

**Microwave Assisted Synthesis of Benzimidazole**  
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## ABSTRACT

*A phenyl ring fused to an imidazole ring is present in the benzimidazole. Because of their special chemical and biological properties, compounds with Benzimidazole nuclei have long piqued the curiosity of synthetic and medicinal chemists. Hoebrecker produced the first Benzimidazole in history in 1872. A few years later, Ladenburg used acetic acid and 3,4-diaminotoluene to reflux the chemical to generate the identical product. Other names for the Benzimidazole are benzoyloxies or Benzimidazole. In light of this nomenclature, Benzimidazole would also be referred to as 2-methyl Benzimidazole and methyl-o-phenylenediamine. Many naturally occurring chemicals and different medications contain benzimidazole derivatives, which are fascinating heterocycles.*

*Effects that are spasmolytic, hypotensive, vasodilator, and local anaesthetic. The goal of research is to provide an overview of the Benzimidazole components of the microwave synthesis processes used to produce benzimidazole derivatives until 2013. This is due to the importance of these heterocyclic compounds.*

**Keywords:** *Urolithiasis, anti urolithiatic activity, calcium oxalate crystal, Phytochemical screening, ethanol extract, kidney stones, herbal medicine, crystal growth inhibition.*

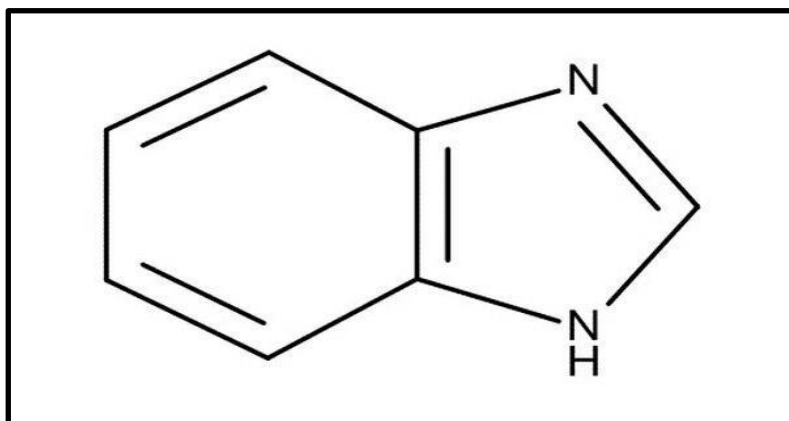
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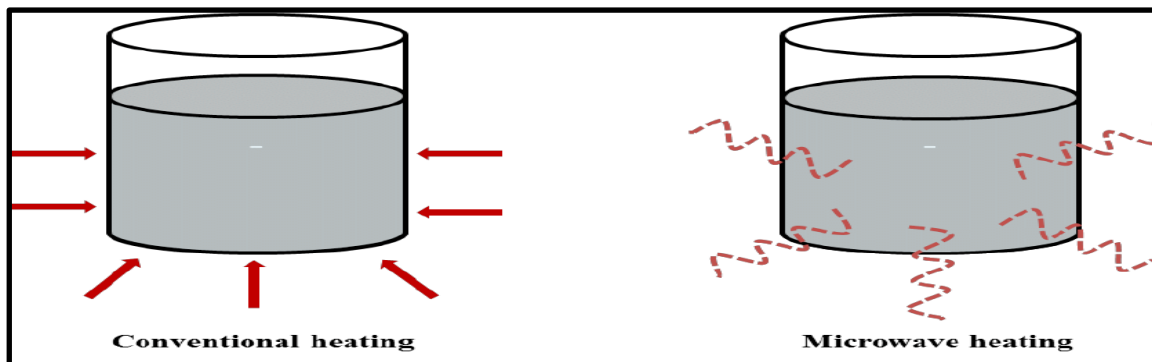
## INTRODUCTION

Microwave chemistry is the study of using microwave radiation to speed up chemical reactions. The development of microwave synthesis is a significant advance in Organic chemistry.. technique; significant shift manner that organic preparation is carried out. In addition to being known for being time-consuming and wasteful, conventional heating has also been shown to be creatively constricting.<sup>1</sup> The results of microwave synthesis provide the provides chemists with more time to be creative, explore new ideas, and create novel procedures. These days, scientists can complete the same process in minutes rather than hours or even days when synthesising a single chemical.<sup>2</sup> Solvent waste disposal has been solved by conducting processes under microwave radiation without the need for a solvent. Microwave irradiation combined with conducted in substance that contains little solvent environments gives that have improved yields, improved selectivity, easier manipulation, and increased reaction speeds. Therefore, microwave synthesis presents itself as a possible instrument for green chemistry.<sup>3</sup>



**Figure No. 1: Benzimidazole**

When it comes to heating or adding energy to the system, microwave irradiation offers an alternative to the traditional techniques. It. Electromagnetic waves are the source of microwave radiation<sup>4</sup>. The microwave radiation area of the The electromagnetic spectrum lies between the infrared and radio wavelengths. Microwaves operate between 0.3 and 300 GHz, with a wavelength of between 1 mm and 1 m. In this area, band frequencies are used by most telecommunications and microwave radar equipment. Through the ability of specific liquids and solids to transform electromagnetic energy into heat, the process of microwave dielectric heating propels chemical reactions.<sup>5</sup> This method opens up new options for synthetic chemists in the form of unique reactions that aren't possible with conventional heating.<sup>6</sup>



**Figure No. 2: Comparison Between Conventional Heating and microwave Heating**

## Benefits

### Speed

Because microwaves produce rapid and consistent heating, reactions can happen significantly faster sometimes in minutes as opposed to hours.

### Efficiency

It uses fewer resources and energy, which makes it more ecologically friendly.

### Larger Yield

Better control over reaction conditions made possible by microwave heating can result in larger yields and purer products.

### Selectivity

In complex processes in particular, microwave irradiation can improve selectivity, enabling more exact control over product production. The scalability of microwave reactors can be advantageous as they can be readily expanded for industrial output.

### Safety

By eliminating the requirement for dangerous chemicals or severe reaction conditions, microwave heating can lower the possibility of adverse reactions and increase safety.<sup>6</sup>

### Enhanced The Microwave Synthesis (EMS)

The term "Enhanced Microwave" refers to a new technique for carrying out microwave-assisted organic reactions. EMS, or "synthesis," investigated. It is possible to directly apply extra energy to the reaction mixture by using compressed air to chill the reaction vessel externally and microwave irradiation at the same time. But once it reaches this point, the microwave power either drops off or stops entirely to keep the bulk temperature at the correct level without going beyond.<sup>7</sup> The complete benefit of microwave-accelerated synthesis is lost when microwave irradiation is turned off and is replaced by classical thermal chemistry.<sup>8</sup> Using CMS, The main method for reaching TB more quickly is microwave irradiation. Chemical processes can only be enhanced by microwaves when microwave energy is applied.<sup>9</sup> Suppressing the use of this energy source is not desirable because it directly chemical reactions of compound activate High, continuous levels of microwave energy are applied thanks to EMS.<sup>10</sup>

The microwave's heating mechanism-Dipolar polarizations, Conduction mechanism, Interfacial polarizations. These are involved in the heating of microwave-absorbing materials, which are crucial for understanding microwave chemistry.<sup>14</sup>

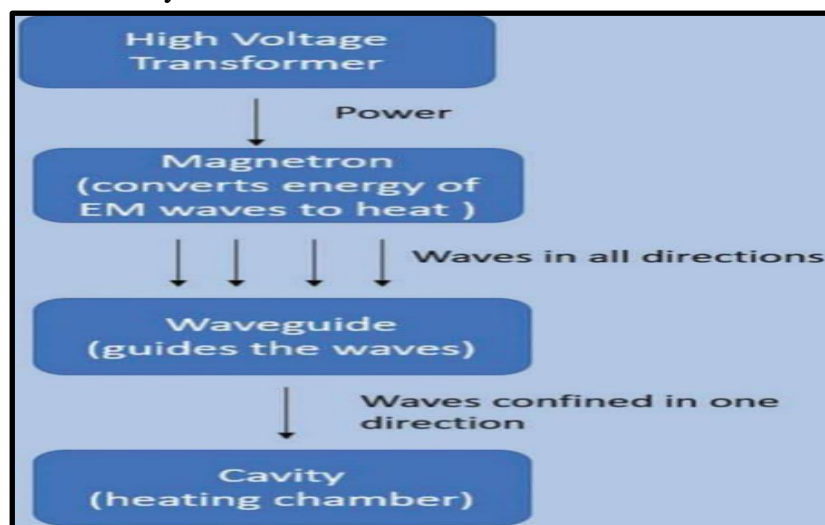


Figure No.3: Mechanism of Microwave Heating

## Dipolar polarizations

When exposed to microwave radiation, a material needs a dipole moment in order to produce heat. It is the A dipole that is trying to reposition itself in relation to an alternating electric field loses energy through molecular friction.<sup>15</sup> Heat can be produced by dipolar polarisation when polar solvent molecules like water, methanol, and ethanol interact with one another, or when Molecules of polar solutes, such as ammonia and formic acid, interact<sup>16</sup>. The frequency range field of the oscillator must be appropriate as the main need for dipolar polarization. to allow for sufficient tiny bit-to-particle contact.<sup>17</sup> the proper frequency range of microwave radiation (0.3-30 GHz) allows for sufficient inter-particle interaction and oscillation of polar particles. As this makes using it to heat polar liquids the perfect option.<sup>18</sup>

## Conduction mechanism

Resistance to an electric current is the conduction mechanism's means of producing heat<sup>19</sup>. The rattling When ions or electrons in a conductor oscillate due to an electromagnetic field, an electric current is produced<sup>20</sup>. Internal resistance causes the conductor to heat up when this current flows through it. since it is possible to assume that the higher the temperature and the more easily microwave radiation is absorbed, the more polar the solvent acquired<sup>21</sup>.

## Interfacial polarizations

The interfacial polarizations technique can be understood as a synthesis of the conduction and dipolar polarizations techniques. systems<sup>22</sup>. For heating systems when a conducting substance is mixed with a non-conducting material, it is crucial<sup>23</sup>. Combining the two results in a good material that absorbs microwaves: Nevertheless, metals must be utilized in powder form for this to occur.<sup>25</sup> This is due to the fact that metal powder effectively absorbs microwave radiation, in contrast to a metal surface. It heats up through a process akin to dipolar polarization and absorbs radiation<sup>26</sup>.

## Materials and Methodology

### Material

Every chemical, including the solvents, was sourced commercially. When required, industry-standard methods documented in literature were employed to purify the compounds. The manufacturer's name is included in parenthesis after the list of compounds used in the synthesis<sup>27</sup>.

O-phenylenediamine

Formic acid

10% Sodium Hydroxide

Potassium Hydroxide

### Experimental Work

#### Synthesis Of Benzimidazole Procedure

Place 17.5 g of 90% formic acid and 27 g of o-phenylenediamine in a 250 mL round-bottom flask. In a water bath, heat the mixture for two hours at 100 degrees Celsius.<sup>28</sup> The liquid should be just barely alkaline to litmus after cooling, at which point you should gradually add 10% sodium hydroxide solution while rotating the flask.<sup>29</sup> With the pump, extract the synthesized crude benzimidazole, rinse with ice cold water, completely drain, and then repeat the process with 25 milliliters of cold water<sup>30</sup>

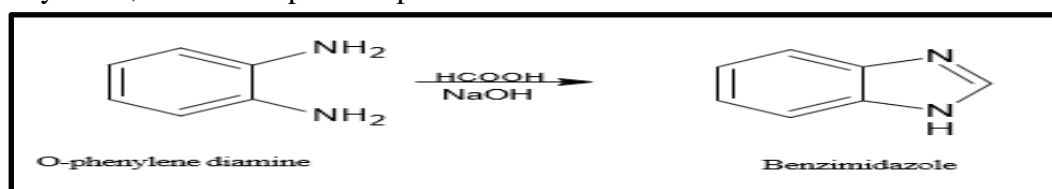


Figure No.4: Chemical Reaction of Benzimidazole

O-phenylenediamine's molecular formula is  $C_6H_8N_2$ .

The molecular formula for benzimidazole is  $C_7H_6N_2$ .

O-phenylenediamine has a molecular weight of 108.1 g/mol.

Benzimidazole has a molecular weight of 118.14 g/mol.

## RESULT

Colour	White to light yellow
PH determination	Acidic
Odour	Aromatic
State	Solid
Melting Point	170-172°C
partition Coefficient	12.8
Reactivity	Nucleophile Substitution, Electrophilic Substitution, Condensation
Appearance	off white coloured crystalline powder
Pka	5.30

**Table No.1: Evaluation parameters**

### Identification test

Sr No.	Test	Observation	Inference
1	Benzimidazole can undergo bromination at the C-2 Position of the heterocyclic ring. Reaction with bromine in acetic acid.	Formation of 2-bromo-benzimidazole	Present
2	Ring Cleavage Test: Benzimidazole undergoes ring Cleavage when treated with concentrated sulfuric acid. resulting in formation of o-phenylenediamine, which can be detected by its reaction with ninhydrin	Formation of purple colour	Present

**Table No2: Identification Test**

## DISCUSSION

Microwave-assisted synthesis is a technique that utilizes microwave irradiation to accelerate chemical reactions, leading to faster reaction times and potentially higher yields compared to traditional methods. In the case of Benzimidazole synthesis, this approach may offer advantages such as improved efficiency and selectivity. It is important to note that the specific conditions and parameters for microwave-assisted synthesis of Benzimidazole may vary depending on the desired outcome and the starting materials involved. Factors such as reaction temperature, time, and choice of solvents can all impact the success of the synthesis process. The time optimum of microwave- assisted synthesis using o-phenylenediamine at minute 7 minute with practical yield 2.97 g. Ring opening of benzimidazole below 280 nm.

## CONCLUSIONS

Research on the microwave-assisted synthesis of benzimidazole is highly promising because of its many benefits, including shorter reaction times, increased yields, and better purity than traditional techniques. With potential uses in agrochemicals, materials science, and medicines, this technique for organic synthesis is effective and sustainable. Its usefulness might be increased and green chemical techniques could be advanced with more research into reaction parameters, mechanistic studies, and scale-up procedures.

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