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NOTE

One Pot Synthesis of 3-Nitro-1,2-dihydroxyanthraquinone and 1-Hydroxy-9,10-dioxo-9,10-dihydroanthracen-2-yl Benzoate Using Microwave Irradiation

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An efficient method for the one pot synthesis of 3-nitro-1,2-dihydroxyanthraquinone and 1-hydroxy-9,10-dioxo-9,10-dihydroanthracen-2-yl benzoate (an anthraquinone derivative) under microwave irradiation conditions. The structures of newly synthesized compounds have been established by analytical data that includes elemental analysis, mass spectra, IR spectra and melting point.

Key Words: Alizarin, Microwave, IR and Mass spectroscopy.

Microwave radiations are now widely used as unconventional source of energy for synthesis of many organic substances and have variety of applications[1-3]. Organic molecules absorb the microwave energy selectively enhancing the rate of reaction. The MORE chemistry was earlier restricted to the use of high boiling point polar solvents like DMSO, DMF *etc*. Later few low boiling point solvents like toluene[4] were used but were found to generate serious hazards. To overcome the problem in microwave synthesis, later on solvent free path[5,6] was developed. This dry media synthesis is now widely used. Verma[7] has carried out solvent free synthesis of thioacetones and thioamides. Bram *et al*.[8] made use of catalysts and solid support. Pasha *et al*.[9] carried out synthesis of amides from carboxylic acids and urea in presence of pyridine under microwave irradiation.

A solvent free synthesis of 2-hydrazinobenzothiazole derivative using microwave has been reported[10]. Some flavones have been synthesized under microwave irradiation. Microwave assisted solvent free synthesis of substituted cromenes was carried out by Meenakshi and Ravichandaran[11]; solid state induced hetrocyclization under microwave irradiation for synthesizing of 2-phenyl-3-hydroxy-quinoline-4(H)-1 was reported by Heravi *et al*.[12]; solvent free improved synthesis of some substituted 1-3-diaryl propones and 3,5-diaryl-6-carbethoxy cyclohexenione under microwave irradiation was carried out by Jhala *et al*.[13]; solvent free synthesis of triphenyl methane dyes was carried out by Mehta[14]. The chemistry of dyes and their derivatives has been an interesting field of study.

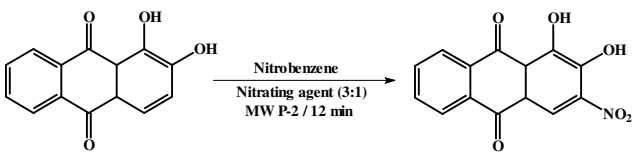
The present work describes the synthesis of 3-nitro-1,2-dihydroxyanthraquinone and 1-hydroxy-9,10-dioxo-9,10-dihydroanthracen-2-yl benzoate by attacking electrophiles and nucleophiles on alizarin.

Melting points were determined by melting point apparatus. The IR spectra of samples were recorded on FT-IR spectrometer in the range 4000-400 cm^{-1} region using nujol technique. Thin layer chromatography was performed on silica gel sheets.

General procedure

Synthesis of 3-nitro-1,2-dihydroxyanthraquinone: 0.01 mol of alizarin was slowly added in nitrating mixture (3:1) and heated for 12 min at P-2 in microwave, cooled and poured into a crushed ice. The solid product was separated out. Recrystallized the solid from ethanol. m.p. = 245 ºC, yield = 64.2 %, m.f. $C_{14}H_7NO_6$, m.w. = 285.

Elemental analysis (%): C 53.84, H 26.92. IR (Nujol, ν_{max}, cm^{-1}): 3468, 3431 (-OH), 3103 (ArCH=CH), 1764 (C=O), 1864 (C=O), 1348-1536 (-NO_2); $M^{\pm}$ = 285, $M^{\pm}$ (NO_2) = 239, $M^{\pm}$ (NO_2, -OH) = 222, $M^{\pm}$ (NO_2, -OH, -OH) = 205.

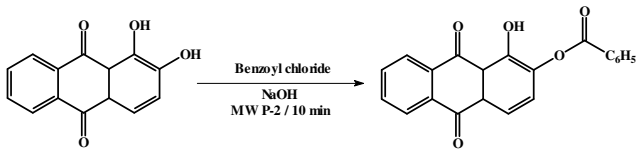


Compound **1**

Synthesis of 1-hydroxy-9,10-dioxo-9,10-dihydroanthracen-2-yl benzoate: Mixture of alizarin (0.01 mol), benzoyl

chloride and added 2 mL of 10 % NaOH and then heated at P-2 for 10 min in microwave. Cooled the reaction mixture at room temperature, the solid product is obtained and recrystallized from ethanol. m.p. = 262 ºC, yield = 59.2 %, m.f. $C_{21}H_{12}O_5$, m.w. = 344.

Elemental analysis (%): C 58.12, H 26.92. IR (Nujol, ν_{max}, cm^{-1}) 3441 (-OH), 1291 (C-O-C), 1712 (C=O), 1735 (C=O), 3130 (Ar CH=CH), 1755 (C=O), 1126 (=O-H), 1686 (C=C), 1496 (C-C); $M^{\pm}$ = 344, $M^{\pm}$ (-OH) = 328.



Compound **2**

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