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3,3'-[Diselenobis(methylene)]bis[5-hydroxy-6-methyl-4-pyridinemethanol

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)

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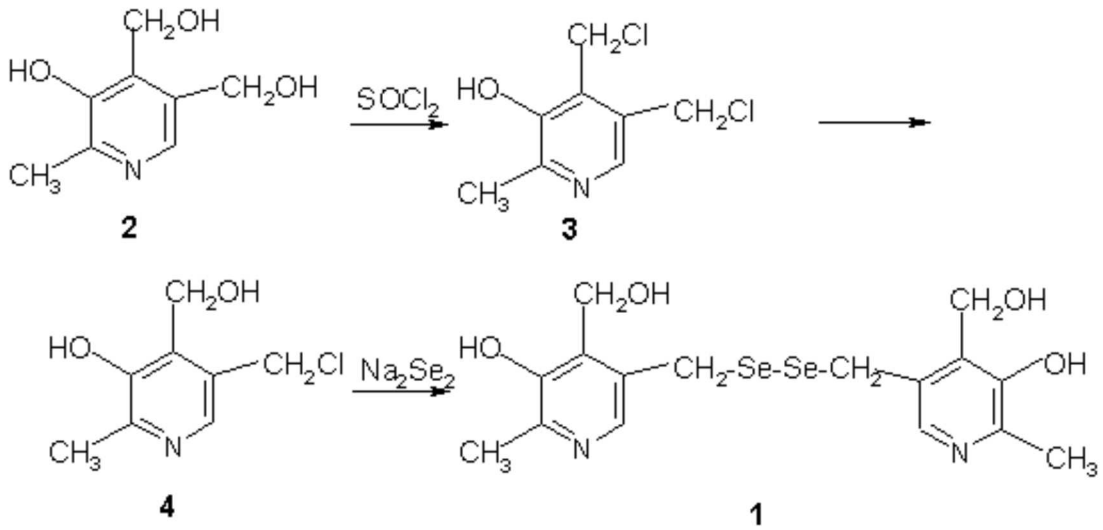

The intermediate **3** was synthesized utilizing the methodology of *Deng* [1]. A mixture of **3** (3.4g, 14mmol) in 60ml of H_2O was stirred at 60-65deg.C for 1.4h and cooled to room temperature. Then 25ml of Na_2Se_2 solution (19mmol) was added dropwise. After adjustment to pH 8, the mixture was stirred at 60deg.C for 2h and cooled to room temperature. The precipitate was filtered off and washed with water. The solid was solved in 20ml of 10% HCl, and stirred for 0.5h, filtered and washed with 10% HCl. The mother liquor was adjusted to pH 8 with 40% NaOH to give a precipitate which was filtered off and washed with water. Yellow solid 3,3'-[diselenobis(methylene)]bis[5-hydroxy-6-methyl-4-pyridine-methanol (**1**) 2.5g (77.3%). Mp:192-3deg.C.

IR(KBr) n (cm^{-1}): 3100 (-OH), 2706, 2628, 1560, 1405, 1236, 1039, 772, 716.

^{1}H-NMR (DMSO-d6) d_H (ppm): 2.32 (s, 6H, -CH_3), 3.98 (s, 4H, -CH_2Se-), 4.75 (s, 4H, -CH_2O), 7.70 (s, 2H, H-2).

MS (m/z,%): 462 (M^+, 2%) 231, 213, 152, 136, 122, 106, 94, 77, 66, 53, 39 (100%).

Anal.($C_{16}H_{20}N_2Se_2O_4$) C, H, N.

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Reference

1. Deng L. *Chinese Journal of Pharmaceuticals* **1996**, *27*(8), 342.

Sample Availability: Available from MDPI.