2-Iodoxybenzoic Acid (IBX): A Versatile Reagent

Compiled by Indresh Kumar

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Introduction

The importance of hypervalent iodine reagents in organic chemistry has been demonstrated in recent years, and they have been found to have several desirable properties: they are mild, selective, efficient and eco-friendly. 2-Iodoxybenzoic acid (IBX) has been developed as a powerful reagent for several organic transformations, and a recent surge in interest was driven by the publication of an improved method for its synthesis. IBX is a powerful single-electron transfer oxidant that readily accepts a new heteroatom-based ligand, and has been applied successfully for the construction of novel heterocycles.

Preparation

According to a new improved procedure, IBX can be prepared in very good yield by the oxidation of 2-iodobenzoic acid with Oxone; this shows advantages over the previously reported methods.

Abstracts

(A) IBX oxidizes 1° and 2° alcohols to the corresponding aldehydes and ketones, without any over-oxidation, in DMSO at room temperature. Using different solvent systems and higher temperatures, yields of 90–100% can be obtained. Environmentally benign ionic liquids have also been used as solvents for this transformation.

(B) IBX has been used to oxidize oximes and tosyl hydrazones to the corresponding carbonyl compounds.
(C) IBX was used to oxidize saturated alcohols and carbonyl compounds\(^7\) to the corresponding \(\alpha,\beta\)-unsaturated carbonyl system in one pot. It can also be used to oxidize the benzylic position.\(^8\)

\[
\begin{align*}
\text{OH} & \xrightarrow{(\text{IBX, 3.5 equiv})} \text{O} \quad (\text{IBX, 1.5 equiv}) \\
65 \, ^\circ \text{C}, 4-24 \, \text{h} & \quad 40-80\% \\
\text{R} & \xrightarrow{\text{DMSO, 55-95} \, ^\circ \text{C}} \text{O} \quad (\text{IBX, 1.5 equiv}) \\
& \quad 65 \, ^\circ \text{C}, 4-24 \, \text{h} \\
\text{R} & \xrightarrow{\text{DMSO, 55-95} \, ^\circ \text{C}} \text{O} \quad 60-85\% \\
& \quad 22 \, \text{examples}
\end{align*}
\]

(D) IBX reacts with certain unsaturated N-aryl amides (anilides) to form novel heterocycles such as \(\delta\)-lactams, cyclic urethanes, hydroxylamine and aminosugar building blocks.\(^8\)

\[
\begin{align*}
\text{Ar} & \xrightarrow{\text{IBX, THF-DMSO}} \text{X} = \text{O, CH}, \text{NR} \\
(10^1), 90 \, ^\circ \text{C}, 24 \, \text{h} & \quad 70-95\%, 29 \, \text{examples}
\end{align*}
\]

(E) In combination with an N-oxide (MPO), IBX was used to oxidize a carbonyl\(^9\) and its silyl enol ether\(^10\) to the corresponding \(\alpha,\beta\)-unsaturated compounds in high yield at ambient temperature.

\[
\begin{align*}
\text{O} & \xrightarrow{\text{IBX-MPO (2-4 equiv)}} \text{O} \\
\text{DMSO, r.t., 15-48 h} & \quad 70-96\% \\
\text{17 examples} & \quad \text{IBX-MPO (1.5-4 equiv)} \\
\text{DMSO, r.t., 40 min} & \quad 80-97\% \\
\text{13 examples}
\end{align*}
\]

(F) A regioselective oxidation of phenols to \(\omega\)-quinones was performed with IBX.\(^11\)

\[
\begin{align*}
\text{Y} & \xrightarrow{\text{(IBX, 1 equiv)}} \text{O} \\
\text{r.t.} & \quad 85-92\% \\
\text{11 examples}
\end{align*}
\]

(G) Recently, IBX was used to convert nitrogen- and sulfur-containing substrates to synthetically useful intermediates.\(^11\)

\[
\begin{align*}
\text{R} & \xrightarrow{\text{(IBX, 1.1 equiv)}} \text{R} \\
\text{DMSO, 45} \, ^\circ \text{C} & \quad 80-98\% \\
\text{29 examples} & \quad \text{(IBX, 2 equiv)} \\
\text{H}_2\text{O-DMSO (1:9), 25} \, ^\circ \text{C} & \quad 96-99\%
\end{align*}
\]

References