ELECTRONIC SUPPLEMENTARY INFORMATION

Synthesis of Inositol Phosphate-Based Competitive Antagonists of Inositol 1,4,5-Trisphosphate Receptors

Vera Konieczny,[a] John. G. Stefanakis,[b] Efstratios D. Sitsanidis,[b] Natalia-Anastasia T. Ioannidou,[b] Nikolaos V. Papadopoulos,[b] Konstantina C. Fylaktakidou,[c] Colin W. Taylor,[a]* and Alexandros E. Koumbis[b]*

[a]*Department of Pharmacology, Tennis Court Road, Cambridge, CB2 1PD, United Kingdom. E-mail: cwt1000@cam.ac.uk*
[b]*Laboratory of Organic Chemistry, Chemistry Department, Aristotle University of Thessaloniki, 54124 Thessaloniki, Greece. E-mail: akoumbis@chem.auth.gr*
[c]*Laboratory of Organic, Bioorganic and Natural Product Chemistry, Molecular Biology and Genetics Department, Democritus University of Thrace, 68100 Alexandroupolis, Greece.*

Table of contents

Table S1. Summary of dimerization reactions of **21** with malonic (**M**) and succinic acid (**S**).

Entry	Acid	Reagents	Solvent	Products (yields %)[a]
1	**M**	DCC/DMAP[b]	CH_2Cl_2	**22** (37)
2	**M**	DCC/DMAP[b]	Et_2O	**22** (12)
3	**M**	DIC/DMAP[b]	CH_2Cl_2	**22** (41)
4	**M**	DCC/DIPEA[b]	CH_2Cl_2	**24b** (6)
5	**M**	DCC[c]	Et_2O	**23a** (62), **24b** (12)[d]
6	**M**	DIC[c]	Et_2O	**23a** (37), **24a** (7)
7	**M**	DIC[c]	CH_2Cl_2	**23a** (39), **24a** (7)
8	**S**	DCC[c]	Et_2O	recovered **21** (100)
9	**S**	DCC/DMAP[b]	CH_2Cl_2	**23b** (56), **24c** (28)[d]
10	**S**	EDC/DMAP[b]	CH_2Cl_2	**23b** (55)

[a] Yields refer to pure products obtained after chromatographic purification, unless otherwise stated.
[b] 4 equivalents of carbodiimide and 0.4 equivalents of base were used.
[c] 4 equivalents of carbodiimide were used.
[d] These compounds were obtained as an inseparable mixture and their ratio was determined from the ^{1}H NMR spectrum.

Table S2. Ca^{2+} release evoked by inositol phosphates **1**-**4** and their binding to IP_3R1.[a]

Compound	Ca^{2+} release			Binding			
	pEC_{50} /M	EC_{50} nM	Maximal release %	pK_d /M	K_d nM	pEC_{50}-pK_d /M	EC_{50} /K_d
(1,4,5)IP_3 (**1**)	6.97 ± 0.04	107	68 ± 5	6.90 ± 0.19	127	0.07 ± 0.06	0.85
(1,3,4,6)IP_4 (**2**)	5.64 ± 0.07*	2291	68 ± 5	5.35 ± 0.07*	4490	0.29 ± 0.08	0.51
2-*O*-butyryl (1,3,4,6)IP_4 (**3**)	5.21 ± 0.01*	7934	49 ± 6*	5.32 ± 0.06*	4800	-	-
(1,2,3,4,6)IP_5 (**4**)	-	-	0	4.46 ± 0.03*	22900	-	-

[a]Summary results from Fig. 2B, C show Ca^{2+} release evoked by maximally effective concentrations of each analog and pEC_{50} values as means ± s.e.m., and EC_{50} values determined in Ca^{2+} release experiments (n = 3). pK_d (means ± s.e.m.) and K_d values were derived from equilibrium-competition binding experiments (n = 3). pEC_{50}-pK_d values (means ± s.e.m.) were calculated as described in the Experimental Section. Statistical differences were determined by one-way ANOVA and Tukey's post hoc test and refer to (1,4,5)IP_3 (**1**), **P* < 0.05. Hill coefficients were not significantly different.

Table S3. (1,2,3,4,6)IP_5 (**4**) and a dimer of (1,2,3,4,6)IP_5 (**6**) are competitive antagonists of IP_3R.[a]

Compound	pEC_{50} /M	ΔpEC_{50} /M	EC_{50} nM	Maximal release %	n_H	K_d nM
control	7.33 ± 0.21	-	47	83 ± 1	1.74 ± 0.38	-
+(1,2,3,4,6)IP_5 (**4**)	6.94 ± 0.22*	0.38 ± 0.04	115	78 ± 2	1.36 ± 0.13	69
+(1,2,3,4,6)IP_5 dimer (**6**)	6.01 ± 0.22*	1.32 ± 0.01	977	75 ± 3	1.36 ± 0.21	5

[a] Summary results from Figure 3B. Ca^{2+} release evoked by maximally effective concentrations of (1,4,5)IP_3 alone (control) or the presence of 100 μM of the indicated analogs, pEC_{50} and ΔpEC_{50}s ($pEC_{50}^{control}$ - $pEC_{50}^{antagonist}$) values and Hill coefficients (n_H) (means ± s.e.m.), and EC_{50} values for (1,4,5)IP_3 (n = 3). K_d is the equilibrium dissociation constant for the antagonist calculated from the Ca^{2+} release experiments as described in the Experimetnal Section. Statistical differences were determined by one-way ANOVA and Tukey's post hoc test and refer to the control antagonist, **P* < 0.05.

1,6:3,4-bis-[*O*-(2,3-dimethoxybutane-2,3-diyl)]-*myo*-inositol (14). Diol **14** was prepared from *myo*-inositol (**13**) in 33% yield, following a known procedure.[S1]

Tetrasodium 1,3,4,6-*myo*-inositol tetrakisphosphate (2). Phosphate **2** was prepared from diol **14**, following a known four-step reaction sequence.[S2]

2-*O*-benzyl-1,6:3,4-bis-[*O*-(2,3-dimethoxybutane-2,3-diyl)]-*myo*-inositol (15a). Pentaol **15a** was prepared from *myo*-inositol (**13**), following a known three-step reaction sequence.[S3]

Decabenzyl 1,2,3,4,6-(5-*O*-benzyl-*myo*-inosityl) pentakisphosphate (15b). Benzyl phosphate **15b** was prepared from pentaol **15a** according to General Procedure E. Yield: 73%. Colorless thick oil. R_f = 0.15 (hexanes/EtOAc 1:1). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 (d, J = 7.3 Hz, 2H, 2×ArH), 7.23–7.02 (m, 49H, 49×ArH), 6.84 (d, J = 7.1 Hz, 4H, 4×ArH), 5.56 (br d, $^3J_{HP}$ = 8.7 Hz, 1H, H-2), 5.14–4.75 (m, 22H, 10×CH_2Ph & H-4 & H-6), 4.51 (dd, J = 11.7, 9.5 Hz, 2H, CH_2Ph), 4.32 (br t, $^3J_{HP}$ = 9.3 Hz, 2H, H-1 & H-3), 3.46 (t, J = 9.4 Hz, 1H, H-5) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 137.7 (C-*i*Ar), 135.8 (d, $^3J_{CP}$ = 7.2 Hz, 2×C-*i*Ar), 135.6 (d, $^3J_{CP}$ = 9.0 Hz, 2×C-*i*Ar), 135.54 (d, $^3J_{CP}$ = 6.8 Hz, 4×C-*i*Ar), 135.49 (d, $^3J_{CP}$ = 7.2 Hz, 2×C-*i*Ar), 128.4, 128.30, 128.28, 128.21, 128.16, 128.07, 128.06, 128.03, 127.96, 127.8, 127.7, 127.1 (C-*o*Ar, C-*m*Ar, C-*p*Ar), 78.8 (C-5), 76.7 (t, $^3J_{CP}$ = 6.1 Hz, C-4 & C-6), 76.0 (d, $^3J_{CP}$ = 5.0 Hz, C-2), 74.6 (*C*H_2Ph), 73.7 (br s, C-1 & C-3), 70.02 (d, $^2J_{CP}$ = 5.6 Hz, 2×*C*H_2Ph), 69.74 (d, $^2J_{CP}$ = 6.6 Hz, 2×*C*H_2Ph), 69.69 (d, $^2J_{CP}$ = 6.2 Hz, 2×*C*H_2Ph), 69.46 (d, $^2J_{CP}$ = 5.7 Hz, 2×*C*H_2Ph), 69.25 (d, $^2J_{CP}$ = 5.2 Hz, 2×*C*H_2Ph) ppm. ^{31}P NMR (202 MHz, $CDCl_3$, ^{31}P-^{1}H decoupled): –0.77 (2P), –1.53 (2P), –2.81 (1P) ppm. HRMS (ESI) calcd. for $C_{83}H_{83}NaO_{21}P_5$ $[M+Na]^+$ 1593.4007; found 1593.4019.

Pentasodium 1,2,3,4,6-*myo*-inosityl pentakisphosphate (3). Phosphate **3** was prepared from benzyl phosphate **15b**, according to General Procedure F. Reaction time: 48 h. Yield: 100%. White amorphous solid. ^{1}H NMR (500 MHz, D_2O) δ 4.72 (br d, $^3J_{HP}$ = 8.8 Hz, 1H, H-2), 4.24 (q, $^3J_{HP}$ = $^3J_{HH}$ = 9.1 Hz, 2H, H-4 & H-6), 4.04 (br t, $^3J_{HP}$ = 8.9 Hz, 2H, H-1 & H-3), 3.55 (t, J = 8.8 Hz, 1H, H-5) ppm. ^{13}C NMR (126 MHz, D_2O) δ 76.5 (br t, $^2J_{CP}$ = 5.9 Hz, C-4 & C-6), 75.3 (br d, $^2J_{CP}$ = 5.7 Hz, C-2), 73.3 (br s, C-1 & C-3), 72.9 (br s, C-5) ppm. ^{31}P NMR (202 MHz, D_2O) ^{31}P-^{1}H decoupled): 0.77 (2P), 0.63 (2P), –0.11 (1P) ppm. HRMS (ESI) calcd. for $C_6H_{12}Na_4O_{21}P_5$ $[M–Na]^–$ 666.8155; found 666.8147.

2-*O*-*p*-Methoxybenzyl-1,6:3,4-bis-[*O*-(2,3-dimethoxybutane-2,3-diyl)]-*myo*-inositol (16a). A solution of diol **14** (410 mg, 1 mmol) in dry DMF (10 mL) was cooled to 0 °C under an Ar atmosphere. 90% NaH (30 mg, 1.1 mmol) was added in one portion and the resulting slurry was stirred at the same temperature for 1 h. Then, PMBCl (150 mL, 1.1 mmol) was added dropwise and the mixture was stirred for 12 h, while it was left to warm up to room temperature. MeOH (0.2 mL) was added and the mixture was stirred at room temperature for 1 h. CH_2Cl_2 (10 mL) was added and the resulting solution was washed with H_2O (10 mL). The aqueous phase was back-extracted with CH_2Cl_2 (10 mL) and the combined organic phases were washed with saturated brine (20 mL), dried over Na_2SO_4, and concentrated *in vacuo*. The residue was purified with flash column chromatography (hexanes/EtOAc 7:1 to 2:1) to give PMB-ether **16a** (355 mg, 67%) as a white amorphous solid. R_f = 0.18 (hexanes/EtOAc 1:1). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (d, J = 8.4 Hz, 2H, 2×ArH), 6.85 (d, J = 8.4 Hz, 2H, 2×ArH), 4.78 (s, 2H, CH_2Ar), 4.04 (t, J = 9.8 Hz, 2H, H-4 & H-6), 3.80 (s, 3H, CH_3OAr), 3.78 (t, J = 1.8 Hz, 1H, H-2), 3.65 (t, J = 9.4 Hz, 1H, H-5), 3.52 (dd, J = 10.3, 1.8 Hz, 2H, H-1 & H-3), 3.27 (s, 6H, 2×OCH_3), 3.22 (s, 6H, 2×OCH_3), 2.66 (br s, 1H, OH), 1.31 (s, 12H, 4×CH_3) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 158.8 (*i*-C_{Ar}), 131.5 (*i*-C_{Ar}), 129.3 (*o*-C_{Ar}), 113.3(*m*-C_{Ar}), 99.6 (-OCO-), 99.1 (-OCO-), 75.7 (OCH_2), 73.4 (C-2), 70.6 (C-5), 69.4 (C-4 & C-6), 69.1 (C-1 & C-3), 55.2 ($OCH_{3(PMB)}$), 47.92 (2×OCH_3), 47.87 (2×OCH_3), 17.74 (2×CH_3), 17.67 (2×CH_3) ppm. HRMS (ESI) calcd. for $C_{26}H_{40}NaO_{11}$ $[M+Na]^+$ 551.2463; found 551.2470.

5-*O*-Benzyl-2-*O*-*p*-methoxybenzyl-1,6:3,4-bis-[*O*-(2,3-dimethoxybutane-2,3-diyl)]-*myo*-inositol (16b). A solution of alcohol **16a** (265 mg, 0.5 mmol) in dry DMF (5 mL) was cooled to 0 °C under an Ar atmosphere. 90% NaH (20 mg, 0.7 mmol) was added in one portion and the resulting slurry was stirred at the same temperature for 1 h. Then, BnBr (95 µL, 0.8 mmol)

was added dropwise and the mixture was stirred for 12 h, while it was left to warm up to room temperature. MeOH (0.1 mL) was added and the mixture was stirred at room temperature for 1 h. CH_2Cl_2 (10 mL) was added and the resulting solution was washed with H_2O (10 mL). The aqueous phase was back-extracted with CH_2Cl_2 (10 mL) and the combined organic phases were washed with saturated brine (10 mL), dried over Na_2SO_4, and concentrated *in vacuo*. The residue was purified with flash column chromatography (hexanes/EtOAc 10:1 to 7:1) to give benzyl ether **16b** (278 mg, 90%) as a colorless thick oil. R_f = 0.66 (hexanes/EtOAc 1:1). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45 (d, J = 8.4 Hz, 2H, 2×ArH_{PMB}), 7.41 (d, J = 7.4 Hz, 2H, 2×ArH_{Bn}), 7.31 (t, J = 7.4 Hz, 2H, 2×ArH_{Bn}), 7.24 (t, J = 7.4 Hz, 1H, 1×ArH_{Bn}), 6.86 (d, J = 8.4 Hz, 2H, 2×ArH_{PMB}), 4.87 (s, 2H, C$H_2$$Ar_{Bn}$), 4.79 (s, 2H, C$H_2$$Ar_{PMB}$), 4.19 (t, J = 9.8 Hz, 2H, H-4 & H-6), 3.81 (s, 3H, CH_3OAr), 3.80 (t, J = 1.7 Hz, 1H, H-2), 3.57 (dd, J = 10.5, 1.7 Hz, 2H, H-1 & H-3), 3.55 (t, J = 9.4 Hz, 1H, H-5), 3.27 (s, 6H, 2×OCH_3), 3.25 (s, 6H, 2×OCH_3), 1.34 (s, 6H, 2×CH_3), 1.33 (s, 6H, 2×CH_3) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 156.7 (*i*-$C_{Ar(PMB)}$), 139.6 (*i*-$C_{Ar(Bn)}$), 131.5 (*i*-$C_{Ar(PMB)}$), 129.4 (*o*-$C_{Ar(PMB)}$), 128.0 (*m*-$C_{Ar(Bn)}$), 127.4 (*o*-$C_{Ar(Bn)}$), 127.1 (*p*-$C_{Ar(Bn)}$), 113.2 (*m*-$C_{Ar(PMB)}$), 99.6 (-OCO-), 99.0 (-OCO-), 78.8 (C-5), 75.6 (OCH_2), 74.9 (OCH_2), 73.4 (C-2), 70.0 (C-4 & C-6), 69.3 (C-1 & C-3), 55.2 ($OCH_{3(PMB)}$), 47.9 (2×OCH_3), 47.8 (2×OCH_3), 17.9 (2×CH_3), 17.7 (2×CH_3) ppm. HRMS (ESI) calcd. for $C_{33}H_{46}NaO_{11}$ $[M+Na]^+$ 641.2932; found 641.2945.

5-*O*-Benzyl-1,6:3,4-bis-[*O*-(2,3-dimethoxybutane-2,3-diyl)]-*myo*-inositol (16c). DDQ (135 mg, 0.6 mmol) was added to a solution of PMP-ether **16b** (250 mg, 0.4 mmol) in CH_2Cl_2 (10 mL) and H_2O (1 mL). The mixture was stirred at 25 °C for 24 h. Then, CH_2Cl_2 (40 mL) and a saturated aqueous $NaHCO_3$ solution (20 mL) were added and the resulting slurry was vigorously stirred at 25 °C for 30 min. The organic phase was further washed with a saturated aqueous $NaHCO_3$ solution (10 mL) and saturated brine (10 mL), dried over Na_2SO_4, and concentrated *in vacuo*. The residue was purified with flash column chromatography (hexanes/EtOAc 7:1 to 4:1) to give alcohol **16c** (140 mg, 71%) as a colorless thick oil. R_f = 0.50 (hexanes/EtOAc 1:1). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 (d, J = 7.4 Hz, 2H, 2×*o*-ArH), 7.31 (t, J = 7.4 Hz, 2H, 2×*m*-ArH), 7.24 (t, J = 7.4 Hz, 1H, *p*-ArH), 4.86 (s, 2H, CH_2Ar), 4.12 (t, J = 9.9 Hz, 2H, H-4 & H-6), 4.02 (t, J = 2.4 Hz, 1H, H-2), 3.58 (dd, J = 10.4, 2.5 Hz, 2H, H-1 & H-3), 3.55 (t, J = 9.5 Hz, 1H, H-5), 3.27 (s, 6H, 2×OCH_3), 3.26 (s, 6H, 2×OCH_3), 2.43 (br s, 1H, OH), 1.34 (s, 6H, 2×CH_3), 1.33 (s, 6H, 2×CH_3) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 139.5 (*i*-C_{Ar}), 128.1 (*m*-C_{Ar}), 127.4 (*o*-C_{Ar}), 127.2 (*p*-C_{Ar}), 100.0 (-OCO-), 99.2 (-OCO-), 78.2 (C-5), 74.9 (OCH_2), 69.4 (C-4 & C-6), 68.9 (C-2), 68.7 (C-1 & C-3), 48.1 (2×OCH_3), 47.9 (2×OCH_3), 17.9 (2×CH_3), 17.6 (2×CH_3) ppm. HRMS (ESI) calcd. for $C_{25}H_{38}NaO_{10}$ $[M+Na]^+$ 521.2357; found 521.2361.

5-*O*-Benzyl-2-*O*-butyryl-1,6:3,4-bis-[*O*-(2,3-dimethoxybutane-2,3-diyl)]-*myo*-inositol (16d). Dry Et_3N (85 µL, 0.6 mmol) and DMAP (12 mg, 0.1 mmol) were added to a solution of alcohol **16c** (150 mg, 0.3 mmol) in dry CH_2Cl_2 (3 mL) under an Ar atmosphere at room temperature. Butyric anhydride (65 µL, 0.4 mmol) was added and the mixture was stirred at room temperature for 12 h. The reaction mixture was diluted with CH_2Cl_2 (10 mL) and successively washed with saturated aqueous sodium bicarbonate solution (3×3 mL) and saturated brine (5 mL). The aqueous phase was back-extracted with CH_2Cl_2 (10 mL) and the combined organic phases were dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified with flash column chromatography (hexanes/EtOAc 10:1 to 7:1) to give butyrate **16d** (155 mg, 91%) as a white thick oil. R_f = 0.69 (hexanes/EtOAc 1:1). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42 (d, J = 7.2 Hz, 2H, 2×*o*-ArH), 7.31 (t, J = 7.4 Hz, 2H, 2×*m*-ArH), 7.26 (t, J = 7.2 Hz, 1H, *p*-ArH), 5.41 (t, J = 2.5 Hz, 1H, H-2), 4.86 (s, 2H, CH_2Ar), 4.03 (t, J = 9.9 Hz, 2H, H-4 & H-6), 3.66 (dd, J = 10.3, 2.6 Hz, 2H, H-1 & H-3), 3.58 (t, J = 9.4 Hz, 1H, H-5), 3.26 (s, 6H, 2×OCH_3), 3.24 (s, 6H, 2×OCH_3), 2.34 (t, J = 7.2 Hz, 2H, COCH_2), 1.77–1.65 (m, 2H, C$H_2$$CH_3$), 1.32 (s, 6H, 2×$CH_3$), 1.23 (s, 6H, 2×$CH_3$), 0.98 (t, J = 7.4 Hz, 3H, CH_2CH_3) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.9 (CO), 139.3 (*i*-C_{Ar}), 128.1 (*m*-C_{Ar}), 127.7 (*o*-C_{Ar}), 127.4 (*p*-C_{Ar}), 99.8 (-OCO-), 99.2 (-OCO-), 78.3 (C-5), 75.3 (OCH_2), 70.0 (C-4 & C-6), 68.9 (C-2), 67.2 (C-1 & C-3), 48.1 (2×OCH_3), 47.8 (2×OCH_3), 36.8

($CO\mathit{C}H_2$), 18.9 ($\mathit{C}H_2CH_3$), 17.8 (2×CH_3), 17.4 (2×CH_3), 13.5 ($CH_2\mathit{C}H_3$) ppm. HRMS (ESI) calcd. for $C_{29}H_{44}NaO_{11}$ $[M+Na]^+$ 591.2776; found 591.2789.

5-*O*-Benzyl-2-*O*-butyryl-*myo*-inositol (17a). Tetraol **17a** was prepared from bisacetal **16d**, according to General Procedure D. Yield: 100%. White amorphous solid. ^{1}H NMR (500 MHz, CD_3OD) δ 7.44 (d, J = 7.3 Hz, 2H, 2×*o*-ArH), 7.31 (t, J = 7.4 Hz, 2H, 2×*m*-ArH), 7.24 (t, J = 7.3 Hz, 1H, *p*-ArH), 5.43 (t, J = 2.8 Hz, 1H, H-2), 4.88 (s, 2H, $C\mathit{H}_2$Ar), 3.67 (t, J = 9.6 Hz, 2H, H-4 & H-6), 3.54 (dd, J = 9.9, 2.9 Hz, 2H, H-1 & H-3), 3.21 (t, J = 9.2 Hz, 1H, H-5), 2.36 (t, J = 7.4 Hz, 2H, $COCH_2$), 1.83–1.53 (m, 2H, $C\mathit{H}_2CH_3$), 0.97 (t, J = 7.4 Hz, 3H, $CH_2C\mathit{H}_3$) ppm. ^{13}C NMR (126 MHz, CD_3OD) δ 175.1 (CO), 140.5 (*i*-C_{Ar}), 129.2 (*o*-C_{Ar} & *m*-C_{Ar}), 128.5 (*p*-C_{Ar}), 85.1 (C-5), 76.2 (OCH_2), 75.3 (C-2), 74.7 (C-4 & C-6), 71.8 (C-1 & C-2), 37.2 ($CO\mathit{C}H_2$), 19.5 ($\mathit{C}H_2CH_3$), 14.04 ($CH_2\mathit{C}H_3$) ppm. HRMS (ESI) calcd. for $C_{17}H_{24}NaO_7$ $[M+Na]^+$ 363.1414; found 363.1405.

Octabenzyl 1,3,4,6-(5-*O*-benzyl-2-*O*-butyryl-*myo*-inositol) tetrakisphosphate (17b). Benzyl phosphate **17b** was prepared from tetraol **17a**, according to General Procedure E. Yield: 63%. Colorless thick oil. R_f = 0.57 (hexanes/EtOAc 1:2). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 (d, J = 7.3 Hz, 2H, 2×ArH), 7.19–7.05 (m, 39H, 39×ArH), 6.88 (d, J = 7.2 Hz, 4H, 4×ArH), 6.07 (br s, 1H, H-2), 4.93–4.74 (m, 18H, 8×CH_2Ph & H-4 & H-6), 4.53 (dd, J = 11.6, 9.4 Hz, 2H, CH_2Ph), 4.34 (br t, J = 9.2 Hz, 2H, H-1 & H-3), 3.45 (t, J = 8.9 Hz, 1H, H-5), 2.23 (t, J = 7.4 Hz, 2H, $COCH_2$), 1.62–1.42 (m, 2H, CH_2CH_3), 0.83 (t, J = 7.4 Hz, 3H, $CH_2C\mathit{H}_3$) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.7 (CO), 137.9 (C-*i*Ar), 135.9 (d, $^3J_{CP}$ = 7.1 Hz, 2×C-*i*Ar), 137.7–135.6 (m, 6×C-*i*Ar), 128.5, 128.4,3, 128.39, 128.32, 128.27, 128.20, 128.17, 128.13, 128.06, 128.00, 127.9, 127.8 (C-*o*Ar, C-*m*Ar, C-*p*Ar), 78.9 (C-5), 77.1 (br s, C-4 & C-6), 74.4 ($\mathit{C}H_2$Ph), 73.6 (br s, C-1 &C-3), 67.0 (d, $^2J_{CP}$ = 5.6 Hz, 2×$\mathit{C}H_2$Ph), 69.6 (d, $^2J_{CP}$ = 4.9 Hz, 2×$\mathit{C}H_2$Ph), 69.5 (d, $^2J_{CP}$ = 5.6 Hz, 2×$\mathit{C}H_2$Ph), 69.3 (d, $^2J_{CP}$ = 5.2 Hz, 2×$\mathit{C}H_2$Ph), 69.2 (C-2), 35.8 ($CO\mathit{C}H_2$), 18.5 ($\mathit{C}H_2CH_3$), 13.5 ($CH_2\mathit{C}H_3$) ppm. ^{31}P NMR (202 MHz, $CDCl_3$, ^{31}P-^{1}H decoupled): –1.28 (2P), –1.83 (2P) ppm. HRMS (ESI) calcd. for $C_{73}H_{76}NaO_{19}P_4$ $[M+Na]^+$ 1403.3823; found 1403.3819.

Tetrasodium (2-*O*-butyryl-1,3,4,6-*myo*-inositol) tetrakisphosphate (4). Phosphate **4** was prepared from benzyl phosphate **17b**, according to General Procedure F. Reaction time: 96 h. Yield: 100%. White amorphous solid. ^{1}H NMR (500 MHz, D_2O) δ 5.53 (br s, 1H, H-2), 4.19 (q, $^3J_{HP}$ = $^3J_{HH}$ = 8.9 Hz, 2H, H-4 & H-6), 4.03 (br t, $^3J_{HP}$ = $^3J_{HH}$ = 8.9 Hz, 2H, H-1 & H-3), 3.54 (t, J = 8.8 Hz, 1H, H-5), 2.39 (t, J = 7.6 Hz, 2H, $COCH_2$), 1.58–1.51 (m, 2H, CH_2CH_3), 0.83 (t, J = 7.4 Hz, 3H, $CH_2C\mathit{H}_3$) ppm. ^{13}C NMR (126 MHz, D_2O) δ 176.0 (CO), 76.6 (C-4 & C-6), 74.0 (C-5), 73.5 (C-2), 71.9 (C-1 & C-3), 57.4 ($CO\mathit{C}H_2$), 17.8 ($\mathit{C}H_2CH_3$), 13.0 ($CH_2\mathit{C}H_3$) ppm. ^{31}P NMR (202 MHz, D_2O, ^{31}P-^{1}H decoupled): 0.05 (2P), 0.00 (2P) ppm. HRMS (ESI) calcd. for $C_{10}H_{18}Na_3O_{19}P_4$ $[M–Na]^-$ 634.9091; found 634.9101.

2-*O*-benzyl-1,6:3,4-bis-[*O*-(2,3-dimethoxybutane-2,3-diyl)]-*myo*-inositol (21). Benzyl ether **21** was prepared from diol **14** in 64% yield, following known procedures.[S4,S5]

5-*O*-Acetyl-2-*O*-benzyl-1,6:3,4-bis-[*O*-(2,3-dimethoxybutane-2,3-diyl)]-*myo*-inositol (22). This compound was isolated from the reaction of malonic acid with alcohol **21** in the presence of DIC/DMAP. Yield: 41%. Colorless thick oil. R_f = 0.48 (hexanes/EtOAc 1:1). FTIR (KBr): 1765 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53 (d, J = 7.1 Hz, 2H, 2×*o*-ArH), 7.33 (t, J = 7.4 Hz, 2H, 2×*m*-ArH), 7.26 (t, J = 7.3 Hz, 1H, *p*-ArH), 5.09 (t, J = 9.8 Hz, 1H, H-5), 4.86 (s, 2H, CH_2Ph), 4.13 (t, J = 10.0 Hz, 2H, H-4 & H-6), 3.82 (t, J = 2.3 Hz, 1H, H-2), 3.61 (dd, J = 10.3, 2.4 Hz, 2H, H-1 & H-3), 3.21 (s, 6H, 2×OCH_3), 3.20 (s, 6H, 2×OCH_3), 2.06 (s, 3H, CH_3CO), 1.30 (s, 6H, 2×CH_3), 1.23 (s, 6H, 2×CH_3) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 169.7 (CO), 139.5 (*i*-C_{Ar}), 127.8 (*m*-C_{Ar}), 127.7 (*o*-C_{Ar}), 127.0 (*p*-C_{Ar}), 99.5 (-OCO-), 99.1 (-OCO-), 76.1 (C-2), 74.0 ($\mathit{C}H_2$Ph), 70.9 (C-5), 69.1 (C-1 & C-3), 67.6 (C-4 & C-6), 47.8 (OCH_3), 47.4 (OCH_3), 20.8 ($\mathit{C}H_3CO$), 17.7 (CH_3), 17.6 (CH_3) ppm. HRMS (ESI) calcd. for $C_{27}H_{40}NaO_{11}$ $[M+Na]^+$ 540.2571; found 540.2582.

5,5'-Bis[2-*O*-benzyl-1,6:3,4-bis-*O*-(2,3-dimethoxybutane-2,3-diyl)-*myo*-inositolyl] malonate (23a). Colorless thick oil. R_f = 0.68 (hexanes/EtOAc 1:1). FTIR (KBr): 1765 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51 (d, J = 7.0 Hz, 4H, 4×*o*-ArH), 7.34−7.22 (m, 6H, 4×*m*-ArH & 2×*p*-ArH), 5.08

(t, *J* = 9.8 Hz, 2H, H-5), 4.83 (s, 4H, 2×C*H*$_2$Ph), 4.12 (t, *J* = 9.8 Hz, 4H, H-4 & H-6), 3.79 (t, *J* = 2.3 Hz, 2H, H-2), 3.59 (dd, *J* = 10.2, 2.3 Hz, 4H, H-1 & H-3), 3.35 (s, 2H, COCH_2CO), 3.20 (s, 12H, 4×OCH_3), 3.16 (s, 12H, 4×OCH_3), 1.27 (s, 12H, 4×CH_3), 1.19 (s, 12H, 4×CH_3) ppm. ^{13}C NMR (75 MHz, CDCl_3) *δ* 164.8 (CO), 139.5 (*i*-C$_{Ar}$), 127.9 (*m*-C$_{Ar}$), 127.8 (*o*-C$_{Ar}$), 127.0 (*p*-C$_{Ar}$), 99.7 (-OCO-), 99.1 (-OCO-), 76.2 (C-2), 74.0 (*C*H$_2$Ph), 72.1 (C-5), 69.1 (C-1 & C-3), 67.5 (C-4 & C-6), 47.8 (4×OCH_3), 47.7 (4×OCH_3), 41.3 (CO*C*H$_2$CO), 17.6 (4×CH_3), 17.5 (4×CH_3) ppm. HRMS (ESI) calcd. for $C_{53}H_{76}NaO_{22}$ [M+Na]$^+$ 1087.4720; found 1087.4738.

5-[2-*O*-benzyl-1,6:3,4-bis-*O*-(2,3-dimethoxybutane-2,3-diyl)-*myo*-inositolyl]-3-(1,3-diisopropylureido)-3-oxopropanoate (24a). Colorless thick oil. R$_f$ = 0.40 (hexanes/EtOAc 1:1). ^{1}H NMR (300 MHz, CDCl_3) *δ* 7.51 (d, *J* = 7.1 Hz, 2H, 2×*o*-ArH), 7.35-7.23 (m, 3H, 2×*m*-ArH & *p*-ArH), 5.13 (t, *J* = 9.8 Hz, 1H, H-5), 4.85 (s, 2H, C*H*$_2$Ph), 4.43–4.34 (m, 1H, CHN), 4.18 (t, *J* = 10.0 Hz, 2H, H-4 & H-6), 3.94–3.89 (m, 1H, CHNH), 3.81 (t, *J* = 2.3 Hz, 1H, H-2), 3.61 (dd, *J* = 10.3, 2.3 Hz, 2H, H-1 & H-3), 3.51 (s, 2H, COCH_2), 3.21 (s, 12H, 4×OCH_3), 1.64 (br s, 1H, NH), 1.35 (d, *J* = 6.8 Hz, 6H, (C*H*$_3$)$_2$CHN), 1.29 (s, 6H, 2×CH_3), 1.22 (s, 6H, 2×CH_3), 1.17 (d, *J* = 6.6 Hz, 6H, (C*H*$_3$)$_2$CHNH) ppm. ^{13}C NMR (75 MHz, CDCl_3) *δ* 167.8 (OCO), 164.6 (NCO), 153.6 (NCONH), 139.5 (*i*-C$_{Ar}$), 127.9 (*m*-C$_{Ar}$), 127.7 (*o*-C$_{Ar}$), 127.1 (*p*-C$_{Ar}$), 99.7 (-OCO-), 99.2 (-OCO-), 76.1 (C-2), 74.1 (*C*H$_2$Ph), 72.8 (C-5), 69.1 (C-1 & C-3), 67.4 (C-4 & C-6), 48.3 (CHNH), 47.9 (OCH_3), 47.7 (OCH_3), 43.6 (CHN), 43.1 (CO*C*H$_2$), 22.1 [(*C*H$_3$)$_2$CHNH], 20.5 [(*C*H$_3$)$_2$CHN], 17.7 (CH_3), 17.6 (CH_3) ppm. HRMS (ESI) calcd. for $C_{35}H_{54}N_2NaO_{13}$ [M+Na]$^+$ 733.3518; found 733.3521.

5-[2-*O*-benzyl-1,6:3,4-bis-*O*-(2,3-dimethoxybutane-2,3-diyl)-*myo*-inositolyl]-3-(1,3-dicyclohexylureido)-3-oxopropanoate (24b). This compound was isolated from the reaction of malonic acid with alcohol **21** in the presence of DCC (12% yield). Colorless thick oil. R$_f$ = 0.68 (hexanes/EtOAc 1:1). ^{1}H NMR (300 MHz, CDCl_3) *δ* 7.51 (d, *J* = 7.2 Hz, 2H, 2×*o*-ArH), 7.35−7.23 (m, 3H, 2×*m*-ArH & *p*-ArH), 5.11 (t, *J* = 9.7 Hz, 1H, H-5), 4.85 (s, 2H, C*H*$_2$Ph), 4.18 (t, *J* = 10.0 Hz, 2H, H-4 & H-6), 4.06–3.97 (m, 1H, CHN), 3.81 (t, *J* = 2.2 Hz, 1H, H-2), 3.67–3.58 (m, 1H, CHN), 3.60 (dd, *J* = 10.2, 2.3 Hz, 2H, H-1 & H-3), 3.48 (s, 2H, COCH_2), 3.21 (s, 12H, 4×OCH_3), 2.05–1.53 (m, 21H, 10×C*H*$_{2cyclohex}$ & NH), 1.29 (s, 6H, 2×CH_3), 1.22 (s, 6H, 2×CH_3) ppm. ^{13}C NMR (75 MHz, CDCl_3) *δ* 168.1 (OCO), 161.8 (NCO), 153.7 (NCONH), 139.5 (*i*-C$_{Ar}$), 127.9 (*m*-C$_{Ar}$), 127.7 (*o*-C$_{Ar}$), 127.1 (*p*-C$_{Ar}$), 99.7 (-OCO-), 99.2 (-OCO-), 76.1 (C-2), 74.0 (*C*H$_2$Ph), 72.9 (C-5), 69.1 (C-1 & C-3), 67.5 (C-4 & C-6), 50.2 (CHN), 47.9 (OCH_3), 47.8 (CHNH), 47.7 (OCH_3), 43.2 (CO*C*H$_2$), 34.0, 32.8, 32.3, 30.6, 29.7, 26.5, 26.1, 25.4, 24.9, 24.7 (*C*$_{cyclohex}$), 17.7 (CH_3), 17.6 (CH_3) ppm. HRMS (ESI) calcd. for $C_{41}H_{62}N_2NaO_{13}$ [M+Na]$^+$ 813.4144; found 813.4131.

5,5'-Bis[2-*O*-benzyl-1,6:3,4-bis-*O*-(2,3-dimethoxybutane-2,3-diyl)-*myo*-inositolyl] succinate (23b). Colorless thick oil. R$_f$ = 0.47 (hexanes/EtOAc 1:1). FTIR (KBr): 1754 cm^{-1}. ^{1}H NMR (300 MHz, CDCl_3) *δ* 7.52 (d, *J* = 7.0 Hz, 4H, 4×*o*-ArH), 7.35–7.22 (m, 6H, 4×*m*-ArH & 2×*p*-ArH), 5.08 (t, *J* = 9.8 Hz, 2H, H-5), 4.84 (s, 4H, 2×C*H*$_2$Ph), 4.12 (t, *J* = 10.0 Hz, 4H, H-4 & H-6), 3.80 (t, *J* = 2.3 Hz, 2H, H-2), 3.59 (dd, *J* = 10.3, 2.3 Hz, 4H, H-1 & H-3), 3.20 (s, 12H, 4×OCH_3), 3.17 (s, 12H, 4×OCH_3), 2.65 (s, 4H, COCH_2CH_2CO), 1.28 (s, 12H, 4×CH_3), 1.21 (s, 12H, 4×CH_3) ppm. ^{13}C NMR (75 MHz, CDCl_3) *δ* 170.8 (CO), 139.6 (*i*-C$_{Ar}$), 127.9 (*m*-C$_{Ar}$), 127.7 (*o*-C$_{Ar}$), 127.0 (*p*-C$_{Ar}$), 99.7 (-OCO-), 99.1 (-OCO-), 76.3 (C-2), 74.0 (*C*H$_2$Ph), 71.4 (C-5), 69.2 (C-1 & C-3), 67.6 (C-4 & C-6), 47.8 (OCH_3), 47.5 (OCH_3), 29.4 (CO*C*H$_2$), 17.7 (CH_3), 17.6 (CH_3) ppm. HRMS (ESI) calcd. for $C_{54}H_{78}NaO_{22}$ [M+Na]$^+$ 1101.4877; found 1101.4888.

5-[2-*O*-benzyl-1,6:3,4-bis-*O*-(2,3-dimethoxybutane-2,3-diyl)-*myo*-inositolyl]-3-(1,3-dicyclohexylureido)-4-oxobutanoate (24c). This compound was isolated from the reaction of succinic acid with alcohol **21** in the presence of DCC/DMAP. Yield: 28%. Colorless thick oil. R$_f$ = 0.47 (hexanes/EtOAc 1:1). ^{1}H NMR (300 MHz, CDCl_3) *δ* 7.51 (d, *J* = 7.2 Hz, 2H, 2×*o*-ArH), 7.35−7.22 (m, 3H, 2×*m*-ArH & *p*-ArH), 5.07 (t, *J* = 9.0 Hz, 1H, H-5), 4.84 (s, 2H, C*H*$_2$Ph), 4.15 (t, *J* = 9.0 Hz, 2H, H-4 & H-6), 4.09–4.01 (m, 1H, CHN), 3.80 (t, *J* = 2.2 Hz, 1H, H-2), 3.59 (dd, *J* = 9.0, 3.0 Hz, 2H, H-1 & H-3), 3.52–3.45 (m, 1H, CHN), 3.22 (s, 6H, 2×OCH_3), 3.21 (s, 6H, 2×OCH_3), 2.74 (t, *J* = 6.0 Hz, 2H, COCH_2), 2.65 (t, *J* = 6.0 Hz, 2H, COCH_2), 1.96–1.92 (m, 4H,

4×CH$H_{cyclohex}$), 1.80-1.59 (m, 17H, 16×CH$H_{cyclohex}$ & NH), 1.29 (s, 6H, 2×CH_3), 1.23 (s, 6H, 2×CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.5 (OCO), 154.2 (NCO), 149.1 (NCONH), 139.6 (*i*-C_{Ar}), 127.9 (*m*-C_{Ar}), 127.7 (*o*-C_{Ar}), 127.0 (*p*-C_{Ar}), 99.7 (-OCO-), 99.2 (-OCO-), 76.2 (C-2), 74.0 (CH_2Ph), 71.6 (C-5), 69.2 (C-1 & C-3), 67.6 (C-4 & C-6), 50.1 (CHN), 47.9 (OCH_3), 47.7 (OCH_3), 47.5 (CHNH), (COCH_2), 34.0, 32.5, 30.7, 29.6, 26.2, 25.7, 25.5, 25.4, 24.9, 24.8 ($C_{cyclohex}$ & 2×COCH_2), 17.7 (CH_3), 17.6 (CH_3) ppm. HRMS (ESI) calcd. for $C_{42}H_{64}N_2NaO_{13}$ $[M+Na]^+$ 827.4301; found 827.4309.

5*O*,5'*O*-(Butane-1,4-diyl)bis[2-*O*-benzyl-1,6:3,4-bis-*O*-(2,3-dimethoxybutane-2,3-diyl)-*myo*-inositol] (25a). Prepared from alcohol **21** and **26**[S6] according to General Procedure A. Yield: 45%. White thick oil. R_f = 0.24 (hexanes/EtOAc 1:1). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.49 (d, *J* = 7.0 Hz, 4H, 4×*o*-ArH), 7.32–7.20 (m, 6H, 4×*m*-ArH & 2×*p*-ArH), 4.82 (s, 4H, 2×CH_2Ph), 4.05 (t, *J* = 9.8 Hz, 4H, H-4 & H-6), 3.78−3.75 (m, 6H, H-2 & 2×OCH_2CH$_2$), 3.51 (dd, *J* = 10.3, 2.3 Hz, 4H, H-1 & H-3), 3.28 (t, *J* = 9.3 Hz, 2H, H-5), 3.23 (s, 12H, 4×OCH_3), 3.21 (s, 12H, 4×OCH_3), 1.63 (m, 4H, 2×OCH$_2$CH_2), 1.29 (s, 12H, 4×CH_3), 1.26 (s, 12H, 4×CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.7 (*i*-C_{Ar}), 127.8 (*m*-C_{Ar}), 127.7 (*o*-C_{Ar}), 126.8 (*p*-C_{Ar}), 99.6 (-OCO-), 98.9 (-OCO-), 78.4 (C-5), 76.3 (C-2), 73.7 (CH_2Ph), 72.6 (OCH$_2$CH$_2$), 69.9 (C-4 & C-6), 69.4 (C-1 & C-3), 47.8 (OCH_3), 47.7 (OCH_3), 26.8 (OCH$_2$CH$_2$), 17.8 (CH_3), 17.6 (CH_3) ppm. HRMS (ESI) calcd. for $C_{54}H_{82}NaO_{20}$ $[M+Na]^+$ 1073.5292; found 1073.5294.

5*O*,5'*O*-(Pentane-1,5-diyl)bis[2-*O*-benzyl-1,6:3,4-bis-*O*-(2,3-dimethoxybutane-2,3-diyl)-*myo*-inositol] (25b). Prepared from alcohol **21** and **27**[S7] according to General Procedure A. Yield: 62%. White thick oil. R_f = 0.21 (hexanes/EtOAc 1:1). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.49 (d, *J* = 7.0 Hz, 4H, 4×*o*-ArH), 7.32–7.22 (m, 6H, 4×*m*-ArH & 2×*p*-ArH), 4.83 (s, 4H, 2×CH_2Ph), 4.05 (t, *J* = 9.9 Hz, 4H, H-4 & H-6), 3.75 (t, *J* = 2.2 Hz, 2H, H-2), 3.73 (t, *J* = 6.3 Hz, 4H, 2×OCH_2CH$_2$), 3.51 (dd, *J* = 10.4, 2.3 Hz, 4H, H-1 & H-3), 3.28 (t, *J* = 9.3 Hz, 2H, H-5), 3.24 (s, 12H, 4×OCH_3), 3.21 (s, 12H, 4×OCH_3), 1.61–1.51 (m, 4H, 2×OCH$_2$CH_2), 1.47–1.40 (m, 2H, OCH$_2$CH$_2$CH_2), 1.29 (s, 12H, 4×CH_3), 1.26 (s, 12H, 4×CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.5 (*i*-C_{Ar}), 127.8 (*m*-C_{Ar}), 127.7 (*o*-C_{Ar}), 126.8 (*p*-C_{Ar}), 99.5 (-OCO-), 98.9 (-OCO-), 78.3 (C-5), 76.1 (C-2), 73.7 (CH_2Ph), 72.8 (OCH$_2$CH$_2$), 69.8 (C-4 & C-6), 69.3 (C-1 & C-3), 47.8 (OCH_3), 47.7 (OCH_3), 29.9 (OCH$_2$CH$_2$), 22.2 (OCH$_2$CH$_2$CH$_2$), 17.8 (CH_3), 17.6 (CH_3) ppm. HRMS (ESI) calcd. for $C_{55}H_{84}NaO_{20}$ $[M+Na]^+$ 1087.5448; found 1087.5459.

5,5'-Bis[1,6:3,4-bis-*O*-(2,3-dimethoxybutane-2,3-diyl)-*myo*-inositolyl] malonate (28a). Prepared from dibenzyl ether **23a** according to General Procedure B. Yield: 100%. White amorphous solid. R_f = 0.15 (hexanes/EtOAc 1:2). FTIR (KBr): 3441, 1800 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.11 (t, *J* = 9.9 Hz, 2H, H-5), 4.10 (t, *J* = 10.0 Hz, 4H, H-4 & H-6), 4.03 (t, *J* = 2.5 Hz, 2H, H-2), 3.62 (dd, *J* = 10.2, 2.5 Hz, 4H, H-1 & H-3), 3.39 (s, 2H, CH_2), 3.23 (s, 12H, 4×OCH_3), 3.20 (s, 12H, 4×OCH_3), 2.33 (br s, 2H, 2×OH), 1.31 (s, 12H, 4×CH_3), 1.23 (s, 12H, 4×CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 164.8 (CO), 100.1 (-OCO-), 99.3 (-OCO-), 71.6 (C-5), 68.9 (C-2), 68.5 (C-1 & C-3), 66.9 (C-4 & C-6), 47.9 (4×OCH_3), 47.7 (4×OCH_3), 41.2 (COCH_2CO), 17.63 (4×CH_3), 17.56 (4×CH_3) ppm. HRMS (ESI) calcd. for $C_{39}H_{64}NaO_{22}$ $[M+Na]^+$ 907.3781; found 907.3788.

5,5'-Bis[1,6:3,4-bis-*O*-(2,3-dimethoxybutane-2,3-diyl)-*myo*-inositolyl] succinate (28b). Prepared from dibenzyl ether **23b** according to General Procedure B. Yield: 100%. White amorphous solid. R_f = 0.09 (hexanes/EtOAc 1:1). FTIR (KBr): 3483, 1754 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.10 (t, *J* = 9.9 Hz, 2H, H-5), 4.09 (t, *J* = 10.0 Hz, 4H, H-4 & H-6), 4.03 (t, *J* = 2.5 Hz, 2H, H-2), 3.62 (dd, *J* = 10.2, 2.6 Hz, 4H, H-1 & H-3), 3.23 (s, 12H, 4×OCH_3), 3.20 (s, 12H, 4×OCH_3), 2.68 (s, 4H, 2×CH_2), 2.38 (s, 2H, 2×OH), 1.31 (s, 12H, 4×CH_3), 1.23 (s, 12H, 4×CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.8 (CO), 100.1 (-OCO-), 99.3 (-OCO-), 70.8 (C-5), 68.9 (C-2), 68.6 (C-1 & C-3), 67.0 (C-4 & C-6), 47.9 (4×OCH_3), 47.6 (4×OCH_3), 29.2 (COCH_2), 17.63 (4×CH_3), 17.56 (4×CH_3) ppm. HRMS (ESI) calcd. for $C_{40}H_{66}NaO_{22}$ $[M+Na]^+$ 921.3938; found 921.3928.

5*O*,5'*O*-(Butane-1,4-diyl)bis[1,6:3,4-bis-*O*-(2,3-dimethoxybutane-2,3-diyl)-*myo*-inositol] (28c). Prepared from dibenzyl ether **25a** according to General Procedure B. Yield: 93%. White amorphous solid. R_f = 0.08 (hexanes/EtOAc 1:1). FTIR (KBr): 3448, 1038 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 3.98 (t, 4H, H-4 & H-6), 3.98 (2H, H-2, obscured), 3.77 (br s, 4H, 2×OCH_2), 3.53 (dd, J = 10.3, 2.5 Hz, 4H, H-1 & H-3), 3.31 (t, J = 9.4 Hz, 2H, H-5), 3.25 (s, 24H, 8×OCH_3), 2.32 (br s, 2H, 2×OH), 1.64 (br s, 4H, 2×OCH_2CH_2), 1.32 (s, 12H, 4×CH_3), 1.28 (s, 12H, 4×CH_3) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 100.0 (-OCO-), 99.1 (-OCO-), 77.7 (C-5), 72.5 (OCH_2), 69.4 (C-4 & C-6), 69.0 (C-2), 68.7 (C-1 & C-3), 48.0 (4×OCH_3), 47.7 (4×OCH_3), 26.7 (OCH_2CH_2), 17.8 (4×CH_3), 17.6 (4×CH_3) ppm. HRMS (ESI) calcd. for $C_{40}H_{70}NaO_{20}$ $[M+Na]^+$ 893.4353; found 893.4348.

5*O*,5'*O*-(Pentane-1,5-diyl)bis[1,6:3,4-bis-*O*-(2,3-dimethoxybutane-2,3-diyl)-*myo*-inositol] (28d). Prepared from dibenzyl ether **25b** according to General Procedure B. Yield: 96%. White amorphous solid. R_f = 0.08 (hexanes/EtOAc 1:1). FTIR (KBr): 3450, 1037 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 3.99 (t, J = 9.9 Hz, 4H, H-4 & H-6), 3.99 (2H, H-2, obscured), 3.72 (t, J = 6.3 Hz, 4H, 2×OCH_2), 3.52 (dd, J = 10.3, 2.5 Hz, 4H, H-1 & H-3), 3.30 (t, J = 9.5 Hz, 2H, H-5), 3.25 (s, 12H, 4×OCH_3), 3.24 (s, 12H, 4×OCH_3), 1.80 (br s, 2H, 2×OH), 1.57–1.52 (m, 4H, 2×OCH_2CH_2), 1.46–1.41 (m, 2H, $OCH_2CH_2CH_2$), 1.31 (s, 12H, 4×CH_3), 1.27 (s, 12H, 4×CH_3) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 99.9 (-OCO-), 99.1 (-OCO-), 77.7 (C-5), 72.8 (OCH_2), 69.3 (C-4 & C-6), 68.9 (C-2), 68.7 (C-1 & C-3), 48.0 (4×OCH_3), 47.7 (4×OCH_3), 29.9 (OCH_2CH_2), 22.2 ($OCH_2CH_2CH_2$), 17.8 (4×CH_3), 17.6 (4×CH_3) ppm. HRMS (ESI) calcd. for $C_{41}H_{72}NaO_{20}$ $[M+Na]^+$ 907.4509; found 907.4500.

5,5'-Bis[2-*O*-butyryl-1,6:3,4-bis-*O*-(2,3-dimethoxybutane-2,3-diyl)-*myo*-inositolyl] malonate (29a). Prepared from diol **28a** according to General Procedure C. Yield: 79%. White solid. m.p. 170.2-172.2 °C. R_f = 0.39 (hexanes/EtOAc 1:1). FTIR (KBr): 1752 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 5.43 (t, J = 2.6 Hz, 2H, H-2), 5.12 (t, J = 9.9 Hz, 2H, H-5), 3.99 (t, J = 10.1 Hz, 4H, H-4 & H-6), 3.70 (dd, J = 10.2, 2.7 Hz, 4H, H-1 & H-3), 3.42 (s, 2H, $COCH_2CO$), 3.21 (s, 12H, 4×OCH_3), 3.20 (s, 12H, 4×OCH_3), 2.37 (t, J = 7.2 Hz, 4H, 2×$COCH_2$), 1.77–1.64 (m, 4H, 2×$COCH_2CH_2$), 1.22 (s, 12H, 4×CH_3), 1.20 (s, 12H, 4×CH_3), 1.00 (t, J = 7.4 Hz, 6H, 2×CH_2CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.6 (CO_{but}), 164.7 (CO_{mal}), 99.9 (-OCO-), 99.3 (-OCO-), 71.6 (C-5), 68.6 (C-2), 67.5 (C-4 & C-6), 67.0 (C-1 & C-3), 48.0 (4×OCH_3), 47.6 (4×OCH_3), 41.1 ($COCH_2CO$), 36.7 ($COCH_2$), 18.8 ($COCH_2CH_2$), 17.6 (4×CH_3), 17.4 (4×CH_3), 13.5 (CH_2CH_3) ppm. HRMS (ESI) calcd. for $C_{47}H_{76}NaO_{24}$ $[M+Na]^+$ 1047.4619; found 1047.4601.

5,5'-Bis[2-*O*-butyryl-1,6:3,4-bis-*O*-(2,3-dimethoxybutane-2,3-diyl)-*myo*-inositolyl] succinate (29b). Prepared from diol **28b** according to General Procedure C. Yield: 97%. White solid. m.p. 242.4-243.5 °C. R_f = 0.64 (hexanes/EtOAc 1:1). FTIR (KBr): 1753, 1742 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ 5.43 (t, J = 2.7 Hz, 2H, H-2), 5.11 (t, J = 9.9 Hz, 2H, H-5), 3.97 (t, J = 10.1 Hz, 4H, H-4 & H-6), 3.69 (dd, J = 10.2, 2.7 Hz, 4H, H-1 & H-3), 3.21 (s, 12H, 4×OCH_3), 3.20 (s, 12H, 4×OCH_3), 2.69 (s, 4H, $COCH_2CH_2CO$), 2.38 (t, J = 7.2 Hz, 4H, 2×$COCH_2$), 1.78–1.66 (m, 4H, 2×$COCH_2CH_2$), 1.20 (s, 12H, 4×CH_3), 1.20 (s, 12H, 4×CH_3), 1.01 (t, J = 7.4 Hz, 6H, 2×CH_2CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.6 (CO_{but}), 170.8 (CO_{suc}), 99.8 (-OCO-), 99.3 (-OCO-), 70.8 (C-5), 68.6 (C-2), 67.6 (C-4 & C-6), 67.1 (C-1 & C-3), 47.9 (4×OCH_3), 47.5 (4×OCH_3), 36.7 ($COCH_2$), 29.1 ($COCH_2CH_2CO$, 18.8 ($COCH_2CH_2$), 17.6 (4×CH_3), 17.4 (4×CH_3), 13.5 (CH_2CH_3) ppm. HRMS (ESI) calcd. for $C_{48}H_{78}NaO_{24}$ $[M+Na]^+$ 1061.4775; found 1061.4786.

5*O*,5'*O*-(Butane-1,4-diyl)bis[2-*O*-butyryl-1,6:3,4-bis-*O*-(2,3-dimethoxybutane-2,3-diyl)-*myo*-inositol] (29c). Prepared from diol **28c** according to General Procedure C. Yield: 87%. White solid. m.p. 195.3 °C. R_f = 0.66 (hexanes/EtOAc 1:1). FTIR (KBr): 1740, 1039 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 5.38 (t, J = 2.7 Hz, 2H, H-2), 3.88 (t, J = 9.9 Hz, 4H, H-4 & H-6), 3.79 (br s, 4H, 2×OCH_2), 3.60 (dd, J = 10.3, 2.7 Hz, 4H, H-1 & H-3), 3.33 (t, J = 9.4 Hz, 2H, H-5), 3.23 (s, 12H, 4×OCH_3), 3.22 (s, 12H, 4×OCH_3), 2.34 (t, J = 7.2 Hz, 4H, 2×$COCH_2$), 1.73–1.67 (m, 4H, 2×$COCH_2CH_2$), 1.66 (br s, 4H, 2×OCH_2CH_2), 1.25 (s, 12H, 4×CH_3), 1.20 (s, 12H, 4×CH_3), 0.98 (t, J = 7.4 Hz, 6H, 2×CH_2CH_3) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.9 (CO), 99.7 (-OCO-), 99.1 (-

OCO-), 77.6 (C-5), 72.6 (OCH_2), 69.9 (C-4 & C-6), 68.9 (C-2), 67.2 (C-1 & C-3), 48.0 (4×OCH_3), 47.7 (4×OCH_3), 36.8 ($COCH_2$), 26.6 (OCH_2CH_2), 18.9 ($COCH_2CH_2$), 17.8 (4×CH_3), 17.4 (4×CH_3), 13.5 (CH_2CH_3) ppm. HRMS (ESI) calcd. for $C_{48}H_{82}NaO_{22}$ $[M+Na]^+$ 1033.5190; found 1033.5203.

5*O*,5'*O*-(Pentane-1,5-diyl)bis[2-*O*-butyryl-1,6:3,4-bis-*O*-(2,3-dimethoxybutane-2,3-diyl)-*myo*-inositol] (29d). Prepared from diol **28d** according to General Procedure C. Yield: 79%. White solid. m.p. 213-214 °C. R_f = 0.59 (hexanes/EtOAc 1:1). FTIR (KBr): 1754, 1039 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 5.39 (t, *J* = 2.7 Hz, 2H, H-2), 3.90 (t, *J* = 9.9 Hz, 4H, H-4 & H-6), 3.76 (t, *J* = 6.4 Hz, 4H, 2×OCH_2), 3.62 (dd, *J* = 10.3, 2.7 Hz, 4H, H-1 & H-3), 3.34 (t, *J* = 9.4 Hz, 2H, H-5), 3.25 (s, 12H, 4×OCH_3), 3.23 (s, 12H, 4×OCH_3), 2.35 (t, *J* = 7.2 Hz, 4H, 2×$COCH_2$), 1.74–1.66 (m, 4H, 2×$COCH_2CH_2$), 1.61–1.55 (m, 4H, 2×OCH_2CH_2), 1.50–1.44 (m, 2H, $OCH_2CH_2CH_2$), 1.26 (s, 12H, 4×CH_3), 1.21 (s, 12H, 4×CH_3), 0.99 (t, *J*=7.4 Hz, 6H, 2×CH_2CH_3) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.9 (CO), 99.7 (-OCO-), 99.0 (-OCO-), 77.8 (C-5), 72.9 (OCH_2), 69.9 (C-4 & C-6), 68.9 (C-2), 67.1 (C-1 & C-3), 48.0 (4×OCH_3), 47.7 (4×OCH_3), 36.8 ($COCH_2$), 29.9 (OCH_2CH_2), 22.1 ($OCH_2CH_2CH_2$), 18.9 ($COCH_2CH_2$), 17.8 (4×CH_3), 17.4 (4×CH_3), 13.5 (CH_2CH_3) ppm. HRMS (ESI) calcd. for $C_{49}H_{84}NaO_{22}$ $[M+Na]^+$ 1047.5346; found 1047.5333.

5,5'-Bis(2-*O*-butyryl-*myo*-inositolyl) malonate (30a). Prepared from tetrakisacetal **29a** according to General Procedure D. Yield: 100%. White solid. ^1H NMR (500 MHz, CD_3OD) δ 5.46 (t, *J* = 2.6 Hz, 2H, H-2), 4.85 (t, *J* = 9.1 Hz, 2H, H-5), 3.67 (t, *J* = 9.9 Hz, 4H, H-4 & H-6), 3.63 (dd, *J* = 9.9, 2.8 Hz, 4H, H-1 & H-3), 3.61 (s, 2H, $COCH_2CO$), 2.38 (t, *J* = 7.4 Hz, 4H, 2×$COCH_2$), 1.70–1.62 (m, 4H, 2×$COCH_2CH_2$), 0.98 (t, *J* = 7.4 Hz, 6H, 2×CH_3) ppm. ^{13}C NMR (126 MHz, CD_3OD) δ 174.8 (CO_{but}), 168.0 (CO_{mal}), 78.9 (C-5), 75.0 (C-2), 72.5 (C-4 & C-6), 71.3 (C-1 & C-3), 42.9 ($COCH_2CO$), 37.1 ($COCH_2$), 19.4 ($COCH_2CH_2$), 14.0 (CH_3) ppm. HRMS (ESI) calcd. for $C_{23}H_{36}NaO_{16}$ $[M+Na]^+$ 591.1896; found 591.1891.

5,5'-Bis(*myo*-inositolyl) malonate (30b). Prepared from tetrakisacetal **28a** according to General Procedure D. Yield: 100%. White solid. ^1H NMR (500 MHz, D_2O) δ 4.85 (t, *J* = 9.6 Hz, 2H, H-5), 4.09 (br s, 2H, H-2), 3.80 (t, *J* = 9.9 Hz, 4H, H-4 & H-6), 3.76 (s, 2H, CH_2), 3.64 (dd, *J* = 10.1, 2.9 Hz, 4H, H-1 & H-3) ppm. ^{13}C NMR (126 MHz, D_2O) δ 168.0 (CO), 77.3 (C-5), 71.8 (C-2), 70.8 (C-4 & C-6), 70.3 (C-1 & C-3), 41.2 (CH_2) ppm. HRMS (ESI) calcd. for $C_{15}H_{24}NaO_{14}$ $[M+Na]^+$ 451.1058; found 451.1060.

5,5'-Bis(2-*O*-butyryl-*myo*-inositolyl) succinate (30c). Prepared from tetrakisacetal **29b** according to General Procedure D. Yield: 100%. White solid. ^1H NMR (500 MHz, CD_3OD) δ 5.46 (t, *J* = 2.8 Hz, 2H, H-2), 4.82 (t, *J* = 9.3 Hz, 2H, H-5), 3.66 (t, *J* = 9.8 Hz, 4H, H-4 & H-6), 3.61 (dd, *J* = 9.9, 2.8 Hz, 4H, H-1 & H-3), 2.78 (s, 4H, $COCH_2CH_2CO$), 2.39 (t, *J* = 7.4 Hz, 4H, 2×$COCH_2$), 1.70–1.63 (m, 4H, 2×$COCH_2CH_2$), 0.99 (t, *J* = 7.4 Hz, 6H, 2×CH_3) ppm. ^{13}C NMR (126 MHz, CD_3OD) δ 174.9 (CO_{but}), 174.0 (CO_{suc}), 78.1 (C-5), 75.1 (C-2), 72.7 (C-4 & C-6), 71.5 (C-1 & C-3), 37.1 ($COCH_2$), 30.5 ($COCH_2CH_2CO$), 19.4 ($COCH_2CH_2$), 14.0 (CH_3) ppm. HRMS (ESI) calcd. for $C_{24}H_{38}NaO_{16}$ $[M+Na]^+$ 605.2052; found 605.2041.

5,5'-Bis(*myo*-inositolyl) succinate (30d). Prepared from tetrakisacetal **28b** according to General Procedure D. Yield: 98%. White solid. ^1H NMR (500 MHz, D_2O) δ 4.78 (2H, H-5, obscured), 4.05 (t, *J* = 2.7 Hz, 2H, H-2), 3.75 (t, *J* = 9.9 Hz, 4H, H-4 & H-6), 3.59 (dd, *J*=10.1, 2.8 Hz, 4H, H-1 & H-3), 2.81 (s, 4H, $COCH_2CH_2CO$) ppm. ^{13}C NMR (126 MHz, D_2O) δ 174.4 (CO), 76.5 (C-5), 71.9 (C-2), 70.9 (C-1 & C-3), 70.5 (C-4 & C-6), 28.9 ($COCH_2$) ppm. HRMS (ESI) calcd. for $C_{16}H_{26}NaO_{14}$ $[M+Na]^+$ 465.1215; found 465.1227.

5*O*,5'*O*-(Butane-1,4-diyl)bis(2-*O*-butyryl-*myo*-inositol) (30e). Prepared from tetrakisacetal **29c** according to General Procedure D. Yield: 99%. White solid. ^1H NMR (500 MHz, D_2O) δ 5.38 (t, *J* = 2.6 Hz, 2H, H-2), 3.79 (br s, 4H, 2×OCH_2), 3.66 (dd, *J* = 10.0, 2.7 Hz, 4H, H-1 & H-3), 3.62 (t, *J* = 10.0 Hz, 4H, H-4 & H-6), 3.14 (t, *J* = 9.0 Hz, 2H, H-5), 2.40 (t, *J* = 7.4 Hz, 4H, 2×$COCH_2$), 1.66 (br s, 4H, 2×OCH_2CH_2), 1.62–1.55 (m, 4H, 2×$COCH_2CH_2$), 0.88 (t, *J* = 7.4 Hz, 6H, 2×CH_3) ppm. ^{13}C NMR (75 MHz, D_2O) δ 176.3 (CO), 82.9 (C-5), 73.9 (C-2), 72.8 (OCH_2), 72.3 (C-1 & C-3), 69.7 (C-4 & C-6), 35.8 ($COCH_2$), 25.9 (OCH_2CH_2), 17.9 ($COCH_2CH_2$), 12.8 (CH_3) ppm. HRMS (ESI) calcd. for $C_{24}H_{42}NaO_{14}$ $[M+Na]^+$ 577.2467; found 577.2477.

5*O*,5'*O*-(Butane-1,4-diyl)bis(*myo*-inositol) (30f). Prepared from tetrakisacetal **28c** according to General Procedure D. Yield: 98%. White solid. ^{1}H NMR (500 MHz, D_2O) δ 3.99 (t, J = 2.8 Hz, 2H, H-2), 3.78 (br t, J = 6.1 Hz, 4H, 2×OCH_2), 3.63 (t, J = 9.8 Hz, 4H, H-4 & H-6), 3.48 (dd, J = 10.0, 2.9 Hz, 4H, H-1 & H-3), 3.09 (t, J = 9.5 Hz, 2H, H-5), 1.65 (br t, J = 6.2 Hz, 4H, 2×OCH_2CH_2) ppm. ^{13}C NMR (126 MHz, D_2O) δ 82.9 (C-5), 72.6 (OCH_2), 71.8 (C-4 & C-6), 71.7 (C-2), 71.0 (C-1 & C-3), 25.7 (OCH_2CH_2) ppm. HRMS (ESI) calcd. for $C_{16}H_{30}NaO_{12}$ $[M+Na]^+$ 437.1629; found 437.1641.

5*O*,5'*O*-(Pentane-1,5-diyl)bis(2-*O*-butyryl-*myo*-inositol) (30g). Prepared from tetrakisacetal **29d** according to General Procedure D. Yield: 99%. White solid. ^{1}H NMR (500 MHz, D_2O) δ 5.45 (t, J = 2.6 Hz, 2H, H-2), 3.84 (t, J = 6.6 Hz, 4H, 2×OCH_2), 3.73 (dd, J = 10.0, 2.5 Hz, 4H, H-1 & H-3), 3.68 (t, J = 10.0 Hz, 4H, H-4 & H-6), 2.47 (t, J = 7.4 Hz, 4H, 2×$COCH_2$), 1.70–1.62 (m, 8H, 2×$COCH_2CH_2$ & 2×OCH_2CH_2), 1.50–1.43 (m, 2H, $OCH_2CH_2CH_2$), 1.19 (t, J = 7.1 Hz, 6H, 2×CH_3) ppm. ^{13}C NMR (75 MHz, D_2O) δ 176.3 (CO), 82.8 (C-5), 73.9 (C-2), 73.0 (OCH_2), 72.3 (C-1 & C-3), 69.7 (C-4 & C-6), 35.8 ($COCH_2$), 29.0 (OCH_2CH_2), 21.6 ($OCH_2CH_2CH_2$), 17.9 ($COCH_2CH_2$), 12.8 (CH_3) ppm. HRMS (ESI) calcd. for $C_{25}H_{44}NaO_{14}$ $[M+Na]^+$ 591.2623; found 591.2626.

5*O*,5'*O*-(Pentane-1,5-diyl)bis(*myo*-inositol) (30h). Prepared from tetrakisacetal **28d** according to General Procedure D. Yield: 99%. White solid. ^{1}H NMR (500 MHz, D_2O) δ 4.02 (t, J = 2.9 Hz, 2H, H-2), 3.78 (t, J = 6.8 Hz, 4H, 2×OCH_2), 3.65 (t, J = 9.7 Hz, 4H, H-4 & H-6), 3.51 (dd, J = 10.0, 2.9 Hz, 4H, H-1 & H-3), 3.11 (t, J = 9.5 Hz, 2H, H-5), 1.66–1.60 (m, 4H, 2×OCH_2CH_2), 1.45–1.39 (m, 2H, $OCH_2CH_2CH_2$) ppm. ^{13}C NMR (126 MHz, D_2O) δ 82.9 (C-5), 72.9 (OCH_2), 71.9 (C-4 & C-6), 71.8 (C-2), 71.0 (C-1 & C-3), 28.9 (OCH_2CH_2), 21.6 ($OCH_2CH_2CH_2$) ppm. HRMS (ESI) calcd. for $C_{17}H_{32}NaO_{12}$ $[M+Na]^+$ 451.1786; found 451.1799.

5,5'-Bis[2-*O*-butyryl-1,3,4,6-*O*-tetrakis(bis(benzyloxy)phosphoryl)-*myo*-inositolyl) malonate (31a). Prepared from decaol **30a** according to General Procedure E. Yield: 61%. Colorless thick oil. R_f = 0.18 (hexanes/EtOAc 1:1). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.25–7.13 (m, 80H, 16×CH_2Ph), 6.12 (br s, 2H, H-2), 5.23 (t, J = 9.5 Hz, 2H, H-5), 5.11–4.88 (m, 32H, 16×CH_2Ph), 4.78 (q, $^{3}J_{HP}$ = $^{3}J_{HH}$ = 9.6 Hz, 4H, H-4 & H-6), 4.33 (br td, $^{3}J_{HH}$ = $^{3}J_{HP}$ = 9.7 Hz, $^{3}J_{HH}$ = 2.2 Hz, 4H, H-1 & H-3), 4.10 (s, 2H, $COCH_2CO$), 2.24 (t, J = 7.4 Hz, 4H, 2×$COCH_2$), 1.63–1.50 (m, 4H, 2×$COCH_2CH_2$), 0.88 (t, J=7.4 Hz, 6H, 2×CH_3) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.4 (CO_{but}), 166.0 (CO_{mal}), 135.9 (d, $^{3}J_{CP}$ = 6.0 Hz, 4×C-*i*Ar), 135.7 (d, $^{3}J_{CP}$ = 7.5 Hz, 4×C-*i*Ar), 135.5 (d, $^{3}J_{CP}$ = 7.3 Hz, 4×C-*i*Ar), 135.4 (d, $^{3}J_{CP}$ = 6.9 Hz, 4×C-*i*Ar), 128.35, 128.29, 128.28, 128.22, 128.14, 128.12, 128.0, 127.9, 127.8, 127.7 (C-*o*Ar, C-*m*Ar, C-*p*Ar), 75.0 (br t, $^{2}J_{CP}$ = 6.3 Hz, C-4 & C-6), 73.3 (t, $^{2}J_{CP}$ = 4.6 Hz, C-1 & C-3), 70.9 (br s, C-5), 69.8 (d, $^{2}J_{CP}$ = 5.6 Hz, 4×CH_2Ph), 69.7 (d, $^{2}J_{CP}$ = 5.6 Hz, 4×CH_2Ph), 69.4 (d, $^{2}J_{CP}$ = 5.3 Hz, 4×CH_2Ph), 69.3 (d, $^{2}J_{CP}$ = 5.5 Hz, 4×CH_2Ph), 69.1 (br s, C-2), 39.6 ($COCH_2CO$), 35.6 ($COCH_2$), 18.3 ($COCH_2CH_2$), 13.4 (CH_3) ppm. ^{31}P NMR (121 MHz, $CDCl_3$, ^{31}P-^{1}H decoupled): –1.25 (4P), –1.78 (4P) ppm. HRMS (ESI) calcd. for $C_{135}H_{140}NaO_{40}P_8$ $[M+Na]^+$ 2671.6714; found 2671.6699.

5,5'-Bis[1,2,3,4,6-*O*-pentakis(bis(benzyloxy)phosphoryl)-*myo*-inositolyl) malonate (31b). Prepared from decaol **30b** according to General Procedure E. Yield: 78%. White thick oil. R_f = 0.50 (hexanes/EtOAc 1:2). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.26–7.01 (m, 100H, 20×CH_2Ph), 5.59 (br d, $^{3}J_{HP}$ = 8.8 Hz, 2H, H-2), 5.26 (t, J = 9.6 Hz, 2H, H-5), 5.15 (dd, $^{2}J_{HH}$ = 11.7 Hz, $^{3}J_{HP}$ = 6.3 Hz, 4H, 2×CH_2Ph), 5.07–4.79 (m, 40H, H-4 & H-6 & 18×CH_2Ph), 4.32 (br t, $^{3}J_{HP}$ = $^{3}J_{HH}$ = 9.5 Hz, 4H, H-1 & H-3), 4.06 (s, 2H, CH_2) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.0 (CO), 135.9 (d, $^{3}J_{CP}$ = 5.8 Hz, 4×C-*i*Ar), 135.7 (d, $^{3}J_{CP}$ = 7.2 Hz, 4×C-*i*Ar), 135.64 (d, $^{3}J_{CP}$ = 8.1 Hz, 4×C-*i*Ar), 135.58 (d, $^{3}J_{CP}$ = 6.7 Hz, 4×C-*i*Ar), 135.5 (d, $^{3}J_{CP}$ = 6.8 Hz, 4×C-*i*Ar), 128.42, 128,38, 128.33, 128.31, 128.24, 128.19, 128.1, 128.0, 127.9 (C-*o*Ar, C-*m*Ar, C-*p*Ar), 76.1 (br d, $^{2}J_{CP}$ = 4.7 Hz, C-2), 74.6 (br t, $^{2}J_{CP}$ = 5.7 Hz, C-4 & C-6), 73.6 (br s, C-1 & C-3), 70.9 (br s, C-5), 70.0 (d, $^{2}J_{CP}$ = 5.6 Hz, 4×CH_2Ph), 69.8 (d, $^{2}J_{CP}$ = 5.6 Hz, 4×CH_2Ph), 69.7 (d, $^{2}J_{CP}$ = 5.8 Hz, 4×CH_2Ph), 69.8 (d, $^{2}J_{CP}$ = 6.2 Hz, 4×CH_2Ph), 69.6 (d, $^{2}J_{CP}$ = 5.6 Hz, 4×CH_2Ph), 39.7 (CH_2) ppm. ^{31}P NMR (121 MHz, $CDCl_3$, ^{31}P-^{1}H

decoupled): –0.91 (4P), –1.58 (4P), –2.30 (2P) ppm. HRMS (ESI) calcd. for $C_{155}H_{154}NaO_{44}P_{10}$ $[M+Na]^+$ 3051.7081; found 3051.7102.

5,5'-Bis[2-*O*-butyryl-1,3,4,6-*O*-tetrakis(bis(benzyloxy)phosphoryl)-*myo*-inositolyl) succinate (31c). Prepared from decaol **30c** according to General Procedure E. Yield: 75%. Colorless thick oil. R_f = 0.18 (hexanes/EtOAc 1:1). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.26–7.12 (m, 80H, 16×CH_2*Ph*), 6.12 (br s, 2H, H-2), 5.14 (t, *J* = 9.4 Hz, 2H, H-5), 4.99–4.89 (m, 32H, 16×C*H*$_2$Ph), 4.79 (q, $^3J_{HP}$ = $^3J_{HH}$ = 9.4 Hz, 4H, H-4 & H-6), 4.33 (td, $^3J_{HP}$ = $^3J_{HH}$ = 9.3 Hz, $^3J_{HH}$ = 2.5 Hz, 4H, H-1 & H-3), 2.92 (s, 4H, $COCH_2CH_2CO$), 2.24 (t, *J* = 7.4 Hz, 4H, 2×$COCH_2$), 1.60–1.52 (m, 4H, 2×$COCH_2CH_2$), 0.88 (t, *J* = 7.4 Hz, 6H, 2×CH_3) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.8 (CO_{but}), 171.5 (CO_{suc}), 135.72 (d, $^3J_{CP}$ = 7.2 Hz, 4×C-*i*Ar), 135.66 (d, $^3J_{CP}$ = 6.3 Hz, 4×C-*i*Ar), 135.49 (d, $^3J_{CP}$ = 7.2 Hz, 4×C-*i*Ar), 135.48 (d, $^3J_{CP}$ = 6.9 Hz, 4×C-*i*Ar), 128.39, 128.33, 128.28, 128.24, 128.22, 128.10, 128.06, 127.94, 127.86, 127.76 (C-*o*Ar, C-*m*Ar, C-*p*Ar), 75.2 (br t, $^2J_{CP}$ = 6.5 Hz, C-4 & C-6), 73.3 (br t, $^2J_{CP}$ = 4.8 Hz, C-1 & C-3), 70.6 (br s, C-5), 69.74 (d, $^2J_{CP}$ = 5.6 Hz, 4×*C*H_2Ph), 69.67 (d, $^2J_{CP}$ = 5.7 Hz, 4×*C*H_2Ph), 69.5 (d, $^2J_{CP}$ = 5.4 Hz, 4×*C*H_2Ph), 69.4 (d, $^2J_{CP}$ = 5.7 Hz, 4×*C*H_2Ph), 69.1 (br s, C-2), 35.7 ($COCH_2$), 28.8 ($COCH_2CH_2CO$) 18.4 ($COCH_2CH_2$), 13.4 (CH_3) ppm. ^{31}P NMR (121 MHz, $CDCl_3$, ^{31}P-^{1}H decoupled): –1.50 (4P), –1.76 (4P) ppm. HRMS (ESI) calcd. for $C_{136}H_{142}NaO_{40}P_8$ $[M+Na]^+$ 2685.6871; found 2685.6876.

5,5'-Bis[1,2,3,4,6-*O*-pentakis(bis(benzyloxy)phosphoryl)-*myo*-inositolyl) succinate (31d). Prepared from decaol **30d** according to General Procedure E. Yield: 72%. White thick oil. R_f = 0.11 (hexanes/EtOAc 1:1). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28–7.07 (m, 100H, 20×CH_2*Ph*), 5.60 (br d, $^3J_{HP}$ = 8.0 Hz, 2H, H-2), 5.15 (dd, $^2J_{HH}$ = 11.3 Hz, $^3J_{HP}$ = 4.9 Hz, 4H, 2×C*H*$_2$Ph), 5.15 (2H, H-5, obscured), 5.06–4.89 (m, 36H, 18×C*H*$_2$Ph), 4.86 (q, $^3J_{HP}$ = $^3J_{HH}$ = 9.5 Hz, 4H, H-4 & H-6), 4.34 (t, $^3J_{HP}$ = $^3J_{HH}$ = 9.0 Hz, 4H, H-1 & H-3), 2.88 (s, 4H, $COCH_2CH_2CO$) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.8 (CO), 135.68 (d, $^3J_{CP}$ = 5.0 Hz, 4×C-*i*Ar), 135.68 (d, $^3J_{CP}$ = 5.9 Hz, 4×C-*i*Ar), 135.62 (d, $^3J_{CP}$ = 6.0 Hz, 4×C-*i*Ar), 135.54 (d, $^3J_{CP}$ = 7.3 Hz, 4×C-*i*Ar), 135.52 (d, $^3J_{CP}$ = 6.9 Hz, 4×C-*i*Ar), 128.42, 128.39, 128.36, 128.31, 128.30, 128.28, 128.18, 128.12, 128.11, 128.09, 128.06, 127.84, 127.80 (C-*o*Ar, C-*m*Ar, C-*p*Ar), 76.1 (br d, $^3J_{CP}$ = 5.0 Hz, C-2), 74.8 (br t, $^2J_{CP}$ = 6.1 Hz, C-4 & C-6), 73.6 (br s, C-1 & C-3), 70.6 (br s, C-5), 70.0 (d, $^2J_{CP}$ = 5.6 Hz, 4×*C*H_2Ph), 69.8 (d, $^2J_{CP}$ = 5.3 Hz, 8×*C*H_2Ph), 69.7 (d, $^2J_{CP}$ = 5.7 Hz, 4×*C*H_2Ph), 69.6 (d, $^2J_{CP}$ = 5.6 Hz, 4×*C*H_2Ph), 28.8 (*C*H_2) ppm. ^{31}P NMR (121 MHz, $CDCl_3$, ^{31}P-^{1}H decoupled): –1.07 (4P), –1.54 (4P), –2.38 (2P) ppm. HRMS (ESI) calcd. for $C_{156}H_{156}NaO_{44}P_{10}$ $[M+Na]^+$ 3065.7238; found 3065.7221.

5*O*,5'*O*-(Butane-1,4-diyl)bis[2-*O*-butyryl-1,2,3,4,6-*O*-tetrakis(bis(benzyloxy)phosphoryl)*myo*-inositol] (31e). Prepared from decaol **30e** according to General Procedure E. Yield: 76%. Colorless thick oil. R_f = 0.14 (hexanes/EtOAc 1:2). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30–7.20 (m, 80H, 16×CH_2*Ph*), 6.10 (br s, 2H, H-2), 5.05–4.92 (m, 32H, 16×C*H*$_2$Ph), 4.70 (q, $^3J_{HP}$ = $^3J_{HH}$ = 9.5 Hz, 4H, H-4 & H-6), 4.27 (td, $^3J_{HP}$ = $^3J_{HH}$ = 9.2 Hz, $^3J_{HH}$ = 2.3 Hz, 4H, H-1 & H-3), 3.48 (br s, 4H, 2×OCH_2), 2.97 (t, *J* = 9.4 Hz, 2H, H-5), 2.19 (t, *J* = 7.4 Hz, 4H, 2×$COCH_2$), 1.59–1.47 (m, 4H, 2×$COCH_2CH_2$), 1.47 (br s, 4H, 2×OCH_2CH_2), 0.86 (t, *J*=7.4 Hz, 6H, 2×CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.7 (CO), 136.0 (d, $^3J_{CP}$ = 7.3 Hz, 4×C-*i*Ar), 135.9 (d, $^3J_{CP}$ = 6.8 Hz, 4×C-*i*Ar), 135.7 (d, $^3J_{CP}$ = 5.6 Hz, 4×C-*i*Ar), 135.6 (d, $^3J_{CP}$ = 5.9 Hz, 4×C-*i*Ar), 128.55, 128.49, 128.44, 128.39, 128.28, 128.11, 128.05, 127.9 (C-*o*Ar, C-*m*Ar, C-*p*Ar), 78.9 (br s, C-5), 77.3 (br t, $^2J_{CP}$ = 6.3 Hz, C-4 & C-6), 74.0 (OCH_2), 73.6 (br s, C-1 & C-3), 69.9 (d, $^2J_{CP}$ = 5.7 Hz, 4×*C*H_2Ph), 69.5 (d, $^2J_{CP}$ = 5.3 Hz, 4×*C*H_2Ph), 69.5 (d, $^2J_{CP}$ = 5.5 Hz, 4×*C*H_2Ph), 69.3 (d, $^2J_{CP}$ = 5.2 Hz, 4×*C*H_2Ph), 69.2 (br s, C-2), 35.8 ($COCH_2$), 25.4 (OCH_2CH_2), 18.5 ($COCH_2CH_2$), 13.5 (CH_3) ppm. ^{31}P NMR (121 MHz, $CDCl_3$, ^{31}P-^{1}H decoupled): –1.13 (4P), –1.65 (4P) ppm. HRMS (ESI) calcd. for $C_{136}H_{146}NaO_{38}P_8$ $[M+Na]^+$ 2657.7285; found 2657.7302.

5*O*,5'*O*-(Butane-1,4-diyl)bis[1,2,3,4,6-*O*-pentakis(bis(benzyloxy)phosphoryl)*myo*-inositol] (31f). Prepared from decaol **30f** according to General Procedure E. Yield: 60%. Colorless thick oil. R_f = 0.17 (hexanes/EtOAc 1:2). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30–7.11 (m, 100H, 20×CH_2*Ph*), 5.61 (br d, $^3J_{HP}$ = 8.8 Hz, 2H, H-2), 5.16 (dd, $^2J_{HH}$ = 11.7 Hz, $^3J_{HP}$ = 5.8 Hz, 4H, 2×C*H*$_2$Ph), 5.07–4.92 (m, 36H, 18×C*H*$_2$Ph), 4.87 (q, $^3J_{HP}$ = $^3J_{HH}$ = 9.6 Hz, 4H, H-4 & H-6), 4.30 (br

t, $^{3}J_{HP}$ = $^{3}J_{HH}$ = 9.4 Hz, 4H, H-1 & H-3), 3.48 (br s, 4H, 2×OCH_2), 2.97 (t, *J* = 9.4 Hz, 2H, H-5), 1.45 (br s, 4H, 2×OCH_2CH_2) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 135.8 (d, $^{3}J_{CP}$ = 6.1 Hz, 4×C-*i*Ar), 135.7 (d, $^{3}J_{CP}$ = 5.5 Hz, 4×C-*i*Ar), 135.6 (d, $^{3}J_{CP}$ = 9.0 Hz, 4×C-*i*Ar), 135.52 (d, $^{3}J_{CP}$ = 7.4 Hz, 4×C-*i*Ar), 135.46 (d, $^{3}J_{CP}$ = 7.6 Hz, 4×C-*i*Ar), 128.38, 128.34, 128.27, 128.24, 128.19, 128.18, 128.09, 128.02, 127.92, 127.85, 127.73, 127.67 (C-*o*Ar, C-*m*Ar, C-*p*Ar), 78.7 (br s, C-5), 76.7 (br t, $^{2}J_{CP}$ = 5.9 Hz, C-4 & C-6), 76.0 (br d, $^{2}J_{CP}$ = 5.4 Hz, C-2), 74.0 (OCH_2), 73.6 (br s, C-1 & C-3), 69.9 (d, $^{2}J_{CP}$ = 5.6 Hz, 4×CH_2Ph), 69.7 (d, $^{2}J_{CP}$ = 5.3 Hz, 4×CH_2Ph), 69.5 (d, $^{2}J_{CP}$ = 6.0 Hz, 4×CH_2Ph), 69.4 (d, $^{2}J_{CP}$ = 5.6 Hz, 4×CH_2Ph), 69.2 (d, $^{2}J_{CP}$ = 5.2 Hz, 4×CH_2Ph), 25.2 (OCH_2CH_2) ppm. ^{31}P NMR (202 MHz, $CDCl_3$, ^{31}P-^{1}H decoupled): –0.55 (4P), –1.39 (4P), –2.86 (2P) ppm. HRMS (ESI) calcd. for $C_{156}H_{160}NaO_{42}P_{10}$ $[M+Na]^+$ 3037.7653; found 3037.7640.

5*O*,5'*O*-(Pentane-1,5-diyl)bis[2-*O*-butyryl-1,2,3,4,6-*O*-tetrakis(bis(benzyloxy)phosphoryl)*myo*-inositol] (31g). Prepared from decaol **30g** according to General Procedure E. Yield: 69%. Colorless thick oil. R_f = 0.17 (hexanes/EtOAc 1:2). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.24–7.16 (m, 80H, 16×CH_2Ph), 6.11 (t, *J* = 2.3 Hz, 2H, H-2), 5.04–4.88 (m, 32H, 16×CH_2Ph), 4.75 (q, $^{3}J_{HP}$ = $^{3}J_{HH}$ = 9.5 Hz, 4H, H-4 & H-6), 4.32 (td, $^{3}J_{HP}$ = $^{3}J_{HH}$ = 10.0 Hz, $^{3}J_{HH}$ = 2.5 Hz, 4H, H-1 & H-3), 3.53 (t, *J* = 7.5 Hz, 4H, 2×OCH_2), 3.20 (t, *J* = 9.4 Hz, 2H, H-5), 2.22 (t, *J* = 7.4 Hz, 4H, 2×$COCH_2$), 1.58–1.51 (m, 4H, 2×$COCH_2CH_2$), 1.47–1.41 (m, 4H, 2×OCH_2CH_2), 0.87 (t, *J* = 7.4 Hz, 6H, 2×CH_3), 0.89 – 0.81 (m, 2H, $OCH_2CH_2CH_2$) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.5 (CO), 135.8 (d, $^{3}J_{CP}$ = 7.2 Hz, 4×C-*i*Ar), 135.7 (d, $^{3}J_{CP}$ = 7.0 Hz, 4×C-*i*Ar), 135.6 (d, $^{3}J_{CP}$ = 7.0 Hz, 4×C-*i*Ar), 135.5 (d, $^{3}J_{CP}$ = 7.1 Hz, 4×C-*i*Ar), 128.35, 128.32, 128.29, 128.27, 128.25, 128.23, 128.19, 128.09, 127.9, 127.8, 127.7 (C-*o*Ar, C-*m*Ar, C-*p*Ar), 79.0 (br s, C-5), 76.9 (br t, $^{2}J_{CP}$ = 6.5 Hz, C-4 & C-6), 73.7 (OCH_2), 73.5 (br s, C-1 & C-3), 69.7 (d, $^{2}J_{CP}$ = 5.6 Hz, 4×CH_2Ph), 69.4 (d, $^{2}J_{CP}$ = 5.6 Hz, 4×CH_2Ph), 69.3 (d, $^{2}J_{CP}$ = 6.0 Hz, 4×CH_2Ph), 69.2 (d, $^{2}J_{CP}$ = 5.3 Hz, 4×CH_2Ph), 69.1 (br s, C-2), 35.7 ($COCH_2$), 29.3 (OCH_2CH_2), 21.5 ($OCH_2CH_2CH_2$), 18.3 ($COCH_2CH_2$), 13.4 (CH_3) ppm. ^{31}P NMR (121 MHz, $CDCl_3$, ^{31}P-^{1}H decoupled): –1.46 (4P), –1.69 (4P) ppm. HRMS (ESI) calcd. for $C_{137}H_{148}NaO_{38}P_8$ $[M+Na]^+$ 2671.7442; found 2671.7456.

5*O*,5'*O*-(Pentane-1,5-diyl)bis[1,2,3,4,6-*O*-pentakis(bis(benzyloxy)phosphoryl)*myo*-inositol] (31h). Prepared from decaol **30h** according to General Procedure E. Yield: 67%. Colorless thick oil. R_f = 0.18 (hexanes/EtOAc 1:1). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30–7.14 (m, 100H, 20×CH_2Ph), 5.61 (br d, $^{3}J_{HP}$ = 8.8 Hz, 2H, H-2), 5.16 (dd, $^{2}J_{HH}$ = 11.7 Hz, $^{3}J_{HP}$ = 6.1 Hz, 4H, 2×CH_2Ph), 5.06–4.85 (m, 40H, H-4 & H-6 & 18×CH_2Ph), 4.32 (br t, $^{3}J_{HP}$ = $^{3}J_{HH}$ = 9.4 Hz, 4H, H-1 & H-3), 3.54 (br t, *J* = 7.4 Hz, 4H, 2×OCH_2), 3.21 (t, *J* = 9.3 Hz, 2H, H-5), 1.47–1.37 (m, 4H, 2×OCH_2CH_2), 1.32–1.23 (m, 2H, $OCH_2CH_2CH_2$) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 136.1–135.7 (m, 20C, 20×C-*i*Ar), 128.45, 128.36, 128.30, 128.26, 128.17, 128.15, 128.12, 128.07, 127.98, 127.86, 127.82 (C-*o*Ar, C-*m*Ar, C-*p*Ar), 79.2 (br s, C-5), 76. 8 (br t, $^{2}J_{CP}$ = 6.4 Hz, C-4 & C-6) 76.2 (br d, $^{2}J_{CP}$ = 5.0 Hz, C-2), 74.1 (OCH_2), 73.9 (br s, C-1 & C-3), 70.0 (d, $^{2}J_{CP}$ = 5.7 Hz, 4×CH_2Ph), 69.8 (d, $^{2}J_{CP}$ = 5.9 Hz, 4×CH_2Ph), 69.8 (d, $^{2}J_{CP}$ = 6.7 Hz, 4×CH_2Ph), 69.6 (d, $^{2}J_{CP}$ = 5.6 Hz, 4×CH_2Ph), 69.4 (d, $^{2}J_{CP}$ = 5.3 Hz, 4×CH_2Ph), 29.5 (OCH_2CH_2), 21.6 ($OCH_2CH_2CH_2$) ppm. ^{31}P NMR (121 MHz, $CDCl_3$, ^{31}P-^{1}H decoupled): –0.89 (4P), –1.45 (4P), –2.77 (2P) ppm. HRMS (ESI) calcd. for $C_{157}H_{162}NaO_{42}P_{10}$ $[M+Na]^+$ 3051.7809; found 3051.7827.

5,5'-Bis(tetrasodium 2-*O*-butyryl-1,3,4,6-*O*-tetrakisphosphono-*myo*-inositolyl) malonate (5). Prepared from decaol **31a** according to General Procedure F. Reaction time: 72 h. Yield: 100%. White amorphous solid. FTIR (KBr): 1742, 1647 cm^{-1}. ^{1}H NMR (500 MHz, D_2O)[S8] δ 5.75 (s, 2H, H-2), 5.24 (t, *J* = 9.6 Hz, 2H, H-5), 4.53 (q, $^{3}J_{HP}$ = $^{3}J_{HH}$ = 9.3 Hz, 4H, H-4 & H-6), 4.35 (br t, $^{3}J_{HP}$ = 9.1 Hz, 2H, H-1 & H-3), 2.50 (t, *J* = 7.3 Hz, 4H, 2×$COCH_2$), 1.71–1.64 (m, 4H, 2×$COCH_2CH_2$), 0.95 (t, *J*=7.4 Hz, 6H, 2×CH_3) ppm. ^{13}C NMR (126 MHz, D_2O)[S8] δ 175.7 (CO_{but}), 168.4 (CO_{mal}), 75.0 (br t, $^{2}J_{CP}$ = 5.1 Hz, C-4 & C-6), 73.8 (br s, C-5), 72.6 (br s, C-1 & C-3), 72.2 (br s, C-2), 39.2 (quintet, J_{CD} = 18.0 Hz, CD_2), 36.2 ($COCH_2$), 18.3 ($COCH_2CH_2$), 13.2 (CH_3) ppm. ^{31}P NMR (202 MHz, D_2O, ^{31}P-^{1}H decoupled): –0.12 (4P), –0.17 (4P) ppm. HRMS (ESI) calcd. for $C_{23}H_{36}Na_7O_{40}P_8$ $[M–Na]^–$ 1360.7973; found 1360.7986.

5,5'-Bis(pentasodium 1,2,3,4,6-*O*-pentakisphosphono-*myo*-inositolyl) malonate (6). Prepared from decaol **31b** according to General Procedure F. Reaction time: 48 h. Yield: 100%. White amorphous solid. FTIR (KBr): 1769, 1211 cm^{-1}. ^{1}H NMR (500 MHz, D_2O)[S8] δ 5.17 (t, J = 9.6 Hz, 2H, H-5), 4.86 (br s, 2H, H-2), 4.48 (q, $^{3}J_{HP} = {}^{3}J_{HH}$ = 9.5 Hz, 4H, H-4 & H-6), 4.22 (br t, $^{3}J_{HP} = {}^{3}J_{HH}$ = 8.8 Hz, 4H, H-1 & H-3) ppm. ^{13}C NMR (126 MHz, D_2O)[S8] δ 168.1 (CO), 75.0 (d, $^{2}J_{CP}$ = 5.2 Hz, C-2), 74.2 (br t, $^{2}J_{CP}$ = 5.9 Hz, C-4 & C-6), 73.5 (br s, C-5), 73.2 (br s, C-1 & C-3), 39.8 (quintet, J_{CD} = 18.3 Hz, CD_2) ppm. ^{31}P NMR (121 MHz, D_2O, ^{31}P-^{1}H decoupled): 0.24 (4P), –0.14 (6P) ppm. HRMS (ESI) calcd. for $C_{15}H_{24}Na_9O_{44}P_{10}$ $[M–Na]^-$ 1424.6101; found 1424.6112.

5,5'-Bis(tetrasodium 2-*O*-butyryl-1,3,4,6-*O*-tetrakisphosphono-*myo*-inositolyl) succinate (7). Prepared from decaol **31c** according to General Procedure F. Reaction time: 72 h. Yield: 99%. White amorphous solid. FTIR (KBr): 1735, 1183 cm^{-1}. ^{1}H NMR (500 MHz, D_2O) δ 5.67 (br s, 2H, H-2), 5.12 (t, J = 9.6 Hz, 2H, H-5), 4.47 (q, $^{3}J_{HP} = {}^{3}J_{HH}$ = 9.5 Hz, 4H, H-4 & H-6), 4.28 (td, $^{3}J_{HP} = {}^{3}J_{HH}$ = 9.7 Hz, $^{3}J_{HH}$ = 1.7 Hz, 4H, H-1 & H-3), 2.83 (s, 4H, $COCH_2CH_2CO$), 2.46 (t, J = 7.3 Hz, 4H, 2×$COCH_2$), 1.67–1.59 (m, 4H, 2×$COCH_2CH_2$), 0.91 (t, J = 7.4 Hz, 6H, 2×CH_3) ppm. ^{13}C NMR (126 MHz, D_2O) δ 175.5 (CO_{but}), 174.3 (CO_{suc}), 74.7 (br t, $^{2}J_{CP}$ = 5.3 Hz, C-4 & C-6), 73.1 (br s, C-5), 72.2 (br s, C-1 & C-3), 72.0 (br s, C-2), 35.8 ($COCH_2$), 28.8 ($COCH_2CH_2CO$), 18.0 ($COCH_2CH_2$), 12.9 (CH_3) ppm. ^{31}P NMR (121 MHz, D_2O, ^{31}P-^{1}H decoupled): 0.42 (4P), –0.13 (4P) ppm. HRMS (ESI) calcd. for $C_{24}H_{38}Na_7O_{40}P_8$ $[M–Na]^-$ 1374.8130; found 1374.8126.

5,5'-Bis(pentasodium 1,2,3,4,6-*O*-pentakisphosphono-*myo*-inositolyl) succinate (8). Prepared from decaol **31d** according to General Procedure F. Reaction time: 72 h. Yield: 95%. White amorphous solid. FTIR (KBr): 1732, 1196 cm^{-1}. ^{1}H NMR (500 MHz, D_2O) δ 5.14 (br t, J = 9.5 Hz, 2H, H-5), 4.89 (br s, 2H, H-2), 4.50 (br q, $^{3}J_{HP} = {}^{3}J_{HH}$ = 9.2 Hz, 4H, H-4 & H-6), 4.25 (br t, $^{3}J_{HP} = {}^{3}J_{HH}$ = 8.2 Hz, 4H, H-1 & H-3), 2.83 (s, 4H, 2×$COCH_2$) ppm. ^{13}C NMR (126 MHz, D_2O) δ 174.3 (CO), 75.2 (br s, C-2), 74.3 (br t, $^{2}J_{CP}$ = 5.9 Hz, C-4 & C-6), 73.2 (br s, C-1 & C-3), 73.1 (br s, C-5), 28.9 ($COCH_2$) ppm. ^{31}P NMR (202 MHz, D_2O, ^{31}P-^{1}H decoupled): 0.60 (4P), 0.05 (2P), –0.20 (4P) ppm. HRMS (ESI) calcd. for $C_{16}H_{26}Na_9O_{44}P_{10}$ $[M–Na]^-$ 1438.6258; found 1438.6242.

5*O*,5'*O*-(Butane-1,4-diyl)bis[tetrasodium 2-*O*-butyryl-1,3,4,6-*O*-tetrakisphosphono-*myo*-inositol] (9). Prepared from decaol **31e** according to General Procedure F. Reaction time: 72 h. Yield: 98%. White amorphous solid. FTIR (KBr): 1739, 1203, 1050 cm^{-1}. ^{1}H NMR (500 MHz, D_2O) δ 5.61 (br s, 2H, H-2), 4.37 (q, $^{3}J_{HP} = {}^{3}J_{HH}$ = 9.4 Hz, 4H, H-4_4 & H-6), 4.21 (td, $^{3}J_{HP} = {}^{3}J_{HH}$ = 9.8 Hz, $^{3}J_{HH}$ = 2.4 Hz, 4H, H-1 & H-3), 3.77 (br s, 4H, 2×OCH_2), 3.46 (t, J = 9.4 Hz, 2H, H-5), 2.44 (t, J = 7.3 Hz, 4H, 2×$COCH_2$), 1.65–1.58 (m, 8H, 2×$COCH_2CH_2$ & 2×OCH_2CH_2), 0.90 (t, J = 7.4 Hz, 6H, 2×CH_3) ppm. ^{13}C NMR (126 MHz, D_2O) δ 175.5 (CO), 80.1 (br s, C-5), 76.5 (br t, $^{2}J_{CP}$ = 5.5 Hz, C-4 & C-6), 73.3 (OCH_2), 72.6 (br d, $^{2}J_{CP}$ = 4.3 Hz, C-1 & C-3), 71.9 (br s, C-2), 35.8 ($COCH_2$), 25.5 (OCH_2CH_2), 18.0 ($COCH_2CH_2$), 12.9 (CH_3) ppm. ^{31}P NMR (202 MHz, D_2O, ^{31}P-^{1}H decoupled): 0.08 (4P), –0.59 (4P) ppm. HRMS (ESI) calcd. for $C_{24}H_{42}Na_7O_{38}P_8$ $[M–Na]^-$ 1346.8544; found 1346.8546.

5*O*,5'*O*-(Butane-1,4-diyl)bis[pentasodium 1,2,3,4,6-*O*-pentakisphosphono-*myo*-inositol] (10). Prepared from decaol **31f** according to General Procedure F. Reaction time: 72 h. Yield: 99%. White amorphous solid. FTIR (KBr): 1203, 1062 cm^{-1}. ^{1}H NMR (500 MHz, D_2O) δ 4.79 (2H, H-2, obscured), 4.41 (q, $^{3}J_{HP} = {}^{3}J_{HH}$ = 9.2 Hz, 4H, H-4 & H-6), 4.15 (br t, $^{3}J_{HP}$ = 8.8 Hz, 4H, H-1 & H-3), 3.80 (br s, 4H, 2×OCH_2), 3.47 (br t, J = 9.3 Hz, 2H, H-5), 1.65 (br s, 4H, 2×OCH_2CH_2) ppm. ^{13}C NMR (126 MHz, D_2O) δ 80.3 (m, C-5), 76.2 (br t, $^{2}J_{CP}$ = 6.2 Hz, C-4 & C-6), 75.6–75.5 (m, C-2), 73.52 (m, C-1 & C-3), 73.48 (OCH_2), 25.5 (OCH_2CH_2) ppm. ^{31}P NMR (202 MHz, D_2O, ^{31}P-^{1}H decoupled): 0.52 (4P), –0.49 (6P) ppm. HRMS (ESI) calcd. for $C_{16}H_{30}Na_9O_{42}P_{10}$ $[M–Na]^-$ 1410.6673; found 1410.6653.

5*O*,5'*O*-(Pentane-1,5-diyl)bis[tetrasodium 2-*O*-butyryl-1,3,4,6-*O*-tetrakisphosphono-*myo*-inositol] (11). Prepared from decaol **31g** according to General Procedure F. Reaction time: 72 h. Yield: 100%. White amorphous solid. FTIR (KBr): 1742, 1200, 1055 cm^{-1}. ^{1}H NMR (500

MHz, D_2O) δ 5.61 (s, 2H, H-2), 4.37 (q, $^3J_{HP}$ = $^3J_{HH}$ = 9.3 Hz, 4H, H-4 & H-6), 4.20 (t, $^3J_{HP}$ = $^3J_{HH}$ = 9.7 Hz, 4H, H-1 & H-3), 3.75 (br t, J = 6.9 Hz, 4H, 2×OCH_2), 3.46 (t, J = 9.4 Hz, 2H, H-5), 2.45 (t, J = 7.2 Hz, 4H, 2×$COCH_2$), 1.65–1.60 (m, 8H, 2×$COCH_2CH_2$ & 2×OCH_2CH_2), 1.30–1.25 (m, 2H, $OCH_2CH_2CH_2$), 0.91 (t, J = 7.4 Hz, 6H, 2×OCH_3) ppm. ^{13}C NMR (126 MHz, D_2O) δ 175.5 (CO), 80.1 (br s, C-5), 76.6 (br t, $^2J_{CP}$ = 5.3 Hz, C-4 & C-6), 73.4 (OCH_2), 72.6 (br d, $^2J_{CP}$ = 4.6 Hz, C-1 & C-3), 72.0 (br s, C-2), 35.9 ($COCH_2$), 29.1 (OCH_2CH_2), 21.2 ($OCH_2CH_2CH_2$), 18.0 ($COCH_2CH_2$), 13.0 (CH_3) ppm. ^{31}P NMR (121 MHz, D_2O, ^{31}P-^{1}H decoupled): 0.43 (4P), –0.33 (4P) ppm. HRMS (ESI) calcd. for $C_{25}H_{44}Na_7O_{38}P_8$ $[M–Na]^-$ 1360.8701; found 1360.8693.

5*O*,5'*O*-(Pentane-1,5-diyl)bis[pentasodium 1,2,3,4,6-*O*-pentakisphosphono-*myo*-inositol] (12). Prepared from decaol **31h** according to General Procedure F. Reaction time: 48 h. Yield: 98%. White amorphous solid. FTIR (KBr): 1200, 1055 cm^{-1}. ^{1}H NMR (500 MHz, D_2O) δ 4.79 (2H, H-2, obscured), 4.35 (q, $^3J_{HP}$ = $^3J_{HH}$ = 7.8 Hz, 4H, H-4 & H-6), 4.08 (br t, $^3J_{HP}$ = 8.7 Hz, 4H, H-1 & H-3), 3.75 (t, J = 5.0 Hz, 4H, 2×OCH_2), 3.41 (t, J = 9.2 Hz, 2H, H-5), 1.65–1.59 (m, 4H, 2×OCH_2CH_2), 1.28–1.22 (m, 2H, $OCH_2CH_2CH_2$) ppm. ^{13}C NMR (126 MHz, D_2O) δ 80.5 (br s, C-5), 76.3 (br s, C-4 & C-6), 75.5 (br s, C-2), 73.6 (OCH_2), 73.5 (br s, C-1 & C-3), 29.2 (OCH_2CH_2), 21.2 ($OCH_2CH_2CH_2$) ppm. ^{31}P NMR (202 MHz, D_2O, ^{31}P-^{1}H decoupled): 1.37 (4P), 0.09 (2P), –0.38 (4P) ppm. HRMS (ESI) calcd. for $C_{17}H_{32}Na_9O_{42}P_{10}$ $[M–Na]^-$ 1424.6829; found 1424.6851.

S1 S. J. Mills, A. M. Riley, C. Liu, M. F. Mahon and B. V. L. Potter, *Chem. Eur. J.*, 2003, **9**, 6207.

S2 A. E. Koumbis, C. D. Duarte, C. Nicolau and J.-M. Lehn, *ChemMedChem*, 2011, **6**, 169.

S3 M. T. Rudolf, T. Kaiser, A. H. Guse, G. W. Mayr and C. Schultz, *Liebigs Ann./Recueil*, 1997, 1861.

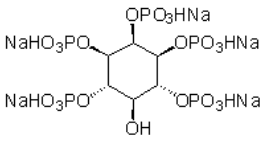
S4 A. M. Riley, H. Wang, J. D. Weaver, S. B. Shears and B. V. L. Potter, *Chem. Commun.*, 2012, **48**, 11292.

S5 H. Wang, H. Y. Godage, A. M. Riley, J. D. Weaver, S. B. Shears and B. V.L. Potter, *Chem. Biol.*, 2014, **21**, 689.

S6 A. E. Martin, T. M. Ford and J. E. Bulkowski, *J. Org. Chem.*, 1982, **47**, 412.

S7 D. H. Bus, D. J. Olszanski, J. C. Stevens, W. P. Schammel, M. Kojima, N. Herron, L. . Zimmer, K. A. Holter and J. Mocak, *J. Am. Chem. Soc.*, 1981, **103**, 1472.

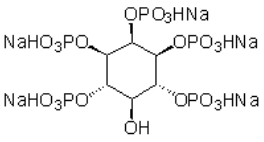
S8 For compounds **5** and **6** a rapid H-D exchange of malonic protons in the NMR solvent (D_2O) occurs. Therefore, these protons disappear in the ^{1}H NMR spectrum, whereas a quintet is observed in the ^{13}C NMR spectrum for the CD_2 group (around 40 ppm). The complete insolubility of these compounds in non-protic solvents did not allow us to run other NMR experiments. The provided data (NMR and HRMS) are consisted with the given structures.



3

D_2O, 500 MHz, 20 °C

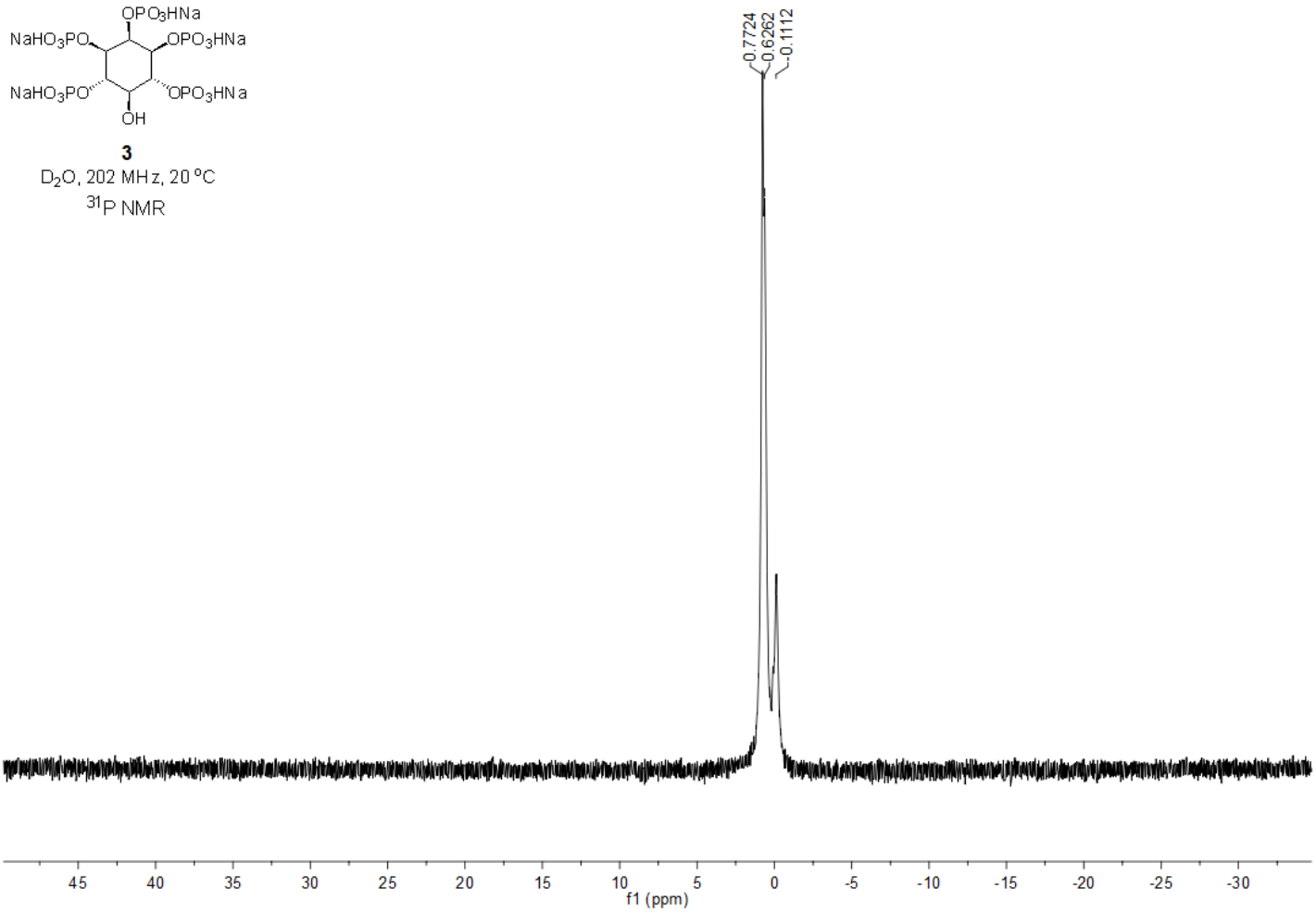
^1H NMR

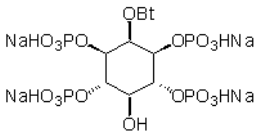


3

D_2O, 126 MHz, 20 °C

^{13}C NMR

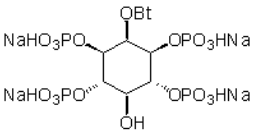

OPO3HNa
NaHO3PO
OPO3HNa
NaHO3PO
OPO3HNa
OH
3
D2O, 202 MHz, 20 °C
31P NMR
0.7724
0.6262
-0.1112
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
f1 (ppm)



4

D_2O, 500 MHz, 20 °C

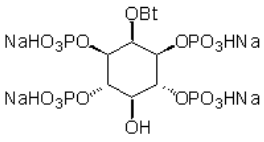
^1H NMR



4

D_2O, 126 MHz, 20 °C

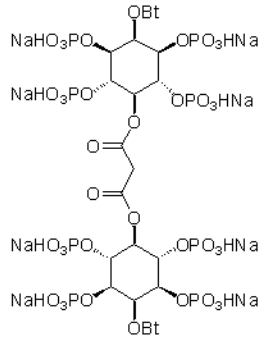
^{13}C NMR



4

D_2O, 202 MHz, 20 °C

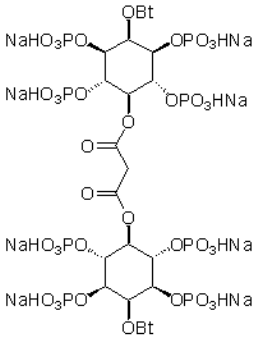
^{31}P NMR



5

D_2O, 500 MHz, 20 °C

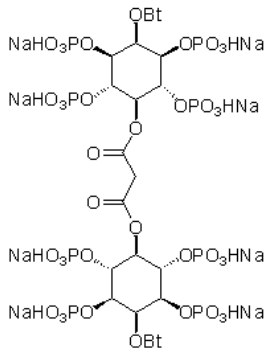
^1H NMR



5

D_2O, 126 MHz, 20 °C

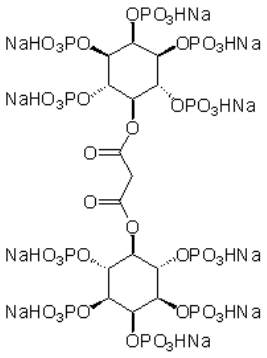
^{13}C NMR



5

D_2O, 202 MHz, 20 °C

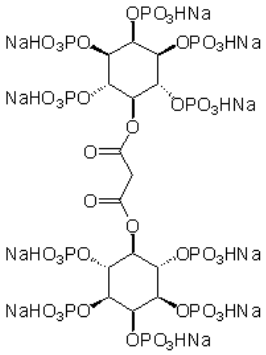
^{31}P NMR



6

D_2O, 500 MHz, 20 °C

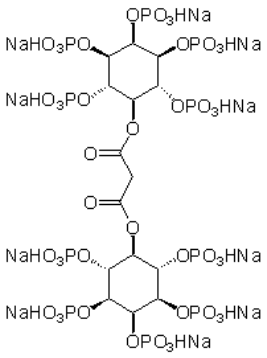
^1H NMR



6

D_2O, 126 MHz, 20 °C

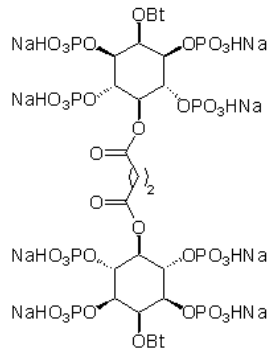
^{13}C NMR



6

D_2O, 121 MHz, 20 °C

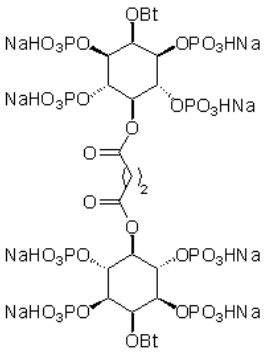
^{31}P NMR



7

D_2O, 500 MHz, 20 °C

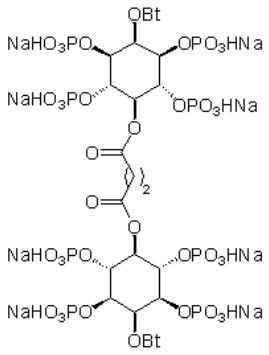
^1H NMR



7

D_2O, 126 MHz, 20 °C

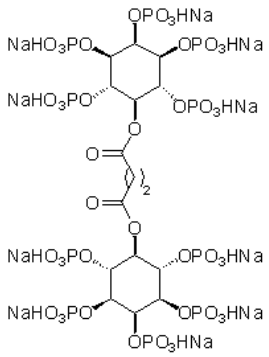
^{13}C NMR



7

D_2O, 121 MHz, 20 °C

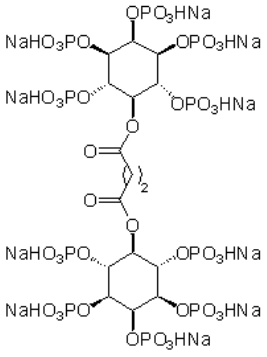
^{31}P NMR



8

D_2O, 500 MHz, 20 °C

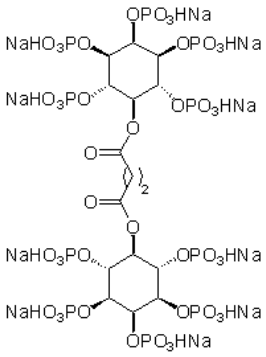
^1H NMR



8

D_2O, 126 MHz, 20 °C

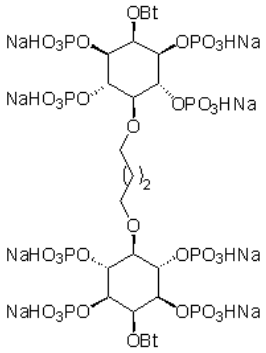
^{13}C NMR



8

D_2O, 202 MHz, 20 °C

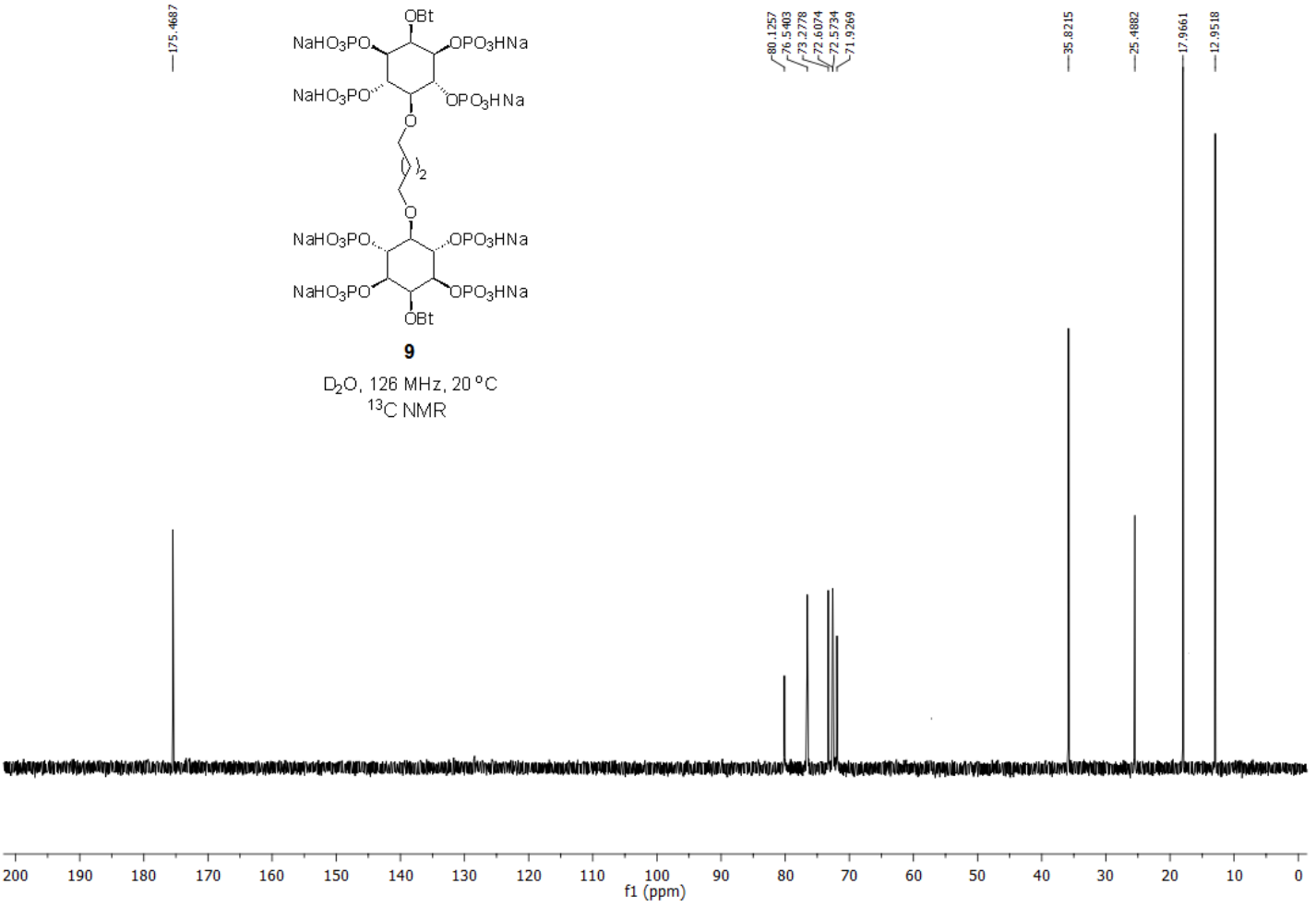
^{31}P NMR



9

D_2O, 500 MHz, 20 °C

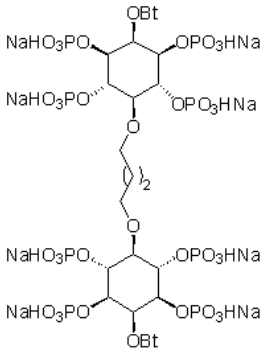
^1H NMR



9

D_2O, 126 MHz, 20 °C

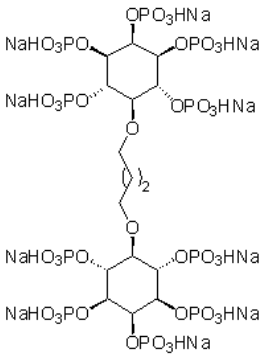
^{13}C NMR



9

D_2O, 202 MHz, 20 °C

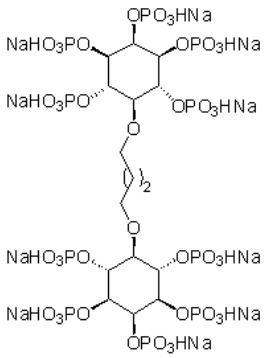
^{31}P NMR



10

D_2O, 500 MHz, 20 °C

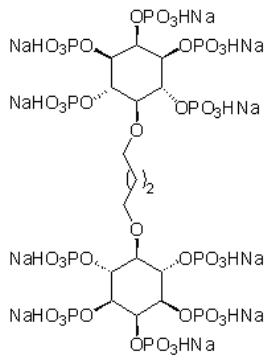
^1H NMR



10

D_2O, 126 MHz, 20 °C

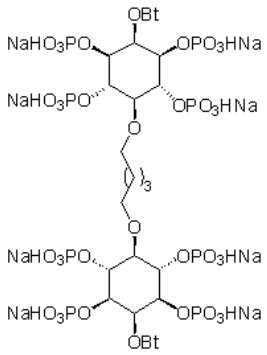
^{13}C NMR



10

D_2O, 202 MHz, 20 °C

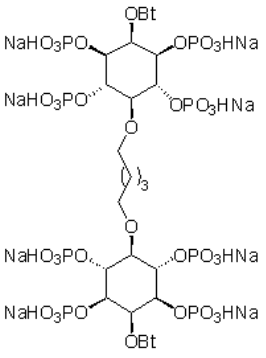
^{31}P NMR



11

D_2O, 500 MHz, 20 °C

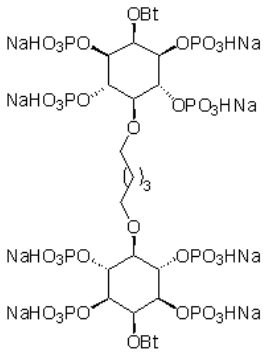
^1H NMR



11

D_2O, 126 MHz, 20 °C

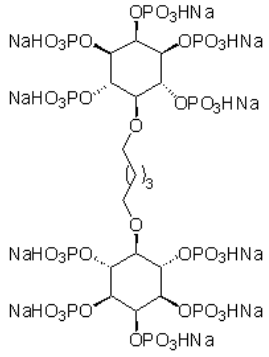
^{13}C NMR



11

D_2O, 121 MHz, 20 °C

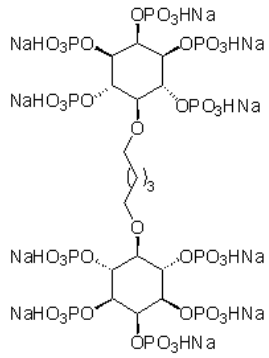
^{31}P NMR



12

D_2O, 500 MHz, 20 °C

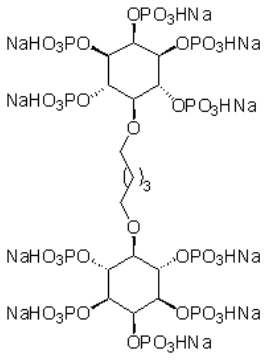
^1H NMR



12

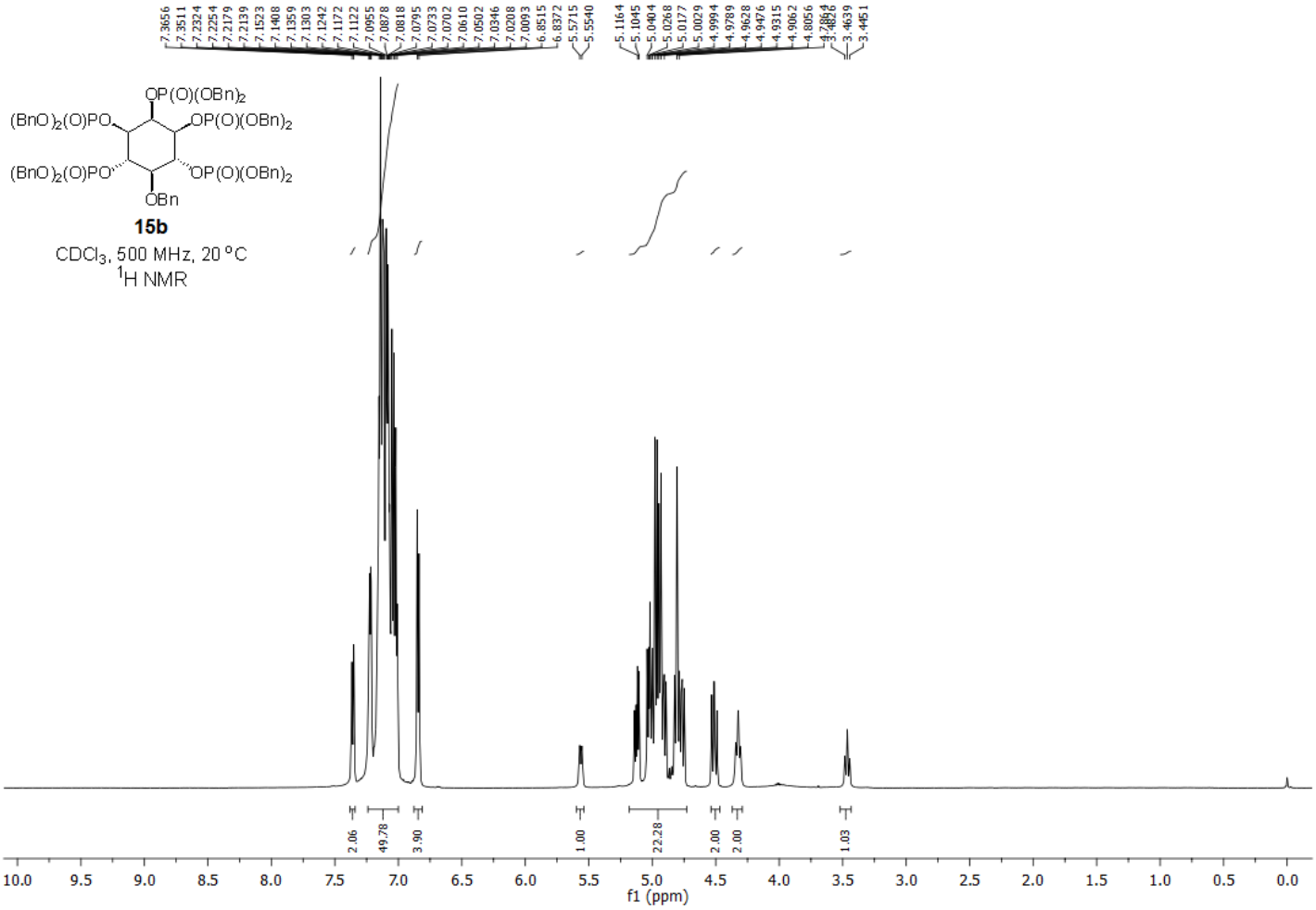
D_2O, 126 MHz, 20 °C

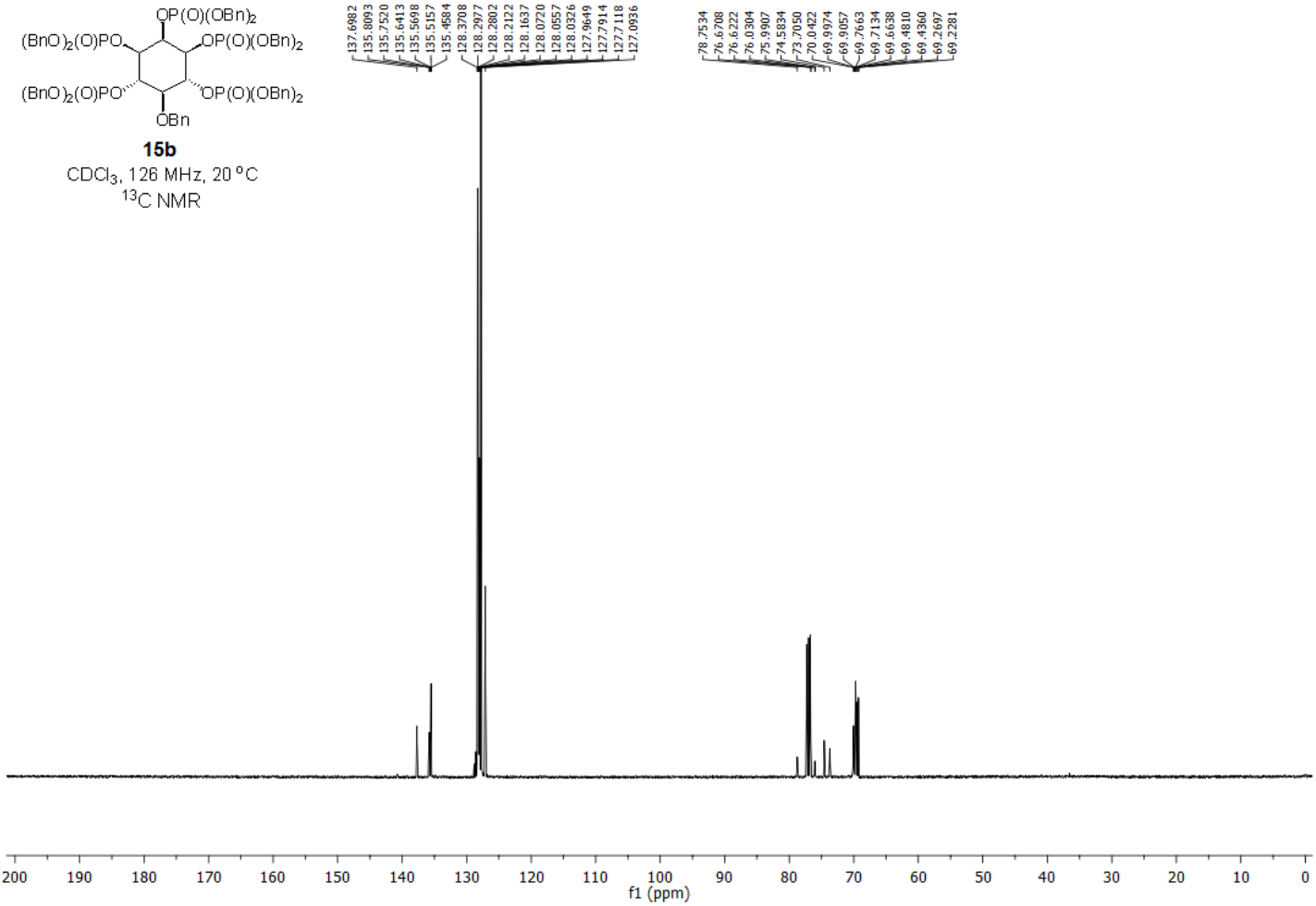
^{13}C NMR

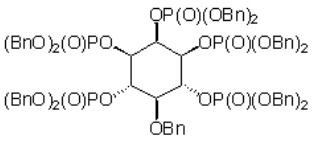


12

D_2O, 202 MHz, 20 °C
^{31}P NMR


OP(O)(OBn)2
(BnO)2(O)PO
OP(O)(OBn)2
(BnO)2(O)PO
OP(O)(OBn)2
OBn
15b
CDCl3, 500 MHz, 20 °C
1H NMR
f1 (ppm)

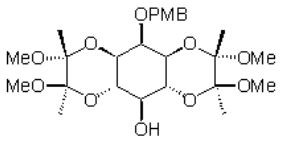

OP(O)(OBn)2
(BnO)2(O)PO
OP(O)(OBn)2
(BnO)2(O)PO
OP(O)(OBn)2
OBn
15b
CDCl3, 126 MHz, 20 °C
13C NMR
f1 (ppm)



15b

$CDCl_3$, 202 MHz, 20 °C

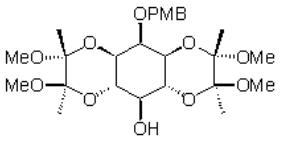
^{31}P NMR



16a

$CDCl_3$, 500 MHz, 20 °C

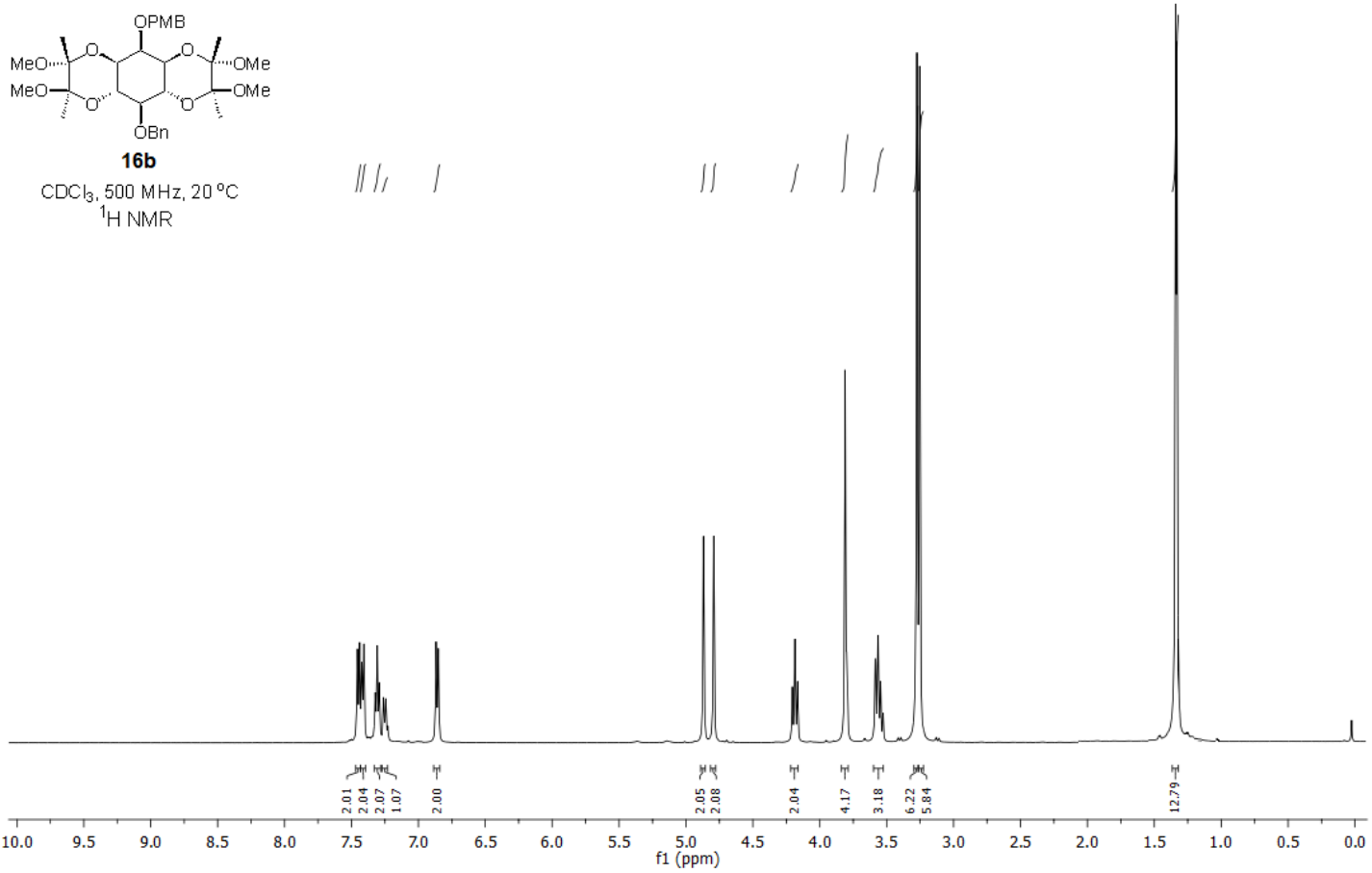
^1H NMR

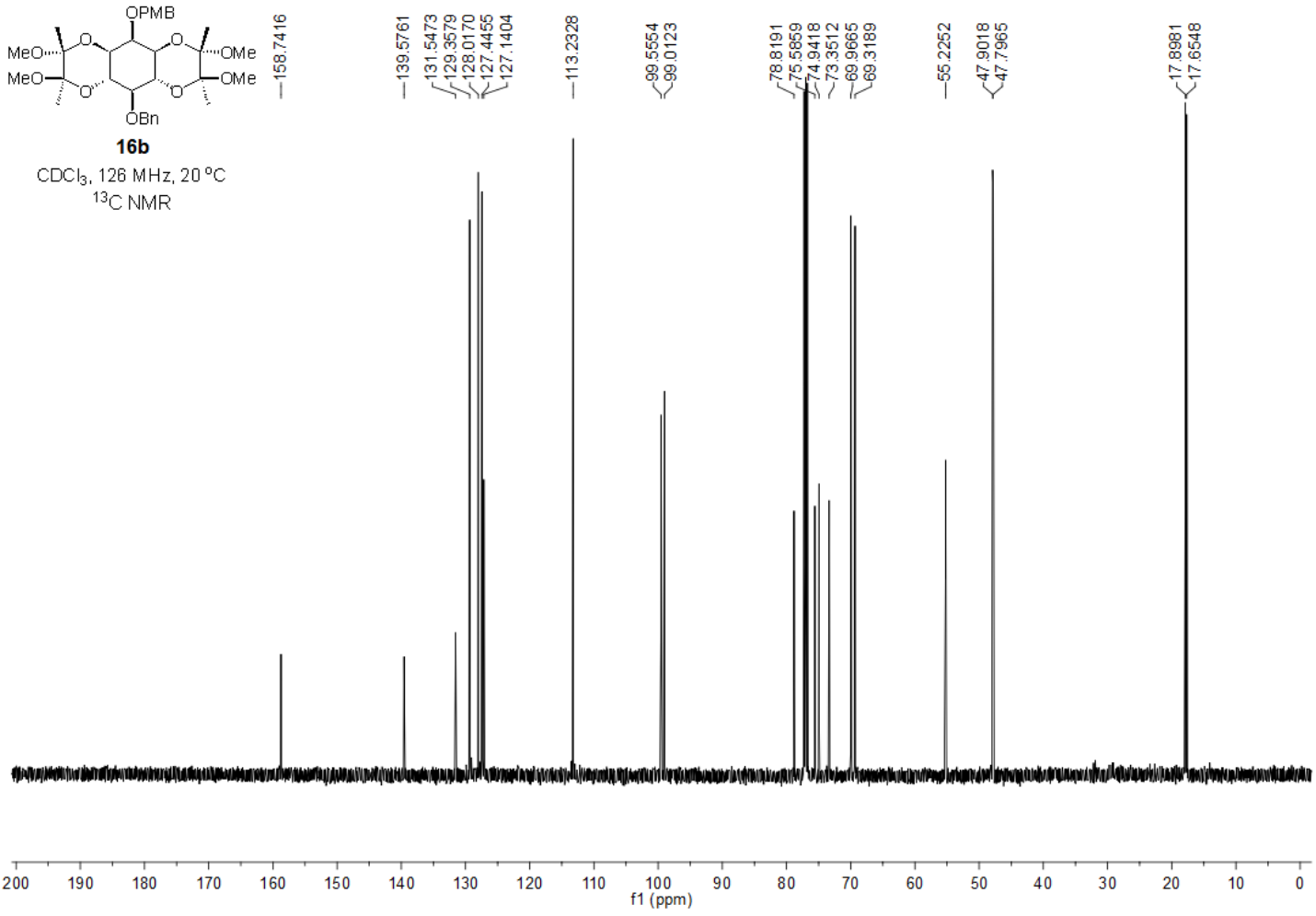


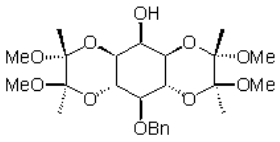
16a

$CDCl_3$, 126 MHz, 20 °C

^{13}C NMR


OPMB
MeO
MeO
OMe
OMe
OBn
16b
CDCl3, 500 MHz, 20 °C
1H NMR
f1 (ppm)

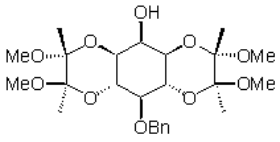

OPMB
MeO
MeO
OMe
OMe
OBn
16b
CDCl3, 126 MHz, 20 °C
13C NMR
f1 (ppm)



16c

$CDCl_3$, 500 MHz, 20 °C

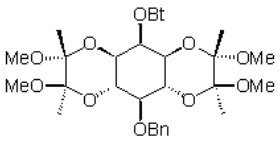
^1H NMR



16c

$CDCl_3$, 126 MHz, 20 °C

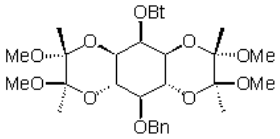
^{13}C NMR



16d

$CDCl_3$, 500 MHz, 20 °C

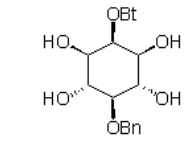
^1H NMR



16d

$CDCl_3$, 126 MHz, 20 °C

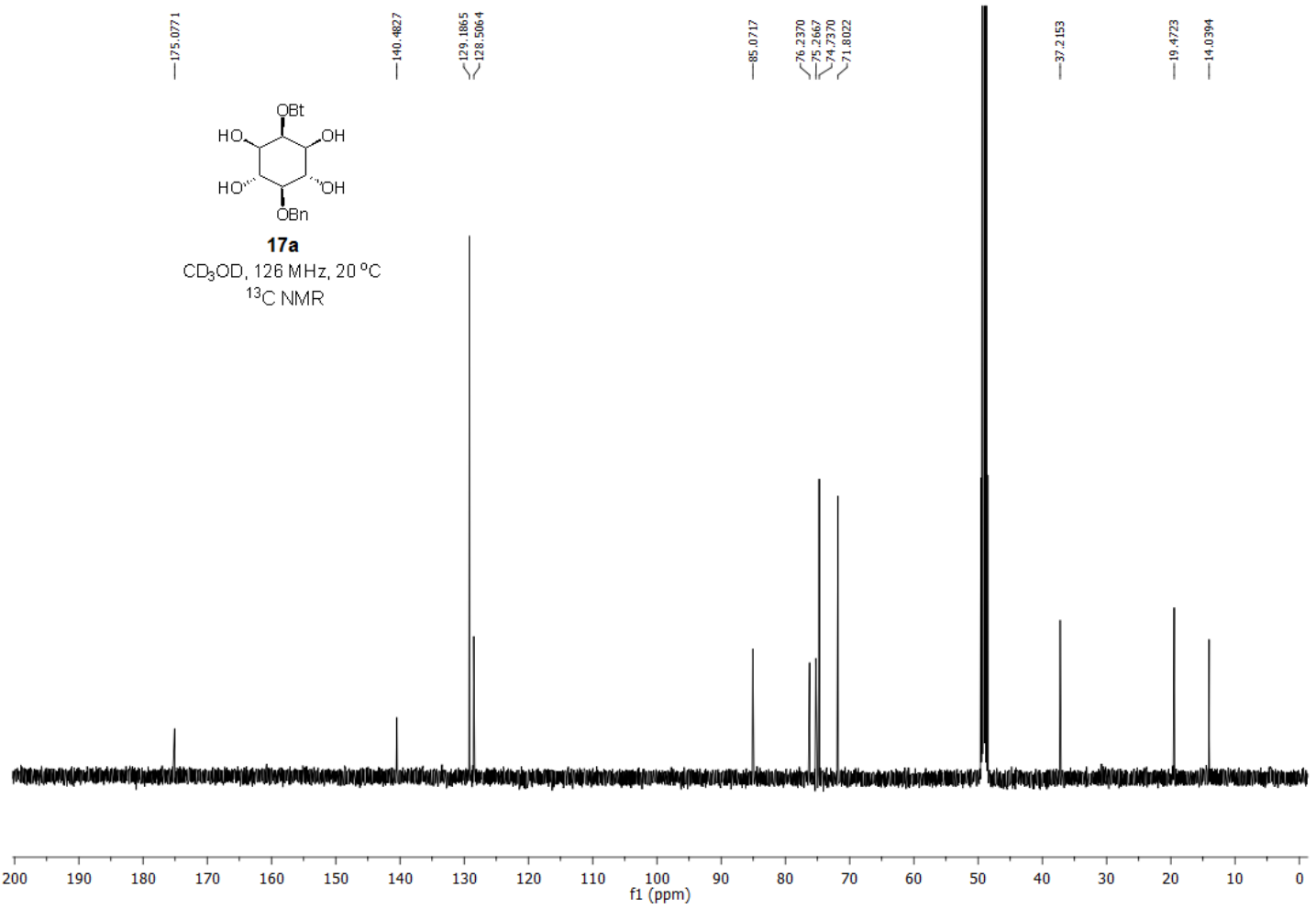
^{13}C NMR



17a

CD_3OD, 500 MHz, 20 °C

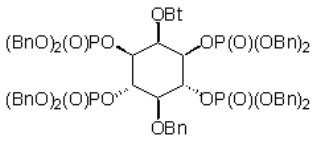
^1H NMR



17a

CD_3OD, 126 MHz, 20 °C

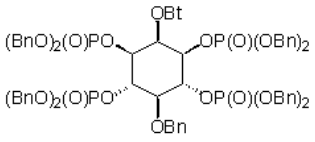
^{13}C NMR



17b

$CDCl_3$, 500 MHz, 20 °C

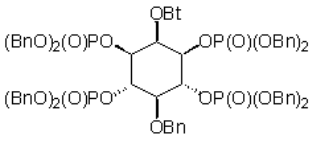
^1H NMR



17b

$CDCl_3$, 126 MHz, 20 °C

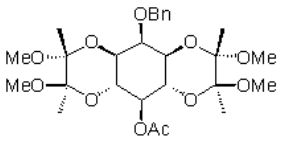
^{13}C NMR



17b

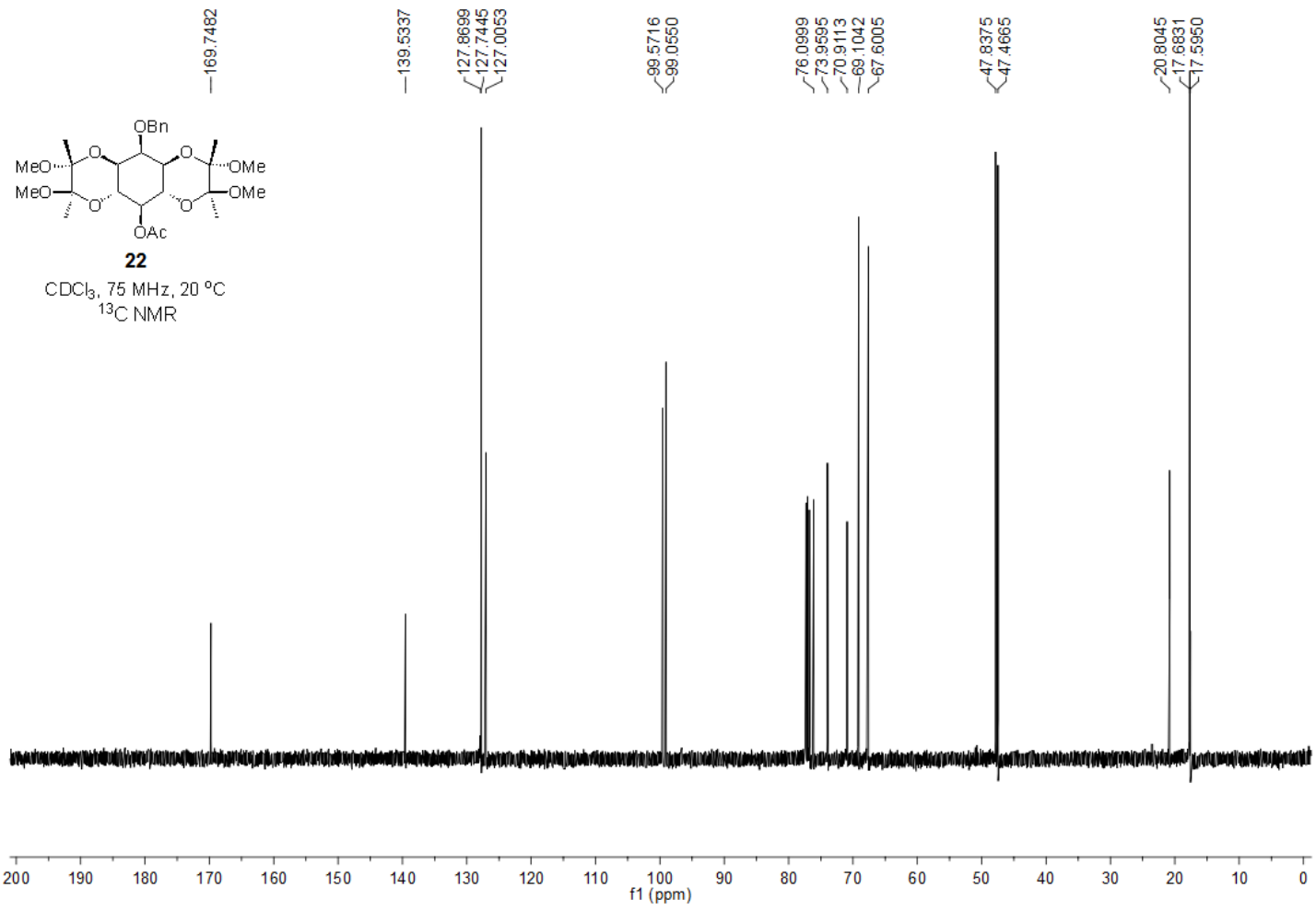
$CDCl_3$, 202 MHz, 20 °C

^{31}P NMR



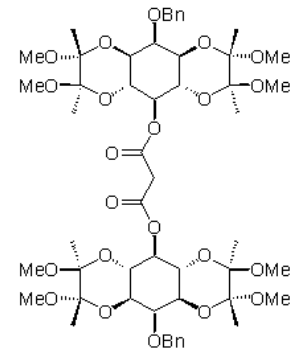
22

$CDCl_3$, 500 MHz, 20 °C
^1H NMR



22

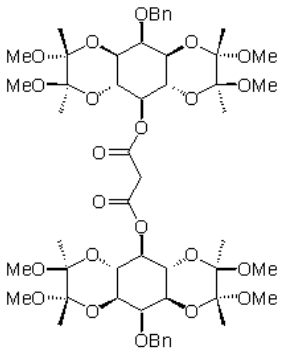
$CDCl_3$, 75 MHz, 20 °C
^{13}C NMR



23a

$CDCl_3$, 300 MHz, 20 °C

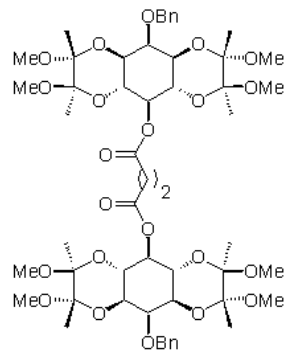
^1H NMR



23a

$CDCl_3$, 75 MHz, 20 °C

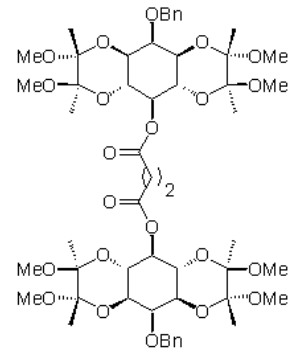
^{13}C NMR



23b

$CDCl_3$, 300 MHz, 20 °C

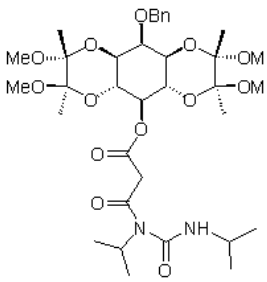
^1H NMR



23b

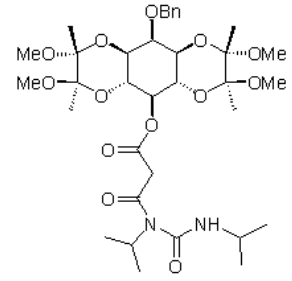
$CDCl_3$, 75 MHz, 20 °C

^{13}C NMR



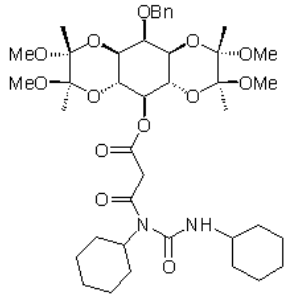
24a

$CDCl_3$, 300 MHz, 20 °C
^1H NMR



24a

$CDCl_3$, 75 MHz, 20 °C
^{13}C NMR



24b

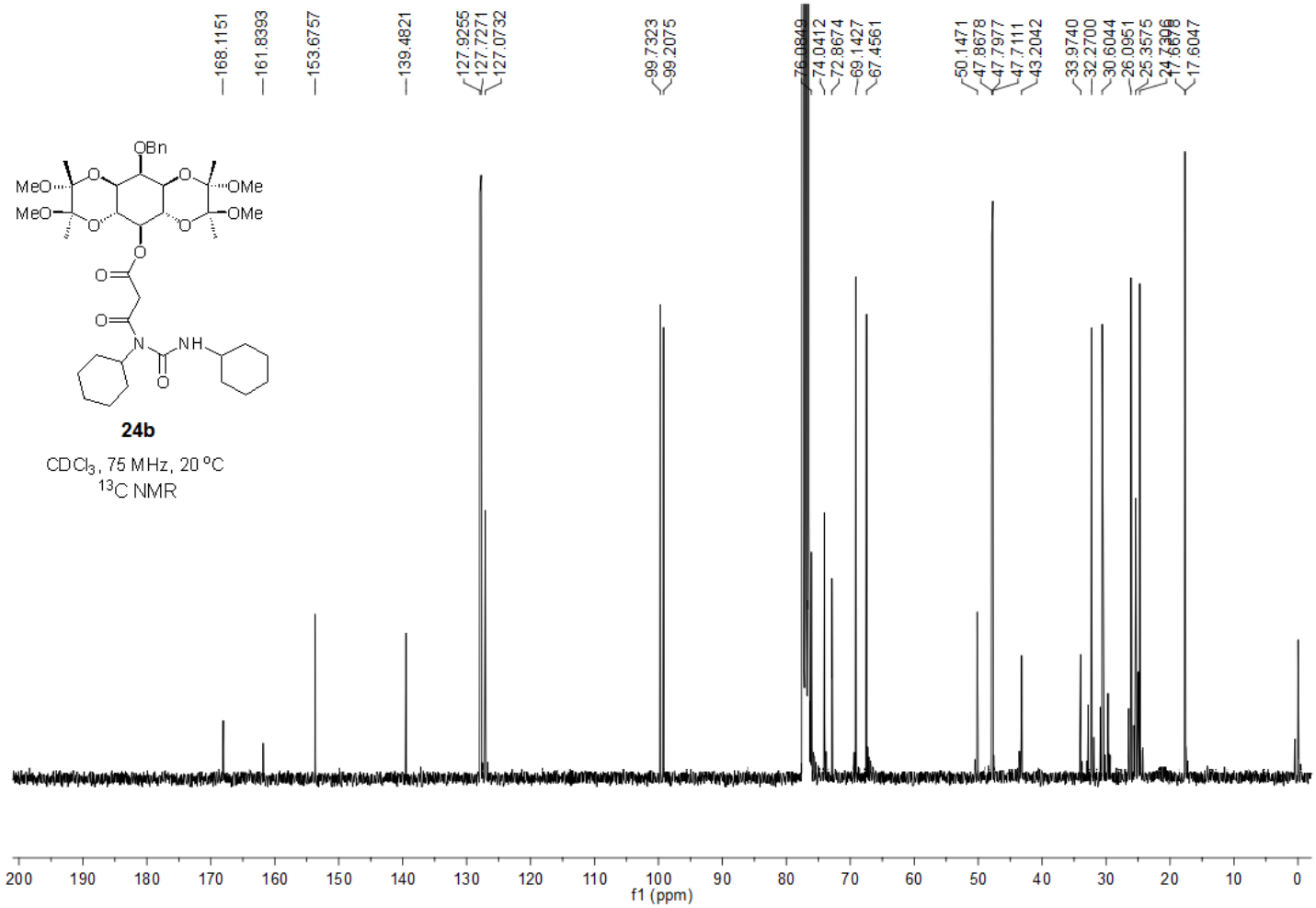
$CDCl_3$, 300 MHz, 20 °C

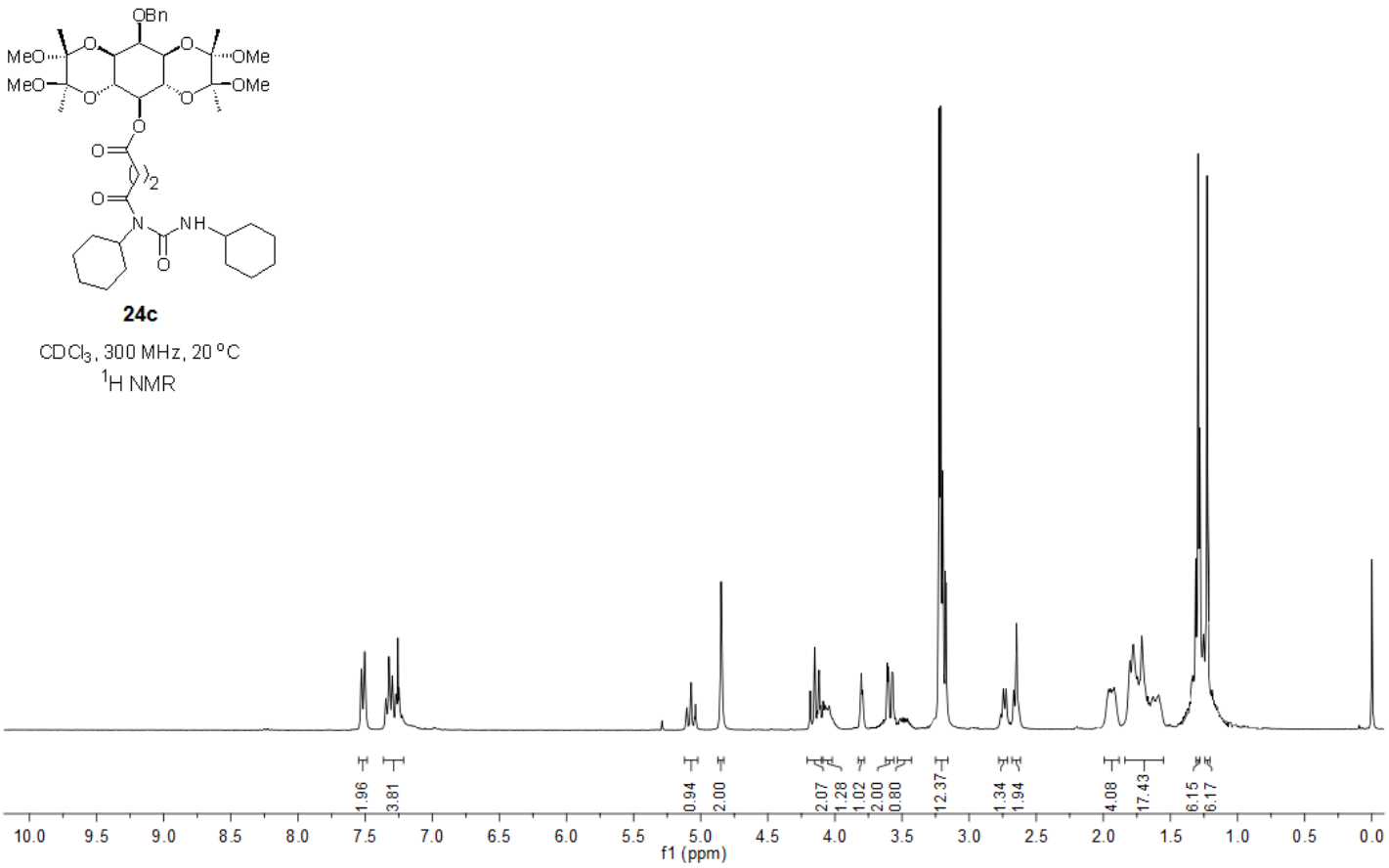
^1H NMR

24b

$CDCl_3$, 75 MHz, 20 °C

^{13}C NMR

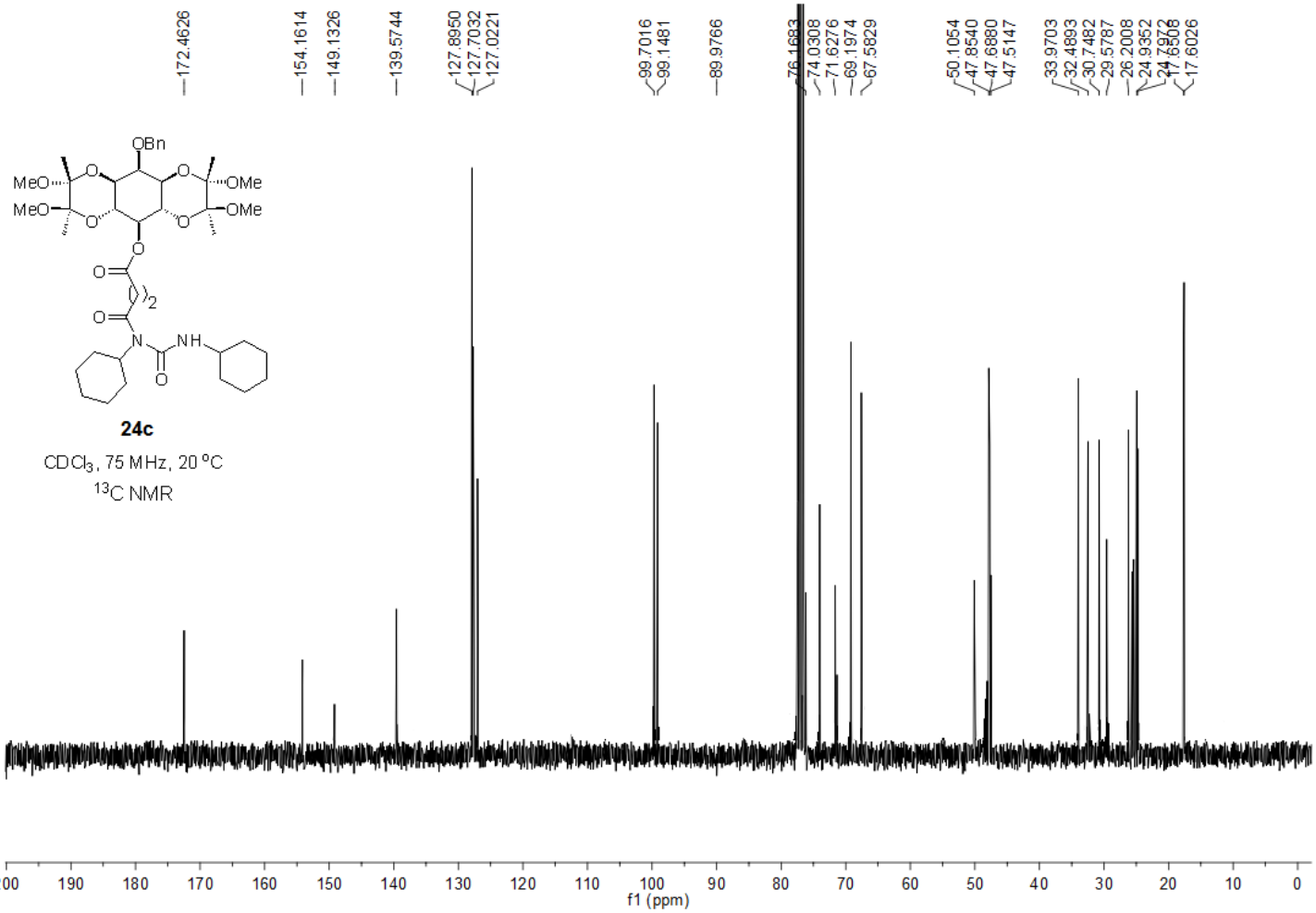




24c

$CDCl_3$, 300 MHz, 20 °C

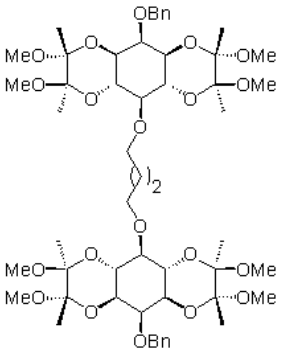
^1H NMR



24c

$CDCl_3$, 75 MHz, 20 °C

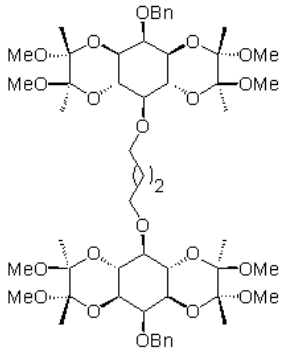
^{13}C NMR



25a

$CDCl_3$, 300 MHz, 20 °C

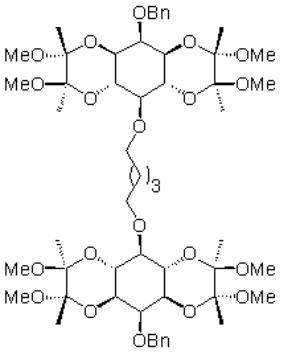
^1H NMR



25a

$CDCl_3$, 75 MHz, 20 °C

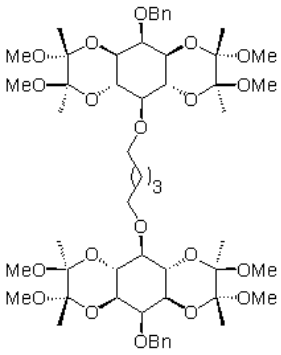
^{13}C NMR



25b

$CDCl_3$, 300 MHz, 20 °C

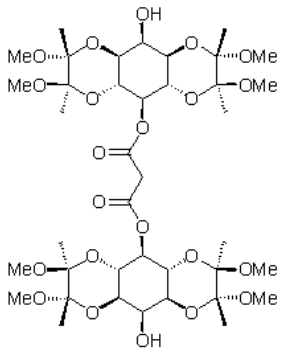
^1H NMR



25b

$CDCl_3$, 75 MHz, 20 °C

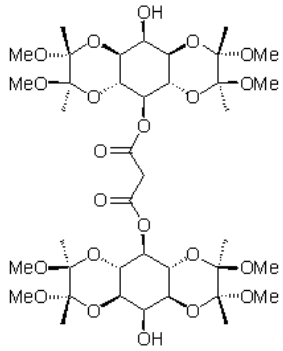
^{13}C NMR



28a

$CDCl_3$, 300 MHz, 20 °C

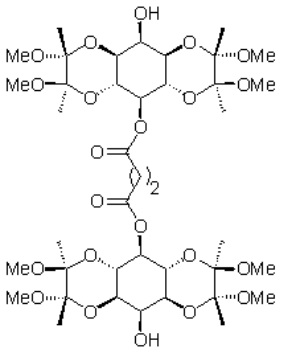
^1H NMR



28a

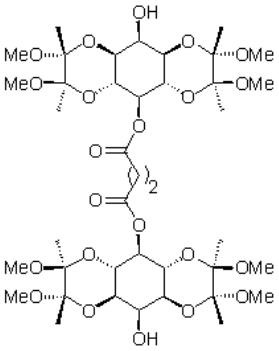
$CDCl_3$, 75 MHz, 20 °C

^{13}C NMR



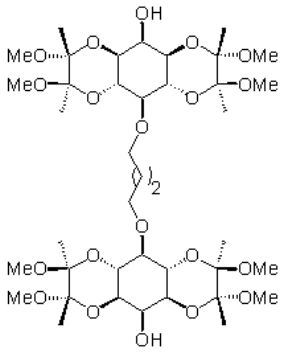
28b

$CDCl_3$, 300 MHz, 20 °C
^1H NMR



28b

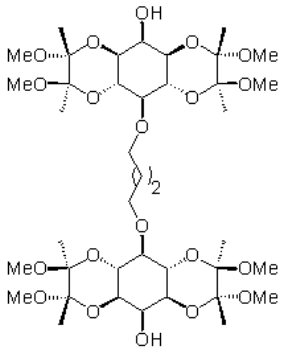
$CDCl_3$, 75 MHz, 20 °C
^{13}C NMR



28c

$CDCl_3$, 500 MHz, 20 °C

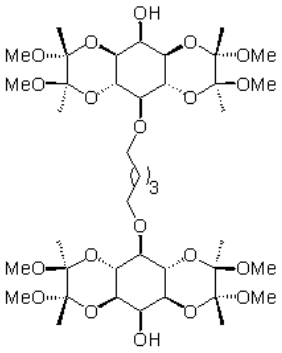
^1H NMR



28c

$CDCl_3$, 126 MHz, 20 °C

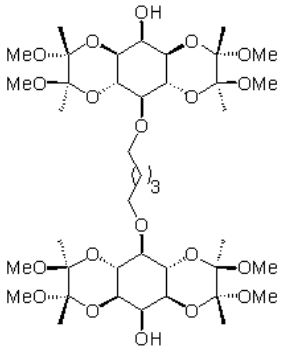
^{13}C NMR



28d

$CDCl_3$, 500 MHz, 20 °C

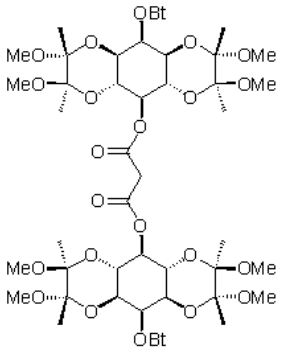
^1H NMR



28d

$CDCl_3$, 126 MHz, 20 °C

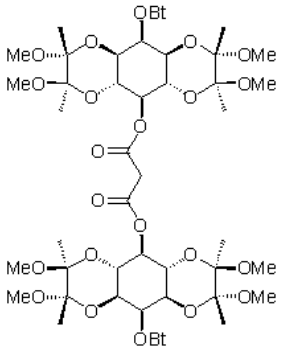
^{13}C NMR



29a

$CDCl_3$, 300 MHz, 20 °C

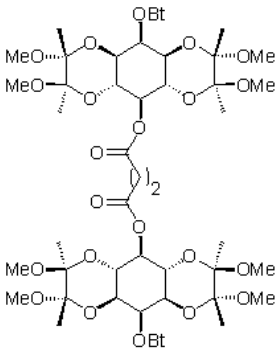
^1H NMR



29a

$CDCl_3$, 75 MHz, 20 °C

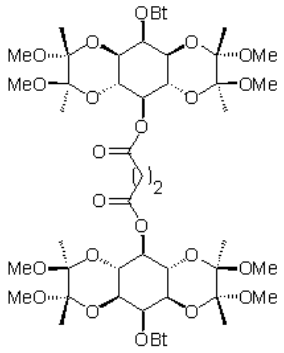
^{13}C NMR



29b

$CDCl_3$, 300 MHz, 20 °C

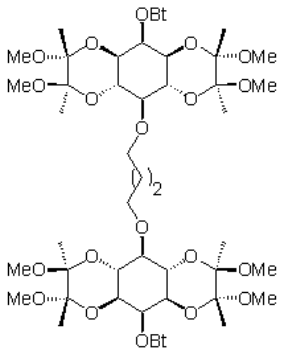
^1H NMR



29b

$CDCl_3$, 75 MHz, 20 °C

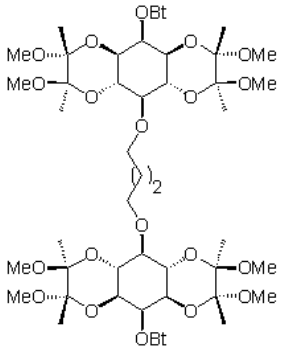
^{13}C NMR



29c

$CDCl_3$, 500 MHz, 20 °C

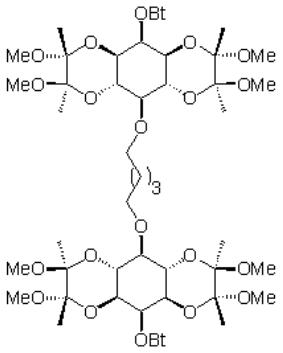
^1H NMR



29c

$CDCl_3$, 126 MHz, 20 °C

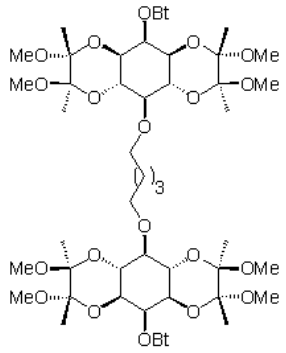
^{13}C NMR



29d

$CDCl_3$, 500 MHz, 20 °C

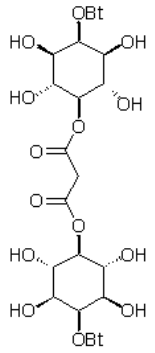
^1H NMR



29d

$CDCl_3$, 126 MHz, 20 °C

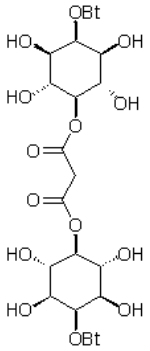
^{13}C NMR



30a

CD_3OD, 500 MHz, 20 °C

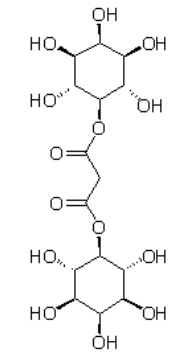
^1H NMR



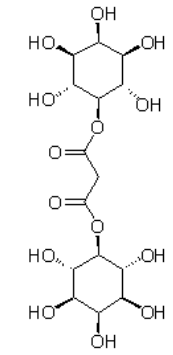
30a

CD_3OD, 126 MHz, 20 °C

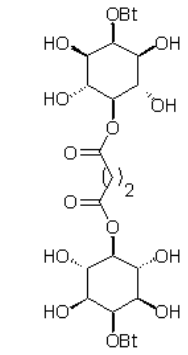
^{13}C NMR



30b
D_2O, 500 MHz, 20 °C
^1H NMR



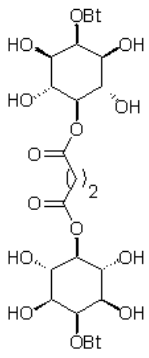
30b
D_2O, 126 MHz, 20 °C
^{13}C NMR



30c

CD_3OD, 500 MHz, 20 °C

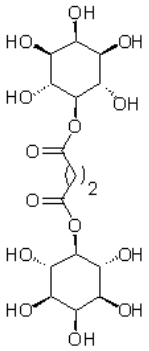
^1H NMR



30c

CD_3OD, 126 MHz, 20 °C

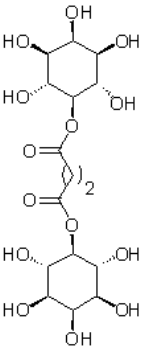
^{13}C NMR



30d

D_2O, 500 MHz, 20 °C

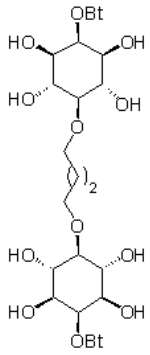
^1H NMR



30d

D_2O, 126 MHz, 20 °C

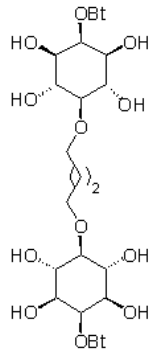
^{13}C NMR



30e

D_2O, 500 MHz, 20 °C

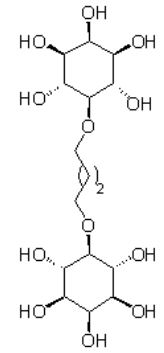
^1H NMR



30e

D_2O, 75 MHz, 20 °C

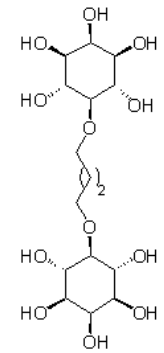
^{13}C NMR



30f

D_2O, 500 MHz, 20 °C

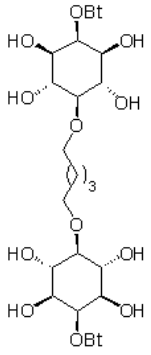
^1H NMR



30f

D_2O, 126 MHz, 20 °C

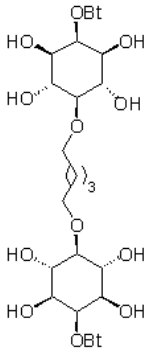
^{13}C NMR



30g

D_2O, 500 MHz, 20 °C

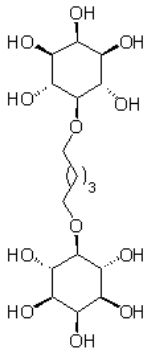
^1H NMR



30g

D_2O, 75 MHz, 20 °C

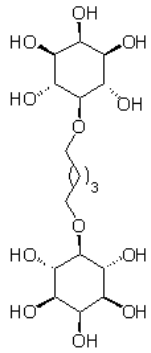
^{13}C NMR



30h

D_2O, 500 MHz, 20 °C

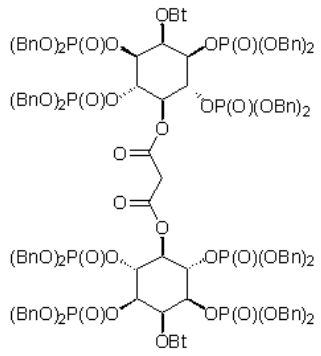
^1H NMR



30h

D_2O, 126 MHz, 20 °C

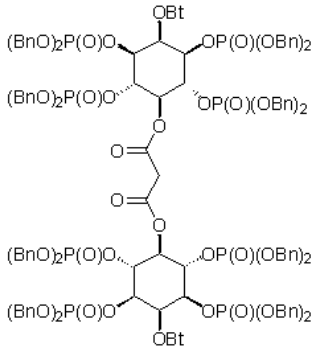
^{13}C NMR



31a

$CDCl_3$, 300 MHz, 20 °C

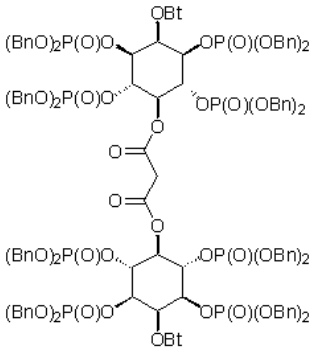
^1H NMR



31a

$CDCl_3$, 126 MHz, 20 °C

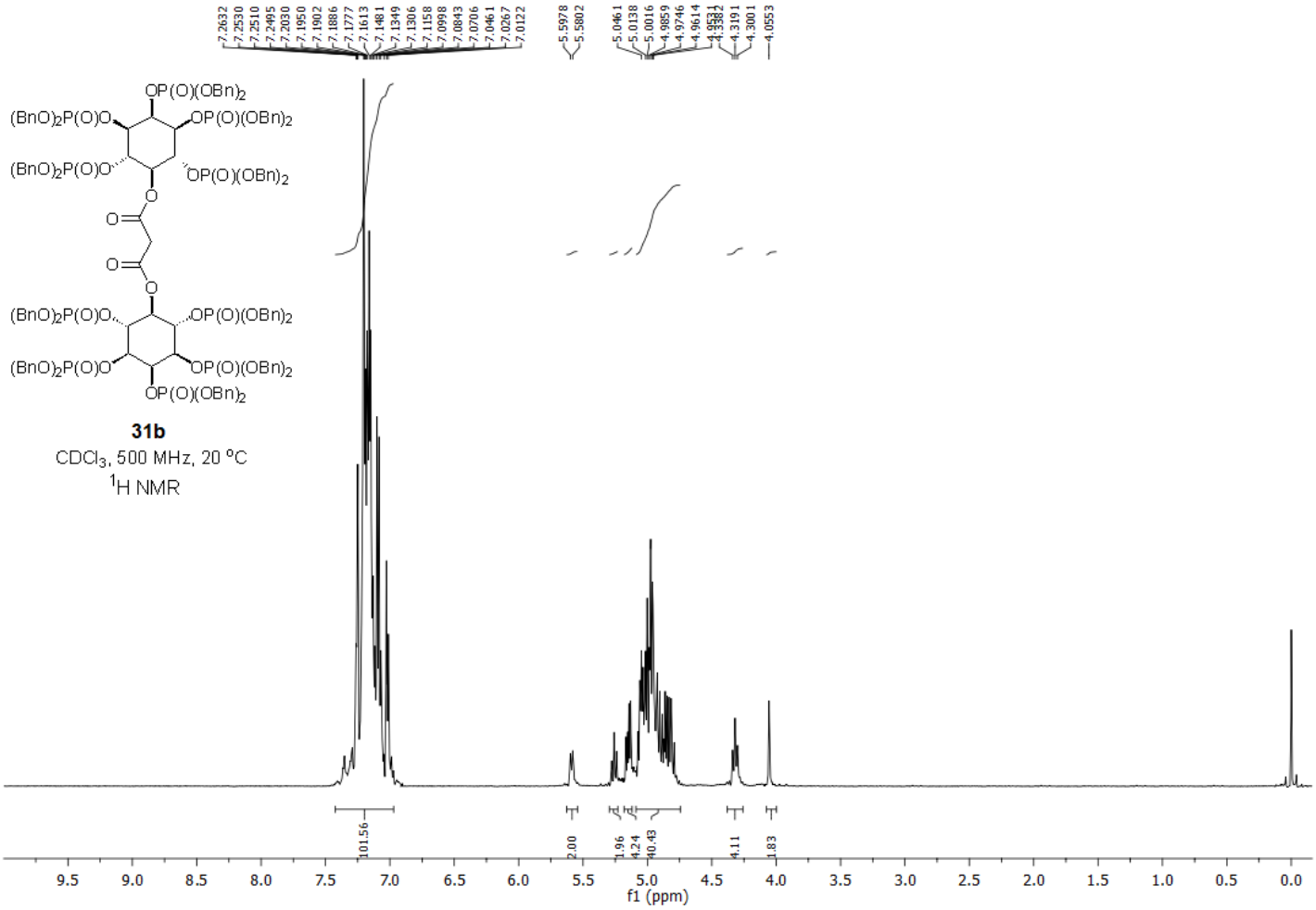
^{13}C NMR

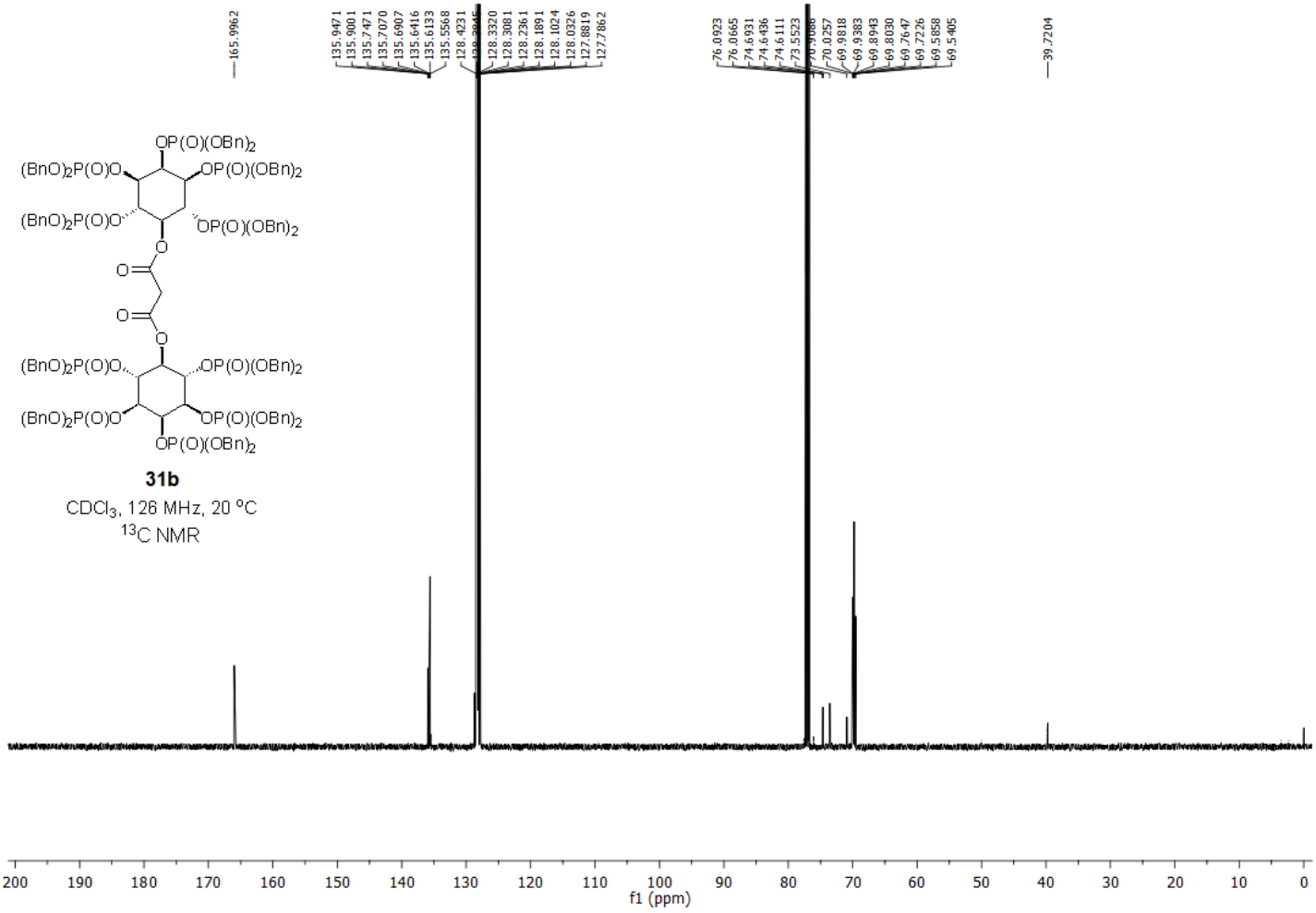


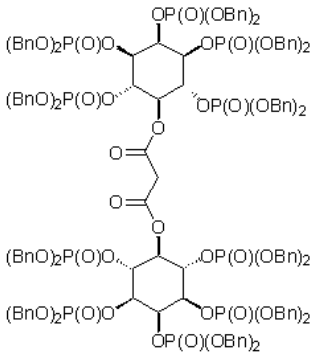
31a

$CDCl_3$, 121 MHz, 20 °C

^{31}P NMR


31b
CDCl3, 500 MHz, 20 °C
1H NMR
f1 (ppm)

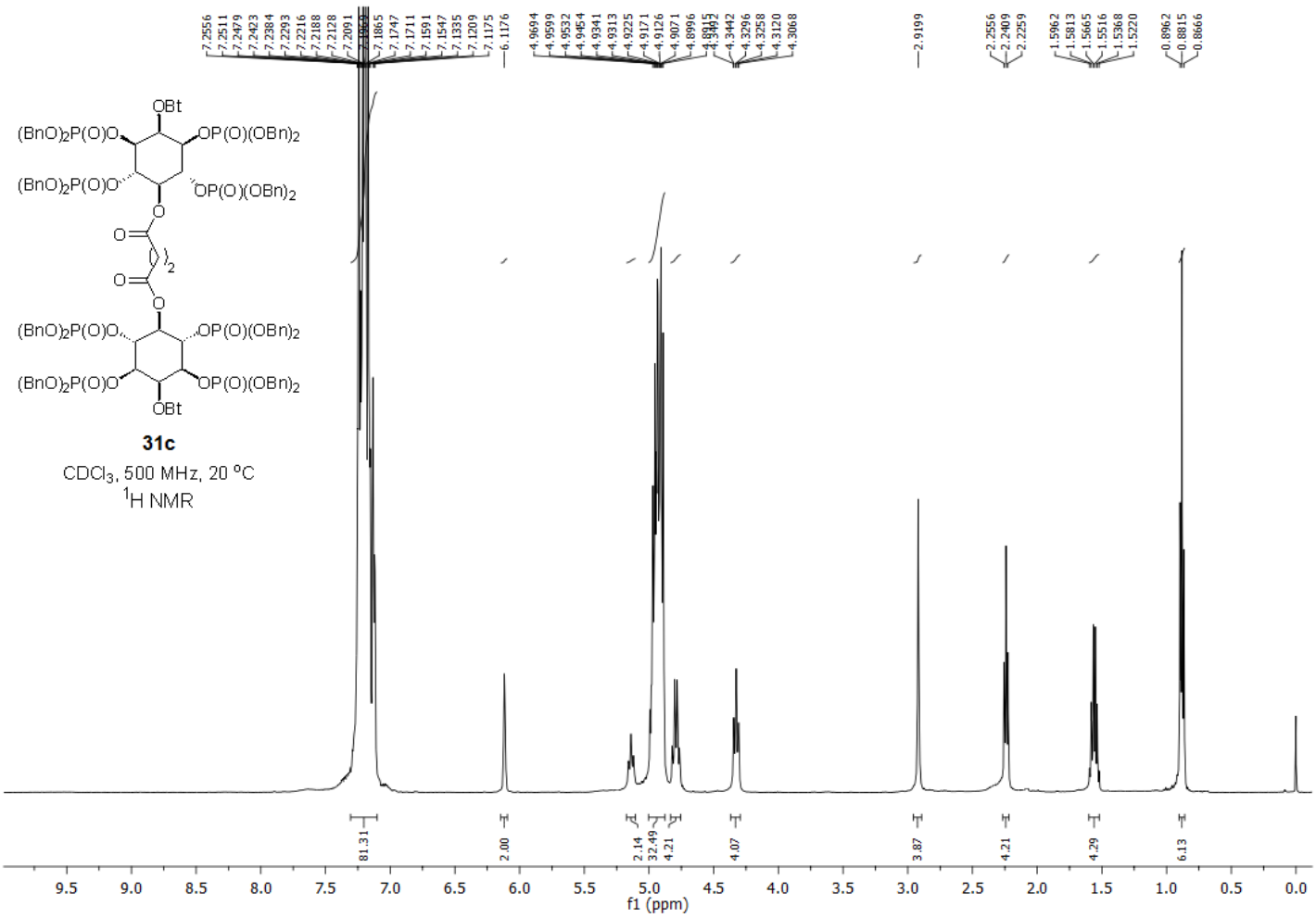

31b
CDCl3, 126 MHz, 20 °C
13C NMR
f1 (ppm)

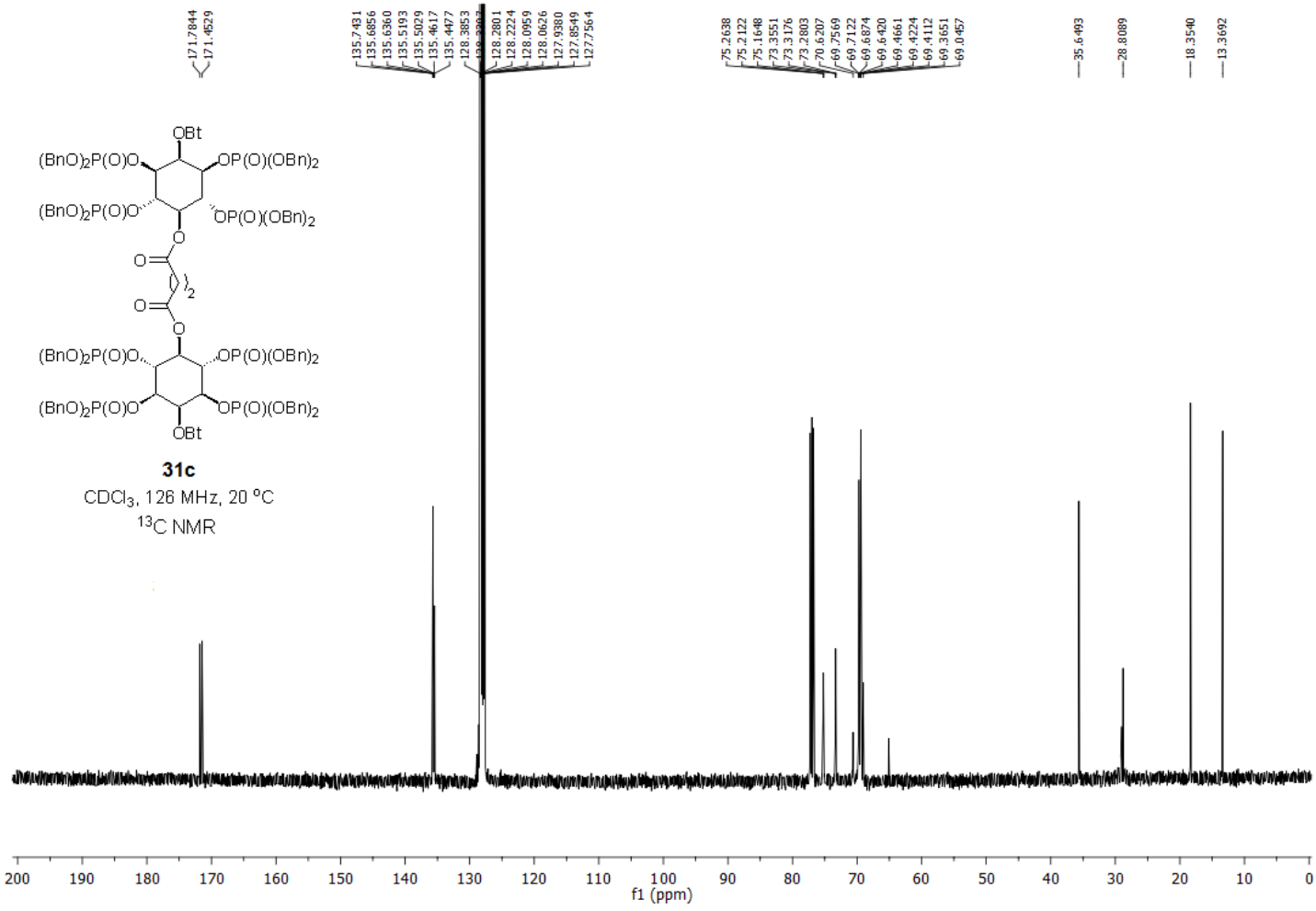


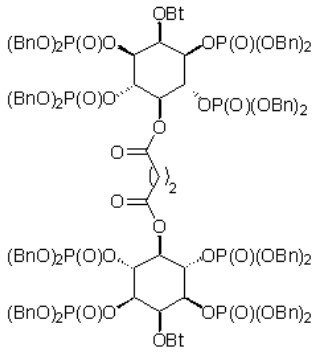
31b

$CDCl_3$, 121 MHz, 20 °C

^{31}P NMR


OBt
(BnO)2P(O)O
OP(O)(OBn)2
(BnO)2P(O)O
OP(O)(OBn)2
O
O
2
O
O
(BnO)2P(O)O
OP(O)(OBn)2
(BnO)2P(O)O
OP(O)(OBn)2
OBt
31c
CDCl3, 500 MHz, 20 °C
1H NMR
f1 (ppm)

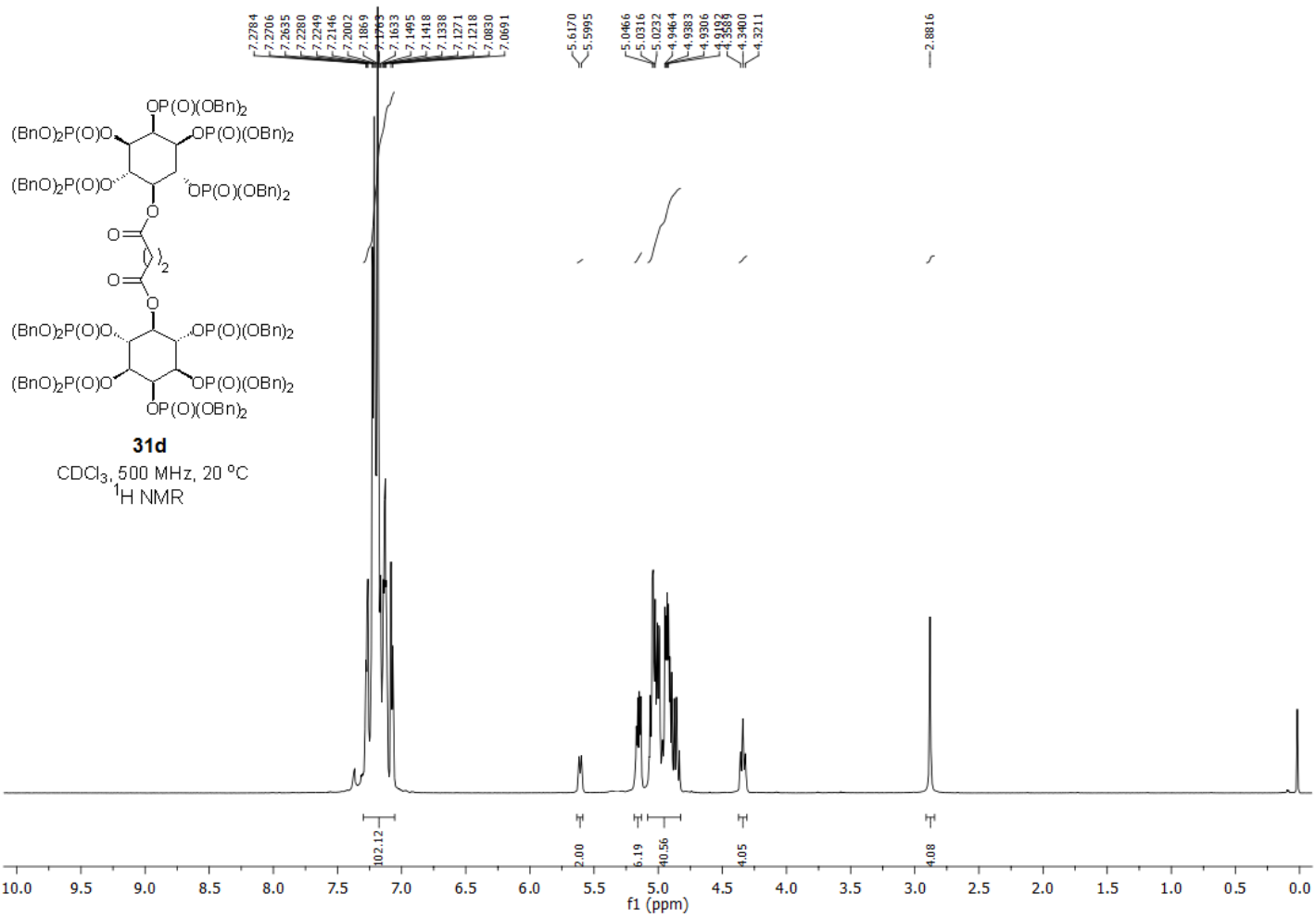

OBt
(BnO)2P(O)O
OP(O)(OBn)2
(BnO)2P(O)O
OP(O)(OBn)2
O
O
2
O
O
(BnO)2P(O)O
OP(O)(OBn)2
(BnO)2P(O)O
OP(O)(OBn)2
OBt
31c
CDCl3, 126 MHz, 20 °C
13C NMR
f1 (ppm)

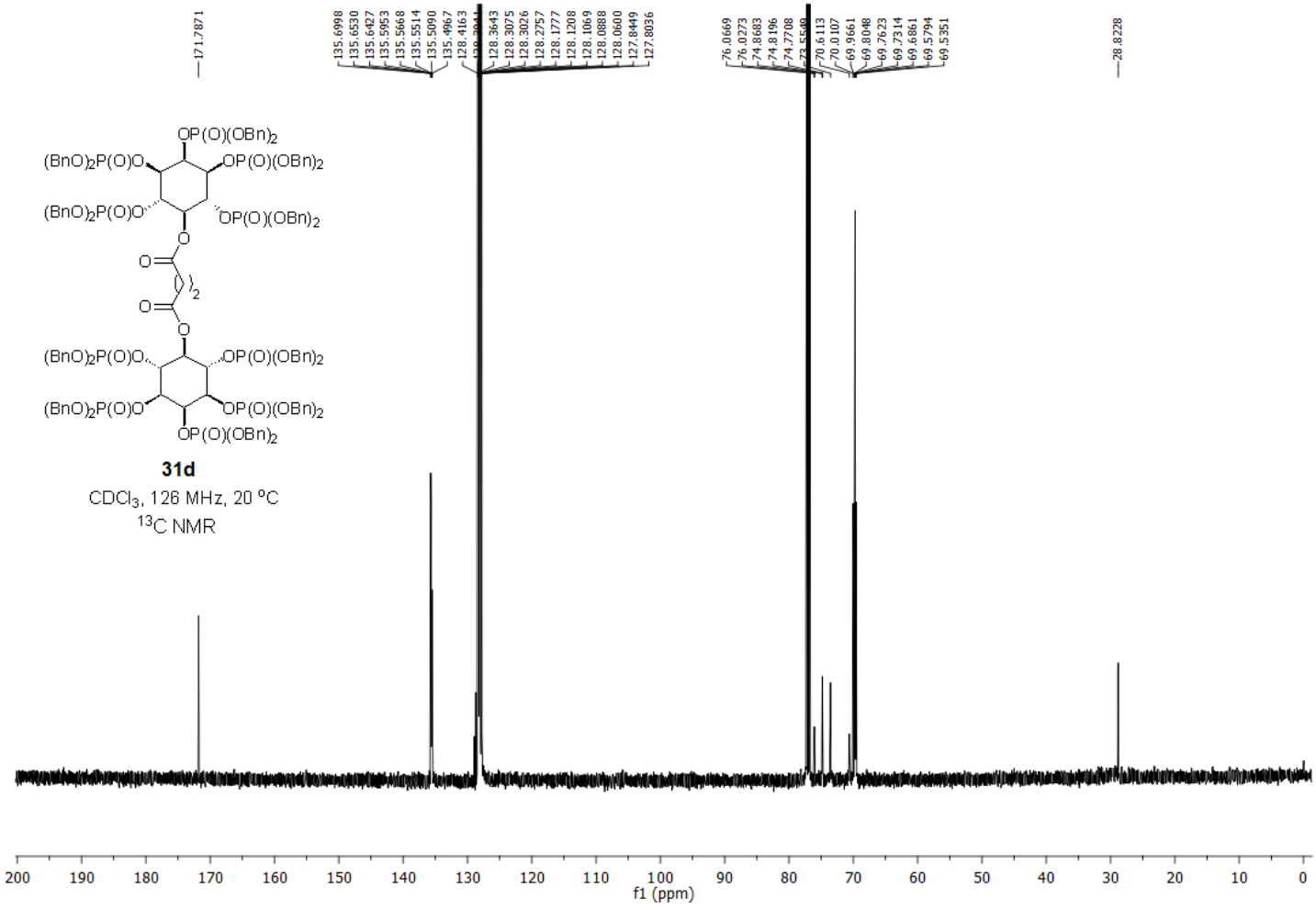


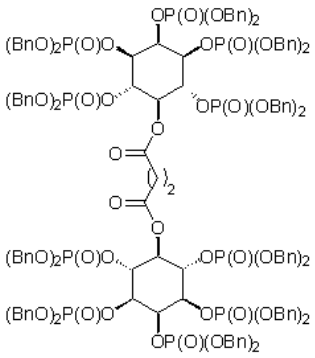
31c

$CDCl_3$, 121 MHz, 20 °C

^{31}P NMR


31d
CDCl3, 500 MHz, 20 °C
1H NMR
f1 (ppm)

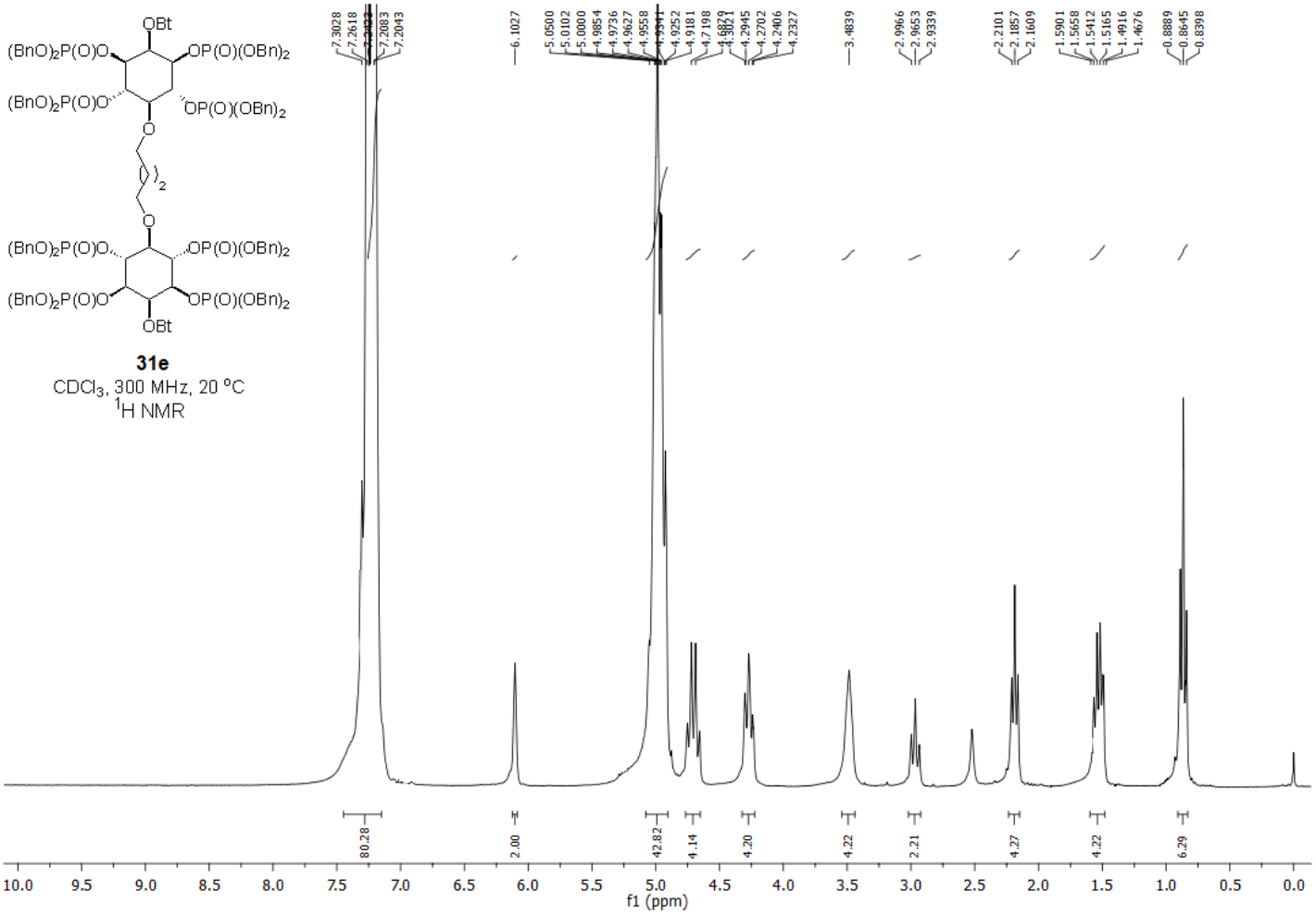

31d
CDCl3, 126 MHz, 20 °C
13C NMR
f1 (ppm)

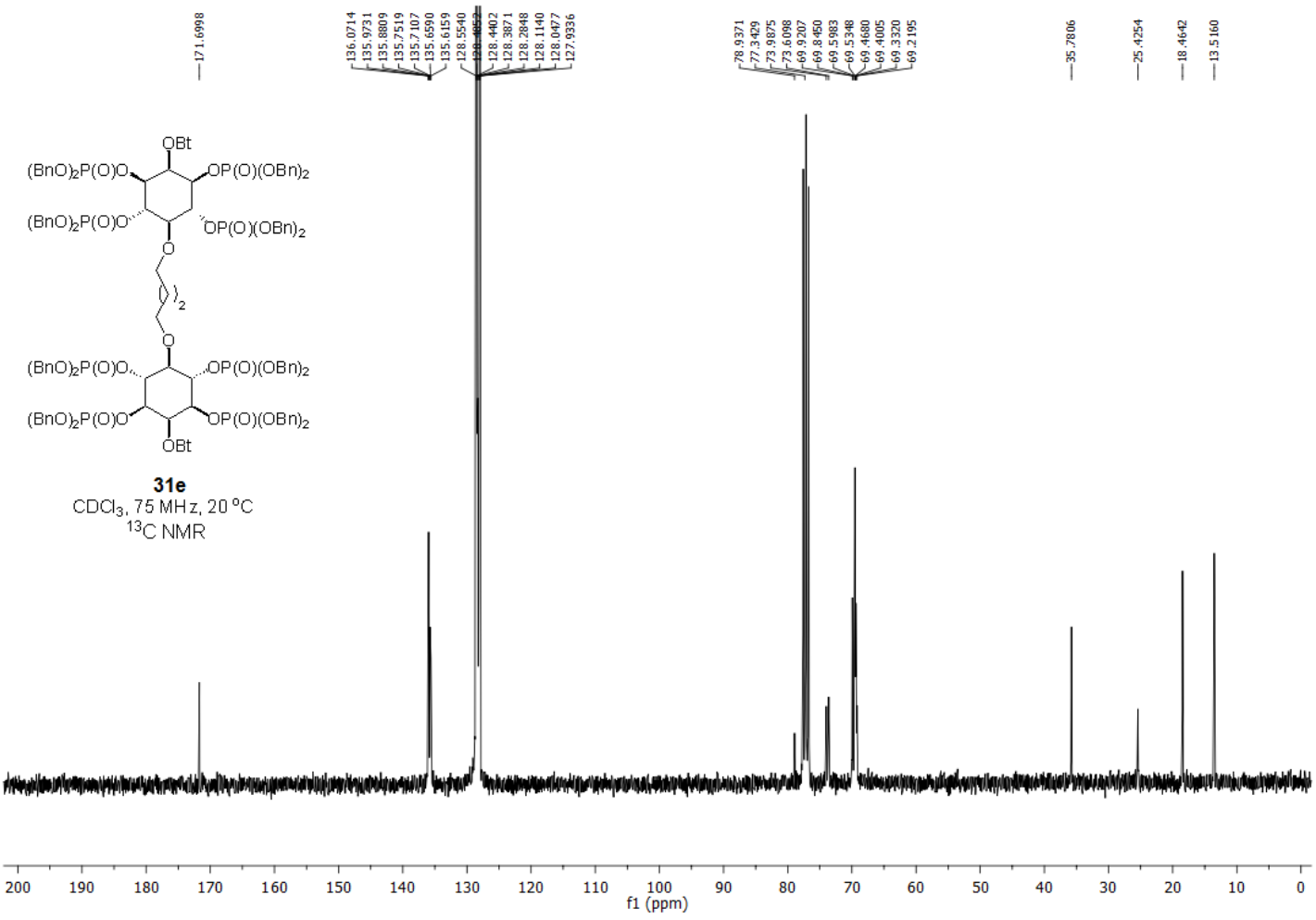


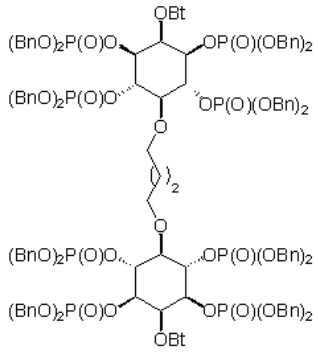
31d

$CDCl_3$, 121 MHz, 20 °C

^{31}P NMR


OBt
(BnO)2P(O)O
OP(O)(OBn)2
(BnO)2P(O)O
OP(O)(OBn)2
O
2
O
(BnO)2P(O)O
OP(O)(OBn)2
(BnO)2P(O)O
OP(O)(OBn)2
OBt
31e
CDCl3, 300 MHz, 20 °C
1H NMR
7.3028
7.2618
7.2083
7.2043
6.1027
5.0500
5.0102
5.0000
4.9854
4.9736
4.9627
4.9558
4.9252
4.9181
4.7198
4.2945
4.2702
4.2406
4.2327
3.4839
2.9966
2.9653
2.9339
2.2101
2.1857
2.1609
1.5901
1.5658
1.5412
1.5165
1.4916
1.4676
0.8889
0.8645
0.8398
80.28
2.00
42.82
4.14
4.20
4.22
2.21
4.27
4.22
6.29
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

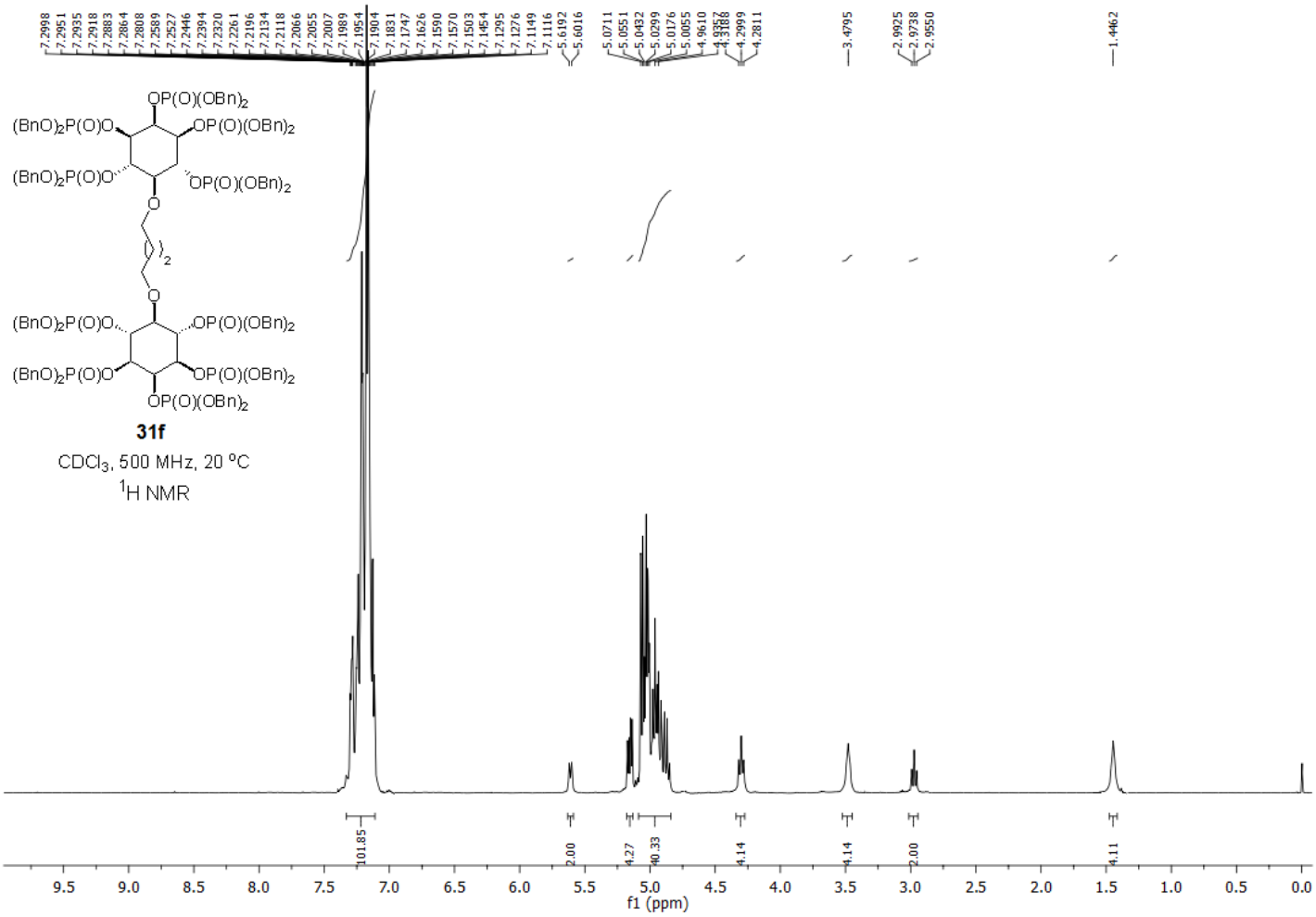

171.6998
136.0714
135.9731
135.8809
135.7519
135.7107
135.6590
135.6159
128.5540
128.4402
128.3871
128.2848
128.1140
128.0477
127.9336
78.9371
77.3429
73.9875
73.6098
69.9207
69.8450
69.5983
69.5348
69.4680
69.4005
69.3320
69.2195
35.7806
25.4254
18.4642
13.5160
OBt
(BnO)2P(O)O
OP(O)(OBn)2
(BnO)2P(O)O
OP(O)(OBn)2
O
2
O
(BnO)2P(O)O
OP(O)(OBn)2
(BnO)2P(O)O
OP(O)(OBn)2
OBt
31e
CDCl3, 75 MHz, 20 °C
13C NMR
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

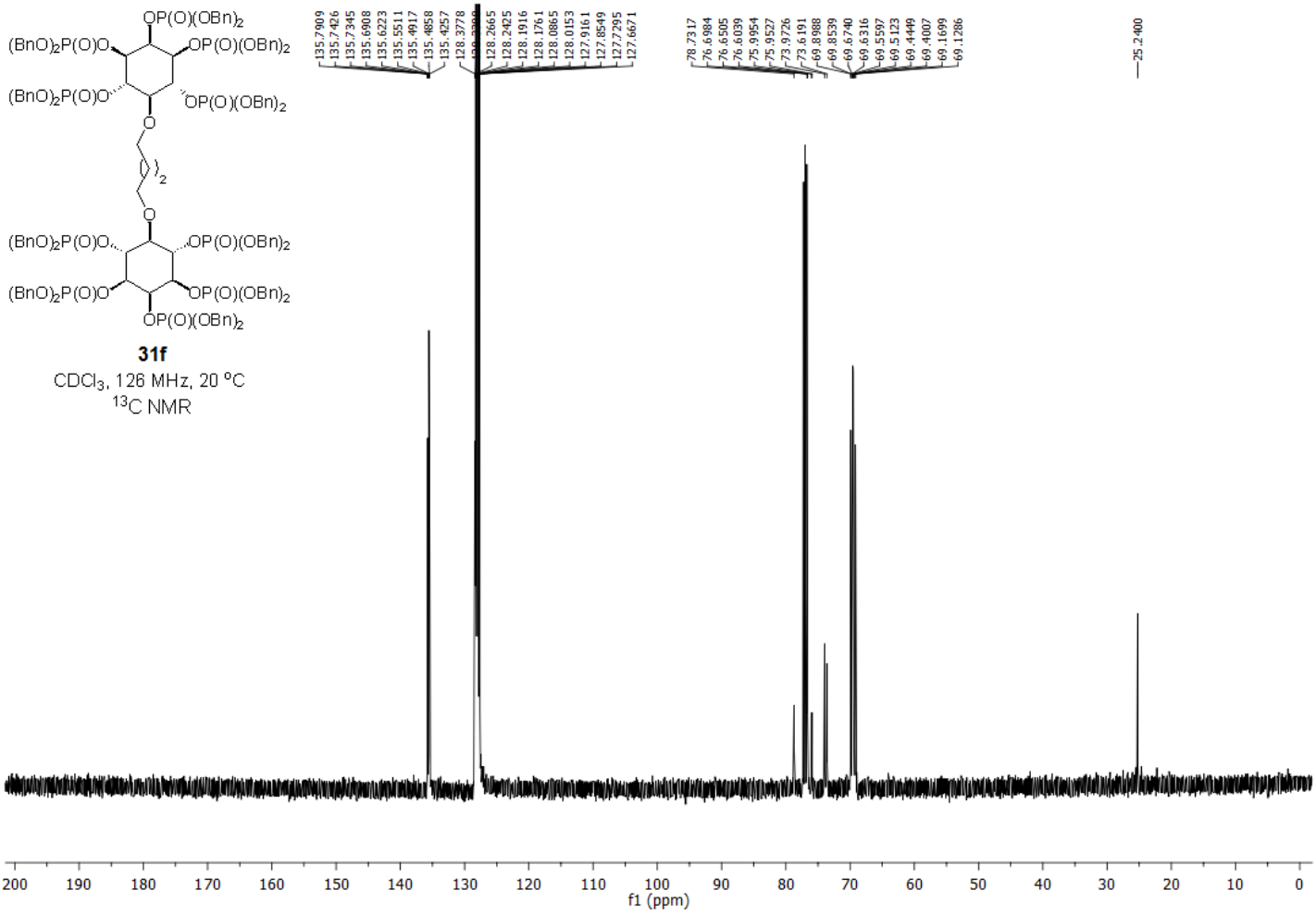


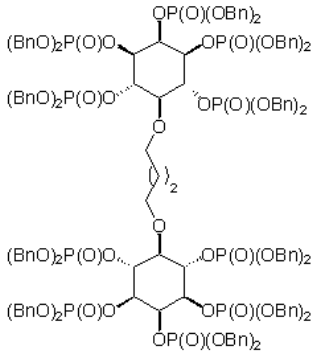
31e

$CDCl_3$, 121 MHz, 20 °C

^{31}P NMR


31f
CDCl3, 500 MHz, 20 °C
1H NMR
f1 (ppm)

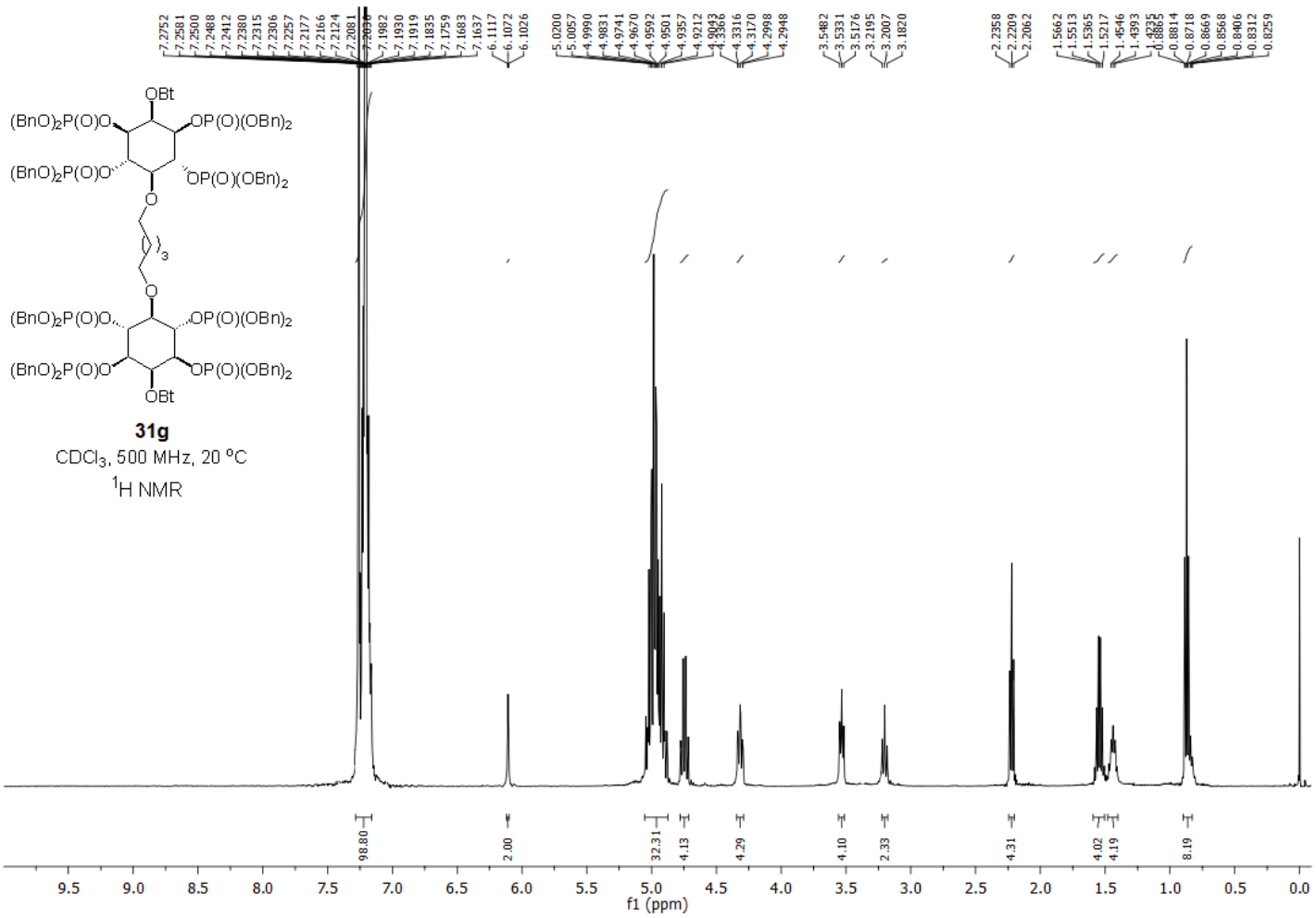

31f
CDCl3, 126 MHz, 20 °C
13C NMR
f1 (ppm)

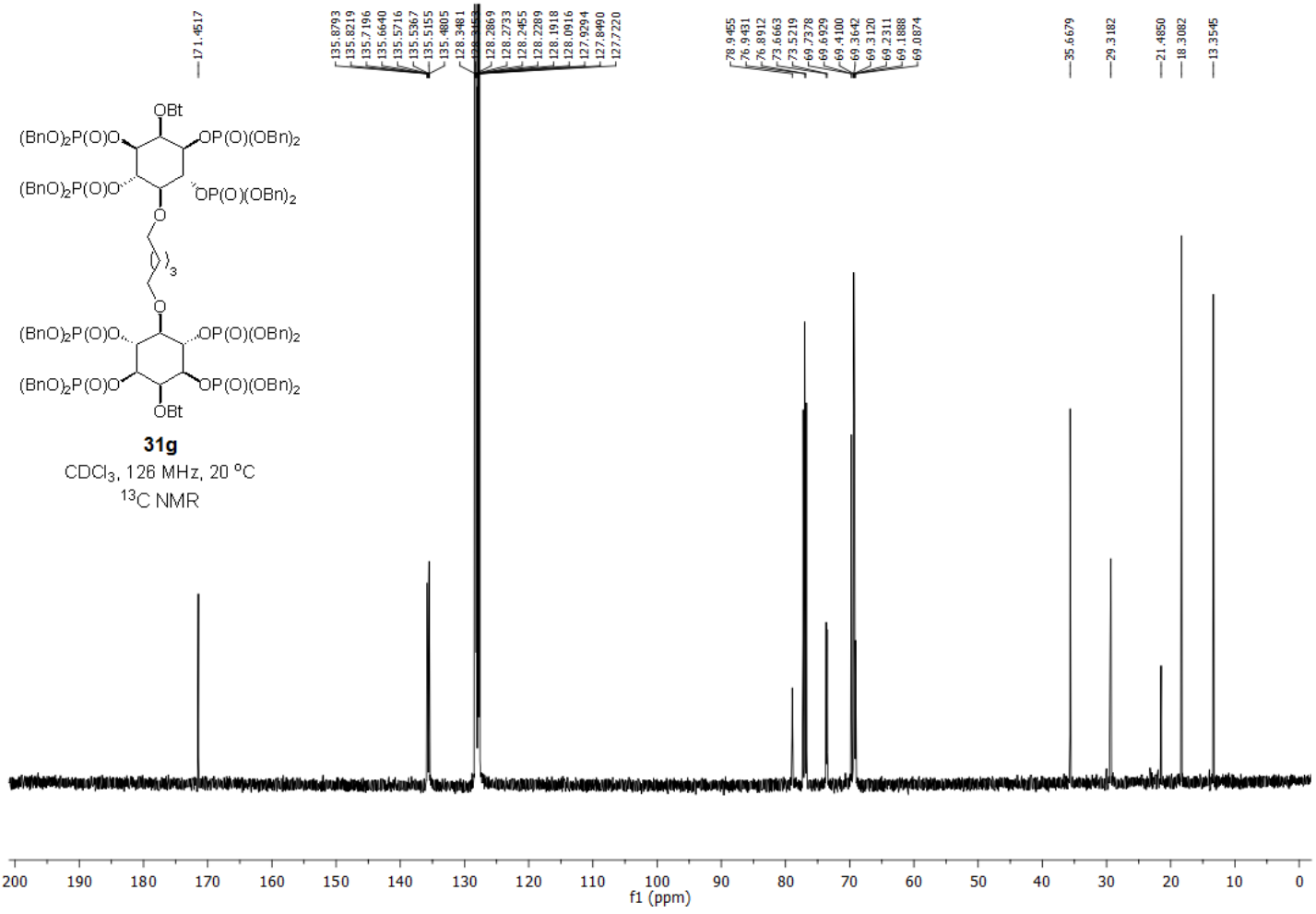


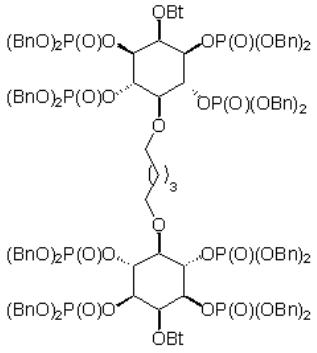
31f

$CDCl_3$, 202 MHz, 20 °C

^{31}P NMR


OBt
(BnO)2P(O)O
OP(O)(OBn)2
(BnO)2P(O)O
OP(O)(OBn)2
O
3
O
(BnO)2P(O)O
OP(O)(OBn)2
(BnO)2P(O)O
OP(O)(OBn)2
OBt
31g
CDCl3, 500 MHz, 20 °C
1H NMR
f1 (ppm)

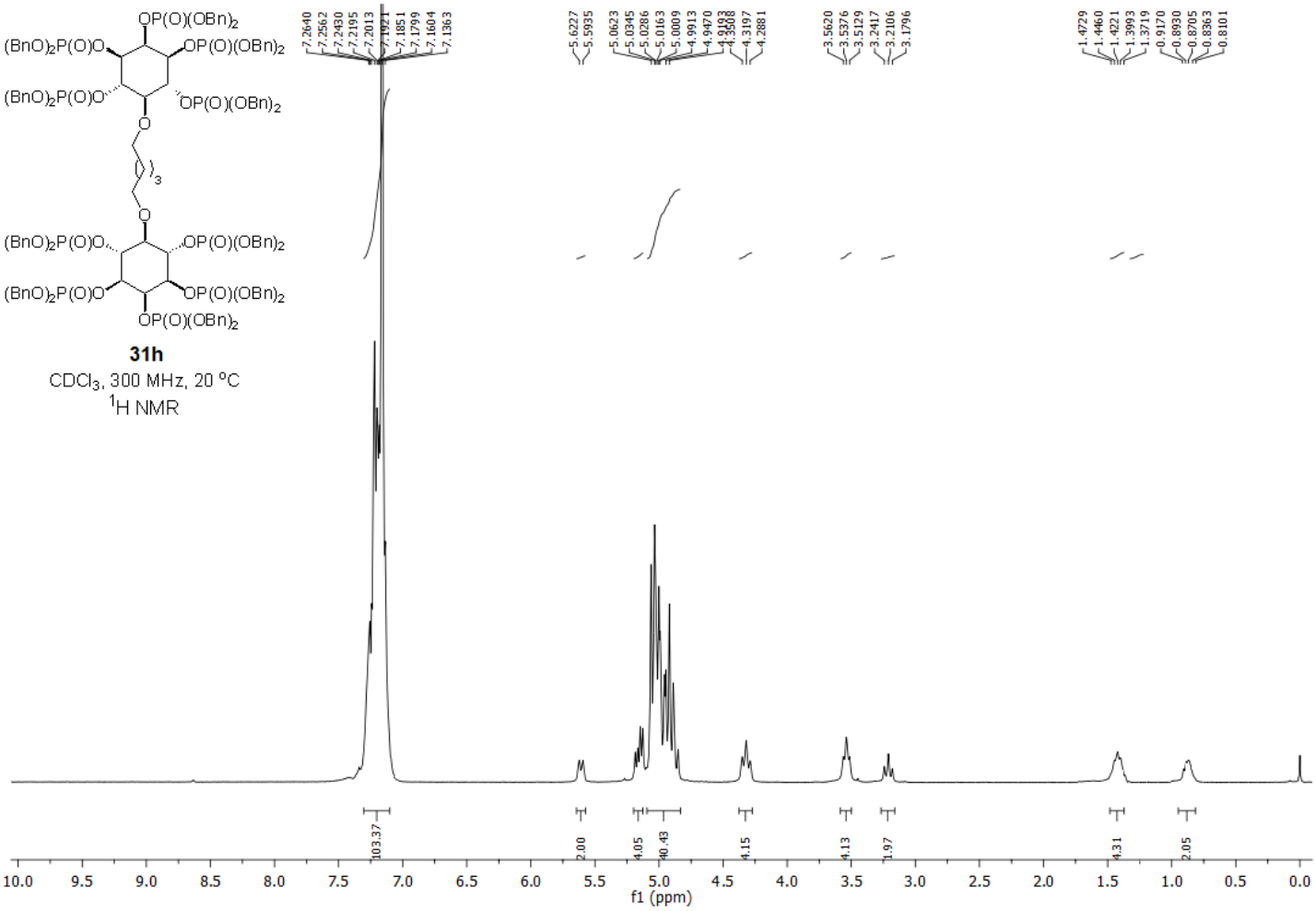

OBt
(BnO)2P(O)O
OP(O)(OBn)2
(BnO)2P(O)O
OP(O)(OBn)2
O
3
O
(BnO)2P(O)O
OP(O)(OBn)2
(BnO)2P(O)O
OP(O)(OBn)2
OBt
31g
CDCl3, 126 MHz, 20 °C
13C NMR
f1 (ppm)



31g

$CDCl_3$, 121 MHz, 20 °C

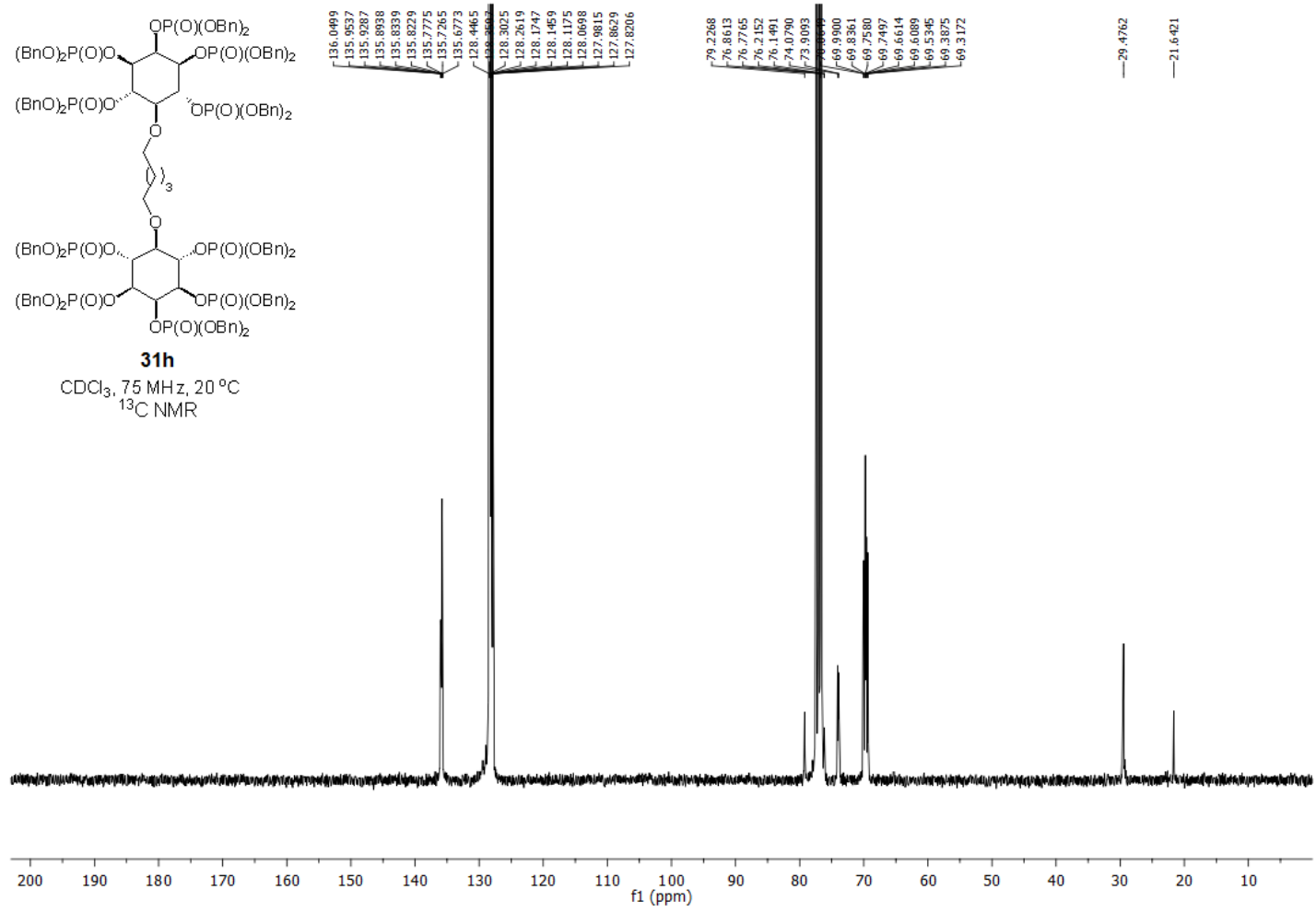
^{31}P NMR



31h

$CDCl_3$, 300 MHz, 20 °C

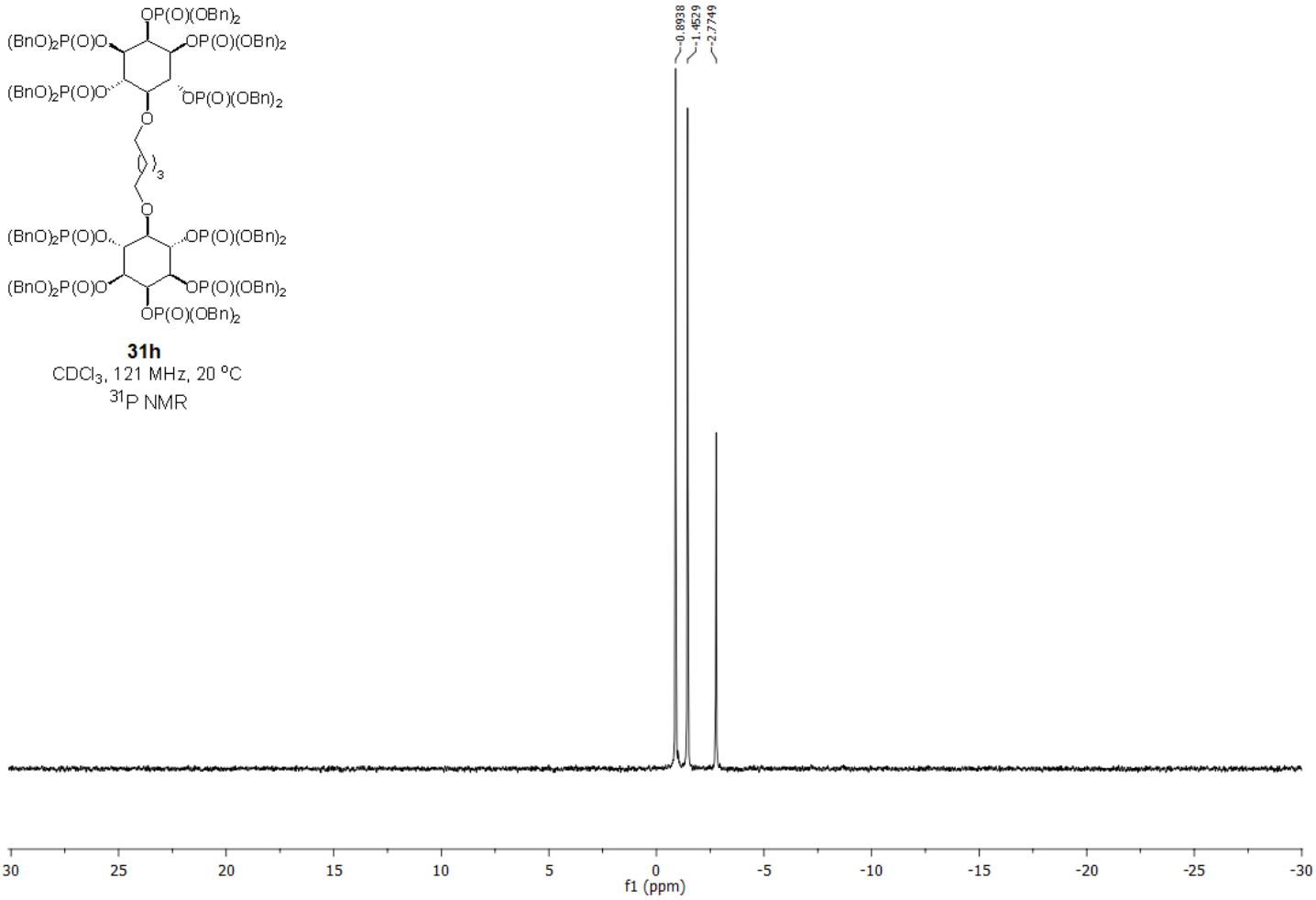
^1H NMR



31h

$CDCl_3$, 75 MHz, 20 °C

^{13}C NMR



31h

$CDCl_3$, 121 MHz, 20 °C

^{31}P NMR