

## Microwave Irradiation Techniques for the Cannizzaro Reaction

Ali Sharifi<sup>1</sup>, Mohammad M. Mojtahedi<sup>2</sup>, Mohammad R. Saidi<sup>2\*</sup>

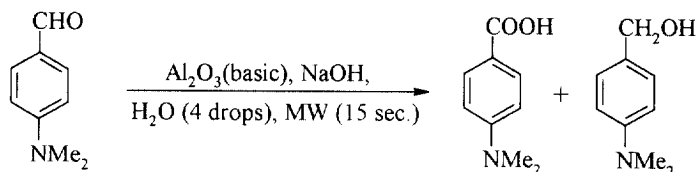
<sup>1</sup>Chemistry & Chemical Engineering Research of Iran, P. O. Box 14355-186; <sup>2</sup>Department of Chemistry, Sharif University of Technology P. O. Box 11365-9516, Teheran, I. R. of Iran

Received 9 September 1998; revised 23 November 1998; accepted 30 November 1998

**Abstract** : Aromatic aldehydes are converted to aromatic alcohols and acids by Cannizzaro reactions under microwave irradiation within 15 sec. in good yields. The reaction rate can be dramatically enhanced by irradiation of a mixture of an aldehyde, sodium hydroxide, and basic alumina, in a commercial microwave oven. © 1999 Elsevier Science Ltd. All rights reserved.

The Cannizzaro reaction is a versatile method for forming an alcohol and an acid from an aldehyde without an  $\alpha$ -hydrogen, usually from aromatic aldehydes.<sup>1-3</sup>

Organic synthesis in dry media coupled with microwave irradiation is currently a matter of increasing interest, and offers several advantages.<sup>4-6</sup> In continuation of our work on synthesis under microwave activation,<sup>7</sup> we now wish to report a very simple, fast, and general method for Cannizzaro reactions without solvent in the presence of basic alumina under microwave irradiation with high to quantitative yields. Eight different aldehydes were mixed with one equivalent of sodium hydroxide, basic alumina, and four drops of water, and then the mixtures were irradiated in a conventional domestic microwave oven for 15 sec. We found that the Cannizzaro reaction rate was enhanced dramatically by microwave irradiation. The results are summarized in Table 1. An example of a microwave-assisted Cannizzaro reaction is shown in Scheme 1.



Scheme 1

### Experimental

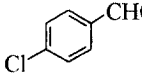
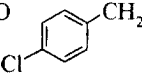
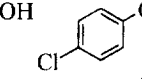
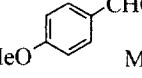
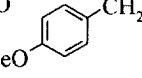
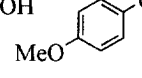
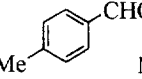
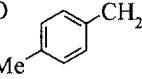
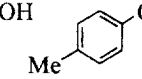
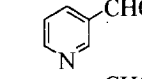
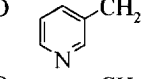
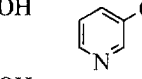
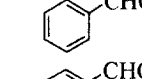
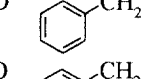
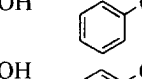
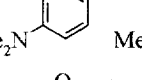
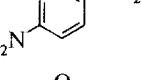
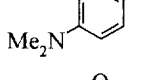
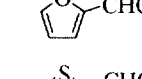
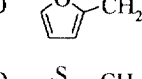
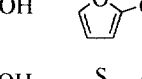
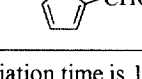
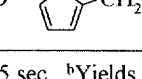
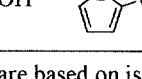
#### General Procedure for the Microwave-assisted Cannizzaro Reaction:

The aldehyde (2 mmol), solid sodium hydroxide (0.5g), and basic alumina (ca.3.0g) were placed in a teflon container. Then four drops of water were added and the reagents were mixed. After irradiation of the mixture for 15 sec., the product was washed by  $\text{CH}_2\text{Cl}_2$  (2x10 mL), and the solvent was evaporated to give the alcohol component. To obtain the acid component, the residue was washed with water (3x10 mL), the filtrate was acidified, extracted with  $\text{CH}_2\text{Cl}_2$ , and dried over magnesium sulphate. Then the solvent was evaporated to give the acid component. Preparative column chromatography with silica gel was used for further purification of the

alcohols, eluting with pet. ether/CH<sub>2</sub>Cl<sub>2</sub> (9:1). The structure of the products were determined by their <sup>1</sup>H and <sup>13</sup>C-NMR, and MS.

In conclusion, we have developed a mild, efficient, and fast, method for the Cannizzaro reaction under microwave irradiation.

Table 1: Microwave-Assisted Cannizzaro Reaction<sup>a</sup>

Entry	Substrate	Products	%Yield <sup>b,c</sup>
1		 	94
2		 	68
3		 	98
4		 	97
5		 	97
6		 	98
7		 	98
8		 	100

<sup>a</sup>Irradiation time is 15 sec. <sup>b</sup>Yields are based on isolated alcohols.

<sup>c</sup>Yield of acid were almost the same as alcohol in each case.

### Acknowledgment

We would like to acknowledge Iranian National Research Council for financial support of this work.

### References

1. March, J., "Advanced Organic Chemistry", p. 1234, J. Wiley & Sons, 4<sup>th</sup> ed., N. Y. 1992.
2. Kellogg, R. M. in "Comprehensive Organic Synthesis", Trost, B. M., Fleming, I., Eds., vol. 8, p 86, Pergamon Press, Oxford, 1991; An, J., Bagnell, L., Cablewski, T., Strauss, C. R., Trainor, R. W., *J. Org. Chem.*, **1997**, 62, 2505.
3. Fuentes, A., Sinisterra, J. V., *Tetrahedron Lett.*, **1986**, 27, 2967.
4. For reviews see: (a) Abramovitch, R. A., *Org. Prep. Proc. Int.*, **1991**, 23, 683; (b) Mingos, D. M. P., Baghurst, D. R., *Chem. Soc. Rev.*, **1991**, 20, 1.
5. Raner, K. D., Strauss, C. R., Trainor, R. W., Thorn, J. S., *J. Org. Chem.*, **1995**, 60, 2456.
6. Cablewski, T., Faux, A. F., Strauss, C. R., *J. Org. Chem.*, **1994**, 59, 3408.
7. Bolourtchian, M., Zadmand, R., Saidi, M. R., *Synth. Commun.*, **1998**, 28, 2017.