Supporting Information

Selective Acylation of Nucleosides, Nucleotides and Glycerol-3-phosphocholine in Water

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Contents

General Information.........................................................................................S2
General protocols for the acetylation of nucleotides.......................................S3
General procedure for the diacylation of glycerol derivatives ......................S4
NMR Studies of acylation reactions..............................................................S5
Characterization data for compounds 5a – 5s.........................................S13
References.....................................................................................................S49
General Experimental

Nucleotides, nucleosides, glycerol-3-phosphocholine and N-acetyl imidazole were purchased from Alfa Aesar, Calbiochem, Carbosynth and Sigma-Aldrich and were used without further purification. Deionised water was obtained from an Elga Option 3 purification system. Flash column chromatography was carried out using a Biotage Isolera Four purification system and a Biotage KP-C18-HS Snap Cartridge. Solution pH values were measured using a Mettler Toledo Seven Compact pH meter with a Mettler Toledo InLab semi-micro pH probe. The readings for D₂O solutions are reported as pD, and corrected according to Covington et al.¹ The readings for H₂O and H₂O/D₂O (1:1) solutions are reported uncorrected. NMR Spectra were recorded on a Bruker AVANCE III (400) or Bruker Avance III (600) spectrometer, equipped with a gradient probe (400) or cryoprobe (600). All chemical shifts (δ) are reported in parts per million (ppm) relative to the residual solvent peak, and ¹H and ¹³C chemical shifts relative to TMS were calibrated using the residual solvent peak: HOD (δH 4.75 ppm) or CDCl₃ (δH 7.26 ppm). Coupling constants (J) are reported in Hertz (Hz). Spin multiplicities are indicated by symbols: s (singlet); d (doublet); t (triplet); q (quartet); ABX (ABX spin system); ABXY (ABXY spin system); obs. (obscured/coincidental signals), or a combination of these. NMR data are reported as follows: chemical shift (multiplicity, coupling constants (J), number of protons, nuclear assignment). Spectra were recorded at 298 K. Signal assignments are all made by COSY, HMBC, HSQC and DEPT experiments. ¹H NMR spectra (H₂O/D₂O) are solvent suppressed (noesygppr1d) with presaturation and spoil gradients. Stacked spectra are offset, x = 0.25 ppm. Infrared (IR) spectra were recorded with a Shimadzu 100 FTIR spectrometer. Absorption maxima are reported in wavenumbers (cm⁻¹). Mass spectra were determined by the University College London mass spectrometry service by electrospray ionisation (ESI) using a Waters LCT Premier XE or Thermo Finnigan MAT 900XP instrument.
General protocols for nucleoside acetylation

**Protocol A:** Nucleoside/nucleotide (2; 100mM) and thioacetic acid (3; 10 eq.) were dissolved in D$_2$O/H$_2$O (1:1) and adjusted to pH 8 by addition of 4M NaOH. Cyanoacetylene 4 (10 eq. or 20 eq., 1M in water) was added and the solution was readjusted to pH 8 by addition of 1M HCl/NaOH. The total nucleotide concentration was volumetrically adjusted to 100mM and the reaction monitored by NMR spectroscopy.

**Protocol B:** Nucleoside/nucleotide (2; 100mM) was dissolved in D$_2$O/H$_2$O (1:1) and the pH adjusted to the desired value by addition of 4M NaOH. The solution was purged with argon for 15 min, potassium ferricyanide (5 – 20 eq.) was added followed by potassium thioacetate (5 – 20 eq.). The pH was stabilised by addition of 1M HCl/NaOH and the reaction monitored by NMR spectroscopy.

**Protocol C:** Nucleoside/nucleotide (2; 100mM) and N-acetyl imidazole (1a; 1-10 eq.) were dissolved in D$_2$O/H$_2$O (1:1). The solution pH was stabilised at the desired value through addition of 4M HCl/NaOH and the reaction monitored by NMR spectroscopy. The product was purified by reverse-phase (C18) flash column chromatography (eluted at pH 4 with 100mM NH$_4$HCO$_3$/MeCN 98:2 to 80:20). The fractions containing 5 were lyophilised to yield a white powder.

General procedure for the diacylation of glycerol 10

**Protocol A:** Glycerol (10, 100mM) and N-acetyl imidazole (1a, 10 eq.) were dissolved in D$_2$O/H$_2$O (1:1) and the pH stabilised at the desired value through addition of 4M HCl/NaOH and the reaction was monitored by NMR spectroscopy.

**Protocol B:** Glycerol (10; 100mM) and N-acyl imidazole (1a-e; 10 eq.) were suspended in D$_2$O and minimal MeCN (1:1 - 20:1 D$_2$O/MeCN) was added to obtain biphasic solutions. The solution pH was measure (pH 7.0 -7.5), but not further adjusted. The suspensions were stirred vigorously for 4 h; no pH change was observed. The biphasic solutions were lyophilised and the lyophilite was dissolved in CDCl$_3$, CDCl$_3$/MeOD or MeOD and analysed by NMR spectroscopy.
Figure S1: $^1$H NMR spectra (400 MHz, D$_2$O/H$_2$O 1:1, 1.5 – 8.5 ppm) to show the reaction of 2a with thioacetic acid (3; 10 eq.) and cyanoacetylene (4; 10 eq.) after: A) 30 min; B) 24 h. Inset: $^1$H NMR spectrum (400 MHz, D$_2$O/H$_2$O 1:1, 1.8 – 2.6) integration for the diacetylated cytidine 5a shown.

Figure S2: $^1$H NMR spectra (600 MHz, D$_2$O/H$_2$O 1:1, 1.5 – 8.5 ppm) for the acetylation of cytidine 2a with: A) thioacetate (3, 10 eq.) and ferricyanide (10 eq.); B) thioacetate (3, 10 eq.) and ferricyanide (20 eq.). Inset: $^{31}$P NMR spectrum (162 MHz, D$_2$O/H$_2$O 1:1, -20 – 20 ppm) showing 5'-phosphate 5a (3.70 ppm) and pyrophosphate 8 (-7.29 ppm) observed upon the reaction of cytidine 2a with thioacetate (3, 10 eq.) and ferricyanide (20 eq.).
Figure S3: $^1$H NMR spectra (600 MHz, D$_2$O/H$_2$O 1:1, 1.5 – 8.5 ppm) showing the reaction of 2a with N-acetylmidazole (1a, 10 eq.) after 1 d in D$_2$O/H$_2$O 1:1: A) pH 5; B) pH 6; C) pH 7; D) pH 8.

Figure S4: $^1$H NMR spectra (600 MHz, D$_2$O/H$_2$O 1:1, 1.5 – 8.5 ppm) showing the reaction of cytidine 2b with N-acetylimidazole (1a, 10 eq.) after 1 d at: A) pH 5; B) pH 6; C) pH 7; D) pH 8.
Figure S5: $^1$H NMR spectra (600 MHz, D$_2$O/H$_2$O 1:1, 1.5 – 8.5 ppm) showing the reaction of uridine 2b with: A) 1 eq. of N-acetyl imidazole (1a); B) 2 eq. of N-acetyl imidazole (1a); C) 3 eq. of N-acetyl imidazole (1a); D) 4 eq. of N-acetyl imidazole (1a); E) 6 eq. of N-acetyl imidazole (1a); F) 8 eq. of N-acetyl imidazole (1a); G) 10 eq. of N-acetyl imidazole (1a) after 1 d at pH 8 and room temperature.
Figure S6: $^1$H NMR spectra (400 MHz, D$_2$O/H$_2$O 1:1, 1.5 – 8.5 ppm) showing the reaction of cytidine 2i with N-acetyl imidazole (1a; 10 eq.) after 1 d at: A) pH 5; B) pH 6; C) pH 7; D) pH 8. Inset: $^1$H NMR spectrum (400 MHz, D$_2$O/H$_2$O 1:1, 1.5 – 2.2 ppm) showing the acetyl groups for the tri-O-acetyl cytidine 5i.
Figure S7: $^1$H NMR spectra (600 MHz, D$_2$O/H$_2$O 1:1, 1.5 – 8.5 ppm) showing the reaction of 2p after 1d at pH 8 with: A) 1 eq. of N-acetyl imidazole (1a); B) 2 eq. of N-acetyl imidazole (1a); C) 3 eq. of N-acetyl imidazole (1a); D) 4 eq. of N-acetyl imidazole (1a); E) 10 eq. of N-acetyl imidazole (1a). Inset: $^1$H NMR spectrum (600 MHz, D$_2$O/H$_2$O 1:1, pH 8, 4.9 – 5.5 ppm) showing H-(C2') and H-(C3') protons for 5p (in blue); H-(C3') for 3'-O-acyl-arabino-cytidine 6p (in green) and H-(C2') for 2'-O-acyl-arabino-cytidine 7p (in orange) as was assigned by $^1$H-$^1$H NMR and $^1$H-$^{13}$C-NMR.
Figure S8: $^1$H NMR spectra (400 MHz, D$_2$O/H$_2$O 1:1, 3.2 – 5.5 ppm) showing the reaction of glycerol 10 with $N$-acetyl imidazole (1a, 10 eq.) after 4 h at: A) pH 5; B) pH 6; C) pH 7; D) pH 8.

Figure S9: $^1$H NMR spectra (400 MHz, 3.3 – 5.5 ppm) showing the reaction of 10 at pH 8 with $N$-acetyl imidazole 1a: A) 10 eq.; B) 20 eq. Inset: $^1$H NMR spectrum (400 MHz, 1.6 – 2.0 ppm) showing the acetyl peaks for the products.
Figure S10: $^1$H NMR spectra (400 MHz, 2.5 – 5.5 ppm) showing the reaction of glycerol 10 with: A) N-acetyl imidazole (1a); B) N-butyryl imidazole (1b); C) N-hexanoyl imidazole (1c); D) N-octanoyl imidazole (1d); E) N-decanoyl imidazole (1e). All reactions were performed at pH 7 and using 10 eq. of acylating agent. Spectrum A was recorded in a mixture D$_2$O/H$_2$O 1:1; spectra B-E were recorded in CDCl$_3$/MeOD after lyophilisation.

Figure S11: $^1$H NMR spectra (400 MHz, D$_2$O/H$_2$O 1:1, 1.5 – 5.5 ppm) showing the reaction of equimolar mixtures of uridine 5'-phosphate 2b and glycerol 10 with N-acetyl imidazole (1a, 10 eq.) at pH 7 after 4 h. Characteristic peaks for diacetyl uridine 5b are indicated in blue; diacetyl glycerol is observed in low yield (9%).
Characterisation data for the acetylated derivatives

2',3'-di-O-acetyl-β-cytidine-5'-phosphate (5a)

Starting from 2a (160 mg, 0.50 mmol), yield = 172 mg (85%) as a white powder. 

\(^1\)H NMR (600 MHz, D\(_2\)O) δ 8.08 (d, J = 7.9 Hz, 1 H, H-(C6)), 6.22 (d, J = 7.9 Hz, 1 H, H-(C5)), 6.11 (d, J = 5.1 Hz, 1 H, H-(C1'))), 5.41 (dd, J = 5.4, 5.1 Hz, 1 H, H-(C2')), 5.38 (dd, J = 5.4, 4.4 Hz, 1 H, H-(C3')), 4.48 (d, J = 4.9, 4.4, 2.4 Hz, 1 H, H-(C4')), 4.13 (ABXY, J = 11.9, 4.9, 2.4 Hz, 1 H, H-(C5'')), 2.09 (s, 3 H, Ac-(C3')). 

\(^{13}\)C NMR (151 MHz, D\(_2\)O) δ 173.4 (OAc), 173.1 (OAc), 160.7 (C4), 150.1 (C2), 144.3 (C6), 96.5 (C5), 88.2 (C1'), 82.5 (C4'), 74.5 (C2'), 74.0 (C2'), 71.5 (C3'), 64.4 (d, C5'), 20.6 (3'-OAc), 20.5 (2'-OAc).

\(^{31}\)P NMR (162 MHz, D\(_2\)O, 1\(^\text{H}\)-decoupled) δ 0.30. IR (neat, cm\(^{-1}\)) 1746, 1660, 1489, 1462, 1375, 1075. HRMS (ESI) (m/z): [M+H\(^+\)] calcd for formula C\(_{13}\)H\(_{19}\)N\(_3\)O\(_{10}\)P, 408.0803; found, 408.0810. 

The spectroscopic properties of this compound were consistent with the data reported in the literature.²

2',3'-di-O-acetyl β-uridine-5'-phosphate (5b)

Starting from 2b (disodium salt; 185 mg, 0.50 mmol), yield = 137 mg (67%) as a white solid. 

\(^1\)H NMR (600 MHz, D\(_2\)O) δ 7.82 (d, J = 8.1 Hz, 1 H, H-(C6)), 6.09 (d, J = 5.1 Hz, 1 H, H-(C1')), 5.86 (d, J = 8.1 Hz, 1 H, H-(C5)), 5.34 - 5.42 (m, 2 H, H-(C2' and H-(C3')), 4.43 (dt, J = 4.8, 2.7 Hz, 1 H, H-(C4')), 4.11 (ABXY, J = 11.9, 4.8, 2.5 Hz, 1 H, H-(C5'')), 2.09 (s, 3 H, Ac-(C3')), 2.03 (s, 3 H, Ac-(C2')).

\(^{13}\)C NMR (151 MHz, D\(_2\)O) δ 173.4 (OAc), 173.2 (OAc), 166.7 (C4), 152.2 (C2), 142.3 (C6), 103.5 (C5), 87.5 (C1'), 82.0 (d, C4'), 74.0 (C2'), 71.6 (C3'), 64.9 (d, C5'), 20.6, 20.4 (3'-OAc and 2'-OAc).

\(^{31}\)P NMR (162 MHz, D\(_2\)O, 1\(^\text{H}\)-decoupled) δ -0.26. IR (neat, cm\(^{-1}\)) 1746, 1689, 1462, 1431, 1378, 1240, 1056. HRMS (ESI) (m/z): [M+H\(^+\)] calcd for formula C\(_{13}\)H\(_{18}\)N\(_2\)O\(_{11}\)P, 409.0643; found, 409.0650.
The spectroscopic properties of this compound were consistent with the data reported in the literature.\(^3\)

2',3'-di-O-acetyl β-adenosine-5'-phosphate (5c)

Starting from 2c (170 mg, 0.50 mmol), yield = 180 mg (85%) as a white solid. 
\(^1\)H NMR (600 MHz, D\(_2\)O) \(\delta 8.40\) (s, 1 H, H-(C8)), 8.11 (s, 1 H, H-(C2)), 6.26 (d, \(J = 6.1\) Hz, 1 H, H-(C1')), 5.67 (dd, \(J = 6.1, 5.4\) Hz, 1 H, H-(C2')), 5.55 (dd, \(J = 5.4, 3.4\) Hz, 1 H, H-(C3')), 4.52 - 4.57 (m, 1 H, H-(C4')), 4.12 (ABXY, \(J = 11.9, 4.8, 2.6\) Hz, 1 H, H-(C5')), 4.08 (ABXY, \(J = 11.9, 5.4, 3.0\) Hz, 1 H, H-(C5'')). 2.14 (s, 3 H, Ac-(C3')). 1.98 (s, 3 H, Ac-(C2')). 
\(^13\)C NMR (151 MHz, D\(_2\)O) \(\delta 173.5\) (3'-OAc), 173.0 (2'-OAc), 155.8 (C6), 153.0 (C2), 149.4 (C4), 140.5 (C8), 119.0 (C5), 85.8 (C1'), 82.9 (d, C4'), 75.0 (C2'), 72.3 (C3'), 64.8 (d, C5'), 20.7, 20.4 (3'-OAc and 2'-OAc). 
\(^31\)P NMR (162 MHz, D\(_2\)O, \(^1\)H-decoupled) \(\delta 0.70\). IR (neat, cm\(^{-1}\)) 1747, 1688, 1431, 1377, 1242, 1072. HRMS (ESI) (m/z): [M+H\(^+\)] calcd for formula C\(_{14}\)H\(_{19}\)N\(_3\)O\(_6\)P, 432.0915; found, 432.0916. 

The spectroscopic properties of this compound were consistent with the data reported in the literature.\(^4\)

2',3'-di-O-acetyl β-guanosine-5'-phosphate (5d)

Starting from 2d (disodium salt; 204 mg, 0.50 mmol), yield = 157 mg (70%) as a white solid. 
\(^1\)H NMR (600 MHz, D\(_2\)O) \(\delta 8.11\) (s, 1 H, H-(C8)), 6.15 (d, \(J = 6.1\) Hz, 1 H, H-(C1')), 5.80 (dd, \(J = 6.1, 5.5\) Hz, 1 H, H-(C2')), 5.62 (dd, \(J = 5.5, 3.7\) Hz, 1 H, H-(C3')), 4.51 - 4.57 (m, 1 H, H-(C4')), 4.10 - 4.18 (m, 2 H, H-(C5')), 2.18 (s, 3 H, Ac-(C3')), 2.06 (s, 3 H, Ac-(C2')). 
\(^13\)C NMR (151 MHz, D\(_2\)O) \(\delta 174.6\) (3'-OAc), 173.1 (2'-OAc), 159.48 (C6), 154.8 (C2), 152.2 (C4), 138.4 (C8), 116.4 (C5), 86.0 (C1'), 82.6 (d, C4'), 74.2 (C2'), 72.1 (C3'), 64.9 (d, C5'), 20.8 (3'-OAc), 20.5 (2'-OAc). 
\(^31\)P NMR (162 MHz, D\(_2\)O, \(^1\)H-decoupled) \(\delta 0.52\). IR (neat, cm\(^{-1}\)) 1746, 1686, 1431, 1376, 1241, 1076. HRMS (ESI) (m/z): [M-H\(^-\)] calcd for formula C\(_{14}\)H\(_{17}\)N\(_3\)O\(_{10}\)P, 446.0719; found, 446.0716.
The spectroscopic properties of this compound were consistent with the data reported in the literature.  

2',3'-di-O-acetyl β-inosine-5'-phosphate (5e)

Starting from 2e (196 mg, 0.50 mmol), yield = 151 mg (71%) as a white solid.  
\[
[\alpha]^20_D = +8.78 \text{ (c 0.5, H}_2\text{O)}; ^1\text{H NMR (600 MHz, D}_2\text{O) \delta 8.44 (s, 1 H, H-(C8)), 8.17 (s, 1 H, H-(C2)), 6.35 (d, J = 6.1 Hz, 1 H, H-(C1')), 5.78 (dd, J = 6.1, 5.5 Hz, 1 H, H-(C2')), 5.64 (dd, J = 5.5, 3.5 Hz, 1 H, H-(C3')), 4.59 (dd, J = 5.5, 3.5, 2.0 Hz, 1 H, H-(C4')), 4.16 (ABX, J = 11.9, 4.8, 2.0 Hz, 1 H, H-(C5')), 4.12 (ABX, J = 11.9, 5.5, 3.1 Hz, 1 H, H-(C5'')), 2.18 (s, 3 H, Ac-(C3')), 2.05 (s, 3 H, Ac-(C2')). ^13\text{C NMR (151 MHz, D}_2\text{O) \delta 173.5 (3'-OAc), 173.1 (2'-OAc), 159.2 (C6), 149.4 (C4), 147.0 (C2), 140.5 (C8), 124.4 (C5), 86.3 (C1'), 83.0 (d, C4'), 75.0 (C2'), 72.2 (C3'), 64.8 (d, C5'), 20.8, 20.4 (3'-OAc and 2'-OAc). ^31\text{P NMR (162 MHz, D}_2\text{O, }^1\text{H-decoupled) \delta 0.88. IR (neat, cm}^{-1}\text{) 1748, 1690, 1428, 1377, 1241, 1072. HRMS (ESI) (m/z): [M+H'] calcd for C}_{14}H_{16}N_3O_{10}P, 431.0610; found, 431.0608.\]

2',3'-di-O-acetyl β-adenosine-5'-triphosphate (5f)

Starting from 2f (disodium salt; 276 mg, 0.50 mmol), yield = 215 mg (73%) as a white solid.  
\[
^1\text{H NMR (600 MHz, D}_2\text{O) \delta 8.51 (s, 1 H, H-(C8)), 8.21 (s, 1 H, H-(C2)), 6.35 (d, J = 6.1 Hz, 1 H, H-(C1')), 5.75 (dd, J = 6.1, 5.4 Hz, 1 H, H-(C2')), 5.63 (dd, J = 5.4, 3.6 Hz, 1 H, H-(C3')), 4.61 - 4.65 (m, 1 H, H-(C4')), 4.25 - 4.35 (m, 2 H, H-(C5')), 2.20 (s, 3 H, Ac-(C3')), 2.03 (s, 3 H, Ac-(C2')). ^13\text{C NMR (151 MHz, D}_2\text{O) \delta 173.5 (3'-OAc) 173.0 (2'-OAc), 156.4 (C6), 153.6 (C2), 149.6 (C4), 140.6 (C8), 119.3 (C5), 85.9 (C1'), 82.6 (d, C4'), 75.0 (C2'), 72.2 (C3'), 65.8 (d, C5'), 20.8 (3'-OAc), 20.4 (2'-OAc). ^31\text{P NMR (162 MHz, D}_2\text{O, }^1\text{H-decoupled) \delta -9.25 (d, J = 17.6 Hz), -11.13 (d, J = 19.6 Hz), -22.48 (t, J = 19.6 Hz). IR (neat, cm}^{-1}\text{) 1745, 1650, 1429, 1374, 1232, 1075, 912. HRMS (ESI) (m/z): [M+H'] calcd for formula C}_{14}H_{21}N_3O_{15}P_3, 592.0242; found, 592.0250.\]
The spectroscopic properties of this compound were consistent with the data reported in the literature.5

2',3'-di-O-acetyl-β-cytidine-5'-triphosphate (5g)

Starting from 2g (disodium salt; 264 mg, 0.50 mmol), yield = 215 mg (76%) as a white solid. [α]20 D = +38.6 (c 1.0, H2O);1H NMR (600 MHz, D2O) δ 8.02 (d, J = 7.7 Hz, 1 H, H-(C6)), 6.22 (d, J = 7.7 Hz, 1 H, H-(C5)), 6.18 (d, J = 5.0 Hz, 1 H, H-(C1')), 5.40 - 5.48 (m, 2 H, H-(C2') and H-(C3')), 4.53 (dd, J = 5.5, 5.1, 3.0 Hz, 1 H, H-(C4')), 4.31 (ABX, J = 11.9, 4.9, 2.3 Hz, 1 H, H-(C5')), 4.21 (ABX, J = 11.9, 5.1, 3.0 Hz, 1 H, H-(C5')), 2.15 (s, 3 H, Ac-(C3')), 2.10 (s, 3 H, Ac-(C2')). 13C NMR (151 MHz, D2O) δ 173.5 (3'-OAc), 173.2 (2'-OAc), 142.9 (C6), 97.4 (C5), 87.9 (C1'), 82.2 (d, C4'), 74.6 (C2'), 71.8 (C3'), 65.5 (d, C5'), 20.7 (3'-OAc), 20.6 (2'-OAc). 31P NMR (162 MHz, D2O, 1H-decoupled) δ -9.21 (d, J=19.6 Hz), -11.15 (d, J=19.6 Hz), -22.46 (t, J=19.6 Hz). IR (neat, cm-1) 1739, 1652, 1430, 1376, 1227, 1074, 906. HRMS (ESI) (m/z): [M+H+] calcd for formula C13H23N3O16P3, 568.0129; found, 568.0134.

2',3'-di-O-acetyl-β-uridine-5'-triphosphate (5h)

Starting from 2h (trisodium salt; 275 mg, 0.50 mmol), yield = 285 mg (90%, as the imidazolium salt) as a white solid.

1H NMR (600 MHz, D2O) δ 8.50 (s, 1 H, Imid), 7.87 (d, J = 8.1 Hz, 1 H, H-(C6)), 7.36 (d, J = 1.0 Hz, 2 H, Imid), 6.11 (d, J = 5.4 Hz, 1 H, H-(C1')), 5.92 (d, J = 8.1 Hz, 1 H, H-(C5)), 5.38 - 5.48 (m, 2 H, H-(C2') and H-(C3')), 4.47 (dd, J = 5.5, 4.5, 2.6 Hz, 1 H, H-(C4')), 4.25 (ABX, J = 11.8, 5.2, 2.6 Hz, 1 H, H-(C5')), 4.18 (ABX, J = 11.8, 5.5, 3.3 Hz, 1 H, H-(C5''))), 2.12 (s, 3 H, Ac-(C3')), 2.06 (s, 3 H, Ac-(C2')). 13C NMR (151 MHz, D2O) δ 173.5 (3'-OAc), 173.2 (2'-OAc), 166.8 (C4), 152.3 (C2'), 142. 4 (C6), 134.5 (CH, Imid), 120.0 (CH=CH, Imid), 103.7 (C5), 87.5 (C1'), 82.1 (d, C4'), 74.1 (C2'), 71.7 (C3'), 65.6 (d, C5'), 20.7 (3'-OAc), 20.5 (2'-OAc). 31P NMR (162 MHz, D2O, 1H-decoupled) δ -7.79 (d, J = 19.6 Hz), -
11.09 (d, $J = 19.6$ Hz), -22.08 (t, $J = 19.6$ Hz). IR (neat, cm$^{-1}$) 1745, 1688, 1460, 1380, 1240, 1077. HRMS (ESI) ($m/z$): [M-H$^{-}$] calcd for formula C$_{13}$H$_{18}$N$_2$O$_7$P$_3$, 566.9824; found, 566.9818.

The spectroscopic properties of this compound were consistent with the data reported in the literature.$^6$

2',3',5'-tri-O-acetyl-$\beta$-cytidine (5i)

Starting from 2i (122 mg, 0.50 mmol), yield = 143 mg (77%) as a white powder.

$^1$H NMR (600 MHz, D$_2$O) δ 7.59 (d, $J = 7.5$ Hz, 1 H, H-(C6)), 5.97 (d, $J = 7.5$ Hz, 1 H, H-(C5)), 5.93 (d, $J = 4.5$ Hz, 1 H, H-(C1')), 5.43 (dd, $J = 5.7$, 4.5 Hz, 1 H, H-(C2')), 5.35 (dd, $J = 5.9$, 5.7 Hz, 1 H, H-(C3')), 4.41 - 4.47 (m, 1 H, H-(C4')), 4.35 (dd, $J = 12.5$, 2.9 Hz, 1 H, H-(C5')), 4.30 (dd, $J = 12.5$, 4.4 Hz, 1 H, H-(C5'')), 2.08 (s, 3 H, Ac-(C3')), 2.07 (s, 3 H, Ac-(C5')), 2.06 (s, 3 H, Ac-(C2')). $^{13}$C NMR (151 MHz, D$_2$O) δ 174.3 (OAc), 173.3 (OAc), 173.2 (OAc), 166.9 (C4), 157.7 (C2), 142.6 (C6), 97.1 (C5), 90.0 (C1'), 79.9 (C4'), 74.2 (C2'), 70.9 (C3'), 63.8 (C5'), 20.8 (OAc), 20.6 (OAc), 20.5 (OAc). IR (neat, cm$^{-1}$) 1741, 1644, 1520, 1488, 1372, 1217, 1094, 1047. HRMS (ESI) ($m/z$): [M-H$^{-}$] calcd for formula C$_{15}$H$_{18}$N$_3$O$_6$, 368.1099; found, 368.1092.

The spectroscopic properties of this compound were consistent with the data reported in the literature.$^7$

2',3',5'-tri-O-acetyl-$\beta$-uridine (5j)

Starting from 2j (122 mg, 0.50 mmol), yield = 137 mg (74%) as a white powder.

$^1$H NMR (600 MHz, D$_2$O) δ 7.69 (d, $J = 8.1$ Hz, 1 H, H-(C6)), 6.00 (d, $J = 4.5$ Hz, 1 H, H-(C1')), 5.89 (d, $J = 8.1$ Hz, 1 H, H-(C5)), 5.54 (dd, $J = 5.9$, 4.5 Hz, 1 H, H-(C2')), 5.44 (dd, $J = 5.9$, 5.8 Hz, 1 H, H-(C3')), 4.51 (ddd, $J = 5.8$, 4.3, 2.8 Hz, 1 H, H-(C4')), 4.42 (dd, $J = 12.3$, 2.8 Hz, 1 H, H-(C5'')), 4.36 (dd, $J = 12.3$, 4.3 Hz, 1 H, H-(C5''')), 2.14 (s, 3 H, Ac-(C5'')), 2.11 - 2.13 (m, 6 H, Ac-(C2') and Ac-(C3')). $^{13}$C NMR (151 MHz, D$_2$O) δ 174.4 (5'-OAc), 173.4
(3′-OAc), 173.3 (2′-OAc), 166.9 (C4), 152.0 (C2), 143.1 (C6), 103.3 (C5), 89.8 (C1′), 80.2 (C4′), 73.8 (C2′), 70.7 (C3′), 63.7 (C5′), 20.9 (OAc), 20.6 (OAc), 20.5 (OAc). IR (neat, cm⁻¹) 1745, 1694, 1457, 1377, 1228, 1095, 1049. HRMS (ESI) (m/z): [M-H]⁻ calcd for formula C₁₆H₁₇N₇O₉, 369.0940; found, 369.0930.

The spectroscopic properties of this compound were consistent with the data reported in the literature.⁸

2′,3′,5′-tri-O-acetyl-β-adenosine (5k)

Starting from 2k (134 mg, 0.50 mmol), yield = 145 mg (74%) as a white powder.

¹H NMR (600 MHz, D₂O) δ 8.29 (s, 1 H, H-(C8)), 8.23 (s, 1 H, H-(C2)), 6.32 (d, J = 5.1 Hz, 1 H, H-(C1′)), 5.91 (dd, J = 5.3, 5.1 Hz, 1 H, H-(C2′)), 5.67 (t, J = 5.3 Hz, 1 H, H-(C3′)), 4.61 - 4.65 (m, 1 H, H-(C4′)), 4.45 (dd, J = 12.8, 3.0 Hz, 1 H, H-(C5′)), 4.40 (dd, J = 12.8, 4.0 Hz, 1 H, H-(C5″)), 2.17 (s, 3 H, Ac-(C3′)), 2.09 (s, 3 H, Ac-(C3″)), 2.06 (s, 3 H, Ac-(C5′)), 13C NMR (151 MHz, D₂O) δ 174.3 (5′-OAc), 173.5 (2′-OAc), 173.1 (3′-OAc) 153.8 (C2), 149.6 (C4), 140.8 (C8), 86.7 (C1′), 80.8 (C4′), 74.2 (C2′), 71.2 (C3′), 63.7 (C5′), 20.8 (OAc), 20.7 (OAc), 20.5 (OAc). IR (neat, cm⁻¹) 1745, 1641, 1597, 1475, 1426, 1372, 1227, 1095, 1047. HRMS (ESI) (m/z): [M+H⁺] calcd for formula C₁₆H₂₀N₇O₇, 394.1357; found, 394.1358.

The spectroscopic properties of this compound were consistent with the data reported in the literature.⁷

2′,3′,5′-tri-O-acetyl-β-guanosine (5l)

Starting from 2l (134 mg, 0.50 mmol), yield = 109 mg (53%) as a white powder.

¹H NMR (600 MHz, CDCl₃) δ 7.91 (s, 1 H, H-(C8)), 6.13 (d, J = 4.5 Hz, 1 H, H-(C1′)), 5.89 (dd, J = 5.6, 4.5 Hz, 1 H, H-(C2′)), 5.70 (dd, J = 5.8, 5.6 Hz, 1 H, H-(C3′)), 4.52 - 4.58 (m, 1 H, H-(C4′)), 4.43 (dd, J = 12.6, 3.0 Hz, 1 H, H-(C5′)), 4.37 (dd, J = 12.6, 4.4 Hz, 1 H, H-(C5″)), 2.16 (s, 3 H, Ac-(C3′)), 2.12 (s, 3 H, Ac-(C3″)), 2.04 (s, 3 H, Ac-(C5′)), 13C NMR
(151 MHz, CDCl₃) δ 174.4 (5'-OAc), 173.5 (3'-OAc), 173.2 (2'-OAc), 159.7 (C6), 154.6 (C2), 152.2 (C4), 138.6 (C8), 117.2 (C5), 86.9 (C1'), 80.3 (C4'), 74.0 (C2'), 71.0 (C3'), 63.5 (C5'), 20.8 (OAc), 20.6 (OAc), 20.5 (OAc). IR (neat, cm⁻¹) 1747, 1693, 1604, 1535, 1374, 1232, 1049. HRMS (ESI) (m/z): [M+H⁺] calcd for formula C₁₆H₂₀N₅O₈, 410.1306; found, 410.1315.

The spectroscopic properties of this compound were consistent with the data reported in the literature.⁹

2',3',5'-tri-O-acetyl-β-inosine (5m)

Starting from 2m (140 mg, 0.50 mmol), yield = 137 mg (70%) as a white powder.

¹H NMR (600 MHz, CDCl₃) δ 8.21 (s, 1 H, H-(C2)), 8.04 (s, 1 H, H-(C8)), 6.17 (d, J = 5.3 Hz, 1 H, H-(C1')), 5.88 (dd, J = 5.3, 5.2 Hz, 1 H, H-(C2')), 5.61 (dd, J = 5.2, 4.9 Hz, 1 H, H-(C3')), 4.42 - 4.50 (m, 2 H, H-(C4') and H-(C5')), 4.38 (dd, J = 12.3, 4.5 Hz, 1 H, H-(C5'')), 2.16 (s, 3 H, H-(C5')), 2.15 (s, 3 H, H-(C3')), 2.11 (s, 3 H, H-(C2')). ¹³C NMR (151 MHz, CDCl₃) δ 170.5 (5'-OAc), 169.7 (3'-OAc), 169.4 (2'-OAc), 159.0 (C6), 148.8 (C4), 145.5 (C2), 138.7 (C8), 125.5 (C5), 86.7 (C1'), 80.5 (C4'), 73.5 (C2'), 70.6 (C3'), 63.1 (C5'), 20.9 (OAc), 20.7 (OAc), 20.5 (OAc). ¹H NMR (600 MHz, D₂O) δ 8.27 (s, 1 H, H-(C8)), 8.19 (s, 1 H, H-(C2)), 6.34 (d, J = 4.8 Hz, 1 H, H-(C1')), 5.92 (dd, J = 5.3, 4.8 Hz, 1 H, H-(C2')), 5.69 (dd, J = 5.3, 5.2 Hz, 1 H, H-(C3')), 4.59 - 4.67 (m, 1 H, H-(C4') partially obscure by HOD signal), 4.46 (dd, J = 12.5, 2.9 Hz, 1 H, H-(C5')), 4.41 (dd, J = 12.5, 4.0 Hz, 1 H, H-(C5'')), 2.17 (s, 3 H, H-(C3')), 2.11 (s, 3 H, H-(C2')), 2.07 (s, 3 H, H-(C5')). ¹³C NMR (151 MHz, D₂O) δ 174.4 (5'-OAc), 173.5 (3'-OAc), 173.2 (2'-OAc), 159.4 (C6), 149.3 (C4), 147.0 (C2), 140.9 (C8), 124.9 (C5), 87.2 (C1'), 80.8 (C4'), 74.3 (C2'), 71.2 (C3'), 63.7 (C5'), 20.8 (2'-OAc), 20.6 (3'-OAc), 20.5 (5'-OAc). IR (neat, cm⁻¹) 1745, 1697, 1587, 1549, 1513, 1421, 1374, 1227, 1095, 1051. HRMS (ESI) (m/z): [M-H⁻] calcd for formula C₁₆H₁₇N₄O₈, 393.1052; found, 393.1042.

The spectroscopic properties of this compound were consistent with the data reported in the literature.¹⁰
2',3',5'-tri-O-acetyl-β-xanthosine (5n)

Starting from 2n (dehydrate, 160 mg, 0.50 mmol), yield = 129 mg (63%) as a white powder. 

$^1$H NMR (600 MHz, D$_2$O) δ 7.96 (s, 1 H, H-(C8)), 6.14 (d, $J = 4.7$ Hz, 1 H, H-(C1'))), 5.67 (dd, $J = 5.2$, 4.7 Hz, 1 H, H-(C2')), 5.50 (dd, $J = 5.2$, 5.1 Hz, 1 H, H-(C3')), 4.59 - 4.68 (m, 1 H, H-(C4') partially obscure by HOD signal), 4.33 - 4.44 (m, 2 H, H-(C5')), 2.15 (s, 3 H, Ac-(C3')), 2.14 (s, 3 H, Ac-(C2')), 2.06 (s, 3 H, Ac-(C5')). 

$^{13}$C NMR (151 MHz, D$_2$O) δ 174.1 (5'-OAc), 173.3 (3'-OAc), 173.1 (2'-OAc), 160.4 (C6), 153.0 (C2), 141.7 (C4), 136.0 (C8), 116.5 (C5), 87.2 (C1'), 81.3 (C4'), 74.7 (C2'), 71.0 (C3'), 63.6 (C5'), 20.8 (OAc), 20.6 (OAc), 20.5 (OAc). IR (neat, cm$^{-1}$) 1744, 1710, 1608, 1570, 1431, 1373, 1227, 1050. HRMS (ESI) (m/z): [M-H]$^-$ calcd for formula C$_{16}$H$_{17}$N$_4$O$_9$, 409.1001; found, 409.0990.

The spectroscopic properties of this compound were consistent with the data reported in the literature.$^{11}$

2',3'-di-O-acetyl β-araadenosine-5'-phosphate (5o)

Starting from 2o (170 mg, 0.50 mmol), yield = 191 mg (91%) as a white solid. 

[$\alpha$]$^D_{20}$ = +21.7 (c 0.5, H$_2$O);$^1$H NMR (600 MHz, D$_2$O) δ 8.36 (s, 1 H, H-(C8)), 8.12 (s, 1 H, H-(C2)), 6.53 (d, $J = 4.8$ Hz, 1 H, H-(C1')), 5.53 (dd, $J = 4.8$, 3.2 Hz, 1 H, H-(C2')), 5.44 (dd, $J = 4.7$, 3.2 Hz, 1 H, H-(C3')), 4.38 - 4.45 (m, 1 H, H-(C4')), 4.16 - 4.21 (m, 1 H, H-(C5')), 4.10 - 4.15 (m, 1 H, H-(C5'')), 2.11 (s, 3 H, Ac-(C3')), 1.74 (s, 3 H, Ac-(C2')). $^{13}$C NMR (151 MHz, D$_2$O) δ 173.5 (3'-OAc), 172.3 (2'-OAc), 155.9 (C6), 153.1 (C2), 148.9 (C4), 141.6 (C8), 118.6 (C5), 84.0 (C1'), 81.1 (d, C4'), 76.0 (C3'), 75.9 (C2'), 64.4 (d, C5'), 20.9 (3'-OAc), 20.1 (2'-OAc). $^{31}$P NMR (162 MHz, D$_2$O, $^1$H-decoupled) δ 0.68. IR (neat, cm$^{-1}$) 1744, 1603, 1604, 1426, 1374, 1232, 1053. HRMS (ESI) (m/z): [M-H]$^-$ calcd for formula C$_{14}$H$_{17}$N$_3$O$_9$P, 430.0769; found, 430.0764.
2',3'-di-O-acetyl-β-aracytidine-5'-phosphate (5p)

Starting from 2p (120 mg, 0.50 mmol), yield = 99 mg (66%) as a white solid.

\[ \alpha \] \text{D}^{20} = +82.8 (c 0.5, H2O); \text{H} NMR (600 MHz, D2O) \delta 7.83 (d, J = 7.6 Hz, 1 H, H-(C6)), 6.28 (d, J = 4.4 Hz, 1 H, H-(C1')), 5.99 (d, J = 7.6 Hz, 1 H, H-(C5)), 5.42 (dd, J = 4.4, 2.2 Hz, 1 H, H-(C2')), 5.21 (dd, J = 3.5, 2.2 Hz, 1 H, H-(C3')), 4.30 - 4.36 (m, 1 H, H-(C4')), 4.07 - 4.13 (m, 1 H, H-(C5')), 4.00 - 4.07 (m, 1 H, H-(C5'')), 2.08 (s, 3 H, Ac-(C3')), 1.92 (s, 3 H, Ac-(C2')).

\[ \delta \text{C} \] NMR (151 MHz, D2O) \delta 173.5 (3′-OAc), 172.4 (2′-OAc), 166.5 (C4), 157.2 (C2), 143.1 (C6), 96.2 (C5), 85.7 (C1'), 81.5 (d, C4'), 76.7 (C3'), 75.3 (C2'), 64.4 (d, C5'), 20.9 (3′-OAc), 20.4 (2′-OAc). 31P NMR (162 MHz, D2O, 1H-decoupled) \delta 0.68. IR (neat, cm⁻¹) 1745, 1650, 1485, 1429, 1373, 1233, 1054. HRMS (ESI) (m/z): [M+H⁺] calcd for formula C13H19N3O10P, 408.0803; found, 408.0810.

The spectroscopic properties of this compound were consistent with the data reported in the literature.¹²

2',3',5'-tri-O-acetyl-β-arauridine (5q)

Starting from 2q (122 mg, 0.50 mmol), yield = 144 mg (78%) as a white powder.

\text{H} NMR (600 MHz, D2O) \delta 7.74 (d, J = 8.1 Hz, 1 H, H-(C6)), 6.33 (d, J = 4.3 Hz, 1 H, H-(C1')), 5.87 (d, J = 8.1 Hz, 1 H, H-(C5)), 5.49 (dd, J = 4.3, 2.3 Hz, 1 H, H-(C2')), 5.29 (dd, J = 3.4, 2.3 Hz, 1 H, H-(C3')), 4.45 - 4.50 (m, 1 H, H-(C5')), 4.39 - 4.45 (m, 2 H, H-(C4') and H-(C5'')), 2.14 (s, 6 H, Ac-(C3') and Ac-(C5')). 13C NMR (151 MHz, D2O) \delta 174.5 (5′-OAc), 173.4 (3′-OAc), 172.6 (2′-OAc), 166.6 (C4), 151.7 (C2), 142.8 (C6), 102.3 (C5), 85.2 (C1'), 80.5 (C4'), 76.7 (C3'), 75.5 (C2'), 63.9 (C5'), 21.0 (OAc), 20.8 (OAc), 20.5 (OAc). IR (neat, cm⁻¹) 1745, 1693, 1454, 1372, 1283, 1223, 1047. HRMS (ESI) (m/z): [M+H⁺] calcd for formula C13H19N3O6, 371.1085; found, 371.1093.

The spectroscopic properties of this compound were consistent with the data reported in the literature.¹²
2',3',5'-tri-O-acetyl-β-araadenosine (5r)

![Chemical structure of 2',3',5'-tri-O-acetyl-β-araadenosine (5r)](image)

Starting from 2r (134 mg, 0.50 mmol), yield = 93 mg (47%) as a white powder.

\(^1\)H NMR (600 MHz, D\(_2\)O) \(\delta\) 8.10 (s, 1 H, H-(C8)), 8.02 (s, 1 H, H-(C2)), 6.39 (d, \(J = 4.7\) Hz, 1 H, H-(C1')), 5.48 (dd, \(J = 4.7, 3.2\) Hz, 1 H, H-(C2')), 5.37 (dd, \(J = 4.4, 3.7\) Hz, 1 H, H-(C3')), 4.32 - 4.41 (m, 3 H, H-(C4') and H-(C5')), 2.09 (s, 3 H, Ac-(C3')), 2.07 (s, 3 H, Ac-(C5')), 1.72 (s, 3 H, Ac-(C2')). \(^13\)C NMR (151 MHz, D\(_2\)O) \(\delta\) 174.3 (5'-OAc), 173.4 (3'-OAc), 172.0 (2'-OAc), 156.0 (C6), 153.5 (C3), 148.8 (C4), 140.9 (C8), 118.4 (C5), 83.9 (C1'), 80.0 (C4'), 76.2 (C3'), 75.5 (C2'), 63.8 (C5'), 20.9 (OAc), 20.8 (OAc), 20.1 (2'-OAc). IR (neat, cm\(^{-1}\)) 1742, 1640, 1598, 1774, 1370, 1220, 1046. HRMS (ESI) (m/z): [M+H\(^+\)] calcd for formula C\(_{16}\)H\(_{20}\)N\(_3\)O\(_7\), 394.1357; found, 394.1360.

The spectroscopic properties of this compound were consistent with the data reported in the literature.\(^7\)

2',3',5'-tri-O-acetyl-β-arahypoxanthine (5s)

![Chemical structure of 2',3',5'-tri-O-acetyl-β-arahypoxanthine (5s)](image)

Starting from 2s (134 mg, 0.50 mmol), yield = 145 mg (74%) as a white powder.

\(^1\)H NMR (600 MHz, D\(_2\)O) \(\delta\) 8.28 (s, 1 H, H-(C8)), 8.20 (s, 1 H, H-(C2)), 6.64 (d, \(J = 4.9\) Hz, 1 H, H-(C1')), 5.65 (dd, \(J = 4.9, 3.6\) Hz, 1 H, H-(C2')), 5.57 (dd, \(J = 4.4, 3.6\) Hz, 1 H, H-(C3')), 4.44 - 4.52 (m, 3 H, H-(C4') and H-(C5')), 2.17 (s, 3 H, Ac-(C3')), 2.14 (s, 3 H, Ac-(C5')), 1.86 (s, 3 H, Ac-(C2')). \(^13\)C NMR (151 MHz, D\(_2\)O) \(\delta\) 174.5 (5'-OAc), 173.5 (3'-OAc), 172.4 (2'-OAc), 159.2 (C6), 149.1 (C4), 147.2 (C2), 141.2 (C8), 124.0 (C5), 84.1 (C1'), 80.0 (C4'), 76.1 (C3'), 75.8 (C2'), 63.9 (C5'), 21.0 (OAc), 20.9 (OAc), 20.2 (2'-OAc). IR (neat, cm\(^{-1}\)) 1745, 1702, 1588, 1549, 1512, 1372, 1222, 1044. HRMS (ESI) (m/z): [M+H\(^+\)] calcd for formula C\(_{16}\)H\(_{19}\)N\(_4\)O\(_8\), 395.1197; found, 395.1195.

The spectroscopic properties of this compound were consistent with the data reported in the literature.\(^13\)
Numbering for the assignment of NMR signals of glycerophosphocholine derivatives

![Diagram of glycerophosphocholine structure]

1,2-\textit{O}-\textit{di}-hexanoyl-\textit{sn}-glycero-3-phosphocholine (11c)

Starting from 10 (65 mg, 0.25 mmol), yield = 42 mg (36%) as a white powder.

$^1$H NMR (600 MHz, CD$_3$OD) $\delta$ 5.19 - 5.28 (m, 1 H, H-(C2)), 4.42 (ABX, $J = 12.0, 3.3$ Hz, 1 H, H-(C1)), 4.23 - 4.34 (m, 2 H, H-(C1')), 4.18 (ABX, $J = 12.0, 6.7$ Hz, 1 H, H-(C1)), 3.98 - 4.04 (m, 2 H, H-(C3)), 3.62 - 3.68 (m, 2 H, H-(C2')), 3.23 (s, 9 H, N(CH$_3$)$_3$), 2.28 - 2.39 (m, 4 H, COCH$_2$), 1.55 - 1.68 (m, 4 H, CH$_2$(CH$_2$)$_2$CH$_3$), 1.25 - 1.40 (m, 8 H, CH$_2$(CH$_2$)$_2$CH$_3$) 0.87 - 0.98 (m, 6 H, CH$_2$CH$_3$). $^{13}$C NMR (151 MHz, CD$_3$OD) $\delta$ 175.2 (COCH$_2$), 174.9 (COCH$_2$), 72.0 (C2), 67.6 (C2'), 65.0 (C3), 63.8 (C1), 60.6 (C1'), 54.9 (N(CH$_3$)$_3$), 35.2, 35.0 (COCH$_2$), 32.5, 25.8, 23.5 (COCH$_2$(CH$_2$)$_2$CH$_3$), 14.4 (CH$_2$CH$_3$). $^{31}$P NMR (162 MHz, D$_2$O, $^1$H-decoupled) $\delta$ -0.59. HRMS (ESI) ($m/z$): [M+H$^+$] calcd for formula C$_{20}$H$_{41}$NO$_8$P, 454.2564; found, 454.2571.

The spectroscopic properties of this compound were identical to commercial samples.

1,2-\textit{O}-\textit{di}-octanoyl-\textit{sn}-glycero-3-phosphocholine (11d)

Starting from 10 (130 mg, 0.50 mmol), yield = 173 mg (67%) as a white powder.

$^1$H NMR (600 MHz, CD$_3$OD) $\delta$ 5.09 - 5.16 (m, 1 H, H-(C2)), 4.32 (ABX, $J = 12.0, 3.3$ Hz, 1 H, H-(C1)), 4.12 - 4.22 (m, 2 H, H-(C1')), 4.07 (ABX, $J = 12.0, 6.8$ Hz, 1 H, H-(C1)), 3.86 - 3.93 (m, 2 H, H-(C3)), 3.49 - 3.57 (m, 2 H, H-(C2')), 3.12 (s, 9 H, N(CH$_3$)$_3$), 2.18 - 2.27 (m, 4 H, COCH$_2$), 1.44 - 1.56 (m, 4 H, CH$_2$), 1.15 - 1.28 (m, 16 H, (CH$_2$)$_4$), 0.76 - 0.84 (m, 6 H, CH$_2$CH$_3$). $^{13}$C NMR (151 MHz, CD$_3$OD) $\delta$ 175.1 (COCH$_2$), 174.8 (COCH$_2$), 72.0 (C2), 67.6
(C2′), 65.0 (C3), 63.8 (C1), 60.6 (C1′), 54.8 (N(CH3)3), 35.2, 35.0 (COCH2), 33.0, 30.3, 30.2, 26.2, 26.1, 23.8 ((CH2)6), 14.57 (CH2CH3). 31P NMR (162 MHz, D2O, 1H-decoupled) δ -0.57. HRMS (ESI) (m/z): [M+H+] calcd for formula C28H57NO8P, 566.3816; found 566.3835.

The spectroscopic properties of this compound were identical to commercial samples.

1.2-O-di-decanoyl-sn-glycero-3-phosphocholine (11e)

Starting from 10 (130 mg, 0.50 mmol), yield = 63 mg (22%) as a white powder.

1H NMR (600 MHz, CD3OD) δ 5.21 - 5.28 (m, 1 H, H-(C2)), 4.43 (ABX, J = 12.0, 3.2 Hz, 1 H, H-(C1)), 4.23 - 4.32 (m, 2 H, H-(C1′)), 4.18 (ABX, J = 12.0, 6.9 Hz, 1 H, H-(C1)), 3.97 - 4.05 (m, 2 H, H-(C3)), 3.61 - 3.68 (m, 2 H, H-(C2′)), 3.23 (s, 9 H, N(CH3)3), 2.28 - 2.37 (m, 4 H, COCH2), 1.55 - 1.67 (m, 4 H, CH2), 1.24 - 1.39 (m, 24 H, (CH2)6), 0.86 - 0.94 (m, 6 H, CH2CH3). 13C NMR (151 MHz, CD3OD) δ 175.2 (COCH2), 174.8 (COCH2), 72.0 (C2), 67.6 (C2′), 65.0 (C3), 63.8 (C1), 60.6 (C1′), 54.9 (N(CH3)3), 35.2, 35.1 (COCH2), 33.2, 30.8, 30.7, 30.6, 30.5, 30.4, 30.3, 26.2, 26.1, 23.9 ((CH2)7), 14.6 (CH2CH3). 31P NMR (162 MHz, D2O, 1H-decoupled) δ -0.57. HRMS (ESI) (m/z): [M+H+] calcd for formula C28H57NO8P, 566.3816; found 566.3835.

The spectroscopic properties of this compound were identical to commercial samples.
2′,3′-di-O-acetyl-β-cytidine-5′-phosphate (5a)

Figure S12: $^1$H NMR spectra (600 MHz, D$_2$O, 1.5 – 9.0 ppm) for cytidine 5a.

Figure S13: $^{13}$C NMR spectra (600 MHz, D$_2$O, 0 – 200 ppm) for cytidine 5a.
2',3'-di-O-acetyl β-uridine-5'-phosphate (5b)

Figure S14: $^1$H NMR spectra (600 MHz, D$_2$O, 1.5 – 9.0 ppm) for uridine 5b.

Figure S15: $^{13}$C NMR spectra (600 MHz, D$_2$O, 0 – 200 ppm) for uridine 5b.
2',3'-di-O-acetyl β-adenosine-5'-phosphate (5c)

Figure S16: $^1$H NMR spectra (600 MHz, D$_2$O, 1.5 – 9.0 ppm) for adenosine 5c.

Figure S17: $^{13}$C NMR spectra (600 MHz, D$_2$O, 0 – 200 ppm) for adenosine 5c.
2′,3′-di-O-acetyl β-guanosine-5′-phosphate (5d)

Figure S18: $^1$H NMR spectra (600 MHz, D$_2$O, 1.5 – 9.0 ppm) for guanosine 5d.

Figure S19: $^{13}$C NMR spectra (600 MHz, D$_2$O, 0 – 200 ppm) for guanosine 5d.
2',3'-di-O-acetyl β-inosine-5'-phosphate (5e)

Figure S20: $^1$H NMR spectra (600 MHz, D$_2$O, 1.5 – 9.0 ppm) for inosine 5e.

Figure S21: $^{13}$C NMR spectra (600 MHz, D$_2$O, 0 – 200 ppm) for inosine 5e.
2',3'-di-O-acetyl β-adenosine-5'-triphosphate (5f)

Figure S22: $^1$H NMR spectra (600 MHz, D$_2$O, 1.5 – 9.0 ppm) for adenosine 5f.

Figure S23: $^{13}$C NMR spectra (600 MHz, D$_2$O, 0 – 200 ppm) for adenosine 5f.
2′,3′-di-O-acetyl-β-cytidine-5′-triphosphate (5g)

Figure S24: $^1$H NMR spectra (600 MHz, D$_2$O, 1.5 – 9.0 ppm) for cytidine 5g.

Figure S25: $^{13}$C NMR spectra (600 MHz, D$_2$O, 0 – 200 ppm) for cytidine 5g.
2',3'-di-\(O\)-acetyl-\(\beta\)-uridine-5'-triphosphate (5h)

Figure S26: \(^1\)H NMR spectra (600 MHz, D\(_2\)O, 1.5 – 9.0 ppm) for uridine 5h. 10% mono acetylated products.

Figure S27: \(^{13}\)C NMR spectra (600 MHz, D\(_2\)O, 0 – 200 ppm) for uridine 5h.
2',3',5'-tri-O-acetyl-β-cytidine (5i)

Figure S28: $^1$H NMR spectra (600 MHz, D$_2$O, 1.5 – 9.0 ppm) for cytidine 5i.

Figure S29: $^{13}$C NMR spectra (600 MHz, D$_2$O, 0 – 200 ppm) for cytidine 5i.
2',3',5'-tri-O-acetyl-β-uridine (5j)

Figure S30: $^1$H NMR spectra (600 MHz, D$_2$O, 1.5 – 9.0 ppm) for uridine 5j.

Figure S31: $^{13}$C NMR spectra (600 MHz, D$_2$O, 0 – 200 ppm) for uridine 5j.
2',3',5'-tri-O-acetyl-β-adenosine (5k)

Figure S32: $^1$H NMR spectra (600 MHz, D$_2$O, 1.5 – 9.0 ppm) for adenosine 5k. 7% 2',3'-di-O-acetyl adenosine.

Figure S33: $^{13}$C NMR spectra (600 MHz, D$_2$O, 0 – 200 ppm) for adenosine 5k.
2',3',5'-tri-O-acetyl-β-guanosine (51)

Figure S34: $^1$H NMR spectra (600 MHz, D$_2$O, 1.5 – 9.0 ppm) for guanosine 51. 10% 2',3'-di-O-acetyl guanosine.

Figure S35: $^{13}$C NMR spectra (600 MHz, D$_2$O, 0 – 200 ppm) for guanosine 51.
2',3',5'-tri-O-acetyl-β-inosine (5m)

Figure S36: $^1$H NMR spectra (600 MHz, CDCl$_3$, 1.5 – 9.0 ppm) for inosine 5m.

Figure S37: $^{13}$C NMR spectra (600 MHz, CDCl$_3$, 0 – 200 ppm) for inosine 5m.
Figure S38. $^1$H NMR spectra (600 MHz, D$_2$O, 1.5 – 9.0 ppm) for inosine 5m.

Figure S39. $^{13}$C NMR spectra (600 MHz, D$_2$O, 0 – 200 ppm) for inosine 5m.
2',3',5'-tri-O-acetyl-β-xanthosine (5n)

Figure S40: $^1$H NMR spectra (600 MHz, D$_2$O, 1.5 – 9.0 ppm) for xanthosine 5n. 2% 2',3'-di-O-acetyl xanthosine.

Figure S41: $^{13}$C NMR spectra (600 MHz, D$_2$O, 0 – 200 ppm) for xanthosine 5n.
2',3'-di-O-acetyl β-araadenosine-5'-phosphate (5o)

Figure S42: $^1$H NMR spectra (600 MHz, D$_2$O, 1.5 – 9.0 ppm) for adenosine 5o. 10% 3'-O-acetyl adenosine 6o.

Figure S43: $^{13}$C NMR spectra (600 MHz, D$_2$O, 0 – 200 ppm) for adenosine 5o.
2',3'-di-O-acetyl-β-aracytidine-5'-phosphate (5p)

Figure S44: $^1$H NMR spectra (600 MHz, D$_2$O, 1.5 – 9.0 ppm) for cytidine 5p. 12% mono acetylated cytidines 6p and 7p.

Figure S45: $^{13}$C NMR spectra (600 MHz, D$_2$O, 0 – 200 ppm) for cytidine 5p.
2',3',5'-tri-O-acetyl-β-arauridine (5q)

Figure S46: $^1$H NMR spectra (600 MHz, D$_2$O, 1.5 – 9.0 ppm) for uridine 5q.

Figure S47: $^{13}$C NMR spectra (600 MHz, D$_2$O, 0 – 200 ppm) for uridine 5q.
2',3',5'-tri-O-acetyl-β-araadenosine (5r)

Figure S48: $^1$H NMR spectra (600 MHz, D$_2$O, 1.5 – 9.0 ppm) for adenosine 5r. 9% diacetyl adenosine.

Figure S49: $^{13}$C NMR spectra (600 MHz, D$_2$O, 0 – 200 ppm) for adenosine 5r.
2',3',5'-tri-O-acetyl-β-arahypoxanthine (5s)

Figure S50: $^1$H NMR spectra (600 MHz, D$_2$O, 1.5 – 9.0 ppm) for hypoxanthine 5s. 5% diacetyl hypoxanthine 6s.

Figure S51: $^{13}$C NMR spectra (600 MHz, D$_2$O, 0 – 200 ppm) for hypoxanthine 5s.
1,2-O-di-hexanoyl-sn-glycero-3-phosphocholine (11c)

![Structure of 1,2-O-di-hexanoyl-sn-glycero-3-phosphocholine (11c)](image)

Figure S52: $^1$H NMR spectra (600 MHz, D$_2$O, 0 – 7.0 ppm) for glycerol 11c.

![13C NMR spectra for glycerol 11c](image)

Figure S53: $^{13}$C NMR spectra (600 MHz, D$_2$O, 0 – 200 ppm) for glycerol 11c.
Figure S54: $^1$H-$^{31}$P NMR spectra (D$_2$O, 3.2 – 5.5 ppm and -8 – 8 ppm) for glycerol 11c.
1,2-\textit{O}-di-octanoyl-\textit{sn}-glycero-3-phosphocholine (11d)

Figure S55: $^1$H NMR spectra (600 MHz, D$_2$O, 0 – 7.0 ppm) for glycerol 11d.

Figure S56: $^{13}$C NMR spectra (600 MHz, D$_2$O, 0 – 200 ppm) for glycerol 11d.
Figure S57: $^1$H-$^{31}$P NMR spectra (D$_2$O, 3.2 – 5.5 ppm and -8 – 8 ppm) for glycerol 11d.
1,2-\textit{O}-di-decanoyl-\textit{sn}-glycero-3-phosphocholine (11e)

Figure S58: $^1$H NMR spectra (600 MHz, D$_2$O, 0 – 7.0 ppm) for glycerol 11e.

Figure S59: $^{13}$C NMR spectra (600 MHz, D$_2$O, 0 – 200 ppm) for glycerol 11e.
Figure S60: $^1$H-$^{31}$P NMR spectra (D$_2$O, 3.2 – 5.5 ppm and -8 – 8 ppm) for glycerol 11e.
References


