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BRIEF COMMUNICATIONS

Synthesis of 2-Amino-4-phenylthiazole under Conditions of Microwave Irradiation

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Abstract—The possibility of preparing 2-amino-4-phenylthiazole from acetophenone, iodine, and thiourea under the conditions of microwave irradiation was studied.

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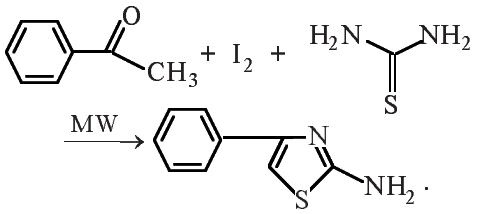
2-Amino-4-phenylthiazole derivatives exhibit high biological activity [1–3]. Published methods for preparing 2-amino-4-phenylthiazole are time-consuming and involve the use of highly toxic solvents [4, 5]. Therefore, the development of new synthetic routes to thiazole derivatives is of practical interest.

Here we suggest a new procedure for preparing 2-amino-4-phenylthiazole in a high yield:



We found that microwave irradiation allows the reaction time to be considerably shortened (by a factor of 280) compared to the traditional process and ensures the product yield as high as 92%.

We performed an independent synthesis of 2-amino-4-phenylthiazole (yield 94%) by the procedure described in [4]. Comparison of the melting points and IR spectra of the samples proved full identity of the substances obtained by the traditional procedure and under the conditions of microwave activation.

EXPERIMENTAL

A beaker flask made of heat-resistant glass was charged with a mixture of 7.2 g (0.06 mol) of acetophenone, 9.13 g (0.12 mol) of thiourea, and 15.23 g (0.06 mol) of iodine. The mixture was thoroughly stirred, loosely closed with a glass lid, and placed in a microwave oven at a radiator power of 70 W. The vessel was radiated for five 1-min periods with 30-s pauses. After that, 100 ml of water was added, and the mixture was heated at a power of 150 W for 5–7 min until the precipitate dissolved. Then the resulting yellow solution was decanted from a tarry liquid formed on the flask bottom and was filtered to separate a small amount of sulfur. After cooling, the filtrate was alkalized with aqueous ammonia to a weakly alkaline reaction. The precipitate formed in the process was filtered off on a glass frit, recrystallized from ethanol, and washed with diethyl ether. Yield of the crude product 92%, mp 148–149°C. IR spectrum, ν, cm^{-1}: 3420, 3240 (NH_2, two bands); 3170 (CH of C_3HNS); 3100 (CH of C_6H_5); 420, 1500, 1575 (C=C of C_6H_5); 1460 (C=N).

Found, %: C 61.45, H 4.47, N 15.55.
$C_9H_8N_2S$. Calculated, %: C 61.34, H 4.58, N 15.90.

The IR spectra were recorded on a UR-20 spectrophotometer (pellets).

CONCLUSIONS

(1) 2-Amino-4-phenylthiazole was prepared in a >90% yield by condensation of acetophenone with thiourea in the presence of iodine under the conditions of microwave irradiation.

(2) The reaction time is 280 times shorter compared to the traditional procedure.

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