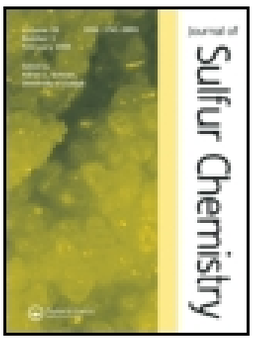

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High yielding protocol for direct conversion of thiols to sulfonyl chlorides and sulfonamides

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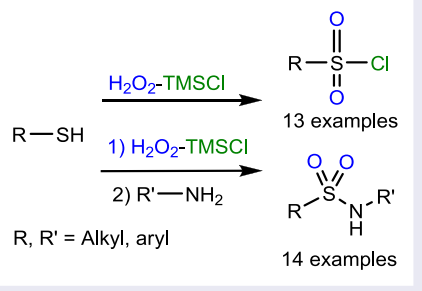
ABSTRACT

In this paper, a new method for oxidative chlorination of thiols to sulfonyl chlorides and sulfonamides using H_2O_2 in the presence of TMSCl is reported. The excellent yields, short reaction times, excellent efficiencies, low costs, and easy separation of products are the most important advantages of this method.



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Sulfonyl chlorides; sulfonamides; oxidative chlorination; chlorotrimethylsilane; hydrogen peroxide

1. Introduction

A large of group of physiologically active compounds based on various sulfonyl chlorides have been synthesized [1]. Sulfonyl chlorides are also promising compounds as reagents or intermediates in organic synthesis [2].

Sulfonamides are a class of significantly important compounds that have been widely applied as building blocks in medical chemistry [3]. These compounds are found in many remedial agents including drugs for the treatment of bacterial [4] and viral [5] putrefaction. Sulfonamides are used as an anticancer agent [6], HIV protease inhibitory activities [7] and in Alzheimer's disease [8] (Figure 1).

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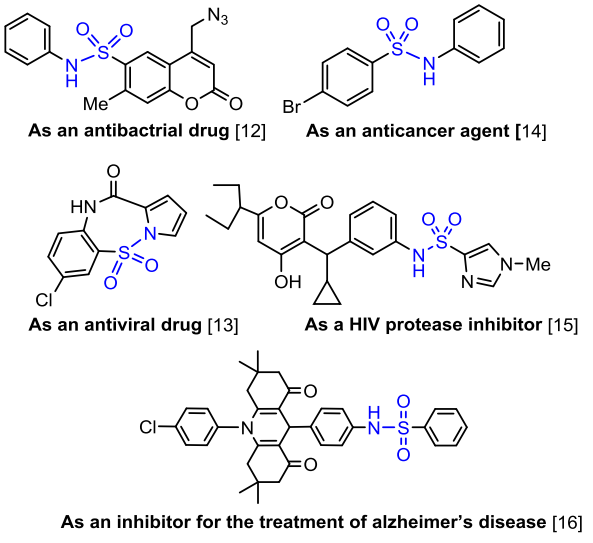
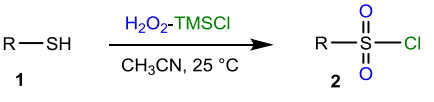


Figure 1. Examples of sulfonamide frameworks as drug candidates.

Due to significance sulfonyl chlorides, over the last several years many endeavors have been made for the synthesis of these compounds using many different methods. Traditionally, sulfonyl chlorides can be prepared in several different ways [9–20]. Of these and for years, the oxidative chlorination of sulfur compounds such as thiols, thioethers, thioacetates, and thiocarbamates with aqueous chlorine [9] has been the most typical synthetic methods used for the formation of sulfonyl chlorides. Although these methods are efficient for the synthesis of sulfonyl chlorides, most of them suffer from one or several disadvantages such as harsh and complex reaction conditions, poor yields, limitations in starting materials, long reaction time, side reactions, slow reactivity, poor functional group tolerability and tedious purification procedures.

Therefore, to overcome these problems it is highly desired to develop a novel and sustainable method for the preparation of sulfonyl chlorides. In continuation of our work on the use of TMSCl in organic transformation [21], herein, we report an efficient and rapid method for the synthesis of sulfonyl chlorides from thiols by the TMSCl-H_2O_2 reagent system (Scheme 1).

Chlorotrimethylsilane (TMSCl) is a chemical reagent with low toxicity, low cost, and high reactivity, which has attracted considerable attention. However, currently no report has been published utilizing TMSCl as promoter in the conversion of thiols to sulfonyl chlorides.



Scheme 1. Synthesis of sulfonyl chlorides from thiols.

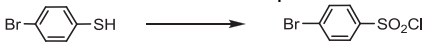
2. Results and discussion

To investigate the effects of various amounts of H_2O_2 and TMSCl in acetonitrile at room temperature, 4-bromothiophenol was selected as a sample for optimization of the reaction conditions. According to Table 1, the best yield (97%) was achieved by performing the reaction with three equivalents of H_2O_2 in the presence of one equivalent of TMSCl for 2 min. Using a lower amount of H_2O_2 resulted in lower yields, while higher amounts did not affect the reaction time and yield.

To select the best solvent, the oxidative chlorination of 4-bromothiophenol was done under the same conditions using various organic solvents such as chloroform, toluene, acetonitrile and 1,4-dioxane. In this step of work, acetonitrile gave the best results and was chosen for this purpose.

Investigation of the reactions of different thiols was performed using the above-mentioned optimized reaction conditions. As seen in Table 2, aromatic thiols carrying either electron-donating or electron-withdrawing substituents led to excellent yields of products in high purity as proved by NMR spectroscopy. Moreover, aliphatic thiols like cyclohexanethiol and 1-octanethiol led to the formation of sulfonyl chlorides in excellent

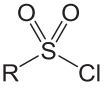
Table 1. Effect of increasing the amounts of H_2O_2 and TMSCl on the oxidative chlorination of 4-bromothiophenol.



Entry	TMSCl (mmol)	30%H_2O_2 (mmol)	Yield (%)[a]
1	0	4	0
2	0.5	4	51
3	0.7	4	63
4	0.8	4	80
5	1	2	74
6	1	3	97
7	1	4	97

Note: Reaction conditions: 4-bromothiophenol (1 mmol), 3 min, 25 °C.
[a]Isolated yields.

Table 2. Oxidative chlorination of thiol derivatives.



2a: R = C_6H_5; 96% (2 min); oil [39]
2b: R = 4-MeC_6H_4; 97% (2 min); mp = 68–69 [39]
2c: R = 4-MeOC_6H_4; 93% (4 min); mp = 39 [40]
2d: R = 4-ClC_6H_4; 97% (3 min); mp = 48–51 [39]
2e: R = 4-BrC_6H_4; 95% (3 min); mp = 70 [40]
2f: R = 4-FC_6H_4; 96% (3 min); mp = 34 [40]
2g: R = 3-MeOC_6H_4; 90% (4 min); oil [41]
2h: R = 4-$O_2N$$C_6H_4$; 90% (2 min); mp = 71–72 [42]
2i: R = 2-Naphthyl; 93% (3 min); mp = 73–75 [39]
2j: R = Benzyl; 96% (3 min); mp = 91 [43]
2k: R = 2-Benzoimidazole; 0% (10 min); mixture of products [44]
2l: R = Cyclohexyl; 90% (10 min); oil [45]
2m: R = *n*-Octyl; 92% (10 min); oil [43]

Notes: The products were characterized by comparison of their spectroscopic and physical data with authentic samples synthesized by reported procedures. Yields refer to pure isolated products.

yields (**2 l** and **2 m**). This reaction was also compatible with other functional groups such as halogens, nitro, and ether linkages (Table 2).

The heterocyclic thiol, 2-mercaptobenzimidazole was examined under the same conditions and failed to produce the desired sulfonyl chloride (**2k**). According to a reported paper [22], benzimidazole-2-sulfonyl chloride is unstable at room temperature and rapidly decomposes to 2-chlorobenzimidazole and 2-hydroxybenzimidazole as detected by comparison to commercial samples.

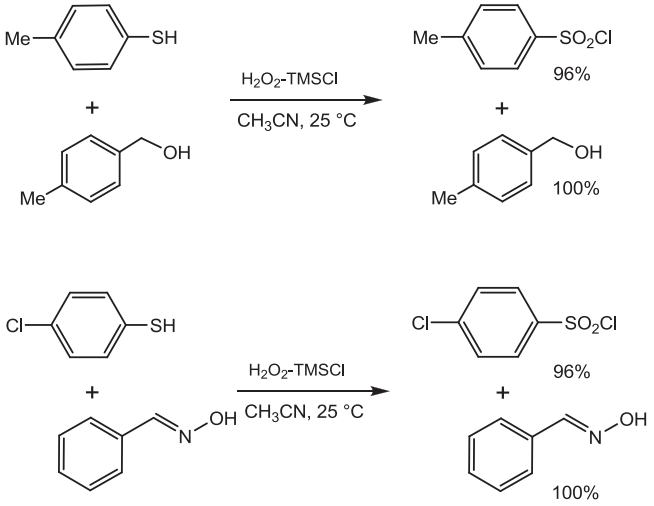
We also studied the oxidative chlorination of thiols in the presence of alcohol and oxime (Scheme 2). The findings confirm that this procedure can be employed for the oxidative chlorination of thiols in the presence of alcohol and oxime functional groups in multifunctional molecules.

These observations can be rationalized by the reaction mechanism proposed in Scheme 3. The reactions proceed, in general, *via* the intermediacy of disulfide **6** and thiosulfonate **9** which produces sulfonyl chloride **3**. Formation of **9** *via* **8** from **7** has been well substantiated and acknowledged [23].

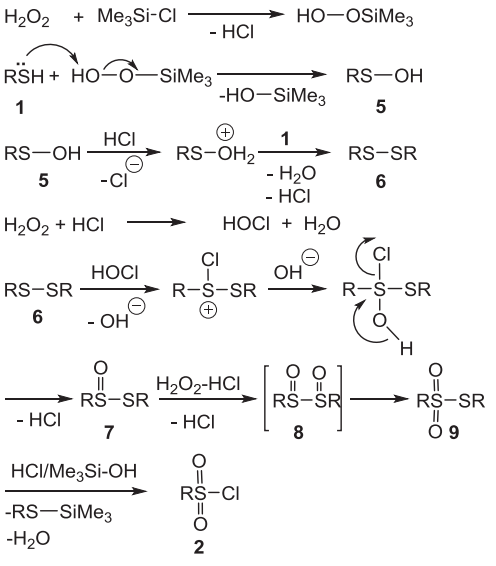
An investigation confirmed the presence of the corresponding disulfide as the main intermediate in this reaction. Thus, the reaction of thiophenol (1 mmol) with H_2O_2/TMSCl in 1:1 molar ratio for 1 min produced diphenyl disulfide in high yield (see supporting information).

Various synthetic methods have been reported for the preparation of sulfonamides [24–29], but the most traditional method is the reaction of an amine with a sulfonyl chloride in the presence of a base in an aprotic solvent [30]. Although, this procedure is sufficient, its utility is limited by the formation of undesired disulfonamides with primary amines, and to need for harsh reaction conditions for less nucleophilic amines such as anilines [31] and also the availability of sulfonyl chlorides, some of which are difficult to handle or store.

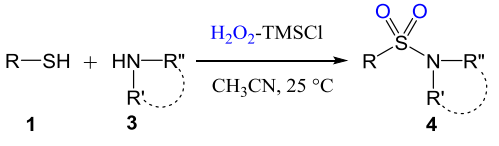
In view of the importance of sulfonamides in chemistry and biology, the synthesis of these compounds is of great interest in synthetic and medicinal chemistry.



Scheme 2. Reagents and conditions: molar ratio of substrates: H_2O_2: TMSCl (1:1:3:1), CH_3CN, 25 °C.



Scheme 3. A proposed mechanism for the preparation of sulfonyl chloride from a thiol with the H_2O_2-TMSCl reagent system.

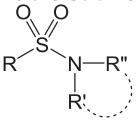


Scheme 4. Conversion of thiols to sulfonamides.

In recent work, we have shown H_2O_2 in combination with $SOCl_2$ [32], $ZrCl_4$ [33] and $POCl_3$ [34] as an efficient system for the direct conversion of thiols into sulfonamides. As an extension of this work and also in continuation of our program directed to the development of efficient reagents for use under mild conditions [35–38] herein, we report a new mild and efficient synthesis of such compounds *via* the reaction of amines (1 mmol) and thiols (1 mmol) in the presence of H_2O_2 (3 mmol), TMSCl (1 mmol) and pyridine (0.5 mL) in acetonitrile at room temperature. To the best of our knowledge, such a reagent system for the preparation of sulfonamides has not been described in the literature. The route for the preparation of sulfonamides from thiols is shown in Scheme 4.

According to Scheme 4, the reaction of structurally and electronically diverse thiols and amines were evaluated by using optimized reaction conditions. The data in Table 3 show that all reaction performed efficiently and the desired sulfonamides were formed in excellent yields and high purity (detected by NMR spectroscopy). As can be seen, aryl thiols carrying either electron-donating or electron-withdrawing substituents reacted very well. In addition, it was discovered that amines are insensitive to substitution and primary (**4a**) and secondary (**4n**) alkyl amines, as well as ammonia (4j), undergo this reaction with the same efficiency. As shown in Table 3, this method proved to be very useful even for sterically hindered amines such as diphenylamine and piperidine, the corresponding sulfonamides were obtained in 92% and 93% yields, respectively (**4e** and **4k**). Furthermore,

Table 3. Direct formation of sulfonamides from thiols.



4a: R = C_6H_5; R′ = H; R″ = C_6H_5; 99% (8 min); mp = 109–110 [46]
4b: R = 4-MeC_6H_4; R′ = H; R″ = 4-MeC_6H_4; 98% (8 min); mp = 112–114 [46]
4c: R = 4-BrC_6H_4; R′ = H; R″ = 3-$O_2NC_6H_4$; 95% (10 min); mp = 119–120 [32]
4d: R = 1-Naphthyl; R′ = H; R″ = C_6H_5; 95% (9 min); mp = 158–159 [47]
4e: R = C_6H_5; R′ = C_6H_5; R″ = C_6H_5; 92% (11 min); mp = 123–125 [48]
4f: R = C_6H_5; R′ = H; R″ = 1-Naphthyl; 95% (9 min); mp = 165–166 [49]
4g: R = 4-BrC_6H_4; R′ = H; R″ = C_6H_{11}; 97% (9 min); mp = 99 [33]
4h: R = $ClSO_2(CH_2)_3$; R′ = H; R″ = 4-MeC_6H_4; 91% (11 min); mp = oil [34]
4i: R = $C_6H_{11}NHSO_2(CH_2)_3$; R′ = H; R″ = C_6H_{11}; 94% (10 min); mp = oil [31]
4j: R = Benzyl; R′ = H; R″ = H; 95% (9 min); mp = 97–98 [50]
4k: R = 4-MeC_6H_4; R′ . . . . ..R″ = $CH_2(CH_2)_4CH_2$; 93% (11 min); mp = 92 [34]
4l: R = 4-MeC_6H_4; R′ = H; R″ = H; 96% (9 min); mp = 137–139 [51]
4m: R = 4-MeC_6H_4; R′ = H; R″ = C_6H_5NH; 93% (8 min); mp = 156–157 [52]
4n: R = C_6H_5; R′ = Ethyl; R″ = Ethyl; 97% (10 min); mp = 42 [53]

Notes: The products were characterized by comparison of their spectroscopic and physical data with authentic samples synthesized by reported procedures. Yields refer to pure isolated products.

1-naphthyl amine (**4f**) transformed into the desired sulfonamide with 95% yield. Moreover, this procedure worked efficiently in the sulfonylation of phenylhydrazine (**4 m**) to provide an excellent yield of the product without the formation of any side products. Interestingly, propan-1,3-dithiol worked well in this method and monosulfamide and disulfamide products were obtained in excellent yields with high selectivity (**4h** and **4i**).

To assess the probability of applying this procedure on a preparative scale, we performed the reaction of 4-methylthiophenol with 4-methylaniline on a 20 mmol scale. As expected, the reaction proceeded smoothly, similar to the case in a smaller scale (Table 3, entry **4b**), and the desired product was obtained in 96% isolated yield.

3. Conclusions

In summary, a new and simple oxidative chlorination method using the H_2O_2-TMSCl system is described and found to be very practical for the direct conversion of thiol compounds into the desired sulfonyl chlorides in excellent yields and short reaction times. This reagent system was also found to be an effective reagent system for the direct synthesis of sulfonamides from thiols in excellent yields. The notable advantages offered by this protocol are simplicity in operation, general applicability, mild reaction conditions, high regioselectivity, compatibility of a wide range of functionalities, which includes the ether, alcohol; and oxime moieties, very short reaction times, and the high yield of products. Further utilization of this procedure is in progress in our laboratory.

4. Experimental

4.1. Materials and physical measurements

The starting materials for this work were purchased from the Merck chemical company. Products are known compounds and were characterized by comparison of their spectral

data (NMR) and physical properties (melting point) with reported samples. The spectra were recorded on Bruker NMR spectrometer (200 and 300 MHz) in $CDCl_3$. Thin layer chromatography (TLC) was carried out on silica gel polygrams SIL G/UV 254 plates. All melting points were corrected and determined with the open capillary method using Gallen. Kamp melting point apparatus. Yield refers to the isolated pure products.

4.2. General procedure for the conversion of thiols to sulfonyl chlorides

To a mixture of appropriate thiol (2 mmol) and TMSCl (2 mmol) in CH_3CN, was added 30% H_2O_2 (6 mmol, 0.6 mL), and stirred at room temperature for the times mentioned in Table 2. On completion of the reaction followed by TLC, H_2O (10 mL) was added and the mixture was then extracted with ethyl acetate (4 × 5 mL). The extract was dried using $MgSO_4$. After filtration and removal of the solvents under reduced pressure, the pure product was obtained in excellent yield.

4.3. General procedure for the preparation of sulfonamides from thiols

To a soln of thiol compound (2 mmol) and TMSCl (2 mmol) in CH_3CN, was added H_2O_2 (30%, 6 mmol, 0.6 mL) and stirred at room temperature. The progress of the reaction was followed by TLC examination (*n*-hexane:ethyl acetate, 6:2). At the end of the reaction, appropriate amine (2 mmol) in pyridine (1 mL) was added and the stirring was continued until the starting material was disappeared (Table 3). The reaction mixture was then acidified by aqueous 2N HCl and extracted using ethyl acetate. After washing organic layer with H_2O and brine, the mixture was dried over $MgSO_4$. After filtration, the solvents were removed under vacuum to afford the desired sulfonamide in excellent yield. Recrystallization from a mixture of ethanol and water produces a more pure final product.

Disclosure statement

No potential conflict of interest was reported by the authors.

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