



## Oxidative iodination of carbonyl compounds using ammonium iodide and oxone<sup>®</sup>

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### ABSTRACT

A simple, efficient, mild, and regioselective method for oxyiodination of carbonyl compounds has been reported by using  $\text{NH}_4\text{I}$  as the source of iodine and Oxone<sup>®</sup> as an oxidant. Various carbonyl compounds such as aralkyl ketones, aliphatic ketones (acyclic and cyclic), and  $\beta$ -keto esters proceeded to the respective  $\alpha$ -monoiodinated products in moderate to excellent yields. Unsymmetrical aliphatic ketones reacted smoothly yielding a mixture of 1-iodo and 3-iodo ketones with the predominant formation of 1-iodoproduct.

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The chemistry dealing with selective introduction of an iodine atom into organic molecules has attracted considerable interest in the wider scientific community,<sup>1</sup> due to the importance of iodo-substituted organic compounds as valuable synthones or precursors in organic synthesis and in carbon–carbon, carbon–oxygen, and carbon–nitrogen bond formation.<sup>2</sup> Moreover, iodinated compounds are widely used in medical diagnostics as contrast agents or radioactively labeled markers.<sup>3</sup>  $\alpha$ -Iodo ketones are among the most versatile intermediates in organic synthesis and their high reactivity makes them available to react with a large number of nucleophiles to provide a variety of useful compounds.<sup>4</sup> Therefore,  $\alpha$ -iodo ketones and their application as reactive intermediates have attracted considerable attention in various organic transformations.  $\alpha$ -Iodo ketones are usually prepared indirectly by oxidative-iodination of olefins<sup>5</sup>, electrophilic iodination of ketone derivatives (enol ethers, and acetates)<sup>6</sup> or by halogen interchange of bromo compounds with sodium iodide.<sup>7</sup> Due to the difficulties in the synthesis and purification of enol silyl ethers and acetals, direct conversion of carbonyl compounds into  $\alpha$ -iodo compounds has received considerable attention.

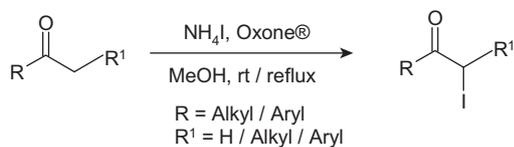
Generally, the direct conversion of carbonyl compounds into  $\alpha$ -iodo compounds is commonly achieved using iodine–cerium (IV) ammonium nitrate,<sup>8</sup> iodine–mercury (II) chloride<sup>9</sup>, and iodine–selenium dioxide.<sup>10</sup> In recent years a wide variety of methods have been developed for the direct  $\alpha$ -iodination of carbonyl compounds using different iodonium donating systems, such as,  $\text{I}_2$ -*m*-iodosylbenzoic acid<sup>11</sup>,  $\text{I}_2$ - $\text{NaNO}_2$ - $\text{O}_2$ -silica supported  $\text{H}_2\text{SO}_4$ <sup>12</sup>,  $\text{KI}$ - $\text{NaNO}_2$ -

$\text{O}_2$ - $\text{H}_2\text{SO}_4$ <sup>13</sup>,  $\text{NIS}$ - $\text{PTSA}$ <sup>14</sup>,  $\text{I}_2$ - $\text{TMOF}$ <sup>15</sup>,  $\text{NIS}$ -ionic liquids<sup>16</sup>,  $\text{I}_2$ - $\text{CuO}$ <sup>17</sup>,  $\text{I}_2$ -urea- $\text{H}_2\text{O}_2$ <sup>18</sup>,  $\text{I}_2$ - $\text{DME}$ <sup>19</sup>,  $\text{HIO}_4$ - $\text{Al}_2\text{O}_3$ <sup>20</sup>,  $\text{HTIB}$ - $\text{MgI}_2$ - $\text{MW}$ <sup>21</sup>,  $\text{I}_2$ -selectfluor<sup>TM</sup>- $\text{TEDA}$ - $\text{BF}_4$ <sup>22</sup>, and  $\text{NIS}$ -Lewis acid.<sup>23</sup> However, most of these methods have drawbacks, such as use of expensive, hazardous or toxic reagents, tedious work-up procedures, and high reaction temperatures for long reaction times. Therefore, the development of an efficient, environmentally friendly, atom economic (100% with respect to iodine), and selective procedure for the  $\alpha$ -monoiodination of ketones is still desirable. Most methods for transforming carbonyl compounds to their related  $\alpha$ -iodo derivatives rely on the modification of molecular iodine or *N*-iodo-succinimide while the use of other reagents has been less investigated.

Oxone<sup>®</sup>, a potassium triple salt containing potassium peroxy monosulfate, is an effective oxidant. Due to its stability, water-solubility, ease of transport, nontoxic 'green' nature, nonpolluting byproducts, and cost-effectiveness, this solid reagent has become an increasingly popular reagent for oxidative transformations.<sup>24–28</sup>

In our on-going efforts to achieve environmentally friendlier halogenation procedures, earlier we have reported a method for the ring iodination of aromatic compounds using  $\text{NH}_4\text{I}$ -Oxone<sup>®</sup> system.<sup>29</sup> Now, we wish to explore this reagent system for the  $\alpha$ -iodination of carbonyl compounds. Herewith, we report a milder, efficient, and environmentally benign procedure for the  $\alpha$ -iodination of carbonyl compounds using  $\text{NH}_4\text{I}$  and Oxone<sup>®</sup> at room temperature/reflux temperature without the use of catalyst and as a possible alternative to overcome the disadvantages described in the earlier methods (Scheme 1). However, to the best of our knowledge, method for  $\alpha$ -iodination of carbonyl compounds using  $\text{NH}_4\text{I}$ /Oxone<sup>®</sup> system has been unprecedented to date.

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**Scheme 1.**  $\alpha$ -Iodination of ketones.

We first investigated the effect of different solvents on the iodination of acetophenone using  $\text{NH}_4\text{I}$  as iodine source and Oxone<sup>®</sup> as an oxidant. The best results were obtained when methanol was used as a solvent compared to others (DCM,  $\text{CHCl}_3$ ,  $\text{CCl}_4$ , EtOH, *i*-PrOH, *t*-BuOH, hexane, and THF).

To assess the generality of this method, a variety of ketones (aralkyl, alkyl, cyclic ketones, and  $\beta$ -keto esters) were investigated

**Table 1**  
Iodination of aralkyl ketones using  $\text{NH}_4\text{I}$  and Oxone<sup>®a</sup>

Entry	Substrate	Time (h)	Product	Yield <sup>b</sup> (%)
1		24 <sup>c</sup> 2.3 <sup>d</sup>		86 (4) <sup>e</sup> 92 (6) <sup>e</sup>
2		48 <sup>c</sup> 3 <sup>d</sup>		79 (8) <sup>e</sup> 92 (5) <sup>e</sup>
3		46 <sup>c</sup> 2.45 <sup>d</sup>		84 (7) <sup>e</sup> 86 (4) <sup>e</sup>
4		48 <sup>c</sup> 3.3 <sup>d</sup>		81 (9) <sup>e</sup> 92 (5) <sup>e</sup>
5		48 <sup>c</sup> 4 <sup>d</sup>		79 (6) <sup>e</sup> 86 (9) <sup>e</sup>
6		24 <sup>c</sup> 2.3 <sup>d</sup>		52 (26) <sup>f</sup> 47 (35) <sup>f</sup>
7		47 <sup>c</sup> 3.3 <sup>d</sup>		- 72 (15) <sup>g</sup>
8		22 <sup>c</sup> 2 <sup>d</sup>		71 (6) <sup>e</sup> (14) <sup>h</sup> 83 (4) <sup>e</sup> (8) <sup>h</sup>
9		49 <sup>c</sup> 8.3 <sup>d</sup>		46 79 (4) <sup>e</sup>
10		48 <sup>c</sup> 8 <sup>d</sup>		81 (2) <sup>e</sup> 93 (4) <sup>e</sup>
11		48 <sup>c</sup> 5.3 <sup>d</sup>		70 (4) <sup>e</sup> 86 (4) <sup>e</sup>
12		48 <sup>c</sup> 6.3 <sup>d</sup>		55 80 (2) <sup>e</sup>

(continued on next page)

Table 1 (continued)

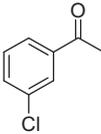
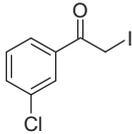
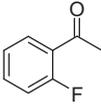
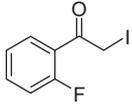
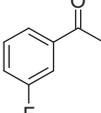
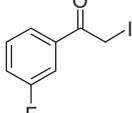
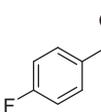
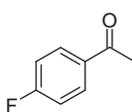
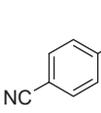
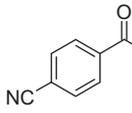
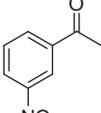
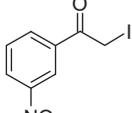
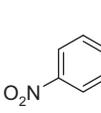
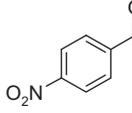
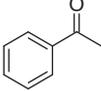
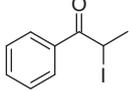
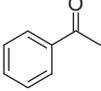
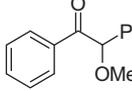
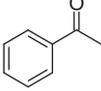
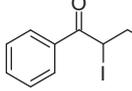
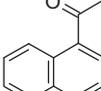
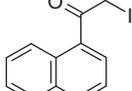
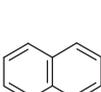
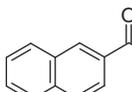
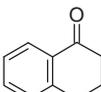
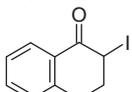
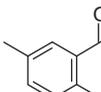
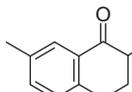
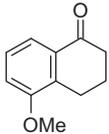
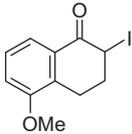
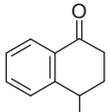
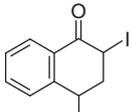
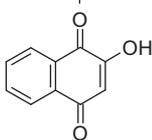
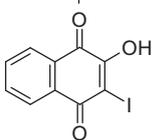
Entry	Substrate	Time (h)	Product	Yield <sup>b</sup> (%)
13		48 <sup>c</sup>		68
		6 <sup>d</sup>		92 (2) <sup>e</sup>
14		48 <sup>c</sup>		70 (3) <sup>e</sup>
		8 <sup>d</sup>		92 (5) <sup>e</sup>
15		48 <sup>c</sup>		68 (5) <sup>e</sup>
		7 <sup>d</sup>		97 (2) <sup>e</sup>
16		48 <sup>c</sup>		74 (4) <sup>e</sup>
		4 <sup>d</sup>		92 (5) <sup>e</sup>
17		48 <sup>c</sup>		52
		9.3 <sup>d</sup>		92 (3) <sup>e</sup>
18		48 <sup>c</sup>		26
		10 <sup>d</sup>		81
19		48 <sup>c</sup>		35
		10 <sup>d</sup>		72
20		48 <sup>c</sup>		35
		10 <sup>d</sup>		90 (8) <sup>e</sup>
21		48 <sup>c</sup>		19
		10 <sup>d</sup>		94
22		48 <sup>c</sup>		-
		11 <sup>d</sup>		74
23		48 <sup>c</sup>		74 (4) <sup>e</sup>
		9 <sup>d</sup>		91 (3) <sup>e</sup>
24		48 <sup>c</sup>		84 (2) <sup>e</sup>
		4 <sup>d</sup>		91 (5) <sup>e</sup>
25		11 <sup>c</sup>		80 (4) <sup>e</sup>
		3.3 <sup>d</sup>		86 (5) <sup>e</sup>
26		48 <sup>c</sup>		84 (3) <sup>e</sup>
		2.3 <sup>d</sup>		84 (4) <sup>e</sup>

Table 1 (continued)

Entry	Substrate	Time (h)	Product	Yield <sup>b</sup> (%)
27		48 <sup>c</sup> 2 <sup>d</sup>		65 (3) <sup>e</sup> 89 (2) <sup>e</sup>
28		22 <sup>c</sup> 2 <sup>d</sup>		93 (4) <sup>e</sup> 92 (3) <sup>e</sup>
29		3.3 <sup>c</sup> 1 <sup>d</sup>		81 92

<sup>a</sup> Reagents conditions: substrate (2 mmol), NH<sub>4</sub>I (2.2 mmol), Oxone<sup>®</sup> (2.2 mmol), MeOH (10 ml).

<sup>b</sup> Products were characterized by <sup>1</sup>H NMR, Mass spectra, and quantified by GC.

<sup>c</sup> Room temperature.

<sup>d</sup> Reflux temperature.

<sup>e</sup>  $\alpha$ -Methoxy product.

<sup>f</sup> 1-(2-Hydroxy-5-iodophenyl)ethanone.

<sup>g</sup> 1-(3-Hydroxy-4-iodophenyl)-2-iodoethanone.

<sup>h</sup> 2-Iodo-1-(3-iodo-4-methoxyphenyl)ethanone.

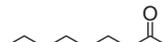
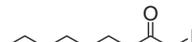
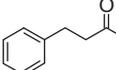
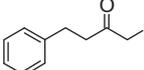
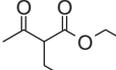
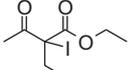
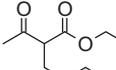
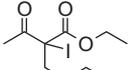
for the reaction with NH<sub>4</sub>I-Oxone<sup>®</sup> reagent system and the results are summarized in Tables 1 and 2. All the substrates treated with 1.1 equiv of NH<sub>4</sub>I and 1.1 equiv of Oxone<sup>®</sup> in methanol at room temperature (or reflux temperature) afforded the corresponding iodinated compounds in good to excellent yields.<sup>30</sup> High yields were obtained in short reaction time at a reflux temperature.

The iodination of a variety of aralkyl ketones proceeded efficiently, to afford the corresponding  $\alpha$ -iodo ketones in moderate to excellent yields (Table 1). The reactivity profile of the substituted acetophenones is noteworthy. Acetophenone produced the corresponding  $\alpha$ -iodo product in high yields (Table 1, entry 1). Moderately activating (Table 1, entries 2–5) and deactivating (Table 1, entries 9–16) groups present on the aromatic ring of acetophenone gave moderate to high yields of the corresponding  $\alpha$ -iodo products, along with small amount of  $\alpha$ -methoxy substituted product. The position of substitution on the phenyl ring of acetophenone slightly affects the reaction yield (Table 1, entries 9–13). A moderate yield was obtained when 2-bromo acetophenone was used (Table 1, entry 9), while high yield of desired product was obtained when 3-bromoacetophenone was used (Table 1, entry 10). Introduction of highly deactivating groups (NO<sub>2</sub>, CN) on the aromatic ring of acetophenone furnished less yields of the desired product under the similar reaction conditions (Table 1, entries 17–19).

Highly activating groups (OCH<sub>3</sub>, OH) present on the aromatic ring of acetophenone gave very interesting results (Table 1, entries 6–8). Aralkyl ketones having an activated aromatic ring possess at least two reactive sites convenient for electrophilic functionality, the aromatic ring and the atom alpha to the carbonyl functionality. A distinct difference of regioselectivity was observed with 2- and 3-hydroxyacetophenones. In case of 2-hydroxyacetophenone the ring was regioselectively iodinated and yielded 1-(2-hydroxy-3-iodophenyl)ethanone and 1-(2-hydroxy-5-iodophenyl)ethanone in 52%, 26%, and 47%, 35% yields, respectively at room temperature and reflux temperature. Whereas 3-hydroxyacetophenone was converted to the respective  $\alpha$ -iodinated product and diiodo product (1-(3-hydroxy-4-iodophenyl)-2-iodoethanone) in 72%, 15% yield, respectively. When 4-methoxyacetophenone subjected to iodination under similar conditions provided the corresponding

Table 2

Iodination of aliphatic ketones and  $\beta$ -keto esters using NH<sub>4</sub>I and Oxone<sup>®</sup><sup>a</sup>

Entry	Substrate	Time (h)	Product	Yield <sup>b</sup> (%)
1		48		52
2		23		69
3		11		61 (28) <sup>c</sup>
4		13		76 (15) <sup>c</sup>
5		24		55 (17) <sup>c</sup>
6		28		64 (16) <sup>d</sup>
7		2		28 (55) <sup>d</sup>

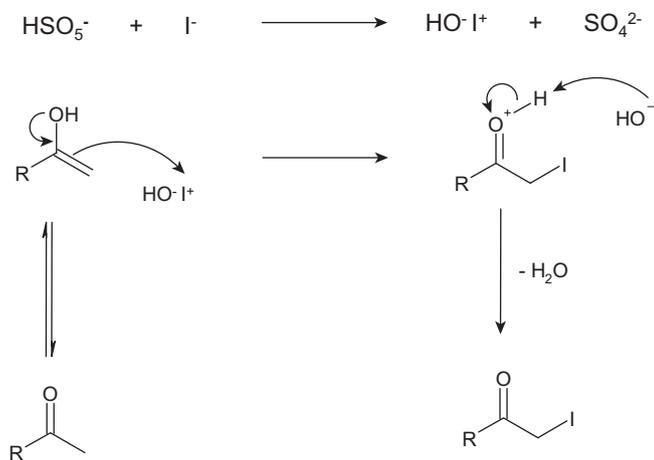
<sup>a</sup> Reagents conditions: substrate (2 mmol), NH<sub>4</sub>I (2.2 mmol), Oxone<sup>®</sup> (2.2 mmol), MeOH (10 ml), room temperature.

<sup>b</sup> Products were characterized by <sup>1</sup>H NMR, mass spectra, and quantified by GC.

<sup>c</sup> 3-Iodo ketones.

<sup>d</sup>  $\alpha$ -Hydroxy- $\beta$ -keto esters.

$\alpha$ -iodinated product in 71% yield, accompanied by a significant amount of respective diiodo product (2-iodo-1-(3-iodo-4-methoxyphenyl)ethanone) (14% yield) and small amount of  $\alpha$ -methoxy substituted product. Propiophenone yielded the corresponding  $\alpha$ -iodinated product in moderate yield at room temperature and high yield at reflux temperature (Table 1, entry 20). Interestingly, 1,2-diphenylethanone forms the solvolytic product (1,2-diphenyl-2-



Scheme 2. Plausible reaction mechanism.

methoxyethanone) instead of the expected  $\alpha$ -iodinated product (1,2-diphenyl-2-iodoethanone) in low yield at room temperature and excellent yield at reflux temperature (Table 1, entry 21). Furthermore, 1- and 2-acetonaphthones also led to the respective  $\alpha$ -iodinated products in excellent yields (Table 1, entries 23 and 24).

The nonmethyl ketone, 1-tetralone was also effectively  $\alpha$ -iodinated using this reagent system and afforded the excellent yield of desired product (Table 1, entry 25). In order to generalize these results, further we selected a wide range of substituted 1-tetralone derivatives, bearing on aromatic ring activated by methyl, methoxy or cycloalkyl part of the molecule substituted by a methyl group (Table 1, entries 26–28). Methyl and methoxy groups present on the aromatic ring direct the ring iodination. But in this case alpha iodination achieved in high yields without forming ring iodination products. In this series, 5,7-dimethyl-3,4-dihydro-1(2H)-naphthalenone and 5-methoxy-3,4-dihydro-1(2H)-naphthalenone were iodinated to 2-iodo substituted products in high yields. 2-Hydroxy-1,4-naphthoquinone showed good reactivity with this reagent system and produced the 2-hydroxy-3-iodo-1,4-naphthoquinone, which is used in the synthesis of naphtho[2,3-*b*]furan-4,9-dione natural product<sup>31</sup> (Table 1, entry 29).

The investigation was additionally extended to aliphatic ketones (alkyl and cyclic ketones) and  $\beta$ -keto esters and the results are presented in Table 2. When cyclic ketones such as cyclohexanone and cycloheptanone are treated in the same reaction conditions as above corresponding  $\alpha$ -iodo ketones are obtained in moderate yields (Table 2, entries 1 and 2). In the case of unsymmetrical aliphatic ketones  $\alpha$ -iodination proceeded smoothly at room temperature leading to a mixture of two monoiodinated regiomers, but a slight preference for the substitution at the primary carbon atom was observed with the 1-iodo isomer as the major product (Table 2, entries 3–5). For example, the reaction of 2-nonanone with this reagent system at room temperature for 11 h proceeded to 1-iodo-2-nonanone and 3-iodo-2-nonanone in 61% and 28% yield, respectively.  $\alpha$ -Unsubstituted- $\beta$ -keto esters provided highly unstable products in this reaction conditions and resulted in a complex mixture of unidentified products. Whereas  $\alpha$ -monosubstituted- $\beta$ -keto esters (Table 2, entries 6 and 7) furnished a mixture of corresponding  $\alpha$ -iodo and  $\alpha$ -hydroxy products, respectively.

In a blank experiment, no reaction occurred between acetophenone and  $\text{NH}_4\text{I}$  without Oxone<sup>®</sup> (oxidant) under similar reaction conditions. Thus the role played by the Oxone<sup>®</sup> is justified. Concerning the reaction mechanism, it is assumed that Oxone<sup>®</sup> efficiently oxidizes the  $\text{I}^\ominus(\text{NH}_4\text{I})$  to  $\text{I}^\ominus(\text{HOI})$ , which further reacts with enol form of carbonyl compound to afford the corresponding  $\alpha$ -iodinated product (Scheme 2).

In conclusion, we have developed an efficient new protocol for the selective  $\alpha$ -monoiodination of the carbonyl compounds using  $\text{NH}_4\text{I}$  and Oxone<sup>®</sup> without metal or mineral acid catalyst. Various ketones and  $\beta$ -keto esters can be iodinated by this reagent system under mild conditions affording the corresponding  $\alpha$ -iodo products in moderate to high yields. The present method, which is more attractive than the existing methods, offers several advantages, such as commercial availability of the reagents, simple reaction conditions, no evolution of hydrogen iodide, high yields, economical process with easier setup/work-up procedures, and environmentally friendly nature making our method more valuable from preparative point of view.

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## Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.tetlet.2011.09.106.

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30. General procedure for the iodination of carbonyl compounds: Oxone® (2.2 mmol) was slowly added to a well stirred solution of NH<sub>4</sub>I (2.2 mmol) and carbonyl compound (2 mmol) in methanol and the reaction mixture was allowed to stir at room or reflux temperature. After disappearance of the reactant (reaction was monitored by TLC) or after the appropriate time, the reaction mixture was filtered and the solvent was removed under reduced pressure. The residue was dissolved in ethyl acetate, insoluble material filtered off, the solution washed with aqueous sodium thiosulfate pentahydrate (5%) and water. The organic layer was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated in vacuo. The crude reaction mixture was further purified by column chromatography using silica gel (230–400 mesh) gave the target products. All the products were identified by their <sup>1</sup>H NMR, <sup>13</sup>C NMR and mass spectra.
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