Available online at www.derpharmachemica.com

Scholars Research Library

Der Pharma Chemica, 2011, 3(2): 206-208
(http://derpharmachemica.com/archive.html)

ISSN 0975-413X
CODEN (USA): PCHHAX

A Novel Synthesis for Meso Substituted Dipyrromethanes

Sayyed Sultan Qasim* and Syed Shahed Ali

Organic Reasearch Laboratory, Maulana Azad College, Dr. Rafiq Zakariya Campus, Dr. Babasaheb Ambedkar Marathwada University, Aurangabad, India

---

ABSTRACT

Dipyrromethanes were obtained in moderate fields by the condensation of Ketone and pyrrole in the presence of iodine as a catalyst with chloroform without using any strong acid and minimize organic solvent.

Keywords: Meso substituted dipyrromethanes, ketone, Iodine, stirring.

INTRODUCTION

Polypyrrolic compounds are of wide interest in several areas, namely in porphyrins and related macrocycles [1], science materials [2], optics [3], and medicine [4], meso substituted dipyrromethanes are important building blocks for the organic synthesis and pharmaceuticals [5,6]. Generally, the synthesis of dipyrromethanes was carried out by the condensation of aldehyde/ ketones with excess of pyrrole in the presence of strong acid such as HCl, TFA, methylsulfuric acid [7,8]. The reaction usually needs high temperature refluxing in harmful organic solvent. To avoid using liquid acid and to minimize the amount of harmful organic solvents. However, these methods require prolonged reaction time and exotic reaction condition. Thus, the development of a new method for the synthesis of meso substituted dipyrromethanes derivatives would be highly desirable. In recent years, iodine has gained special attention as a catalyst in organic synthesis because many advantage such as excellent yield, operationally simplicity, no toxicity, low cost, eco-friendly nature, readily available and high reactivity. Recently, several synthetically useful organic transformations using iodine as a catalyst have been reported in the literature [9, 10].

MATERIALS AND METHODS

The uncorrected melting points of compounds were taken in an open capillary in a paraffin bath. IR spectra were recorded on Perkin Elmer FT spectrophotometer in KBr disc. ^{1}H NMR Spectra were recorded on an 80 MHz FT-NMR Spectrometer in $CDCl_3$.

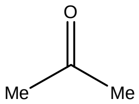
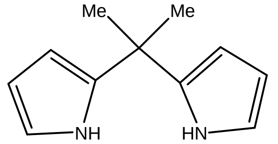
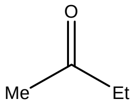
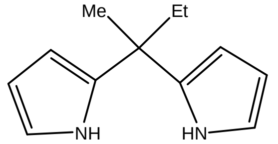
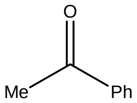
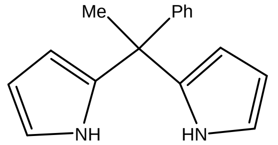
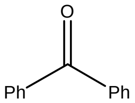
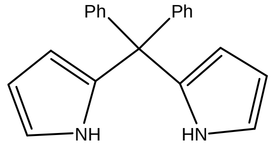
General procedure for the synthesis of meso substituted dipyrromethanes (3a-d)

A solution of acetone (1 mmol) and pyrrole (2 mmol) and Iodine (3%) mol, chloroform (10 ml) was added dropwise in round bottom flask at room temperate. The reaction mixture was stirred at room temperature for appropriate time (**Table 1**). After completion of the reaction confirmed by TLC, the mixture was treated with distilled water to furnish the crude products. The crude was further purified by column chromatography and get the corresponding meso substituted dipyrromethanes (**3a-d**). The products (**3a-d**) were conformed by comparison with authentic sample, IR, ^{1}H NMR, Mass and melting points.

RESULTS AND DISCUSSION

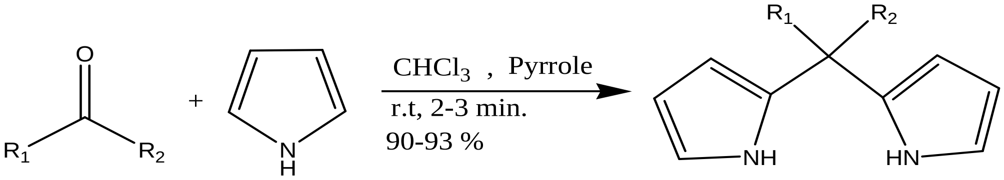
The condensation of ketone with a excess of pyrrole (1:2 Ketone/ pyrrole mmol ratio) catalyzed by iodine affords meso substituted dipyrrometahnes (**3a-d**) Table 1.

Table 1. Reaction of ketones with pyrrole to give dipyrromethanes

Entry	Ketones	Time (min)	Product	Yield (%)^{a,b}
1		2		93
2		3		90
3		2		92
4		2		93

[a]Yield of isolated pure products. [b]Product were characterized by IR, NMR, Mass elemental analysis and comparison with authentic sample.

Under these conditions, pyrrole serves both as the reactant in excess and as the solvent for the reaction complete consumption of the ketone was observed after stirring the mixture for 2-3 min at room temperature.



(3 a-d)

Scheme 1

The meso substituted dipyrromethanes (**3a-d**) were isolated by flash chromatography in 90, 92, 93% yields respectively. A mildly basic medium (n-hexane / ethyle acetate/ triethylamine ~ 1%) was used as eluant to prevent the decomposition of the dipyrromethane on the silica column used dipyrromethanes (**3a-d**) are stable in the purified from it stored at 0 °C under nitrogen atmospheres and in the absence of light. The result show that this condensation reaction is compatible with aromatic ketones bearing benzene ring group, widening the broad scope of these reactions [6, 11]

REFERENCES

[1] a) C-H. Lee, F. Li, K. Iwamoto, J. Dedok, A. A.Bothner, by J. S. Lindsey, *Tetrahedron*, **1995**, 51, 11645. b) S. Shanmugthasan, C. Edwards, R. W. Boyle, *Tetrahedron* **2000**, 56, 1025.
[2] C. M. Drain, J. T. Hupp, K. S. Suslick, M. R. Wasielewski, X. Chem, *J. Porphyrins phthalocyanines* **2002**, 6, 243-258.
[3] I. Okura, *J. Porphyrins Phthalocyanines* **2002**, 6, 268-270.
[4] a) R. Bonnett, *Chem.. Soc. Rev.* **1995**, 4151. b) E. D. Sternbery, D. Dolphin, C. Bruckner, *Tetrahedron*, **1998**, 54, 4151. c) I. J. MacDonald, T. J. Dougherty, *J. Porphyrins Phthalocyanines* **2001**, 5, 105-129.
[5] B. J. Littler, M. A. Miller, C. H. Hung, et al, *J.Org. Chem* **1999**, 64, 1391.
[6] C. H. Lee, J. S. Lindsey, *Tetrahedron*, **1994**, 11427.
[7] G. Gao, I. Korobkov, S. Gambarotta, *Inorg. Chem*. **2004**, 1108
[8] T. Dube, S. Conoci, S. Gambarotta, G. P. A., Yap, *Angew, Chem. Int Ed.* **1999**, 3657.
[9] M. Kidwai, P. Mothsra, V. Bansal, R. Goyal, *Mont. Fur. Chem*. **2006**, 137, 1189.
[10] B. P. Bandgar, K. A. Shaikh, *Tetrahedron Lett*. **2003**, 44, 1959-1961.
[11] E. N. Durantini, J. J. Silber, *Synth. Commun.* **1999**, 29, 3353.