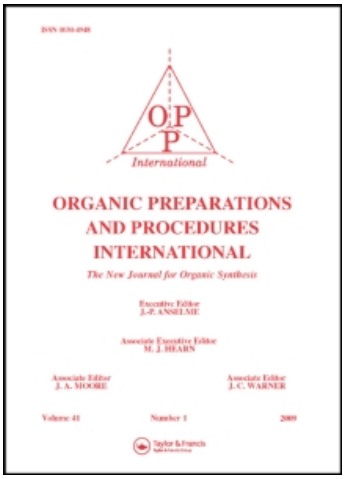

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AN IMPROVED PROCEDURE FOR SYNTHESIS OF 2-ACETYL-3-(2-METHYL-4-AMINOPYRIMIDIN-5-YL)METHYL-3A-METHYLPERHYDROFURO[2,3-D]THIAZOLE

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AN IMPROVED PROCEDURE FOR SYNTHESIS OF 2-ACETYL-3-(2-METHYL-4-AMINOPYRIMIDIN-5-YL)METHYL-3a-METHYLPERHYDROFURO[2,3-d]THIAZOLE

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(8/8/77)
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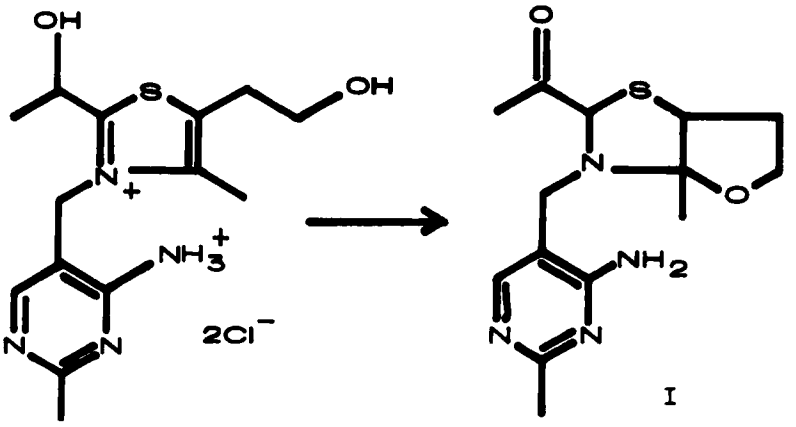
An improved procedure results in substantially higher yield of 2-acetyl-3-(2-methyl-4-aminopyrimidin-5-yl)methyl-3a-methylperhydrofuro[2,3-d]thiazole(I) than that reported earlier.[1]



EXPERIMENTAL

2-Hydroxyethylthiomine hydrochloride (10.00 g., 26.3 mmoles)[2] and two equivalents of potassium carbonate (7.29 g., 52.7 mmoles) were suspended in 100 ml of DMF and the reaction mixture was stirred under nitrogen for 6 hrs. The cloudy yellow suspension obtained was filtered and the filtrate was extracted with dichloromethane. The solvents were removed in vacuo and the thick yellow syrup was dissolved in 100 ml of dichloromethane and extracted with 3 ml of water. The aqueous layer was extracted with four 50 ml portions of dichloromethane. The combined organic phase was dried over sodium

sulfate and the solvent was removed in vacuo. The yellow syrup obtained was then extracted with six 50 ml portions of a 5:1 mixture of dichloromethane-carbon tetrachloride and 5 ml of water. The organic phase was dried and evaporated in vacuo to give white crystals. Recrystallization from hot ethyl acetate afforded 5.8 g (71%) mp. 155-157°.

Anal. Calcd. for $C_{14}H_{20}N_4O_2S$: C, 54.54; H, 6.49; N, 18.18.

Found: C, 54.75; H, 6.03; N, 18.72.

IR($CHCl_3$): 920, 1670, 1680 cm^{-1}

NMR ($CDCl_3$): 7.85 (s, 1H), 6.30 (m, 2H), 4.56 (s, 1H), 4.08 (m, 1H), 3.90 (s, 2H), 3.80 (m, 2H), 2.49 (s, 3H), 2.05 (m, 2H), 2.05 (s, 3H), 1.68 (s, 3H).

REFERENCES

1. A. Takamizawa, K. Hirai, Y. Hamashima and S. Matsumoto, Tetrahedron Letters, 5071 (1967).

2. G. E. Risinger, W. E. Gore and K. Pulver, Synthesis, 659 (1974).

A CONVENIENT METHOD FOR THE PREPARATION OF 2-AMINO-3',4,4',5-TETRACHLOROBIPHENYL

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2-Amino-3',4,4'5-tetrachlorobiphenyl (previously prepared in 24% yield by Hickmott and Hudson[1] from 3,3'-dichloro-6-nitrobenzidine) needed as a radioimmunoassay hapten, has been synthesized in 83% yield by the nitration of 3,3',4,4'-tetrachlorobiphenyl with nitric acid in acetic anhydride and