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Synthesis of Some Bromo-Substituted 3-Aroyl Flavanones and Flavones

P. R. MAHALLE and N. T. KHATY*

Priyardarshini College of Engineering, Nagpur, India.
prmahalle@rediffmail.com

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Abstract: A series of bromo-substituted 3-aroyl flavanones and flavones have been synthesized. The identities of the new compounds synthesized have been developed on the basis of usual chemical transformation and IR, NMR spectral studies.

Keywords: Bromo-substituted compounds, 3-Aroyl flavanones, Flavones, Synthesis.

Introduction

Chalcones, flavanones and flavones were reported to have antibacterial, antifungal, anticancer, anti -HIV and anti-oxidant properties[1-4]. Flavanones and flavones play a pivotal role in the field of heterocyclic chemistry. These acts as a backbone for the compounds which possess diverse pharmacological and microbial activity[5,6].

The present communication deals with the synthesis of bromo-substituted 3-aroyl flavones by employing two procedures *i. e.* one by the oxidation of 3-aroyl flavanones in presence SeO_2 as oxidizing agent which involves refluxing period of about 18 h and other method by using iodine crystal as the oxidizing agent which requires only 30 min. The reaction was carried out in different solvents like DMSO, THF and dioxane.

Experimental

All the melting points reported are uncorrected. All the compounds synthesized were characterized on the basis of usual chemical transformation and IR, NMR spectral studies[7]. The IR spectra were recorded on a Perkin - Elmer FT-IR infrared spectrophotometer. The ^{1}HNMR spectra were recorded on a Bruker-DRX 300 MHz NMR spectrophotometer using TMS as an internal reference in DMSO-d6 as a solvent.

*1-(2-Hydroxy -3-bromo-5-methyl-phenyl)-3-phenyl– 1, 3-propadione (**1**)*

It was prepared by the classical Baker -Venkatraman transformation[8,9] from 2-benzoyloxy -3-bromo-5-methyl acetophenone using pulverized KOH in pyridine.

*3-Benzoyl-6-methyl-8-bromo flavanone(**3**) and 4'Methoxy-3-benzoyl-6-methyl-8-bromo flavanone(**5**)*

3-Benzoyl-6-methyl-8-bromo flavanone **(3)** and 4'methoxy-3-benzoyl-6-methyl-8-bromo flavanone **(5)** were prepared from the condensation of 1-(2-hydroxy-3-bromo-5-methyl-phenyl)-3-phenyl 1,3-propadione (**1**, 0.005 M) with benzaldehyde (2, 0.005 M) and anisaldehyde (4, 0.005 M) respectively in ethanol in presence of few drops of piperidine (0.5 mL). The reactions were carried out for 2 h. The products were recrystallised form ethanol- acetic acid mixture.

*3-Benzoyl-6-methyl-8-bromo flavone (6) and 4'methoxy-3-benzoyl-6-methyl-8-bromo flavone(**7**)*

The target products were prepared by the oxidation of 3-benzoyl-6-methyl-8-bromo flavanone **(3)** and 4'methoxy-3-benzoyl-6-methyl-8-bromo flavanone **(5)**, respectively with SeO_2 in different solvents like DMF, THF and DMSO. The reaction mixture was refluxed for 18 h and the formation of the products was ensured with the help of TLC. The same products were prepared using Iodine as an oxidizing agent which just takes 30 min for the completion of the reaction. The Spectral data of the synthesized product are described as below:

IR (cm^{-1})**:** 1670(C=O), 2340(Ar-H), 1070(C-O-C), 1440(C=C). **NMR:** δ 7.0-7.4 (m, 12H Ar-H); δ 5.5-6.0(m, 2H,CH), δ 2.2-2.3(m,3H, CH_3).

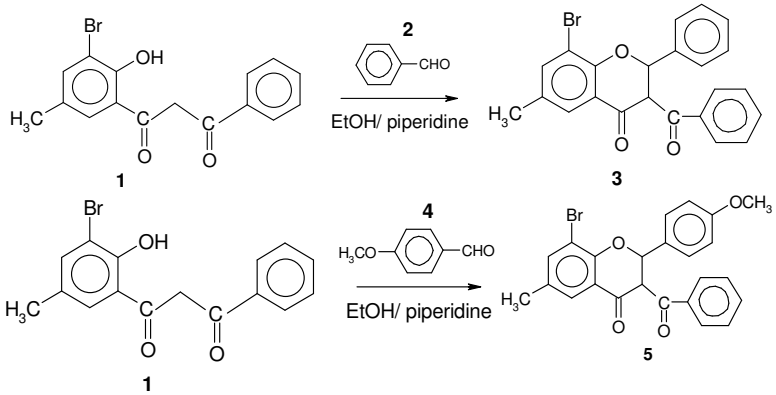
IR (cm^{-1}) **:** 1690(C=O), 2360(Ar-H), 1050(C-O-C), 1450(C=C). **NMR:** δ 7.32-7.97 (m, 11H Ar-H); δ 5.5-5.9 (m, 2H, CH), δ 3.4-3.6(m, 3H, OCH_3); δ 2.2-2.4(m, 3H,CH_3).

IR (cm^{-1}): 1650(C=O), 2370(Ar-H), 1060(C-O-C), 1450(C=C). **NMR:** δ 7.2-7.8 (m, 12H Ar-H); δ 2.2-2.3(m, 3H, CH_3).

IR (cm^{-1}): 1650(C=O), 2370(Ar-H), 1060(C-O-C), 1450(C=C). **NMR:** δ.4-8.0 (m,11H,Ar-H); δ3.6-3.8(m,3H,OCH_3),δ2.34-2.46(m, 3H,CH_3)

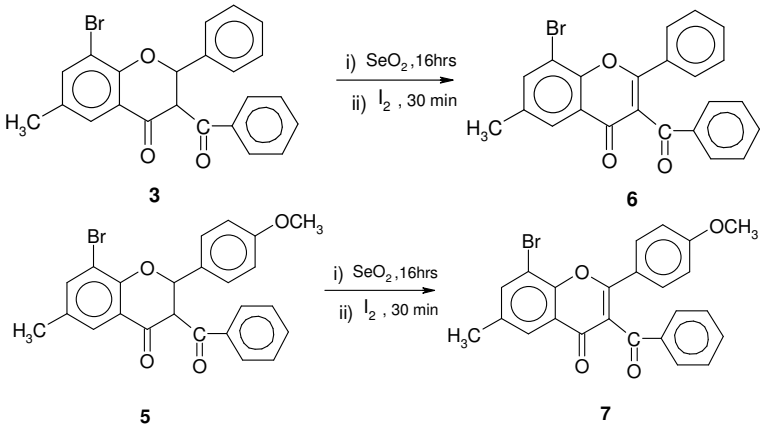
Results and Discussion

The bromo-substituted 3-aroyl flavanones were prepared from the condensation of diketone with an aromatic aldehyde. The diketone was prepared by employing the classical Bekar-Venkatraman synthesis. Thus 3-benzoyl-6-methyl-8-bromo flavanone **(3)** and 4'-methoxy-3-benzoyl-6-methyl-8-bromo flavanone **(5**) were synthesized by the condensation of 1-(2-hydroxy-3-bromo-5-methyl phenyl)-3-phenyl-1,3-propadione **(1)** with benzaldehyde (**2)** and anisaldehyde **(4)** respectively in ethanol for two hours in presence of few drops of piperidine (Scheme 1)**.** The products were recrystallised from ethanol-acetic acid mixture.



Scheme 1

In order to obtain the bromo-substituted 3-aroyl flavones, the flavanones **3** and **5** were oxidized using SeO_2 for the reaction time of 16 h in various solvents like THF, DMSO and dioxane. Similarly, the same products were oxidized in presence of a single crystal of iodine (I_2) and it was observed that the reaction was completed just in 30 min.



Scheme 2

Table 1. Characteristic data of the synthesized compounds

Product	Molecular Formula	Yield, %	M.p., °C	Rf (*n*-hexane: EtOAc)	C, H analysis %	
					C, Found (Calcd.)	H, Found (Calcd.)
3	$C_{23}H_{17}BrO_3$	75	137-138	0.76(3:2)	65.61(65.57)	4.05(4.07)
5	$C_{24}H_{19}BrO_4$	75	149-151	0.79(3:2)	63.90(63.87)	4.22(4.24)
6	$C_{23}H_{15}BrO_3$	65	189-191	0.86(3:2)	65.85(65.89)	3.59(3.61)
7	$C_{24}H_{17}BrO_4$	70	196-199	0.64(3:2)	64.10(64.16)	3.83(3.81)

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