6-Amino-1-(2’-deoxy-b-D-ribofuranosyl)-4,5-dihydro-8H-imidazo[4,5-e][1,3]diazepine-4,8-dione

Ramesh K. Sood 2, Vishweshwar S. Bhadti 2, Huan-Ming Chen 1 and Ramachandra S. Hosmane1*

1. Laboratory for Drug Design and Synthesis, Department of Chemistry & Biochemistry, University of Maryland, Baltimore County, 1000 Hilltop Circle, Baltimore, MD 21250, USA. Tel. 410-455-2520, Fax 410-455-1148. Email: hosmane@umbc.edu
2. Nabi, 12280 Wilkins Avenue, Rockville, MD 20852, USA.

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The starting methyl 1-(2’-deoxy-b-D-ribofuranosyl)imidazole-4,5-dicarboxylate (1) was prepared by deoxygenation of the corresponding riboside, employing the literature procedure [1]. To a solution of 1 (150 mg, 0.5 mmol) and guanidine hydrochloride (240 mg, 2.5 mmol) in 30 mL of absolute methanol was added 1 mL of 25% (w/v) sodium methoxide solution in methanol. The reaction mixture was stirred for 24 h at room temperature, then 1 g of silica gel was added. The resulting mixture was evaporated to dryness and the product was isolated by silica gel flash chromatography. The column was successively eluted with chloroform/methanol (10:1) and methanol. The proper fraction was collected and evaporated to afford title product 2 as a colorless solid. Yield 110 mg (74.5%).

M.p. >250°C.

1H NMR (DMSO-d6): d 10.65 (brs, 1H, NH, exchangeable with D2O), 8.47 (s, 1H, imidazole), 7.62 (brs, 1H, NH, exchangeable with D2O), 6.65 (t, 1H, J=5.7 Hz, 1H), 6.54 (brs, 1H, NH, exchangeable with D2O), 5.27 (d, 1H, J=4.5 Hz, OH, exchangeable with D2O), 5.09 (t, 1H, J=4.8 Hz, OH, exchangeable with D2O), 4.24 (m, 1H, 4H), 3.84 (m, 1H, 3H), 3.60 (m, 2H, 5H), 2.18 (dt, 1H, J=13.5 and 5.7 Hz, 2H), 2.02 (d, 1H, J=13.5 Hz, 2H).


Anal. Calcd. For C11H13N5O5·0.25 H2O: C, 44.09; H, 4.51; N, 23.37. Found: C, 44.08; H, 4.54; N, 23.36.

Reference

Sample Availability: Available from the authors and from MDPI.