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**Research Article**

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# A Rapid and Efficient Microwave-Assisted Synthesis of 2-Arylbenzoxazoles by Using 1,3-Dibromo-5,5-Dimethylhydantoin (DBH) Under Solvent-Free Condition

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**ABSTRACT**

*A simple, rapid and efficient procedure for the synthesis of 2 -arylbenzoxazole derivatives has been developed from 2-aminophenol and arylaldehydes by using 1, 3-dibromo-5, 5-dimethylhydantoin (DBH) in moderate to good yields under solvent-free microwave irradiation. The remarkable efficiency of DBH under solvent free microwave irradiation leads to many advantages like, the use of non-corrosive and inexpensive reagents, simple workup procedure and good yields in addition to the eco-friendly "green chemistry" economical and environmental impacts.*

**Keywords:** 2-arylbenzoxazoles, 1, 3-dibromo-5, 5-dimethylhydantoin (DBH), Microwave assisted chemistry, Solvent free condition.

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## INTRODUCTION

Worldwide demand for environmentally friendly chemical processes and products requires the development of novel and cost-effective approaches to pollution prevention. One of the most attractive concepts in chemistry for sustainability is green chemistry, which is the utilization of a set of principles that reduces or eliminates the use or generation of hazardous substances in the design, manufacture and applications of chemical products [1]. It should be noted that the rapid development of green chemistry is due to the recognition that environmentally friendly products and processes will be economical on a long term. One of the key areas of green chemistry is the elimination of solvents in chemical processes or the replacement of hazardous solvents with environmentally benign solvents.

In recent years, the concept of microwave-assisted organic synthesis has attracted considerable interest, due to the generally short reaction times as well as both the high purity and yields of the resulting products [2]. Specially, microwave accelerated solvent-free reactions have received much attention because of safety problems minimization [3].

2-Arylbenzoxazoles are an important group of target molecules by virtue of their special photophysical properties [4-6] and it is the key structure feature of a large number of biologically active natural products and pharmaceutical compounds [7]. Generally, the synthesis of 2-arylbenzoxazoles can proceed by two strategies. One is the coupling of 2-aminophenols with carboxylic acid derivatives, which is either catalyzed by strong acids [8] or requires microwave conditions or mediated by hexachloroethane and triphenylphosphine [9-10]. The other way is the oxidative cycalisation of phenolic schiff bases derived from the condensation of 2-aminophenols and aldehydes. In the latter case, various oxidants have been used, such as DDQ [11], $O_2$ (promoted by activated carbon or catalyzed by Cu-nanoparticle) [12-13], Mn-$(OAc)_3$ [14], $PhI(OAc)_2$ [15-16], Th+$ClO_4$- [17-18], $BaMnO_4$ [19], $NiO_2$ [20], Pb-$(OAc)_4$ [21], deoxo-fluoro reagent [22], $[Cp^*IrI_2]_2$ [23], dess-martin periodinane [24], tert-butyl hypochlorite [25], o-iodobenzoic acid (IBX) [26] and also arylbenzoxazole containing amino acids prepared by using $Pb(OAc)_4$ was

reported as the best oxidant [27], but it is toxic and deleterious to the environment. However, all of these oxidants, some are very costly, some are toxic and deleterious to the environment, some are not easy for work up and purification, even though required in stoichiometric or excess amounts relative to their respective substrates. Recently, some microwave-assisted methods were reported for the synthesis of 2-substituted benzothiazoles or benzoxazoles [28].

In ongoing research on 1, 3-dibromobromo-5, 5-dimethylhydantoin (DBH) is an inexpensive, commercially available and versatile convenient reagent used in various transformations [29-33] and also in order to avoid the drawbacks related to the previously reported methods for the synthesis of 2-arylbenzooxazole such as the use of acidic media, high cost, tedious work up and purification of the products from reaction mixtures, we wish herein, to report on the reagent DBH as a more robust and efficient reagent for the synthesis of 2-arylbenzooxazoles. Optimizing the reaction conditions in the use of organic solvents such as acetonitrile, dimethyl sulphoxide, dioxane, toluene or carbon tetrachloride is common but these all are high boiling and contribute to environmental pollution and health. It is therefore desirable to see if these reactions can be carried out under solvent-free conditions. Microwave-assisted organic reactions have been applied to a wide range of reaction types like substitution, addition, cycalisation reactions etc. It accelerates a variety of synthetic transformations via time and energy-saving protocols. We began the preliminary investigation by examining microwave- assisted synthesis of 2-arylbenzoxazoles in which we report 1, 3-dibromobromo-5, 5-dimethylhydantoin (DBH) as an oxidant.

## EXPERIMENTAL SECTION

### General

All reagents were obtained from commercial sources and used without further purification. Flash column chromatography was performed with Rankem Silica Gel (230–400 mesh size). Thin layer chromatography (TLC) was performed on Merck pre-coated TLC aluminum sheets with Silica Gel 60 F254. The $^{1}H$ & $^{13}C$ NMR spectra were recorded on a Bruker Avance 300 MHz spectrometer, and chemical shifts (δ) are given in ppm and are referenced to the corresponding solvent residual peak. The LC/ES-MS analysis was performed using a Thermo Finnigan LCQ Advantage Max spectrometer. GC–MS analysis was carried out on a Thermo Focus GC spectrometer. Melting points were determined with (PEW-340MP) melting point apparatus.

***General procedure for synthesis of 2-arylbenzooxazole derivatives (3a-3o)***

To a mixture of 2-aminophenol (2 mmol) and respective aldehyde (2 mmol) in 5ml Biotage microwave vial was added DBH (2 mmol) a little exothermic reaction was observed. After that vial was sealed and irradiate at 120 $^{o}C$ for 20 min, the resulting mixture was diluted with ethyl acetate (10 mL) and washed sequentially with water (10 mL×2) and brine (10 mL). The organic layer was dried over anhydrous $Na_2SO_4$. After evaporation, the crude was purified by flash column chromatography (10% EtOAc in hexane) to afford the desired product.

***2-phenylbenzo[d]oxazole (3a) (Table 2, entry 1)*** off white solid, yield 71%, mp 100-101 $^{o}C$ (lit. (13) 102-104 $^{o}C$); $^{1}H$ & $^{13}C$ NMR (lit. 13); IR (KBr) νmax/$cm^{-1}$ 3065 (C-H Ar str.), 1550 (C=N), 1515 (C=C), 1259 (C-O), 824 (C-N), 701 (C-H Ar bend); LC–ESMS m/z 196.8 $[M+H]^{+}$; GC–MS (EI) m/z 195.03 [M+].

***2-(m-tolyl) benzo[d]oxazole (3b) (Table 2, entry 2)*** off white solid, yield 68%, mp 79-80 °C (lit. (34) 82-83 $^{o}C$); $^{1}H$ & $^{13}C$ NMR (lit. 34); IR (KBr) νmax/$cm^{-1}$ 3060 (C-H Ar str.), 2958 (C-H alkane str.), 1614 (C=N), 1548 (C=C), 1458 (C-H alkane bend), 1241 (C-O), 824 (C-N), 747 (C-H Ar bend); LC–ESMS m/z 210.17 $[M+H]^{+}$; GC–MS (EI) m/z 209.03 [M+].

***2-(3-methoxyphenyl) benzo[d]oxazole (3c) (Table 2, entry 3)*** White solid, yield 67%, mp 79-80 °C (lit. (35) 82-83 °C); $^{1}H$ & $^{13}C$ NMR (lit. 35); IR (KBr) νmax/$cm^{-1}$ 3048 (C-H Ar str.), 2862 (C-H alkane str.), 1645 (C=N), 1543 (C=C), 1451 (C-H alkane bend), 1223 (C-O), 846 (C-N), 740 (C-H Ar bend); LC–ESMS m/z 226.17 $[M+H]^{+}$; GC–MS (EI) m/z 225.02 [M+].

***3-(benzo[d]oxazol-2-yl) phenol (3d) (Table 2, entry 4)*** White solid, yield 68%, mp 127-129 $^{o}C$, $^{1}H$ & $^{13}C$ NMR (lit. 36); IR (KBr) νmax/$cm^{-1}$ 3115 (O-H), 3029 (C-H Ar str.), 1600 (C=N), 1552 (C=C), 1245 (C-O), 885 (C-N), 795 (C-H Ar bend); LC–ESMS m/z 212.17 $[M+H]^{+}$; GC–MS (EI) m/z 210.96 [M+].

***2-(3-nitrophenyl) benzo[d]oxazole (3e) (Table 2, entry5)*** Yellow solid, yield 78%, mp 210-211 $^{o}C$ (lit. (37) 211.5-211.6 °C); $^{1}H$ NMR (DMSO-$d_6$) δ: 8.92 (t, J = 1.9 Hz, 1H), 8.65 (ddd, J = 8.1, 1.3, 1.1 Hz, 1H), 8.25 - 8.57 (m, 1H), 7.86 - 8.07 (m, 3H), 7.31 -7.65 (m, 2H); $^{13}C$ NMR (300 MHz, $CDCl_3$) 162.7, 150.4, 142.2, 131.4, 128.9, 127.3, 127.2, 125.0, 124.7, 119.7, 110.3. IR (KBr) νmax/cm–1 3093 (C-H Ar str.), 1604 (C=N), 1528 (C=C), 1351 (N=O), 1230 (C-O), 808 (C-N), 744 (C-H Ar bend); LC–ESMS m/z 241.02 $[M+H]^{+}$; GC–MS (EI) m/z 240.02 [M+].

***2-(4-chloro-3-nitrophenyl) benzo[d]oxazole (3f) (Table 2, entry 6)*** Yellow solid, yield 84%, mp 215–216 $^{o}$C; IR (KBr) νmax/cm$^{-1}$ 3088 (C-H Ar str.), 1615 (C=N), 1529 (C=C), 1345 (N=O), 1240 (C-O), 808 (C-N), 751 (C-Cl), 722 (C-H Ar bend); LC–ESMS m/z 275.17 [M+H]$^{+}$; GC–MS (EI) m/z 273.88 [M+].

***2-(4-fluoro-3-nitrophenyl) benzo[d]oxazole (3g) (Table 2, entry 7)*** Yellow solid, yield 85%, mp 219–220 $^{o}$C; IR (KBr) νmax/cm$^{-1}$ 3101 (C-H Ar str.), 1609 (C=N), 1534 (C=C), 1348 (N=O), 1265 (C-O), 1243 (C-F), 844 (C-N), 798 (C-H Ar bend); LC–ESMS m/z 258.98 [M+H]$^{+}$; GC–MS (EI) m/z 257.95 [M+].

***2-(3-bromophenyl) benzo[d]oxazole (3h) (Table 2, entry 8)*** White Solid, yield 73%, mp 124-126 $^{o}$C (lit. (38) 128–130 $^{o}$C); $^{1}$H & $^{13}$C NMR (lit. 38); IR (KBr) νmax/cm$^{-1}$ 3075 (C-H Ar str.), 1550 (C=N), 1472 (C=C), 1193 (C-O), 842 (C-N), 790 (C-H Ar bend), 673 (C-Br); LC–ESMS m/z 274.88 [M+H]$^{+}$; GC–MS (EI) m/z 274.08 [M+].

***2-(3-fluorophenyl) benzo[d]oxazole (3i) (Table 2, entry 9)*** White solid, yield 74%, mp 95–96 $^{o}$C (lit. (38) 92–94 $^{o}$C); $^{1}$H & $^{13}$C NMR (lit. 38); IR (KBr) νmax/cm$^{-1}$ 3073 (C-H Ar str.), 1619 (C=N), 1498 (C=C), 1236 (C-O), 1155 (C-F), 841 (C-N), 798 (C-H Ar bend); LC–ESMS m/z 214.08 [M+H]$^{+}$; GC–MS (EI) m/z 212.96 [M+].

***2-(3-chlorophenyl) benzo[d]oxazole (3j) (Table 2, entry 10)*** White solid, yield 73%, mp 122-123 °C (lit. (38) 124–125˚C); $^{1}$H & $^{13}$C NMR (lit. 38); IR (KBr) νmax/cm$^{-1}$ 3086 (C-H Ar str.), 1572 (C=N), 1481 (C=C), 1260 (C-O), 833 (C-N), 742 (C-Cl), 725 (C-H Ar bend); LC–ESMS m/z 230.08 [M+H]$^{+}$; GC–MS (EI) m/z 228.88 [M+].

***3-(benzo[d]oxazol-2-yl) benzoic acid (3k) (Table 2, entry 11)*** White solid, yield 78%, mp 218–220 $^{o}$C; IR (KBr) νmax/cm$^{-1}$ 3032 (C-H Ar str.), 2830 (O-H), 1710 (C=O), 1630 (C=N), 1532 (C=C), 1225 (C-O), 865 (C-N), 765 (C-H Ar bend); LC–ESMS m/z 240.17 [M+H]$^{+}$; GC–MS (EI) m/z 239.08 [M+].

***3-(benzo[d]oxazol-2-yl) benzonitrile (3l) (Table 2, entry 12)*** Light Brown solid, Yield 77%, mp 205-207 °C, $^{1}$H & $^{13}$C NMR (lit. 39); IR (KBr) νmax/cm$^{-1}$ 3086 (C-H Ar str.), 2229 (CN), 1604 (C=N), 1495 (C=C), 1244 (C-O), 843 (C-N), 751 (C-H Ar bend); LC–ESMS m/z 221.08 [M+H]$^{+}$; GC–MS (EI) m/z 219.82 [M+].

***5-methyl-2-(3-nitrophenyl) benzo[d]oxazole (3m) (Table 2, entry 13)*** Pale yellow solid, yield 86%, mp 212–214 $^{o}$C; IR (KBr) νmax/cm$^{-1}$ 3104 (C-H Ar str.), 2921 (C-H alkane str.), 1607 (C=N), 1526 (C=C), 1470 (C-H alkane bend), 1352 (N=O), 1195 (C-O), 809 (C-N), 707 (C-H Ar bend); LC–ESMS m/z 255.17 [M+H]$^{+}$; GC–MS (EI) m/z 253.95 [M+].

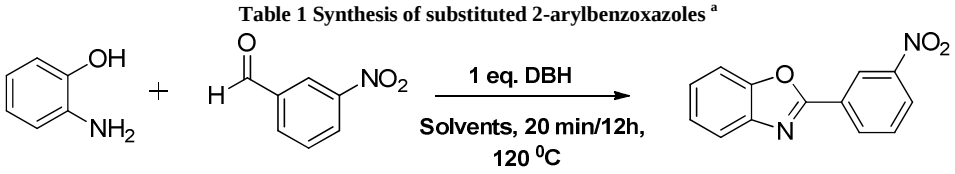
***5-methoxy-2-(3-nitrophenyl) benzo[d]oxazole (3n) (Table 2, entry 14)*** Pale yellow solid, yield 88%, mp 208–209 $^{o}$C; IR (KBr) νmax/cm$^{-}$1 3068 (C-H Ar str.), 2882 (C-H alkane str.), 1615 (C=N), 1533 (C=C), 1471 (C-H alkane bend), 1352 (N=O), 1203 (C-O), 811 (C-N), 730 (C-H Ar bend); LC–ESMS m/z 271.17 [M+H]$^{+}$; GC–MS (EI) m/z 270.08 [M+].

***5-chloro-2-(3-nitrophenyl) benzo[d]oxazole (3o) (Table 2, entry 15)*** Light yellow solid, yield 67%, mp 186-187 °C (lit., (40) 184–186 °C); $^{1}$H & $^{13}$C NMR (lit. 40); IR (KBr) νmax/cm$^{-1}$ 3060 (C-H Ar str.), 1606 (C=N), 1515 (C=C), 1335 (N=O), 1230 (C-O), 828 (C-N), 761 (C-Cl), 728 (C-H Ar bend); LC–ESMS m/z 275.17 [M+H]$^{+}$; GC–MS (EI) m/z 273.88 [M+].

## RESULTS AND DISCUSSION

According to the results shown in the Table 1 (entries 1-6), the reactions proceed within 20 min at 120 °C under solvent free microwave irradiation in moderate yields. Numerous repetitions of the reactions under different solvents and conditions indicated that the most effective conversion occurs when equimolar amounts of 2-aminophenol, arylaldehydes and DBH are used in the reactions under solvent free microwave irradiation. Longer reaction time gives small amount of brominated product with expected product while less reaction time gives incompletion of reaction with schiff's' base. It is also important to note that, low eq. of DBH results into incompletion of reaction or high eq. of DBH gives brominated product in 20 min at 120 °C under solvent free microwave irradiation.

**Table 1 Synthesis of substituted 2-arylbenzoxazoles [a]**



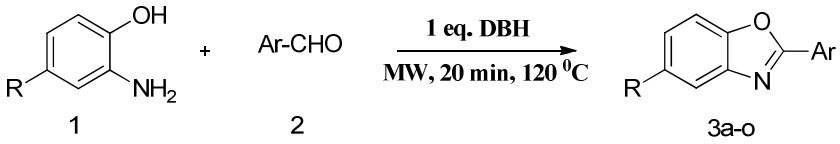
| Entry | Solvent | Time | Condition | Yield (%) [b] |
|---|---|---|---|---|
| 1 | Acetonitrile | 20 min /12h | MW/CH | 54/44 |
| 2 | DMSO | 20 min /12h | MW/CH | 62/50 |
| 3 | Dioxane | 20 min /12h | MW/CH | 52/39 |
| 4 | Toluene | 20 min /12h | MW/CH | 49/35 |
| 5 | $CCl_4$ | 20 min /12h | MW/CH | 35/30 |
| 6 | Neat | 20 min /12h | MW/CH | 78/18 |

[a] *Reaction condition 2-amino phenol (1.0 mmol), 3-nitro benzaldehyde (1.0 mmol), DBH (1.0 mmol), 120 °C, MW, Time 20 min / CH, Time 12h.*

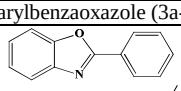
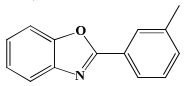
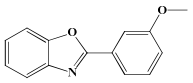
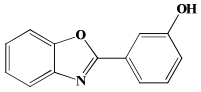
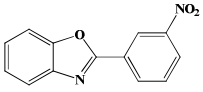
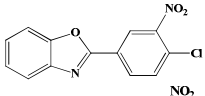
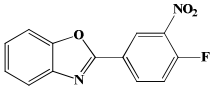
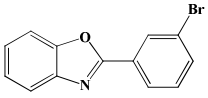
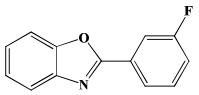
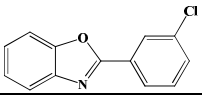
[b] *Yield of isolated product after flash chromatography : yield by MW Heating / yield by CH-Conventional Heating.*

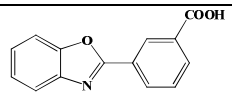
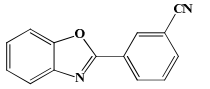
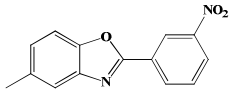
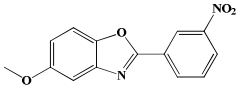
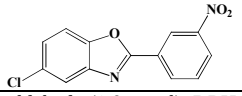
Following the above optimized condition (Table 1) different substituted 2-aminophenols were coupled with substituted benzaldehydes and the results are summarized in Table 2. The results indicate that the aromatic aldehydes having an electron-donating group or electron-withdrawing group, the reactions proceed smoothly to give the corresponding benzoxazoles **3a-3o** in moderate to good yields (Table 2, entries **1–15**). When aromatic aldehyde with an electron-withdrawing groups like $NO_2$, COOH, and CN gives better yield (Table 2, entries **5, 11, 12**) than those having electron-donating groups like Me, OMe and OH (Table 2, entries **2, 3, 4**) while aromatic aldehyde with halogens (Table 2, entries **8, 9, 10**) gives higher yield than electron-donating groups. Aromatic aldehydes having 3-$NO_2$ with Cl or F at 4 position (Table 2, entries **6, 7**) gives higher yield in comparison with electron-withdrawing groups, due to the presence of two electron-withdrawing groups. Additionally, electron donating substituent on 2-aminophenol with 3-niro benzaldehyde (Table 2, entries **13, 14**) were also investigated, and the desired products were obtained in higher yields than halogen substituted 2-aminophenol (Table 2, entry **15**). So we observed that the electron withdrawing groups on aldehyde with electron donating group on 2-aminophenol gives highest yield than any other entry in Table 2.

**Table 2 Synthesis of 2 -arylbenzoxazole derivatives from 2-aminophenol and arylaldehydes by using 1, 3-dibromo-5, 5-dimethylhydantoin (DBH) [a]**




1 2 3a-o

| Entry | *R* | Ar | 2-arylbenzaoxazole (3a-o) | Yield (%) [b] |
|---|---|---|---|---|
| 1 | H | Ph |  | 71 |
| 2 | H | *m*-Me–$C_6H_4$ |  | 68 |
| 3 | H | *m*-OMe–$C_6H_4$ |  | 67 |
| 4 | H | *m*-OH–$C_6H_4$ |  | 68 |
| 5 | H | *m*-$NO_2$–$C_6H_4$ |  | 78 |
| 6 | H | *p*-Cl, *m*-$NO_2$–$C_6H_4$ |  | 84 |
| 7 | H | *p*-F, *m*-$NO_2$–$C_6H_4$ |  | 85 |
| 8 | H | *m*-Br–$C_6H_4$ |  | 73 |
| 9 | H | *m*-F–$C_6H_4$ |  | 74 |
| 10 | H | *m*-Cl–$C_6H_4$ |  | 73 |

| | | | | |
|---|---|---|---|---|
| 11 | H | *m*-COOH–$C_6H_4$ |  | 78 |
| 12 | H | *m*-CN–$C_6H_4$ |  | 77 |
| 13 | Me | *m*-$NO_2$–$C_6H_4$ |  | 86 |
| 14 | OMe | *m*-$NO_2$–$C_6H_4$ |  | 88 |
| 15 | Cl | *m*-$NO_2$–$C_6H_4$ |  | 67 |

*[a] Reaction condition: - 2-amino phenol (1.0 mmol), arylbenzaldehyde (1.0 mmol), DBH (1.0 mmol), 120°C, MW, Time (20 min).*
*[b] Yield of isolated product after flash chromatography.*

## CONCLUSION

In summary, we have developed an expedient and straightforward approach for the synthesis of 2-arylbenzooxazole derivatives from 2-aminophenol and arylaldehydes by using eco-friendly and inexpensive reagent 1, 3-dibromo-5, 5-dimethylhydantoin (DBH) in moderate to good yield under solvent-free microwave irradiation. This new direct synthesis 2-arylbenzooxazole includes in situ preparation of the schiff's' base which undergo oxidative cycalisation whereas no metal oxides, organic oxidizing agents or strong acids such as polyphosphoric acid required. In addition, our method is devoid of expensive reagents and solvents.

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