A facile and sustainable protocol to the preparation of aryl iodides using stable arenediazonium bis(trifluoromethylsulfonyl) imide salts via the telescopic process

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Abstract
The preparation of aryl iodides in a telescopic reaction using tert-butyl nitrite as a diazotization reagent and a mixture of bis(trifluoromethane) sulfonamide and glacial acetic acid as a mild acidic agent in ethanol followed by iododediazoniation with tetraethylammonium iodide in water was investigated. The current method has other advantages such as minimized waste by avoiding solvent for the purification of products in diazotization step, simple experimental procedure, and good yield of the sterically hindered aryl amines, metal and strong acid-free waste and environmentally benign conditions. The noteworthy features of this study are the preparation of stable arenediazonium bis(trifluoromethylsulfonyl) imide salts that can be used with no significant loss activity after 1 week and bis(trifluoromethane)sulfonamide was recovered in high yields from reactions.

1 | INTRODUCTION

Arenediazonium salts play an important role in organic synthesis as intermediate and a wide variety of aromatic compounds have been prepared using them.\cite{1,2} A serious drawback of arenediazonium salts is their instability in a dry state; therefore, they must be stored and handled carefully to avoid spontaneous explosion and other hazard events.\cite{3,4,5} The nature of the counterion of arenediazonium salts is of utmost importance as it defines their stability; for example, the arenediazonium salts containing low nucleophilic counterions such as \( \text{BF}_4^- \), \( \text{PF}_6^- \), \( \alpha \)-benzenedisulfonimide, aryl sulfonates,\cite{6} and bis(trifluoromethane)sulfonamide\cite{7} can be isolated and stored for long periods of time. However, these arenediazonium salts have some drawbacks such as demanding for expensive, toxic, fatal reagents and very laborious steps to synthesis,\cite{8} arduous separation and purification steps,\cite{9} sensitivity to air, decomposing on heating, commercial unavailability of reagents. Use of metal nitrates as a diazotizing reagent is not promising in large scale; therefore, nitrite-exchange resin (Resin-\( \text{NO}_2^- \)) was investigated as a polymer-supported diazotizing reagent.\cite{10,11} In this procedure, the filtered resin after preparation must be washed with water until the pH of the filtrate became neutral which generates large amounts of salt waste and greatly increases the risk of environmental pollution.\cite{12,13} Considering the aforementioned drawbacks, the search continues for a better and more efficient method for preparation of stable arenediazonium salts in terms of operational simplicity, environmental acceptability, and metal free conditions.

Over the past decade, bis(trifluoromethane)sulfonamide (TFSI-H) and its salts have attracted much interest for their use as catalyst during the ring-opening polymerization of octamethylocotetrasiloxane in the presence of hexamethyldisiloxane,\cite{14} the synthesis of various non-reducing saccharides via ketopyranosylation of 2,3,4,6-tetra-O-benzyl-1-C-methyl-D-hexopyranoses,\cite{15} esterification carboxylic acid with alkyl halides,\cite{16} reactant for preparing the N-fluorobis[(trifluoromethyl)sulfonyl]imide (TFSI-F) as an efficient fluorinating agent,\cite{17,18} and as a component of solid polymer electrolytes.\cite{19} Synthesis, properties, and reactions of TFSI-H were previously studied.\cite{20} The salts of bis-trifluoromethane sulfonimide are widely used as non-aqueous electrolytes in electrochemical applications such as batteries,\cite{21} fuel cells,\cite{22} In this context, their conductivity and wide electrochemical window of bis(trifluoromethylsulfonyl)imide,