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

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(Received: June 30, 2012; Accepted: August 20, 2012)

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 biocidal¹-³, pharmaceutical ⁴-⁷anti- cancer⁸ and anti-inflammatory⁹¹⁰ effects. Dibenzoyl methane reacts in DMF medium or with bromine in DMF to give 3-halo flavones¹¹. 1, 3 diketones was transformed into 7-hydroxy-6-nitro flavone¹². Some new biologically active chalcones and flavones have been synthesized¹³. 3-chloro flavones were prepared by action of thionyl chloride or sulphuryl chloride with flavones¹⁴-¹⁶. Antibacterial activity of some new chalcones and flavones having 2-chloro-8-methoxyquinolinyl moiety¹⁷.

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 dibenzoylmethane (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, H¹ NMR spectra were recorded in CDCl₃ 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°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)](image)

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

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Compounds</th>
<th>R₁</th>
<th>R₂</th>
<th>M.P(ºC)</th>
<th>Yields in %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>IIa</td>
<td>Br</td>
<td>NO₂</td>
<td>111</td>
<td>60</td>
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<tr>
<td>2</td>
<td>IIb</td>
<td>Br</td>
<td>Cl</td>
<td>178</td>
<td>76</td>
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<tr>
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<td>IIc</td>
<td>Br</td>
<td>NH₂</td>
<td>138</td>
<td>69</td>
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<tr>
<td>4</td>
<td>IId</td>
<td>NO₂</td>
<td>NO₂</td>
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<tr>
<td>5</td>
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<td>NO₂</td>
<td>Cl</td>
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<tr>
<td>6</td>
<td>IIf</td>
<td>NO₂</td>
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<td>IIg</td>
<td>H</td>
<td>NO₂</td>
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<td>8</td>
<td>IIh</td>
<td>H</td>
<td>Cl</td>
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<tr>
<td>9</td>
<td>IIi</td>
<td>H</td>
<td>NH₂</td>
<td>261</td>
<td>71</td>
</tr>
</tbody>
</table>

**Spectral interpretation of (2i)**

IR spectrum was recorded in Nujol.

- 1663 cm⁻¹ (C=O).
- 1590 cm⁻¹ (C=C).
- 1249 cm⁻¹ (C-O-C).
- 702 cm⁻¹ (-C-Br).

**NMR**

H PMR was recorded in CDCl₃ with TMS as internal standard.

- 6.85-7.97 δ (m, 6H, Ar-H).

**ACKNOWLEDGMENTS**

The authors are thankful to Department of Chemistry ShriShivaji College Akola & Mahatma Fule Arts, Commerce And SitaramjiChaudhari Science Mahavidyalaya, Warud for their valuable Support and necessary laboratory facilities during research work.
REFERENCES