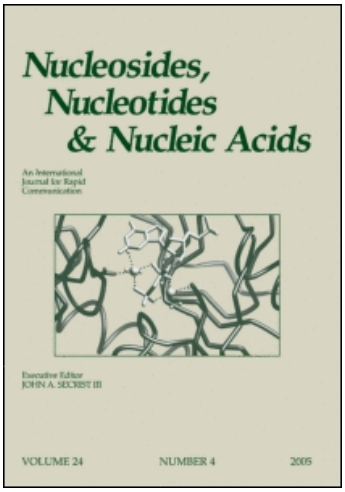

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Synthesis of Nucleosides Using Trimethylsilyl Perfluoroethoxyethanesulphonate as Catalyst

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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 Vorbrüggen[1] 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 realy became a widely used compound.

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

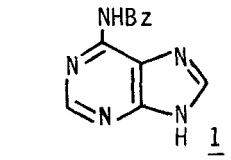
$$CF_3CF_2OCF_2CF_2SO_3H + (CH_3)_4Si \xrightarrow[2h]{\Delta} CF_3CF_2OCF_2CF_2SO_3Si(CH_3)_3 + CH_4\uparrow$$

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 Wright[2] 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



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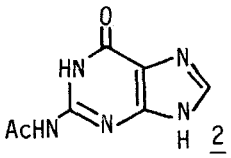

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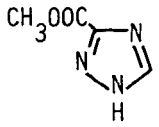

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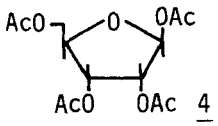
1.BSA/CH_3CN
2.4 (5 or 6),TPEE
3.NH_3/MeOH

Adenosine
Guanosine
XyloA
Virazole



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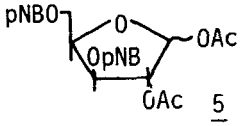

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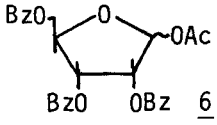

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pNB = p-nitrobenzoyl; TPEE = $CF_3CF_2OCF_2CF_2SO_3Si(CH_3)_3$;

BSA = $CH_3C(OSi(CH_3)_3)=NSi(CH_3)_3$

allowing to obtain maximal yields of N_9-isomers of purine nucleosides and N_1-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 have to be mentioned that step of the condensation proceeded quantitatively (TLC) and in the case of guanosine only traces of N_7-isomer were noticed (NMR).

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