Molecules **1998**, *3*, M74

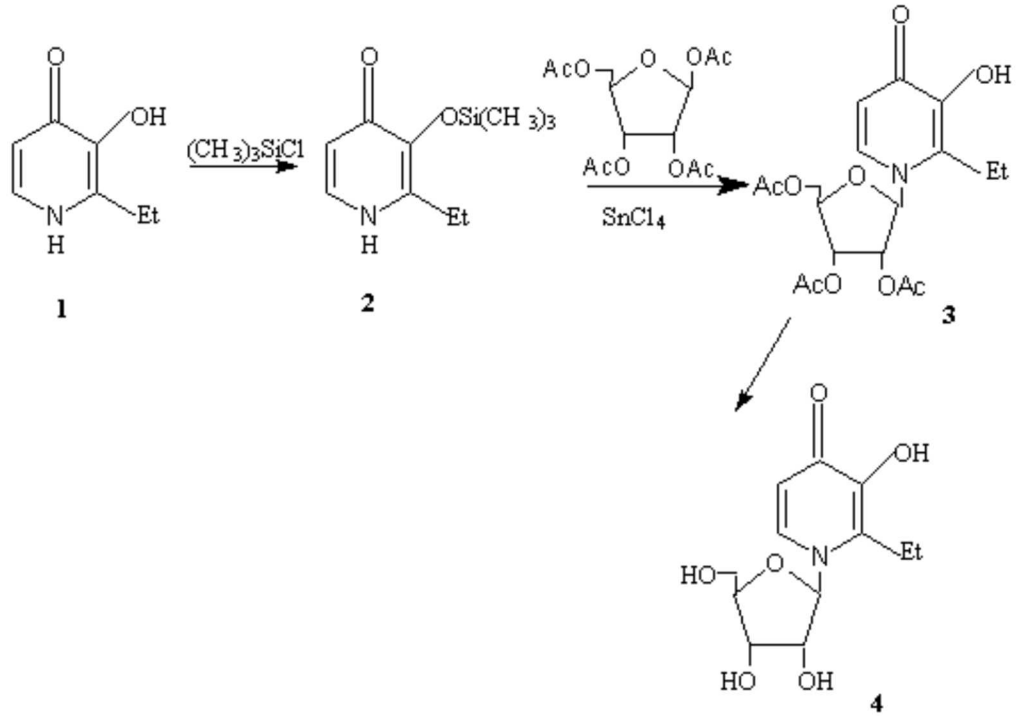
1-D-Ribofuranosyl-2-ethyl-3-hydroxy-4-pyridone

Wei-Min Chen [1] and Long-Mei Zeng [2]

[1] Institute of Pharmaceutical Sciences, The First Military Medical University, Guangzhou 510515, P. R China. Tel. & Fax +86-20-87705671 (chenwm@fimmu.edu.cn)

[2]Department of Chemistry , Zhongshan University, Guangzhou, 510275, P. R China, Tel. +86 20 84185447 (ceszlm@zsu.edu.cn)

Received: 9 March 1998 / Published: 15 April 1998



The intermediate **3** and the product **4** were prepared according to a reported procedure [1].

*1-(2,3,5-Tri-O-acetyl-D-ribofuranosyl)-3-hydroxy-2-ethyl-4-pyridone (**3**)*

2-Ethyl-3-hydroxy-4-pyridone (**1**, 5.2g, 37mmol) was suspended in 50ml of hexamethyldisilazane (HMDS), and 1 ml of chlorotrimethylsilane was added. The mixture was heated at reflux with exclusion of moisture for 2h to obtain a clear solution. Solid NH_4Cl deposited in the reflux condenser. Excess reagent was removed under vacuum, and the residue was dissolved in 250ml of anhydrous methylene chloride. To this solution, which was protected with nitrogen, were added the protected 1,2,3,5-tetra-O-acetyl-D-ribofuranose (10.85g, 34mmol) and 10g of trimethylsilyl trifluoromethanesulfonate (TMSOTf). The solution was stirred at ambient temperature for 24h. The reaction mixture was then diluted with 90ml of methylene chloride and washed twice with 90ml of ice-cold, saturated $NaHCO_3$ solution as several portions and finally once with brine. The organic layer was dried (Na_2SO_4),and the solvent was removed under reduced pressure to give a foam, **3** (11.0g, 75%). ^{1}HNMR($CDCl_3$): 1.25 (3H, t, -CH_3), 2.04 (3H, s, CH_3CO), 2.09 (3H, s, CH_3CO), 2.15 (3H, s,CH_3CO), 4.15 (2H, q, -CH_2), 4.40 (2H, m, H-5), 4.7 (1H, m, H-4), 5.40 (2H, m, H-2, 3), 5.90 (1H, d, H-1), 6.5 (1H, d, H-5), 7.7 (1H, d, H-6).

*1-D-Ribofuranosyl-2-ethyl-3-hydroxy-4-pyridone (**4**)*

Protected nucleoside (**3**,10.5g, 26.4mmol) was dissolved in 500ml of a solution of saturated ammonia in methanol and the solution was kept in a sealed container at 4 deg. for 40h. It was then evaporated to give a red foam. Recrystallization from isopropanol /H_2O gave 4.8g of **4** (67%).

^{1}HNMR ($CDCl_3$): 1.18 (3H, t,-CH_3), 3.62 (2H, q, -CH_2), 3.92 (2H, m, H-5), 4.2 (3H, m, H-2, 3, 4), 5.65 (1H, d, H-1), 6.0 (1H, d, H-5), 8.6 (1H, d, H-6).

MS (FAB): 272 (M+1, 14%), 136 (92%).

Anal. ($C_{12}H_{17}NO_6$) C, H, N.

Acknowledgment: This research was supported by the natural sciences foundation of Guangdong province.

Reference

1. Mao, D. T.; Driscoll, J. S.; Marquez. V. E. *J. Med. Chem.* **1984**, *27*, 160.

Sample Availability: Available from MDPI: **4**, 0.5g.