

NOTE

Synthesis of Some New Phenolic Azo Schiff Bases—Part II

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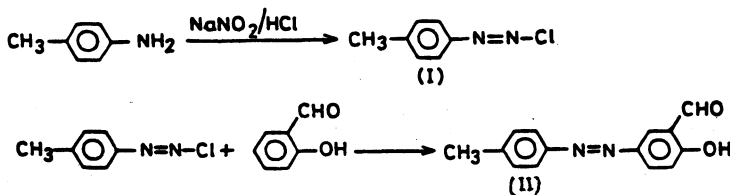
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p-Toluidine on diazotisation gives *p*-methyl benzene diazonium chloride (I) which on condensation with salicylaldehyde gives 2-hydroxy-5-(4-methyl phenylazo) benzaldehyde (II). This II on condensation with substituted aniline gives 2-hydroxy-5-(4-methyl phenylazo) benzyldine substituted aniline, *i.e.*, substituted phenolic azo Schiff bases.

Anticancer Schiff bases have been synthesised by condensation of aniline with substituted benzaldehyde¹. Benzaldehyde-2-hydroxy aniline was prepared by condensation of *o*-aminophenol with substituted benzaldehyde in ethanol in presence of 2-3 drops of concentrated H₂SO₄.² Benzoxazoles were prepared by cyclisation of O,N-diacetyl derivative of *o*-aminophenol at lower temperature by treating aminophenol with carboxylic acid or its derivative³. It was suggested that azomethine linkage might be responsible for biological activities of Schiff bases⁴. Some oxime derivatives having ethoxy group have been reported to possess antiinflammatory activities⁵. Some 2-oxothiazoline hydrazones from 3-methoxy, Δ -allyloxy-benzaldehyde are endowed with anti-HIV activity⁶. Some new azo pyrazoles have been synthesised by Jolly *et al.*⁷ Recently some new azo Schiff bases have been synthesised by the reaction of 5-*p*-methoxy phenylazo salicylaldehyde with primary aromatic amines⁸. Hence it was thought interesting to prepare 2-hydroxy-5-(4-methyl phenylazo) benzyldine substituted aniline from 2-hydroxy-5-(4-methylphenylazo) benzaldehyde..

Synthesis of 2-hydroxy-5-(4-methyl phenylazo) benzaldehyde.

p-Toluidine (0.01 M) was dissolved in aq. HCl (5 mL, 5 N) and diazotized by using sodium nitrite (0.88 M). A cold solution of salicylaldehyde (1 g, 6 N) in aqueous sodium hydroxide (8 mL, 2 N) was added slowly with constant stirring below 5°C. The resulting crude solid was washed with water and recrystallised from ethanol to get 2-hydroxy-5-(4-methyl phenylazo) benzaldehyde.

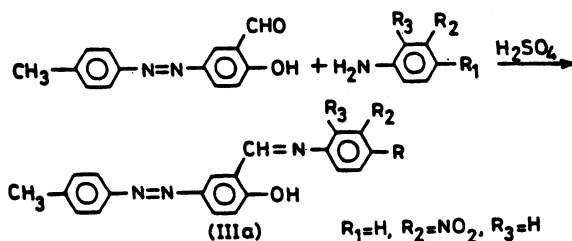


Compound II: m.w. = 240, m.p. = 152°C, Colour = Yellow

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Synthesis of 2-hydroxy-5-(4-methyl phenyl-azo) benzylidine *meta*-nitroaniline.

2-Hydroxy-5-(4-methyl phenylazo) benzaldehyde condensed with *meta*-nitroaniline in ethanolic medium in presence of conc. H_2SO_4 when compound IIIa was obtained.



Where $R_1 = H, R_2 = NO_2, R_3 = H$; Compound IIIa: m.f. = $C_{20}H_{16}N_4O_3$, m.w. = 360, m.p. = $181^\circ C$, Colour = Yellow; N %, Found (Calcd.) 15.3 (15.5)

TABLE-I
PHYSICAL DATA OF PHENOLIC AZO SCHIFF BASES

S.No.	R_1	R_2	R_3	m.p. $^\circ C$	Colour	m.w.
IIIa	H	NO_2	H	181	Yellow	360
IIIb	NO_2	H	H	185	Yellow	360
IIIc	H	H	NO_2	142	Yellow	360
IIId	CH_3	H	H	170	Dark brown	329
IIIe	H	CH_3	H	160	Orange Red	329
IIIf	H	H	CH_3	120	Brown	329
IIIg	H	H	H	145	Yellow brown	310

I.R. Spectrum of compound IIIa (Nujol): $3100 \nu(-OH)$, $1580 \nu(N=N \text{ azo phenyl})$, $1620 \nu(CH=N \text{ azomethane})$, 1530, 1550 ($ArNO_2$ asymmetric and symmetric stretching); MR (in $CDCl_3$), 2.1 (s, 3H $Ar.CH_3$) 6.3–7.8 (m, 11H, $Ar-OH$), 8.3 (s, 1H, $-CH=N$ azomethane), 12.1 (s, $Ar-OH$).

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