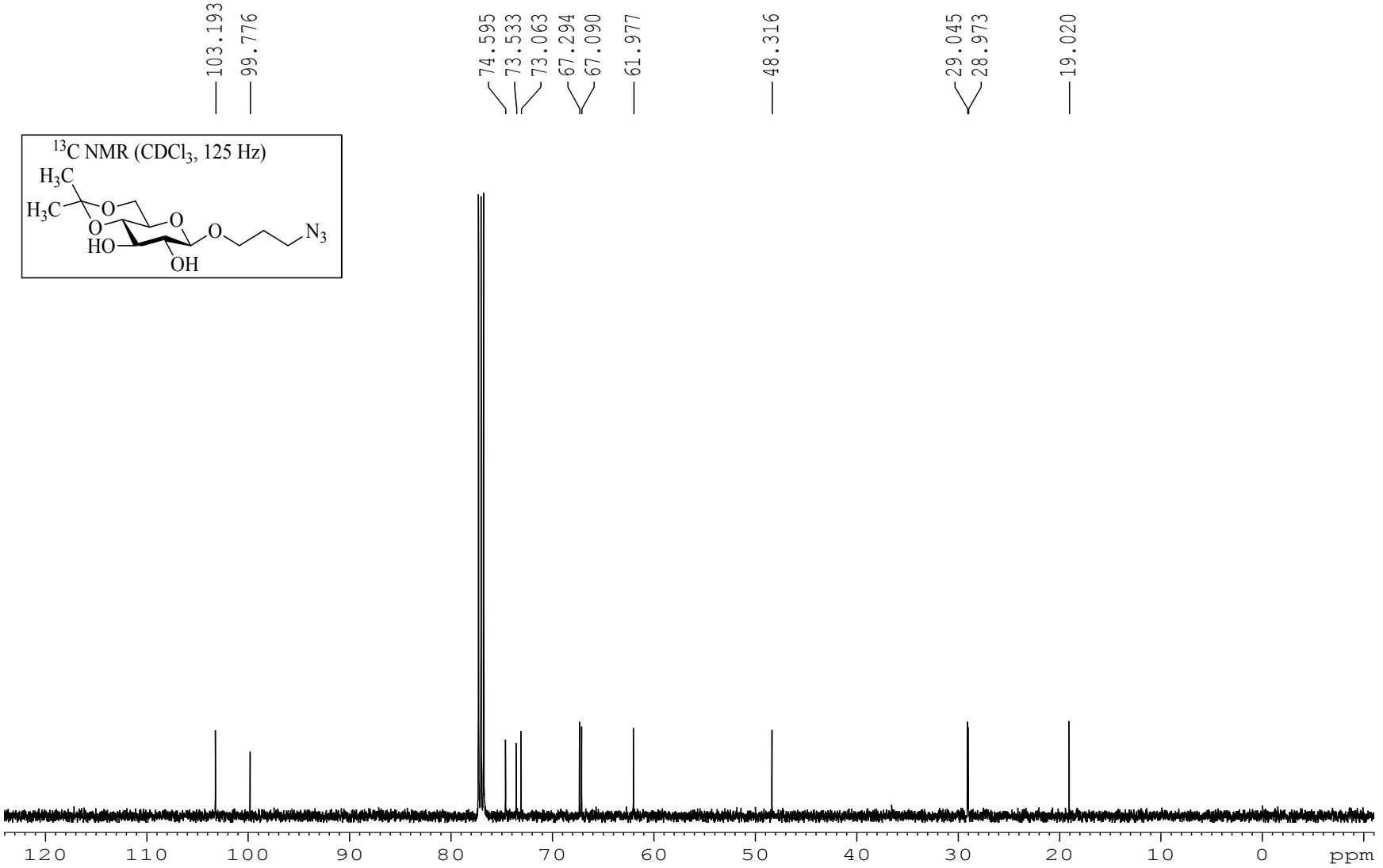
3.803
3.782
3.760
3.686
3.674
3.666
3.658
3.640
3.588
3.569
3.550
3.459
3.442
3.424
3.410
3.293
3.282
3.273
3.263
3.254
3.243
2.953
2.877
1.915
1.903
1.890
1.877
1.865
1.503
1.434
^{1}H NMR ($CDCl_3$, 500 Hz)
H_3C
H_3C
O
O
O
HO
OH
O
N_3
7
6
5
4
3
2
1
0
ppm
1.00
2.02
1.06
2.03
1.09
3.04
1.03
2.14
3.04
3.05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99.776
74.595
73.533
73.063
67.294
67.090
61.977
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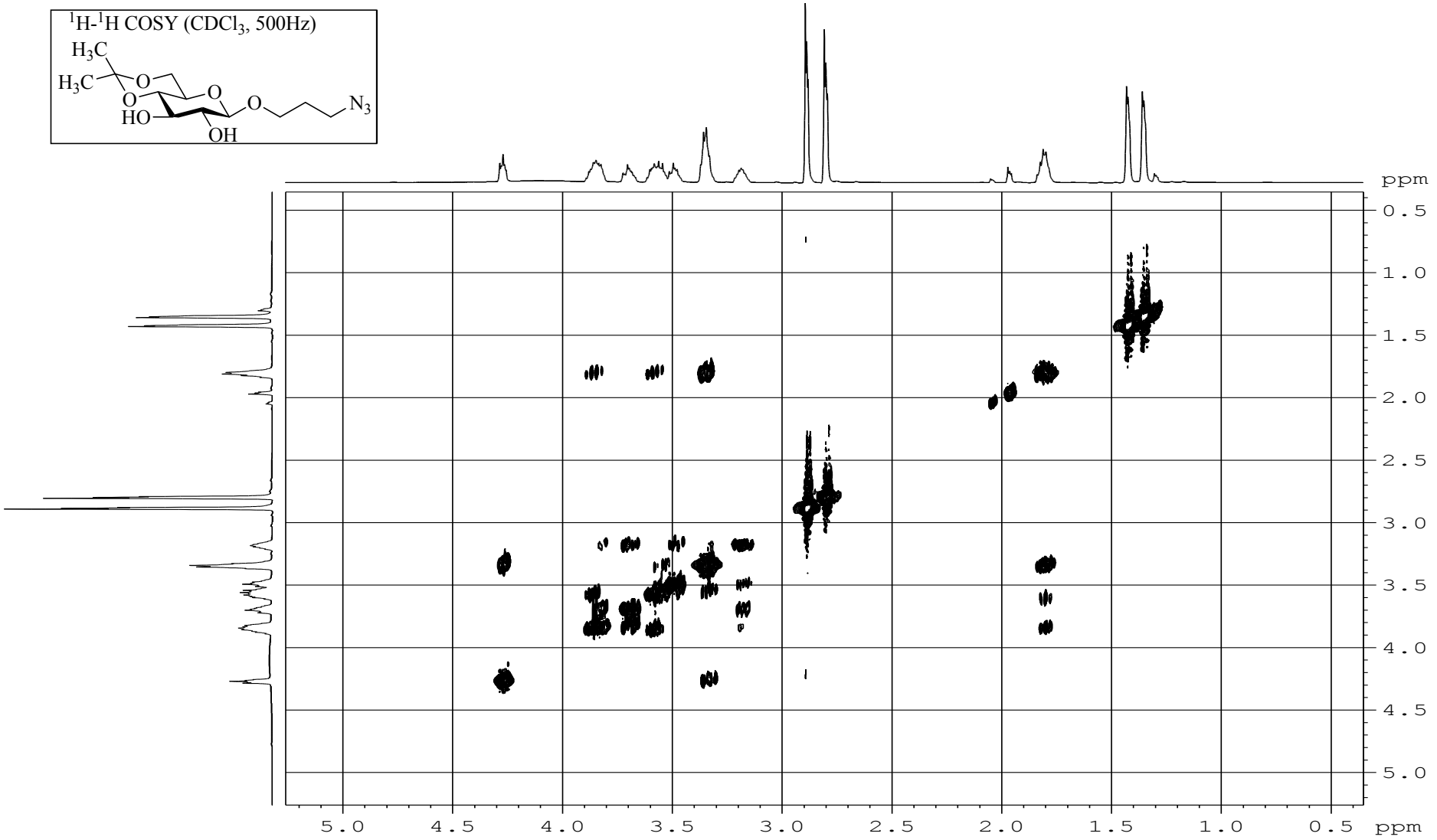
29.045
28.973
19.020
13C NMR (CDCl3, 125 Hz)
H3C
H3C
O
O
O
HO
OH
O
N3
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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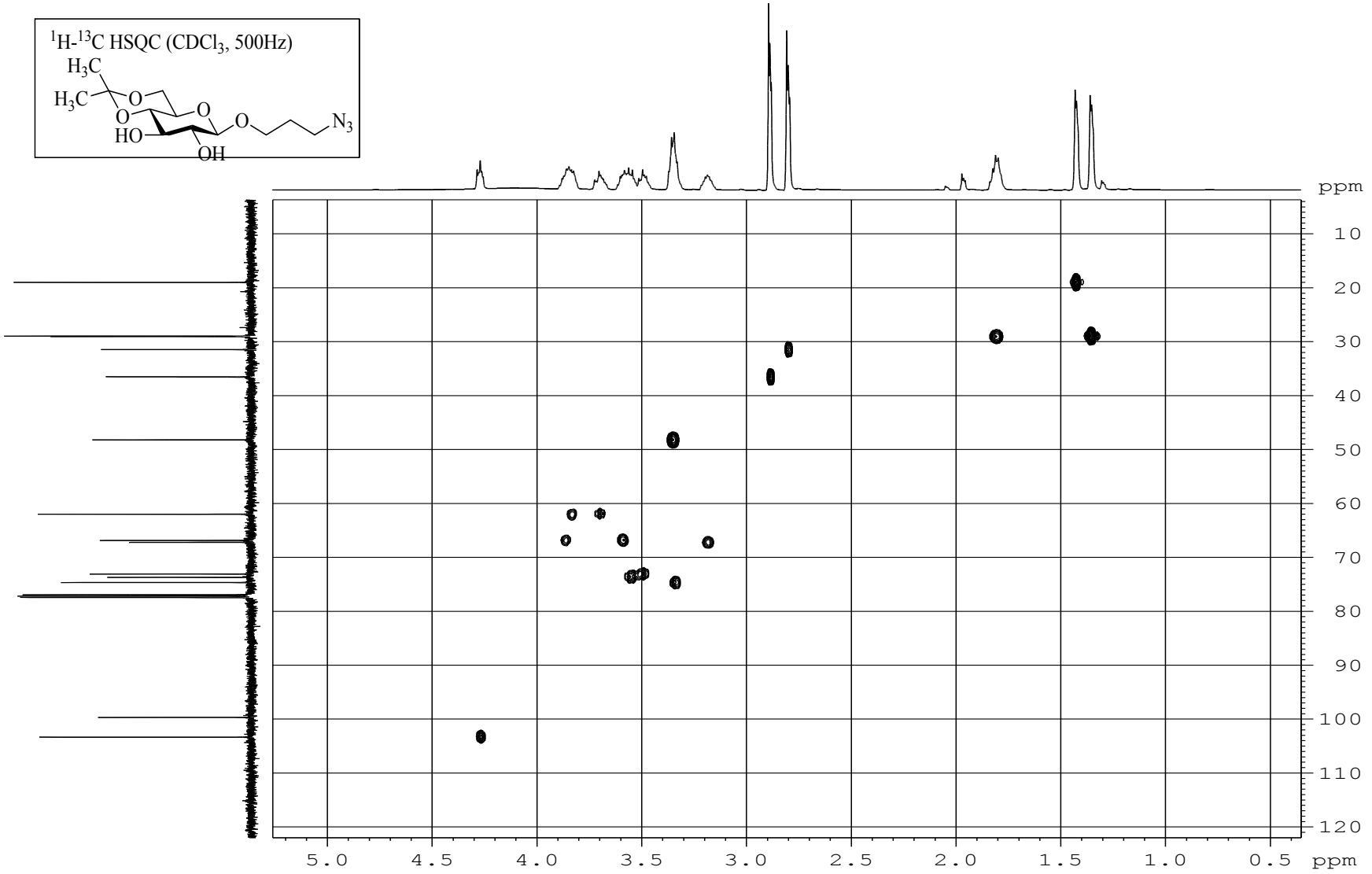
H_3C
H_3C
O
O
O
O
HO
OH
O
N_3
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm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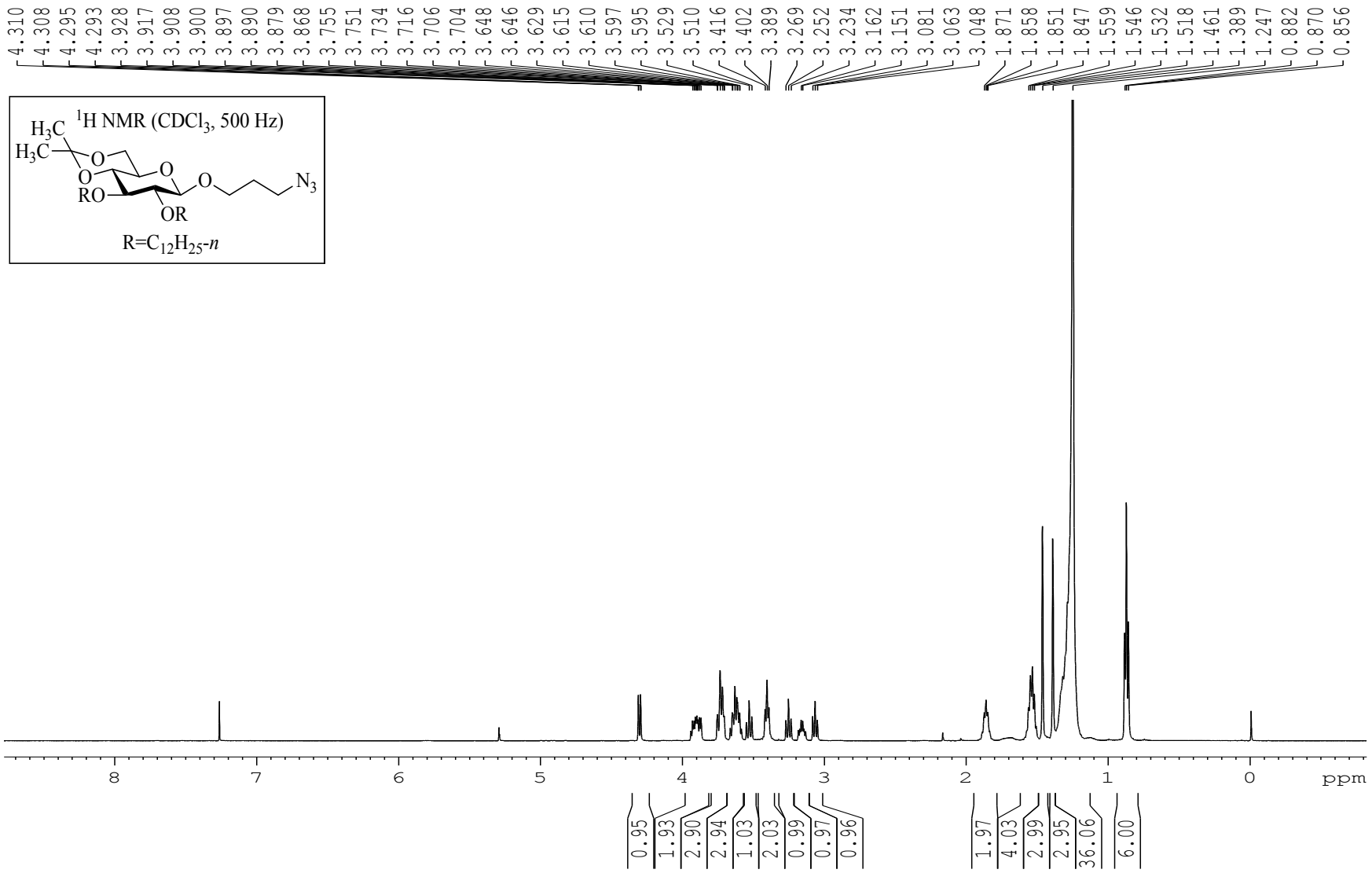
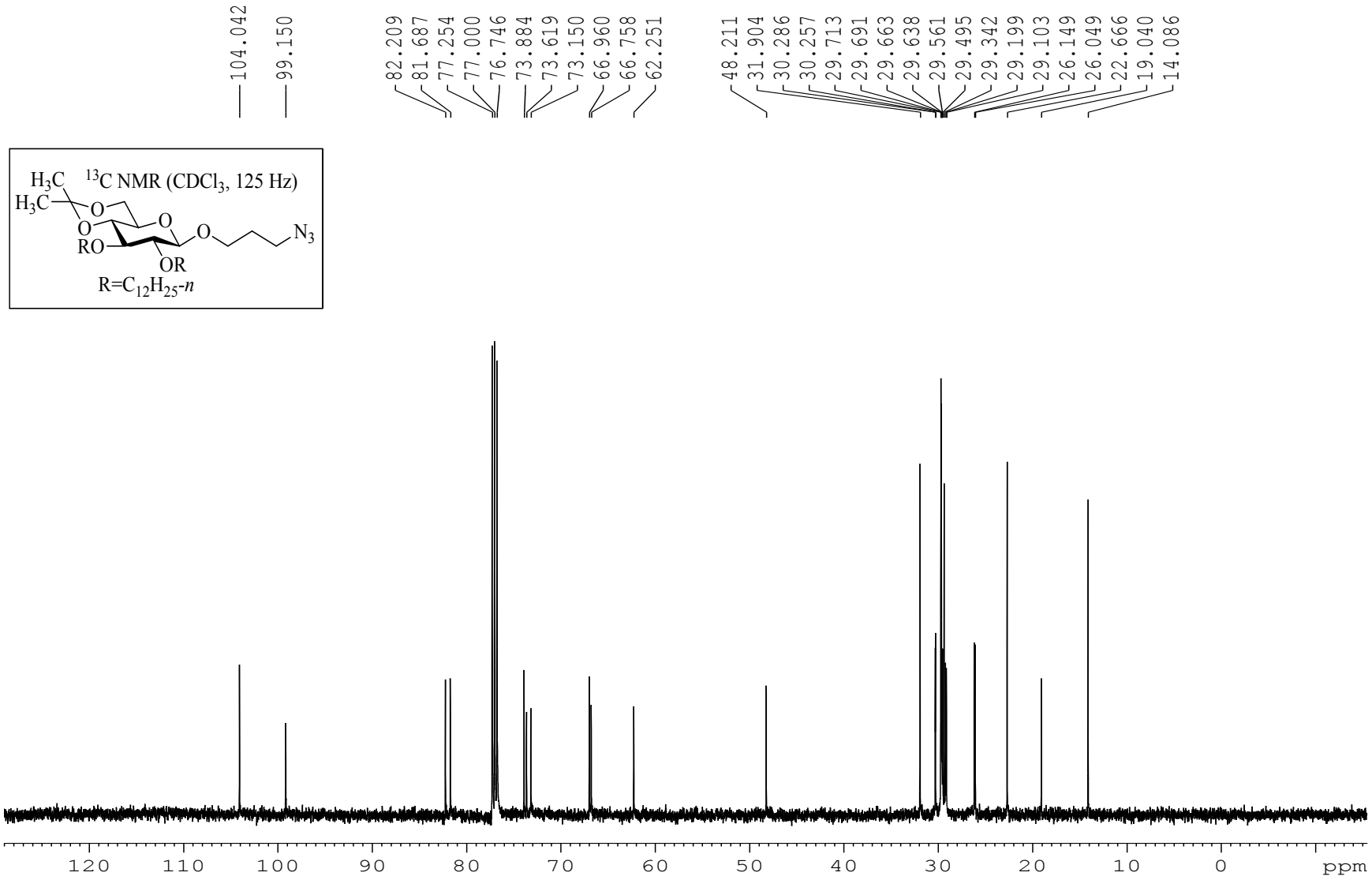
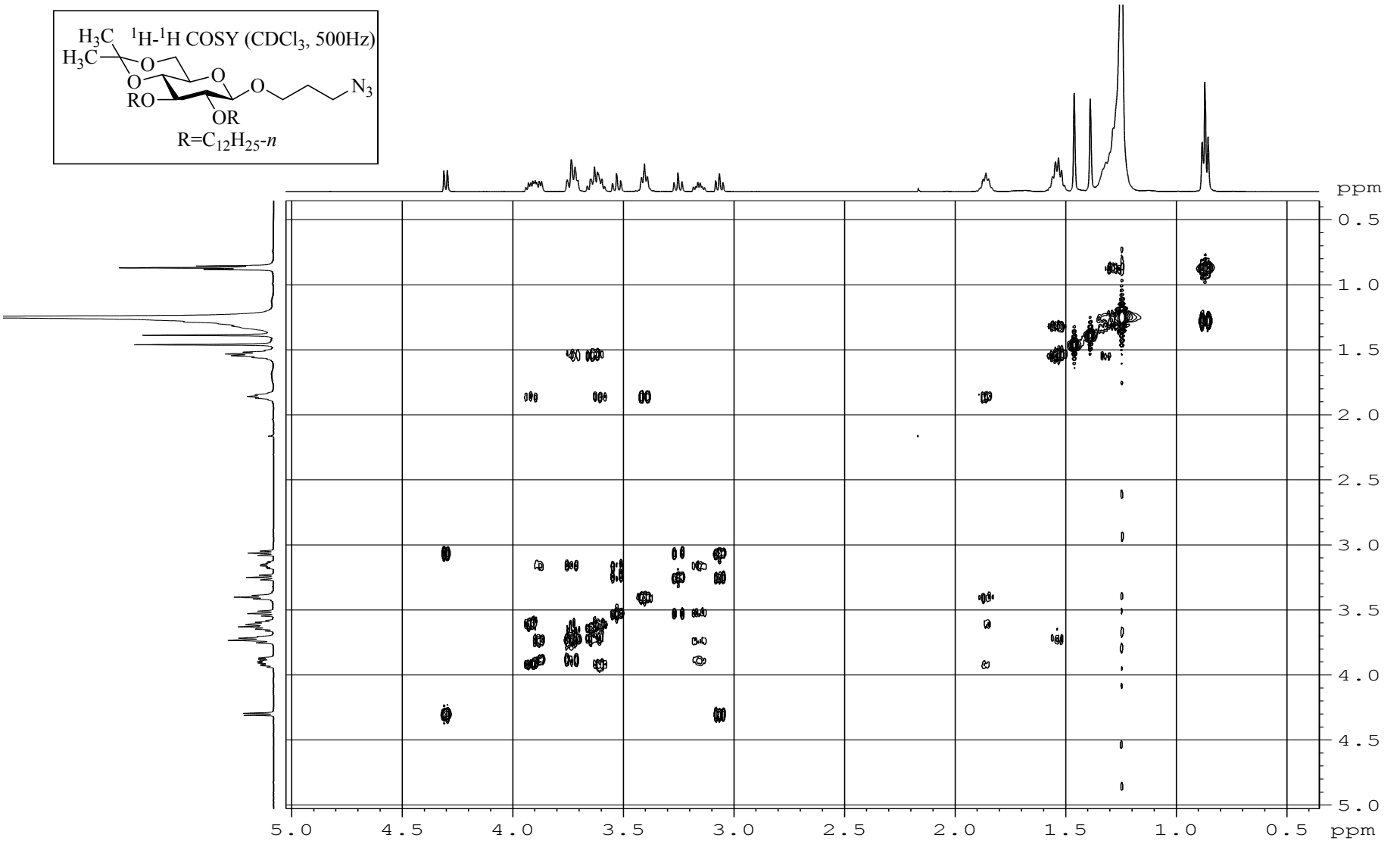
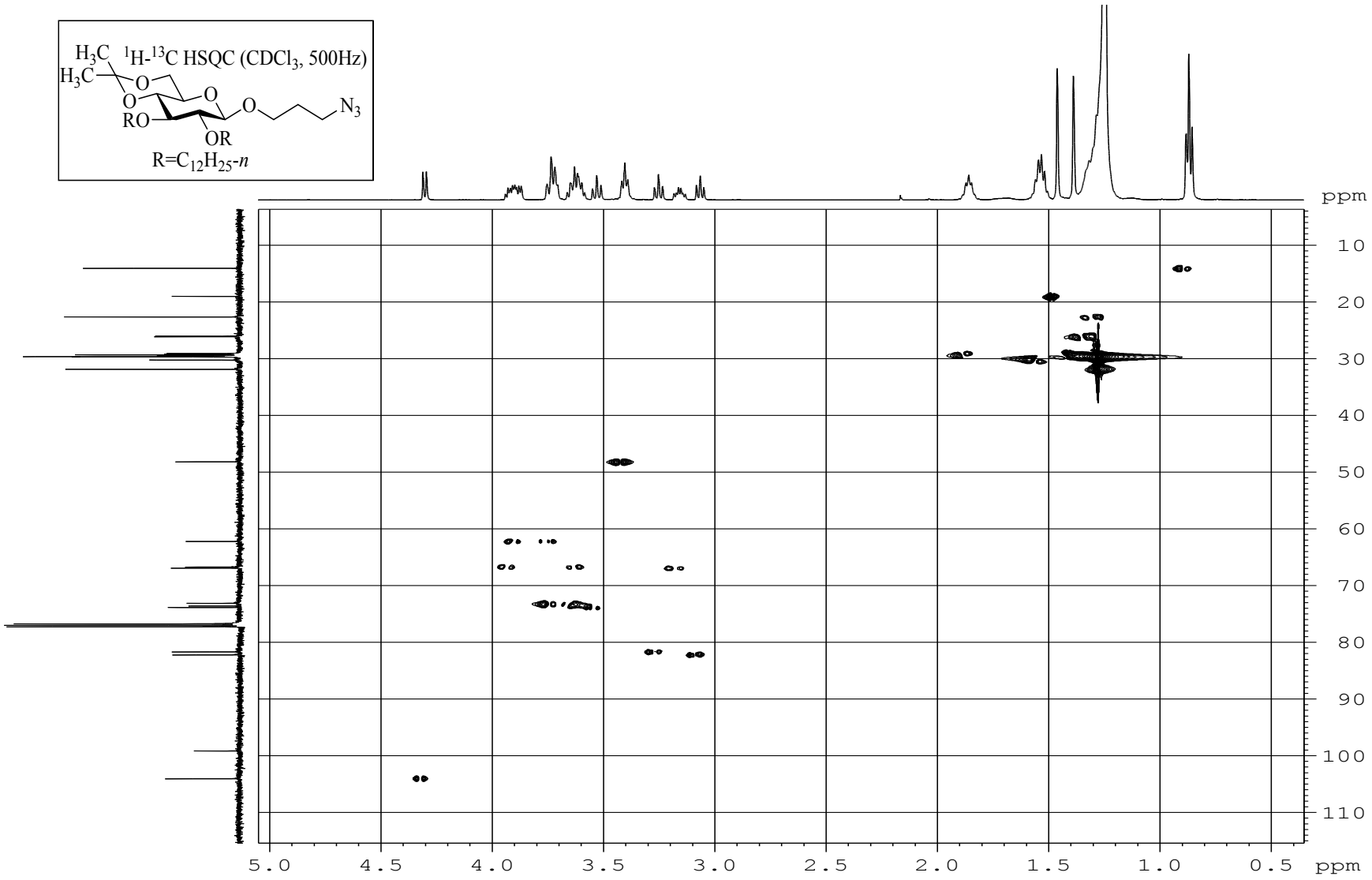
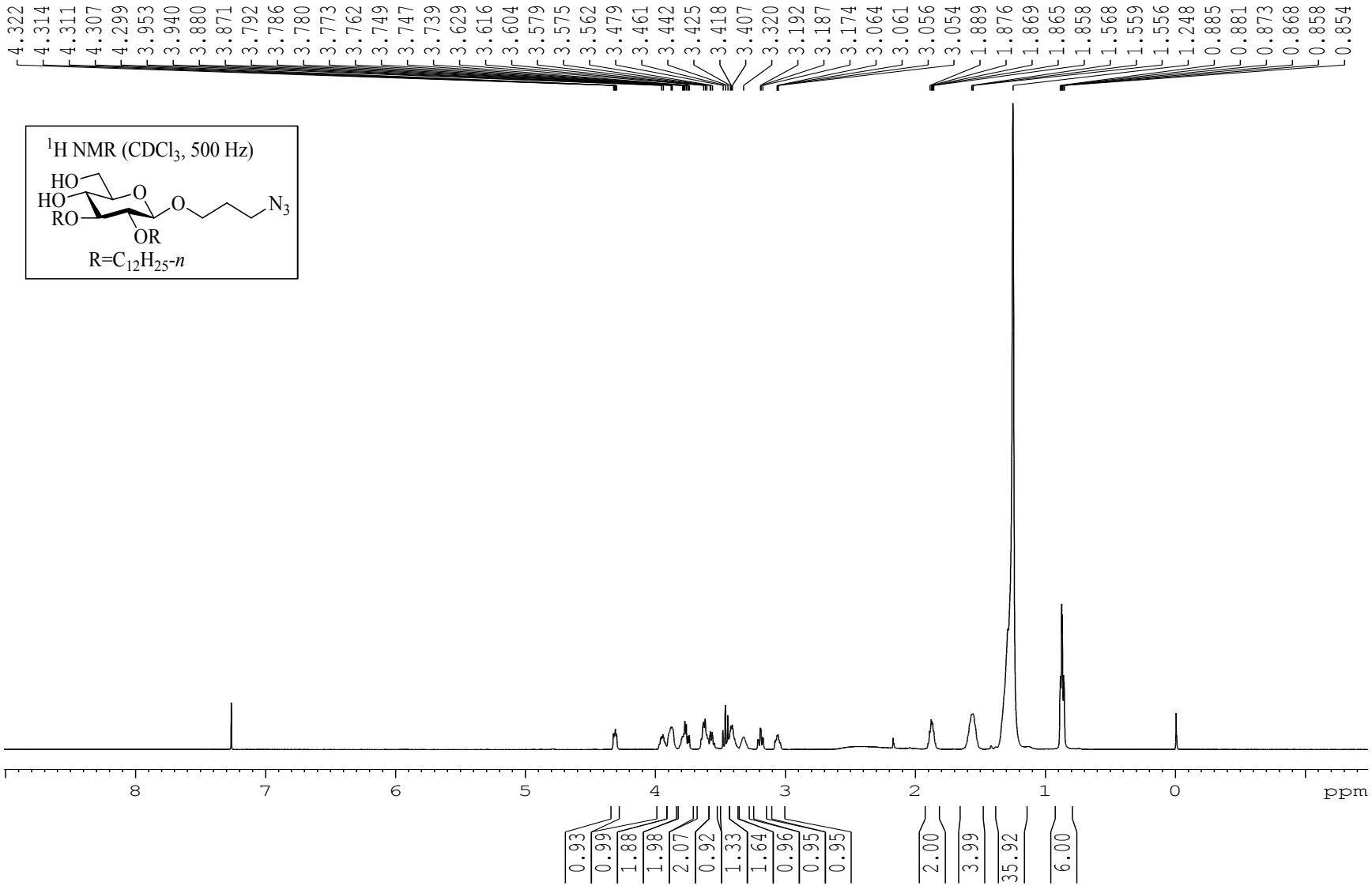
H3C
H3C
O
O
HO
OH
O
O
N3
ppm
10
20
30
40
50
60
70
80
90
100
110
120
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

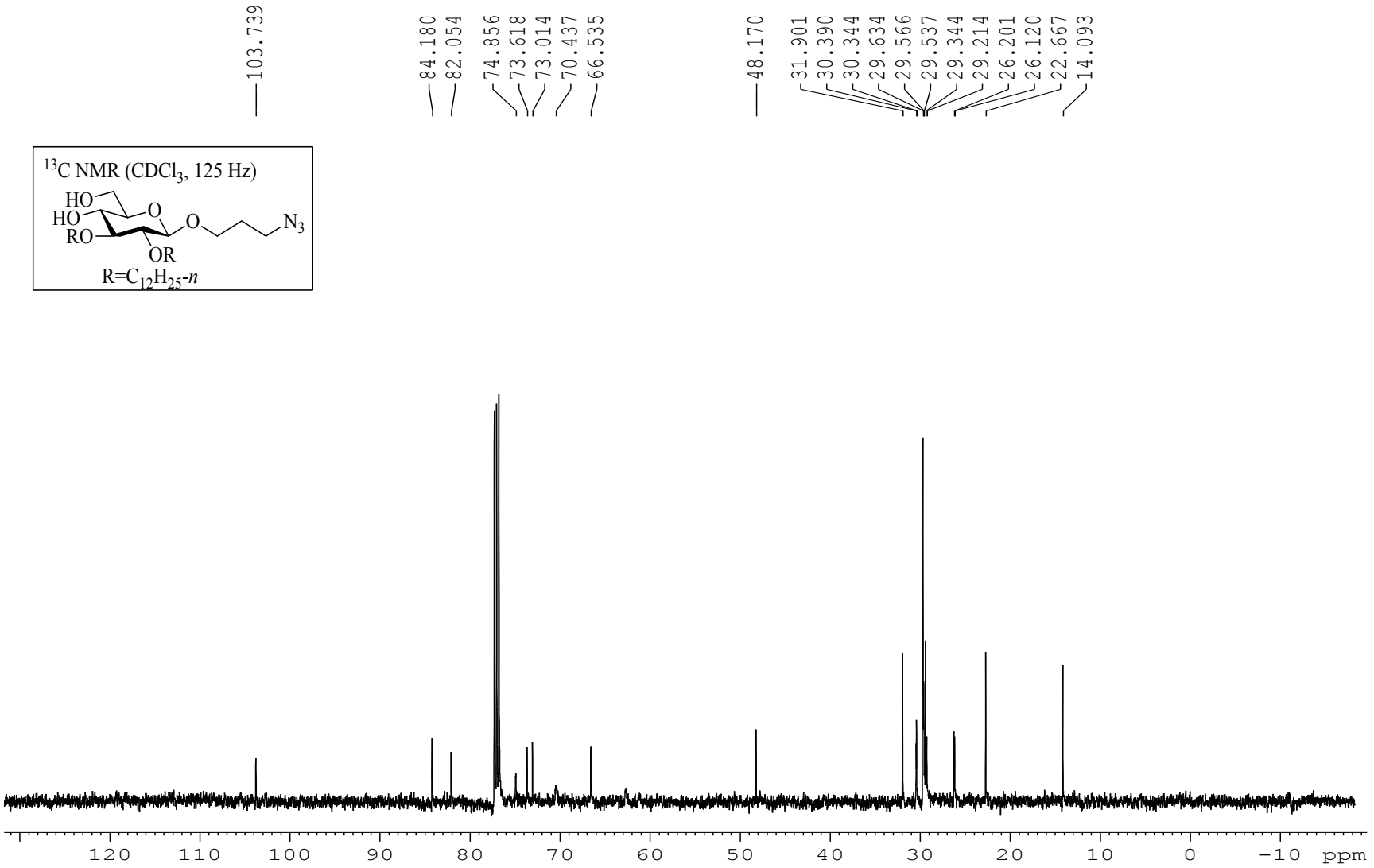

4.310
4.308
4.295
4.293
3.928
3.917
3.908
3.900
3.897
3.890
3.879
3.868
3.755
3.751
3.734
3.716
3.706
3.704
3.648
3.646
3.629
3.615
3.610
3.597
3.595
3.529
3.510
3.416
3.402
3.389
3.269
3.252
3.234
3.162
3.151
3.081
3.063
3.048
1.871
1.858
1.851
1.847
1.559
1.546
1.532
1.518
1.461
1.389
1.247
0.882
0.870
0.856
1H NMR (CDCl3, 500 Hz)
H3C
H3C
O
O
RO
O
O
O
N3
OR
R=C12H25-n
8
7
6
5
4
3
2
1
0
ppm
0.95
1.93
2.90
2.94
1.03
2.03
0.99
0.97
0.96
1.97
4.03
2.99
2.95
36.06
6.00

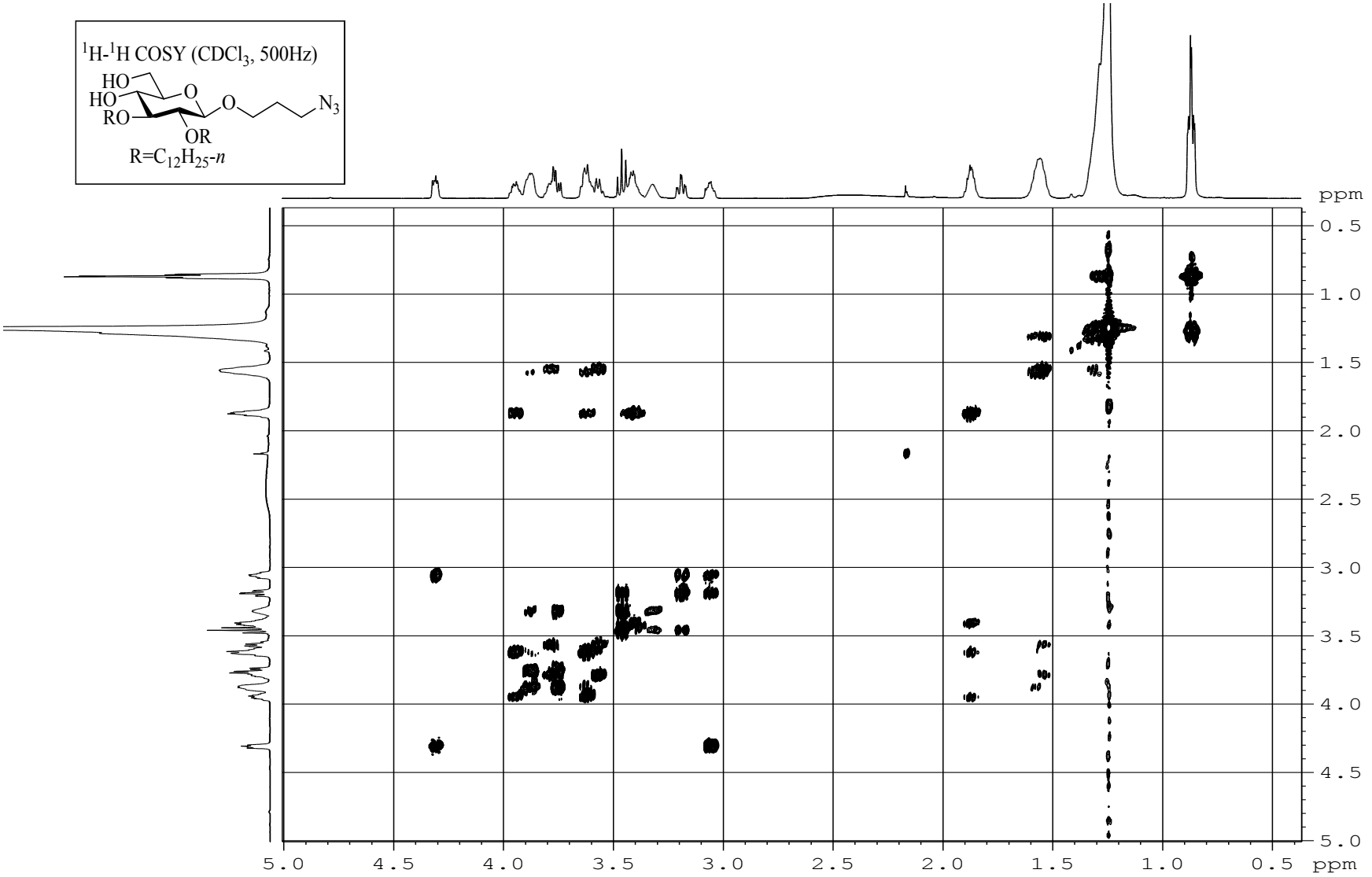

104.042
99.150
82.209
81.687
77.254
77.000
76.746
73.884
73.619
73.150
66.960
66.758
62.251
48.211
31.904
30.286
30.257
29.713
29.691
29.663
29.638
29.561
29.495
29.342
29.199
29.103
26.149
26.049
22.666
19.040
14.086
H3C
H3C
O
O
O
O
RO
OR
O
N3
R=C12H25-n
13C NMR (CDCl3, 125 Hz)
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

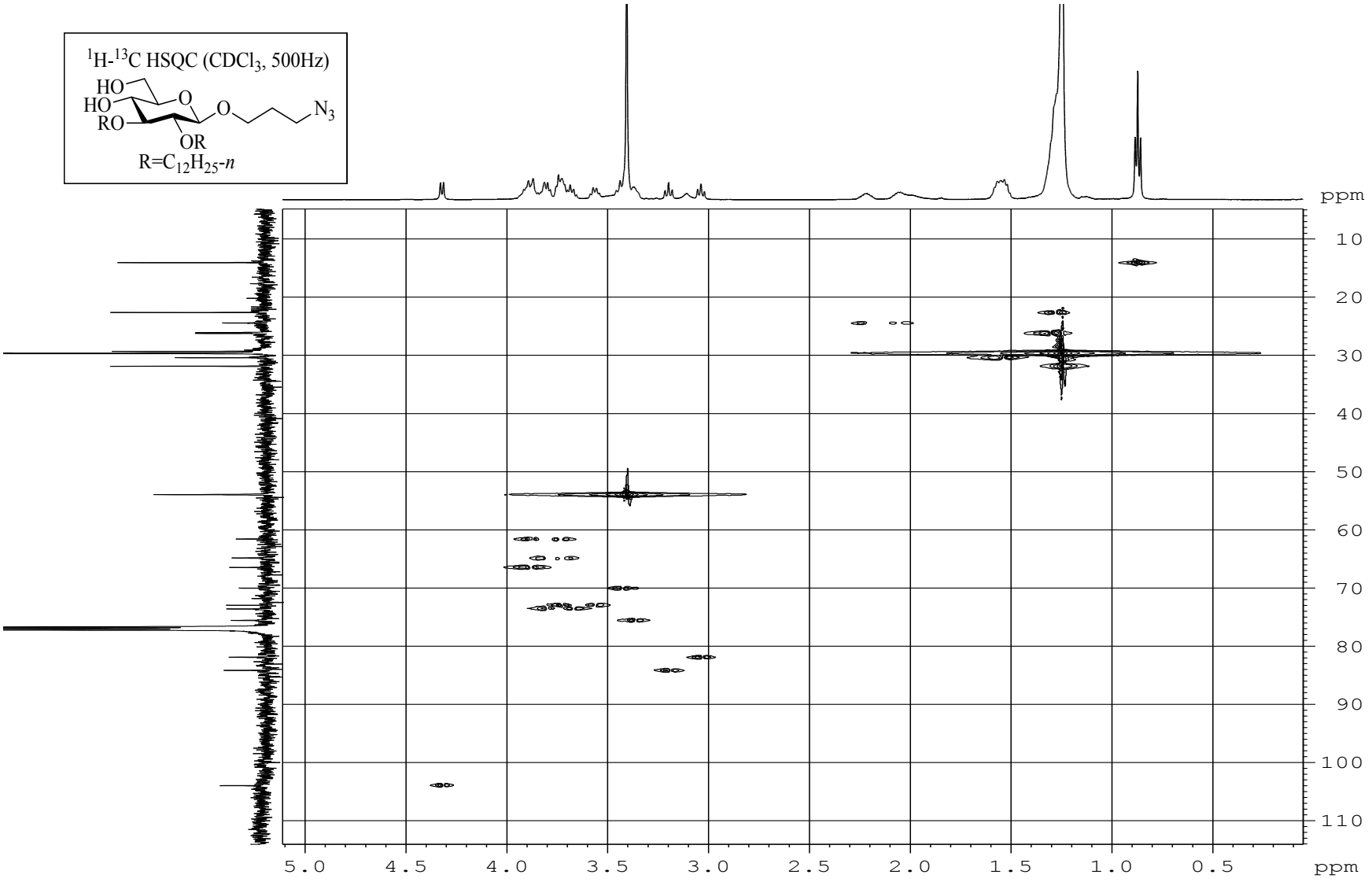

^{1}H-^{1}H COSY (CDCl$_3$, 500Hz)
R=C$_{12}$H$_{25}$-*n*
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

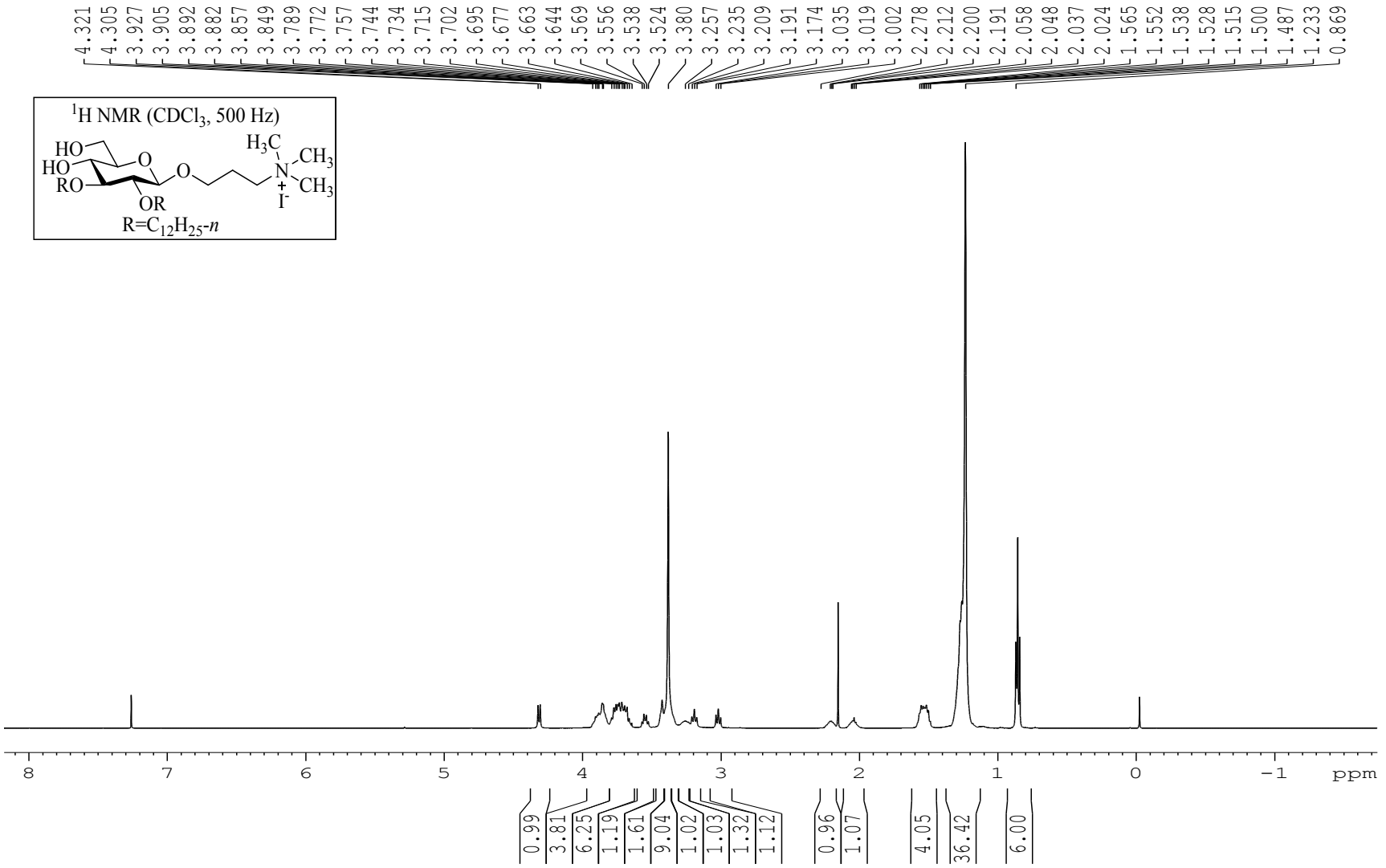

H3C
H3C
O
O
RO
O
O
O
N3
OR
R=C12H25-n
1H-13C HSQC (CDCl3, 500Hz)
ppm
10
20
30
40
50
60
70
80
90
100
110
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

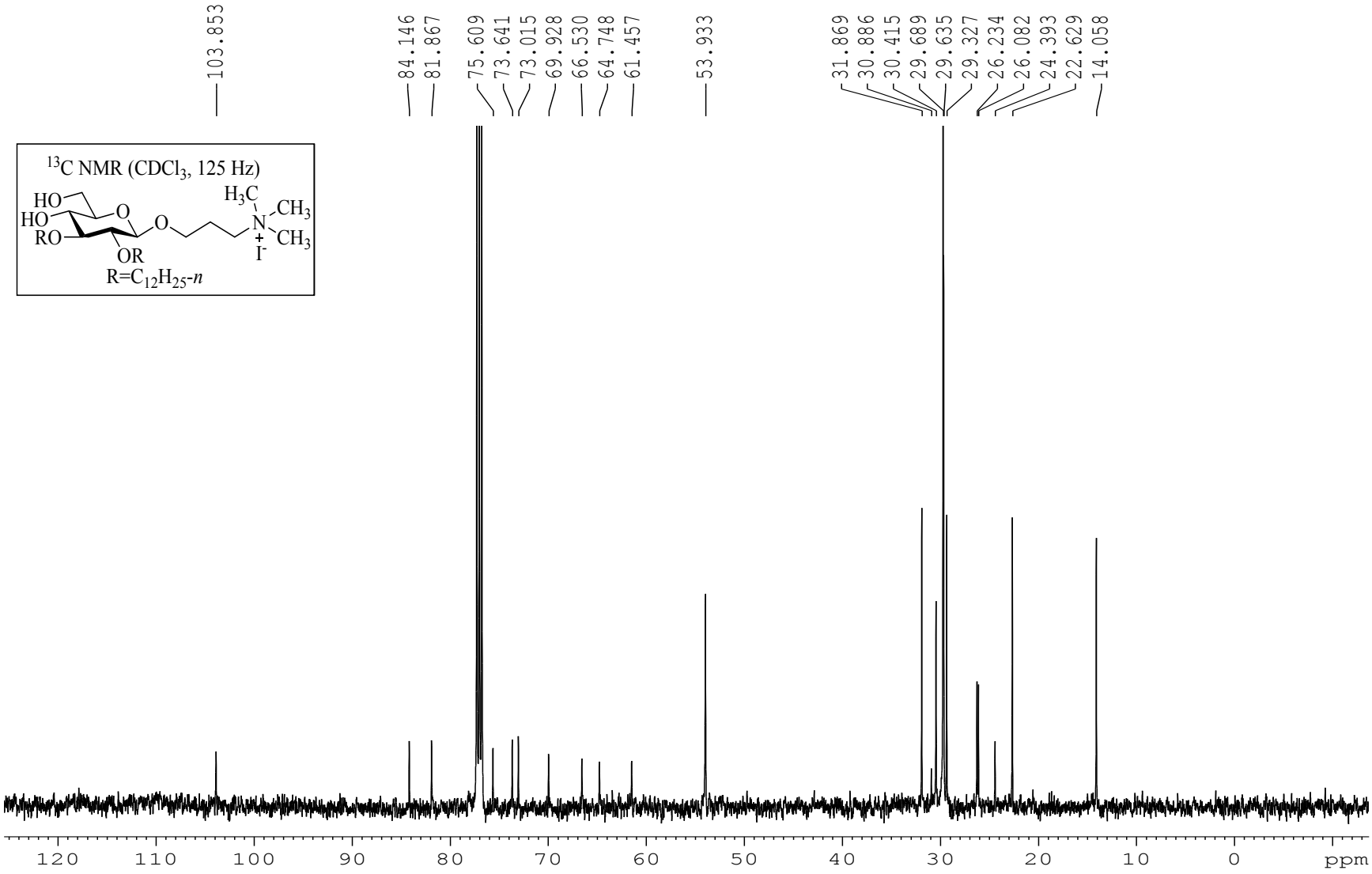

4.322
4.314
4.311
4.307
4.299
3.953
3.940
3.880
3.871
3.792
3.786
3.780
3.773
3.762
3.749
3.747
3.739
3.629
3.616
3.604
3.579
3.575
3.562
3.479
3.461
3.442
3.425
3.418
3.407
3.320
3.192
3.187
3.174
3.064
3.061
3.056
3.054
1.889
1.876
1.869
1.865
1.858
1.568
1.559
1.556
1.248
0.885
0.881
0.873
0.868
0.858
0.854
^{1}H NMR ($CDCl_3$, 500 Hz)
HO
HO
RO
O
O
N_3
OR
R=$C_{12}H_{25}$-*n*
8
7
6
5
4
3
2
1
0
ppm
0.93
0.99
1.88
1.98
2.07
0.92
1.33
1.64
0.96
0.95
0.95
2.00
3.99
35.92
6.00

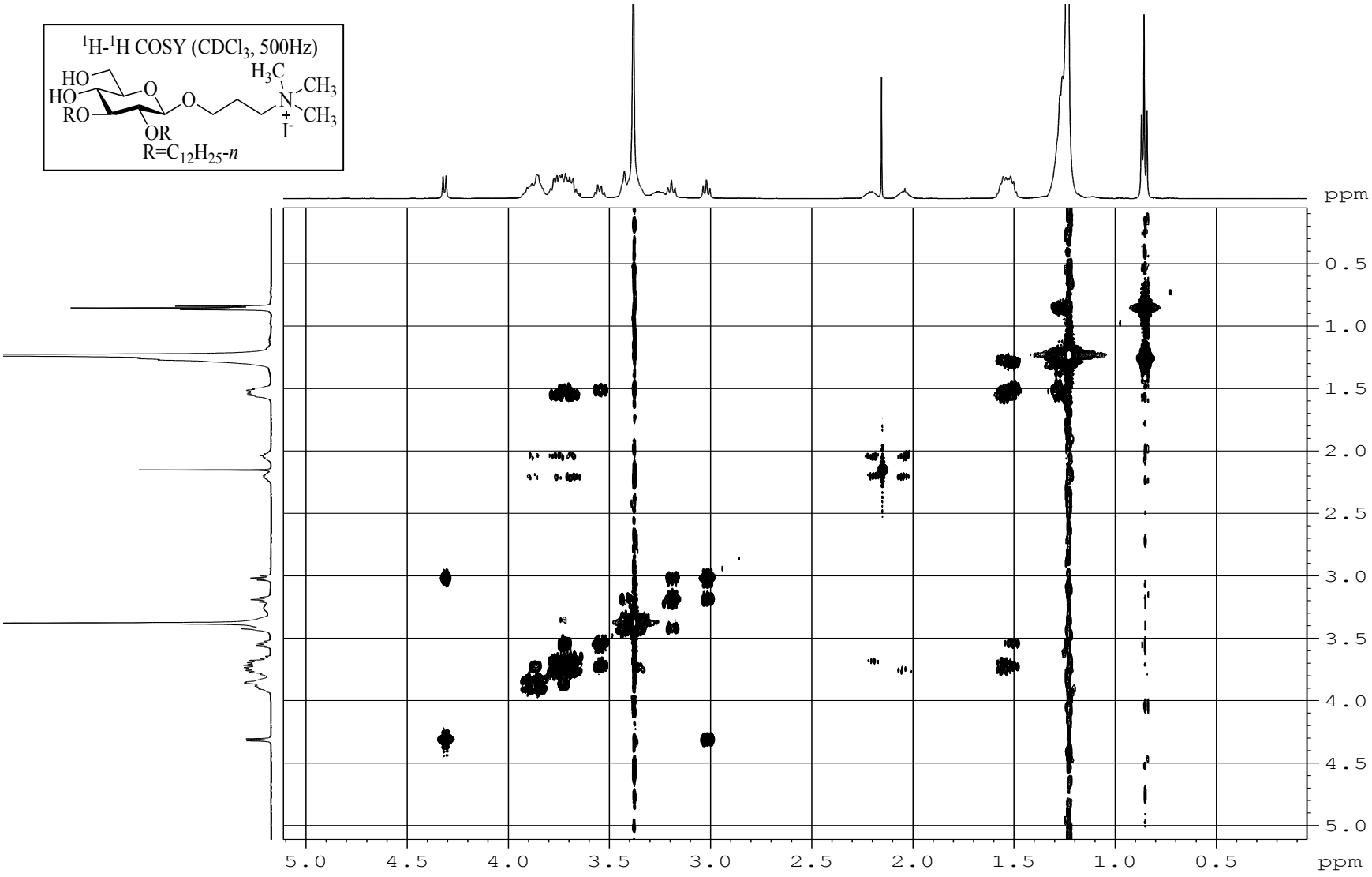

103.739
84.180
82.054
74.856
73.618
73.014
70.437
66.535
48.170
31.901
30.390
30.344
29.634
29.566
29.537
29.344
29.214
26.201
26.120
22.667
14.093
13C NMR (CDCl3, 125 Hz)
HO
HO
RO
O
O
N3
OR
R=C12H25-n
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
ppm

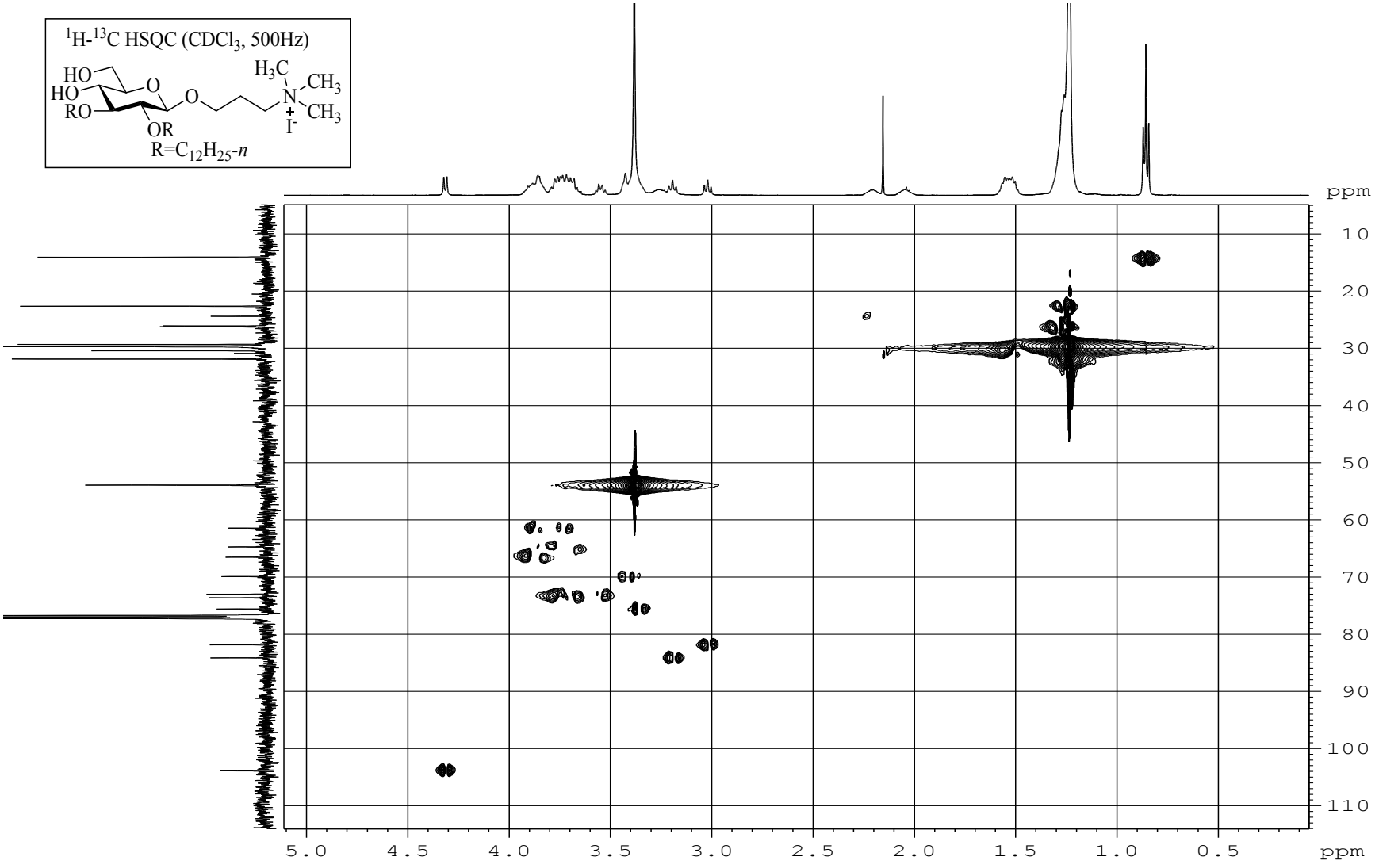

^{1}H-^{1}H COSY (CDCl$_3$, 500Hz)
HO
HO
RO
O
O
N$_3$
OR
R=C$_{12}$H$_{25}$-*n*
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

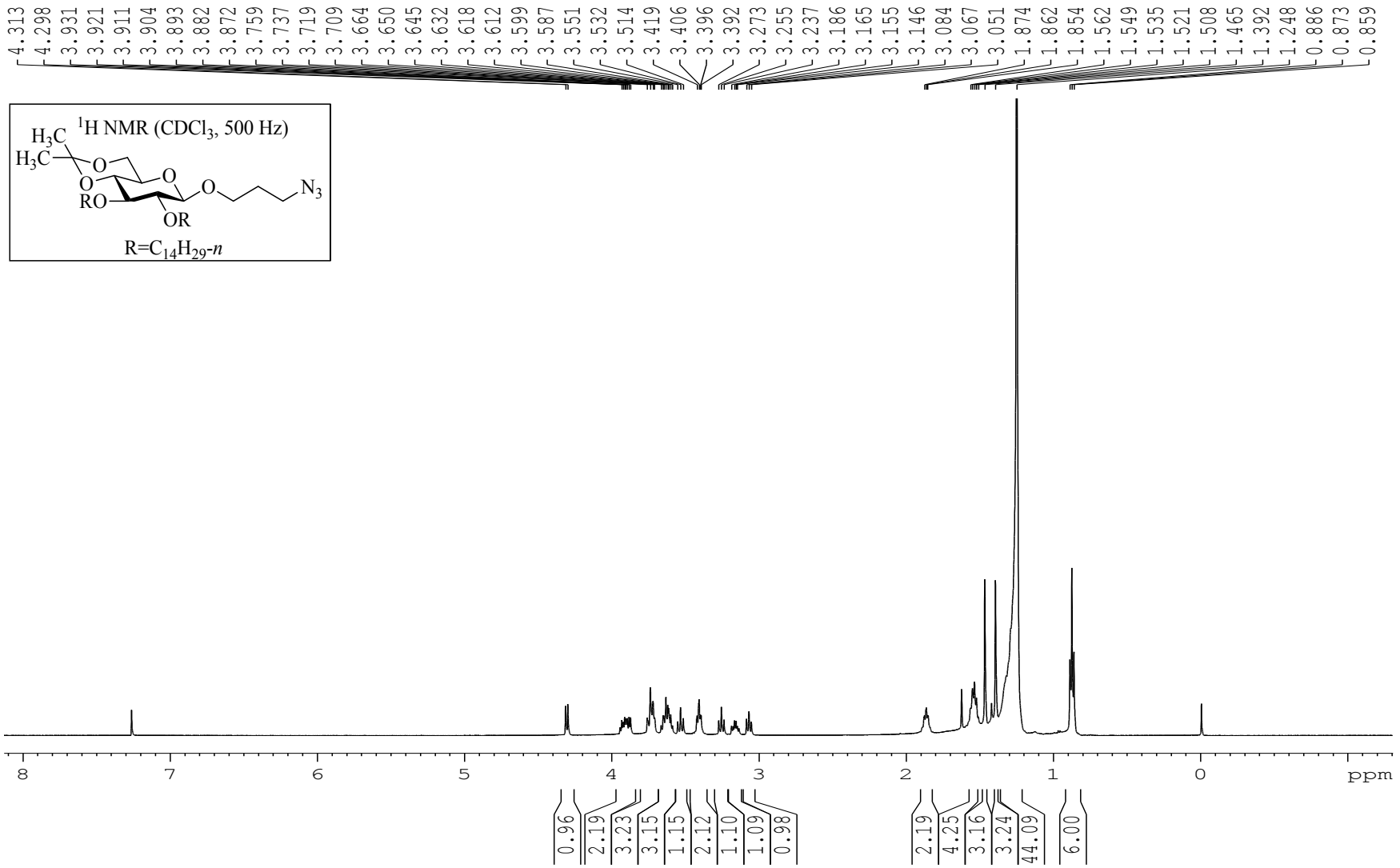

^{1}H-^{13}C HSQC (CDCl$_3$, 500Hz)
HO
HO
RO
O
O
N$_3$
OR
R=C$_{12}$H$_{25}$-*n*
ppm
10
20
30
40
50
60
70
80
90
100
110
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

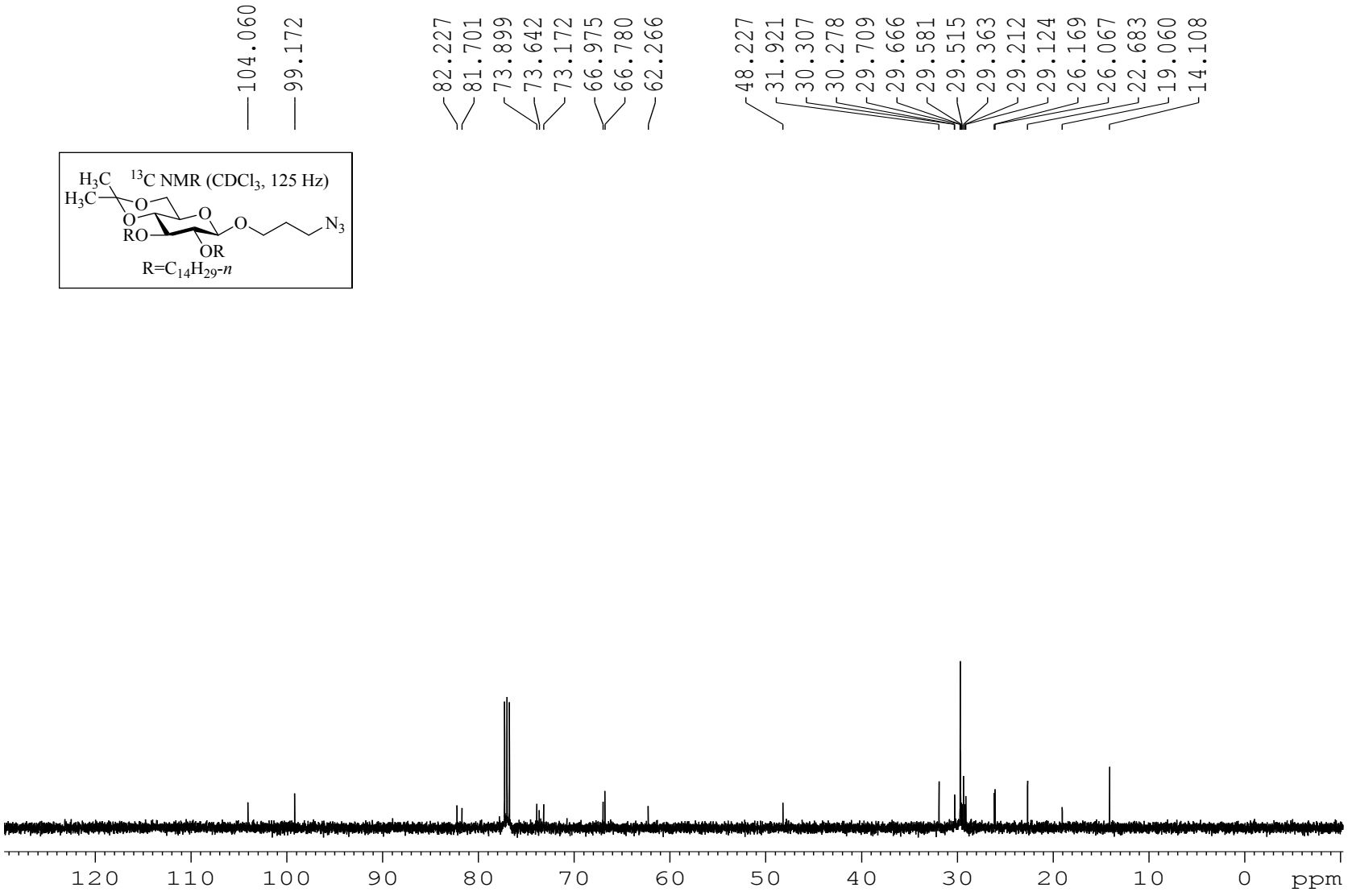

4.321
4.305
3.927
3.905
3.892
3.882
3.857
3.849
3.789
3.772
3.757
3.744
3.734
3.715
3.702
3.695
3.677
3.663
3.644
3.569
3.556
3.538
3.524
3.380
3.257
3.235
3.209
3.191
3.174
3.035
3.019
3.002
2.278
2.212
2.200
2.191
2.058
2.048
2.037
2.024
1.565
1.552
1.538
1.528
1.515
1.500
1.487
1.233
0.869
1H NMR (CDCl3, 500 Hz)
HO
HO
RO
OR
O
O
H3C
CH3
N+
CH3
I-
R=C12H25-n
8
7
6
5
4
3
2
1
0
−1
ppm
0.99
3.81
6.25
1.19
1.61
9.04
1.02
1.03
1.32
1.12
0.96
1.07
4.05
36.42
6.00

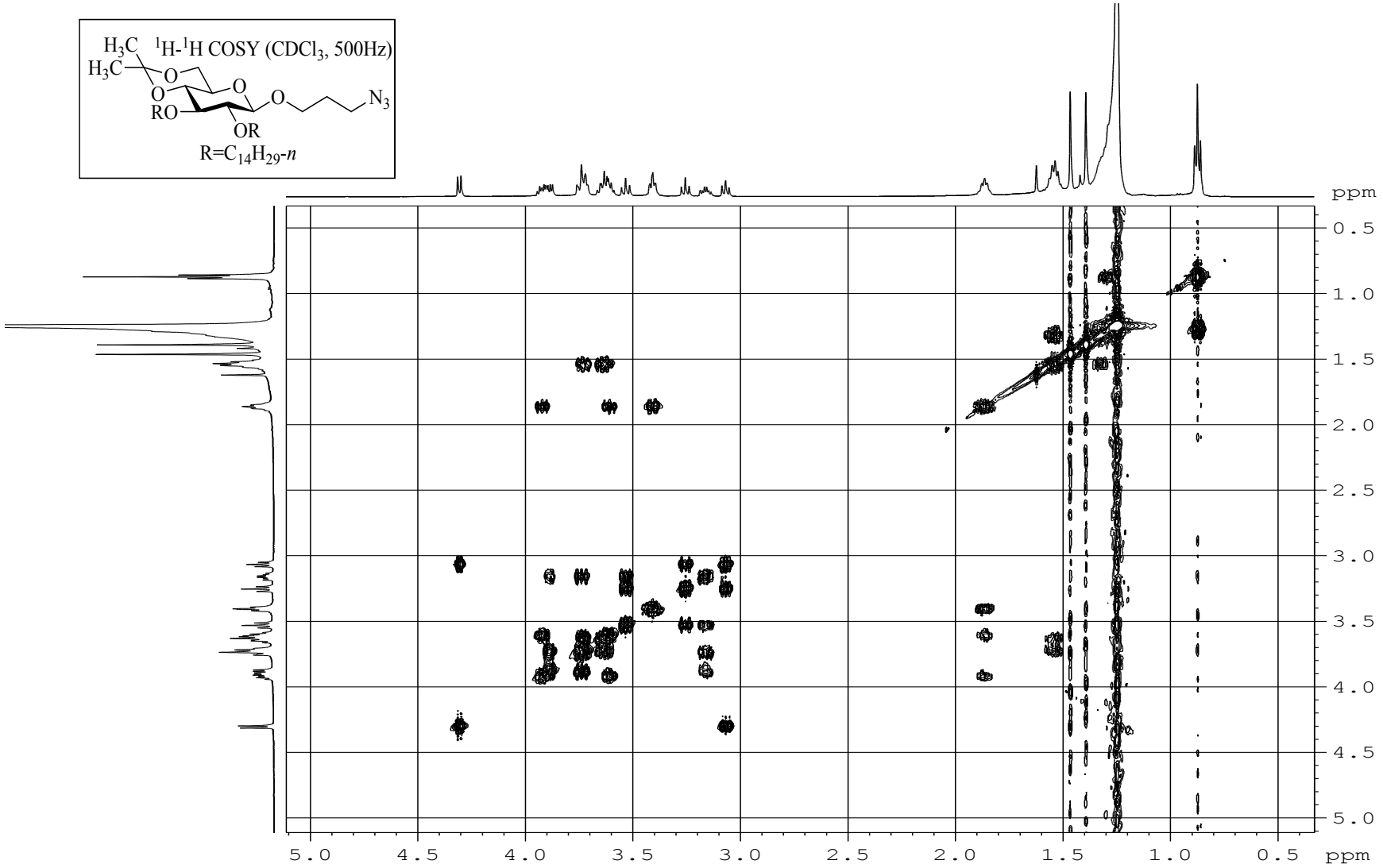

103.853
84.146
81.867
75.609
73.641
73.015
69.928
66.530
64.748
61.457
53.933
31.869
30.886
30.415
29.689
29.635
29.327
26.234
26.082
24.393
22.629
14.058
13C NMR (CDCl3, 125 Hz)
HO
HO
RO
OR
O
O
H3C
N
CH3
CH3
I-
R=C12H25-n
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

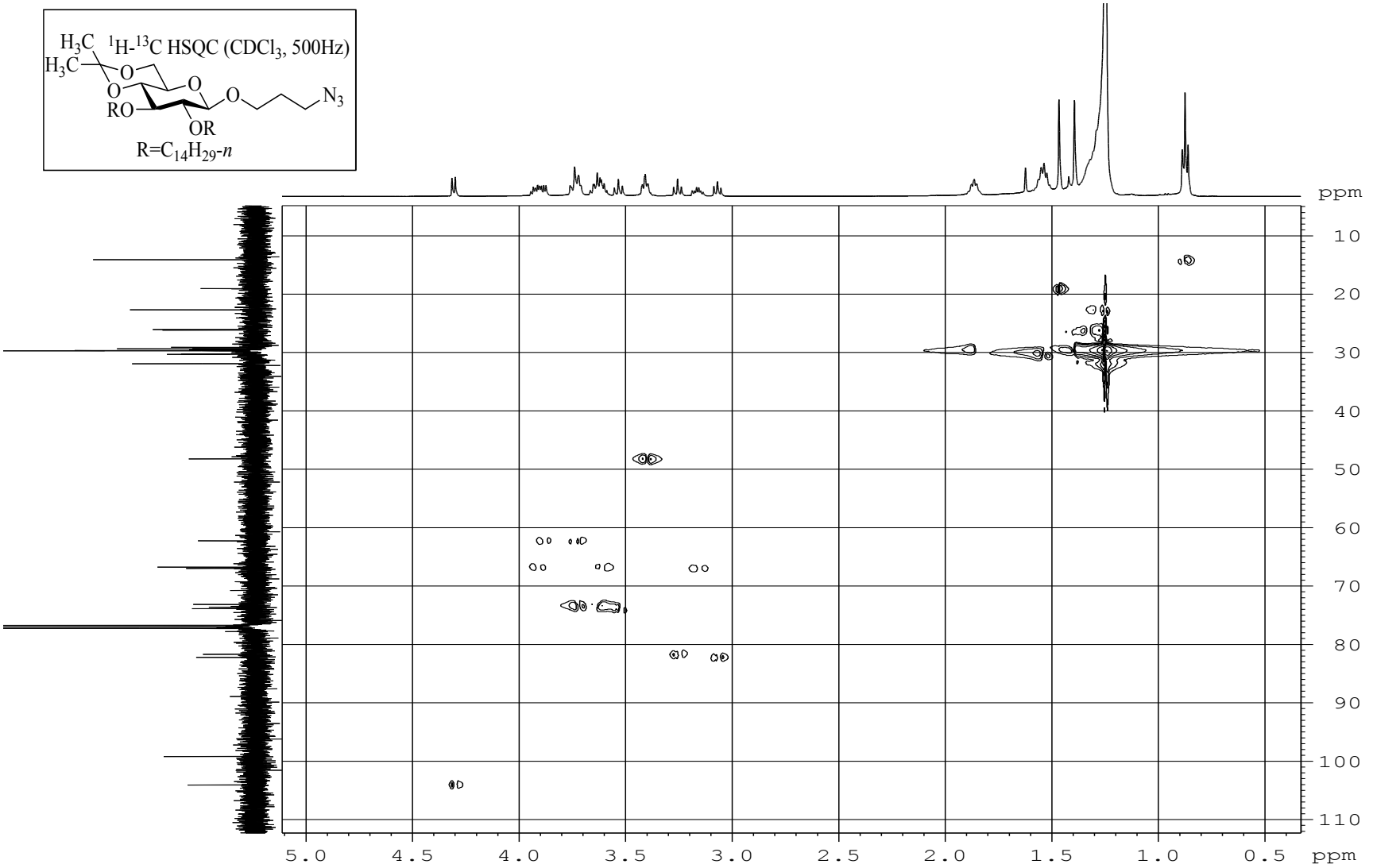

^{1}H-^{1}H COSY (CDCl$_3$, 500Hz)
HO
HO
RO
OR
O
O
R=C$_{12}$H$_{25}$-*n*
H$_3$C
CH$_3$
N
+
CH$_3$
I$^-$
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

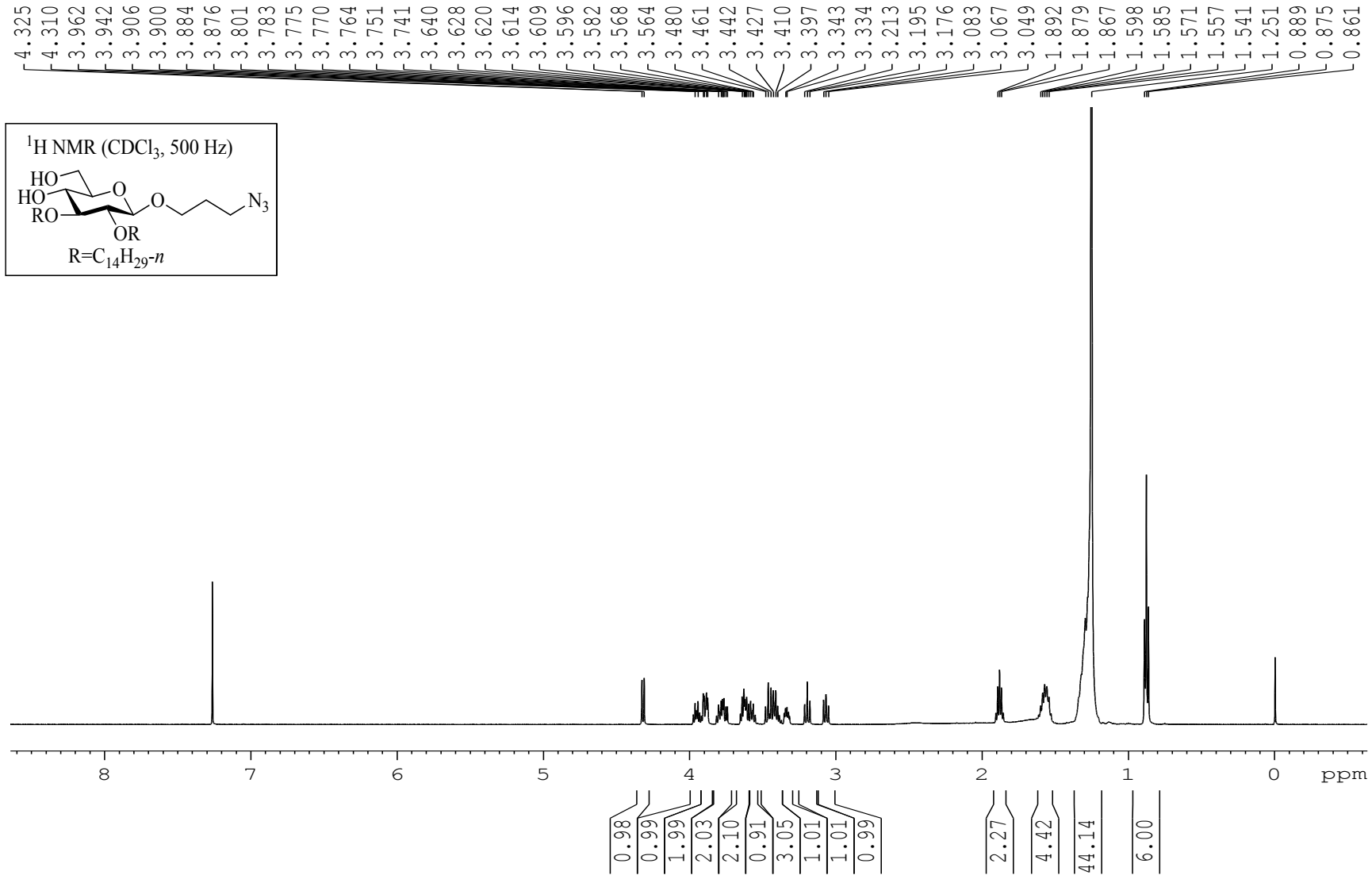

^{1}H-^{13}C HSQC (CDCl$_3$, 500Hz)
HO
HO
RO
OR
O
O
H$_3$C
N
CH$_3$
CH$_3$
I$^-$
R=C$_{12}$H$_{25}$-*n*
ppm
10
20
30
40
50
60
70
80
90
100
110
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

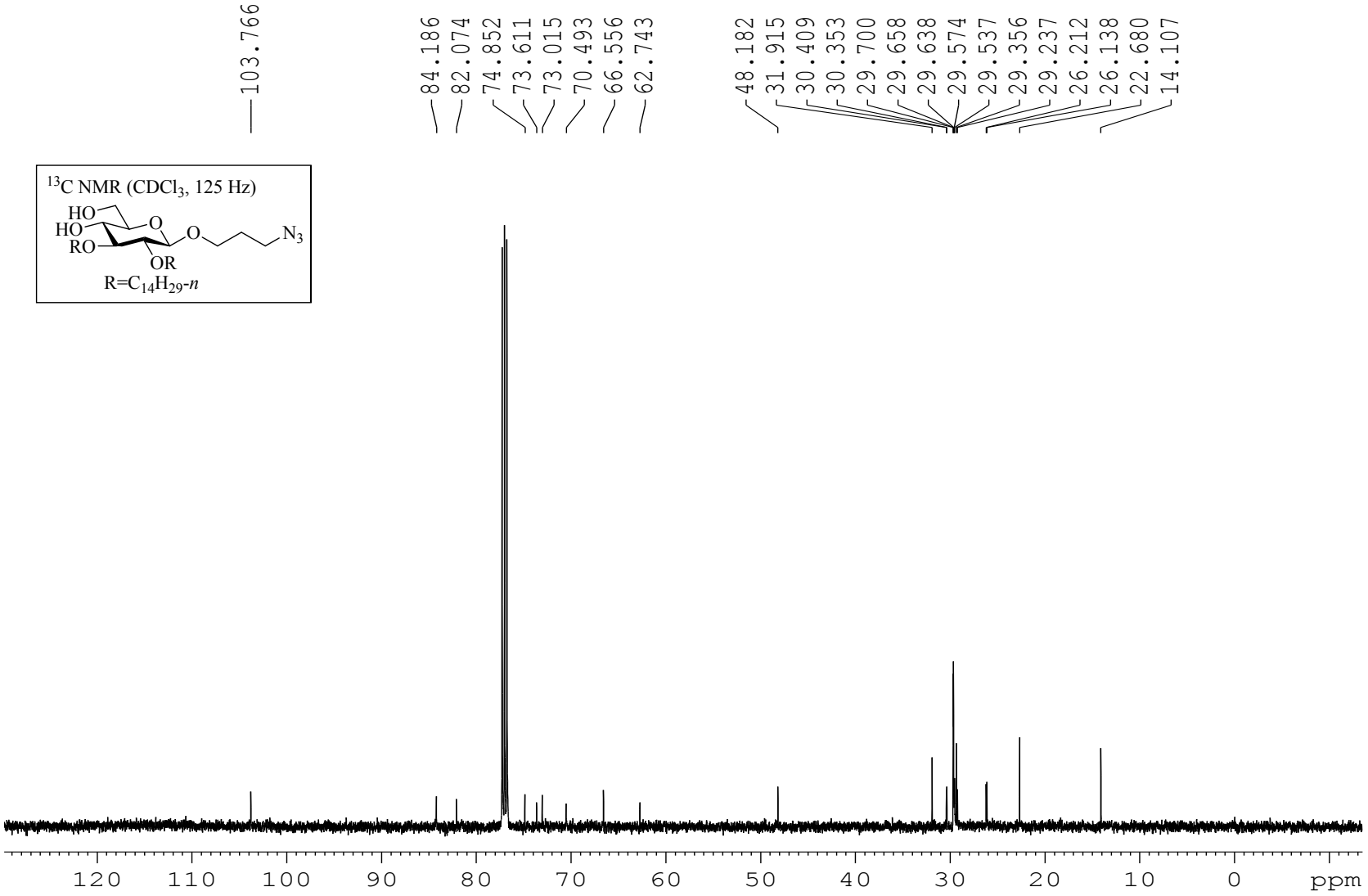

4.313
4.298
3.931
3.921
3.911
3.904
3.893
3.882
3.872
3.759
3.737
3.719
3.709
3.664
3.650
3.645
3.632
3.618
3.612
3.599
3.587
3.551
3.532
3.514
3.419
3.406
3.396
3.392
3.273
3.255
3.237
3.186
3.165
3.155
3.146
3.084
3.067
3.051
1.874
1.862
1.854
1.562
1.549
1.535
1.521
1.508
1.465
1.392
1.248
0.886
0.873
0.859
^{1}H NMR ($CDCl_3$, 500 Hz)
H_3C
H_3C
O
O
O
O
RO
O
N_3
OR
R=$C_{14}H_{29}$-*n*
8
7
6
5
4
3
2
1
0
ppm
0.96
2.19
3.23
3.15
1.15
2.12
1.10
1.09
0.98
2.19
4.25
3.16
3.24
44.09
6.00

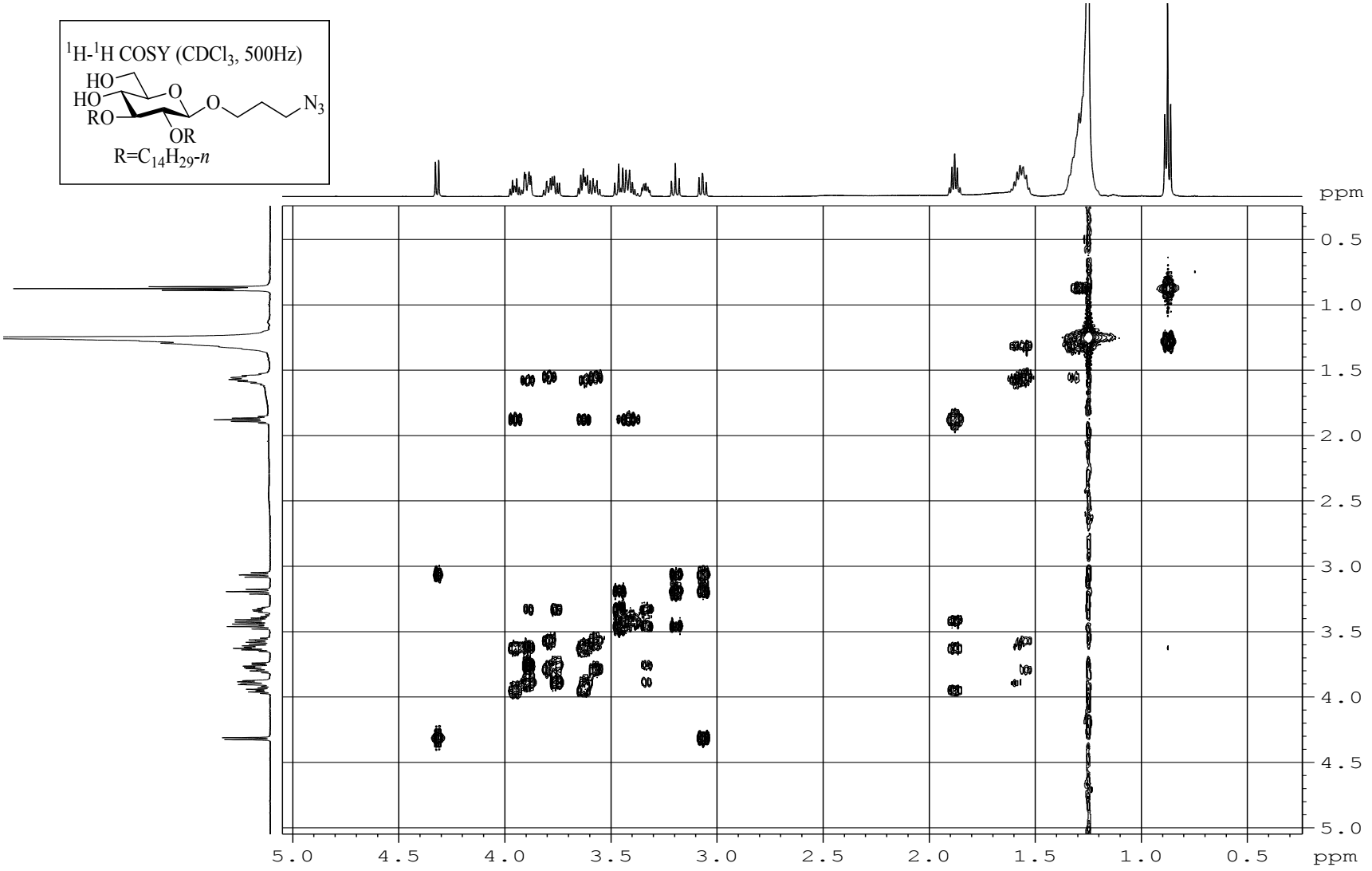

104.060
99.172
82.227
81.701
73.899
73.642
73.172
66.975
66.780
62.266
48.227
31.921
30.307
30.278
29.709
29.666
29.581
29.515
29.363
29.212
29.124
26.169
26.067
22.683
19.060
14.108
^{13}C NMR ($CDCl_3$, 125 Hz)
H_3C
H_3C
O
O
O
O
RO
OR
O
N_3
R=$C_{14}H_{29}$-*n*
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

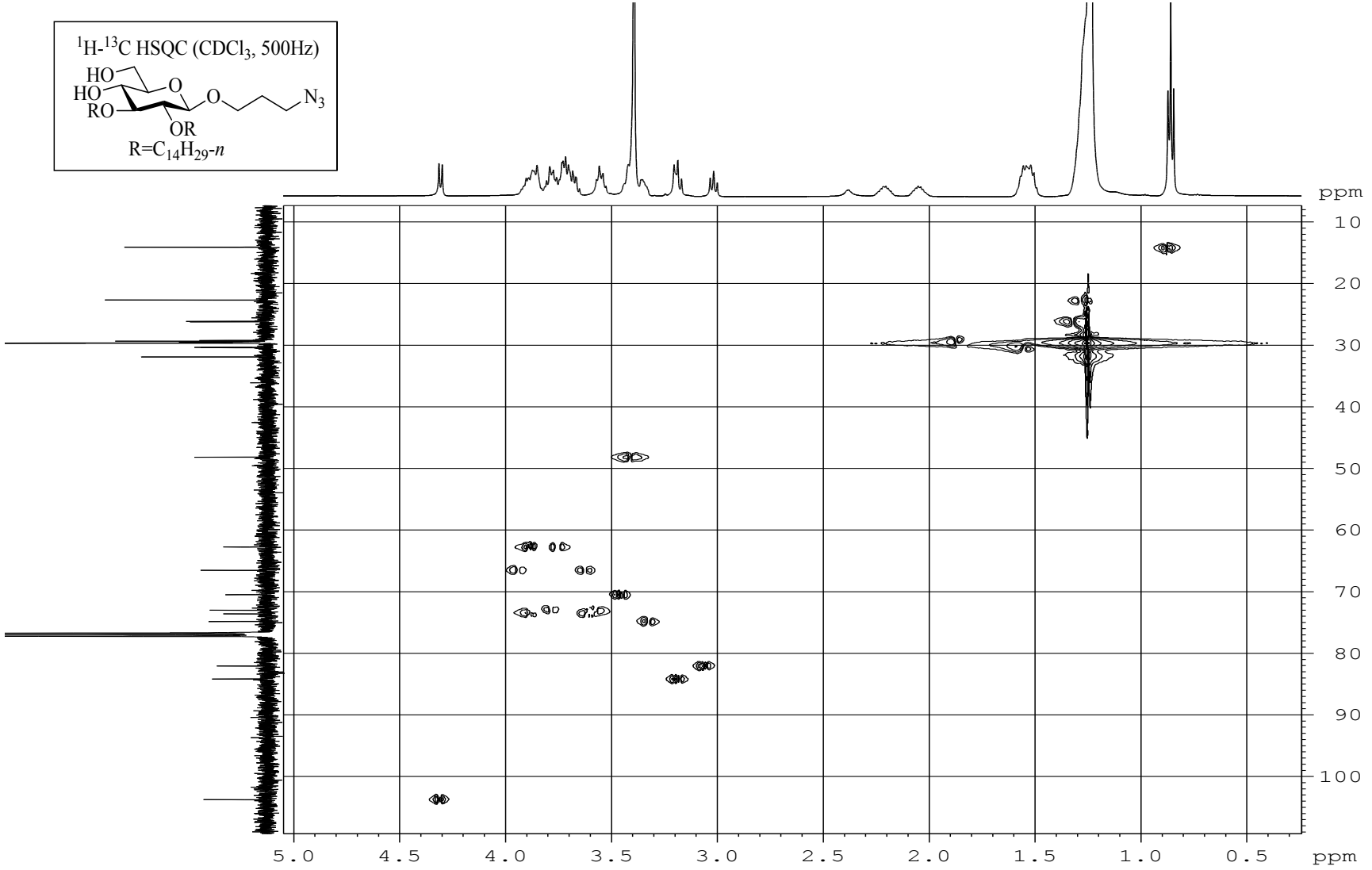

^{1}H-^{1}H COSY ($CDCl_3$, 500Hz)
R=$C_{14}H_{29}$-*n*
ppm
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

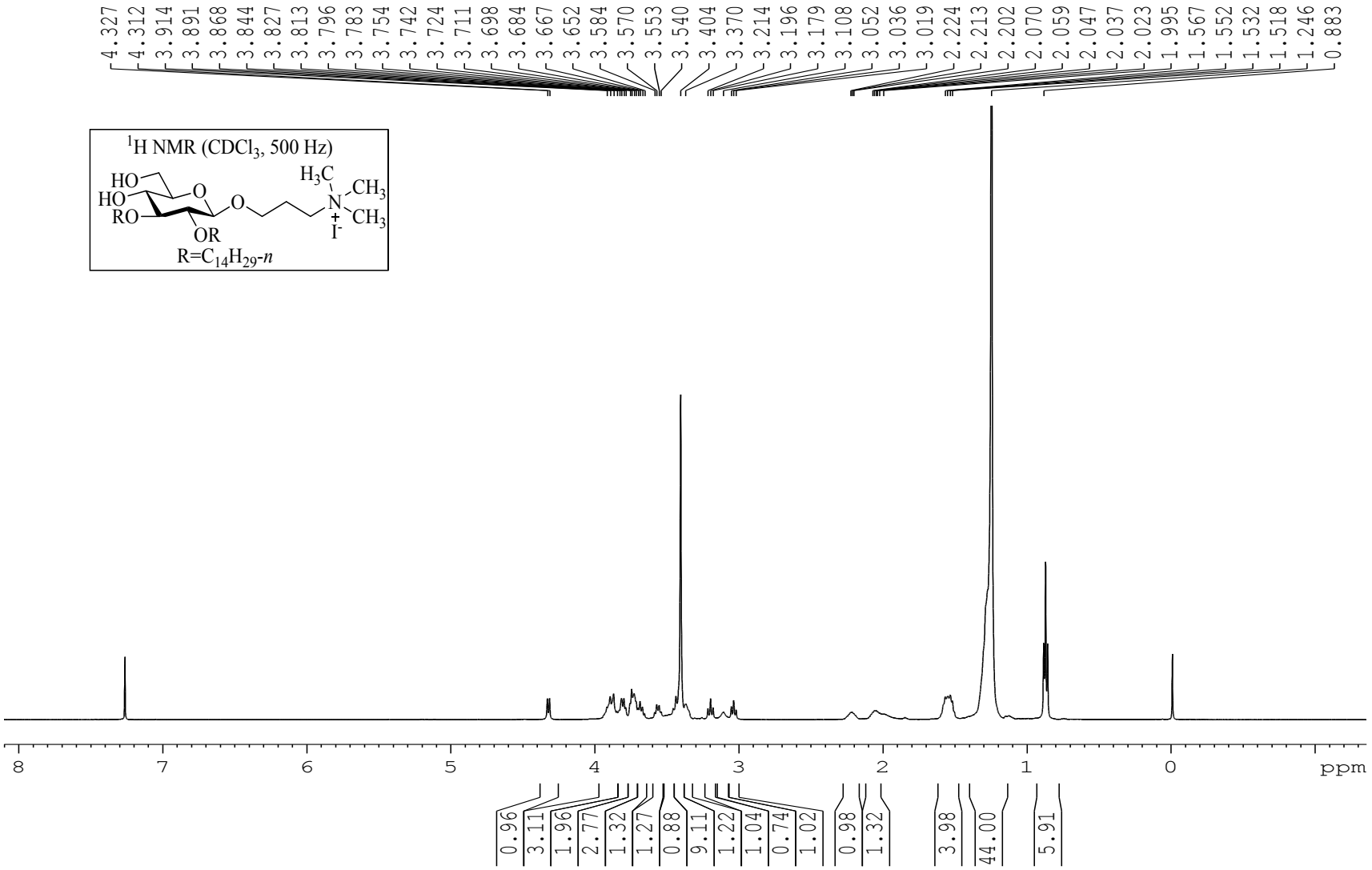

^{1}H-^{13}C HSQC ($CDCl_3$, 500Hz)
H_3C
H_3C
O
O
O
O
O
N_3
RO
OR
R=$C_{14}H_{29}$-*n*
ppm
10
20
30
40
50
60
70
80
90
100
110
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

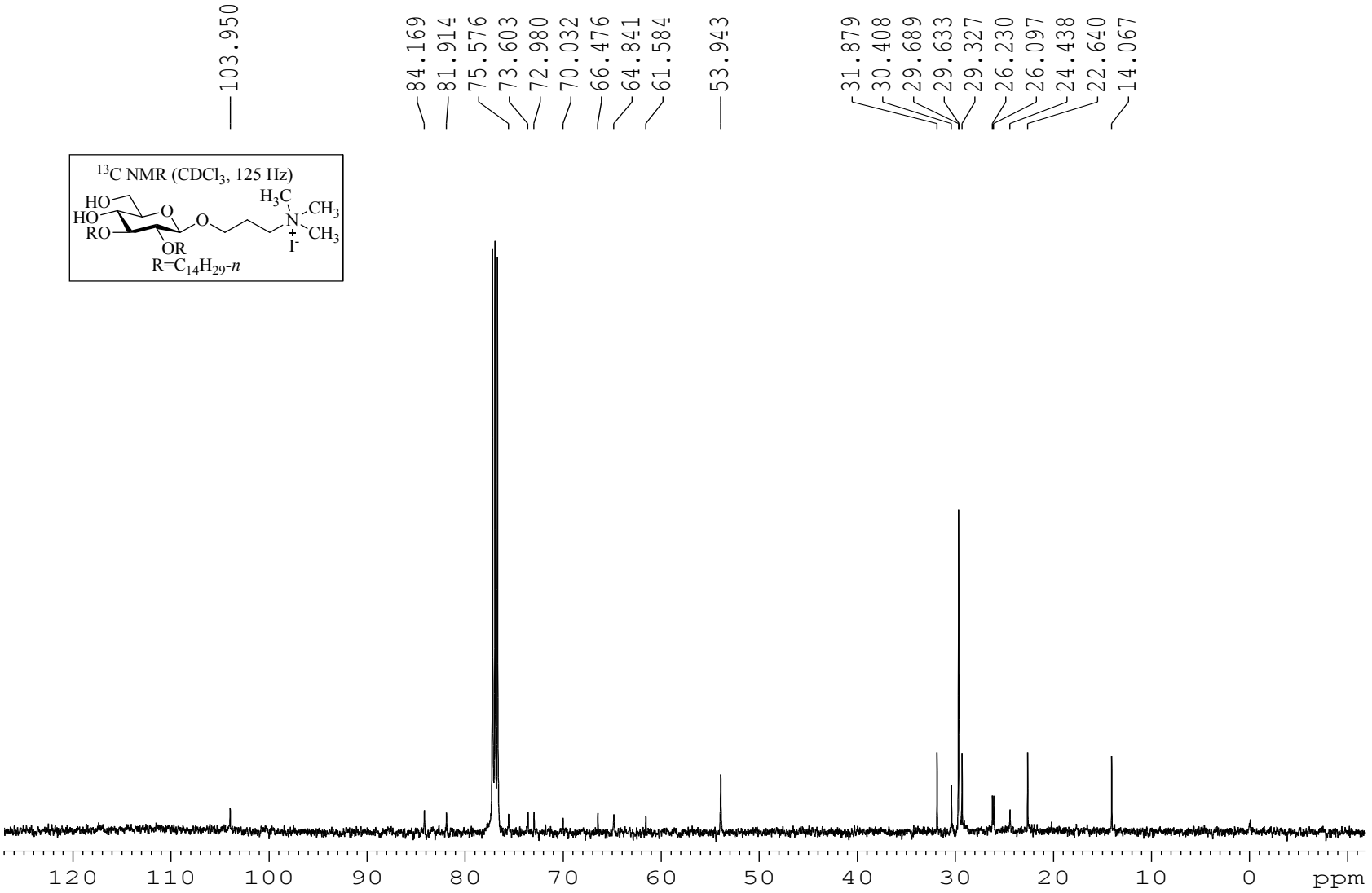

4.325
4.310
3.962
3.942
3.906
3.900
3.884
3.876
3.801
3.783
3.775
3.770
3.764
3.751
3.741
3.640
3.628
3.620
3.614
3.609
3.596
3.582
3.568
3.564
3.480
3.461
3.442
3.427
3.410
3.397
3.343
3.334
3.213
3.195
3.176
3.083
3.067
3.049
1.892
1.879
1.867
1.598
1.585
1.571
1.557
1.541
1.251
0.889
0.875
0.861
1H NMR (CDCl3, 500 Hz)
HO
HO
RO
O
O
N3
OR
R=C14H29-n
8
7
6
5
4
3
2
1
0
ppm
0.98
0.99
1.99
2.03
2.10
0.91
3.05
1.01
1.01
0.99
2.27
4.42
44.14
6.00

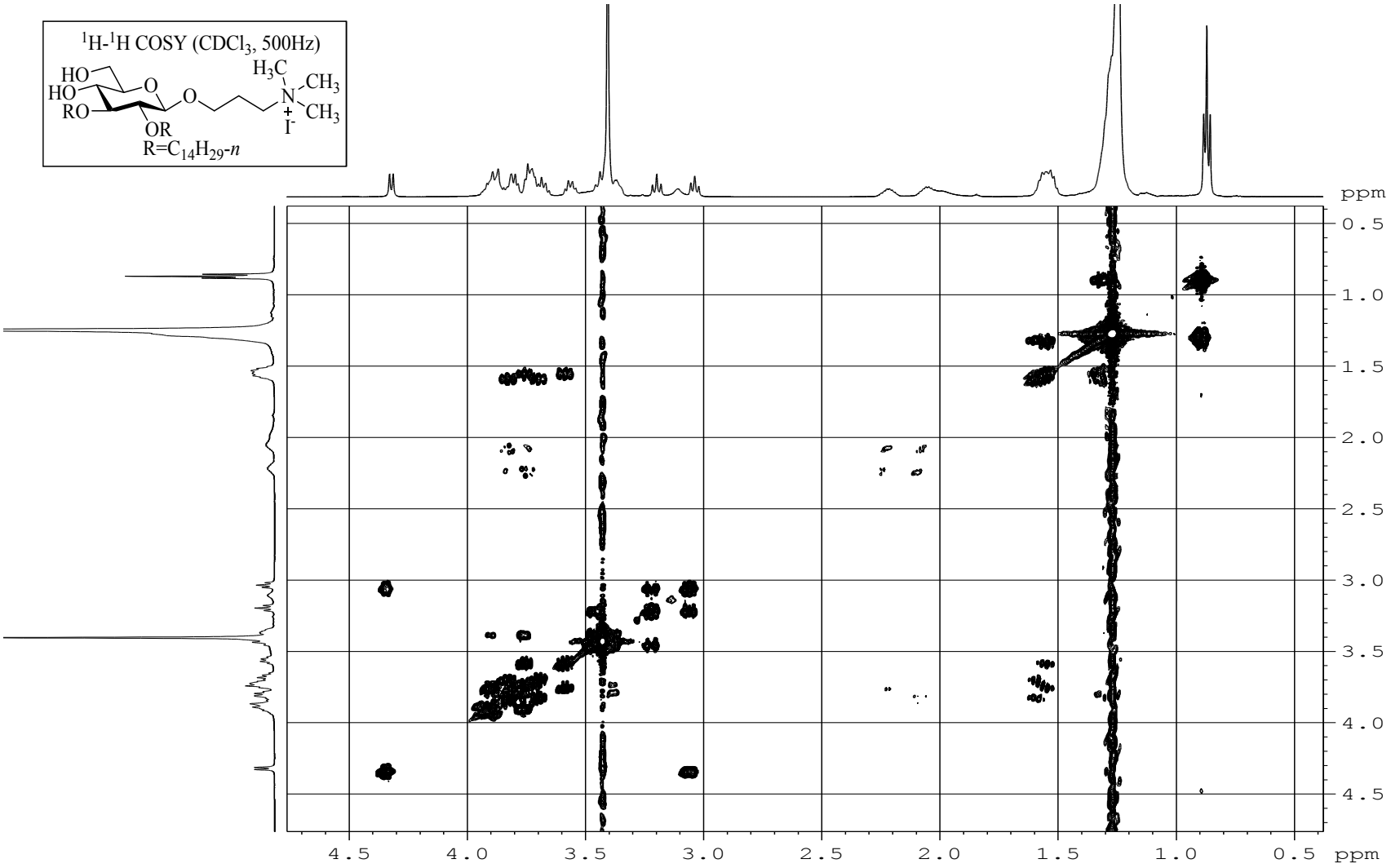

103.766
84.186
82.074
74.852
73.611
73.015
70.493
66.556
62.743
48.182
31.915
30.409
30.353
29.700
29.658
29.638
29.574
29.537
29.356
29.237
26.212
26.138
22.680
14.107
13C NMR (CDCl3, 125 Hz)
HO
HO
RO
O
O
N3
OR
R=C14H29-n
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

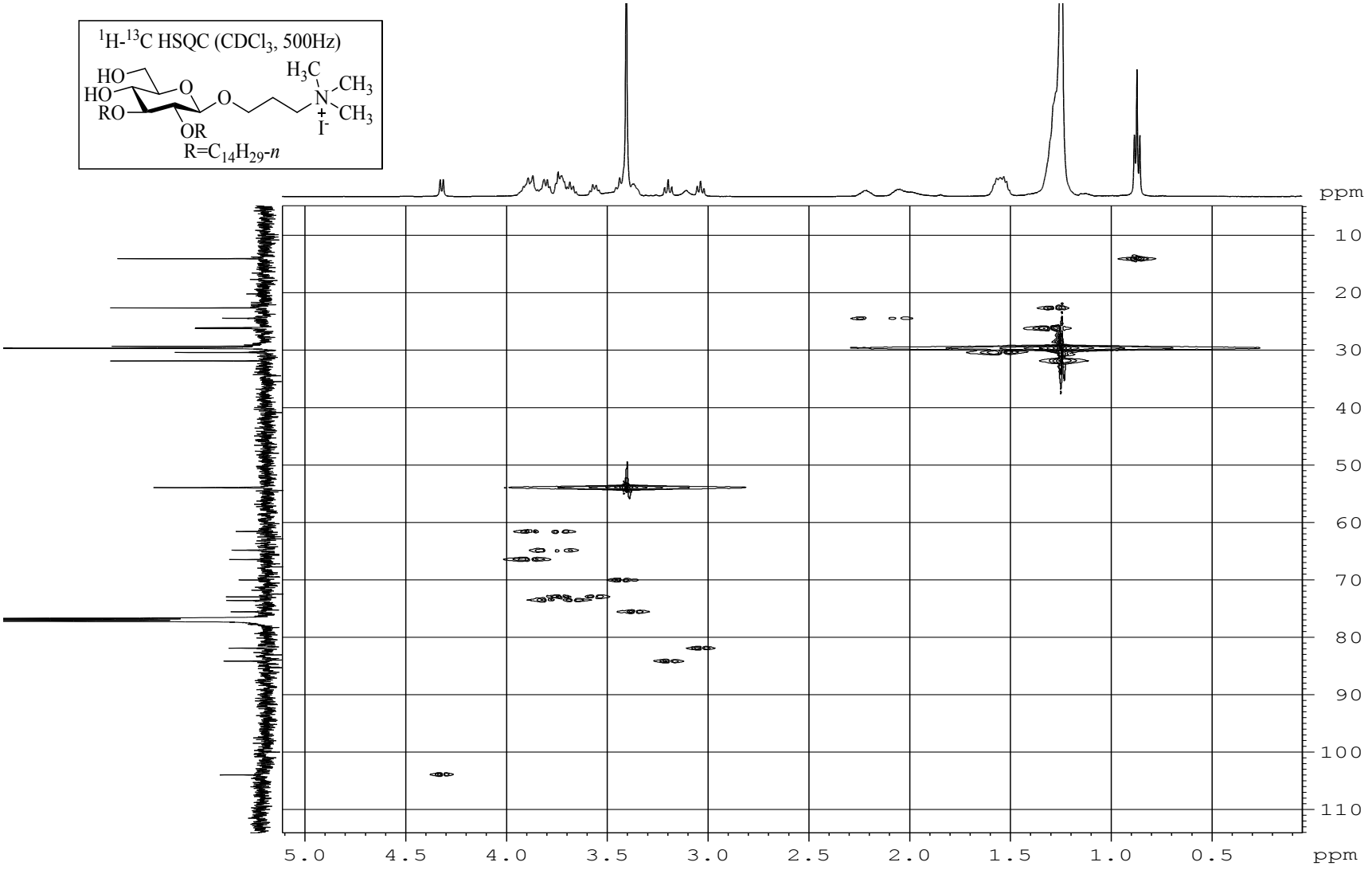

^{1}H-^{1}H COSY (CDCl$_3$, 500Hz)
HO
HO
RO
O
O
N$_3$
OR
R=C$_{14}$H$_{29}$-*n*
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

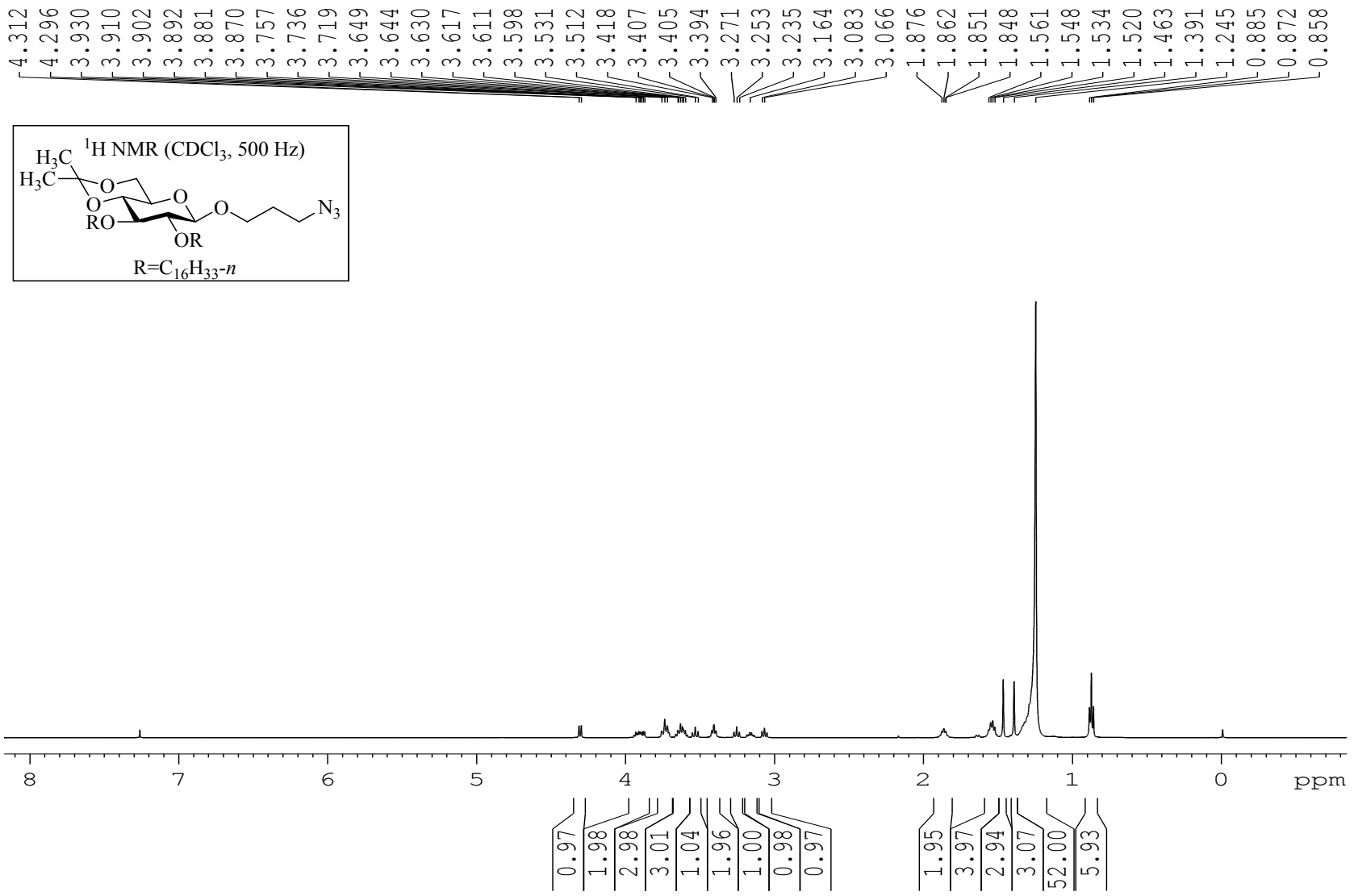

^{1}H-^{13}C HSQC (CDCl$_3$, 500Hz)
HO
HO
RO
O
O
N$_3$
OR
R=C$_{14}$H$_{29}$-*n*
ppm
10
20
30
40
50
60
70
80
90
100
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

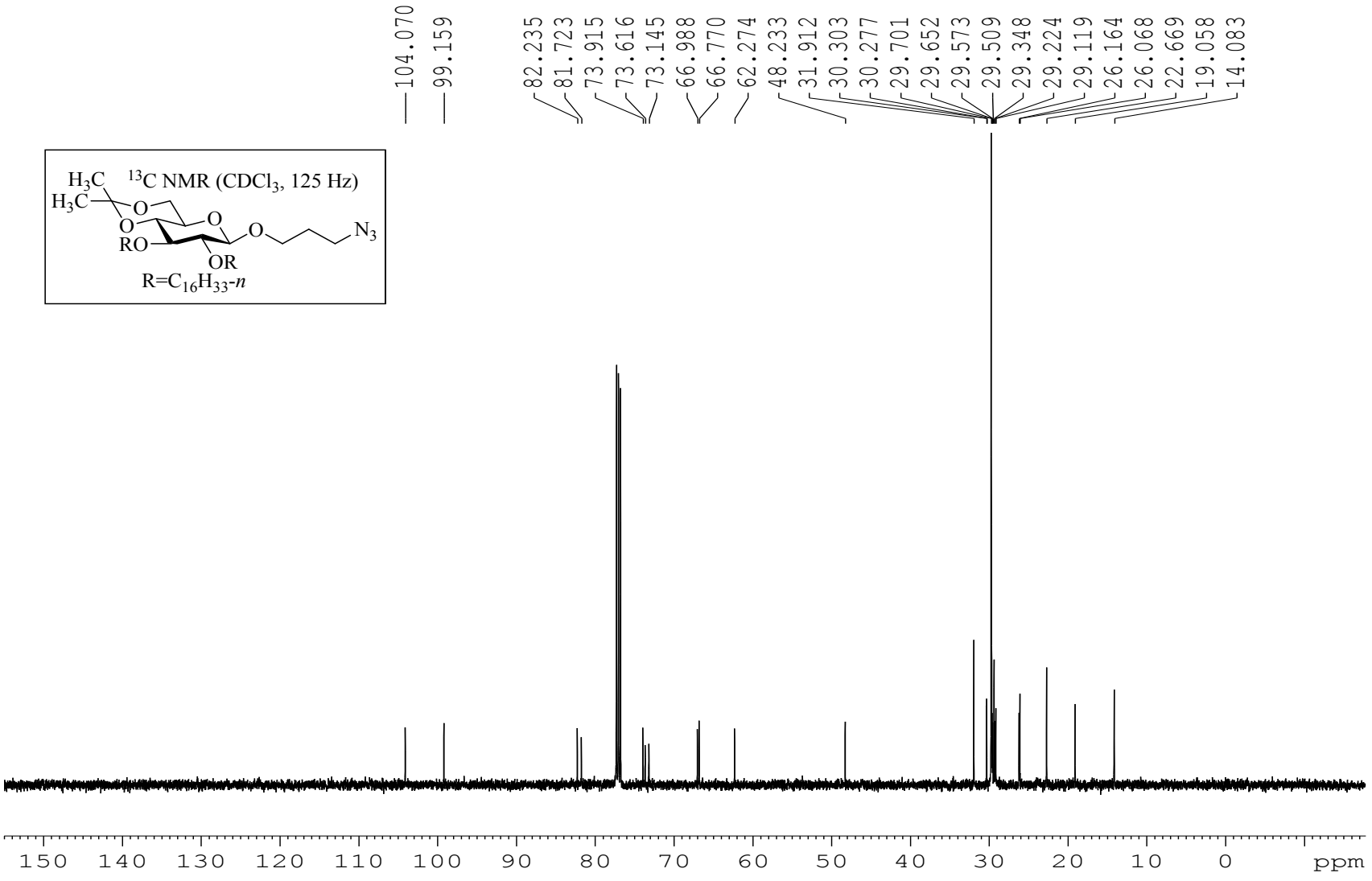

4.327
4.312
3.914
3.891
3.868
3.844
3.827
3.813
3.796
3.783
3.754
3.742
3.724
3.711
3.698
3.684
3.667
3.652
3.584
3.570
3.553
3.540
3.404
3.370
3.214
3.196
3.179
3.108
3.052
3.036
3.019
2.224
2.213
2.202
2.070
2.059
2.047
2.037
2.023
1.995
1.567
1.552
1.532
1.518
1.246
0.883
1H NMR (CDCl3, 500 Hz)
HO
HO
RO
OR
O
O
H3C
CH3
N
CH3
+
I-
R=C14H29-n
8
7
6
5
4
3
2
1
0
ppm
0.96
3.11
1.96
2.77
1.32
1.27
0.88
9.11
1.22
1.04
0.74
1.02
0.98
1.32
3.98
44.00
5.91

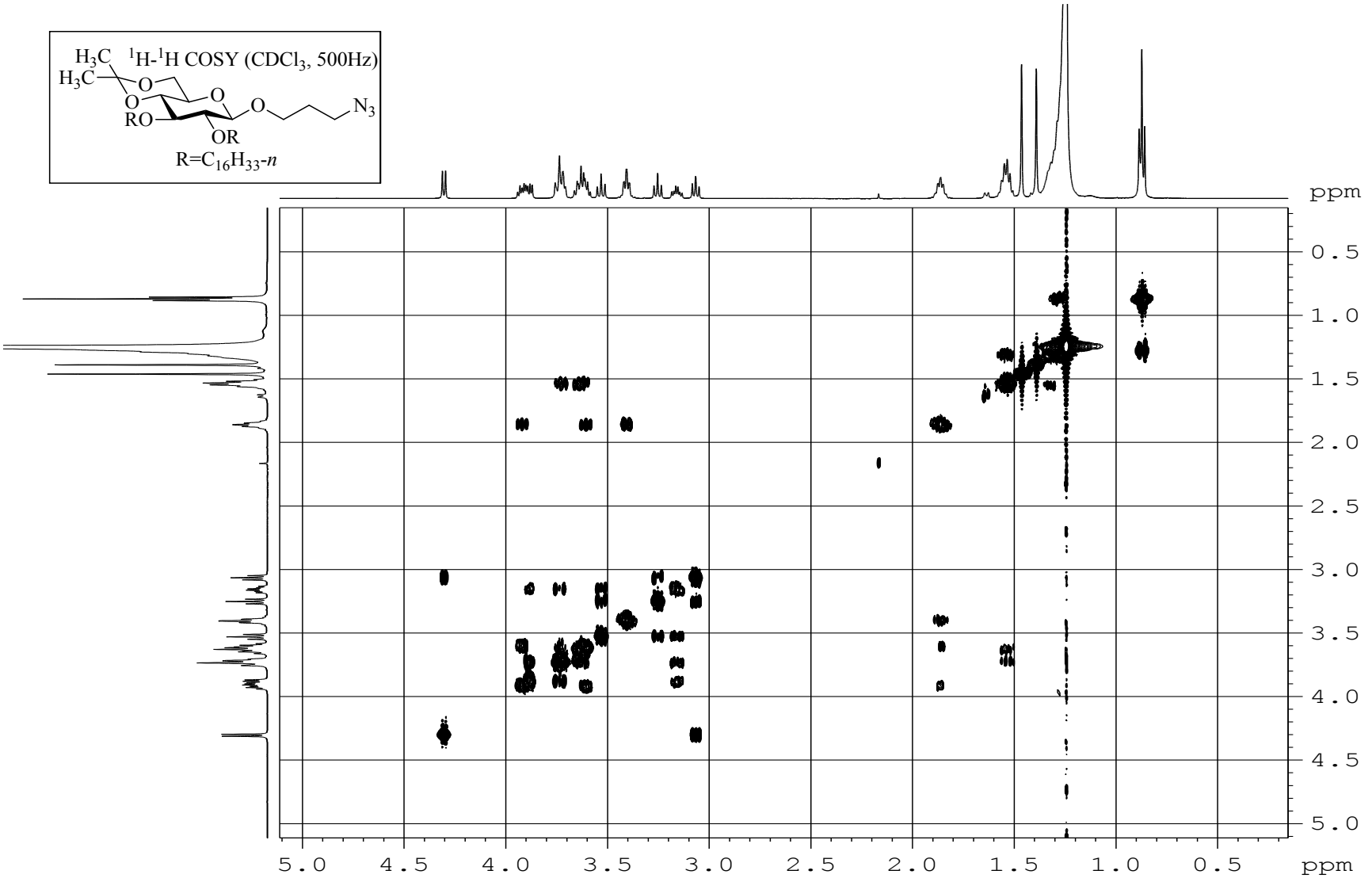

103.950
84.169
81.914
75.576
73.603
72.980
70.032
66.476
64.841
61.584
53.943
31.879
30.408
29.689
29.633
29.327
26.230
26.097
24.438
22.640
14.067
13C NMR (CDCl3, 125 Hz)
HO
HO
RO
OR
O
O
H3C
N
CH3
CH3
I-
R=C14H29-n
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

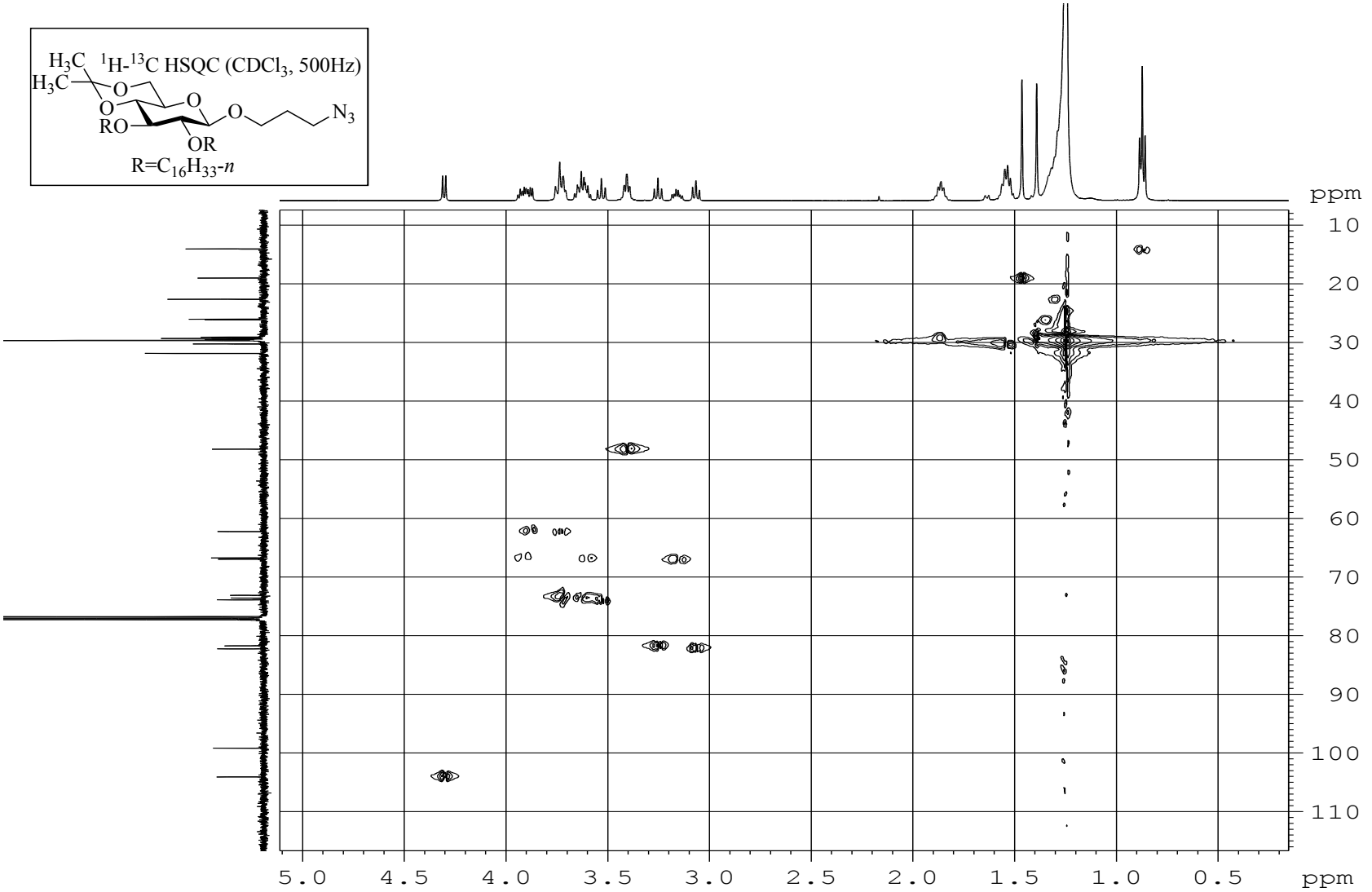

^{1}H-^{1}H COSY (CDCl$_3$, 500Hz)
HO
HO
RO
OR
O
O
H$_3$C
N
CH$_3$
CH$_3$
I$^-$
R=C$_{14}$H$_{29}$-*n*
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

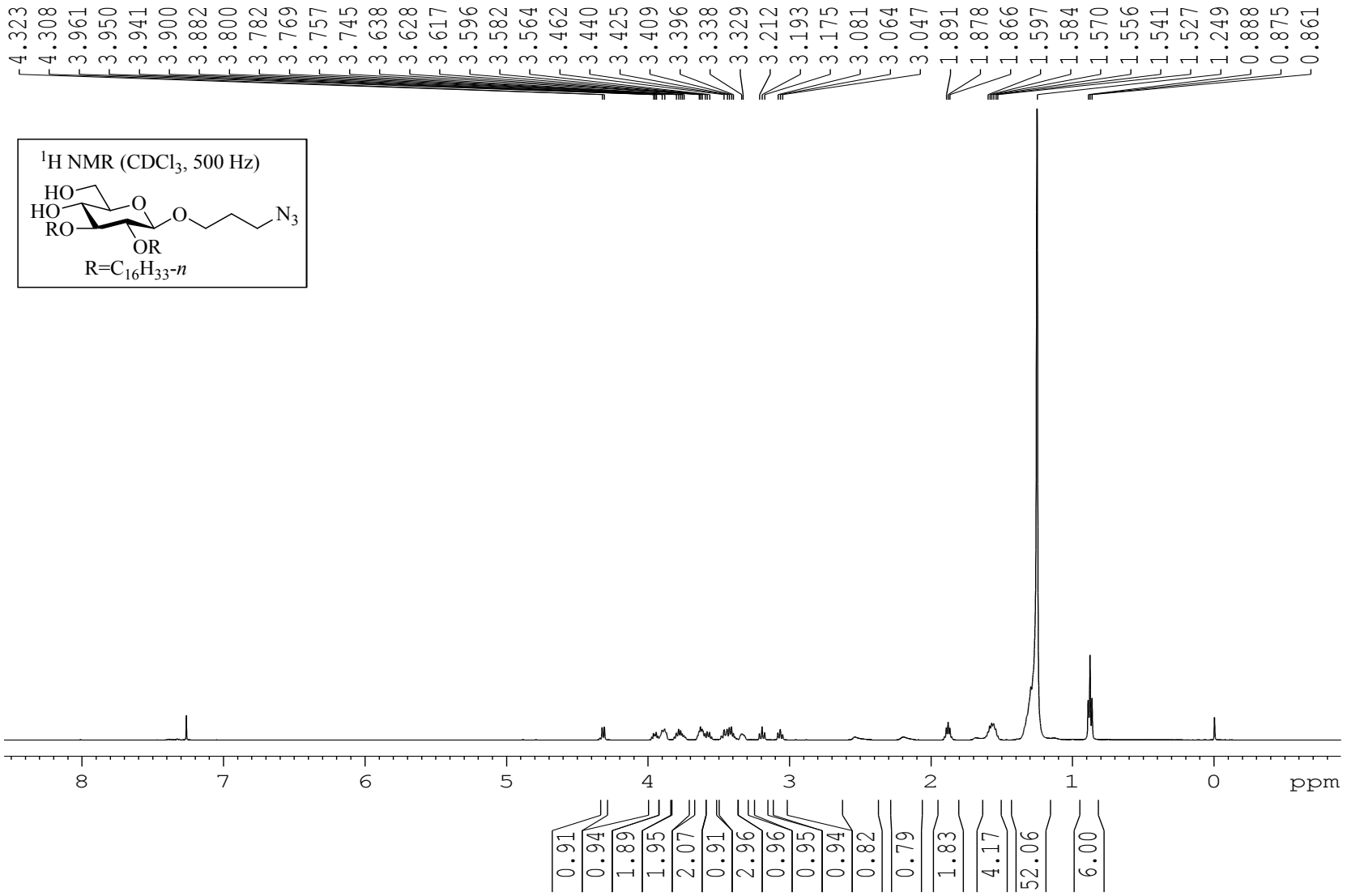

^{1}H-^{13}C HSQC (CDCl$_3$, 500Hz)
HO
HO
RO
O
O
OR
R=C$_{14}$H$_{29}$-*n*
H$_3$C
N
CH$_3$
CH$_3$
+
I$^-$
ppm
10
20
30
40
50
60
70
80
90
100
110
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

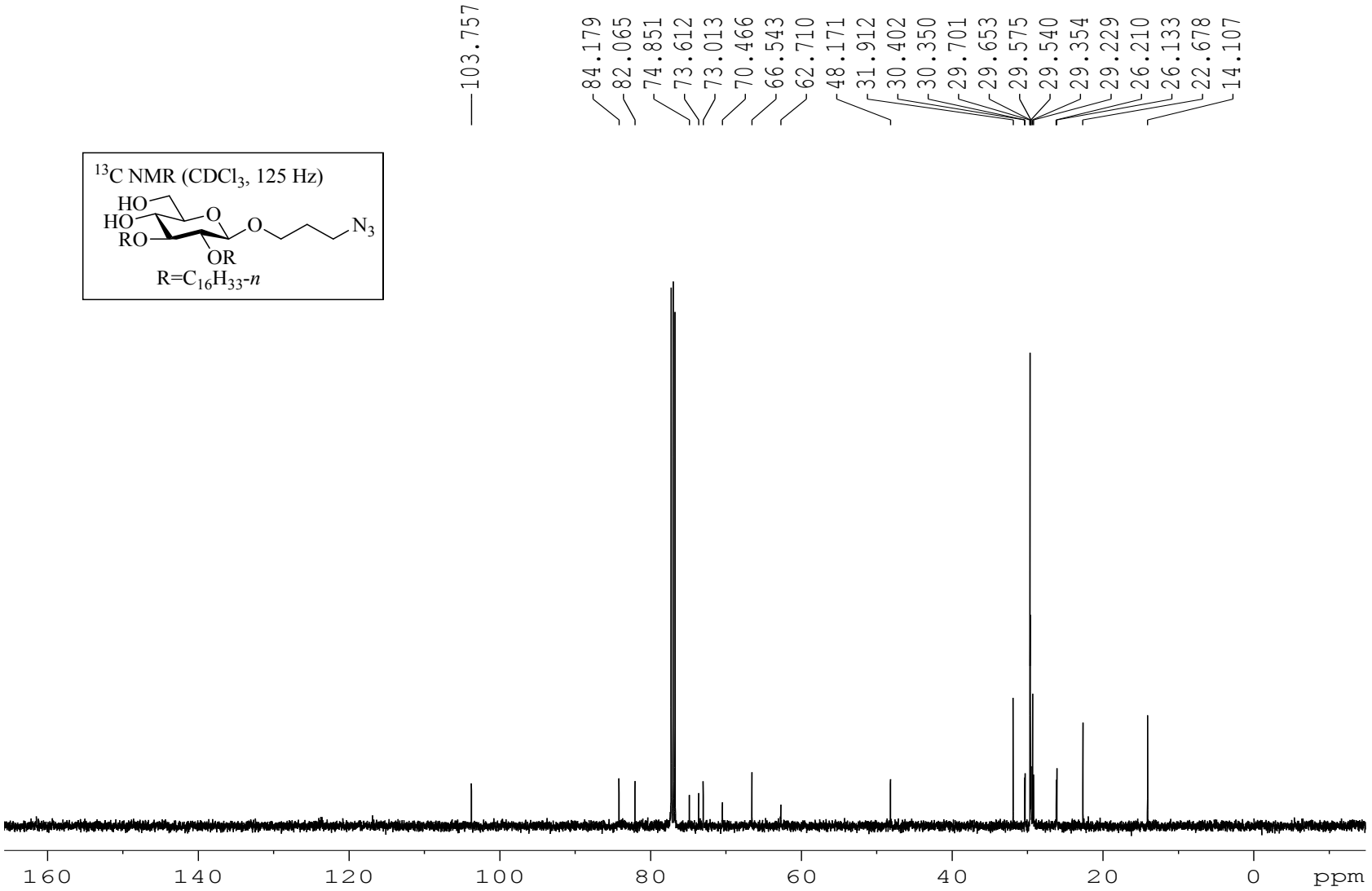

4.312
4.296
3.930
3.910
3.902
3.892
3.881
3.870
3.757
3.736
3.719
3.649
3.644
3.630
3.617
3.611
3.598
3.531
3.512
3.418
3.407
3.405
3.394
3.271
3.253
3.235
3.164
3.083
3.066
1.876
1.862
1.851
1.848
1.561
1.548
1.534
1.520
1.463
1.391
1.245
0.885
0.872
0.858
^{1}H NMR ($CDCl_3$, 500 Hz)
H_3C
H_3C
O
O
O
O
RO
OR
O
N_3
R=$C_{16}H_{33}$-*n*
8
7
6
5
4
3
2
1
0
ppm
0.97
1.98
2.98
3.01
1.04
1.96
1.00
0.98
0.97
1.95
3.97
2.94
3.07
52.00
5.93

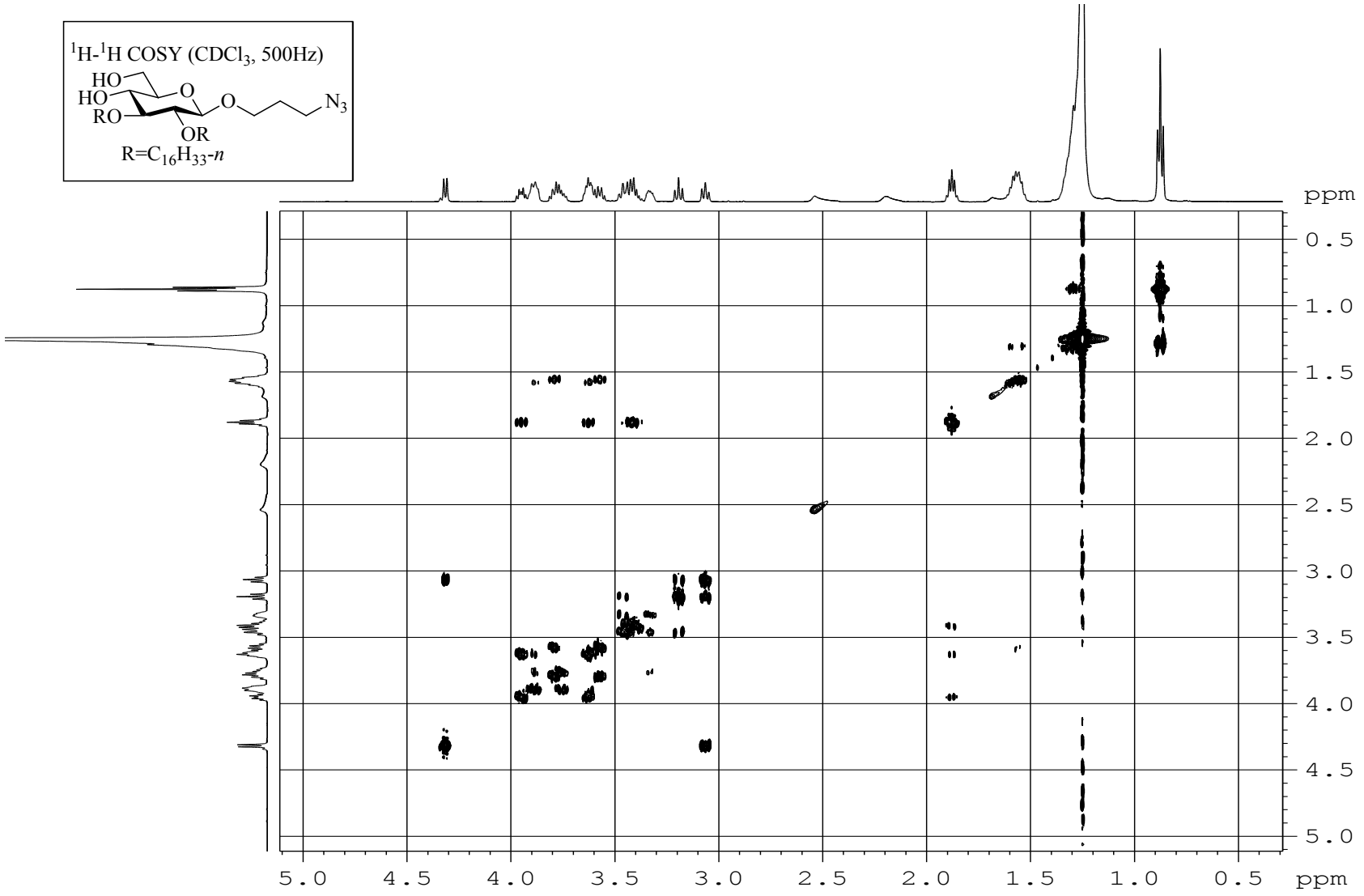

104.070
99.159
82.235
81.723
73.915
73.616
73.145
66.988
66.770
62.274
48.233
31.912
30.303
30.277
29.701
29.652
29.573
29.509
29.348
29.224
29.119
26.164
26.068
22.669
19.058
14.083
^{13}C NMR ($CDCl_3$, 125 Hz)
H_3C
H_3C
O
O
O
O
RO
OR
O
N_3
R=$C_{16}H_{33}$-*n*
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

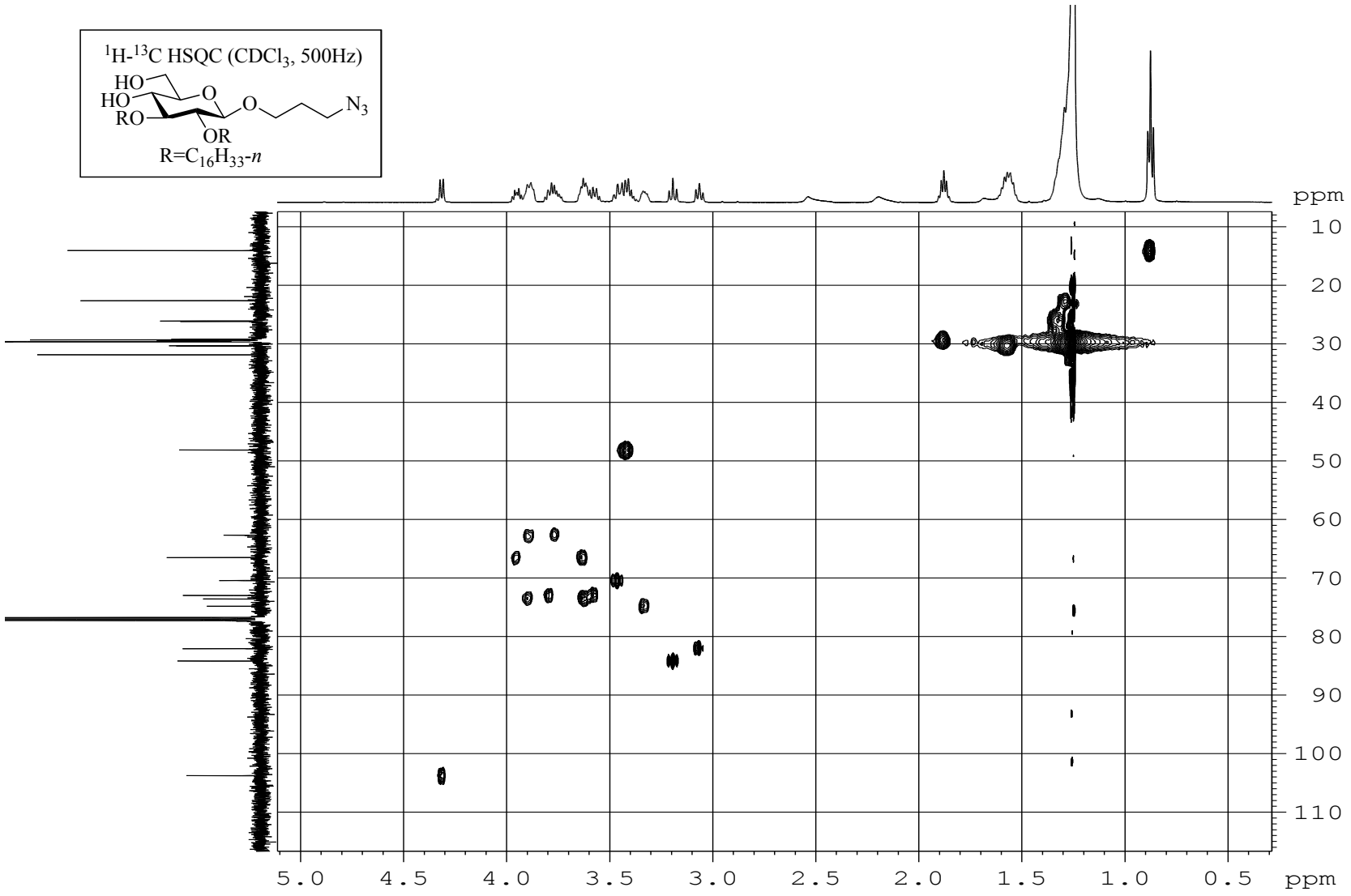

H3C
H3C
O
O
RO
O
O
O
N3
OR
R=C16H33-n
1H-1H COSY (CDCl3, 500Hz)
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

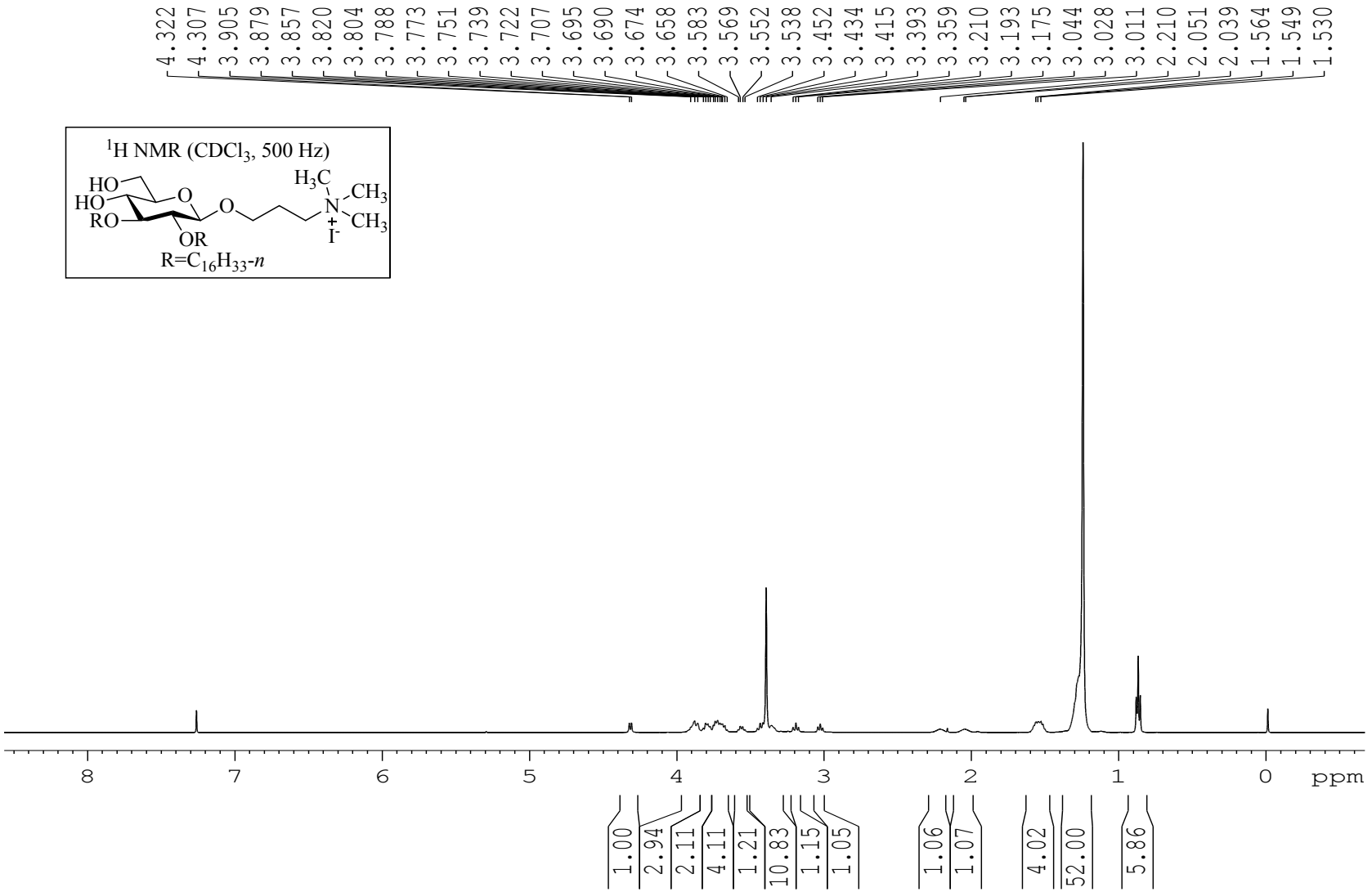

H_3C
H_3C
O
O
RO
O
O
N_3
OR
R=$C_{16}H_{33}$-*n*
^{1}H-^{13}C HSQC ($CDCl_3$, 500Hz)
ppm
10
20
30
40
50
60
70
80
90
100
110
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

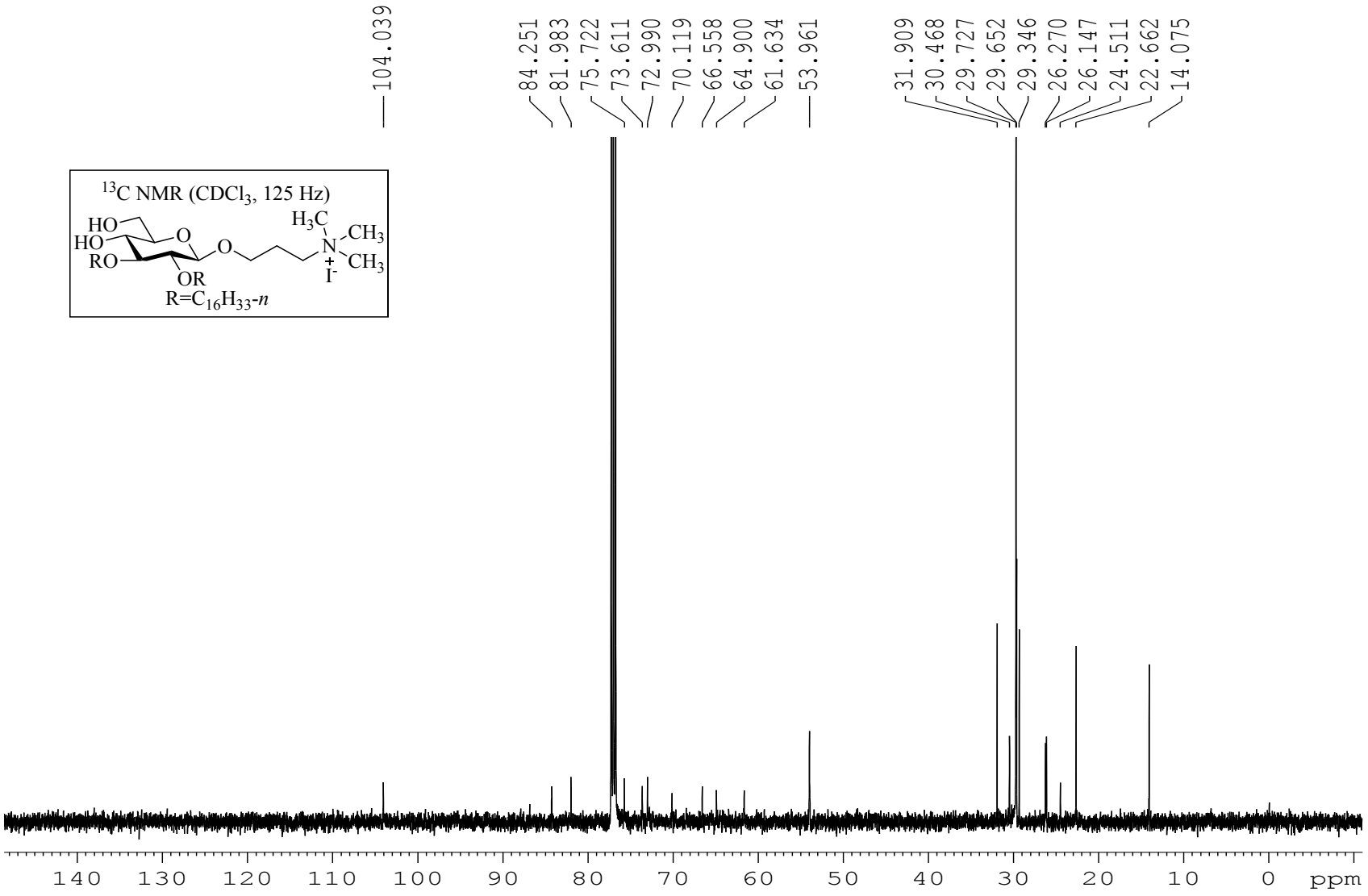

4.323
4.308
3.961
3.950
3.941
3.900
3.882
3.800
3.782
3.769
3.757
3.745
3.638
3.628
3.617
3.596
3.582
3.564
3.462
3.440
3.425
3.409
3.396
3.338
3.329
3.212
3.193
3.175
3.081
3.064
3.047
1.891
1.878
1.866
1.597
1.584
1.570
1.556
1.541
1.527
1.249
0.888
0.875
0.861
1H NMR (CDCl3, 500 Hz)
HO
HO
RO
O
O
N3
OR
R=C16H33-n
8
7
6
5
4
3
2
1
0
ppm
0.91
0.94
1.89
1.95
2.07
0.91
2.96
0.96
0.95
0.94
0.82
0.79
1.83
4.17
52.06
6.00

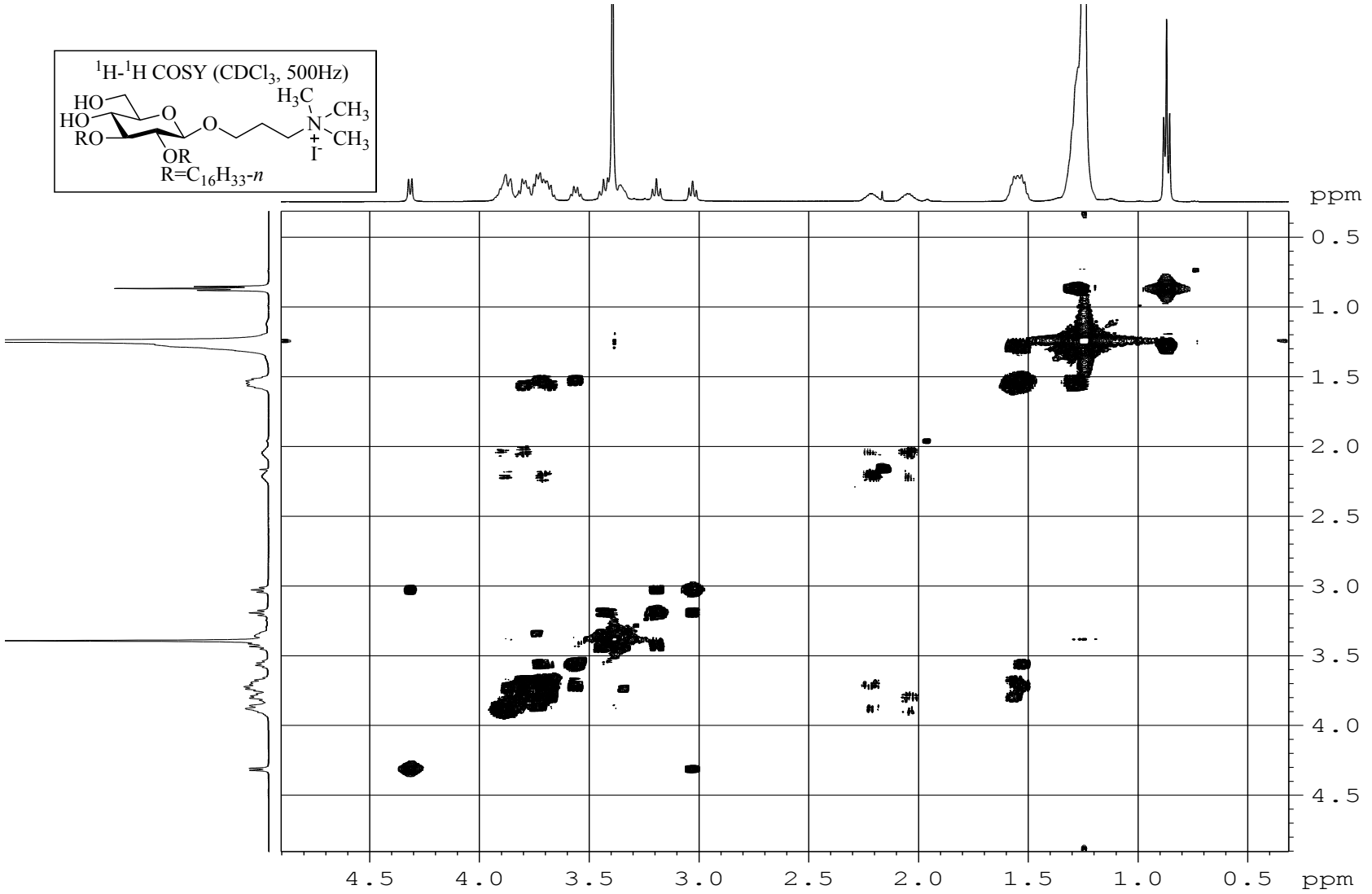

103.757
84.179
82.065
74.851
73.612
73.013
70.466
66.543
62.710
48.171
31.912
30.402
30.350
29.701
29.653
29.575
29.540
29.354
29.229
26.210
26.133
22.678
14.107
13C NMR (CDCl3, 125 Hz)
HO
HO
RO
O
O
N3
OR
R=C16H33-n
160
140
120
100
80
60
40
20
0
ppm

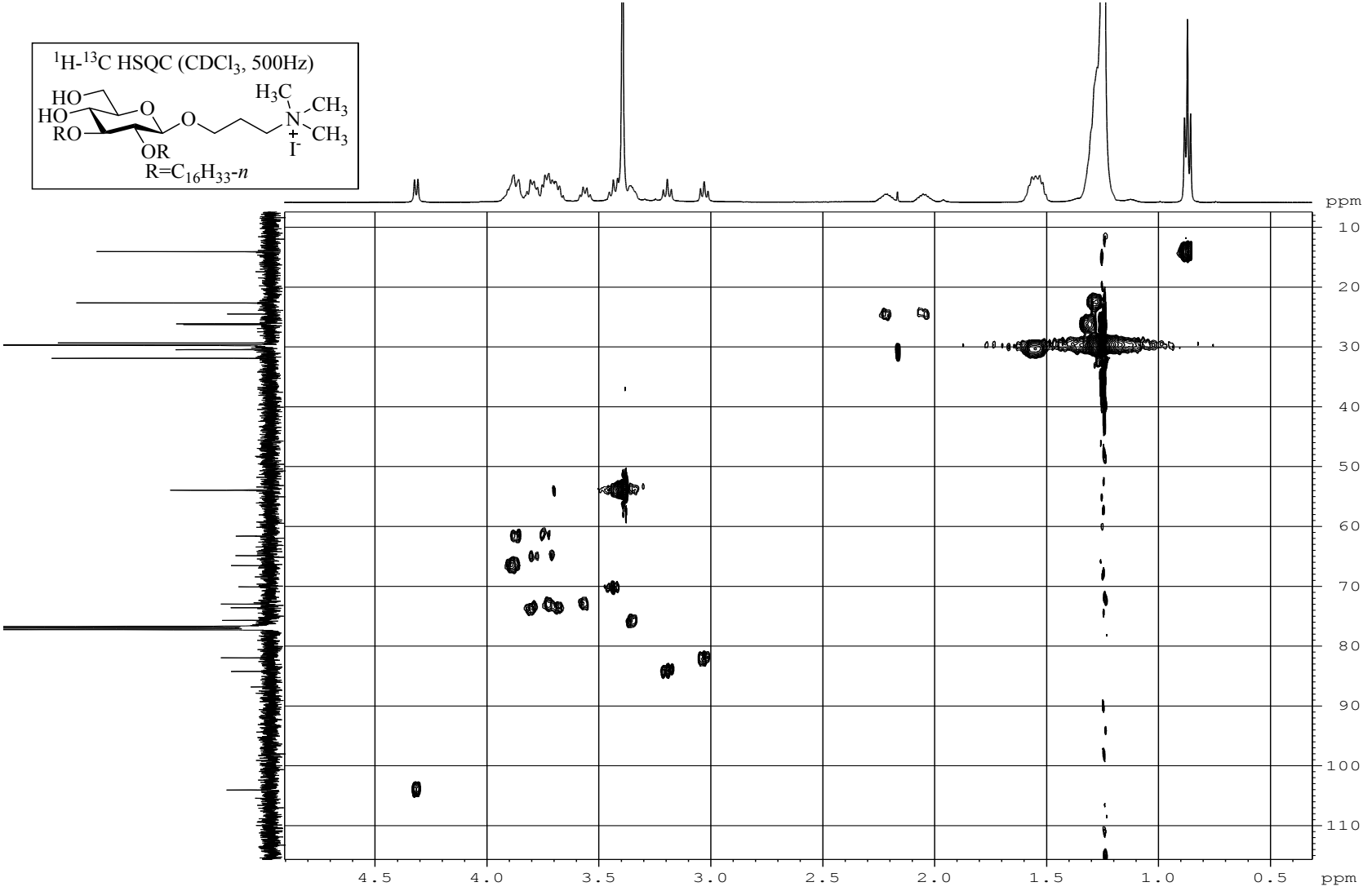

^{1}H-^{1}H COSY (CDCl$_3$, 500Hz)
HO
HO
RO
O
O
N$_3$
OR
R=C$_{16}$H$_{33}$-*n*
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

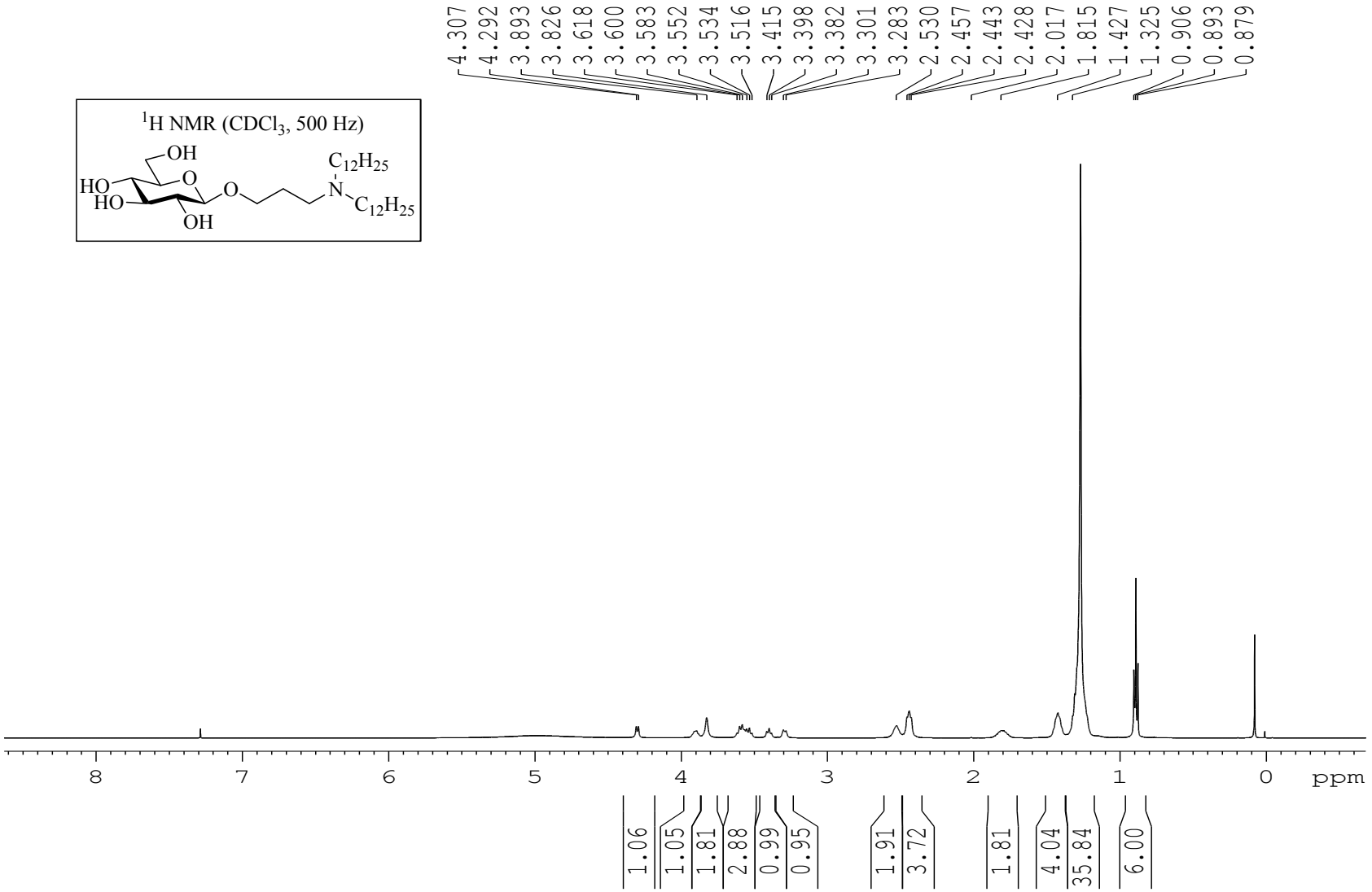

^{1}H-^{13}C HSQC ($CDCl_3$, 500Hz)
HO
HO
RO
O
O
N_3
OR
R=$C_{16}H_{33}$-*n*
ppm
10
20
30
40
50
60
70
80
90
100
110
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

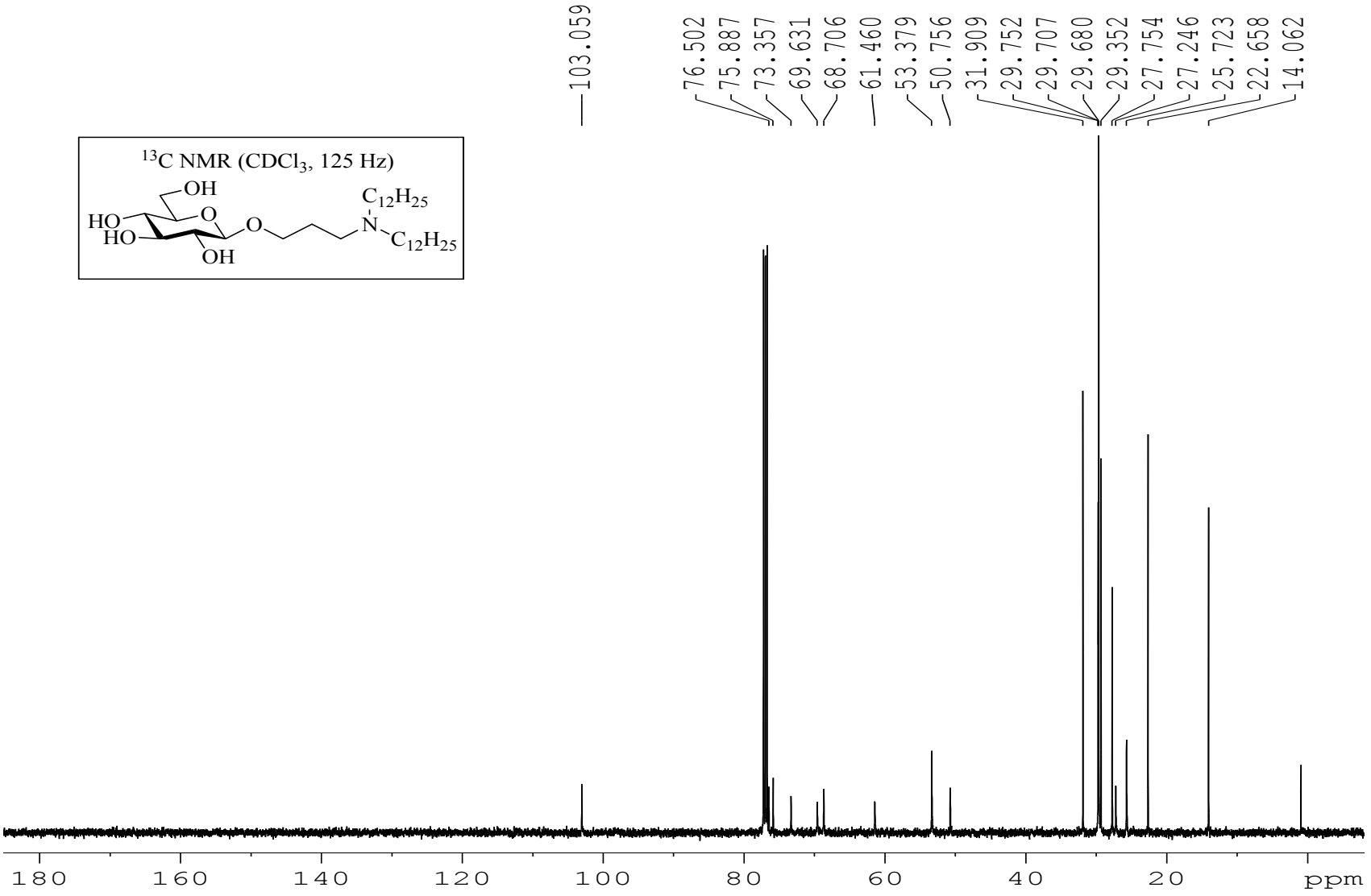

4.322
4.307
3.905
3.879
3.857
3.820
3.804
3.788
3.773
3.751
3.739
3.722
3.707
3.695
3.690
3.674
3.658
3.583
3.569
3.552
3.538
3.452
3.434
3.415
3.393
3.359
3.210
3.193
3.175
3.044
3.028
3.011
2.210
2.051
2.039
1.564
1.549
1.530
1H NMR (CDCl3, 500 Hz)
HO
HO
RO
O
O
OR
H3C
CH3
N
CH3
I-
R=C16H33-n
8
7
6
5
4
3
2
1
0
ppm
1.00
2.94
2.11
4.11
1.21
10.83
1.15
1.05
1.06
1.07
4.02
52.00
5.86

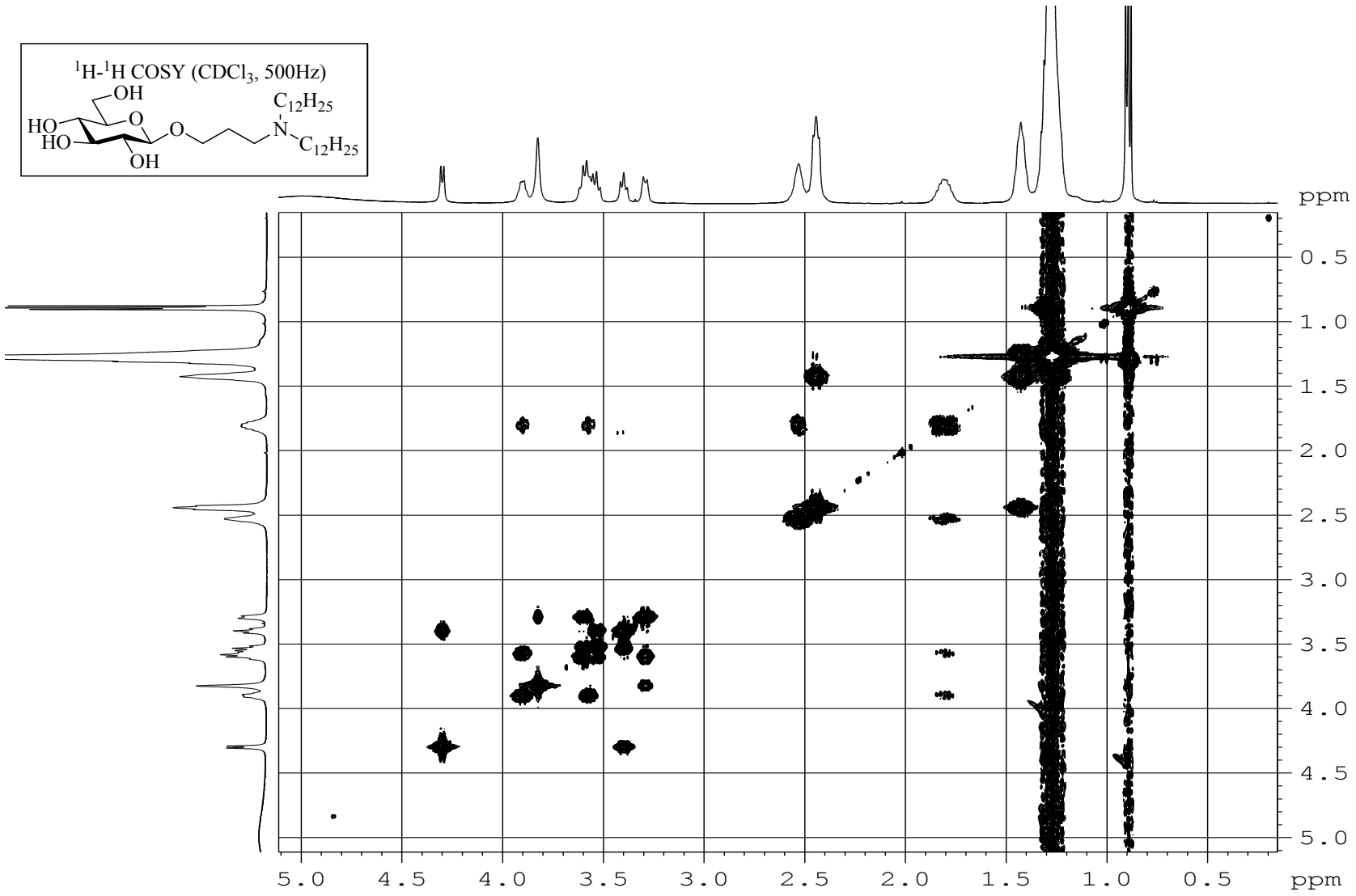

104.039
84.251
81.983
75.722
73.611
72.990
70.119
66.558
64.900
61.634
53.961
31.909
30.468
29.727
29.652
29.346
26.270
26.147
24.511
22.662
14.075
13C NMR (CDCl3, 125 Hz)
HO
HO
RO
OR
O
O
H3C
N
CH3
CH3
I-
R=C16H33-n
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

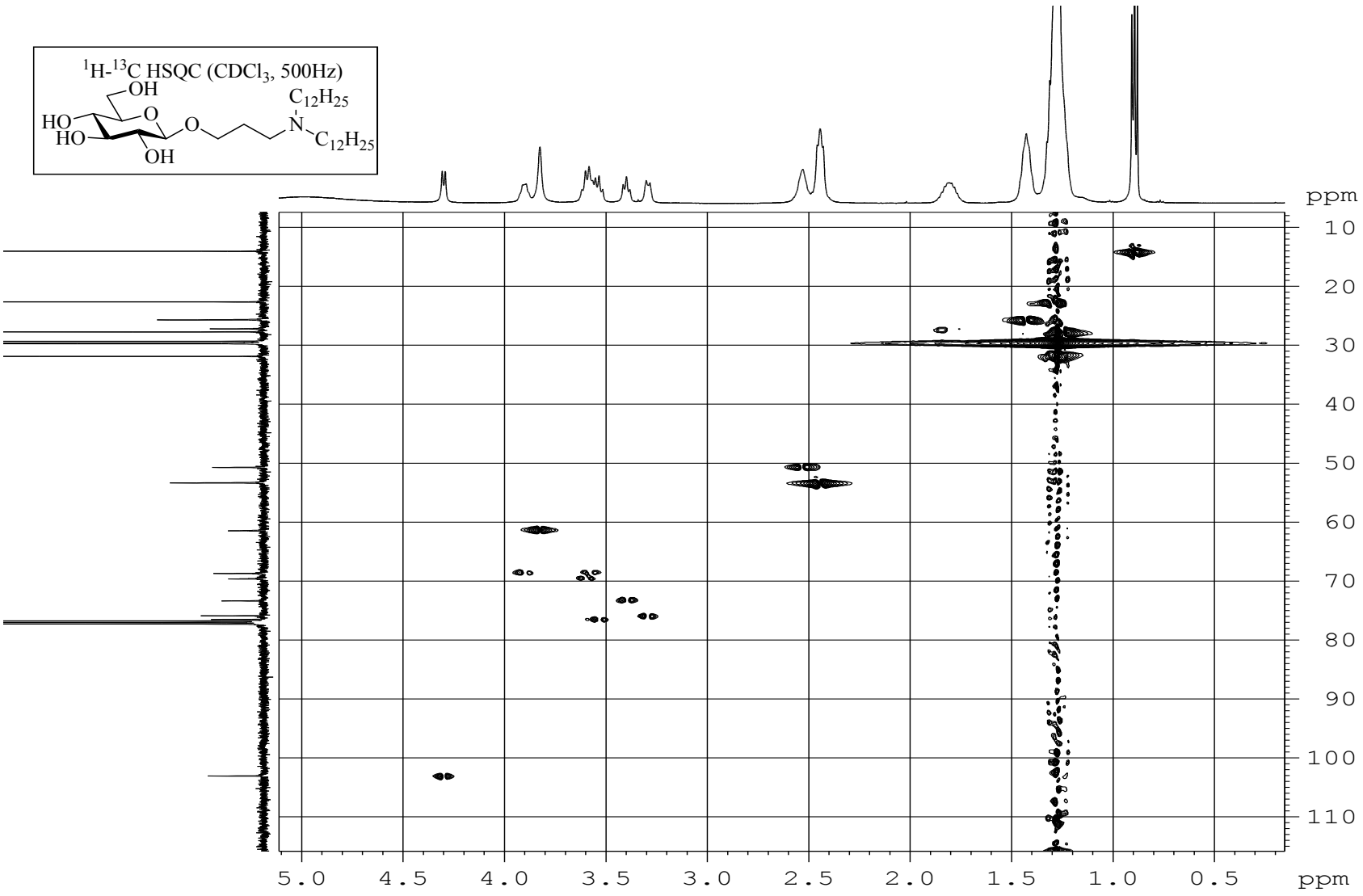

^{1}H-^{1}H COSY (CDCl$_3$, 500Hz)
HO
HO
RO
OR
O
O
H_3C
CH_3
N
CH_3
I^-
R=$C_{16}H_{33}$-*n*
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

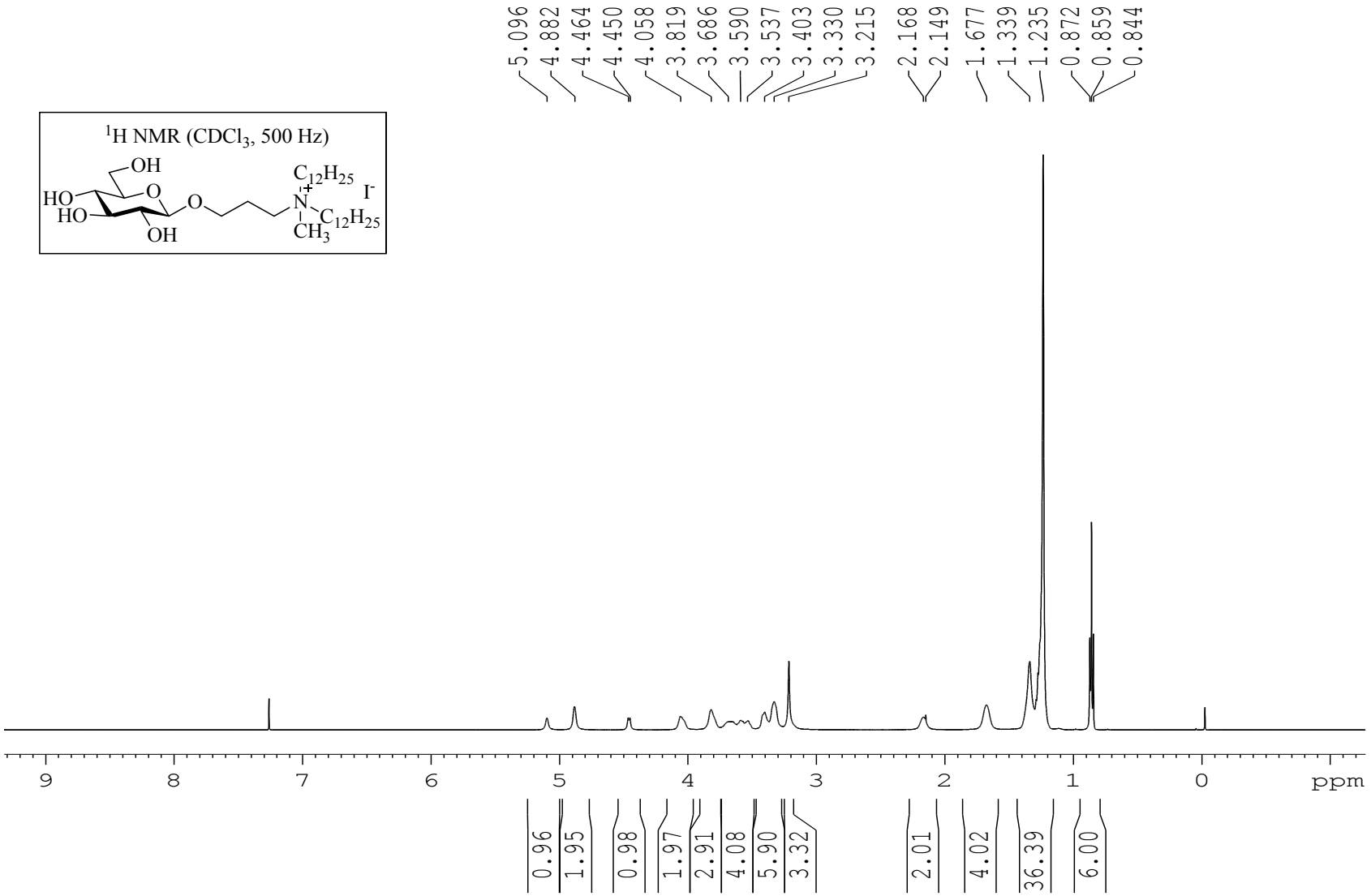

^{1}H-^{13}C HSQC (CDCl$_3$, 500Hz)
HO
HO
RO
OR
O
O
H$_3$C
N
CH$_3$
CH$_3$
I$^-$
R=C$_{16}$H$_{33}$-*n*
ppm
10
20
30
40
50
60
70
80
90
100
110
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

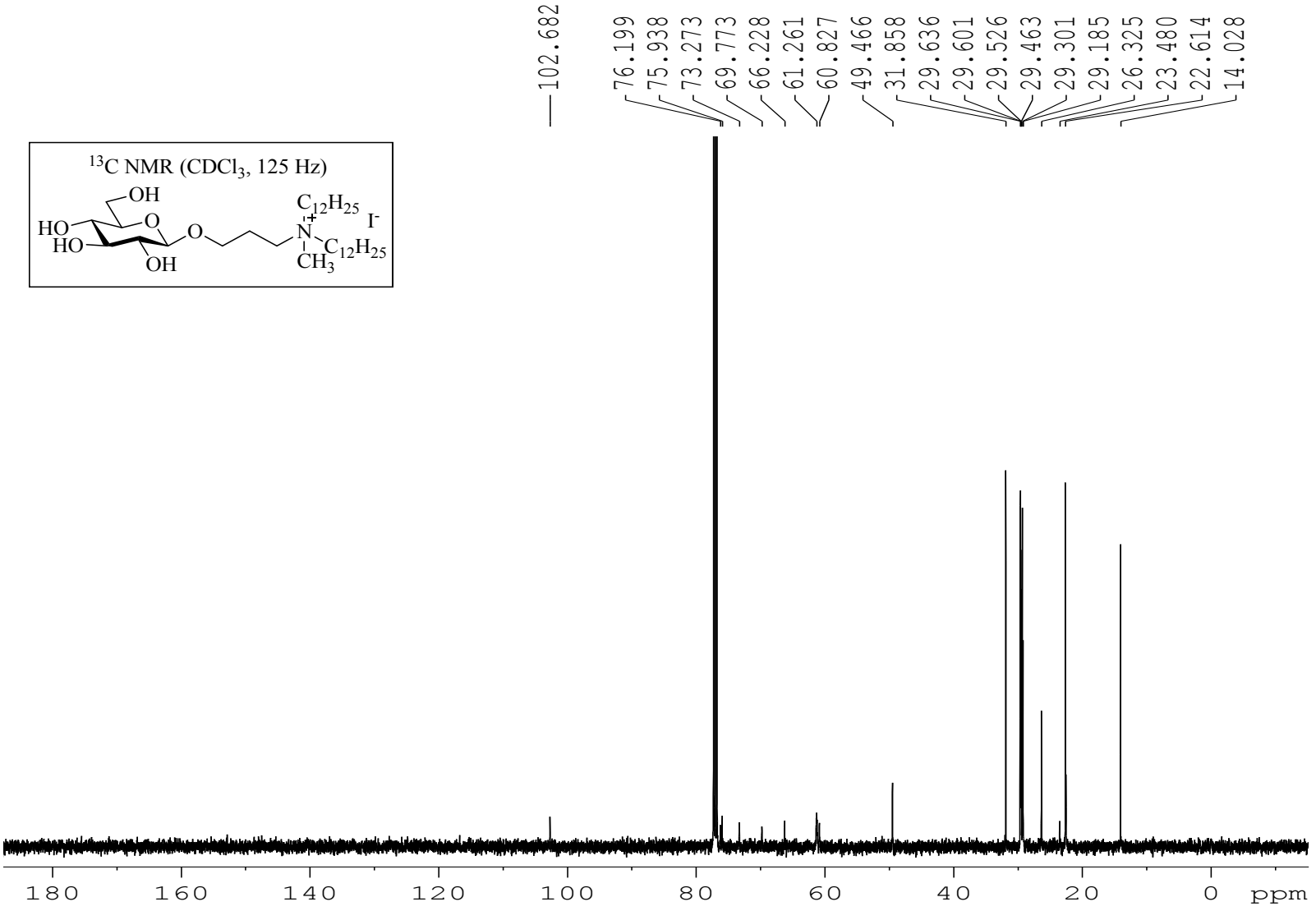

4.307
4.292
3.893
3.826
3.618
3.600
3.583
3.552
3.534
3.516
3.415
3.398
3.382
3.301
3.283
2.530
2.457
2.443
2.428
2.017
1.815
1.427
1.325
0.906
0.893
0.879
^{1}H NMR (CDCl$_3$, 500 Hz)
OH
HO
HO
O
O
OH
$C_{12}H_{25}$
N
$C_{12}H_{25}$
8
7
6
5
4
3
2
1
0
ppm
1.06
1.05
1.81
2.88
0.99
0.95
1.91
3.72
1.81
4.04
35.84
6.00

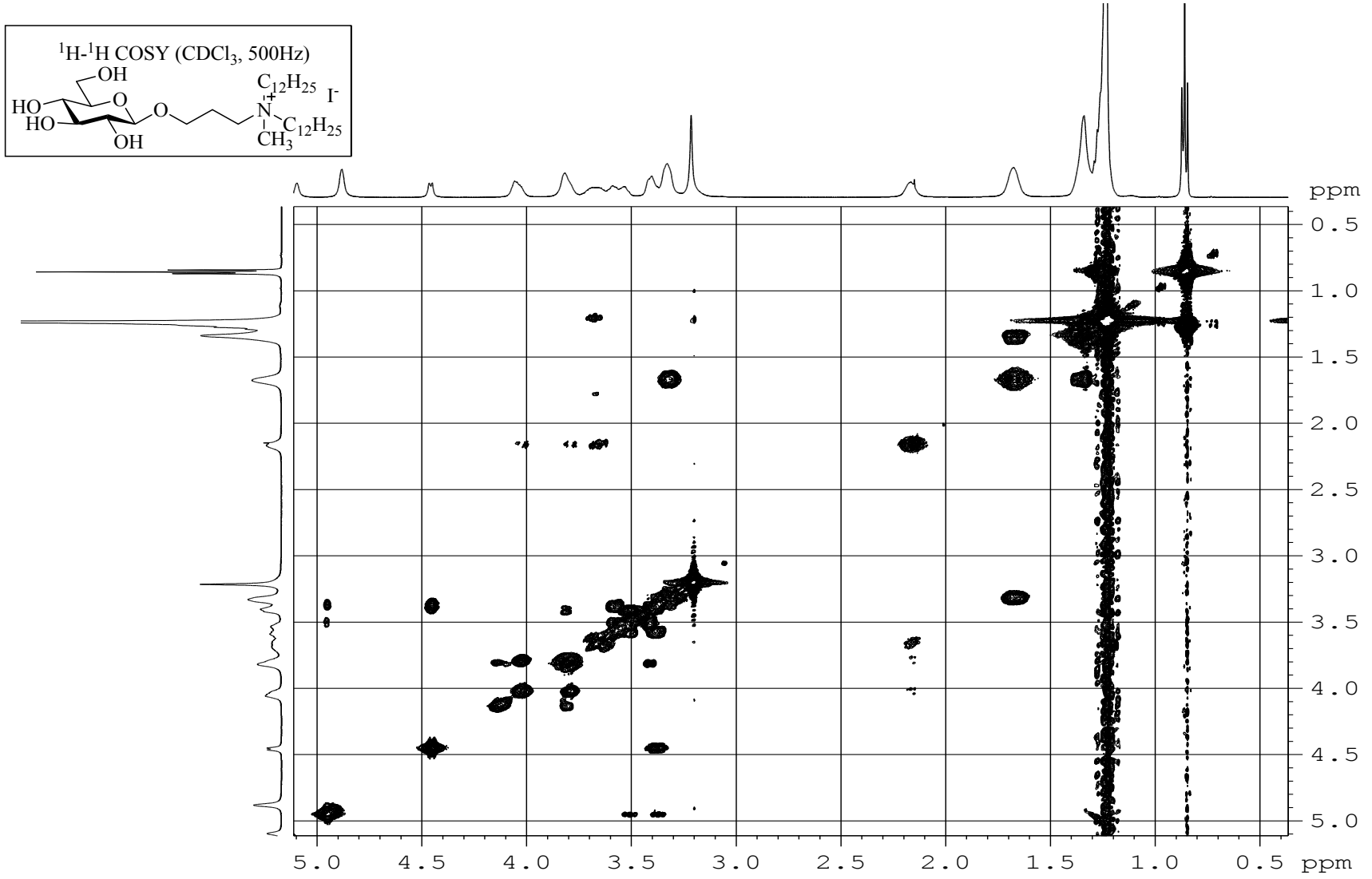

103.059
76.502
75.887
73.357
69.631
68.706
61.460
53.379
50.756
31.909
29.752
29.707
29.680
29.352
27.754
27.246
25.723
22.658
14.062
13C NMR (CDCl3, 125 Hz)
OH
HO
HO
O
O
OH
C12H25
N
C12H25
180
160
140
120
100
80
60
40
20
ppm

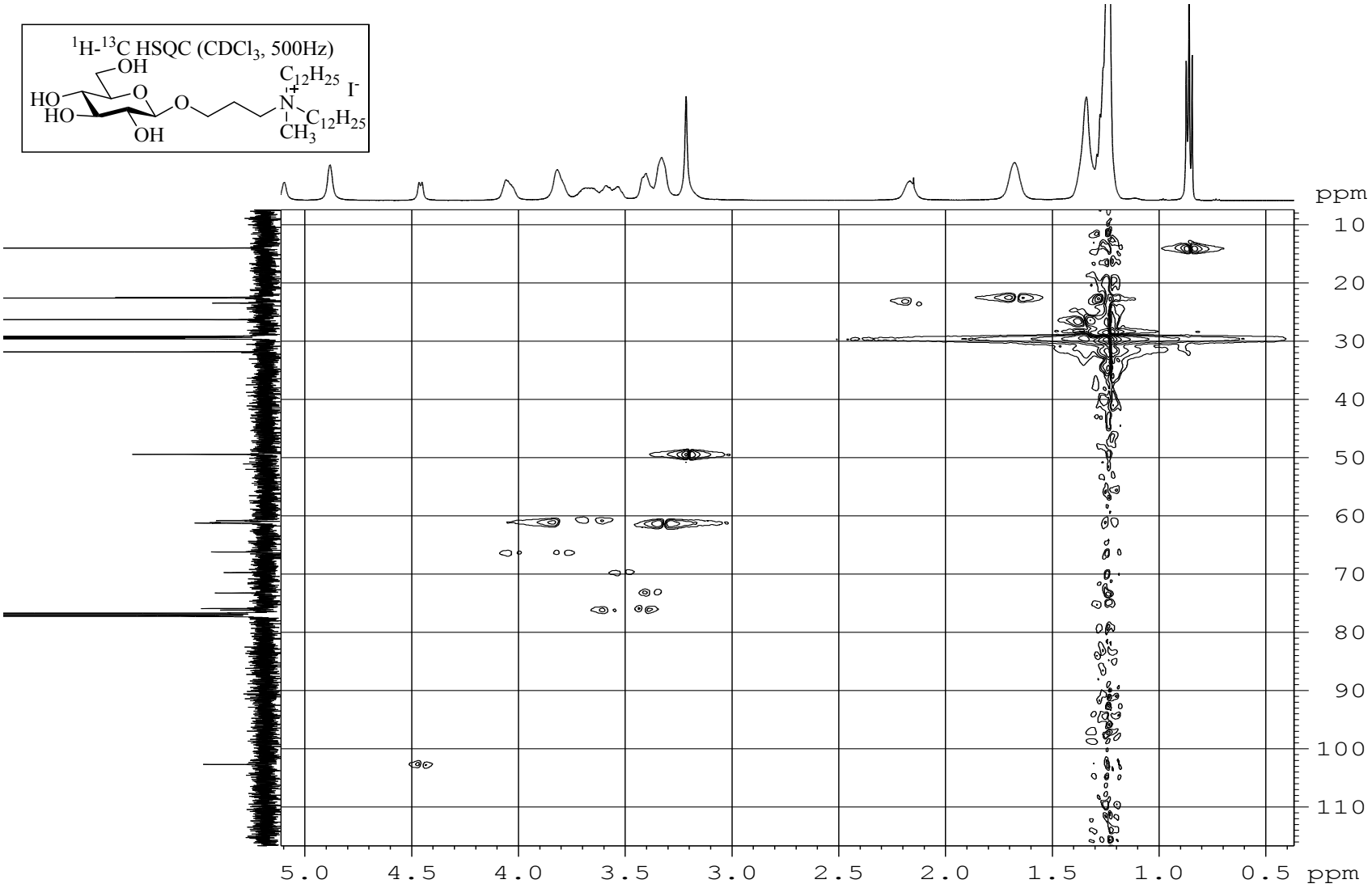

^{1}H-^{1}H COSY (CDCl$_3$, 500Hz)
OH
HO
HO
O
O
OH
$C_{12}H_{25}$
N
$C_{12}H_{25}$
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

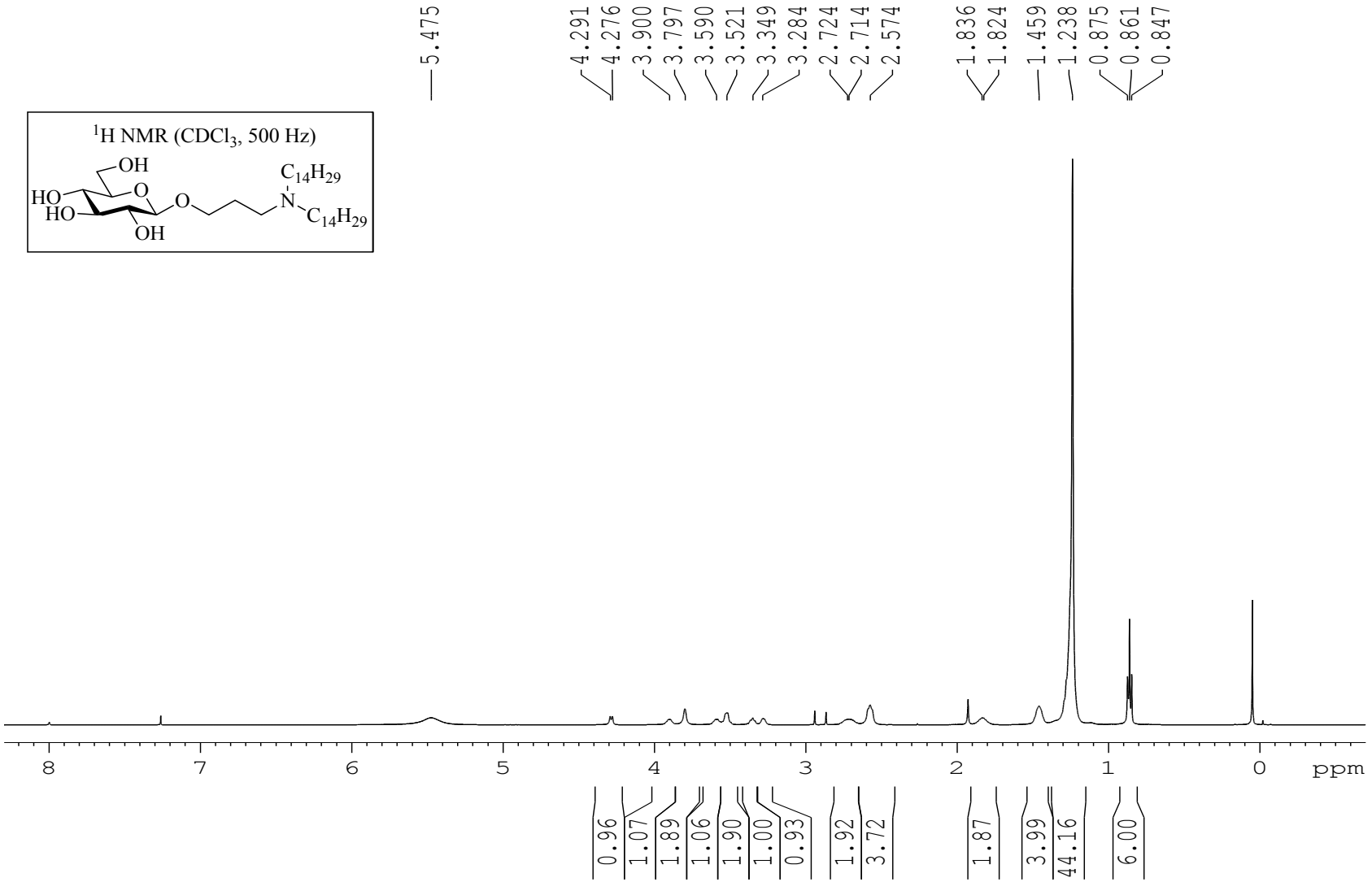

^{1}H-^{13}C HSQC ($CDCl_3$, 500Hz)
OH
HO
HO
OH
O
O
$C_{12}H_{25}$
N
$C_{12}H_{25}$
ppm
10
20
30
40
50
60
70
80
90
100
110
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

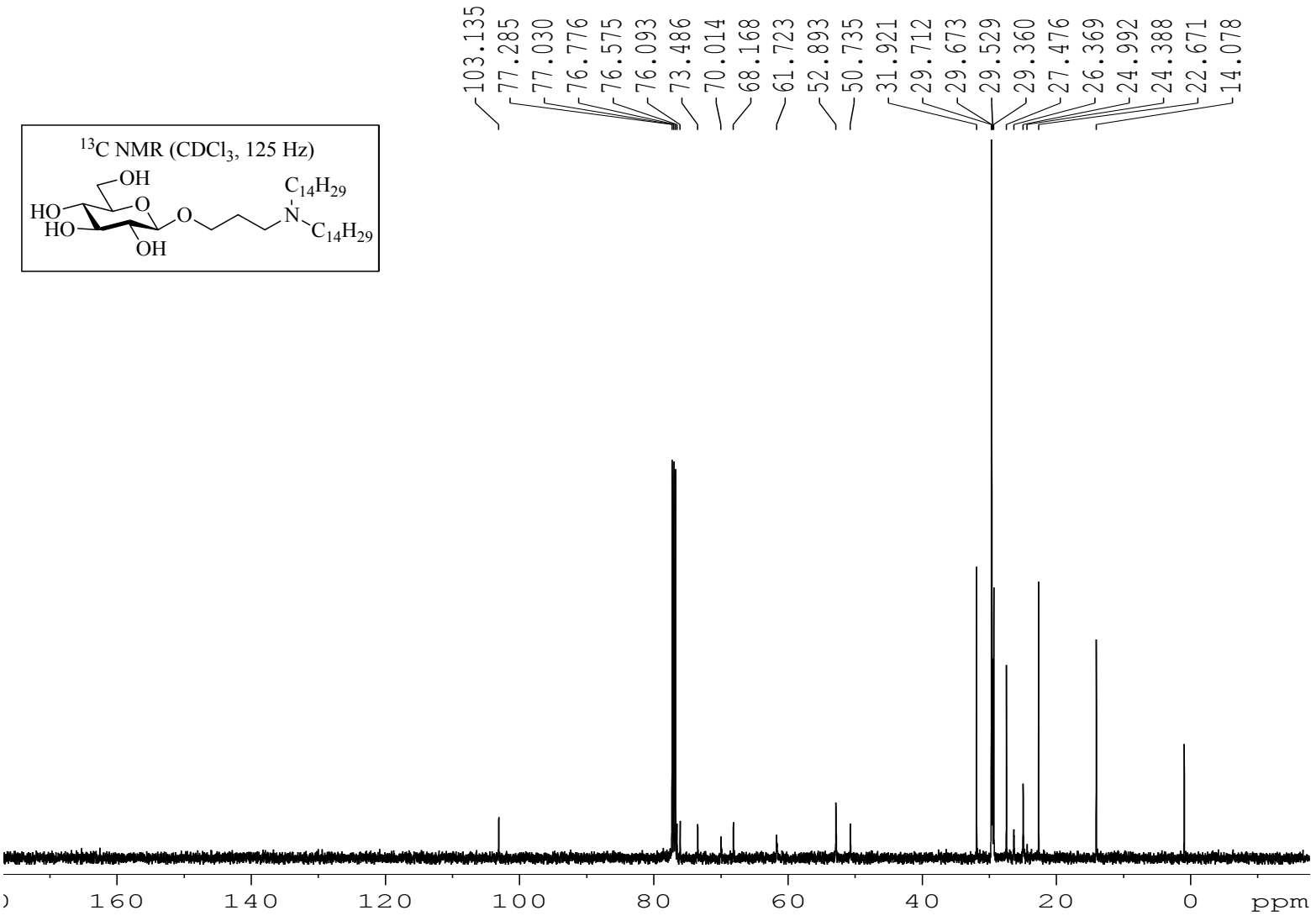

5.096
4.882
4.464
4.450
4.058
3.819
3.686
3.590
3.537
3.403
3.330
3.215
2.168
2.149
1.677
1.339
1.235
0.872
0.859
0.844
^{1}H NMR ($CDCl_3$, 500 Hz)
OH
HO
HO
O
O
OH
$C_{12}H_{25}$
N
I^-
$C_{12}H_{25}$
CH_3
9
8
7
6
5
4
3
2
1
0
ppm
0.96
1.95
0.98
1.97
2.91
4.08
5.90
3.32
2.01
4.02
36.39
6.00

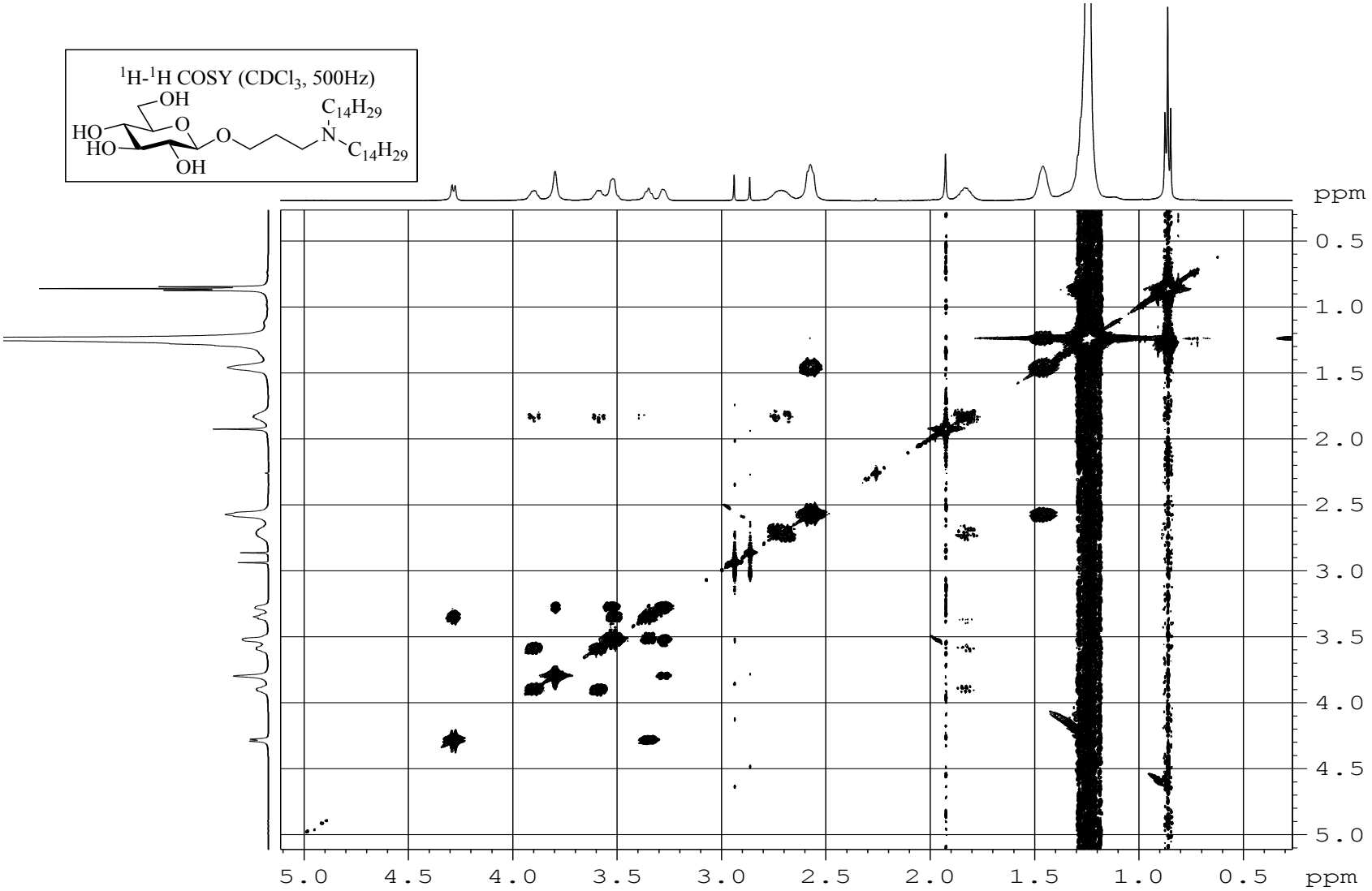

102.682
76.199
75.938
73.273
69.773
66.228
61.261
60.827
49.466
31.858
29.636
29.601
29.526
29.463
29.301
29.185
26.325
23.480
22.614
14.028
13C NMR (CDCl3, 125 Hz)
OH
HO
HO
O
O
OH
C12H25
N+
I-
C12H25
CH3
180
160
140
120
100
80
60
40
20
0
ppm

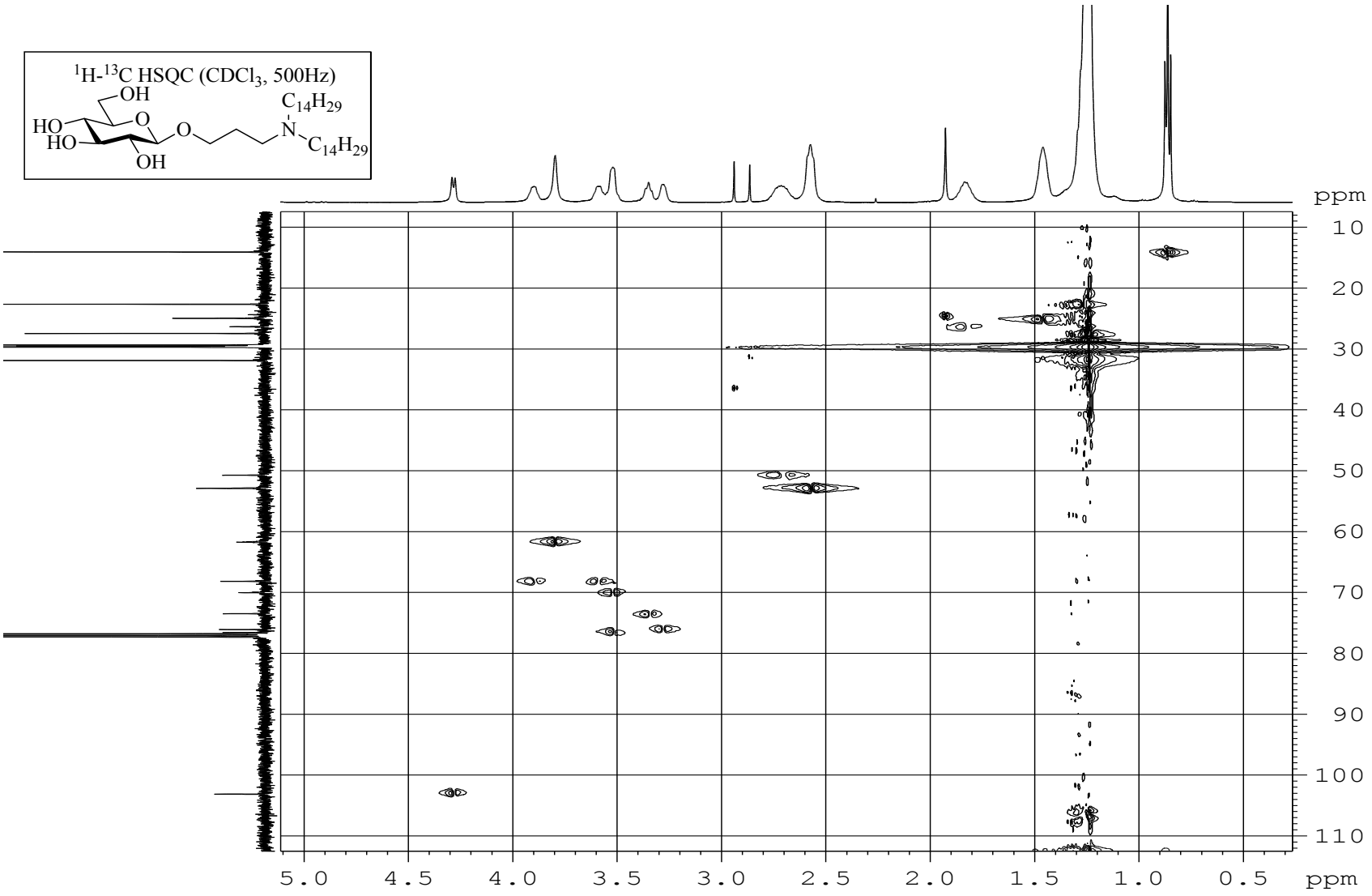

^{1}H-^{1}H COSY (CDCl$_3$, 500Hz)
OH
HO
HO
O
O
OH
$C_{12}H_{25}$
N$^+$
$C_{12}H_{25}$
CH_3
I$^-$
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

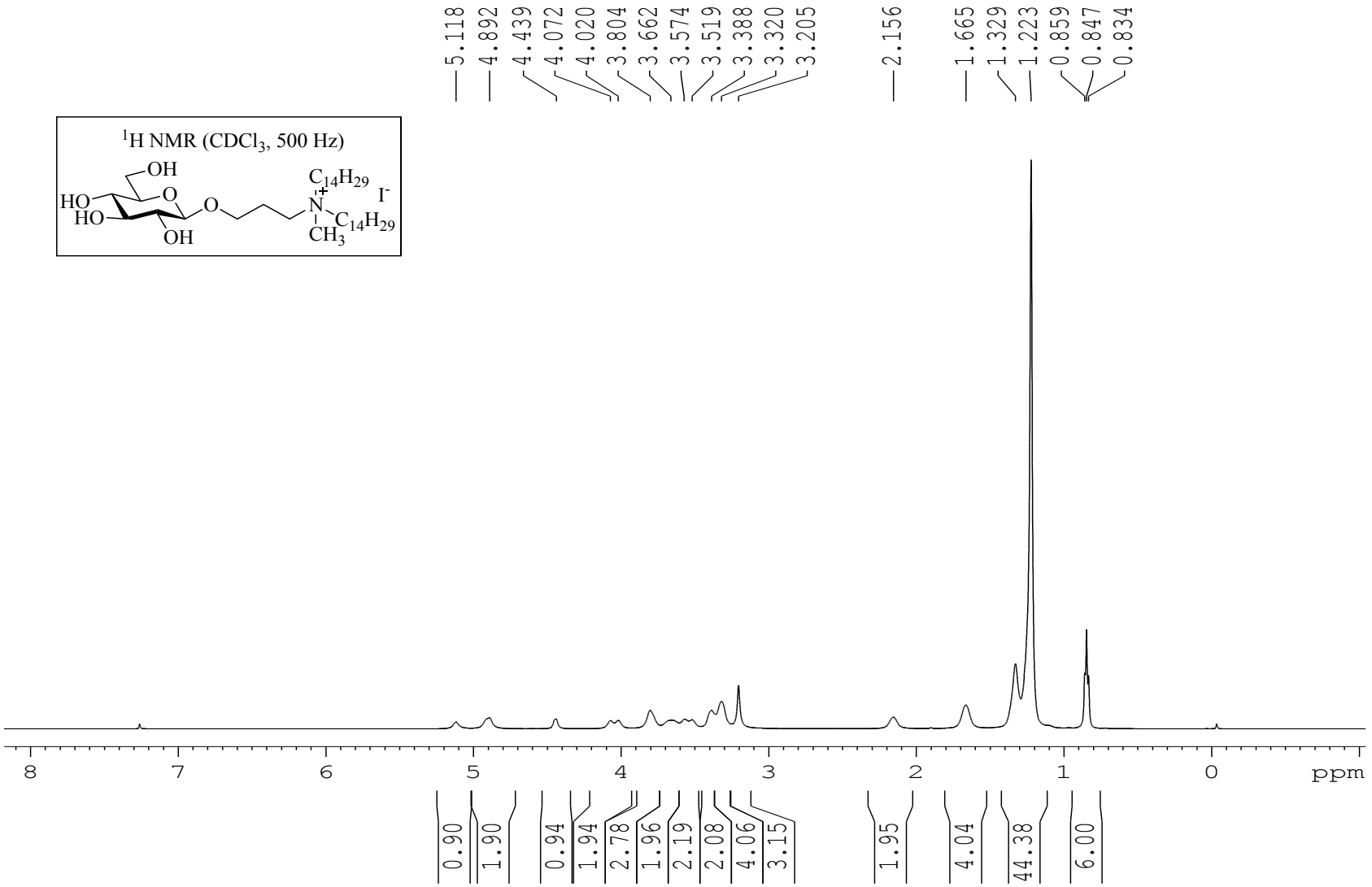

^{1}H-^{13}C HSQC (CDCl$_3$, 500Hz)
OH
HO
HO
O
O
OH
$C_{12}H_{25}$
I^-
N
$C_{12}H_{25}$
CH_3
ppm
10
20
30
40
50
60
70
80
90
100
110
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

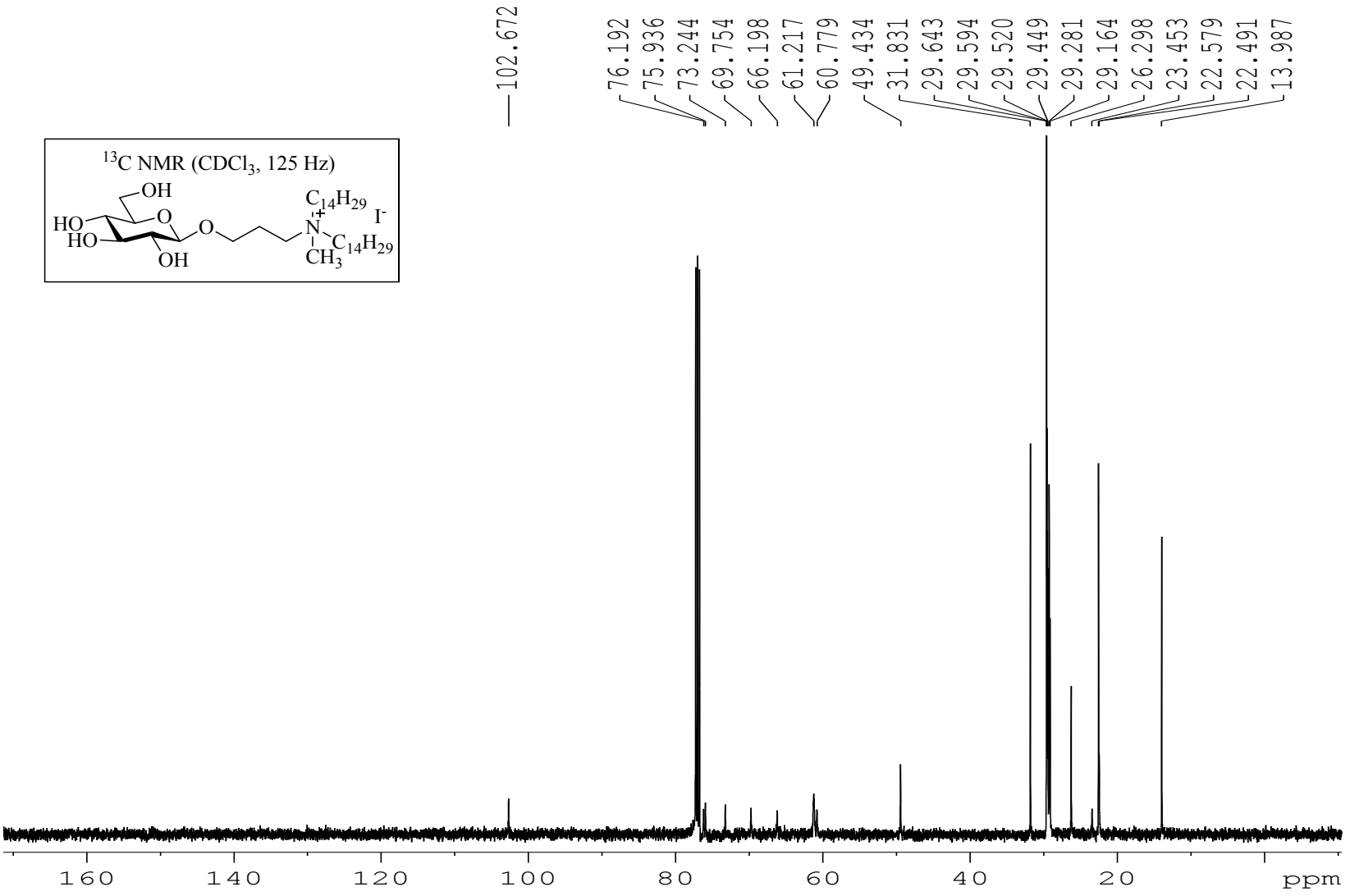

5.475
4.291
4.276
3.900
3.797
3.590
3.521
3.349
3.284
2.724
2.714
2.574
1.836
1.824
1.459
1.238
0.875
0.861
0.847
^{1}H NMR ($CDCl_3$, 500 Hz)
OH
HO
HO
O
OH
O
$C_{14}H_{29}$
N
$C_{14}H_{29}$
8
7
6
5
4
3
2
1
0
ppm
0.96
1.07
1.89
1.06
1.90
1.00
0.93
1.92
3.72
1.87
3.99
44.16
6.00

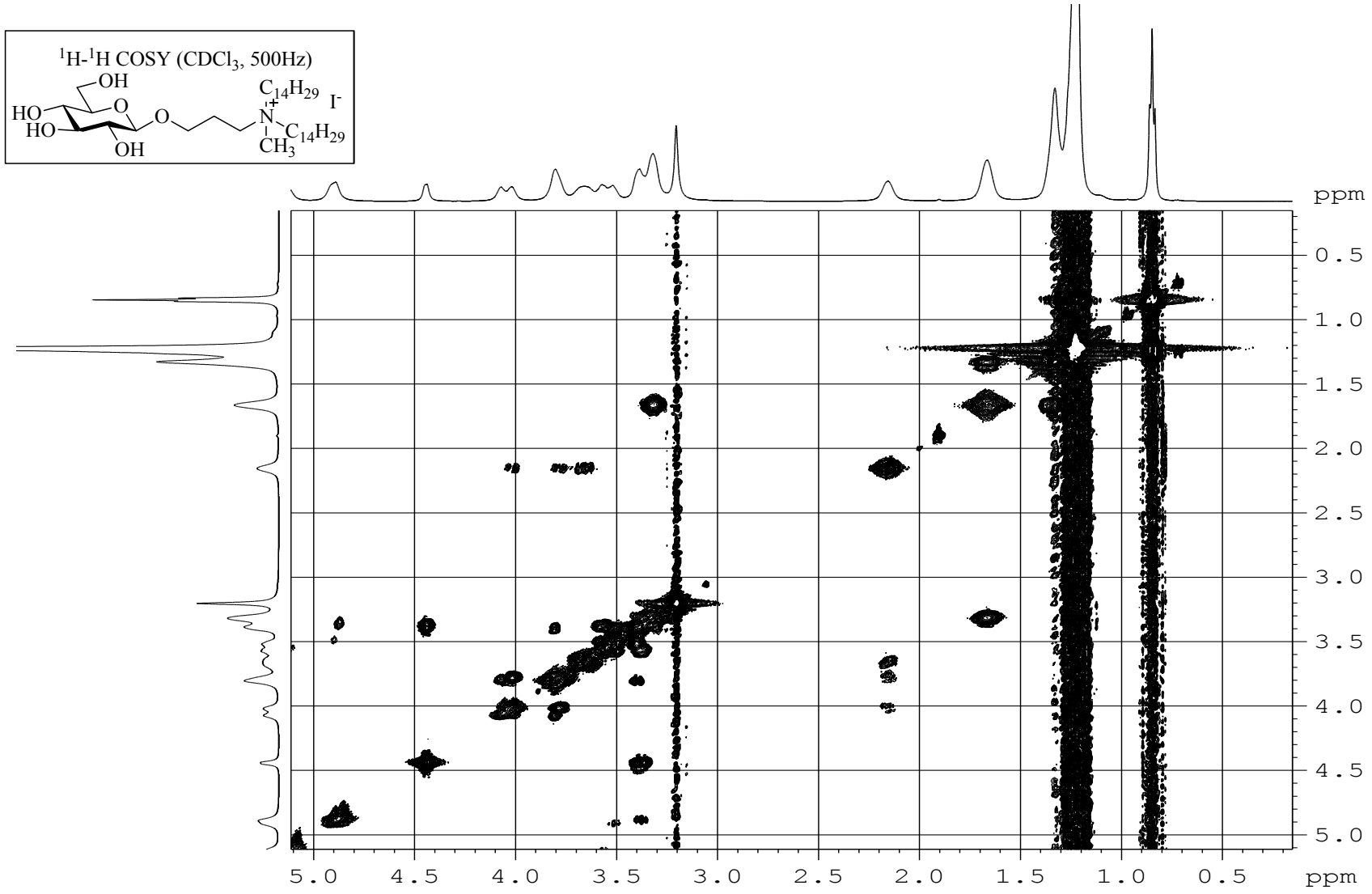

103.135
77.285
77.030
76.776
76.575
76.093
73.486
70.014
68.168
61.723
52.893
50.735
31.921
29.712
29.673
29.529
29.360
27.476
26.369
24.992
24.388
22.671
14.078
13C NMR (CDCl3, 125 Hz)
OH
HO
HO
O
OH
O
C14H29
N
C14H29
160
140
120
100
80
60
40
20
0
ppm

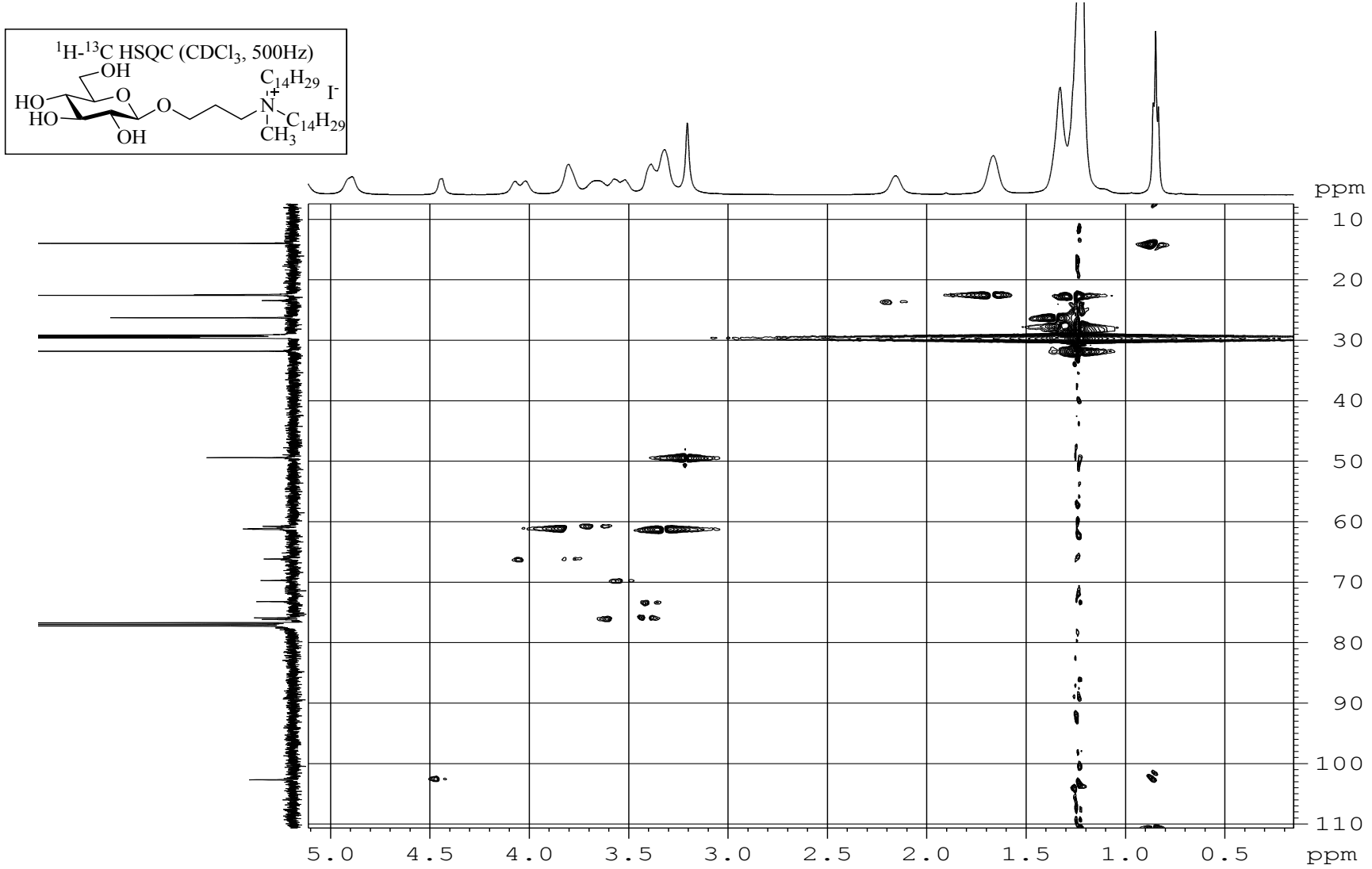

^{1}H-^{1}H COSY ($CDCl_3$, 500Hz)
OH
HO
HO
O
O
OH
$C_{14}H_{29}$
N
$C_{14}H_{29}$
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

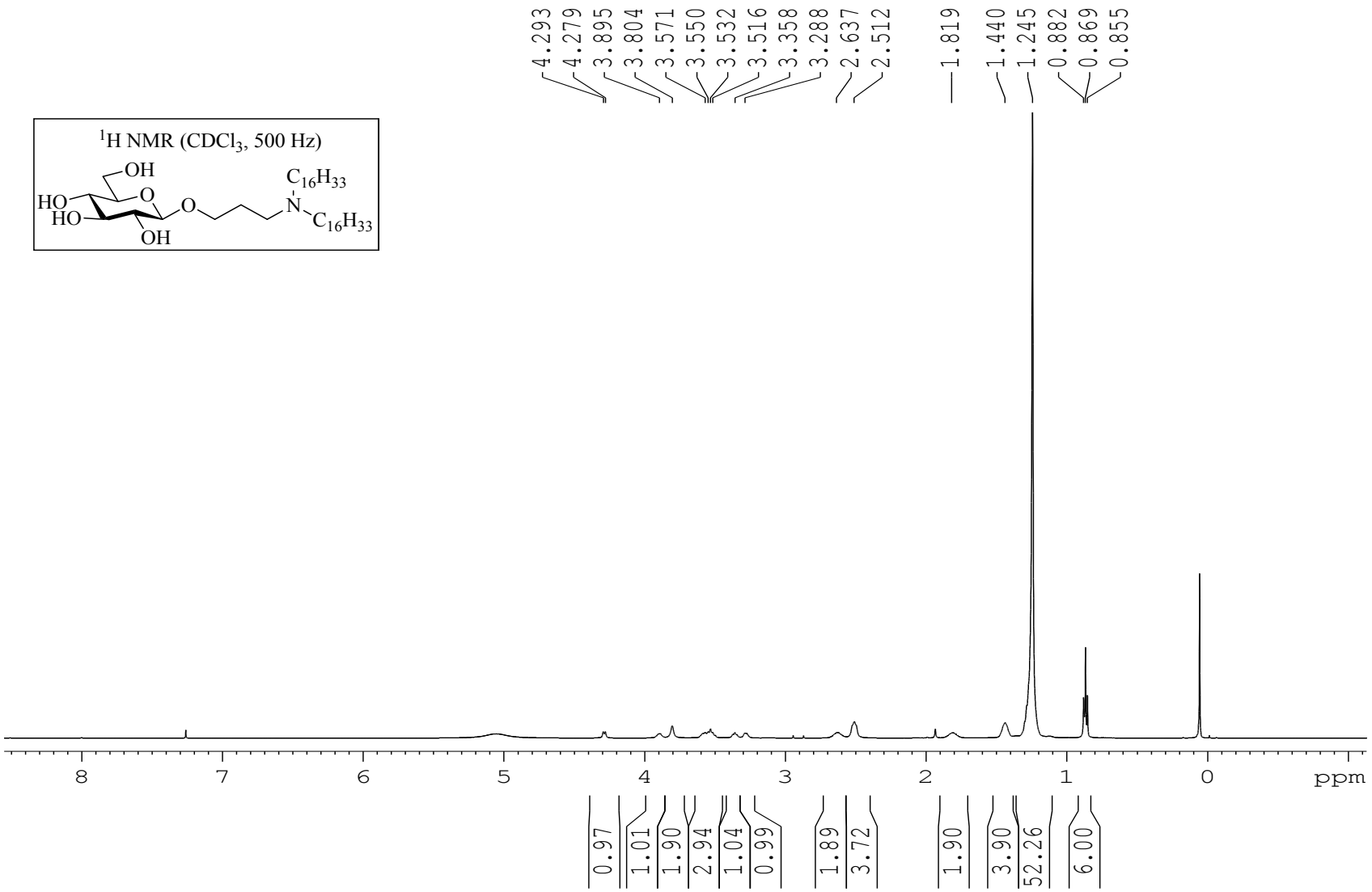

^{1}H-^{13}C HSQC (CDCl$_3$, 500Hz)
OH
HO
HO
O
O
OH
$C_{14}H_{29}$
N
$C_{14}H_{29}$
ppm
10
20
30
40
50
60
70
80
90
100
110
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

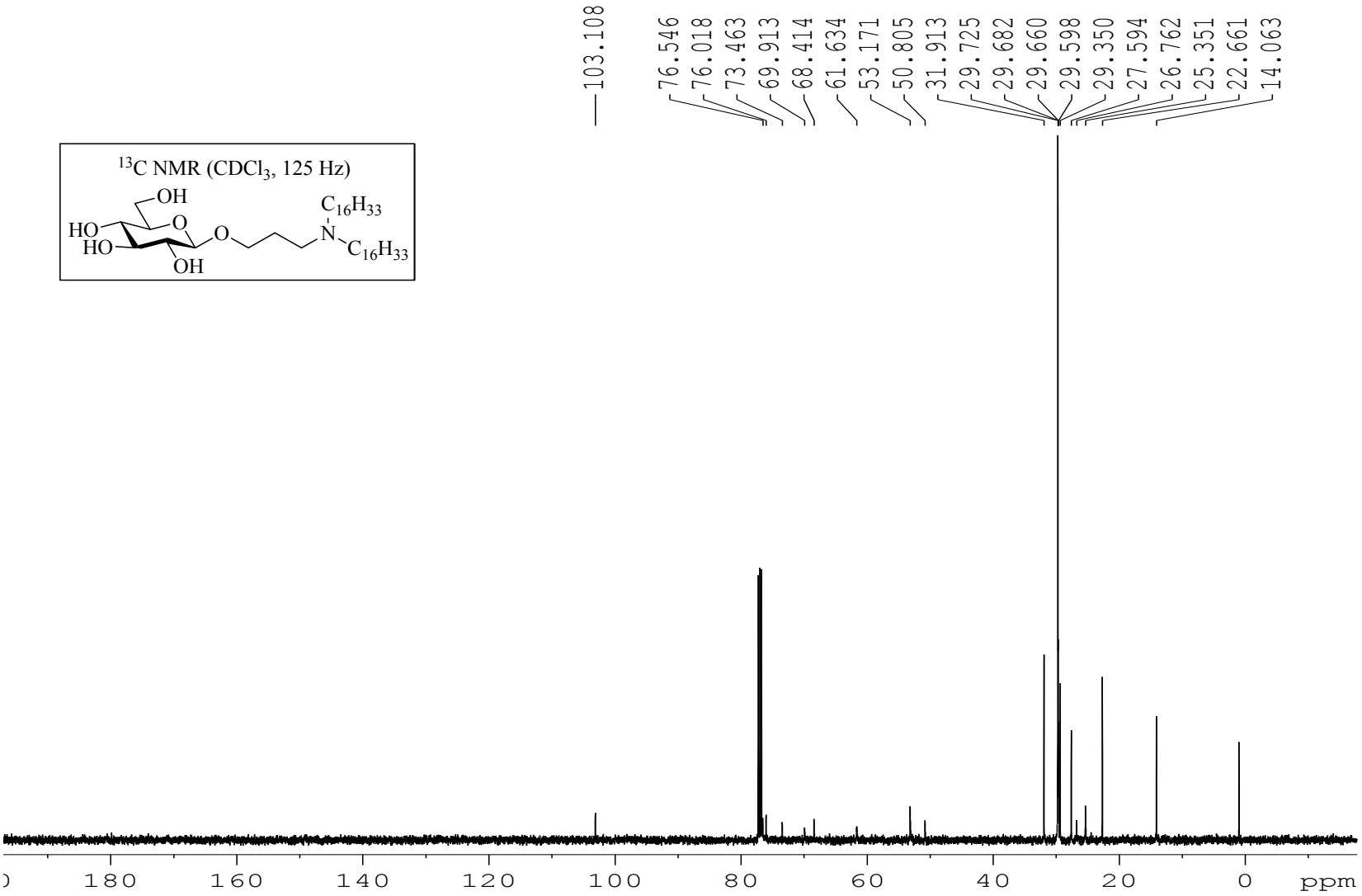

5.118
4.892
4.439
4.072
4.020
3.804
3.662
3.574
3.519
3.388
3.320
3.205
2.156
1.665
1.329
1.223
0.859
0.847
0.834
1H NMR (CDCl3, 500 Hz)
OH
HO
HO
O
OH
O
C14H29
N+
CH3
C14H29
I-
8
7
6
5
4
3
2
1
0
ppm
0.90
1.90
0.94
1.94
2.78
1.96
2.19
2.08
4.06
3.15
1.95
4.04
44.38
6.00

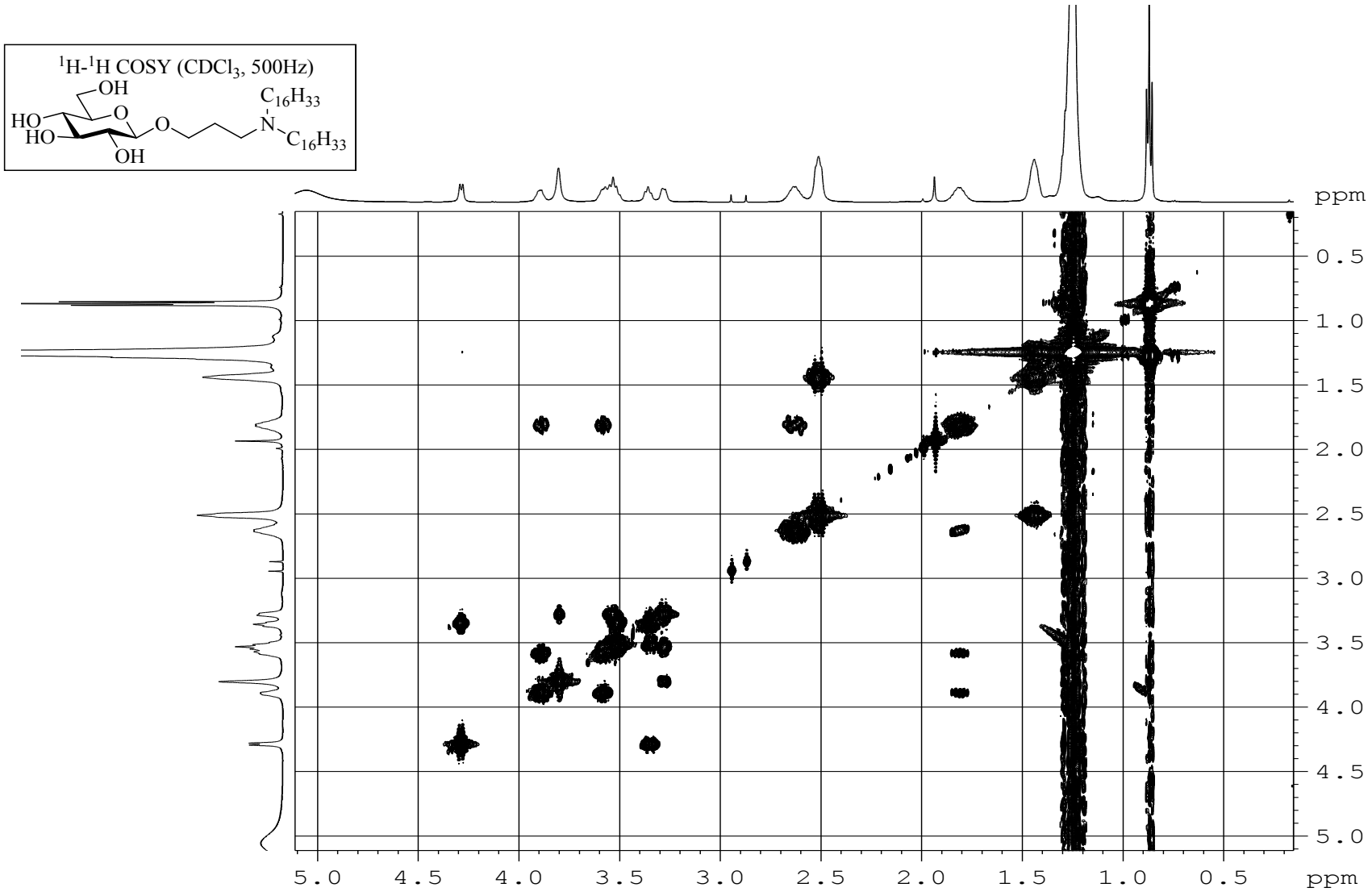

102.672
76.192
75.936
73.244
69.754
66.198
61.217
60.779
49.434
31.831
29.643
29.594
29.520
29.449
29.281
29.164
26.298
23.453
22.579
22.491
13.987
13C NMR (CDCl3, 125 Hz)
OH
HO
HO
O
O
OH
C14H29
N+
I-
CH3
C14H29
160
140
120
100
80
60
40
20
ppm

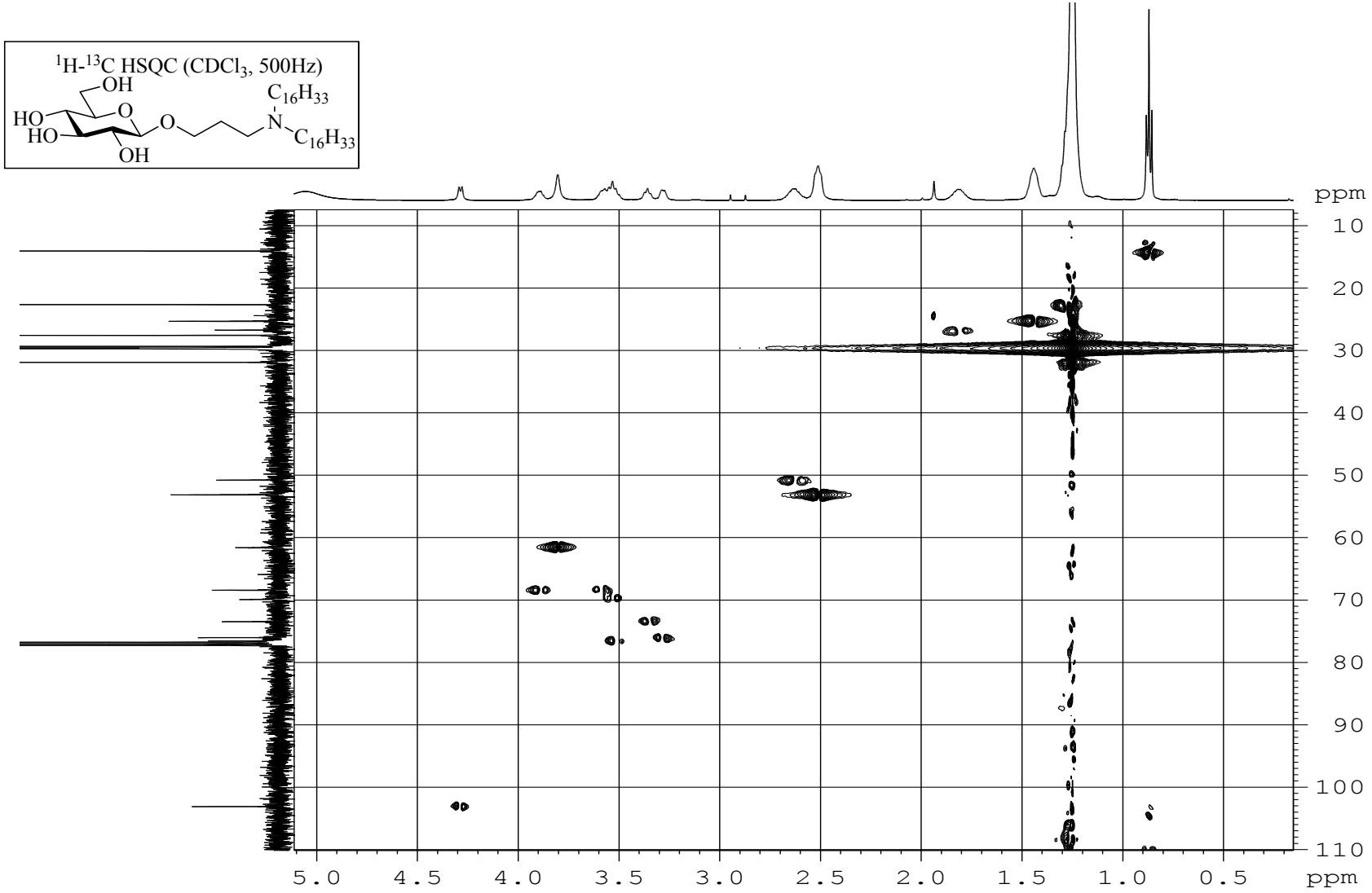

^{1}H-^{1}H COSY (CDCl$_3$, 500Hz)
OH
HO
HO
O
O
OH
$C_{14}H_{29}$
N$^+$
CH_3
$C_{14}H_{29}$
I$^-$
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

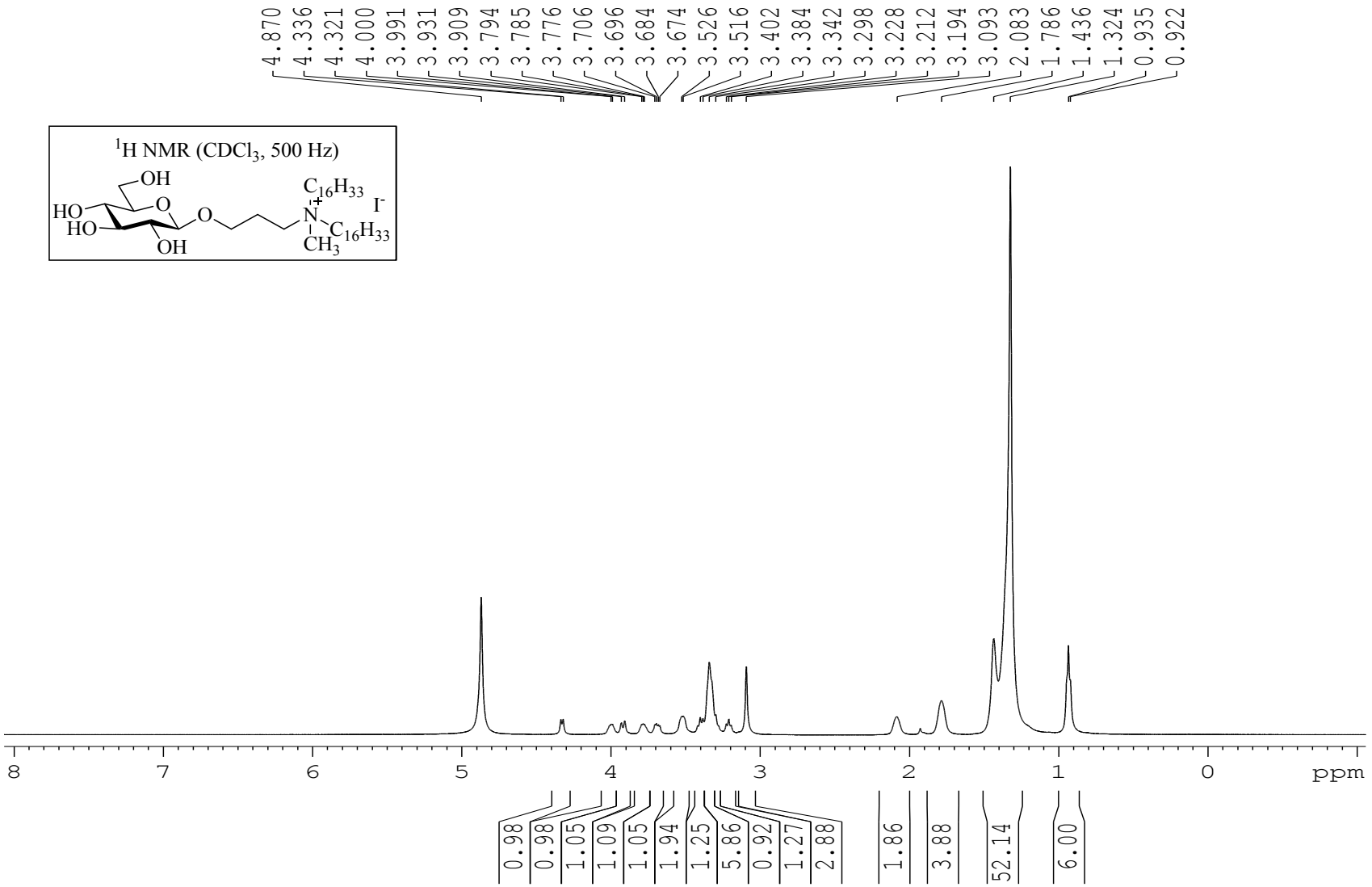

^{1}H-^{13}C HSQC ($CDCl_3$, 500Hz)
OH
HO
HO
O
O
OH
$C_{14}H_{29}$
N
I^-
$C_{14}H_{29}$
CH_3
ppm
10
20
30
40
50
60
70
80
90
100
110
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

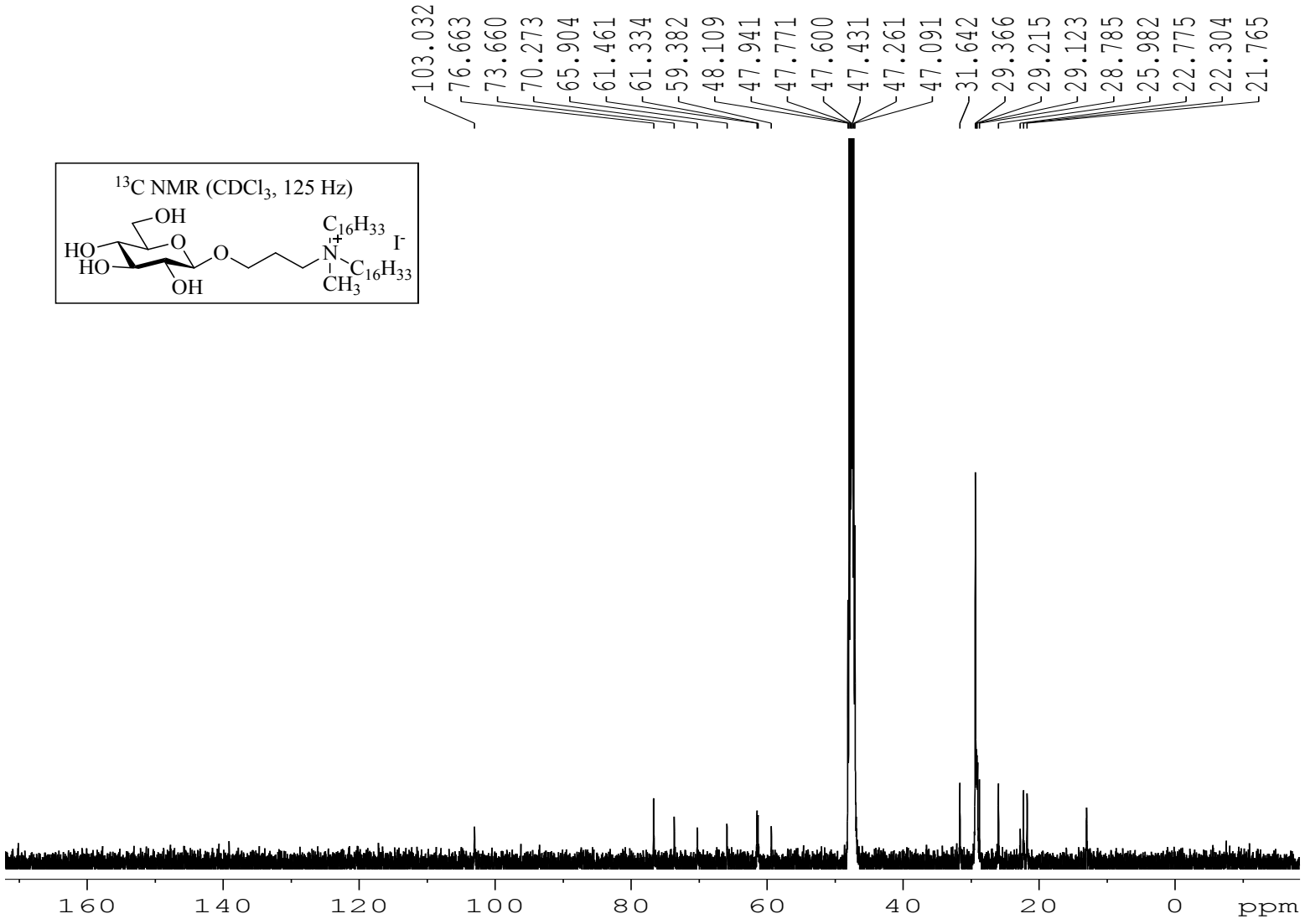

4.293
4.279
3.895
3.804
3.571
3.550
3.532
3.516
3.358
3.288
2.637
2.512
1.819
1.440
1.245
0.882
0.869
0.855
^{1}H NMR ($CDCl_3$, 500 Hz)
OH
HO
HO
O
O
OH
$C_{16}H_{33}$
N
$C_{16}H_{33}$
8
7
6
5
4
3
2
1
0
ppm
0.97
1.01
1.90
2.94
1.04
0.99
1.89
3.72
1.90
3.90
52.26
6.00

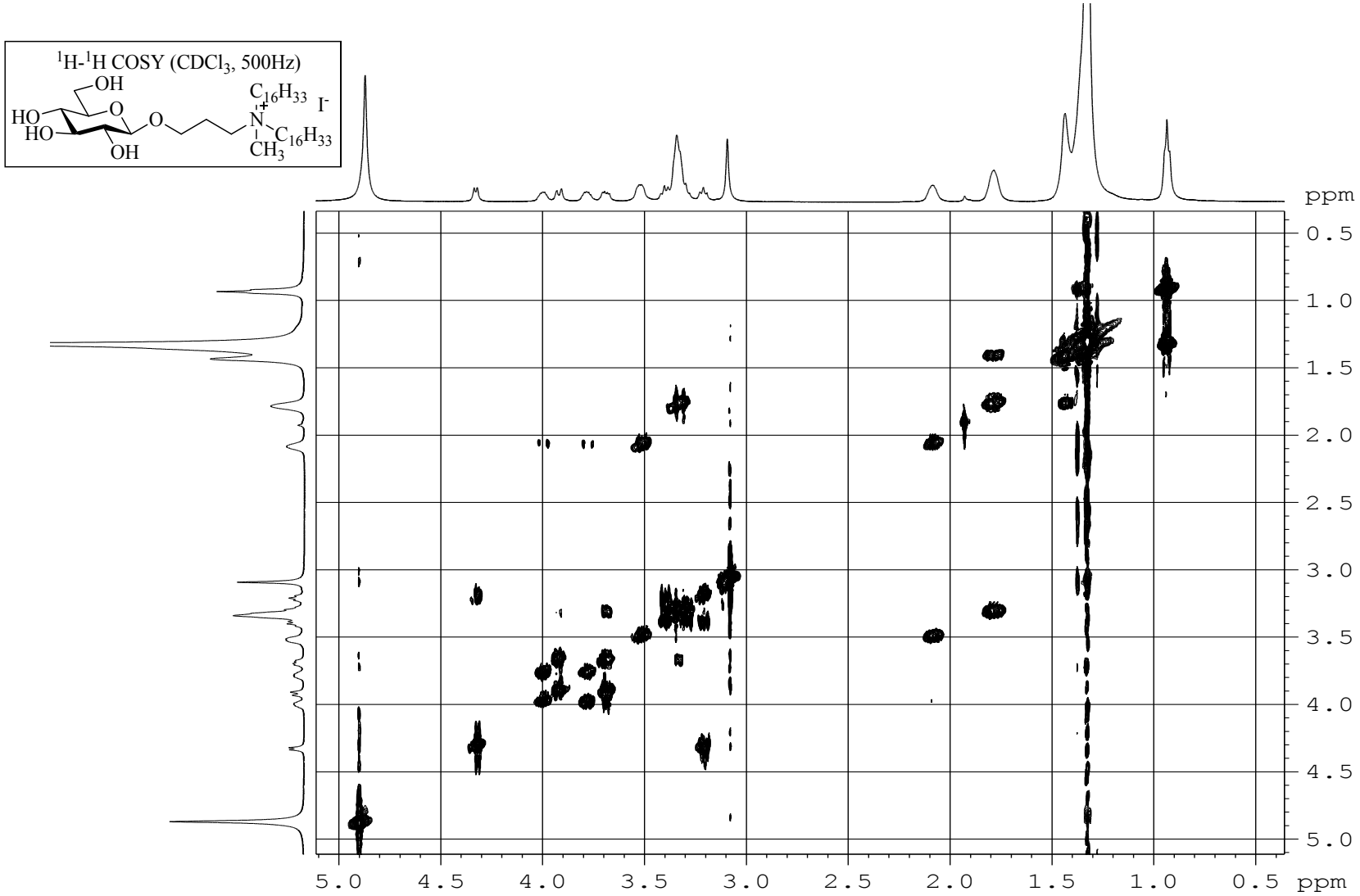

103.108
76.546
76.018
73.463
69.913
68.414
61.634
53.171
50.805
31.913
29.725
29.682
29.660
29.598
29.350
27.594
26.762
25.351
22.661
14.063
13C NMR (CDCl3, 125 Hz)
OH
HO
HO
O
O
OH
C16H33
N
C16H33
180
160
140
120
100
80
60
40
20
0
ppm

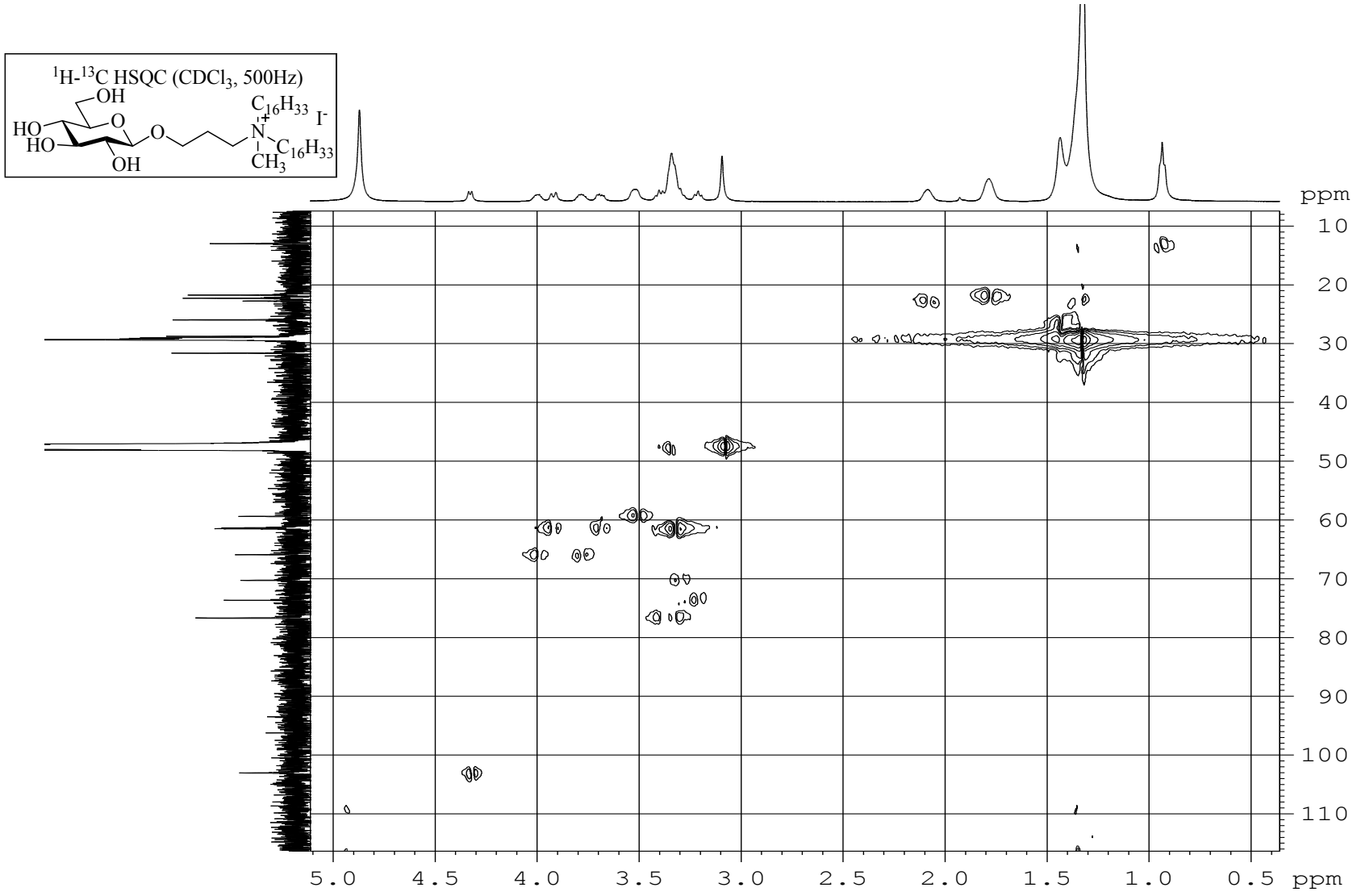

^{1}H-^{1}H COSY (CDCl$_3$, 500Hz)
OH
HO
HO
O
O
OH
$C_{16}H_{33}$
N
$C_{16}H_{33}$
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
ppm


^{1}H-^{13}C HSQC (CDCl$_3$, 500Hz)
OH
HO
HO
O
O
OH
$C_{16}H_{33}$
N
$C_{16}H_{33}$
ppm
10
20
30
40
50
60
70
80
90
100
110
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm


4.870
4.336
4.321
4.000
3.991
3.931
3.909
3.794
3.785
3.776
3.706
3.696
3.684
3.674
3.526
3.516
3.402
3.384
3.342
3.298
3.228
3.212
3.194
3.093
2.083
1.786
1.436
1.324
0.935
0.922
1H NMR (CDCl3, 500 Hz)
OH
HO
HO
O
OH
O
C16H33
N+
CH3
C16H33
I-
8
7
6
5
4
3
2
1
0
ppm
0.98
0.98
1.05
1.09
1.05
1.94
1.25
5.86
0.92
1.27
2.88
1.86
3.88
52.14
6.00


103.032
76.663
73.660
70.273
65.904
61.461
61.334
59.382
48.109
47.941
47.771
47.600
47.431
47.261
47.091
31.642
29.366
29.215
29.123
28.785
25.982
22.775
22.304
21.765
13C NMR (CDCl3, 125 Hz)
OH
HO
HO
O
OH
O
C16H33
N+
I-
C16H33
CH3
160
140
120
100
80
60
40
20
0
ppm


^{1}H-^{1}H COSY (CDCl$_3$, 500Hz)
OH
HO
HO
O
OH
O
$C_{16}H_{33}$
N$^+$
$C_{16}H_{33}$
CH_3
I$^-$
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm


^{1}H-^{13}C HSQC ($CDCl_3$, 500Hz)
OH
HO
HO
O
O
OH
$C_{16}H_{33}$
N+
$C_{16}H_{33}$
CH_3
I-
ppm
10
20
30
40
50
60
70
80
90
100
110
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

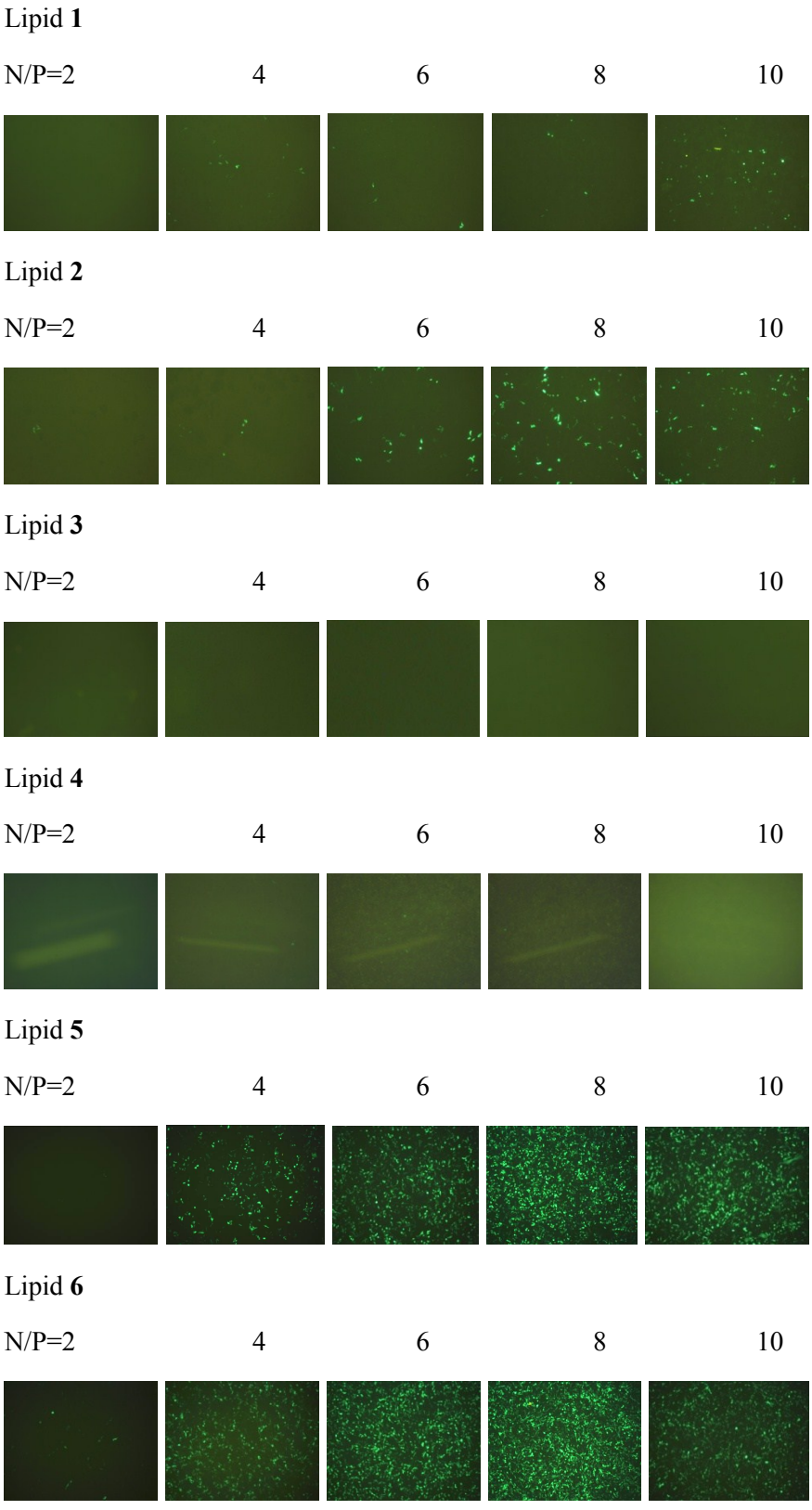
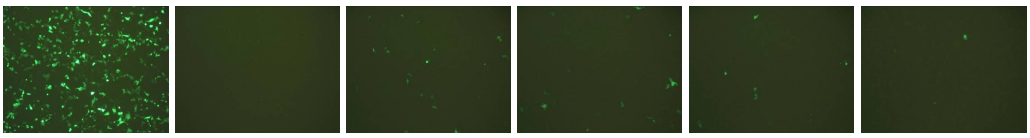
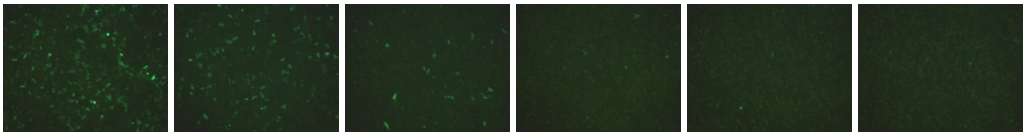


Fig. S1. The fluorescence microscope images of HEK293 cells transfected by glycolipid/DNA complexes in the other four levels of N/P ratios.



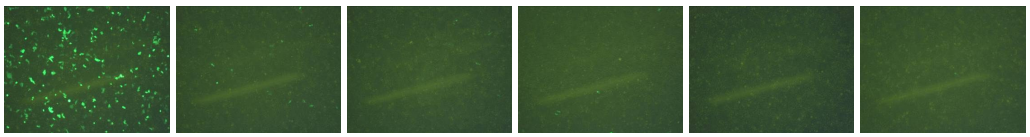
Lipo2000 N/P=2 N/P=4 N/P=6 N/P=8 N/P=10

a) Transfection efficiency of lipid **2**/DNA complexes in Hela cells under different N/P ratios.



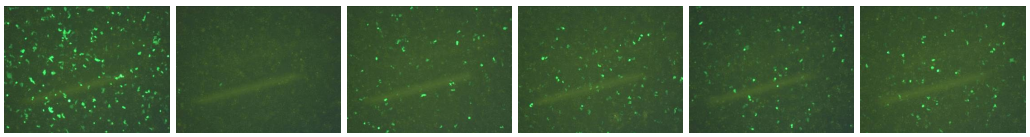
Lipo2000 N/P=2 N/P=4 N/P=6 N/P=8 N/P=10

b) Transfection efficiency of lipid **3**/DNA complexes in Hela cells under different N/P ratios.



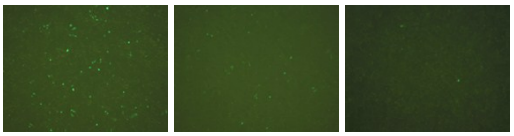
Lipo2000 N/P=2 N/P=4 N/P=6 N/P=8 N/P=10

c) Transfection efficiency of lipid **5**/DNA complexes in HepG2 cells under different N/P ratios.



Lipo2000 N/P=2 N/P=4 N/P=6 N/P=8 N/P=10

d) Transfection efficiency of lipid **6**/DNA complexes in cells under different N/P ratios.



Lipo2000 N/P=2 N/P=4

e) Transfection efficiency of lipid **6**/DNA complexes in SW480 cells under different N/P ratios.

Fig. S2. The fluorescence microscope images of transfected by glycolipid/DNA complexes in Hela, HepG2 and SW480 cells-ines.