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### Silphox [ $\text{POCl}_3 \cdot (\text{SiO}_2)_n$ ] as a New, Efficient, and Heterogeneous Reagent for the Synthesis of Benzimidazole Derivatives Under Microwave Irradiation

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## Silphox [ $\text{POCl}_{3-n}(\text{SiO}_2)_n$ ] as a New, Efficient, and Heterogeneous Reagent for the Synthesis of Benzimidazole Derivatives Under Microwave Irradiation

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*Silphox [ $\text{POCl}_{3-n}(\text{SiO}_2)_n$ ] efficiently catalyzes the condensation of benzene-1,2-diamine with mono and dicarboxylic acids under microwave irradiation to afford benzimidazole derivatives in high yields and short reaction times.*

**Keywords** Benzene-1,2-diamine; benzimidazole derivatives; carboxylic acid; microwave; silphox.

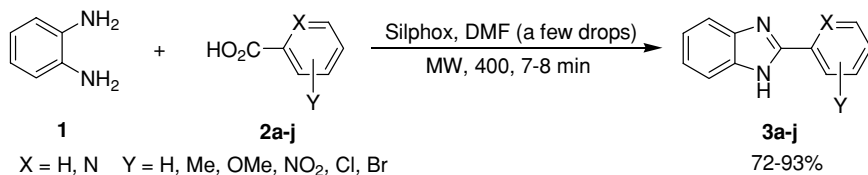
## INTRODUCTION

Benzo-fused heterocyclic systems such as benzimidazole are well-known because they possess a wide range of biological properties and clinical applications.<sup>1</sup> These heterocyclic ring systems are present in numerous antiparasitic, fungicidal, anthelmintic, and anti-inflammatory drugs.<sup>2</sup> Antibacterial and antifungal activity has been observed for benzimidazole derivatives.<sup>3</sup> 2-Aryl and 2-alkyl benzimidazoles exhibit activity against HIV<sup>4</sup> and influenza viruses.<sup>5</sup> Thus, the synthesis of these compounds has received considerable attention in diverse areas of chemistry. A number of synthetic methods have been developed to uncover a variety of new reagents for the preparation

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### SCHEME 1

of benzimidazoles.<sup>6–10</sup> The most commonly used synthetic approaches typically entail the condensation of *o*-arylenediamines with carbonyl compounds, such as aldehydes, carboxylic acids, and their derivatives.<sup>7</sup> In addition, there have been several reports on benzimidazole synthesis via the reductive cyclization of *o*-nitroanilines with aldehydes,<sup>8</sup> cyclization of *o*-nitroaniline derivatives with aryl isothiocyanates,<sup>9</sup> and Baker's yeast reduction of 2,4-dinitroacyl anilines.<sup>10</sup> Recently, Salehi et al. reported that the reaction of benzene-1,2-diamine with aldehydes in the presence of silica sulfuric acid produced 2-aryl-1-arylmethyl-1*H*-1,3-benzimidazoles.<sup>11</sup> However, many of the reported methods have several drawbacks, including low yields, long reaction times, the use of expensive reagents, tedious workup procedures involving more than one step in their synthesis, and co-occurrence of several side reactions.

Microwave irradiation has been extensively used for the rapid synthesis of a variety of heterocyclic compounds.<sup>12</sup> The beneficial effects of microwave irradiation are finding an increased role in process chemistry, especially when conventional methods require forcing conditions or prolonged reaction times.<sup>12</sup> Moreover, the possibilities offered by this technology are particularly attractive where high-yielding protocols and avoidance or ease of purification are highly desirable.<sup>12</sup>

On the basis of the above facts, and also along with our previous studies on the applications of phosphorus-containing reagents<sup>13</sup> as well as microwave technology in organic synthesis,<sup>14</sup> herein, we wish to report an efficient, rapid, and simple method for the preparation of benzimidazole derivatives from benzene-1,2-diamine, mono and dicarboxylic acids in the presence of silphox [ $\text{POCl}_3\text{-n}(\text{SiO}_2)_n$ , silica phosphin oxide] and a few drops of *N,N*-dimethylformamide (DMF) under microwave irradiation (Schemes 1 and 2).

## RESULTS AND DISCUSSION

We have previously used phosphorus-containing reagents for several organic transformations.<sup>13</sup> One of these reagents is silphox, which is easily prepared by the reaction of plate silica gel with  $\text{POCl}_3$ .<sup>13a</sup> We have

applied this reagent successfully for the condensation of sulfonamides with carbonyl compounds to produce *N*-sulfonyl imines.<sup>13a</sup> As a part of our studies on applications of phosphorus-containing reagents in organic synthesis, we have decided to employ silphox for the synthesis of benzimidazole derivatives via the reaction of benzene-1,2-diamine with carboxylic acids. Thus, at first, the reaction of benzene-1,2-diamine (1 mmol) with benzoic acid (1 mmol) in the presence of silphox (1 g) and DMF (10 drops) as a model was examined at the range 70–130°C. The results are summarized in Table I. As it can be seen from Table I, the respective benzimidazole **3a** was obtained in 86% yield after 240 min at 110°C. The same reaction was also tested in the presence of plate silica gel as well as POCl<sub>3</sub> at 110°C separately (Table II). The results showed that these reagents alone were not efficient for the synthesis of benzimidazoles. Furthermore, the reaction of benzene-1,2-diamine with benzoic acid was examined using P<sub>2</sub>O<sub>5</sub>/SiO<sub>2</sub> in the presence of a few drops of DMF at 110°C; however, compound **3a** did not produce at all, even by prolonging the reaction time and increasing the temperature (Table II). In these conditions, the starting materials remained intact plus two undesired products, the structures of which have not been identified.

**TABLE I** The Condensation of Benzene-1,2-diamine with Benzoic Acid in the Presence of Silphox at Different Temperatures

Entry	Temperature (°C)	Time (min)	Yield (%) <sup>a</sup>
1	70	1000	26
2	80	720	40
3	90	540	63
4	100	360	74
5	110	240	86
6	120	210	81
7	130	150	73

<sup>a</sup>Isolated yield.

**TABLE II The Reaction of Benzene-1,2-diamine with Benzoic Acid in the Presence of Several Reagents at 110°C**

Entry	Reagent	Time (min)	Yield <sup>a</sup> (%)
1	Silphox	240	86
2	Silica gel	720	–
3	POCl <sub>3</sub>	240	32
4	P <sub>2</sub> O <sub>5</sub> /SiO <sub>2</sub>	360	–
5	P <sub>2</sub> O <sub>5</sub> /SiO <sub>2</sub>	720	–

<sup>a</sup>Isolated yield.

**TABLE III The Reaction of Benzene-1,2-diamine with Benzoic Acid in the Presence of Silphox Promoted by Microwave Irradiation (MW)**

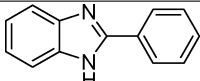
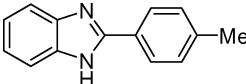
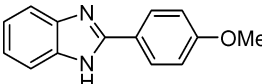
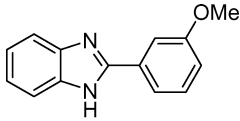
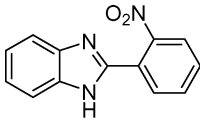
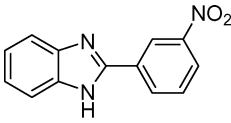
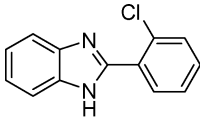
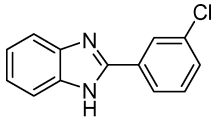
Entry	Microwave Power	Time (min)	Yield <sup>a</sup> (%)
1	100	25	32
2	200	18	63
3	300	12	79
4	400	8	93
5	500	5	85
6	600	3	74

<sup>a</sup>Isolated yield.

As Tables I and II show, the reaction yield under conventional thermal conditions was relatively high; however, the reaction time was long. To improve the reaction time, the model reaction was checked under microwave irradiation at the range 100–600 W of microwave power (Table III). As it is shown in Table III (entry 4), microwave irradiation significantly decreased the reaction time and also increased the yield. Therefore, we selected the microwave conditions as our main method for all reactions.

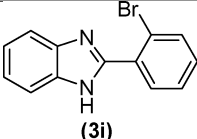
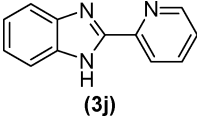
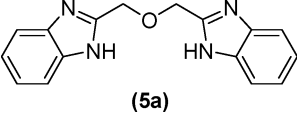
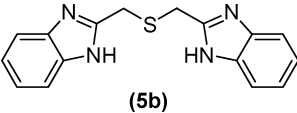
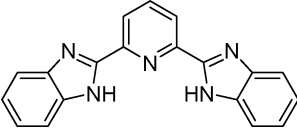
To realize the efficiency of silphox for the synthesis of benzimidazoles, benzene-1,2-diamine was condensed with structurally diverse carboxylic and dicarboxylic acids under microwave irradiation. The results are displayed in Table IV. As is clear from Table IV, all reactions proceeded efficiently, and the products were produced in good to high yields and short reaction times. The effect of electron-releasing and electron-withdrawing substituents on the aromatic ring of carboxylic acids upon the reaction was investigated. It has been observed that electron-releasing groups had no significant effect on the yields and the reaction times (Table IV, entries 2–4). The electron-withdrawing

**TABLE IV Preparation of Benzimidazole Derivatives from Benzene-1,2-diamine, Mono and Dicarboxylic Acids Under Microwave Conditions (400 W)**

Entry	Product	Time (min)	Yield <sup>a</sup> (%)	M.p. °C (Lit.)
1	 (3a)	8	93	288–290 (292–294) <sup>7a</sup>
2	 (3b)	8	90	265–267 (270) <sup>7b</sup>
3	 (3c)	8	87	223–225 (226) <sup>7b</sup>
4	 (3d)	8	89	203–205 (201–204) <sup>7a</sup>
5	 (3e)	7	78	260–263 (264–265) <sup>6a</sup>
6	 (3f)	7	72	207–209 (204–207) <sup>7a</sup>
7	 (3g)	7	90	233–235 (234) <sup>6b</sup>
8	 (3h)	7	92	236–239 (238) <sup>6b</sup>

(Continued on next page)

**TABLE IV Preparation of Benzimidazole Derivatives from Benzene-1,2-diamine, Mono and Dicarboxylic Acids Under Microwave Conditions (400 W) (Continued)**

Entry	Product	Time (min)	Yield <sup>a</sup> (%)	M.p. °C (Lit.)
9	 (3i)	7	89	245–247 (241–244) <sup>7a</sup>
10	 (3j)	8	89	216–218 (217–219) <sup>7d</sup>
11 <sup>b</sup>	 (5a)	15	87	[294–296 (dec.)] [296–298 (dec.)] <sup>7d</sup>
12 <sup>b</sup>	 (5b)	17	84	[210–212 (dec.)] [212 (dec.)] <sup>6c</sup>
13 <sup>b</sup>	 (5c)	17	82	273–276 (>250) <sup>7d</sup>

<sup>a</sup>Isolated yield.

substituent did not affect on the reaction times while decreased the yields (Table IV, entries 5 and 6). Moreover, the results showed that the presence of a halogen on the aromatic ring of carboxylic acids had negligible influence on the reaction results (Table IV, entries 7–9). This method was also applied successfully for the condensation of benzene-1,2-diamine (2 eq.) with dicarboxylic acids (1 eq.) to afford bis-benzimidazoles in high yields and short reaction times (Table IV, entries 11–13).

The efficiency and applicability of this new procedure was compared with some reported methods for the preparation of 2-substituted benzimidazoles (Table V). As Table V demonstrates, our method afforded the better results.

**TABLE V The Comparative Synthesis of Compound 3a Using the Reported Methods Versus the Present Method**

Entry	Reagent	Time (min)	Yield <sup>a</sup> (%)	Ref.
1 <sup>b</sup>	Silphox	8	93	—
2	Zeolite	180	81	7a
3	Pyridine/SOCl <sub>2</sub>	1080	92	7b
4	In(OTf) <sub>3</sub>	30	95	7c
5	Alumina-methanesulfonic acid	8	87	7d
6	Air in capped tube	960	85	7g

<sup>a</sup>Isolated yield.<sup>b</sup>Our method.

In summary, we have introduced an efficient reagent for the condensation of benzene-1,2-diamine with carboxylic acids under microwave irradiation. The promising points for the presented methodology are high conversion, ease of handling, and low cost of the reagent, and cleaner reaction profile and short reaction times, which makes it a useful and attractive process for the rapid synthesis of benzimidazoles and bis-benzimidazoles as biologically interesting compounds.

## EXPERIMENTAL

All chemicals were purchased from Merck or Fluka Chemical Companies. Plate silica gel (Mesh 60, 15–40  $\mu$ m) was used as support. All known compounds were identified by comparison of their melting points and <sup>1</sup>H NMR data with those in the authentic samples.<sup>6a,6b,6c,7a,7b,7d</sup> All reactions were carried out using a domestic microwave oven (MB 245, Butan Industrial Co., Iran). The <sup>1</sup>H NMR (250 MHz) and <sup>13</sup>C NMR (62.5 MHz) spectra were run on a Bruker Avance DPX-250, FT-NMR spectrometer ( $\delta$  in ppm). Melting points were recorded on a Stuart Scientific Apparatus SMP3 (UK) in open capillary tubes.

### Procedure for the Preparation of Silphox<sup>13a</sup>

Under nitrogen atmosphere, to a flask containing dry plate silica gel (6.0 g, 0.1 mol), POCl<sub>3</sub> (64 g, 0.5 mol) at room temperature was added and stirred slowly with a mechanical stirrer for 30 min. The mixture was then heated to 60°C while it was stirring under pressure of nitrogen for 3 h to remove all produced HCl. Afterward, the reaction mixture was cooled to room temperature, filtered, and the resulting precipitate was washed with dry CH<sub>2</sub>Cl<sub>2</sub> (50 mL) to give silphox as a white solid (9.1–9.3 g), which was stored in a capped bottle. The reagent can be kept without any change for months.



## General Procedure for the Synthesis of Benzimidazoles via Condensation of Benzene-1,2-diamine with Carboxylic Acids Under Microwave Irradiation

To a well-ground mixture of benzene-1,2-diamine (0.11 g, 1 mmol), carboxylic acid (1 mmol), and silphox (1 g) in a test tube, DMF (10 drops) was added and mixed carefully with a small rod. The resulting mixture was irradiated in a microwave oven at 400 W for the times reported in Table IV. Afterward, the reaction mixture was cooled to room temperature, and warm EtOAc (50 mL) was added to the reaction mixture, filtered, and the filtrate was washed with saturated NaHCO<sub>3</sub> (2 × 50 mL) and H<sub>2</sub>O (2 × 50 mL). After evaporation of the solvent, the resulting solid was recrystallized from EtOH to give the pure product.

## Some Selected Spectral and Physical Data of Benzimidazole Derivatives

### 2-Phenyl-1H-benzo[d]imidazole (3a)

White solid; m.p. 288–290°C (Lit.<sup>7a</sup> 292–294°C); <sup>1</sup>H NMR (DMSO-d<sub>6</sub>): δ 7.12–7.19 (m, 2H), 7.49–7.55 (m, 5H), 7.98 (m, 2H), 12.56 (s, 1H); <sup>13</sup>C NMR (DMSO-d<sub>6</sub>): δ 114.2, 121.6, 125.3, 126.2, 130.6, 131.2, 134.7, 148.8.

### 2-[[[(1H-Benzo[d]imidazol-2-yl)methoxy)methyl]-1H-benzo[d]imidazole (5a)

White solid; m.p. 294–296°C (dec.) [Lit.<sup>7a</sup> 296–298°C (dec.)]; <sup>1</sup>H NMR (DMSO-d<sub>6</sub>): δ 4.89 (s, 4H), 7.14–7.22 (m, 4H), 7.53–7.58 (m, 4H), 12.63 (s, 2H); <sup>13</sup>C NMR (DMSO-d<sub>6</sub>): δ 65.9, 113.0, 119.4, 129.1, 149.2.

### 2-(6-(1H-Benzo[d]imidazol-2-yl)pyridin-2-yl)-1H-benzo[d]imidazole (5c)

White solid; m.p. 273–276°C (Lit.<sup>7d</sup> >250°C); <sup>1</sup>H NMR (DMSO-d<sub>6</sub>): δ 7.23–7.27 (m, 4H), 7.66–7.70 (m, 4H), 8.03 (m, 2H), 8.29 (d, 1H, *J* = 7.3 Hz), 12.55 (s, 2H); <sup>13</sup>C NMR (DMSO-d<sub>6</sub>): δ 119.6, 123.9, 125.1, 138.1, 149.2, 151.2, 152.6.

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