

Regioselective bromination and iodination of aromatic substrates promoted by *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane

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Abstract Selective and efficient bromination and iodination of aromatic compounds by ammonium bromide and ammonium iodide, respectively, under promotion of *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane have been explored. Mild reaction conditions, high selectivity and yield, and high reaction rate are some of the major advantages of this synthetic method.

Keywords *Trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane · Aromatic substrate · Bromination · Iodination · Ammonium bromide · Ammonium iodide

Introduction

Bromination and iodination of aromatic substrates have received a great deal of interest in recent years [1–6]. This interest stems from the commercial and biological importance of aryl halides as industrial intermediates for the manufacture of specialty chemicals, agrochemicals, pharmaceuticals [7], and also as potent antitumor, antibacterial, antifungal, antineoplastic, antiviral, and antioxidants [8]. Also, these compounds are key intermediates in the preparation of organometallic reagents [9, 10], and play important role in transition metal-mediated coupling reactions [11–14]. Often, bromination of aromatic substrates performed with elemental bromine poses practical

problems such as difficulty in handling, formation of hydrobromic acid as by-product which can reduce the atom efficiency by 50%. Also, the hydrobromic acid generated requires to be neutralized before it is allowed to enter the atmosphere. Likewise, the elemental iodine lacks enough reactivity for direct iodination of aromatic substrates and the hydriodic acid generated in the course of the reaction can cause proteolytic cleavage of sensitive groups. As a result, the presence of some activating agents in the reaction is required to produce iodonium (I^+) as a strong electrophile. In this regard, iodination of aromatic substrates has been carried out with elemental iodine combined with strong oxidizing agents such as nitric acid, sulfuric acid, iodic acid, sulfur trioxide and hydrogen peroxide [15, 16]. In recent years, several protocols have been developed employing iodonium (I^+) generating systems such as iodine nitrogen dioxide [17], I_2/HgO [18], iodine monochloride [19], bis(pyridine)iodonium (I) tetrafluoroborate- CF_3SO_3OH [20], $NaOCl-NaI$ [21], $I_2-Na_2S_2O_8$ [22], $I_2-(NH_4)_2S_2O_8-CuCl_2-AgSO_4$ [23], I_2 -tetrabutylammonium peroxydisulfate [24], I_2 -diiodine pentoxide [25], I_2 -lead acetate [26], I_2 -thallium acetate [27], I_2 -TEDA- BF_4 [28], $BuLi-F_3CCH_2I$ [29], KI -oxone [30], bis(symocollidine)-iodine(I) hexafluorophosphate [31], $KI-H_2O_2-H_2SO_4$ [32], $ICl-In(OTf)_3$ [33], HIO_4/Al_2O_3 [34], NaI /chloramine T [35], KI/H_2O_2 or sodium perborate/sodium tungstate [36], $I_2/O_2/H_3PV_2Mo_{10}O_{40}$ [37], $KI/KIO_3/H^+$ [38], and I_2/UHP [39]. However, most of these methods are subject to certain drawbacks such as high cost, toxicity of the transition metal catalysts, low yield and selectivity.

Similarly, to avoid the limitations associated with direct bromination, a variety of brominating agents have been reported [40, 41]. Recent reports describe the use of NBS-sulfonic acid-functionalized silica [42], NBS- NH_4OAc [43], NBS/ BF_3-H_2O [44], NBS/ Al_2O_3 [45], NBS-TEAB [46],

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NBS–DMF (or THF) [47], NBS–Pd(OAc)₂ [48], 2:1 bromide/bromate [49], *N*-methylpyrrolidin-2-one hydrotribromide–H₂O₂ [50], Br₂/SO₂Cl₂/zeolite [51], 1,2-dipyridiniumdibromide–ethane [52], [Bmim]Br₃ [53], alkylpyridinium tribromide [54], IBX amide-resin–TEAB [55], poly(4-vinylpyridine)-supported bromate [56], NH₄VO₃–H₂O₂–HBr [57], bromodichloroisocyanuric acid [58], CuBr₂ [59], ZrBr₄/diazene [60], etc. Recently, we described the efficient use of 1,3-dibromo-5,5-dimethyl hydantoin (DBH) in selective bromination of sydnones [61]. However, in order to avoid the limitations allocated to these reported methods, more robust and convenient methods and reagents for bromination of aromatic compounds are still in demand. In this regard, the use of bromine ion (Br⁺) generated in situ in the presence of oxidizing agents such as hydrogen peroxide and sodium perborate has been reported for bromination of aromatic substrates [62].

Experimental

Chemicals were obtained from Merck chemical company. FT-IR spectra were recorded on a Shimadzu 435-U-04

spectrophotometer (KBr pellets). NMR spectra were recorded on a 200 (50) MHz Varian spectrometers, and are reported in CDCl₃ using TMS as internal standard. Melting points were measured on a SMPI apparatus.

Caution Although we did not encounter any problem with *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane **1**, 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 and heating should be avoided.

General procedure for bromination and iodination of aromatic substrates

To a mixture of aromatic substrate (1 mmol) and appropriate amount of NH₄Br (Table 1) in AcOH (or MeCN for anilines) (4 mL) was added *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane **1** (0.5 mmol) and the mixture was stirred at room temperature for an appropriate time (Table 1). After completion of the reaction as monitored by TLC (*n*-hexane/EtOAc; 2:1), 1 M aqueous solution of Na₂SO₃ (3 mL) was added to quench the reaction and stirring was continued for further 10 min. Then, the reaction mixture was diluted with

Table 1 Bromination of aromatic substrates with NH₄Br promoted by *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane at room temperature

Entry	Substrate	Product	NH ₄ Br (mmol)	Time (h)	Yield (%) ^a	Mp (°C)	
						Found	Reported [60, 62]
1	C ₆ H ₅ OH	4-BrC ₆ H ₄ OH	1.1	0.5	92	62–64	63
2	C ₆ H ₅ OH	2,4-Br ₂ C ₆ H ₃ OH	2.2	1	94	41–43	40
3	C ₆ H ₅ OH	2,4,6-Br ₃ C ₆ H ₂ OH	3.5	2	90	88–90	85–86
4	2-BrC ₆ H ₄ OH	2,4-Br ₂ C ₆ H ₃ OH	1.1	1.5	87	39–41	40
5	4-BrC ₆ H ₄ OH	2,4-Br ₂ C ₆ H ₃ OH	1.1	1	90	39–41	40
6	2,6-Br ₂ C ₆ H ₃ OH	2,4,6-Br ₃ C ₆ H ₂ OH	1.1	2	85	88–90	85–86
7	2-ClC ₆ H ₄ OH	4-Br-2-ClC ₆ H ₃ OH	1.1	3	82	52–54	55
8	2,4-Cl ₂ C ₆ H ₃ OH	6-Br-2,4-Cl ₂ C ₆ H ₂ OH	1.1	4	80	64–66	62
9	2,6-Cl ₂ C ₆ H ₃ OH	4-Br-2,6-Cl ₂ C ₆ H ₂ OH	1.1	4	84	68–70	66
10	3-MeC ₆ H ₄ OH	4-Br-3-MeC ₆ H ₃ OH	1.1	1	78	56–58	54
11	C ₆ H ₅ OMe	4-BrC ₆ H ₄ OMe	1.1	1	76	17–18	15
12	4-ClC ₆ H ₄ OH	2-Br-2-ClC ₆ H ₃ OH	1.1	4	88	32–34	32–34
13	2-MeC ₆ H ₄ OH	4-Br-2-MeC ₆ H ₃ OH	1.1	1.2	97	64–66	63–64
14	2,6-Me ₂ C ₆ H ₃ OH	4-Br-2,6-Me ₂ C ₆ H ₂ OH	1.1	1	95	80–82	78–79
15	2,4-Me ₂ C ₆ H ₃ OH	6-Br-2,4-Me ₂ C ₆ H ₂ OH	1.1	2	98	232–234	232
16	4-NO ₂ C ₆ H ₄ OH	2-Br-4-NO ₂ C ₆ H ₃ OH	1.5	5	87	117–119	114–115
17	2-NO ₂ C ₆ H ₄ OH	4-Br-2-NO ₂ C ₆ H ₃ OH	1.3	4	86	94–96	91–93
18	C ₆ H ₅ NMe ₂	4-BrC ₆ H ₄ NMe ₂	1	0.25	88	56–58	55–56
19	C ₆ H ₅ NH ₂	4-BrC ₆ H ₄ NH ₂	1	0.25	81	56–58	57–58
20	4-BrC ₆ H ₄ NH ₂	2,4,6-Br ₃ C ₆ H ₂ NH ₂	2.2	1	92	124–126	121–123
21	C ₆ H ₅ NH ₂	2,4,6-Br ₃ C ₆ H ₂ NH ₂	3.5	2	90	124–126	124–126
22	2-ClC ₆ H ₄ NH ₂	4-Br-2-ClC ₆ H ₃ NH ₂	1.1	1.3	82	73–75	71–73
23	4-ClC ₆ H ₄ NH ₂	2-Br-4-ClC ₆ H ₃ NH ₂	1.1	1.5	85	66–68	64–66
24	4-CNC ₆ H ₄ NH ₂	2-Br-4-CNC ₆ H ₃ NH ₂	1.3	3.5	93	110–112	108–110

Conditions: substrate (1 mmol), oxidant **1** (0.5 mmol; 1 mmol for entry 2 and 1.5 mmol for entry 3), room temperature, AcOH as the solvent (reactions with anilines were conducted in MeCN)

^a Isolated yields

Table 2 Iodination of aromatic substrates with NH₄I promoted by *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane at room temperature

Entry	Substrate	Product	NH ₄ Br (mmol)	Time (h)	Yield (%) ^a	Mp (°C)	
						Found	Reported [39, 63]
1	C ₆ H ₅ NH ₂	4-IC ₆ H ₄ NH ₂	1	2	88	64–66	63–65
2	C ₆ H ₅ NH ₂	2,4-I ₂ C ₆ H ₃ NH ₂	2.2	2	86	94–96	94–95
3	C ₆ H ₅ NMe ₂	4-IC ₆ H ₄ NMe ₂	1	1.5	95	82–84	81–82
4	C ₆ H ₅ NEt ₂	4-IC ₆ H ₄ NEt ₂	1	2	85	29–30	26–29
5	4-IC ₆ H ₄ NH ₂	2,4-I ₂ C ₆ H ₃ NH ₂	1.2	2	80	94–96	94–95
6	2-ClC ₆ H ₄ NH ₂	2-Cl-4-IC ₆ H ₃ NH ₂	1.2	4	75	72–74	70–73
7	2-MeC ₆ H ₄ NH ₂	4-I-2-MeC ₆ H ₃ NH ₂	1.1	2	92	88–90	86–88
8	4-MeOC ₆ H ₄ CO ₂ H	3-I-4-MeOC ₆ H ₃ CO ₂ H	1.4	5	70	228–230	225–228
9	4-BrC ₆ H ₄ NH ₂	4-Br-2-IC ₆ H ₃ NH ₂	1.5	4	65	72–74	69–70
10	C ₆ H ₆	1,4-I ₂ C ₆ H ₄	2.5	5	72	124–126	126–128
11	C ₆ H ₅ I	1,4-I ₂ C ₆ H ₄	1.3	5	80	124–126	126–128
12	C ₆ H ₅ CO ₂ Me	3-IC ₆ H ₄ CO ₂ Me	1.4	5	94	52–54	50–52
13	4-MeC ₆ H ₄ CO ₂ H	4-Me-3-IC ₆ H ₃ CO ₂ H	1.2	5	95	206–208	208–210
14	4-NO ₂ C ₆ H ₄ Me	2-I-4-NO ₂ C ₆ H ₃ Me	1.5	6	88	55–57	53–55

Conditions: substrate (1 mmol), oxidant **1** (0.5 mmol; 1 mmol for entries 2 and 10), room temperature, AcOH as the solvent (reactions with anilines were conducted in MeCN)

^a Isolated yields

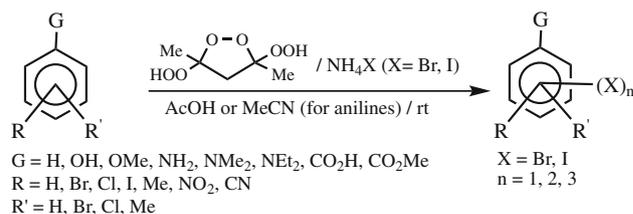
distilled water (15 mL). The precipitated product was filtered and washed with water (2 × 3 mL) to yield almost pure product.

For dibromination reactions use of excess amounts of NH₄Br and the oxidant **1** are required (Table 1). All the products were characterized by their physical and spectral (IR, ¹H NMR and ¹³C NMR) analysis and compared with the reported data [58, 60].

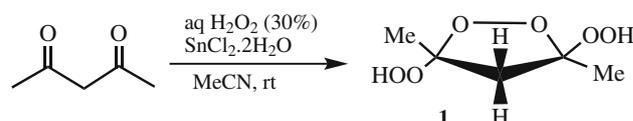
Exactly the same procedure was applied for iodination reactions except that NH₄I has been used instead of NH₄Br and the experimental results are summarized in Table 2. The physical and spectral data obtained for the iodinated products are in accord with those reported [39, 63].

Results and discussion

In connection with our continuing interest in the synthesis of *gem*-dihydroperoxides [64, 65], and their applications in various organic transformations [66–68], herein, we wish to report the novel use of *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane as a versatile and highly potent oxidant to promote the in situ generation of Br⁺ and I⁺ ions for bromination and iodination of aromatic substrates, respectively. In this method, NH₄Br and NH₄I have been used as the sources for HOBr and HOI, respectively, upon the initial reaction with *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane. The reactions proceeded selectively rapidly under mild conditions at room temperature in acetic acid or acetonitrile (for anilines only) to afford the corresponding brominated or iodinated aromatic products in high yields (Scheme 1). The experimental results obtained are summarized in Table 1. The products were



Scheme 1



Scheme 2

characterized based on their physical and spectral (IR, ¹H NMR and ¹³C NMR) analysis and compared with the reported data [60, 62].

Trans-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane (**1**) has been prepared in this laboratory following the reported procedure as a white crystalline compound in high yield (85%) from SnCl₂·2H₂O-catalyzed reaction of acetylacetone with aqueous (30%) hydrogen peroxide (Scheme 2) [66].

In order to establish the conditions for the titled reactions, we initially examined the bromination of phenol (1 mmol) as test compound. The effects of solvent and the oxidant **1** were studied using different solvents such as AcOH, CH₂Cl₂, CHCl₃, CCl₄, and MeCN, under various amounts of the oxidant **1**. It was noticed that, the best results for monobromination (92%, entry 1), dibromination (94%, entry 2) and tribromination (90%, entry 3) using 1.1, 2.2 and 3.5 mmols of NH₄Br, respectively, were obtained when the

reactions were conducted with stirring at room temperature in acetic acid as the solvent of choice although comparable yields were obtained with acetonitrile as well. Similarly, optimization for iodination reaction was carried out for aniline (1 mmol) as the test compound with NH_4I as the source of iodine ion under the promotion of the oxidant **1**. The optimum conditions found for iodination reaction were similar to those of the bromination reaction (0.5 mmol of oxidant **1**, room temperature, AcOH or MeCN as solvents). However, considering the probable acetylation of the amino group with acetic acid in anilines, the bromination and iodination reactions of the aniline substrates were preferably conducted in acetonitrile rather than in acetic acid. It is also likely that, the partial protonation of the amino group in anilines with acetic acid could occur which can reduce the reactivity of the anilines toward electrophilic halogenation reactions. Protonation of the amino group with acetic acid can also render the separation of the aniline products difficult in the course of work up due to the increase in their solubility in aqueous layer.

To develop the scope of these reactions, several other aromatic substrates were subjected to bromination and iodination under the optimized conditions. The experimental results are summarized in Tables 1 and 2. Almost all the reactions proceeded smoothly with high selectivity in relatively short reaction times to afford the corresponding brominated and iodinated products. It is important to note that, neither bromination nor iodination occurred with NH_4Br and NH_4I , respectively, in the absence of the oxidant **1**. This substantiates the importance of the oxidant **1** likely in promoting the in situ generation of Br^+ and I^+ ions which act as main brominating and iodinating species in these reactions. This protocol is associated with the advantages of mild conditions, high selectivity, easy manipulation, easy disposal of the remaining excess reagent in the reaction mixture owing to its solubility in water, easy control on further continuation of halogenation to di- and trihalo derivatives, and avoidance of the harmful and corrosive elemental bromine or hydrobromic acid in the reactions. In addition, as shown in

Table 3 Comparison of the present method with some other previously reported procedures using model bromination reactions

Substrate	Product	Reaction conditions	Time (h)	Yield (%) ^a	Reference
$\text{C}_6\text{H}_5\text{OH}$	4- $\text{BrC}_6\text{H}_4\text{OH}$	$\text{NH}_4\text{Br}/\text{trans-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane}/\text{rt}$	0.5	92	Present method
$\text{C}_6\text{H}_5\text{OH}$	4- $\text{BrC}_6\text{H}_4\text{OH}$	$\text{ZrBr}_4/\text{diazene mixture}/\text{rt}$	0.67	82	[60]
$\text{C}_6\text{H}_5\text{OH}$	4- $\text{BrC}_6\text{H}_4\text{OH}$	HMTA- $\text{Br}_2/-15^\circ\text{C}$	1.5	45	[69]
$\text{C}_6\text{H}_5\text{OH}$	4- $\text{BrC}_6\text{H}_4\text{OH}$	$\text{KBr}/\text{H}_2\text{O}_2/\text{Zeolite}$	5	58	[70]
$\text{C}_6\text{H}_5\text{OH}$	4- $\text{BrC}_6\text{H}_4\text{OH}$	Heteropoly acid cesium salt/ cetyltrimethylammonium bromide/ Br_2	15	94	[71]
$\text{C}_6\text{H}_5\text{OH}$	4- $\text{BrC}_6\text{H}_4\text{OH}$	$\text{KBr}/\text{PhCH}_2\text{Ph}_3\text{PHSO}_5/\text{H}_2\text{O}_2$	5	88	[62]
$\text{C}_6\text{H}_5\text{NH}_2$	4- $\text{BrC}_6\text{H}_4\text{NH}_2$	$\text{NH}_4\text{Br}/\text{trans-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane}/\text{rt}$	0.25	81	Present method
$\text{C}_6\text{H}_5\text{NH}_2$	4- $\text{BrC}_6\text{H}_4\text{NH}_2$	$\text{ZrBr}_4/\text{diazene mixture}/\text{rt}$	6	66	[60]
$\text{C}_6\text{H}_5\text{NH}_2$	4- $\text{BrC}_6\text{H}_4\text{NH}_2$	TBAB/ $\text{V}_2\text{O}_5\text{-H}_2\text{O}_2$	0.5	82	[72]
$\text{C}_6\text{H}_5\text{NH}_2$	4- $\text{BrC}_6\text{H}_4\text{NH}_2$	Tetrabutylammonium peroxydisulfate	1	80	[73]
$\text{C}_6\text{H}_5\text{NH}_2$	4- $\text{BrC}_6\text{H}_4\text{NH}_2$	HMTA- $\text{Br}_2/-15^\circ\text{C}$	0.75	75	[69]
$\text{C}_6\text{H}_5\text{NH}_2$	4- $\text{BrC}_6\text{H}_4\text{NH}_2$	Heteropoly acid cesium salt/ cetyltrimethylammonium bromide/ Br_2	15	43	[71]

^a Isolated yield

Scheme 3

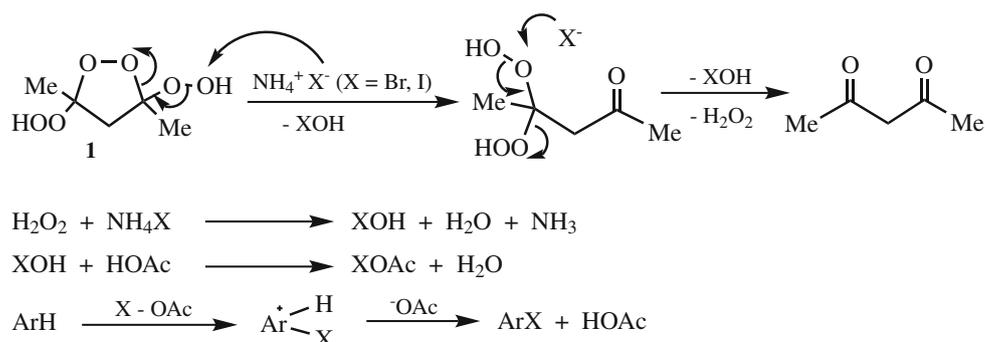


Table 3, the preference of our method over other previously published methodologies is due to the higher reaction rates and yields obtained from our model reactions in comparison with those reported.

A possible mechanism to explain the bromination and iodination of the aromatic substrates is depicted in Scheme 3. As shown in this scheme, these reactions are probably affected by Br^+ and I^+ ions as the main electrophiles which are likely generated in situ by initial reaction of NH_4Br or NH_4I , respectively, with *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane **1**.

Conclusions

In summary, *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane has been conveniently used as an efficient and highly potent oxidant for in situ generation of Br^+ and I^+ ions in reaction with NH_4Br and NH_4I , respectively. These ions can subsequently act as reactive electrophiles towards the aromatic substrates to afford the corresponding brominated or iodinated products in good to quantitative yields. The reactions proceed under mild conditions at room temperature with a simple workup procedure. This protocol is considered as environmentally benign since no toxic residues or corrosive acids are introduced to the environment.

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