



## NOVEL SYNTHESIS AND CHARACTERISATION OF AURONES

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### ABSTRACT

2-Hydroxy-5-bromo acetophenone, 2-hydroxy-3,5-dibromo acetophenone and 2-hydroxy-3-nitro-5-bromo acetophenone in glacial acetic acid at its boiling point treated with ICl affords ω-iodo-2-hydroxy-5-bromo acetophenone (Ia), ω-iodo-2-hydroxy-3,5-dibromo acetophenone (Ib) and ω-iodo-2-hydroxy-3-nitro-5-bromo acetophenone (Ic) condensed with substituted benzaldehyde in 40% NaOH gives aurones (IIa-l). The structure of aurones was confirmed by chemical and spectral data.

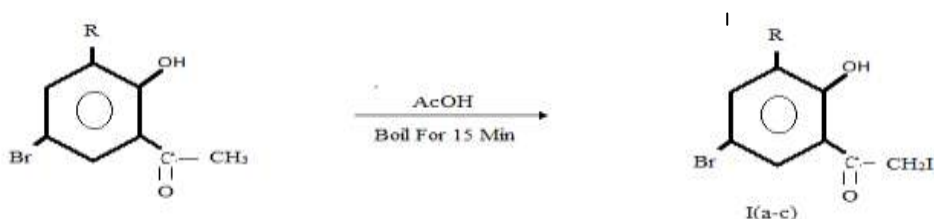
**Key words :** 2-hydroxy chalcones, 2-benzylidene coumaran-3-one

### INTRODUCTION –

2-Hydroxy chalcones in acetic acid reacts with  $Mn(OAc)_2$  affords 2-benzylidene coumaran-3-one (aurones)<sup>1</sup>. Chalcone reacts with mercuric acetate in DMSO solvent gives 2-benzylidene coumaran-3-one<sup>2,3,4</sup>. Chalcone bromide is kept in cold ethanol for 24 hours then treated with alkali gives aurones<sup>5,6</sup>. Some chalcones are directly oxidized by air to give aurones<sup>7</sup>. ω-bromo-2-hydroxy acetophenone condenses with substituted benzaldehyde in 40% NaOH affords 2-substituted benzylidene coumaran-3-one<sup>8,9</sup> ω-bromo-2-hydroxy acetophenone and substituted benzaldehyde dissolved in ethanol and the solution treated with triethanolamine<sup>10</sup>. Gives to 2-substituted benzylidene coumaran-3-one. ω-bromo acetophenone used for preparation aurones. Hence it was thought interesting to prepare ω-iodo acetophenone is used to prepare 2-benzylidene coumaran-3-one.

**Preparation of  $\omega$ - iodo-2-hydroxy-5-bromo acetophenone :**

2 -Hydroxy- 5- bromo acetophenone (0.01mole) was dissolved in 10 ml glacial acetic acid. To this mixture ICl in acetic acid (0.001mole) was added dropwise, with constant stirring boiled for 15 minutes, allowed to stand for 1 hour diluted with water. The mixture was extracted from benzene/ether. The benzene/ether was evaporated to get solid mass. Finally crystallized from ethanol to get  $\omega$ - iodo-2-hydroxy-5-bromo acetophenone (Ia) M.P. 46°C yield 78%. Similarly other compounds were prepared by above method. They are reported in Table-1.

**Properties of the compounds(Ia)**

1. It is brown coloured compound M.P. 46°C
2. Alcoholic solution of Ia gives red colouration with neutral ferric chloride solution indicates that it contains phenolic - OH group.
3. From the analytical data the molecular formula was found to be  $C_8H_6O_2BrI$ . The molecular weight being found to be 339 g.

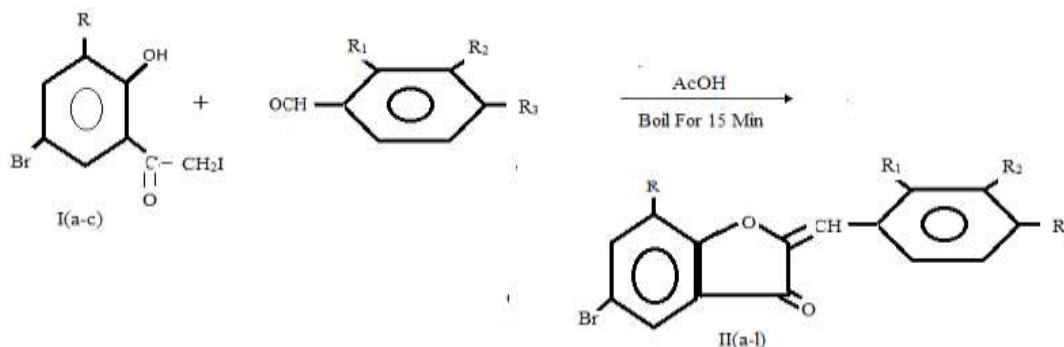
**Table-1:  $\omega$ - iodo-3 -substituted -5-bromo acetophenone**

Sr.No.	R	M.P. °C	Yield %
Ia	H	46	78
Ib	Br	95	62
Ic	$NO_2$	101	70

**Properties of 2-benzylidene- 5-bromo coumaran- 3- one :**

$\omega$ - iodo-2-hydroxy-5-bromo acetophenone (Ia) (0.01mole) and benzaldehyde (0.012mole) was dissolved in 20 ml ethanol. The solution warmed and 40% NaOH(6-8ml) added with constant stirring till red colour persist. The mixture was allowed to stand for 6 hours. Diluted the

solid the mass with 1:1 HCl solid separated crystallised from ethanol to get compound (IIa). M.P.88°C. Similarly other compound were prepared by above method and they are reported in the Table -2



### Properties of the compounds(IIa)-

1. It is golden yellow coloured crystalline compound M.P.88°C .
2. It gives negative ferric chloride test indicating involvement of phenolic - OH group in cyclization.
3. From the analytical data the molecular formula was found to be C<sub>15</sub>H<sub>9</sub>O<sub>2</sub>Br. The molecular weight being found to be 299.5 g.
4. The IR spectrum was recorded in nujol

1724	(C=O stretching in 5 membered ring)
1576.7, 1463.8	(C=C in aromatic ring)
1180	(C-O stretching) and
640	(C-Br stretching)

5. The PMR was recorded in CDCl<sub>3</sub> with TMS as internal standard

4.5δ	(S, H,	= CH-)
6.9 – 8.1 δ	(m, 8H,	Ar-H)

From chemical and spectral data compound (IIa) is 2-benzylidene- 5-bromo coumaran- 3- one

**Table-2 : Synthesized aurone M.P.(s), yield %**

Sr.No	R	R <sub>1</sub>	R <sub>2</sub>	R <sub>3</sub>	M.P. °C	Yield %
IIa	H	H	H	H	89	70
IIb	H	H	H	OCH <sub>3</sub>	152	65
IIc	H	OH	H	H	133	60
IId	H	H	NO <sub>2</sub>	H	160	72
IIE	Br	H	H	H	111	82
IIf	Br	H	H	OCH <sub>3</sub>	182	70
IIg	Br	OH	H	H	207	72
IIh	Br	H	NO <sub>2</sub>	H	170	73
Iii	NO <sub>2</sub>	H	H	H	151	55
IIj	NO <sub>2</sub>	H	H	OCH <sub>3</sub>	150	60
IIk	NO <sub>2</sub>	OH	H	H	88	62
III	NO <sub>2</sub>	H	NO <sub>2</sub>	H	173	65

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**REFERENCES :**

- Kurosawa Kerze, Bull. Chem. Soc .Japn 42,(5)
- Doifode S.K. And Doshi A.G. Orient. (1995) J.Chem. 11 (2),189-190.
- Kadu V.B. And Doshi A.G. Orient. (1997) J.Chem. 13 (3),281-284.
- Sahathrabuddhe A.S., (1992) Ph.D. Thesis, Nagpur University.
- Wheeler T.S.,et.al., (1935) Pruc.Ind.Acad.Sci.,2,439.
- Marathe M.G.J. (1952) Uni. Poona, 2,7.
- Simokoriyand M. (1957) J. Am. Chem.Soc. 79 and 399, (1958) ibid 75,1900.
- Doifode K.B. And Marathe M.G. (1964) J.Org.Chem., 29, 2025.
- Parajape M.V.And Wadokar K.N., (1981) Indian. J. Chem.20B, 808- 809.
- Soni. (1977) P.A. Study of Bromination and Debromination on flavanoids, chalcones and Aurones Ph.D.Thesis Nagpur University.