

Original Article: Ammonium Salts as Economical and Eco-friendly N-Sources Applied to Green, Simple and Scale-up Synthesis of Trialkyl Amines in Water



Elahe Ghasbeigi | Mohammad Soleiman-Beigi*

Department of Chemistry, Ilam University, P.O. BOX 69315-516, Ilam, Iran



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ABSTRACT

We have introduced a selective synthesis of tertiary amines using alkyl halide and ammonium salts as the amine sources in water. This green process has outstanding superiorities, such as being eco-friendly, possessing ammonium salts, and using water as a green solvent in the absence of organic ligands or catalysts. It is worth mentioning that the presence of *t*-Butyl alcohol and potassium hydroxide leads to synthesize tertiary amines, while under other conditions we witnessed the formation of byproducts. Other factors affecting the synthesis of various tertiary amines are temperature ranges. Note that various tertiary amines and the process of scale-up were synthesized in moderate to high yields.

Introduction

Since the green chemistry's aim is to use water as the solvent of reactions, significant efforts have been made for N-alkylation of amines in water [1].

Between nitrogen-containing organic compounds, amines are important molecules that have many applications in the industry, medicine, agriculture, paints and organic synthesis. They have also been known as building blocks for material chemistry and ligands [2-4]. Tertiary amines are utilized in the textile industry, polymer, and composite [5,6]. Moreover, they have been used as ion-pairing reagents [7], catalysts [8] and base.

Historically, traditional routes for the synthesis of amines are involved in Gabriel [9], Hofmann [10], Mitsunobu reaction [11], Buchwald-Hartwig [12], Curtius rearrangement methods [13] and N-alkylation [14]. N-Alkylation is the most common reaction for the synthesis of all amines types, that is to say, it is performed by the reaction of the N-source (amines and liquid ammonia) with alcohols and alkyl halides [15,16] in the presence of acids/bases catalysts [17]. Ammonia which is a chemical substance, toxic [18], has a most common pollutant in water and industrial wastewater [19], and a threat to aquatic and human life [20]. In this regard, researchers

*Corresponding Author: Mohammad Soleiman-Beigi (m.soleimanbeigi@ilam.ac.ir)

use eco-friendly methods by applying solid and safer nitrogen-donor reagents such as 5-methyl 1, 3, 4-thiadiazole-2-amine, lithium amide and urea in the N-alkylation reaction for the synthesis of amines [21]. Ammonium salts as solid, green and safer N-donors are used in organic reaction [22], heterocycles synthesis [23,24], and N-alkylation of benzylic alcohols [25,26]; additionally, they are also used as buffer [27], base, phase transfer and food additive [28]. In order to introduce a green, efficient eco-friendly and commercial procedure for the tertiary amines synthesis, reactions of alkyl halides and ammonium salts in water were successfully investigated.

Experimental

General

The chemicals used in this work were from Sigma-Aldrich Merck. The ^1H NMR and ^{13}C NMR spectra were recorded at 400 and 100 MHz, respectively, in CDCl_3 or D_2O as the solvent and tetramethylsilane (TMS) as the reference. Other analyses including the FT-IR spectra were measured using the KBr. Besides, melting points were determined by Electrothermal 9100 apparatus. Monitoring products was performed by thin-layer chromatography (TLC) technique (Merck silica gel 60 F_{254} , under UV light at 365 nm). The residue oil products and crud solid were purified by plate chromatography (silica gel 60 GF_{254}) and crystallization, respectively.

Typical procedure for the synthesis of tribenzylamine (3a-3h)

A mixture of alkyl halide (1.25 mmol), ammonium acetate (0.7 mmol), *t*-Butyl alcohol (4 mmol) and KOH (2 mmol) in water (2 mL) was stirred at 100 °C temperature for 2.5 h. After consumption of the reactants and completion of the reaction by TLC (ethyl acetate/ *n*-hexane 1:9) monitoring, the reaction mixture was cooled and, then, it was extracted by dichloromethane (15 mL x 3). Afterwards, the reaction mixture was washed with HCl (5%, 5 mL) and water. The organic layer then was separated and dried over anhydrous Na_2SO_4 . In order to obtain pure tri alkyl amines, the solvent was evaporated and the residue crud solid was purified by

becoming crystallized in ethanol. The oil products were purified by the preparative chromatography (ethyl acetate/ *n*-hexane 1:9).

General procedure for the synthesis of 4-methyl benzene sulfonamide (7)

Ammonium acetate (0.7 mmol) and *t*-Butyl alcohol (4 mmol) in H_2O (2 mL) KOH (2 mmol) was added to a mixture of 4-methyl benzenesulfonyl chloride (1.25 mmol). The reaction mixture was stirred at a temperature of 100 °C for 2.5 h. After cooling to room temperature, the mixture was extracted with EtOAc: H_2O (15 mL x 3). The organic layer was separated and then dried over anhydrous Na_2SO_4 . Then, the residue crud solid was purified by becoming crystallized in ethanol in order to give the desired product **7** in 90% yields, mp: 122-124 °C.

General procedure for the synthesis of *N*-benzyl-4 methylbenzenesulfonamide (8)

A 2 mL H_2O reaction vessel was charged with 4-methyl benzenesulfonyl chloride (0.7 mmol), benzyl chloride (0.7 mmol), ammonium acetate (0.7 mmol), *t*-Butyl alcohol (4 mmol) and KOH (2 mmol). The resulting solution was stirred at 100 °C for 2.5 h. After cooling to room temperature, the mixture was extracted with EtOAc: H_2O (15 mL x 3). The combined organic phase was dried over anhydrous Na_2SO_4 . Purification was done by plate chromatography (ethyl acetate/ *n*-hexane 8:1) to give the desired product **8** in 40% yields as a white solid, mp: 110-113 °C.

General procedure for the synthesis of 2,2',2''-nitrilotriacetic acid (10)

2,2',2''-Nitrilotriacetic acid was obtained via the reaction with ethyl bromoacetate (1.2 mmol), ammonium acetate (0.7 mmol), *t*-Butyl alcohol (4 mmol) and KOH (2 mmol). The reaction was monitored in water under reflux condition in 2.5 h. After cooling to room temperature, the mixture was extracted with EtOAc: H_2O (15 mL x 3). Then hydrochloric acid (2 mL) was added to the water phase, and the mixture was heated for 1 h. After standing overnight, the acid was crystallized out and the precipitate was filtered by ethanol to give

the desired product **10** in 70% yields as a white solid, mp: 229-232 decomposition.

Results and discussion

We began our investigation using the reaction of benzyl chloride **1a**, ammonium acetate **2a** and *t*-BuOH as co-solvents in order to find the best base, solvent, and temperature. Accordingly, in the absence of water and base, the desired product **3a** was obtained with 32% yields (Entry 5, Table 1) in *t*-BuOH. As shown in Table 1, under the reaction conditions in the presence of water and in an alkaline environment, benzyl chloride may be converted into three products: tribenzyl amine **3a**, dibenzyl ether **4a** and benzyl alcohol **5a**. In the absence of the base and in water and DMF, trace amount of tribenzyl amine **3a** (Entries 2 and 4, Table 1) was detected. The influences of the *t*-BuOH and temperature on the yield of **3a** were also studied; by decreasing the reaction temperature from 100 °C to 85 °C, the desired product's (**3a**) yield was reduced from 90 % to 55% (Entries 1 and 6, Table 1).

It was observed that *t*-BuOK as a base was also found to be effective for the synthesis of tribenzyl amine. The results show that the base and temperature are very important in this procedure, as it has been clarified, the reaction was not performed in the absence of *t*-BuOK or at room temperature in water (Entries 12 and 15, Table 1). In the presence of *t*-BuOK, tri-benzylamine can be regarded as the main reaction product, while by changing base to NaOH and K₂CO₃, a mixture of the three products is created (Entries 13 and 14, Table 1). It should be noted that, herein, the reaction temperature, affects the efficiency of the reaction and has the key role in the progress of the reaction as shown in Entries 8-11, Table 1. As it is deduced from Table 1, at a temperature of less than 100 °C, the reaction efficiency is reduced. Moreover, the procedure works fine with both of *t*-BuOK and *t*-BuOH/KOH systems in water. In this sense, we selected the utility of the *t*-BuOH/ KOH system as an efficient, inexpensive, mild and green for the tribenzyl amine synthesis. In addition, synthesis of dibenzyl ether is also possible by changing the base/ solvent and temperature of the reaction (Entry 16, Table 1).

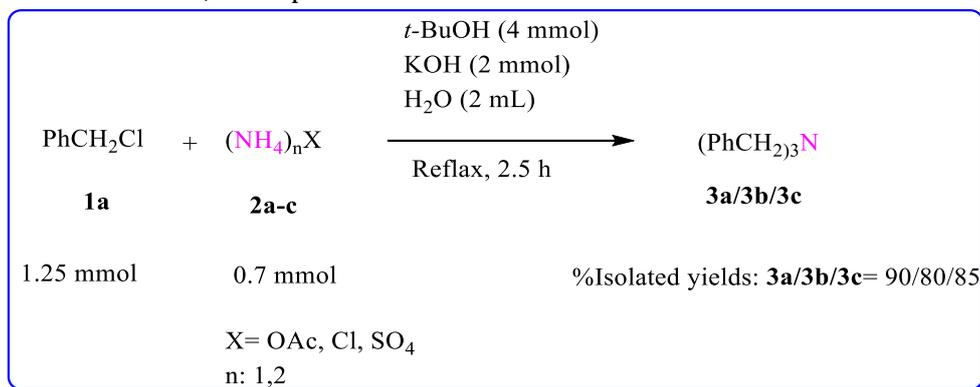
Table 1. Optimization of the reaction conditions

Entry	Base (mmol)	Solvent (2 mL)	*Co-solvent (mmol)	Temperature (°C)	Yield% ^a 3a: 4a: 5a
1	KOH (2)	H ₂ O	4	100	90: 0: 0
2	KOH (2)	DMF	4	100	trace: 30: 20
3	KOH (3)	H ₂ O	6	100	30: 30: 30
4	-	H ₂ O	6	100	trace: 0: 0
5	-	<i>t</i> -BuOH	-	100	32: 0: 0
6	KOH (2)	H ₂ O	6	85	55: 15: 20
7	NaOH (2)	H ₂ O	6	85	52: 18: 20
8	<i>t</i> -BuOK (2)	DMF	-	130	90: 0: 10
9	<i>t</i> -BuOK (2)	H ₂ O	-	100	80: 0: 10
10	<i>t</i> -BuOK (2)	H ₂ O	-	70	60: 0: 10
11	<i>t</i> -BuOK (2)	H ₂ O	-	50	40: 40: 10
12	<i>t</i> -BuOK (2)	H ₂ O	-	r. t.	N. R
13	K ₂ CO ₃ (2)	H ₂ O	-	100	40: 40: 10
14	NaOH (2)	H ₂ O	-	100	40: 40: 10
15	-	H ₂ O	-	100	N.R
**16	K ₂ CO ₃ (1.25)	DMF	-	80	10: 80: 10

Reaction conditions: Alkyl halides (1.25 mmol), NH₄OAc (0.7 mmol), **NH₄OAc (1.25 mmol), aIsolated yield, * *t*-BuOH

Herein, in order to find the best N-donor of the reaction between the available nitrogen-donors, the model reaction for tribenzyl amine **3a** synthesis with various ammonium salts was investigated. NH_4OAc , NH_4Cl and $(\text{NH}_4)_2\text{SO}_4$ are available, inexpensive and

effective nitrogen sources which can be given ammonia under the reaction conditions. As shown in Scheme 1, the desired product was obtained with more than 80% yields from ammonium salts.

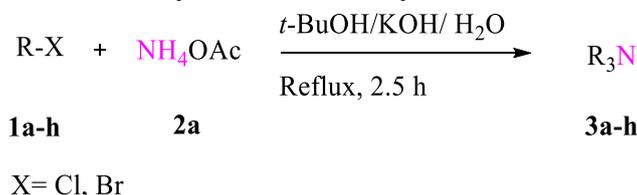


Scheme 1. Synthesis of symmetric tertiary amines from alkyl halides and ammonium salts

According to the above descriptions, optimal reaction conditions for tertiary amine synthesis was obtained with benzyl chloride (1.25 mmol), *t*-Butyl alcohol (4 mmol), potassium hydroxide (2 mmol), and ammonium acetate (0.7 mmol) as N-source, in 2 mL water and at 100 °C. After obtaining the optimized conditions, synthesis of tertiary amines by the reaction of ammonium acetate with alkyl halides was tested. It is noteworthy that different alkyl halides could be used as primary materials for tertiary amines' synthesis with high yields (Table 2). Tris(2-

phenylethyl) amine and styrene were obtained from the reaction of phenylethyl bromide and ammonium acetate in the presence of potassium hydroxide and *t*-Butyl alcohol. However, Tris (2-phenylethyl) amine was synthesized in water in better yield (75%) (Entry 3, Table 2). Because of the fact that bromobutane has a lower boiling point as compared to other alkyl halides, tributylamine synthesis was not done at 100 °C temperature; However, it was synthesized with 40% efficiency with a lower temperature at 70 °C (24 h) (Entry 8, Table 2).

Table 2. Synthesis of tertiary amines from alkyl halides and ammonium acetate

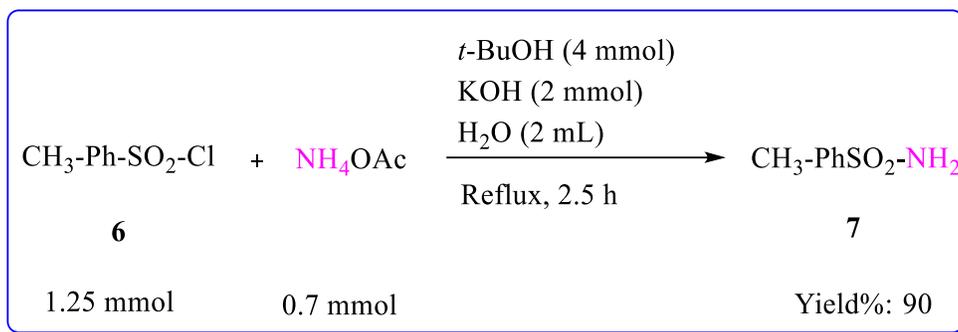


Entry	R-X	R ₃ N	Yield% a	M.p. (lit. m.p.)/ °C	Ref
1	PhCH ₂ Cl (1a)	(PhCH ₂) ₃ N (3a)	90	89-91 (90-91)	[29a]
2	PhCH ₂ Br (1b)	(PhCH ₂) ₃ N (3b)	92	89-91 (90-91)	[29a]
3	PhCH ₂ CH ₂ Br (1c)	(PhCH ₂ CH ₂) ₃ N (3c)	75	29 (31)	[29b]
4	PhCH ₂ CH ₂ CH ₂ Br (1d)	(PhCH ₂ CH ₂ CH ₂) ₃ N (3d)	89	Oil	[29c]
5	4-CH ₃ -PhCH ₂ -Br (1e)	(4-CH ₃ -PhCH ₂) ₃ N (3e)	91	67-68 (55-57)	[29d]
6	2-Cl-PhCH ₂ Cl (1f)	(2-Cl-PhCH ₂) ₃ N (3f)	93	88-89 (90-91)	[29d]
7	CH ₂ =CHCH ₂ Br (1g)	(CH ₂ =CHCH ₂) ₃ N (3g)	40	Oil	[29e]
8	^b CH ₃ CH ₂ CH ₂ CH ₂ Br (1h)	(CH ₃ CH ₂ CH ₂ CH ₂) ₃ N (3h)	40	Oil	[29f]

Reaction conditions: Alkyl halides (1.25 mmol), NH_4OAc (0.7 mmol), *t*-Butyl alcohol (4 mmol), KOH (2 mmol), H_2O (2 mL), at 2.5 h, under Reflux conditions. ^aIsolated yield. ^b 24 h, 70 °

Other important features of this process would apply the other alkyl halides for the synthesis of various tertiary, secondary and primary amines in moderate to excellent yields. Considering the above notions, sulfonamides are synthesized by this procedure, due to sulfonamide unit as an

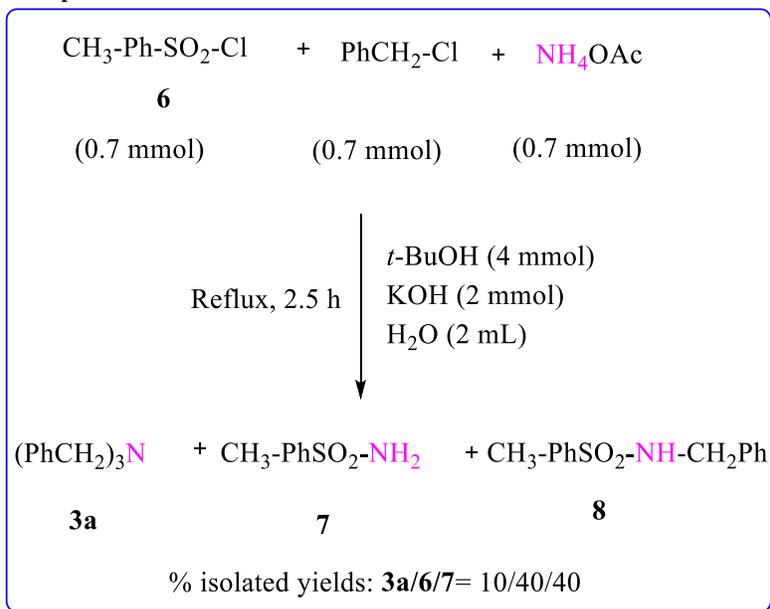
amine protecting group in organic chemistry, medicinal chemistry, drugs, and antitumor [30,31]. 4-Methyl benzenesulfonamide was synthesized successfully from the reactions of 4-methyl benzenesulfonyl chloride and ammonium acetate (Scheme 2).



Scheme 2. Synthesis of primary amines from 4-methyl benzenesulfonyl chloride and ammonium acetate

The mentioned procedure was examined for the one-pot synthesis of N-benzyl sulfonamide in water. The desired product **8** was obtained

together with the two side products **3a** and **7** (Scheme 3).



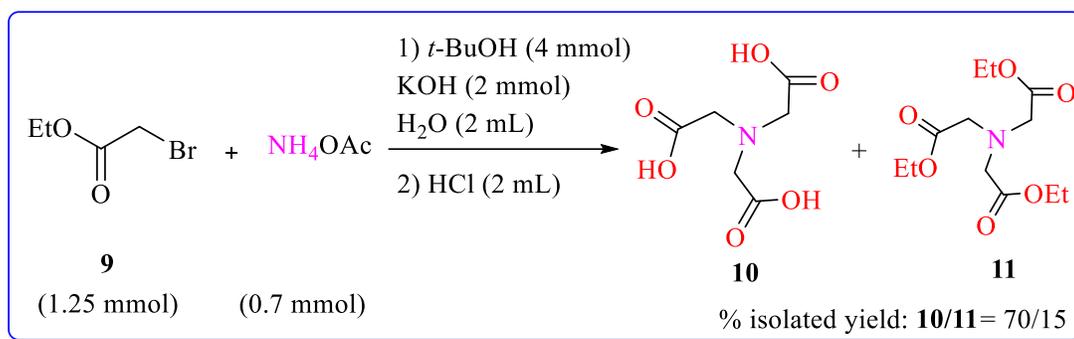
Scheme 3. Synthesis of tertiary, secondary and primary amines from benzyl chloride and 4-methyl benzenesulfonyl chloride and ammonium acetate

The procedure was also extended to the reaction of ammonium acetate with alkyl halides involving other active functional

groups such as ethyl bromoacetate and epichlorohydrin. Interestingly, 2,2',2''-Nitrilotriacetic acid (H₃NTA) was obtained

from the reactions of ethyl bromoacetate with ammonium acetate under the optimized reaction conditions in 70% yields. In this reaction, triethyl

nitrilotriacetate was yielded as a by-product (Scheme 4) while 2,2',2''-Nitrilotriacetic acid was used in the cleaning products [32].



Scheme 4. Synthesis of tertiary amines from ethyl bromoacetate and ammonium acetate

In continuation of our study, the procedure was scaled-up for the synthesis of tribenzylamine by the reaction of benzyl chloride and ammonium acetate in water with high efficiency. We checked the process of

scale-up up to 20 times and observed the increasing substrates under the optimized laboratory condition. Conclusions are illustrated in Table 3.

Table 3. Synthesis of tribenzylamine from benzyl chloride and ammonium acetate

PhCH₂Cl + **NH₄OAc**

(1.25 mmol) (0.7 mmol)

t-BuOH (4 mmol)
KOH (2 mmol)
H₂O (2 mL)
Reflux, 2.5 h

3a

Entry	PhCH ₂ Cl (mmol)	NH ₄ OAc (mmol)	<i>t</i> -BuOH (mmol)	KOH (mmol)	Yield% ^a
1	1.25	0.7	4	2	90
2	6.25	3.5	20	10	90
3	12.5	7	40	20	90
4	18.75	10.5	60	30	89
5	25	14	80	40	85

^aIsolated yield

The comparison between this procedure and the ones reported in the literature shows that trialkyl amines are synthesized in a short reaction times, with high yield and without using the catalyst. Moreover, ammonium salts were effective as solid, available, portable and green nitrogen donors, while other nitrogen donors are not like this. In this regard, ammonium salts can be used as the best N-

donor for tertiary amines synthesis (Table 4, SI).

Conclusion

In this paper, we have achieved a simple and green method for trialkyl amine synthesis from alkyl halide and ammonium acetate in water.

The procedure is not only applicable for the synthesis of tertiary amines but also is efficient for the synthesis of 2,2',2''-Nitrilotriacetic acid, triethyl nitrilotriacetate, N-benzyl-4-methylbenzenesulfonamide and 4-methylbenzenesulfonamide from the related halides. *T*-Butyl alcohol and temperature both were found as effective factors in the progress of the reactions. The main advantages of this process are, 1) the use of water as a green solvent and eco-friendly conditions, 2) the use of ammonium salts as a cheaper, available and appropriate N-source, 3) economical conditions, 4) the avoiding use of toxic reagents, 5) easy purification and handle and 6) short reaction times. Additionally, the procedure was scaled-up for the tribenzylamine synthesis by 85-90 % efficiency after 20 times increasing of the substrates.

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