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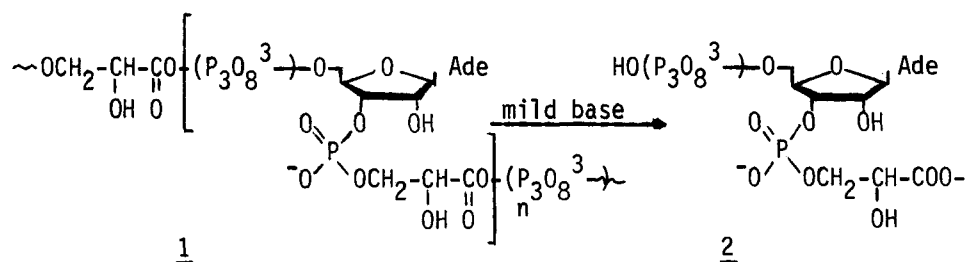
# THE CHEMICAL SYNTHESIS OF ATP-DERIVATIVE OF PHOSPHOGLYCERIC ACID

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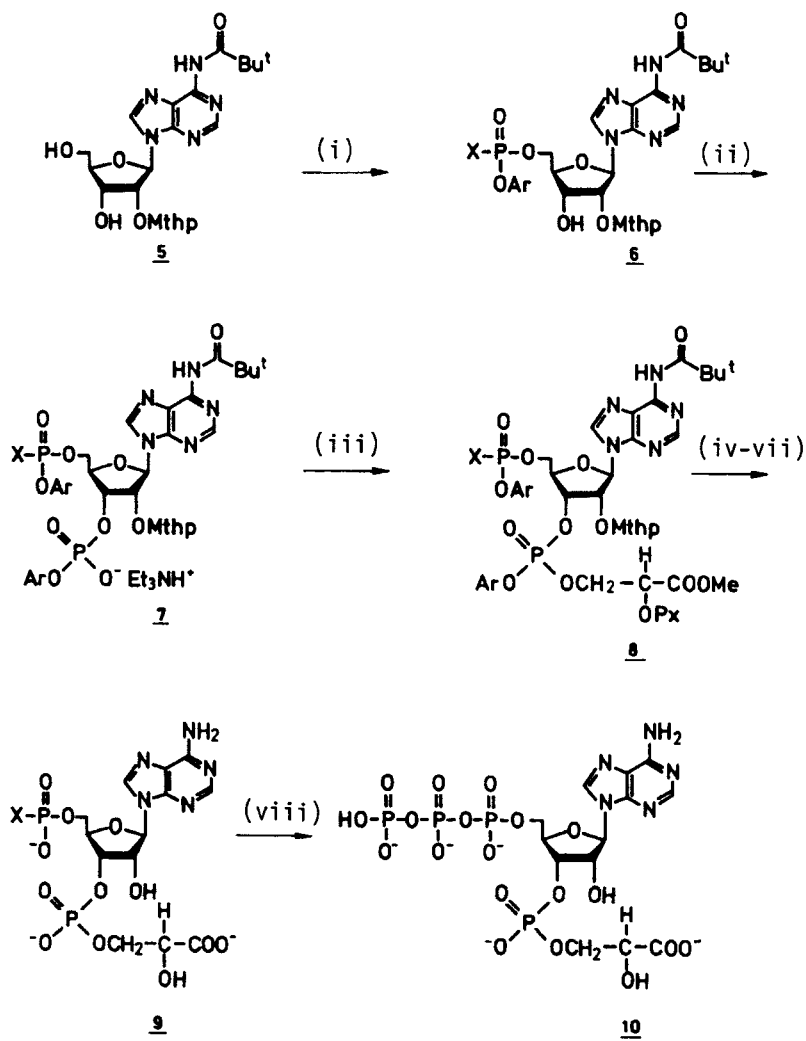
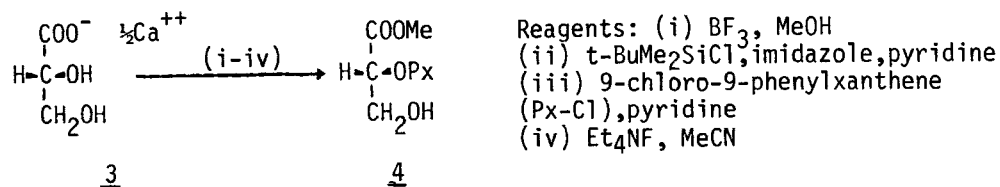
**Abstract:** A rapidly metabolized polymeric tetraphosphate derivative of adenosine was found in perfused rat heart [1]. The putative structure of this oligomeric derivative of phosphoglyceric acid and ATP (**1**) was proposed on the basis of enzymatic analysis and <sup>31</sup>P NMR data [2]. Hydrolysis of **1** under mildly basic conditions (Scheme 1) is believed to yield the corresponding monomer **2**. We therefore undertook a chemical synthesis of **2** (Scheme 2) in order to confirm its structure.

The protected D(+)-glyceric acid (**4**) was obtained from calcium D(+)-glycerate (**3**). 6-N-(trimethylacetyl)-2'-O-(4-methoxytetrahydropyran-4-yl) adenosine (**5**) was treated with 4-nitrophenyl phosphoromorpholinochloridate in pyridine leading to **6** in 88% isolated yield. The latter material was phosphorylated to give **7** in virtually quantitative yield. Reaction



Scheme 1

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X = morpholin-4-yl; Ar = 2-ClC<sub>6</sub>H<sub>4</sub>

Scheme 2

between 7, the protected D(+)-glyceric acid derivative 4 and 1-(mesitylene-2-sulphonyl)-3-nitro-1,2,4-triazole (MSNT) in pyridine solution gave the fully-protected product 8 in 85% yield. The latter material was then subjected to a three-step unblocking procedure and purified by chromatography on Sephadex A-25. 9 was allowed to react with tri-*n*-butylammonium pyrophosphate in anhydrous DMF solution. The product obtained (10) was deprotected by an analogous three-step unblocking procedure.

#### Reagents:

(i) 4-nitrophenylphosphoromorpholinochloridate, 1-methylimidazole, pyridine  
(ii) a) 2-chlorophenyl phosphoro di-(1,2,4-triazolide), 1-methylimidazole, THF b) aqueous Et<sub>3</sub>N; (iii) 4, MSNT/pyridine; (iv) E-2-nitrobenzaldehyde, N<sup>1</sup>,N<sup>1</sup>,N<sup>3</sup>,N<sup>3</sup>-tetramethylguanidine, dioxane-water (9:1) v/v; (v) aqueous NH<sub>3</sub> (d 0.88); (vi) 0.2M NaOH (dioxane/water 1:1 v/v); (vii) 0.01M-hydrochloric acid; (viii) tri-*n*-butylammonium pyrophosphate, DMF

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2. J.Mowbray *et al.*, Biochem.J. (1986) 234, 623.