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RESEARCH ARTICLE

STUDIES IN THE SYNTHESIS OF SUBSTITUTED 2-HYDROXYBENZOINOXIMES

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ABSTRACT

Recently, the synthesis of 2-hydroxybenzoinoxime, 2-hydroxy-benzoinhydrazone, 2-hydroxybenzoinphenylhydrazone and 2-hydroxybenzoin-semicarbazone with hydroxylamine hydrochloride, hydrazine hydrate, phenyl hydrazine and semicarbazide hydrochloride in presence of aqueous sodium hydroxide in DMF-water (80%) medium respectively. Similarly, 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 2-hydroxybenzoin and 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.

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INTRODUCTION

Benzoin and oxime nucleus containing heterocyclic compounds possess pharmaceutical, medical, agricultural, industrial significance (Paria and Majumdar, 1979; Ali and Hafez, 1994; Saarinem and Orama, 1998; Chandra, 2001; De *et al*., 2009; Prasad and Agrawal, 2008; Naik *et al*., 2008; Monfared *et al*., 2009; Starikor *et al*., 2009. The important reactions of carbonyl with hydroxylamine, semicarbazide and various hydrazines were briefly studied in presence of strong base in ethanol medium (Pavia *et al*., 2004; Hegde *et al*., 2007; Hassan *et al*., 2005. 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. The present work described suitable and somewhat direct method for the synthesis of new series of substituted 2-hydroxybenzoin hydrazone, 2-hydroxybenzoin phenylhydrazone by the condensation of 2-hydroxyamino-benzoin with hydroxylamine hydrochloride and semicarbazide hydrochloride in presence of aqueous sodium hydroxide in DMF-water, Dioxane-water (80%) medium respectively. (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)

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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.

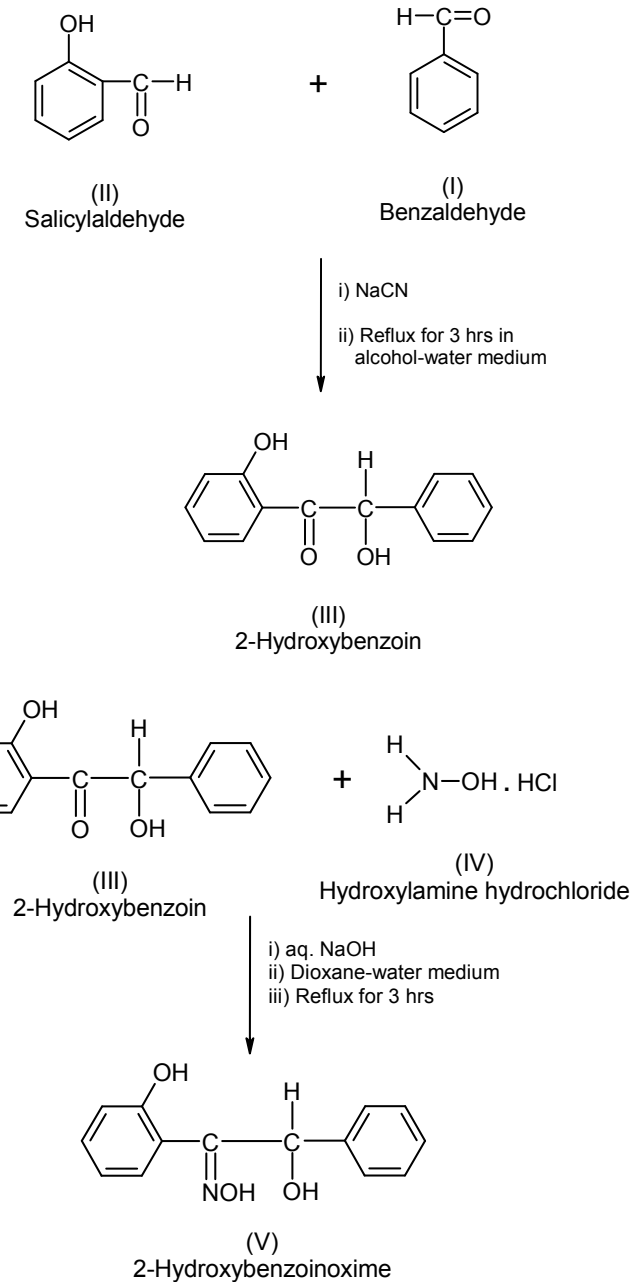
Preparation of 2-hydroxybenzoin (III)

2-Hydroxybenzoin (III) was synthesized by refluxing benzaldehyde (I), salicylaldehyde (II) in presence of NaCN in alcohol-water medium for one hour on water bath.

Table 1

Sr. No.	2-Hydroxybenzoinoxime (VII)	Colour	Yield (%)	m.p. (°C)
1.	2-Hydroxybenzoin hydrazone (VIII)	Brown	80	79
2.	2-Hydroxybenzoinphenyl hydrazone (IX)	Light brown	70	80
3.	2-Hydroxybenzoin semicarbazone (X)	Brown	50	82

Scheme I



This reaction mixture was poured in ice-cold water to obtain sticky semisolid. The sticky product was kept in dessicator containing anhydrous calcium oxide for half an hour, after that it was dissolved in alcohol. The alcohol was distilled off to isolate yellowish product. It was again refluxed in 80% dioxane-water medium on water bath for one hour and the reaction mixture was again poured in ice-cold water. After acidification with dilute hydrochloric acid, the yellow crystals were obtained, they were recrystallized with alcohol, yield 79%, m.p. 80°C (Scheme-I).

Examination of the product

It is yellow crystalline solid having m.p. 80°C. It gave positive test for alcoholic (–OH) group. It gave positive test with sodium bisulphate which clearly indicate presence of carbonyl group. It gave satisfactory elemental analysis.

Synthesis of 2-hydroxybenzoinoxime (V)

2-Hydroxybenzoinoxime (V) was synthesized by refluxing 2-hydroxybenzoin (III), hydroxylamine hydrochloride (IV) in 1:1

molar proportion in presence of aqueous sodium hydroxide in dioxane-water mixture for half an hour. This reaction mixture was then poured in ice-cold water to obtain yellow coloured 4-dimethylamino benzoinoxime (VIII), yield 79%, m.p. 95°C.

Examination of the product

It is yellow crystalline solid having m.p. 95°C. It gave positive test for nitrogen. It gave positive test for alcoholic group. Elemental Analysis: C [(found 70.09%) calculated 71.11], H [(found 5.18% calculated 6.66%], N [(found 10.36% calculated 10.37].

IR Spectra: The spectra was carried out in KBr pellets and the important absorption can be correlated as (cm^{-1}) 3412 (O–H stretching), 3068 (Ar–H stretching), 1666 (C=N stretching in oxime), 1450.1 (C=C stretching), 1463.5 (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 Ar–H protons at δ 9.1054 ppm, Ar–H protons at δ 7.5503-6.6787 ppm, =NOH proton at δ 5.61-5.133 ppm, –CH proton at δ 2.1690 ppm. Similarly, reaction between 2-hydroxybenzoin (III) with hydrazine hydrate (VI), phenyl hydrazine (VI), semicarbazide hydrochloride (VII) were carried out as mentioned above. The products obtained were given below in Table 1.

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