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One-Pot Synthesis of 1,8-Dioxo-octahydroxanthene Derivatives Using $Zn(NO_3)_2$ under Solvent-free Conditions

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ABSTRACT

$Zn(NO_3)_2$ as an efficient catalyst have been used for the one-pot synthesis of 1,8-Dioxo-octahydroxanthene Derivatives via multi-component reactions between dimedone and various aromatic aldehydes under solvent-free conditions. The presented method is available, environmentally friendly, cheap and highly effective to give the products in good to excellent yields.

Key words: Xanthenes, Solvent-free, Multicomponent reactions, $Zn(NO_3)_2$.

INTRODUCTION

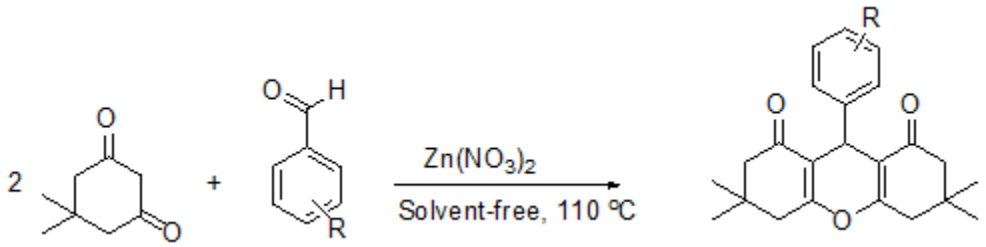
Multicomponent reactions (MCRs) refers to a reaction in which two or more ingredients are combined within a single process and the products they create, which is part of all the components are present[1-3]. Since the multi-component reactions for the synthesis of organic compounds and these compounds can be used as a drug and precursor multicomponent reactions, so to investigate them out is important[4].

In our ongoing research prompted by our interest in multiple component reactions and as part of programs in the area of heterocyclic compounds containing oxygen, and due to the resultant pharmacological interest in compounds which belong to the xanthene Derivatives, although this reaction done[5].

Xanthene derivatives are one of the important classes of organic compounds which are biologically important drug intermediates in the field of medicinal chemistry for their biologically active properties, such as antimalarial, antibacterial, antiinflammatory, and antiviral properties and have been used as dyes, fluorescent material and in laser technologies[6-9]. Recently, several improved methodologies have been developed that use triethylbenzyl ammonium chloride[10], *p*-dodecyl benzenesulfonic acid[11], phosphomolybdic acid supported on silica gel[12], sulfonic acid on silica gel[13], $HClO_4$–SiO_2 [14] and ZnO [15] among others. Previously, we have synthesized a number of heterocyclic compounds[16-26].

In this study, we have used of $Zn(NO_3)_2$ as a catalysts to develop a new and easy methodology for the synthesis of xanthene derivatives. The

experiments were started with the study of one pot reaction, a short time with high yields, easy separation of product, and a 3-component method, mild and efficient method for the preparation of the xanthenes by using $Zn(NO_3)_2$ as a catalyst (Scheme1).



Scheme 1:

EXPERIMENTAL

All chemicals were obtained from Merck or Fluka without further purification. Silica gel SILG/UV 254 plates were used for TLC. IR spectra were measured on a Shimadzu IR-470 Spectrophotometer. ^{1}H NMR spectra were determined on Bruker 400 DRX AVANCE instrument at 400 MHz, respectively.

General procedure for preparation of A1

A mixture of aldehyde (1 mmol), dimedone (2 mmol), and $Zn(NO_3)_2$ (6 mol %) as a catalyst was stirred at 110 °C for 10 min. The progress of reaction was monitored by TLC. After finishing, recrystallized from ethanol 95% to give pure products (A1)

Spectral data

3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-9-(3-nitrophenyl)-2H-xanthene-1,8(5H,9H)-dione (A1)

White crystals, Yield: (90%), mp 171-174°C. IR (ν_{max}/ cm^{-1}) (KBr): 3074 (arom. CH Str.); 2990 (aliph. CH Str.); 1660 (C=O Str.); 1622 (C=C Str.); 1500, 1360 (NO2,Str). ^{1}H NMR (400.22 MHz $CDCl_3$): δ=1.00 (6H, s, $2CH_3$); 1.12 (6H, s, $2CH_3$); 2.19 (4H, ABq, ^3J=16.4Hz, $2CH_2$); 2.52 (4H, s, $2CH_2$); 4.84 (H, s, CH); 7.28 8.04- (4H, m, CH).

RESULTS AND DISCUSSION

We have been able to introduce an efficient and environmentally friendly for the synthesis of xanthene derivatives via condensation of dimedone with various aromatic aldehydes and ammonium acetate. Therefore, reported $Zn(NO_3)_2$ as catalyst which could provide an efficient, cheap, easy separation, high yield and simple route under solvent-free condition for the synthesis of 1,8-Dioxo-octahydroxanthenes.

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