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Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/lsyc20

Facile Synthesis of Nitriles from Aromatic Aldehydes Using DMSO-I_2

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Published online: 25 Jul 2007.

To cite this article: Digambar D. Gaikwad , Sameer V. Renukdas , Babasaheb V. Kendre , Suresh U. Shisodia , Ravikumar M. Borade , Praveen S. Shinde , Sunil S. Chaudhary & Rajendra P. Pawar (2007) Facile Synthesis of Nitriles from Aromatic Aldehydes Using DMSO-I_2 , Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry, 37:2, 257-259, DOI: 10.1080/00397910601033500

To link to this article: http://dx.doi.org/10.1080/00397910601033500

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Synthetic Communications®, 37: 257–259, 2007

ISSN 0039-7911 print/1532-2432 online
DOI: 10.1080/00397910601033500

Facile Synthesis of Nitriles from Aromatic Aldehydes Using DMSO-I_2

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Abstract: Nitriles have been prepared from different aromatic aldehydes by a one-pot process using hydroxylamine hydrocloride and molecular iodine in the presence of dimethylsulphoxide.

Keywords: acetonitrile, aromatic aldehydes, dimethylsulphoxide, hydroxylamine-hydrochloride, molecular iodine

INTRODUCTION

The efficient and mild transformations of aldehydes to nitriles were extensively studied, and many methods were reported. For example, aldehydes were transformed into nitriles by using Zn/I_2 in acetonitrile,[1] ammonia water and iodine in ionic liquid,[2] acetonitrile,[3] NH_4OAc under microwave irradiation,[4] $NH_2OH{\cdot}HCl$ under microwave irradiation,[5] $I_2/NH_3/THF$[6] catalyst, $NaN_3/AlCl_3$[7] catalyst, HY-zeolite catalyst under microwave irradiation[8] and $H_2O_2/NH_2OH/Cu$[9] (Scheme 1).

In the present work benzaldehyde (**1**) was stirred at room temperature with hydroxylamine hydrochloride (**2**) and molecular iodine in the presence of dimethylsulphoxide. After an aqueous workup, benzonitrile (**3**) was isolated. Other aromatic nitriles have been similarly synthesized and are

Received June 16, 2006

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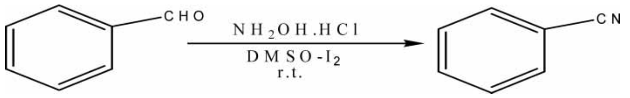
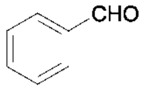
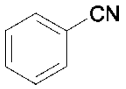
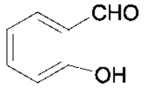
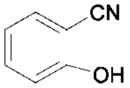
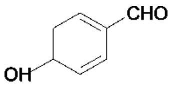
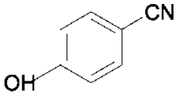
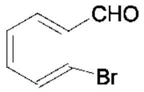
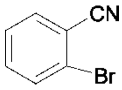
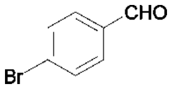
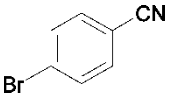
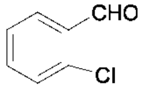
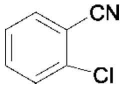
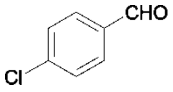
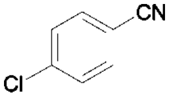
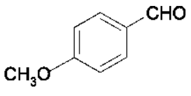
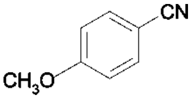
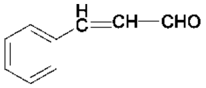
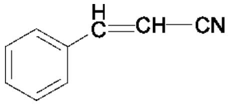


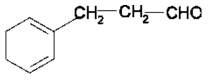
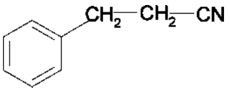
Scheme 1.

Table 1. Conversion of aromatic aldehydes to nitriles using two different solvents

Entry	Starting material (**1**)	Products (**3**)	Mp/bp (°C)	Yield (%) DMSO	Yield (%) CH_3CN
1			193	90	40
2			98	85	45
3			113	87	45
4			252	85	34
5			225	89	45
6			45	80	43
7			98	85	47
8			58	83	43
9			135	84	48

(*continued*)

Table 1. Continued

Entry	Starting material (**1**)	Products (**3**)	Mp/bp (°C)	Yield (%) DMSO	Yield (%) CH_3CN
10			265	89	38

Note: All products are known compounds and identified by comparing their melting points with standards described in the literature.

listed in Table 1. The structures of nitriles prepared were confirmed on the basis of IR and ^1H NMR spectral data. Although other organic solvents were tried, the best results were obtained with dimethylsulphoxide (DMSO) as a solvent.

EXPERIMENTAL

A solution of benzaldehyde (1.06 g) in DMSO (5 ml), hydroxylamine hydrochloride (0.69 g), and iodine (10 mg) was stirred at room temperature for 4 h. The progress of the reaction was checked on thin-layer chromatography (TLC) (hexane/ethylacetate, 8 : 2). After the completion of reaction, the reaction mixture was poured over crushed ice and extracted with ether. The ether layer was washed with brine, dried over anhydrous sodium sulphate, and evaporated to give benzonitrile (0.93 g, 90%).

ACKNOWLEDGMENTS

The authors are thankful to the principal, Vaidyanath College, Parli Vaijnath, for providing laboratory facilities. The authors are also thankful to A. W. Vadal for help during the work and to the Department of Chemistry, Pune University, for providing spectral data.

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