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Study of some New 3-(2-Hydroxy-5-Methyl phenyl)-5,5-Dialkyl/5,5-Diaryl/5-ARYL ISOXAZOLINES

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Abstract

In this paper Seven different chalcones I(a)-I(g) were synthesised by literature method from 2-hydroxy-5-methyl acetophenone. They reacted with hydroxyl-amine hydrochloride in pyridine to get seven 3-(2-hydroxy-5-methylphenyl)-5,5-dialkyl/5,5-diaryl/5-aryl-isoxazolines II(a)-II(g). The structure of these compounds were confirmed on the basis of chemical and spectral analysis.

Keywords: 3-(2-Hydroxy-5-Methyl phenyl)

Introduction

Isoxazolines can effectively used antibacterial¹, antitubercular, antiviral, antifungal, herbicidal and insecticidal agents²⁻⁵. Synthesis of isoxazolines has been reported by the action of $\text{NH}_2\text{OH}\cdot\text{HCL}$ on hydroxyl-chalcones and flavanones⁶. Borkhades⁷ synthesised isoxazolines from o-hydroxychalcones and corresponding flavanones. Gimil Aziz et.al.⁸ synthesised isoxazolines from flurochalcones. Jamode⁹ reported the synthesis of 3,5-diaryl isoxazolines using ethylenediamine from 2'-hydroxy chalcones, flavanones and 3-arylidine flavanones. Rajput¹⁰ and Jamode have synthesised 3,5-diaryl isoxazolines from 2'-hydroxy-3-chlorochalcones in ethanol containing piperidine. Kedar¹¹ have reported synthesis of 3,5-diarylisoxazolines in EtOH in presence of alkali. We tried to synthesise 3-(2-hydroxy-5-methylphenyl)-5,5-dialkyl/5,5-diaryl/5-aryl isoxazolines using pyridine.

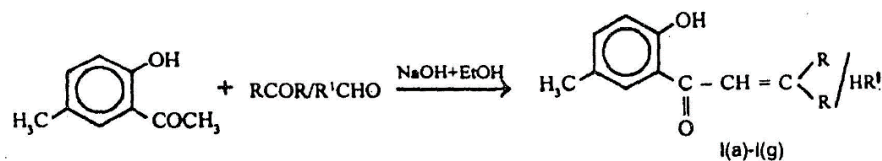
Experimental

Melting points of all compounds were determined on Tempo Melting Point apparatus and are uncorrected. Compounds I and II were prepared in laboratory by known methods, m.p., purity of compounds were checked by TLC on silica-gel G plates.

The structures of compounds I(c), I(a), II(a) and II(g) were confirmed by chemical analysis, IR and NMR spectra.

Synthesis of compounds I(a)-I(g)

The compounds were synthesised by known literature method from 2-hydroxy-5-methylacetophenone with two ketones and five aromatic aldehydes in ethanol in presence of alkali. Physical data of compounds I(a)-I(g) are given in Table-1



where $\text{C} \begin{array}{l} \diagup \text{R} \\ \diagdown \text{R}'/\text{HR}' \end{array}$ as shown in Table 1.

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Table 1: Physical data of compounds I(a)-I(g)

S. No.	Comp. No.	$\langle R / R^1 \rangle$	Molecular formula	m.p. ^o c	Yield (%)
1.	I(a)		C ₁₂ H ₁₄ O ₂	138	70
2.	I(b)		C ₂₂ H ₁₈ O ₂	90	72
3.	I(c)		C ₁₆ H ₁₄ O ₂	98.5	76
4.	I(d)		C ₁₇ H ₁₆ O ₃	100.5	81
5.	I(e)		C ₁₇ H ₁₂ O ₃	86	69
6.	I(f)		C ₁₈ H ₁₆ O ₂	133	68
7.	I(g)		C ₁₆ H ₁₃ O ₄	114	72

IR Absorption observed (cm⁻¹) for I(c): 3021 (s) strongly hydrogen bonded-OH stretching, 1639(s) C=O stretching, 1569(s) C-CH=CH stretching, 1267(d) Ar-O stretching, 1184 C-O stretching in phenol.

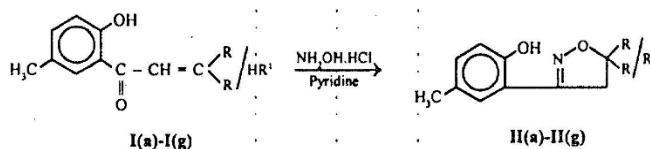
NMR for I(c):(2.35s, 3H,AR-CH₃) (6.9-7dd, 1H, CH_A),

(7.33 dd, 1H, =CH_B), (12.61, s, 1H, OH phenolic) (7.35-7.95(m), 8H, Ar-H)

IR for I(a) (m¹): 3306 -Hbonded -OH, 1600 -CH stretching, 1495 -C=C- in aryl, 1700 -C=O stretching, 1306 =C(CH₃)₂.
NMR for I(a): (2.25-2.35 s), 9H, Ar-CH₃ and =C(CH₃)₂, (6.8-7.3, m, 3H, Ar-H), (7.85, d, 1H, C-H), (11.25 Ar-OH)

Synthesis of compound II(a)-II(g)

The chalcones I(a)-II(g) (0.01 M) were refluxed in 20 ml pyridine using (0.02 M) NH₂OH.HCl for 3 to 4 hours. Further processing as per known literature method¹¹ to give seven [II(a)-II(g)] isoxazolines from corresponding chalcones I(a)-II(g). The physical data for II(a)-II(g) are given in Table 2.



where $\langle R / HR^1 \rangle$ as shown in Table 2

Table 2: Physical data of compounds II(a)-II(g)

S. No.	Compd. No.	$\langle R / R^1 \rangle$	Molecular formula	m.p. ^o c	Yield (%)
1.	I(a)		C ₁₂ H ₁₅ NO ₂	111.5	70
2.	I(b)		C ₂₂ H ₁₉ NO ₂	131.5	69
3.	I(c)		C ₁₆ H ₁₅ NO ₂	162	66
4.	I(d)		C ₁₇ H ₁₇ O ₃ N	173	70
5.	I(e)		C ₁₄ H ₁₃ O ₃ N at 220 liquified and polymerise (blue-black)	120 (decomposes)	60
6.	I(f)		C ₁₈ H ₁₇ O ₂ N	73 (decomposes)	58
7.	I(g)		C ₁₆ H ₁₄ O ₄ N ₂	142	74

IR for compound II(a): (Absorption observed in cm⁻¹)-3333 strong hydrogen bonded -OH, 3038 C-H stretching aromatic, 1636(s) CH₂ stretching, 1686(s) C=N stretching, 1252-1230(m) Ar-O, 1035-1130(d) C(CH₃)₂.

NMR for compound II(a): (Observed peaks in δ)-(2.3(s), 9H, Ar-CH₃ and C(CH₃)₂), (4.7 (dd, 1H, CH_A), (5.15 (dd, 1H, CH_B) (6.8-7.3 m, 3H, Ar-H) (7.85 s, 1H, Ar-OH).

IR for compound II(g): 3244-3651 strongly bonded OH stretching, 2918(s) C-H aromatic stretching, 1617.8(s) CH₂ stretching, 1559 (s) C=N stretching, 1532.5 and 1347.5(s) due to NO₂ group, 1013-1171 (m) CH₃, 973-950(d) C=N-O stretching.

NMR for compound II(g): (2.35(s), 3H, Ar-CH₃), (2.75(dd), 1H, CH_A), (3.65(dd), 1H, CH_B), (5.17(dd), 1H, >CH_X), (6.83-8.25 m, 7H, Ar-H), (8.4(s), 1H, Ar-OH).

Mechanism

Formation of isoxazoline¹² involves 1,2 addition of NH₂OH to carbonyl group giving an adduct. The adduct then loses water molecule to give mono-oxime which on cyclisation and rearrangement gives isoxazoline.

References

1. Ankiwala MD, Hathi MV. J INd Chem Soc 1994;71(9):587-89.

- Robert Howek US. 4, 139, 366, Chem Abstr 1979;90:16958w.
- Antibiot J. (Tokyo) 1992;45:1930
- Kedar RM, Vidhale NN, Chincholkar MM. Orient J Chem 1996, 301.
- Kedar RM. *et al.*, Orient J Chem 1997, 143.
- Sammour AB, Mhd Elkasaby. J Chem UAR 1969;12(1).
- Borkhade KT, Marathe MG. Indian J Chem 1970;8:796.
- Aziz G, Nosser MH, Doss NL, Rizk AB. Indian J Chem 1976;148:266.
- Jamode VS. Ph.D. Thesis "Synthetic studies of Nitrogen and Oxygen heterocyclic compounds", Nagpur University, 1977.
- Rajput PR. "Synthetic of Nitrogen and Oxygen heterocyclic compounds", Ph.D. Thesis, Amravati University 1993.
- Kedar RM. "Synthetic of Oxygen-Nitrogen Sulphur heterocyclic compounds and their physico-chemical studies" Ph.D. Thesis, Amravati University 1998.
- Barnes RP. Spriggs, A.S., J Am Chem Soc 1945;67:134.