



Research Article

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## **Microwave Assisted Synthesis and Characterization of Schiff Base of 2-Amino Benzimidazole**

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### **ABSTRACT**

New Schiff base (2-[(1H-benzimidazol-2-ylimino) methyl]-4,6- diiodophenol) was synthesized by the condensation of aryl/hetero aromatic aldehyde (3,5-diiodosalicylaldehyde) with 2- amino benzimidazole under conventional and microwave conditions and characterized through IR, HNMR and Mass spectral data and CHN analysis.

**Keywords:** Microwave assisted synthesis, Conventional, Diiodosalicylaldehyde, Benzimidazoles.

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### **INTRODUCTION**

In the recent year microwave assisted organic reaction has emerged as new tool in organic synthesis. The reactions are very fast and are completed within short times and purity enhanced as compared with conventional heating methods. [1-3] Among others, microwave (MW)-assisted methodology has matured and can be used safely to substitute for conventional laboratory heating techniques; a number of chemical transformations have been achieved thereby improving many existing protocols with superior results when compared to reactions performed under conventional

heating conditions. Benzimidazoles derivative are related numerous varieties of pharmacokinetic and pharmacodynamics properties. Specifically, this nucleus is a constituent of vitamin B<sub>12</sub>. The medicinal activities of the Benzimidazoles containing moiety have been well documented Albendazole, Mebendazole. [4-5] Benzimidazoles nucleus is one of the bioactive heterocyclic compounds that exhibit a wide range of biological activities. These biological activities include anti-cancer [6-7], bactericidal [8], fungicidal [9-11], analgesic [12-13] and anti-viral properties. [14-16] Some have Anti-hypertensive activity [17] while some derivatives have

been synthesized and evaluated for inhibition of HIV-1 infectivity. [18-19]

Compounds containing azomethine group (-HC=N-) referred to as schiff base. They are condensation product of ketones or aldehydes with primary amines and were initial reportable by Victor Hugo Schiff in 1864. [20] Schiff bases are biological active compounds they possess lots of biological activities like anticancer [21], plant growth inhibitors [22] insecticidal [23], antidepressant [24], antibacterial [25], anti-inflammatory [26], anti-tuberculosis [27], antimicrobial [28] and anticonvulsant drug activity. [29]

Schiff bases have sort of applications viz., synthetic use, identification, detection and determination of aldehydes or ketones, purification of carbonyl or amino compounds, or protection of these groups throughout sophisticated or sensitive reactions. [30]

In present investigation, we synthesized Schiff base from 2-amino benzimidazoles and 3,5 diiodosalicylaldehyde in the presence of acetic acid as catalyst in ethanol medium under microwave irradiation. We aimed to the synthesis of new Schiff bases using microwave irradiation due to easy to workup, improved better yield as well as completion of the reaction time is less.

## MATERIALS AND METHODS

All the used chemicals and solvents were of Analytical grade. All the reagents used for the preparation of the Schiff bases were obtained from Sigma Aldrich. Melting point was determined in open capillary and is uncorrected. The IR studies of the schiff were recorded with 3000 Hyperion Microscope with Vertex 80 FTIR System in KBr pellets or Nicol phase from 4000  $\text{cm}^{-1}$  to

200  $\text{cm}^{-1}$  at SAIF IIT Mumbai. Elemental analysis was carried out on Flash EA 1112 series Elemental Analyser System from IIT, Mumbai. The mass spectra of a Schiff base in this study were recorded at SAIF IIT Madras by (GC-MS Spectrometer Model Joel GC Mate.  $^1\text{H}$ NMR spectra in  $\text{CDCl}_3$  were recorded on NMR spectrophotometer 500 MHz FT NMR Spectrometer at SAIF IIT Madras.

### Conventional Method for Synthesis of Schiff Base

Ethanol solution of 3,5 diiodosalicylaldehyde (0.01 mol) were added drop wise to a ethanolic solution of 2-amino benzimidazoles (0.01 mol). The mixture was refluxed on water bath for 2 hours. The product was recrystallized from ethanol. Yield: 38-45%. The Schiff base ligand exists in crystalline or amorphous form, light yellowish in colour and are stable to air and moisture.

### Microwave assisted Synthesis of schiff base

The Synthesis of Schiff base is schematically presented in (scheme 1).

2-amino Benzimidazoles (0.01 mol) and 3,5-diiodosalicylaldehyde (0.01 mol), and were mixed thoroughly in ethanol and small amount of glacial acetic acid was added and were taken in Erlen Meyer flask capped with a funnel placed in a microwave oven and irradiated an interval of 1 min at 450W for about 8-10 min. After completion the reaction, the reaction mixture was allowed to attain room temperature and solid separated was filtered. The resulting yellow product was then recrystallized with ethanol, finally dried under reduced pressure over anhydrous  $\text{CaCl}_2$  in a desiccator. Yield: 65-78%.



Scheme 1

Table 1: The comparative results of conventional and microwave methods, analytical and physical data of the compounds

S. No	Compound	Reaction time		Yield %	Melting point	Elemental Analysis		
		CM	MW			C%	H%	N%
1	BENI-S C <sub>14</sub> H <sub>9</sub> I <sub>2</sub> N <sub>3</sub> O 489.047	2		38%	175	34.38 (34.11)	1.85 (2.1)	N=8.59 (8.1)
		(10)		78%				

CM = Conventional method, time in hours; MW = Microwave method, time in minutes

## RESULTS AND DISCUSSION

As a result of microwave assisted synthesis, it was observed that the synthesis of schiff (2-[(1H-benzimidazol-2-ylimino) methyl]-4,6-diiodophenol) was completed in a short time with higher yields as compared to the conventional method. Comparative

study results obtained by microwave assisted synthesis; versus conventional heating method is that some reactions which required 2-3 h. by conventional method, was completed within 8-10 min by the microwave irradiation technique, yields have been improved from 30-46 to 76-80%.

**Table 2: Observed IR bands (cm<sup>-1</sup>) of Schiff base ligands.**

Compound	V <sub>1</sub> (O-H)	V <sub>1</sub> (C=N)	Cyclic(C=N)	V <sub>1</sub> (C-O)
BENI-S	3574	1679	1595	1276

### Physical properties

The details of physical properties of the schiff base are tabulated in Table 1. Table 1 showed the comparative results of conventional and microwave methods, analytical and physical data of the compounds

### IR Spectral Studies

Disappearances of carbonyl and amine group peaks from IR spectrum indicated formation of schiff base. In the schiff base strong peaks of carbonyl near 1723 nm and amine near 3315 nm were observed. Both of these peaks were absent in the IR spectra of schiff base. [31] In addition to that another peak was observed near 1679 nm which is an indication of azomethine (CH=N). This reflects that amino acid and aldehydes which are the substrate for synthesis have been converted into schiff base i.e. 2-amino Benzimidazoles and 3,5 diiodosalicyldehyde. In addition, the ligand exhibits a band at 1595 cm<sup>-1</sup> due to  $\nu$  cyclic (C=N) of the imidazole nitrogen. [32] The phenolic C-O stretch band is observed at 1276 cm<sup>-1</sup> in the free ligand. [33] The data

of the IR spectra of investigated Schiff base are listed in Table 2.

### <sup>1</sup>H NMR Spectral Studies

The 1H-NMR Spectra of schiff base are given some signals which are summarised in Table 3.

The NMR spectra of Schiff bases were recorded in CDCl<sub>3</sub> solution, using tetramethylsilane (TMS) as internal standard. The H-NMR spectrum for schiff base showed a peak at 7.4 ppm (s, 1H, -OH), a peak at 9.4 ppm (s, 1H, N=CH), a peak at 9.7 ppm (Benzimidazoles ring N-H) The multi signals within the 8.2-7.0 ppm range are assigned to the aromatic protons of both rings. The free NH<sub>2</sub> protons usually show a broad singlet peak in a region at 4-6 ppm. This NH<sub>2</sub> signal is absent in the observed spectra of Schiff base which indicates the formation of the Schiff base. [34]

### Mass Spectral studies

The mass spectrum of the Schiff base (2-[(1H-benzimidazol-2-ylimino) methyl]-4,6- diiodophenol) shows a molecular ion peak (m<sup>+</sup>) at m/z 489.04 that corresponds to the molecular weight of the Schiff base. Besides this peak, the Schiff base showed a fragment ion peak at m/z 373 that corresponds to (C<sub>7</sub>H<sub>4</sub>I<sub>2</sub>O<sub>2</sub>)<sup>+</sup>, one part of the ligand (i. e. 3,5-diiodosalicyldehyde).

**Table 3: Observed <sup>1</sup>H NMR Peaks (ppm) of schiff base.**

S. No	Compound	H for from azomethine group	H from aromatic group	H from Phenolic proton	H from benzimidazole N-H
1	BENI-S	9.4	8.2-7.0	7.4	9.7

In this report, we described new Schiff base which have been synthesized using condensation of 2-amino Benzimidazoles and 3,5 diiodosalicyldehyde efficiently in an alcoholic medium using acetic acid with excellent yields under microwaves irradiation and characterized by various physicochemical and spectral analyses. In the result of microwave assisted synthesis of schiff base(2-[(1H-benzimidazol-2-ylimino) methyl]-4,6-diiodophenol), it has been observed that the reaction time decreased from hours to minutes and availability of the product within better yields compared.

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