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Synthesis of O- β -D-Ribofuranosyl-(1''-2')-adenosine-5''-O-phosphate

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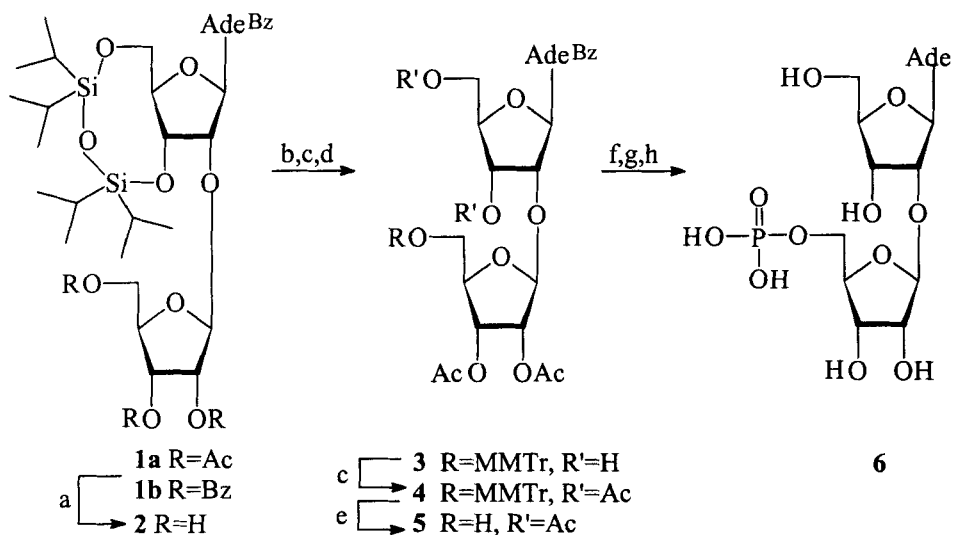
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ABSTRACT: The first synthesis of *O*- β -D-ribofuranosyl-(1''-2'')-adenosine-5''-*O*-phosphate starting from protected 2'-*O*- β -D-ribofuranosyladenosine has been performed.

Recently a minor nucleoside was isolated from yeast methionine initiator tRNA and its structure was determined as *O*-β-D-ribofuranosyl-(1''-2')-adenosine-5''-*O*-phosphate (**6**)^{1,2}. Here we report on the first synthesis of this compound starting from **1**. The synthesis was performed according to the following scheme.



a. 0.1 M NaOMe; b. MMTTrCl/Py; c. Ac₂O/Py; d. Bu₄NF/THF; e. *p*-TsOH/ CHCl₃/MeOH; f. NC(CH₂)₂OPO₃H₂/DCC/Py; g. NH₃/MeOH; h. 1M NaOH.

Fully protected 2'-*O*- β -D-ribofuranosyladenosines (**1**) were prepared by condensation of *N*⁶,3',5'-*O*-protected adenosine with slight excess of 1,2,3,5-tetra-*O*-acetyl(benzoyl)- β -D-ribofuranoses in the presence of 1.2 eq. of tin tetrachloride in dichloroethane (0°C, under nitrogen)^{3,4}. It should be mentioned that the yield of disaccharide with *O*-benzoyl groups was higher (50% and 75% for **1a** and **1b** respectively).

Treatment of **1a** with NaOMe for 10 min gave **2** in 82 % yield. The same deprotection of **1b** proceeded much more slowly and was accompanied by the formation of several products. The overall yields for these two steps using *O*-acetyl and *O*-benzoyl groups were near the same (41-42%). The 5'-hydroxyl group of additional *O*-ribofuranosyl moiety in **2** was protected with monomethoxytrityl group. The conversion of **2** \rightarrow **5** was achieved using standard methods without difficulties. The phosphorylation of **5** with subsequent deprotection gave **6**⁵ in overall good yield. The structures of **1-6** were proven by NMR spectroscopy.

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5. ¹H NMR (400.13 MHz) (D₂O) of **6**: 8.33 s (1H, H-8), 8.17 s (1H, H-2), 6.17 d (1H, J_{1',2'} = 6.3 Hz, H-1' Ado), 4.97 d (1H, J_{1',2'} = 1.0 Hz, H-1' Rib), 4.82 dd (1H, J_{2',3'} = 5.3 Hz, H-2' Ado), 4.56 dd (1H, J_{3',4'} = 3.2 Hz, H-3' Ado) 4.25 ddd (1H, J_{4',5'a} = 2.5 Hz, J_{4',5'b} = 3.6 Hz, H-4' Ado), 4.16 dd (1H, J_{3',2'} = 4.7 Hz, J_{3',4'} = 6.5 Hz, H-3' Rib), 4.13 dd (1H, H-2' Rib), 3.92 ddd (1H, J_{4',5'a} = 4.2 Hz, J_{4',5'b} = 6.3 Hz, H-4' Rib), 3.89 dd (1H, J_{5'a,5'b} = -12.9 Hz, H-5'a Ado), 3.80 dd (1H, H-5'b Ado), 3.68 ddd (1H, J_{5'a,5'b} = -11.6 Hz, J_{5'a,P} = 6.1 Hz, H-5'a Rib), 3.44 ddd (1H, J_{5'b,P} = 6.4 Hz, H-5'b Rib).
³¹P NMR (161.98 MHz) (D₂O): 1.92.