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Synthesis of 3-bromo Flavones from 2-Hydroxy-3, 5-dibromo-4'nitro Dibenzoyl Methane

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ABSTRACT

A new 3-bromo substituted flavones have been synthesized by the reaction of 2- hydroxy-3, 5-dibromo-4'nitro dibenzoyl methane was dissolved in dimethyl formamide (DMF) and pure bromine was added. The mixture was refluxed for 1-3 hours. Then cooled, diluted with ice-cold water and crystallized from alcohol-acetic acid mixture to get 2(4'nitrophenyl)-3,6, 8-tribromo flavones.The structures of all newly synthesized compounds were confirmed on the basis of IR,NMR. The melting points were taken in an open capillary tube.

Key words: Synthesis, Dibenzoyl methane, Bromo-Flavones.

INTRODUCTION

Flavones constitute large segment of natural products.Synthesis of flavones has attracted considerable attention due to their significant biocidal[1-3], pharmaceutical [4-7]anti- cancer[8] and anti-inflammatory [9-10]effects.Dibenzoyl methane reacts in DMF medium or with bromine in DMF to give 3-halo flavones[11]. 1, 3 diketones was transformed into 7-hydroxy-6-nitro flavone[12].Some new biologically active chalcones and flavones have been synthesized [13].3-chloro flavones were prepared by action of thionyl chloride or sulphuryl chloride with flavones[14-16]. Antibacterial activity of some new chalconesand flavones having 2-chloro-8-methoxyquinolinyl moiety[17].

The literature survey clearly indicates that 3-bromo substituted flavones are not yet synthesized. It was therefore thought of interest to synthesis 2(4'nitrophenyl)-3, 6, 8-tribromo flavones from 2- hydroxy-3, 5-dibromo-4'nitro dibenzoylmethane(IIa). 2- hydroxy-3, 5-dibromo-4'nitro dibenzoyl methane (0.01 moles) (Ia) was dissolved in dimethyl formamide (DMF) and pure Bromine (0.01 moles) was added. The mixture was refluxed for 1-3 hours. Then cooled, diluted with ice-cold water and crystallized from alcohol-acetic acid mixture to get 2(4'nitrophenyl)-3, 6, 8-tribromo flavones (IIa). Structures of these compounds have been established by spectral analysis.

EXPERIMENTAL

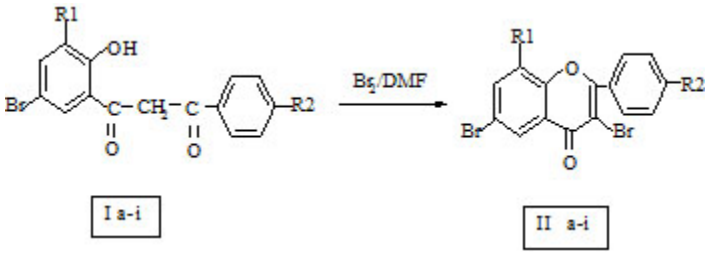
Melting point were taken in Silicon Oil bath instrument in open capillary and are uncorrected . Purity of the compounds was checked by TLC on silica gel G plates. IR spectra were recorded in Nujol, H^1 NMR spectra were recorded in $CDCl_3$ with TMS as an internal standard.

Preparation of 2-(4'-nitrophenyl)-3,6-8-tribromo flavones (IIa)

2- hydroxy-3, 5-dibromo-4'nitro dibenzoyl methane (0.01 moles) was dissolved in dimethyl formamide (DMF) and pure Bromine (0.01 moles) was added. The mixture was refluxed for 1-3 hours. Then cooled, diluted with ice-cold water and crystallized from alcohol-acetic acid mixture to get 2(4'nitrophenyl)-3, 6, 8-tribromo flavones (IIa), m.p.-169^{0}c.

It show negative ferric chloride solution test indicate the involvement of phenolic –OH group in cyclization.

Similarly other compounds (II a-i) were prepared by above method.



Scheme of Compounds (IIa-i)

Table 1: Physical data of synthesized 2-(4'-nitrophenyl)-3,6-8-tribromo flavones (IIa)

S.No.	Compounds	R_1	R_2	M.P(^{0}C)	Yields in %
1	IIa	Br	NO_2	111	60
2	IIb	Br	Cl	178	76
3	IIc	Br	NH_2	138	69
4	IId	NO_2	NO_2	252	75
5	IIe	NO_2	Cl	206	73
6	II_f	NO_2	NH_2	131	80
7	IIg	H	NO_2	293	78
8	IIh	H	Cl	224	72
9	IIi	H	NH_2	261	71

Spectral interpretation of (2i)

IR spectrum was recorded in Nujol.

IR(υmax)cm^{-1}

- 1663 cm^{-1}(C=0).
- 1590cm^{-1} (C=C).
- 1249 cm^{-1} (C-O-C)
- 702 cm^{-1} (-C-Br).

NMR

H PMR was recorded in $CDCl_3$ with TMS as internal standard.

- 6.85-7.97 δ (m, 6H, Ar-H).

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