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*NOTE*

# Synthesis of New-2,3-Disubstituted Quinoxaline

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2-α DIbromo-2-(substituted benzyl)-3-nitro-5-chloro coumaran-3-one (**1a–p**) in methanol condensed with benzene-1,2-diamine in presence of few drops of concentrated $H_2SO_4$ affords 2,3-disubstituted quinoxaline (**IIa-p**). The structural elucidation of compounds were done on the basis of analytical and spectral data.

**Key words: Synthesis, 2,3-Disubstituted quinoxaline**

Quinoxalines are well known for their anti-bacterial[1] antitumor and anti-viral[2] properties. Earlier workers have reported various aziridinyl ketones and their cyclic anils by reaction of chalcone dibromide with benzene-1,2-diamine in presence of triethyl amine and their subsequent acid catalysed isomerisation to quinoxaline[3]. Similarly 2-monoalkyl amino and 2-dialkylamino-4-phenyl benzodiazopines are also reported[4]. Formation of novel Schiff bases contaning tricyclic (7 + 12 + 7) inner ring system has also been reported[5] in this reaction. Chalcone dibromide condensed with benzene-1,2-diamine to give quinoxaline. Aurone with hydrogen peroxide in alkaline methanol or dioxane gives aurone epoxide which on ring opening, gives 2, β aurone, isomeric with 1,2 diketone structure[6] which subsequently condense with BDA to give 2,3-disubstituted quinoxalines.

Chalcone dibromide or flavone on condensation with benzene-1,2-diamine gives 2 benzyl-3-phenyl quinoxaline[7, 8]. Aurone dibromide condense with benzene-1,2-diamine in presence of few drops of conc. $H_2SO_4$ affords quinoxaline[9]. Aurone dibromide are prepared by known method[10, 11].

Melting points were determined in an open capillary tubes and are uncorrected. IR spectra were recorded on Perkin-Elmer-557 Spectrophotometer. PMR spectra were recorded in $CDCl_3$ on a DRX AC 300 F Spectrophotometer at 300 MHz

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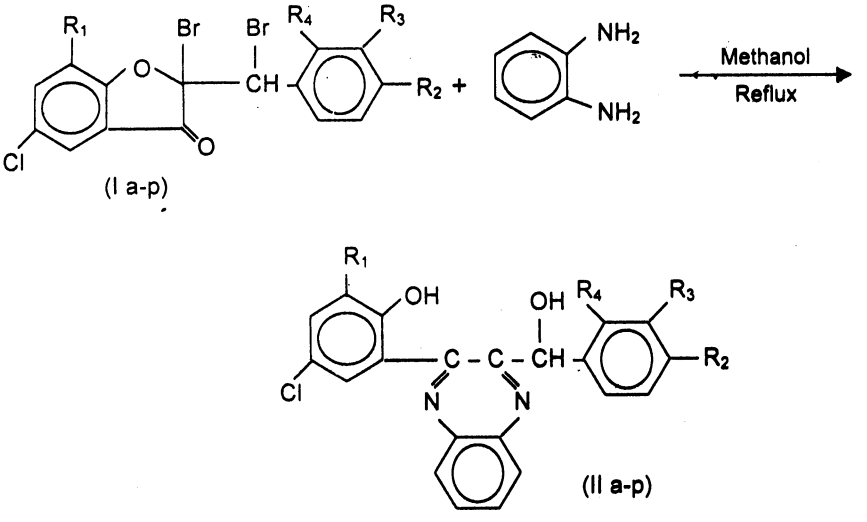
**Synthesis of 2-(2′-hydroxy-3′-nitro-5′-chlorophenyl)-3-(α″-hydroxy-4″-methoxy) benzyl quinoxaline (II a)**

2-α Dibromc-2-(4′-methoxy benzyl)-3-nitro-5-chloro-coumaran-3-one **(Ia)** (0.01 m) and benzene 1,2-diamine (0.01 mole) was dissolved in 25 mL methanol 2,3 drops of concentrated $H_2SO_4$ was added to it. The mixture was refluxed for 3 h, allow to cool, diluted with cold water with constant stirring. Resulting solid was extract with diethyl ether to get 2-(2′-hydroxy-3′-nitro-5′-chlorophenyl)-3-(α″-hydroxy-4″-methoxy)benzyl quinoxaline **(IIa)**.




**Properties of the Compound (IIa)**

It is yellowish green crystalline solid, m.p. 156°C. It shows positive ferric chloride test, indicating presence of phenolic hydroxy group. m.f. $C_{22}H_{16}N_3O_4Cl$; m.w. 421.7; elemental analysis %, found (calcd.): C = 58.32 (58.34), H = 3.76 (3.79), N = 10.22 (10.19) and Cl = 8.45 (8.42).

Infrared spectrum was recorded on Perkin-Elmer 557 Spectrophotometer: 3500–3400 (broad —OH gr. stretching); 1645 (singlet —C=N stretching); 1589 (symmetrical aromatic $—NO_2$ group); 1348 (unsymmetrical aromatic $—NO_2$ group); 1022 ($—O—CH_3$ group stretching); 764 $cm^{-1}$ (—C—Cl group stretching)

The PMR was recorded in $CDCl_3$ with TMS as an internal standard: 0.99 δ (1H, —OH); 1.15 δ (1H, —OH); 3.51 δ (3H, aromatic $—OCH_3$;) 4.86 δ (1H, —CH); 7.23–7.80 δ (10H, aromatic H).

These chemical and spectral data shows that compound **(IIa)** is 2-(2′-hydroxy-3′-nitro-5′-chlorophenyl)-3-(α″-hydroxy-4″-methoxy) benzyl quinoxaline.

Similarly other compounds were prepared by above method and reported in Table-1.

TABLE-1
SYNTHESIS, m.p. AND YIELD (%) OF DI-SUBSTITUTED QUINOXALINE

| Comp. No | $R_1$ | $R_2$ | $R_3$ | $R_4$ | m.p. (°C) | (% Yield) |
|---|---|---|---|---|---|---|
| **IIa** | $NO_2$ | $OCH_3$ | H | H | 156 | 70 |
| **IIb** | $NO_2$ | H | H | H | 140 | 68 |
| **IIc** | $NO_2$ | H | H | OH | 75 | 83 |
| **IId** | $NO_2$ | H | $NO_2$ | H | 138 | 75 |
| **IIe** | H | H | H | H | 57 | 84 |
| **IIf** | H | $OCH_3$ | H | H | 96 | 68 |
| **IIg** | H | H | H | OH | 102 | 60 |
| **IIh** | H | H | $NO_2$ | H | 155 | 70 |
| **IIi** | Br | H | H | H | 139 | 65 |
| **IIj** | Br | $OCH_3$ | H | H | 110 | 72 |
| **IIk** | Br | H | H | OH | 152 | 81 |
| **III** | Br | H | $NO_2$ | H | 149 | 70 |
| **IIm** | Cl | H | H | H | 96 | 59 |
| **IIn** | Cl | $OCH_3$ | H | H | 125 | 86 |
| **IIo** | Cl | H | H | OH | 143 | 65 |
| **IIp** | Cl | H | $NO_2$ | H | 110 | 75 |

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