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# A New and Versatile One-Pot Strategy to Synthesize alpha-Bromoketones from Secondary Alcohols Using Ammonium Bromide and Oxone

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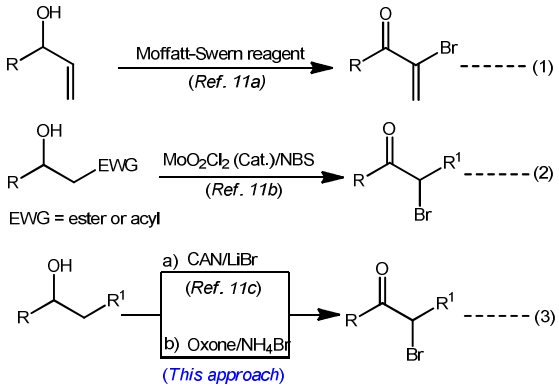
**A new, efficient and green protocol for one-pot synthesis of α-bromoketones from secondary alcohols using cheap, air stable and non-toxic reagents such as $NH_4Br$ and oxone has been developed. This reaction proceeds *via* two consecutive steps such as oxidation of secondary alcohol and oxidative bromination of *in situ* generated ketone.**

Cascade reactions, sequence of at least two consecutive chemical transformations, play an increasingly important role in synthetic organic chemistry because they address fundamental principles of synthetic efficiency and reaction design. The cascade reaction sequences are powerful synthetic tools for creating molecular complexity in one-pot in an economically and ecologically viable manner.[1] The main advantages of these one-pot reactions include the high atom economy, reduction of waste generated by the several chemical processes and minimization of the time and work required to carry out them. Due to these many advantages and increasing demand for more efficient and lower cost chemical processes, a lot of deal has been paid to synthesize complex molecules of both natural and designed by using these reactions. The increased interest in cascade sequences, over the past few decades, can be reflected by the numerous significant review articles published in various scientific journals.[1,2]

α-Bromomethyl ketones are the most versatile and essential building blocks in organic synthesis.[3] Due to the presence of both C-O and C-Br bonds in their structures, they show high electrophilic properties and undergo a series of reactions with a large number of nucleophiles to provide a variety of biologically active compounds.[4] For example, phenacyl bromides have been widely employing as precursors for various pharmaceutically important hetero aromatics such as imidazoles, selenazoles, and oxazoles.[5] Moreover, the derivatives of α-bromoketones have been investigated for their active participation in the inhibition of protein tyrosine phosphatase such as SHP-1 and PTP1B.[6]




**Scheme 1.** Different approaches for the preparation of α-bromoketones *via* tandem oxidation/halogenations.

Owing to the broad synthetic applicability of phenacyl bromides, a large number of methods have been developed for their preparation. Traditional methods involve the bromination of ketones[7] or their derivatives[8] (silyl enol ethers). Other methods employ the 1-bromoalkynes[9] (pre-synthesized from terminal alkynes) or alkenes[10] as starting materials for their preparation. In addition, the aryl allylic alcohols,[11a] β-hydroxy esters,[11b] and secondary alcohols[11c] have also been investigated for the synthesis of α-bromoketones *via* tandem oxidation/halogenations (Scheme 1). Nevertheless, most of these approaches suffer from one or more drawbacks such as

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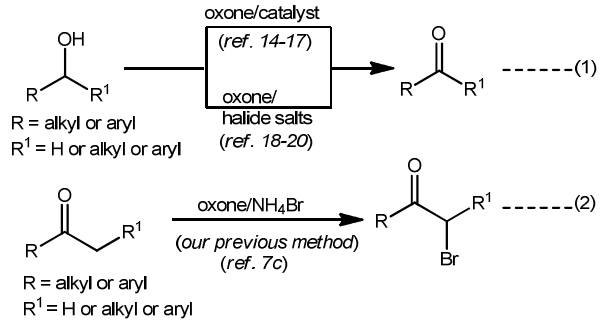
the use of costly or hazardous reagents, harsh reaction conditions, long reaction times, difficult work-up procedures, transition metal catalysts, liberation of excess organic waste originates from stoichiometric oxidants or brominating reagents, over brominations and limited substrate scope. Therefore, there is still strong demand for the development of new, efficient and eco-friendly procedures for their preparation.

Oxone, a triple salt containing potassium peroxymonosulfate, has been considering as an environmentally benign oxidant due to its inherent advantages such as non-toxic nature, ease of transport and handling, cheap, stable, non-polluting by-products and readily availability. It has been widely employing as a mild and versatile reagent in synthetic organic chemistry.[12] However, to the best of knowledge, oxone has not been utilized for the direct preparation of phenacyl bromides from alcohols. In continuation of our research program to develop new and eco-friendly protocols using oxone,[7c,13] we report herein a novel, efficient and inexpensive conventional cascade approach for the synthesis of *α*-bromoketones from alcohols, using ammonium bromide as an electrophilic bromine precursor and oxone as an oxidant, *via* tandem oxidation of secondary alcohols/oxidative halogenation of *in situ* generated ketones in one-pot (Scheme 1, eqn. 3b).

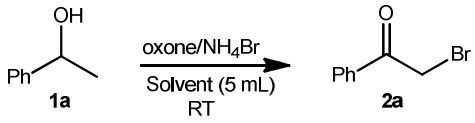
Inspired by the recent progress in the oxidation of alcohols to carbonyl compounds using oxone in presence of catalysts[14-17] or halide ions[18-20] and our previously developed method for oxidative bromination of ketones[7c] (Scheme 2), we envisioned that the oxidation of secondary alcohols and oxidative bromination of *in situ* generated ketones can occur in sequential manner in one-pot. To test our hypothesis, we initiated optimization studies by choosing 1-phenylethanol (**1a**) as model substrate and $NH_4Br$ as bromine source. To our delight, the reaction of **1a** with 2 equivalents of $NH_4Br$ and 2 equivalents of oxone in water yielded the 51 % of desired 2-bromo-1-phenylethanone (**2a**) at room temperature (Table 1, entry 1). The same reaction at 50 $^oC$ provided the 63 % of **2a** within 5 h of reaction time (Table 1, entry 2). Generally, the use of surfactants in aqueous reactions increase the solubility of organic substrates thereby enhances the selectivity and reactivity of a reaction.[13e] Therefore, in order to improve the yield of tandem oxidation/oxidative bromination reaction of **1a**, we employed anionic and cationic (SDS and CTAB, respectively) surfactants in reaction media. But, we did not succeed in improving the reaction yield (Table 1, entries 3-4). Next, we tested the reaction in mixture of solvents consisting of water and organic solvent (Table 1, entries 5-7). Gratifyingly, the co-solvent system containing water/methanol (1:4 ratio) gave the better yield of the reaction (Table 1, entry 7). Finally, we investigated the effect of mole ratios of $NH_4Br$ and oxone on the reaction yield (Table 1, entries 8-9). The findings of this investigation revealed that 2 equivalents of $NH_4Br$ and 3 equivalents of oxone with respect to **1a** were essential to obtain the highest yield of **2a** at room temperature (Table 1, entry 8).

After optimizing the reaction conditions, we then turned our attention to expand the scope of this tandem oxidation/bromination process. In this context, initially, we investigated the various 1-aryl-1-alkanols containing halo, electron withdrawing and electronic donating groups on aromatic ring (Table 2). The position of the substituent's on aromatic ring influenced the reaction yields. The reaction of halo substituent containing 1-aryl-1-alkanols **1b**-**1f** underwent smoothly under optimized conditions to provide the corresponding products **2b**-**2f** in good to excellent yields (Table 2, entries 2-6).




**Scheme 2.** Oxone mediated oxidation of alcohols, and our previous approach for oxidative bromination of ketones.

**Table 1.** Optimization of reaction conditions.[a,b]




| Entry | Solvent | Time (h) | Yield (%) **2a** |
|---|---|---|---|
| 1 | $H_2O$ | 24 | 51 |
| 2[c] | $H_2O$ | 5 | 63 |
| 3[d] | $H_2O$ | 15 | 46 |
| 4[e] | $H_2O$ | 15 | 48 |
| 5 | $H_2O$/DCM (1:4) | 24 | 05 |
| 6 | $H_2O/CH_3CN$ (1:4) | 15 | 18 |
| 7 | $H_2O$/MeOH (1:4) | 15 | 80 |
| **8**[f] | **$H_2O$/MeOH (1:4)** | **15** | **86** |
| 9[g] | $H_2O$/MeOH (1:4) | 15 | 70 |

[a] Reaction Conditions: Unless otherwise mentioned, Substrate **1a** (1 mmol), $NH_4Br$ (2 equiv.), Oxone (2 equiv.), Solvent (5 mL), RT. [b] Isolated yields. [c] Reaction performed at 50 $^oC$. [d] 10 mol% of Sodium dodecyl sulfate (SDS) was used. [e] 10 mol% of Cetyltrimethylammonium bromide (CTAB) was used. [f] 3 equiv. of oxone was used. [g] 1.5 equiv. of $NH_4Br$ was used.

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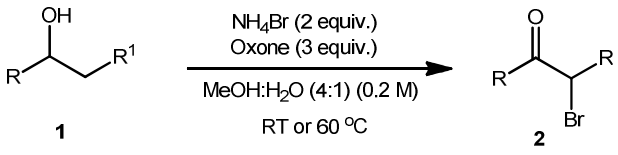
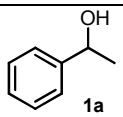
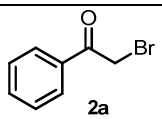
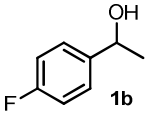
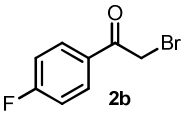
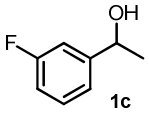
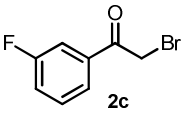
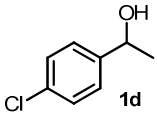
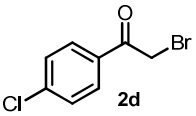
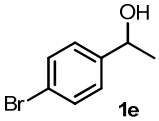
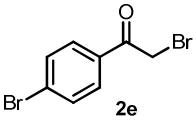
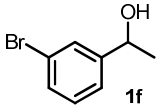
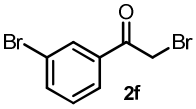
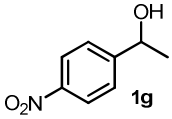
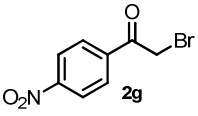
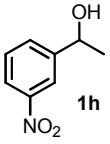
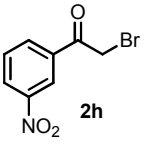
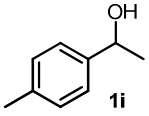
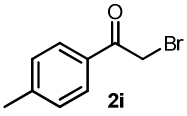
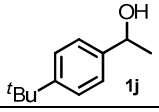
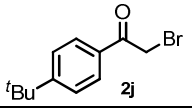
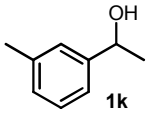
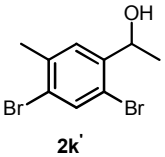
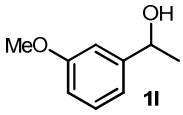
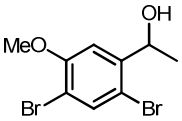
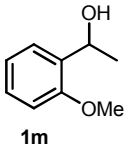
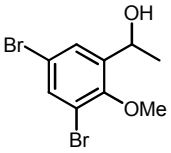
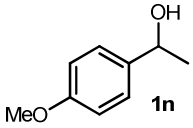
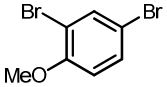
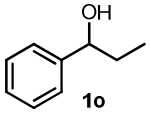
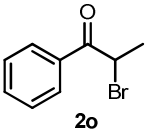
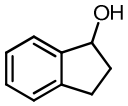
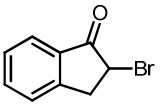
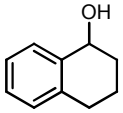
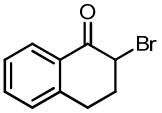
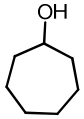
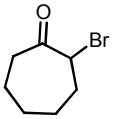
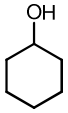
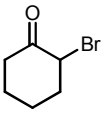
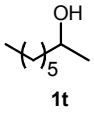
**Table 2**. Substrate scope.[a,b]




| Entry | Substrate (**1**) | Time (h) | Product (**2**) | Yield (%) |
|---|---|---|---|---|
| 1 |  **1a** | 15 |  **2a** | 86 |
| 2 |  **1b** | 24 |  **2b** | 89 |
| 3 |  **1c** | 24 |  **2c** | 77 |
| 4 |  **1d** | 24 |  **2d** | 81 |
| 5 |  **1e** | 24 |  **2e** | 94 |
| 6 |  **1f** | 24 |  **2f** | 74 |
| 7 |  **1g** | 24 |  **2g** | 38 (45)[c] |
| 8 |  **1h** | 24 |  **2h** | 62 |
| 9 |  **1i** | 24 |  **2i** | 45 |
| 10 |  **1j** | 24 |  **2j** | 59 |
| 11 |  **1k** | 24 |  **2k'** | 88 |
| 12 |  **1l** | 24 |  **2l'** | 29 (82)[c] |
| 13 |  **1m** | 24 |  **2m'** | 25 |
| 14 |  **1n** | 20 |  **2n''** | 93 |
| 15 |  **1o** | 48 |  **2o** | 31 (42)[c] |
| 16 |  **1p** | 24 |  **2p** | 52 |
| 17 |  **1q** | 24 |  **2q** | 88 |
| 18 |  **1r** | 24 |  **2r** | 48 |
| 19 |  **1s** | 24 |  **2s** | 04 |
| 20 |  **1t** | 24 |  **2t** | 20 |
| 21 |  **1u** | 24 |  **2u** | 25 |

[a] Reaction Conditions: Unless otherwise mentioned, Substrate **1** (1 mmol), $NH_4Br$ (2 equiv.), Oxone (3 equiv.), Solvent (5 mL), RT. [b] Isolated yields. [c] Reaction performed at 60 °C for 24 h.

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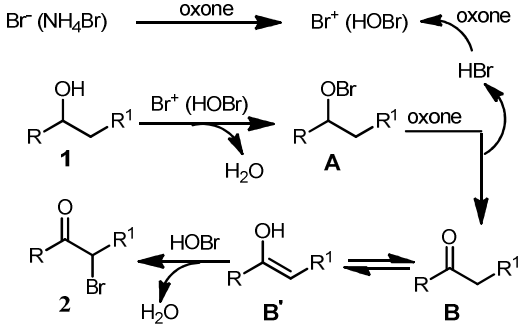
The aromatic secondary alcohol **1g** containing strong electron withdrawing group i.e., the -$NO_2$ group at *para* position led to the formation of **2g** in 38 % yield at room temperature and 45 % yield at 60 $^{o}$C (Table 2, entry 7). Whereas, the -$NO_2$ group at *meta* position of phenyl ring provided the corresponding product **2h** in 62 % yield at room temperature (Table 2, entry 8). The benzylic secondary alcohols with methyl (**1i**) and *tert*-butyl (**1j**) groups at *para* position of aromatic ring generated the corresponding products **2i** and **2j** in 45 and 59 % yields, respectively (Table 2, entries 9-10). Interestingly, 1-(3-methylphenyl)ethanol (**1k**) led to the formation of ring brominated product 1-(2,4-dibromo-5-methylphenyl)ethanol (**2k'**) instead of tandem oxidation/$\alpha$-bromination product **2k** in 88 % yield at room temperature (Table 2, entry 11). In a similar manner, the strong electron donating group i.e., -OMe group at *ortho* or *meta* position on aromatic ring of 1-aryl-1-alkanols **1l**-**1m** directed the ring bromination to give the corresponding products **2l'** and **2m'** (Table 2, entries 12-13). It is noteworthy that, the 1-(4-methoxyphenyl)ethanol (**1n**) under standard reaction conditions yielded the deacylated ring bromination product 2,4-dibromoanisole (**2n"**) in 93 % yield (Table 2, entry 14). The substrate **1o** under optimized conditions afforded the product **2o** in 31 % yield at room temperature and 42 % yield at 60 $^{o}$C (Table 2, entry 15).

The aromatic ring fused and simple cyclic secondary alcohols also examined under optimal conditions. The cyclic secondary alcohols **1p** and **1q** furnished the desired tandem oxidation/oxidative bromination products **2p** and **2q** in 52 and 88 % yields, respectively (Table 2, entries 16-17). The aliphatic cyclic alcohols **1r** and **1s** gave the desired products **2r** and **2s** in low to moderate yields (Table 2, entries 18-19). In addition, acyclic secondary alcohols **1t** and **1u** subjected to the standard conditions to obtain the corresponding products **2t** and **2u** in 20 and 25 % yields, respectively (Table 2, entries 20-21).




**Scheme 3.** Plausible reaction mechanism for the tandem oxidation/oxidative bromination of secondary alcohols to $\alpha$-bromoketones.

A plausible reaction mechanism for the tandem oxidation/oxidative bromination of secondary alcohols to $\alpha$-bromoketones has been depicted in scheme 3. Initially, it is assumed that oxone may oxidize bromide ion to hypobromous acid.[21] This reactive electrophillic bromine species may oxidize secondary alcohol **1** to ketone **B** in presence of oxone *via* intermediate **A**.[18] The ketone **B** may undergo keto-enol tautomerism in aqueous methanolic solution. The enol form **B'** may rapidly reacts with $Br^{+}$ to generate the desired $\alpha$-bromoketone **2**.

In conclusion, we have developed a new, efficient and green approach for the one pot synthesis of $\alpha$-bromoketones from secondary alcohols. The scope and limitations of this protocol were investigated with various secondary alcohols such as 1-aryl-1-alkanols (containing halo, electron withdrawing and electronic donating groups on aromatic ring), aromatic ring fused and aliphatic (cyclic and acylic) alcohols. This reaction proceeds *via* two consecutive steps such as oxidation of secondary alcohol to ketone and oxidative bromination of *in situ* generated ketone to $\alpha$-bromoketone. Moreover, this method precludes the need of metal catalysts, alkali metal salts, moisture sensitive reagents, drastic reaction conditions and costly bromine surrogates. Simple reaction conditions, use of air stable and commercially available reagents, and easy work up procedures make this approach valuable in a preparative point of view.

## Experimental section

### General information

All chemicals used were reagent grade and used as received without further purification. $^{1}$H NMR spectra were recorded at 300 or 500 MHz and $^{13}$C NMR spectra at 75 or 125 MHz in $CDCl_3$. The chemical shifts ($\delta$) are reported in ppm units relative to TMS as an internal standard for $^{1}$H NMR and $CDCl_3$ for $^{13}$C NMR spectra. Coupling constants ($J$) are reported in hertz (Hz) and multiplicities are indicated as follows: s (singlet), br s (broad singlet), d (doublet), dd (doublet of doublet), m (multiplet). TLC inspections were performed on Silica gel 60 $F_{254}$ plates. Column chromatography was performed on silica gel (100-200 mesh) using *n*-hexane-EtOAc as eluent.

### General procedure for the one-pot synthesis of $\alpha$-bromoketones from secondary alcohols

In an oven dried double necked round bottom flask equipped with a magnetic stirring bar, secondary alcohol **1** (1 mmol, 1 equiv.) and water/methanol (1:4; 5 mL) were taken at room temperature and stirred for some time. To this solution, $NH_4Br$ (2 mmol, 2 equiv.) was added. Then, oxone (3 mmol, 3 equiv.) was added slowly and the resulting solution was allowed to stirr at room temperature for the time indicated in the tables. After completion of the reaction, as indicated by the TLC, the reaction was quenched with aqueous $Na_2S_2O_3$ and stirred vigorously until the orange colour was disappeared. The reaction mixture was extracted with DCM (15 x 3 mL) and the organic phase was washed with water (2 x 5 mL) and dried over anhydrous $Na_2SO_4$. After evaporation of the solvent under reduced pressure, the crude reaction mixture was purified by column chromatography (silica gel, hexane/ethyl acetate mixture) to give the corresponding $\alpha$-bromoketone **2**.

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**Keywords:** alcohols • bromination • cascade reaction • oxidation • synthetic methods

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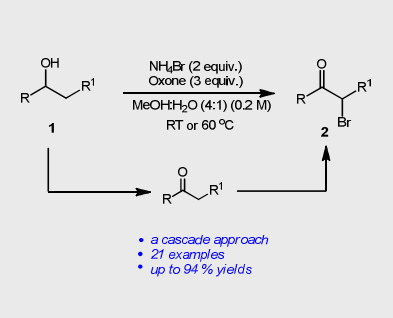
COMMUNICATION

A new, efficient and green protocol for the one-pot synthesis of *α*-bromoketones from readily available secondary alcohols using cheap, air stable and non-toxic reagents is reported. This reaction proceeds *via* two consecutive steps such as oxidation of secondary alcohol to ketone and oxidative bromination of *in situ* generated ketone to *α*-bromoketone.

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**A new and Versatile One-Pot Strategy to Synthesize alpha-Bromoketones from Secondary Alcohols Using Ammonium Bromide and Oxone**