Reduction of Styrene Compounds by Hydrogen Iodide

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Keywords: Styrene compound; Alkene; Hydrogen Iodide; Reduction

Focused on the reduction ability of hydrogen iodide (HI), we have reported the chemoselective reduction of α,β -unsaturated carbonyl and carboxylic compounds with aqueous HI through the addition of HI at olefin and reduction of C-I bond. 1) Herein we report the reduction of styrene derivatives with aqueous HI.

When 1a was treated with 57 wt% aqueous HI (2.5 equiv.) in toluene under refluxing conditions, 2a was obtained in 72% yield (Scheme 1). The improvement of yield was achieved by changing solvent into o-xylene because of raising the temperature (b.p.: toluene, 110 °C; o-xylene, 145 °C). In the case of 1b, the excellent yield (95%) was obtained in toluene.

* Yield was determined by ¹H NMR using *p*-chlorobenzaldehyde as an internal standard

We also examined alkylated styrene derivative (1c). When the reaction was conducted with same conditions for 1a and 1b, the reduction proceeded to give 2c in moderate yield (Table 1, Entry 1). No improvement of yield was observed by prolonged reaction time and raising temperature (Entries 2 and 3). In the case of toluene and o-xylene as a solvent, we found the generation of 3 which was formed by the addition of the solvent through Friedel-Crafts alkylation. When the solvent was exchanged into chlorobenzene (b.p.: 131 °C) to decrease the

nucleophilic ability, the Table 1. Reduction of 1c with aq. HI yield of 2c was increased to give 71% yield (Entry 4). High temperature was required give to because lower yield of 2c (31%) was observed at 80 °C accompanied with the iodinated compound, (1-iodoheptyl)benzene, in 65% yield (Entry 5).

- a) Determined by ¹H NMR using *p*-chlorobenzaldehyde as an internal standard
- b) Reaction temperature was 80 $^{\circ}\text{C}$. (1-lodoheptyl)benzene was obtained in 65% yield.

1) Matsumoto, S.; Marumoto, H.; Akazome, M.; Otani, Y.; Kaiho, T. Bull. Chem. Soc. Jpn. **2021**, 94, 590.