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Graphical Abstract

Iron(0) nanoparticles mediated direct conversion of aryl/heteroaryl amines to chalcogenides <i>via</i> in situ diazotization	Leave this area blank for abstract info.
Subir Panja, Pintu Maity, Debasish Kundu, and Brindaban C. R	anu*
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Iron(0) nanoparticles mediated direct conversion of aryl/heteroaryl amines to chalcogenides *via* in situ diazotization

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ABSTRACT

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Introduction

The aryl/heteroaryl chalcogenides are of much interest as they constitute the framework of many molecules having important biological activities such as anticancer, antioxidant, enzymeinhibiting, and applications as useful materials.^{1,2} They are also employed as useful intermediates in organic reactions.³ Hence, the synthesis of chalcogenides is of continued interest and a plethora of methods have been reported. One of the widely employed procedures is the transition metal catalysed arylchalcogen bond formation. Several transition metals such as Pd,Ni,Cu,Fe,Co,Rh and In have been used as catalysts for the reaction of aryl halides, aryl boronic acid, aryl triflates with thiols, selenols/PhSeNa and diaryl dichalcogenides for the access to diaryl chalcogenides.⁴⁵ In some cases these procedures need long reaction time, higher temperature and stoichiometric or greater amount of metal catalysts and the yields were not always good.⁶ Moreover, aryl halides, aryl boronic acid and aryl triflates are relatively expensive. Hence, amines being less expensive and readily available commercially, have been employed in several recent reactions. Usually aryl amines are converted to stable diazonium fluoroborates which are subsequently subjected to reaction with diaryl dichalcogenides under different conditions such as microwave irradiation,⁷ ball milling⁸ and in the presence of hypophosphorus acid⁹ for carbon-chalcogen bond formation. However the use of aryl diazonium fluoroborate limits the substrate scope because of the practical disadvantage to diazotize the heteroaryl amines in standard acidic medium.¹⁰ To avoid

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A simple procedure for the synthesis of organo-chalcogenides has been developed by the reaction of aryl/heteroaryl amines with di-aryl/heteroaryl dichalcogenides in the presence of 'BuONO and Fe(0) nanoparticles. The reaction proceeds *via* in situ diazotization followed by chalcogenation. A series of functionalized diaryl/aryl heteroaryl/diheteroaryl/aryl-alkyl selenides, sulfides and tellurides have been obtained by this procedure. Significantly, using this procedure 2,4-dinitroaniline is converted to (2,4-dinitrophenyl)(phenyl)selane which is known as thioredoxin reductase (TR) and glutathione reductase (GR) inhibitor. The reaction goes by a radical pathway and a plausible mechanism has been suggested.

this drawback recently our group has developed an alternative route for the synthesis of diaryl/heteroaryl chalcogenides by direct use of aryl/ heteroaryl amines and 'BuONO in visible light (blue LED) photocatalyzed condition.¹¹ Very recently Cai et.alreported synthesis of aryl sulphides from aniline using ascorbic acid as a promoter although selenides and tellurides were not addressed by this procedure.¹² We report here a more convenient and general procedure avoiding any special device such as LED, for the synthesis of aryl/heteroaryl sulfides, selenides and tellurides with a wide substrate scope by Fe(0) nanoparticles mediated direct conversion of aryl/heteoaryl amines *via* in situ diazotization using 'BuONO (Scheme 1). Although there are several reports of Fe(0) mediated reactions¹³ no report of its use for chalcogenide bond formation is known. Iron nanoparticles were prepared by a reported procedure and were used as such.¹⁴



Scheme 1. Synthesis of aryl/heteroaryl selenides, tellurides and sulfides from aryl/heteroaryl amines

Results and Discussion

To optimize the reaction conditions a series of experiments were performed with variation of solvent, temperature and time for a representative reaction of 4-methoxy aniline and diphenyl diselenide in the presence of Fe(0) NPs¹⁴. The results are summarized in Table 1. Among a variety of solvents studied, dimethyl sulfoxide (DMSO) produced the best yield in the presence of Fe(0) NPs (1.0 equiv.) and ^{*t*}BuONO (1.1 equiv.) at

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30 °C for 8 h (Table 1, entry 6). Dimethyl formamide (DMF) is also considerably effective (Table 1, entry 2) whereas, use of Nmethylpyrrolidone (NMP) (Table 1, entry 1), CH₃CN, dimethyl carbonate, toluene and THF (Table 1, entries 7,8,9 and 10) led to lower yield. The change of reaction temperature to 50 °C and 70 °C did not increase the yield further for this reaction (Table 1, entry 11 and 12). The reaction for longer time than 8 h (Table 1, entries 3 and 5) reduced the yield. When Fe(0) NPs was used in more than 1 equivalent no better result was obtained (Table 1, entry 13). In the absence of Fe(0) NPs, the reaction did not occur (Table 1, entry 4). The reaction produced relatively low yield (46%) in presence of Fe powder (Table 1, entry 14). In case of diphenyl ditelluride the reaction afforded the best yield in DMSO in presence of Fe(0) NPs (1.0 equiv.) and ^tBuONO (1.1 equiv.) at 80 °C for 12 h (Table 1, entry 18). The lowering of temperature (50 °C and 30 °C) and time (8 h) did not give satisfactory yield of the product (Table 1, entries 15-17). For diphenyl disulfide the reaction provides the best result in DMSO in the presence of Fe(0) NPs (1.0 equiv.) and ^tBuONO (1.1 equiv.) at 80 °C for 8 h (Table 1, entries 19, 20, 21 and 22).

Table 1

Standardization of reaction conditions ^a

Ĺ		1BuONO	175	E	E Ph		
MeO	solv	rent, tempei time	rature Med	o			
Ent	Ph_2E_2	Fe(0	Solven	Time	Temp	Yield	
ry) nps	t	(h)	(°C)	%	
		(eq.)					
1	Ph_2Se_2	1	NMP	12	30	30	
2	Ph_2Se_2	1	DMF	12	30	70	
3	Ph_2Se_2	1	DMSO	12	30	76	
4	Ph_2Se_2	-	DMSO	10	30	trac	
						е	
5	Ph ₂ Se ₂	1	DMSO	10	30	77	
6	Ph ₂ Se ₂	1	DMSO	8	30	82	
7	Ph_2Se_2	1	CH₃CN	8	30	30	
8	Ph_2Se_2	1	DMC	8	30	65	
9	Ph_2Se_2	1	toluen	8	30	45	
			e				
10	Ph_2Se_2	1	THF	8	30	49	
11	Ph_2Se_2	1	DMSO	8	50	78	
12	Ph_2Se_2	1	DMSO	8	70	44	
13	Ph_2Se_2	2	DMSO	8	30	73	
14 ^b	Ph_2Se_2	1	DMSO	8	30	46	
15	Ph_2Te_2	1	DMSO	8	30	21	
16	Ph_2Te_2	1	DMSO	8	50	40	
17	Ph_2Te_2	1	DMSO	8	80	70	
18	Ph_2Te_2	1	DMSO	12	80	74	
19	Ph_2S_2	1	DMSO	12	30	60	
20	Ph_2S_2	1	DMSO	12	80	73	
21	Ph_2S_2	1	DMSO	8	80	81	
22	Ph_2S_2	1	DMSO	8	70	65	

^a*Reaction conditions*: 4-Methoxy aniline (1 mmol), diaryl dichalcogenides (0.5 mmol), ¹BuONO (1.1 mmol), Fe(0) NPs (1.0 mmol). ^bFe powder (1 equiv.) instead of Fe (0) NPs.

In a typical experimental procedure, a mixture of an amine and diphenyl diselenide/ditelluride/disulfide in DMSO was heated (30-80°C) in the presence of Fe(0) NPs¹⁴ and 'BuONO for a required period of time (monitored by TLC). After evaporation of DMSO, extraction of the residue with ethyl acetate and purification by column chromatography over silica gel provided the pure product.

A wide range of substituted aryl/heteroaryl amines were subjected to reaction with substituted diphenyl diselenides by this procedure to produce the corresponding products. The results are summarized in Table 2. Both electron donating and electron withdrawing groups bearing aryl amines uniformly reacted with substituted diphenyl diselenides and provided the corresponding products (**3a**, **3c**, **3d**, **3e**, **3f**, **3g**, **3h**, **3i**, **3j**, **3k**, **3l**, **3m**, **3n**, **3o**, **3p**,

 Table 2

 Fe (0) mediated C(sp2)-Se bond formation



^a*Reaction conditions*: Aryl amines (1.0 mmol), diaryl diselenides (0.5 mmol), Fe (0) NPs (1.0 equiv.) and ^{*i*}BuONO (1.1 mmol), 30 °C, 8 h, argon atmosphere. ^bIsolated yield.

3q, **3r**). Halogen containing aryl amines were also compatible for this reaction (**3c**, **3g**, **3j**) and needless to say, the presence of halogens provides scope for further functionalization.

Heteroaryl amines successfully participated in this reaction with aryl or heteroaryl diselenides and the corresponding products (**3s**, **3t**, **3u**, **3v**) were obtained.

Table 3

Synthesis of diaryl tellurides from aryl amines^{a, b}





^a*Reaction conditions*: Aryl amines (1.0 mmol), diaryl ditellurides (0.5 mmol), Fe(0) NPs (1.0 equiv.) and ^fBuONO (1.1 mmol), 80 °C, 12 h, argon atmosphere. ^bIsolated yield.

Sterically hindered 2,6-dimethyl aniline also reacts with diselenides (**3i,3t**). Several functional groups such as $-NO_2$, -COMe, $-CO_2Me$, -OMe, -CN are sustainable with the reaction conditions. The substitution of electron donating and electron withdrawing groups on the phenyl ring of aryl amines did not have any appreciable influence on the outcome of the reaction.

The same procedure was followed for the synthesis of diaryl tellurides and sulfides although the reactions were performed at 80°C as optimized. All the products are summarized in Table 3 and Table 4 respectively.

Several substituted aryl/heteroaryl amines have been employed for these reactions. A variety of functional groups such as -F, -NO₂, -COMe, -CN, -CHO, -OMe, -Cl, -I, -CF₃ on aryl amines are compatible for these reactions and the corresponding products were produced without any difficulty.

Table 4





^a Reaction conditions: Aryl amine (1.0 mmol), diaryl disulfide (0.5 mmol), Fe(0) NPs (1.0 equiv.) and ^bBuONO (1.1 mmol), 80°C, 8 h, argon atmosphere. ^b Isolated yield.

In general the reactions are clean and high yielding. The Fe nanoparticles were obtained by a reported procedure using FeCl₃ and sodium borohydride.¹⁴ The Fe(0) NPs obtained by a different procedure using tea extract¹⁵ without using any hydride reducing agent also provides the same result. The chalcogenides are obtained pure and properly characterized

by spectroscopic data. Majority of the compounds were obtained for the first time. This procedure gives diverse diaryl selenides/tellurides/sulfides in good to excellent yield. Moreover pharmaceutically potential² diheteroaryl selenides are also synthesized by this protocol.

2,4-Dinitroaniline reacts with diphenyl diselenide to produce (2,4-dinitrophenyl)(phenyl)selane (Scheme 2, **6a**) which is reported as thioredoxin reductase (TR) and glutathione reductase (GR) inhibitor.^{2f}



Scheme 2. Synthesis of (2,4-dinitrophenyl)(phenyl)selane from 2,4-dinitroaniline

To find the nature (ionic/radical) of the process we have performed a reaction of 4-fluoroacetophenone with diphenyl diselenide in the presence of Fe(0) NPs at 90°C in DMSO for 12 h. The expected product, 1-(4-(phenylselanyl)phenyl)ethanone¹⁶ was not formed indicating that Fe (0) is inactive for Se-Se bond cleavage under this condition. Thus the reaction *via* an ionic pathway involving PhSe is unlikely (Scheme 3).



Scheme 3. Investigation on the cleavage of PhSeSePh by Fe(0) NPs

To check the radical pathway we have performed the reaction of 4-methoxy aniline and diphenyl diselenide under normal reaction conditions in the presence of TEMPO (a radical quencher) and the reaction is partially arrested. Thus to have further conclusive evidence we have performed a blank reaction using only 4methoxy aniline in the absence of diselenide. 4,4'-dimethoxy-1,1'-biphenyl (confirmed by HRMS, See SI) was obtained possibly by the dimerization of the aryl radical generated from aryl diazonium moiety in presence of Fe(0) nanoparticle. We have also carried out a reaction of 2-(allyloxy)aniline with diphenyl diselenide in the presence of t-BuONO and Fe(0) under these conditions and significantly the reaction produced a cyclised product, 3-((phenylselanyl)methyl)-2,3dihydrobenzofuran exclusively (Scheme 4).



Scheme 4. Reaction between 2-(allyloxy) aniline and diphenyldiselenide

The EPR spectra of the reaction intermediate after two hours of reaction indicates the presence of a radical (Figure 1).

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Supplementary Material

Supplementary data (characterization data and ¹ H NMR, ¹³C NMR spectra of all compounds listed in Tables 2, 3, and 4, Schemes 2 and 4) associated with this article can be found, in the online version, at

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Fig. 1. EPR study of the reaction

Thus these observations suggest that the reaction follows a radical pathway. Based on these observations the following reaction mechanism is proposed. Aniline is nitrosated by [']BuONO to produce the diazonium unit (**A**) which successfully produced the corresponding aryl radical (**B**) in the presence of Fe (0). Then this aryl radical reacts with diphenyl diselenide to produce the corresponding diaryl selenide (Scheme 5).

400

500

300

Magnetic Field Strength(G)





In conclusion, we have developed an efficient and general method for the synthesis of aryl/heteroaryl sulfides, selenides and tellurides from aryl/heteroaryl amines *via* in situ diazotization using 'BuONO and subsequent reaction with diaryl dichalcogenides in the presence of Fe(0) nanoparticles by a uniform protocol. This procedure avoids the isolation and handling of hazardous diazonium salt and the reaction was performed in one pot. This reaction is compatible with a broad range of functionalities and heteroaryl amines which are not often addressed.^{7-9,12} The reaction proceeds by a radical pathway and a rationale mechanism has been outlined. The generation of aryl radical by the interaction of diazonium moiety with Fe(0) nanoparticle is not reported earlier and is of potential for further applications.

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3000

2000

1000

-300

dabsorption/dH

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Highlights

- Direct synthesis of chalcogenides from aryl/hereoary amine
- ➤ A general synthetic method for all type of sulfides, selenides and tellurides
- Acctinition Avoids isolation and handling of hazardous diazonium salt involving in situ

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