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Study of some new 3-(2-hydroxy-5-methylphenyl)-5, 5-dialkyl/5, 5-diaryl/5-aryl isoxazolines

Dr. Pushpa Kumari

Abstract
The different chalcones I(a)–I(g) were synthesised by literature method from 2-hydroxy-5-methyl acetophenone. They were reacted with hydroxyl-amine hydrochloride in Pyridine to get seven 3-(2-hydroxy-5-methylphenyl)-5-dialkyl/5-diaryl/5-aryl-sixazolines II(a)-II(g). The structures of these compounds were confirmed on the basis of chemical and spectral analysis.

Keywords: Isoxazolines, 2-hydroxy-5-methylphenyl, NH₂OH, HCl

Introduction
Isoxazolines has been reported by the action of NH₂OH, HCl on hydroxy-chalcones and flavanones. Brokhade-synthesised isoxazolines from O-hydroxychalcones and corresponding flavanones. Gimil Aziz et al. [1–3] synthesised isozabolines from flurochalcones. Jamode [4] repored the synthesis of 3, 5-diaryl isoxazolines using ethylenediamine from 2’-hydroxy chalcones, flavanones and 3-arylidine flavanones. Rajput [5] and Jamode have synthesised 3, 5-diaryl isoxazolines from 2-hydroxy-3-chlorochalcones in ethanol containing piperidine. Kedar” have reported synthesis of 3, 5-diarylisoxazolines in EtOH in presents of alkali. I tried to synthesise 3-(2-hydroxy-5-methylphenyl)-5, 5-dialkyl/5-aryl isoxazolines using pyridine.

Material and Method
Melting points of all compounds were determined on Tempo Melting Point apparatus and are uncorrected. Compound 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 structure 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 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.

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), (6.9-7 dd. 1H, CH₃), (7.33 dd, 1H, = CH B), (12.61, s, 1H, OH phenolic) (7.35-7.95(m), 8H, Ar-H)
**IR for I(a) (cm⁻¹):** 3306 -H bonded -OH, 1600 –CH=C stretching, 1495 -C=C- in aryl, 1700-C=O stretching, 1036= C (CH₃)₂.

**NMR for I(a):** (2.25-2.35s). 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)

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Compd. No.</th>
<th>R ( / ) R'</th>
<th>molecular formula</th>
<th>m.p. °C</th>
<th>Yield (%)</th>
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<tr>
<td>1.</td>
<td>I(a)</td>
<td>C₁₂H₁₄O₂</td>
<td>136</td>
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<td>70</td>
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<tr>
<td>2.</td>
<td>I(b)</td>
<td>C₂₂H₁₈O₂</td>
<td>90</td>
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<td>71</td>
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<tr>
<td>3.</td>
<td>I(c)</td>
<td>C₁₄H₁₄O₂</td>
<td>98.3</td>
<td></td>
<td>74</td>
</tr>
<tr>
<td>4.</td>
<td>I(d)</td>
<td>C₁₇H₁₆O₃</td>
<td>100.3</td>
<td></td>
<td>81</td>
</tr>
<tr>
<td>5.</td>
<td>I(e)</td>
<td>C₁₄H₁₄O₂</td>
<td>84</td>
<td></td>
<td>67</td>
</tr>
<tr>
<td>6.</td>
<td>I(f)</td>
<td>C₁₄H₁₄O₂</td>
<td>131</td>
<td></td>
<td>66</td>
</tr>
<tr>
<td>7.</td>
<td>I(g)</td>
<td>C₁₄H₁₃O₄</td>
<td>112</td>
<td></td>
<td>70</td>
</tr>
</tbody>
</table>

*All compounds I(a)-I(g) gave satisfactory elemental analysis.

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Compd. No.</th>
<th>R ( / ) R'</th>
<th>molecular formula</th>
<th>m.p. °C</th>
<th>Yield (%)</th>
</tr>
</thead>
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<td>1.</td>
<td>I(a)</td>
<td>C₁₂H₁₅NO₂</td>
<td>111.3</td>
<td></td>
<td>68</td>
</tr>
<tr>
<td>2.</td>
<td>I(b)</td>
<td>C₂₂H₁₉NO₂</td>
<td>131.3</td>
<td></td>
<td>67</td>
</tr>
<tr>
<td>3.</td>
<td>I(c)</td>
<td>C₁₆H₁₅NO₂</td>
<td>160</td>
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<td>64</td>
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<tr>
<td>4.</td>
<td>I(d)</td>
<td>C₁₇H₁₇O₃N</td>
<td>171</td>
<td></td>
<td>68</td>
</tr>
<tr>
<td>5.</td>
<td>I(e)</td>
<td>C₁₄H₁₃O₃N</td>
<td>120 (decomposes)</td>
<td></td>
<td>58</td>
</tr>
<tr>
<td>6.</td>
<td>I(f)</td>
<td>C₁₄H₁₃O₂N</td>
<td>73 (decomposes)</td>
<td></td>
<td>56</td>
</tr>
<tr>
<td>7.</td>
<td>I(g)</td>
<td>C₁₆H₁₄O₄N₂</td>
<td>140</td>
<td></td>
<td>73</td>
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</table>

*All compounds II(a)-II(g) gave satisfactory elemental analysis.
Synthesis of Compounds II(a)-II(g)
The chalcones I(a)-I(g) (0.01M) were refluxed in 20ml pyridine using (0.02M) NH₂OH, HCL for 3 to 4 hours. Further processing as per known literature method⁴ to give seven [II(a)-II(g)] isoxazolines from corresponding chalcones I(a)-I(g). The physical data for II(a)-II(g) are given in above table Table.

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 C=N stretching, 1252-1230 (m) Ar-O, 1035-1130 (d) C (CH₃)₂.

NMR for compound II(g): (Observed peaks in δ)- (2.3(s), 9H, Ar-CH₃ and C(CH₃)₂), (4.7 (dd, 1H, CH₈), (5.15 (dd), 1H, CH₉) (6.8-7.3 m, 3H, Ar-H) (7.85 S, 1H, Ar-OH).

IMR for compound II(g): 3244-3651 strongly ended OH stretching, 2918(s) C-H aromatic stretching, 1615.8(s) C-H₂ stretching, 1557 (s) C=N stretching, 1532.5 and 1345.3 and 1345.3(s) due to NO₂ group, 1013-1171(m) CH₃, 973-950(d) C=N-O stretching.

NMR for compound II(g): (2.33(s), 3H, Ar-CH₃), (2.73 (dd), 1H, (CHH₈), (3.65 (dd), 1H, CH₉H₁)(5.17 (dd), 1H, > CH₉), (6.83-8.25m, 7H, Ar-H), (8.4(s), 1H, Ar-OH).

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

References