



A Facile Stereoselective Synthesis of Z-Aldoximes from Benzaldehyde and Hydroxylaminehydrochloride in Dry Media

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ABSTRACT

The synthesis of Z-aryldoximes has been carried out under microwave conditions using CdSO_4 as catalyst. The yields obtained were in the range 84-88%.

Key words: Z-oximes, Aldehydes, CdSO_4 , M W, Stereoselectivity.

INTRODUCTION

Oximes are widely used as protective groups for carbonyl compounds in organic synthesis as they are very stable crystalline substances. Further, they are very important from pharmacological point of view. They can also be converted into useful nitriles. As part of our continued interest in the protective group protocols in organic synthesis¹⁻⁵, we describe herein our results for the synthesis of oximes under microwave conditions in dry media using CdSO_4 .

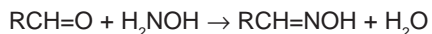
EXPERIMENTAL

In a typical procedure, benzaldehyde (1 mmol), and hydroxylaminehydrochloride (1.2 mmol) were mixed together with CdSO_4 (0.5 g) in an

Erlenmeyer flask and then irradiated at 320 W in a domestic microwave oven (Kenstar OM-9925E, 800W, operating at 2450 MHz) and the reaction was monitored by TLC till completion. The flask was taken out, cooled and dichloromethane (5 mL) added. The catalyst was filtered off and the resultant solution after drying over anhydrous sodium sulfate evaporated to give a residue which was purified by column chromatography to afford the desired Z-oxime.

RESULTS AND DISCUSSION

Oximes are commonly synthesized by condensing carbonyl compounds with hydroxylaminehydrochloride in an acidic medium although many other methods exist. However, this method usually yields a mixture of Z- and E-stereoisomeric oximes.



We have already reported the stereoselective synthesis of Z-oximes using ZnSO₄ as catalyst in this reaction. We have now investigated the use of CdSO₄ as catalyst in this reaction. The results are collected in Table 1. A large variety of variously substituted (EWG and EDG) were investigated. These included benzaldehyde, 4-

Table 1: Synthesis of Z-oximes under MW using CdSO₄

Arylaldehydeoxime	Yield
Benzaldehyde	88%
3-Nitrobenzaldehyde	84%
4-chlorobenzaldehyde	86%
2,6-dichlorobenzaldehyde	85%
4-Hydroxybenzaldehyde	84%
Cinnamaldehyde	85%

nitrobenzaldehyde, 4-chlorobenzaldehyde, 2,6-dichlorobenzaldehyde, 4-hydroxybenzaldehyde, anisaldehyde, tolualdehyde and cinnamaldehyde. The Z-oximes were obtained in 84-88% yield. These were obtained within about five minutes of microwaving the reaction mixture under open vessel conditions. E-oximes were not obtained. All the products were identified on the basis of their spectroscopic data and by comparison of their mps with those reported in literature¹⁻⁴.

Cinnamaldehyde was chosen as the representative example of an alpha, beta-unsaturated arylaldehyde and its Z-stereoisomeric oxime was obtained in 85% yield.

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