Microwave-induced acetylation of some aromatic primary amines using acetic acid

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Microwave-induced synthesis of anilides from aromatic primary amines using acetic acid with considerable reaction rate enhancement and improved yields is reported.

Acetylation of aromatic primary amines¹ is generally carried out by using acetic anhydride. Acetic acid in presence or absence of zinc dust can also be used for the conversion of aniline to acetanilide. However, the method requires high temperature, gas trap and 1.5 h refluxation.

In view of the above and wide applicability of microwave irradiation in chemical reaction enhancement², we report here the microwave-induced synthesis of anilides from some substituted primary aromatic amines using acetic acid and in the absence of zinc dust resulting higher yields, clear reaction products and easier workup. The results are presented in Table 1.

Table 1. Results of microwave enhanced acetylation by acetic acid

Sl no.	Substrate	Product	Time min	Yield %	M.p.* °C
1.	Aniline	Acetanilide	10	85	114
2.	<i>o</i> -Toluidine	<i>o</i> -Methylacetanilide	15	65	110
3.	<i>m</i> -Toluidine	<i>m</i> -Methylacetanilide	15	85	65
4.	<i>p</i> -Toluidine	<i>p</i> -Methylacetanilide	15	90	148
5.	2,4-Dimethylaniline	2,4-Dimethylacetanilide	15	80	127
6.	<i>p</i> -Anisidine	<i>p</i> -Methoxyacetanilide	15	71	127
7.	<i>p</i> -Bromoaniline	<i>p</i> -Bromoacetanilide	20	70	167
8.	<i>p</i> -Nitrophenol	<i>p</i> -Nitrophenylacetate	18	86	81
9.	<i>p</i> -Nitroaniline	<i>p</i> -Nitroacetanilide	18	76	212
10.	α -Naphthylamine	α -Naphthylamineacetate	18	74	159

* Lit.³ m.p.

Experimental

M.ps. were taken in open capillary on an electrically heated metal block and are uncorrected. The reactions were

carried out in a domestic Kenstar MO 9706 microwave oven (output energy, 1200 W; frequency, 2450 MHz).

General procedure : A solution of amine (0.054 mol) in glacial acetic acid (10.0 ml) was taken in a 100-ml Erlenmeyer flask fitted with a funnel as a loose top. The reaction mixture was then irradiated under microwave (17–30% power) for 2 min. Then an interval of 1 min was allowed to avoid excessive evaporation of the solvent. This protocol was repeated until reaching an overall irradiation time of 10–20 min. It was then cooled and the resulting solution was poured in ice-cold water. In the case of substrate Sl. nos. 1, 3, 5, 6 and 8 (Table 1), the acetates separated during the course of reaction. The resulting solid was filtered, washed with water and recrystallized from alcohol. The products were characterized by m.p., co-TLC and superimposable IR-spectra of the authentic samples.

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