

Short Communication

APPLICATION OF MICROWAVE IRRADIATION TECHNIQUES FOR THE WITTING REACTIONM. R. Saidi^{1*}, M. M. Mojtahedi, Sh. Kaamyabi² and M. Bolourtchian²

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Abstract

The Wittig reaction rate can be dramatically enhanced by irradiation of the reaction mixture containing an aldehyde or ketone; methoxymethyl(triphenyl)phosphonium chloride, potassium t-butoxide, and t-BuOH with a commercial microwave oven. Carbonyl compound are converted to Wittig products within three min. in good yields.

Introduction

The Wittig reaction is a highly versatile method for forming alkenes from carbonyl derivatives. Ylides are useful compounds for extending a carbon chain by incorporating the double bond in the molecule and for building up carbon containing frameworks. Triphenylphosphonium ylides are very popular because of their easy preparation and stable ylides can be prepared from methoxymethyl(triphenyl)phosphonium chloride [1,5]. To carry out these reactions, it is usually required to stir at room temperature or reflux the reaction mixture for several hours.

Recently, it was shown that microwave radiation can be applied to many types of organic transformation and offers several advantages [6,8]. Continuing our work on microwave-assisted synthesis [9], we will now report a

very simple, fast, and general method for Wittig reaction. Various aldehydes and ketones are mixed with methoxymethyl(triphenyl)phosphonium chloride with an equivalent of potassium t-butoxide and an optimized amount of t-BuOH which is then subjected to microwave irradiation. We found that the Wittig reaction rate was dramatically enhanced under these conditions (typically 3 min for reaction completion). Nine different aldehydes or ketones were reacted with methoxymethyl(triphenyl)phosphonium chloride and the results are summarized in Table 1. The general reaction is shown in Scheme 1.

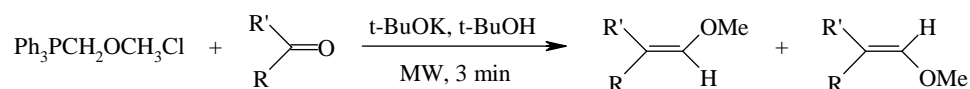
Experimental Section

IR spectra were taken on Matt Son 1000 Unicam FTIR, ¹H and ¹³C-nmr spectra were recorded on Bruker AC 80, ms spectra were obtained on a Varian MAT 311 A and Varian CH 5 spectrometers.

Keywords: Microwave irradiation; Wittig reaction; Alkenes

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Scheme 1

**General Procedure for the Preparation of Alkenes**

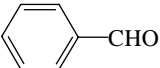
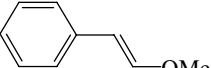
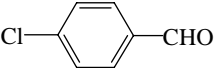
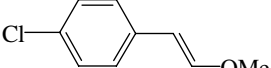
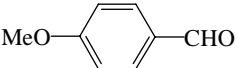
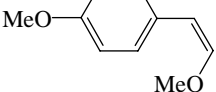
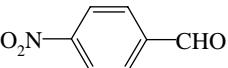
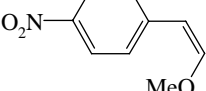

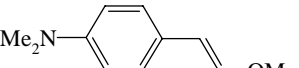
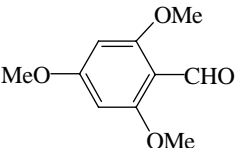
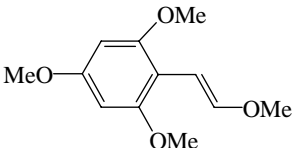
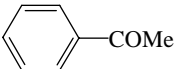
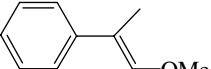
The aldehyde or ketone (1 mmol), methoxymethyl (triphenyl)phosphonium chloride (1 mmol, 0.31 g), potassium t-butoxide (1 mmol, 0.1 g) and t-butyl alcohol (1 ml) were placed in a Teflon container. The mixture was irradiated on high power in a conventional microwave oven for about 3 min. The Teflon container was then cooled to r.t., and water (10 ml) was added. The product was extracted with light pet. ether (3×10 ml). The combined organic solution was dried over Na₂SO₄, and the solvent evaporated to give the

crude product. Further purification was done by preparative column chromatography using silica gel, eluting with pet. ether, CH₂Cl₂ (9:1) if needed. The structure of the new compounds was determined by their ¹H and ¹³C-nmr and their ms.

Acknowledgements

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Table 1. Microwave-assisted Wittig reaction of aldehydes and ketones with methoxymethyl(triphenyl)phosphonium chloride

Entry	Substrate	Major product	E/Z ratio	Isolated yield, %, (time, min.)
1			7:1	52, (3)
2			5:1	53, (4)
3			1:1.7	59, (3)
4			1:1.3	55, (0.7)
5			1.9:1	32, (3)
6			1.1:1	51, (5)
7			1.3:1	33, (3)

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