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Studies in the Synthesis of Substituted Furoinbenzoinoximes

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ABSTRACT

Recently, the synthesis of furoinoxime, furoinhydrazone, furoinphenylhydrazone, furoinsemicarbazone were synthesized by the interactions of furoinbenzoin with hydroxylamine hydrochloride, hydrazine hydrate, phenyl hydrazine and semicarbazide hydrochloride in presence of aqueous sodium hydroxide in DMF-water (80%) medium respectively. The synthesis of furoinbenzoin were carried out by the known literature method. The structure of all the synthesized compounds were justified on the basis of chemical characteristics, elemental and I.R. and NMR spectral analysis.

Introduction

Benzoin and oxime nucleus containing heterocyclic compounds possess pharmaceutical, medical, agricultural, industrial significance.[1-10] The important reactions of carbonyl with hydroxylamine, semicarbazide and various hydrazines were briefly studied in presence of strong base in ethanol medium.[11-14] As benzoin is bifunctional molecule having two reactive sites such as carbonyl and alcoholic groups and phenul ring can also be substituted. The condensation of carbonyl group of benzoin molecule with various amino compounds has been explored for the synthesis of new heterocycles. As these heterocycles possess more stability, specific geometry hence these heterocycles can be easily used for the synthesis of various important co-ordinate molecules. (Scheme-I)

While furoinbenzoinoximes, furoinbenzoin hydrazone, furoinphenyl hydrazone and furoinbenzoin semicarbazone were synthesized by the interaction of furoinbenzoin with hydroxylamine hydrochloride, hydrazine hydrate, phenyl hydrazine and semicarbazide hydrochloride in presence of aqueous sodium hydroxide in DMF-Dioxane-water (80%) medium respectively. (Scheme-II)

Experimental

The melting point of the all the synthesized compounds were recorded using hot paraffin bath. The carbon and hydrogen analysis were carried out on Carlo-Ebra 1106 analyser. Nitrogen estimation was carried out on Colman-N-analyzer-29. IR spectra were recorded on Perkin Elmer spectrometer in range 4000-400 cm^{-1} in KBr pellets. PMR spectra were recorded on Brucker AC 300F spectrometer with TMS as internal standard using $CDCl_3$ and DMSO-d_6 as solvent. The purity of compounds was checked on silica Gel-G pellets by TLC with layer thickness of 0.3 mm. All chemicals used were of AR grade.

Preparation of Furoin (VI)

Furoin was synthesized by refluxing furaldehyde (V) in presence of NaCN in alcoholic medium for three hours on water bath, during refluxing reddish brown colour of the reaction mixture was converted to dark reddish brown. This reaction mixture was poured in ice-cold water when a reddish brown sticky semisolid was obtained. It was separated from water and kept in dessicator containing anhydrous calcium oxide for one hour, then the sticky semisolid product was dissolved in alcohol. It was again refluxed on water bath for half an hour and the reaction mixture was poured in ice-cold water. It was acidified by dilute HCl, the brown colour crystals were separated out, yield 78%, melting point 128°C.

Examination of the Product

It is reddish brown crystalline solid having melting point 128°C. It gave positive test for nitrogen. It gave positive test for alcoholic (□OH) group. It gave satisfactory elemental analysis for carbon and hydrogen. As the compound was known and gave satisfactory chemical and elemental analysis.

Synthesis of Furoinbenzoineoxime (VII)

Furoinoxime (VIII) was synthesized by refluxing furoin (VI), hydroxylaminehydrochloride (IV) in 1:1 molar proportion in presence of aqueous sodium hydroxide in ethanol-water mixture for two hours. After two hours this reaction mixture was poured in ice-cold water to obtain reddish brown crystals of furoinoxime (VIII), yield79%, melting point 207°C. (Scheme-I)

Elemental Analysis

C [(found 56.78%) calculated 57.97], H [(found 3.28% calculated 4.34%], N [(found 5.56% calculated 6.76].

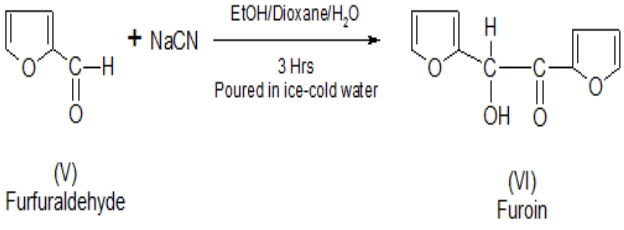
IR Spectra

The spectra was carried out in KBr pellets and the important absorption can be correlated as (cm^{-1}) 3395.4 (O□H stretching), 1651.2 (C=N stretching in oxime), 1420 (C□O stretching).

NMR Spectra

The spectrum was carried out in $CDCl_3$ and DMSO-d_6. This spectrum distinctly displayed the signals due to □NOH protons at □ 6.4652-6.6430 ppm, alcoholic protons at □ 3.3054 ppm, □CH protons at □ 1.2541 ppm.

Scheme - I



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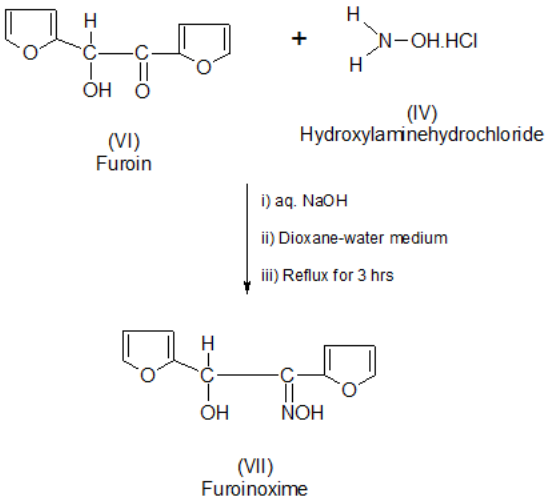

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