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EFFICIENT SYNTHESIS OF BIS(INDOLYL) METHANES BY USING SILICA SUPPORTED TCAA

VISHVANATH D. PATIL*, PRATHAMESH V. GIDH, PRASANNA C. PATIL, NAGESH SUTAR and KETAN P. PATIL

Organic Chemistry Research Laboratory, Department of Chemistry, C. K. Thakur A. C. S. College, New Panvel, RAIGAD (M.S.) INDIA

ABSTRACT

Bis(indolyl) methanes derivatives have been synthesized using a catalytic amount of SiO_2.TCAA at room temperature with excellent yields. The remarkable selectivity under mild, neutral and, inexpensive catalyst are attractive features.

Key words: Bis(indolyl) methanes, SiO_2.TCAA, Aldehydes, Ketones.

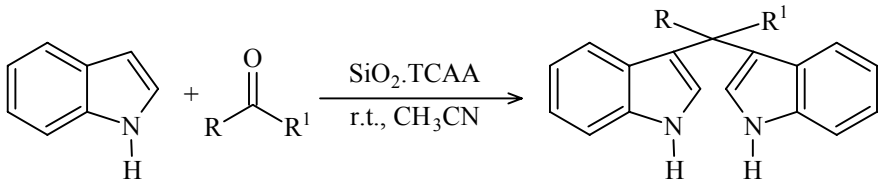
INTRODUCTION

The development of simple, efficient and economically viable chemical process or methodologies for widely used organic compounds are in great demand[1]. Various methods have been developed for their synthesis using Lewis acid catalysts[2-8], ionic liquids[9], trichloro-1,3,5-triazine[10], and potassium hydrogen sulphate[11]. However, many of these reported methods suffer from one or more disadvantages such as harsh experimental procedure and reagents that are expensive and moisture sensitive. A mild and efficient catalyst for the synthesis of bis(indolyl) methanes is highly desirable.

EXPERIMENTAL

In this communication, a synthesis of Bis(indolyl) Methanes by usinig silica supported trichloroacetic acid (TCAA) has been reported. A wide variety of compounds were used at optimal reaction conditions to prepare a wide range of bis(indolyl) methanes (**Scheme 1**).

*Author for correspondence; E-mail: patilvd148@yahoo.in; Fax: 022-7467600



R : Phenyl, Alkyl; R^1: H, Phenyl, Alkyl

Scheme 1

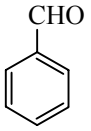
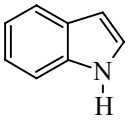
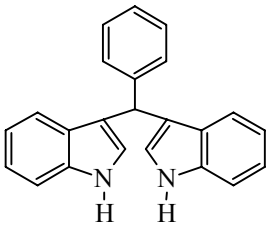
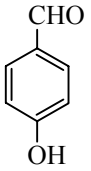
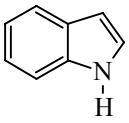
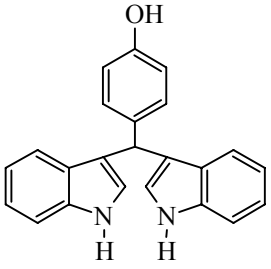
General experimental procedure for Bis(indolyl) methanes

A mixture of benzaldehyde (2 mmol), indole (4 mmol) and [TCAA.SiO_2] (0.1 mmol, 30 mg) was stirred magnetically at room temperature in acetonitrile (1 mL) and the progress of the reaction was monitored by thin layer chromatography. The product was dried over anhydrous Na_2SO_4 and further purification by column chromatography.

RESULTS AND DISCUSSION

The reaction proceeded efficiently and smoothly at room temperature in presence of silica supported trichloroacetic acid as a catalyst, and the products were obtained in excellent yields. Various aromatic aldehydes, aliphatic aldehyde and ketones give the corresponding products with excellent yield (Table 1, entries 1-9).

Table 1: Synthesis of bis(indolyl) methanes

Entry	Aldehyde[a]	Indoles	Product[b]	Time (min)	Yield[c] (%)
1				30	93
2				30	91

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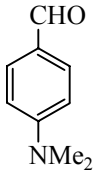
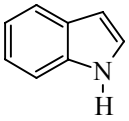
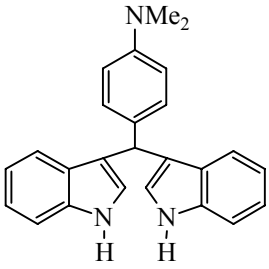
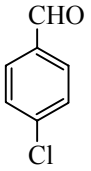
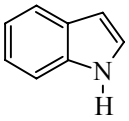
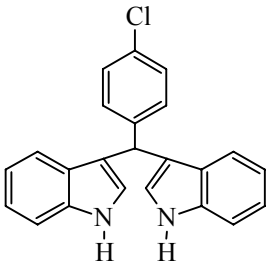
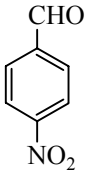
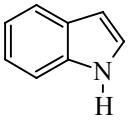
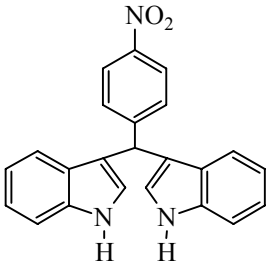
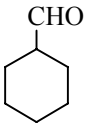
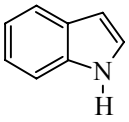
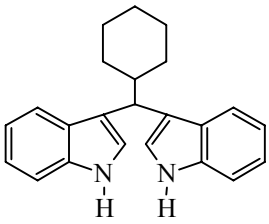
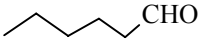
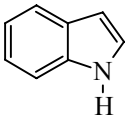
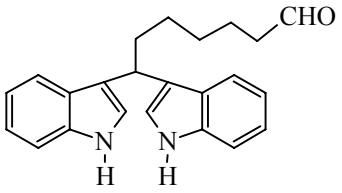
Entry	Aldehyde[a]	Indoles	Product[b]	Time (min)	Yield[c] (%)
3				60	90
4				60	85
5				80	90
6				80	90
7				90	85

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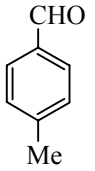
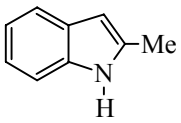
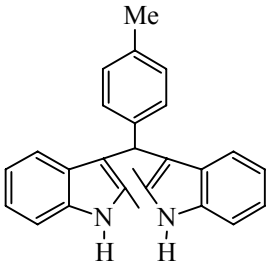
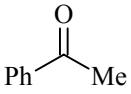
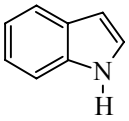
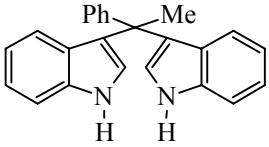
Entry	Aldehyde[a]	Indoles	Product[b]	Time (min)	Yield[c] (%)
8				40	90
9				120	87

[a]The substrate was treated with indole (4 mmol) by stirring at room temperature with SiO_2.TCAA in presence of acetonitrile as solvent; [b]All products were identified by their IR and ^{1}H NMR spectra; [c]Isolated yields after column chromatography

Spectral data

3,3'-Bis(indolyl) phenyl methane (1): Pale-red solid, yield 93%, m.p. 122-124^{o}C;

IR (KBr): 736, 1012, 1173,1336, 1415, 1599, 2848, 3024, 3054 and 3409 cm^{-1};

^{1}H NMR (300MHz, $CDCl_3$): δ = 7.8 (s, 2H); 7.1-7.4 (br m, 8H); 6.3-6.8 (m, 5H); 4.1-4.4 (s, 2NH) and 2.2 (s, H);

^{13}C NMR ($CDCl_3$): 144.1, 136.7, 128.7, 128.6, 127.2, 126.9, 123.7, 121.9, 119.9, 111.1 and 40.2.

EIMS; m/z 322.

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