

## Facile Synthesis of 2-Substituted Benzimidazoles using Waste Aluminium Foil

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### Abstract

*Aromatic aldehydes and o-phenylenediamine in presence of waste aluminium foil synthesizes 2-Substituted benzimidazoles were synthesized in a one pot under microwave condition. at specific temperature it gives good yield and high selectivity.*

**Keywords:** Benzimidazoles, o-Phenylenediamine, Aromatic aldehyde, FeCl<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub>

### Introduction

Benzimidazole is important heterocycles in drug discovery 1 large variety of biological activity benzimidazole have 2-4. Benzimidazole nucleus exhibit wide spread occurrence and exhibit wide range of properties like HIV, RNA, influenza, HCMV, 5 they have Bis benzimidazoles derivatives as mirror groove binding agent for DNA for antitumor activity 6 many times they are used for biological modelling of biological systems 7-8 Benzimidazole are also important in organic synthesis for various reactions 9-10 Therefore the benzimidazole is centre of attraction now a days. preparation of benzimidazoles has gained considerable attention in recent years 11-14. Despite their importance from pharmacological, industrial and synthetic points of view, comparatively few methods for the preparation of benzimidazoles have been reported in chapter 1. The most popular synthetic approaches generally involve the condensation of an arylendiamine with a carbonyl equivalent 15. These includes the condensation of aryl diamines and aldehyde using air as the oxidant 16, the condensation of aryl diamines with carboxylic acids or their derivatives recently by PS-PPh<sub>3</sub>/CCl<sub>3</sub>CN 17, thermal or acid promoted cyclization reactions 18 or the use of microwave irradiation 19-20. In all these approaches, condensation of arylendiamines with aldehyde involves a two step procedure that includes the oxidative cyclodehydration of aniline Schiff's bases, which often generated in situ. Various recent oxidative reagents, such as (bromodimethyl) sulphonium bromide 21, TiCl<sub>4</sub> 22, Sulphamic acid 23, Iodobenzene diacetate 24, H<sub>2</sub>O<sub>2</sub>-HCl 25, silica sulfuric acid 26, FeBr<sub>3</sub> or Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O 27 Oxalic acid. 28 in dioxane solvent by air oxidation at 100 °C 29, Silica supported thionylchloride 30. Some solid-phase synthetic methods are explored in the synthesis of benzimidazole derivatives 31. A number of protocols that involves the condensation of o-phenylenediamine with different substituted aldehydes in the presence of transition metal triflate salts such as Sc(OTf)<sub>3</sub> or Yb(OTf)<sub>3</sub> were reported 32-33.

### Experimental

All chemicals were from S.D. Fine chemicals suppliers and used without further purification. Melting points were uncorrected. <sup>1</sup>H NMR spectra were recorded on a Bruker AVANCE 600 spectrometer using TMS as internal standard and DMSO as solvent.

### Preparation of Catalyst

Waste aluminium foil collected, dried in oven at 50 °C crushed in pestle and mortar to fine powder. Obtained powder is dried at 60 °C in oven, and then stored in glass bottle.

### General procedure for the Synthesis of 2-substituted benzimidazoles

*O*-Phenylenediamine (10 mmol) and aromatic aldehyde (10 mmol) were dissolved in DMF (2 mL) in a 25 mL conical flask, the waste aluminium foil (120 mg, 0.1 mmol) was then added and the mixture sonicated at specific time at 25°C for the specified time as indicated in Table 1. The progress of reaction was followed by TLC. After the completion of the reaction, the mixture was dissolved in dichloromethane, and the catalyst was removed by filtration and washed with dichloromethane. The solvent was evaporated under pressure to give the crude product, which was purified by column chromatography on silica gel and eluted with hexane and ethylacetate. All of the compounds were studied by comparing their melting points references 2,14,17 and characterized by <sup>1</sup>H NMR and mass spectra.

**Observation table 1**

Sr. No	Aromatic group	Time (min)	(%)Yield	MP (°C)
1.	C <sub>6</sub> H <sub>5</sub>	15	88	290
2.	2-ClC <sub>6</sub> H <sub>4</sub>	15	90	134
3.	3- ClC <sub>6</sub> H <sub>4</sub>	14	92	236
4.	4- MeOC <sub>6</sub> H <sub>4</sub>	20	90	227
5.	2-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> rvation	12	94	250
6.	3- NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	12	93	305
7.	4- NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	18	88	306
8.	4-MeC <sub>6</sub> H <sub>5</sub>	14	85	266
9.	4-Me <sub>2</sub> NC <sub>6</sub> H <sub>4</sub>	18	87	237
10.	2-Furanyl	19	78	287

### Spectral analysis

#### 2-(4'-Methylphenyl)benzimidazole

Light yellow crystal. <sup>1</sup>H NMR  $\delta$ : 10.53 (s, 1H), 8.27 (d, 2H, J = 8.1 Hz), 7.34 (s, 1H), 7.22 (s, 1H), 7.46 (d, 2H, J = 7.5 Hz), 7.20 (s, 2H), 2.38 (s, 3H).

#### 2-(2'-Furanylphenyl) benzimidazole

Light yellow crystal. <sup>1</sup>H NMR  $\delta$ : 10.23 (s, 1H), 7.45 (d, 1H, J = 1.2 Hz), 7.57 (s, 2H), 7.59-7.32 (m, 3H), 6.73-6.44 (m, 1H).

### Results and Discussion

In order to get the best experimental condition, we have considered the reaction of *O*-phenylenediamine and Benzaldehyde in the presence of catalyst under sonication at ambient temperature reaction. This reaction gives higher yield with good purity.

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