This feature focuses on a reagent chosen by a postgraduate, highlighting the uses and preparation of the reagent in current research.

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.\(^1\) 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.\(^2a\) 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;\(^2a\) this shows advantages over the previously reported methods.\(^2b\)

Abstracts

(A) IBX oxidizes 1° and 2° alcohols to the corresponding aldehydes and ketones, without any over-oxidation, in DMSO at room temperature.\(^3\) Using different solvent systems and higher temperatures, yields of 90–100% can be obtained.\(^4\) Environmentally benign ionic liquids have also been used as solvents for this transformation.\(^5\)

(B) IBX has been used to oxidize oximes and tosyl hydrazones to the corresponding carbonyl compounds.\(^6\)

SYNLETT 2005, No. 9, pp 1488–1489
Advanced online publication: 27.04.2005
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(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.\(^8\)

\[
\begin{align*}
\text{OH} & \quad \text{IBX, 3.5 equiv} \quad 65^\circ \text{C, 4–24 h} \quad \text{40–80%} \quad 29 \text{ examples} \\
\text{O} & \quad \text{IBX, 1.5 equiv} \quad 65^\circ \text{C, 4–24 h} \\
\end{align*}
\]

(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.\(^8\)

\[
\begin{align*}
\text{H} & \quad \text{IBX, THF–DMSO (10:1), 90 °C, 24 h} \\
\text{X = O, CH}_2, \text{NR} & \quad 70–95%, 29 \text{ examples} \\
\end{align*}
\]

(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.

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

(F) A regioselective oxidation of phenols to \(\omega\)-quinones was performed with IBX.\(^10\)

\[
\begin{align*}
\text{OH} & \quad \text{IBX, 1 equiv} \quad \text{r.t.} \quad 85–92% \quad 11 \text{ examples} \\
\end{align*}
\]

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

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

References


(5) Karthikeyan, G.; Perumal, P. T. *Synlett* 2003, 2249.


