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ChemTech

International Journal of ChemTech Research
CODEN(USA): IJCRGG ISSN : 0974-4290
Vol.2, No.4, pp 2182-2184, Oct-Dec 2010

Calcium chloride catalyzed Microwave Synthesis of some Mannich bases and their Characterisation

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Abstract: The synthesis of Mannich bases N-[1-(piperidinobenzyl) acetamide (**PBA**), N-[1-(morpholinobenzyl) acetamide (**MBA**), N-[1-(piperidinobenzyl) benzamide (**PBB**), N-[1-(morpholinobenzyl) benzamide (**MBB**) have been reported under microwave radiation catalysed by calcium chloride. The product is obtained in good yield.
Key words: Mannich base, PBA, MBA, PBB, MBB, Microwave, economical and ecofriendly.

Introduction

In recent years, the development of more economical and environmental friendly conversion processes is gaining interest in the chemical community.

It is well known from the literature[1-3] that the compounds containing amide moiety exhibit a wide range of biological activities. In the last few years **microwave–induced Organic Reaction Enhancement (MORE)** Chemistry has gained popularity as a non-conventional technique for rapid organic synthesis and many researchers have described accelerated organic reactions, and a large number of papers has appeared forming the synthetic utility of **MORE** chemistry in reactive organic synthesis. It can be termed as **'e-chemistry'** because it is easy effective, economical and eco–friendly and is believed to be a step towards green chemistry.

In continuation of our research programme[4-8], we herein report the microwave assisted synthesis of some Mannich bases like, **PBA, MBA, PBB** and **MBB** catalysed by calcium chloride.The synthesized compounds have been characterized by TLC, Elemental analysis, IR and ^{1}H-NMR Spectroscopy.

Recently use of inorganic solid supports[9-11] as catalysts has been developed for solvent-free reactions resulting in milder conditions and easy experimental procedures. Calcium chloride catalyzed organic reactions[12] are gaining importance owing to their inexpensive nature and special catalytic attributes in heterogeneous reactions.

Experimental

Melting points were determined in an open capillary tube with a Buchi melting point apparatus and are uncorrected. Elemental analyses were carried out using Perkin-Elmer 240C CHN-analyzer. IR spectra were recorded on a Perkin Elmer IR spectrophotometer. ^{1}H- NMR spectra was run in ($CDCl_3$) solvent at 200 MHz on a NMR spectrophotometer (chemical shifts in δ ppm).

General procedure for the Synthesis of Mannich base PBA/MBA/ PBB and MBB

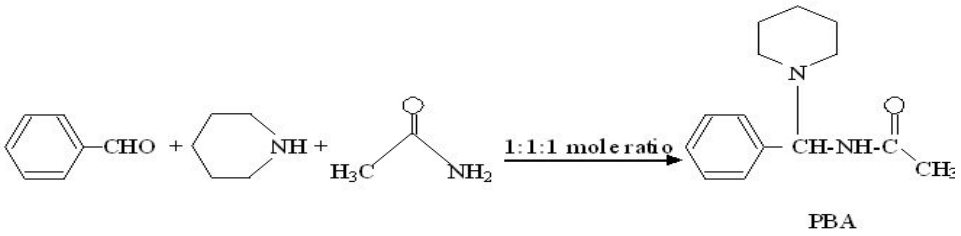
In a typical experimental procedure benzaldehyde, piperidine/ Morpholine and acetamide/ Benzamide were taken in 1:1:1 mole ratio. Piperidine / Morpholine 0.9 mL (10 mM), acetamide 0.6 g (10mM)/ Benzamide (0.9 g) and then 1 mL of benzaldehyde (10 mM) was added in the presence of $CaCl_2$ (0.168 g, 20 mol%) and kept under microwave radiation at 130^{0}C for 2 min. Then the reaction mixture was acidified with 5 mL of concentrated HCl and the lower layer was separated, washing the water layer

with dichloromethane (DCM). The organic layer was dried and, after vaccum distillation, provided the desired Mannich base in reasonable yields, **Table 1** and **Scheme 1**. Thin layer chromatography was used to check the purity of the compound.

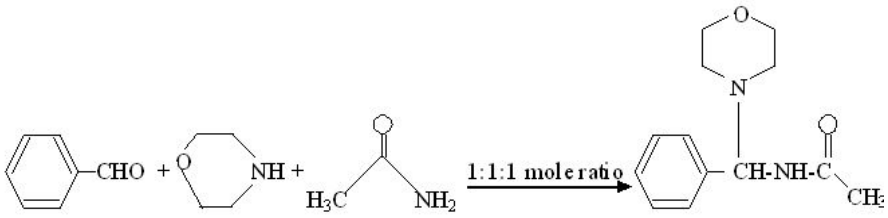
Results and Discussion

The elemental analyses value consistent with the stoichiometry for all the Mannich bases and the elemental analyses value of Mannich bases are given in the Table 1.

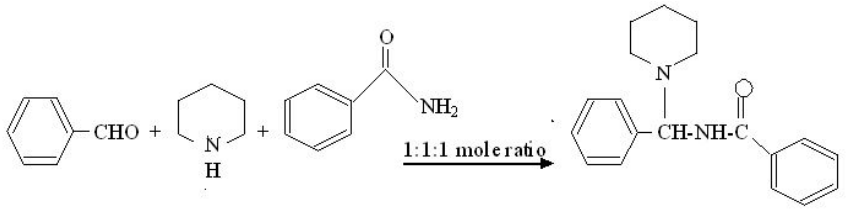
Scheme 1



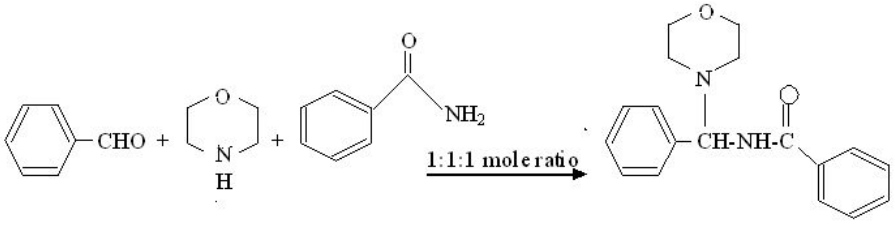
PBA



MBA



PBB



MBB

Table 1. Analytical data of the Mannich bases

Complex	Found (calculated)			m.p.	Yield (%)
	C %	H %	N %		
PBA	71.60 (72.36)	8.10 (8.68)	11.82 (12.06)	167^{0}C	82
MBA	65.80 (66.64)	7.16 (7.74)	11.34 (11.96)	169^{0}C	80
PBB	76.75 (77.52)	7.25 (7.53)	8.62 (9.52)	171^{0}C	81
MBB	71.72 (72.7)	6.84 (7.12)	9.18 (9.42)	177^{0}C	72

The IR-spectral data of the Mannich bases are given in the Table 2.

Table 2. IR spectral data of the Mannich bases ν(cm^{-1})

Compound	$\nu_{(NH2)}$	$\nu_{(C=O)}$	$\nu_{(C-N-C)}$
PBA	3320	1645	1160
MBA	3312	1642	1160
PBB	3361	1635	1140
MBB	3364	1630	1155

The ^{1}H NMR spectral data of the Mannich bases are given in the table 3.

Table 3. ^{1}H NMR spectral data of the Mannich bases

Compound	$\delta_{(`H-Aro)}$	$\delta_{(`H-NH)}$	$\delta\nu_{(Mor\ N-CH2)}$	$\delta\nu_{(Pip\ N-CH2)}$	$\delta_{(Methyl)}$	$\delta\nu_{(Mor-o-CH2)}$
PBA	7.2-7.7	5.56	-	2.5	2.2	
MBA	7.2-7.7	5.5	2.5	-	-	3.6
PBB	7.2-7.7	5.9	-	2.6	2.4	
MBB	7.2-7.7	5.0	2.7	-	-	3.6

In conclusion this condensation method offers an alternative route for the novel synthesis of Mannich bases using the inexpensive, less toxic and commercially available catalyst. Moreover, the present method offered several advantages including high yields, shorter reaction times, simple work-up procedures which make it a useful process for the synthesis of Mannich bases.

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Acknowledgements

SR thanks the Chairman, Director and Principal of Saveetha College of Engineering, Saveetha University, Chennai for their constant support and encouragement. The author also thanks the RSIC, CDRI, Lucknow and RSIC, IIT, Chennai for the use of their instrumentation facilities

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