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NOTE

Synthesis and Characterization of 3,4,5-Trisubstituted Isoxazoline

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3-Aroyl flavanone **(III)** has been prepared by the condensation of dibenzoyl methane **(II)** with aromatic aldehyde in ethanol containing few drops of piperidine. The compound **(II)** on refluxing with $NH_2OH{\cdot}HCl$ in DMF containing small amount of piperidine gives 4-aroyl substituted isoxazoline **(IV)**.

Key Words: Synthesis, 3,4,5-Trisubstitued isoxazoline.

Isoxazolines have been synthesized[1, 2] by reaction with hydroxylamine hydrochloride in pyridine. Recently, isoxazolines have been synthesized by the reaction of $NH_2OH{\cdot}HCl$ on flavanone in DMF[3] medium containing little piperidine. It was interesting to prepare some new substituted 3,5-diaryl-4-aroyl substituted isoxazolines **(IV)** in DMF medium containing little piperidine. The structure of **(IV)** was confirmed on spectral and chemical data.

3-aroyl flavanone was obtained by condensation of dibenzoyl methane[4] **(II)** with aromatic aldehydes in ethanol containing small amount of piperidine[5]. The structure of **(III)** was confirmed on the basis of chemical and spectral data **(Scheme-1)**.

Synthesis of 1-(2′-hydroxy-4′-methyl-5′-chlorophenyl)-3-(2′-chlorophenyl)-1,3-propanedione (II)

2-Hydroxy-4-methyl-5-chloro acetophenone **(a)** (m.p. 55°C) on condensation with 2-chlorobenzoic acid in pyridine medium in presence of $POCl_3$ gives 2-(2′-chlorobenzoyloxy)-4-methyl-5-chloro acetophenone **(I)** (m.p. 135–136°C).

The Baker-Venkatraman transformation of 2-(2′-chlorobenzoyloxy)-4-methyl-5-chloro acetophenone **(I)** gives 1-(2′-hydroxy-4′-methyl-5′-chlorophenyl)-3-(2′-chlorophenyl)-1,3-propanedione **(II)**; yield 87%; m.p. 90–91°C; R_f 0.86; IR (KBr, cm^{-1}): 3094 ν(—OH); 1682.4–1528.9; ($\rangle$C=O ketones), 1282.3–929.3 ν($\rangle$C—O phenols), 761.8–629.5 ν(C—Cl); ^1H NMR ($CDCl_3$):

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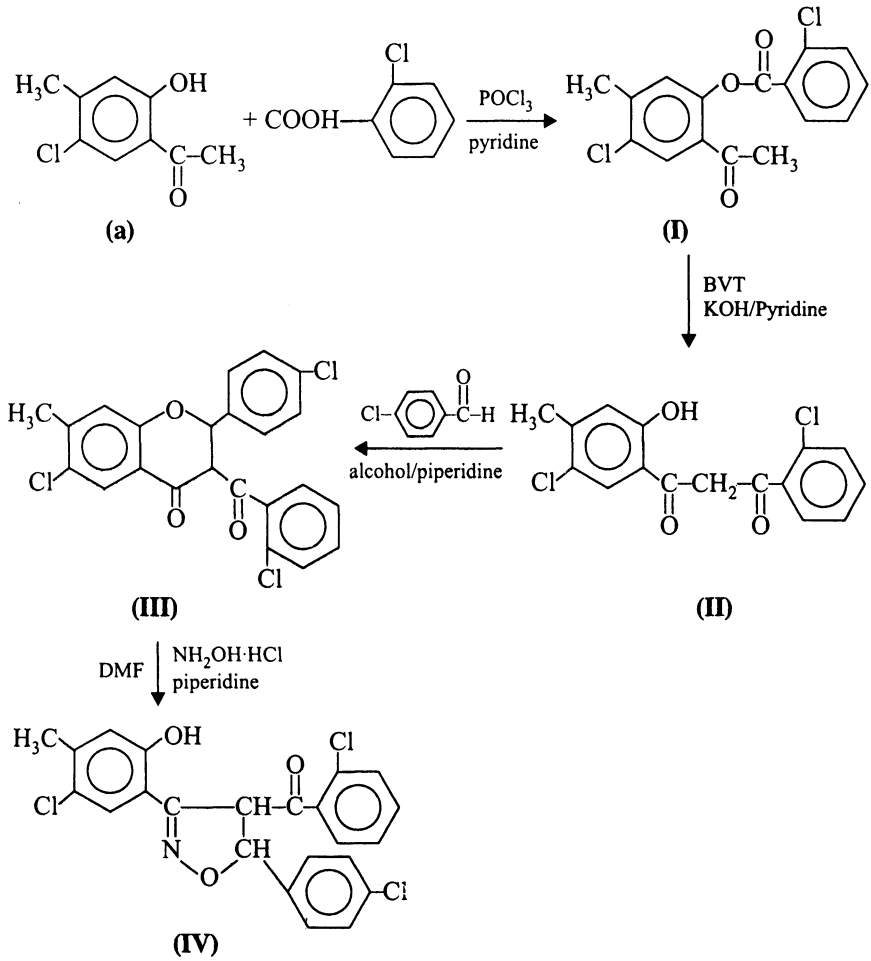
2.8 δ (S, 2H, —C(=O)—CH_2—C(=O)—), 2.5 δ (S, 3H, —CH_3), 12.25 δ (S, 1H, —OH), 7.5–8.5 δ (m, 6H, Ar—H).



Scheme-1

Synthesis of 4′-chloro-3-(2′-chlorobenzoyl)-6-chloro-7-methyl flavanone (III)

1-(2′-Hydroxy-4′-methyl-5′-chlorophenyl)-3-(2′-chlorophenyl)-1,3-propandione (**II**) (0.01 mol) and 4-chlorobenzaldehyde (0.01 mol) was refluxed in ethanol containing few drops of piperidine (0.5 mL) for 1 h. The reaction mixture on cooling gave white crystals, filtered and crystallized from ethanol to give **IIIa**; yield 64%; m.p. 152°C, R_f 0.69, IR (KBr, cm^{-1}): 1684 ν(C=O), 1591 ν(C=O), 1089 ν(C—O—C) and 760 ν(C—Cl); ^{1}H NMR ($CDCl_3$) : 2.35 δ (S, 3H,—CH_3), 5.1 δ (d, 1H, —CH of methine), 6.05 δ (d, 1H, —CH of methine) and 7.4–8.35 δ (m, 10H, Ar—H).

Synthesis of 3-(2′-hydroxy-4′-methyl-5′-chlorophenyl)-4-(2′-chlorobenzoyl)-5-(4′-chlorophenyl)isoxazoline (IV)

A mixture of **III** (0.01 mol) and hydroxylamine hydrochloride (0.02 mol) in DMF (20 mL) and few drops of piperidine (0.5 mL) was refluxed for 1.3 h. The reaction mixture was cooled, acidifed with (1 : 1) HCl, washed with $NaHCO_3$ (2%) solution and washed with water and crystallized from ethanol (50%) to give **IV**; yield 76%; m.p. 208°C; R_f 0.73; IR (KBr, cm^{-1}) : 3093 ν(—OH); 1799 ν(C=O), 1573 ν(C=O), 1491 ν(C=O), 1282 ν(C—N), 1175–1128 ν(C—O), 981 ν(C=N—O) and 820 ν(C—Cl); ^1H NMR ($CDCl_3$) : 2.6 δ (S, 3H—CH_3), 5.2 δ (d, 1H, —CH), 5.9 δ (d, 1H, —CH), 7.35–8.35 δ (m, 10H, ArH).

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