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ORIGINAL PAPER

$KMnO_4$/HOAc system promoted one-pot synthesis of benzoxazoles from *o*-aminophenols or oxidative cyclization of *o*-hydroxyarylidene anilines at room temperature

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Abstract 1,3-Benzoxazoles via oxidative cyclization of corresponding *o*-hydroxyarylidene anilines was synthesized in the presence of $KMnO_4$/HOAc system. This system also was applied for the one-pot synthesis of 1,3-benzoxazoles from *o*-amino phenols and aldehydes. The both protocols were processed at room temperature under solvent-free conditions with good to excellent yields.

Keywords 1,3-Benzoxazoles · *o*-Hydroxyarylidene anilines · $KMnO_4$ · *o*-Amino phenol · Oxidative cyclization

Introduction

Benzoxazoles are one of the important structural motifs in many biologically active compounds such as antibiotics [1], antimicrobials [2], anticancers, anti-HIV-1s [3, 4], antituberculous [5], antimycobacterials [6], anti-inflammatories, and analgesic and kinase (CDK-1, CDK-5 and GSK-3) inhibitors [7]. This class of compounds allowed an access to many demonstrated bio-active agents such as 5-HT3 receptor partial agonist for treatment of diarrhea-predominant irritable bowel syndrome [8], melatonin receptor agonists [9], 5-HT3 receptor partial agonists in the gut [10], cyclooxygenase inhibitors [11], hypoglycemic agents [12], precursors for in vivo imaging of β-amyloid plaques [13], membranes for CO_2 separation [14], heat-resistant polybenzoxazole nanofibers [15], 5-lipooxygenase inhibitor [16], (HIF)-1 transcriptional inhibitor and [17] mPGES-1 inhibitors [18]. Some of biological active benzoxazoles are shown in Scheme 1.

Classical protocols for the preparation of benzoxazoles involved (1) one-pot reaction of carboxylic acids [19, 20], aldehydes [20–22], orthoesters [23] or acid chlorides [24, 25] with *o*-aminophenols (2) cyclization of *o*-haloanilides [26, 27], *o*-nitro phenyl esters [28], *o*-hydroxyarylidene anilines [29–33] and *o*-hydroxyanilides [34], (3) flash vacuum pyrolysis of 2-methoxy-*N*-(arenylidene)anilines [35] and (4) copper-catalyzed reaction of *o*-aryl dihalides with nitriles [36]. Oxidative cyclization of *o*-hydroxyarylidene anilines or phenolic Schiff's bases was done using various oxidants such as hypervalent iodine [29], 2,3-dichloro-5,6-dicyanobenzo-1,4-quinone [30], barium manganite [32], silver (I) oxide [33], PCC/silica [37]. One-pot synthesis of benzoxazoles from aldehydes and *o*-aminophenols was achieved in the presence of acidic catalysts such as $H_5[PMo_{10}V_2O_{40}]$ [20], iodine [21], nanoceria [22], 4-methoxy-TEMPO/O_2 [31], activated carbon/O_2 [38], CN^-/O_2 [39], $K_2S_2O_8$/$CuSO_4$ [40], ABMs/O_2 [41], $Zn(OTf)_2$ [42] and Cu-np, K_2CO_3/O_2 [43].

Experimental

General

Chemicals were purchased from Sigma–Aldrich and Merck chemical companies and were used without any purification. All products were characterized by their FT-IR, ^{1}H-NMR and comparison of their physical properties with those reported in the literature. FT-IR spectra were recorded on a Bruker, Eqinox 55 spectrometer. In all cases, the ^{1}H-NMR spectra were recorded on a Bruker DRX-400

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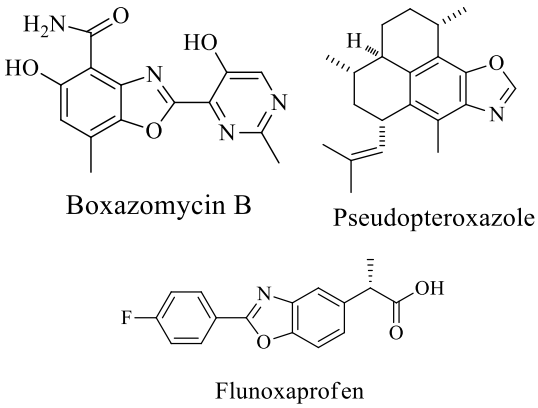


Scheme 1 The structure of some benzoxazole containing drugs

instrument. The elemental analyses was done by Costech ECS 4010 CHNS-O analyzer. Melting points were determined by a Buchi melting point B-540 B.V.CHI apparatus.

Preparation of 2-aryl-1,3-benzoxazoles from o-hydroxyarylidene anilines

In a mortar, *o*-hydroxyarylidene aniline (1 mmol), $KMnO_4$ (1.7 mmol) and HOAc (0.08 mL) were charged and ground for 5 min. The progress of reaction was monitored by TLC (ethyl acetate:*n*-hexane, 20:80). Then, acetone (5 mL) was added to the mortar and filtered the mixture. By adding water to filtrate, 2-aryl-1,3-benzoxazole was appeared as a pure solid.

One-pot preparation of 2-aryl-1,3-benzoxazoles

In a mortar, aldehyde (1 mmol), 2-aminophenol (1 mmol), $KMnO_4$ (3.4 mmol) and HOAc (0.08 mL) were charged and ground for 10 min. The progress of reaction was monitored by TLC (ethyl acetate:*n*-hexane, 20:80). Then, acetone (5 mL) was added to the mortar and filtered the mixture. By adding water to filtrate, 2-aryl-1,3-benzoxazole was appeared as a pure solid.

Some spectroscopic data

2-(4-Nitrophenyl)-1,3-benzoxazole

FT-IR (ATR) = $\bar{\upsilon}$, cm^{-1}: 3112 (C–H), 1598 (C=N), 1555, 1348 (N=O), 1519, 1451 (C=C).

^1H NMR (400 MHz, $CDCl_3$): δ, ppm (*J*, Hz): 8.44 (sbr, 4H), 7.83 (sbr, 1H), 7.65 (sbr, 1H), 7.44 (sbr, 2H).

2-(3-Nitrophenyl)-1,3-benzoxazole

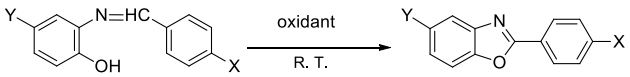
FT-IR (ATR) = $\bar{\upsilon}$, cm^{-1}: 3097 (C–H), 1613 (C=N), 1525, 1351 (N=O), 1474, 1453 (C=C), 1241 (C–O).

^1H NMR (400 MHz, acetone): δ, ppm (*J*, Hz): 8.97 (sbr, 1H), 8.77 (sbr, 1H), 8.64 (sbr, 1H), 7.7-8.0 (m, 3H), 7.47 (sbr, 2H).

2-(2-Nitrophenyl)-1,3-benzoxazole

FT-IR (ATR) = $\bar{\upsilon}$, cm^{-1}: 1620 (C=N), 1534, 1349 (N=O), 1452 (C=C), 1240 (C–O).

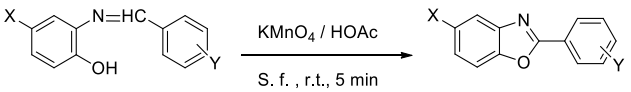
Table 1 Synthesis of 2-(aryl)-benzoxazole from *o*-hydroxyarylidene anilines (1 mmol) under various conditions



Entry	X	Y	Oxidant	Reactant/oxidant	Acid (mL)	Solvent	Time (h)	Yield (%)* References
1	NO_2	H	–	1/–	HOAc (0.08)	–	0.5	5
2	NO_2	H	$KMnO_4$	1/1.5	–	–	0.5	20
3	NO_2	H	$KMnO_4$	1/1.5	HOAc (0.08)	–	0.1	83
4	NO_2	H	$KMnO_4$	1/1.7	HOAc (0.08)	–	0.1	91
5	NO_2	H	$KMnO_4$	1/2	HOAc (0.08)	–	0.1	91
6	NO_2	H	$KMnO_4$	1/1.7	HOAc (0.06)	–	0.1	76
7	NO_2	H	$KMnO_4$	1/1.7	HOAc (0.07)	–	0.1	85
8	NO_2	H	$KMnO_4$	1/1.7	HOAc (0.09)	–	0.1	91
9	NO_2	H	PhI $(OAc)_2$	1/1.1	–	CH_3CN	0.1	93 [29]
10	H	H	DDQ	–	–	CH_2Cl_2	0.5	93 [30]
11	NO_2	H	$BaMnO_4$	1/10	–	$CHCl_3$	14	72 [32]
12	H	H	Ag_2O	–	–	CH_2Cl_2	2–5	76 [33]
13	NO_2	Me	PCC/Silica	1/1.1	–	CH_2Cl_2	0.18	89 [37]

* Isolated yield

Table 2 Synthesis of benzoxazoles via oxidative cyclization of *o*-hydroxyarylidene anilines

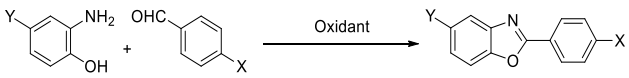


Entry	X	Y	Yield (%)*	m.p (°C)		References
				Observed	Reported	
1	H	4-NO_2	92	259–261	257–263	[44]
2	H	3-NO_2	87	196–198	207	[47]
3	H	2-NO_2	90	204–205	–	
4	H	4-Cl	85	143–146	143–145	[46]
5	H	2,4-Cl_2	88	119–120	118–119	[45]
6	H	2,6-Cl_2	0	–	–	–
7	H	4-Br	91	144–146	142–144	[46]
8	H	4-COOMe	68	189–191	–	–
9	Cl	4-NO_2	83	220–222	–	–
10	Cl	3-NO_2	79	123–125	184–186	[46]
11	Cl	4-Cl	81	177–179	–	–
12	Cl	2,4-Cl_2	78	148–150	–	–

* 1 mmol phenolic schiff's bases, 1.7 mmol $KMnO_4$, 0.08 ml HOAc, solvent free, room temperature, 5 min

** Isolated yields

Table 3 One-pot synthesis of 2-(aryl)-benzoxazole under various conditions



Entry	X	Y	Oxidant	Reactant/catalyst (mmol)	Acid (mL)	Solvent	*T*/°C	Time (h)	Yield (%)
1	NO_2	H	–	1/–	HOAc (0.08)	–	R.T.	0.5	10
2	NO_2	H	$KMnO_4$	1/1.7	–	–	R.T.	0.5	22
3	NO_2	H	$KMnO_4$	1/1.7	HOAc (0.08)	–	R.T.	0.15	65
4	NO_2	H	$KMnO_4$	1/2.5	HOAc (0.08)	–	R.T.	0.15	71
5	NO_2	H	$KMnO_4$	1/3	HOAc (0.08)	–	R.T.	0.15	79
6	NO_2	H	$KMnO_4$	1/3.4	HOAc (0.08)	–	R.T.	0.15	82
7	NO_2	H	$KMnO_4$	1/3.8	HOAc (0.08)	–	R.T.	0.15	82
8	NO_2	H	$KMnO_4$	1/3.4	HOAc (0.06)	–	R.T.	0.15	74
9	NO_2	H	$KMnO_4$	1/3.4	HOAc (0.07)	–	R.T.	0.15	78
10	NO_2	H	$KMnO_4$	1/3.4	HOAc (0.09)	–	R.T.	0.15	82
11	H	H	$H_5[PMO_{10}V_2O_{40}]$	1/0.5	–	THF	Reflux	6.5	88 [20]
12	Cl	H	Iodine	1/0.5	–	–	M.W.	0.05	95 [21]
13	NO_2	H	Nanoceria	1/0.05	–	H_2O	R.T.	0.3	98 [22]
14	NO_2	H	4-Methoxy-TEMPO/O_2	1/0.05	–	Xylene	120	6	91 [31]
15	H	H	Activated carbon/O_2	1/0.125	–	Xylene	120	4	78 [38]
16	H	H	CN^-/O_2	1/1	–	DMF	80	4	78 [39]
17	H	H	$K_2S_2O_8/CuSO_4$	1/0.01	–	SDS/water	60	0.83	90 [40]
18	NO_2	H	$ZnBr_2$/ABM/air	1/100 mg	–	Toluene	110	0.3	92 [41]
19	H	H	$Zn(OTf)_2$	1/0.01	–	Ethanol	80	5	91 [42]
20	NO_2	H	Cu–np, K_2CO_3/O_2	1/0.01	–	MeOH	80	4.6	75 [43]

* *o*-Aminophenol (1 mmol) and aldehyde (1 mmol) were used

^{1}H NMR (400 MHz, $CDCl_3$): δ, ppm (*J*, Hz): 8.43–8.45 (m, 4H), 8.15 (d, *J* = 7.5 Hz, 1H), 7.90 (d, *J* = 7.5 Hz, 1H), 7.82 (sbr, 1H), 7.75–7.71 (m, 2H), 7.58 (sbr, 1H), 7.4 (sbr, 1H).$C_{13}H_8N_2O_3$, calculated: C, 65.00; H, 3.36; N, 11.66. Found: C, 64.6; H, 3.39; N, 11.67 %.

2-(4-Chlorophenyl)-1,3-benzoxazole

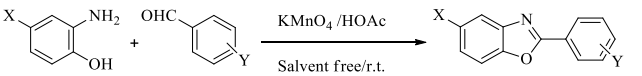
FT-IR (ATR) = ῡ, cm^{-1}: 3060 (C–H), 1617 (C=N), 1574, 1483 (C=C), 1344 (C–N), 1244 (C–O), 1091 (C–Cl).

^{1}H NMR (400 MHz, acetone): δ, ppm (*J*, Hz): 8.26 (sbr, 2H), 7.69–7.77 (m, 3H), 8.64 (sbr, 1H), 7.43–7.5 (m, 3H).

2-(2,4-Dichlorophenyl)-1,3-benzoxazole

FT-IR (ATR) = ῡ, cm^{-1}: 3066 (C–H), 1590 (C=N), 1560, 1471 (C=C), 1375 (C–N), 1191 (C–O), 1027 (C–Cl).

Table 4 One-pot synthesis of 2-(aryl)-benzoxazoles in the presence of $KMnO_4$/HOAc system at room temperature



Entry	X	Y	Yield (%)	References
1	H	4-NO_2	84	[44]
2	H	3-NO_2	76	[47]
3	H	4-Cl	73	[46]
4	H	2,4-Cl_2	68	[45]
5	H	4-Br	80	[46]
6	Cl	4-NO_2	79	–
7	Cl	3-NO_2	71	[46]

* The ratio of *o*-aminophenol (mmol):aldehyde (mmol):$KMnO_4$ (mmol):HOAc (mL) is equal to 1:1:3.4:0.08

^{1}H NMR (400 MHz, acetone): δ, ppm (*J*, Hz): 8.25 (sbr, 1H), 7.8 (m, 3H), 8.64 (sbr, 1H), 7.7 (sbr, 1H), 7.45 (sbr, 2H).

2-(4-Bromophenyl)-1,3-benzoxazole

FT-IR (ATR) = ῡ, cm^{-1}: 3057(C–H), 1616 (C=N), 1592, 1483 (C=C), 1294 (C–N), 1243 (C–O), 1068 (C–Br).

^{1}H NMR (400 MHz, DMSO-d_6): δ, ppm (*J*, Hz): 8.62 (sbr, 2H), 8.24 (sbr, 4H), 7.88 (sbr, 2H).

2-(4-Metoxycarbonyl phenyl) 1,3-benzoxazole

FT-IR (ATR) = ῡ, cm^{-1}: 3163 (C–H), 2955 (C–H), 1726 (C=O), 1605 (C=N), 1557, 1454 (C=C), 1276 (C–O).

^{1}H NMR (400 MHz, DMSO-d_6): δ, ppm (*J*, Hz): 8.71 (sbr, 4H), 8.23 (sbr, 2H), 7.92 (sbr, 2H), 4.35 (s, 3H). $C_{15}H_{11}NO_3$, calculated: C, 71.14; H, 4.38; N, 5.53. Found: C, 70.64; H, 4.42; N, 5.89 %.

5-Chloro, 2-(4-nitrophenyl)-1,3-benzoxazole

FT-IR (ATR) = ῡ, cm^{-1}: 3093 (C–H), 1602 (C=N), 1552, 1348 (N=O), 1519, 1453 (C=C), 1285 (C–O), 1063 (C–Cl).

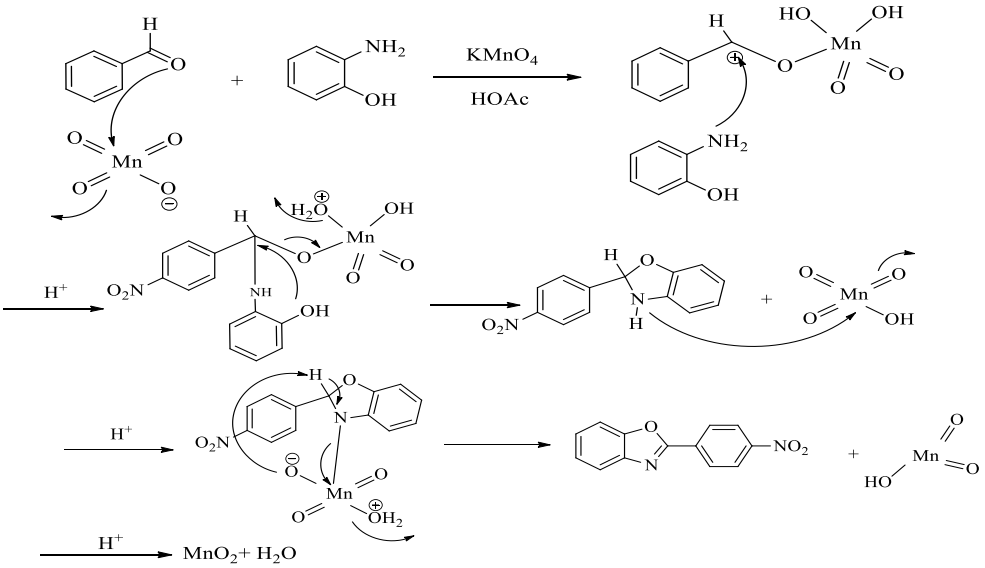
^{1}H NMR (400 MHz, acetone): δ, ppm (*J*, Hz): 8.49 (sbr, 4H), 7.8–8.0 (m, 2H), 7.5 (sbr, 1H).

$C_{13}H_7ClN_2O_3$, calculated: C, 56.85; H, 2.57; N, 10.20. Found: C, 56.75; H, 2.62; N, 10.25 %.

5-Chloro, 2-(3-nitrophenyl)-1,3-benzoxazole

FT-IR (ATR) = ῡ, cm^{-1}: 3093 (C–H), 1621 (C=N), 1527, 1353 (N=O), 1449 (C=C), 1195 (C–O), 1101 (C–Cl).

Scheme 2 A proposed mechanism for synthesis of benzoxazoles using $KMnO_4$/HOAc system



^{1}H NMR (400 MHz, acetone): δ, ppm (*J*, Hz): 9.0 (sbr, 1H), 8.77 (sbr, 1H), 8.5 (sbr, 1H), 7.8 (sbr, 1H), 7.9 (sbr, 1H), 8.0 (sbr, 2H).

5-Chloro, 2-(4-chlorophenyl)-1,3-benzoxazole

FT-IR (ATR) = $\bar{\upsilon}$, cm^{-1}: 3066 (C–H), 1611 (C=N), 1550, 1481 (C=C), 1332 (C–N), 1261 (C–O), 1089 (C–Cl).

^{1}H NMR (400 MHz, acetone): δ, ppm (*J*, Hz): 8.3 (sbr, 2H), 7.6–7.8 (m, 4H), 8.64 (sbr, 1H), 7.5 (sbr, 1H). $C_{13}H_7Cl_2NO$, calculated: C, 59.12; H, 2.67; N, 5.30. Found: C, 58.92; H, 2.45; N, 5.72 %.

5-Chloro, 2-(2,4-dichlorophenyl)-1,3-benzoxazole

FT-IR (ATR) = $\bar{\upsilon}$, cm^{-1}: 3092 (C–H), 1586 (C=N), 1561, 1448 (C=C), 1388 (C–N), 1257 (C–O), 1092 (C–Cl).

^{1}H NMR (400 MHz, $CDCl_3$): δ, ppm (*J*, Hz): 8.25 (sbr, 1H), 7.81 (sbr, 1H), 7.4–7.5 (m, 2H), 7.01–7.12 (m, 2H), ppm. $C_{13}H_6Cl_3NO$, calculated: C, 52.30; H, 2.03; N, 4.69. Found: C, 52.15; H, 2.39; N, 4.48 %.

Results and discussion

To find the optimum conditions for the synthesis of benzoxazoles from *o*-hydroxyarylidene anilines in the presence of $KMnO_4$, we have synthesized 2-(4-nitrophenyl)-1,3-benzoxazole under various conditions. The reactions at different ratios of reactant/oxidant/acid at room temperatures have revealed that the best conditions are solvent free at room temperature with a ratio of phenolic Schiff's bases (mmol): $KMnO_4$ (mmol): HOAc (ml) equal to 1:1.7:0.08 (Table 1).

According to optimized conditions, we have improved the synthesis of benzoxazoles via oxidative cyclization of corresponding *o*-hydroxyarylidene anilines in the presence of $KMnO_4$/HOAc system (Table 2).

On the other hand, we have attempted to prepare benzoxazoles in a one-pot procedure from *o*-aminophenol and aldehyde in the presence $KMnO_4$/HOAc system. Optimum conditions for the one-pot synthesis of benzoxazoles in the presence of $KMnO_4$ and HOAc were achieved by synthesis of 2-(4-nitrophenyl)-1,3-benzoxazole under various conditions. The reactions at different ratios of reactant/oxidant/acid at room temperatures have revealed that the best conditions are solvent free at room temperature with a ratio of *o*-aminophenol (mmol):aldehyde (mmol):$KMnO_4$ (mmol): HOAc (mL) equal to 1:1:3.4:0.08 (Table 3).

According to modified conditions, we have improved the one-pot synthesis of benzoxazoles in the presence of $KMnO_4$/HOAc system under solvent-free condition at room temperature (Table 4).

Our proposed mechanism for 2-(aryl)-benzoxazoles synthesis is shown in Scheme 2. By addition oxygen of aldehyde carbonyl group to $KMnO_4$, addition of 2-aminophenol to aldehyde is accelerated and finally dihydrobenzoxazole is formed. The resulted $HMnO_4$ oxidize dihydrobenzoxazole to benzoxazole.

In conclusion, $KMnO_4$/HOAc system promotes synthesis of 2-(aryl)-benzoxazoles at room temperature under solvent-free condition. High to excellent yields, short reaction times, easy workup and low cost are some advantages of this protocol.

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