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## SOLVENT-FREE MICROWAVE ASSISTED CANNIZZARO REACTION

**Abstract:** Microwave assisted Cannizzaro reactions of seven monosubstituted aromatic aldehydes and of 2-formylpyridine have been made under solvent-free conditions, in the presence of K-10 Montmorillonite, during a short (15 seconds) time of irradiation.

Reactions under solvent-free conditions are of a great importance in view of ecological aspects; to day the considerable increase of their use is to be observed [1,2]. Microwave irradiation is more advantageous than the conventional heating in terms of the ease of reaction conditions, especially of its shorter time [1, 3-5]. Yields of microwave assisted reactions are often higher than those of reactions performed with conventional heating [6]. Having in view the above we have investigated a series of Cannizzaro reactions [2,7,8] in solid state and under microwave irradiation.

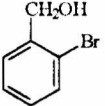
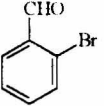
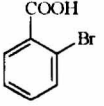
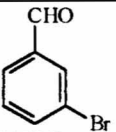
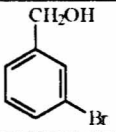
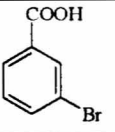
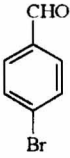
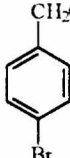

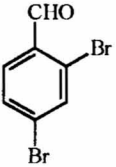
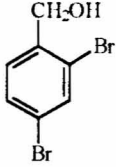
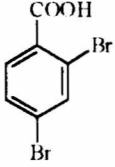
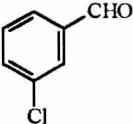
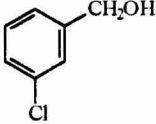
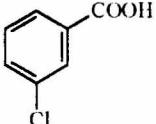
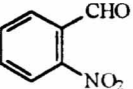
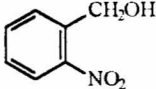
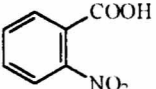
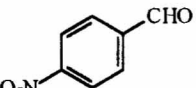
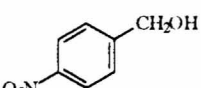
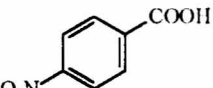
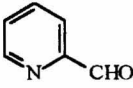
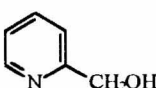
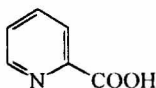
The Cannizzaro reaction involves disproportionation of aldehydes without  $\alpha$ -hydrogen atoms, occurring in the presence of a base:



In this paper we show results of microwave assisted Cannizzaro reactions of substituted aromatic aldehydes 1 - 7 and of 2-formylpyridine 8 performed in the solid state. The above starting products were mixed with NaOH, K-10 Montmorillonite and water and microwave irradiated during 15 seconds. If the reaction time was increased up to 60 seconds, the yield was not higher, and in the case of 7 the explosive decomposition took place. When 2,4-dihydroxybenzaldehyde was subjected to Cannizzaro reaction under the above conditions, the disproportionation products have not been formed, as it could be expected for a disubstituted aromatic aldehyde.

**Table 1**

Aldehydes submitted to microwave assisted Cannizzaro reaction and obtained products

	ALDEHYDE	ALCOHOL	ACID	YIELD (%)*
1				80
2				60
3				65
4				65
5				80
6				60
7				85
8				90

\* yields based on alcohols

Structures of the obtained products have been determined by  $^1\text{H}$  NMR and IR analyses; they are compatible with literature data. Results of the investigated reactions are shown in Table I.

## Experimental

Aldehyde (2 mmol), NaOH (0.5 g) and K-10 Montmorillonite (3 g) placed in a glass container were treated with four drops of water and thoroughly mixed. After 15 seconds of irradiation in a domestic microwave oven the formed product was extracted with chloroform and the solvent removed to give the alcohol component.

In order to isolate the acid component the solid residue was washed with water and filtered. The filtrate was acidified with HCl, extracted with chloroform and the extract dried over  $\text{MgSO}_4$ ; the removal of solvent gave the acid.

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**Reakcje Cannizzaro prowadzone bez rozpuszczalnika  
pod wpływem nagrzewania mikrofalowego**

**Streszczenie:** Przeprowadzono reakcje Cannizzaro siedmiu mono-  
podstawionych aldehydów aromatycznych i 2-fenylopirydyny pod wpływem  
ogrzewania mikrofalowego. Reakcje wykonano w fazie stałej  
w Montmorillonicie K-10 w czasie 15 sekund.