Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Experimental Procedures

Reactions were carried out using oven-dried glassware under an atmosphere of dry N_2 and magnetically stirred, unless noted otherwise. Air- and moisture-sensitive liquids and solutions were transferred via syringe or stainless steel canula.

Reagents were purchased from commercial suppliers (Acros, Aldrich, Fluka, TCI) and used without further purification, unless noted otherwise.

Solvents (methylene chloride, diethyl ether, tetrahydrofuran, acetonitrile, toluene) for reactions were purified by filtration and dried by passage over activated anhydrous neutral A-2 alumina (MBraun solvent purification system) under an atmosphere of dry nitrogen. Analytical grade solvents were used as received for extractions and chromatographic purifications.

Deuterated solvents were obtained from Armar Chemicals, Switzerland, in the indicated purity grade.

Thin Layer Chromatography were used for monitoring reactions and carried out using Merck silica gel 60 F254 plates, visualized with UV light or developed either with phosphormolybdic acid solution or with potassium permanganate solution followed by heating

Flash Chromatography was performed using Fluka silica gel 60 (230-400 Mesh) at a pressure of ca. 0.3 bar. Eluents and $\mathbf{R_f}$ are indicated.

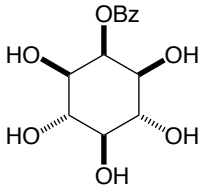
Lyophilizations were performed on a Christ Freeze Dryer Alpha 1-2 LD+.

^{1}H-NMR spectra were recorded on Bruker 400 MHz spectrometers or Bruker 500 MHz spectrometers (equipped with a cryo platform) at 298K in the indicated deuterated solvent. Data are reported as follow: chemical shift (δ, ppm), multiplicity (s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet or not resolved signal; br, broad signal), coupling constant(s) (*J*, Hz), integration. All signals were referenced to the internal solvent signal as standard ($CDCl_3$, δ 7.26; CD_3OD, δ 3.31; DMSO-d_6, δ 2.50).

^{13}C-NMR spectra were recorded with ^{1}H-decoupling on Bruker 101 MHz or Bruker 125 MHz spectrometers (equipped with a cryo platform) at 298K in the indicated deuterated solvent. All signals were referenced to the internal

solvent signal as standard ($CDCl_3$, δ 77.0; CD_3OD, δ 49.0; DMSO-d_6, δ 39.5).
^{31}P-NMR spectra were recorded with proton coupling and 1H-decoupling on Bruker 162 MHz or Bruker 202 MHz spectrometers (equipped with a cryo platform) at 298K in the indicated deuterated solvent. All signals were referenced to an internal standard (PPP)
IR spectra were recorded on a JASCO FT-IR-4100 spectrometer and data are reported in terms of frequency of absorption (cm^{-1}).
Mass spectra were recorded by the Mass spectroscopy Service of UZH on Finnigan MAT95 MS, Bruker EsquireLC MS, Bruker maXis QTof HR MS and Finnigan TSQ700 MS machines.

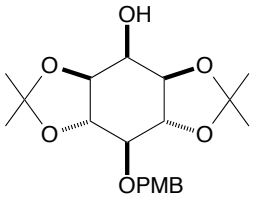
Synthesis of **8**



The compound **8** was synthesized as described before in two steps starting from *myo*-inositol **7**. Analytical data were identical with the values reported in the literature.[13]

[13] H. Y. Godage, A. M. Riley, T. J. Woodman, B. V. L. Potter, *Chem. Comm.* **2006**, 2989-2991.

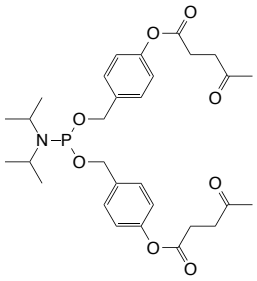
Synthesis of **10**



The compound **10** was synthesized as described before in three steps starting from **8**. PMB group was introduced by a procedure described in a patent by Postech Foundation WO2005/85159 A1, 2005; Analytical data were identical with the values reported in the literature.[8e]

[8e] H. Zhang, J. Thompson, G. D. Prestwich, *Org. Lett.* **2009**, *11*, 1551-1554.

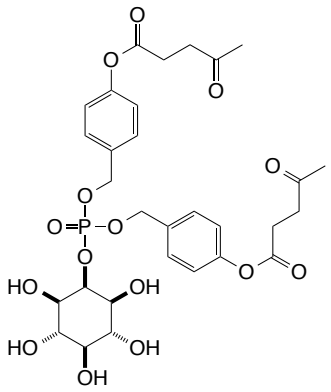
Synthesis of **11**



The compound was synthesized as described before three steps starting from 4-hydroxibenzaldehyde and levulinic acid. Analytical data were identical with the values reported in the literature.[10]

[10] I. Pavlovic, D. T. Thakor, J. R. Vargas, C. J. McKinlay, S. Hauke, P. Anstaett, R. C. Camuna, L. Bigler, G. Gasser, C. Schultz, P. A. Wender, H. J. Jessen, *Nat. Commun.* **2016**, accepted.

Synthesis of **12**



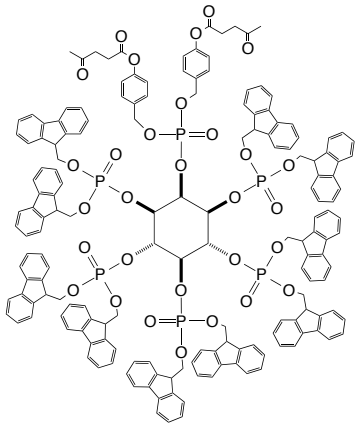
A) 100 mg (0.263 µmol, 1.0 eq.) **10** and 180 mg (0.315 µmol, 1.2 eq) levulinylbenzyl phosphoramidite (LevB-PA) **11** were dissolved in dry MeCN (5 mL). 40.5 mg (0.342 mmol, 1.3 eq) of DCI was added and mixture was stirred for 10 minutes at room temperature. Progress of the reaction was followed by ^{31}P-NMR. Oxidation was achieved by addition of 0.240 ml (1.32 mmol, 5 eq) *t*-BuOOH. Solvent was evaporated and the obtained crude oil **12a** was directly used in the next step.

B) The intermediate was dissolved in DCM (10mL) and 5% of TFA (0.5 ml) and stirred for 5 min at room temperature. It was monitored by TLC and ^{31}P-NMR. After completion of reaction, solvent was concentrated in vacuo and

residue was crystalized from Et_2O and purified by column chromatography (Gradient EtOAc to EtOAc: MeOH, 4:1) to get pure final product as brown solid **12** (99.0 mg, 0.150 mmol, 56 % yield)

TLC (EtOAc:MeOH, 4:1 v/v): $\mathbf{R_f}$ = 0.60; **^{1}H-NMR** (500 MHz, DMSO-d_6): δ 7.44 (d, *J* = 8.5 Hz, 4H), 7.09 (d, *J* = 8.5 Hz, 4H), 5.17 (d, *J* = 3.3 Hz, 1H), 5.12 (d, *J* = 3.1 Hz, 1H), 5.11 (d, *J* = 2.3 Hz, 1H), 4.89 - 4.82 (m, 2H), 4.75 - 4.66 (m, 2H), 3.45 - 3.37 (m, 3H), 2.85 (t, *J* = 6.4 Hz, 4H), 2.73 (t, *J* = 6.4 Hz, 4H), 2.15 (s, 6H); **^{13}C-NMR** (126 MHz, MeOD): δ 207.91, 171.78, 150.89, 133.94, 128.89, 121.40, 82.05, 74.97, 72.94, 70.39, 68.75, 48.10, 48.02, 47.93, 47.81, 47.76, 47.64, 47.59, 47.47, 47.42, 47.25, 47.08, 37.28, 28.21, 27.64; **^{31}P{1H}-NMR** (203 MHz, MeOD): δ -2.08; **^{31}P-NMR** (203 MHz, MeOD): δ -1.37 to -3.14 (m); **IR** (neat, cm-1) 3380.6, 2959.2, 2921.6, 2364.3, 1754.9, 1715.4, 1361.5, 1200.5, 1135.9, 1004.7; **HRMS** (ESI) $[M+Na]^+$ calcd 691.1768 for $C_{30}H_{37}NaO_{15}P$, found 691.1764

Synthesis of **14**

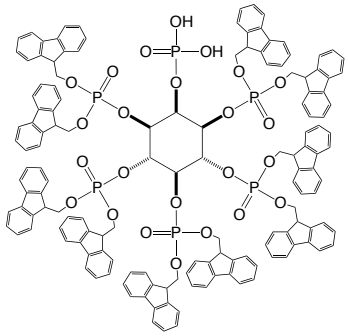


50.0 mg (0.075 mmol, 1.0 eq.) of inositol monophosphate **12** and 0.575 g (1.13 mmol, 15.0 eq.) of 9-fluorenylmethyl phosphoramidite (Fm-PA) **13** were coevaporated with dry MeCN (2 mL). The residue was dissolved in dry THF (2 mL). To this solution 177 mg (1.50 mmol, 20.0 eq.) of DCI was added. Progress of the reaction was monitored by ^{31}P-NMR. After completion of the reaction (30-45 min), oxidation was achieved by slow (!) addition of 276.5 mg (1.13 mmol, 15.0 eq.) *m*CPBA (70% moistened with water) at 0ºC. Mixture was concentrated *in vacuo* and product was crystalized from MeOH (2 x 3 ml)

yielding 155 mg of **14** as a white sticky solid (0.054 mmol, 73%).

TLC (EtOAc:Hexane, 3:2 v/v): $\mathbf{R_f}$ = 0.33; **^{1}H-NMR** (500 MHz, $CDCl_3$): δ 7.65 - 7.51 (m, 24H), 7.44 - 7.34 (m, 9H), 7.34 - 7.29 (m, 6H), 7.27 - 7.20 (m, 12H), 7.21 - 7.13 (m, 8H), 7.12 - 7.06 (m, 5H), 7.05 - 6.98 (m, 21H), 6.85 - 6.81 (m, 3H), 5.66 (d, *J* = 9.0 Hz, 1H), 5.01 (q, *J* = 9.9 Hz, 2H), 4.94 (dd, *J* = 11.7, 7.9 Hz, 2H), 4.73 (dd, *J* = 11.8, 9.7 Hz, 2H), 4.59 - 4.51 (m, 2H), 4.49 - 4.40 (m, 2H), 4.37 - 4.19 (m, 11H), 4.19 - 4.05 (m, 6H), 4.05 - 4.00 (m, 2H), 3.99 - 3.91 (m, 16H), 3.81 (q, *J* = 6.9 Hz, 4H), 2.87 - 2.76 (m, 8H), 2.22 (s, 6H); **^{13}C-NMR** (126 MHz, $CDCl_3$): δ 206.26, 171.04, 150.62, 143.30 - 142.80 (m), 141.39 - 141.11 (m), 133.28, 129.39, 127.71 - 126.83 (m), 125.69 - 125.21 (m), 121.50, 119.89 - 119.64 (m), 77.29, 77.23, 77.03, 76.78, 75.90, 75.63, 74.69, 73.28, 70.05 - 69.10 (m), 47.90, 47.84, 37.89, 29.86, 28.15, 27.35, 21.07, 14.22; **^{31}P{1H}-NMR** (203 MHz, $CDCl_3$) δ -0.61 , -1.08 , -1.42 , -2.81; **^{31}P-NMR** (162 MHz, $CDCl_3$): δ -0.41 - -0.80 (m), -0.88 - -1.22 (m), -1.26 - -1.62 (m), -2.66 - -2.98 (m); **IR** (neat, cm-1) 2923.6, 2853.2, 2362.4, 2341.2, 1709.6, 1449.2, 1283.4, 1020.2, 983.5; **HRMS** (ESI) $[M+2Na]^{2+}$ calcd for $C_{170}H_{142}Na_2O_{30}P_6$, 1447.3898; found, 1447.3906.

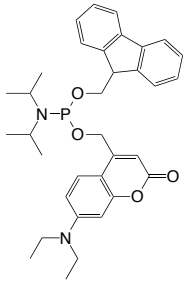
Synthesis of **14a**



155 mg (54.4 μmol, 1.0 eq.) of hexaphosphate **14** was dissolved in CH_2Cl_2 (3.00 mL). TFA (43.6 mL) and mixture of hydrazine acetate 9.75 mg (0.300 mmol, 6.0 eq) in MeOH (0.27 mL) were added. Reaction mixture was stirred overnight at room temperature. It was tracked by ^{31}P-NMR. After completion it was concentrated in vacuo and precipitated from MeOH, crude material **14a** was obtained (115 mg, 0.050 mmol, 87%) and it was used directly in the next step.

TLC (EtOAc): $\mathbf{R_f}$ = 0.43; ^{31}**P{1 H}-NMR** (203 MHz, $CDCl_3$): δ -0.76 , -0.82 , -1.49 , -1.75; ^{31}**P-NMR** (162 MHz, $CDCl_3$): δ -0.79 , -1.50 , -1.77; **HRMS** (ESI) $[M]^-$ calcd for $C_{146}H_{117}O_{24}P_6$, 2439.6364; found, 2439.6349

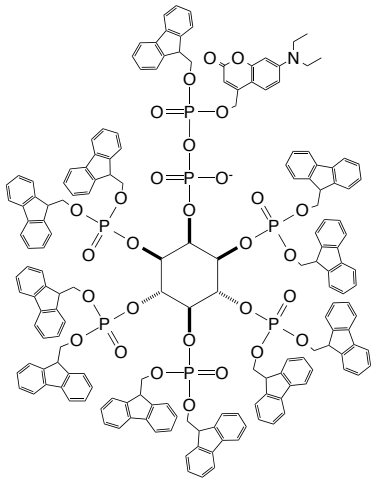
Synthesis of **15**



The compound was synthesized as described before in three steps starting from *myo*-inositol. Analytical data were identical with the values reported in the literature.[17a]

[17a] D. Subramanian, V. Laketa, R. Muller, C. Tischer, S. Zarbakhsh, R. Pepperkok, C. Schultz, *Nat. Chem. Biol.* **2010**, *6*, 324-326

Synthesis of **16a**



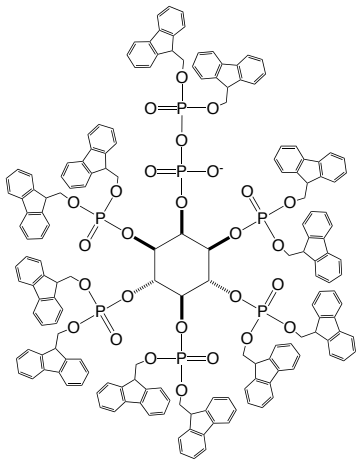
57.0 mg (23.3 µmol, 1.0 eq.) of crude hexaphosphate **14a** were dissolved in dry CH_2Cl_2 (2 mL). Coumarine-9-floreniylmethyl phosphoramidite (DEACM-Fm PA) **15** 26.8 mg (46.6 µmol, 2.0 eq) was added and mixture was coevaporated. Dry mixture was dissolved again in CH_2Cl_2 (2 mL) and DCI 8.25 mg (69.9 µmol, 3.0 eq.) was added. Reaction was stirred for 10 minutes.

Progress of the reaction was monitored by ^{31}P-NMR. After completion of the reaction, oxidation was achieved by slow (!) addition of 11.4 mg (46.6 µmol, 2.00 eq) *m*CPBA (70% moistened with water).

The reaction mixture was concentrated and MeOH (5 ml) was added. White precipitate was formed and it was centrifugated for 5 min. Solvent was removed and precipitate purified by column chromatography (EtOAc:Hexane 1:1 over EtOAc to EtOAc:MeOH=20:1). Yield: 38 mg **16a** as a colorless syrup (15.0 µmol, 55%).

TLC (EtOAc): $\mathbf{R_f}$ = 0.66; **^{1}H-NMR** (400 MHz, $CDCl_3$): δ 7.77 - 7.33 (m, 34H), 7.28 - 6.98 (m, 37H), 6.98 - 6.60 (m, 21H), 5.90 - 5.61 (m, 2H), 5.25 - 4.80 (m, 5H), 4.63 - 3.58 (m, 34H), 3.41 - 2.85 (m, 9H); **^{13}C-NMR** (101 MHz, $CDCl_3$): δ 195.77, 190.49, 187.38, 171.16, 161.41, 155.79, 150.34, 142.85, 141.05, 127.53, 127.16, 126.91, 125.19, 119.69, 119.66, 105.29, 97.19, 77.36, 77.24, 77.04, 76.73, 74.42, 74.15, 73.78, 69.92, 60.42, 50.83, 47.66, 44.37, 29.73, 21.07, 14.22, 12.43; **^{31}P{1 H}-NMR** (162 MHz, $CDCl_3$): δ 1.06 - -0.34 (m), -0.54 - -4.26 (m), -11.49 (d, *J* = 32.2 Hz), -13.11 (d, *J* = 33.8 Hz); **^{31}P-NMR** (162 MHz, $CDCl_3$): δ 1.28 - -0.41 (m), -0.36 - -1.94 (m), -2.24 - -5.41 (m), -10.51 - -12.25 (m), -12.56 - -14.24 (m); **IR** (neat, cm-1) 3122.2, 2931.3, 2362.4, 2239.9, 1639.9, 1653.7, 1600.6, 1422.2, 1240.0, 1009.6, 737.6, 503.3; **HRMS** (ESI) $[M]^+$ calcd for $C_{174}H_{143}NO_{29}P_7$, 2926.7915; found, 2926.7871.

Synthesis of **6b**



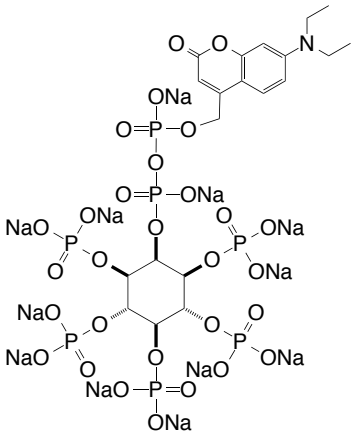
50.0 mg (20.4 µmol, 1.0 eq.) of crude hexaphosphate **14a** were dissolved in

dry CH_2Cl_2 (2 mL). 9-fluorenylmethyl phosphoramidite (Fm-PA) **13** 21.0 mg (40.9 µmol, 2.00 eq) was added and mixture was coevaporated. Dry mixture was dissolved again in CH_2Cl_2 (2 mL) and DCI 4.83 mg (40.9 µmol, 2.00 eq.) was added. Reaction was stirred for 10 minutes. Progress of the reaction was monitored by ^{31}P-NMR. After completion of the reaction, oxidation was achieved by slow (!) addition of 7.10 mg (40.9 µmol, 2.0 eq) *m*CPBA (70% moistened with water).

The reaction mixture was concentrated and MeOH (5 ml) was added. White precipitate was formed and it was centrifugated for 5 min. Solvent was removed and precipitate purified by column chromatography (DCM:MeOH 5:0.1 to DCM:MeOH=5:0.5). Yield: 40 mg **16b** as a colorless syrup (13.9 µmol, 68%).

TLC (EtOAc): $\mathbf{R_f}$ = 0.60; **^{1}H-NMR** (500 MHz, $CDCl_3$): δ 7.69 - 7.31 (m, 36H), 7.27 - 6.95 (m, 40H), 6.96 - 6.65 (m, 20H), 5.23 - 5.00 (m, 3H), 4.54 - 3.58 (m, 39H), (contains solvents: DCM, MeOH); **^{13}C-NMR** (126 MHz, $CDCl_3$): δ 142.89, 141.06, 127.54, 126.92, 125.24, 119.68, 99.98, 77.34, 77.02, 76.75, 76.70, 74.03, 70.65, 69.90, 53.43, 50.84, 47.67, 29.70; **^{31}P{1H}-NMR** (203 MHz, $CDCl_3$): δ 0.17 , -0.07 , -2.68 , -11.20 , -12.15; **^{31}P-NMR** (203 MHz, $CDCl_3$): δ -0.73 , -0.95 - -5.31 (m), -12.54 , -14.28; **IR** (neat, cm-1) 3365.2, 2923.6, 2337.3, 1717.3, 1449.2, 1265.1, 1017.3, 736.7; **HRMS** (ESI) $[M]^+$ calcd for $C_{174}H_{138}O_{27}P_7$, 2875.7594; found, 2875.7581.

Synthesis of **17**

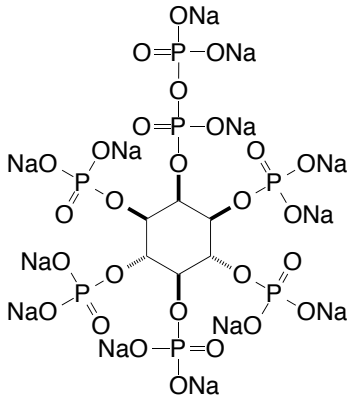


20.0 mg (6.83 µmol, 1.0 eq.) of **16a** were dissolved in DMF (3 mL) and

piperidine (0.5 ml) was added. The solution was stirred 40 minutes at room temperature. After completion of the deprotection solution was concentrated and product precipitated with Et_2O (10 mL). The precipitate was centrifugated and separated by decantation of solvent. The precipitate was once more dissolved in MeOH and crystallized by addition of Et_2O. Centrifugation process was repeated and yellowish crystals were obtained that were dried in vacuo. Piperidinium counter ions were exchanged to sodium by addition of excess NaI to MeOH solution of **17**. After 30 minutes of stirring sodium salt of **17** was precipitated: Yield: 8.02 mg of **17** (8.59 µmol, 95%).

^{1}H-NMR (500 MHz, D_2O): δ 7.58 (dd, *J* = 9.9 Hz, 1H), 6.85 (dd, 2H), 6.64 (s, 1H), 6.29 (s, 1H), 5.26 (d, *J* = 7.8 Hz, 2H), 4.98 (dd, *J* = 9.2 Hz, 9.2 Hz, 1H), 4.48 (ddd, *J* = 28.6, 28.6, 28.6 Hz, 2H), 4.17 (ddd, *J* = 19.1, 19.1, 19.1 Hz, 3H), 3.42 (q, *J* = 7.0 Hz, 4H), 1.13 (t, *J* = 7.2 Hz, 6H); **^{13}C-NMR** (126 MHz, 300 K, D_2O): δ 165.94, 155.61, 154.83, 154.77, 151.27, 125.61, 110.37, 106.13, 103.99, 97.39, 77.55, 76.63, 75.82, 73.23, 63.91, 48.88, 44.51, 22.25, 11.52; **^{31}P{1H}-NMR** (203 MHz, D_2O): δ 1.81 , 0.99 (d, *J* = 21.1 Hz), 0.46 (d, *J* = 24.1 Hz), -10.74 (d, *J* = 16.0 Hz), -11.08 (d, *J* = 16.4 Hz); **^{31}P-NMR** (203 MHz, 300 K, D_2O): δ 1.81 (d, *J* = 9.7 Hz), 0.99 (dd, *J* = 20.8, 9.2 Hz), 0.46 (dd, *J* = 24.4, 9.7 Hz), -10.76 (dd, *J* = 15.7, 8.1 Hz), -10.91 to -11.23 (m); **IR** (neat, cm-1) 3372.9, 2301.4, 1604.5, 1245.8, 1071.3, 948.8; **HRMS** (ESI) calcd for 483.4617 (M^{2-}, $C_{20}H_{32}NO_{29}P_7$), found 483.4617,

Synthesis of **2**

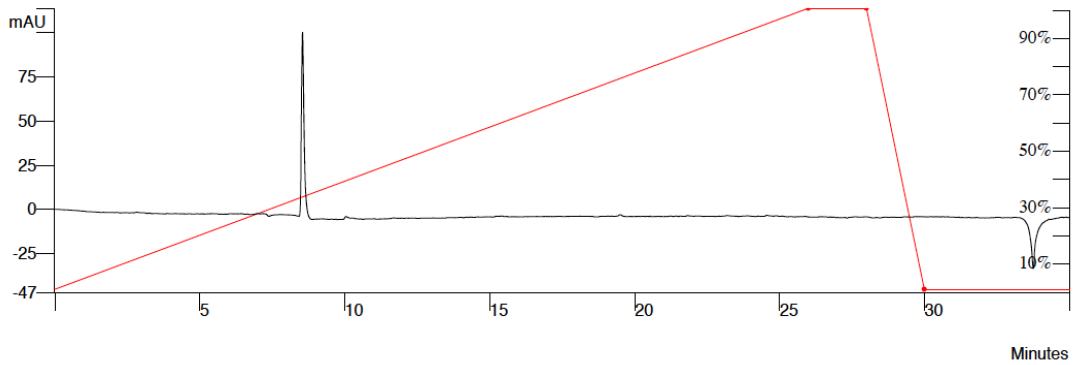


20.0 mg (6.95 µmol, 1.00 eq.) of **16b** were dissolved in DMF (3 mL) and piperidine (0.5 ml) was added. The solution was stirred 40 minutes at room

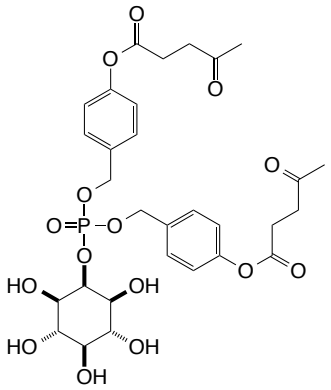
temperature. After completion of the deprotection solution was concentrated and product precipitated with Et_2O (10 mL). The precipitate was centrifugated and separated by decantation of solvent. The precipitate was once more dissolved in MeOH and crystallized by addition of Et_2O. Centrifugation process was repeated and yellowish crystals were obtained that were dried in vacuo. Piperidinium counter ions were exchanged to sodium by addition of excess NaI to MeOH solution of **2**. After 30 minutes of stirring sodium salt of **2** was precipitated: Yield: 6.91 mg of **2** (6.98 µmol, 97%).

^{1}H-NMR (500 MHz, D_2O): δ 5.06 - 5.04 (m, 2H), 4.91 (1H, peak assigned by 1H-31P HSQC correlation spectra (not shown)), 4.55 (dd, *J* = 18.9 Hz, 18.9, 1H), 4.27 (ddd, *J* = 15.9 Hz, 15.9 Hz, 15.9 Hz 2H); **^{13}C-NMR** (126 MHz, D_2O): δ 77.63, 76.06, 73.25; **^{31}P{1H}-NMR** (203 MHz, D_2O): δ 1.72 , 1.05 , 0.79 , -8.38 , -11.72 (d, *J* = 17.2 Hz); **^{31}P-NMR** (203 MHz, D_2O): δ 1.72 (d, *J* = 9.2 Hz), 1.04 (d, *J* = 9.6 Hz), 0.80 , -8.36 , -11.70; **IR** (neat, cm-1): 3410.5, 2361.4, 1635.3, 1056.8, 918.9; **HRMS** (ESI) $[M]^{2-}$ calcd for $C_6H_6O_{27}P_7$, 368.9066; found, 368.9068

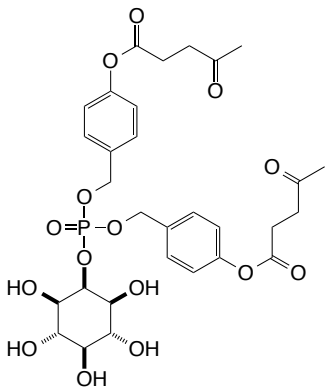
Purity Analysis of **17** by analytical Varian Pro Star HPLC and a C-5 reverse-phase column (Phenomenex C5 3.5 µm, 150 x 4.6 mm) using a 0 – 100 % gradient of CH_3CN in water over 30 minutes run a flow rate of 0.5 ml min^{-1}.



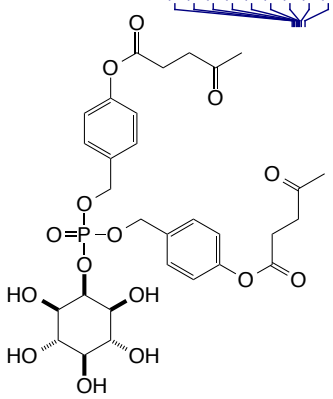
Compound 12, 31P 1H decoupled



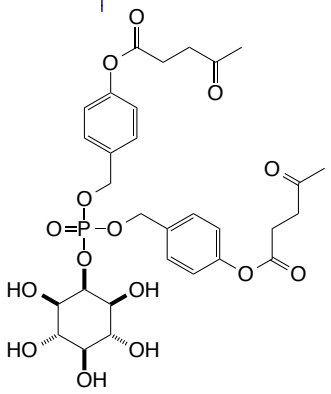
Compound 12, 31P 1H coupled



Compound 12, 1H

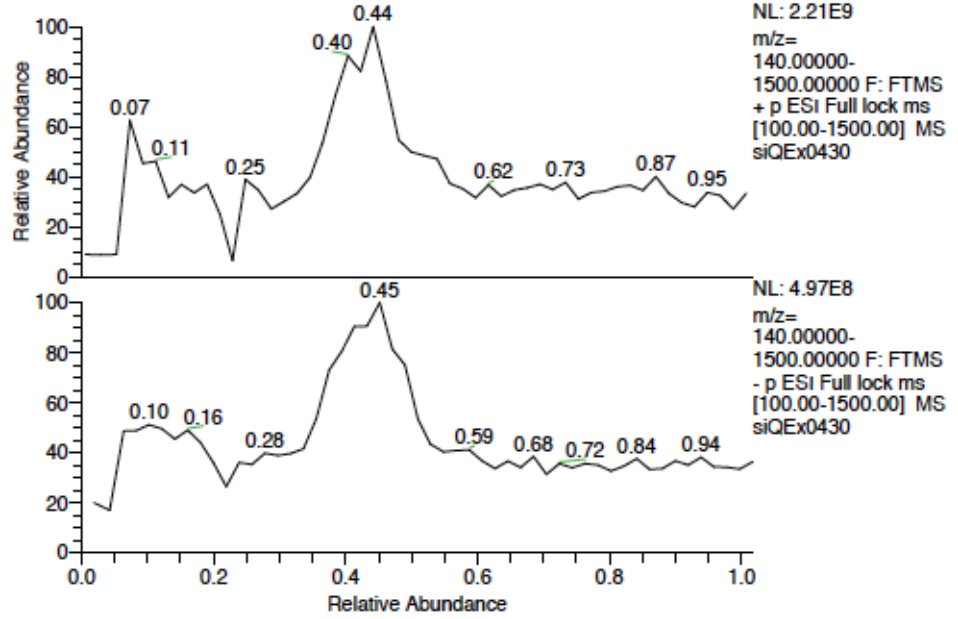


Compound 12, 13C



D:\Data\ASamp_Service\siQEx0430 05/18/15 11:24:02 Sample: IP-8-26P1
Client: Pavlovic (+)-HR-ESI-MS Solvent: MeOH

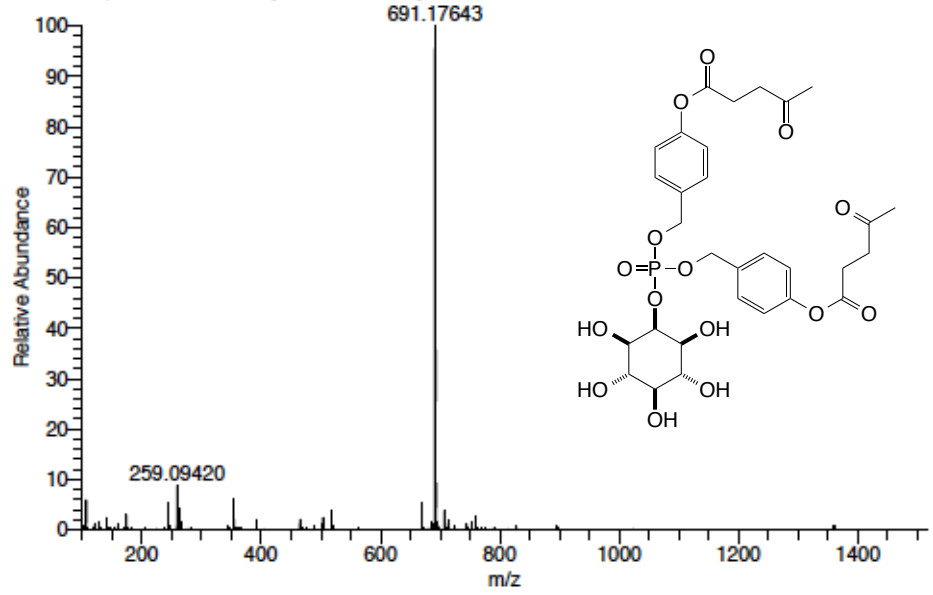
RT: 0.00 - 1.02



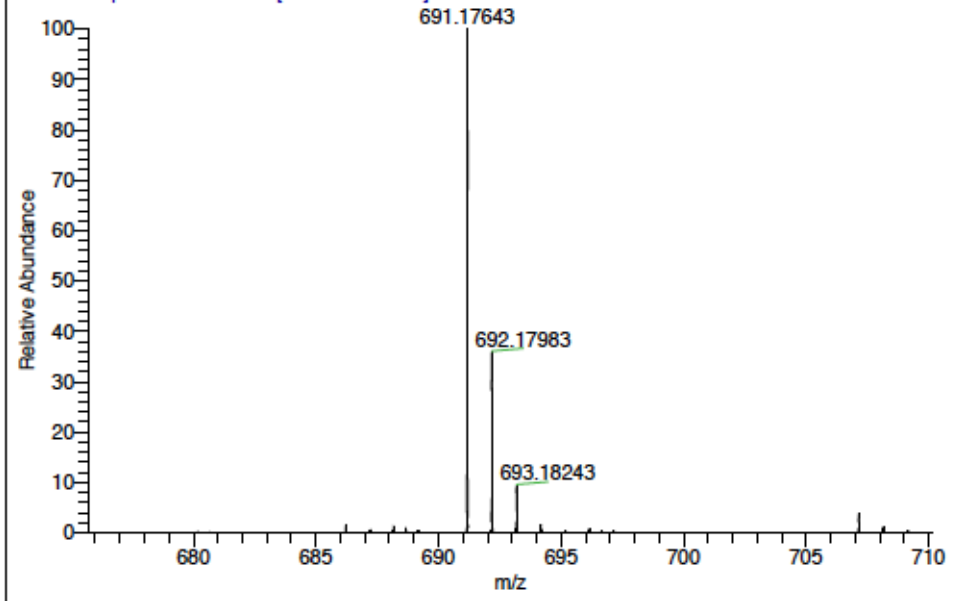
siQEx0430#47-49 RT: 0.46-0.48 AV: 2
SB: 10 0.06-0.25
T: FTMS + p ESI Full lock ms [100.00-1500.00]

m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
691.17643	372378688.0	100.00	691.17642	0.00	4.5	$C_{20}H_{36}O_{21}N_4Na$
			691.17623	0.29	12.5	$C_{30}H_{37}O_{15}NaP$
			691.17622	0.30	18.0	$C_{29}H_{31}O_{10}N_7NaP$
			691.17615	0.40	3.0	$C_{19}H_{37}O_{24}N_3$
			691.17614	0.41	8.5	$C_{18}H_{31}O_{19}N_{10}$

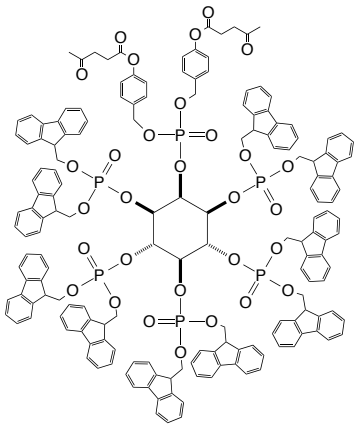
siQEx0430 #46-49 RT: 0.46-0.48 AV: 2 SB: 10 0.06-0.25 NL: 3.58E8
T: FTMS + p ESI Full lock ms [100.00-1500.00]



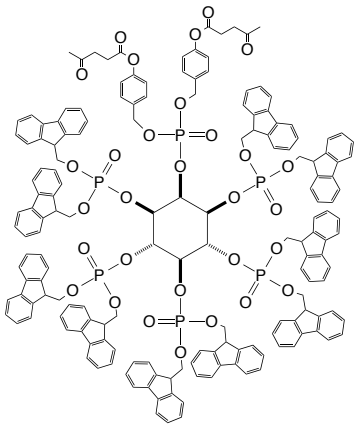
siQEx0430 #47-49 RT: 0.46-0.48 AV: 2 SB: 10 0.06-0.25 NL: 3.58E8
T: FTMS + p ESI Full lock ms [100.00-1500.00]



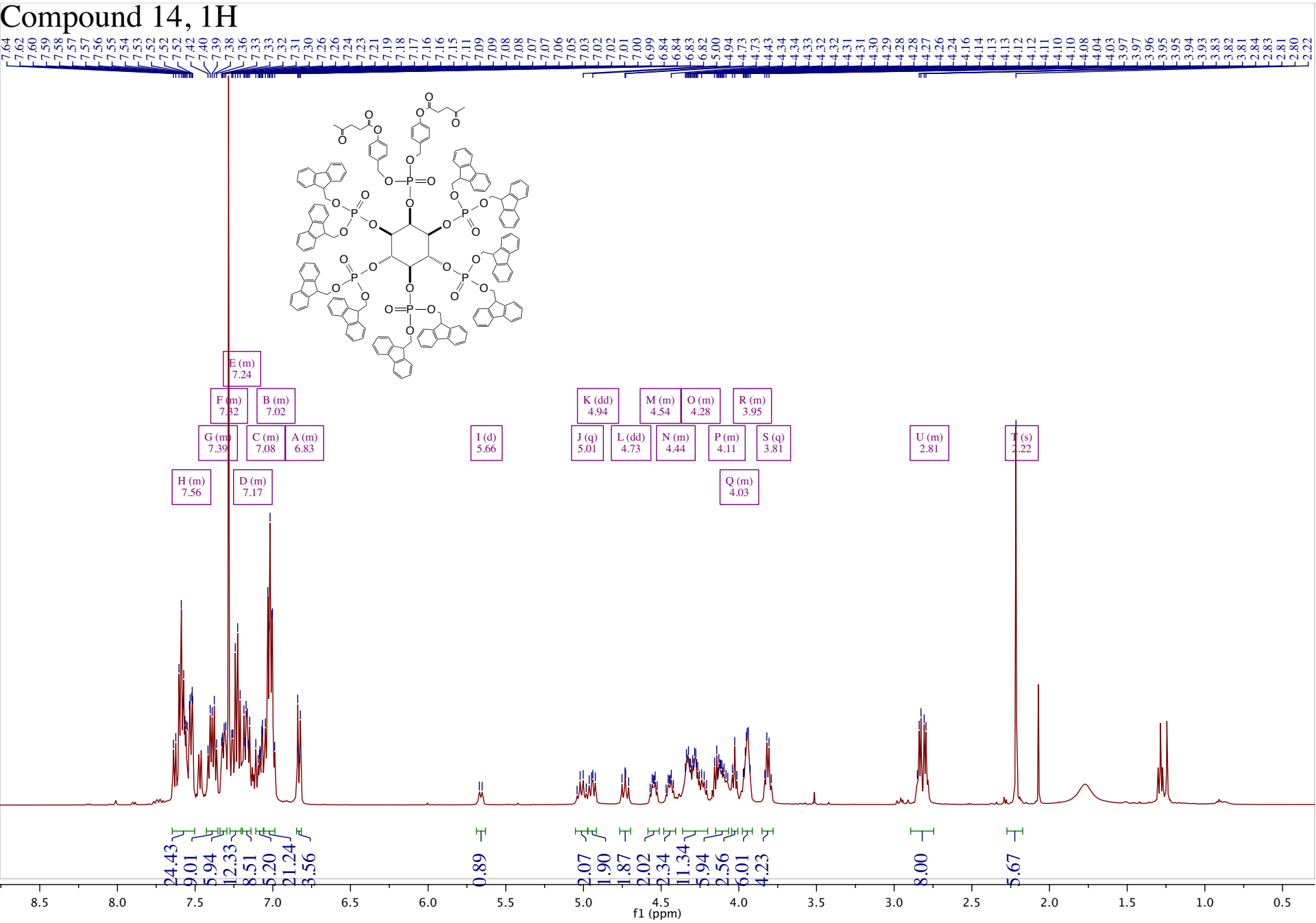
Compound 14, 31P 1H decoupled



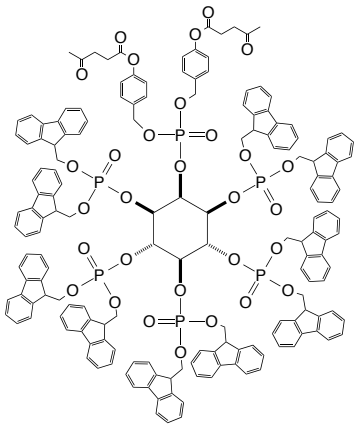
Compound 14, 31P 1H coupled



Compound 14, 1H



Compound 14, 13C



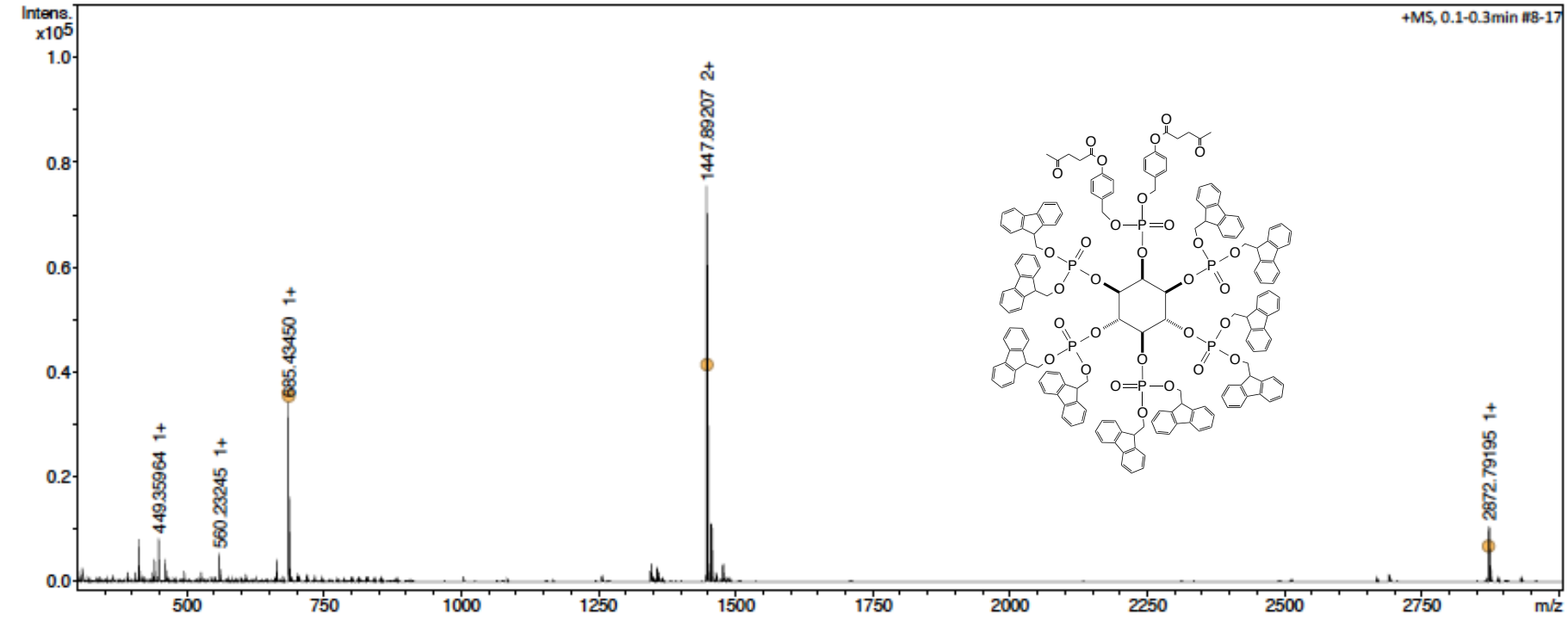
HR-ESI-MS (Bruker maXis)

Analysis Info

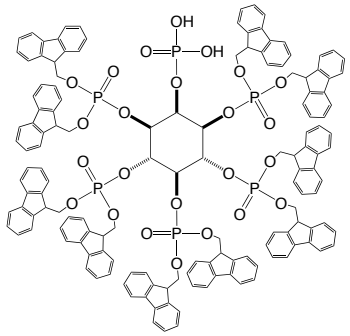
Analysis Name	D:\Data\Service\8017jehres.d	Acquisition Date	6/8/2015 3:10:14 PM	
Method	tune_low_modified_09_01_14_pos.m	Operator	ust	
Sample Name	mm-2074sn	Instrument	maXis	255552.00033
Comment	Solvent: MeCN Client: Pavlovic			

Acquisition Parameter

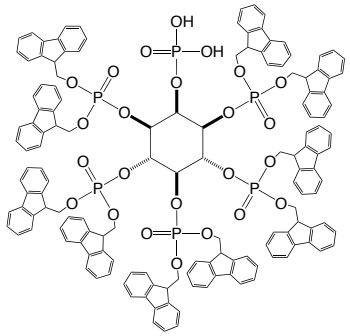
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.5 Bar
Scan Begin	50 m/z	Set Capillary	5000 V	Set Dry Heater	180 °C
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min



Compound 14a, 31P 1H decoupled (crude)



Compound 14a, 31P 1H coupled (crude)



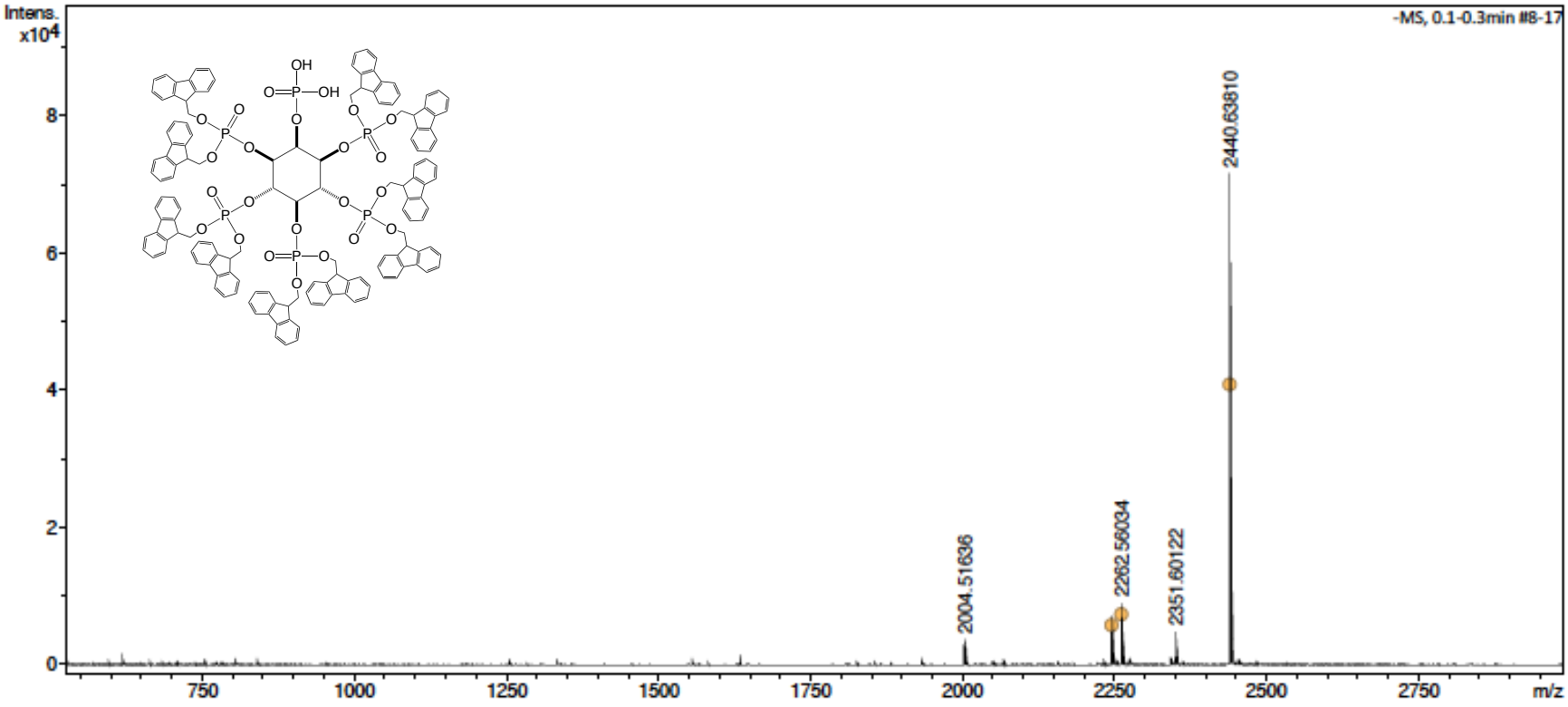
HR-ESI-MS (Bruker maXis)

Analysis Info

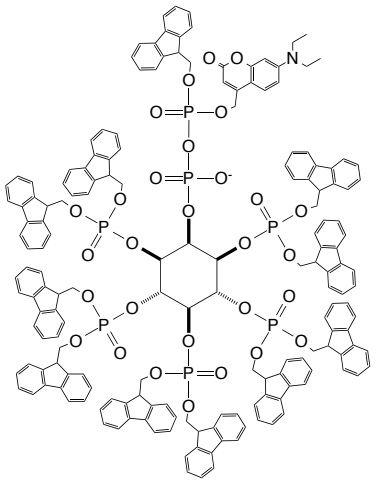
Analysis Name	D:\Data\Service\8035jehres.d	Acquisition Date	6/19/2015 3:30:21 PM
Method	tune_low_neg_Aug2014.m	Operator	ust
Sample Name	IP-8-58P1	Instrument	maXis 255552.00033
Comment	Solvent: MeCN Client: Pavlovic		

Acquisition Parameter

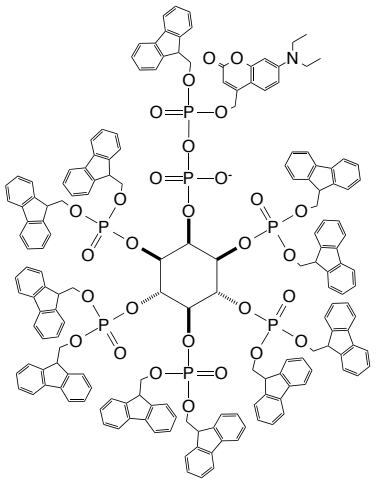
Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	1.0 Bar
Scan Begin	50 m/z	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min



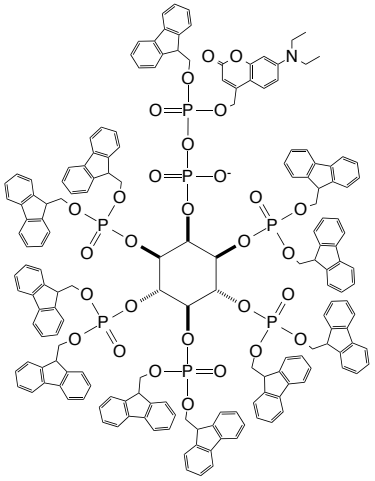
Compound 16a, 31P 1H decoupled (mixture of diastereomers)

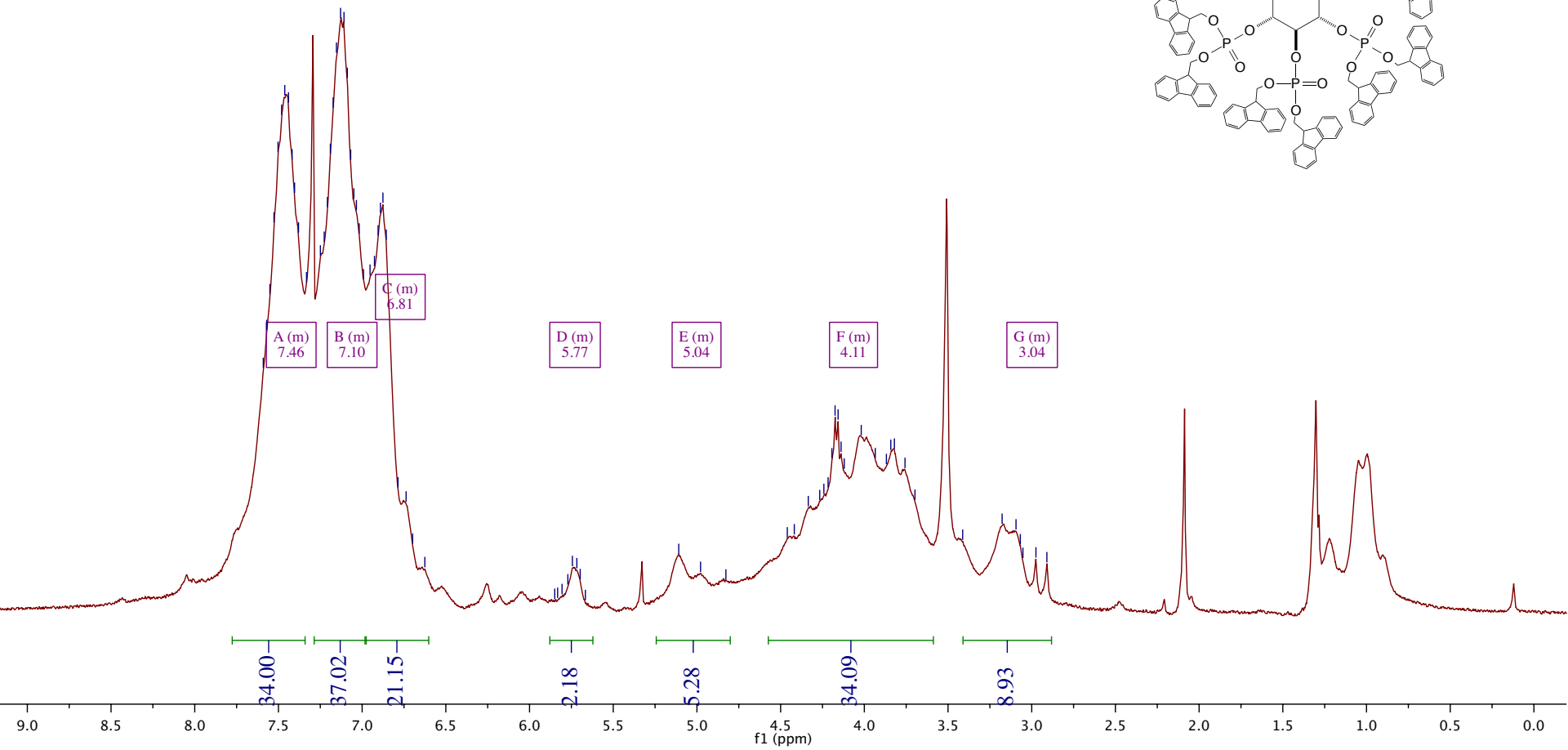


Compound 16a, 31P 1H coupled (mixture of diastereomers)

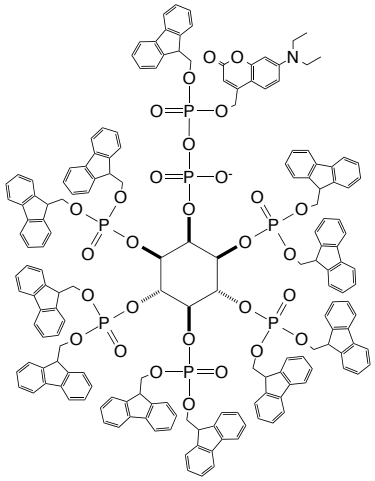


Compound 16a, 1H (mixture of diastereomers)





Compound 16a, 13C (mixture of diastereomers)



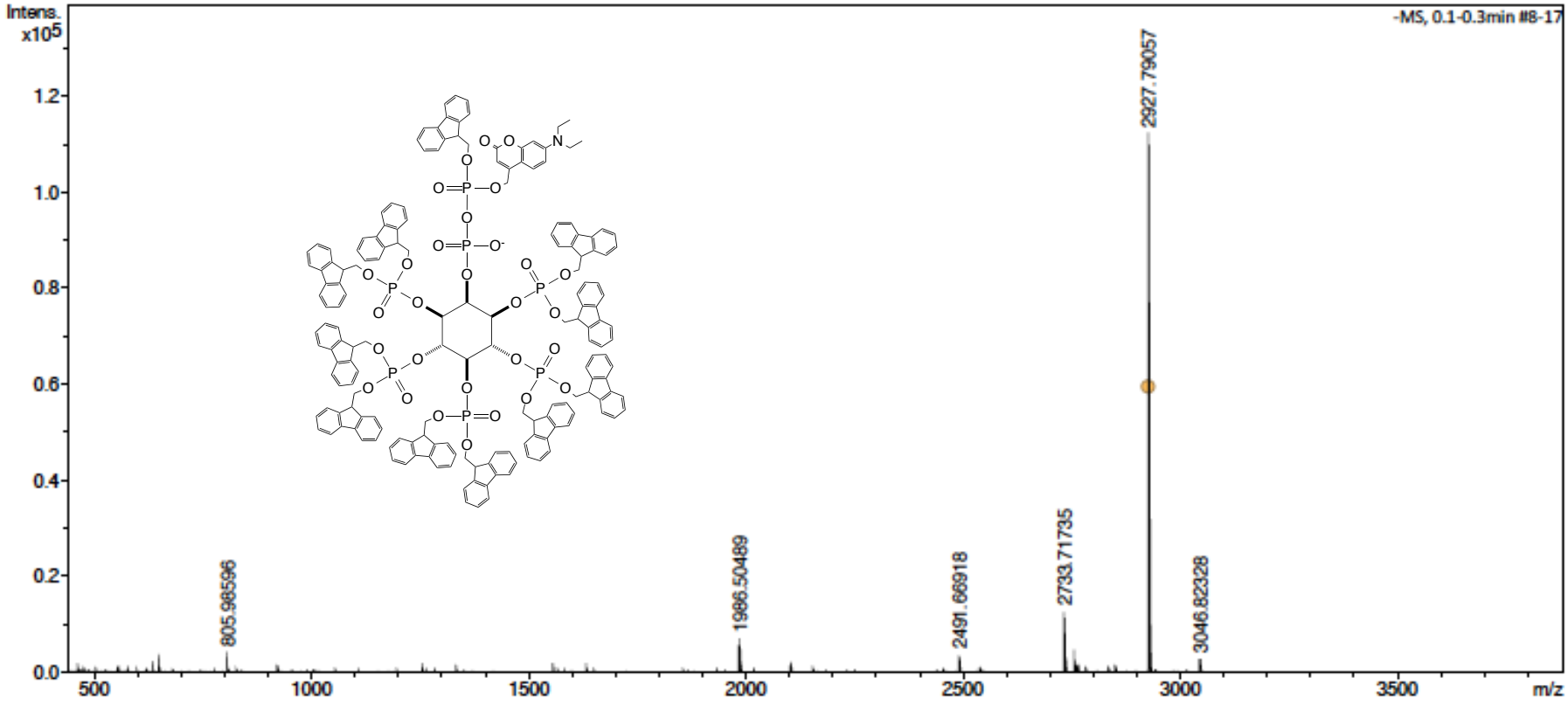
HR-ESI-MS (Bruker maXis)

Analysis Info

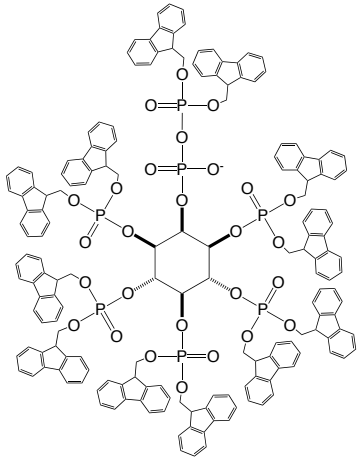
Analysis Name	D:\Data\Service\8045jehres.d	Acquisition Date	6/25/2015 1:13:07 PM
Method	tune_low_neg_Aug2014_TuneMix.m	Operator	ust
Sample Name	IP-8-63P1	Instrument	maXis 255552.00033
Comment	Solvent: H2O Client: Pavlovic		

Acquisition Parameter

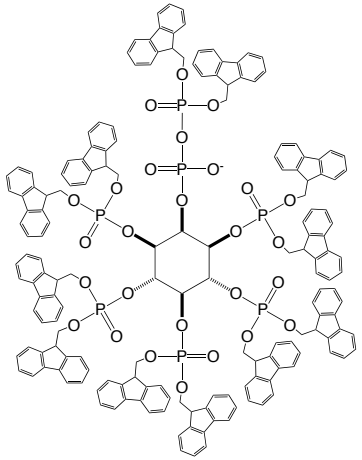
Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	1.0 Bar
Scan Begin	50 m/z	Set Capillary	5000 V	Set Dry Heater	180 °C
Scan End	3500 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min



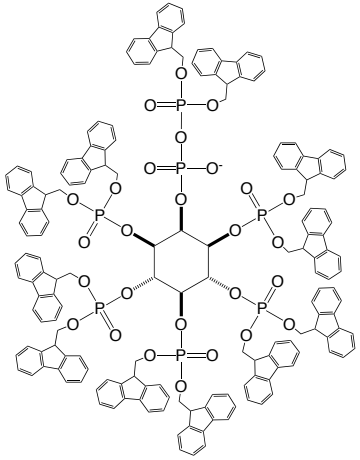
Compound 16b, 31P 1H decoupled



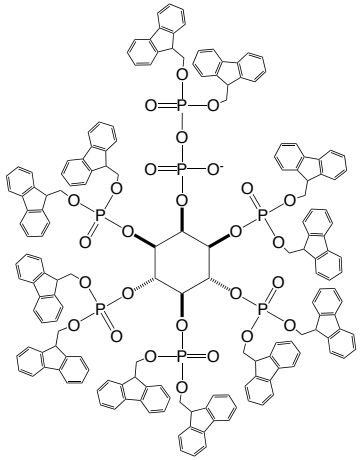
Compound 16b, 31P 1H coupled



Compound 16b, 1H



Compound 16b, 13C



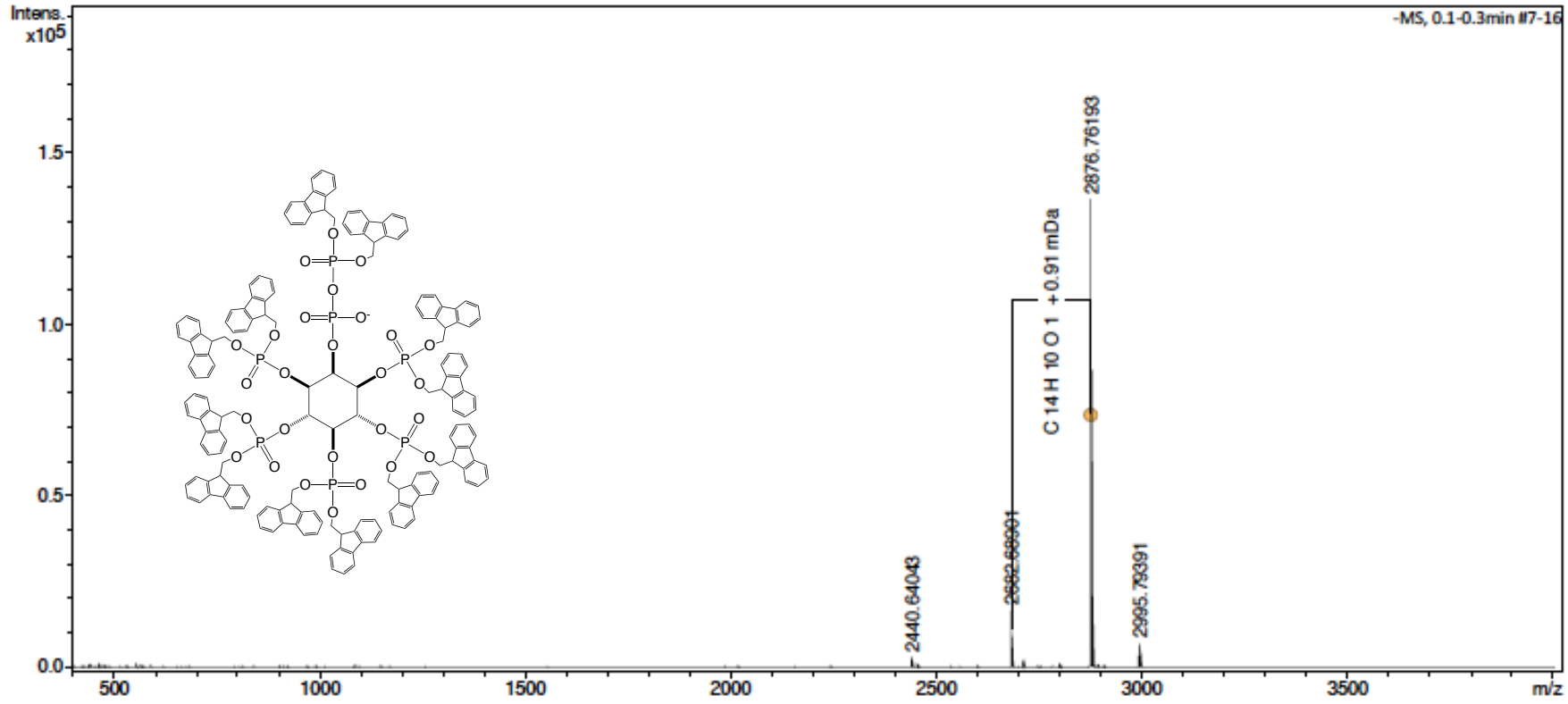
HR-ESI-MS (Bruker maXis)

Analysis Info

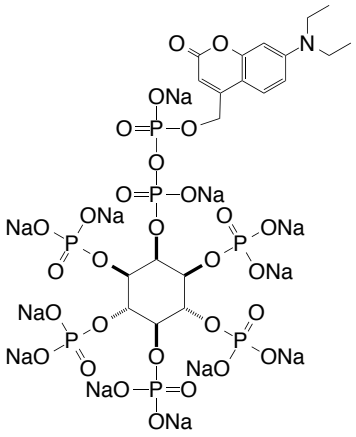
Analysis Name	D:\Data\Service\8039jehres.d	Acquisition Date	6/23/2015 3:19:22 PM
Method	tune_low_neg_Aug2014.m	Operator	ust
Sample Name	IP-8-62P1	Instrument	maXis 255552.00033
Comment	Solvent: MeOH/CHCl3 3:2 Client: Pavlovic		

Acquisition Parameter

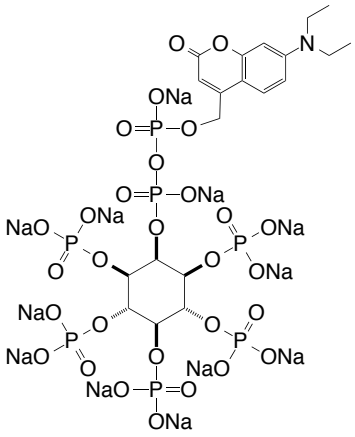
Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.5 Bar
Scan Begin	50 m/z	Set Capillary	5000 V	Set Dry Heater	180 °C
Scan End	4000 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min



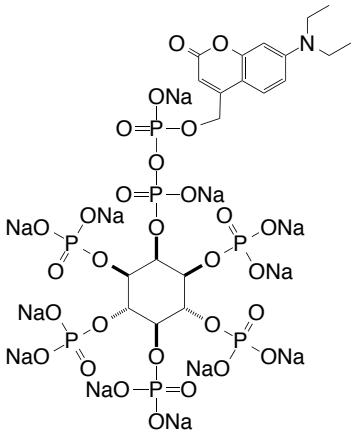
Compound 17, 31P 1H decoupled



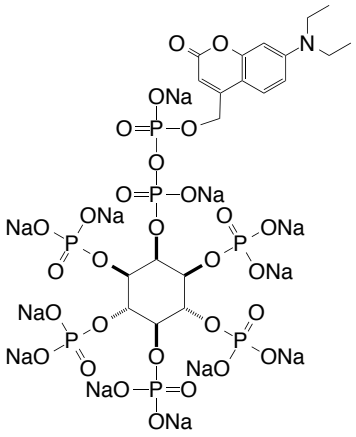
Compound 17, 31P 1H coupled



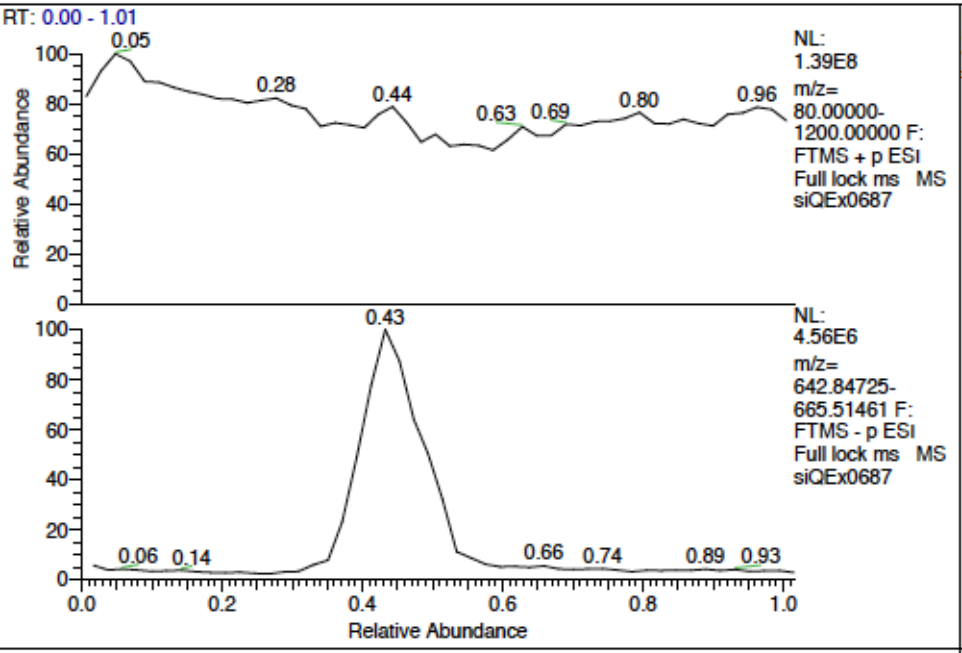
Compound 17, 1H



Compound 17, 13C



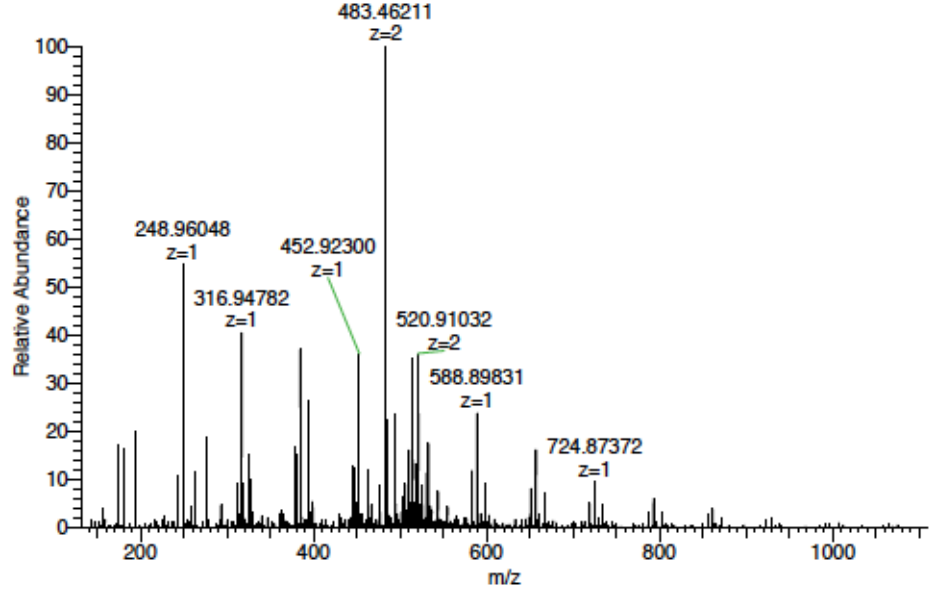
D:\Data\Service\Data\siQEx0687 06/23/15 09:59:26 Sample: IP-8-64P1
Client: Pavlovic (+)-HR-ESI-MS Solvent: Water



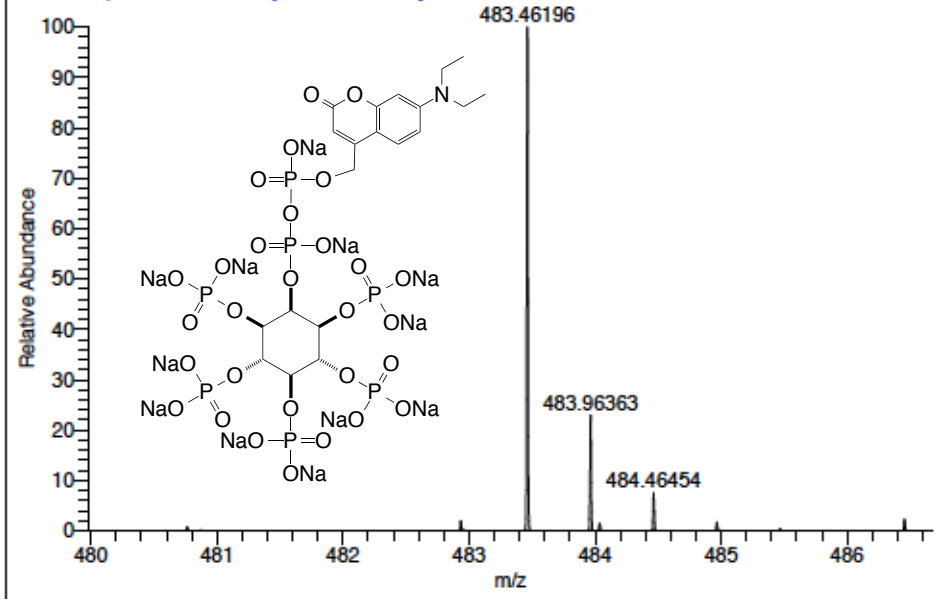
siQEx0687#42-46 RT: 0.43-0.47 AV: 3
SB: 12 0.08-0.31
T: FTMS - p ESI Full lock ms [133.40-2000.00]
m/z= 483.04487-483.67802

m/z	Intensity	Relative	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
483.46195	8917291.0	100.00	483.46192	0.02	13.5	$C_{21}H_{33}O_{18}N_6P_{10}$
			483.46202	-0.08	5.5	$C_{11}H_{32}O_{24}N_{10}P_9$
			483.46187	0.08	4.5	$C_{16}H_{35}O_{29}N_2P_8$
			483.46202	-0.08	0.0	$C_{12}H_{38}O_{29}N_3P_9$
			483.46187	0.08	10.0	$C_{15}H_{29}O_{24}N_9P_8$
			483.46207	-0.12	28.5	$C_{28}H_{16}O_{20}N_{10}P_5$
			483.46207	-0.13	23.0	$C_{29}H_{22}O_{25}N_3P_5$
			483.46177	0.18	18.0	$C_{25}H_{30}O_{18}N_5P_9$
			483.46171	0.23	9.0	$C_{20}H_{32}O_{29}NP_7$
			483.46171	0.24	14.5	$C_{19}H_{26}O_{24}N_8P_7$

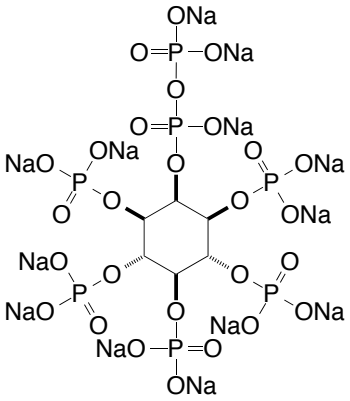
siQEx0687 #44 RT: 0.45 AV: 1 SB: 24 0.07-0.29 , 0.71-0.98 NL: 9.65E6
T: FTMS - p ESI Full lock ms [133.40-2000.00]



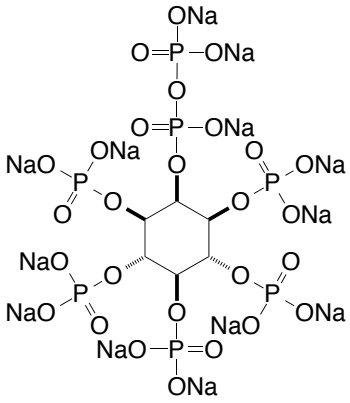
siQEx0687 #39-43 RT: 0.41-0.43 AV: 2 SB: 24 0.07-0.29 , 0.71-0.97 NL: 5.71E6
T: FTMS - p ESI Full lock ms [133.40-2000.00]



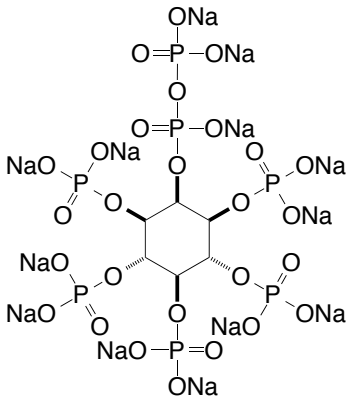
Compound 2, 31P 1H decoupled



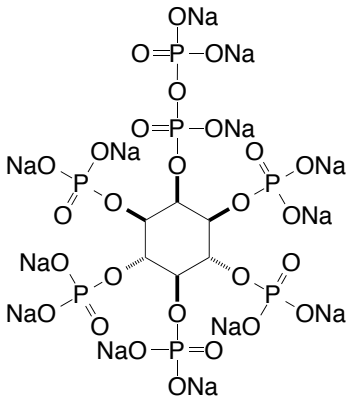
Compound 2, 31P 1H coupled



Compound 2, 1H



Compound 2, 13C



HR-ESI-MS (Bruker maXis)

Analysis Info

Analysis Name	D:\Data\Service\7961jehres.d	Acquisition Date	4/17/2015 1:15:46 PM
Method	tune_low_neg_Aug2014.m	Operator	ust
Sample Name	IP-8-36T1	Instrument	maXis 255552.00033
Comment	Solvent: H2O Client: Pavlovic		

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.5 Bar
Scan Begin	50 m/z	Set Capillary	6000 V	Set Dry Heater	180 °C
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min