Chemistry of Heterocyclic Compounds, Vol.35, No. 2, 1999

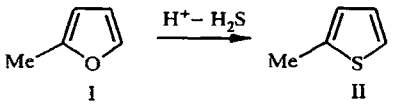
LETTERS TO THE EDITOR

NEW APPROACH TO SYNTHESIS OF 2-METHYLTHIOPHENE

T. I. Gubina, S. M. Rogacheva, and V. G. Kharchenko

2-Methylthiophene is widely used in synthesis of thiophene derivatives, and it is also used in veterinary science [1]. We have shown that 2-methylthiophene can be prepared by recycling silvan in acid media at 60°C, but the yield of the target product did not exceed 5%. At the same time its oligomerization product has been isolated in considerable amounts [2].

We have proposed a new method of conducting the reaction in a closed system and have studied the influence of various factors (nature and concentration of the acidic component, temperature, and initial 2-methylfuran concentration) as regards the selectivity of the process:



I II

We have determined the optimum conditions for converting 2-methylfuran (I) into 2-methylthiophene (II), which have enabled us to obtain the product with a yield up to 60%, initial concentration of substrate I amounting to 0.125-0.215 M, acidic component concentration - to 2.66-2.80 N (36% hydrochloric acid or dry hydrogen chloride), and temperature of 50-60°C. We have found that transferring from the system employing dry hydrogen chloride in absolute ethanol had hardly any effect.

2-Methylthiophene (II, C_5H_7S). In a long-necked flask thermostatically controlled at 50-60°C set up in a shaker, we placed 20 ml of the solvent containing the acid component (2.66-2.8 N), which had been previously saturated with hydrogen sulfide for 1 h, and to this we added 2 g (0.025 mol) of sylvan (I). At the end of reaction (2 h), the mixture was neutralized with solution of alkali and extracted with ether. The solvent was evaporated off and the residue was distilled at atmospheric pressure. Yield 1.4 g (60%); bp 112-114°C, n_D^{20} 1,5200; published data [2]: bp 112,5°C, n_D^{20} 1,5190.

REFERENCES

1. O. Z. Iskhakov, A. S. Selivanova, V. S. Avsyukevich, A. N. Sinyavin, N. P. Biryukova, and A. G. Boldyrev, "Methiphen," Sound Veterinary Use of Drug Preparations [in Russian], Rossel'khozizdat, Moscow (1984).
2. V. G. Kharchenko, T. I. Gubina, S. P. Voronin, and I. A. Markushina, Khim. Geterotsikl. Soedin., No. 11, 1453 (1986).

Chernyshevskii Saratov State University, Saratov 410026, Russia. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 2, p. 274, February, 1999. Original article submitted January 6, 1999.

 0009-3122/99/3502-0244$22.00