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ORIGINAL PAPER

Direct oxidative conversion of benzylhalides, -amines, -alcohols, and arylaldehydes to nitriles with *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane activated by NH_4Br

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Abstract A simple and efficient oxidative conversion of benzyl derivatives of halides, amines, alcohols, and aldehydes into corresponding nitriles is described using *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane in the presence of NH_4Br. The reactions proceeded smoothly at room temperature to afford the products in high-to-excellent yields.

Keywords *Trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane · DHPDMDO · Oxidation · Nitrile · Amine · Halide · Alcohol · Aldehyde

Introduction

Nitriles are known as important and versatile intermediates in synthetic organic chemistry for conversion into various functional groups such as amines, ketones, amides, amidines, carboxylic acids, esters, *N*-heterocycles, etc. [1–6]. In addition, nitriles are industrially useful building blocks of dyes, natural products, herbicides, and agrochemicals [7, 8]. Nitriles are also of pharmaceutical importance as HIV protease inhibitors, 5-lipoxygenase inhibitors, and many other bioactive molecules [9–12]. Several methods are known for the preparation of nitriles that include (1) nucleophilic displacement of various groups such as halogens, sulfonates, alcohols, esters, ethers, nitro or amino, and diazonium groups with inorganic cyanides [13–22]; (2) dihydration of amides [23, 24] and aldoximes [25–27]; and (3) direct conversion of aldehydes into nitriles via the corresponding aldoximes [28–32], or using reagents such as trimethylsilyl azide [33], triazidochlorosilane [34], sodium azide and aluminum chloride [35]. The transformation of aldehydes into their corresponding nitriles using ammonium salts in combination with an appropriate oxidant has emerged as an expedient method. Reagents reported to effect this conversion are $NH_3/H_2O_2/CuCl$ [36], $NH_3/O_2/CuCl_2.2H_2O/MeONa$ [37], $NH_3/NBS/H_2O$ [38], NH_3/I_2 [39], $NH_3/Pb(OAc)_4$ [40], $NH_3/S_8/NaNO_2$ [41], and $MeReO_3/H_2O_2$ [42]. Recently, the synthesis of nitriles has been developed by treating aldehydes with hydroxylamine hydrochloride in the presence of catalysts such as ZnO [43] and KF/Al_2O_3 [44]. In addition, a few reports have been published on one-pot oxidation of alcohols to nitriles [45–47]. The most common approach for this conversion is based on Mitsunobu's reaction using HCN/Ph_3P/diethyl azadicarboxylate (DEAD) [48–50]. Other reagents used for direct conversion of alcohols to nitriles include Ph_3P/*n*-Bu_4NCN/2,3-dichloro-5,6-dicyanobenzoquinone (DDQ) [51], $CCl_4/Ph_3P/NaCN$ [52], *n*-$Bu_3P/CCl_4/KCN$/18-crown-6 [53], $Me_3SiCl/NaI/NaCN$ [54, 55], and NH_3/1,3-diiodo-5,5-dimethyl hydantoin [56]. Huang et al. have recently reported the use of ruthenium hydroxide in a highly selective and green aerobic oxidation of amines to nitriles in water [57]. Also, the use of a ruthenium(III) complex bearing phenylpyridino as an efficient catalyst in an aerobic dehydrogenation of benzylamines to the corresponding benzonitriles has been reported [58]. However, most of the reported methods are subject to certain drawbacks including prolonged reaction times, low yield, use of highly toxic reagents such as HCN or metal cyanides, necessity of using costly and excess catalysts, and harsh reaction conditions. Nevertheless, the development of alternative milder and environmentally green approaches to aromatic and aliphatic nitriles appears as interesting challenge.

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Experimental

Material and instruments

Chemicals were purchased from the Merck company and used without further purification. ^{1}H and ^{13}C NMR spectra were obtained on a Bruker 500 MHz Avance spectrometer with $CDCl_3$ as solvent. FT-IR spectra on were recorded a Bruker 500 scientific spectrometer in KBr pellets. *Trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane was prepared following our previously reported procedure [59–62].

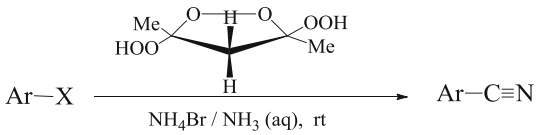
Caution Although we did not encounter any problem with *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2 dioxolane, it is potentially explosive and should be handled with precautions; all reactions should be carried out behind a safety shield inside a fume hood and transition metal salts or heating should be avoided.

Preparation of *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane

To a stirred solution of acetylacetone (100 mg, 1 mmol) in CH_3CN (5 mL) was added $SnCl_2{\cdot}2H_2O$ (45 mg, 0.2 mmol) and the resulting mixture was stirred for 5 min at room temperature. Then, aqueous 30 % H_2O_2 (5 mmol) was added to the reaction mixture and stirred for 12 h at room temperature. After completion of the reaction as monitored by TLC, water (15 mL) was added and the product was extracted with ethylacetate (2 × 10 mL). The combined organic layer was dried over anhydrous $MgSO_4$ and evaporated under reduced pressure to give almost pure white crystalline product in 85 % yield (140 mg); mp 98–100 °C.

General experimental procedure for the conversion of benzyl derivatives of alcohols, amines, halides, and aryl aldehydes into corresponding nitriles

To a mixture of benzyl substrate or aryl aldehyde (1 mmol), aqueous ammonia (3.0 mL, 45 mmol) and NH_4Br (0.11 g, 1.1 mmol) was added *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane (166 mg, 1 mmol). The reaction mixture was stirred at room temperature for an appropriate time (Tables 3, 4, 5). After completion of the reaction as monitored by TLC, the mixture was treated with aqueous Na_2SO_3 solution (3 mL) with stirring for 10 min to neutralize the remaining peroxide. The resulting mixture was diluted with distilled water (15 mL) and then the product was extracted with Et_2O (2 × 10 mL). The combined organic layer was dried over anhydrous $MgSO_4$ and evaporated under reduced pressure to leave the pure product. Physical and spectroscopic (IR, ^{1}H NMR, and ^{13}C NMR) data of all the products matched those reported.



(X = CH_2OH; CH_2NRR'; CH_2Hal; CHO)

Scheme 1 Oxidative conversion of benzyl derivatives into nitriles with *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane

Table 1 Effect of various oxidants on the NH_4Br-activated conversion of benzyl alcohol (**1a**) into benzonitrile (**2a**) in aqueous ammonia

Entry	Oxidant	Time (min)	Yield (%)[a]
1	H_2O_2	300	–
2	TBHP	300	29
3	NaOCl	300	–
4	DHPDMDO	100	95
5	Al_2O_3	300	25
6	CuO	300	15
7	–	300	–

Conditions: $PhCH_2OH$ (1 mmol), aqueous NH_3 (3 mL, 45 mmol), NH_4Br (1.1 mmol), oxidant (1 mmol), rt

[a] Isolated yield

Results and discussion

In continuation of our efforts in the synthesis of *gem*-dihydroperoxides [59–62], and their applications in various organic transformations [63–66], herein, we wish to report for the first time the versatile oxidative capability of *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane (DHPDMDO) as a non-toxic oxidant with high atom-efficiency in direct oxidative conversion of various benzyl derivatives including alcohols, amines, halides as well as aldehydes to their corresponding nitriles in the presence of aqueous ammonia and NH_4Br at room temperature (Scheme 1).

To establish the reaction conditions, we initially examined the oxidative conversion of benzyl alcohol (**1a**) to benzonitrile (**2a**) as the model reaction. To screen the effect of the oxidant in this reaction, various oxidants including H_2O_2, *t*-butyl hydrogen peroxide (TBHP), NaOCl, Al_2O_3, CuO as well as *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane (DHPDMDO) were examined in the presence of NH_4Br as the promoter in aqueous ammonia at room temperature. The results are summarized in Table 1.

As shown in Table 1, *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane (DHPDMDO) appeared as the most efficient oxidant when activated by NH_4Br to afford

the highest yield (95 %) in a short reaction time (100 min) in aqueous ammonia (entry 4). It was noticed that, no reaction occurs when conducted in the absence of the oxidant (entry 7).

In the later step, we studied the activating role of the salt on the model reaction using DHPDMDO with examining the catalytic potency of several inorganic salts such as KF, KI, NH_4Br and NH_4I under different loadings (Table 2). The results summarized in Table 2 demonstrate that, among different salts examined in this reaction, NH_4Br with a loading of 1.1 mmol was found to be the most efficient salt in terms of the reaction rate and the yield to promote the reaction (entry 6). It was also noticed that, the yield was increasingly enhanced by increasing the amount of NH_4Br in the reaction (entries 4–6) until the maximum yield (95 %) was reached (entry 6). Moreover, not only the yield was not improved by further increasing the amount of NH_4Br, but considerable diminution of the yield was observed after longer reaction times (entries 7, 8). The significance of the catalyst NH_4Br in the reaction was approved by conducting the reaction in the absence of the catalyst that resulted in no detectable amount of the product after a long reaction time (entry 9).

Table 2 Effect of various catalysts on the conversion of benzyl alcohol into benzonitrile with DHPDMDO in aqueous ammonia

Entry	Catalyst (mmol)	Time (min)	Yield (%)[a]
1	KI (0.1)	100	50
2	KF (0.1)	100	35
3	NH_4I (0.1)	100	65
4	NH_4Br (0.1)	100	70
5	NH_4Br (0.5)	100	80
6	NH_4Br (1.1)	100	95
7	NH_4Br (1.5)	150	50
8	NH_4Br (2.2)	150	50
9	–	150	–

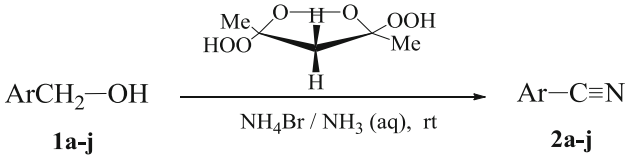
Conditions: $PhCH_2OH$ (1 mmol), aqueous NH_3 (3 mL, 45 mmol), oxidant (1 mmol), rt

[a] Isolated yield

To explore the generality of this method, we applied the optimized conditions, i.e., NH_4Br (1.1 mmol), DHPDMDO (1 mmol), aqueous NH_3 (45 mmol), room temperature, to a series of differently substituted benzyl alcohols **1a–j** into their corresponding nitriles **2a–j** as summarized in Table 3.

As seen in Table 3, the electron-withdrawing groups attached to the phenyl ring generally reduce the yields, whereas the electron-releasing groups cause slight improvement of the yields. It is noticed that, both the hydroxymethyl groups present in 1,4-di(hydroxymethyl)benzene **1d** undergo conversion into nitrile groups in this reaction (entry d). However, other groups attached to the phenyl ring such as halogens, nitro and aldehyde groups remained intact and only the hydroxymethyl group was selectively converted to nitrile group. Nevertheless, all the reactions proceeded smoothly under mild conditions to afford the respective products in high-to-excellent yields (98–84 %).

Table 3 Direct conversion of benzyl alcohols **1a–j** to benzyl nitriles **2a–j** with DHPDMDO using NH_4Br in aqueous NH_3

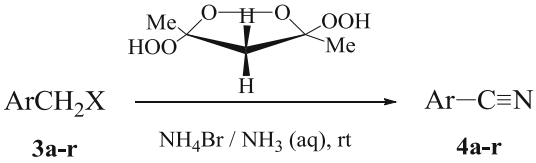


Entry	Ar	Product **2**	Time (min)	Yield (%)[a]
a	C_6H_5	C_6H_5CN	100	95
b	2-MeC_6H_4	2-MeC_6H_4CN	100	96
c	4-$MeOC_6H_4$	4-$MeOC_6H_4CN$	90	95
d	4-$HOCH_2C_6H_4$	4-CNC_6H_4CN	90	98
e	4-$NO_2C_6H_4$	4-$NO_2C_6H_4CN$	80	86
f	3-BrC_6H_4	3-BrC_6H_4CN	80	88
g	4-IC_6H_4	4-IC_6H_4CN	90	87
h	4-CNC_6H_4	4-CNC_6H_4CN	100	84
i	4-$CHOC_6H_4$	4-$CHOC_6H_4CN$	100	84
j	2-Cl-4-$NO_2C_6H_3$	2-Cl-4-$NO_2C_6H_3CN$	100	86

Conditions: $ArCH_2OH$ (1 mmol), aqueous NH_3 (3 mL, 45 mmol), NH_4Br (1.1 mmol), DHPDMDO (1 mmol), rt

[a] Isolated yield

Table 4 Conversion of benzyl amines **3a–i** and benzyl halides **3j–r** into corresponding nitriles with DHPDMDO/NH_4Br

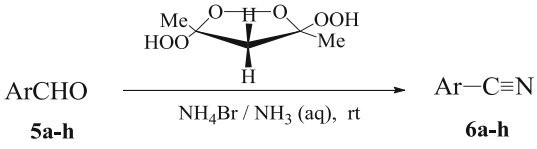


Entry	Ar	X	Product **4**	Time (min)	Yield (%)[a]
a	C_6H_5	NH_2	C_6H_5CN	120	78
b	4-$NO_2C_6H_4$	NH_2	4-$NO_2C_6H_4CN$	90	95
c	3-BrC_6H_4	NH_2	3-BrC_6H_4CN	100	83
d	4-IC_6H_4	NH_2	4-IC_6H_4CN	120	82
e	4-CNC_6H_4	NH_2	4-CNC_6H_4CN	90	98
f	4-$CHOC_6H_4$	NH_2	4-$CHOC_6H_4CN$	90	96
g	2-Cl-4-$NO_2C_6H_3$	NH_2	2-Cl-4-$NO_2C_6H_3CN$	100	86
h	C_6H_5	$NH(CH_3)$	C_6H_5CN	150	83
i	C_6H_5	$N(CH_3)_2$	C_6H_5CN	200	83
j	C_6H_5	Cl	C_6H_5CN	140	75
k	C_6H_5	Br	C_6H_5CN	100	83
l	4-$NO_2C_6H_4$	Cl	4-$NO_2C_6H_4CN$	90	78
m	3-BrC_6H_4	Cl	3-BrC_6H_4CN	120	78
n	4-IC_6H_4	Cl	4-IC_6H_4CN	100	88
o	4-CNC_6H_4	Cl	4-CNC_6H_4CN	90	78
p	4-$CHOC_6H_4$	Cl	4-$CHOC_6H_4CN$	90	80
q	2-Cl-4-$NO_2C_6H_3$	Cl	2-Cl-4-$NO_2C_6H_3CN$	120	80
r	2-Cl-4-$NO_2C_6H_3$	Br	2-Cl-4-$NO_2C_6H_3CN$	90	85

Conditions: $ArCH_2X$ (1 mmol), aqueous NH_3 (3 mL, 45 mmol), NH_4Br (1.1 mmol), DHPDMDO (1 mmol), rt

[a] Isolated yield

Table 5 Direct conversion of aryl aldehydes **5a–h** to benzonitriles **6a–h** with DHPDMDO using NH_4Br in aqueous ammonia



Entry	Ar	Product **6**	Time (min)	Yield (%)[a]
a	C_6H_5	C_6H_5CN	50	94
b	2-MeC_6H_4	2-MeC_6H_4CN	50	95
c	4-$NO_2C_6H_4$	4-$NO_2C_6H_4CN$	45	83
d	3-BrC_6H_4	3-BrC_6H_4CN	45	83
e	4-IC_6H_4	4-IC_6H_4CN	40	78
f	4-CNC_6H_4	4-CNC_6H_4CN	40	82
g	4-$CHOC_6H_4$	4-$CHOC_6H_4CN$	50	78
h	2-Cl-4-$NO_2C_6H_3$	2-Cl-4-$NO_2C_6H_3CN$	50	78

Conditions: $ArCH_2OH$ (1 mmol), aqueous NH_3 (3 mL, 45 mmol), NH_4Br (1.1 mmol), DHPDMDO (1 mmol), rt

[a] Isolated yield

The successful accomplishment of the oxidation of benzyl alcohols into corresponding nitriles with *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane encouraged us to explore the suitability of this reagent for oxidative conversion of other benzyl-bearing functionalities including amines, halides as well as aldehydes. We preliminarily studied the oxidative conversion of benzyl amine **3a** as the test compound into the corresponding benzonitrile with *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane. The reaction was conducted under the aforementioned optimized conditions which led to the formation of the desired product **4a** in high yield (78 %). The crucial roles played by both the oxidant DHPDMDO and NH_4Br in this reaction were evaluated by carrying out the reaction in the absence of either the oxidant or NH_4Br and no expected transformation was observed in either case. The generality of this reaction was examined by extending it to a series of benzyl amines **3a–i** bearing different groups in the aromatic ring. According to the results summarized in Table 4, all the reactions proceeded smoothly to furnish the respective nitriles **4a–i** in high-to-excellent yields (78–98 %). Similarly, this method was found equally convenient for conversion of different benzyl halides **3j–r** into corresponding nitriles **4j–r** in high yields (75–88 %) as summarized in Table 4 (entries **j–r**). As seen in this table, these reactions also occur selectively while the other groups attached to the phenyl ring remain resistant. Alternatively, this method can conveniently provide a direct and environmentally more benign approach to aromatic nitriles in comparison with other previously reported methods such as the Sandmeyer reaction of diazonium compounds with toxic CuCN or dehydration of aromatic amides with dehydrating agents.

Finally, in order to further generalize the present method and extend it to conversion of other functional groups, we were encouraged to continue our research with direct transformation of aryl aldehydes into corresponding nitriles by treatment with *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane under the promotion of NH_4Br in aqueous NH_3 at room temperature. Several aryl aldehydes **5a–h** were readily converted into the corresponding nitriles **6a–h** in high yields as summarized in Table 5. As seen in this table, all these reactions proceeded in shorter reaction times in comparison with the aforementioned reactions. It is interesting to note that, from two aldehyde groups

attached to the phenyl ring in 1,4-benzendicarboxaldehyde (entry **g**) only one is converted to afford the mononitrilated product **6g**.

Conclusions

In summary, different benzyl derivatives of amines, alcohols, halides and also aryl aldehydes have been effectively converted to their corresponding nitriles with *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane as an effective and high potent oxidant under the promotion of NH_4Br in aqueous ammonia. The reactions proceeded under mild conditions at room temperature to afford the respective products in good to excellent yields. This protocol can be considered as environmentally benign since neither the reagent nor the catalyst used in this method is toxic.

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