Diazotization-Iodination of Aromatic Amines in Water Mediated by Crosslinked Poly(4-vinylpyridine) Supported Sodium Nitrite

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ABSTRACT: An Efficient and simple method for the diazotization of a wide range of aryl amines has been developed by using a polymer-supported sodium nitrite. The diazotization of aryl amines occurred with crosslinked poly(4-vinylpyridine)-supported sodium nitrite, in the presence of concentrated H_2SO_4, at low temperature (0–5°C). The obtained diazonium salt, followed by treatment with KI in water at room temperature or at 60°C to produce a various of aryl iodides. The spent polymeric reagent can be regenerated and reused for several times without losing its activity. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 121: 2621–2625, 2011

Key words: diazotization; aryl iodide; aryl amine; diazonium salt

INTRODUCTION

In 1963, Merrifield[1] introduced a modified technique that overcame the problems associated with classical multistep synthesis in solution. This technique has been used in the production of large amount of products. However, recently, the chemistry of functional polymers has received great attention and became a practical method for the efficient preparation of novel chemical libraries.[2–7] These polymeric reagents are generally used in single-step reactions; their main advantage over nonpolymeric reagent is their insolubility in the reaction medium and consequently their easier work-up by a simple filtration. The reactions often can be driven to completion using excess amounts of reagents without the fear of separation of the excess of reagents from the products. The spent polymeric reagents usually can be removed quantitatively and regenerated. In addition, anions bound to the macroporous resin have the advantage that often react successfully in nonpolar solvents. These reagents usually can be prepared from commercial anion exchange resins or polymers such as crosslinked poly(4-vinylpyridine) as support. We have reported several different types of polymer-supported reagents from crosslinked poly(4-vinylpyridine) and used in organic synthesis.[8–21]

Aromatic diazonium salts are important building blocks not only in classical organic synthesis but also in the preparation of modern organic nanocompounds and the grafting of organic molecules onto metallic or nonmetallic surfaces.[22–27] Aromatic diazonium salts have gained attention in synthetic chemistry due to their powerful synthetic and industrial importance.[28–30] Despite wide applicability in the synthesis of compounds with a diazonium motif, diazonium salts have a serious drawbacks; their intrinsic instability and explosive nature. Because of this instability, subsequent reactions with diazonium salts must be carried out in the same medium in which they were produced. This restricts many chemists from approaching potentially important transformations of this salts.[22]

It is well known that aromatic halides are important building blocks in organic transformations, such as the Suzuki cross coupling and Heck-type reactions,[31–37] and one of the most renowned methods for the synthesis of aryl halides by aryl amines is the traditional Sandmeyer rection.[26,38–41]

Aromatic iodides are important building blocks in modern organic synthesis, especially for carbon–carbon bond formation via crosscoupling reactions.[33–37,42–45] In addition, many aryl iodides are biologically active molecules that are used as drug or diagnostic aids. For example, some aryl halides are used as X-ray contrast agents or radioactively labeled markers in radioimmunoassays.[46,47] One of the most commonly used methods for preparing aromatic iodides is the substitution of a diazonium group by iodine, the so-called Sandmeyer reaction.[22,25,26,41] The process of diazotization is usually

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carried out with sodium nitrite at low temperature in aqueous hydrochloric or sulfuric acid,[33–36,42–48] and then aromatic diazonium salts are reacted with KI in a subsequent reaction, known as iodination, sometimes in the presence of copper salts.[22,25,26,41] Alternative procedures involve more expensive alkyl nitrites as diazotizing agents in the presence of diiodomethane or other sources of iodine.[49,50] However, most iodination methods developed, thus, far have drawbacks such as increased waste or require organic solvents.

Although polymer-supported reagents especially anion exchange resins have been widely applied in organic synthesis,[1–21] but a literature search shows that there are a few reports in the preparation of aryl iodides by using a polymeric reagent.[51,52] Filimonov and coworkers reported unusually stable arenediazonium tosylates and used Amberlyst A-26 NO_2^- form (Resin-NO_2^-) for diazotization-iodination. With using polymeric quaternary ammonium nitrite, the reaction time is very long (in some cases, many hours).

Zarei et al. reported one-pot method for the iodination of aryl amines via stable aryl diazonium silica sulfates under solvent-free conditions.

In general, these reagents suffer at least from one of the following drawbacks: (1) yield of the reactions are low; (2) the reactions must be carried out in the presence of phase transfer catalysts; (3) the reaction times are long; (4) the work-up of the reaction mixtures are difficult; (5) copper salts as catalyst is required; and (6) the by-products are formed.

In this study, we wish to report an efficient and simple method for synthesis of aryl halides from aryl amines using a polymer-supported sodium nitrite under mild and heterogeneous conditions, with high yields (Scheme 1).

Previously, crosslinked poly(4-vinylpyridine) supported sodium nitrite, [P_4-VP]NO_2, was prepared and used for synthesis of nitro alkanes,[10] N-nitrosation of secondary amines,[11,12] nitration of activated aromatic compounds,[21] and synthesis of azo chromophores by diazotization–diazocoupling reactions of aniline and its substituted derivatives with a coupling component.[20] In continuing our studies on the development of the application of [P_4-VP]NO_2 in organic synthesis, herein, we wish to report an improved and general method for preparation of aryl iodide from aryl amines using [P_4-VP]NO_2 under mild and heterogeneous conditions. The fundamental advantage of this method, over the direct electrophilic iodination[53,54] of aromatic compound, is the selective introduction of iodine atoms into a specific position of the aromatic ring. Direct electrophilic iodination, on the other hand, frequently gives a mixture of regioisomers. In this article, we wish to report a facile and environmentally friendly one-pot method for the diazotization and subsequent iodination of aryl amines.
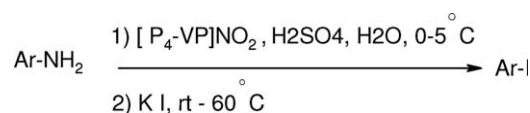
Scheme 1 One-pot method for the diazotization–iodination of aromatic amines.

EXPERIMENTAL

General

Chemicals were either prepared in our laboratory or were purchased from Fluka (Buchs, Switzerland), Aldrich (Milwaukee, WI) and Merck (Germany) chemical companies. Poly(4-vinylpyridine) crosslinked with 2% divinyl benzene (DVB), [P4-VP] 2% DVB, was purchased from Fluka (Buchs, Switzerland). Crosslinked poly(N-methyl-4-vinylpyridinium) iodide, [P_4-VP]I, and crosslinked poly(4-vinylpyridine) supported sodium nitrite, [P_4-VP]NO_2, were synthesized according to our reported procedures[11–13] (Scheme 1). Progress of the reaction was followed by thin layer chromatography (TLC) using silica gel Poly Gram SIL G/UV 254 plates. All products were characterized by comparison of their melting point, FTIR, and ^{1}H-NMR spectral data, with those of known samples and all yields refer to the isolated pure products. Melting points were determined with a Buchi melting point B-540 B.V. CHI apparatus. FTIR spectra were obtained by using a Bruker, Equinox (model 55), and NMR spectra were recorded on a Bruker AC 400, Avance DPX spectrophotometer at 400 MHz in CDCl$_3$ solutions.

Preparation of [P_4-VP]I and [P_4-VP]NO_2

Poly(4-vinylpyridine) crosslinked with 2% DVB (white powder, 100–200 mesh) (1.0 g) was added to a solution of methyl iodide (20 mmol, 3.24 g) in acetonitrile (10 mL), and the mixture was slowly stirred for 24 h at room temperature. The yellow quaternized polymer, [P_4-VP]I, was filtered and was washed with distilled water and acetonitrile. It was then dried under vacuum in the presence of P_2O_5 at 40°C overnight (Scheme 2, Step 1).

The obtained [P_4-VP]I was added to 40 mL of a 3M solution of sodium nitrite and slowly was stirred for 24 h. The prepared resin, [P_4-VP]NO_2, was filtered off and was washed rapidly with distilled water until the filtrate gave a negative test for NO_2^-. It was then washed with diethyl ether and dried under vacuum in the presence of P_2O_5 at 40°C overnight. The activity of the polymer was determined by potentiometric titration of the filtrates with a 0.1N solution of silver nitrate, and it was found to be 3.7 mmol g^{-1} of the polymer.
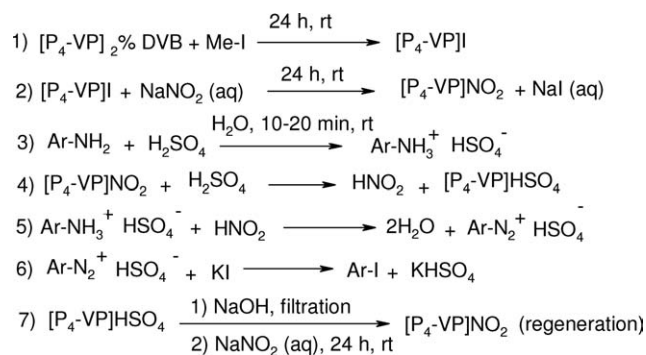
Scheme 2 Preparation of $[P_4\text{-VP}]NO_2$, mechanism of diazotization–iodination of aromatic amines in the presence of $[P_4\text{-VP}]NO_2/H_2SO_4/KI$, and regeneration of polymer.

General procedure for diazotization-iodination of aromatic amines with $[P_4\text{-VP}]\,NO_2$ / H_2SO_4/KI in water at 0.0–60°C, under heterogeneous conditions

An aryl amine (1 mmol) was added to a solution of concentrated H_2SO_4 (2 mmol) in water (5 mL), and the mixture was slowly stirred magnetically at 0–5°C for appropriate time (10–20 min) until the ammonium salt was formed and the aromatic amine was disappeared. The progress of the reaction was monitored by TLC [eluent: n-hexane/ethyl acetate (80 : 20)]. Then, 0.54 g (2 mmol) of $[P_4\text{-VP}]NO_2$, was added and stirring was continued until, the diazonium salt was formed. After completion of the reaction, the suspension was filtered, and was washed with very cold water (3 × 5 mL). Then, 415 mg of KI (2.5 mmol) was added to the filtrates (the salient of nitrogen gas is observed), and the mixture was slowly stirred magnetically at room temperature or at 60°C for appropriate time as indicated in Table I (40–100 min). The crude product was treated with aqueous solution of Na_2SO_3 (10%, 8 mL), and the precipitated aromatic iodide product was filtered and was dried. If further purification is needed recrystallization in $EtOH/H_2O$ (5 : 1), or column chromatography on silica gel, [eluent: n-hexane/ethyl acetate (91 : 9)] provides highly pure products. Only when the product was liquid, the reaction mixture was extracted with diethyl ether (3 × 8 mL) that, after drying of the organic layer (with $MgSO_4$) and evaporation of the solvent, highly pure product is obtained (Table I, entries 7, 11, 15, 19). FTIR and ^{1}H-NMR spectra data of some selected prepared aryl iodides are given below:

P-Bromoiodobenzene

^{1}H-NMR (400 MHz, CDCl3) δ (ppm) =7.24 (d, 2H), 7.55 (d, 2H); FTIR (neat) ν (cm^{-1}): 3071, 1618, 994, 798.

4-Iodophenyl methyl ketone

^{1}H-NMR (400 MHz, CDCl3) δ (ppm) =2.60 (s, 3H), 7.66 (d, 2H), 7.83 (d, 2H); FTIR (cm^{-1}) 3002, 1671, 1581, 816.

m-Iodonitrobenzene

^{1}H-NMR (400 MHz, CDCl3) δ (ppm) =7.27 (t, 1H), 7.98 (d, 1H), 8.15 (d, 1H), 8.46(s, 1H); FTIR (neat) ν (cm^{-1}) 3072, 1520, 1490, 1341, 862, 797.

4-Iodoanisole

^{1}H-NMR (400 MHz, CDCl3) δ (ppm) =3.78 (s, 3H, OCH_3), 6.68 (d, 2H), 7.56 (d, 2H); FTIR (neat) ν (cm^{-1}) 3007, 2838, 1585, 1243, 1026, 808.

p-Iodonitrobenzene

^{1}H-NMR (400 MHz, CDCl3) δ (ppm) =7.92 (d, 2H), 7.95 (d, 2H); FTIR (neat) ν (cm^{-1}) 3092, 1593, 1568, 1504, 1337, 850, 843.

Regeneration of $[P_4\text{-VP}]NO_2$

The spent cream-colored polymer (1 g) was washed with aqueous NaOH and then added to a 40 mL of a $3M$ solution of sodium nitrite and was slowly stirred for 24 h. The mixture was filtered, washed several times with distilled water, and dried under vacuum in the presence of P_2O_5 at 40°C overnight (Scheme 2, Step 7). The capacity of the regenerated polymer was determined and was found that, it had the same capacity as the original form (3.7 mmol g^{-1} of polymer). The regenerated polymer can be reused several times, without losing its activity. Although, the use of the $[P_4\text{-VP}]NO_2$ that is recycled for fourth time are given in Table I (entries 3-6) but also the regenerated $[P_4\text{-VP}]NO_2$was used for preparation of other aryl iodides.

RESULTS AND DISCUSSION

Crosslinked poly(4-vinylPyridine) supported sodium nitrite was prepared according to our previous reported procedure[10,11] and used as an efficient procedure for diazotization-iodination of aromatic amines (Scheme 1). It is important to note that this polymeric reagent is stable and can be stored for long time (months), without losing its activity and can be readily used for diazotization-iodination of anilines.

To increase the yield of iodo compounds optimization of the reaction conditions was accomplished. 4-Nitroaniline (1 mmol) was chosen as a model substrate and was treated with different molar ratio of

TABLE I
Diazotization-Iodination of Aromatic Amines by [P$_4$-VP]NO$_2$/H$_2$SO$_4$/KI in Water at 0–60°C

$$Ar-NH_2 \xrightarrow[\text{2) KI, rt–60°C}]{\text{1) [P}_4\text{–VP]NO}_2,\ \text{H2SO4, H2O, 0–5°C}} Ar-I$$
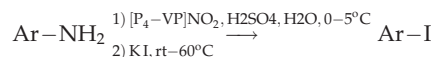
Entries	Ar-NH$_2$	Ar-I	Diazotization/Iodination Time (min)	Isolated yield (%)	M.P./Lit (°C)
1	Ph-NH$_2$	Ph-I	40/100[a]	0.0	–
2	4-NO$_2$C$_6$H$_4$NH$_2$	4-NO$_2$C$_6$H$_4$I	25/50	79	173/172–174
3	4-NO$_2$C$_6$H$_4$NH$_2$	4-NO$_2$C$_6$H$_4$I	25/50[b]	79	173/172–174
4	4-NO$_2$C$_6$H$_4$NH$_2$	4-NO$_2$C$_6$H$_4$I	25/51[b]	79	173/172–174
5	4-NO$_2$C$_6$H$_4$NH$_2$	4-NO$_2$C$_6$H$_4$I	25/52[b]	79	173/172–174
6	4-NO$_2$C$_6$H$_4$NH$_2$	4-NO$_2$C$_6$H$_4$I	25/50[b]	79	173/172–174
7	3-NO$_2$C$_6$H$_4$NH$_2$	3-NO$_2$C$_6$H$_4$I	25/65	75	Oil/Oil
8	2-NO$_2$C$_6$H$_4$NH$_2$	2-NO$_2$C$_6$H$_4$I	30/70 [a]	74	50/49–51
9	4-NCC$_6$H$_4$NH$_2$	4-NCC$_6$H$_4$I	20/40	89	125/124–128
10	4-IC$_6$H$_4$NH$_2$	4-IC$_6$H$_4$I	20/45	88	127/130
11	2-IC$_6$H$_4$NH$_2$	2-IC$_6$H$_4$I	30/50[a]	80	Oil/Oil
12	4-BrC$_6$H$_4$NH$_2$	4-BrC$_6$H$_4$I	25/45	81	88/89–91
13	4-ClC$_6$H$_4$NH$_2$	4-ClC$_6$H$_4$I	25/75	61	54/52–56
14	4-MeC$_6$H$_4$NH$_2$	4-MeC$_6$H$_4$I	25/80[a]	67	30/33–35
15	2-BrC$_6$H$_4$NH$_2$	2-BrC$_6$H$_4$I	30/85[a]	62	Oil/Oil
16	4-HOOCC$_6$H$_4$NH$_2$	4-HOOCC$_6$H$_4$I	20/40	85	270/270–273
17	4-MeOC$_6$H$_4$NH$_2$	4-MeOC$_6$H$_4$I	20/60	78	49/50–53
18	4-MeCOC$_6$H$_4$NH$_2$	4-MeCOC$_6$H$_4$I	20/45	84	83/82–84
19	1-Naphthyl amine	1-Iodonaphthalene	35/90[a]	68	Oil/Oil
20	2,4 (NO$_2$)$_2$C$_6$H$_3$NH$_2$	2,4-(NO$_2$)$_2$C$_6$H$_3$I	40/100[a]	41	–

[a] The reaction was carried out at 60°C.
[b] The entries 3-6, refer to the use of the [P$_4$-VP]NO$_2$ that is recycled first, second, third, and fourth time, respectively, under identical conditions.

[P$_4$-VP]NO$_2$/H$_2$SO$_4$/KI and in different solvents. It was observed that the 2/2/2.5 molar ratio and water as solvent were the best.

The diazotization–iodination reactions were investigated under mild and completely heterogeneous conditions at room temperature and at 60°C, and the results are summarized in Table I. A good range of available aromatic amines were also subjected to diazotization–iodination by using [P$_4$-VP]NO$_2$/H$_2$SO$_4$/KI in water at 0–60°C. This new, simple method can be successfully applied for the synthesis of a wide range of aryl iodides starting from the corresponding aryl amines. Various aromatic amines, with electron-withdrawing groups and electron-donating groups, were transformed into aromatic iodides in good yields (Table I). However, when aniline was subjected to this novel diazotization–iodination even after 100 min, no iodo product was separated, and a few by-products are formed that are not identified (Table I, entry 1). However, there is no reported result for diazotization–iodination of aniline in the other reported methods for halogenation of arenediazonium salts, such as arenediazonium tosylates with nucleophilic reagents,[51] efficient and economic halogenation of aryl amines via arenediazonium tosylate salts,[53] a simple and efficient

procedure for diazotization–iodination of aromatic amines in aqueous pastes by the action of sodium nitrite and sodium hydrogen sulfate,[54] and iodination of aryl amines in a water-paste via stable aryl diazonium tosylates.[55] Also, Iodination of 2,4-dinitroanilline provided only moderate yield of the corresponding iodo derivative. This result is in agreement with previously reported methods.[54,55]

We have also successfully applied this new method on a large scale. For example, up to 15 mmol of *p*-nitroaniline (entry 2) could be converted into 4-iodonitrobenzene without any loss of efficiency. In this method, the reaction time will be developed, which is almost shorter than previously reported methods.[51–55] This can probably be attributed to the local concentration of iodide ion species inside the pores.

The plausible mechanism of diazotization–iodination of aromatic amine is given in Scheme 2.

The advantages of this method over conventional classical methods[56,57] are mild reaction conditions, safe handling, mildness of polymeric nitrating reagent and very simple work-up. In addition, there is current research and general interest in heterogeneous systems because of the importance such systems have in industry and in developing technologies.[58]

CONCLUSIONS

In conclusion, this research demonstrates the synthesis of aromatic iodides from various aniline derivatives via a polymer-supported sodium nitrite and concentrated H_2SO_4 in the presence of KI. The protocol is operationally simple and efficient method under heterogeneous conditions, free-catalyst (Cu salt), and at room temperature in water with good to high yield in most cases.

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