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LETTERS TO THE EDITOR

Aminomethylation of Phenylacetylene under Microwave Irradiation

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The procedure of Mannich aminomethylation of acetylenes is being permanently improved due to the great practical significance of the resulting products. According to Gevorkyan et al. [1], the principal disadvantage of this reaction is its long time, which was explained by the necessity of using a solvent. The referees carried out aminomethylation of phenylacetylene in the absence of solvent, which allowed the reaction time to be decreased from 60 to 4–6 h.

$$Ph–C{\equiv}CH + CH_2O + HNR_2 \xrightarrow[CuCl]{MWI} Ph–C{\equiv}CCH_2NR_2.$$

We found that under microwave irradiation (MWI) phenylacetylene can be successfully aminomethylated within 2–3 min in solvent-free conditions.

To this end, a mixture of 0.01 mol of phenylacetylene, 0.013 mol of Paraform, and 0.013 mol of a secondary amine was prepared in a 250-ml heat-resistant conical flask, and then 3 drops of a catalyst (a suspension of 1 g of copper chloride in 3.5 ml of DMF) was added. The resulting mixture was carefully shaken and placed into a household microwave oven (350 W). The reaction duration was 3 min. The products were isolated by conventional methods.

This procedure was used to synthesize the following acetylenic amines: **3-(morpholin-4-yl)-1-phenylprop-1-yne,** bp 131–133°C (2 mm Hg), yield 78.4%; **3-phenyl-1-(piperidin-1-yl)prop-1-yne,** bp 132–135°C (2 mm Hg), yield 77.6%; **3-(diethylamino)-1-phenylprop-1-yne,** bp 150–155°C (5 mm Hg), yield 66.5%; **1-phenyl-3-[2-(3-pyridyl)piperidin-1-yl]-prop-1-yne,** bp 205–207°C (2 mm Hg), yield 57%. The structure of the listed compounds was confirmed by independent synthesis by the procedure in [1].

REFERENCES

1. Gevorkyan, A.A., Arakelyan, A.S., Movsisyan, A.A., Dzhandzhulyan, L.Zh., and Petrosyan, K.A., *Zh. Obshch. Khim.,* 2006, vol. 76, no. 7, p. 1223.