Section 1 of supporting information for

Synthesis of Surfactants Based on Pentaerythritol. Part I. Cationic and Zwitterionic Gemini Surfactants

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Note: spectra from compound **1.24** to the end of series **3** are in section 1b

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General Methods. *N*,*N*-Dimethylformamide was stored over activated molecular sieves for 72 hours, then distilled with reduced pressure over more activated molecular sieves. Methanol was dried with magnesium methoxide. Toluene was dried by reflux over calcium hydride for 10 min followed by distillation from calcium hydride. Sodium hydride was a 60% oil dispersion that was washed with dry hexane under nitrogen before use. Reactions involving sodium hydride were performed in flame-dried glassware. ^{1}H and ^{13}C NMR spectra were recorded at 300 K in 5 mm NMR tubes on NMR spectrometers operating at 250.13 and 62.9, respectively or at 500.13 and 125.08 MHz, respectively, on solutions in chloroform-*d*, unless otherwise indicated. Chemical shifts are given in parts per million (ppm)(+/-0.01 ppm) relative to that of tetramethylsilane (TMS) (0.00 ppm) in the case of ^{1}H NMR spectra, and to the central line of chloroform-*d* (∗ 77.16) for the ^{13}C NMR spectra. All assignments were made with the aid of COSY, HETCOR, and/or long-range HETCOR experiments at 250 MHz or DGF COSY, HSQC or HMBC experiments at 500 MHz. High resolution electrospray mass spectra (HR ESI MS) were recorded on samples dissolved in methanol using trilysine KKK or rifampicin or the Tuning Mix from Agilent as references. Most TLC was performed on aluminum-backed plates bearing 200 um silica gel 60 F_{254}. Benzylidene acetals were visualized by quenching of fluoresence or by spraying the plate with a solution[22] of 0.2 % *p*-methoxyphenol in ethanol/2N H_2SO_4 (1/1, v/v) or an acidic solution of anisaldehyde in ethanol [ethanol (9 mL), anisaldehyde (0.5 mL), and conc. sulfuric acid (0.5 mL)][23] or a solution of 2% ceric sulfate in 1M sulfuric acid and followed, for all spray reagents, by heating on a hot plate until colour developed. TLC for quaternary ammonium salts was performed on aluminum-backed plates bearing 200 um basic alumina and developed with the Dragendorf reagent. Diethyl (*N*,*N*-dimethylcarbamoyl)methylphosphonate was prepared as described by Bartlett et al[16] for the dimethyl analog and had physical properties as described.[24]

General Method for Alkylation: 5,5'-Bis(octyloxymethyl)-2-phenyl-1,3-dioxane (5). A hexanes-washed sodium hydride oil dispersion (60% oil dispersion, 8.6 g, 0.22 mol, 2.0 eq) was added in

portions slowly to a stirred solution of mono-*O*-benzylidenepentaerythritol[12] (**4**) (24.11 g, 0.1076 mol) in dry DMF (600 mL) under a nitrogen atmosphere. The stirred reaction mixture was cooled with an ice water bath for one hour, then 1-bromooctane (46.76 mL, 51.90 g, 0.268 mol, 2.5 eq) was added dropwise over 2 h. After the reaction mixture had been stirred 12 h, another addition of sodium hydride (4.5 g, 0.11 mol, 1.0 eq) and 1-bromooctane (20 mL, 0.11 mol, 1.0 eq) was made. If after the reaction mixture had been stirred a further 12 h, TLC showed that some mono-*O*-octyl product was present, another identical addition was made. When all of the mono-*O*-octyl derivative had been consumed, the reaction mixture was quenched by the addition of methanol dropwise until foaming ceased. The reaction mixture was filtered under vacuum and the solid was washed with dichloromethane (~150 mL). The combined filtrate and washings were concentrated and the residue was extracted with hexanes (300 mL, then 200 mL). The combined extracts were concentrated to an oily residue that was passed through a short silica gel column using hexanes, then 5% ethyl acetate/95% hexanes as eluents. The title compound (**5**) was a colorless oil (44.71 g, 85 %): R_F 0.46 (hexanes : ethyl acetate 94 : 6); ^{1}H NMR (500.13 MHz) δ 0.88, 0.89 (2 t, 6H, J = 6.5 Hz, 2 x Me), 1.20-1.35 (br m, 20H, 10 x CH_2), 1.54, 1.57 (2 pentet, 4H, J = 6.8 Hz, 2 $OCH_2C\mathit{H}_2$), 3.22 (s, 2H, eq CCH_2O), 3.35 (t, 2H, J = 6.5 Hz, eq octyl OCH_2), 3.45 (t, 2H, J = 6.6 Hz, ax octyl OCH_2), 3.71 (s, 2H, ax OCH_2C), 3.88, 4.09 (2d, 4H, J = 11.5 Hz, H-4,H-4', H-6,H-6'), 5.42 (s, 1H, acetal H), 7.31-7.49 (m, 5H, Ph); ^{13}C NMR δ 138.5 (q Ph) , 128.8 (para Ph), 128.3 (2C, mPh), 126.1 (2C, oPh), 101.7 (acetal C), 71.8 (eq OCH_2CH_2), 71.7 (ax OCH_2CH_2), 70.8 (eq OCH_2C), 70.2 (C-4 and C-6), 69.4 (ax OCH_2C), 38.9 (q C), 2 x 31.89 ($CH_2CH_2CH_3$), 29.68, 29.54, 29.51, 29.45, 2 x 29.34 (6 octyl CH_2), 26.22, 26.19 ($CH_2CH_2CH_2O$), 2 x 22.70 (CH_2CH_3), 14.3 (Me); HR ESI MS *m/z* calcd for $C_{28}H_{48}O_4Na$ (M+Na) 471.3445, found 471.3448.

5,5'-Bis(decyloxymethyl)-2-phenyl-1,3-dioxane (6). The title compound was prepared as above (89% yield) as a colorless oil that was crystallized from methanol: mp 29-30 ^{o}C; R_F 0.48 (94: 6 hexanes:ethyl acetate); ^{1}H NMR δ 0.88 (t, 6H, J = 6.4 Hz, 2 x Me), 1.20-1.35 (br s, 28H, 14 x CH_2), 1.54 (pentet, 4H, J = 6.7 Hz, 2 $OCH_2C\mathit{H}_2$), 3.23 (s, 2H, eq CCH_2O), 3.36 (t, 2H, J = 6.4 Hz, eq decyl OCH_2), 3.46 (t, 2H, J = 6.6 Hz, ax decyl OCH_2), 3.71 (s, 2H, ax OCH_2C), 3.88, 4.09 (2d, 4H, J = 11.7 Hz, H-4, H-4', H-6, H-6'), 5.42 (s, 1H, acetal H), 7.31-7.48 (m, 5H, Ph); ^{13}C NMR δ 138.6 (q Ph) , 129.0 (para Ph), 128.4 (2C, mPh), 126.2 (2C, oPh), 101.9 (acetal C), 71.9 (eq OCH_2CH_2), 71.8 (ax OCH_2CH_2), 70.9 (eq OCH_2C), 70.4 (C-4 and C-6), 69.4 (ax OCH_2C), 39.0 (q C), 2 x 32.06 ($CH_2CH_2CH_3$), 2 x 29.81, 29.79, 2 x 29.76, 29.69, 29.65, 29.62, 2 x 29.50 (10 decyl CH_2), 2 x 26.3 ($CH_2CH_2CH_2O$), 2 x 22.8 (CH_2CH_3), 14.3 (Me); LR ESI MS *m/z* calcd for $C_{32}H_{57}O_4$ (M+H) 505.4, found 505.1. Anal. Calcd. for $C_{32}H_{56}O_4$: C, 76.14; H, 11.18. Found: C, 76.03; H, 10.97.

5,5'-Bis(dodecyloxymethyl)-2-phenyl-1,3-dioxane (7). The title compound was prepared as above (77% yield) as a solid that was crystalized from methanol to give colorless needles: R_F 0.51 (94:6 hexanes:ethyl acetate); mp 37.5 – 38.5 ^oC; ^1H NMR δ 0.88 (t, 6H, J = 6.4 Hz, 2 x Me), 1.20-1.35 (br s, 36H, 18 x CH_2), 1.54 (pentet, 4H, J = 6.3 Hz, 2 OCH_2CH_2), 3.22 (s, 2H, eq CCH_2O), 3.35 (t, 2H, J = 6.5 Hz, eq dodecyl OCH_2), 3.46 (t, 2H, J = 6.6 Hz, ax dodecyl OCH_2), 3.71(s, 2H, ax OCH_2C), 3.88, 4.09 (2d, 4H, J = 11.7 Hz, H-4, H-4', H-6, H-6'), 5.41 (s, 1H, acetal H), 7.31-7.48 (m, 5H, Ph); ^{13}C NMR δ 138.5 (q Ph), 128.8 (para Ph), 128.2 (2C, mPh), 126.1 (2C, oPh), 101.7 (acetal C), 71.7 (eq CH_2CH_2OC), 71.6 (ax CH_2CH_2OC), 70.7 (eq OCH_2C), 70.2 (C-4 and C-6), 69.3 (ax OCH_2C), 38.9 (q C), 2 x 31.8 ($CH_2CH_2CH_3$), 29.70, 29.66 , 29.61, 29.53, 29.22 (14 dodecyl CH_2), 26.2 ($CH_2CH_2CH_2O$), 2 x 22.7 (CH_2CH_3), 2 x 14.1 (Me); LR ESI MS *m/z* calcd for $C_{36}H_{65}O_4$ (M+H) 561.49, found 561.3; calcd for $C_{36}H_{64}O_4Na^+$ 583.47, found 583.5; calcd for $C_{36}H_{64}O_4K^+$ 599.44, found 599.3. Anal. Calcd. for $C_{36}H_{64}O_4$: C, 77.09, H, 11.50. Found: C, 77.04, H, 11.92.

2-Phenyl-5,5'-bis(tetradecyloxymethyl)-1,3-dioxane (8). The title compound was prepared as above (89% yield) as a solid that was crystallized from ethyl acetate: R_F 0.53 (94:6 hexanes:ethyl acetate); mp 45 ^oC; ^1H NMR (500 MHz) δ 0.88 (t, 6H, J = 6.6 Hz, 2 x Me), 1.16-1.37 (br s, 44H, 18 x CH_2), 1.55 (br pentet, 4H, J = 6.4 Hz, 2 OCH_2CH_2), 3.23 (s, 2H, eq CCH_2O), 3.35 (t, 2H, J = 6.4 Hz, eq OCH_2CH_2), 3.46 (t, 2H, J = 6.4 Hz, ax OCH_2CH_2), 3.71(s, 2H, ax OCH_2C), 3.88, 4.09 (2d, 4H, J = 11.3 Hz, H-4,H-4', H-6,H-6'), 5.41 (s, 1H, acetal H), 7.31-7.48 (m, 5H, Ph); ^{13}C NMR δ 139.3 (q Ph) , 128.9 (para Ph), 128.4 (2C, mPh), 126.2 (2C, oPh), 101.8 (acetal C), 71.9 (eq CH_2CH_2OC), 71.8 (ax CH_2CH_2OC), 70.9 (eq OCH_2C), 70.4 (C-4 and C-6), 69.5 (ax OCH_2C), 39.0 (q C), 2 x 32.1 ($CH_2CH_2CH_3$), 2 x 29.86, 2 x 29.83, 6 x 29.81, 2 x 29.77, 2 x 29.75, 2 x 29.66, 2 x 29.51 (18 tetradecyl CH_2), 2 x 26.3 ($CH_2CH_2CH_2O$), 2 x 22.8 (CH_2CH_3), 2 x 14.2 (Me); HR ESI MS *m/z* calcd for $C_{40}H_{72}O_4Na$ (M+Na) 639.5323, found 639.5305.

General method for hydrogenolysis: 2,2-bis(octyloxymethyl)-1,3-propanediol (9). To a solution of compound **5** (10.02 g, 22.3 mmol) in ethyl acetate (100 mL) was added 10% Pd/C (Degussa type, 0.2 g). The mixture was stirred vigorously under atmospheric pressure H_2(g) for 1 h. More 10% Pd/C (Degussa type, 0.5 g) was added and the solution stirred until uptake of H_2(g) ceased (2h). The mixture was filtered and the residue washed with dichloromethane (50 mL), then dichloromethane containing 20% methanol (2 x 50 mL). The filtrate and washings were concentrated to a colorless solid, yield 6.89 g, 85%. Crystallization from isopropanol gave colorless crystals:, R_F 0.40 (dichloromethane: methanol 96:4); mp 35 ^oC; ^1H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.20-1.36 (br m, 20H, 10 x CH_2), 1.56 (pentet, 4H, J = 6.8 Hz, 2 OCH_2CH_2), 2.82 (t, 2H, J = 6.1 Hz, OH), 3.42 (t, 4H, J = 6.5 Hz, octyl OCH_2), 3.50 (s, 4H, OCH_2C), 3.65 (d, 4H, CH_2OH); ^{13}C NMR δ 73.1 ($CCH_2OCH_2CH_2$), 72.2 (CH_2CH_2OC), 65.5 (CH_2OH), 44.7 (q C), 31.9 ($CH_2CH_2CH_3$), 29.64 (OCH_2CH_2), 29.50, 29.35 (2 octyl CH_2), 26.3 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.2 (Me); LR ESI MS *m/z* calcd for $C_{21}H_{45}O_4$ (M+H) 361.33, found 361.1. Anal. Calcd. for $C_{21}H_{44}O_4$: C, 69.95; H, 12.30. Found: C, 69.62; H, 12.68.

2,2-Bis(decyloxymethyl)-1,3-propanediol (10). Hydrogenolysis of compound **6** (10.23 g, 20.3 mmol) as for compound **5** above in ethyl acetate (100 mL) using 10% Pd/C (Degussa type, 0.2 g) gave a colorless solid that was crystallized from methanol: yield 6.81 g, 81%, R_F 0.42 (dichloromethane: methanol 96:4); mp 44.5-45.0 ^oC; ^1H NMR δ 0.88 (t, 6H, J = 6.6 Hz, 2 x Me), 1.20-1.35 (br s, 28H, 18 x CH_2), 1.55 (pentet, 4H, J = 6.5 Hz, 2 OCH_2CH_2), 2.88 (t, 2H, J= 6.1 Hz, OH), 3.42 (t, 4H, J = 6.5 Hz, decyl OCH_2), 3.51 (s, 4H, OCH_2C), 3.65 (d, 4H, CH_2OH); ^{13}C NMR δ 73.3 ($CCH_2OCH_2CH_2$), 72.2 (CH_2CH_2OC), 65.6 (CH_2OH), 44.6 (q C), 32.0 ($CH_2CH_2CH_3$), 2 x 29.7, 29.66, 29.56, 29.47 (5 decyl CH_2), 26.3 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.2 (Me); LR ESI MS: *m/z* calcd for $C_{25}H_{53}O_4$ 417.39, found 417.1. Anal. Calcd. for $C_{25}H_{52}O_4$: C, 72.06; H, 12.58. Found: C, 71.98; H, 12.45.

2,2-Bis(dodecyloxymethyl)-1,3-propanediol (11). Hydrogenolysis of compound **7** (10.23 g, 20.3 mmol) as for compound **5** above in ethyl acetate (100 mL) using 10% Pd/C (Degussa type, 0.2 g) gave a colorless solid that was crystallized from ethyl acetate: yield 6.81 g, 81%; R_F 0.45 (dichloromethane: methanol 96:4); mp 54 –55 ^oC; ^1H NMR δ 0.88 (t, 6H, J = 6.6 Hz, 2 x Me), 1.20-1.35 (br s, 36H, 18 x CH_2), 1.56 (pentet, 4H, J = 6.4 Hz, 2 OCH_2CH_2), 2.69 (br s, 2H, OH), 3.42 (t, 4H, J = 6.5 Hz, dodecyl OCH_2), 3.51(s, 4H, OCH_2C), 3.64 (s, 4H, CH_2OH); ^{13}C NMR δ 73.3 (CCH_2OCH_2C), 72.2 (CH_2CH_2OC), 65.6 (CH_2OH), 44.6 (q C), 32.1 ($CH_2CH_2CH_3$), 29.81, 29.78, 29.77, 29.73, 29.58, 29.50 (6 dodecyl CH_2), 29.66 (OCH_2CH_2), 26.3 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.3 (Me); LR ESI MS *m/z* calcd for $C_{29}H_{61}O_4$ (M+H) 473.46, found 473.3. Anal. Calcd. for $C_{29}H_{60}O_4$: C, 73.67; H, 12.79. Found: C, 73.31; H, 12.68.

2,2-Bis(tetradecyloxymethyl)-1,3-propanediol (12). Hydrogenolysis of compound **8** (10.0 g, 16.2 mmol) in ethyl acetate (200 mL) containing 10% Pd/C (Degussa type, 0.5 g) as for compound **5** above gave a colorless solid that was crystallized from ethyl acetate: yield 7.88 g, 92%; R_F 0.47 (dichloromethane: methanol 96:4); mp 63-64 ^oC; ^1H NMR δ 0.88 (t, 6H, J = 6.6 Hz, 2 x Me), 1.20-1.35 (br s, 44H, 18 x CH_2), 1.56 (pentet, 4H, J = 6.4 Hz, 2 OCH_2CH_2), 2.83 (br s, 2H, OH), 3.42 (t, 4H, J = 6.5 Hz, dodecyl OCH_2), 3.51(s, 4H, OCH_2C), 3.64 (s, 4H, CH_2OH); ^{13}C NMR δ 73.4 (CCH_2OCH_2C), 72.2 (CH_2CH_2OC), 65.7 (CH_2OH), 44.6 (q C), 32.1 ($CH_2CH_2CH_3$), 29.85, 2 x 29.83, 29.81, 29.78, 29.75, 29.59, 29.51 (8 tetradecyl CH_2), 29.65 (OCH_2CH_2), 26.3 ($CH_2CH_2CH_2O$), 22.7 (CH_2CH_3), 14.3 (Me); LR ESI MS *m/z* calcd for $C_{33}H_{69}O_4$ (M+H) 529.52, found 529.3. Anal. Calcd. for $C_{33}H_{68}O_4$: C, 74.94, H, 12.96. Found: C, 74.55, H, 13.03.

General procedure for formation of iodides: 1,3-diiodo-2,2-bis(octyloxymethyl)propane (13). Iodine (10.2 g, 0.0415 mol, 2.5 eq), imidazole (2.73 g, 0.0415 mol, 2.5 eq) and triphenylphosphine (8.94 g, 0.0353 mol, 2.2 eq) were added to a solution of compound **9** (5.80 g, 0.0161 mol) in anhydrous toluene (200 mL) and the reaction mixture was refluxed for 3 h. More iodine was then

added to consume excess triphenyl phosphine and reflux was continued for 1 h. The cooled reaction mixture was stirred for 10 min each with saturated sodium bicarbonate (100 mL) and 10% aqueous sodium thiosulfate (200 mL) solutions. The organic layer was washed with water (3 x 50 mL), dried ($MgSO_4$) and concentrated. The residue was taken up in hexanes and the solution was passed a short silica gel column. Concentration gave the title compound as a colorless oil: 7.89 g, 86%; R_F 0.34 (98:2 hexanes: dichloromethane); ^{1}H NMR δ 0.89 (t, 6H, J = 6.3 Hz, 2 x Me), 1.22-1.36 (br s, 20H, 10 x CH_2), 1.55 (pentet, 4H, J = 6.1 Hz, 2 OCH_2CH_2), 3.33 (s, 4H, CH_2I), 3.35 (s, 4H, OCH_2C), 3.42 (t, 4H, octyl OCH_2); ^{13}C NMR δ 71.7 (CH_2CH_2OC), 70.9 (CCH_2OCH_2C), 41.7 (q C), 32.0 ($CH_2CH_2CH_3$), 29.69, 29.56, 29.47 (3 octyl CH_2), 26.3 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.3 (Me), 12.1 (CH_2I); HR ESI MS *m/z* calcd for $C_{21}H_{43}I_2O_2$ (M+H) 581.1353, found 581.1351.

2,2-Bis(decyloxymethyl)-1,3-diiodopropane (14). A solution of compound **10** (10.0 g, 24.3 mmol) in toluene (300 mL) was reacted with iodine (16.3 g, 25.9 mmol, 2.7 eq), imidazole (4.08 g, 60.0 mmol, 2.5 eq) and triphenyl phosphine (13.4 g, 50.4 mmol, 2.1 eq) as above to give the title compound as an oil: 14.4 g, 94%; R_F 0.43 (98:2 hexanes: dichloromethane); ^{1}H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.22-1.36 (br s, 28H, 14 x CH_2), 1.54 (pentet, 4H, J = 6.8 Hz, 2 CH_2CH_2O), 3.32 (s, 4H, CH_2I), 3.35 (s, 4H, OCH_2C), 3.41 (t, 4H, J = 6.5 Hz, 2 decyl OCH_2); ^{13}C NMR δ 71.7 (CH_2CH_2OC), 70.9 (CCH_2OCH_2C), 41.7 (q C), 32.1 ($CH_2CH_2CH_3$), 29.65, 29.63, 29.48, 29.37 (4 decyl CH_2), 29.58 (OCH_2CH_2), 26.3 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.3 (Me), 12.1 (CH_2I); HR ESI MS *m/z* calcd for $C_{25}H_{51}I_2O_2$ (M+H) 637.1979, found 637.1976.

2,2-Bis(dodecyloxymethyl)-1,3-diiodopropane (15). Treatment of compound **11** (10.0 g, 21.2 mmol) in toluene (300 mL) with iodine (14.0 g, 52.8 mmol, 2.5 eq), imidazole (3.58 g, 52.8 mol, 2.5 eq) and triphenyl phosphine (13.82 g, 52.8 mol, 2.5 eq) as above gave the title compound as an oil: 14.0 g, 95%; R_F 0.44 (98:2 hexanes: dichloromethane); ^{1}H NMR δ 0.88 (t, 6H, J = 6.5 Hz, 2 x Me), 1.22-1.36 (br s, 36H, 18 x CH_2), 1.55 (pentet, 4H, J = 6.4 Hz, 2 OCH_2CH_2), 3.32 (s, 4H, CH_2I), 3.35 (s, 4H,

OCH_2C), 3.41 (t, 4H, dodecyl OCH_2); ^{13}C NMR δ 71.7 (CH_2CH_2OC), 70.9 (CCH_2OCH_2C), 41.7 (q C), 32.1 ($CH_2CH_2CH_3$), 29.92, 3 x 29.88, 29.79, 29.59 (6 dodecyl CH_2), 29.68 (OCH_2CH_2), 26.3 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.3 (Me), 12.1 (CH_2I); HR ESI MS *m/z* calcd for $C_{29}H_{59}O_2I_2$ (M+H) 693.2598, found 693.2605.

1,3-Diiodo-2,2-bis(tetradecyloxymethyl)propane (16). Treatment of compound **12** (5.0 g, 9.5 mmol) in toluene (200 mL) with iodine (7.20 g, 28.4 mmol, 3.0 eq), imidizole (1.60 g, 23.6 mmol, 2.5 eq) and triphenyl phosphine (7.19 g, 28.4 mmol, 2.9 eq) as above gave the title compound as a colorless solid: yield 6.36 g, 90 %; mp 27-28 °C; R_F 0.45 (98:2 hexanes: dichloromethane); ^1H NMR δ 0.88 (t, 6H, J = 7.0 Hz, 2 x Me), 1.22-1.36 (br s, 44H, 22 x CH_2), 1.53 (pentet, 4H, J = 6.9 Hz, 2 OCH_2CH_2), 3.33 (s, 4H, CH_2I), 3.35 (s, 4H, OCH_2C), 3.41(t, 4H, tetradecyl OCH_2); ^{13}C NMR δ 71.8 (CH_2CH_2OC), 71.0 (CCH_2OCH_2C), 41.8 (q C), 32.1 ($CH_2CH_2CH_3$), 29.87, 29.86, 29.85, 29.82, 2 x 29.81, 29.72, 29.52 (8 tetradecyl CH_2), 29.61 (OCH_2CH_2), 26.4 ($CH_2CH_2CH_2O$), 22.9 (CH_2CH_3), 14.3 (Me), 12.1 (CH_2I); LR ESI MS *m/z* calcd for $C_{33}H_{67}I_2O_2$ (M+H) 749.32, found 749.1. Anal. Calcd. for $C_{33}H_{66}I_2O_2$: C, 52.94, H, 8.89. Found: C, 53.14, H, 9.20.

General procedure for iodide displacement by dimethylamine: *N*,*N*,*N*',*N*'-Tetramethyl-2,2-bis(octyloxymethyl)-1,3-propanediaminium dichloride (1.5). Compound **13** (9.98 g, 17.2 mmol), dimethylamine in THF (2 M, 87 mL, 0.17 mol, 10 eq) and potassium carbonate (5.9 g, 43 mmol, 2.5 eq) were added to a sealed tube with the aid of THF (10 mL). The reaction mixture was stirred at 160~170 °C. After one week, all of the starting material had been consumed and the reaction mixture was filtered and the solvent was removed in vaccuo at 35~40 °C to give a yellow oil, *N*,*N*,*N*',*N*'-tetramethyl-2,2-bis(octyloxymethyl)-1,3-propanediamine (**1.1**). The crude oil was taken up in dichloromethane (50 mL) and the resulting solution was shaken with ice cold 2 M HCl (75 mL). The aqueous layer was extracted with dichloromethane (2 x 50 mL), then the combined organic layers were washed with water (20 mL), dried ($MgSO_4$), and concentrated to give the title compound as a light

yellow crystalline solid that was recrystallized from ethyl acetate, acetone 15:1 to give clear rectangular crystals: yield 5.62 g, 67%; mp 145 ^{o}C; R_F 0.29 on basic alumina (hexanes, ethyl acetate, methanol 96: 4: 0.4); ^{1}H NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.26 -1.33 (br s, 20H, 10 x CH_2), 1.56 (pentet, 4H, J = 6.6 Hz, 2 OCH_2CH_2), 2.97 (s, 12H, 2 x $N(CH_3)_2$), 3.47 (t, 4H, J = 6.6 Hz, octyl OCH_2); 3.68 (s, 4H, OCH_2C), 3.79 (s, 4H, CH_2N), 11.78 (brs, HN); ^{13}C NMR δ 71.7 (CH_2CH_2OC), 66.9 (CCH_2OCH_2C), 58.6 (CH_2N), 47.5 ($N(CH_3)_2$), 44.5 (q C), 31.8 ($CH_2CH_2CH_3$), 29.5, 29.3, 29.2, (3 octyl CH_2), 26.2 ($CH_2CH_2CH_2O$), 22.6 (CH_2CH_3), 14.1 (Me); HR ESI MS *m/z* calcd for $C_{25}H_{55}N_2O_2$ (M-H-2Cl) 415.4264, found 415.4260.

2,2-Bis(decyloxymethyl)-*N*,*N*,*N*',*N*'-tetramethyl-1,3-propanediaminium dichloride (1.6). Compound **14** (6.88 g, 10.8 mmol), 2 M dimethylamine in THF (54 mL, 0.11 mol, 10 eq), and potassium carbonate (3.72 g, 27.0 mmol, 2.5 eq) in THF were reacted as above to give a yellow oil, 2,2-bis(decyloxymethyl)-*N*,*N*,*N*',*N*'-tetramethyl-1,3-propanediamine (**1.2**) that was converted to the title bishydrochloride as above. The colorless crystals were recrystallized from ethyl acetate/acetone 2/1 to give colorless needles: yield 3.95 g, 68 %; mp 130-133 ^{o}C; R_F 0.32 on basic alumina (hexanes, ethyl acetate, methanol 96: 4: 0.4); ^{1}H NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.26 -1.32 (br s, 28H, 14 x CH_2), 1.55 (pentet, 4H, J = 6.6 Hz, 2 OCH_2CH_2), 2.97 (s, 12H, 2 x $N(CH_3)_2$), 3.47 (t, 4H, J = 6.6 Hz, decyl OCH_2), 3.67 (s, 4H, OCH_2C), 3.81 (s, 4H, CH_2N), 11.85 (brs, HN); ^{13}C NMR δ 71.8 (CH_2CH_2OC), 66.9 (CCH_2OCH_2C), 58.6 (CH_2N), 47.7 ($N(CH_3)_2$), 44.6 (q C), 32.0 ($CH_2CH_2CH_3$), 29.7, 29.7, 29.6, 29.5, 29.4, (decyl CH_2), 26.3 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.2 (Me); HR ESI MS *m/z* calcd for $C_{29}H_{63}N_2O_2$ (M-H-2Cl) 471.4890, found 471.4886.

2,2-Bis(dodecyloxymethyl)-*N*,*N*,*N*',*N*'-tetramethyl-1,3-propanediaminium dichloride (1.7). Compound **15** (11.72 g, 16.9 mmol), 2 M dimethylamine in THF (85 mL, 0.17 mol, 10 eq), potassium carbonate (5.83 g, 42.2 mmol), and THF (10 mL) were reacted as for compound **1.5** to give a yellow crystalline solid, 2,2-bis(dodecyloxymethyl)-*N*,*N*,*N*',*N*'-tetramethyl-1,3-propanediamine (**1.3**) that was

converted to the title compound, an off white crystalline solid. It was recrystallized from ethyl acetate acetone to give colorless rectangular crystals: yield 6.90 g, 68 %; mp 128-130 ^{o}C; R_F 0.34 on basic alumina (hexanes, ethyl acetate, methanol 96: 4: 0.4); ^{1}H NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.26 -1.31 (br s, 36H, 18 x CH_2), 1.55 (pentet, 4H, J = 6.5 Hz, 2 OCH_2CH_2), 2.97 (s, 12H, 2 x $N(CH_3)_2$), 3.47 (t, 4H, J = 6.6 Hz, dodecyl OCH_2), 3.67 (s, 4H, OCH_2C), 3.78 (s, 4H, CH_2N), 11.69 (brs, HN); ^{13}C NMR δ 71.7 (CH_2CH_2OC), 66.9 (CCH_2OCH_2C), 58.6 (CH_2N), 47.6 ($N(CH_3)_2$), 44.5 (q C), 31.9 ($CH_2CH_2CH_3$), 29.7, 29.6, 29.6, 29.6, 29.5, 29.4, 29.3 (dodecyl CH_2), 26.2 ($CH_2CH_2CH_2O$), 22.7 (CH_2CH_3), 14.1 (Me); HR ESI MS *m/z* calcd for $C_{33}H_{71}N_2O_2$ (M-H-2Cl) 527.5516, found 527.5517.

***N*,*N*,*N*',*N*'-Tetramethyl-2,2-bis(tetradecyloxymethyl)-1,3-propanediaminium dichloride (1.8)** A mixture of compound **16** (15.24 g, 20.37 mmol), 2 M dimethyl amine in THF (51 mL, 0.10 mol, 5.0 eq), and potassium carbonate (7.03 g, 50.9 mmol) were heated in a sealed tube as for compound 1.5, except that the same amount of dimethylamine was added after 48 h, to give an offwhite crystalline solid, *N*,*N*,*N*',*N*'-tetramethyl-2,2-bis(tetradecyloxymethyl)-1,3-propanediamine (**1.4**) that was converted to the title bishydrochloride as above. The crystalline solid was recrystallized from ethyl acetate to give colorless crystals: yield 9.97 g, 75%; mp 129-130 ^{o}C; R_F 0.36 on basic alumina (hexanes, ethyl acetate, methanol 96: 4: 0.4); ^{1}H NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.26 - 1.31 (br s, 44H, 22 x CH_2), 1.55 (pentet, 4H, J = 6.5 Hz, 2 OCH_2CH_2), 2.97 (s, 6H, 2 x $N(CH_3)_2$, 3.47 (t, 4H, J = 6.6 Hz, tetradecyl OCH_2), 3.67 (s, 4H, OCH_2C), 3.82 (s, 4H, CH_2N), 11.88 (brs, HN); ^{13}C NMR δ 71.9 (CH_2CH_2OC), 66.9 (CCH_2OCH_2C), 58.6 (CH_2N), 47.7 ($N(CH_3)_2$), 44.7 (q C), 32.1 ($CH_2CH_2CH_3$), 29.8, 29.6, 29.5, 29.5, (tetradecyl CH_2), 26.4 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.3 (Me); HR ESI MS *m/z* calcd for $C_{37}H_{79}N_2O_2$ (M-2Cl-H) 583.6142, found 583.6139.

General procedure for formation of quaternary ammonium salts: *N*,*N*,*N*,*N*',*N*',*N*'-hexamethyl-2,2-bis(octyloxymethyl)-1,3-propanediammonium diiodide (1.9). An aqueous NaOH solution (2 M,

30 mL) was added to salt **1.5** (4.44 g, 9.1 mmol) and the resulting mixture was extracted with dichloromethane (3 x 50 mL). The combined extracts were washed with water and dried ($MgSO_4$) and concentrated to a colorless syrup, *N*,*N*,*N*',*N*'-tetramethyl-2,2-bis(octyloxymethyl)-1,3-propanediamine (**1.1**): yield: 2.45 g, 65 %; R_F 0.39 on basic alumina (hexanes, ethyl acetate, methanol 96: 4: 0.4); ^{1}H NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.26 -1.32 (br s, 20H, 10 x CH_2), 1.54 (pentet, 4H, J = 7.0 Hz, 2 OCH_2CH_2), 2.26 (s, 12H, 2 x $N(CH_3)_2$), 3.26 (s, 4H, 2 x NCH_2), 3.26 (s, 4H, OCH_2C), 3.33 (t, J = 6.5 Hz, 4H, octyl OCH_2); ^{13}C NMR δ 71.2 (CH_2CH_2OC), 70.8 (CCH_2OCH_2C), 59.9 (CH_2N), 48.7 ($N(CH_3)_2$), 45.8 (q C), 32.0 ($CH_2CH_2CH_3$), 29.9, 29.6, 29.5, (3 octyl CH_2), 26.5 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.2 (Me).

Methyl iodide (3.6 mL, 57.9 mmol, 10.0 eq) was added to a stirred solution of compound **1.1** (2.4 g, 5.79 mmol) in dry THF (15 mL) and the resulting solution was refluxed for 24 h, then concentrated. The title compound, a light yellow crystalline solid, was recrystallized from ethyl acetate and acetone to give colorless crystals: yield 3.01 g, 75 %; mp 160-162 ^oC; R_F on basic alumina 0.53 (chloroform acetone methanol 2 1 1); ^{1}H NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.27 - 1.29 (br s, 20H, 10 x CH_2), 1.60 (pentet, 4H, J = 6.6 Hz, 2 OCH_2CH_2), 3.51 (t, 4H, J = 6.7 Hz, octyl OCH_2), 3.65 (s, 18H, 6 x CH_3), 3.91 (s, 4H, OCH_2C), 4.45 (s, 4H, CH_2N); ^{13}C NMR δ 72.2 (CH_2CH_2OC), 68.2 (CCH_2OCH_2C), 67.7 (CH_2N), 56.5 ($N(CH_3)_3$), 49.1(q C), 31.9 ($CH_2CH_2CH_3$), 29.7, 29.4, 29.3, (octyl CH_2), 26.4 ($CH_2CH_2CH_2O$), 22.7 (CH_2CH_3), 14.2 (Me); HR ESI MS *m/z* calcd for $C_{27}H_{60}N_2IO_2$ (M-I) 571.3700, found 571.3702; calcd for $C_{54}H_{120}N_4I_3O_4$ (2M-I) 1269.6444, found 1269.6443.

2,2-Bis(decyloxymethyl)-*N*,*N*,*N*,*N*',*N*',*N*'-hexamethyl-1,3-propanediammonium diiodide (1.10). An aqueous NaOH solution (2 M, 20 mL) and salt **1.5** (2.5 g, 4.4 mmol) were reacted as for compound **1.9** to yield a colorless syrup of 2,2-bis(decyloxymethyl)-*N*,*N*,*N*',*N*'-tetramethyl-1,3-propanediamine (**1.2**), yield: 1.7 g, 79%; R_F 0.42 on basic alumina (hexanes, ethyl acetate, methanol 96: 4: 0.4); ^{1}H

NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.26 -1.28 (br s, 28H, 14 x CH_2), 1.54 (br m, 4H, 2 OCH_2CH_2), 2.26 (s, 16H, 2 x $N(CH_3)_2$, 2 x NCH_2), 3.26 (s, 4H, OCH_2C), 3.38 (t, J = 6.5 Hz, 4H, decyl OCH_2); ^{13}C NMR δ 71.1 (CH_2CH_2OC), 70.7 (CCH_2OCH_2C), 59.9 (CH_2N), 48.6 ($N(CH_3)_2$), 45.6 (q C), 32.1 ($CH_2CH_2CH_3$), 29.9, 29.8, 29.8, 29.7, 29.5 (decyl CH_2), 26.5 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.2 (Me).

Alkylation of compound **1.2** (1.7 g, 3.6 mmol) in dry THF (25 mL) with methyl iodide (2.24 mL, 36.1 mmol, 10 eq) as for compound **1.9** gave the title compound as a light yellow crystalline solid that was recrystallized from ether and acetone to give colorless crystals: yield 2.4 g, 73 %; mp 75 °C; R_F on basic alumina 0.57 (chloroform acetone methanol 2 1 1); ^1H NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.26 -1.30 (br s, 28H, 14 x CH_2), 1.60 (pentet, 4H, J = 6.5 Hz, 2 OCH_2CH_2), 3.51 (t, 4H, J= 6.9 Hz, decyl OCH_2), 3.65 (s, 18H, 6 x CH_3), 3.91 (s, 4H, OCH_2C), 4.44 (s, 4H, CH_2N); ^{13}C NMR δ 71.9 (CH_2CH_2OC), 68.2 (CCH_2OCH_2C), 67.9 (CH_2N), 56.1 ($N(CH_3)_3$), 48.7 (q C), 31.7 ($CH_2CH_2CH_3$), 29.45, 29.43, 29.37, 29.14 (4 decyl CH_2), 29.25 (OCH_2CH_2), 26.2 ($CH_2CH_2CH_2O$), 22.5 (CH_2CH_3), 14.0 (Me); HR ESI MS *m/z* calcd for $C_{31}H_{68}N_2IO_2$ (M-I) 627.4326, found 627.4326; calcd for $C_{62}H_{136}I_3N_4O_4$ (2M-I) 1381.7696, found 1381.7694.

2,2-Bis(dodecyloxymethyl)-*N*,*N*,*N*,*N*',*N*',*N*'-hexamethyl-1,3-propanediammonium diiodide (1.11). Reaction of an aqueous NaOH solution (2 M, 50 mL) with salt **1.7** (4.75 g, 7.9 mmol) as for compound **1.9** yielded a colorless syrup, 2,2-bis(dodecyloxymethyl)-*N*,*N*,*N*',*N*'-tetramethyl-1,3-propanediamine (**1.3**): yield 3.27 g, 78%; R_F 0.48 on basic alumina (hexanes, ethyl acetate, methanol 96: 4: 0.4); ^1H NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.26 -1.33 (br s, 36H, 18 x CH_2), 1.53 (pentet, 4H, J = 7.0 Hz, 2 OCH_2CH_2), 2.26 (2s, 16H, 2 x $N(CH_3)_2$, 2 x NCH_2), 3.26 (s, 4H, OCH_2C), 3.33 (t, J = 6.5 Hz, 4H, dodedecyl OCH_2); ^{13}C NMR δ 71.2 (CH_2CH_2OC), 70.7 (CCH_2OCH_2C), 59.9 (CH_2N), 48.7 ($N(CH_3)_2$), 45.7 (q C), 32.1 ($CH_2CH_2CH_3$),29.9, 29.8, 29.8, 29.7, 29.5 (dodecyl CH_2), 26.5 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.3 (Me**).**

Alkylation of compound **1.2.3** (3.25 g, 6.18 mmol) in dry THF (20 mL) with methyl iodide (3.9 mL, 61.7 mmol, 10 eq) as above gave **1.11** as a light yellow crystalline solid that was recrystallized from ethyl acetate and acetone to give colorless crystals: yield 3.75 g, 76 %; R_F on basic alumina 0.60 (chloroform acetone methanol 2 1 1); mp 130-132 ^oC; ^1H NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.26 -1.30 (br s, 36H, 18x CH_2), 1.59 (pentet , 4H, J = 6.4 Hz, 2 OCH_2CH_2), 3.51 (t, 4H, J = 6.7 Hz, dodecyl OCH_2), 3.65 (s, 18H, 6 x CH_3), 3.91 (s, 4H, OCH_2C), 4.45 (s, 4H, CH_2N); ^{13}C NMR δ 72.1 (CH_2CH_2OC), 68.2 (CCH_2OCH_2C), 67.9 (CH_2N), 56.4 ($N(CH_3)_3$), 48.9 (q C), 31.9($CH_2CH_2CH_3$), 29.6, 29.5, 29.4, 29.4 (dodecyl CH_2), 26.3 ($CH_2CH_2CH_2O$), 22.7 (CH_2CH_3), 14.1 (Me); LR ESI MS *m/z* calcd for $C_{35}H_{76}N_2IO_2$ (M-I) 683.49, found 683.3. Anal. Calcd. for $C_{35}H_{76}N_2O_2I_2$: C, 51.85, H, 9.45, N, 3.46. Found: C, 51.43, H, 9.32, N, 3.71.

***N*,*N*,*N*',*N'*,*N*'-Hexamethyl-2,2-bis(tetradecyloxymethyl)-1,3-propanediammonium diiodide (1.12).** Treatment of salt **1.8** (5.96 g, 9.11 mmol) with an aqueous NaOH solution (2 M, 40 mL) as above gave a colorless syrup, *N*,*N*,*N*',*N*'-tetramethyl-2,2-bis(tetradecyloxymethyl)-1,3-propanediamine (**1.4**): yield 4.69 g, 89 %; R_F 0.51 on basic alumina (hexanes, ethyl acetate, methanol 96: 4: 0.4); ^1H NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.26 -1.31 (br s, 44H, 22 x CH_2), 1.51 (pentet, 4H, J = 7.0 Hz, 2 OCH_2CH_2), 2.26 (s, 16H, 2 x $N(CH_3)_2$, 2 x NCH_2), 3.26 (s, 4H, OCH_2C), 3.33 (t, J = 6.5 Hz, 4H, tetradecyl OCH_2); ^{13}C NMR δ 71.2 (CH_2CH_2OC), 70.7 (CCH_2OCH_2C), 59.9 (CH_2N), 48.7 ($N(CH_3)_2$), 45.7 (q C), 32.1 ($CH_2CH_2CH_3$), 29.91, 29.86, 29.85, 29.82, 29.81, 29.80, 29.65 (8 tetradecyl CH_2), 29.52 (OCH_2CH_2), 26.5 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.3 (Me); LR ESI MS *m/z* calcd for $C_{37}H_{79}N_2O_2$ 583.61, found 583.5.

Alkylation of compound **1.4** (4.0 g, 6.9 mmol) in dry THF (20 mL) with methyl iodide (4.3 mL, 68.7 mmol, 10 eq) as above gave the title compound as a light yellow crystalline solid, that was recrystallized from ethyl acetate and acetone to give colorless crystals: yield 5.7 g, 96%; R_F on basic alumina 0.63 (chloroform acetone methanol 2 1 1); mp 127-128 ^oC; ^1H NMR δ 0.88 ppm (t, 6H, J =

7.0 Hz, 2 x Me), 1.26 -1.30 (br s, 44H, 22 x CH_2), 1.57 (pentet, 4H, J = 7.1 Hz, 2 OCH_2CH_2), 3.50 (t, 4H, J = 6.8 Hz, tetradecyl OCH_2), 3.63 (s, 18H, 6 x CH_3), 3.93 (s, 4H, OCH_2C), 4.48 (s, 4H, CH_2N); ^{13}C NMR δ 72.2 (CH_2CH_2OC), 68.2 (CCH_2OCH_2C), 67.7 (CH_2N), 56.6 ($N(CH_3)_3$), 49.1 (q C), 32.0 ($CH_2CH_2CH_3$), 29.83, 29.80, 29.78, 29.78, 29.76, 29.54, 29.48 (8 tetradecyl CH_2), 29.68 (OCH_2CH_2), 26.5 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.23 (Me); LR ESI MS *m/z* calcd for $C_{39}H_{84}N_2O_2I$ (M-I) 739.56, found 739.3. Anal. Calcd. for $C_{39}H_{84}N_2O_2I_2$: C, 54.03, H, 9.77, N, 3.23. Found: C, 53.78, H, 9.76, N, 3.09.

General procedure for displacement of iodides by pyrrolidine: 1,3-Bis(1-azacyclopentyl)-2,2-bis(octyloxymethyl)propane dihydrochloride (1.17). A stirred solution of compound **13** (18.0 g, 31.0 mmol) in pyrrolidine (100 mL) containing potassium carbonate (10.7 g, 31.0 mmol, 2.5 eq) was refluxed under nitrogen for 48 h, allowed to cool to rt, then filtered. The solid was washed with dichloromethane (2 x 10 mL) and the filtrate and washings were combined and diluted with dichloromethane (100 mL). The resulting solution was washed with water (3 x 100 mL), dried ($MgSO_4$) and concentrated at 30 ^oC to give crude 1,3-bis(1-azacyclopentyl)-2,2-bis(octyloxymethyl)propane (**1.13**): yield 14.20 g. This product was taken up in dichloromethane (100 mL) and the resulting solution was shaken with ice cold 2 M HCl (100 mL). The aqueous layer was extracted with dichloromethane (2 x 100 mL), then the combined organic layers were washed with water (20 mL), dried ($MgSO_4$) and concentrated to give the title compound as a light yellow crystalline solid that was recrystallized from hexanes, ethyl acetate 2:1 to give colorless crystals: yield 12.5 g, 75 %; mp 124-125 ^oC, R_F 0.22 on basic alumina (hexanes, ethyl acetate, methanol, 96:4:0.4); ^1H NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.26-1.31 (br s, 20H, 10 x CH_2), 1.55 (pentet, 4H, J = 6.6 Hz, 2 OCH_2CH_2), 2.06 (YY' part of AA'BB'XX'YY' pattern, 4H, 1/2 of 2 $N(CH_2CH_2)_2$), 2.25 (XX' part of AA'BB'XX'YY' pattern, 4H, 1/2 of 2 $N(CH_2CH_2)_2$), 3.21 (BB' part of AA'BB'XX'YY' pattern, 4H, 1/2 of 2 $N(CH_2)$), 3.46 (t, 4H, J = 6.6 Hz, octyl OCH_2), 3.59 (s, 4H, OCH_2C), 3.84 (s, 4H, CH_2N), 3.91

(AA' part of AA'BB'XX'YY' pattern, 4H, 1/2 of 2 $N(CH_2)$), 11.51 (br s, HN); ^{13}C NMR δ 71.5 (CH_2CH_2OC), 67.5 ($CCH_2OCH_2CH_2$), 57.8 (NCH_2), 56.9 (CH_2N), 44.3 (q C), 31.7 ($CH_2CH_2CH_3$), 29.41, 29.25, 29.18 (3 octyl CH_2), 26.2 ($CH_2CH_2CH_2O$), 23.5(NCH_2CH_2), 22.6 (CH_2CH_3), 14.0 (Me); HR ESI MS *m/z* calcd for $C_{29}H_{59}N_2O_2$ (M-H-2Cl) 467.4577, found 467.4578.

1,3-Bis(1-azacyclopentyl)-2,2-bis(decyloxymethyl)propane dihydrochloride (1.18). Treatment of a solution of compound **14** (16.0 g, 25.0 mmol) in pyrrolidine (100 mL) containing potassium carbonate (8.69 g, 62.8 mmol, 2.5 eq) as for compound **1.17** gave a light brown syrup, 1,3-bis(1-azacyclopentyl)-2,2-bis(decyloxymethyl)propane (**1.14**): yield 11.93 g. Addition of ice cold 2 M HCl (100 mL) gave a light yellow crystalline solid that was recrystallized from ethyl acetate to give colorless crystals: yield 10.9 g, 76 %; mp 125-126 ^oC, R_F 0.24 on basic alumina (hexanes, ethyl acetate, methanol, 96:4:0.4); ^1H NMR δ 0.88 ppm (t, 6H, J = 6.8 Hz, 2 x Me), 1.26-1.28 (br s, 28H, 14 x CH_2), 1.55 (pentet, 4H, J = 6.3 Hz, 2 OCH_2CH_2), 2.05 (YY' part of AA'BB'XX'YY' pattern, 4H, 1/2 of 2 2 $N(CH_2CH_2)_2$), 2.25 (XX' part of AA'BB'XX'YY' pattern, 4H, 1/2 of 2 2 $N(CH_2CH_2)_2$), 3.21 (BB' part of AA'BB'XX'YY' pattern, 4H, $N(CH_2)_2$), 3.45 (t, 4H , J = 6.6 Hz, decyl OCH_2), 3.58 (s, 4H, OCH_2C), 3.84 (s, 4H, CH_2N), 3.91 (BB' part of AA'BB'XX'YY' pattern, 4H, $N(CH_2)_2$), 11.5 (br s, HN); ^{13}C NMR δ 71.7 (CH_2CH_2OC), 67.5 (CCH_2O), 57.9 (NCH_2), 57.0 ($(CH_2)_2N$), 44.5 (q C), 32.0 ($CH_2CH_2CH_3$), 29.71, 29.66, 29.59, 29.47, 29.40 (6 decyl CH_2), 26.3 ($CH_2CH_2CH_2O$), 23.6 ($N(CH_2CH_2)_2$), 22.8 (CH_2CH_3), 14.2 (Me); HR ESI MS *m/z* calcd for $C_{33}H_{67}N_2O_2$ (M-H-2Cl) 523.5203, found 523.5204.

1,3-Bis(1-azacyclopentyl)-2,2-bis(dodecyloxymethyl)propane dihydrochloride (1.19). Treatment of a solution of compound **15** (12.2 g, 17.6 mmol) in pyrrolidine (100mL) containing potassium carbonate (6.1 g, 44 mmol, 2.5 eq) as for compound **1.17** gave an orange syrup, 1,3-bis(1-azacyclopentyl)-2,2-bis(dodecyloxymethyl)propane (**1.15**): yield 12.9 g. Addition of ice cold 2 M HCl (100 mL) and dichloromethane (30 mL) provided a light pink crystalline solid that was dissolved in dichloromethane (30 mL). The dichloromethane solution was washed with distilled water (10 mL),

dried ($MgSO_4$) and concentrated to a crystalline solid that was recrystallized from ethyl acetate to give colorless crystals: yield 8.8 g, 77 %; mp 127 ^oC; R_F 0.28 on basic alumina (hexanes, ethyl acetate, methanol, 96:4:0.4); ^1H NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.26-1.28 (br s, 36H, 18xCH_2), 1.55 (pentet, 4H, J = 6.4 Hz, 2 OCH_2CH_2), 2.06 (YY' part of AA'BB'XX'YY' pattern, 4H, 1/2 of 2 2 $N(CH_2CH_2)_2$), 2.25 (XX' part of AA'BB'XX'YY' pattern, 4H, 1/2 of 2 2 $N(CH_2CH_2)_2$), 3.21 (BB' part of AA'BB'XX'YY' pattern, 4H, $N(CH_2)_2$), 3.45 (t, 4H, J = 6.5 Hz , dodecyl OCH_2), 3.58 (s, 4H, OCH_2C), 3.83 (s, 4H, CH_2N), 3.91 (AA' part of AA'BB'XX'YY' pattern, 4H, $N(CH_2)_2$), 11.5 (br s, HN); ^{13}C NMR δ 71.7 (CH_2CH_2OC), 67.6 (CCH_2O), 58.0 (NCH_2), 57.1 ($(CH_2)_2N$), 44.5 (q C), 32.0 ($CH_2CH_2CH_3$), 29.7, 29.6, 29.5, 29.4, (dodecyl CH_2), 26.3 ($CH_2CH_2CH_2O$), 23.6 ($N(CH_2CH_2)_2$), 22.8 (CH_2CH_3), 14.2 (Me); HR ESI MS *m/z* calcd for $C_{37}H_{75}N_2O_2$ (M-H-2Cl) 579.5829, found 579.5826.

1,3-Bis(1-azacyclopentyl)-2,2-bis(tetradecyloxymethyl)propane dihydrochloride (1.20). Treatment of a solution of compound 16 (14.96 g, 31.29 mmol) in pyrrolidine (100 mL) containing potassium carbonate (10.78 g, 78.0 mmol, 2.5 eq) as for compound **1.17** gave a brown solid, 1,3-bis(1-azacyclopentyl)-2,2-bis(tetradecyloxymethyl)propane (**1.16**), yield 17.26 g. Addition of ice cold 2 M HCl (100 mL) and dichloromethane (30 mL) provided a yellow crystalline solid (18.11 g), that was dissolved in dichloromethane (30 mL). The solution was dried ($MgSO_4$) and concentrated to colorless crystalline solid that was recrystallized from ethyl acetate to give the title compound as colorless crystals: yield 17.5 g, 79 %; mp 128 ^oC; R_F 0.33 on basic alumina (hexanes, ethyl acetate, methanol, 96:4:0.4); ^1H NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.26-1.28 (br s, 44H, 22 x CH_2), 1.55 (pentet, 4H, J = 6.3 Hz, 2 OCH_2CH_2), 2.05 (YY' part of AA'BB'XX'YY' pattern, 4H, 1/2 of 2 2 $N(CH_2CH_2)_2$), 2.25 (XX' part of AA'BB'XX'YY' pattern, 4H, 1/2 of 2 2 $N(CH_2CH_2)_2$), 3.21 (BB' part of AA'BB'XX'YY' pattern, 4H, $N(CH_2)_2$), 3.45 (t, 4H, J = 6.5 Hz , tetradecyl OCH_2), 3.58 (s, 4H, OCH_2C), 3.84 (s, 4H, CH_2N), 3.91 (AA' part of AA'BB'XX'YY' pattern, 4H, $N(CH_2)_2$), 11.5 (br s, HN); ^{13}C NMR δ 71.7 (CH_2CH_2OC), 67.6 (CCH_2O), 58.0 (NCH_2), 57.1 ($(CH_2)_2$)N), 44.6 (q C), 32.1

($CH_2CH_2CH_3$), 29.83, 29.82, 29.79, 29.65, 29.57, 29.49 (8 tetradecyl CH_2), 26.4 ($CH_2CH_2CH_2O$), 23.7 ($N(CH_2CH_2)_2$), 22.8 (CH_2CH_3), 14.2 (Me); HR ESI MS *m/z* calcd for $C_{41}H_{83}N_2O_2$ (M-H-2Cl) 635.6455, found 635.6454.

General procedure for methylation of the pyrrolidine derivatives: 1,3-bis(1-methyl-1-azoniacyclopentyl)-2,2-bis(octyloxymethyl)propane diiodide (1.21). An aqueous 2 M NaOH solution (50 mL) was added to compound **1.17** (12.5 g, 23.2 mmol) and the resulting mixture was extracted with dichloromethane (3 x 50 mL). The combined extracts were washed with water and dried ($MgSO_4$), and concentrated to give the free base, 1,3-bis(1-azacyclopentyl)-2,2-bis(octyloxymethyl)propane (**1.13**) as a light yellow syrup: yield 10.3 g, 72%; R_F 0.32 on basic alumina (hexanes, ethyl acetate, methanol, 96:4:0.4); ^{1}H NMR δ 0.88 ppm (t, 6H, J = 6.8 Hz, 2 x Me), 1.27-1.34 (br s, 20H, 10 x CH_2), 1.53 (pentet, 4H, J = 6.7 Hz, 2 OCH_2CH_2), 1.68 (m, 8H, $2NCH_2CH_2$), 2.48 (s ,4H CH_2N), 2.56 (m, 8H, $2N(CH_2)_2$, 3.27 (s, 4H, OCH_2C), 3.32 (t, 4H, J = 6.4 Hz, octyl OCH_2); ^{13}C NMR δ 71.7 ($CCH_2OCH_2CH_2$), 71.1 (CH_2CH_2OC), 56.81 (CH_2N), 56.84 (NCH_2), 45.7 (q C), 31.9 ($CH_2CH_2CH_3$), 29.87, 29.55, 29.43 (3 octyl CH_2), 26.4 ($CH_2CH_2CH_2O$), 24.3 (NCH_2CH_2), 22.8 (CH_2CH_3), 14.1 (Me).

Methyl iodide (13.3 g, 21.3 mmol, 10 eq) was added to a stirred solution of compound **1.13** (9.90 g, 21.3 mmol) in dry THF (50 mL). The resulting mixture was refluxed under nitrogen for 48 h, then concentrated. The title compound, a light brown crystalline solid, was recrystallized from ethyl acetate to give colorless crystals: yield 13 g, 81%; mp 92 ^{o}C; R_F 0.69 on basic alumina (chloroform, acetone, methanol, ammonia 2:2:1:0.5); ^{1}H NMR δ 0.89 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.26-1.30 (br s, 20H, 10 x CH_2), 1.57 (pentet, 4H, J = 6.5 Hz, 2 OCH_2CH_2), 2.18 (YY' part of AA'BB'XX'YY' pattern, 4H, 1/2 of 2 2 $N(CH_2CH_2)_2$), 2.38 (XX' part of AA'BB'XX'YY' pattern, 4H, 1/2 of 2 2 $N(CH_2CH_2)_2$)), 3.46 (s, 6H, 2 NCH_3), 3.49 (t, 4H, J = 6.6 Hz , octyl OCH_2), 3.96 (s, 4H, OCH_2C), 4.04 (m, 8H, 2 $N(CH_2)_2$), 4.63 (s, 4H, 2 CH_2N); ^{13}C NMR δ 71.9 (CH_2CH_2OC), 68.3 ($CCH_2OCH_2CH_2$),

66.9 (NCH_2), 65.4 (CH_2N), 48.5 (CH_3N), 48.4 (q C), 31.8 ($CH_2CH_2CH_3$), 29.5, 29.3, 29.2 (3 octylCH_2), 26.3 ($CH_2CH_2CH_2O$), 22.6 (CH_2CH_3), 21.1 (NCH_2CH_2), 14.1 (Me); HR ESI-MS *m/z* calcd for $C_{31}H_{64}IN_2O_2$ 623.4013 (M-I), found 623.4011.

2,2-Bis(decyloxymethyl)-1,3-bis(1-methyl-1-azoniacyclopentyl)propane diiodide (1.22). Aqueous 2 M NaOH solution (50 mL) and compound **1.18** (10.9 g, 18.3 mmol) were reacted as for **1.21** above to give a colorless syrup, 1,3-bis(1-azacyclopentyl)-2,2-bis(decyloxymethyl)propane (**1.14**): yield 9.50 g, 72.5%; R_F 0.34 on basic alumina (hexanes, ethyl acetate, methanol, 96:4:0.4); ^{1}H NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.26-1.31 (br s, 28H, 14 x CH_2), 1.53 (pentet, 4H, J = Hz, 2 OCH_2CH_2), 1.68 (m, 8H, 2 $N(CH_2CH_2)$), 2.49 (s , 4H, NCH_2C, 2.57 (m , 8H, 2 $N(CH_2)_2$), 3.29 (s, 4H, OCH_2C), 3.33 (t, 4H, J = 6.5 Hz, decyl OCH_2); ^{13}C NMR δ 71.9 ($CCH_2OCH_2CH_2$), 71.3 (CH_2CH_2OC), 57.2 (CH_2N), 57.0 ($N(CH_2)_2$), 45.8 (q C), 32.1 ($CH_2CH_2CH_3$), 29.93, 29.85, 29.78 29.68, 29.52 (6 decyl CH_2), 26.5 ($CH_2CH_2CH_2O$), 24.4 ($N(CH_2CH_2)_2$), 22.8 (CH_2CH_3), 14.3 (Me).

Methyl iodide (16.6 g, 117 mmol, 10 eq) and compound **1.14** (6.1 g, 11.6 mmol) in dry THF (50 mL) was treated as for compound **1.21** to give a light brown crystalline solid that was recrystallized from ethyl acetate to give an off white crystalline solid: yield 6.0 g, 81%; mp 95 - 96 °C; R_F 0.71 on basic alumina (chloroform, acetone, methanol, ammonia 2:2:1:0.5); ^{1}H NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.26-1.30 (br s, 28H, 14 x CH_2), 1.57 (pentet, 4H, J = 6.5 Hz, 2 OCH_2CH_2), 2.18 (YY' part of AA'BB'XX'YY' pattern, 4H, 1/2 of 2 2 $N(CH_2CH_2)_2$), 2.38 (XX' part of AA'BB'XX'YY' pattern, 4H, 1/2 of 2 2 $N(CH_2CH_2)_2$)), 3.46 (s, 6H, 2 NCH_3), 3.49 (t, 4H, J = 6.6 Hz , decyl OCH_2), 3.96 (s, 4H, OCH_2C), 4.04 (m , 8H, $2N(CH_2)_2$), 4.63 (s, 4H, 2 CH_2N); ^{13}C NMR δ 72.1 (CH_2CH_2OC), 68.2 ($CCH_2OCH_2CH_2$), 66.9 (NCH_2), 65.2 (CH_2N), 48.6 (CH_3N), 48.3 (q C), 32.0 ($CH_2CH_2CH_3$), 29.71, 29.67, 29.66, 29.47, 29.39 (5 decyl CH_2), 26.5 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 21.2 (NCH_2CH_2), 14.2 (Me); LR ESI MS *m/z* calcd for $C_{35}H_{72}IN_2O_2$ (M-I) 679.64, found 679.3, calcd

for (M-2I) 276.3, found 276.3. Anal. Calcd. for $C_{35}H_{72}I_2N_2O_2$: C, 52.11, H, 9.00, N, 3.47. Found: C, 52.08, H, 9.17, N, 3.69.

2,2-Bis(dodecyloxymethyl)-1,3-bis(1-methyl-1-azoniacyclopentyl)propane diiodide (1.23). Reaction of 2 M NaOH (50 mL) with salt **1.19** (8.8 g, 13.5 mmol) as above gave 1,3-bis(1-azacyclopentyl)-2,2-bis(dodecyloxymethyl)propane (**1.15**) as a colorless syrup: yield 7.5 g, 74%; R_F 0.36 on basic alumina (hexanes, ethyl acetate, methanol, 96:4:0.4); ^{1}H NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.26-1.31 (br s, 36H, 18 x CH_2), 1.53 (pentet, 4H, J = 7.0 Hz, 2 OCH_2CH_2), 1.69 (m, 8H, 2 $N(CH_2CH_2)_2$), 2.49 (s, 4H, CH_2N), 2.57 (m, 8H , 2 $N(CH_2)_2$), 3.28 (s, 4H, OCH_2C), 3.33 (t, 4H, J = 6.5 Hz , dodecyl OCH_2); ^{13}C NMR δ 71.9 (CCH_2O), 71.3 (CH_2CH_2OC), 57.3 (CH_2N), 57.0 ($N(CH_2)_2$), 45.8 (q C), 32.0 ($CH_2CH_2CH_3$), 29.3, 29.8, 29.7 (dodecyl CH_2), 26.4 ($CH_2CH_2CH_2O$), 24.2 ($N(CH_2CH_2)_2$), 22.9 (CH_2CH_3), 14.3 (Me).

Methyl iodide (8 mL, 129.7 mmol, 10 eq) was reacted with compound **1.15** (7.5 g, 13 mmol) in dry THF (50 mL) as above to give the title compound, as an offwhite crystalline solid, that was recrystallized from ethyl acetate to give colorless crystals: yield 7.5 g, 84%; mp 100-101 °C; R_F 0.74 on basic alumina (chloroform, acetone, methanol, ammonia 2:2:1:0.5); ^{1}H NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.25-1.23 (br s, 36H, 18 x CH_2), 1.57 (pentet, 4H, J = 6.6 Hz, 2 OCH_2CH_2), 2.17 (YY' part of AA'BB'XX'YY' pattern, 4H, 1/2 of 2 2 $N(CH_2CH_2)_2$), 2.38 (XX' part of AA'BB'XX'YY' pattern, 4H, 1/2 of 2 2 $N(CH_2CH_2)_2$), 3.45 (s, 2 NCH_3), 3.49 (t, 4H , J = 6.6 Hz , 2 dodecyl OCH_2), 3.96 (s, 4H, OCH_2C), 4.05 (m , 8H , NCH_2), 4.64 (s, 4H, CH_2N); ^{13}C NMR δ 71.9 (CH_2CH_2OC), 68.3 ($CCH_2OCH_2CH_2$), 66.9 (NCH_2), 65.5 (CH_2N) , 48.4 (CH_3N), 48.3 (q C), 31.8 ($CH_2CH_2CH_3$), 29.6, 29.5, 29.3, 29.3 (dodecyl CH_2), 26.3 ($CH_2CH_2CH_2O$), 22.6 (CH_2CH_3), 21.1 (NCH_2CH_2), 14.0 (Me); LR ESI MS *m/z* calcd for $C_{39}H_{80}IN_2O_2$ (M-I) 735.53, found 735.3; calcd for $C_{38}H_{77}N_2O_2$ (M-2I-Me) 593.60, found 593.4; calcd for (M-2I)/2 304.31, found 304.3. Anal. Calcd. for $C_{39}H_{80}I_2N_2O_2$: C, 54.29, H, 9.34, N, 3.47. Found: C, 54.16, H, 9.77, N, 3.38.

1,3-Bis(1-methyl-1-azoniacyclopentyl)-2,2-bis(tetradecyloxymethyl)propane iodide (1.24). Reaction of 2 M NaOH (50 mL) with salt **1.20** (17.5 g, 24.7 mmol) as above gave 1,3-bis(1-azacyclopentyl)-2,2-bis(tetradecyloxymethyl)propane (**1.16**) as a light yellow crystalline solid: yield 14.3 g, 72 %; mp 100-102 ^{o}C; R_F 0.38 on basic alumina (hexanes, ethyl acetate, methanol, 96:4:0.4); ^{1}H NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.26-1.31 (br s, 44H, 22 x CH_2), 1.53 (pentet, 4H, J = 7.0 Hz, 2 OCH_2CH_2), 1.68 (m, 8H, 2 $N(CH_2CH_2)_2$), 2.49 (s, 4H, CH_2N), 2.57 (m, 8H, 2 $N(CH_2)_2$), 3.28 (s, 4H, OCH_2C), 3.33 (t, 4H, J = 6.5 Hz , tetradecyl OCH_2); ^{13}C NMR δ 71.7 (CCH_2O), 71.1 (CH_2CH_2OC), 57.1 (CH_2N), 56.9 ($N(CH_2)_2$), 45.6 (q C), 32.0 ($CH_2CH_2CH_3$), 29.80, 29.74, 29.70 29.55, 29.40 (8 tetradecyl CH_2), 26.4 ($CH_2CH_2CH_2O$), 24.2 ($N(CH_2CH_2)_2$), 22.7 (CH_2CH_3), 14.1 (Me).

Methylation of compound **1.16** (7.15 g, 11.3 mmol) in THF (50 mL) with methyl iodide (15.8 g, 112 mmol, 10 eq) as above gave the compound, a light brown crystalline solid, that was recrystallized from ethyl acetate to give an off-white crystalline solid: yield 7.6 g, 94%; mp 108 ^{o}C; R_F 0.76 on basic alumina (chloroform, acetone, methanol, ammonia 2:2:1:0.5); ^{1}H NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.26-1.29 (br s, 44H, 22 x CH_2), 1.57 (pentet, 4H, J = 6.5 Hz, 2 OCH_2CH_2), 2.17 (YY' part of AA'BB'XX'YY' pattern, 4H, 1/2 of 2 2 $N(CH_2CH_2)_2$), 2.38 (XX' part of AA'BB'XX'YY' pattern, 4H, 1/2 of 2 2 $N(CH_2CH_2)_2$), 3.45 (s, 2 NCH_3), 3.49 (t, 4H , J = 6.6 Hz, tetradecyl OCH_2), 3.97 (s, 4H, OCH_2C), 4.05 (m , 8H , NCH_2), 4.67 (s, 4H, CH_2N); ^{13}C NMR δ 72.0 (CH_2CH_2OC), 68.3 ($CCH_2OCH_2CH_2$), 66.9 (NCH_2), 65.3 (CH_2N) , 48.6 (CH_3N), 48.3 (q C), 32.0 ($CH_2CH_2CH_3$), 29.72, 29.69, 29.62, 29.44, 29.39 (5 tetradecyl CH_2), 26.4 ($CH_2CH_2CH_2O$), 22.7 (CH_2CH_3), 21.2 (NCH_2CH_2), 14.2 (Me); LR ESI MS *m/z* calcd for $C_{43}H_{88}IN_2O_2$ (M-I) 791.59, found 791.3; calcd for M/2: 332.34, found 332.4; calcd for $C_{86}H_{176}I_3N_4O_4$ (2 M –I) 1710.1, found 1709.3. Anal. Calcd for $C_{43}H_{88}I_2N_2O_2.H_2O$: C, 55.12, H, 9.68, N, 2.99. Found: C, 55.26, H, 9.46, N, 3.30.

General procedure for displacement of iodides by cyanide: 3,3-bis(octyloxymethyl)pentanedinitrile (2.1). Potassium cyanide (0.80 g, 12.0 mmol, 3.0 eq) was added

to a stirred solution of compound **13** (2.4 g, 4.1 mmol) in dry DMF (25 mL). The resulting mixture was stirred at 80 ^{o}C for 24 h, and then allowed to cool to rt. A solid was deposited and the solution was decanted. The solid was washed with dichloromethane (2 x 10 mL). The combined solution and washings were concentrated to yellow oil that was taken up in dichloromethane (30 mL). The resulting solution was washed with water (3 x 25 mL), dried ($MgSO_4$) and concentrated to give the title compound as a light yellow oil: yield 1.3 g, 83%; R_F 0.42 (hexanes: ethyl acetate 9: 1); ^{1}H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.23-1.33 (br s, 20H, 10 x CH_2), 1.55 (pentet, 4H, J = 7.0 Hz, 2 OCH_2CH_2), 2.57 (s, 4H, CH_2CN), 3.42 (s, 4H, OCH_2C), 3.44 (t, J = 6.5 Hz, 4H, octyl OCH_2); ^{13}C NMR δ 116.7 (CN), 71.9 (CH_2CH_2OC), 70.8 (CCH_2OCH_2C), 41.3 (q C), 32.9 ($CH_2CH_2CH_3$), 29.45, 29.34 (2 octyl CH_2), 29.47 (OCH_2CH_2), 26.2 ($CH_2CH_2CH_2O$), 22.7 (CH_2CH_3), 21.7 (CH_2CN) 14.2 (Me); HR ESI MS *m/z* calcd for $C_{23}H_{42}N_2O_2Na$ (M+Na) 401.3138, found 401.3152.

3,3-Bis(decyloxymethyl)pentanedinitrile (2.2). Treatment of compound **14** (5.9 g, 9.3 mmol) in dry DMF (30 mL) with potassium cyanide (1.8 g, 28.0 mmol, 3.0 eq) as above gave the title compound as a light yellow oil: yield 3.6 g, 91 %; R_F 0.46 (hexanes: ethyl acetate 9:1); ^{1}H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.26-1.31 (br s, 28H, 14 x CH_2), 1.53 (pentet, 4H, J = 7.2 Hz, 2 OCH_2CH_2), 2.58 (s, 4H, CH_2N_3), 3.43 (s, 4H, OCH_2C), 3.45 (t, 4H, J = 6.5 Hz, decyl OCH_2); ^{13}C NMR δ 116.8 (CN), 72.0 (CH_2CH_2OC), 70.9 (OCH_2C), 41.3 (q C), 32.1 ($CH_2CH_2CH_3$), 29.75, 29.72, 29.53, 29.47 (4 decyl CH_2), 29.55 (OCH_2CH_2), 26.2 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 21.7 (CH_2CN) 14.3 (Me); HR ESI MS *m/z* calcd for $C_{27}H_{50}N_2O_2Na$ (M+Na) 457.3764, found 457.3781.

3,3-Bis(dodecyloxymethyl)pentanedinitrile (2.3). Treatment of compound **15** (2.3 g, 3.3 mmol) in dry DMF (30 mL) with potassium cyanide (0.5 g, 8.4 mmol, 3.0 eq) as above gave the title compound as a light yellow oil: yield 1.5 g, 91 %; R_F 0.49 (hexanes: ethyl acetate 9:1); ^{1}H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.26-1.32 (br s, 36H, 18 x CH_2), 1.54-1.58 (pentet, 4H, J = 7.0 Hz, 2 OCH_2CH_2), 2.57 (s, 4H, CH_2CN), 3.38 (s, 4H, OCH_2C), 3.45 (t, 4H, J = 7.0 Hz, 2 OCH_2CH_2), 2.57 (s, 4H, CH_2CN),

3.38 (s, 4H, OCH_2C), 3.45 (t, 4H, J = 6.5 Hz, dodecyl OCH_2); ^{13}C NMR δ 116.7 (CN), 72.0 (CH_2CH_2OC), 70.8 (CCH_2OCH_2C), 41.3 (q C), 32.0 ($CH_2CH_2CH_3$), 2 x 29.72, 2 x 29.70, 29.51, 29.45 (6 dodecyl CH_2), 29.53 (OCH_2CH_2), 26.2 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 21.7 (CH_2CN) 14.3 (Me); HR ESI MS *m/z* calcd for $C_{31}H_{58}N_2O_2Na$ (M+Na) 513.4391, found 513.4373.

3,3-Bis(tetradecyloxymethyl)pentanedinitrile (2.4). Treatment of compound **16** (5.0 g, 6.7 mmol) in dry DMF (30 mL) with potassium cyanide (1.0 g, 16.7 mmol, 2.5 eq) as above gave a light yellow oil that was taken up in hot 95% ethanol (50 mL). When this solution was kept at 5 °C, the title compound (**2.4**) precipitated as an amorphous solid, yield 2.24 g. Flash column chromatography of the residue yielded an additional 0.57 g, total yield 2.81 g, 77%: R_F 0.56 (hexanes: ethyl acetate 9: 1); mp 33-35 °C; ^1H NMR δ 0.88 (t, 6H, J = 6.8 Hz, 2 x Me), 1.22-1.36 (br s, 44H, 22 x CH_2), 1.56 (pentet, 4H, J = 6.7 Hz, 2 OCH_2CH_2), 2.58 (s, 4H, CH_2CN), 3.43 (s, 4H, OCH_2C), 3.45 (t, J = 6.5 Hz, 4H, tetradecyl OCH_2); ^{13}C NMR δ 116.7 (CN), 72.0 (CH_2CH_2OC), 70.8 (CCH_2OCH_2C), 41.3 (q C), 32.1 ($CH_2CH_2CH_3$), 29.84, 29.78, 29.76, 29.72, 29.54, 29.51 (8 tetradecyl CH_2), 29.56 (OCH_2CH_2), 26.2 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 21.8 (CH_2CN) 14.3 (Me); HR ESI MS *m/z* calcd for $C_{35}H_{66}N_2O_2Na$ (M+Na) 569.5017, found 569.5002.

General procedure for cyanide hydrolysis: 3,3-bis(octyloxymethyl)pentanedioic acid (2.5). A mixture of compound **2.1** (2.5 g, 5.61 mmol) in 1-propanol (40 mL) containing 35% NaOH (10 mL) was refluxed for 36 h. The reaction mixture was concentrated then the resulting aqueous reaction mixture was refluxed for another 24 h. The reaction mixture was cooled to 10 °C, acidified by adding a dilute HCl solution until the pH was 5 (pH paper). The mixture was extracted with ethyl acetate (2 x 50 mL) and the combined organic layers were washed with water (2 x 30 mL), brine (20 mL), dried (Na_2SO_4), and concentrated to give the crude product (**2.5**). The product was purified by flash column chromatography on silica gel using a gradient changing from 5% EtOAc in hexanes to 15% EtOAc in hexanes as eluent to give as a thick colorless syrup: yield 1.75 g (63%); R_F 0.5 (EtOAc: hexanes 1:1);

^{1}H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 6.5 Hz, CH_3), 1.24-1.31 (m, 20H, 10 x CH_2), 1.53 (pentet, 4H, J = 6.5 Hz, OCH_2CH_2), 2.61 (s, 4H, $COCH_2$), 3.40 (t, 4H, J = 6.5, OCH_2), 3.46 (s, 4H, OCH_2), 10.92(bs, 2H, COOH); ^{13}C NMR ($CDCl_3$) δ 176.8 (COOH), 73.2 (CCH_2O), 71.8 (OCH_2CH_2), 41.1 (q C), 37.5 ($HOOCCH_2$), 32.0 ($CH_2CH_2CH_3$), 29.60, 29.55, 29.42 (3 octyl CH_2), 26.3 ($OCH_2CH_2CH_2$), 22.8 (CH_3CH_2), 14.2 (CH_3); HR ESI MS *m/z* calcd for $C_{23}H_{43}O_6$ (M-H) 415.3065, found 415.3042.

3,3-Bis(decyloxymethyl)pentanedioic acid (2.6). Reaction of compound **2.2** (2.5 g, 5.8 mmol) in 1-propanol (40 mL) containing 35% NaOH (10 mL) as above gave the title compound as a colorless solid: yield 1.2 g (44%); mp 68-71 °C, R_F 0.4 (EtOAc: hexanes 1:1); ^{1}H NMR ($CDCl_3$) δ 0.87 (t, 6H, J = 6.5 Hz, CH_3), 1.23-1.32 (m, 28H, 14 x CH_2), 1.53 (pentet, 4H, J = 6.5 Hz, OCH_2CH_2), 2.60 (s, 4H, $COCH_2$), 3.41 (t, 4H, J = 6.5, OCH_2), 3.47 (s, 4H, OCH_2), 10.50(bs, 2H, COOH); ^{13}C NMR $CDCl_3$ δ 176.4 (COOH), 73.2 (CCH_2O), 71.8 (OCH_2CH_2), 41.1 (q C), 37.6 ($HOOCCH_2$), 32.1 ($CH_2CH_2CH_3$), 3 x 29.75, 29.60, 29.49 (5 decyl CH_2), 26.3 ($OCH_2CH_2CH_2$), 22.8 (CH_3CH_2), 14.3 (CH_3); HR ESI MS *m/z* calcd for $C_{27}H_{52}O_6$ (M-H) 471.3691, found 471.3667.

3,3-Bis(dodecyloxymethyl)pentanedioic acid (2.7). A mixture of compound **2.3** (2.5 g, 5.8 mmol) in 1-propanol (40 mL) containing 35% NaOH (10 mL) was treated as in the preparation of compound **2.5** above except that the aqueous mixture was refluxed for 36 h to give the product as a colorless solid: yield 2.2 g (46%); mp 80-82 °C; R_F 0.46 (ethyl acetate: hexanes 1:1); ^{1}H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 6.5 Hz, CH_3), 1.24-1.32 (m, 36H, 18 x CH_2), 1.53 (pentet, 4H, J = 6.5 Hz, OCH_2CH_2), 2.60 (s, 4H, $COCH_2$), 3.40 (t, 4H, J = 6.5 Hz, OCH_2), 3.46 (s, 4H, OCH_2), 10.6(bs,2H,COOH); ^{13}C NMR ($CDCl_3$) δ 176.5 (COOH), 73.2 (CCH_2O), 71.8 (OCH_2CH_2), 41.1 (qC), 37.6 ($HOOCCH_2$), 32.1 ($CH_2CH_2CH_3$), 3 x 29.84, 2 x 29.81, 29.61, 29.52 (7 dodecyl CH_2), 26.3 ($OCH_2CH_2CH_2$), 22.8 (CH_3CH_2), 14.3 (CH_3); HR ESI MS *m/z* calcd for $C_{31}H_{59}O_6$ (M-H) 527.4317, found 527.4324.

3, 3-Bis (tetradecyloxymethyl)pentanedioic acid (2.8). A mixture of compound **2.4** (5.0 g, 6.7 mmol) in 1-propanol (40 mL) containing 35% NaOH (10 mL) was treated as in the preparation of

compound **2.7** above to give the product as a colorless solid: yield: 2.2 g (46%); mp 84-86°C; R_F 0.51 (ethyl acetate: hexanes 1:1); ^{1}H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 6.5 Hz, CH_3), 1.22-1.32 (m, 44 H, 22 x CH_2), 1.54 (pentet, 4H, J = 6.5 Hz, OCH_2CH_2), 2.60 (s, 4H, $COCH_2$), 3.41 (t, 4H, J = 6.5 Hz, OCH_2), 3.47 (s, 4H, OCH_2),10.6 (bs, 2H, COOH); ^{13}C NMR ($CDCl_3$) δ 175.6 (COOH), 73.3 (CCH_2O), 71.9 (OCH_2CH_2), 41.1 (qC), 37.8 ($HOOCCH_2$), 32.1 ($CH_2CH_2CH_3$), 29.86, 29.82, 29.60, 29.52 (tetradecyl CH_2), 26.3 ($OCH_2CH_2CH_2$), 22.8 (CH_3CH_2), 14.3 (CH_3); HR ESI MS *m/z* calcd for $C_{35}H_{68}O_6$ (M-H) 583.4943, found 583.4978.

General procedure for formation of *N*,*N*-dimethyl amides: *N*,*N*,*N*',*N*'-tetramethyl-3,3-bis(octyloxymethyl)pentanediamide (2.9). 1-Hydroxybenzotriazole (HOBT, 1.03 g, 7.69 mmol) and *N*-(3-dimethylamino-propyl)-*N*'-ethylcarbodiimide.hydrochloride (EDC.HCl, 1.54 g, 8.07 mmol) were added to a stirred solution of diacid **2.5** (1.6 g, 3.9 mmol) and the reaction mixture was stirred for 1 h. Dimethylamine hydrochloride (1.25 g, 15.4 mmol) and triethylamine (2.7 g, 27 mmol) were added and the reaction mixture was stirred for another 24 h, then diluted with dichloromethane (50 mL). This mixture was washed with water (2 x 30 mL), brine (20 mL), dried (Na_2SO_4) and concentrated. The residue was purified by flash column chromatography using a gradient of 15 to 30% EtOAc in hexanes as eluent, giving compound **2.9** as a viscous liquid: yield 1.63 g (91%); R_F 0.5 (ethyl acetate: hexanes 2:1); ^{1}H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 6.5 Hz, CH_3), 1.26-1.30 (m, 20 H, 10 x CH_2), 1.51 (pentet, 4H, J = 6.5 Hz, OCH_2CH_2), 2.65 (s, 4H, $COCH_2$), 2.89 (s, 6H, NCH_3), 3.03 (s, 6H, NCH_3), 3.36 (t, 4H, J = 6.5 Hz, OCH_2), 3.53 (s, 4H, OCH_2); ^{13}C NMR ($CDCl_3$) δ 172.4 (CO), 72.7 (CCH_2O), 71.4 (OCH_2CH_2), 42.1 (qC), 37.9 (NCH_3), 35.5 (NCH_3), 33.6 ($NCOCH_2$), 32.0 ($CH_2CH_2CH_2$), 29.87, 29.63, 29.51 (2 x 3 octyl CH_2), 26.4 ($OCH_2CH_2CH_2$), 22.8 (CH_3CH_2), 14.2 (CH_3); HR ESI MS *m/z* calcd for $C_{27}H_{54}N_2O_4Na$ (M+Na) 493.3976, found 493.3966.

3,3-Bis(decyloxymethyl)-*N*,*N*,*N*',*N*'-tetramethylpentanediamide (2.10). 1-Hydroxybenzotriazole (HOBT, 1.14 g, 8.45 mmol), EDC.HCl (1.61 g, 8.45 mmol), diacid **2.6** (1.9 g, 4.0 mmol),

dimethylamine hydrochloride (1.31 g, 16.1 mmol) and triethylamine (3.26 g, 32.2 mmol) were reacted as for compound **2.9** above, giving compound **2.10** as a viscous liquid: yield 2.0 g (95%); R_F 0.5 (ethyl acetate: hexanes 2:1); ^{1}H NMR ($CDCl_3$) δ 0.89 (t, 6H, J = 6.5 Hz, CH_3), 1.26-1.35 (m, 28 H, 14 x CH_2), 1.53 (pentet, 4H, J = 6.5 Hz, OCH_2CH_2), 2.67 (s, 4H, $COCH_2$), 2.91 (s, 6H, NCH_3), 3.04 (s, 6H, NCH_3), 3.38 (t, 4H, J = 6.5 Hz, OCH_2), 3.54 (s, 4H, OCH_2); ^{13}C NMR ($CDCl_3$) δ 172.4 (CO), 72.7 (CCH_2O), 71.4 (OCH_2CH_2), 42.1 (qC), 38.0 (NCH_3), 35.5 (NCH_3), 33.6 ($NCOCH_2$), 32.1 ($CH_3CH_2CH_2$), 2 x 29.87, 29.78, 29.69, 29.52 (2 x 4 decyl CH_2), 26.4 ($OCH_2CH_2CH_2$), 22.8 (CH_3CH_2), 14.3 (CH_3); HR ESI MS *m/z* calcd for $C_{31}H_{62}N_2O_4Na$ (M+Na) 549.4602, found 549.4580.

3,3-Bis(dodecyloxymethyl)-*N*,*N*,*N*',*N*'-tetramethylpentanediamide (2.11). 1-Hydroxybenzotriazole (HOBT, 0.96 g, 7.2 mmol), EDC.HCl (1.36 g, 7.15 mmol), diacid **2.7** (1.8 g, 3.4 mmol), dimethylamine hydrochloride (1.11 g, 13.6 mmol) and triethylamine (2.06 g, 20.5 mmol) were reacted as for compound **2.9** above to give the title compound as a viscous liquid: yield 1.7 g (86%); R_F 0.5 (ethyl acetate: hexanes 2:1); ^{1}H NMR ($CDCl_3$) δ 0.89 (t, 6H, J = 6.5 Hz, CH_3), 1.22-1.34 (m, 36 H, 18 x CH_2), 1.50 (pentet, 4H, J = 6.5 Hz, OCH_2CH_2), 2.65 (s, 4H, $COCH_2$), 2.89 (s, 6H, NCH_3), 3.03 (s, 6H, NCH_3), 3.36 (t, 4H, J = 6.5 Hz, OCH_2), 3.52 (s, 4H, OCH_2); ^{13}C NMR ($CDCl_3$) δ 172.4 (CO), 72.7 (CCH_2O), 71.4 (OCH_2CH_2), 42.1 (qC), 37.9 (NCH_3), 35.5 (NCH_3), 33.5 ($NCOCH_2$), 32.1 ($CH_3CH_2CH_2$), 29.88 (x3), 29.83 (x2), 29.70, 29.52 (7 dodecyl CH_2), 26.4 ($OCH_2CH_2CH_2$), 22.8 (CH_3CH_2), 14.2 (CH_3); HR ESI MS *m/z* calcd for $C_{35}H_{70}N_2O_4Na$ (M+Na) 605.5228, found 605.5183.

***N*,*N*,*N*',*N*'-Tetramethyl-3,3-bis(tetradecyloxymethyl)pentanediamide (2.12).** 1-Hydroxybenzotriazole (HOBT, 2.0 g, 15 mmol), EDC.HCl (2.89 g, 15.1 mmol), diacid **2.8** (4.2 g, 7.2 mmol), dimethylamine hydrochloride (2.40 g, 29.5 mmol) and triethylamine (5.81 g, 57.5 mmol) were reacted as for compound **2.9** above to give the title compound (**2.12**) as a solid: yield 4.1 g (89%); mp 52-54 ^{o}C; R_F 0.46 (ethyl acetate: hexanes 2:1); ^{1}H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 7.0 Hz, CH_3), 1.26-1.42 (m, 44 H, 22 x CH_2), 1.51 (p, 4H, J = 6.5 Hz, OCH_2CH_2), 2.65 (s, 4H, $COCH_2$), 2.89 (s, 6H,

NCH_3), 3.02 (s, 6H, NCH_3), 3.36 (t, 4H, J = 6.5 Hz, OCH_2), 3.52 (s, 4H, OCH_2); ^{13}C NMR ($CDCl_3$) δ 172.4 (CO), 72.7 (CCH_2O), 71.4 (OCH_2CH_2), 42.1 (qC), 37.9 (NCH_3), 35.5 (NCH_3), 33.6 ($NCOCH_2$), 32.1 ($CH_3CH_2CH_2$), 29.9 - 29.8 (7C), 29.69, 29.52 (2 x 9 tetradecyl CH_2), 26.4 ($OCH_2CH_2CH_2$), 22.9 (CH_3CH_2), 14.3 (CH_3); HR ESI MS *m/z* calcd for $C_{39}H_{78}N_2O_4Na$ (M+Na) 661.5854, found 661.5823.

General procedure for amide reduction: *N*,*N*,*N*',*N*'-Tetramethyl-3,3-bis(octyloxymethyl)-1,5-pentanediamine (2.13). Diamide **2.9** (0.9 g, 1.91 mmol) was added dropwise to a stirred suspension of $LiAlH_4$ (0.29 g, 7.65 mmol) in THF (50 mL) at 0 °C, then the reaction mixture was stirred at rt for 6 h. Ethyl acetate (50 mL) was added dropwise, followed by water (0.3 mL), then 1M NaOH (0.3 mL). The mixture was filtered on a bed of celite that was washed with hot ethyl acetate. The combined filtrate and washings were dried (Na_2SO_4), filtered and concentrated to a residue that was purified by flash column chromatography. Elution using a gradient of 5 to 15% MeOH in dichloromethane gave **2.13** as a light brown liquid: yield: 0.67 g (80%); R_F on basic alumina 0.46 (dichloromethane: methanol 96:4); ^1H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 7.0 Hz, CH_3), 1.22-1.38 (m, 20 H, 10x CH_2), 1.43-1.54 (m, 8H, NCH_2CH_2, OCH_2CH_2), 2.21 (s, 12H, NCH_3), 2.27 (t, 4H, J = 8.0 Hz, NCH_2), 3.19 (s, 4H, OCH_2), 3.34 (t, 4H, J = 6.5 Hz, OCH_2); ^{13}C NMR ($CDCl_3$) δ 73.8 (CCH_2O), 71.5 (OCH_2CH_2), 54.5 (NCH_2), 45.8 (NCH_3), 40.2 (qC), 32.0 ($CH_3CH_2CH_2$), 30.3 (NCH_2CH_2), 29.89, 29.64, 29.50 (2 x 3 octyl CH_2), 26.5 ($OCH_2CH_2CH_2$), 22.8 (CH_3CH_2), 14.3 (CH_3); HR ESI MS *m/z* calcd for $C_{27}H_{58}N_2O_2$ (M+1) 443.4571, found 443.4558.

3,3-Bis(decyloxymethyl)-*N*,*N*,*N*',*N*'-tetramethyl-1,5-pentanediamine (2.14). Reaction of diamide **2.10** (1.9 g, 3.6 mmol) with $LiAlH_4$ (0.54 g, 14 mmol) in THF (50 mL) as for **2.13** gave **2.14** as light brown liquid: yield: 1.1 g (61%); R_F on basic alumina 0.44 (dichloromethane: methanol 96:4); ^1H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 7.0 Hz, CH_3), 1.22-1.45 (m, 28 H, 14x CH_2), 1.49-1.54 (m, 8H, NCH_2CH_2, OCH_2CH_2), 2.27 (s, 12H, NCH_3), 2.37 (t, 4H, J = 7.5, NCH_2), 3.19 (s, 4H, OCH_2), 3.34 (t, 4H, J = 6.5 Hz, OCH_2); ^{13}C NMR ($CDCl_3$) δ 73.8 (CCH_2O), 71.6 (OCH_2CH_2), 54.5 (NCH_2), 45.5

(NCH_3), 40.3(qC), 32.1 ($CH_3CH_2CH_2$), 30.01 (NCH_2CH_2), 29.87, 29.85, 29.79, 29.68, 29.52 (2 x 5 decyl CH_2), 26.5 ($OCH_2CH_2CH_2$), 22.9 (CH_3CH_2), 14.3 (CH_3); HR ESI MS *m/z* calcd for $C_{27}H_{58}N_2O_2$ (M+1) 443.4571, found 443.4558.

3,3-Bis(dodecyloxymethyl)-*N*,*N*,*N*',*N*'-tetramethyl-1,5-pentanediamine (2.15). Reaction of diamide **2.11** (1.9 g, 3.3 mmol) was reacted with $LiAlH_4$ (0.62 g, 16 mmol) in THF (50 mL) as for **2.13** gave **2.15** as a light brown liquid: yield 1.3 g (72%); R_F on basic alumina 0.52 (DCM: methanol 95:5); ^{1}H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 7.0 Hz, CH_3), 1.22-1.45 (m, 36 H, 18x CH_2), 1.45-1.54 (m, 8H, NCH_2CH_2, OCH_2CH_2), 2.21 (s, 12H, NCH_3), 2.26-2.29 (m, 4H, J = 8.5 Hz, NCH_2), 3.18 (s, 4H, OCH_2), 3.34 (t, 4H, J = 6.5 Hz, OCH_2); ^{13}C NMR ($CDCl_3$) δ 73.8 (CCH_2O), 71.5 (OCH_2CH_2), 54.5 (NCH_2), 45.8 (NCH_3), 40.2 (qC), 32.1 ($CH_2CH_2CH_2$), 30.2 (NCH_2CH_2), 29.9 -20.8 (4C), 29.69, 29.52 (2 x 7 dodecyl CH_2), 26.5 ($OCH_2CH_2CH_2$), 22.8 (CH_3CH_2), 14.3 (CH_3); HR ESI MS *m/z* calcd for $C_{35}H_{74}N_2O_2$ (M+1) 555.5823, found 555.5854.

***N*,*N*,*N*',*N*'-Tetramethyl-3,3-bis(tetradecyloxymethyl)-1,5-pentanediamine (2.16)** Reaction of diamide **2.12** (2.2 g, 3.4 mmol) with $LiAlH_4$ (0.65 g, 17 mmol) in THF (50 mL) as for **2.13** gave a residue that was purified by flash column chromatography. Elution using a gradient of 10 to 15% MeOH in DCM gave **2.16** as a light brown liquid: yield 1.7 g (81%); R_F on basic alumina 0.55 (DCM: methanol 95:5); ^{1}H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 7.0 Hz, CH_3), 1.21-1.37 (m, 44 H, 22x CH_2), 1.46-1.54 (m, 8H, NCH_2CH_2, OCH_2CH_2), 2.23 (s, 12H, NCH_3), 2.30-2.33 (m, 4H, J = 8.0 Hz, NCH_2), 3.18 (s, 4H, OCH_2), 3.34 (t, 4H, J = 6.5 Hz, OCH_2); ^{13}C NMR ($CDCl_3$) δ 73.7 (CCH_2O), 71.5 (OCH_2CH_2), 54.4 (NCH_2), 45.5 (NCH_3), 40.2 (qC), 32.1 ($CH_2CH_2CH_3$), 30.0 (NCH_2CH_2), 29.9-29.8, 29.68, 29.51 (tetradecyl CH_2), 26.9 ($OCH_2CH_2CH_2$), 22.8 (CH_3CH_2), 14.3 (CH_3); HR ESI MS *m/z* calcd for $C_{39}H_{82}N_2O_2$ (M+1) 611.6449, found 611.6466.

General procedure for alkylation: *N*,*N*,*N*,*N*',*N*',*N*'-hexamethyl-3,3-bis(octyloxymethyl)-1,5-pentanediammonium diiodide (2.17). A solution of methyl iodide (1.6 g, 11 mmol) and diamine

2.13 (0.50 g, 1.1 mmol) in THF (20 mL) was refluxed for 36 h, then concentrated. The solid residue was purified by flash column chromatography using 10 % methanol in dichloromethane as eluent to give the title product as an off-white solid: yield 0.70 g (85%); mp 223-225 ^{o}C; R_F 0.5 on basic alumina (8% methanol in dichloromethane); ^{1}H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 7.0 Hz, CH_3), 1.22-1.28 (m, 20 H, 10 x CH_2), 1.45 (t, 4H, J = 6.0 Hz, OCH_2CH_2), 1.83 (AA′ part of AA'XX' pattern, 4H, NCH_2CH_2), 3.13 (s, 4H, OCH_2), 3.15 (s, 18H, NCH_3), 3.18 (t, 4H, J= 7.0 Hz, OCH_2), 3.73 (XX part of AA'XX' pattern, 4H, NCH_2); ^{13}C NMR ($CDCl_3$) δ 71.7 (CCH_2O), 71.5 (OCH_2CH_2), 62.8 ($CH_2N(CH_3)_3$), 54.2 (NCH_3), 41.6 (qC), 32.0 ($CH_2CH_2CH_2$), 29.82, 29.58, 29.50 (3 octyl CH_2), 26.6 ($OCH_2CH_2CH_2$), 24.6 (NCH_2CH_2), 22.8 (CH_3CH_2), 14.3 (CH_3); HR ESI MS *m/z* calcd for $C_{29}H_{64}IN_2O_2$ (M-I) 599.4007, found 599.3993.

3,3-Bis(decyloxymethyl)-*N*,*N*,*N*,*N*',*N*',*N*'-hexamethyl-1,5-pentanediammonium diiodide (2.18). Reaction of methyl iodide (1.93 g, 13.7 mmol) and diamine **2.14** (0.68 g, 1.4 mmol) in THF as above gave the title product as an off-white solid: yield 0.80 g (80%); mp 214-216 ^{o}C; R_F 0.5 on basic alumina (7% methanol in dichloromethane); ^{1}H NMR (CDCl3) δ 0.88 (t, 6H, J = 7.0 Hz, CH_3), 1.24-1.35 (m, 28 H, 14 x CH_2), 1.52 (t, 4H, J = 6.5 Hz, OCH_2CH_2), 1.93 (AA′ part of AA'XX' pattern, 4H, NCH_2CH_2), 3.39 (s, 4H, OCH_2), 3.42 (s, 18H, NCH_3), 3.44 (t, 4H, J= 7.0 Hz, OCH_2), 4.02 (XX part of AA'XX' pattern, 4H, NCH_2); ^{13}C NMR ($CDCl_3$) δ 71.7 (CCH_2O), 71.5 (OCH_2CH_2), 62.8 ($CH_2N(CH_3)_3$), 54.2 (NCH_3), 41.5 (qC), 32.1 ($CH_2CH_2CH_2$), 29.85, 29.81, 29.77, 29.62, 29.47 (5 decyl CH_2), 26.5 ($OCH_2CH_2CH_2$), 24.6 (NCH_2CH_2), 22.9 (CH_3CH_2), 14.4 (CH_3); HR ESI MS *m/z* calcd for $C_{33}H_{72}IN_2O_2$ (M-I) 655.4633, found 655.4614.

3,3-Bis(dodecyloxymethyl)-*N*,*N*,*N*,*N*',*N*',*N*'-hexamethyl-1,5-pentanediammonium diiodide (2.19). Reaction of methyl iodide (2.56 g, 18.0 mmol) with diamine **2.14** (1.00 g, 1.80 mmol) as for compound **2.17** above gave the title product as an off-white solid: yield 1.20 g (78%); mp 220-223 ^{o}C; R_F 0.5 on basic alumina (7% methanol in dichloromethane); ^{1}H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 7 .0 Hz, CH_3),

1.23-1.36 (m, 36 H, 18 x CH_2), 1.52 (t, 4H, J = 6.5 Hz, OCH_2CH_2), 1.89 (AA′ part of AA'XX' pattern, 4H, NCH_2CH_2), 3.38 (s, 4H, OCH_2), 3.44 (t, 4H, J = 7.0 Hz, OCH_2), 3.45 (s, 18H, NCH_3), 3.94 (XX' part of AA'XX' pattern, 4H, NCH_2); ^{13}C NMR ($CDCl_3$) 71.7 (CCH_2O), 71.5 (OCH_2CH_2), 62.8 ($CH_2N(CH_3)_3$), 54.2 (NCH_3), 41.5 (qC), 32.1 ($CH_2CH_2CH_2$), 29.90 -29.75 (5C), 29.62, 29.47 (7 dodecyl CH_2), 26.5 ($OCH_2CH_2CH_2$), 24.6 (NCH_2CH_2), 22.9 (CH_3CH_2), 14.3 (CH_3); HR ESI MS *m/z* calcd for $C_{37}H_{80}IN_2O_2$ (M-I) 711.5259, found 711.5239.

***N*,*N*,*N*',*N*',*N*'-Hexamethyl-3,3-bis(tetradecyloxymethyl)-1,5-pentanediammonium diiodide (2.20).** Reaction of methyl iodide (2.32 g, 16.4 mmol) and diamine **2.15** (1.0 g, 1.6 mmol) as for compound **2.17** above (without chromatography) gave a solid residue that was crystallized from dichloromethane and dried under vacuum to give compound **2.20** as an off-white shiny solid: yield 1.2 g (82%); mp 218-221 ^oC; R_F 0.6 on basic alumina (7% methanol in dichloromethane); ^1H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 7.0 Hz, CH_3), 1.27 (m, 44 H, 22x CH_2), 1.52 (t, 4H, J = 6.5 Hz, OCH_2CH_2), 1.93(AA′ part of AA'XX' pattern, 4H, NCH_2CH_2), 3.39 (s, 4H, OCH_2), 3.43 (s, 18H, NCH_3), 3.44 (t, 4H, J = 7.0Hz, OCH_2), 4.03 (XX' part of AA'XX' pattern, 4H, NCH_2); ^{13}C NMR ($CDCl_3$) δ 71.7 (CCH_2O), 71.5 (OCH_2), 62.8 ($CH_2N(CH_3)_3$), 54.1 (NCH_3), 41.4 (qC), 32.1 ($CH_2CH_2CH_2$), 29.90 – 29.75 (7C), 29.64, 29.50 (tetradecyl CH_2), 26.5 ($OCH_2CH_2CH_2$), 24.6 (NCH_2CH_2), 22.8 (CH_3CH_2), 14.2 (CH_3); HR ESI MS *m/z* calcd for $C_{41}H_{88}IN_2O_2$ (M-1) 767.5885, found 767.5907.

General conditions for the Wadsworth-Horner-Emmons reaction: *N*,*N*,*N*′,*N*′-tetramethyl-4,4-bis(octyloxymethyl)-2,5-heptadienediamide (**3.1**). A solution of dry dimethyl sulfoxide (DMSO) (0.47 g, 6.11 mmol) in dichloromethane (2 mL) was added dropwise to a stirred solution of oxalyl chloride (0.38 g, 3.0 mmol) in dichloromethane (5 mL) at -78 ^oC. After the reaction mixture had been stirred for 30 min, a solution of diol **9** (0.50 g, 1.4 mmol) was added and the reaction mixture was stirred for 1.5 h -78 ^oC, then Et_3N (0.98 g, 9.7 mmol) was added slowly. The reaction mixture was stirred for 30 min, allowed to warm to rt, the quenched by addition of a saturated NH_4Cl solution (10

mL). The reaction mixture was extracted with dichloromethane (3 x 50 mL), and the combined organic layers were washed with 2M HCl (5 mL), water (2 x 5mL), brine (5 mL), then dried (Na_2SO_4) and concentrated to give 2,2-bis(octyloxymethyl)propanedial (**17**) as a colorless viscous oil: yield 0.47g, 95%; R_F 0.56 (hexanes: ethyl acetate 8:2); ^{1}H NMR δ 0.88 (t, 6H, J = 7.0 Hz, 2 x Me), 1.26-1.32 (br s, 20H, 10 x CH_2), 1.51 (p, 4H, J = 7.2 Hz, 2 OCH_2CH_2), 3.42 (t, 4H, J = 6.5 Hz, decyl OCH_2), 3.87 (s, 4H, OCH_2C), 9.74 (s, 2H,CHO); ^{13}C NMR δ 199.4 (CHO), 72.3 (CH_2CH_2OC), 68.6 (OCH_2C), 43.8 (q C), 31.7 ($CH_2CH_2CH_3$), 29.76, 29.62, 29.48, 29.44, 29.37 (5 decyl CH_2), 26.12 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.2 (Me); LR ESI *m/z* calcd for $C_{21}H_{40}O_4Na$.MeOH 411.31, found 411.3; for $C_{21}H_{40}O_4Na$.2MeOH 443.33, found 443.3; for 2 $C_{21}H_{40}O_4$+Na+H_2O 753.54, found 753.6.

Diethyl *N*,*N*-dimethylcarbamoylmethylphosphonate[16,22] (4.1 g, 18 mmol) in THF (10 mL) was added in portion to a stirred suspension of NaH (0.45 g, 18 mmol) in THF (80 mL) at rt and the reaction mixture was stirred for 2 h, then cooled to 0 ^{o}C. A solution of dialdehyde **17** (1.6 g, 4.5 mmol) in THF (10 mL) was added and the reaction mixture was stirred for 24 h at rt. A saturated aqueous ammonium chloride solution (20 mL) was added to reaction mixture and then the volatile organic components were removed by concentration. The resulting solution was extracted with ethyl acetate (3 x 30 mL). The combined organic layers were washed with water (2 x 20 mL), brine (20 mL), dried (Na_2SO_4), filtered and concentrated**.** The residue was purified by flash column chromatography using a gradient of 80 to 90% EtOAc in hexanes as eluent, yielding **3.1** as a viscous liquid: yield 1.4g (63%); R_F 0.35 (dichloromethane: methanol 94:6); ^{1}H NMR δ 0.88 (t, 6H, J = 7 Hz, 2 x Me), 1.26-1.34 (br s, 20H, 10 x CH_2), 1.53 (pentet, 4H, J = 7 Hz, 2 OCH_2CH_2), 2.99 (s, 3H, NCH_3), 3.05 (s, 3H, NCH_3), 3.39 (t, 4H, J = 6.5 Hz, decyl OCH_2), 3.49 (s, 4H, OCH_2C), 6.36 (d, 2H, J = 16 Hz, COCHCH), 6.86 (d, 2H, J = 16 Hz, COCHCH); ^{13}C NMR δ 166.8 (C=O), 145.3 (CH=CHC), 121.9 (COCH=), 72.9 (CH_2CH_2OC), 71.9 (OCH_2C), 48.9 (q C), 37.5 (NCH_3), 35.8 (NCH_3), 32.0 ($CH_2CH_2CH_3$), 29.77,

29.61, 29.45 (3 octyl CH_2), 26.4 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.2 (Me); HR ESI MS *m/z* calcd for $C_{29}H_{54}N_2O_4Na$ (M+Na) 517.3976, found 517.3971.

4,4-Bis(decyloxymethyl)-*N*,*N*,*N*',*N*'-tetramethyl-2,5-heptadienediamide (**3.2**)**.** Treatment of diol **10** (2.3 g, 5.5 mmol) in dry dichloromethane with the Swern oxidation mixture [DMSO (1.89 g, 24.3 mmol), oxalyl chloride (1.54 g, 12.2 mmol)] followed by workup as for dial **17** gave 2,2-bis(decyloxymethyl)propanedial (**18**) as a light yellow oil: yield 2.1 g, 92 %; R_F 0.7 (hexanes: ethyl acetate 8:2); ^{1}H NMR δ 0.88 (t, 6H, J = 6.5 Hz, 2 x Me), 1.25-1.31 (brs, 28H, 14 x CH_2), 1.51 (pentet, 4H, J = 7.2 Hz, 2 OCH_2CH_2), 3.41 (t, 4H, J = 6.5 Hz, decyl OCH_2), 3.87 (s, 4H, OCH_2C), 9.74(s, 2H,CHO); ^{13}C NMR δ 199.5 (CHO), 72.3 (CH_2CH_2OC), 68.6 (OCH_2C), 43.7 (q C), 32.0 ($CH_2CH_2CH_3$), 29.72, 29.53, 29.46 (decyl CH_2), 26.13 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.3 (Me); LR ESI MS *m/z* calcd for $C_{25}H_{48}O_4Na$.MeOH 467.37, found 467.4.

A mixture of diethyl *N*,*N*-dimethylcarbamoylmethylphosphonate (4.54 g, 20.4 mmol), NaH (0.49 g, 20.6 mmol) and dialdehyde **18** (2.0 g, 4.8 mmol) in THF (100 mL) was treated as in the preparation of compound **3.1** to give compound **3.2** as a colorless liquid: yield, 1.7 g (64%); R_F 0.39 (dichloromethane: methanol 95:5); ^{1}H NMR δ 0.88 (t, 6H, J = 7 Hz, 2 x Me), 1.26-1.31 (br s, 28H, 14 x CH_2), 1.52 (pentet, 4H, J = 7 Hz, 2 OCH_2CH_2), 2.99 (s, 3H, NCH_3), 3.02 (s, 3H, NCH_3), 3.39 (t, 4H, J = 6.5 Hz, decyl OCH_2), 3.49 (s, 4H, OCH_2C), 6.36 (d, 2H, J = 15.5 Hz, COCH=), 6.86 (d, 2H, J = 15.5 Hz, =CHC); ^{13}C NMR δ 166.8 (C=O), 145.3 (CH=*C*HC), 121.9 (CO*C*H=), 72.9 (CH_2CH_2OC), 71.9 (OCH_2C), 48.9 (q C), 37.5 (NCH_3), 35.8 (NCH_3), 32.0 ($CH_2CH_2CH_3$), 29.79, 29.76, 29.66, 29.49 (5 decyl CH_2), 26.4 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.2 (Me); HR ESI MS *m/z* calcd for $C_{33}H_{62}N_2O_4Na$ (M+Na) 573.4602, found 573.4592.

4,4-Bis(dodecyloxymethyl)-*N*,*N*,*N*',*N*'-tetramethyl-2,5-heptadienediamide (**3.3**). Treatment of diol **11** (1.3 g, 2.8 mmol) in dry dichloromethane (30 mL) with the Swern oxidation mixture [DMSO (1.74 g, 22.3 mmol), oxalyl chloride (1.42 g, 11.2 mmol)] as for dial **17** gave 2,2-

bis(dodecyloxymethyl)propanedial (**19**) as a light yellow oil: yield 1.2 g, 92 %; R_F 0.7 (hexanes: ethyl acetate 8:2); ^{1}H NMR δ 0.88 (t, 6H, J = 7 Hz, 2 x Me), 1.25-1.32 (br s, 36H, 18 x CH_2), 1.52 (pentet, 4H, J = 7.2 Hz, 2 OCH_2CH_2), 3.41 (t, 4H, J = 6.5 Hz, decyl OCH_2), 3.87 (s, 4H, OCH_2C), 9.74 (s, 2H,CHO); ^{13}C NMR δ 199.4 (CHO), 72.3 (CH_2CH_2OC), 68.6 (OCH_2C), 43.7 (q C), 32.1 ($CH_2CH_2CH_3$), 29.81, 29.78, 29.72, 29.53, 29.44, (decyl CH_2), 26.1 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.2 (Me).

A mixture of diethyl *N*,*N*-dimethylcarbamoylmethylphosphonate (2.67 g, 11.9 mmol), NaH (0.29 g,12 mmol) and dialdehyde **19** (1.1 g, 2.3 mmol) in THF(100 mL) was treated as in the preparation of compound **3.1** to give compound **3.3** as a colorless solid: yield 1.25 g (69%); mp 52-54 ^oC; R_F 0.42 (dichloromethane: methanol 95:5); ^{1}H NMR δ 0.88 (t, 6H, J = 7 Hz, 2 x Me), 1.26-1.32 (br s, 36H, 18 x CH_2), 1.52 (pentet, 4H, J = 7 Hz, 2 OCH_2CH_2), 2.99 (s, 3H, NCH_3), 3.05 (s, 3H, NCH_3), 3.39 (t, 4H, J = 6.5 Hz, decyl OCH_2), 3.49 (s, 4H, OCH_2C), 6.36 (d, 2H, J = 15.5 Hz, COCH=), 6.86 (d, 2H, J = 15.5 Hz, =CHC); ^{13}C NMR δ 166.8 (C=O), 145.3 (CH=*C*HC), 121.9 (CO*C*H=), 72.9 (CH_2CH_2OC), 71.9 (OCH_2C), 48.9 (qC), 37.5 (NCH_3), 35.8 (NCH_3), 32.1 ($CH_2CH_2CH_3$), 29.86, 29.82, 29.68, 29.52 (5 dodecyl CH_2), 26.4 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.3 (Me); HR ESI MS *m/z* calcd for $C_{37}H_{70}N_2O_4Na$ (M+Na) 629.5228, found 629.5244.

***N*,*N*,*N′*,*N′*-Tetramethyl-4,4-bis(tetradecyloxymethyl)-2,5-heptadienediamide** (**3.4**). Treatment of diol **12** (3.0 g, 5.7 mmol) in dry DCM (30 mL) with the Swern oxidation mixture [DMSO (2.03 g, 26.1 mmol), oxalyl chloride (1.65 g, 13.0 mmol)] as for dial **17** gave 2,2-bis(tetradecyloxymethyl)propanedial (**20**) as a light yellow oil: yield 2.5 g, 86 %; R_F 0.7 (hexanes: ethyl acetate 8:2); ^{1}H NMR δ 0. 0.88 (t, 6H, J = 7 Hz, 2 x Me), 1.25-1.32 (br s, 36H, 18 x CH_2), 1.51 (pentet, 4H, J = 7.2 Hz, 2 OCH_2CH_2), 3.40 (t, 4H, J = 6.5 Hz, decyl OCH_2), 3.87 (s, 4H, OCH_2C), 9.74 (s, 2H,CHO); ^{13}C NMR δ 199.5 (CHO), 72.4 (CH_2CH_2OC), 68.6 (OCH_2C), 43.7 (q C), 32.1

($CH_2CH_2CH_3$), 29.81, 29.77, 29.52, 29.45 (decyl CH_2), 26.1 ($CH_2CH_2CH_2O$), 22.9 (CH_2CH_3), 14.2 (Me); LR ESI MS *m/z* calcd for $C_{33}H_{64}O_4Na$.MeOH, 579.50, found 579.5.

A mixture of diethyl *N*,*N*-dimethylcarbamoylmethylphosphonate (4.68 g, 20.9 mmol), NaH (0.51 g, 21 mmol) and dialdehyde **20** (2.5 g, 4.7 mmol) in THF (100 mL) was treated as in the preparation of compound **3.1** to give compound **3.4** as a colorless solid: yield 2.0 g (64%); mp 59-61 ^oC.; R_F 0.43 (dichloromethane: methanol 95:5); ^1H NMR δ 0.88 (t, 6H, J = 7 Hz, 2 x Me), 1.26-1.32 (br s, 44H, 22 x CH_2), 1.53 (pentet, 4H, J = 7 Hz, 2 OCH_2CH_2), 2.99 (s, 3H, NCH_3), 3.05 (s, 3H, NCH_3), 3.39 (t, 4H, J = 6.5 Hz, decyl OCH_2), 3.50 (s, 4H, OCH_2C), 6.37 (d, 2H, J = 15.5 Hz, COCH=), 6.86 (d, 2H, J = 15.5 Hz, =CHC); ^{13}C NMR δ 166.8 (C=O), 145.3 (CH=*C*HC), 121.9 (CO*C*H=), 72.9 (CH_2CH_2OC), 71.9 (OCH_2C), 48.9 (q C), 37.5 (NCH_3), 35.8 (NCH_3), 32.1 ($CH_2CH_2CH_3$), 29.86, 29.82, 29.77, 29.68, 29.52 (5 tetradecyl CH_2), 26.4 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.3 (Me); HR ESI MS *m/z* calcd for$C_{41}H_{78}N_2O_4Na$ (M+Na) 685.5854, found 685.5814.

General procedure for hydrogenation: *N*,*N*,*N′*,*N′*-Tetramethyl-4,4-bis(octyloxymethyl)heptanediamide (**3.5**). A mixture of **3.1** (0.5 g, 1.0 mmol) and 10% Pd/C wet Degusa type catalyst (50 mg) in ethyl acetate (50 mL) was stirred under H_2 at atmospheric pressure for 24 h. The reaction mixture was filtered using a celite bed and the filtrate was concentrated to give compound **3.5** as a colorless liquid: yield 0.46 g (91%); R_F 0.5 (dichloromethane: methanol 95:5); ^1H NMR δ 0.88 (t, 6H, J = 6.5 Hz, 2 x Me), 1.25-1.31 (br s, 20H, 10 x CH_2), 1.50 (pentet, 4H, J = 7 Hz, 2 OCH_2CH_2), 1.61 (4H, AA′XX′ pattern, CCH_2), 2.32 (4H, AA′XX′ pattern, $COCH_2$), 2.99 (s, 3H, NCH_3), 3.07 (s, 3H, NCH_3), 3.22 (s, 4H, OCH_2C), 3.34 (t, 4H, J = 6.5 Hz, decyl OCH_2); ^{13}C NMR δ 173.8 (C=O), 73.9 (CH_2CH_2OC), 71.7 (OCH_2C), 40.6 (qC), 37.4 (NCH_3), 35.6 (NCH_3), 32.0 ($CH_2CH_2CH_3$), 29.90, 29.64, 29.48 (3 octyl CH_2), 28.04, 27.94 ($COCH_2CH_2$, $COCH_2$), 26.5 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.3 (Me); HR ESI MS *m/z* calcd for $C_{29}H_{58}N_2O_4Na$ (M+Na) 521.4289, found 521.4278.

4,4-Bis(decyloxymethyl)-*N*,*N*,*N*',*N*'-tetramethyl-2,5-heptanediamide (**3.6**). Hydrogenation of compound **3.2** (1.0 g, 1.8 mmol) as for compound **3.1** gave the title compound as a colorless liquid: yield 0.90 g (89%); R_F 0.39 (dichloromethane: methanol 95:5); ^{1}H NMR δ 0.88 (t, 6H, J = 7 Hz, 2 x Me), 1.25-1.31 (br s, 28H, 14 x CH_2), 1.51 (pentet, 4H, J = 7 Hz, 2 OCH_2CH_2), 1.61 (4H, AA′XX′ pattern, CCH_2), 2.32 (4H, AA′XX′ pattern, $COCH_2$), 2.92 (s, 3H, NCH_3), 3.00 (s, 3H, NCH_3), 3.22 (s, 4H, OCH_2C), 3.34 (t, 4H, J = 6.5 Hz, decyl OCH_2); ^{13}C NMR δ 173.8 (C=O), 73.9 (CH_2CH_2OC), 71.7 (OCH_2C), 40.6 (q C), 37.4 (NCH_3), 35.6 (NCH_3), 32.0 ($CH_2CH_2CH_3$), 29.90, 29.82, 29.77, 29.67, 29.51 (5 decyl CH_2), 28.04, 27.94 ($COCH_2CH_2$, $COCH_2$), 26.5 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.3 (Me); HR ESI MS *m/z* calcd for $C_{33}H_{66}N_2O_4Na$ 577.4915, found 577.4918.

4,4-Bis(dodecyloxymethyl)-*N*,*N*,*N*',*N*'-tetramethyl-2,5-heptanediamide (**3.7**). Hydrogenation of compound **3.3** (0.70 g, 1.2 mmol) as for compound **3.1** gave compound **3.7** as a colorless solid: yield 0.65 g (92%); mp 46-48 °C; R_F 0.42 (dichloromethane: methanol 95:5); ^{1}H NMR δ 0.88 (t, 6H, J = 7 Hz, 2 x Me), 1.25-1.32 (br s, 36H, 18 x CH_2), 1.52 (pentet, 4H, J = 7 Hz, 2 OCH_2CH_2), 1.62 (4H, AA′XX′ pattern, CCH_2), 2.33 (4H, AA′XX′ pattern, $COCH_2$), 2.92 (s, 3H, NCH_3), 3.00 (s, 3H, NCH_3), 3.22 (s, 4H, OCH_2C), 3.34 (t, 4H, J = 6.5 Hz, decyl OCH_2); ^{13}C NMR δ 173.8 (C=O), 73.9 (CH_2CH_2OC), 71.7 (OCH_2C), 40.6 (q C), 37.4 (NCH_3), 35.5 (NCH_3), 32.1 ($CH_2CH_2CH_3$), 29.90, 29.83, 29.70, 29.52 (7 dodecyl CH_2), 28.05, 27.94 ($COCH_2CH_2$, $COCH_2$), 26.5 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.3 (Me); HR ESI MS *m/z* calcd for $C_{37}H_{74}N_2O_4Na$ (M+Na) 633.5541, found 633.5562.

***N*,*N*,*N*′,*N*′-Tetramethyl-4,4-bis(tetradecyloxymethyl)heptanediamide** (**3.8**). Hydrogenation of compound **3.3** (1.60 g, 2.4 mmol) as for compound **3.1** gave the title compound as a colorless solid: yield, 1.50 g (94%); mp 47-49 °C; R_F 0.43 (dichloromethane: methanol 95:5); ^{1}H NMR δ 0.88 (t, 6H, J = 7 Hz, 2 x Me), 1.25-1.32 (br s, 36H, 18 x CH_2), 1.50 (pentet, 4H, J = 7 Hz, 2 OCH_2CH_2), 1.62 (4H, AA′XX′ pattern, CCH_2), 2.33 (4H, AA′XX′ pattern, $COCH_2$), 2.92 (s, 3H, NCH_3), 3.00 (s, 3H, NCH_3), 3.22 (s, 4H, OCH_2C), 3.33 (t, 4H, J = 6.5 Hz, decyl OCH_2); ^{13}C NMR δ 173.8 (C=O), 74.0

(CH_2CH_2OC), 71.7 (OCH_2C), 40.6 (q C), 37.4 (NCH_3), 35.5 (NCH_3), 32.1 ($CH_2CH_2CH_3$), 29.87, 29.84, 29.70, 29.53 (9 tetradecyl CH_2), 28.04, 27.94 ($COCH_2CH_2$, $COCH_2$), 26.5 ($CH_2CH_2CH_2O$), 22.9 (CH_2CH_3), 14.4 (Me); HR ESI MS *m/z* calcd for $C_{41}H_{82}N_2O_4Na$ (M+Na) 689.6167, found 689.6152.

General procedure for amide reduction: *N*,*N*,*N*',*N*'-Tetramethyl-4,4-bis(octyloxymethyl)-1,7-heptanediamine (3.9). Diamide **3.5** (0.6 g, 1.2 mmol) was added dropwise to a stirred suspension of $LiAlH_4$ (0.18 g, 4.8 mmol) in THF at 0 °C. The reaction mixture was stirred at rt for 6 h, then the excess of LAH was decomposed by dropwise addition of ethyl acetate (50 mL), water (0.3 mL), then 1M NaOH (0.3 mL) at 10 °C. The mixture was filtered on a bed of celite, which was then washed with hot ethyl acetate. The combined filtrate and washings were dried (Na_2SO_4), then concentrated to a residue that was purified by flash column chromatography. Elution using a gradient of 5 to 15% MeOH in dichloromethane gave the title compound as a light brown liquid: yield 0.45 g (80%); R_F on basic alumina 0.71 (dichloromethane: methanol 94:6); ^1H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 7.0 Hz, CH_3), 1.21-1.33 (m, 24 H, 10 octyl CH_2, 2 CCH_2), 1.37-1.43 (m, 4H, NCH_2CH_2), 1.51 (pentet, 4H, J = 7 Hz, OCH_2CH_2), 2.201 (t, 4H, J = 7.5 Hz, NCH_2), 2.206 (s, 12H, NCH_3), 3.17 (s, 4H, OCH_2), 3.33 (t, 4H, J = 6.5 Hz, OCH_2); ^{13}C NMR ($CDCl_3$) δ 73.5 (CCH_2O), 71.5 (OCH_2), 60.9 (NCH_2), 45.7 (NCH_3), 41.0 (qC), 32.0 ($CH_2CH_2CH_3$), 29.85, 29.76, 29.65, 29.50 (3 octyl CH_2, CCH_2), 26.5 ($OCH_2CH_2CH_2$), 22.8 (CH_2CH_3), 21.45 (NCH_2CH_2), 14.3 (Me); HR ESI MS *m/z* calcd for $C_{29}H_{63}N_2O_2$ (M+H) 471.4884, found 471.4885.

4,4-Bis(decyloxymethyl)-*N*,*N*,*N*',*N*'-tetramethyl-1,7-heptanediamine (3.10). Diamide **3.6** (0.90 g, 1.6 mmol) was reacted with $LiAlH_4$ (0.3 g, 8 mmol) as above to give the title compound as a light brown liquid: yield 0.60 g (70%); R_F on basic alumina 0.52 (DCM: methanol 95:5); ^1H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 7.0 Hz, CH_3), 1.21-1.33 (m, 32 H, 14 decyl CH_2, 2 CCH_2), 1.39-1.44 (m, 4H, NCH_2CH_2), 1.51 (pentet, 4H, J = 7 Hz, 2 x OCH_2CH_2), 2.202 (t, 4H, J = 7.5 Hz, NCH_2), 2.205 (s, 12H,

NCH_3), 3.17 (s, 4H, OCH_2), 3.33 (t, 4H, J = 6.5 Hz, OCH_2); ^{13}C NMR ($CDCl_3$) δ 73.5 (C*C*H_2O), 71.53 (OCH_2), 60.9 (NCH_2), 45.7 (NCH_3), 41.0 (qC), 32.1 (*C*$H_2CH_2CH_3$), 29.85, 29.77, 29.70, 29.52 (5 decyl CH_2, C*C*H_2), 26.5 ($OCH_2CH_2CH_2$), 22.8 (*C*H_2CH_3), 21.4 (NCH_2*C*H_2), 14.3 (Me); HR ESI MS *m/z* calcd for $C_{33}H_{71}N_2O_2$ (M+H) 527.5510, found 527.5502.

4,4-Bis(dodecyloxymethyl)-*N*,*N*,*N*',*N*'-tetramethyl-1,7-heptanediamine (3.11). Diamide **3.7** (0.64 g, 1.0 mmol) was reacted with $LiAlH_4$ (0.16 g, 4.2 mmol) as above to give the title compound as a light brown liquid: yield 0.55 g (90%); R_F on basic alumina 0.7 (DCM: methanol 93:7); ^1H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 7.0 Hz, CH_3), 1.21-1.33 (m, 40 H, 18 dodecyl CH_2, 2 CCH_2), 1.37-1.43 (m, 4H, NCH_2C*H*$_2$), 1.51 (pentet, 4H, J = 7 Hz, 2 x OCH_2C*H*$_2$), 2.186 (t, 4H, J = 7.5 Hz, NCH_2), 2.189 (s, 12H, NCH_3), 3.17 (s, 4H, OCH_2), 3.33 (t, 4H, J = 6.5 Hz, OCH_2); ^{13}C NMR ($CDCl_3$) δ 73.5 (C*C*H_2O), 71.5 (OCH_2), 60.9 (NCH_2), 45.7 (NCH_3), 40.9 (qC), 32.1 (*C*$H_2CH_2CH_3$)**,** 29.85, 29.82, 29.74, 29.52 (7 dodecyl CH_2, C*C*H_2), 26.5 ($OCH_2CH_2CH_2$), 22.8 (CH_3*C*H_2), 21.4 (NCH_2*C*H_2), 14.3 (Me); HR ESI MS *m/z* calcd for $C_{37}H_{79}N_2O_2$ (M+H) 583.6136, found 583.6123.

***N*,*N*,*N*',*N*'-Tetramethyl-4,4-bis(tetradecyloxymethyl)-1,7-heptanediamine (3.12).** Diamide **3.8** (1.0 g, 1.5 mmol) was reacted with $LiAlH_4$ (0.23 g, 6.0 mmol) as above to give the title compound as a light brown liquid: yield 0.60 g (68%); R_F on basic alumina 0.52 (dichloromethane: methanol 96:4); ^1H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 7.0 Hz, CH_3), 1.21-1.35 (m, 48 H, 22 tetradecyl CH_2, 2 CCH_2), 1.38-1.43 (m, 4H, NCH_2C*H*$_2$), 1.51 (pentet, 4H, J = 7 Hz, 2 x OCH_2C*H*$_2$), 2.209 (t, 4H, J = 7.5 Hz, NCH_2), 2.211 (s, 12H, NCH_3), 3.17 (s, 4H, OCH_2), 3.33 (t, 4H, J = 6.5 Hz, OCH_2); ^{13}C NMR ($CDCl_3$) δ 73.5 (C*C*H_2O), 71.5 (OCH_2), 60.9 (NCH_2), 45.6 (NCH_3), 40.9 (qC), 32.1 (*C*$H_2CH_2CH_3$), 29.87, 29.77, 29.71, 29.52 (9 tetradecyl CH_2, C*C*H_2), 26.5 ($OCH_2CH_2CH_2$), 22.9 (*C*H_2CH_3), 21.4 (NCH_2*C*H_2), 14.3 (Me); HR ESI MS *m/z* calcd for $C_{41}H_{87}N_2O_2$ (M+1) 639.6762, found 639.6744.

General procedure for alkylation: *N*,*N*,*N*,*N*',*N*',*N*'-Hexamethyl-4,4-bis(octyloxymethyl)-1,7-heptanediammonium diiodide (3.13). Methyl iodide (1.51 g, 10.6 mmol) was added to a stirred

solution of amine **3.9** (0.5 g, 1.0 mmol) in THF (30 mL). The reaction mixture was refluxed for 12 h, then concentrated. The solid residue was purified by flash column chromatography using 10 % methanol in dichloromethane as eluent to give the title compound as an off-white solid, yield: 0.75 g (94%); mp 233-236 ^{o}C; R_F 0.5 on basic alumina (7% methanol in dichloromethane); ^{1}H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 7.0 Hz, CH_3), 1.23-1.31 (m, 20 H, 10 x CH_2), 1.43 (m, 4H, J = 8.2 Hz $NCH_2CH_2C\mathit{H}_2$), 1.51 (pentet, 4H, J = 6.5 Hz, 2 x $OCH_2C\mathit{H}_2$), 1.88 (m, 4H, $NCH_2C\mathit{H}_2$), 3.21 (s, 4H, OCH_2), 3.36 (t, 4H, J = 6.5 Hz, OCH_2), 3.44 (s, 18H, NCH_3), 3.73 (m, 2H, NCH_2); ^{13}C NMR ($CDCl_3$) δ 72.5 ($C\mathit{C}H_2O$), 71.6 (OCH_2), 67.8 (NCH_2), 54.5 (NCH_3), 41.4 (qC), 32.0 ($\mathit{C}H_2CH_2CH_3$), 29.82, 29.62, 29.53 (octyl CH_2), 26.7 ($C\mathit{C}H_2$), 26.5 ($OCH_2CH_2\mathit{C}H_2$), 22.8 ($CH_3\mathit{C}H_2$), 17.8 ($NCH_2\mathit{C}H_2$), 14.3 (Me); HR ESI MS *m/z* calcd for $C_{31}H_{68}IN_2O_2$ (M-I) 627.4320, found 627.4267.

4,4-Bis(decyloxymethyl)-*N*,*N*,*N*,*N*',*N*',*N*'-hexamethyl-1,7-heptanediammonium diiodide (3.14). Alkylation of amine **3.10** (0.5 g, 0.9 mmol) with methyl iodide (1.34 g, 9.43 mmol) as above gave the title product as an off-white solid: yield 0.70 g (91%); mp 240-243 ^{o}C; R_F 0.5 on basic alumina (7% methanol in dichloromethane); ^{1}H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 7.0 Hz, CH_3), 1.23-1.33 (m, 28 H, 14 x CH_2), 1.43 (m, 4H, $NCH_2CH_2C\mathit{H}_2$), 1.51 (pentet, 4H, J = 6.5 Hz, 2 x $OCH_2C\mathit{H}_2$), 1.88 (m, 4H, $NCH_2C\mathit{H}_2$), 3.22 (s, 4H, OCH_2), 3.37 (t, 4H, J = 6.5 Hz, OCH_2), 3.45 (s, 18H, NCH_3), 3.72 (m, 2H, NCH_2); ^{13}C NMR ($CDCl_3$) δ 72.5 ($C\mathit{C}H_2O$), 71.6 (OCH_2), 67.8 (NCH_2), 54.4 (NCH_3), 41.4 (qC), 32.1 ($\mathit{C}H_2CH_2CH_3$), 29.88, 29.79, 29.67, 29.50 (5 decyl CH_2), 26.7 ($C\mathit{C}H_2$), 26.5 ($OCH_2CH_2CH_2$), 22.8 ($CH_3\mathit{C}H_2$), 17.9 ($NCH_2\mathit{C}H_2$), 14.3 (Me); HR ESI MS *m/z* calcd for $C_{35}H_{76}IN_2O_2$ (M-I) 683.4946, found 683.4895.

4,4-Bis(dodecyloxymethyl)-*N*,*N*,*N*,*N*',*N*',*N*'-hexamethyl-1,7-heptanediammonium diiodide (3.15). Alkylation of amine **3.11** (0.50 g, 0.8 mmol) with methyl iodide (1.2 g, 8.4 mmol) as above gave the title product as an off-white solid: yield 0.65 g (88%); mp 252-254 ^{o}C; R_F 0.5 on basic alumina (7% methanol in dichloromethane); ^{1}H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 7.0 Hz, CH_3), 1.22-1.34 (m, 20 H,

10 x CH_2), 1.41 (m, 4H, $NCH_2CH_2CH_2$), 1.51 (pentet, 4H, J = 6.5 Hz, 2 x OCH_2CH_2), 1.87 (m, 4H, NCH_2CH_2), 3.22 (s, 4H, OCH_2), 3.37 (t, 4H, J = 6.5 Hz, OCH_2), 3.46 (s, 18H, NCH_3), 3.71 (m, 4H, 2H, NCH_2); ^{13}C NMR ($CDCl_3$) δ 72.5 (CCH_2O), 71.6 (OCH_2), 67.8 (NCH_2), 54.4 (NCH_3), 41.3 (qC), 32.1 ($CH_2CH_2CH_3$), 29.86, 29.82, 29.69, 29.51 (decyl CH_2), 26.72, 26.48 ($OCH_2CH_2CH_2$), 22.83 (CH_3CH_2), 17.8 (NCH_2CH_2), 14.3 (Me); HR ESI MS *m/z* calcd for $C_{39}H_{84}IN_2O_2$ (M-I) 739.5572, found 739.5548.

***N*,*N*,*N*,*N*',*N*',*N*'-Hexamethyl-4,4-bis(tetradecyloxymethyl)-1,7-heptanediammonium diiodide (3.16).** Alkylation of amine **3.12** (0.5 g, 0.9 mmol) with methyl iodide (0.89 g, 6.2 mmol) as above gave the title product as an off-white solid: yield 0.51 g (88%); mp 238 – 241 °C; R_F 0.47 on basic alumina (8% methanol in dichloromethane); ^1H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 7.0 Hz, CH_3), 1.22-1.32 (m, 44 H, 22 x CH_2), 1.44 (m, 4H, $NCH_2CH_2CH_2$), 1.51 (pentet, 4H, J = 6.5 Hz, 2 x OCH_2CH_2), 1.89 (m, 4H, NCH_2CH_2), 3.22 (s, 4H, OCH_2), 3.37 (t, 4H, J = 6.5 Hz, OCH_2), 3.44 (s, 18H, NCH_3), 3.74 (m, 4H, NCH_2); ^{13}C NMR ($CDCl_3$) δ 72.4 (CCH_2O), 71.6 (OCH_2), 67.8 (NCH_2), 54.5 (NCH_3), 41.3 (qC), 32.1 (OCH_2CH_2), 29.87, 29.82, 29.70, 29.52 (decyl CH_2), 26.61, 26.48 ($OCH_2CH_2CH_2$), 22.8 (CH3CH_2), 17.9 (NCH_2CH_2), 14.3 (Me); HR ESI MS *m/z* calcd for $C_{43}H_{92}IN_2O_2$, 795.6198, found 795.6215.

General procedure for reaction of dialkoxides with 2-(dimethylamino)ethyl chloride hydrochloride: *N*,*N*,*N'*,*N'*-tetramethyl-5,5-bis(octyloxymethyl)-3,7-dioxa-1,9-nonanediaminium dichloride (4.5). Sodium hydride (3.36 g, 0.14 mol, 10 eq) was added slowly to a stirred solution of 2,2-dioctyloxymethyl-1,3-propanediol (**9**) (5.05 g, 0.014 mol) in DMF (500 mL) under nitrogen gas at rt and the mixture was stirred vigorously for 1 h. 2-(Dimethylamino)ethyl chloride hydrochloride (8.08 g, 0.05 mol, 4 eq) was added and the reaction mixture was stirred at 50 °C under an N_2 atmosphere for 12 h, then quenched with methanol. The mixture was filtered and concentrated. The residue was taken up in diethyl ether (50 mL) and the resulting solution was washed with brine (50 mL), dried ($MgSO_4$) and concentrated at 30-35 °C to give *N*,*N*,*N'*,*N'*-tetramethyl-5,5-

bis(octyloxymethyl)-3,7-dioxa-1,9-nonanediamine (**4.1**) as a orange oil. The crude product was taken up in dichloromethane (50 mL) and the resulting solution was shaken with ice cold 2 M HCl (30 mL). The aqueous layer was diluted with brine and this solution was extracted with dichloromethane (5 x 30 mL). The combined organic layers were dried ($MgSO_4$) and concentrated to give the title compound (**4.5**) as a light yellow solid, that was crystallized from ethyl acetate and acetone to give colorless crystals: yield 4.90 g, 61 %; mp 150-152 ^oC; R_F on basic alumina 0.47 (dichloromethane: ethanol 96 :4); ^1H NMR δ 0.88 (t, 6H, J = 6.8 Hz, 2 x Me), 1.27 (brs, 20H, 10 x CH_2), 1.50 (pentet, 4H, J = 6.3 Hz, 2 OCH_2CH_2), 2.92 (s, 12H, 2 x $N(CH_3)_2$), 3.33 (t, 4H, J = 6.7 Hz, octyl OCH_2), 3.34 (s over broad pattern, 8H, octylOCH_2C and OCH_2CH_2N), 3.51 (s, 4H, $N(CH_2)_2OCH_2C$), 3.90 (XX` part of AA`XX`pattern, 4H, OCH_2CH_2N), 12.0 (br s, 2H, NH); ^{13}C NMR δ 71.7 (CH_2CH_2OC), 70.6 ($CCH_2O(CH_2)_2N$), 69.5 (CCH_2O octyl), 65.8 (OCH_2CH_2N), 56.7 (NCH_2CH_2), 45.1 (q C), 43.6 ($N(CH_3)_2$), 32.0 ($CH_2CH_2CH_3$), 29.7, 29.5, 29.3, (octyl CH_2), 26.2 ($CH_2CH_2CH_2O$), 22.7 (CH_2CH_3), 14.2 (Me); HR ESI MS *m/z* calcd for $C_{29}H_{63}N_2O_4$ (M+H) 503.4788, found 503.4784.

5,5-Bis(decyloxymethyl)-*N,N,N′,N′*-tetramethyl-3,7-dioxa-1,9-nonanediaminium dichloride (4.6)

Compound **10** (5.03 g, 0.012 mol) in DMF (700 mL) with sodium hydride (4.83 g, 0.12 mol, 10 eq) and 2-(dimethylamino)ethyl chloride hydrochloride (6.91 g, 0.05 mol, 4 eq) as above gave 5,5-bis(decyloxymethyl)-*N,N,N′,N′*-tetramethyl-3,7-dioxa-1,9-nonanediamine (**4.2**) as a orange oil. Treatment with ice cold 2 M HCl (30 mL) as above gave the title compound (**4.6**) as a yellow crystalline solid, that was crystallized from ethyl acetate and acetone to give light yellow crystals: yield 5.0 g, 66 %; mp 154-155 ^oC; R_F on basic alumina 0.52 (dichloromethane: ethanol 96 : 4); ^1H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.26 (brs, 28H, 14 x CH_2), 1.50 (pentet, 4H, J = 6.4 Hz, 2 OCH_2CH_2), 2.91 (s, 12H, 2 x $N(CH_3)_2$), 3.30 (br AA`part of AA`XX` pattern 4H, $OCH_2CH_2N$), 3.33 (t, 4H, J = 6.6 Hz, decyl $OCH_2$), 3.34 (s, 4H, decyl$OCH_2C$), 3.51 (s, 4H, $N(CH_2)_2OCH_2C$), 3.90 (XX` part of AA`XX`pattern, 4H, OCH_2CH_2N); 12.2 (br s, 2H, NH);^{13}C NMR δ 71.8 (CH_2CH_2OC), 70.7

(C*C*H_2O(CH_2)$_2$N), 69.5 (C*C*H_2Odecyl), 65.8 (O*C*$H_2$$CH_2$N), 56.8 (N*C*$H_2$$CH_2$), 45.2 (q C), 43.7 (N($CH_3$)$_2$), 32.0 (*C*$H_2$$CH_2$$CH_3$), 29.73, 29.71, 29.68, 29.57, (decyl CH_2), 29.40 (OCH_2*C*H_2), 26.3 (*C*$H_2$$CH_2$$CH_2$O), 22.7 (*C*$H_2$$CH_3$), 14.2 (Me), HR ESI MS *m/z* calcd for $C_{33}H_{71}N_2O_4$ (M+H) 559.5411, found 559.5411.

5,5-Bis(dodecyloxymethyl)-*N,N,N′,N′*-tetramethyl-3,7-dioxa-1,9-nonanediaminium dichloride (4.7). Treatment of compound **11** (5.040 g, 0.0106 mol) in DMF (700 mL) with sodium hydride (4.26 g, 0.106 mol, 10 eq) and 2-(dimethylamino)ethyl chloride hydrochloride (6.10 g, 0.042 mol, 4 eq) as above gave 5,5-bis(dodecyloxymethyl)-*N,N,N′,N′*-tetramethyl-3,7-dioxa-1,9-nonanediamine (**4.3**) as a orange oil, that was taken up in dichloromethane (30 mL). This solution was shaken with ice cold 2 M HCl (30 mL) as for **4.5** to give a colorless crystalline solid that was recrystallized from ethyl acetate and acetone to give colorless crystals: yield 5.20 g, 71%; mp 145 ^{o}C; R_F on basic alumina 0.55 (dichloromethane: ethanol 96 :4); ^{1}H NMR δ 0.88 (t, 6H, J = 6.8 Hz, 2 x Me), 1.26 (br s, 36H, 10 x CH_2), 1.50 (pentet, 4H, J = 6.3 Hz, 2 OCH$_2$C*H*$_2$), 2.90 (s, 12H, 2 x N(CH_3)$_2$), 3.30 (AA`part of AA`XX` pattern, 4H, $J_{AX}$ + $J_{A'X}$ = 9.3Hz OCH$_2$*CH*$_2$N ), 3.33 (t, 4H, J = 6.5 Hz, dodecyl OCH$_2$), 3.34 (s, 4H, dodecylOCH$_2$C), 3.51 (s, 4H, N(CH$_2$)$_2$OC*H*$_2$C), 3.90 (XX` part of AA`XX`pattern, 4H, J_{AX} + $J_{A'X}$ = 9.3 Hz OC*H*$_2$CH$_2$N); 12.2 (br s, 2H, NH); ^{13}C NMR δ 71.7 (CH$_2$*C*H$_2$OC), 70.6 (C*C*H$_2$O(CH$_2$)$_2$N), 69.5 (C*C*H$_2$Ododecyl), 65.7 (O*C*H$_2$CH$_2$N), 56.7 (N*C*H$_2$CH$_2$), 45.1 (q C), 43.6 (N(CH_3)$_2$), 31.9 (*C*H$_2$CH$_2$CH$_3$), 29.7, 29.5, 29.4 (dodecyl CH_2), 26.2 (*C*H$_2$CH$_2$CH$_2$O), 22.7 (*C*H$_2$CH$_3$), 14.1 (Me), HR ESI MS *m/z* calcd for $C_{37}H_{79}N_2O_4$ (M+H) 615.6040, found 615.6046.

***N,N,N′,N′*-Tetramethyl-3,7-dioxa-5,5-bis(tetradecyloxymethyl)-1,9-nonanediaminium dichloride (4.8).** Treatment of compound **12** (5.28 g, 0.01 mol) in DMF (700 mL) with sodium hydride (4.0 g, 0.10 mol, 10 eq) and 2-(dimethylamino)ethyl chloride hydrochloride (5.7 g, 0.040 mol, 4 eq) as above gave *N,N,N′,N′*-tetramethyl-3,7-dioxa-5,5-bis(tetradecyloxymethyl)-1,9-nonanediamine (**4.4**) as a orange oil. Treatment with ice cold 2 M HCl (30 mL) as above gave the title compound (**4.8**) as a

colorless crystalline solid, that was recrystallized from ethyl acetate and acetone to give colorless crystals: yield 5.55 g, 75 % ; mp 148-150 ^{o}C; R_F on basic alumina 0.57 (dichloromethane: ethanol 96 :4); ^{1}H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.26 (br s, 44H, 22 x CH_2), 1.50 (pentet, 4H, J = 6.4 Hz, 2 OCH_2CH_2), 2.93 (s, 12H, 2 x $N(CH_3)_2$), 3.30 (br AA`part of AA`XX` pattern, 4H, $OCH_2CH_2N$ ), 3.33 (t, 4H, J = 6.5 Hz, tetradecyl $OCH_2$), 3.34 (s, 4H, tetradecyl$OCH_2C$), 3.51 (s, 4H, $N(CH_2)_2OCH_2C$), 3.90 (XX` part of AA`XX`pattern, 4H, OCH_2CH_2N); 12.0 (br s, 2H, NH);^{13}C NMR δ 71.7 (CH_2CH_2OC), 70.6 ($CCH_2O(CH_2)_2N$), 69.4 (CCH_2O tetradecyl), 65.7 (OCH_2CH_2N), 56.7 (NCH_2CH_2), 45.1 (q C), 43.6 ($N(CH_3)_2$), 31.9 ($CH_2CH_2CH_3$), 29.7, 29.7, 29.5, (tetradecyl CH_2), 26.2 ($CH_2CH_2CH_2O$), 22.7 (CH_2CH_3), 14.1 (Me); HR ESI MS *m/z* calcd for $C_{41}H_{87}N_2O_4$ (M+H) 671.6666, found 671.6662.

General procedure for alkylation with methyl iodide: *N,N,N,N′,N′,N′*-hexamethyl-5,5-bis(octyloxymethyl)-3,7-dioxa-1,9-nonanediammonium diiodide (4.9). Salt **4.5** (4.1 g, 7.1 mmol) was dissolved in a NaOH solution (2 M, 30 mL) and the resulting mixture was extracted with diethyl ether (2 x 30 mL). The combined extracts were washed with brine (10mL), dried ($MgSO_4$), and concentrated to give a light yellow syrup, *N,N,N′,N′*-tetramethyl-5,5-bis(octyloxymethyl)-3,7-dioxa-1,9-nonanediamine (**4.1**): yield 3.20 g, 89%; R_F on basic alumina 0.44 (chloroform: ethanol 98:2); ^{1}H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.25-1.33 (br m, 20H, 10 x CH_2), 1.51 (pentet, 4H, J = 6.8 Hz, 2 x OCH_2CH_2), 2.26 (s, 12H, 2 x $N(CH_3)_2$), 2.49 (t, 4H, J = 5.8 Hz, OCH_2CH_2N), 3.35 (t, 4H, J = 6.5 Hz, octyl OCH_2), 3.36 (s, 4H, octylOCH_2C), 3.39 (s, 4H, $NCH_2CH_2OCH_2C$), 3.49 (t, 4H, J = 6.0 Hz , OCH_2CH_2N); ^{13}C NMR δ 71.9 (CH_2CH_2OC), 70.5 ($NCH_2CH_2OCH_2$), 70.3 (OCH_2CH_2N), 69.8 (CCH_2Odecyl), 58.8 (OCH_2CH_2N), 46.1 ($N(CH_3)_2$), 45.6 (q C), 32.0 ($CH_2CH_2CH_3$), 29.70, 29.57, 29.54 (3 octyl CH_2), 29.40 (OCH_2CH_2), 26.3 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.2 (Me); LR ESI MS *m/z* calcd for $C_{29}H_{63}N_2O_4$ (M+H) 503.47, found 503.4; calcd for M+Na 525.46, found 525.5.

Compound **4.1** (7.49 g, 0.0149 mol) was shaken with methyl iodide (8.28 g, 0.0584 mol, 4 eq) for 2 min, then dichloromethane (100 mL) was added and shaking was continued for 5 min. The reaction mixture was concentrated to a yellow solid, that was washed with acetone, then crystallized from toluene : ethanol 10 : 1 to give colorless crystals: yield 10.17 g, 87 %; recrystallized from ethyl acetate: methanol; R_F on basic alumina 0.54 (butanol: water: methanol 20: 5 : 2); mp softens 180 ^oC, melts 188 - 189 ^oC; ^1H NMR δ 0.89 (t, 6H, J = 6.9 Hz, 2 x Me), 1.28 -1.32 (brs, 20H, 10 x CH_2), 1.51 (pentet, 4H, J = 6.3 Hz, 2 OCH_2CH_2), 3.30 (s, 4H, octylOCH_2C), 3.33 (t, 4H, J = 6.6 Hz, octyl OCH_2), 3.51 (s, 4H, $N(CH_2)_2OCH_2C$), 3.54 (s, 18H, 2 x $N(CH_3)_3$), 3.94 (br s, 4H, OCH_2), 4.02 (br m, 4H, CH_2N); ^{13}C NMR δ 71.8 (CH_2CH_2OC), 70.9 ($CCH_2O(CH_2)_2N$), 69.4 (CCH_2Ooctyl), 65.8 (CH_2N), 65.4 (OCH_2CH_2N), 54.9 ($N(CH_3)_3$), 45.2 (q C), 31.9 ($CH_2CH_2CH_3$), 29.66, 29.41, 29.32 (3 octyl CH_2), 29.46 (OCH_2CH_2), 26.2 ($CH_2CH_2CH_2O$), 22.7 (CH_2CH_3), 14.2 (Me); LR ESI MS *m/z* calcd for $C_{31}H_{68}N_2O_4I$ (M-I) 659.45, found 659.1. Anal. Calcd for $C_{31}H_{68}N_2O_4I_2$: C, 47.33; H, 8.71; N, 3.56. Found: C, 47.34; H, 8.51; N 3.29.

5,5-Bis(decyloxymethyl)-*N,N,N,N′,N′,N′*-hexamethyl-3,7-dioxa-1,9-nonanediammonium diiodide (4.10). Salt **4.6** (2.06 g, 3.26 mmol) was dissolved in a NaOH solution (2 M, 20 mL) and the resulting mixture was extracted with dichloromethane (3 x 20 mL). The combined extracts were washed with water (5 mL), dried ($MgSO_4$), and concentrated to give a light yellow syrup of 5,5-bis(decyloxymethyl)-*N,N,N′,N′*-tetramethyl-3,7-dioxa-1,9-nonanediamine (**4.2**): yield 1.6 g, 87 %; R_F on basic alumina 0.46 (chloroform : ethanol 98 : 2); ^1H NMR δ 0.88 (t, 6H, J = 6.8 Hz, 2 x Me), 1.26 (br s, 28H, 14 x CH_2), 1.52 (pentet, 4H, J = 6.6 Hz, 2 OCH_2CH_2), 2.27 (s, 12H, 2 x $N(CH_3)_2$), 2.51 (t, 4H, J = 5.8 Hz, OCH_2CH_2N), 3.35 (t, 4H, J = 6.5 Hz, decyl OCH_2), 3.36 (s, 4H, $CH_2CH_2CH_2OCH_2C$), 3.39 (s, 4H, $NCH_2CH_2OCH_2C$), 3.51 (t, 4H, J = 5.8 Hz, OCH_2CH_2N); ^{13}C NMR δ 71.6 (CH_2CH_2OC), 70.4 ($NCH_2CH_2OCH_2$), 70.1 (OCH_2CH_2N), 69.8 ($CCH_2OCH_2CH_2C$), 58.7 (OCH_2CH_2N), 46.0 ($N(CH_3)_2$), 45.5 (q C), 32.1 ($CH_2CH_2CH_3$), 29.81, 29.79, 29.75, 29.73, 29.65 (5 decyl CH_2), 29.48

(OCH_2CH_2), 26.4 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.2 (Me); LR ESI MS *m/z* calcd for $C_{33}H_{71}N_2O_4$ (M+H) 559.54, found 559.5; *m/z* calcd for M+Na, 581.52, found 581.5.

Compound **4.2** (11.0 g, 0.0197 mol) was shaken with methyl iodide (4.91 mL, 11.2 g, 0.0716 mol, 4 eq) for 2 min, then dichloromethane (250 mL) was added and shaking was continued for 5 min. The reaction mixture was concentrated to a solid that was crystallized from toluene: ethanol 10:1 to give colorless crystals: yield 16 g, 96%, that were recrystallized from ethyl acetate-methanol to give colorless translucent cubes: R_F on basic alumina 0.59 (butanol: water: methanol 20: 5 : 2); mp 100-110 ^oC becomes opaque, 180 – 183 ^oC, clears, 186 ^oC, melts; ^1H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.27 -1.31 (brs, 28H, 14 x CH_2), 1.50 (pentet, 4H, J = 6.3 Hz, 2 OCH_2CH_2), 3.29 (s, 4H, decylOCH_2C) , 3.33 (t, 4H, J = 6.6 Hz, decyl OCH_2), 3.51 (s, 4H, $N(CH_2)_2OCH_2C$), 3.53 (s, 18H, 2 x $N(CH_3)_3$), 3.94 (br s, 4H, OCH_2), 4.02 (br m, 4H, CH_2N); ^{13}C NMR δ 71.8 (CH_2CH_2OC), 70.9 ($CCH_2O(CH_2)_2N$), 69.4 ($CCH_2Odecyl$), 65.8 ($CH_2N(CH_3)_3$), 65.4 (OCH_2CH_2N), 54.9 ($N(CH_3)_3$), 45.1 (q C), 31.9 ($CH_2CH_2CH_3$), 29.65, 29.63, 29.60, 29.41 (5 decyl CH_2), 29.49 (OCH_2CH_2), 26.2 ($CH_2CH_2CH_2O$), 22.7 (CH_2CH_3), 14.1 (Me); LR ESI MS *m/z* calcd for $C_{35}H_{77}N_2O_4I$ (M-I) 715.52, found 715.3. Anal. Calcd for $C_{35}H_{76}N_2O_4I_2$: 49.88, H 9.09, N 3.32. Found: C 49.84, H 9.16, N 3.14.

5,5-Bis(dodecyloxymethyl)-*N,N,N,N′,N′,N′*-hexamethyl-3,7-dioxa-1,9-nonanediammonium diiodide (4.11). Salt **4.7** (5.2 g, 7.5 mmol) was dissolved in a NaOH solution (2 M, 40 mL) and the resulting mixture was extracted with dichloromethane (3 x 40 mL). The combined extracts were washed with water (10 mL), dried ($MgSO_4$), and concentrated to give a colorless semi-solid, 5,5-bis(dodecyloxymethyl)-*N,N,N′,N′*-tetramethyl-3,7-dioxa-1,9-nonanediamine (**4.3**), yield 4.34 g, 94%; R_F on basic alumina 0.51 (chloroform : ethanol 98 : 2); ^1H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.26-1.29 (br s, 36H, 10 x CH_2), 1.52 (pentet, 4H, J = 6.6 Hz, 2 x OCH_2CH_2), 2.27 (s, 12H, 2 x $N(CH_3)_2$) , 2.51 (t, 4H, J = 5.9 Hz, OCH_2CH_2N), 3.35 (t, 4H, J = 6.5 Hz, dodecyl OCH_2), 3.36 (s, 4H, $CH_2CH_2CH_2OCH_2C$), 3.39 (s, 4H, $NCH_2CH_2OCH_2C$), 3.51 (t, 4H, J = 5.9 Hz, OCH_2CH_2N); ^{13}C NMR

δ 71.6 (CH_2CH_2OC), 70.4 ($NCH_2CH_2OCH_2C$), 70.1 (OCH_2CH_2N), 69.8 ($CCH_2OCH_2CH_2C$), 58.8 (OCH_2CH_2N), 45.9 ($N(CH_3)_2$), 45.4 (q C), 32.0 ($CH_2CH_2CH_3$), 29.82, 29.80, 29.79, 29.78, 29.77, 29.65 (6 decylCH_2), 29.48 (OCH_2CH_2), 26.4 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.2 (Me); LR ESI MS *m/z* calcd for $C_{37}H_{79}N_2O_4$ (M+H) 615.60, found 615.5; calcd for M+Na 637.59, found 637.6.

Treatment of compound **4.3** (17.6 g, 0.0286 mol) with methyl iodide (7.14 mL, 16.2 g, 0.114 mol, 4 eq) as for compound **4.9** above gave the title compound (**4.11**) as colorless crystals: yield 21.44 g, 85 %; recrystallized from ethyl acetate - methanol to give colorless needles: R_F on basic alumina 0.61 (butanol: water: methanol 20: 5: 2) ; mp 100-115 ^oC becomes opaque, 155 – 180 ^oC, clears, 183 ^oC, melts; ^1H NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.26 - 1.31 (br s, 36H, 18 x CH_2), 1.50 (pentet, 4H, J = 6.3 Hz, 2 OCH_2CH_2), 3.29 (s, 4H, dodecylOCH_2C), 3.33 (t, 4H, J = 6.6 Hz, dodecyl OCH_2), 3.51 (s, 4H, $N(CH_2)_2OCH_2C$), 3.53 (s, 18H, 2 x $N(CH_3)_3$), 3.94 (br s, 4H, OCH_2), 4.02 (br m, 4H, 2 x $CH_2N(CH_3)_3$); ^{13}C NMR δ 71.8 (CH_2CH_2OC), 70.9 ($N(CH_2)_2OCH_2C$), 69.4 (CCH_2Ododecyl), 65.9 (CH_2N), 65.4 (OCH_2CH_2N), 55.0 ($N(CH_3)_3$), 45.2 (q C), 32.0 ($CH_2CH_2CH_3$), 29.75, 29.75, 29.72, 29.41 (7 dodecyl CH_2), 29.58 (OCH_2CH_2), 26.3 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.2 (Me); LR ESI MS *m/z* calcd for $C_{39}H_{84}N_2O_4I$ (M-I) 771.58, found 771.3. Anal. Calcd. for $C_{39}H_{84}N_2O_4I_2$: C, 52.11; H, 9.42; N, 3.12. Found: C, 52.11; H, 9.24; N, 2.99.

***N,N,N,N′,N′,N′*-Hexamethyl-3,7-dioxa-5,5-bis(tetradecyloxymethyl)-1,9-nonanediammonium diiodide (4.12)** Salt **4.8** (3.59 g, 4.83 mmol) was dissolved in a NaOH solution (2 M, 30 mL) and the resulting mixture was extracted with dichloromethane (3 x 30 mL). The combined extracts were washed with water (10 mL), dried ($MgSO_4$), and concentrated to a colorless semi-solid, *N,N,N′,N′*-tetramethyl-3,7-dioxa-5,5-bis(tetradecyloxymethyl)-1,9-nonanediamine (**4.4**), yield 3.12 g, 97%; R_F on basic alumina 0.53 (chloroform : ethanol 98 : 2); ^1H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.24-1.32 (br, 44H, 22 x CH_2), 1.52 (pentet, 4H, J = 6.8 Hz, 2 OCH_2CH_2), 2.26 (s, 12H, 2 x $N(CH_3)_2$), 2.49 (t, 4H, J = 5.9 Hz, OCH_2CH_2N), 3.35 (t, 4H, J = 6.5 Hz, tetradecyl OCH_2), 3.36 (s, 4H,

$CH_2CH_2CH_2OCOC\mathit{H}_2C$), 3.39 (s, 4H, $NCH_2CH_2OC\mathit{H}_2C$), 3.50 (t, 4H, J = 5.9 Hz, $OC\mathit{H}_2CH_2N$); ^{13}C NMR δ 71.7 ($CH_2\mathit{C}H_2OC$), 70.4 ($NCH_2CH_2O\mathit{C}H_2C$), 70.3 ($O\mathit{C}H_2CH_2N$), 69.8 ($C\mathit{C}H_2OCH_2CH_2C$), 58.9 ($OCH_2\mathit{C}H_2N$), 46.2 (2 x $N(CH_3)_2$), 45.5 (q C), 32.1 ($\mathit{C}H_2CH_2CH_3$), 3 x 29.85, 29.83, 29.82, 2 x 29.80, 29.68 (8 tetradecyl CH_2), 29.51 ($OCH_2\mathit{C}H_2$), 26.4 ($\mathit{C}H_2CH_2CH_2O$), 22.8 ($\mathit{C}H_2CH_3$), 14.3 (Me); LR ESI MS *m/z* calcd for $C_{41}H_{87}N_2O_4$ (M+H) 671.67, found 671.6; calcd for M+Na 693.65, found 693.7.

Treatment of compound **4.8** (3.63 g, 0.00570 mol) with methyl iodide (1.42 mL, 3.236 g, 0.0228 mol, 4 eq) as for compound **4.9** above gave the title compound (**4.12**) as colorless crystals: yield 3.7 g, 69 %; recrystallized from ethyl acetate-methanol as opaque colorless crystals; R_F on basic alumina 0.63 (butanol: water: methanol 20: 5 : 2); mp 160–180 °C, becomes transparent, 185 °C, melts; ^1H NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.26 -1.31 (brs, 44H, 22 x CH_2), 1.50 (pentet, 4H, J = 6.3 Hz, 2 $OCH_2C\mathit{H}_2$), 3.29 (s, 4H, 2 x tetradecylOCH_2C), 3.33 (t, 4H, J = 6.6 Hz, tetradecyl OCH_2), 3.52 (s, 4H, 2 x $N(CH_2)_2OC\mathit{H}_2C$), 3.53 (s, 18H, 2 x $N(CH_3)_3$), 3.93 (br s, 4H, 2 x OCH_2CH_2N), 4.02 (br m, 4H, 2 x CH_2N); ^{13}C NMR δ 71.9 ($CH_2\mathit{C}H_2OC$), 70.9 ($C\mathit{C}H_2O(CH_2)_2N$), 69.4 ($C\mathit{C}H_2O$tetradecyl), 65.8 (CH_2N), 65.5 ($O\mathit{C}H_2CH_2N$), 54.9 ($N(CH_3)_3$), 45.2 (q C), 32.0 ($\mathit{C}H_2CH_2CH_3$), 29.81, 29.75, 29.64, 29.41 (9 tetradecyl CH_2), 29.45 (OCH_2CH_2), 26.3 ($\mathit{C}H_2CH_2CH_2O$), 22.8 ($\mathit{C}H_2CH_3$), 14.2 (Me); LR ESI MS *m/z* calcd for $C_{43}H_{91}N_2O_4I$ (M-I) 827.59, found 827.3. Anal. Calcd for $C_{43}H_{92}N_2O_4I_2$: C, 54.08; H, 9.71; N 2.93. Found: C, 54.16; H, 9.53; N, 2.83.

General procedure for reaction of dialkoxides with 2-bromo-*N*,*N*-diethylethylamine hydrobromide: *N*,*N*,*N'*,*N'*-tetraethyl-5,5-bis(octyloxymethyl)-3,7-dioxa-1,9-nonanediaminium dihydrochloride (4.17). Sodium hydride (6.10 g, 0.15 mol, 10 eq) was added slowly to a stirred solution of 2,2-dioctyloxymethyl-1,3-propanediol (**9**) (5.50 g, 0.015 mol) in THF (500 mL) under N_2 gas at rt. When foaming ceased, the mixture was stirred vigorously at 60 °C for 1 h. The reaction mixture was cooled to rt, then 2-bromo-*N*,*N*-diethylethylamine hydrobromide (15.95 g, 0.061 mol, 4.0

eq) was added. The reaction mixture was stirred at 60 ^{o}C under N_2 gas for 12 h, then quenched with methanol. The reaction mixture was filtered and the filtrate concentrated to a syrupy residue. The residue was taken up in diethyl ether (50mL) and the resulting solution was washed with brine (50 mL), dried ($MgSO_4$) and concentrated at 30-35 ^{o}C to give crude *N,N,N′,N′*-tetraethyl-5,5-bis(octyloxymethyl)-3,7-dioxa-1,9-nonanediamine (**4.13**). The crude product was taken up in dichloromethane (50 mL) and the resulting solution was shaken with ice cold 2 M HCl (30 mL). The aqueous layer was diluted with brine (20 mL) and this solution was extracted with dichloromethane (5 x mL). The combined organic layers were dried ($MgSO_4$) and concentrated to give the title compound (**4.17**) as a colorless solid, that was crystallized from ethyl acetate and acetone to give colorless granules: yield 7.20 g, 75 %; mp 155 ^{o}C; R_F 0.5 (96:4 dichloromethane : ethanol); ^{1}H NMR δ 0.88 (t, 6H, J = 6.8 Hz, 2 x Me), 1.27 (brs, 20H, 10 x CH_2), 1.42 (t, 12H, J = 7.2 Hz, 4 x Me), 1.51 (pentet, 4H, J = 6.3 Hz, 2 OCH_2CH_2), 3.21 (very br AB part of ABX_3 pattern, 8H, NCH_2CH_3), 3.26 (br t, 4H, NCH_2CH_2O), 3.31 (s, 4H, octyl OCH_2C), 3.33 (t, 4H, J = 6.6 Hz, octyl OCH_2), 3.44 (s, 4H, $N(CH_2)_2OCH_2C$), 3.91 (t, 4H, J = 4.3 Hz, OCH_2CH_2N); 12.0 (br s, 2H, NH); ^{13}C NMR δ 71.7 (CH_2CH_2OC), 70.6 ($CCH_2O(CH_2)_2N$), 69.4 (CCH_2O octyl), 65.8 (OCH_2CH_2N), 50.9 (NCH_2CH_2), 47.3 (NCH_2CH_3), 45.1 (q C), 32.0 ($CH_2CH_2CH_3$), 29.6, 29.5, 29.3 (octyl CH_2), 26.2 ($CH_2CH_2CH_2O$), 22.7 (CH_2CH_3), 14.1 (Me), 8.8 (Me); HR ESI MS *m/z* calcd for $C_{33}H_{72}N_2O_4$/2 (M+2H/2) 280.2741, found 280.2741.

5,5-Bis(decyloxymethyl)-*N,N,N′,N′*-tetraethyl-3,7-dioxa-1,9-nonanediaminium dihydrochloride (4.18). A mixture of sodium hydride (0.96 g, 0.024 mol, 10 eq) and compound **10** (1.0 g, 0.0024 mol) in THF (50 mL) under nitrogen gas were reacted with 2-bromo-*N,N*-diethylethylamine hydrobromide (2.5 g, 0.0096 mol, 4.0 eq) for 18 h as above to give crude 5,5-bis(decyloxymethyl)-*N,N,N′,N′*-tetraethyl-3,7-dioxa-1,9-nonanediamine (**4.14**) that was converted to the title hydrochloride (**4.18**) as above. It was a colorless solid, yield 0.92 g, that was crystallized from ethyl acetate and acetone to

give colorless granules: yield 0.84 g, 51%; mp 144 °C; R_F 0.63 (96:4 dichloromethane : ethanol); ^{1}H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.26 (brs, 28H, 14 x CH_2), 1.42 (t, 12H, J = 7.3 Hz, 4 x Me), 1.51 (pentet, 4H, J = 6.4 Hz, 2 OCH_2CH_2), 3.21 (very br AB part of ABX_3 pattern, 8H, NCH_2CH_3), 3.26 (br t, 4H, NCH_2CH_2O), 3.30 (s, 4H, decylOCH_2C), 3.33 (t, 4H, J = 6.6 Hz, decyl OCH_2), 3.44 (s, 4H, $N(CH_2)_2OCH_2C$), 3.92 (t, 4H, J = 4.6 Hz, OCH_2CH_2N); 12.05 (br s, 2H, NH); ^{13}C NMR δ 71.8 (CH_2CH_2OC), 70.7 ($CCH_2O(CH_2)_2N$), 69.5 (CCH_2Odecyl), 65.9 (OCH_2CH_2N), 51.1 (NCH_2CH_2), 47.4 (NCH_2CH_3), 45.2 (q C), 32.0 ($CH_2CH_2CH_3$), 29.73, 29.70, 29.57 (decyl CH_2), 29.40 (OCH_2CH_2), 26.3 ($CH_2CH_2CH_2O$), 22.7 (CH_2CH_3), 14.2 (Me), 8.8 (Me); HR ESI MS *m/z* calcd for $C_{37}H_{79}N_2O_4$ (M+H) 615.6040, found 615.6036.

5,5-Bis(dodecyloxymethyl)-*N,N,N′,N′*-tetraethyl-3,7-dioxa-1,9-nonanediaminium dihydrochloride (4.19) Treatment of compound **11** (9.38 g, 0.0198 mol) in THF (500 mL) with sodium hydride (7.90 g, 0.198 mol, 10 eq) and 2-bromo-*N,N*-diethylethylamine hydrobromide (20.7 g, 0.079 mol, 4 eq) as above gave a crude product, 5,5-bis(dodecyloxymethyl)-*N,N,N′,N′*-tetraethyl-3,7-dioxa-1,9-nonanediamine (**4.15**) that was converted to the title hydrochloride (**4.19**), a colorless solid that was crystallized from ethyl acetate and acetone to give colorless granules: yield 10.4 g, 71%; mp 150 °C; R_F 0.65 (96:4 dichloromethane : ethanol); ^{1}H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.26 (brs, 36H, 18 x CH_2), 1.42 (t, 12H, J = 7.3 Hz, 4 x Me), 1.51 (pentet, 4H, J = 6.4 Hz, 2 OCH_2CH_2), 3.21 (very br AB part of ABX_3 pattern, 8H, NCH_2CH_3), 3.26 (br t, 4H, J = 4.4 Hz, NCH_2CH_2), 3.30 (s, 4H, dodecylOCH_2C), 3.33 (t, 4H, J = 6.6 Hz, dodecyl OCH_2), 3.44 (s, 4H, $N(CH_2)_2OCH_2C$), 3.92 (t, 4H, J = 4.6 Hz, OCH_2CH_2); 12.05 (br s, 2H, NH); ^{13}C NMR δ 71.9 (CH_2CH_2OC), 70.8 ($CCH_2O(CH_2)_2N$), 69.5 (CCH_2Ododecyl), 66.9 (OCH_2CH_2N), 51.1 (NCH_2CH_2), 47.4 (NCH_2CH_3), 45.2 (q C), 32.0 ($CH_2CH_2CH_3$), 29.79, 29.63, 29.46 (dodecyl CH_2), 26.3 ($CH_2CH_2CH_2O$), 22.9 (CH_2CH_3), 14.2 (Me), 8.8 (Me); HR ESI MS *m/z* calcd for $C_{41}H_{87}N_2O_4$ (M+H) 671.6666, found 671.6669.

***N,N,N′,N′*-Tetraethyl-3,7-dioxa-5,5-bis(tetradecyloxymethyl)-1,9-nonanediaminium dihydrochloride (4.20)** Treatment of compound **12** (10.0 g, 0.0189 mol) in THF (500 mL) with sodium hydride (7.57 g, 0.189 mol, 10 eq) and 2-bromo-*N,N*-diethylethylamine hydrobromide (19.7 g, 0.076 mol, 4 eq) as above to give a crude light yellow syrup, *N,N,N′,N′*-tetraethyl-5,5-bis(tetradecyloxymethyl)-3,7-dioxa-1,9-nonanediamine (**4.16**). The syrup was taken up in dichloromethane (50 mL) and the solution was shaken with ice cold 2 M HCl (50 mL) as for **4.17** to give a colorless solid that was crystallized from ethyl acetate and acetone to give colorless granules: yield 11.5 g, 76%; mp 146 ^{o}C; R_F 0.69 (96:4 dichloromethane : ethanol); ^{1}H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.26 (brs, 44H, 22 x CH_2), 1.41 (t, 12H, J = 7.2 Hz, 4 x Me), 1.51 (pentet, 4H, J = 6.4 Hz, 2 OCH_2CH_2), 3.20 (very br AB part of ABX_3 pattern, 8H, NCH_2CH_3), 3.24 (br t, NCH_2CH_2), 3.30 (s, 4H, tetradecylOCH_2C), 3.33 (t, 4H, J = 6.6 Hz, tetradecyl OCH_2), 3.44 (s, 4H, $N(CH_2)_2OCH_2C$), 3.91 (t, 4H, J = 4.5 Hz, OCH_2CH_2N), 12.05 (brs, 2H, NH); ^{13}C NMR δ 71.8 (CH_2CH_2OC), 70.8 ($CCH_2O(CH_2)_2N$), 69.5 (CCH_2Otetradecyl), 66.1 (OCH_2CH_2N), 51.2 (NCH_2CH_2), 47.2 (NCH_2CH_3), 45.2 (q C), 32.0 ($CH_2CH_2CH_3$), 29.72, 29.77, 29.65, 29.47 (tetradecyl CH_2), 26.4 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.2 (Me), 8.9 (Me); HR ESI MS *m/z* calcd for $C_{45}H_{95}N_2O_4$ (M+H) 727.7292, found 727.7293.

General procedure for reaction of amines with ethyl bromide: *N,N,N,N′,N′,N′*-Hexaethyl-5,5-bis(octyloxymethyl)-3,7-dioxa-1,9-nonanediammonium dibromide (4.21). Salt **4.17** (1.44g, 2.28 mmol) was dissolved in a NaOH solution (2 M, 15 mL). The resulting mixture was extracted with diethyl ether (3 x 5 mL) to yield a colorless syrup of *N,N,N′,N′*-tetraethyl-5,5-bis(octyloxymethyl)-3,7-dioxa-1,9-nonanediamine (**4.13**), yield 1.08 g, 85 %; R_F 0.43 on basic alumina (dichloromethane : ethanol, 98:2); ^{1}H NMR δ 0.88 (t, 6H, J = 6.8 Hz, 2 x Me), 1.03 (t, J = 7.2 Hz, 12H, 4 x Me) 1.28 (br s, 20H, 10 x CH_2), 1.51 (pentet , 4H, J = 6.8 Hz, 2 OCH_2CH_2), 2.57 (q, 8H, J = 7.1 Hz, 2 x $N(CH_2CH_3)_2$), 2.65 (t , 4H, J = 6.2 Hz, $2NCH_2CH_2$) , 3.35 (t, J = 6.5 Hz, 4H, octyl OCH_2), 3.35 (s, 4H,

decylOCH_2C), 3.38 (s, 4H, CCH_2O(CH_2)$_2$N), 3.47 (t, J = 6.3 Hz, 4H, 2 OCH_2CH_2); ^{13}C NMR δ 71.6 (CH_2CH$_2$OC), 70.4 (CCH$_2$O(CH_2)$_2$N), 70.4 (CH_2CH_2N), 69.8 (CCH$_2$O octyl), 52.1 (NCH_2CH_2), 47.8 (NCH_2CH$_3$), 45.4 (q C), 32.0 (CH$_2$CH_2CH$_3$), 29.8, 29.6, 29.5, 29.3 (octyl CH$_2$), 26.3 (CH$_2$CH_2CH$_2$O), 22.8 (CH$_2$CH$_3$), 14.2 (Me), 12.1 (CH$_2$CH$_3$).

Ethyl bromide (98%, 2.78 mL, 37.3 mmol, 20.0 eq) was added to a stirred solution of compound **4.13** (1.04 g, 1.86 mmol) in a mixture of THF and ethanol (6 mL) (2:1). Potassium carbonate (0.5 g, 3.7 mmol, 2 eq) was added and the resulting mixture was refluxed for 26 h, then cooled to rt, filtered, and the filtrate was concentrated to give the title compound (**4.21**) as a colorless sticky solid (1.92 g). The salt precipitated from ethyl acetate containing a few drops of methanol, yield 1.13 g, 78 %; mp 157 ^{o}C; R_F 0.53 on basic alumina (butanol: water: methanol 20: 5: 2); ^{1}H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.28 (br s, 20H, 10 x CH$_2$), 1.41 (t, J = 7.2 Hz, 12H, 6 x Me), 1.51 (pentet , 4H, J = 6.5 Hz, 2 OCH$_2$CH_2), 3.28 (s, 4H, octylOCH$_2$C), 3.33 (t, J = 6.6 Hz, 4H, octyl OCH$_2$), 3.47 (s, 4H, CCH_2O(CH_2)$_2$N), 3.4 7 (q, 12H, J = 7.2 Hz, 2 N (CH_2CH$_3$)$_2$), 3.79 (AA` part of AA`BB` pattern, 4H, 2 NC$H_2$C$H_2$), 3.94 (BB` part of AA`BB` pattern, 4H, 2OCH$_2$CH_2); ^{13}C NMR δ 71.9 (CH_2CH$_2$OC), 71.1(CCH$_2$O(CH_2)$_2$N), 69.5 (CCH$_2$O octyl), 65.1 (OCH$_2$CH_2N), 57.8 (NCH$_2$CH_2), 54.4 (NCH$_2$CH$_3$), 45.3 (q C), 31.9 (CH$_2$CH_2CH$_3$), 29.7, 29.6 , 29.4, (octyl CH$_2$), 26.3 (CH$_2$CH_2CH$_2$O), 22.7 (CH$_2$CH$_3$), 14.2 (Me), 8.4 (CH$_2$CH$_3$); HR ESI MS *m/z* calcd for $C_{37}H_{80}N_2O_4$/2 (M-2Br)/2 308.3054, found 308.3038.

5,5-Bis(decyloxymethyl)-*N,N,N,N′,N′,N′*-hexaethyl-3,7-dioxa-1,9-nonanediammonium dibromide (4.22). Salt **4.18** (0.87 g, 1.26 mmol) was dissolved in a NaOH solution (2 M, 10 mL). The resulting mixture was extracted with dichloromethane (3 x 5 mL) to yield a colorless syrup of 5,5-bis(decyloxymethyl)-*N,N,N′,N′*-tetraethyl-3,7-dioxa-1,9-nonanediamine (**4.14**), yield 0.69 g, 89%; R_F 0.44 on basic alumina (dichloromethane : ethanol, 98:2); ^{1}H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.03 (t, J = 7.2 Hz, 12H, 4 x Me) 1.26 (br s, 28H, 14 x CH$_2$), 1.52 (pentet , 4H, J = 6.7 Hz, 2

OCH_2CH_2), 2.57 (q, 8H, J = 7.14 Hz, 2 x $N(CH_2CH_3)_2$), 2.65 (t , 4H, J = 6.2 Hz, $2NCH_2CH_2$) , 3.35 (t, J = 6.5 Hz, 4H, decyl OCH_2), 3.35 (s, 4H, decylOCH_2C), 3.38 (s, 4H, $CCH_2O(CH_2)_2N$), 3.47 (t, J = 6.2 Hz, 4H, 2 OCH_2CH_2); ^{13}C NMR δ 71.7 (CH_2CH_2OC), 70.4 ($CCH_2O(CH_2)_2N$), 70.4 (CH_2CH_2N), 69.8 ($CCH_2Odecyl$), 52.2 (NCH_2CH_2), 47.6 (NCH_2CH_3), 45.3 (q C), 32.0 ($CH_2CH_2CH_3$), 29.7, 29.6, 29.5, 29.3 (decyl CH_2), 26.2 ($CH_2CH_2CH_2O$), 22.6 (CH_2CH_3), 14.0 (Me), 11.90 (CH_2CH_3).

Ethyl bromide (98%, 1.71 mL, 22.5 mmol, 20.0 eq) was added to a stirred solution of compound **4.14** (0.69 g, 1.12 mmol) in a mixture of THF and ethanol (6 mL) (2:1). Potassium carbonate (0.13 g, 2.24 mmol, 2.0 eq) was added and the resulting mixture was refluxed for 26 h, then cooled to rt, filtered, and the filtrate was concentrated to give the title compound (**4.22**) as a colorless crystalline solid, yield 0.78 g, 86%; R_F on basic alumina 0.43 (butanol: water: methanol 20: 5: 2); ^1H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.27 (br s, 28H, 14 x CH_2), 1.40 (t, J = 7.2 Hz, 12H, 6 x Me), 1.51 (pentet , 4H, J = 6.3 Hz, 2 OCH_2CH_2), 3.28 (s, 4H, decylOCH_2C), 3.33 (t, J = 6.6 Hz, 4H, decyl OCH_2), 3.46 (s, 4H, $CCH_2O(CH_2)_2N$), 3.57 (q, 12H, J = 7.2 Hz, 2 N $(CH_2CH_3)_2$), 3.78 (AA` part of AA`BB` pattern, 4H, 2 $NCH_2CH_2$), 3.90 (BB` part of AA`BB` pattern, 4H, $2OCH_2CH_2$); ^{13}C NMR δ 71.7 (CH_2CH_2OC), 70.9 ($CCH_2O(CH_2)_2N$), 69.3 ($CCH_2Odecyl$), 64.8 (OCH_2CH_2N), 57.5 (NCH_2CH_2), 54.2 (NCH_2CH_3), 45.0 (q C), 31.8 ($CH_2CH_2CH_3$), 29.5, 29.5 , 29.5, 29.4, 29.2 (decyl CH_2), 26.1 ($CH_2CH_2CH_2O$), 22.5 (CH_2CH_3), 14.0 (Me), 8.2 (CH_2CH_3); HR ESI MS *m/z* calcd for $C_{41}H_{88}BrN_2O_4$ (M-Br) 751.5927, found 751.5925.

5,5-Bis(dodecyloxymethyl)-*N,N,N,N′,N′,N′*-hexaethyl-3,7-dioxa-1,9-nonanediammonium dibromide (4.23). Salt **4.19** (5.5 g, 7.4 mmol) was dissolved in a NaOH solution (2 M, 30 mL). The resulting mixture was extracted with diethyl ether (2 x 25 mL) to yield a colorless syrup of 5,5-bis(dodecyloxymethyl)-*N,N,N′,N′*-tetraethyl-3,7-dioxa-1,9-nonanediamine (**4.15**), yield 4.66 g, 94%; R_F 0.46 on basic alumina (dichloromethane : ethanol, 98:2), ^1H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.03 (t, J = 7.1 Hz, 12H, 4 x Me) 1.27 (brs, 36H, 18 x CH_2), 1.52 (pentet, 4H, J = 6.6 Hz, 2 OCH_2CH_2),

2.58 (q, 8H, J = 7.1 Hz, 2 x N(CH_2CH_3)$_2$), 2.67 (t, 4H, J = 6.1 Hz, 2 NCH_2CH_2) , 3.35 (t, J = 6.6 Hz, 4H, dodecyl OCH_2), 3.35 (s, 4H, dodecylOCH_2C), 3.39 (s, 4H, CCH_2O(CH_2)$_2$N), 3.48 (t, J = 6.1 Hz, 4H, 2 OCH_2CH_2N); ^{13}C NMR δ 71.3 (CH_2CH_2OC), 70.0 (CCH_2O(CH_2)$_2$N), 70.0 (OCH_2CH_2N), 69.5 (CCH_2Ododecyl), 51.9 (NCH_2CH_2), 47.5 (NCH_2CH_3), 45.2 (q C), 31.9 ($CH_2CH_2CH_3$), 29.5, 29.4, 29.2 (dodecyl CH_2), 26.1 ($CH_2CH_2CH_2$O), 22.5 (CH_2CH_3), 13.9 (Me), 11.8 (CH_2CH_3).

Ethyl bromide (21.0 mL, 276 mmol, 40.0 eq) was added to a stirred solution of compound **4.15** (4.66 g, 6.96 mmol) in a mixture of THF and ethanol (3:2) (50 mL). Potassium carbonate (2.37 g, 17.2 mmol, 2.5 eq) was added and the resulting mixture was refluxed for 33 h, cooled to rt, filtered, and the filtrate was concentrated to give the title compound (**4.23**) as a colorless solid. Crystallization from ethyl acetate and acetone gave colorless granules: yield 4.79 g, 78%; mp 185 ^{o}C; R_F on basic alumina 0.46 (butanol: water: methanol 20: 5 : 2); ^{1}H NMR δ 0.88 (t, 6H, J = 6.8 Hz, 2 x Me), 1.27 (brs, 36H, 18 x CH_2), 1.41 (t, J = 7.2 Hz, 12H, 6 x Me), 1.51 (pentet, 4H, J = 6.3 Hz, 2 OCH_2CH_2), 3.27 (s, 4H, dodecylOCH_2C), 3.32 (t, J = 6.6 Hz, 4H, dodecyl OCH_2), 3.48 (s, 4H, CCH_2O(CH_2)$_2$N), 3.58 (q, 12H, J = 7.2 Hz, 2 N (CH_2CH_3)$_2$), 3.81 (AA` part of AA`BB` pattern, 4H, 2 NC$H_2CH_2$O), 3.95 (BB` part of AA`BB` pattern, 4H, 2 OCH_2CH_2N); ^{13}C NMR δ 71.8 (CH_2CH_2OC), 71.0 (CCH_2O(CH_2)$_2$N), 69.5 (CCH_2Ododecyl), 64.9 (OCH_2CH_2N), 57.6 (NCH_2CH_2O), 54.3 (NCH_2CH_3), 45.2 (q C), 31.9 ($CH_2CH_2CH_3$), 29.7, 29.7, 29.6, 29.4 (dodecyl CH_2), 26.3 ($CH_2CH_2CH_2$O), 22.7 (CH_2CH_3), 14.1 (Me), 8.3 (CH_2CH_3); HR ESI MS *m/z* calcd for $C_{45}H_{96}BrN_2O_4$ (M-Br) 807.6553, found 807.6549.

***N,N,N,N′,N′,N′*-Hexaethyl-3,7-dioxa-5,5-bis(tetradecyloxymethyl)-1,9-nonanediammonium dibromide (4.24).** Salt **4.20** (5.42g, 6.79 mmol) was dissolved in a 2 M NaOH solution (30 mL). The resulting mixture was extracted with diethyl ether (2 x 25 mL) to yield a colorless syrup of *N,N,N′,N′*-tetraethyl-5,5-bis(tetradecyloxymethyl)-3,7-dioxa-1,9-nonanediamine (**4.16**): yield 4.11 g, 84%; R_F 0.48 on basic alumina (dichloromethane : ethanol 98:2), ^{1}H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.02 (t, 12H, J = 7.1 Hz, 4 x Me), 1.28 (brs, 44H, 22 x CH_2), 1.52 (pentet, 4H, J = 6.8 Hz, 2 OCH_2CH_2),

2.57 (q, 8H, J = 7.1 Hz, 2 N(C*H*$_2$CH$_3$)$_2$), 2.65 (t , 4H, J = 6.2 Hz, 2 NCH$_2$CH$_2$), 3.35 (t, J = 6.5 Hz, 4H, tetradecyl OCH$_2$), 3.35 (s, 4H, tetradecylOCH$_2$C), 3.39 (s, 4H, CC*H*$_2$O(CH$_2$)$_2$N), 3.48 (t, J = 6.3 Hz, 4H, 2 OCH$_2$CH$_2$); ^{13}C NMR δ 71.5 (CH$_2$*C*H$_2$OC), 70.4 (C*C*H$_2$O(CH$_2$)$_2$N), 70.3 (O*C*H$_2$CH$_2$N), 69.7 (C*C*H$_2$Otetradecyl), 52.1 (N*C*H$_2$CH$_2$), 47.8 (N*C*H$_2$CH$_3$), 45.4 (q C), 32.0 (*C*H$_2$CH$_2$CH$_3$), 29.8, 29.7, 29.6, 29.4 (tetradecyl CH$_2$), 26.3 (*C*H$_2$CH$_2$CH$_2$O), 22.8 (*C*H$_2$CH$_3$), 14.1 (Me), 12.1(CH$_2$*C*H$_3$).

Treatment of a mixture of ethyl bromide (16.9 mL, 224 mmol, 40.0 eq) and compound **4.16** (4.11 g, 5.66 mmol) in a THF ethanol solution (3:2) (50 mL) containing potassium carbonate (1.95 g, 14.2 mmol, 2.5 eq) as above gave the title compound (**4.24**), as colorless solid. It was recrystallized from ethyl acetate and acetone to give colorless granules: yield 4.50 g, 85%; mp 180 ^{o}C; R$_F$ on basic alumina 0.49 (butanol: water: methanol 20: 5 : 2); ^{1}H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.26 (brs, 44H, 22 x CH$_2$), 1.41 (t, J = 7.2 Hz, 12H, 6 x Me), 1.50 (pentet, 4H, J = 6.3 Hz, 2 x OCH$_2$C*H*$_2$), 3.27 (s, 4H, 2 x tetradecylOCH$_2$C), 3.32 (t, J = 6.6 Hz, 4H, decyl OCH$_2$), 3.48 (s, 4H, CC*H*$_2$O(CH$_2$)$_2$N), 3.58 (q, 12H, J = 7.2 Hz, 2 N(C*H*$_2$CH$_3$)$_2$), 3.81 (AA` part of an AA`BB` pattern, 4H, 2 NCH$_2$CH$_2$), 3.95 (BB`part of AA`BB`pattern, 4H, 2 OCH$_2$CH$_2$); ^{13}C NMR δ 71.7 (CH$_2$*C*H$_2$OC), 70.9 (C*C*H$_2$O(CH$_2$)$_2$N), 69.4 (CCH$_2$Otetradecyl), 64.9 (O*C*H$_2$CH$_2$N), 57.6 (N*C*H$_2$CH$_2$), 54.3 (N*C*H$_2$CH$_3$), 45.0 (q C), 31.8 (*C*H$_2$CH$_2$CH$_3$), 29.6, 29.6, 29.5, 29.3 (tetradecyl CH$_2$), 26.2 (*C*H$_2$CH$_2$CH$_2$O), 22.6 (*C*H$_2$CH$_3$), 14.0 (Me), 8.3 (CH$_2$*C*H$_3$); HR ESI MS *m/z* calcd for C$_{49}$H$_{104}$BrN$_2$O$_4$ (M-Br) 863.7179, found 863.7178.

5,5-Bis(dodecyloxymethyl)-*N*,*N*′-diethyl-*N*,*N*,*N*′,*N*′-dimethyl-3,7-dioxa-1,9-nonanediammonium dibromide (4.25). Ethyl bromide (2.3 mL, 31 mmol, 10 eq), then sodium bicarbonate (1.30 g, 15.5 mmol, 5.0 eq) were added to a stirred solution of compound **4.3** (2.13 g, 3.10 mmol) in THF (30 mL) and the resulting mixture was refluxed for 12 h, cooled to rt, filtered, and the filtrate was concentrated to give the title compound as a colorless solid. Crystallization from ethyl acetate and acetone gave colorless granules: yield 2.37 g, 91.1%; mp 185 ^{o}C; R$_F$ on basic alumina 0.45 (butanol, water,

methanol 20: 5: 2); ^{1}H NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.27-1.31 (br s, 36H, 18 x CH_2), 1.45 (t, J = 7.2 Hz, 6H, 2 x NCH_2CH_3) 1.51 (pentet, 4H, J = 6.2 Hz, 2 OCH_2CH_2), 3.28 (s, 4H, dodecyl OCH_2C), 3.32 (t, J = 6.6 Hz, 4H, dodecyl OCH_2), 3.43 (s, 12H, 2 x $N(CH_3)_2$), 3.47 (s, 4H, $CCH_2O(CH_2)_2N$), 3.81 (q, 4H, J = 7.3 Hz, 2 x $N(CH_2CH_3)_2$), 3.92 (AA` part of AA`BB` pattern, 4H, 2 $OCH_2CH_2N$), 3.97 (BB` part of AA`BB` pattern, 4H, 2 NCH_2CH_2O); ^{13}C NMR δ 71.8 (CH_2CH_2OC), 70.9 ($CCH_2O(CH_2)_2N$), 69.4 ($CCH_2Ododecyl$), 65.3 (OCH_2CH_2N), 63.1 (NCH_2CH_2O), 61.0 (NCH_2CH_3) 51.3 $(NCH_3)_2$, 45.1 (q C), 31.9 ($CH_2CH_2CH_3$), 29.7, 29.7, 29.5, 29.4 (dodecyl CH_2), 26.3 ($CH_2CH_2CH_2O$), 22.7 (CH_2CH_3), 14.2 (Me), 8.9 (NCH_2CH_3); HR ESI MS *m/z* calcd for $C_{41}H_{88}BrN_2O_4$ (M-Br) 751.5927, found 751.5922 .

5,5-Bis(dodecyloxymethyl)-*N,N,N′,N′*-dimethyl-3,7-dioxa-*N,N′*-dipropyl-1,9-nonanediammonium dibromide (4.26). 1-Bromopropane (5.4 mL, 59 mmol, 10 eq), then sodium bicarbonate (2.47 g, 29.4 mmol, 5.0 eq) were added to a stirred solution of compound **4.3** (4.05 g, 5.89 mmol) in THF (50 mL) and the resulting mixture was refluxed for 26 h, cooled to rt, filtered, and the filtrate was concentrated to give the title compound as a colorless solid. Crystallization from ethyl acetate and acetone gave colorless crystals: yield 4.83 g, 95.5%; mp 62 ^{o}C; R_F on basic alumina 0.50 (butanol, water, methanol 20: 5: 2); ^{1}H NMR δ 0.88 ppm (t, 6H, J = 6.7 Hz, 2 x Me), 1.05 (t, J = 7.3 Hz, 6H, 2 x N $(CH_2)_2CH_3$) 1.26-1.31 (br s, 36H, 18 x CH_2), 1.51 (pentet, 4H, J = 6.2 Hz, 2 OCH_2CH_2), 1.86 (AA` part of AA`XX` pattern, 4H, 2 x $NCH_2CH_2CH_3$), 3.28 (s, 4H, dodecyl$OCH_2C$ ), 3.32 (t, J = 6.6 Hz, 4H, dodecyl $OCH_2$), 3.44 (s, 12H, 2x$N(CH_3)_2$), 3.48 (s, 4H, $CCH_2O(CH_2)_2N$), 3.63 (XX `part of AA`XX` pattern 4H, 2 x $NCH_2CH_2CH_3$), 3.93 (AA` part of AA`BB` pattern, 4H, 2, $OCH_2CH_2N$,) 3.99 (BB` part of AA`BB` pattern, 4H, 2,NCH_2CH_2O); ^{13}C NMR δ 71.8 (CH_2CH_2OC), 70.9 ($CCH_2O(CH_2)_2N$), 69.5 ($CCH_2Ododecyl$), 67.1 ($NCH_2CH_2CH_3$), 65.4 (OCH_2CH_2N), 63.6 (NCH_2CH_2O), 51.9 $(NCH_3)_2$, 45.2 (q C), 31.9 ($CH_2CH_2CH_3$), 29.7, 29.7, 29.5, 29.4 (dodecyl CH_2), 26.2 ($CH_2CH_2CH_2O$), 22.7 (CH_2CH_3),

16.5 ($NCH_2CH_2CH_3$), 14.2 (Me), 10.8 ($N(CH_2)_2CH_3$); HR ESI MS *m/z* calcd for $C_{43}H_{92}BrN_2O_4$ (M-Br) 779.6234, found 779.6209.

***N,N′*-Dibutyl-5,5-bis(dodecyloxymethyl)-*N,N,N′,N′*-dimethyl-3,7-dioxa-1,9-nonanediammonium dibromide (4.27).** 1-Bromobutane (4.70 mL, 44.0 mmol, 10 eq), then sodium bicarbonate (1.84 g, 22.0 mmol, 5.0 eq), were added to a stirred solution of compound **4.3** (3.03 g, 4.4 mmol) in THF (40 mL) and the resulting mixture was refluxed for 33 h, cooled to rt, filtered, and the filtrate was concentrated to give the title compound as a colorless solid. Crystallization from ethyl acetate and methanol gave colorless crystals: yield 3.46 g, 88.7 %; mp 110 ^oC; R_F on basic alumina 0.53 (butanol, water, methanol 20: 5: 2); ^1H NMR δ 0.88 ppm (t, 6H, J = 6.9 Hz, 2 x Me), 1.01 (t, J = 7.3 Hz, 6H, 2 x N $(CH_2)_3CH_3$) 1.26-1.31 (br s, 36H, 18 x CH_2), 1.48 (sextet, 4H, J = 7.4 Hz, 2 x $NCH_2CH_2CH_2CH_3$), 1.51 (pentet, 4H, J = 6.2 Hz, 2 OCH_2CH_2), 1.78 (AA` part of AA`XX` pattern, 4H, 2 x $NCH_2CH_2CH_2CH_3$), 3.28 (s, 4H, dodecyl$OCH_2C$), 3.32 (t, J = 6.6 Hz, 4H, dodecyl $OCH_2$), 3.35 (s, 4H, $N(CH_2)_2OCH_2C$), 3.44 (s, 12H, $2xN(CH_3)_2$) 3.48 (s, 4H, $CCH_2O(CH_2)_2N$), 3.65 (XX `part of AA`XX` pattern, 4H, 2 x $NCH_2CH_2CH_2CH_3$), 3.94 (AA` part of AA`BB` pattern, 4H, 2 $OCH_2CH_2N$), 3.99 (BB` part of AA`BB` pattern, 4H, 2 NCH_2CH_2O); ^{13}C NMR δ 71.9 (CH_2CH_2OC), 71.0 ($CCH_2O(CH_2)_2N$), 69.5 (CCH_2Ododecyl), 65.6 ($NCH_2CH_2CH_2CH_3$), 65.4 (OCH_2CH_2N), 63.7 (NCH_2CH_2O), 51.8 $(NCH_3)_2$, 45.2 (q C), 32.0 ($CH_2CH_2CH_3$), 29.8, 29.7, 29.6, 29.4 (dodecyl CH_2), 26.3 ($CH_2CH_2CH_2O$), 24.9 ($NCH_2CH_2CH_2CH_3$), 22.7 (CH_2CH_3), 14.2 (Me), 13.9 (N $(CH_2)_3CH_3$); HR ESI MS *m/z* calcd for $C_{45}H_{96}BrN_2O_4$ (M-Br) 807.6553, found 807.6548.

General procedure for reaction with 3-chloro-*N*,*N*-dimethyl-1-propanamine hydrochloride: *N*,*N*,*N*',*N*'-Tetramethyl-6,6-bis(octyloxymethyl)-4,8-dioxa-1,11-undecanediamine (5.1). Sodium hydride (1.33 g, 55.4 mmol) was added slowly to a stirred solution of 2,2-dioctyloxymethyl-1,3-propanediol (**9**) (2.0 g, 5.5 mmol) in DMF (100 mL) under an N_2 atmosphere at 50 ^oC. The mixture was then stirred at 80 ^oC for 1 h. After the reaction mixture had been allowed to cool to rt, 3-chloro-*N*,*N*-dimethyl-1-propanamine hydrochloride (1.93 g, 12.2 mmol) was added in portions and the reaction mixture was stirred at 80 ^oC under nitrogen for another 24 h, then quenched with methanol and filtered. The filtrate was concentrated, and the residue was taken up in ethyl acetate (50 mL). This solution was washed with water (2 x 20 mL) and brine (20 mL), then dried (Na_2SO_4) and concentrated to a residue that was purified by flash column chromatography. Elution using a gradient of 10 to 15% methanol in dichloromethane gave compound **5.1** as a light brown liquid: yield 1.6 g (54%); R_F on basic alumina 0.6 (dichloromethane: methanol 93:7); ^1H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 6.5 Hz, 2 CH_3), 1.26-1.35 (m, 20 H, 10 x CH_2), 1.52 (p, 4H, J = 7.0 Hz, 2 OCH_2CH_2), 1.71 (p, 4H, J = 6.5 Hz, 2 NCH_2CH_2), 2.22 (s, 12H, 2 $N(CH_3)_2$), 2.32 (t, 4H, J = 7.5 Hz, 2 NCH_2), 3.34 (t, 4H, J = 6.5 Hz, 2 CH_2CH_2O), 3.35 (s, 4H, 2 OCH_2), 3.37 (s, 4H, 2 OCH_2), 3.41 (t, 4H, J = 6.5 Hz, OCH_2); ^{13}C NMR ($CDCl_3$) δ 71.7 (octyl CH_2O), 70.0 ($OCH_2CH_2CH_2N$), 69.85 (OCH_2C), 69.78 (OCH_2C), 57.0 (NCH_2), 45.64 ($N(CH_3)_2$), 45.59 (qC), 32.0 ($CH_3CH_2CH_2$), 29.82, 29.63, 29.49 (3 octyl CH_2), 28.2 (NCH_2CH_2), 26.4 ($OCH_2CH_2CH_2$), 22.9 (CH_3CH_2), 14.2 (CH_3); HR ESI MS *m/z* calcd for $C_{31}H_{67}N_2O_4$ (M+H) 531.5095, found 531.5087.

6,6-Bis(decyloxymethyl)-*N*,*N*,*N*',*N*'-tetramethyl-4,8-dioxa-1,11-undecanediamine (5.2). Sodium hydride (0.57 g, 24.0 mmol), 2,2-didecyloxymethyl-1,3-propanediol (**10**) (1.0 g, 2.4 mmol) in DMF (100 mL) and 3-chloro-*N*,*N*-dimethyl-1-propanamine hydrochloride (1.8 g, 12 mmol) were reacted as above to give compound **5.2** as a light brown liquid: yield 0.7 g (50%); R_F on basic alumina 0.44 (dichloromethane: methanol 95:5); ^1H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 6.5 Hz, CH_3), 1.28-1.32 (m, 28

H, 14 x CH_2), 1.52 (p, 4H, J = 7.0 Hz, OCH_2CH_2), 1.70 (p, 4H, J = 6.5 Hz, NCH_2CH_2), 2.22 (s, 12H, 2 $N(CH_3)_2$), 2.31 (t, 4H, J = 7.5 Hz, NCH_2), 3.34 (t, 4H, J = 6.5 Hz, CH_2O), 3.35 (s, 4H, OCH_2), 3.37 (s, 4H, OCH_2), 3.40 (t, 4H, J = 6.5 Hz, OCH_2); ^{13}C NMR ($CDCl_3$) δ 71.7 (decyl CH_2O), 70.0 ($OCH_2CH_2CH_2N$), 69.9 (OCH_2C), 69.8 (OCH_2C), 57.0 (NCH_2), 45.7 ($N(CH_3)_2$), 45.6 (qC), 32.1 ($CH_3CH_2CH_2$), 29.85 (x2), 29.79, 29.70, 29.52 (5 decyl CH_2), 28.20 (NCH_2CH_2), 26.4 ($OCH_2CH_2CH_2$), 22.9 (CH_3CH_2), 14.3 (CH_3); ESI MS *m/z* calcd for $C_{35}H_{75}N_2O_4$ (M+H) 587.5712, found 587.5742.

6,6-Bis(dodecyloxymethyl)-*N*,*N*,*N*',*N*'-tetramethyl-4,8-dioxa-1,11-undecanediamine (5.3). Sodium hydride (1.0 g, 42 mmol), 2,2-didodecyloxymethyl-1,3-propanediol (**11**) (2.0 g, 4.2 mmol), and 3-chloro-*N*,*N*-dimethyl-1-propanamine hydrochloride (2.60 g, 16.9 mmol) was reacted together as above. The reaction mixture was stirred at 90 ^oC under N_2 for another 36 h, then quenched with methanol, and filtered and worked up as above to give **5.3** as a light brown liquid: yield 1.4 g (51%); R_F on basic alumina 0.5 (dichloromethane: methanol 97:5); ^1H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 6.5 Hz, CH_3), 1.24-1.38 (m, 36 H, 18 x CH_2), 1.52 (pentet, 4H, J = 7.0 Hz, OCH_2CH_2), 1.72 (pentet, 4H, J = 6.5 Hz, NCH_2CH_2), 2.22 (s, 12H, $N(CH_3)_2$), 2.32 (t, 4H, J = 7.5, NCH_2), 3.35 (t, 4H, J = 6.5 Hz, CH_2O), 3.36 (s, 4H, OCH_2), 3.37 (s, 4H, OCH_2), 3.41 (t, 4H, J = 6.5 Hz, OCH_2); ^{13}C NMR ($CDCl_3$) δ 71.6 (dodecyl CH_2O), 69.9 ($OCH_2CH_2CH_2N$), 69.72 (OCH_2C), 69.67 (OCH_2C), 56.9 (NCH_2), 45.5 ($N(CH_3)_2$), 45.5 (qC), 31.9 ($CH_3CH_2CH_2$), 29.75 – 29.65 (5C), 29.56, 29.38 (7 dodecyl CH_2), 28.0 (NCH_2CH_2), 26.3 ($OCH_2CH_2CH_2$), 22.7 (CH_3CH_2), 14.1 (CH_3); HR ESI MS *m/z* calcd for $C_{39}H_{83}N_2O_4$ (M+H) 643.6347, found 643.6331.

***N*,*N*,*N*',*N*'-Tetramethyl-4,8-dioxa-6,6-bis(tetradecyloxymethyl)-1,11-undecanediamine (5.4).** Sodium hydride (2.2 g, 95 mmol), 2,2-tetradecyloxymethyl-1,3-propanediol (**12**) (5.0 g, 9.4 mmol), and 3-chloro-*N*,*N*-dimethyl-1-propanamine hydrochloride (5.98 g, 37.9 mmol) were reacted as for compound **5.3** and worked up as previously to give compound **5.4** as a light brown liquid: yield 2.2 g

(35%); R_F on basic alumina 0.50 (dichloromethane: methanol 97:5); ^{1}H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 6.5 Hz, CH_3), 1.22-1.34 (m, 44 H, 22 x CH_2), 1.52 (p, 4H, J = 6.5 Hz, OCH_2CH_2), 1.71 (p, 4H, J = 6.5 Hz, NCH_2CH_2), 2.23 (s, 12H, $N(CH_3)_2$), 2.33 (t, 4H, J = 7.5, NCH_2), 3.34 (t, 4H, J = 6.5 Hz, CH_2O), 3.35 (s, 4H, OCH_2), 3.36 (s, 4H, OCH_2), 3.41 (t, 4H, J = 6.5 Hz, OCH_2); ^{13}C NMR ($CDCl_3$) δ 71.7 (tetradecyl CH_2O), 70.0 ($OCH_2CH_2CH_2N$), 69.9 (OCH_2C), 69.8 (OCH_2C), 57.0 (NCH_2), 45.7 $N(CH_3)_2$, 44.8 (qC), 32.1 (OCH_2CH_2), 29.9 (NCH_2CH_2), 29.72, 29.53, 28.14, 26.4 (decyl CH_2), 22.9 (CH_3CH_2), 14.3 (CH_3); HR ESI MS *m/z* calcd for $C_{43}H_{91}N_2O_4$ (M+H) 699.6973, found 699.6986.

***N,N,N,N',N',N'*-Hexamethyl-6,6-bis(octyloxymethyl)-4,8-dioxa-1,11-undecanediammonium diiodide (5.5).** Methyl iodide (1.6 g, 11 mmol) was added to a stirred solution of amine **5.1** (0.6 g, 1.1 mmol) in THF (50 mL) and the reaction mixture was stirred for 36 h, then allowed to cool to rt. The reaction mixture was concentrated and the residue was purified by flash chromatography using as eluant a gradient of 10 to 15% methanol in dichloromethane to give the title salt as an off-white solid: yield 0.7 g (68%); mp 212-215 ^{o}C; R_F 0.4 on basic alumina (8% methanol in dichloromethane); ^{1}H NMR (DMSO-d_6) δ 0.88 (t, 6H, J = 6.5 Hz, CH_3), 1.25-1.37 (m, 20 H, 10 x CH_2), 1.49 (p, 4H, J = 6.5 Hz, OCH_2CH_2), 1.96 (4H, AA' part of AA′BB′ pattern, NCH_2CH_2), 3.11 (s, 18H, $N(CH_3)_3$), 3.31 (s, 4H, octylOCH_2C), 3.33 (t, 4H, J = 6.5 Hz, OCH_2), 3.36 (s, 4H, CH_2OCH_2C), 3.38 (4H, BB' part of AA′BB′ pattern, NCH_2), 3.42 (t, 4H, J = 6.0 Hz, OCH_2); ^{13}C NMR ($CDCl_3$) δ 70.7 (octyl CH_2O), 69.4 ($OCH_2CH_2CH_2N$), 68.8 (OCH_2C), 67.7 (OCH_2C), 63.3 (NCH_2), 52.3 ($N(CH_3)_3$), 45.0 (qC), 31.1 (OCH_2CH_2), 28.74, 28.65, 25.63 (decyl CH_2), 23.0 (NCH_2CH_2), 22.0 (CH_3CH_2), 13.9 (CH_3); HR ESI MS *m/z* calcd for $C_{33}H_{72}IN_2O_4$ (M-I) 687.4531, found 687.4522.

6,6-Bis(decyloxymethyl)-*N,N,N,N',N',N'*-hexamethyl-4,8-dioxa-1,11-undecanediammmonium diiodide (5.6). Methyl iodide (1.3 g, 9.5 mmol) was added to a stirred solution of amine **5.2** (0.7 g, 1.2 mmol) in THF (70 mL) and the reaction mixture was stirred for 36 h, then allowed to cool to rt. The reaction mixture was concentrated and the residue was purified by flash chromatography using as

eluant a gradient of 10 to 15% methanol in dichloromethane to give 1.5.10 as an off-white solid: yield 0.7 g (68%); mp 219-222 ^{o}C; R_F 0.5 on basic alumina (7% methanol in dichloromethane); ^{1}H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 6.5 Hz, CH_3), 1.22-1.35 (m, 28 H, 14 x CH_2), 1.50 (p, 4H, J = 6.5 Hz, OCH_2CH_2), 2.10 (m, 4H, $NCH_2C\mathit{H}_2$), 3.31 (s, 4H, decylOCH_2C), 3.36 (t, 4H, J = 6.5 Hz, OCH_2), 3.41 (s, 4H, CH_2OCH_2C), 3.48 (s, 18H, $N(CH_3)_3$), 3.60 (t, 4H, J = 6.0 Hz, OCH_2), 3.85 (m, 4H, NCH_2); ^{13}C NMR ($CDCl_3$) δ 71.8 (decyl CH_2O), 70.3 ($OC\mathit{H}_2CH_2CH_2N$), 69.3 (OCH_2C), 67.8 (OCH_2C), 65.2 (NCH_2), 54.2 ($N(CH_3)_3$), 45.6 (q,C), 32.0 ($OCH_2\mathit{C}H_2$), 29.87, 29.81, 29.72, 28.51, 26.4 (decyl CH_2), 24.3 ($NCH_2\mathit{C}H_2$), 22.8 ($\mathit{C}H_2CH_3$), 14.2 (CH_3); HR ESI MS *m/z* calcd for $C_{37}H_{80}IN_2O_4$ (M-I) 743.5157, found 743.5130.

6,6-Bis(dodecyloxymethyl)-*N*,*N*,*N*,*N*',*N*',*N*'-hexamethyl-4,8-dioxa-1,11-undecanediammmonium diiodide (5.7). Methyl iodide (1.5 g, 11 mmol) was added to a stirred solution of amine **5.3** (0.70 g, 1.1 mmol) in THF (70 mL) and the reaction mixture was stirred for 36 h, then allowed to cool to rt. The reaction mixture was concentrated and the residue was purified by flash chromatography using as eluant a gradient of 10 to 15% methanol in dichloromethane .to give compound **5.7** as an off-white solid: yield 0.82 g (82%); mp 230-234 ^{o}C; R_F 0.58 on basic alumina (7% methanol in dichloromethane); ^{1}H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 6.5 Hz, CH_3), 1.24-1.35 (m, 36 H, 18 x CH_2), 1.51 (p, 4H, J = 6.5 Hz, $OCH_2C\mathit{H}_2$), 2.10 (m, 4H, $NCH_2C\mathit{H}_2$), 3.31 (s, 4H, decylOCH_2C), 3.35 (t, 4H, J = 6.5 Hz, OCH_2), 3.40 (s, 4H, CH_2OCH_2C), 3.48 (s, 18H, $N(CH_3)_3$), 3.60 (t, 4H, J = 6.0Hz, OCH_2), 3.82 (m, 4H, $NC\mathit{H}_2$); ^{13}C NMR ($CDCl_3$) δ 71.8 (dodecyl CH_2O), 70.4 ($OC\mathit{H}_2CH_2CH_2N$), 69.4 (OCH_2C), 67.9 (OCH_2C), 65.3 (NCH_2), 54.3 ($N(CH_3)_3$), 45.7 (qC), 32.1 ($OCH_2\mathit{C}H_2$), 29.89, 29.74, 29.54, 26.4 (decyl CH_2), 24.4 ($NCH_2\mathit{C}H_2$), 22.9 ($CH_3\mathit{C}H_2$), 14.3 (CH_3); HR ESI MS *m/z* calcd for $C_{41}H_{88}IN_2O_4$ (M-I) 799.5783, found 799.5815.

***N*,*N*,*N*,*N*',*N*',*N*'-Hexamethyl-4,8-dioxa-6,6-bis(tetradecyloxymethyl)-1,11-undecanediammmonium diiodide (5.8).** Methyl iodide (3.1 g, 22 mmol) was added to a stirred

solution of amine **5.4** (1.50 g, 2.23 mmol) in THF (100 mL) and the reaction mixture was stirred for 36 h then allowed to cool to rt. The reaction mixture was concentrated and the solid residue was crystallized from dichloromethane to give pure **5.8** as a shiny white solid: yield 1.5 g (70%); mp 222-225 ^{o}C; R_F 0.6 on basic alumina (7% methanol in dichloromethane); ^{1}H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 6.5 Hz, CH_3), 1.22-1.37 (m, 44 H, 22x CH_2), 1.51 (p, 4H, J = 6.5 Hz, OCH_2CH_2), 2.09 (m, 4H, NCH_2CH_2), 3.31 (s, 4H, decylOCH_2C), 3.35 (t, 4H, J = 6.5 Hz, OCH_2), 3.41 (s, 4H, CH_2OCH_2C), 3.48 (s, 18H, $N(CH_3)_3$), 3.61 (t, 4H, J = 6.0 Hz, OCH_2), 3.90 (m, 4H, NCH_2); ^{13}C NMR ($CDCl_3$) δ 71.8 (tetradecyl CH_2O), 70.4 ($OCH_2CH_2CH_2N$), 69.4 (OCH_2C), 67.8 (OCH_2C), 65.2 (NCH_2), 54.2 ($N(CH_3)_3$), 45.7 (qC), 32.1 (OCH_2CH_2), 29.87, 29.72, 29.51, 26.4 (tetradecyl CH_2), 24.4 (NCH_2CH_2), 22.8 (CH_3CH_2), 14.3 (CH_3); HR ESI MS *m/z* calcd for $C_{45}H_{96}IN_2O_4$ (M-I) 855.6409, found 855.6432.

2,2-Bis(decyloxymethyl)-7-dimethylamino-4-oxa-1-heptanol (5.9). Sodium hydride (0.57 g, 24.0 mmol) was added slowly to a stirred solution of 2,2-didecyloxymethyl-1,3-propanediol (**10**) (1.0 g, 2.4 mmol) in DMF (100 mL) under an N_2 atmosphere at 50 ^{o}C. The mixture was then stirred at 80 ^{o}C for 1 h. The reaction mixture was cooled to rt and 3-(dimethylamino)propyl chloride hydrochloride (1.89 g, 12 mmol) was added in portions. The reaction mixture was stirred at 80 ^{o}C under N_2 for another 12 h, then quenched with methanol. The mixture was filtered, concentrated and the residue was taken up in ethyl acetate (50 mL). This solution was washed with water (2 x 20 mL) and brine (20 mL), then dried (Na_2SO_4) and concentrated to a residue that was purified by flash column chromatography. Elution using a gradient of 3 to 5% MeOH in dichloromethane gave **5.9** as a light brown liquid: yield 0.56 g (40%); R_F on basic alumina 0.55 (dichloromethane: methanol 96: 4); ^{1}H NMR ($CDCl_3$) δ 0.88 (t, 6H, J = 6.5 Hz, CH_3), 1.28-1.32 (m, 28 H, 14 x CH_2), 1.52 (p, 4H, J = 7.0 Hz, OCH_2CH_2), 1.71 (p, 2H, J = 6.0 Hz, NCH_2CH_2), 2.22 (s, 6H, NCH_3), 2.33 (t, 2H, J = 7.5 Hz, NCH_2), 3.38 (t, 4H, J = 6.5 Hz, CH_2O), 3.42 (s, 6H, OCH_2), 3.44 (t, 2H, J = 6.5 Hz, OCH_2), 3.42 (s, 2H, OCH_2); ^{13}C NMR ($CDCl_3$) δ 71.8 (CH_2CH_2O), 71.5 ($OCH_2CH_2CH_2N$), 67.0 (OCH_2C), 66.3 (OCH_2C), 62.8 ($HOCH_2C$), 56.9

(NCH_2), 45.5 (NCH_3), 44.9 (qC), 31.9 (OCH_2), 29.66, 29.61, 29.50, 29.36, 27.8, 26.2 (decyl CH_2), 22.7 (CH_3CH_2), 14.1 (CH_3).

General procedure for reaction with ethyl bromoacetate: diethyl 3,13-diazonia-3,3,13,13-tetramethyl-8,8-bis(octyloxymethyl)-6,10-dioxa-pentadecanedioate dibromide (6.1). When a solution of compound **4.1** (0.61g, 1.21 mmol) and ethyl bromoacetate (0.31 mL, 2.79 mmol, 2.3 eq) in diethyl ether (20 mL) was stirred for 26 h, a colorless solid formed that was isolated by filtration, then washed with ether. The solid was crystallized from ethyl acetate and methanol to give the title compound as a colorless crystalline solid: yield 0.88 g, 84 %; mp 110-111 ^oC; R_F on basic alumina 0.59 (butanol: water: methanol 20: 5 : 2); ^1H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.27 (brs, 20H, 10 x CH_2), 1.32 (t, 6H, J = 7.2 Hz, 2 x Me), 1.50 (pentet, 4H, J = 6.0 Hz, 2 OCH_2CH_2), 3.28 (s, 4H, octyl OCH_2C), 3.33 (t, 4H, J = 6.6 Hz, octyl OCH_2C), 3.50 (s, 4H, $CCH_2O(CH_2)_2N$), 3.71 (s, 12H, $2N(CH_3)_2$), 3.96 (br AA` part of AA`BB` pattern, 4H, 2 $OCH_2CH_2N$), 4.27 (q, 4H, J = 7.2 Hz, $OCH_2CH_3$), 4.32 (br BB` part of AA`BB` pattern, 4H, 2 NCH_2CH_2O), 4.96 (s, 4H, CH_2COOR); ^{13}C NMR δ 164.9 (CH_2COOR), 71.8 (CH_2CH_2OC), 71.1 ($CCH_2O(CH_2)_2N$), 69.4 (CCH_2O octyl), 65.4 (OCH_2CH_2N), 64.1 (NCH_2CH_2O), 62.7 ($CH_2COOCH_2CH_3$), 62.3 ($CH_2COOCH_2CH_3$), 52.4 ($N(CH_3)_2$), 45.1(qC), 32.0 ($CH_2CH_2CH_3$), 29.6, 29.5, 29.3, (octyl CH_2), 26.2 ($CH_2CH_2CH_2O$), 22.7 (CH_2CH_3), 14.12 (Me), 14.06 (CH_3CH_2); HR ESI MS *m/z* calc for $C_{37}H_{76}N_2O_8/2$ ((M-2Br)/2) 338.2795, found 338.2811.

Diethyl 3,13-diazonia-8,8-bis(decyloxymethyl)-3,3,13,13-tetramethyl-6,10-dioxapentadecane-dioate dibromide (6.2). A solution of compound **4.2** (1.86 g, 3.33 mmol) and ethyl bromoacetate (0.85 mL,7.66 mmol, 2.3 eq) in diethyl ether (35 mL) was stirred for 26 h then filtered and concentrated to give a colorless solid that was crystallized from ethyl acetate and methanol to give the title compound as a colorless crystalline solid, yield: 2.66 g, 90 %; mp 114-115 ^oC; R_F on basic alumina 0.61 (butanol: water: methanol 20: 5 : 2); ^1H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.27

(brs, 28H, 14 x CH_2), 1.32 (t, 6H, J = 7.2 Hz, 2 x Me), 1.50 (pentet, 4H, J = 6.0 Hz, 2 OCH_2CH_2), 3.27 (s, 4H, decyl, OCH_2C), 3.32 (t, 4H, J = 6.5 Hz, decyl OCH_2C), 3.49 (s, 4H, $CCH_2O(CH_2)_2N$), 3.71 (s, 12H, $2N(CH_3)_2$), 3.94 (br AA` part of AA`BB` pattern, 4H, 2 $OCH_2CH_2N$), 4.27 (q, 4H, J = 7.2 Hz, $OCH_2CH_3$), 4.31 (br BB` part of AA`BB` pattern, 4H, 2 NCH_2CH_2O), 4.96 (s, 4H, CH_2COOR); ^{13}C NMR δ 165.0 (CH_2COOR), 71.9 (CH_2CH_2OC), 71.1 ($CCH_2O(CH_2)_2N$), 69.4 (CCH_2O decyl), 65.4 (OCH_2CH_2N), 64.3 (NCH_2CH_2O), 62.8 ($CH_2COOCH_2CH_3$), 62.4 ($CH_2COOCH_2CH_3$), 52.5 ($N(CH_3)_2$), 45.2 (qC), 32.0 ($CH_2CH_2CH_3$), 29.8, 29.7, 29.6, 29.5 (decylCH_2), 26.4 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.2 (Me), 14.1 (CH_3CH_2); HR ESI MS *m/z* calc for $C_{41}H_{84}N_2O_8/2$ ((M-2Br)/2) 366.3108, found 366.3112.

Diethyl 3,13-diazonia-8,8-bis(dodecyloxymethyl)-3,3,13,13-tetramethyl-6,10-dioxapentadecane-dioate dibromide (6.3). A solution of compound **4.3** (1.77 g, 2.88 mmol) and ethyl bromoacetate (0.73 mL, 6.60 mmol, 2.3 eq) in diethyl ether (35 mL) was treated as above to give the title compound as a colorless solid that was crystallized from ethyl acetate and methanol to give a colorless crystalline solid, yield: 2.05 g, 73%; mp 119-120 ^oC; R_F on basic alumina 0.63 (butanol: water: methanol 20: 5 : 2); ^1H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.26 (brs, 36H, 18 x CH_2), 1.32 (t, 6H, J = 7.2 Hz, 2 x Me), 1.51 (pentet, 4H, J = 6.4 Hz, 2 OCH_2CH_2), 3.27 (s, 4H, dodecyl, OCH_2C), 3.33 (t, 4H, J = 6.6 Hz, dodecyl, OCH_2C), 3.49 (s, 4H, $CCH_2O(CH_2)_2N$), 3.70 (s, 12H, $2N(CH_3)_2$), 3.95 (br AA` part of AA`BB` pattern, 4H, 2 $OCH_2CH_2N$), 4.26 (q, 4H, J = 7.2 Hz, $OCH_2CH_3$), 4.31 (br BB` part of AA`BB` pattern, 4H, 2 NCH_2CH_2O), 4.94 (s, 4H, CH_2COOR); ^{13}C NMR δ 164.9 (CH_2COOR), 71.8 (CH_2CH_2OC), 71.1 ($CCH_2O(CH_2)_2N$), 69.4 (CCH_2O dodecyl), 65.4 (OCH_2CH_2N), 64.1 (NCH_2CH_2O), 62.7 ($CH_2COOCH_2CH_3$), 62.3 ($CH_2COOCH_2CH_3$), 52.4 ($N(CH_3)_2$), 45.1 (qC), 32.0 ($CH_2CH_2CH_3$), 29.7, 29.5, 29.4 (dodecylCH_2), 26.3 ($CH_2CH_2CH_2O$), 22.7 (CH_2CH_3), 14.12 (Me), 14.06 (CH_3CH_2); HR ESI MS *m/z* calcd for $C_{45}H_{92}N_2O_8/2$ ((M-2Br)/2) 394.3421, found 394.3424.

Diethyl 3,13-diazonia-3,3,13,13-tetramethyl-6,10-dioxa-8,8-bis(tetradecyloxymethyl)penta-decanedioate dibromide (**6.4**). A solution of compound **4.4** (0.78 g, 1.22 mmol) and ethyl bromoacetate (0.31 mL, 2.80 mmol, 2.3 eq) in diethyl ether (25 mL) was treated as above to give the title compound (**6.4**) as a colorless solid that precipitated from ethyl acetate and methanol to give a colorless amorphous solid, yield: 0.89 g, 71 %; mp 116-117 ^{o}C; R_F on basic alumina 0.66 (butanol: water: methanol 20: 5 : 2); ^{1}H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.26 (brs, 44H, 22 x CH_2), 1.32 (t, 6H, J = 7.2 Hz, 2 x Me), 1.50 (pentet, 4H, J = 6.0 Hz, 2 OCH_2CH_2), 3.27 (s, 4H, tetradecyl, OCH_2C), 3.32 (t, 4H, J = 6.6 Hz, tetradecyl, OCH_2C), 3.48 (s, 4H, $CCH_2O(CH_2)_2N$), 3.71 (s, 12H, $2N(CH_3)_2$), 3.93 (br AA` part of AA`BB` pattern, 4H, 2 $OCH_2CH_2N$), 4.26 (q, 4H, J = 7.2 Hz, $COCH_2CH_3$), 4.30 (br BB` part of AA`BB` pattern, 4H, 2 NCH_2CH_2O), 4.95 (s, 4H, CH_2COOR); ^{13}C NMR δ 164.9 (CH_2COOR), 71.8 (CH_2CH_2OC), 71.1 ($CCH_2O(CH_2)_2N$), 69.4 (CCH_2O tetradecyl), 65.4 (OCH_2CH_2N), 64.2 (NCH_2CH_2O), 62.7 ($CH_2COOCH_2CH_3$), 62.4 ($CH_2COOCH_2CH_3$), 52.5 ($N(CH_3)_2$), 45.1 (qC), 32.0 ($CH_2CH_2CH_3$), 29.8, 29.7, 29.6, 29.4 (tetradecyl CH_2), 26.3 ($CH_2CH_2CH_2O$), 22.8 (CH_2CH_3), 14.2 (Me), 14.1 (CH_3CH_2O); HR ESI MS *m/z* calcd for $C_{49}H_{100}N_2O_8/2$ ((M-2Br)/2) 422.3734, found 422.3721.

General procedure for ester hydrolysis: 3,13-Diazonia-3,3,13,13-tetramethyl-8,8-bis(octyloxymethyl)-6,10-dioxapentadecanedioate (6.5). Compound **6.1** (0.44 g, 0.51 mmol) and IRA-400 anion-exchange resin (OH^-) (11.0 g) in ethanol (30 mL) were stirred at rt for 24 h. The reaction mixture was filtered and the filtrate was concentrated to a semi-solid residue that precipitated from ethyl acetate and methanol to give the title compound as a colorless waxy mass, yield: 0.28 g, 90 %; mp 183 ^oC; R_F on basic alumina 0.37 (butanol: water: methanol 20: 5 : 2); ^1H NMR δ 0.88 (t, 6H, J = 6.7 Hz, 2 x Me), 1.28-1.32 (brs, 20H, 10 x CH_2), 1.51 (pentet, 4H, J = 6.2 Hz, 2 OCH_2CH_2), 3.29 (s, 4H, octyl OCH_2C), 3.33 (t, 4H, J = 6.4 Hz, octyl OCH_2), 3.39 (s, 4H, $CCH_2O(CH_2)_2N$), 3.39 (s, 12H, $2N(CH_3)_2$), 3.82 (br AA` part of AA`BB` pattern, 4H, 2 $NCH_2CH_2O$), 3.99 (BB` part of AA`BB` pattern, 4H, 2 OCH_2CH_2N), 3.99 (s, 4H, CH_2COO^-); ^{13}C NMR δ 166.2 (CH_2COO^-), 71.4 (CH_2CH_2OC), 70.6 ($CCH_2O(CH_2)_2N$), 69.2 (CCH_2O octyl), 65.46 (NCH_2CH_2O), 65.3 (OCH_2CH_2N), 62.4 (CH_2COO^-), 52.0 ($N(CH_3)_2$), 44.7 (q C), 31.6 ($CH_2CH_2C H_3$), 29.3, 29.2, 29.1 (octyl CH_2), 25.9 ($CH_2CH_2CH_2O$), 22.4 (CH_2CH_3), 13.8 (Me); HR ESI MS *m/z* calcd for $C_{33}H_{67}N_2O_8$ (M+H) 619.4892, found 619.4859.

3,13-Diazonia-8,8-bis(decyloxymethyl)-3,3,13,13-tetramethyl-6,10-dioxapentadecanedioate (6.6). Compound **6.2** (2.21 g, 2.55 mmol) and IRA-400 anion-exchange resin (OH^-) (11.6 g) in ethanol (40 mL) were stirred at rt as above. The reaction mixture was filtered and concentrated to a semi-solid residue that precipitated from ethyl acetate and methanol to give the title compound as a colorless waxy solid, yield: 1.47 g, 89 %; mp 175 ^oC; R_F on basic alumina 0.35 (butanol: water: methanol 20: 5 : 2); ^1H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.21-1.32 (brs, 28H, 14 x CH_2,), 1.51 (pentet, 4H, J = 6.5 Hz, 2 OCH_2CH_2), 3.28 (s, 4H, decylOCH_2C), 3.33 (t, 4H, J = 6.4 Hz, decyl OCH_2), 3.38 (s, 12H, $2N(CH_3)_2$), 3.39 (s, 4H, $CCH_2O(CH_2)_2N$), 3.81 (br AA` part of AA`BB` pattern, 4H, 2 $NCH_2CH_2O$), 3.95 (BB` part of AA`BB` pattern, 4H, 2 OCH_2CH_2N), 4.12 (s, 4H, CH_2COO^-); ^{13}C NMR δ 167.9 (CH_2COO^-), 71.7 (CH_2CH_2OC), 70.8 ($CCH_2O(CH_2)_2N$), 69.3 (CCH_2Odecyl), 65.6 (NCH_2CH_2O), 65.5

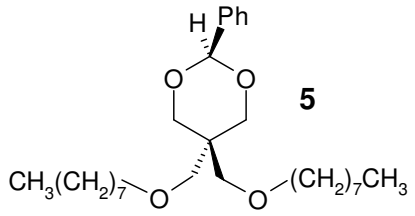
(O*C*$H_2$$CH_2$N), 63.1 (C*H*$_2COO^-$), 52.1 (N ($CH_3$)$_2$), 45.1(q C), 32.0 (*C*$H_2$$CH_2$$CH_3$), 29.7, 29.7, 29.6, 29.4 (decyl CH_2), 26.3 (*C*$H_2$$CH_2$$CH_2$O), 22.7 (*C*$H_2$$CH_3$), 14.1 (Me); HR ESI MS *m/z* calcd for $C_{37}H_{74}N_2O_8Na$ (M+Na) 697.5337, found 697.5304.

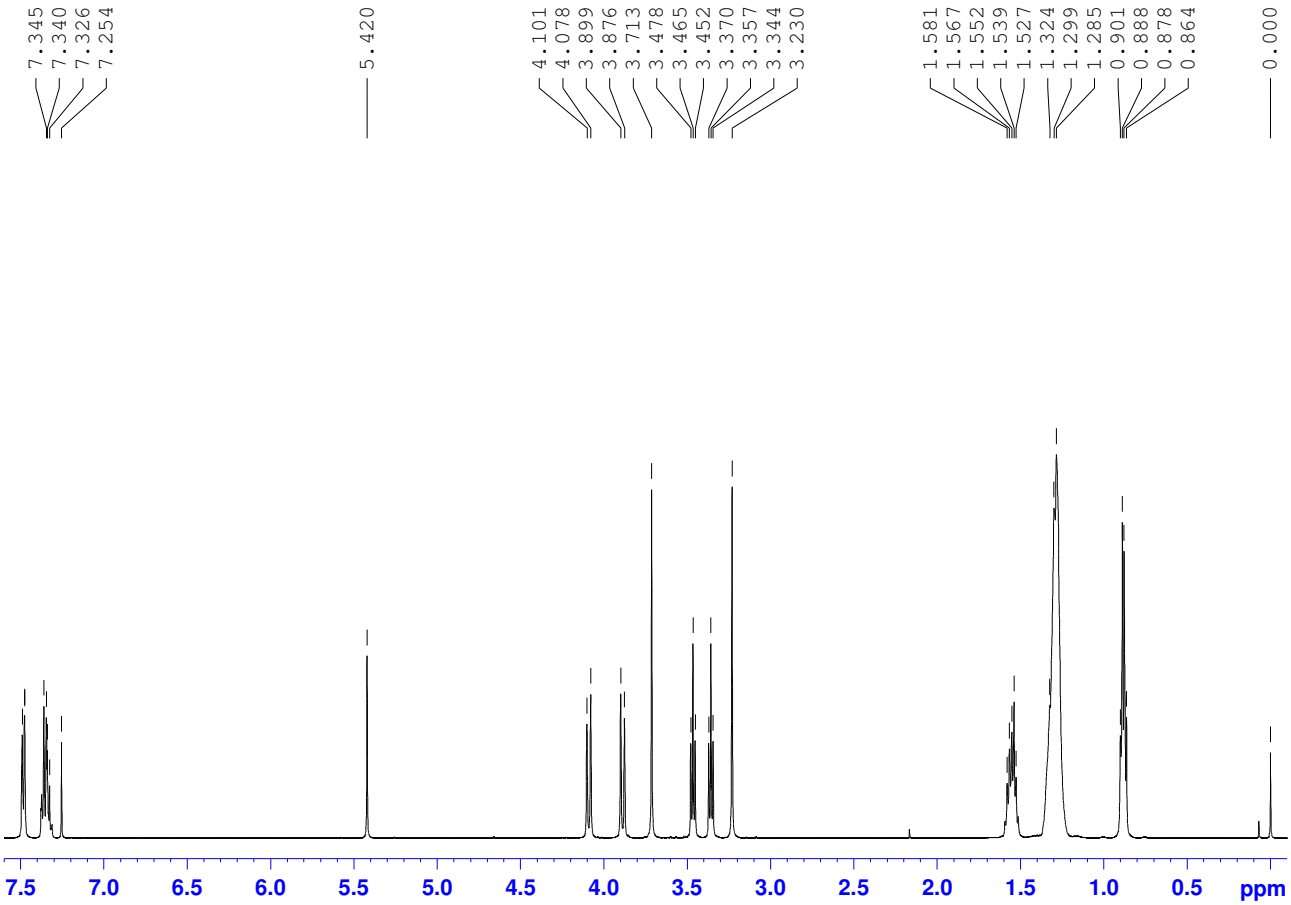
3,13-Diazonia-8,8-bis(dodecyloxymethyl)-3,3,13,13-tetramethyl-6,10-dioxapentadecanedioate (6.7). Compound **6.3** (1.54 g, 1.62 mmol) and IRA-400 anion-exchange resin (OH$^-$) (12.0 g) in ethanol (50 mL) were stirred at rt as above. The reaction mixture was filtered and concentrated to semi-solid residue that was crystallized from ethyl acetate and methanol to give the title compound (**6.7**) as a colorless granules: yield 1.1 g, 93 %; mp 170-171 ^{o}C; R_F on basic alumina 0.33 (butanol: water: methanol 20: 5 : 2); ^{1}H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.26 (brs, 36H, 18 x CH_2), 1.51 (pentet, 4H, J = 6.5 Hz, 2 OCH_2C*H*$_2$), 3.28 (s, 4H, dodecyl OCH_2C), 3.33 (t, 4H, J = 6.4 Hz, dodecyl OCH_2), 3.39 (s, 12H, 2N(CH_3)$_2$) 3.4 (s, 4H, CC*H*$_2$O(CH_2)$_2$N), 3.83 (br AA` part of AA`BB` pattern, 4H, 2 NC*H*$_2$$CH_2$O), 3.99 (BB` part of AA`BB` pattern, 4H, 2 OC*H*$_2$$CH_2$N), 3.99 (s, 4H, C*H*$_2COO^-$); ^{13}C NMR δ 166.3 ($CH_2$*C*OO$^-$), 71.8 ($CH_2$*C*$H_2$OC), 70.9 (C*C*$H_2$O($CH_2$)$_2$N), 69.5 (C*C*$H_2$O dodecyl), 65.7 (N*C*$H_2$$CH_2$O), 65.5 (O*C*$H_2$$CH_2$N), 62.7 (C*H*$_2COO^-$), 52.4 (N($CH_3$)$_2$), 45.1(q C), 32.0 (*C*$H_2$$CH_2$$CH_3$), 29.7, 29.6, 29.4 (dodecyl CH_2), 26.3 (*C*$H_2$$CH_2$$CH_2$O), 22.7 (*C*$H_2$$CH_3$), 14.1 (Me); HR ESI MS *m/z* calcd for $C_{41}H_{83}N_2O_8$ (M+H) 731.6144, found 731.6117.

3,13-Diazonia-3,3,13,13-tetramethyl-6,10-dioxa-8,8-bis(tetradecyloxymethyl)pentadecanedioate (6.8). Compound **6.4** (0.89 g, 0.88 mmol) and IRA-400 anion-exchange resin (OH$^-$) (11.2 g) in ethanol (30 mL) were stirred at rt for 24 h. The reaction mixture was filtered and the filtrate was concentrated to a colorless semi-solid residue that was crystallized from ethyl acetate and methanol to give the title compound as a colorless powder: yield 0.65 g, 92 %; mp 168 ^{o}C; R_F on basic alumina 0.30 (butanol: water: methanol 20: 5 : 2); ^{1}H NMR δ 0.88 (t, 6H, J = 6.9 Hz, 2 x Me), 1.26-1.31 (brs, 44H, 22 x CH_2), 1.50 (pentet, 4H, J = 6.2 Hz, 2 OCH_2C*H*$_2$), 3.28 (s, 4H, tetradecylOCH_2C), 3.33 (t, 4H, J = 6.4 Hz, tetradecyl OCH_2), 3.40 (s, 12H, 2N(CH_3)$_2$), 3.40 (s, 4H, CC*H*$_2$O(CH_2)$_2$N), 3.83 (br AA` part of

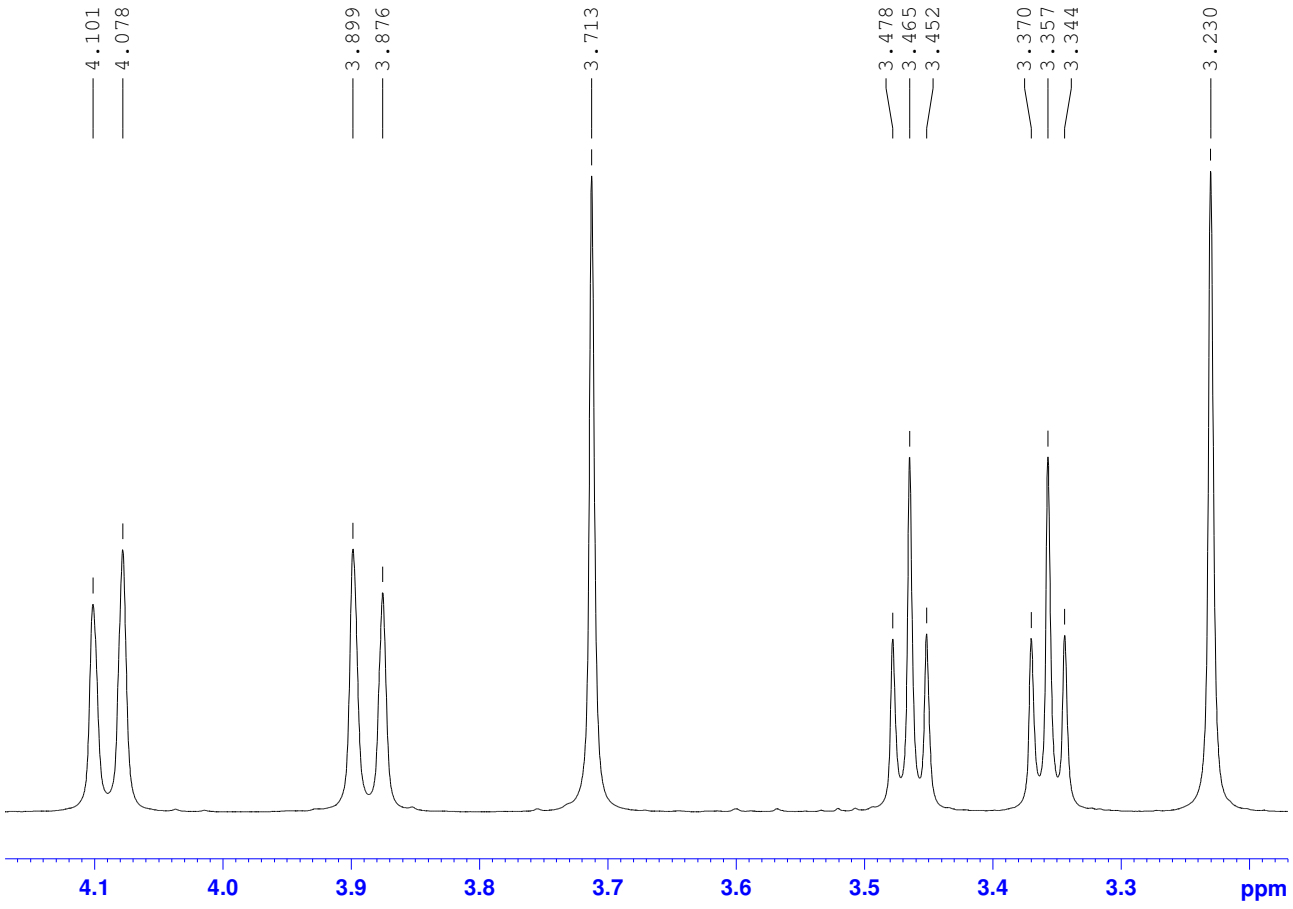
AA`BB` pattern, 4H, 2 NCH_2CH$_2$O), 3.98 (BB` part of AA`BB` pattern, 4H, 2 OCH_2CH$_2$N), 4.10 (s, 4H, CH_2COO$^-$); ^{13}C NMR δ 166.1 (CH$_2$*C*OO$^-$), 71.8 (CH$_2$*C*H$_2$OC), 70.9 (C*C*H$_2$O(CH$_2$)$_2$N), 69.5 (C*C*H$_2$Odecyl), 65.7 (N*C*H$_2$CH$_2$O), 65.5 (O*C*H$_2$CH$_2$N), 62.9 (CH_2COO$^-$), 52.4 (N (CH$_3$)$_2$), 45.2 (q C), 32.1 (*C*H$_2$CH$_2$CH$_3$), 29.9, 29.8, 29.7, 29.5 (tetradecyl CH$_2$), 26.4 (*C*H$_2$CH$_2$CH$_2$O), 22.8 (*C*H$_2$CH$_3$), 14.2 (Me); HR ESI MS *m/z* calcd for $C_{45}H_{90}N_2O_8Na$ (M+Na) 809.6589, found 809.6567.

500.1 MHz ^{1}H NMR spectrum of 5,5'-bis(octyloxymethyl)-2-phenyl-1,3-dioxane (**5**) in $CDCl_3$

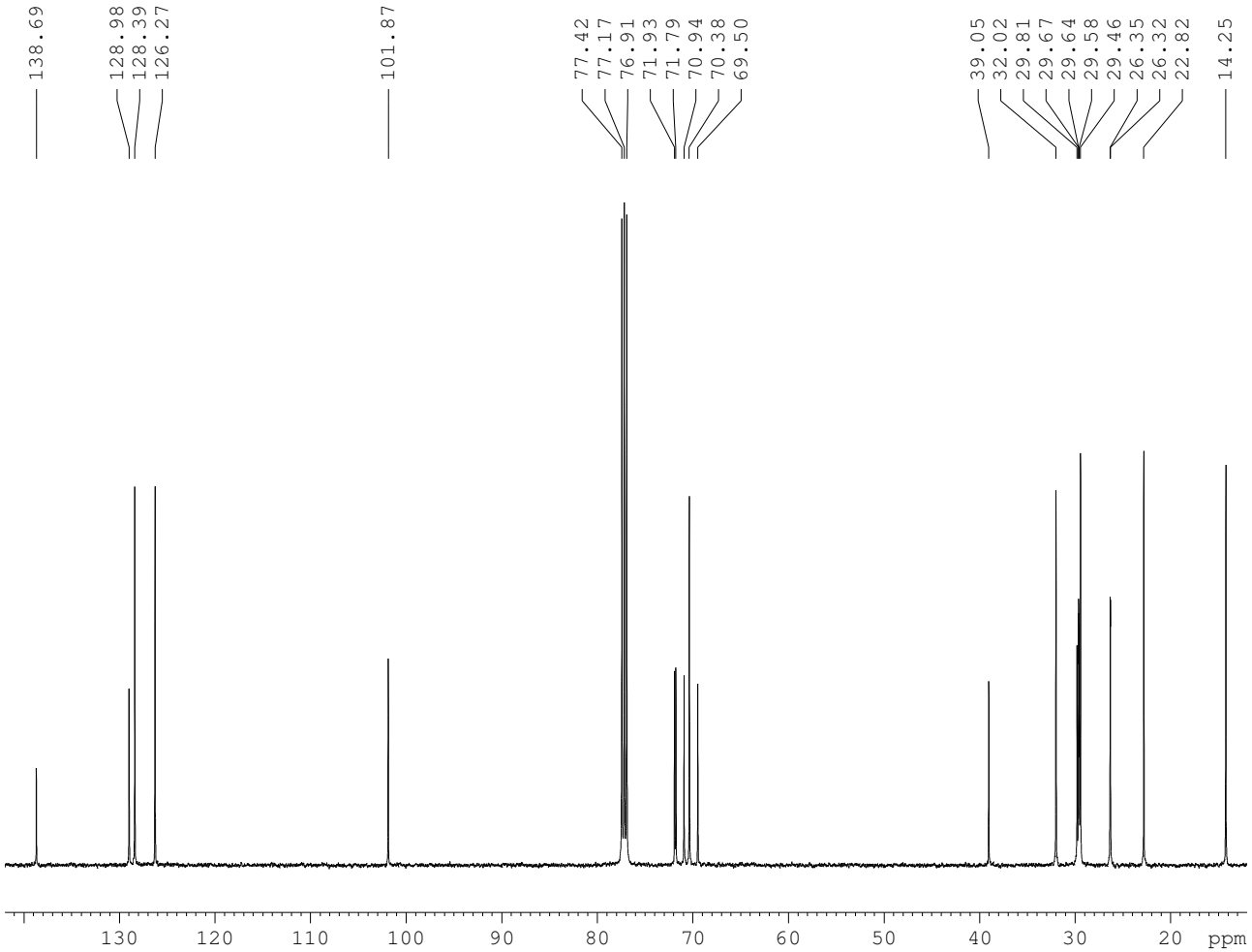




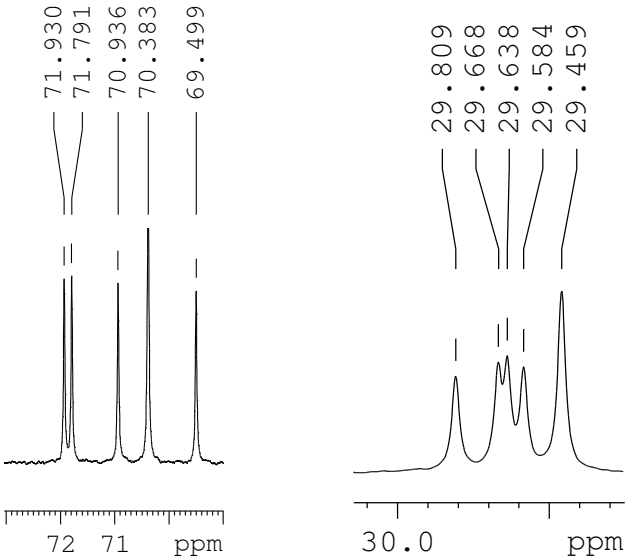
Expansion of part of the 500.1 MHz ^{1}H NMR spectrum of 5,5'-bis(octyloxymethyl)-2-phenyl-1,3-dioxane (**5**) in chloroform-*d*



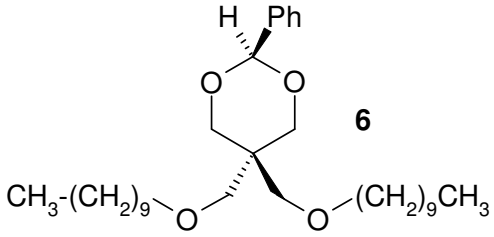
125.7 MHz ^{13}CNMR spectrum of 5,5'-bis(octyloxymethyl)-2-phenyl-1,3-dioxane (**5**) in chloroform-*d*

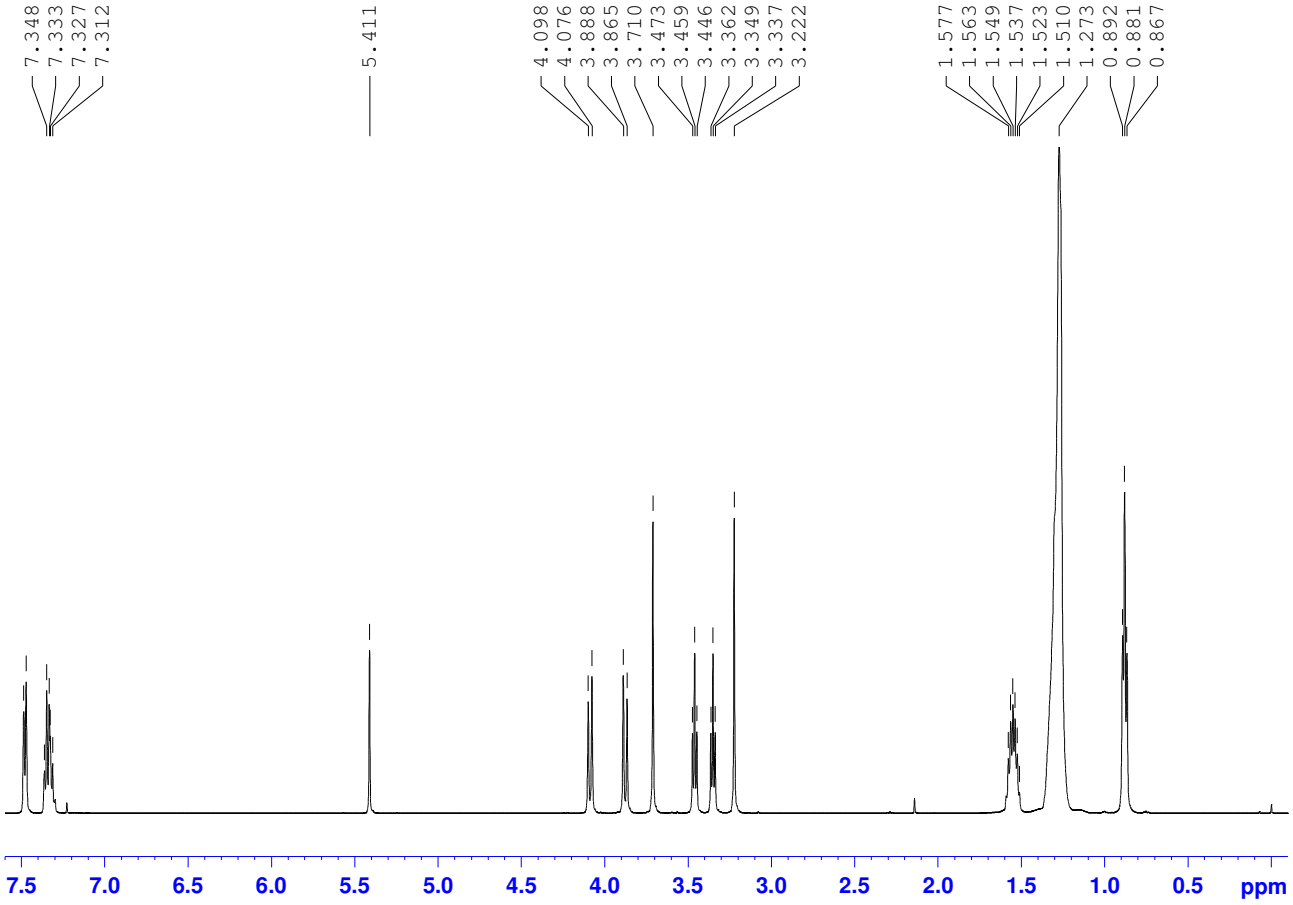


Expansions of parts of the 125.7 MHz ^{13}C NMR spectrum of 5,5'-bis(octyloxymethyl)-2-phenyl-1,3-dioxane (**5**) in $CDCl_3$

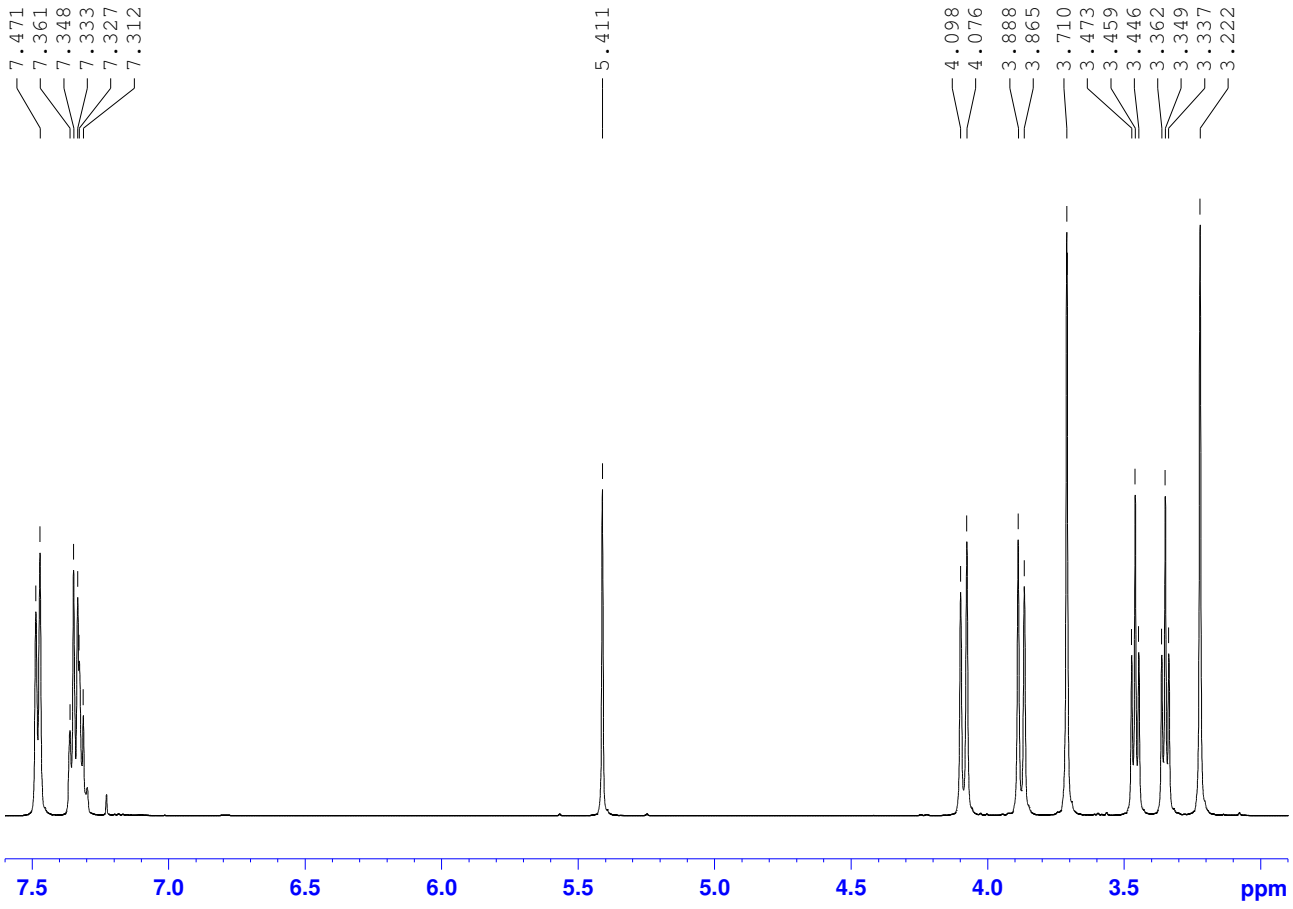


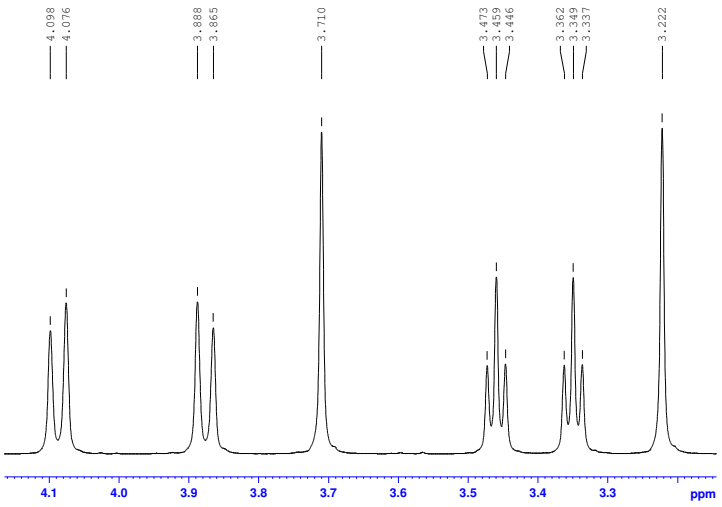
500.1 MHz ^{1}H NMR spectrum of 5,5'-bis(decyloxymethyl)-2-phenyl-1,3-dioxane (**6**) in chloroform-*d*



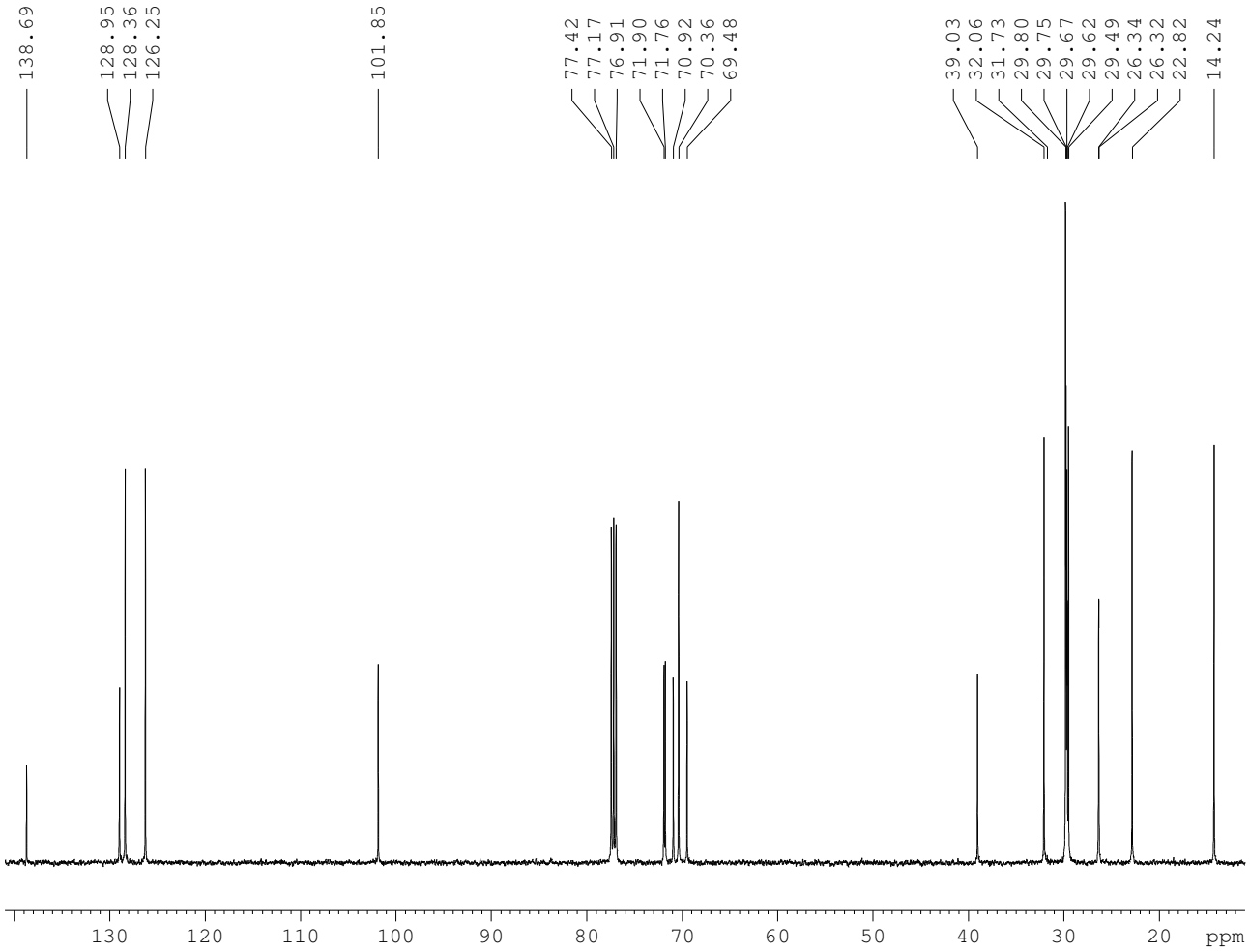


Expansions of parts of the 500.1 MHz ^{1}H NMR spectrum of 5,5'-bis(decyloxymethyl)-2-phenyl-1,3-dioxane (**6**) in $CDCl_3$

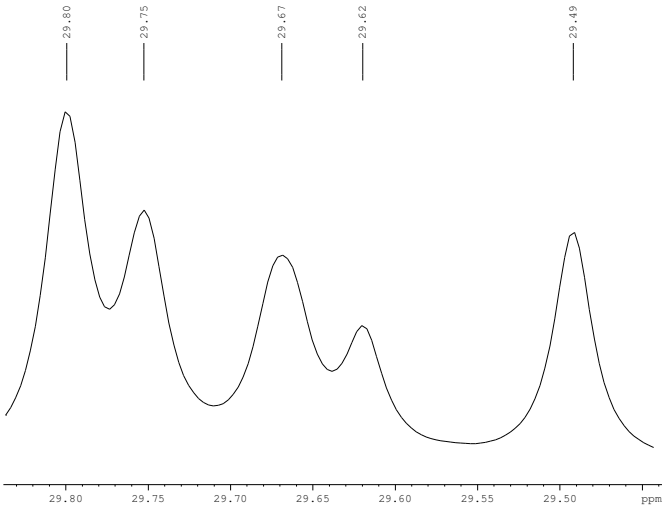




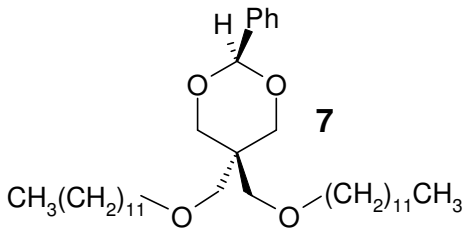
125.7 MHz ^{13}CNMR spectrum of 5,5'-bis(decyloxymethyl)-2-phenyl-1,3-dioxane (**6**) in chloroform-*d*

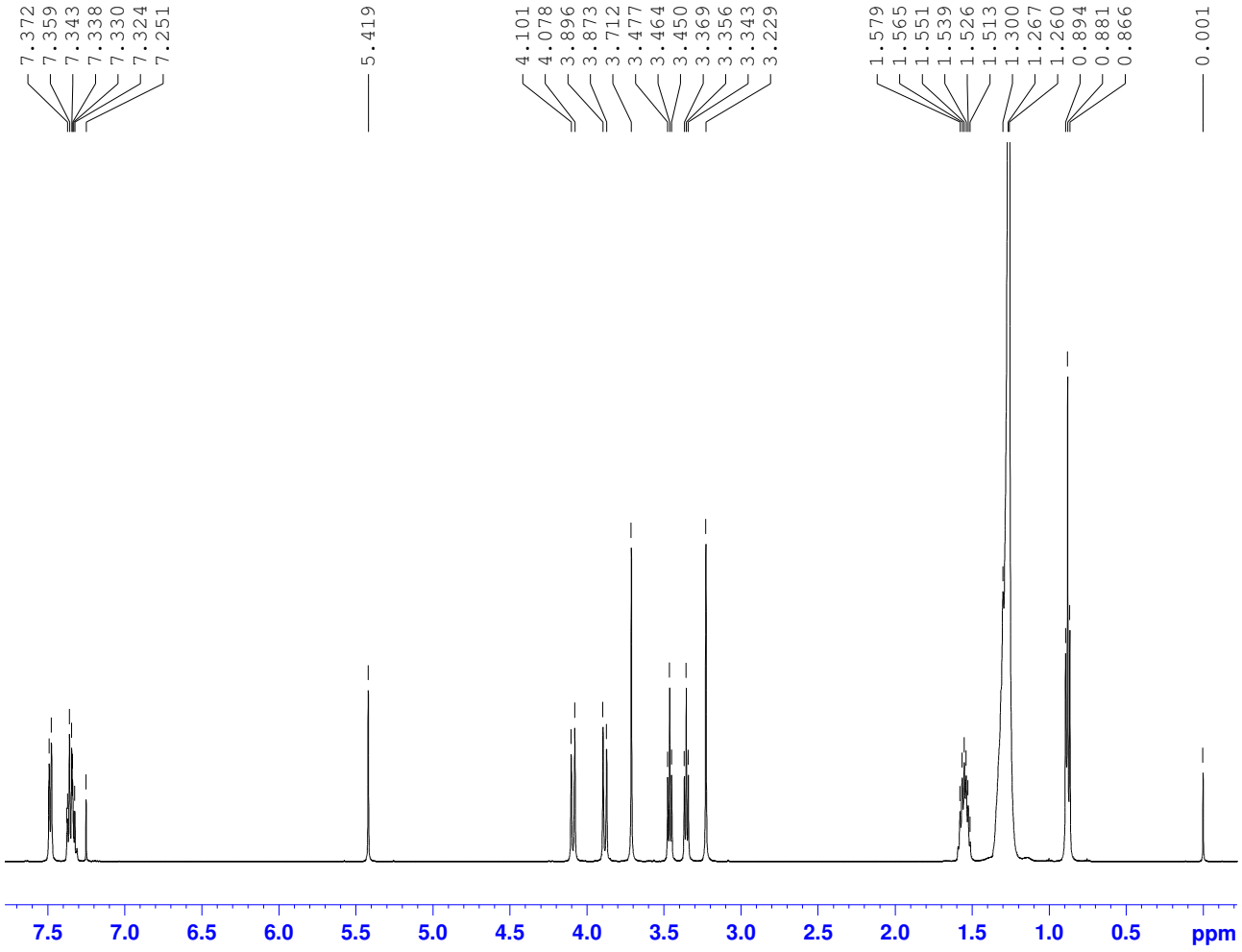


Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of 5,5'-bis(decyloxymethyl)-2-phenyl-1,3-dioxane (**6**) in $CDCl_3$

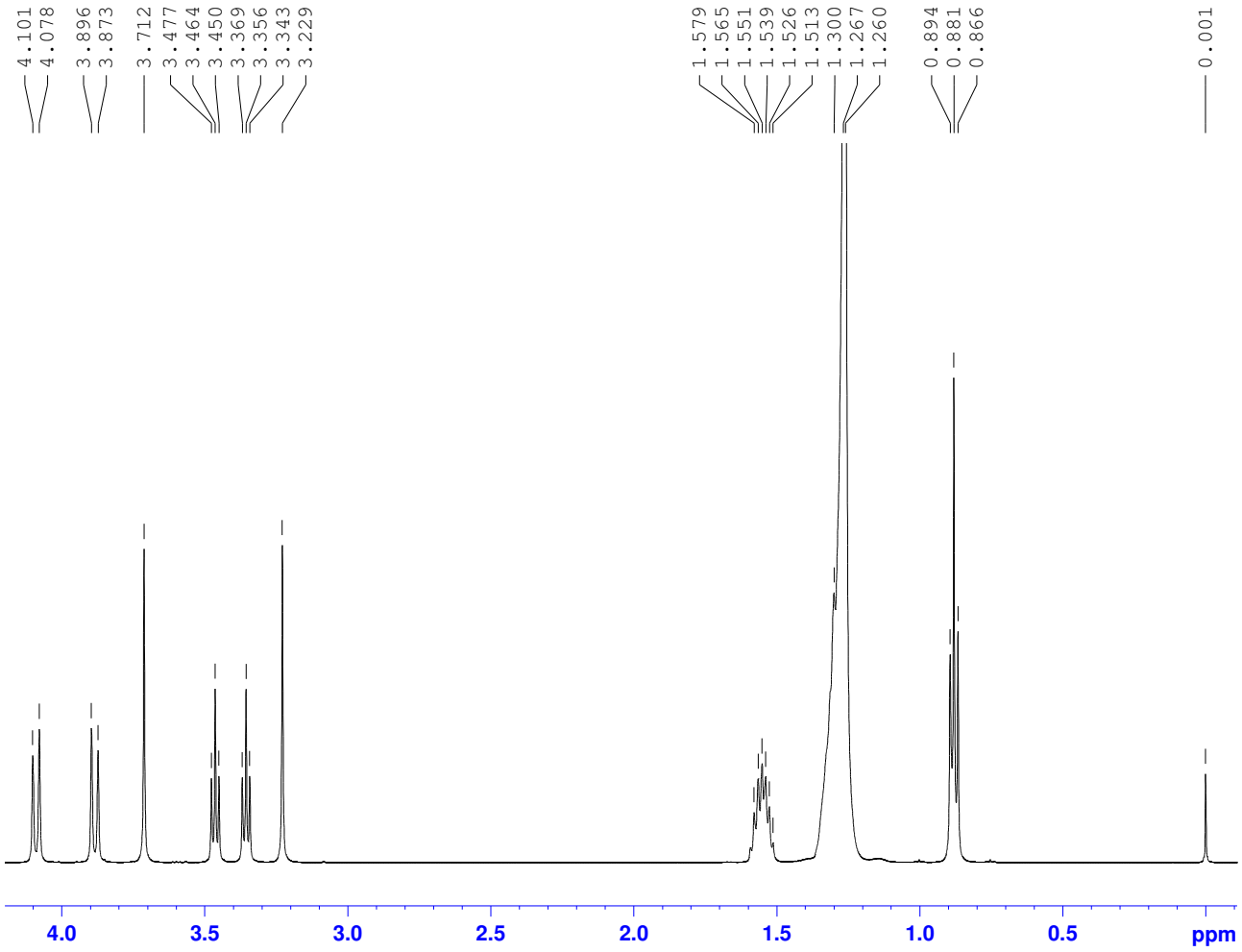


500.1 MHz ^{1}H NMR spectrum of 5,5'-bis(dodecyloxymethyl)-2-phenyl-1,3-dioxane (**7**)

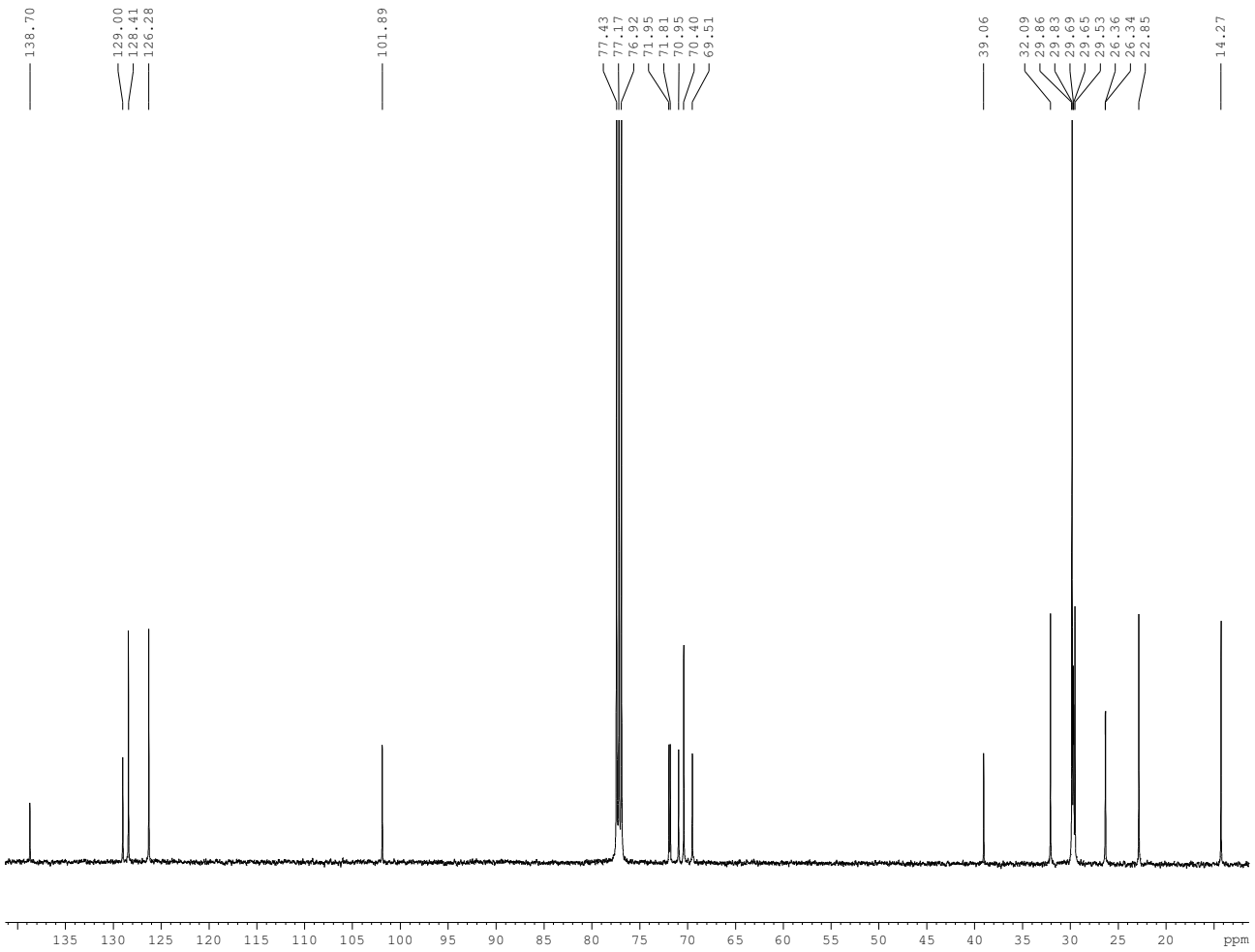




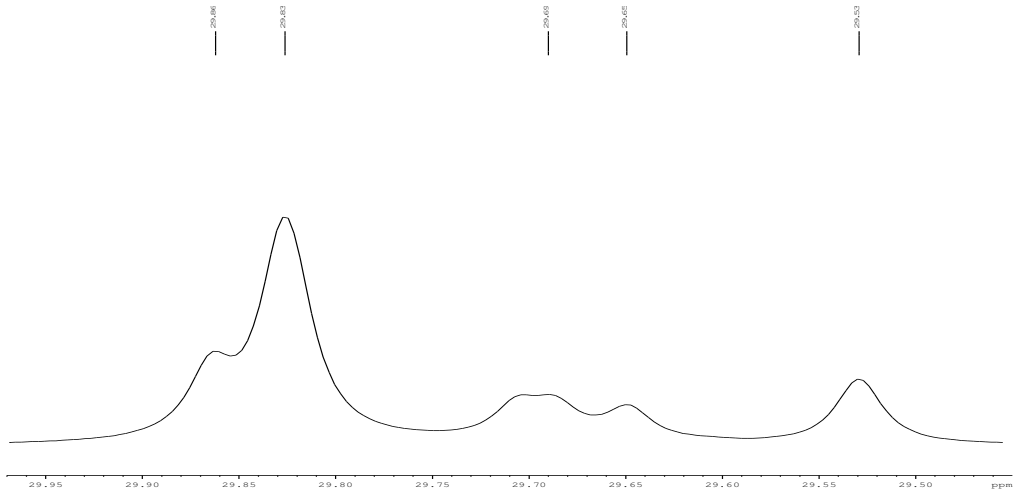
Expansion of part of the 500.1 MHz ^{1}H NMR spectrum of 5,5'-bis(dodecyloxymethyl)-2-phenyl-1,3-dioxane (**7**) in chloroform-*d*



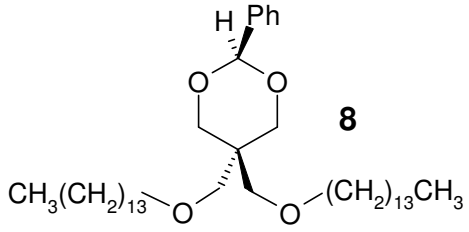
125.7 MHz ^{13}CNMR spectrum of 5,5'-bis(dodecyloxymethyl)-2-phenyl-1,3-dioxane (**7**) in chloroform-*d*

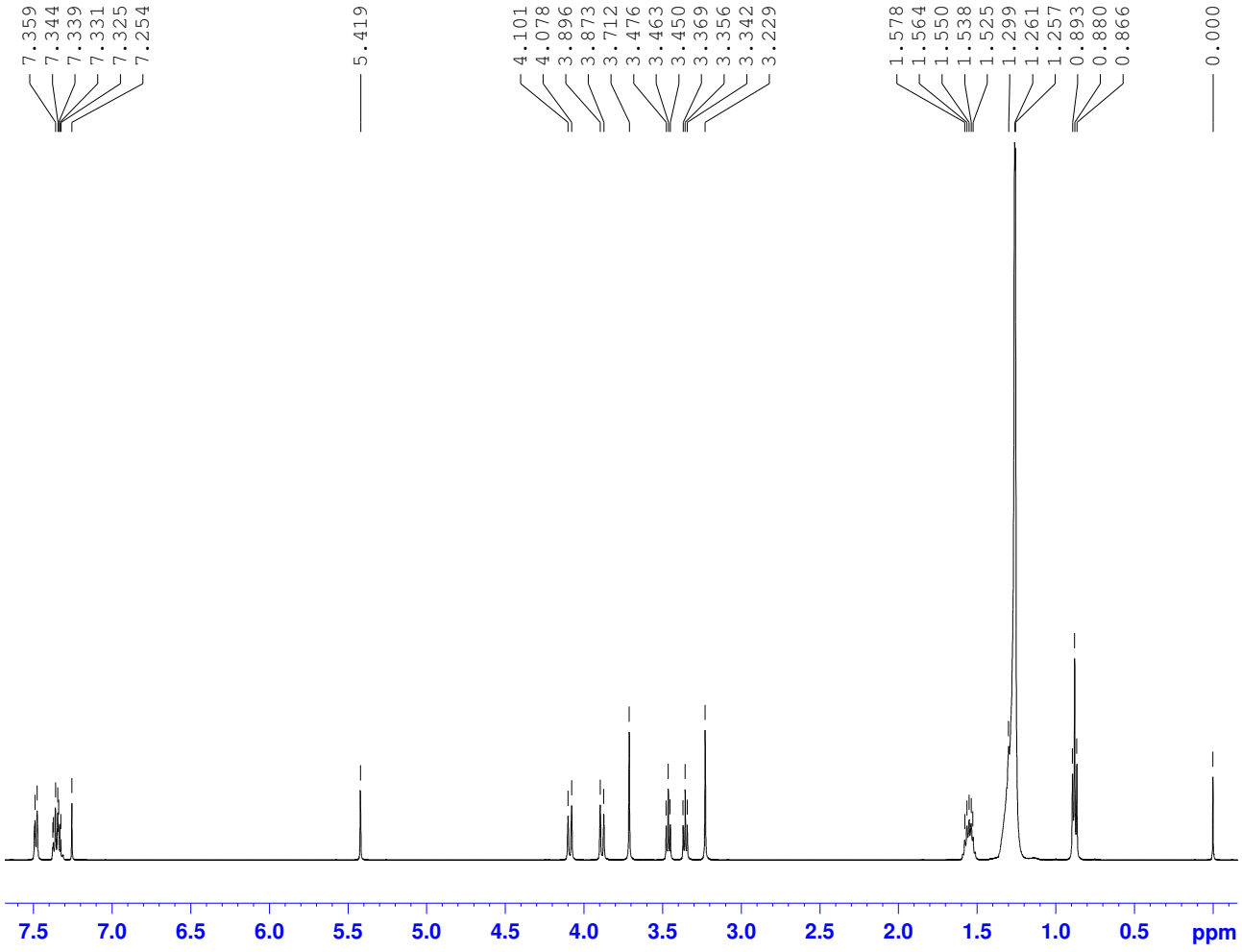


Exapansion of part of the 125.7 MHz ^{13}CNMR spectrum of 5,5'-bis(dodecyloxymethyl)-2-phenyl-1,3-dioxane (**7**) in chloroform-*d*

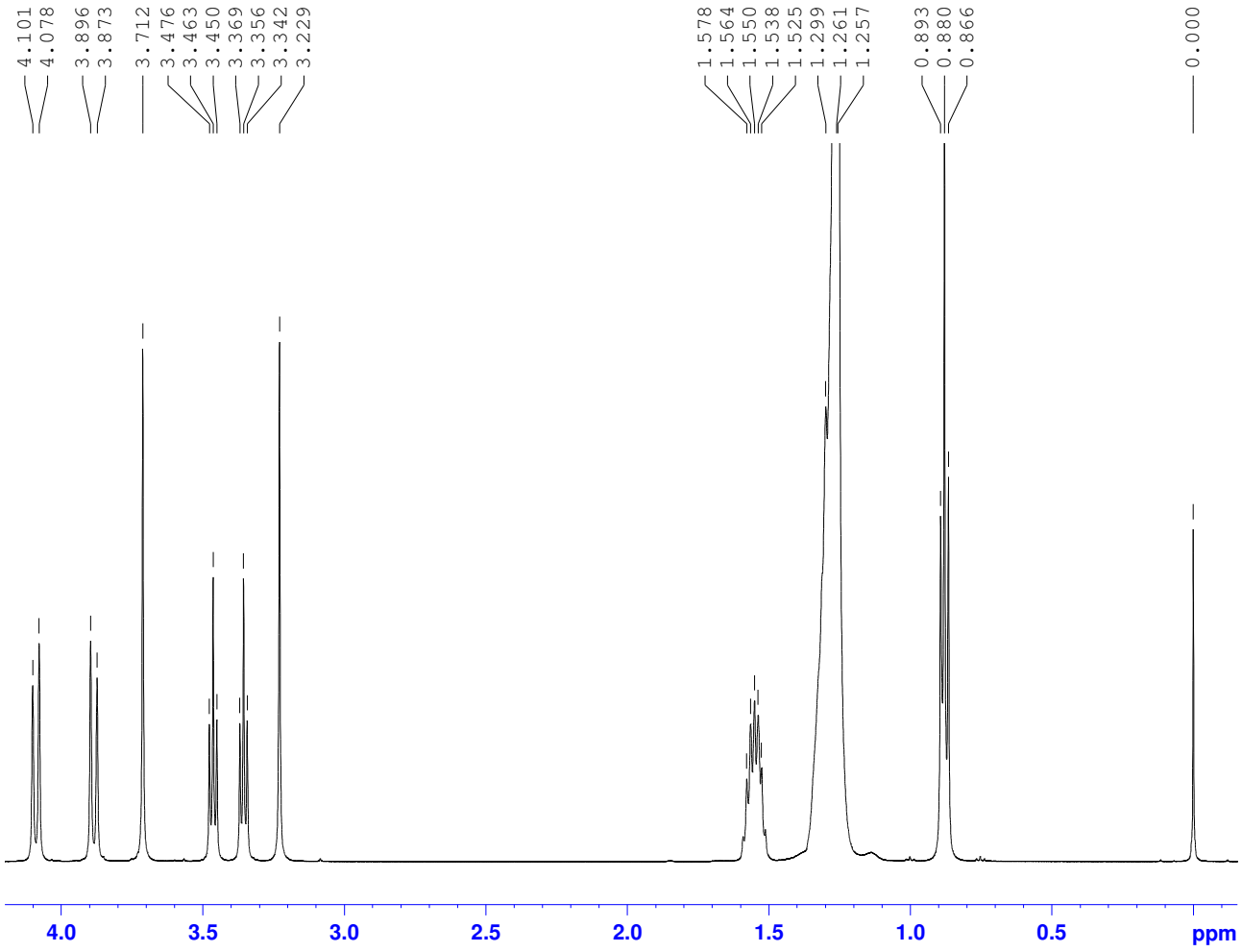


500.1 MHz ^{1}H NMR spectrum of 2-phenyl-5,5'-bis(tetradecyloxymethyl)-1,3-dioxane (**8**) in chloroform-*d*

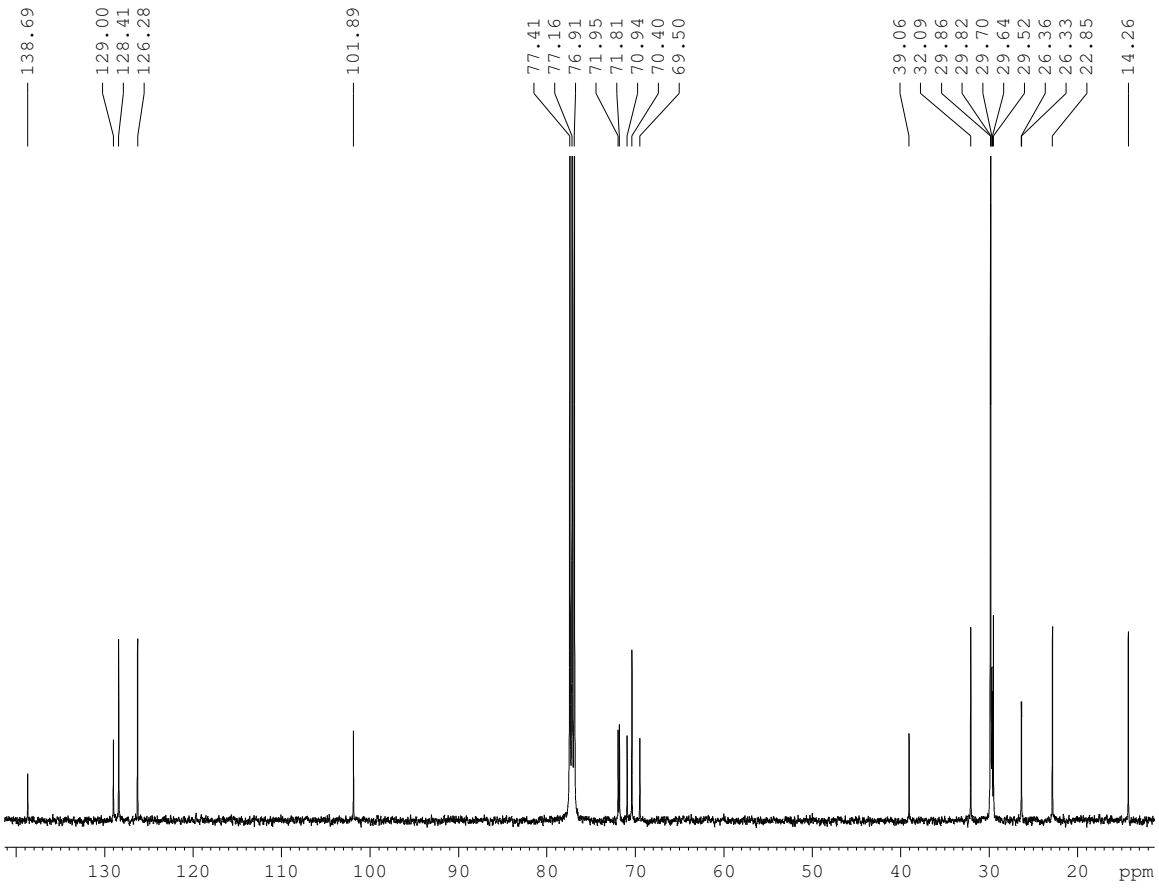




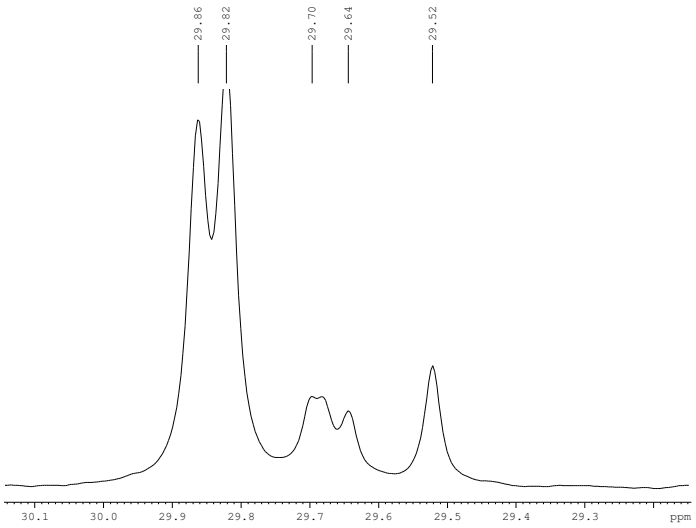
Expansion of part of the 500.1 MHz ^{1}H NMR spectrum of 2-phenyl-5,5'-bis(tetradecyloxymethyl)-1,3-dioxane (**8**) in chloroform-*d*



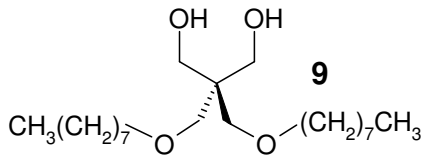
The 125.7 MHz ^{13}C NMR spectrum of 2-phenyl-5,5'-bis(tetradecyloxymethyl)-1,3-dioxane (**8**) in chloroform-*d*

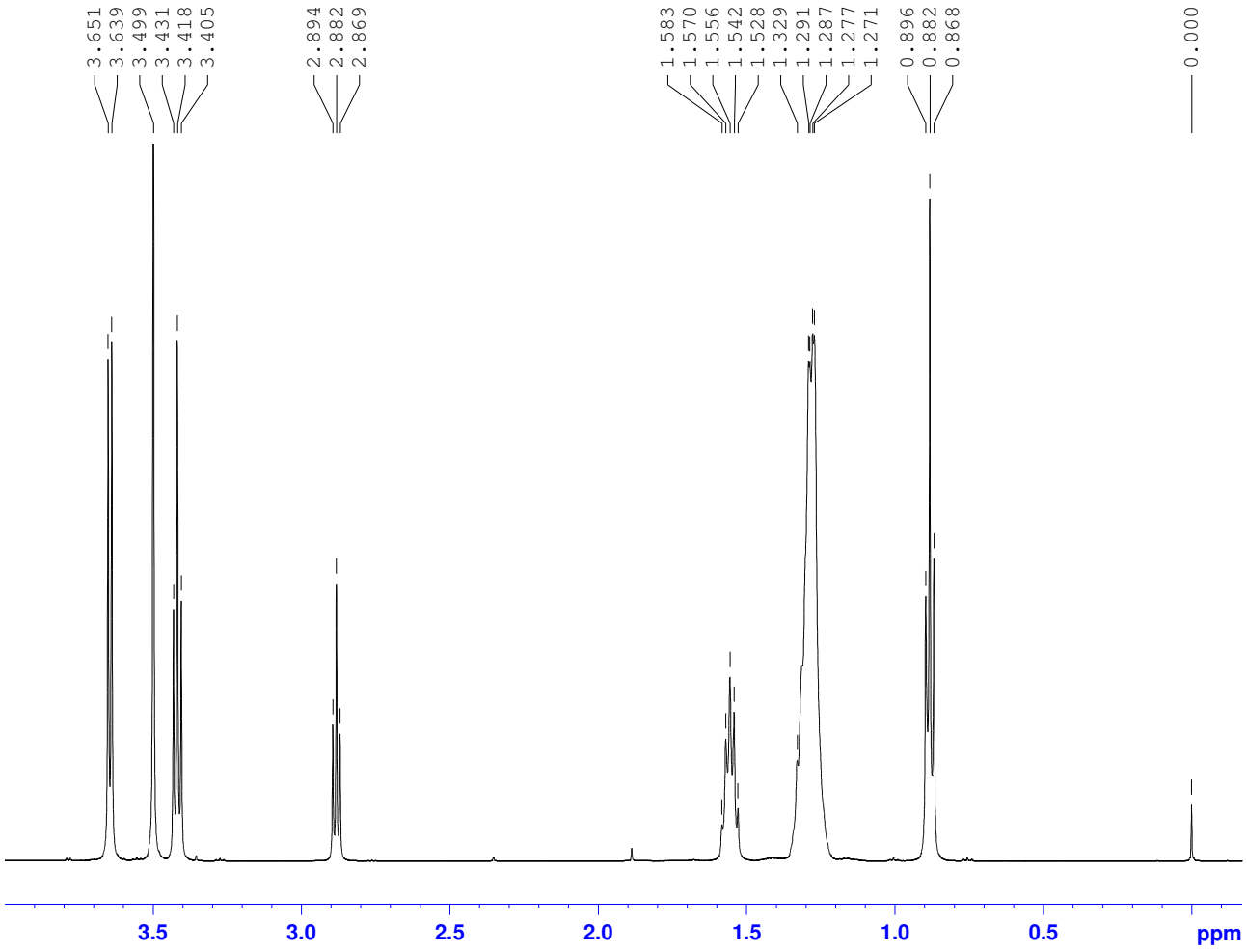


Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of 2-phenyl-5,5'-bis(tetradecyloxymethyl)-1,3-dioxane (**8**) in $CDCl_3$

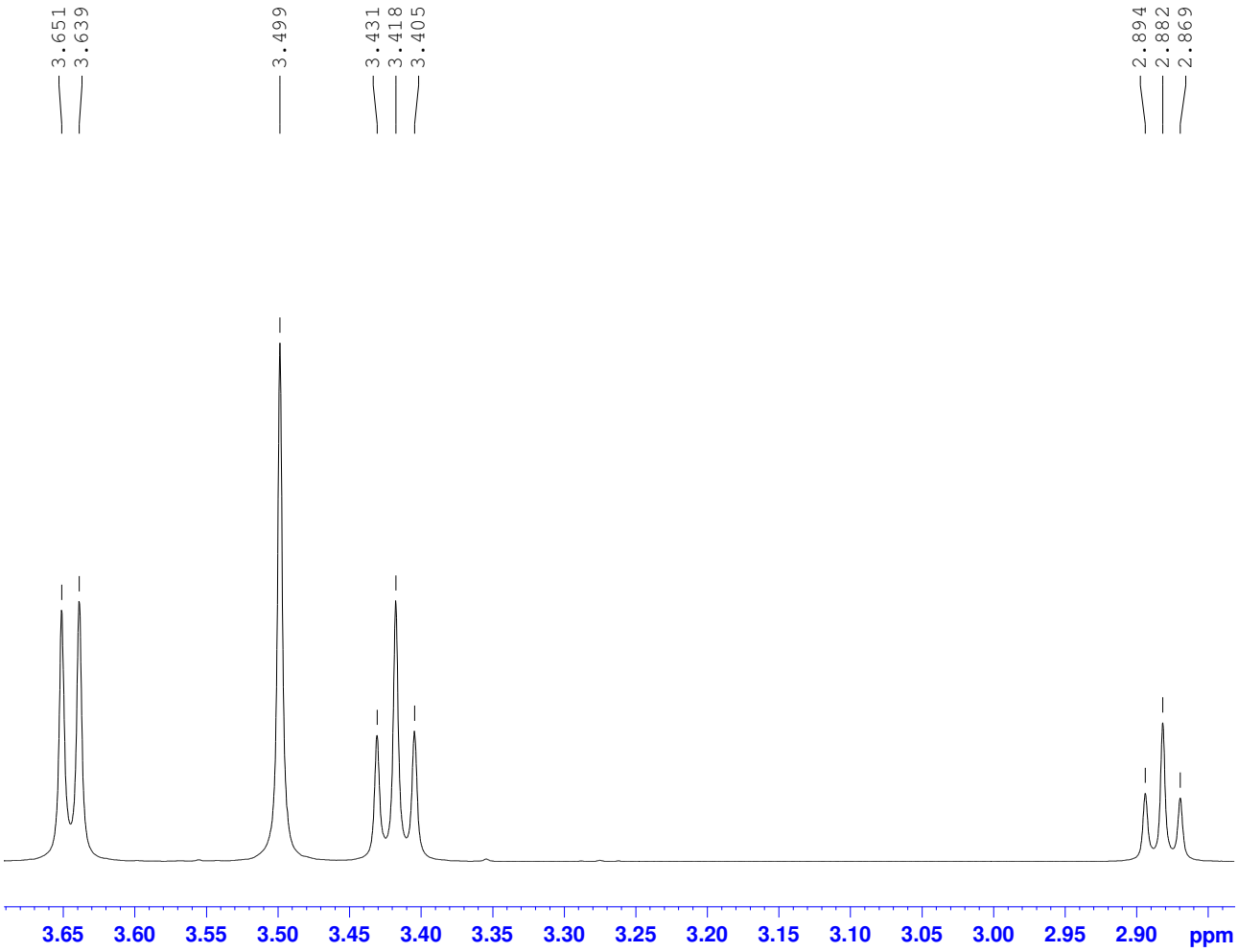


500.1 MHz ^{1}H NMR spectrum of 2,2'-bis(octyloxymethyl)-1,3-propanediol (**9**) in chloroform-*d*

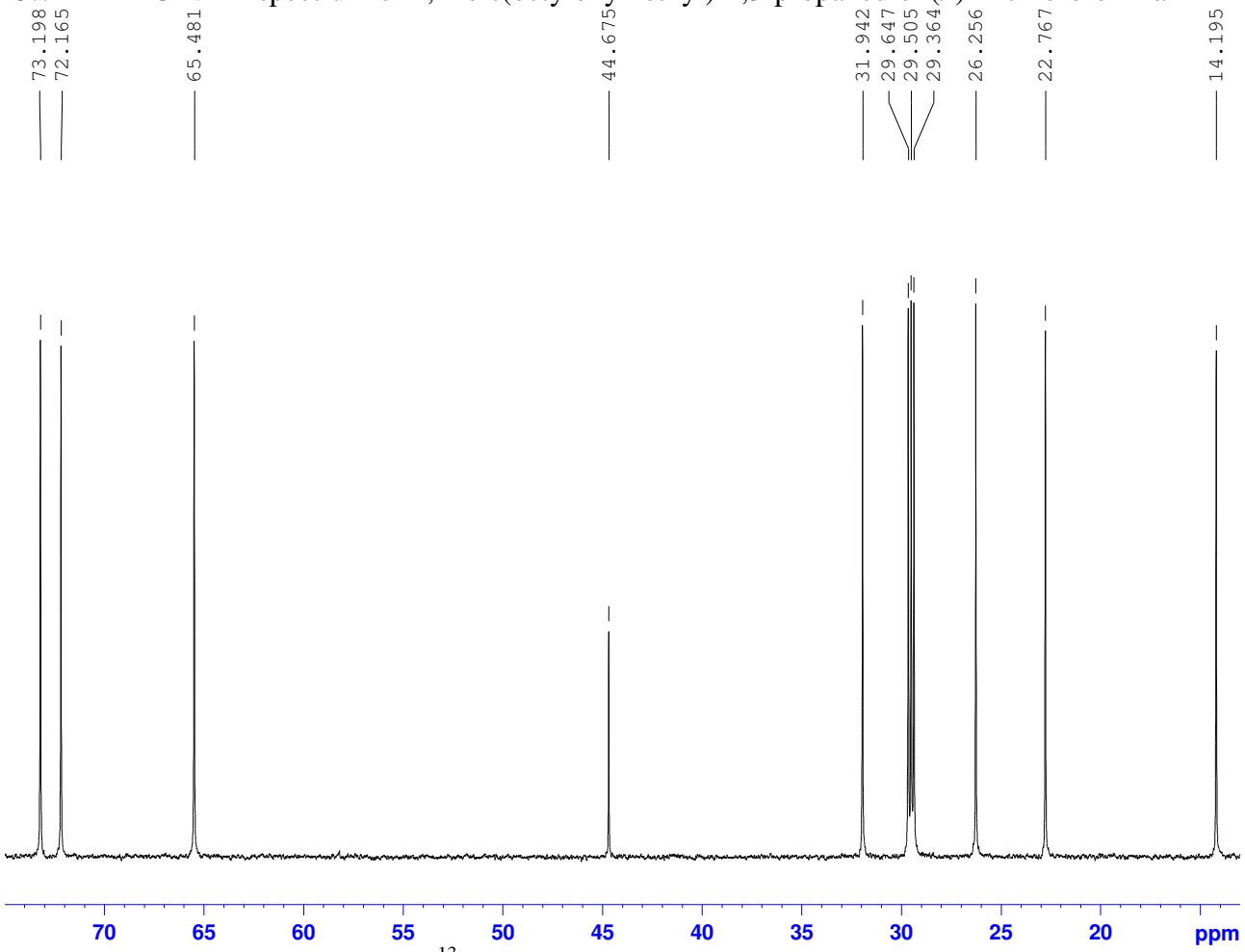




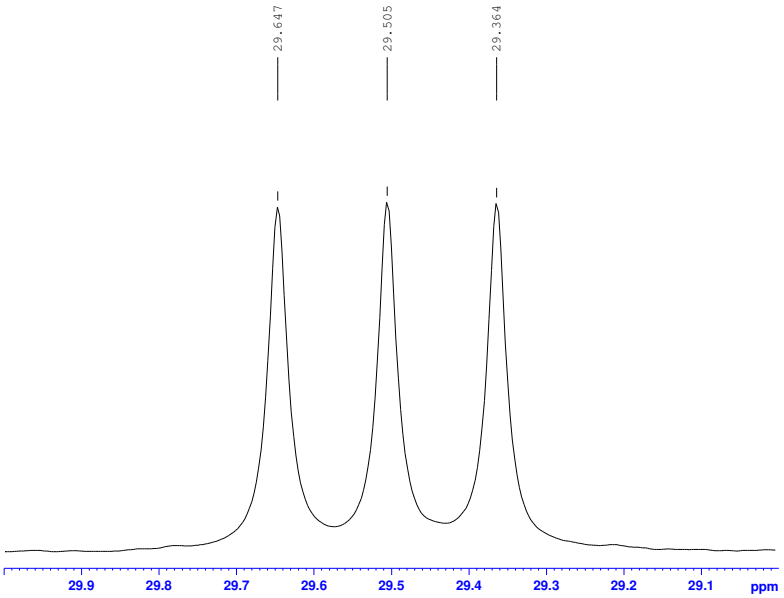
Expansion of part of the 500.1 MHz ^{1}H NMR spectrum of 2,2'-bis(octyloxymethyl)-1,3-propanediol (**9**) in $CDCl_3$



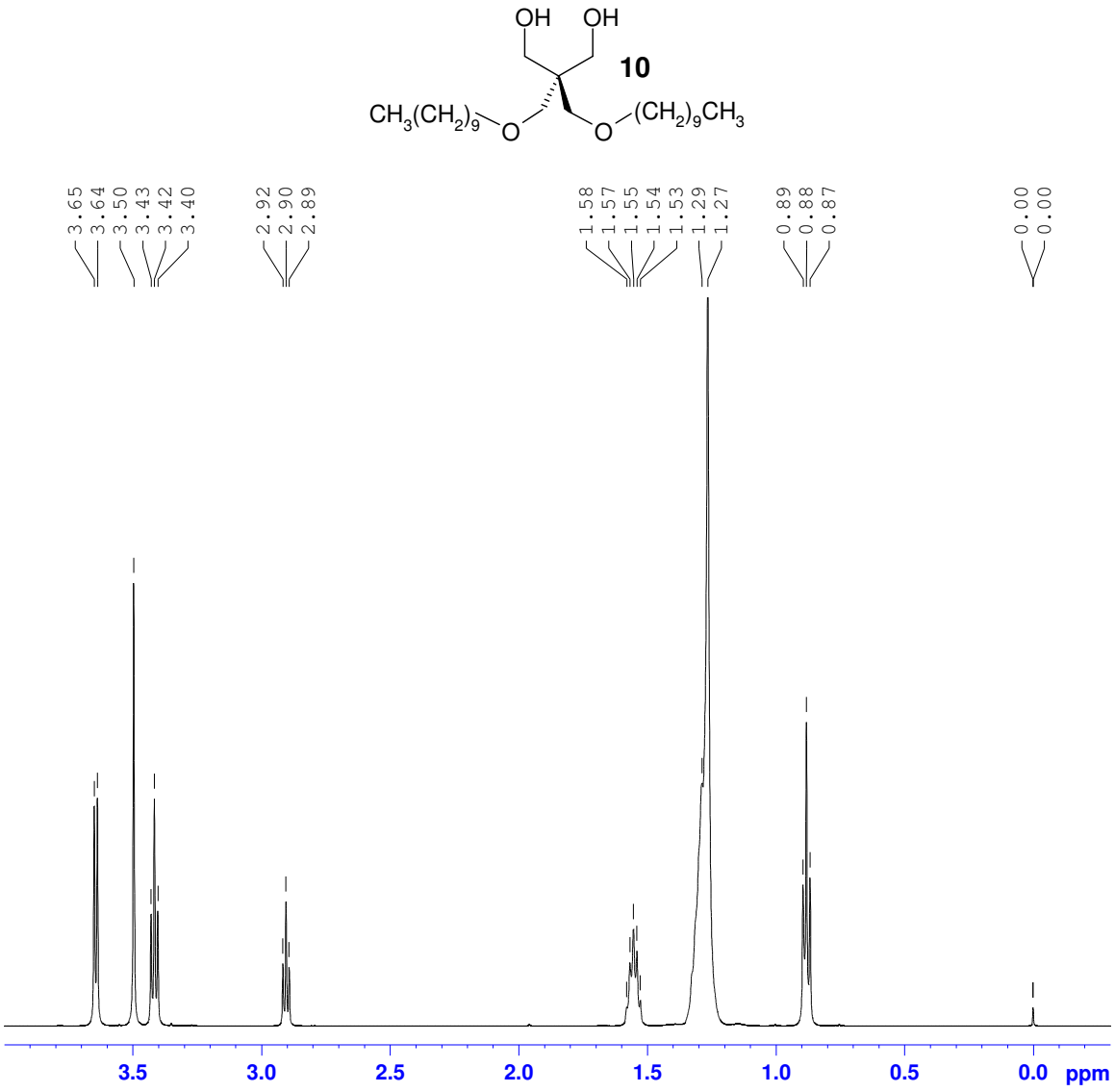
125.7 MHz ^{13}C NMR spectrum of 2,2'-bis(octyloxymethyl)-1,3-propanediol (**9**) in chloroform-*d*



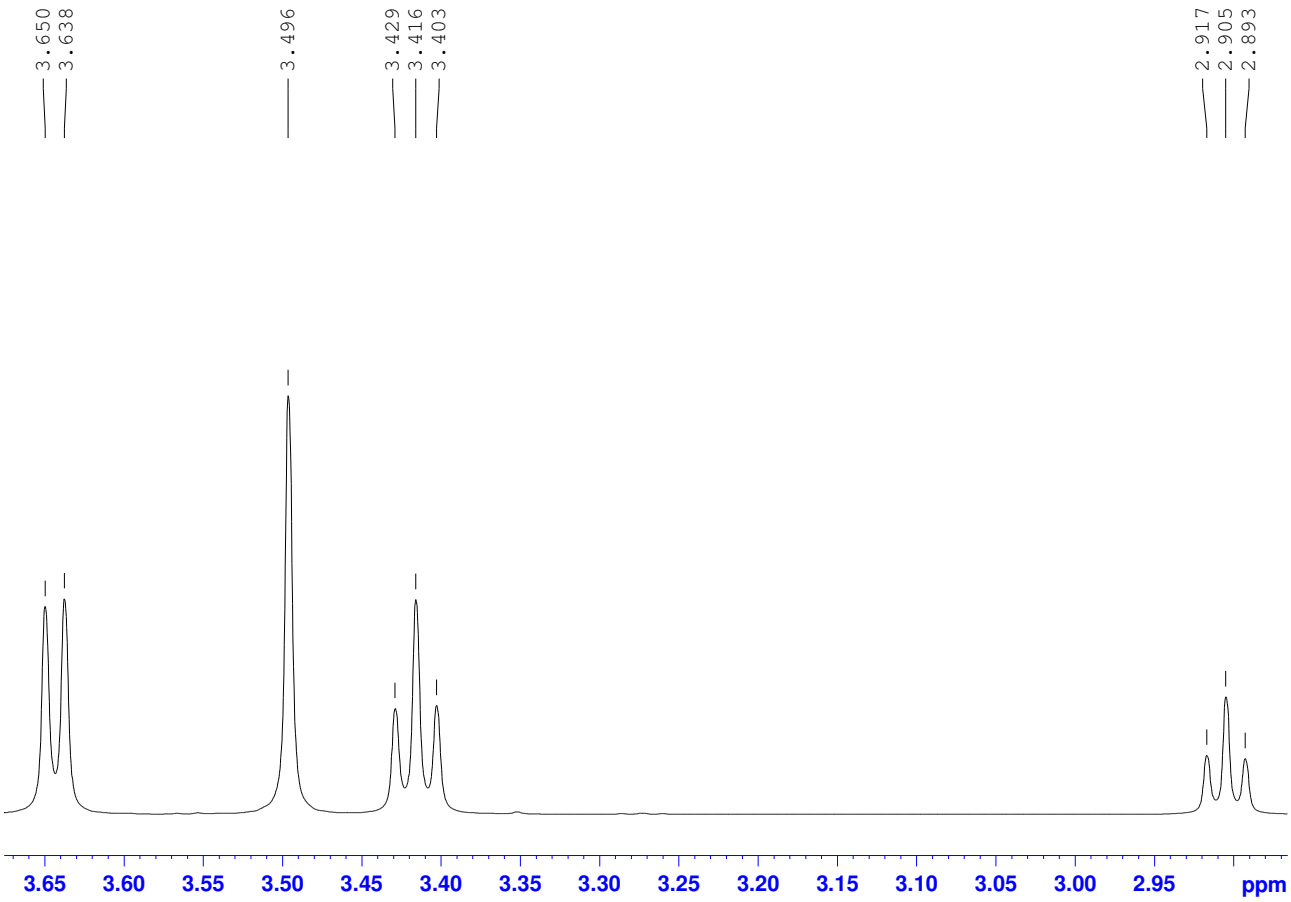
Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of 2,2'-bis(octyloxymethyl)-1,3-propanediol (**9**) in $CDCl_3$



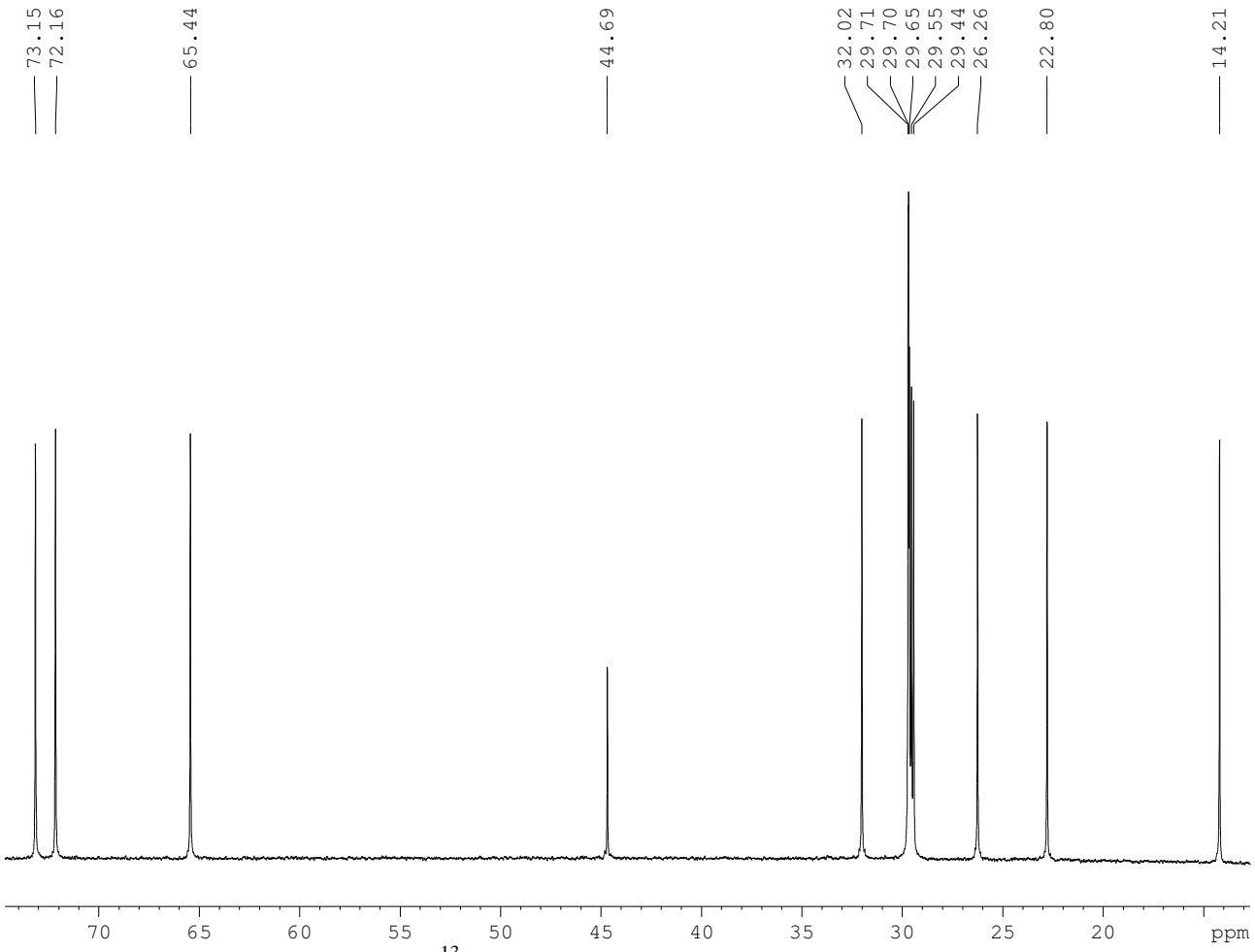
500.1 MHz ^{1}H NMR spectrum of 2,2'-bis(decyoxymethyl)-1,3-propanediol (**10**) in chloroform-*d*



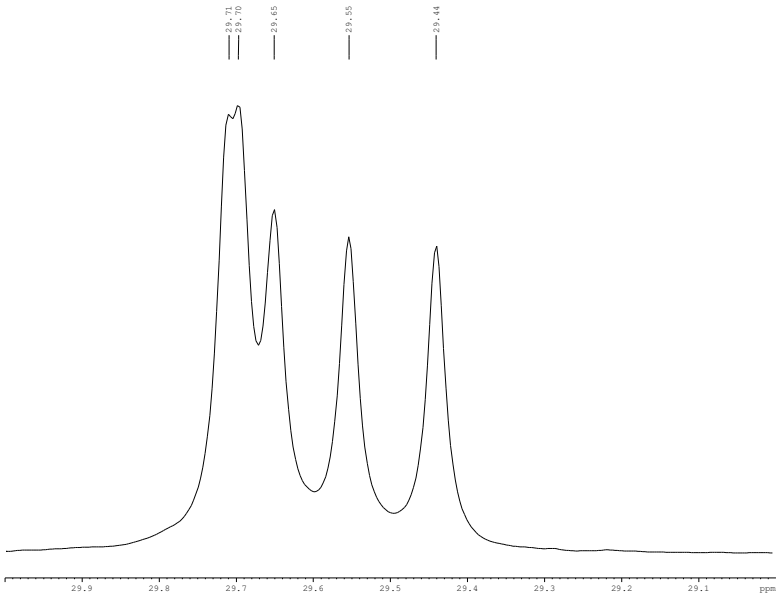
Expansion of part of the 500.1 MHz ^{1}H NMR spectrum of 2,2'-bis(decyoxymethyl)-1,3-propanediol (**10**) in $CDCl_3$



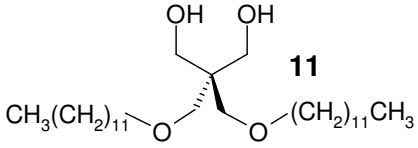
125.7 MHz ^{13}C NMR spectrum of 2,2'-bis(decyoxymethyl)-1,3-propanediol (**10**) in chloroform-*d*

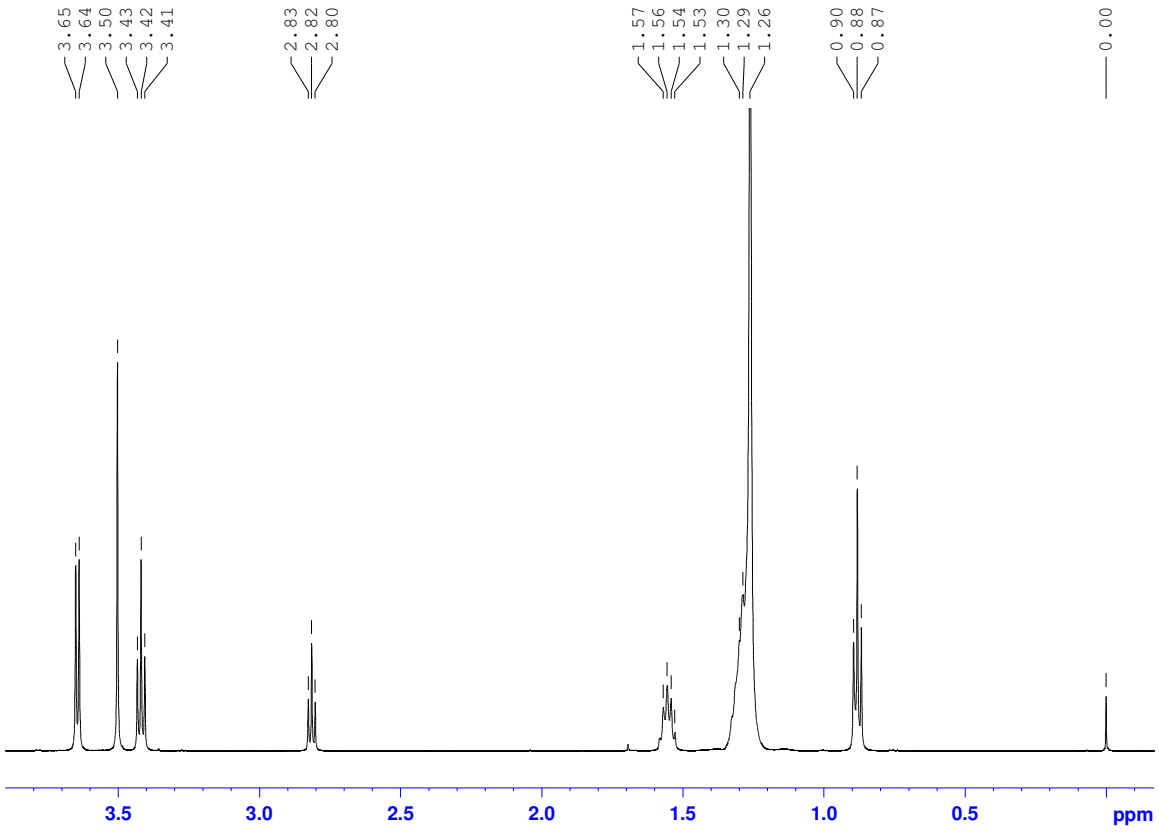


Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of 2,2'-bis(decyoxymethyl)-1,3-propanediol (**10**) in chloroform-*d*

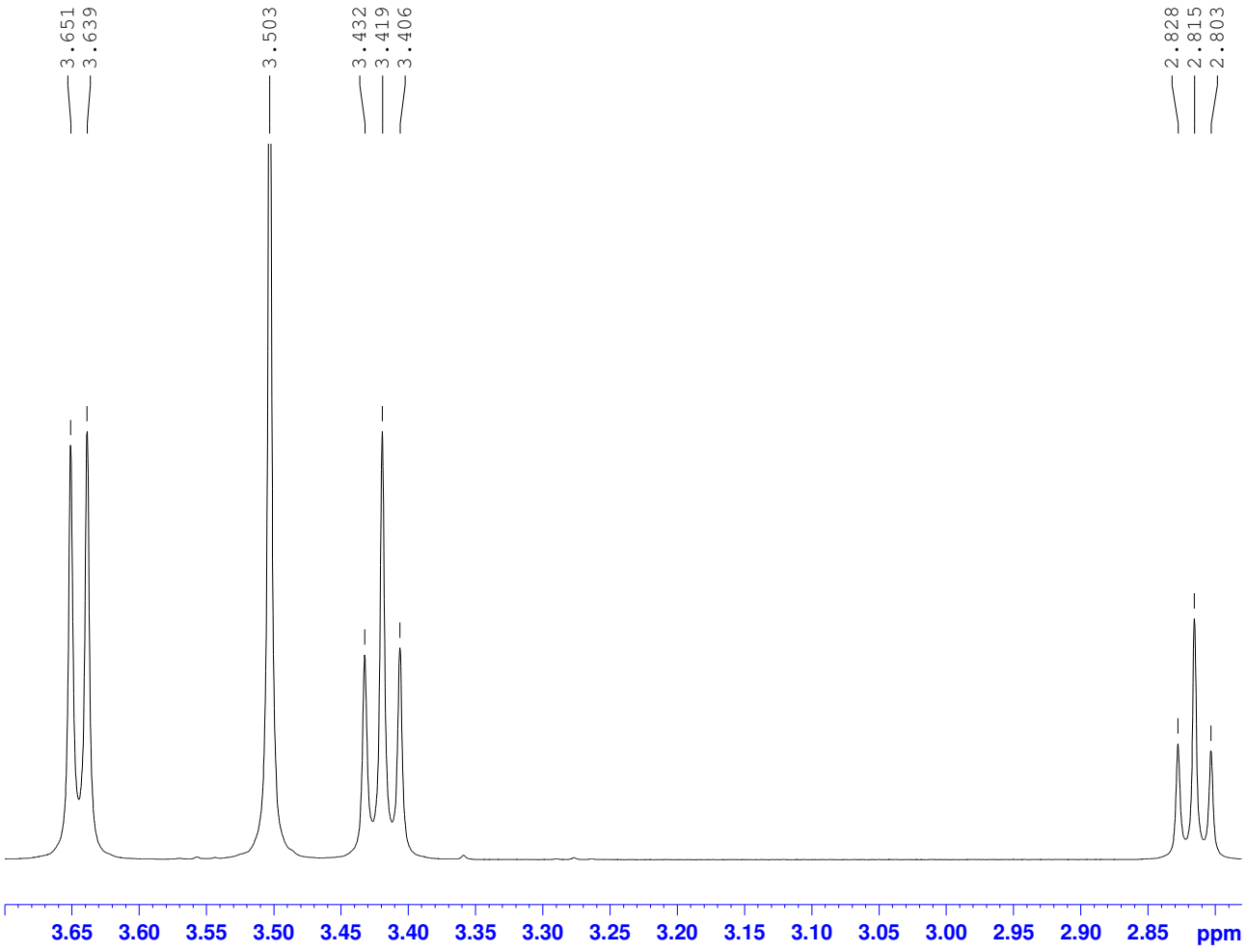


500.1 MHz ^{1}H NMR spectrum of 2,2'-bis(dodecyoxymethyl)-1,3-propanediol (**11**) in chloroform-*d*

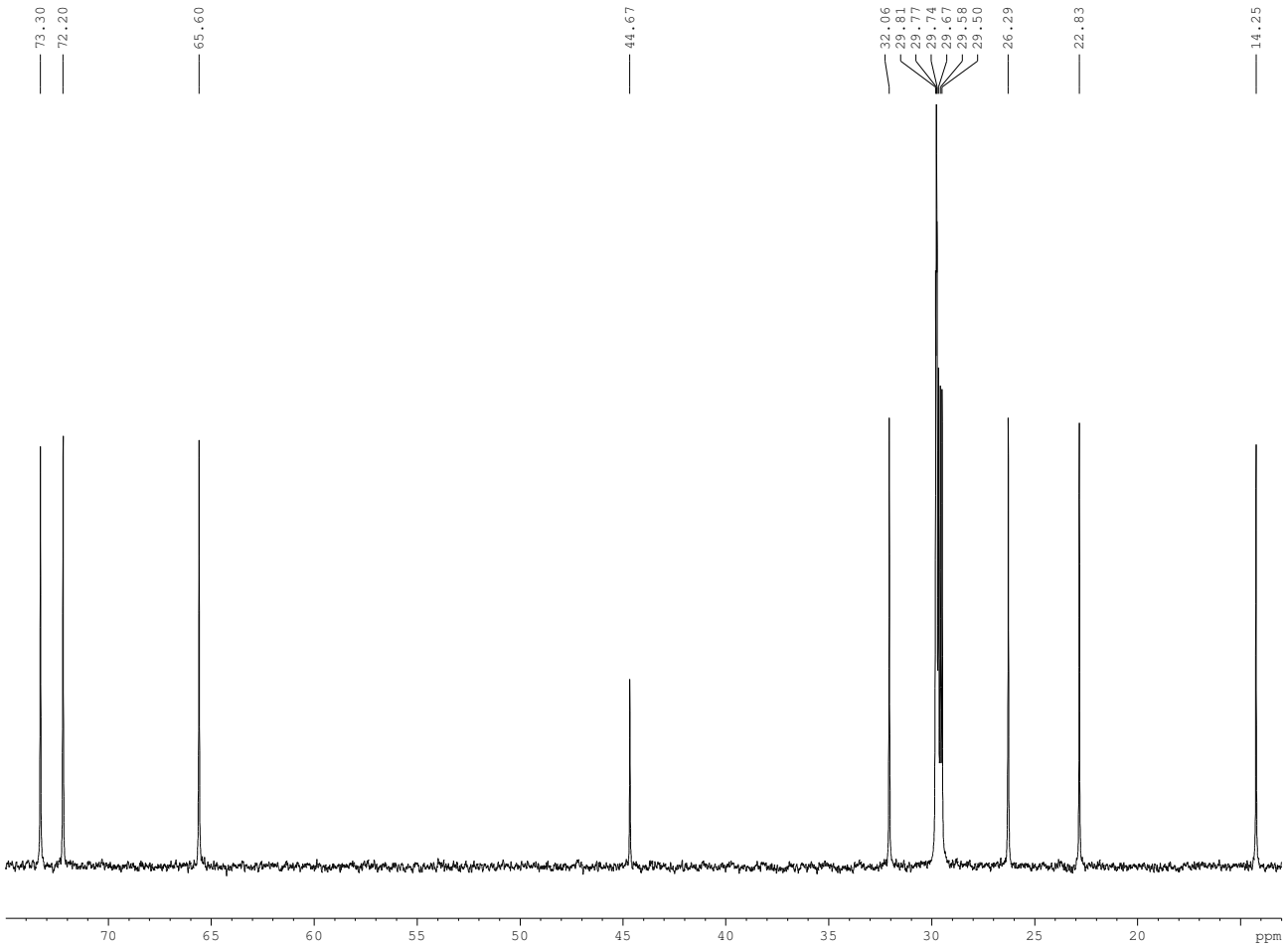




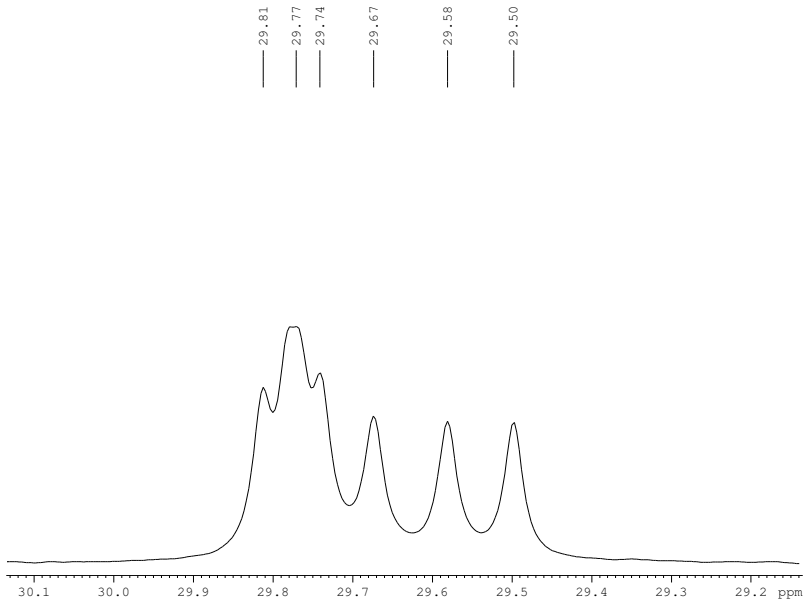
Expansion of part of the 500.1 MHz ^{1}H NMR spectrum of 2,2'-bis(dodecyoxymethyl)-1,3-propanediol (**11**) in $CDCl_3$



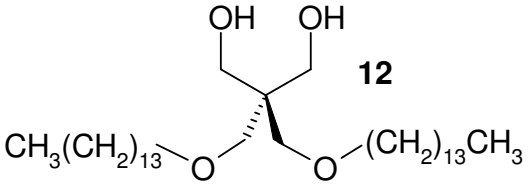
125.7 MHz ^{13}C NMR spectrum of 2,2'-bis(dodecyoxymethyl)-1,3-propanediol (**11**) in chloroform-*d*

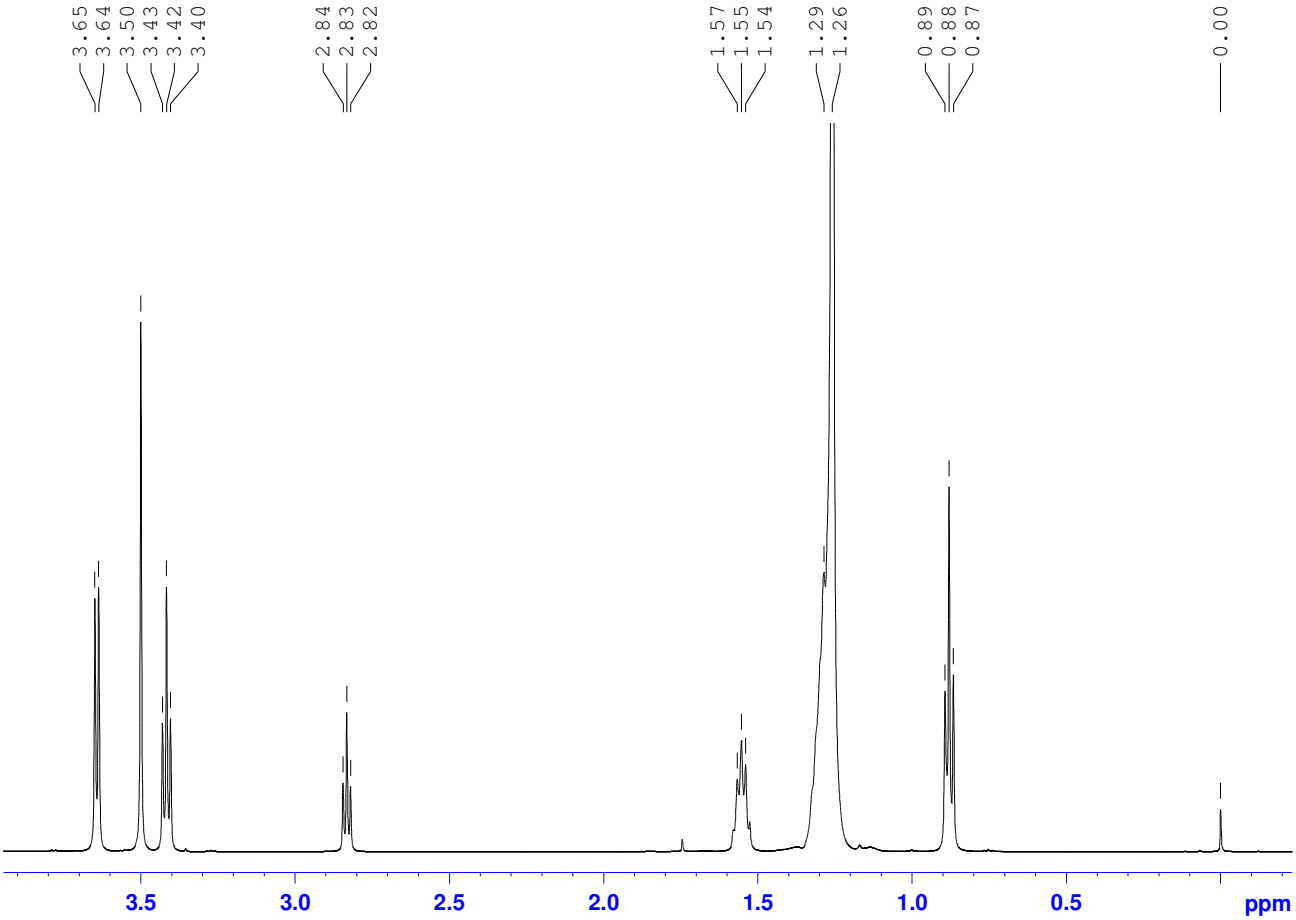


Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of 2,2'-bis(dodecyoxymethyl)-1,3-propanediol (**11**) in chloroform-*d*

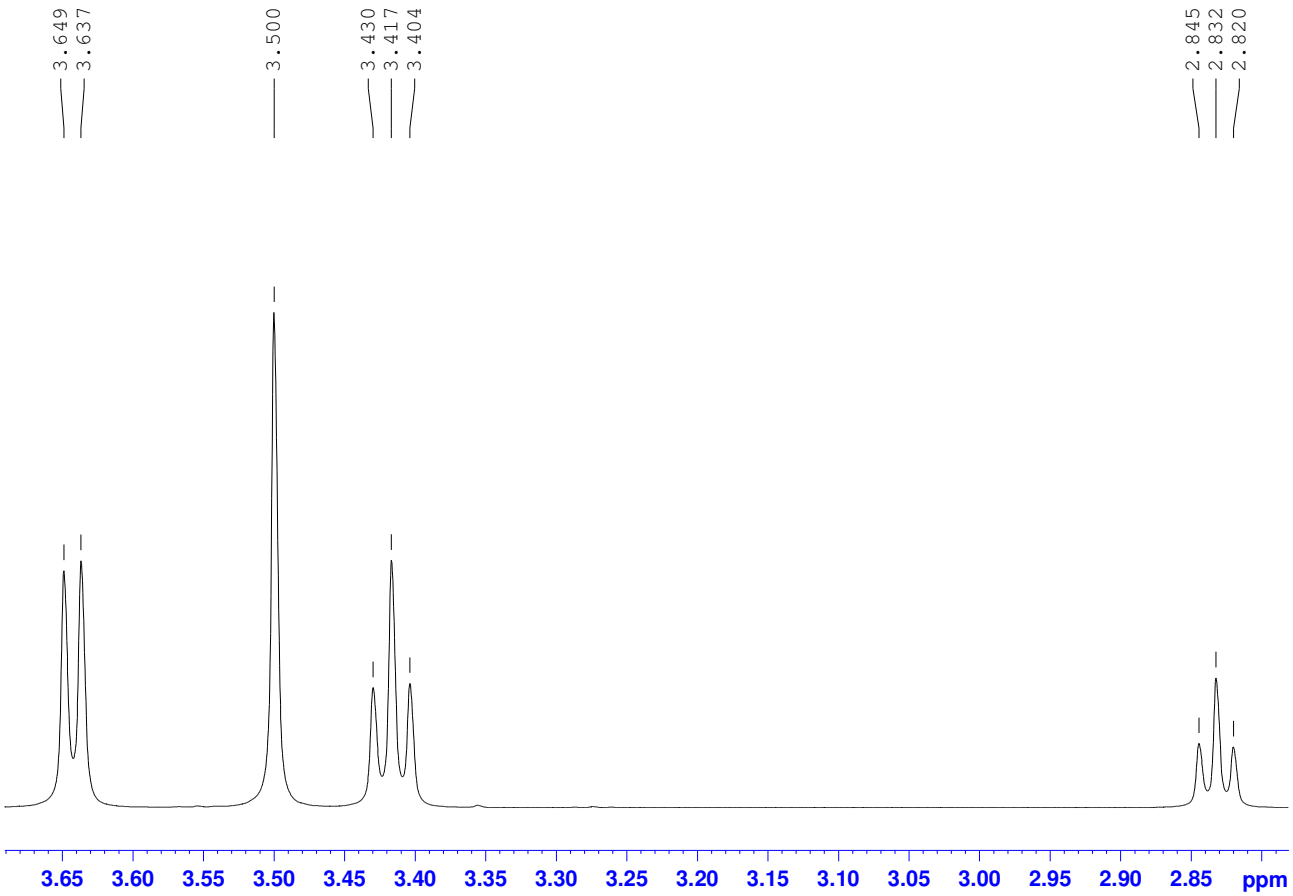


500.1 MHz ^{1}H NMR spectrum of 2,2'-bis(tetradecyloxymethyl)-1,3-propanediol(**12**) in chloroform-*d*

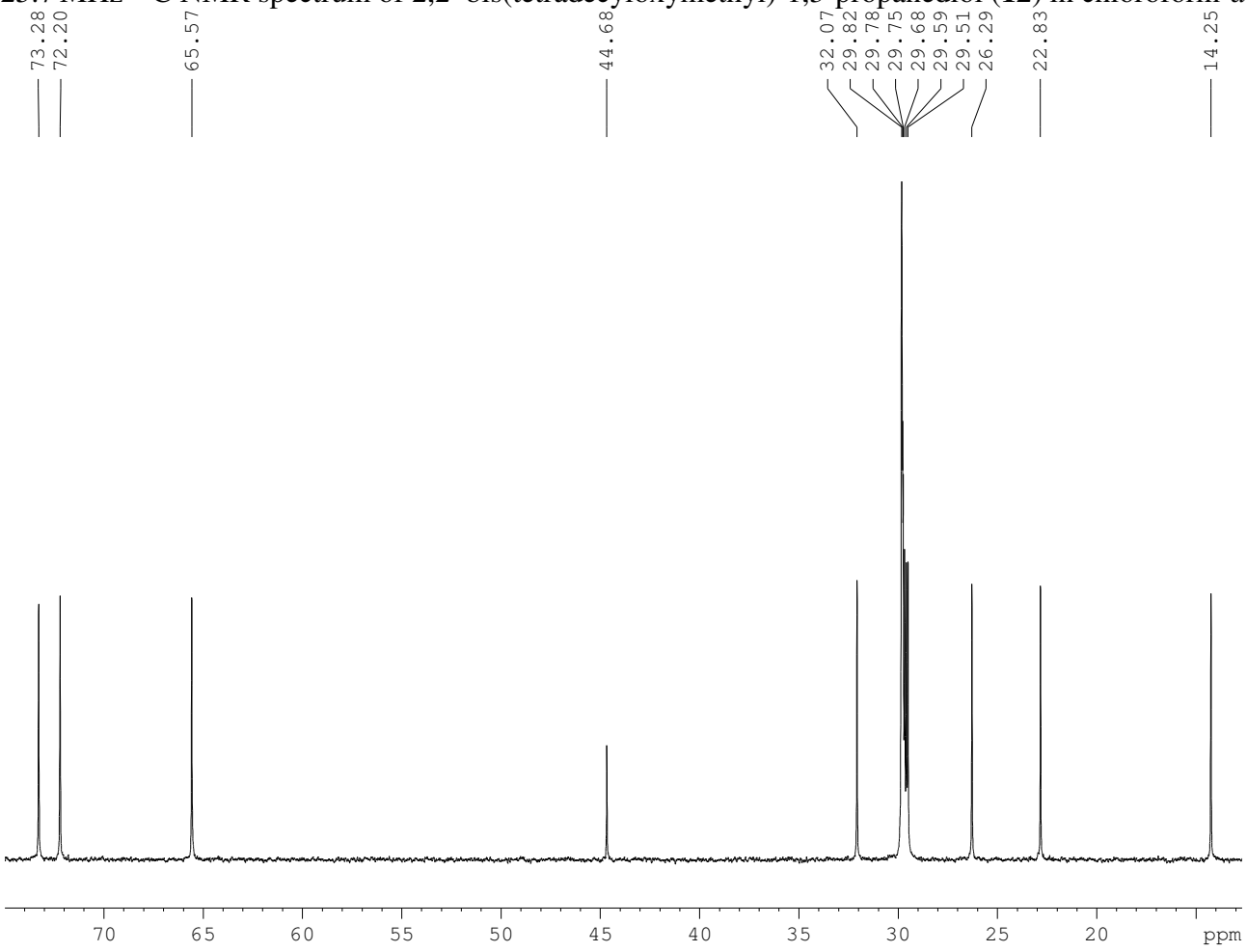




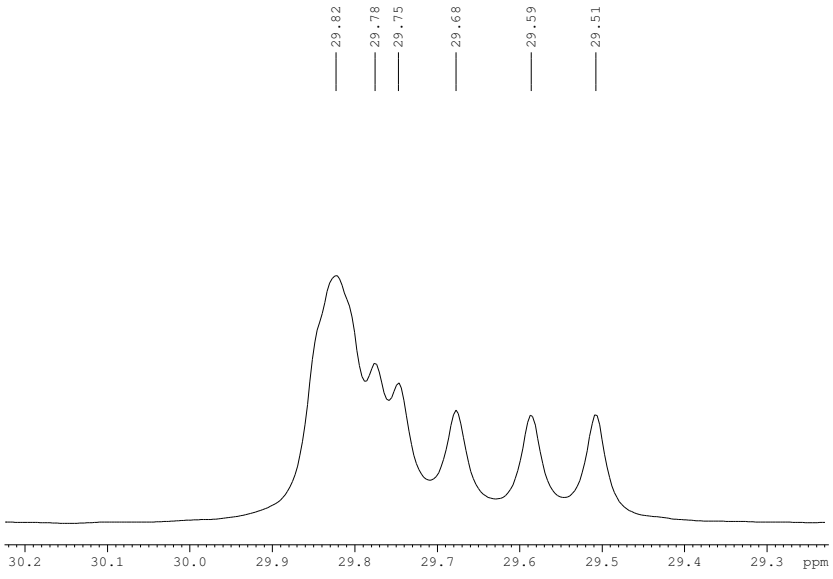
Expansion of part of the 500.1 MHz ^{1}H NMR spectrum of 2,2'-bis(dodecyoxymethyl)-1,3-propanediol (**12**) in $CDCl_3$



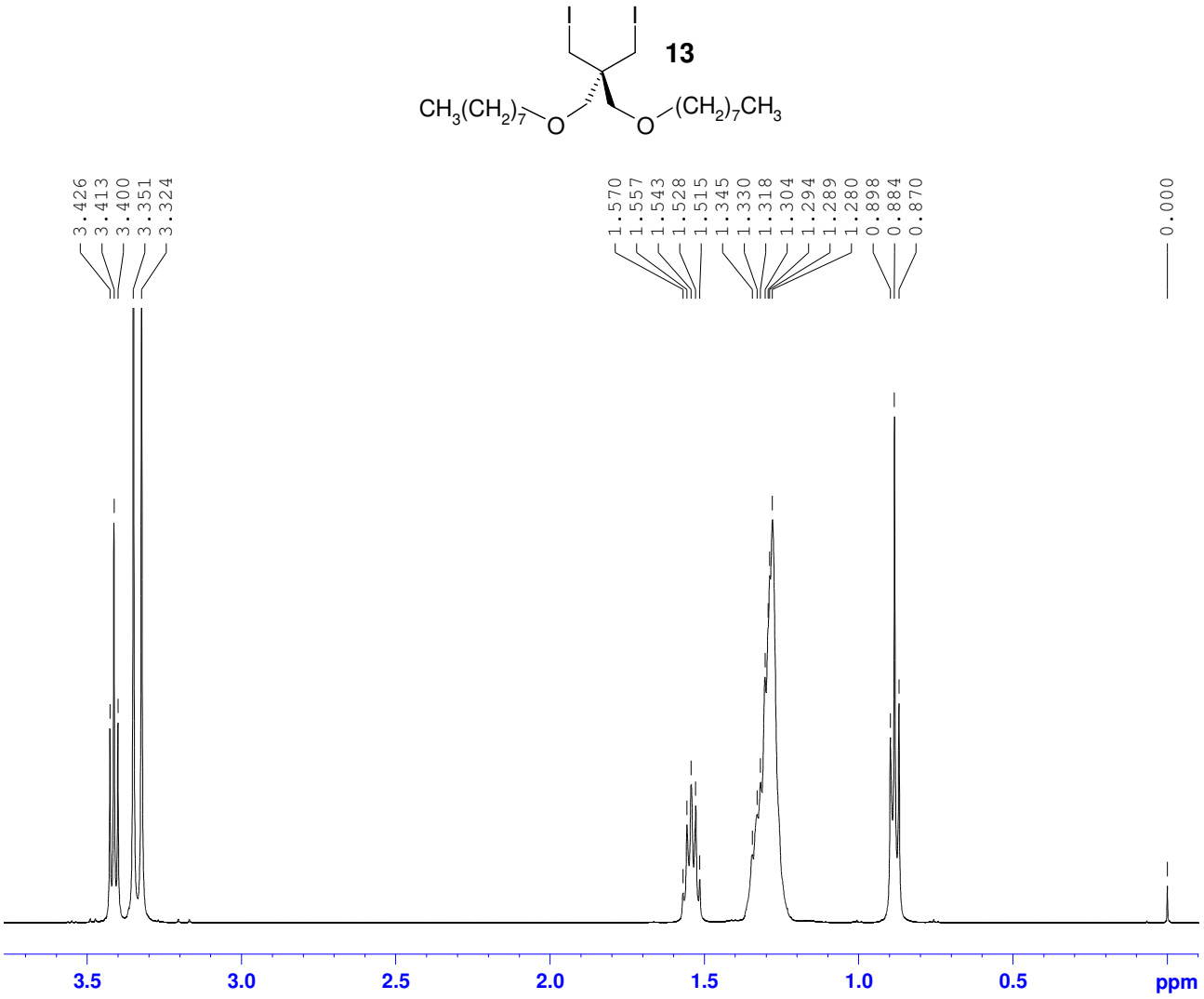
125.7 MHz ^{13}C NMR spectrum of 2,2'-bis(tetradecyloxymethyl)-1,3-propanediol (**12**) in chloroform-*d*



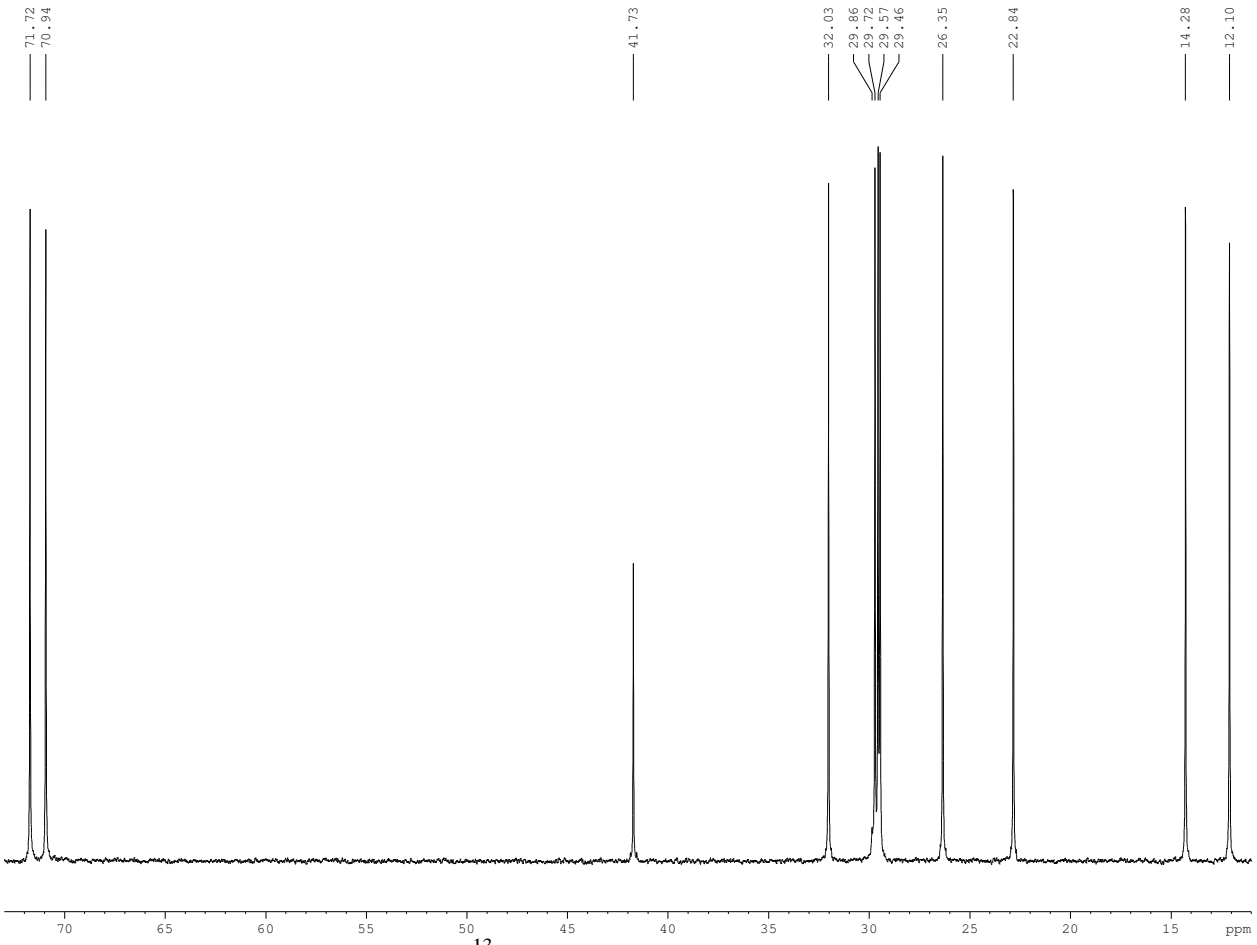
Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of 2,2'-bis(tetradecyloxymethyl)-1,3-propanediol (**12**) in chloroform-*d*



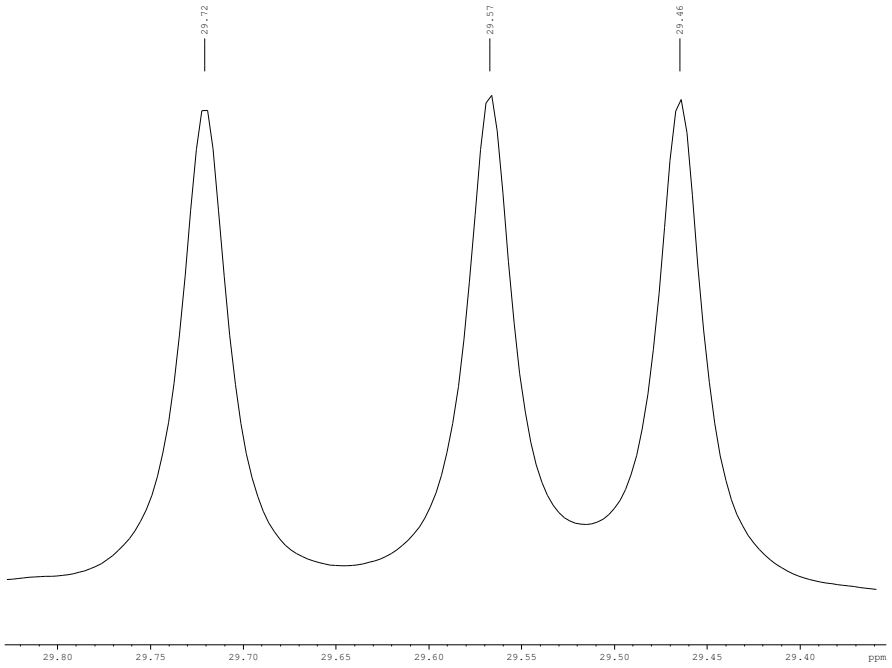
500.1 MHz ^{1}H NMR spectrum of 1,3-diiodo-2,2'-bis(octyloxymethyl)propane (**13**) in chloroform-*d*



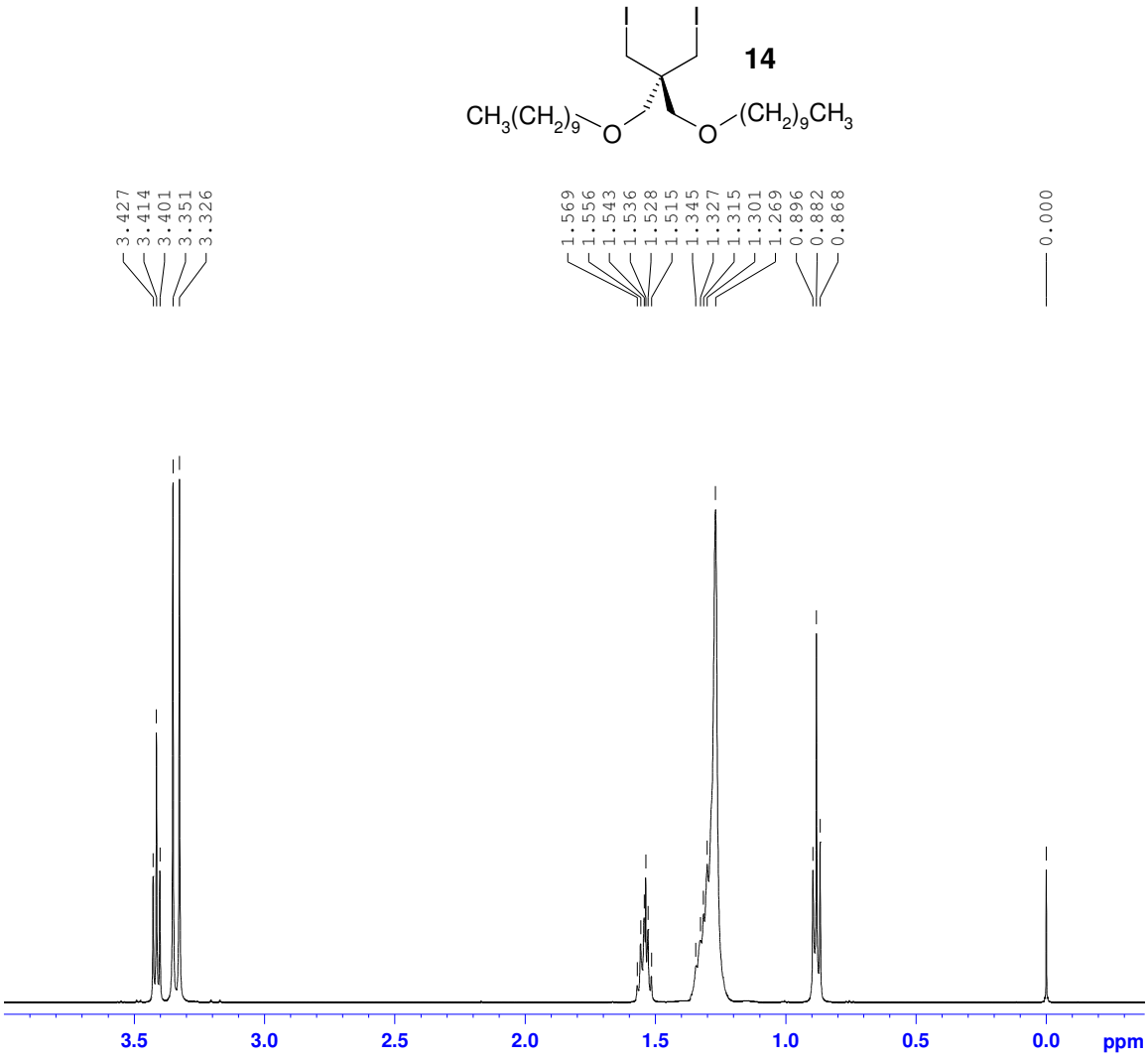
125.7 MHz ^{13}C NMR spectrum of 1,3-diiodo-2,2'-bis(octyloxymethyl)propane (**13**) in chloroform-*d*



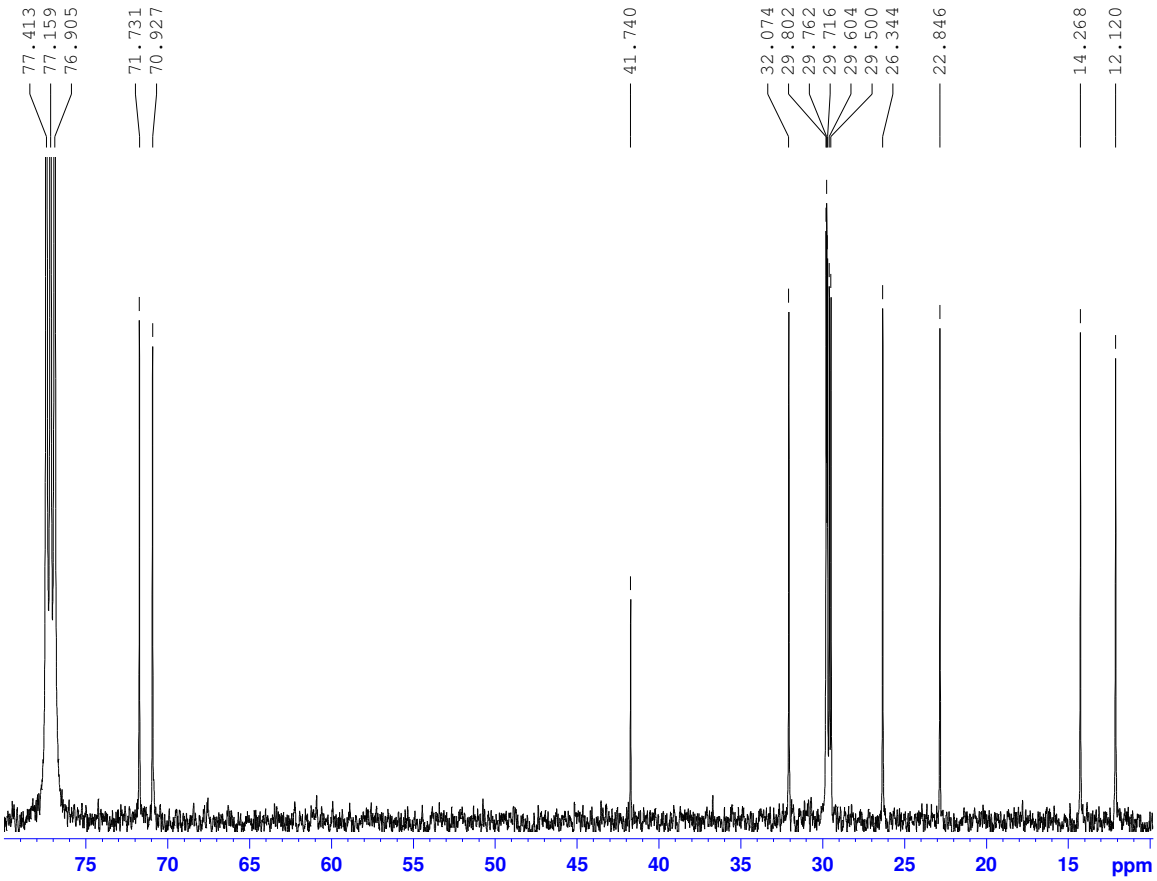
Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of 1,3-diiodo-2,2'-bis(octyloxymethyl)propane (**13**) in $CDCl_3$



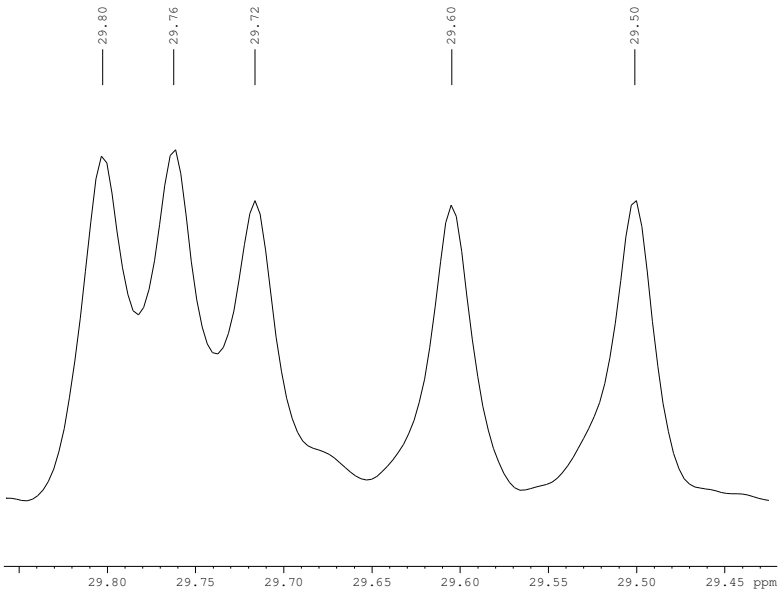
500.1 MHz ^{1}H NMR spectrum of 2,2'-bis(decyloxymethyl)-1,3-diiodopropane (**14**) in chloroform-*d*



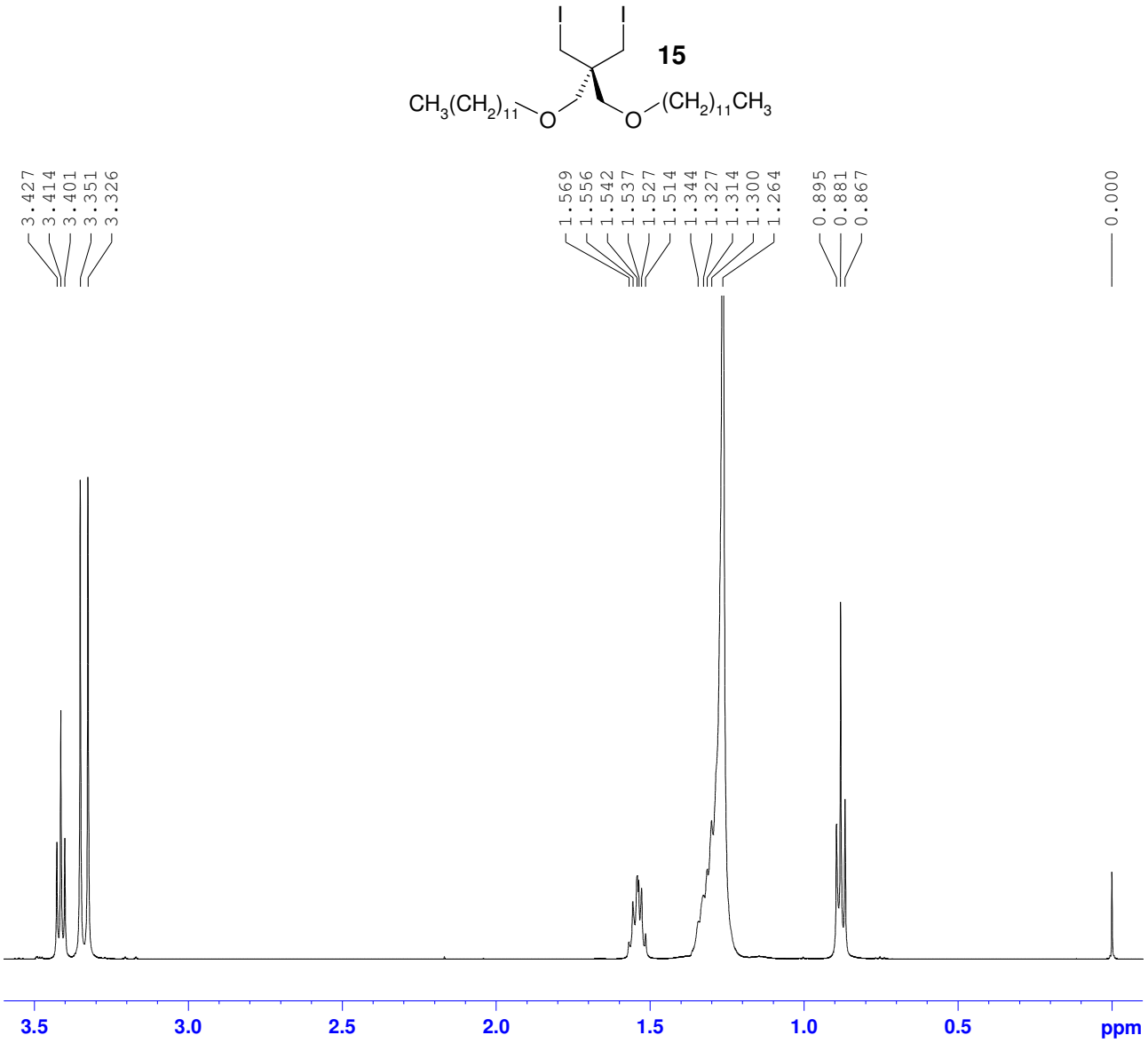
125.7 MHz ^{13}C NMR spectrum of 2,2'-bis(decyloxymethyl)-1,3-diiodopropane (**14**) in chloroform-*d*



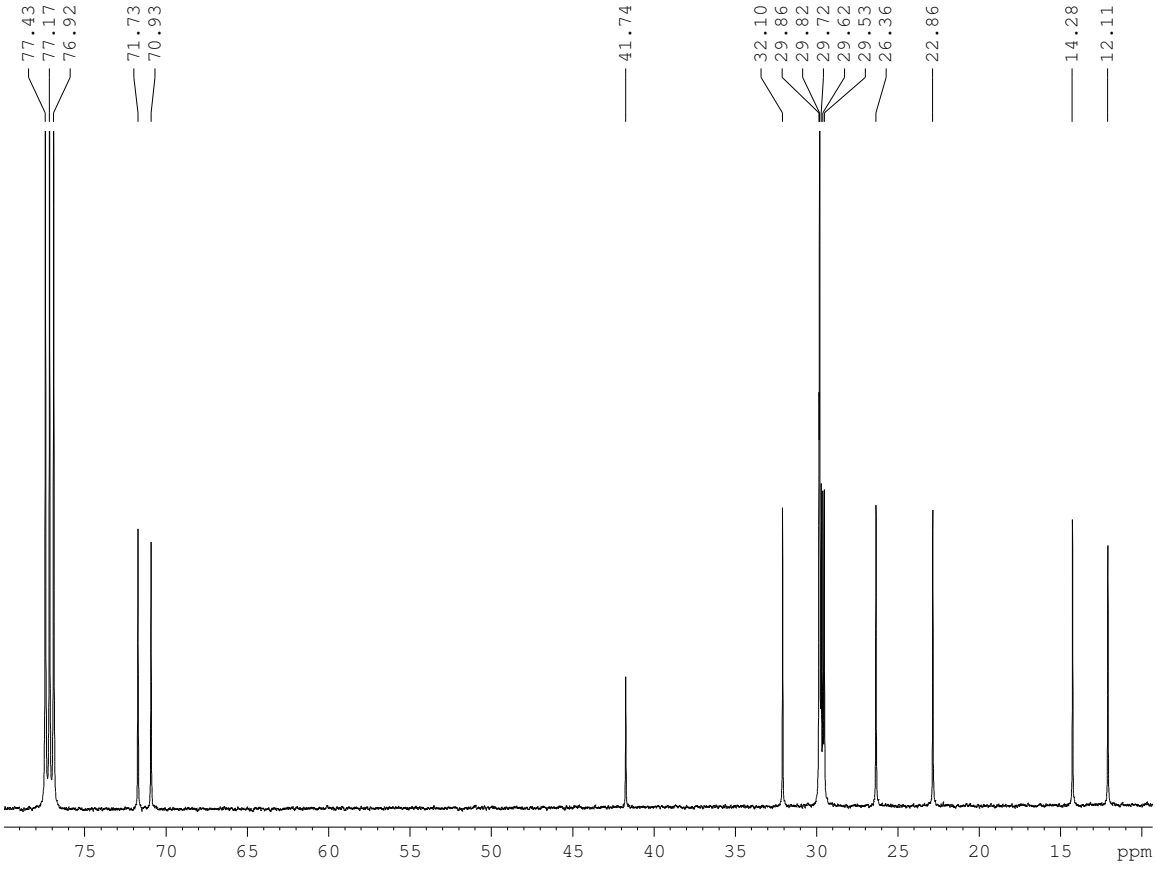
Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of 2,2'-bis(decyloxymethyl)-1,3-diiodopropane (**14**) in $CDCl_3$



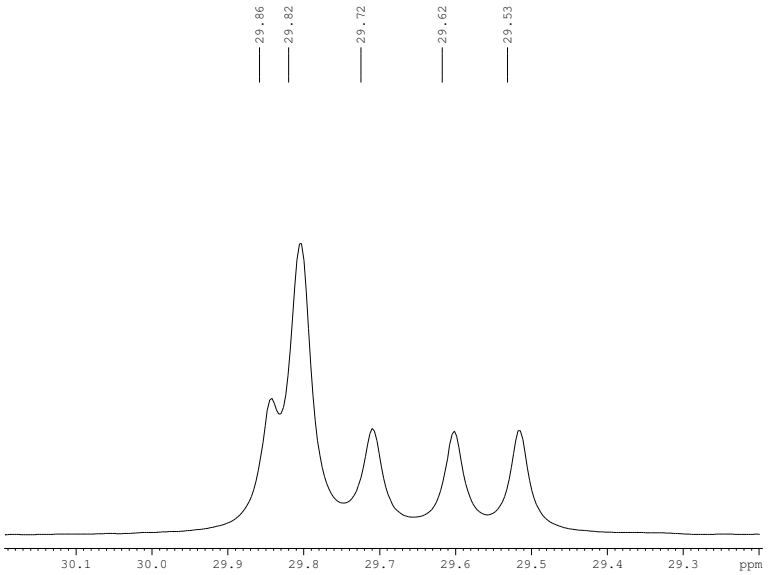
500.1 MHz ^{1}H NMR spectrum of 2,2'-bis(dodecyloxymethyl)-1,3-diiodopropane (**15**) in chloroform-*d*



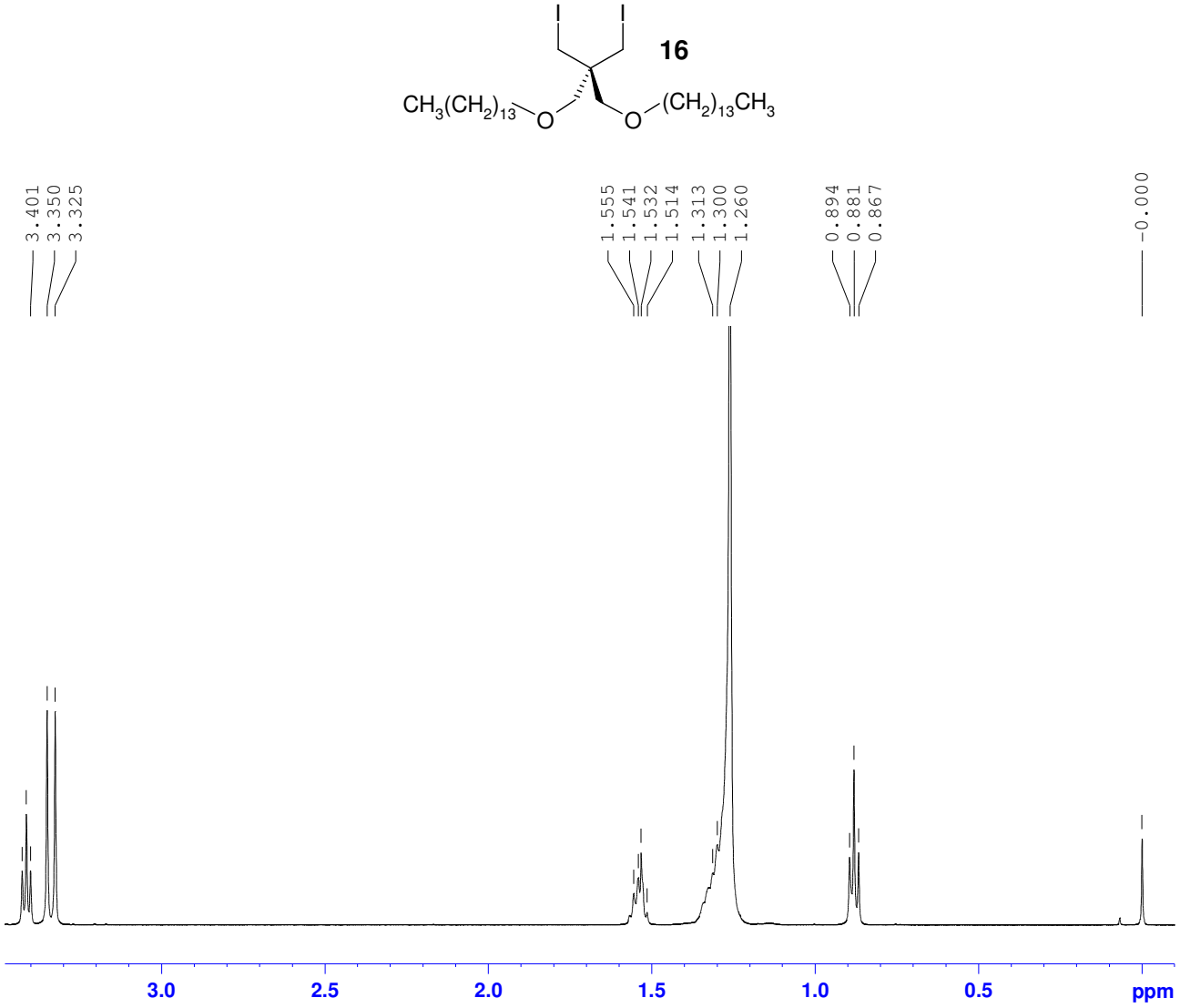
125.7 MHz ^{13}C NMR spectrum of 2,2'-bis(dodecyloxymethyl)-1,3-diiodopropane (**15**) in chloroform-*d*



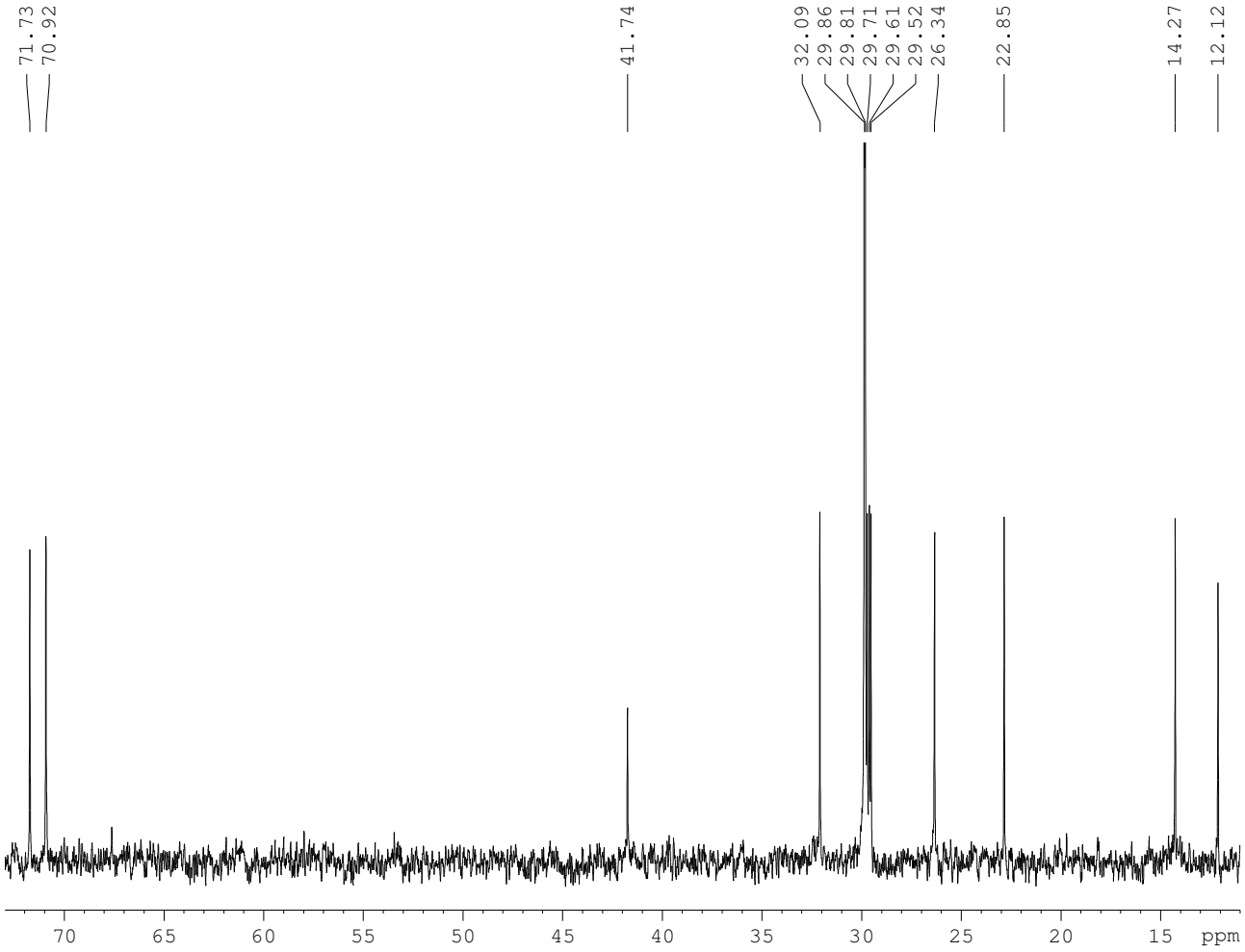
Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of 2,2'-bis(dodecyloxymethyl)-1,3-diiodopropane (**15**) in $CDCl_3$



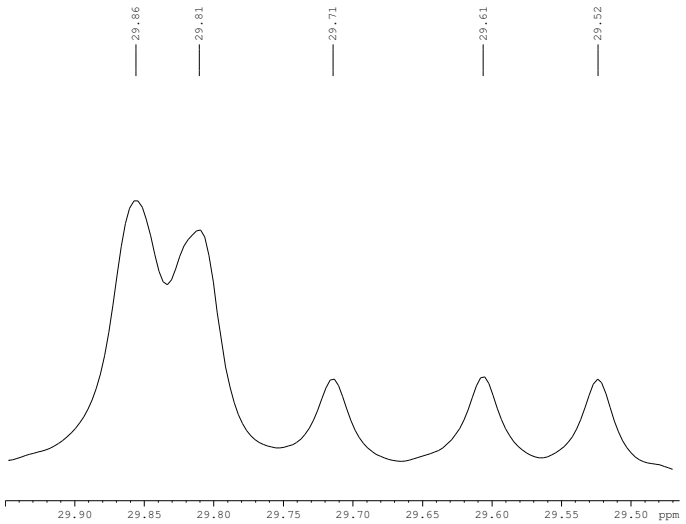
500.1 MHz ^{1}H NMR spectrum of 1,3-diiodo-2,2'-bis(tetradecyloxymethyl)propane (**16**) in chloroform-*d*



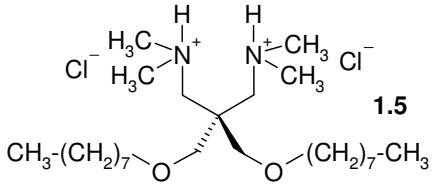
125.75 MHz ^{13}C NMR spectrum of 1,3-diiodo-2,2'-bis(tetradecyloxymethyl)propane (**16**) in chloroform-*d*



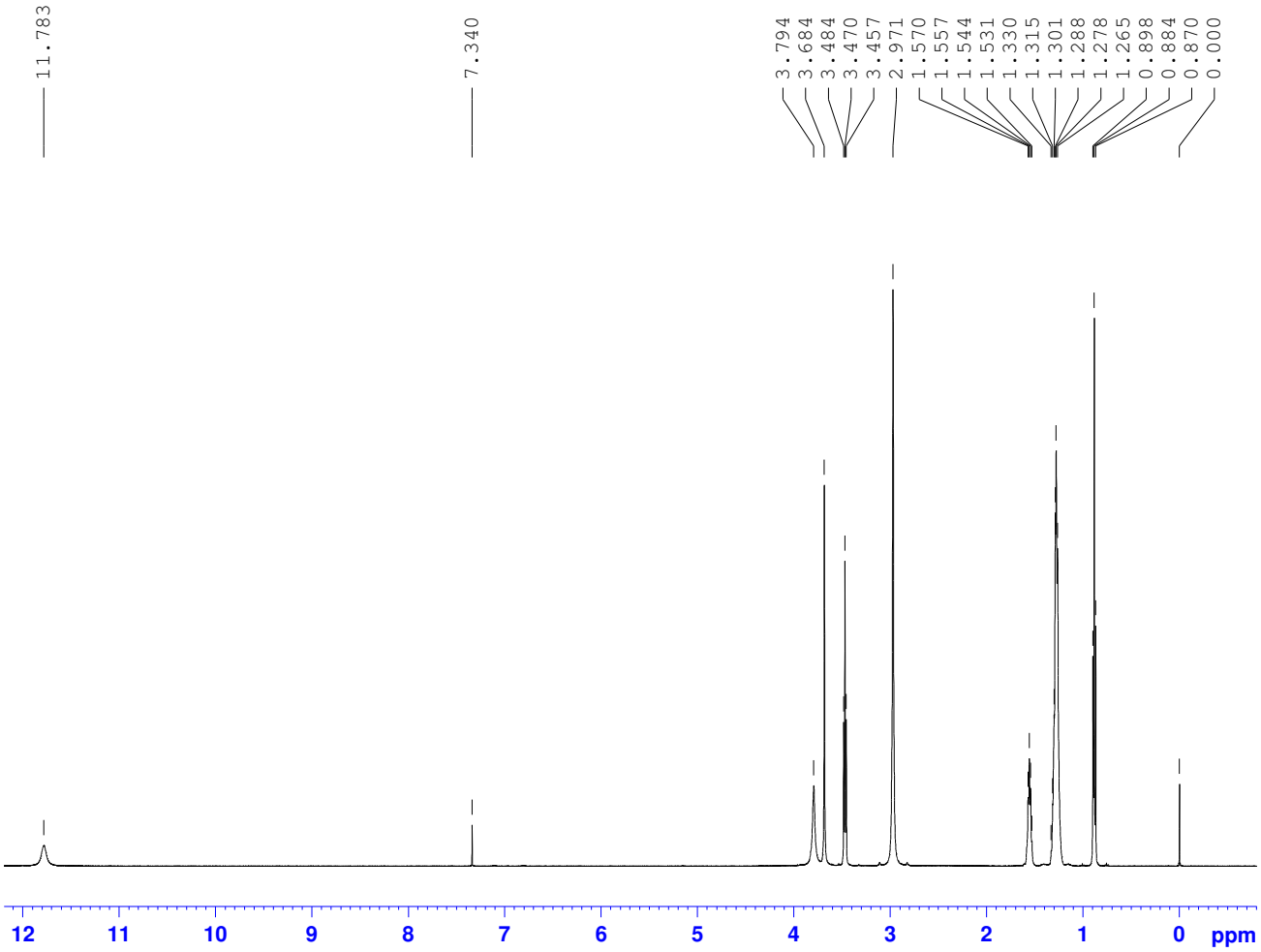
Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of 1,3-diiodo-2,2'-bis(tetradecyloxymethyl)propane (**16**) in $CDCl_3$



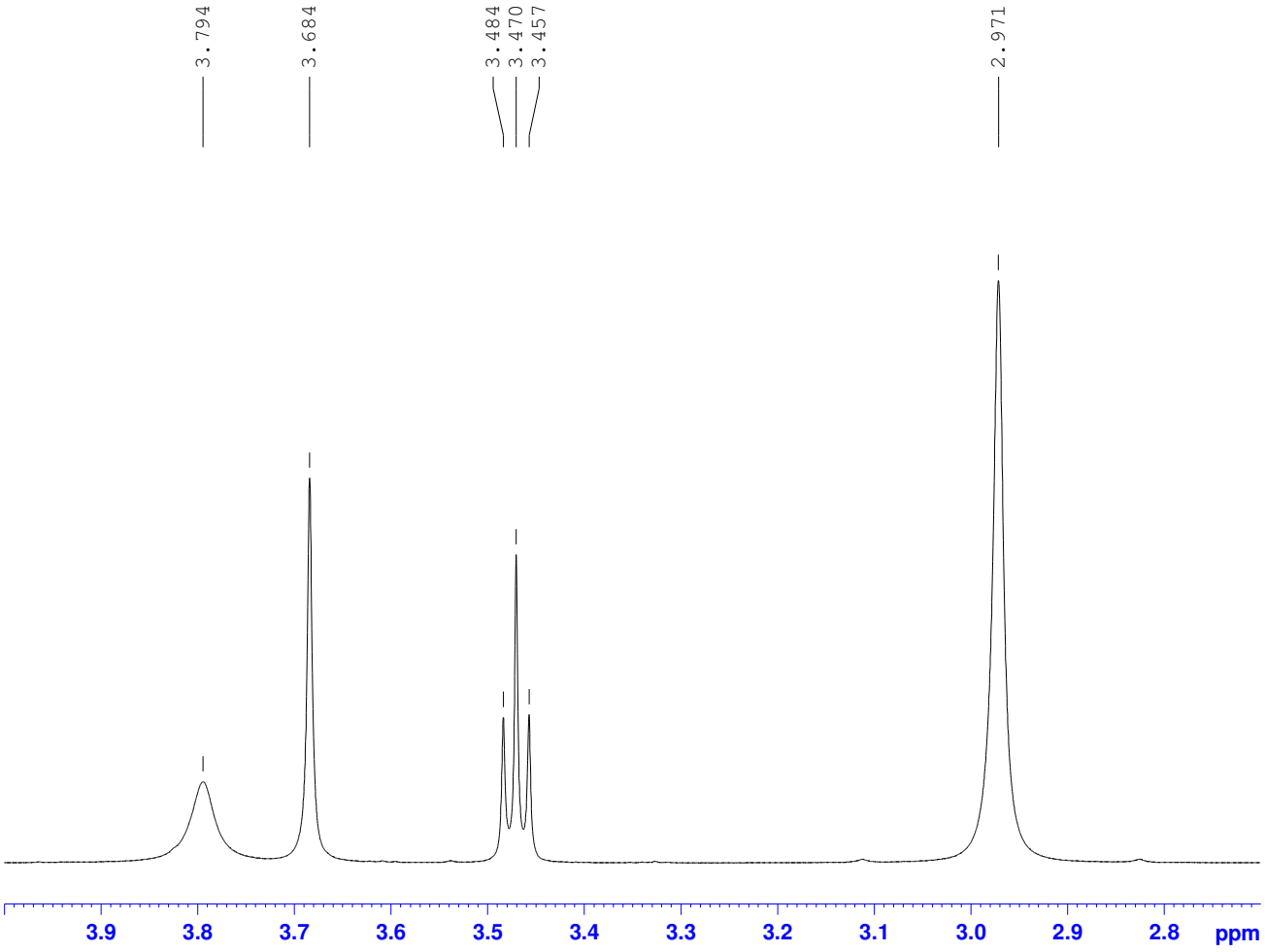
500.1 MHz ^{1}H NMR spectrum of *N*,*N*,*N*',*N*'-tetramethyl-2,2-bis(octyloxymethyl)-1,3-propanediaminium dichloride (**1.5**) in chloroform-*d*



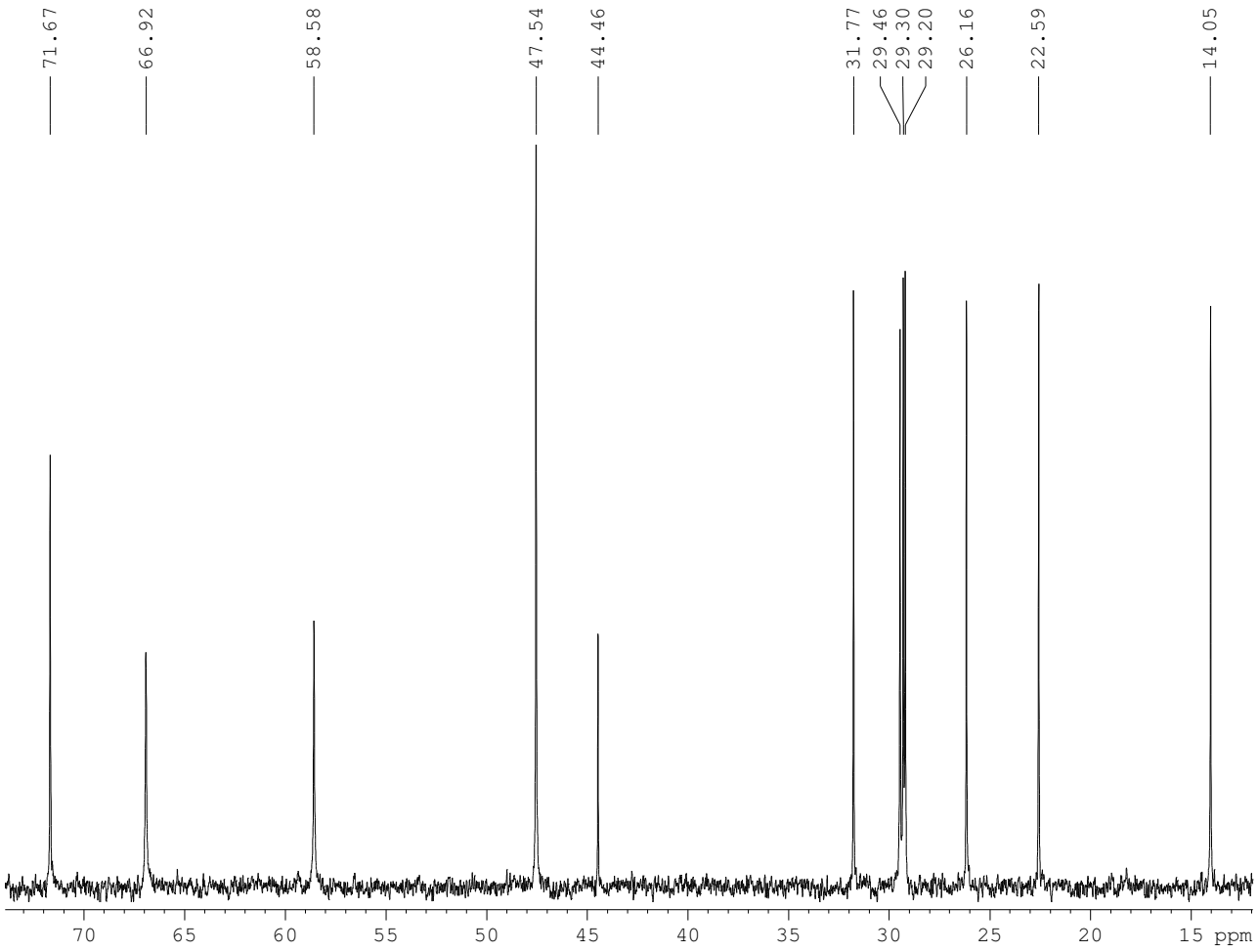
1.5



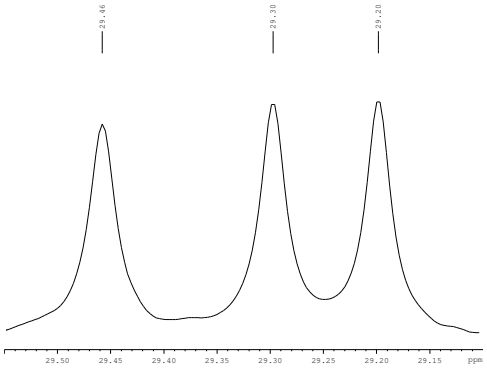
Expansion of part of the 500.1 MHz ^{1}H NMR spectrum of *N*,*N*,*N*',*N*'-tetramethyl-2,2-bis(octyloxymethyl)-1,3-propanediaminium dichloride(**1.5**) in $CDCl_3$



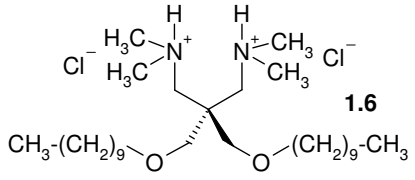
125.7 MHz ^{13}C NMR spectrum of *N*,*N*,*N*',*N*'-tetramethyl-2,2-bis(octyloxymethyl)-1,3-propanediaminium dichloride (**1.5**) in chloroform-*d*



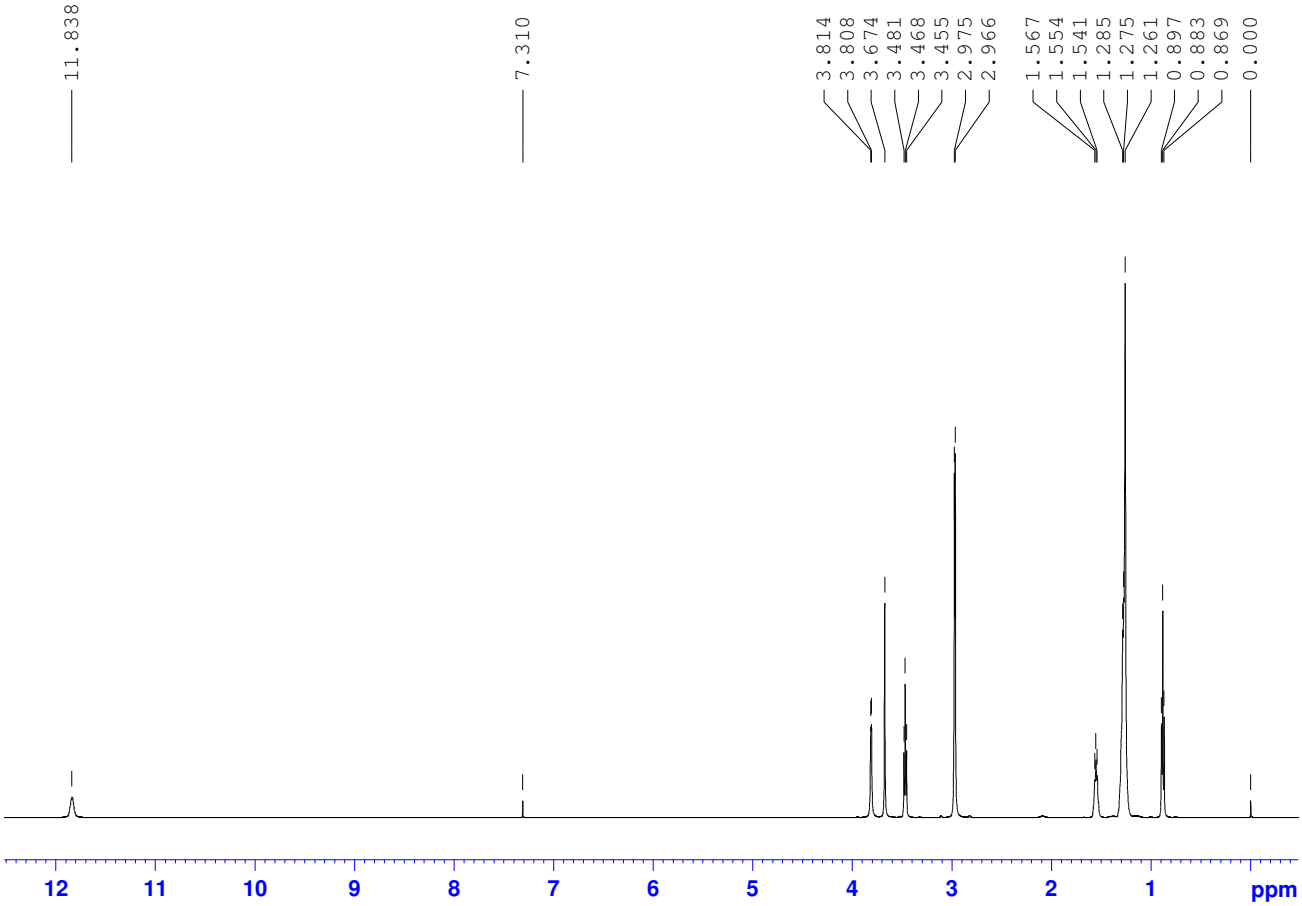
Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of *N*,*N*,*N*',*N*'-tetramethyl-2,2-bis(octyloxymethyl)-1,3-propanediaminium dichloride (**1.5**) in $CDCl_3$



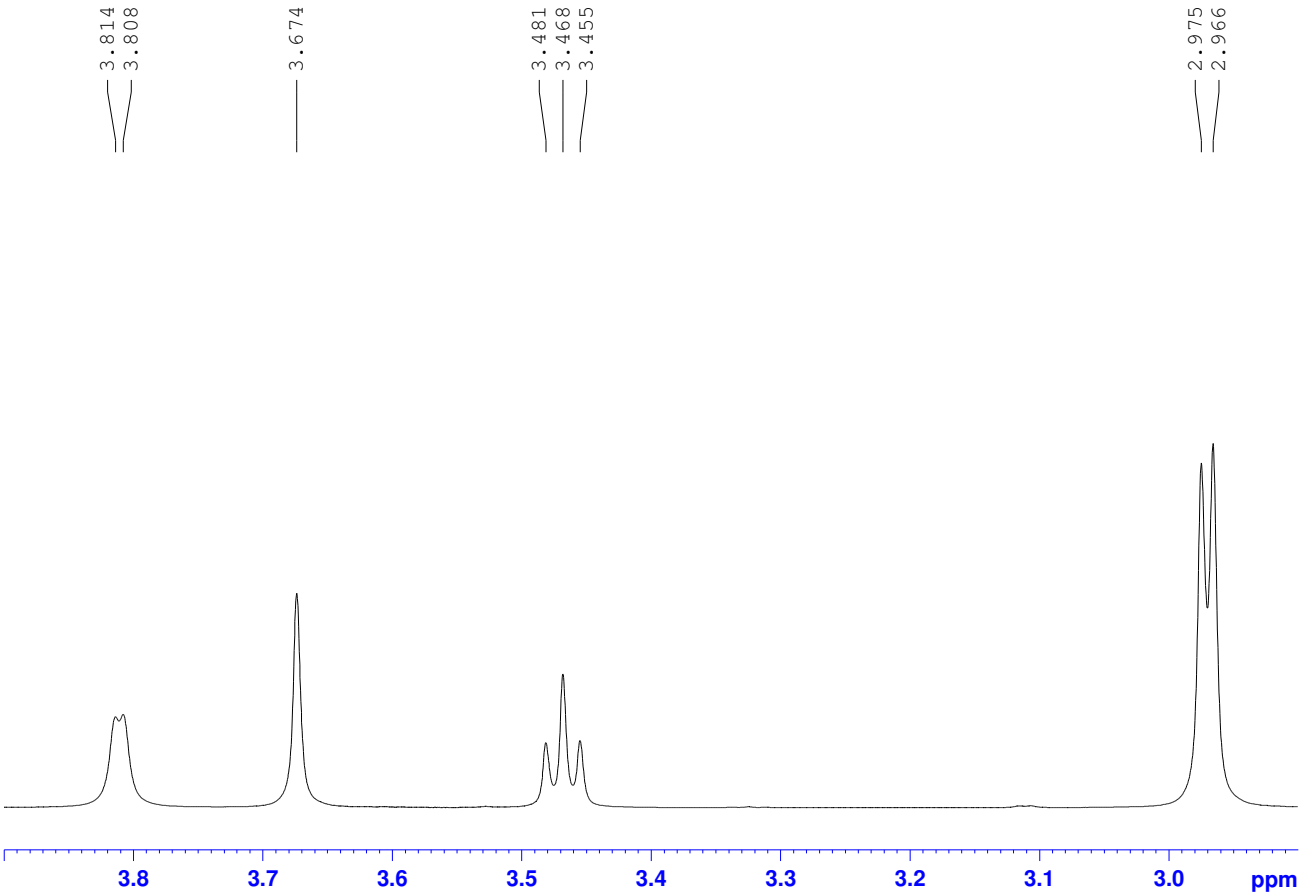
500.1 MHz ^{1}H NMR spectrum of *N*,*N*,*N*',*N*'-tetramethyl-2,2-bis(decyloxymethyl)-1,3-propanediaminium dichloride (**1.6**) in chloroform-*d*



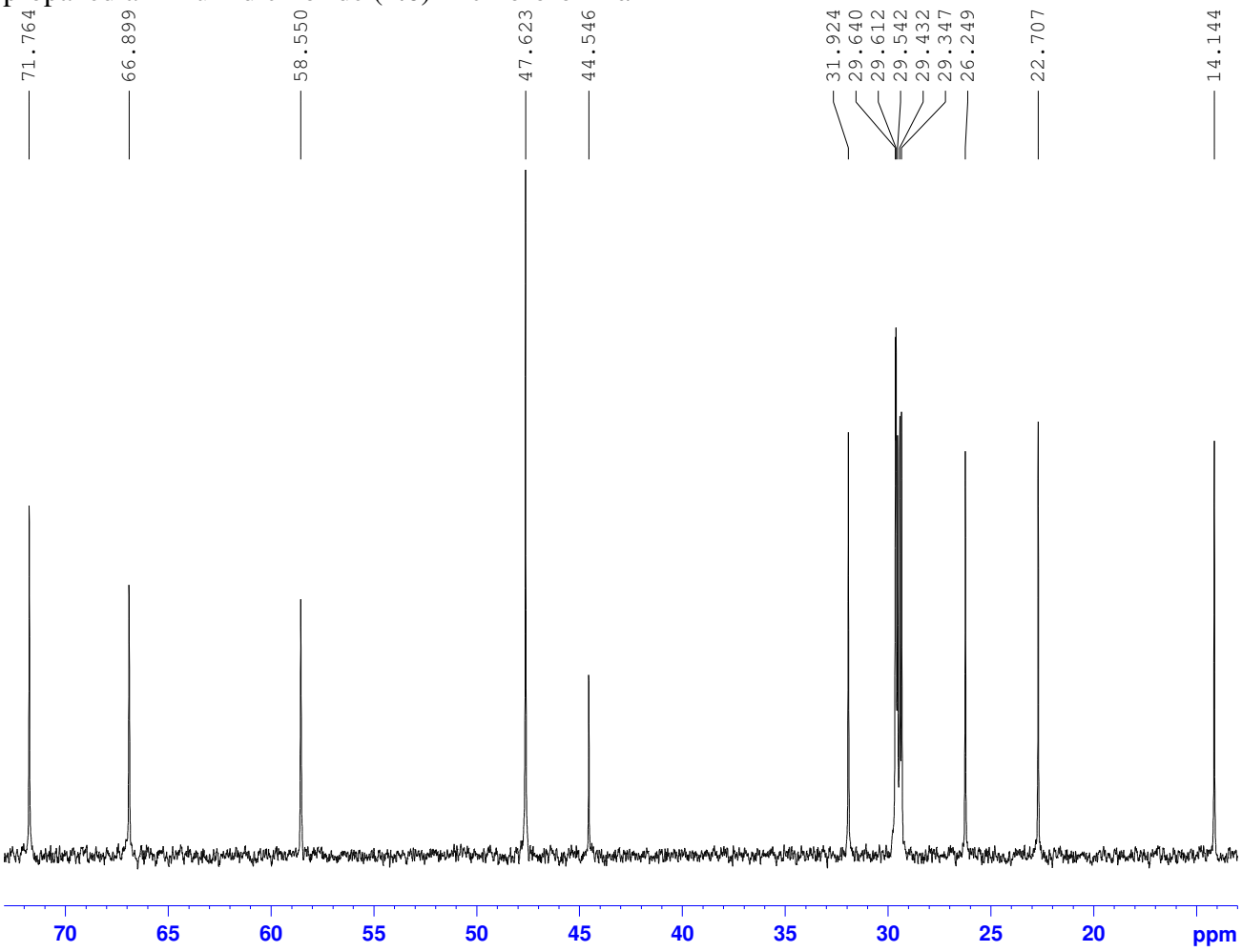
1.6



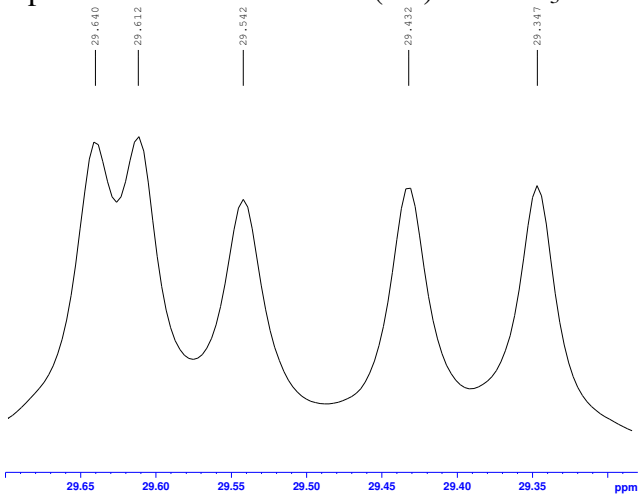
Expansion of part of the 500.1 MHz ^{1}H NMR spectrum of *N*,*N*,*N*',*N*'-tetramethyl-2,2-bis(decyloxymethyl)-1,3-propanediaminium dichloride (**1.6**) in $CDCl_3$



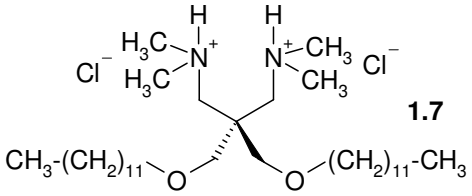
125.7 MHz ^{13}C NMR spectrum of *N*,*N*,*N*',*N*'-tetramethyl-2,2-bis(decyloxymethyl)-1,3-propanediaminium dichloride (**1.6**) in chloroform-*d*



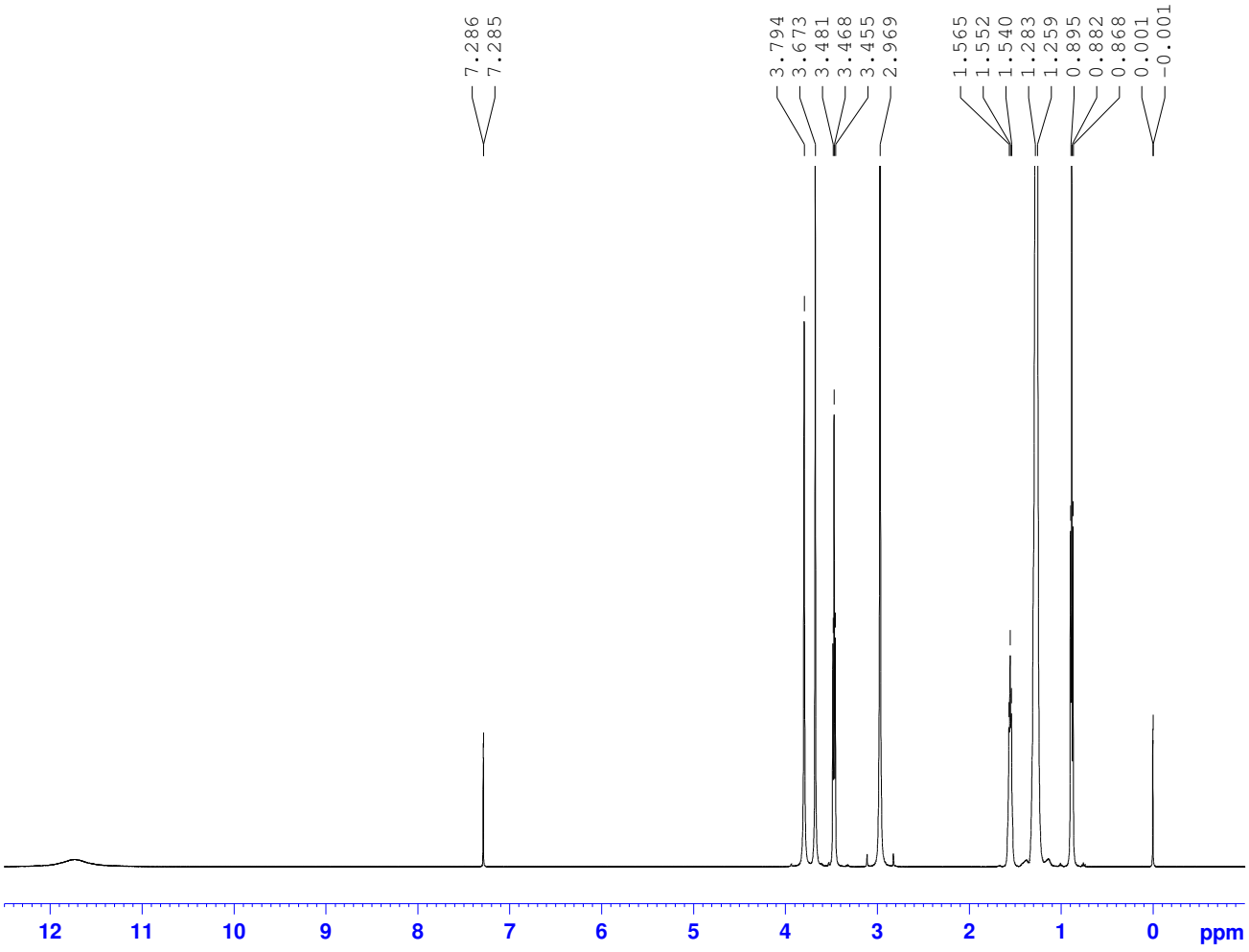
Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of *N*,*N*,*N*',*N*'-tetramethyl-2,2-bis(decyloxymethyl)-1,3-propanediaminium dichloride (**1.6**) in $CDCl_3$



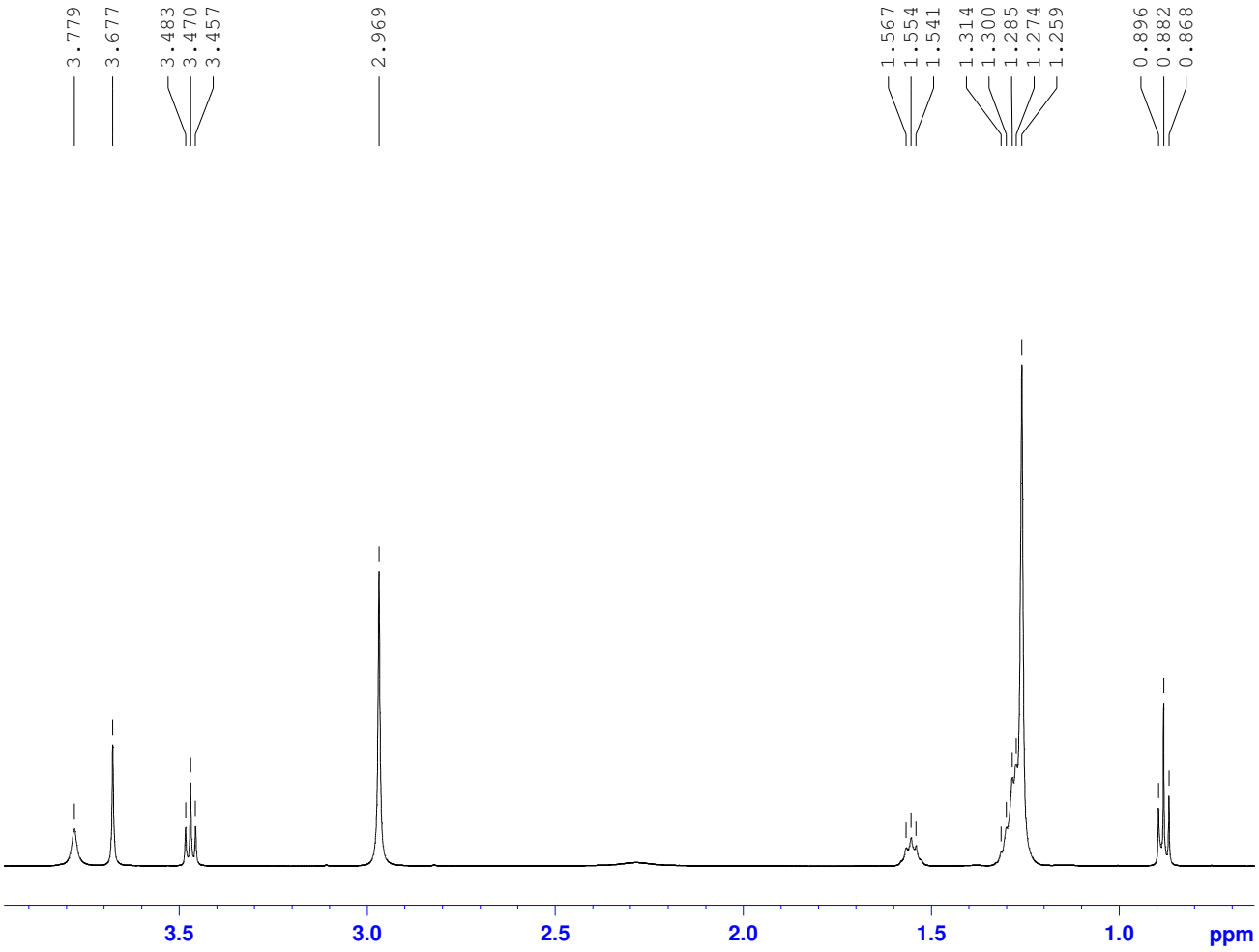
500.1 MHz ^{1}H NMR spectrum of *N*,*N*,*N*',*N*'-tetramethyl-2,2-bis(dodecyloxymethyl)-1,3-propanediaminium dichloride (**1.7**) in chloroform-*d*



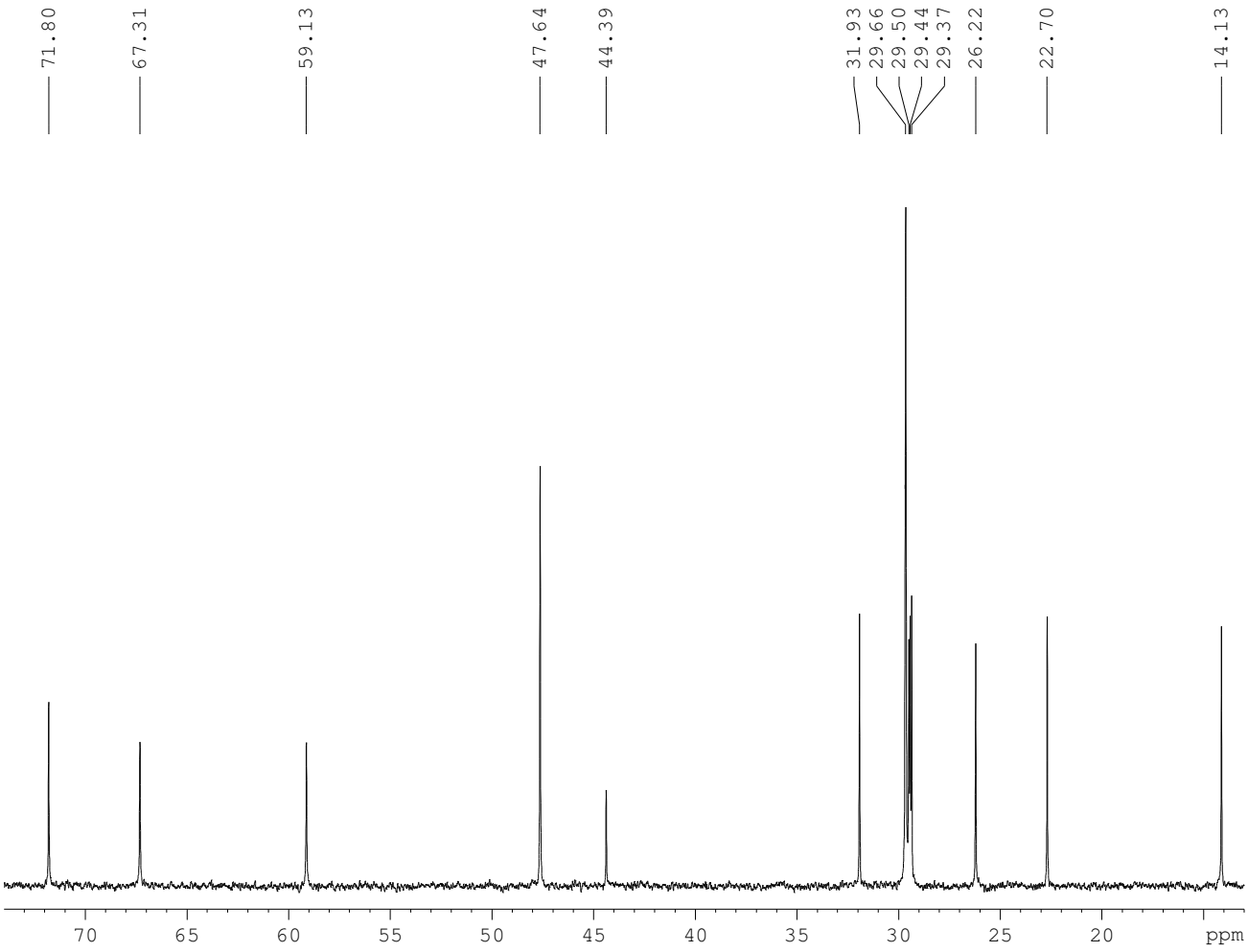
1.7



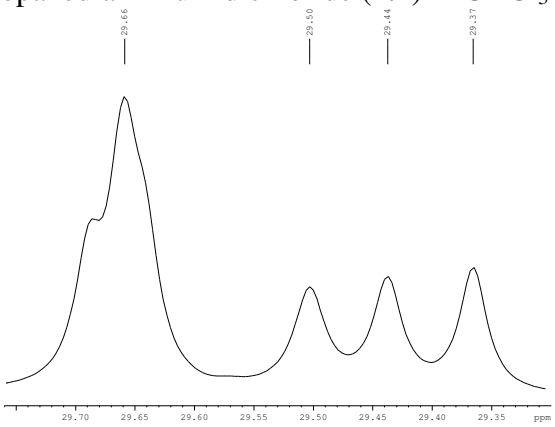
Expansion of part of the 500.1 MHz ^{1}H NMR spectrum of *N*,*N*,*N*',*N*'-tetramethyl-2,2-bis(dodecyloxymethyl)-1,3-propanediaminium dichloride (**1.7**) in $CDCl_3$



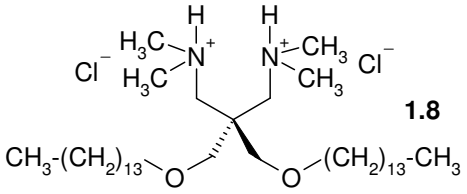
125.7 MHz ^{13}C NMR spectrum of *N*,*N*,*N*',*N*'-tetramethyl-2,2-bis(dodecyloxymethyl)-1,3-propanediaminium dichloride (**1.7**) in chloroform-*d*



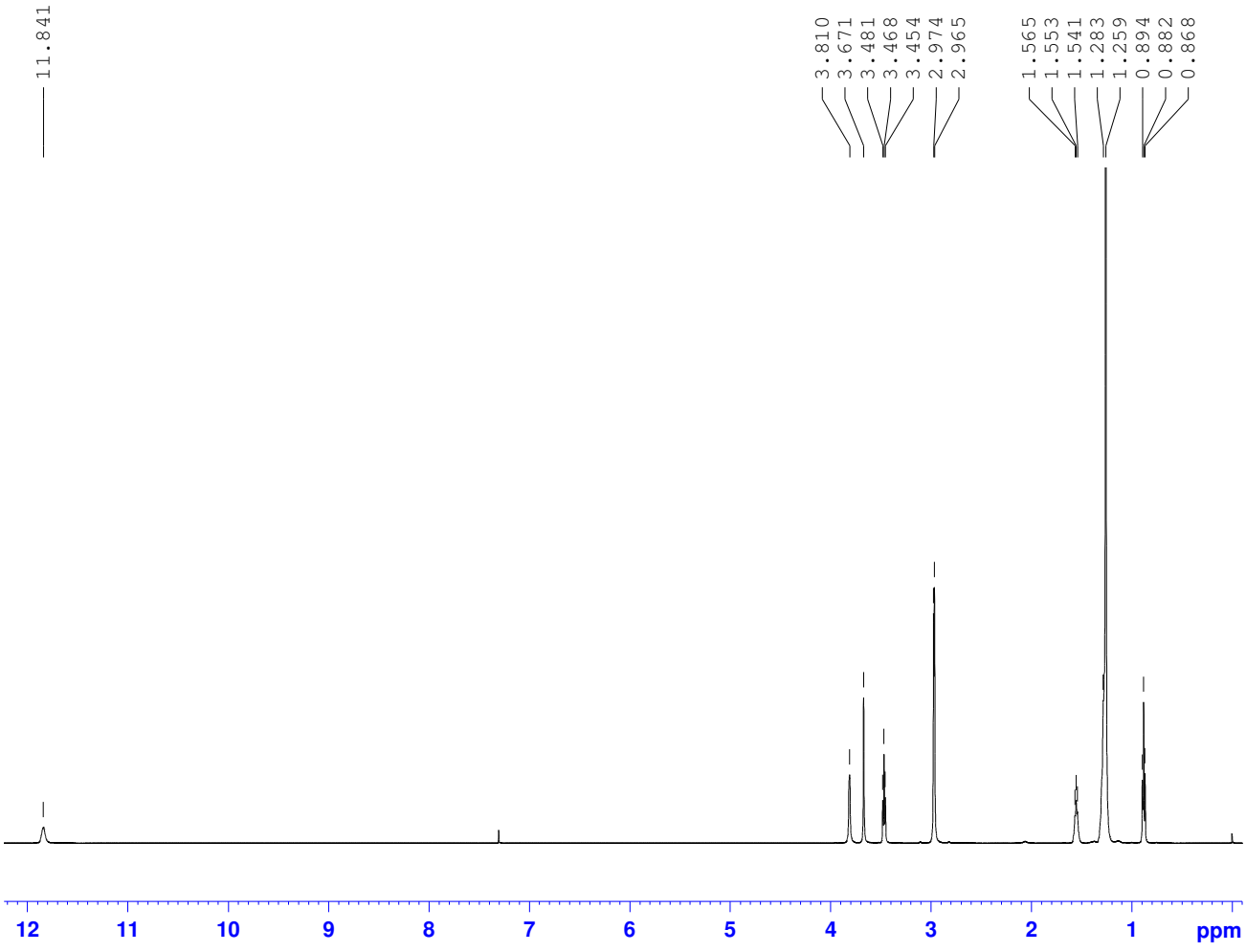
Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of *N*,*N*,*N*',*N*'-tetramethyl-2,2-bis(dodecyloxymethyl)-1,3-propanediaminium dichloride (**1.7**) in $CDCl_3$



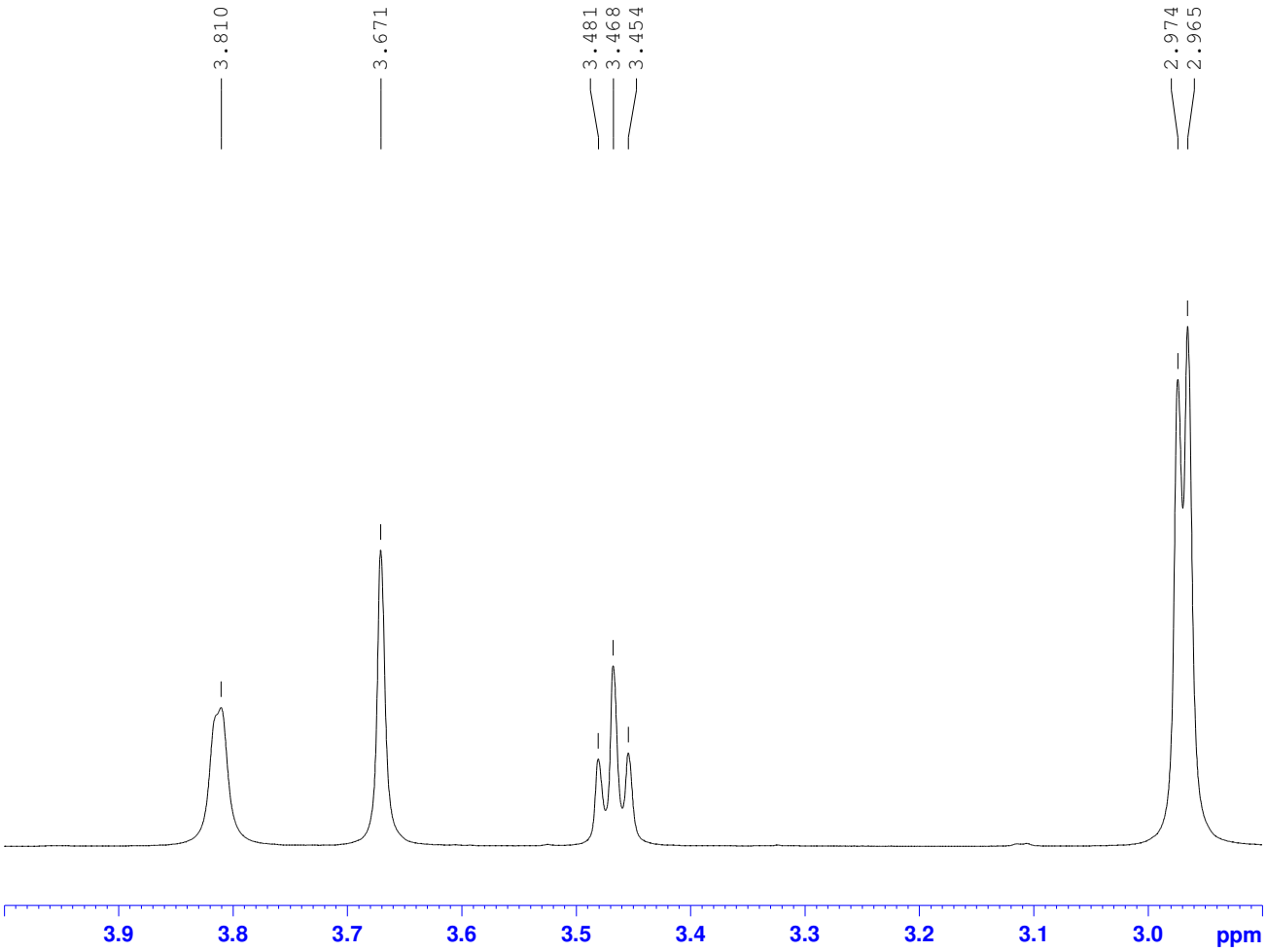
500.1 MHz ^{1}H NMR spectrum of *N*,*N*,*N*',*N*'-tetramethyl-2,2-bis(tetradecyloxymethyl)-1,3-propanediaminium dichloride (**1.8**) in chloroform-*d*



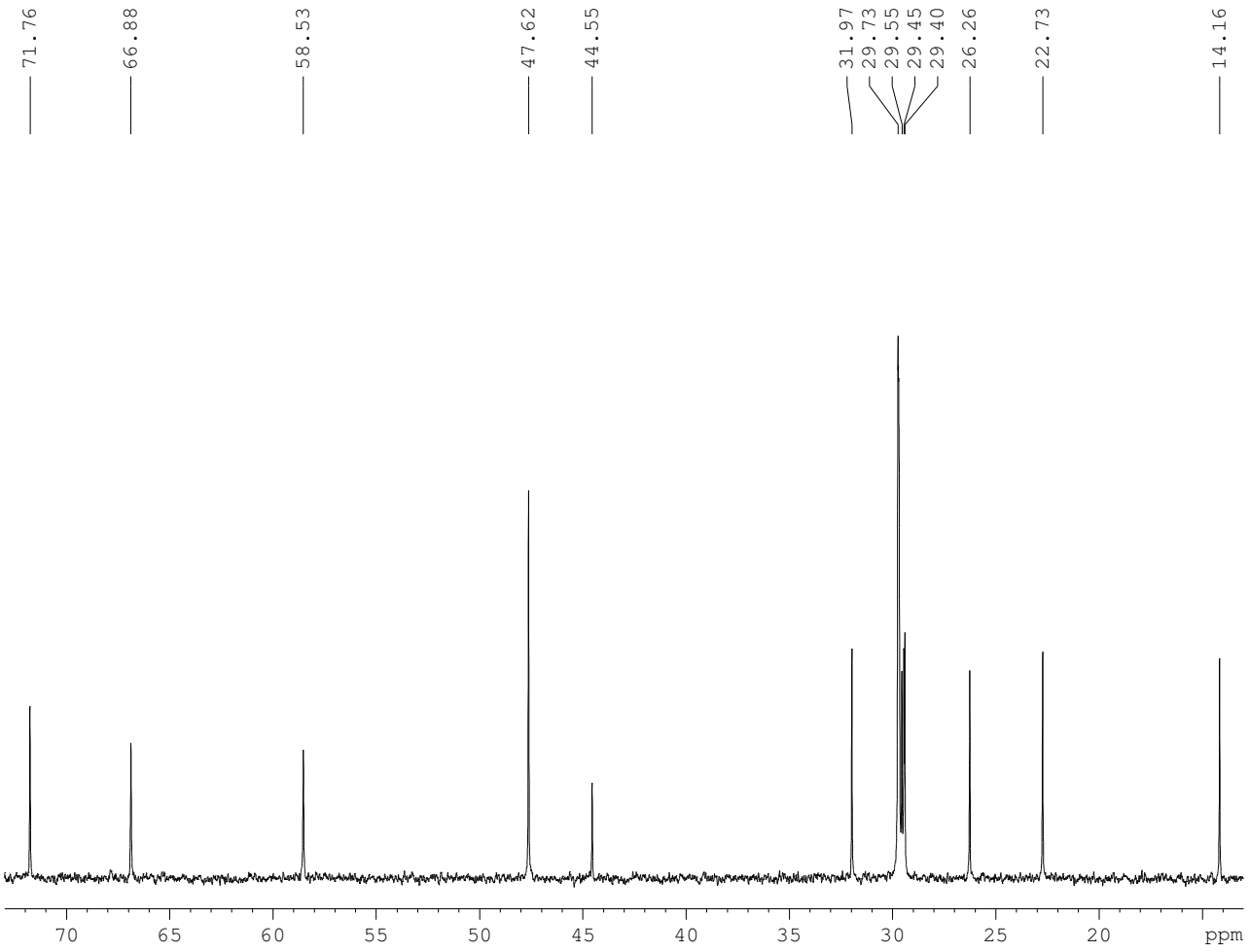
1.8



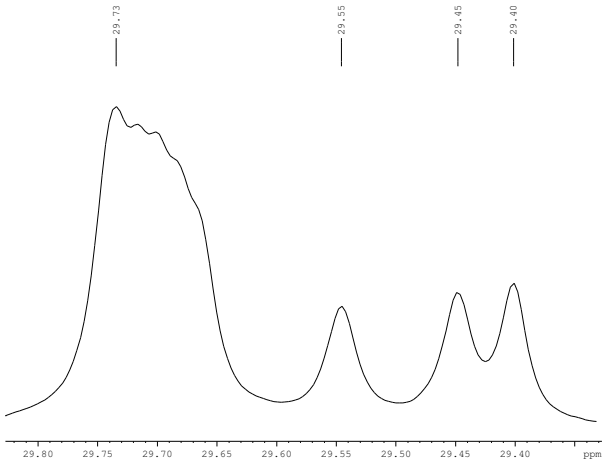
Expansion of part of the 500.1 MHz ^{1}H NMR spectrum of *N*,*N*,*N*',*N*'-tetramethyl-2,2-bis(tetradecyloxymethyl)-1,3-propanediaminium dichloride (**1.8**) in $CDCl_3$



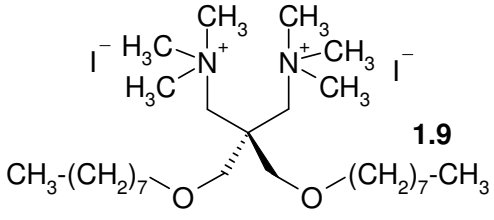
125.7 MHz ^{13}C NMR spectrum of *N*,*N*,*N*',*N*'-tetramethyl-2,2-bis(tetradecyloxymethyl)-1,3-propanediaminium dichloride (**1.8**) in chloroform-*d*

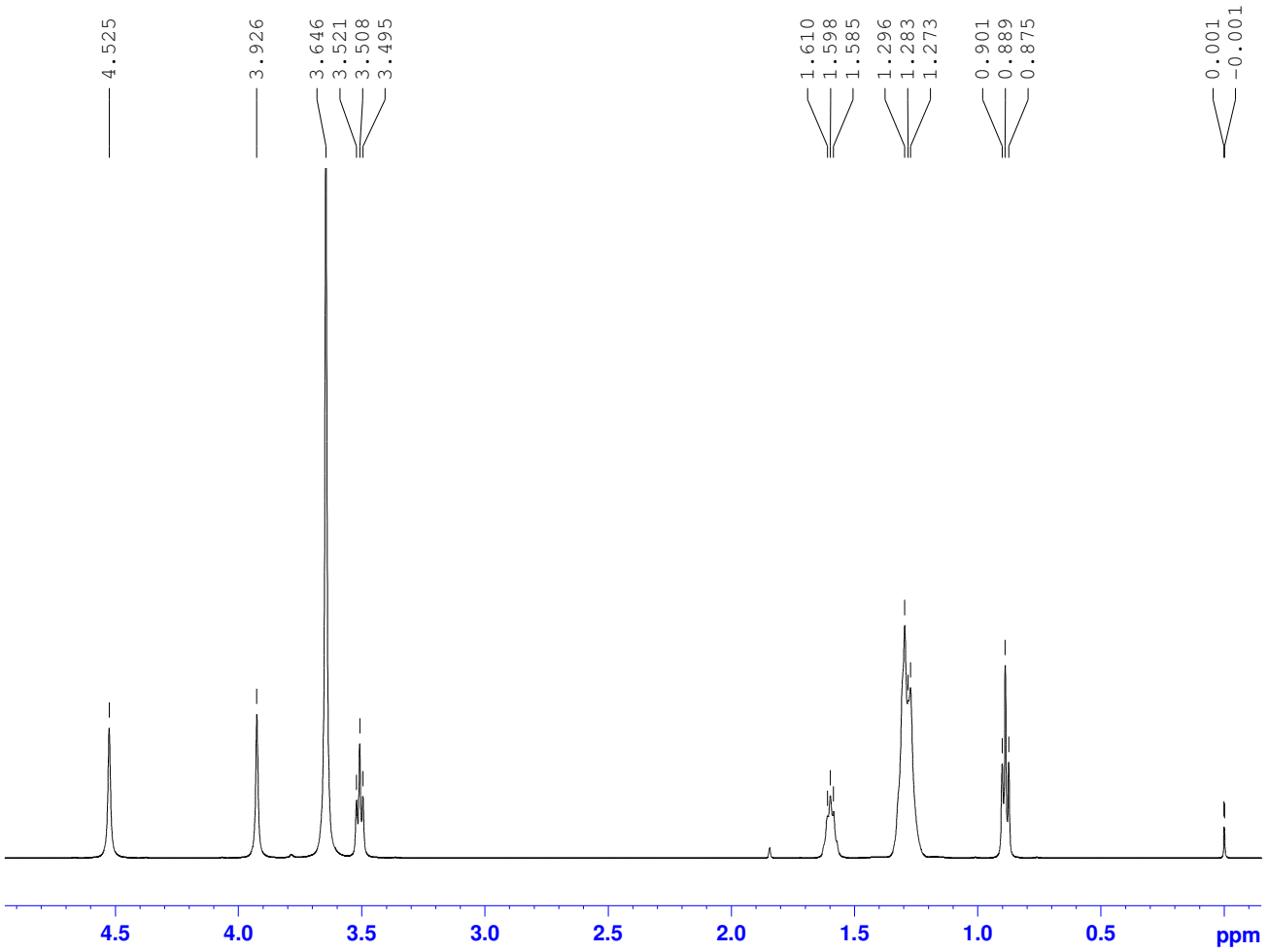


Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of *N*,*N*,*N*',*N*'-tetramethyl-2,2-bis(tetradecyloxymethyl)-1,3-propanediaminium dichloride (**1.8**) in $CDCl_3$

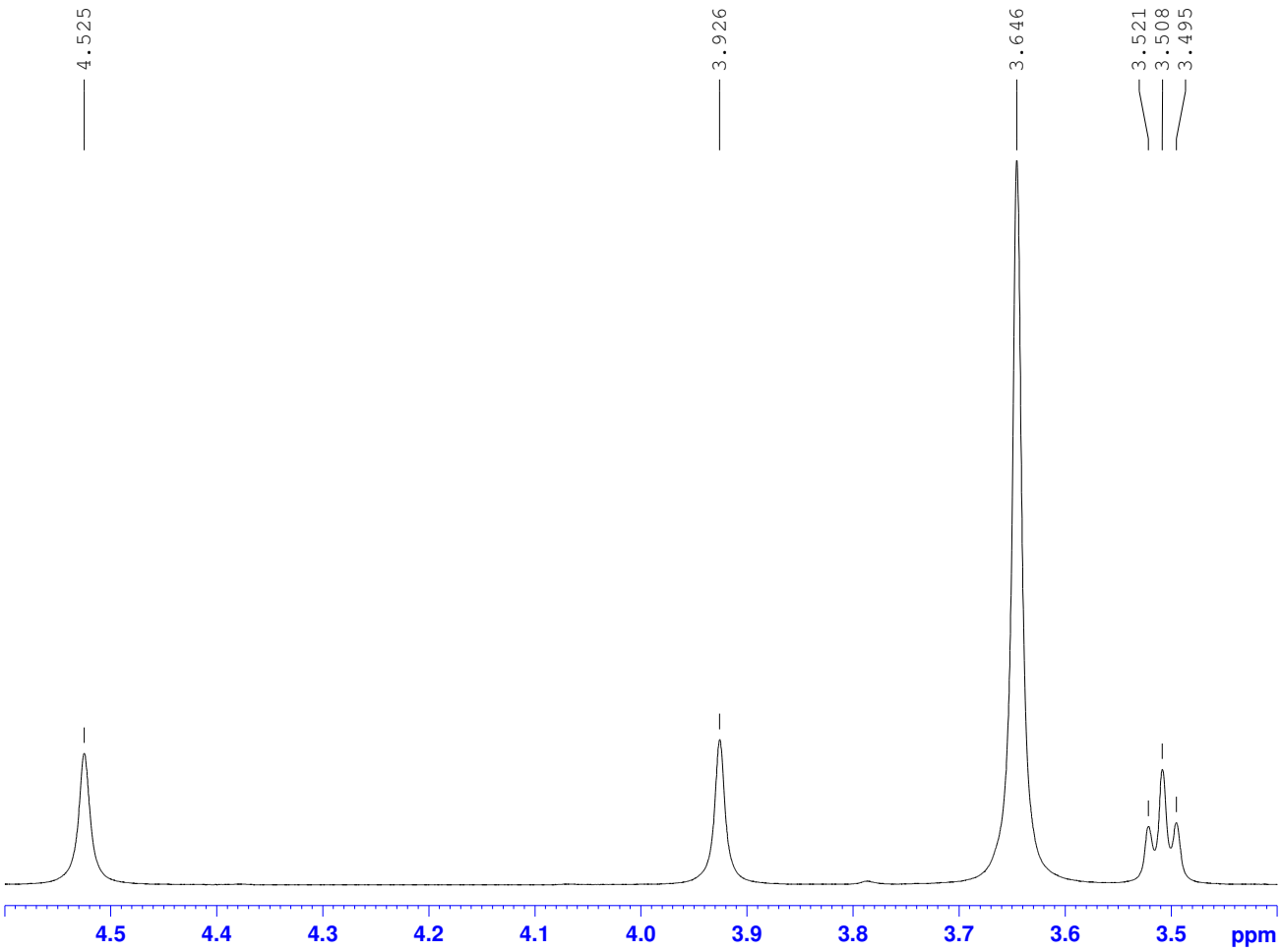


500.1 MHz ^{1}H NMR spectrum of *N*,*N*,*N*',*N'*,*N*'-hexamethyl-2,2-bis(octyloxymethyl)-1,3-propanediammonium diiodide (**1.9**) in chloroform-*d*

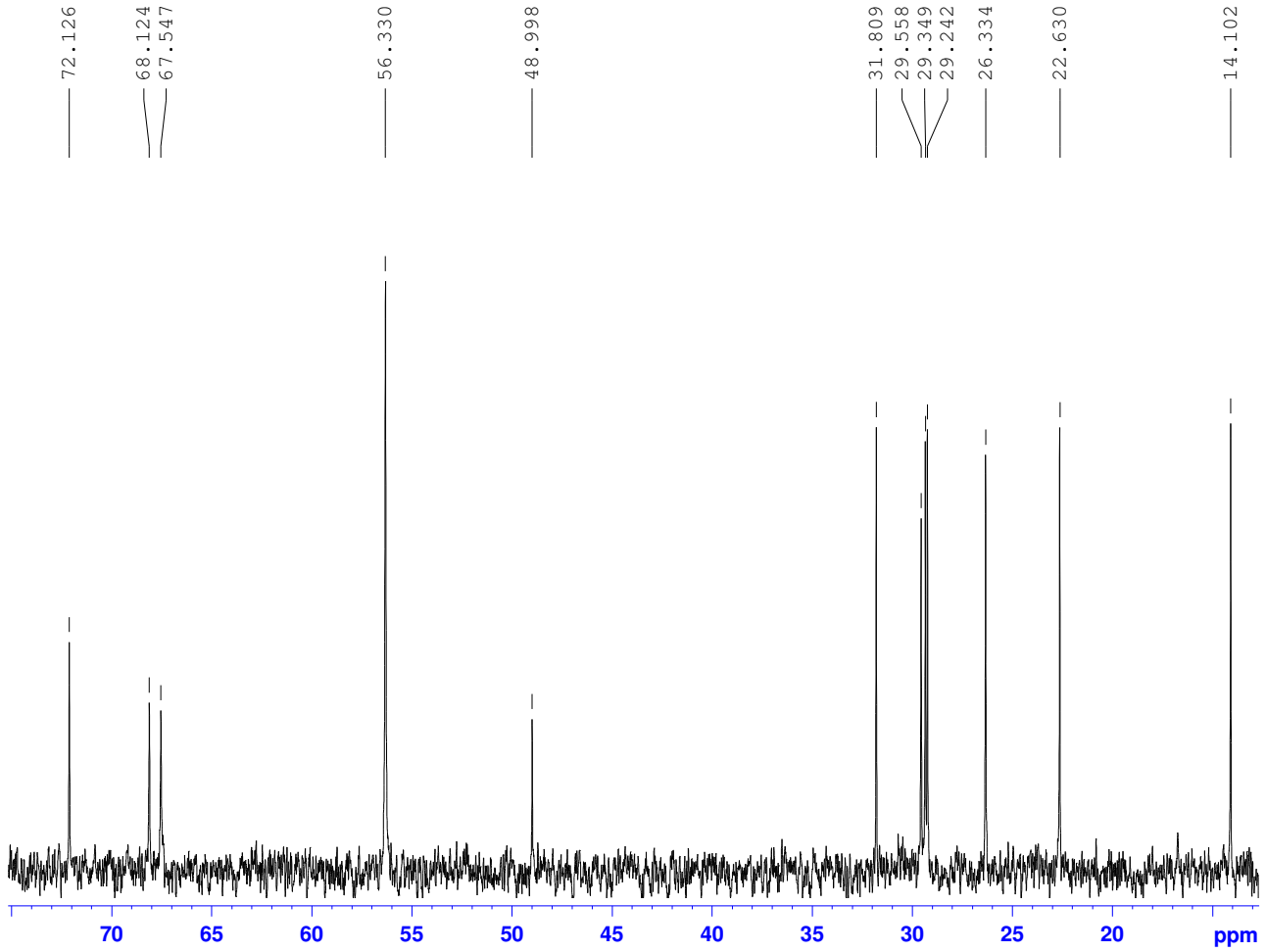




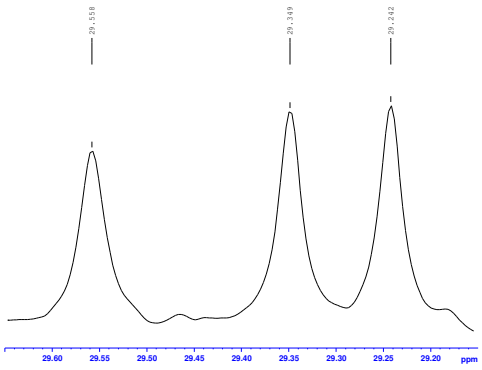
Expansion of part of the 500.1 MHz ^{1}H NMR spectrum of *N*,*N*,*N*',*N*',*N*'-hexamethyl-2,2-bis(octyloxymethyl)-1,3-propanediammonium diiodide (**1.9**) in chloroform-*d*



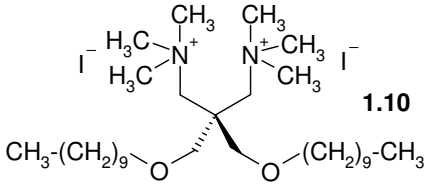
125.7 MHz ^{13}C NMR spectrum of *N*,*N*,*N*,*N*',*N*',*N*'-hexamethyl-2,2-bis(octyloxymethyl)-1,3-propanediammonium diiodide (**1.9**) in chloroform-*d*



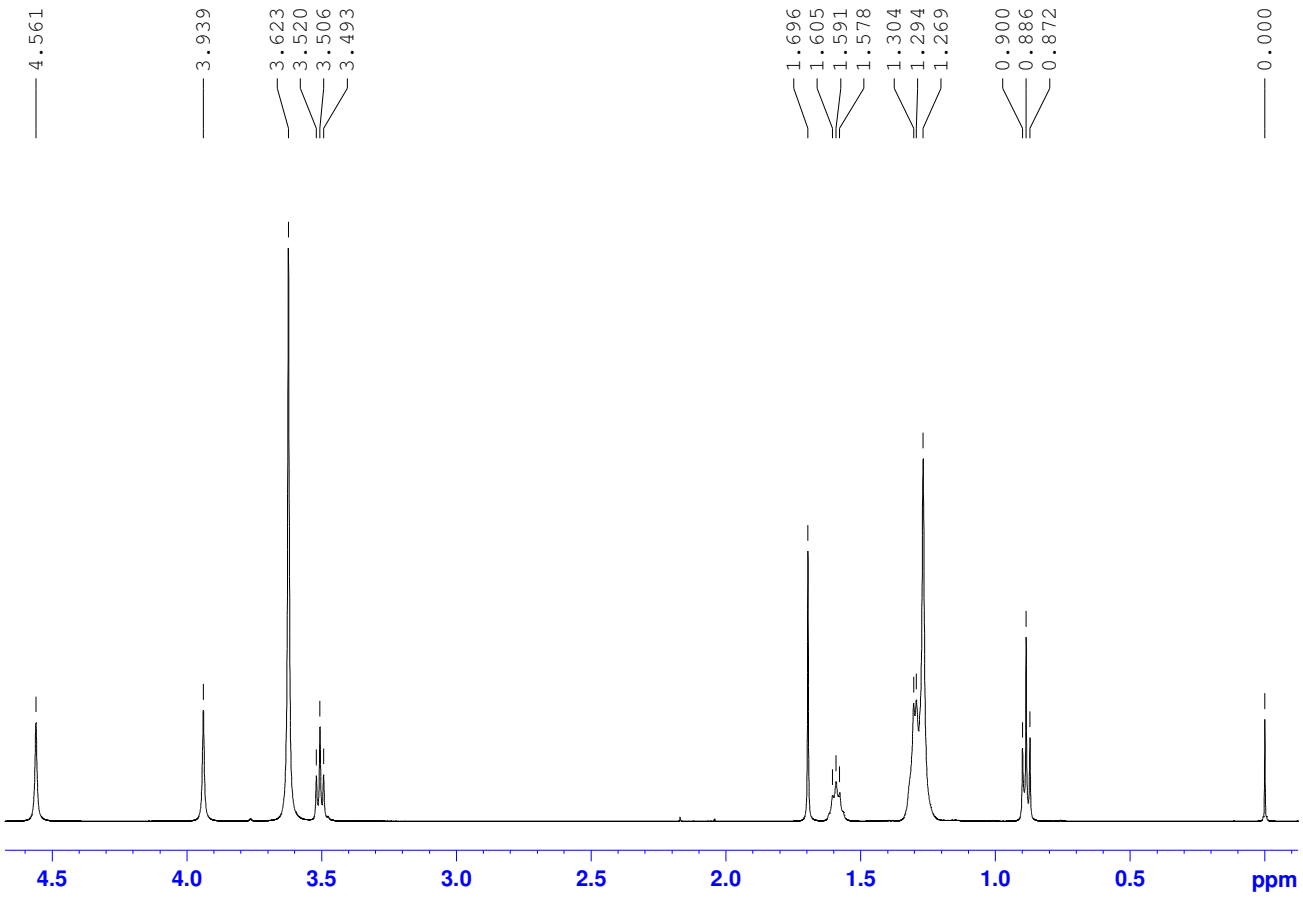
Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of *N*,*N*,*N*,*N*',*N*',*N*'-hexamethyl-2,2-bis(octyloxymethyl)-1,3-propanediammonium diiodide (**1.9**) in $CDCl_3$



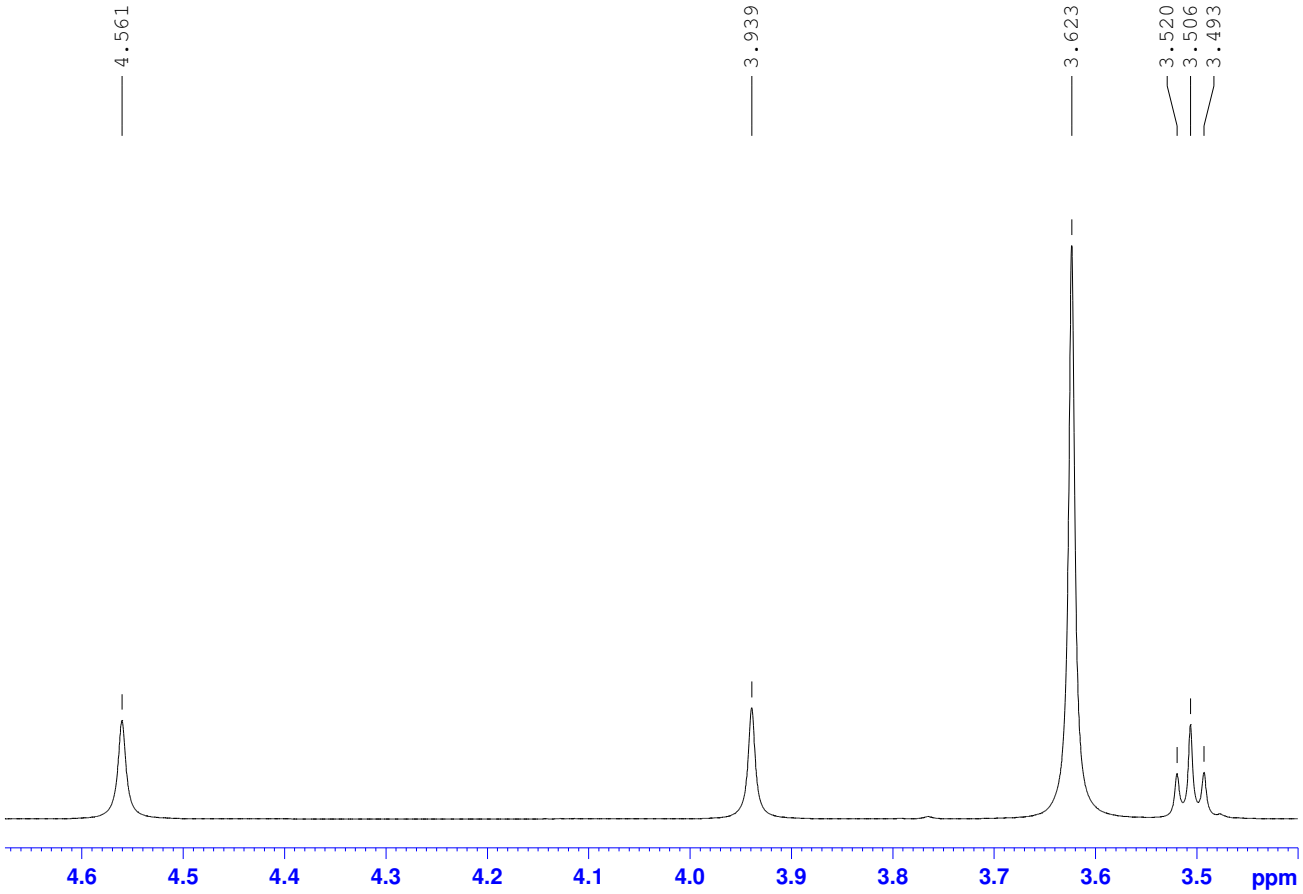
500.1 MHz ^{1}H NMR spectrum of 2,2-bis(decyloxymethyl)-*N*,*N*,*N*,*N*',*N*',*N*'-hexamethyl-1,3-propanediammonium diiodide (**1.10**) in chloroform-*d*



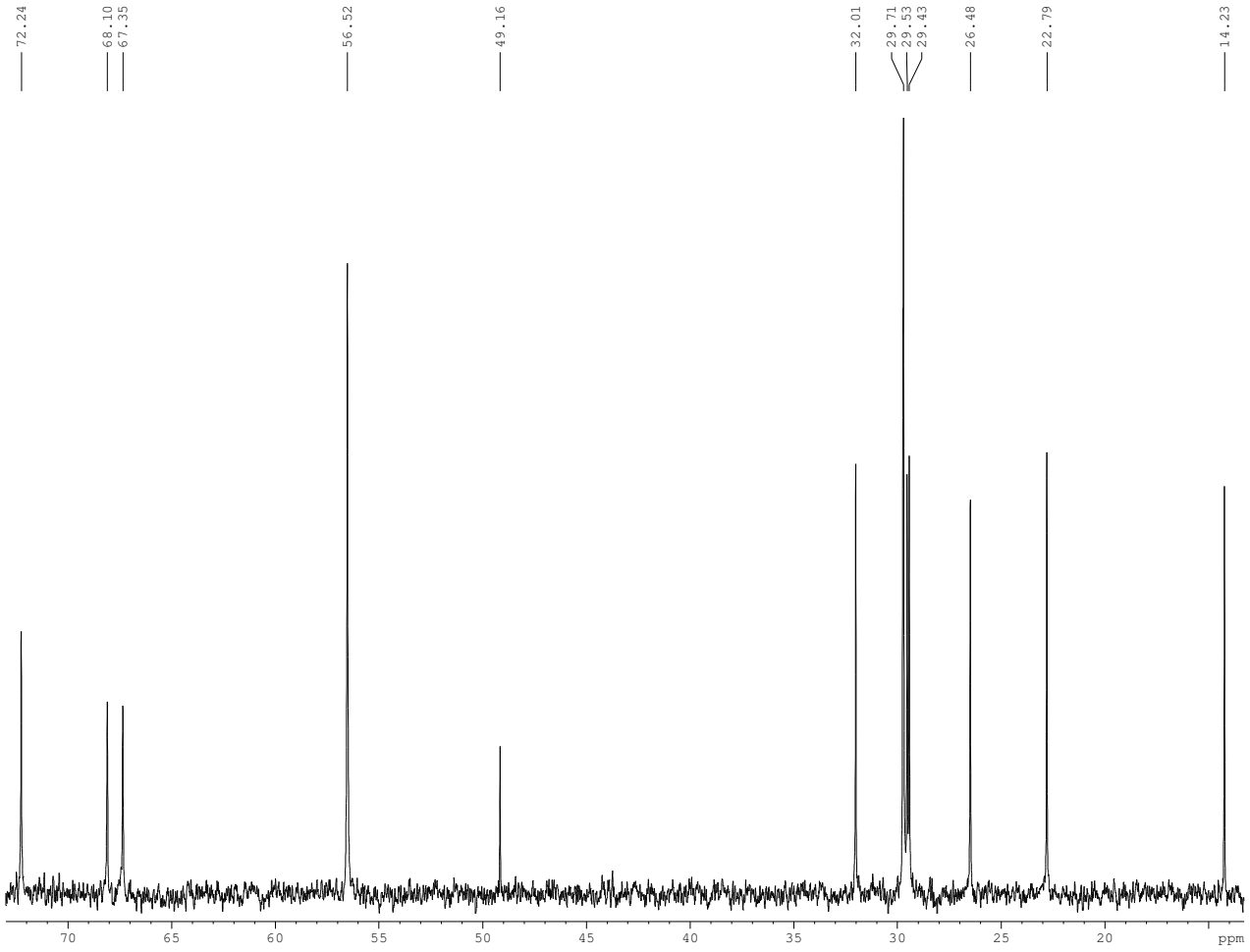
1.10



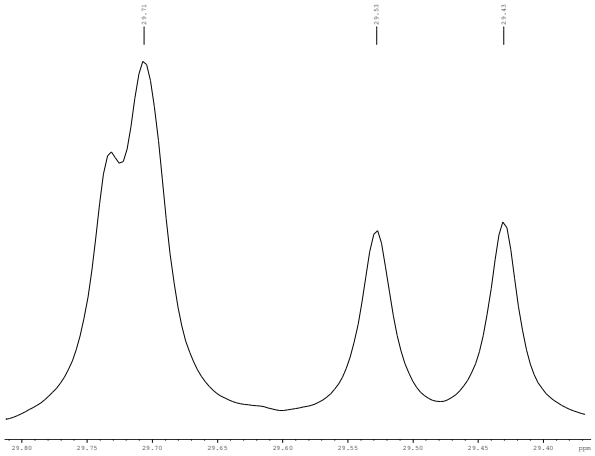
Expansion of part of the 500.1 MHz ^{1}H NMR spectrum of 2,2-bis(decyloxymethyl)-*N*,*N*,*N*,*N*',*N*',*N*'-hexamethyl-1,3-propanediammonium diiodide (**1.10**) in $CDCl_3$



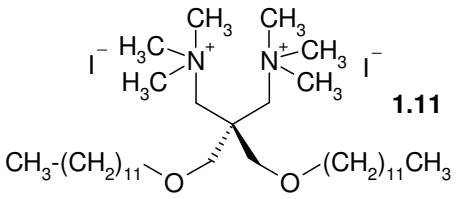
125.7 MHz ^{13}C NMR spectrum of 2,2-bis(decyloxymethyl)-*N*,*N*,*N*,*N*',*N'*,*N*'-hexamethyl-1,3-propanediammonium diiodide (**1.10**) in chloroform-*d*



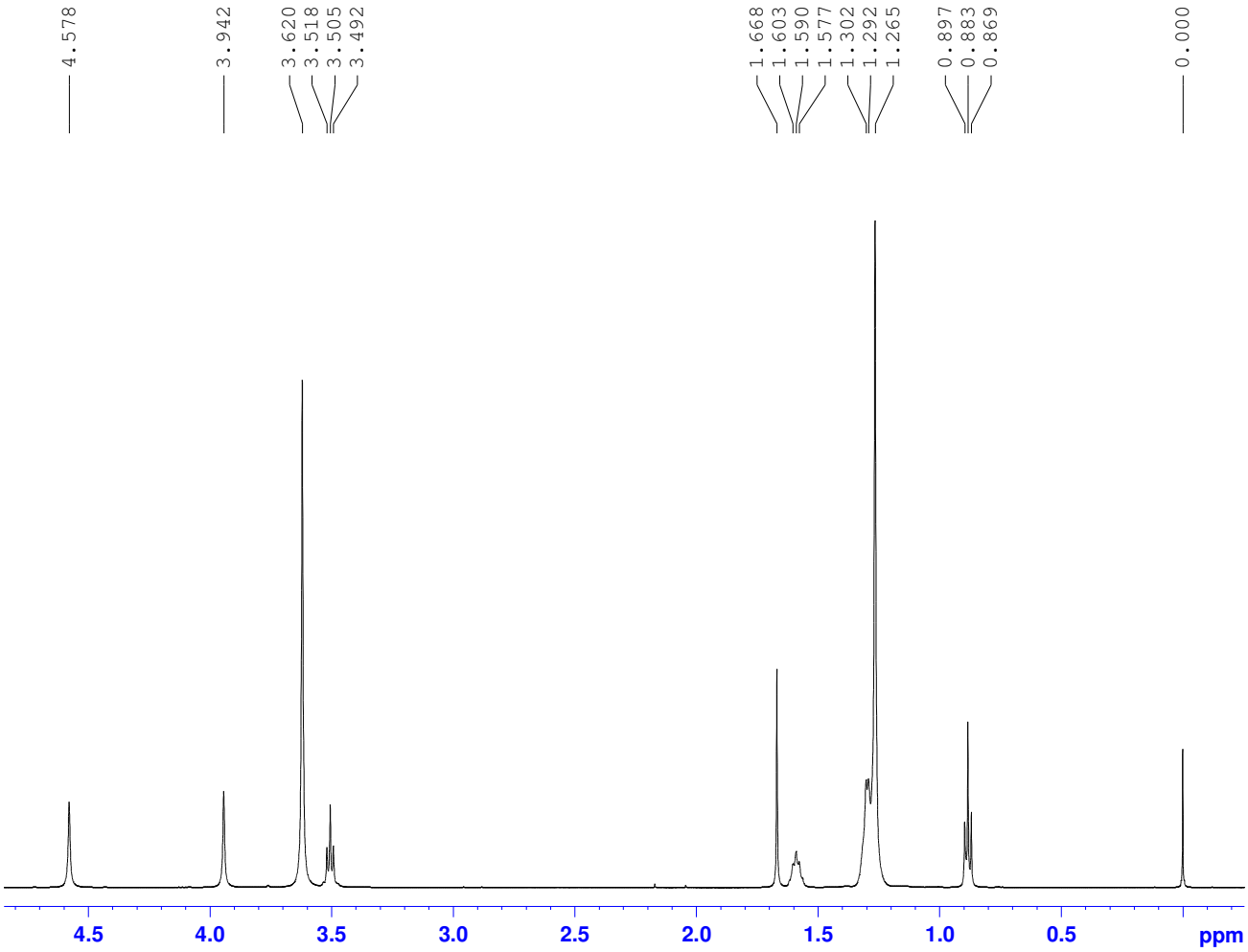
Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of bis(decyloxymethyl)-*N*,*N*,*N*,*N*',*N'*,*N*'-hexamethyl-2,2-1,3-propanediammonium diiodide (**1.10**) in $CDCl_3$



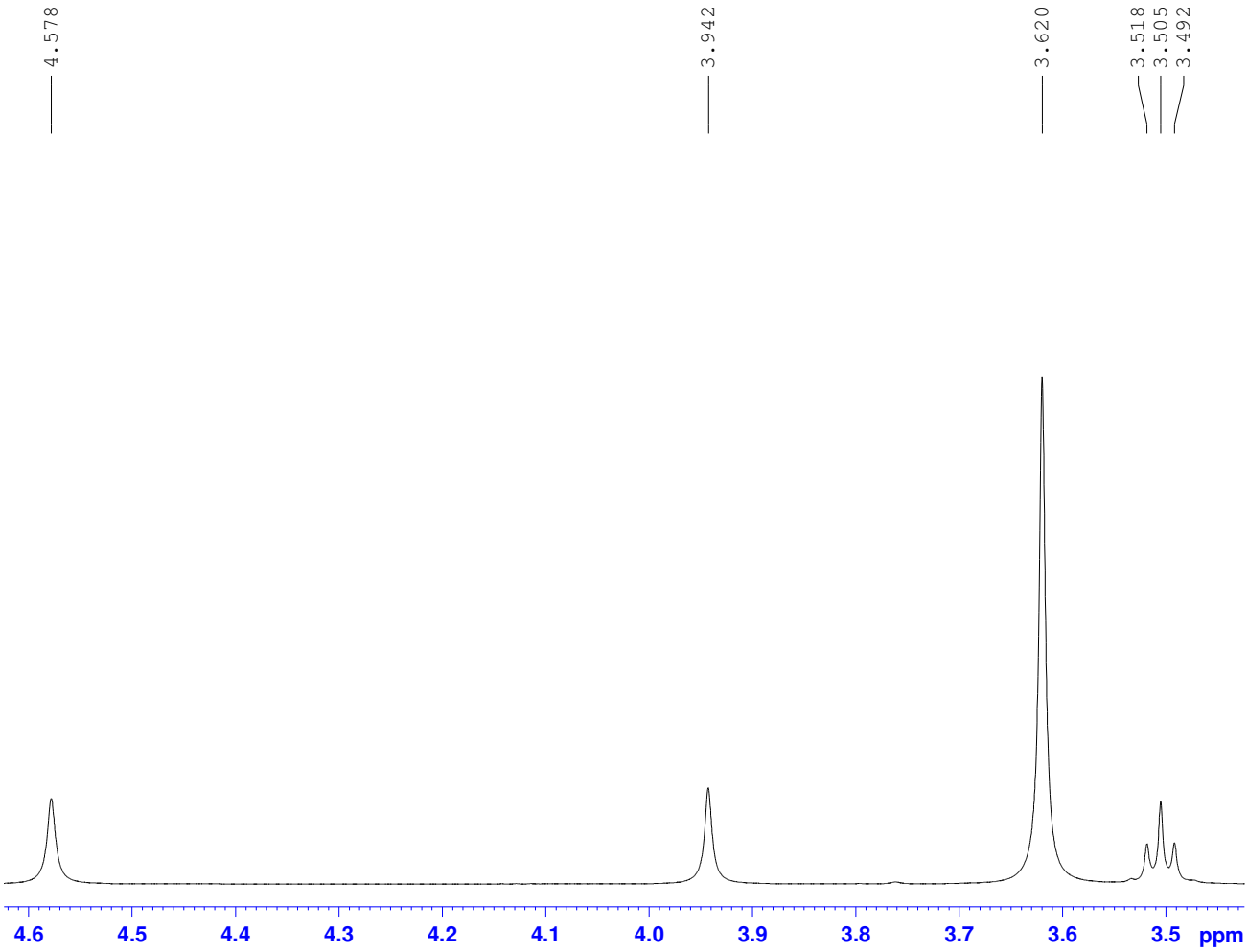
500.1 MHz ^{1}H NMR spectrum of 2,2-bis(dodecyloxymethyl)-*N*,*N*,*N*,*N*',*N*',*N*'-hexamethyl-1,3-propanediammonium diiodide (**1.11**) in chloroform-*d*



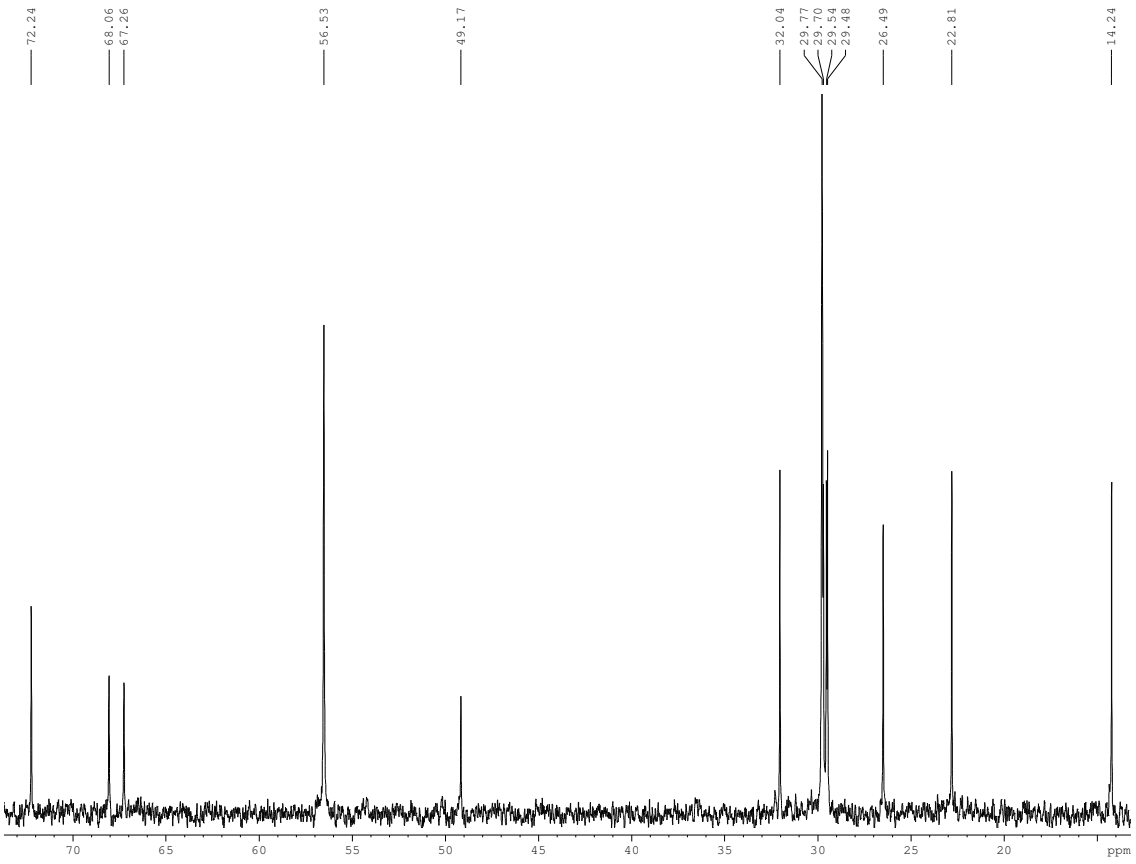
1.11



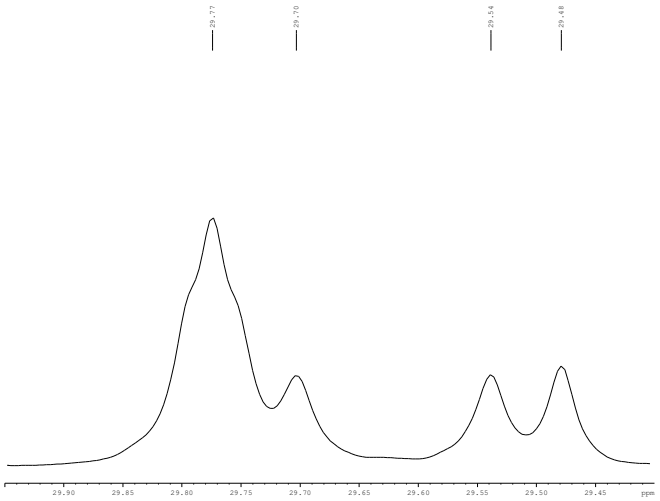
Expansion of part of the 500.1 MHz ^{1}H NMR spectrum of 2,2-bis(dodecyloxymethyl)-*N*,*N*,*N*,*N*',*N*',*N*'-hexamethyl-1,3-propanediammonium diiodide (**1.11**) in $CDCl_3$



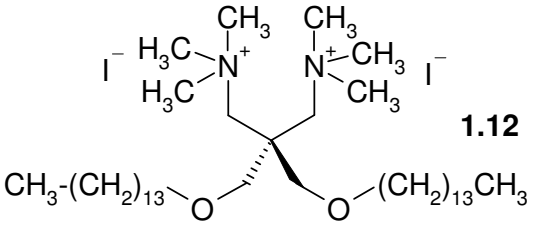
125.7 MHz ^{13}C NMR spectrum of 2,2-bis(dodecyloxymethyl)-*N*,*N*,*N*,*N*',*N'*,*N*'-hexamethyl-1,3-propanediammonium diiodide (**1.11**) in chloroform-*d*



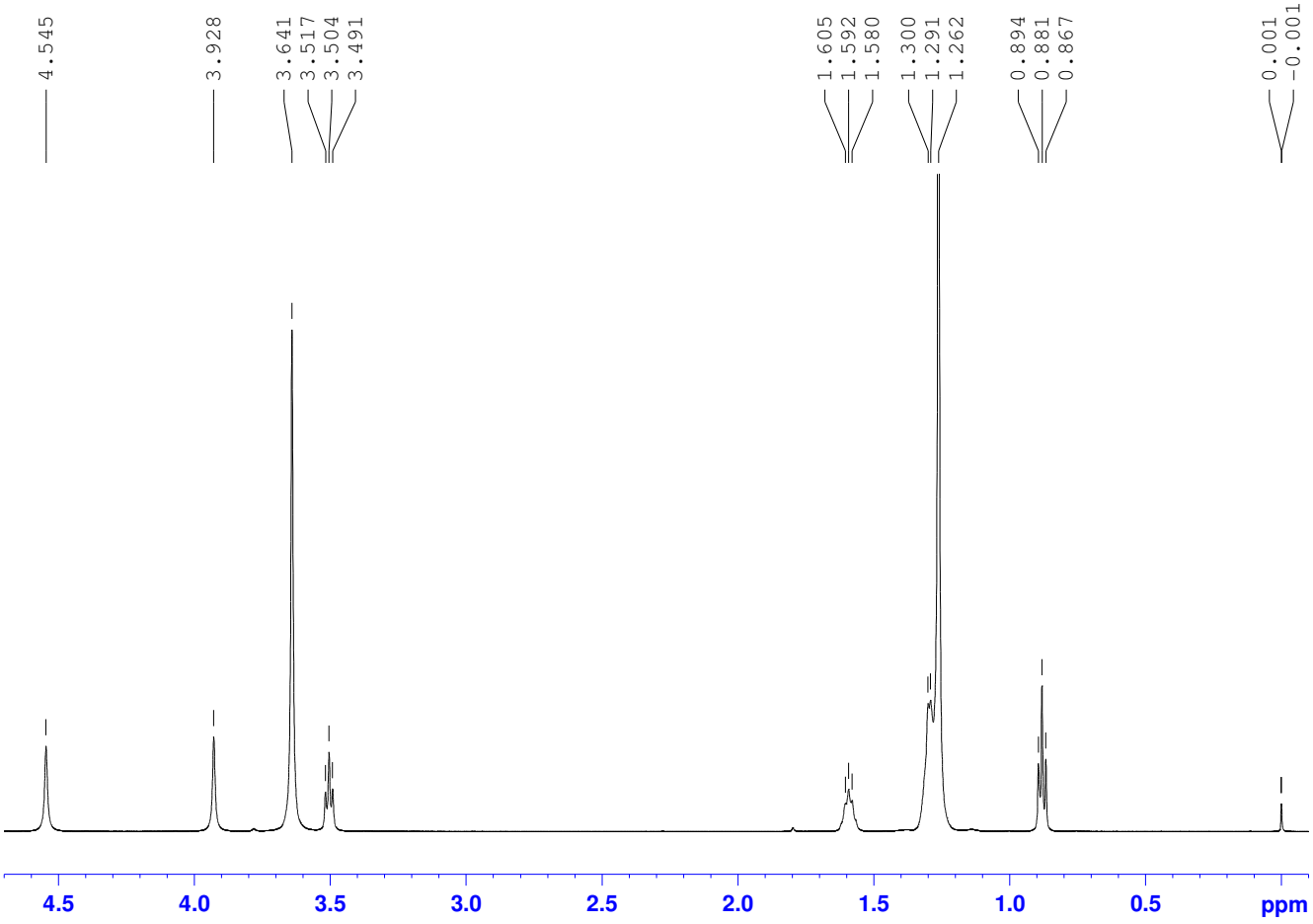
Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of 2,2-bis(dodecyloxymethyl)-*N*,*N*,*N*,*N*',*N'*,*N*'-hexamethyl-1,3-propanediammonium diiodide (**1.11**) in $CDCl_3$



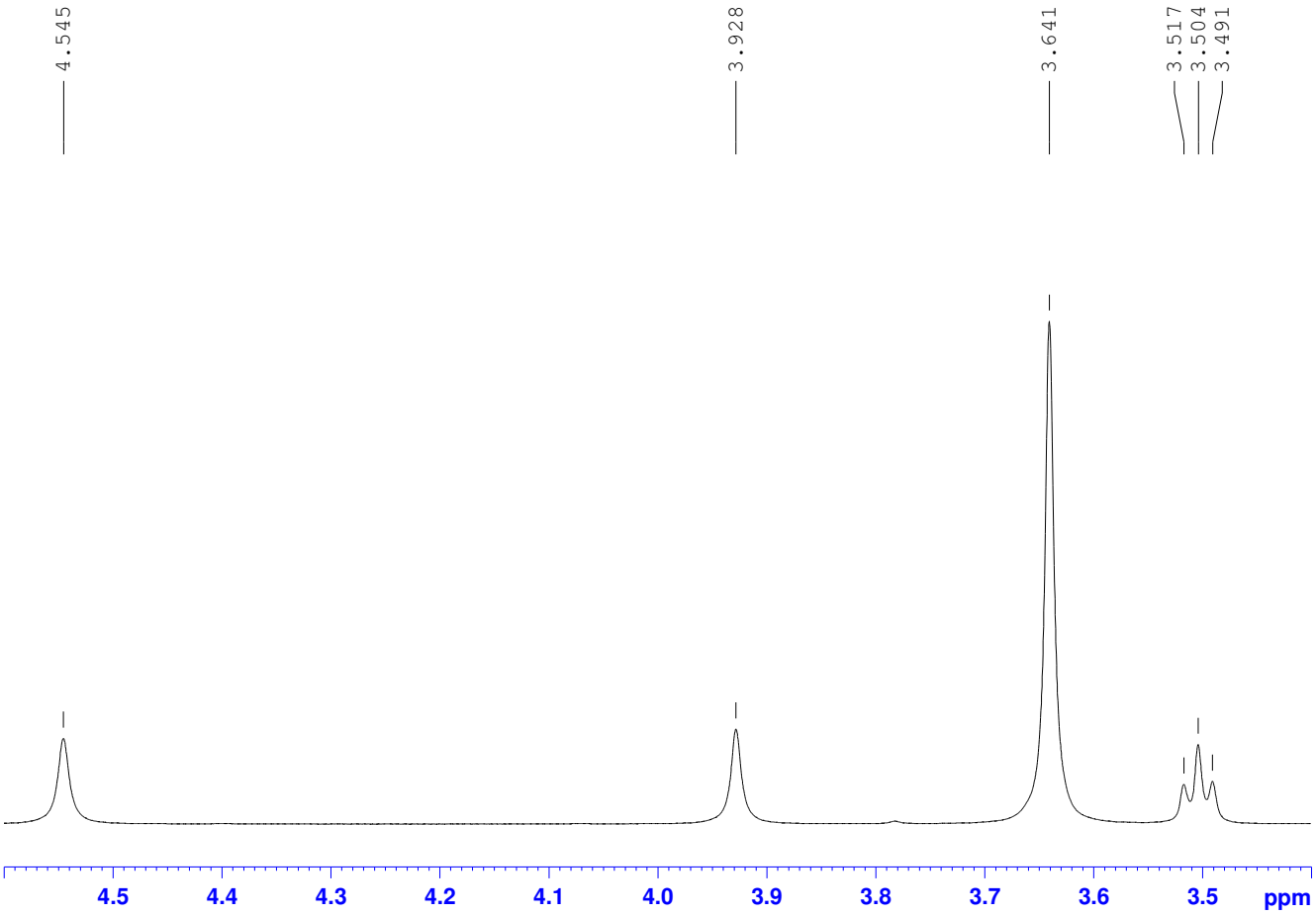
500.1 MHz ^{1}H NMR spectrum of *N*,*N*,*N*,*N*',*N*',*N*'-hexamethyl-2,2-bis(tetradecyloxymethyl)-1,3-propanediammonium diiodide (**1.12**) in chloroform-*d*



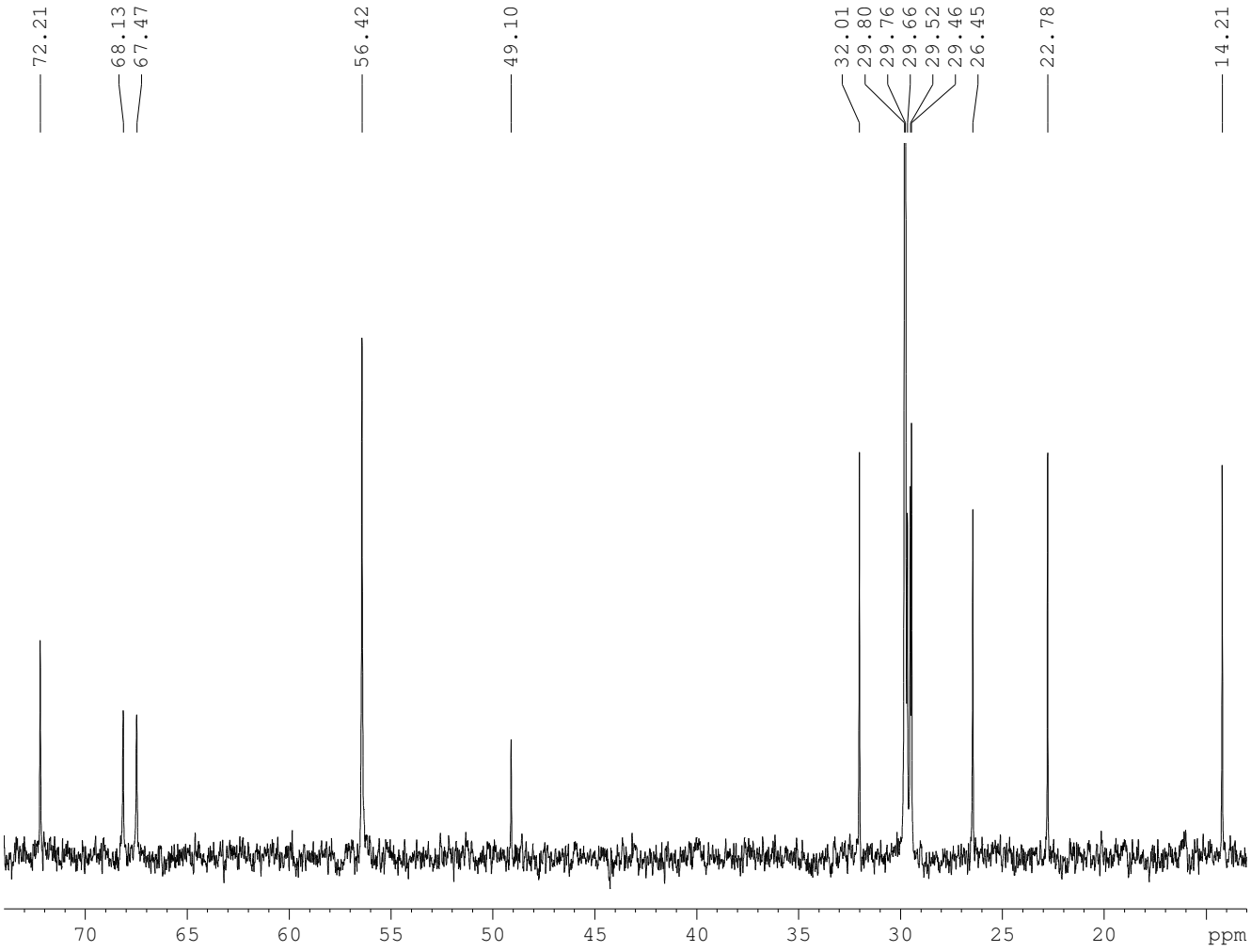
1.12



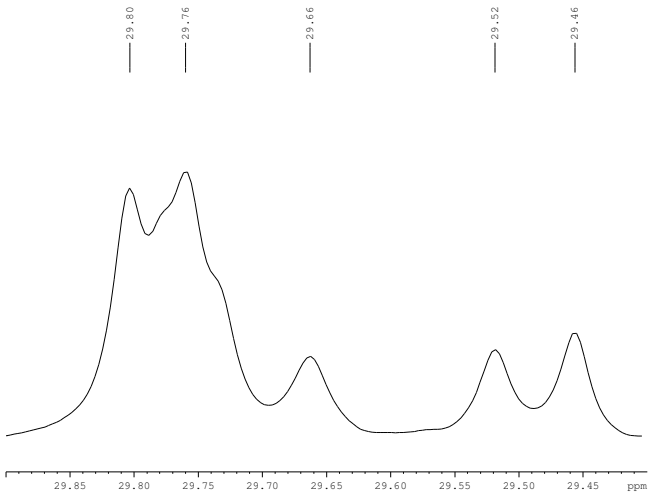
Expansion of part of the 500.1 MHz ^{1}H NMR spectrum of *N*,*N*,*N*',*N*',*N*'-hexamethyl-2,2-bis(tetradecyloxymethyl)-1,3-propanediammonium diiodide (**1.12**) in chloroform-*d*



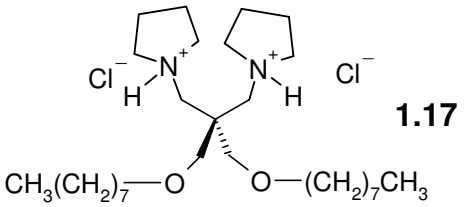
125.7 MHz ^{13}C NMR spectrum of *N*,*N*,*N*,*N*',*N'*,*N*'-hexamethyl-2,2-bis(tetradecyloxymethyl)-1,3-propanediammonium diiodide (**1.12**) in chloroform-*d*



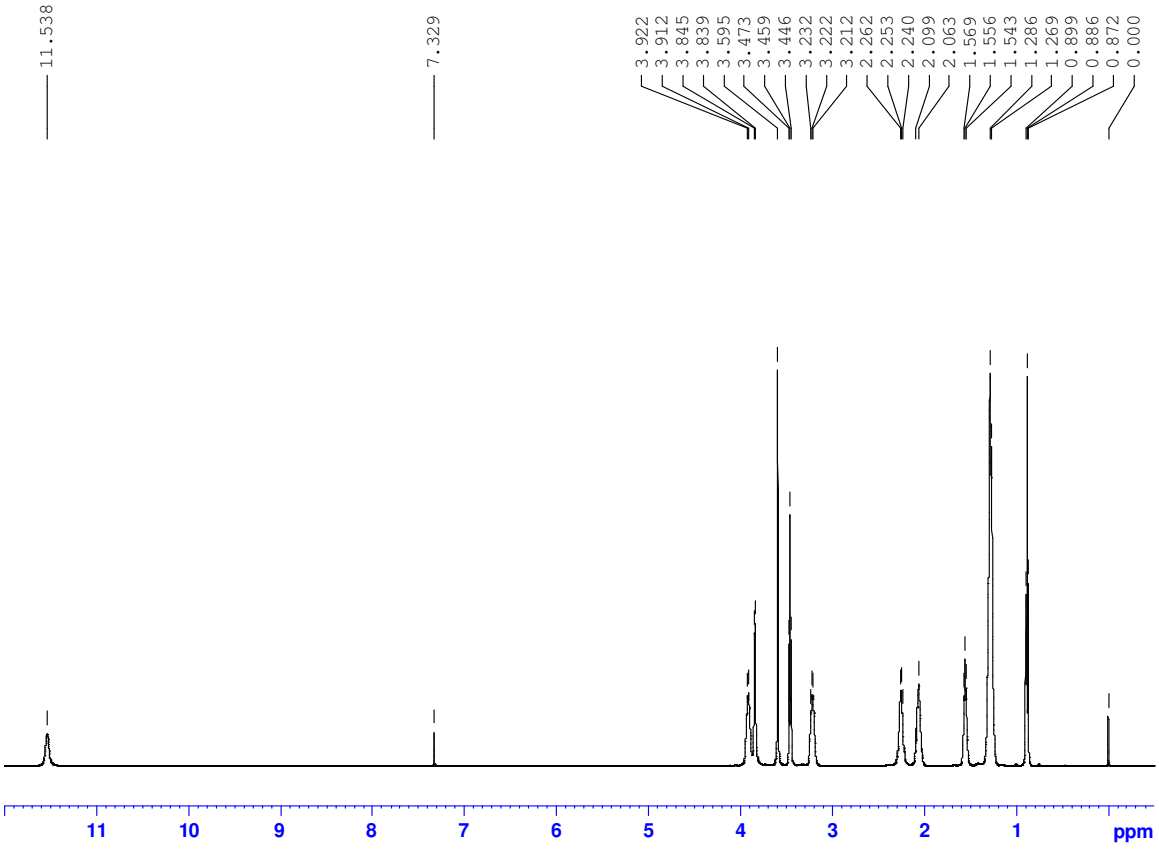
Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of *N*,*N*,*N*,*N*',*N'*,*N*'-hexamethyl-2,2-bis(tertradecyloxymethyl)-1,3-propanediammonium diiodide (**1.12**) in chloroform-*d*



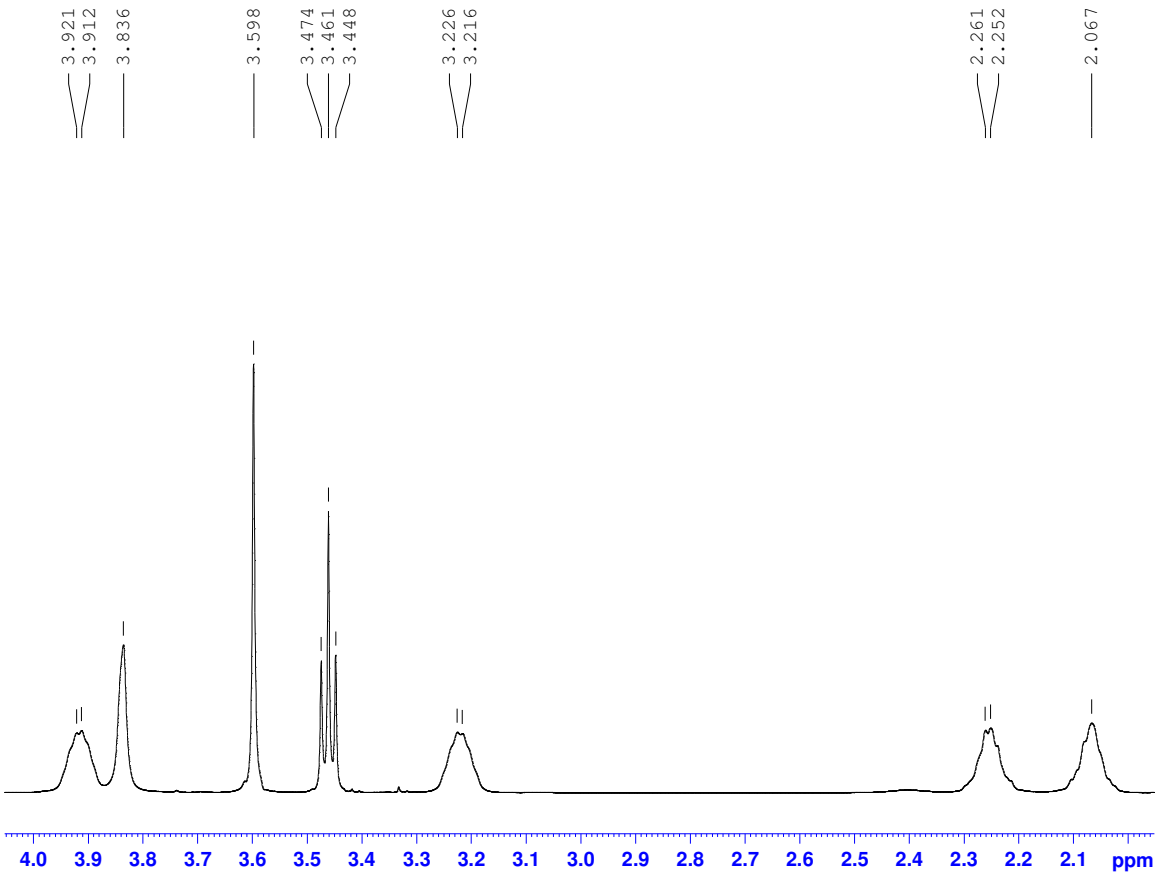
500.1 MHz ^{1}H NMR spectrum of 1,3-bis(1-azacyclopentyl)-2,2-bis(octyloxymethyl)propane dihydrochloride **(1.17)** in chloroform-*d*



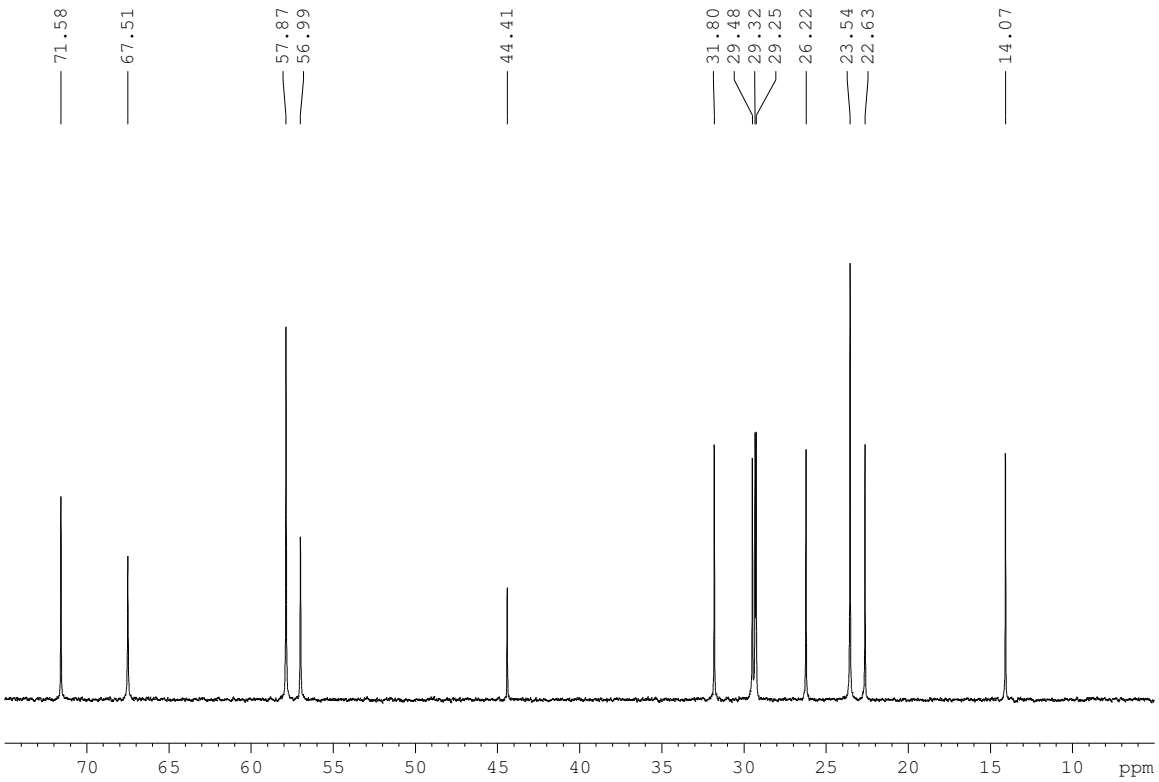
1.17



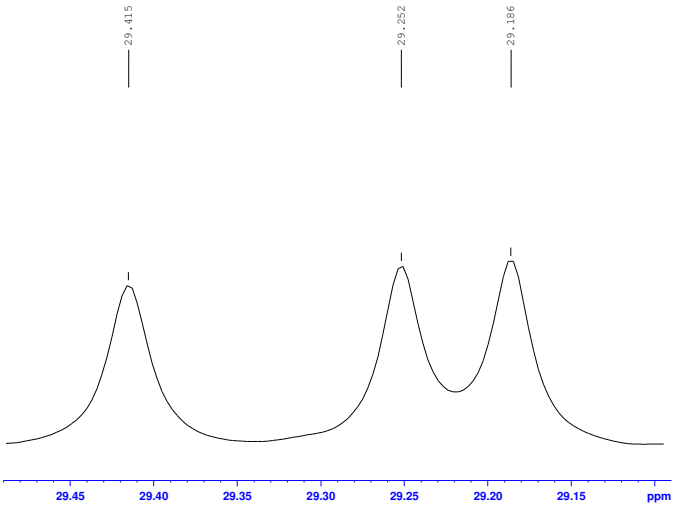
Expansion of part of the 500.1 MHz ^{1}H NMR spectrum of 1,3-bis(1-azacyclopentyl)-2,2-bis(octyloxymethyl)propane dihydrochloride (**1.17**) in $CDCl_3$



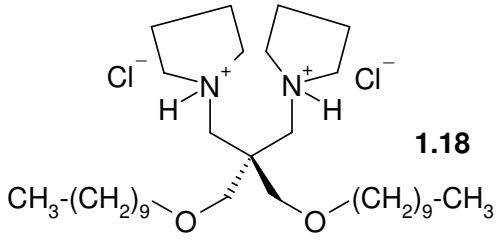
125.7 MHz ^{13}C NMR spectrum of 1,3-bis(1-azacyclopentyl)-2,2-bis(octyloxymethyl)propane dihydrochloride (**1.17**) in chloroform-*d*



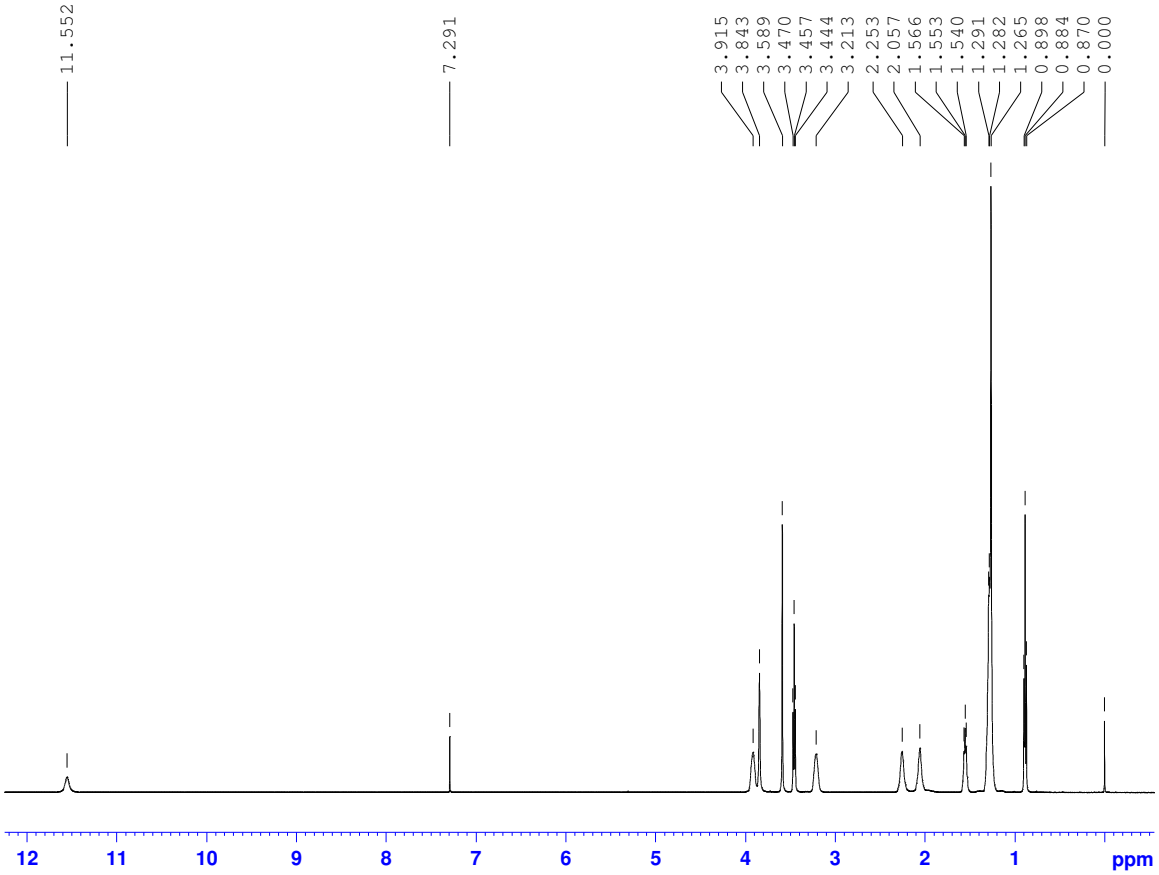
Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of 1,3-bis(1-azacyclopentyl)-2,2-bis(octyloxymethyl)propane dihydrochloride (**1.17**) in $CDCl_3$



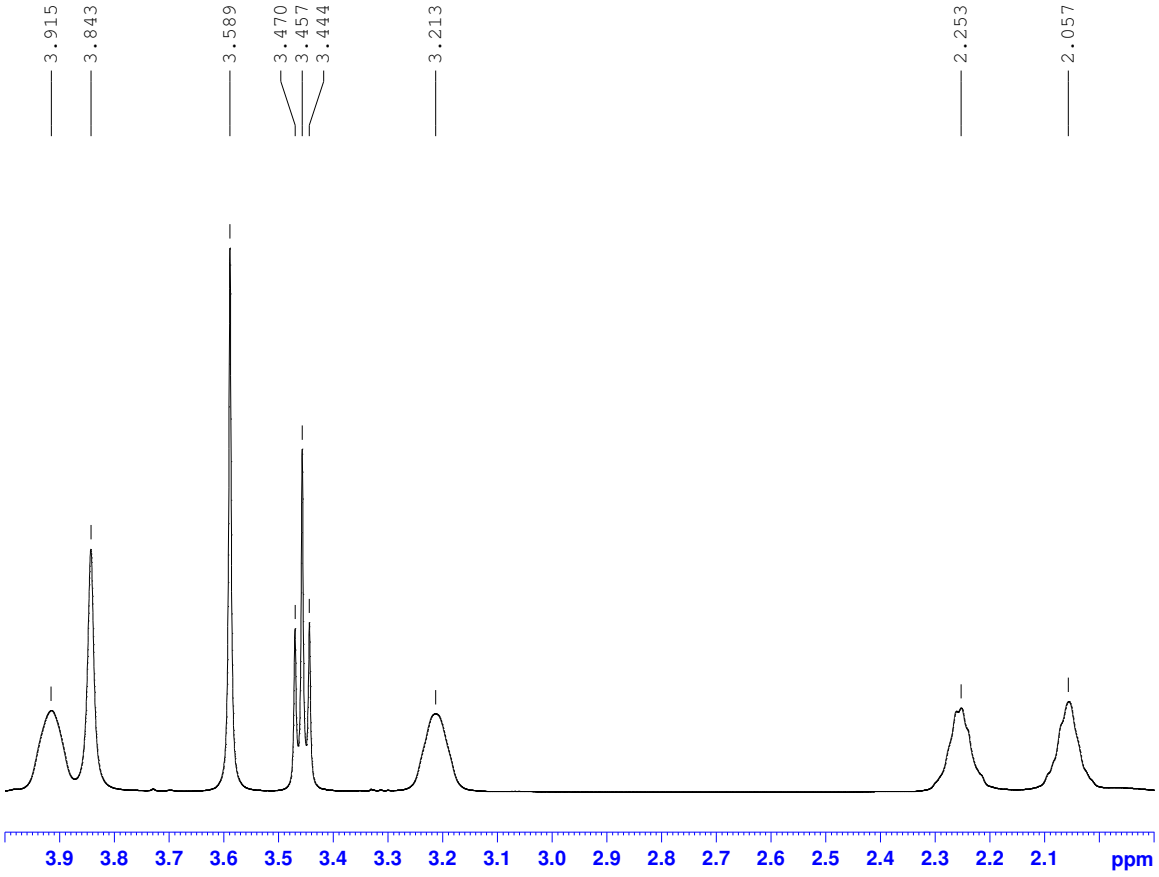
500.1 MHz ^{1}H NMR spectrum of 1,3-bis(1-azacyclopentyl)-2,2-bis(decyloxymethyl)propane dihydrochloride (**1.18**) in $CDCl_3$



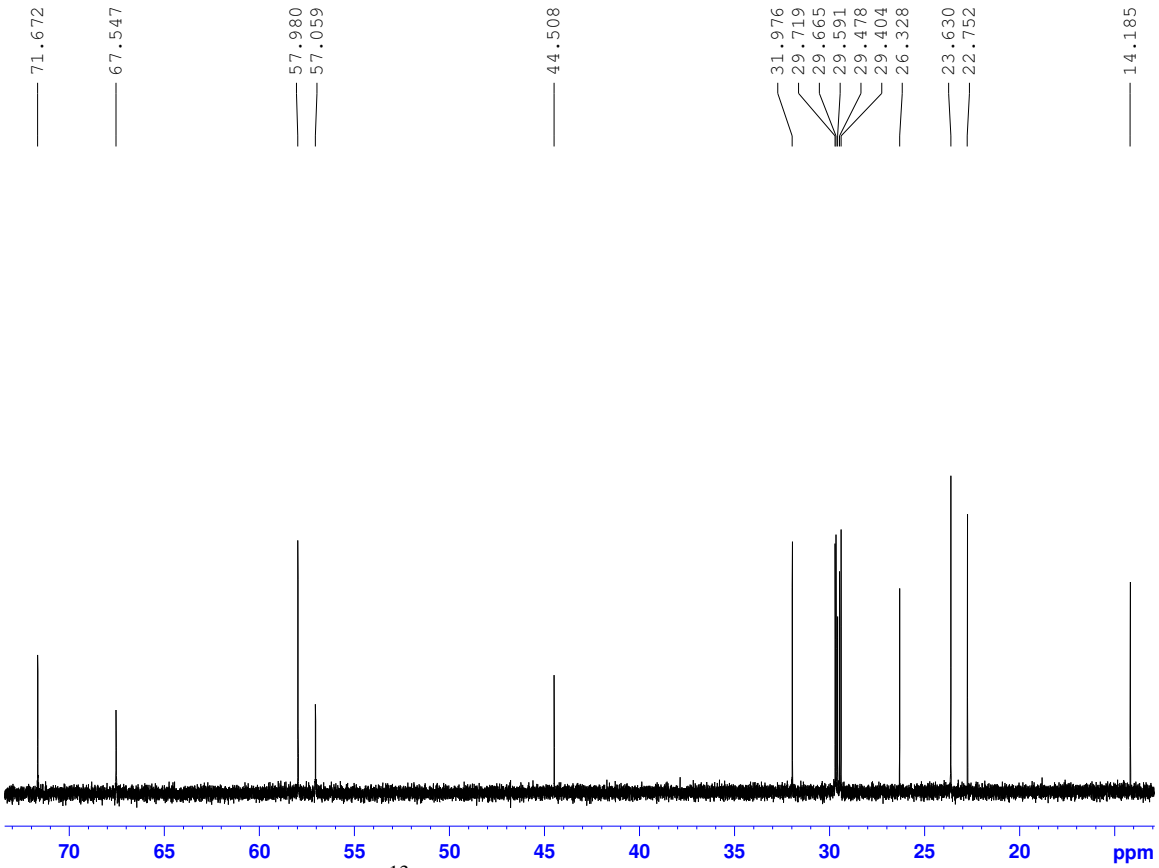
1.18



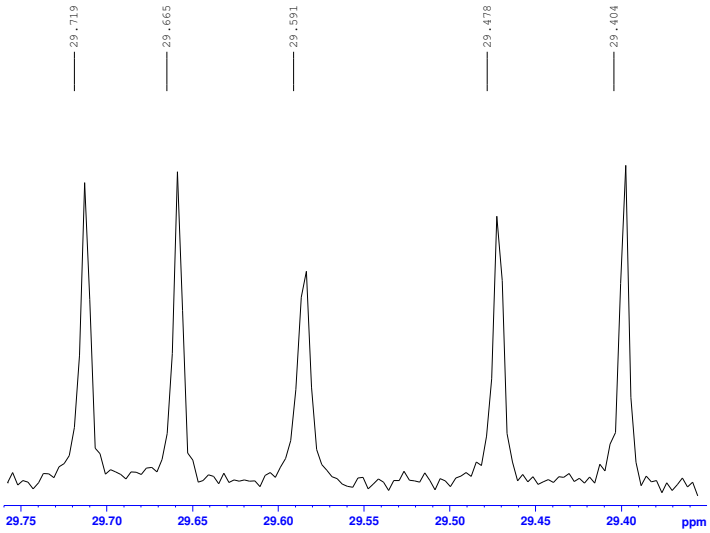
Expansion of part of the 500.1 MHz ^{1}H NMR spectrum of 1,3-bis(1-azacyclopentyl)-2,2-bis(decyloxymethyl)propane dihydrochloride (**1.18**) in $CDCl_3$



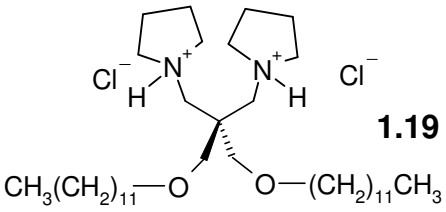
125.7 MHz ^{13}C NMR spectrum of 1,3-bis(1-azacyclopentyl)-2,2-bis(decyloxymethyl)propane dihydrochloride (**1.18**) in $CDCl_3$



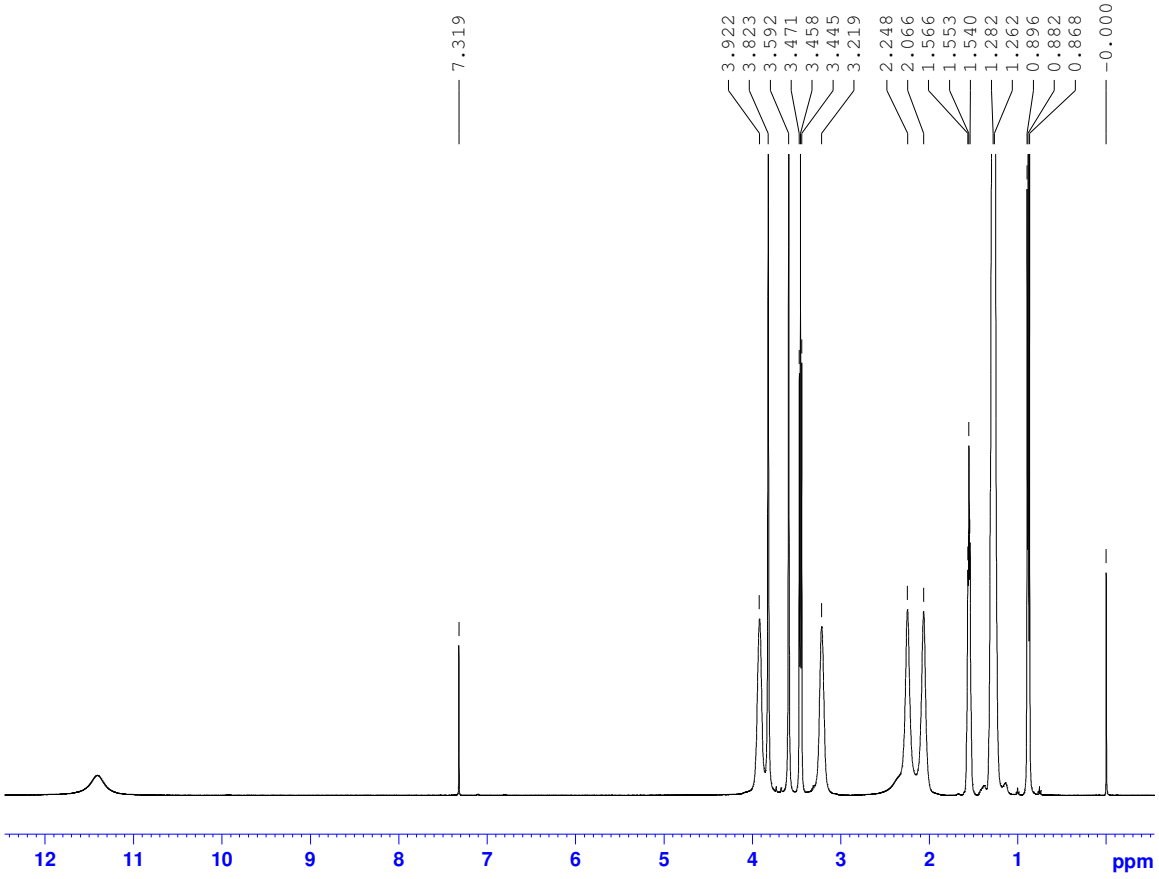
Expansion of part of the 125.7 MHz ^{13}CNMR spectrum of 1,3-bis(1-azacyclopentyl)-2,2-bis(decyloxymethyl)propane dihydrochloride (**1.18**) in $CDCl_3$



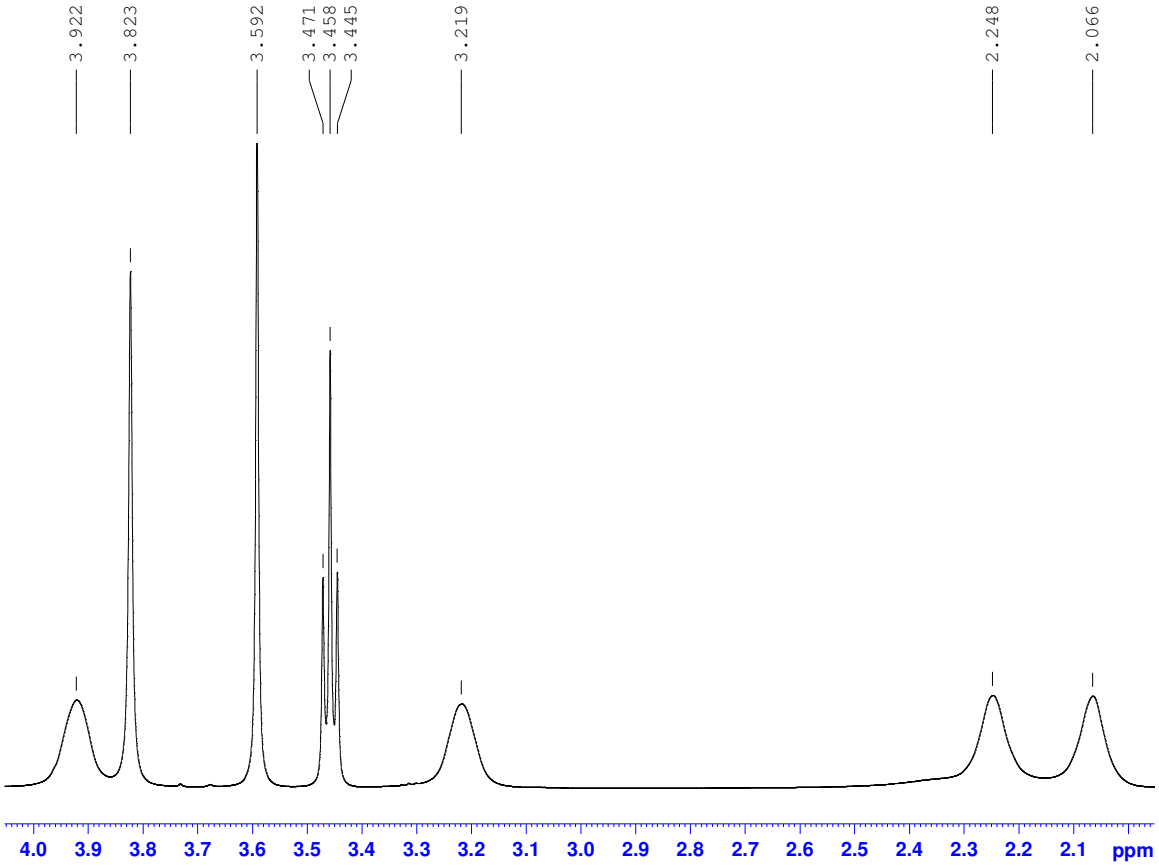
500.1 MHz ^{1}H NMR spectrum of 1,3-bis(1-azacyclopentyl)-2,2-bis(dodecyloxymethyl)propane dihydrochloride (**1.19**) in chloroform-*d*



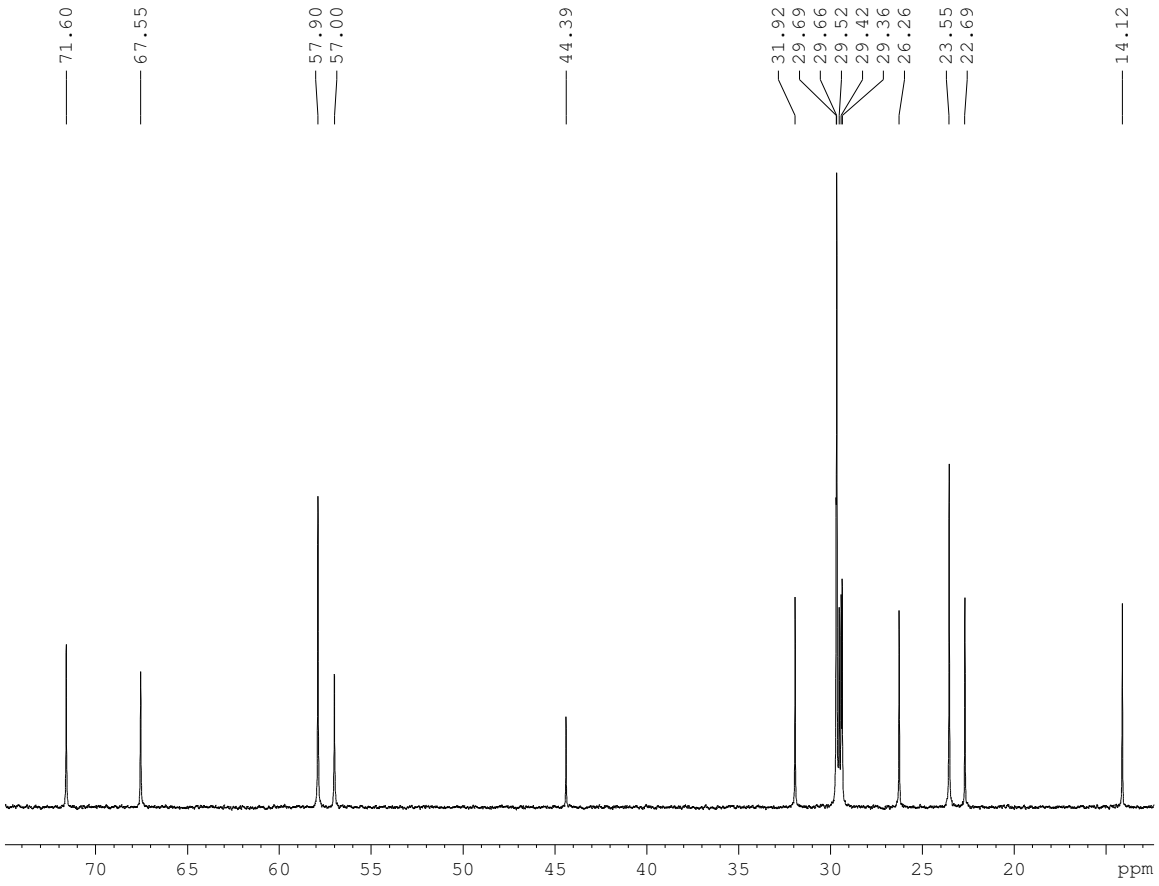
1.19



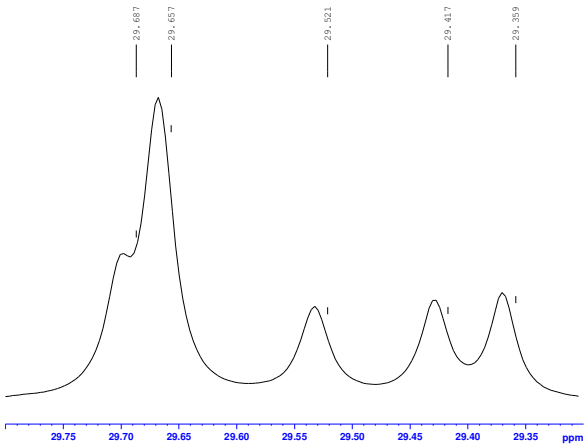
Expansion of part of the 500.1 MHz ^{1}H NMR spectrum of 1,3-bis(1-azacyclopentyl)-2,2-bis(dodecyloxymethyl)propane dihydrochloride (**1.19**) in $CDCl_3$



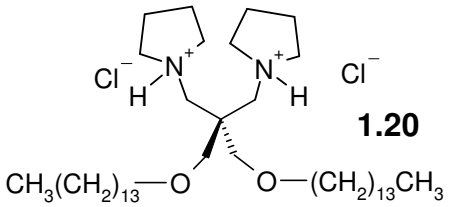
125.7 MHz ^{13}C NMR spectrum of 1,3-bis(1-azacyclopentyl)-2,2-bis(dodecyloxymethyl)propane dihydrochloride (**1.19**) in chloroform-*d*



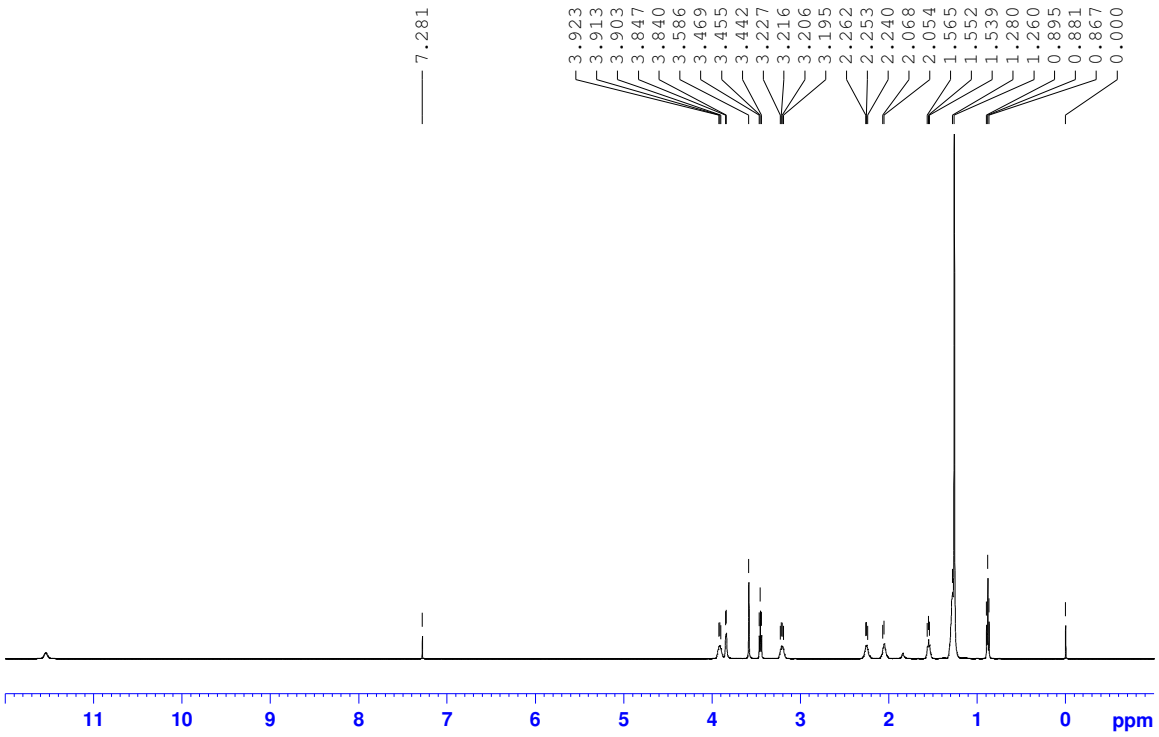
Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of 1,3-bis(1-azacyclopentyl)-2,2-bis(dodecyloxymethyl)propane dihydrochloride (**1.19**) in $CDCl_3$



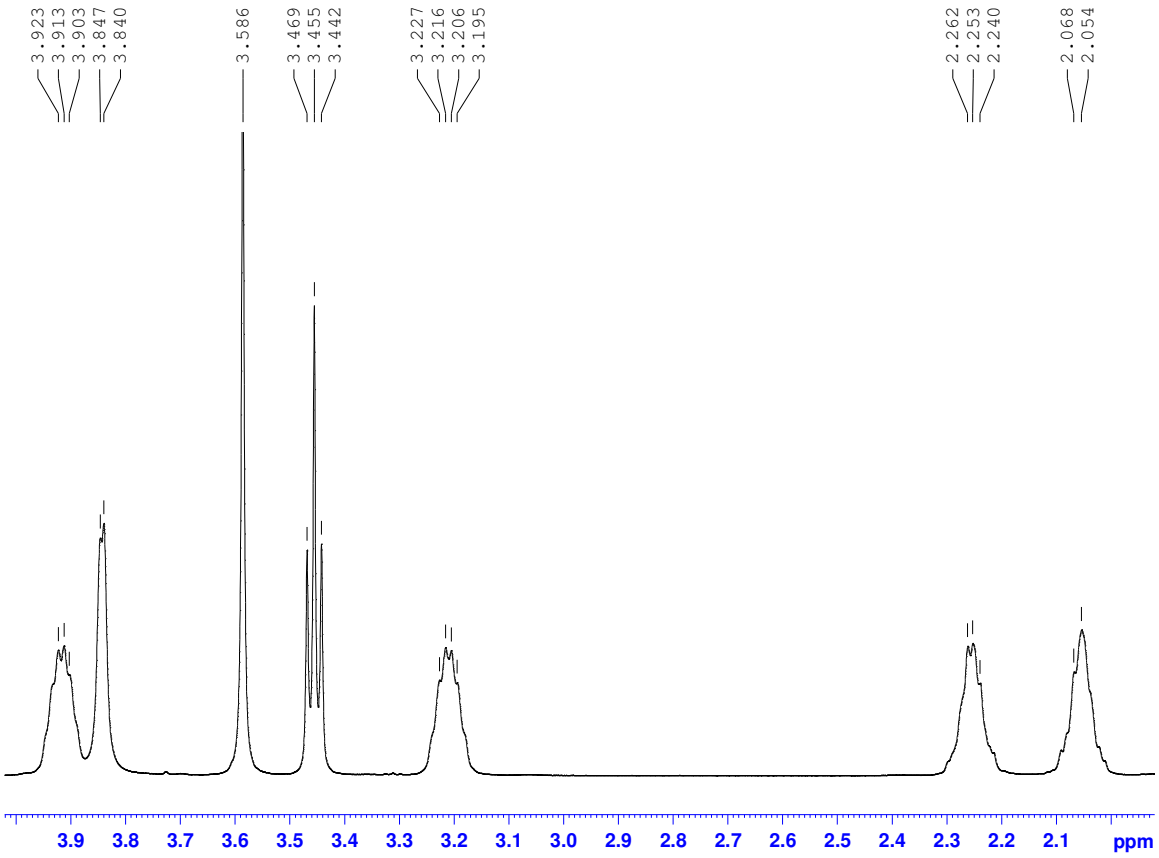
500.1 MHz ^{1}H NMR spectrum of 1,3-bis(1-azacyclopentyl)-2,2-bis(tetradecyloxymethyl)propane dihydrochloride (**1.20**) in chloroform-*d*



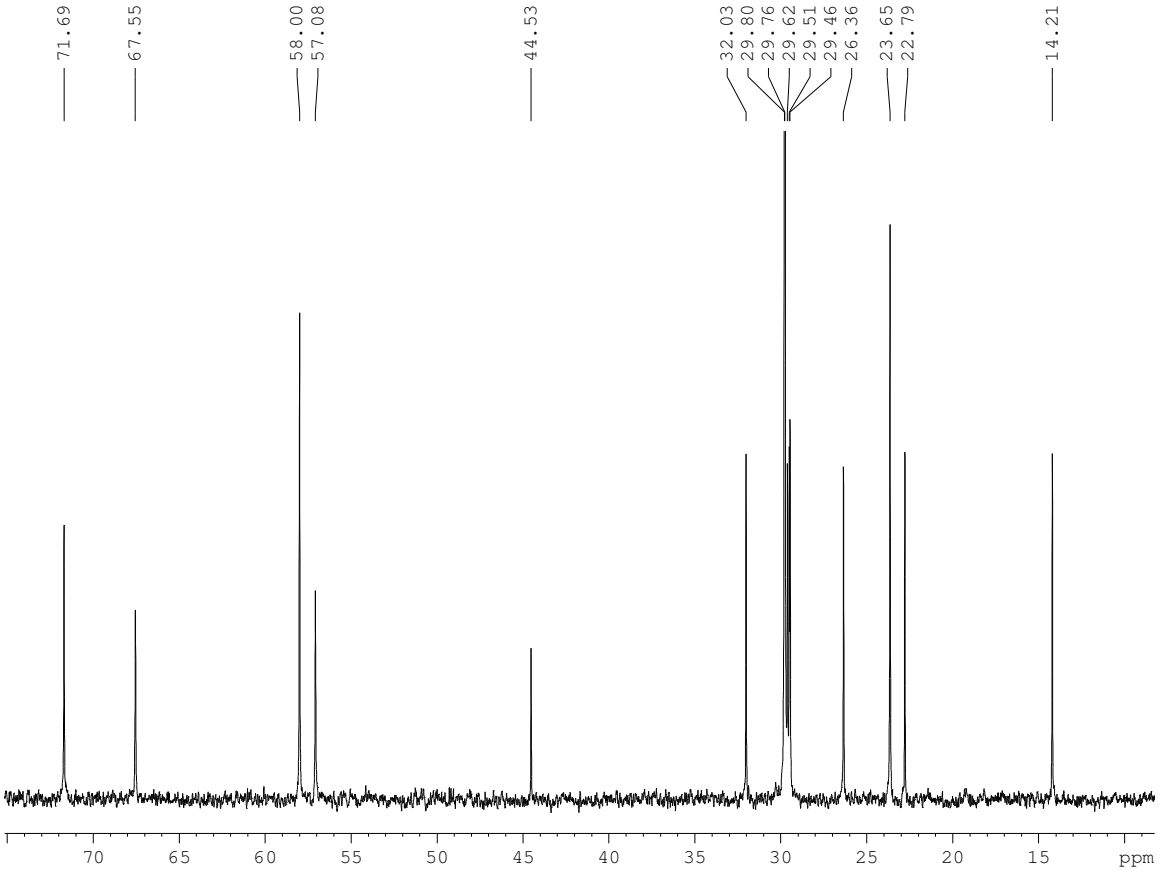
1.20



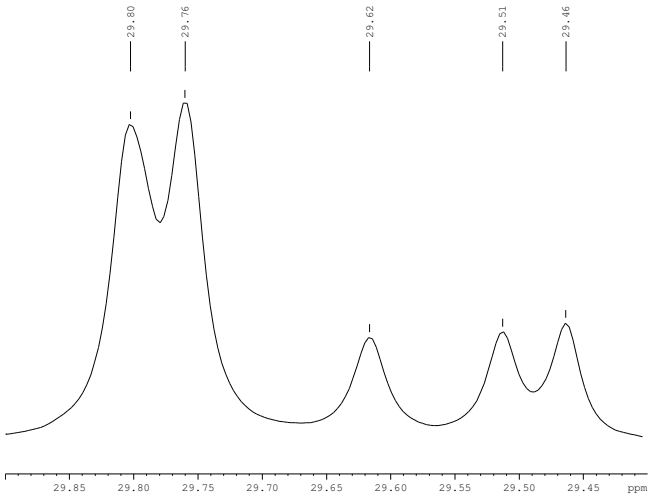
Expansion of part of the 500.1 MHz ^{1}H NMR spectrum of 1,3-bis(1-azacyclopentyl)-2,2-bis(tetradecyloxymethyl)propane dihydrochloride (**1.20**) in $CDCl_3$



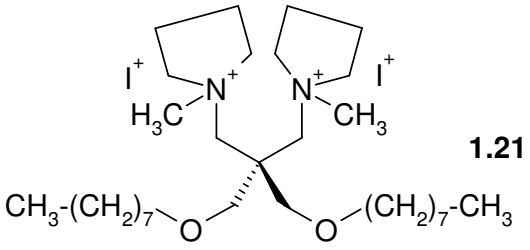
125.7 MHz ^{13}C NMR spectrum of 1,3-bis(1-azacyclopentyl)-2,2-bis(tetradecyloxymethyl)propane dihydrochloride (**1.20**) in chloroform-*d*



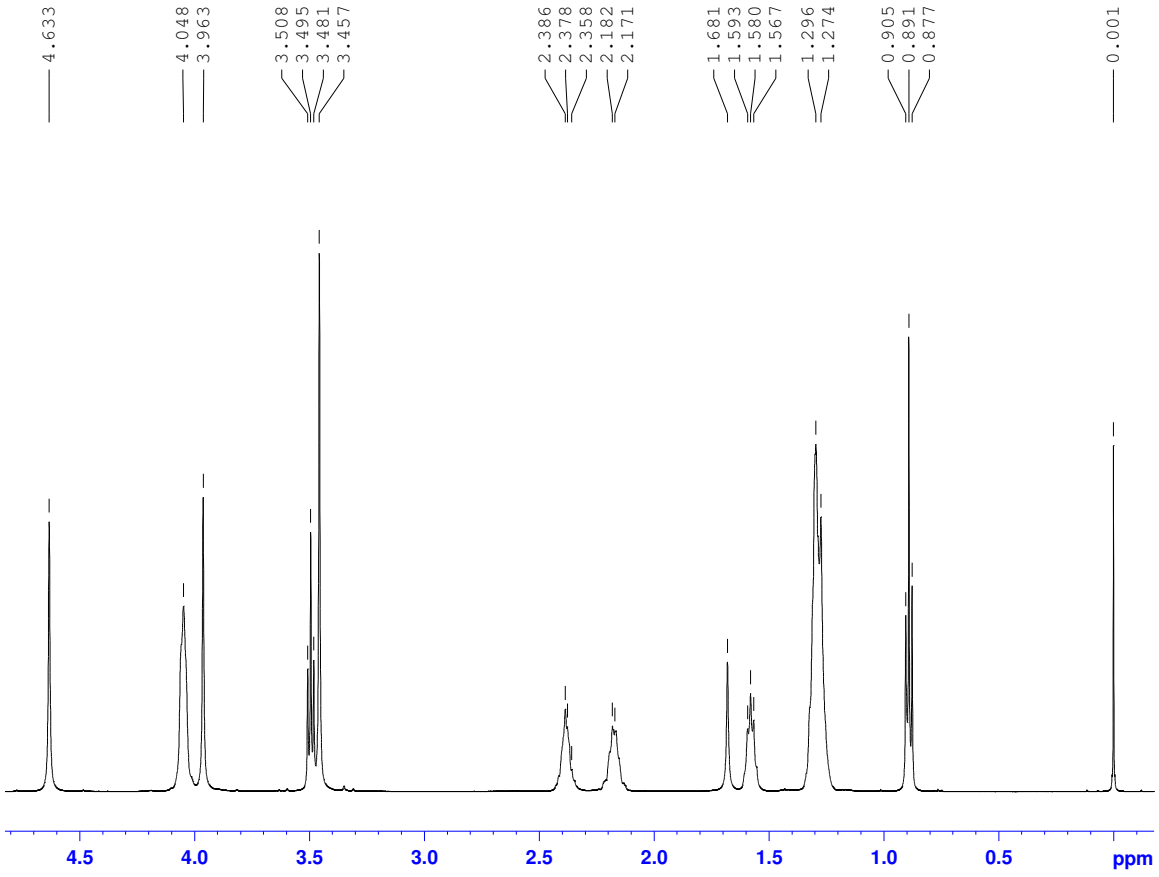
Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of 1,3-bis(1-azacyclopentyl)-2,2-bis(tetradecyloxymethyl)propane dihydrochloride (**1.20**) in $CDCl_3$



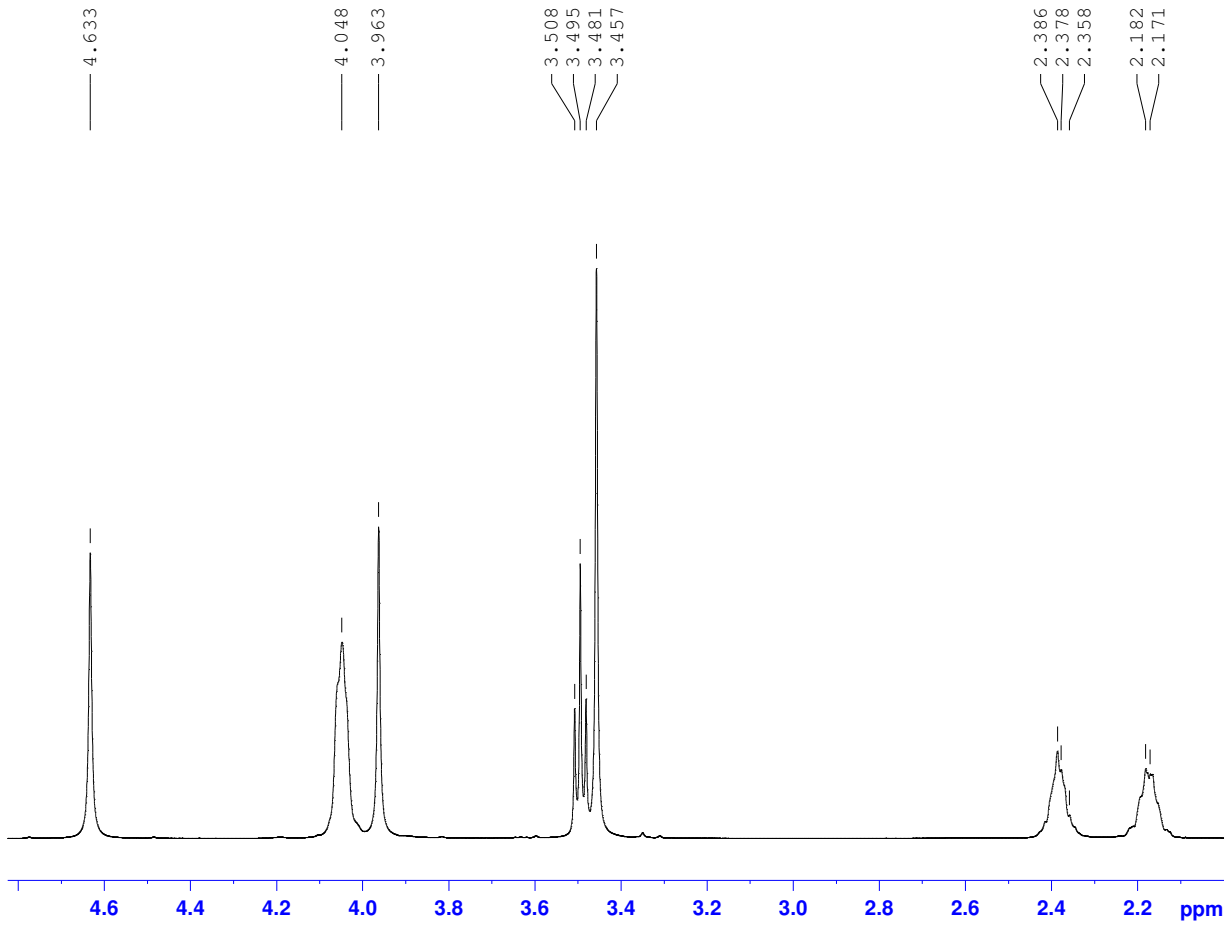
500.1 MHz ^{1}H NMR spectrum of 1,3-bis(1-methyl-1-azoniacyclopentyl)-2,2 bis(octyloxymethyl)propane diiodide (**1.21**) in chloroform-*d*



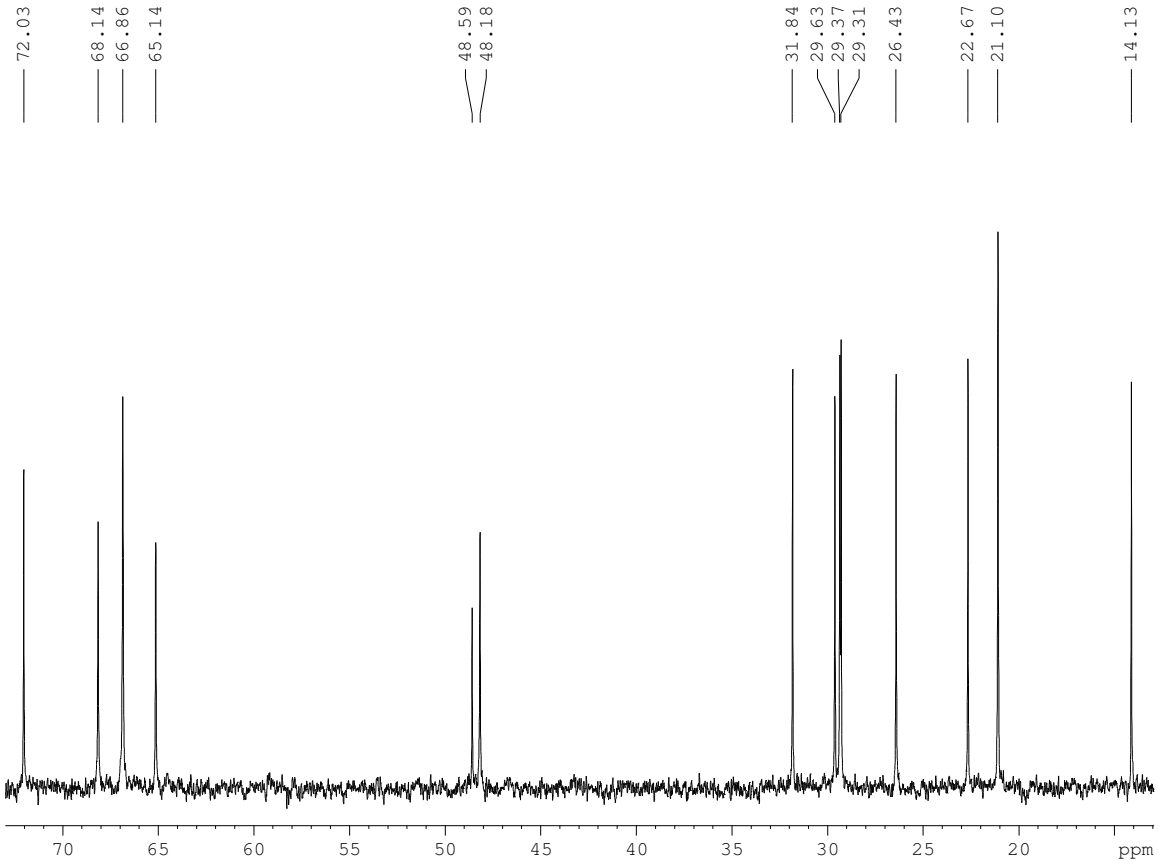
1.21



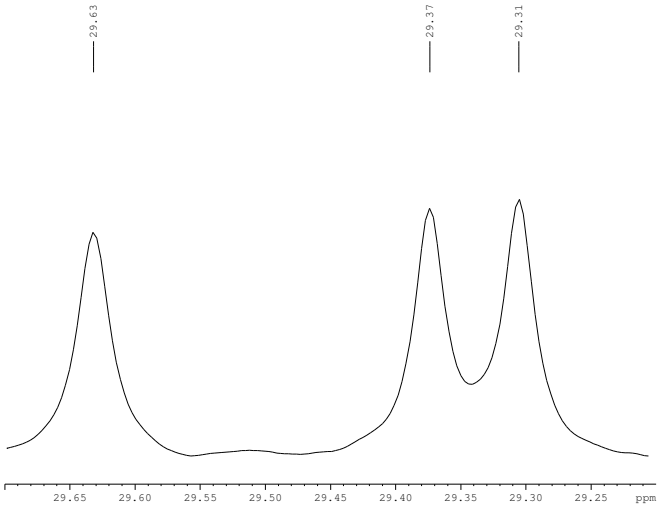
Expansion of part of the 500.1 MHz ^{1}H NMR spectrum of 1,3-bis(1-methyl-1-azoniacyclopentyl)-2,2 bis(octyloxymethyl) propane diiodide (**1.21**) in $CDCl_3$



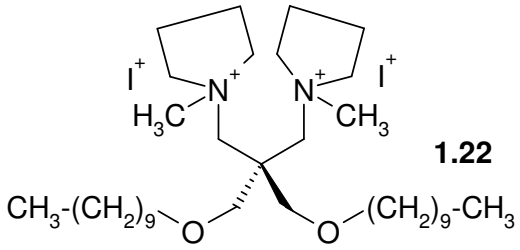
125.7 MHz ^{13}C NMR spectrum of 1,3-bis(1-methyl-1-azoniacyclopentyl)-2,2 bis(octyloxymethyl)propane diiodide (**1.21**) in chloroform-*d*



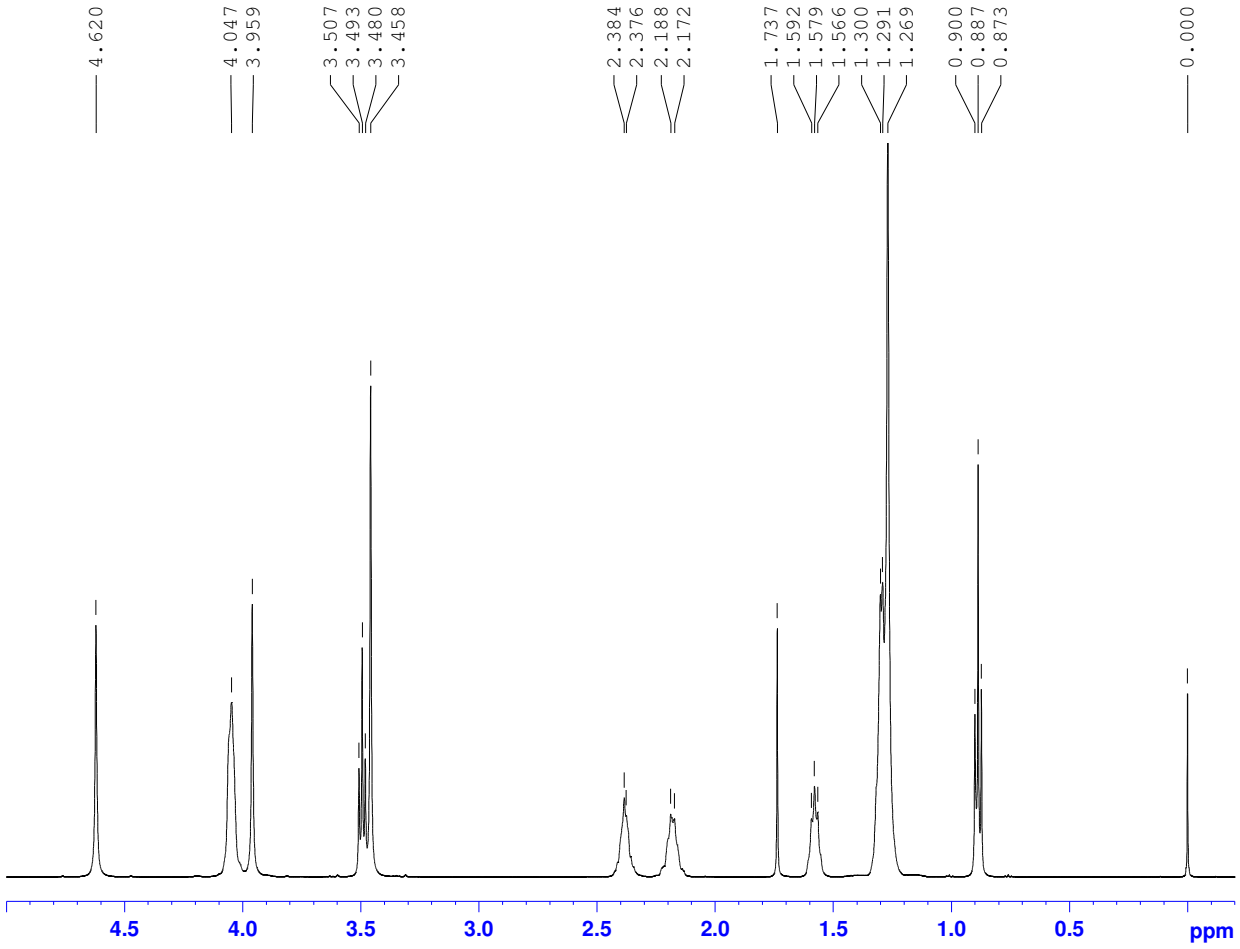
Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of 1,3-bis(1-methyl-1-azoniacyclopentyl)-2,2 bis(octyloxymethyl)propane diiodide (**1.21**) in $CDCl_3$



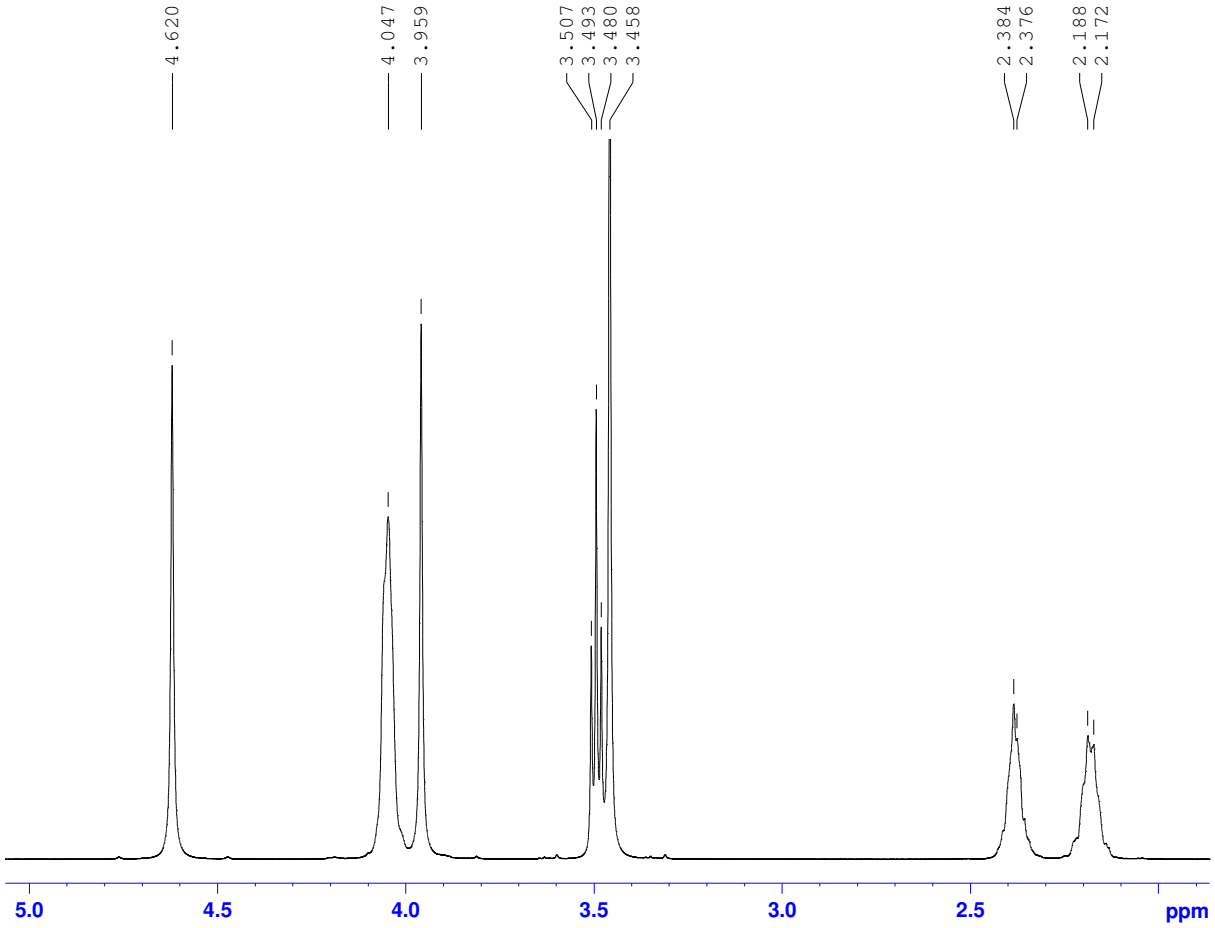
500.1 MHz ^{1}H NMR spectrum of 2,2-bis(decyloxymethyl)-1,3-bis(1-methyl-1-azoniacyclopentyl)-propane diiodide (**1.22**) in chloroform-*d*



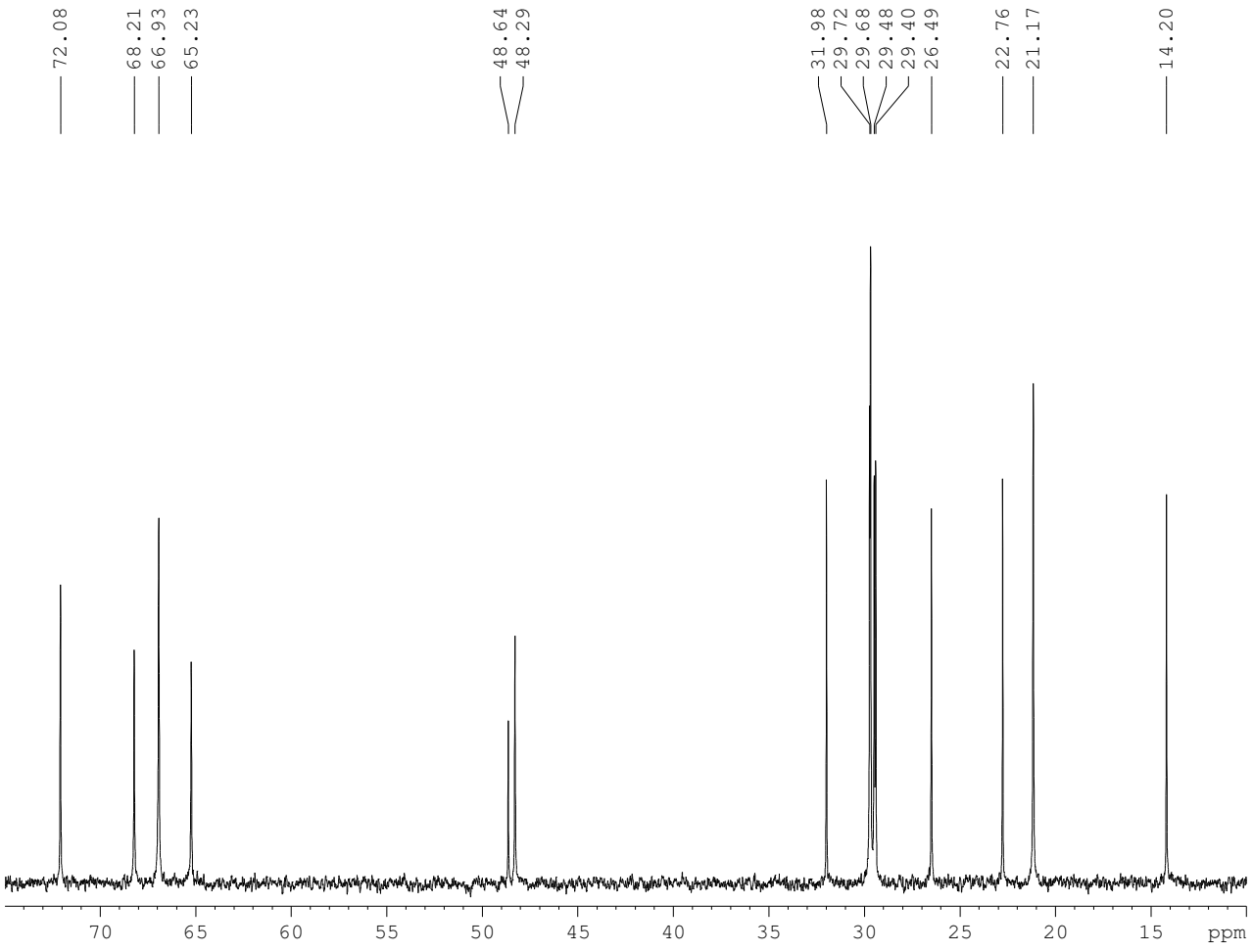
1.22



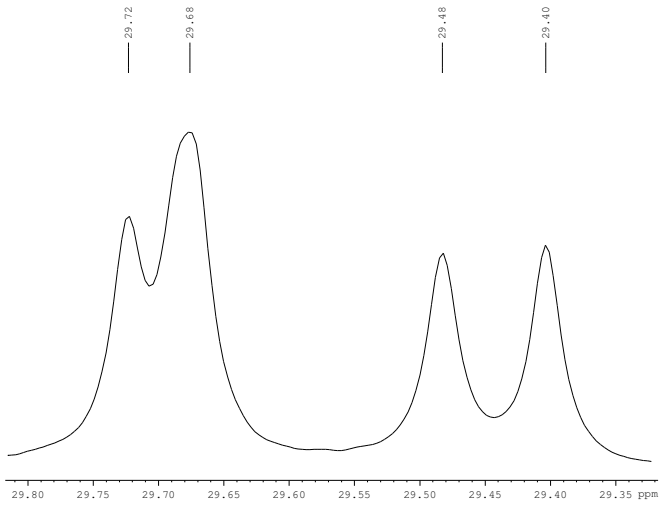
Expansion of part of the 500.1 MHz ^{1}H NMR spectrum of 2,2-bis(decyloxymethyl)-1,3-bis(1-methyl-1-azoniacyclopentyl)propane diiodide (**1.22**) in $CDCl_3$



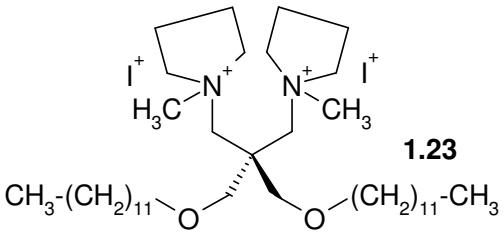
125.7 MHz ^{13}C NMR spectrum of 2,2-bis(decyloxymethyl)-1,3-bis(1-methyl-1-azoniacyclopentyl)-propane diiodide (**1.22**) in chloroform-*d*



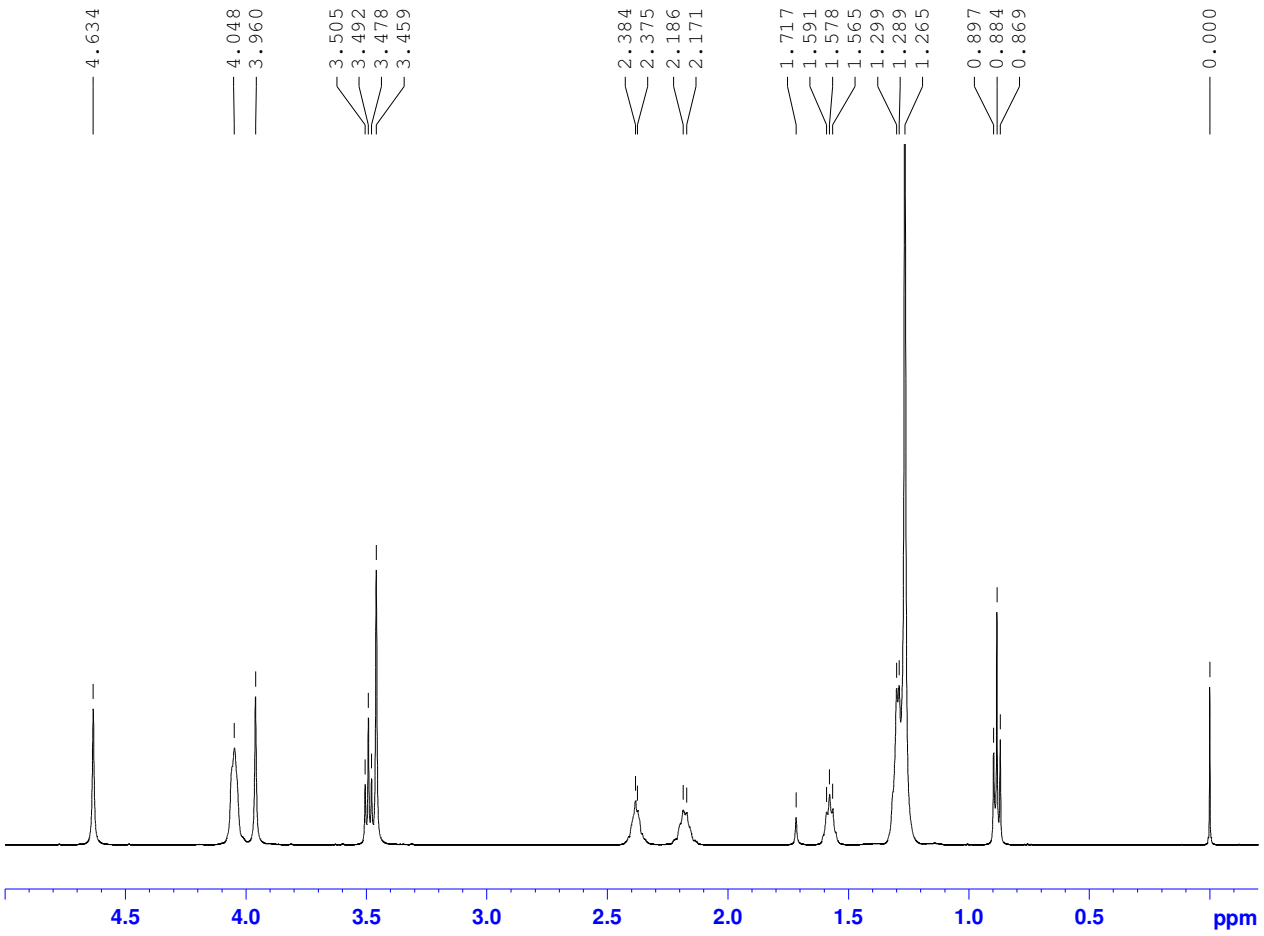
Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of 2,2 bis(decyloxymethyl)-1,3-bis(1-methyl-1-azoniacyclopentyl)propane diiodide (**1.22**) in $CDCl_3$



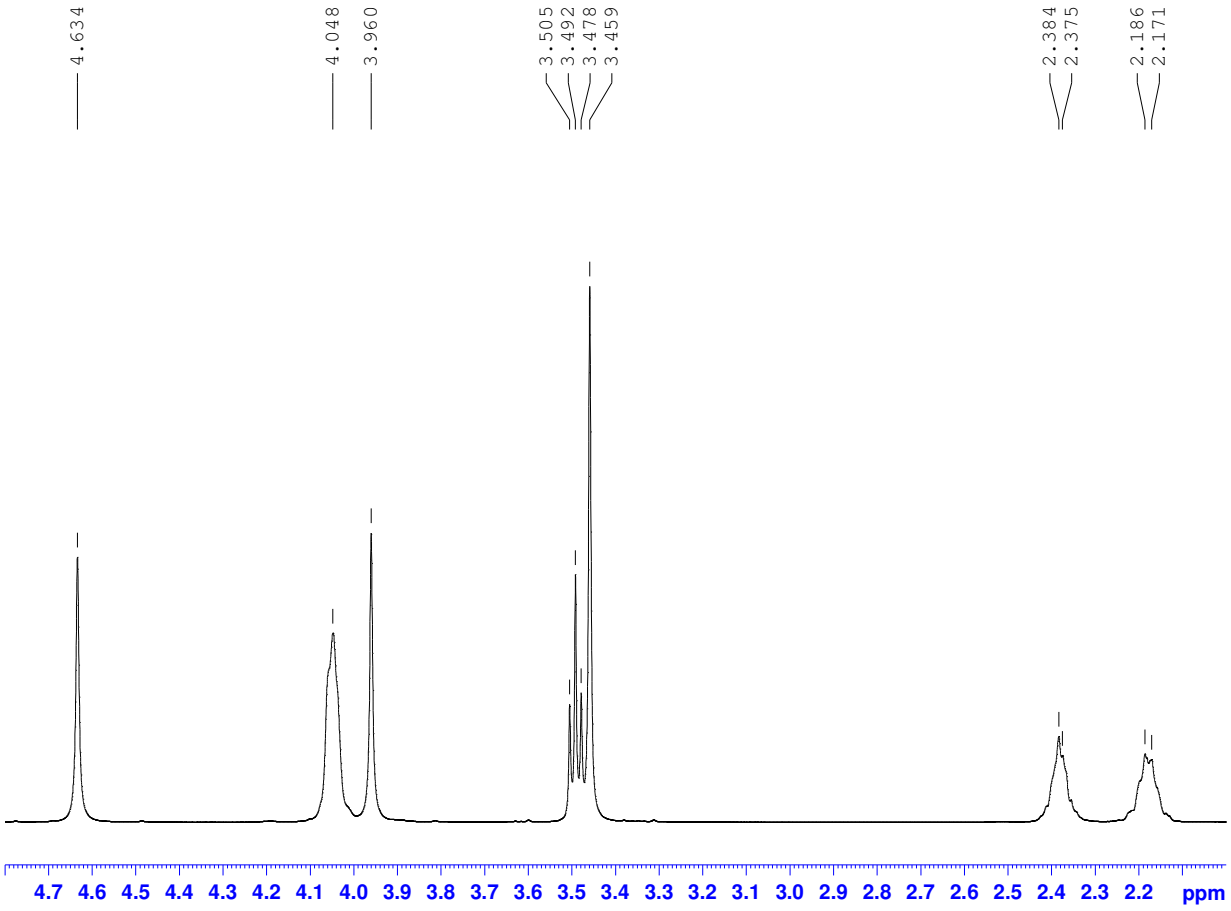
500.1 MHz ^{1}H NMR spectrum of 2,2-bis(dodecyloxymethyl)-1,3-bis(1-methyl-1-azoniacyclopentyl)propane diiodide (**1.23**) in chloroform-*d*



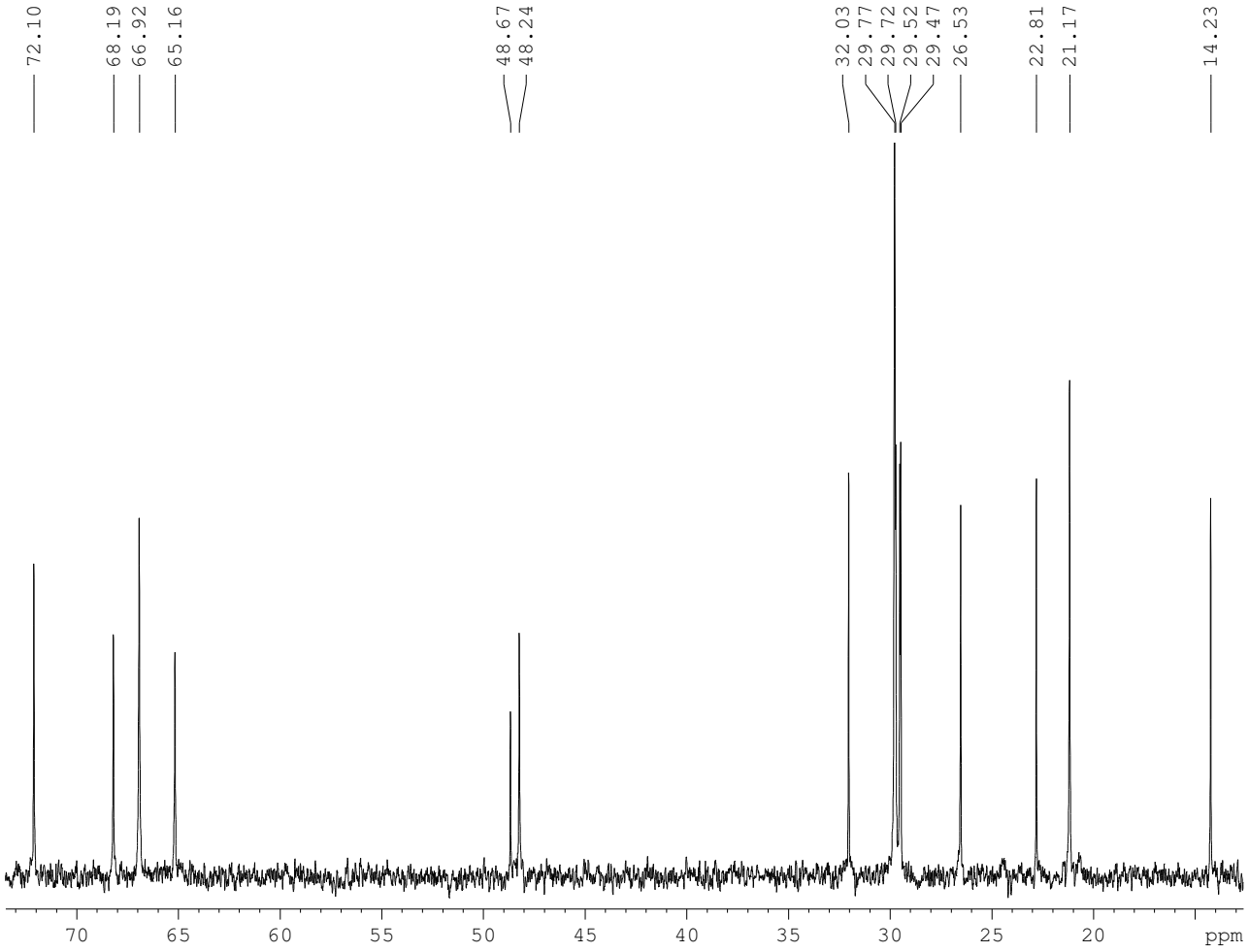
1.23



Expansion of part of the 500.1 MHz ^{1}H NMR spectrum of 2,2-bis(dodecyloxymethyl)-1,3-bis(1-methyl-1-azoniacyclopentyl)propane diiodide (**1.23**) in chloroform-*d*



125.7 MHz ^{13}C NMR spectrum of 2,2-bis(dodecyloxymethyl)-1,3-bis(1-methyl-1-azoniacyclopentyl)propane diiodide (**1.23**) in chloroform-*d*



Expansion of part of the 125.7 MHz ^{13}C NMR spectrum of 2,2-bis(dodecyloxymethyl)-1,3-bis(1-methyl-1-azoniacyclopentyl)propane diiodide (**1.23**) in $CDCl_3$