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Nucleosides, Nucleotides and Nucleic Acids

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Synthesis of Nucleosides Using Trimethylsilyl Perfluoroethoxyethanesulphonate as Catalyst

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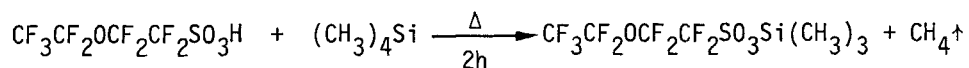
SYNTHESIS OF NUCLEOSIDES USING TRIMETHYLSILYL
PERFLUOROETHOXYETHANESULPHONATE AS CATALYST

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Abstract - New catalyst - trimethylsilyl perfluoroethoxyethanesulphonate was used for the synthesis of adenosine, guanosine, 9-β-D-xylofuranosyladenine and 1-β-D-ribofuranosyl-1,2,4-triazole-3-carboxamide by silyl method.

Since Vorbrüggen¹ introduced into practical use trimethylsilyl esters of perfluoroalkanesulphonic acids as catalysts for the synthesis of different derivatives of nucleosides, only one of them - trimethylsilyl trifluoromethanesulphonate has really become a widely used compound.

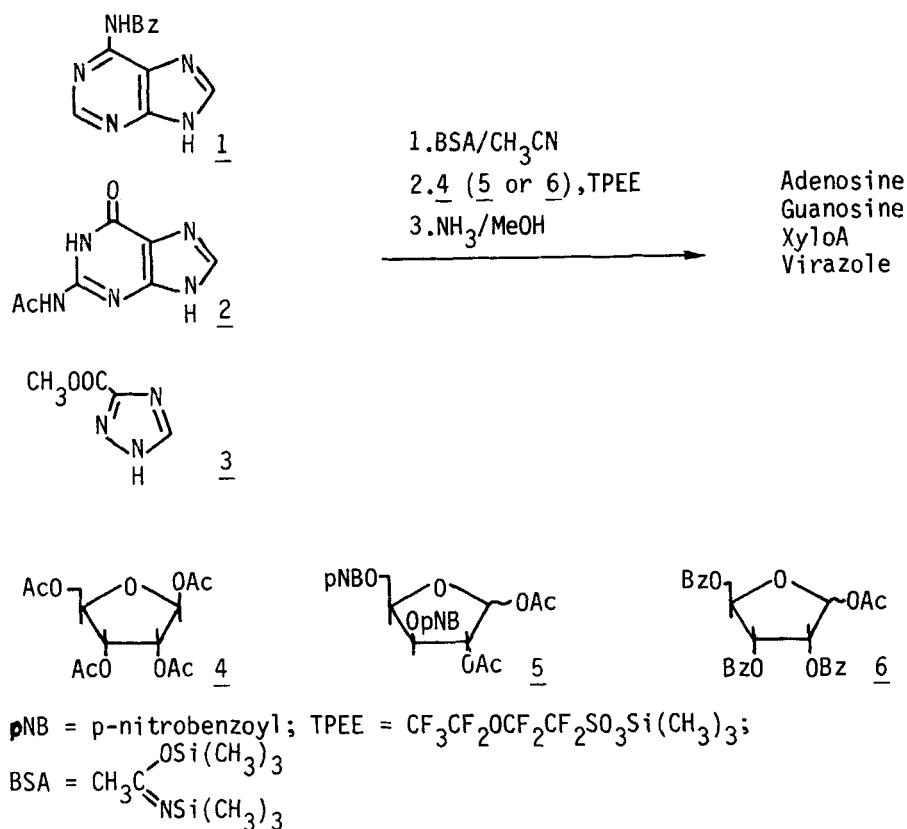
Now we wish to report another catalyst - trimethylsilyl perfluoroethoxyethanesulphonate (TPEE), readily obtainable from perfluoroethoxyethanesulphonic acid and tetramethylsilane:



We tested suitability of TPEE for the synthesis of adenosine, guanosine as well as 9-β-D-xylofuranosyladenine (XyloA) and 1-β-D-ribofuranosyl-1,2,4-triazole-3-carboxamide (Virazole) as model compounds.

We used the standard one pot procedure introduced by Dudych and Wright² consisting from following steps:

1. Silylation of heterocyclic base with N,O-bis(trimethylsilyl)-acetamide in acetonitrile.
2. Condensation of silylated heterocycle with fully acylated carbohydrates in the presence of TPEE. We found out the optimal conditions



allowing to obtain maximal yields of N₉-isomers of purine nucleosides and N₁-isomer of virazole. The molar ratio of the reagents: purine 1 or 2 - sugar 4 or 5 - TPEE 1:1:1.4 and triazole 3 - sugar 4 or 6 - TPEE 1:1:2. All the reactions were carried out in boiling acetonitrile from 20 min to 3 hours.

3. Deacylation with methanolic ammonia.

Yield of nucleosides after recrystallization was as follows: adenosine - 70%, guanosine - 60%, XyloA - 80%, virazole - 60%. It has to be mentioned that step of the condensation proceeded quantitatively (TLC) and in the case of guanosine only traces of N₇-isomer were noticed (NMR).

REFERENCES

1. H. Vorbrüggen, K. Krolikiewicz, B. Bennua Chem. Ber., 114, 1234 (1981).
2. L. W. Dudycz, G. E. Wright Nucleosides and Nucleotides, 3, 33 (1984).