A new synthetic route to 4-arylidene-2-phenyl-2-imidazolin-5-ones

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Condensation of aromatic aldehydes with benzoylglycine amide in saturated potassium carbonate solution yields imidazolinones. Eight imidazolinones have been synthesized by this method in 44-60% yields. The reaction follows pathway (i) which has been proved by isolation and identification of the intermediate (IA) formed.

4-Arylidene-2-imidazolin-5-ones are an important class of compounds as they can be converted into amino acids and their derivatives by reduction and hydrolysis. In the present work a new method for their synthesis is described. Benzoylglycine amide and an aromatic aldehyde on boiling for 3 hr in saturated aq. solution of potassium carbonate yielded 4-arylidene-2-phenyl-2-imidazolin-5-ones (Table I). The reaction may follow either pathway (i) (condensation followed by cyclization) or pathway (ii) (cyclization followed by condensation) (Scheme I). The reaction occurred by pathway (i) was proved as follows. In the condensation of benzaldehyde the intermediate IA could be isolated and identified as α-benzoylaminocinnamic acid amide. An authentic sample of this compound was prepared by heating the azlactone of α-benzoylaminocinnamic acid and ammonia in alcohol. The IR spectrum and melting point of these compounds were identical and mixed melting point did not show any depression.

Wieland and Biener obtained IB by heating benzoylglycine amide to 170°C in presence of a base. But under the reaction conditions we employed the reaction did not follow pathway (ii). It was proved by heating benzoylglycine amide alone under reflux for 3 hr in saturated potassium carbonate solution. No colour change was observed even after heating for 3 hr. Formation of IB would have resulted in red colouration.

The identity of all the imidazolinones prepared were confirmed from their melting points, undepressed mixed melting points and comparison of their IR spectra with authentic samples.

Experimental Section
Benzoylglycine amide. This starting material was prepared by the benzoylation of glycine amide under Schotten-Baumann conditions. Glycine amide for this
purpose could be prepared by the reaction between glycine ethyl ester and methanolic ammonia. Glycine amide (14.8 g, 0.2 mole) was dissolved in ice cold 10% NaOH solution (100 mL) and to this was added benzyol chloride (25.8 mL, 0.22 mole) in five equal portions and the mixture shaken well under the tap so that the temperature could be kept low. After cooling in ice for 2 hr the product was filtered and washed thrice with 5 mL portions of water and dried in the oven, m.p. 180-82°C (Reported m.p. 183°C).

4-Arylidene-2-phenyl-2-imidazolin-5-ones 2. Finely powdered benzyolglycine amide (1.96 g, 0.011 mole), aromatic aldehyde (0.01 mole) and saturated potassium carbonate solution (20 mL) were taken in a 50 mL round-bottomed flask and heated under reflux (in the case of solid aldehydes they were ground along with benzyolglycine amide). Within half an hour yellow crystals of imidazolone began to separate. After 3 hr of refluxing the reaction mixture was allowed to cool to room temperature and the product filtered. It was washed thoroughly with water and finally with 10 mL of ethanol and dried in the oven.

α-Benzoylaminocinnamic acid amide. The procedure under 4-arylidene-2-phenyl-2-imidazolin-5-ones was followed using benzaldehyde. After refluxing for 15 min the reaction mixture was cooled and filtered to get dull white crystals. Alternatively it was prepared by heating azalactone of α-benzoylaminocinnamic acid and alcoholic ammonia. Both the samples melted at 223°C.

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References

4. Erlenmeyer E, Ber, 33, 1900, 2036.