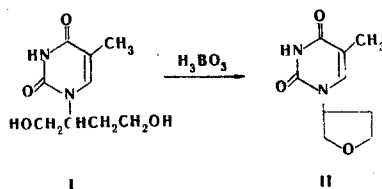


SYNTHESIS OF N₁-(3-FURANIDYL)THYMINE

S. A. Giller and Z. A. Shomshtein

UDC 547.722.3'854.4

It is known that olefins are formed by the action of boric acid on alcohols at 270–350°C [1, 2]. Proceeding from this, we proposed that N₁-(1,4-butadien-2-yl)thymine would be formed under similar conditions from N₁-(1,4-dihydroxy-2-butyl)thymine (I). In fact, we found that N₁-(3-furanidyl)thymine (II) (65% yield) is sublimed when I is heated with boric acid at 300–325°. At higher temperatures (350–450°) we obtained a mixture of several products, from which a substance with a UV spectrum characteristic for divinyl derivatives (λ_{\max} 215 nm and a shoulder at 260 nm, $D_{215}/D_{260}=1.96$) was isolated by extraction with pentane. Furanidylthymine II is stable under acid hydrolysis conditions, in contrast to N₁-(2-furanidyl)thymine, which decomposes on similar treatment.



EXPERIMENTAL

N₁-(3-Furanidyl)thymine (II). A solution of 14.05 mmole of I and 9.38 mmole of boric acid in 35 ml of dimethylformamide (DMFA) was refluxed for 4 h with slow removal of the DMFA by distillation. The residue was heated in vacuo (10 mm) at 170–180° for 45 min. The resulting solid white mass was heated in a vacuum sublimation apparatus (10 mm) while slowly raising the temperature to 300–325°. Sublimation product II was recrystallized from alcohol to give colorless crystals with mp 181–182° and R_f 0.54 [Silufol, chloroform–ethanol (9.2:0.8)] in 65% yield. UV spectrum: λ_{\max} 271 nm, $\log \epsilon$ 4.01. IR spectrum: ν 1050 (C–O), 1680 cm^{-1} (C=O). PMR spectrum: δ 2.74 (singlet, CH=C), 4.95 (octet, >N–CH), 5.8–6.5 (multiplet, CH₂–O), 7.1–8.2 (multiplet, CH₂–C), and 8.21 ppm (broad singlet, CH₃).

LITERATURE CITED

1. W. Brandenburg and A. Galat, *J. Amer. Chem. Soc.*, **72**, 3275 (1950).
2. G. L. O'Connor and H. R. Nacl, *J. Amer. Chem. Soc.*, **77**, 1578 (1955).
3. R. A. Zhuk, A. É. Berzinya, G. G. Volynkina, and S. A. Giller, *Khim. Geterotsikl. Soedin.*, 550 (1970).

Institute of Organic Synthesis, Academy of Sciences of the Latvian SSR, Riga. Translated from *Khimiya Geterotsiklicheskikh Soedinenii*, No. 12, pp. 1700–1701, December, 1974. Original article submitted May 27, 1974.

©1976 Plenum Publishing Corporation, 227 West 17th Street, New York, N.Y. 10011. No part of this publication may be reproduced, stored in a retrieval system, or transmitted, in any form or by any means, electronic, mechanical, photocopying, microfilming, recording or otherwise, without written permission of the publisher. A copy of this article is available from the publisher for \$15.00.