Synthesis of Sulfonyl Chlorides and Sulfonic Acids in SDS Micelles

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Abstract: $H_2O_2/POCl_3$ is found to be a reactive reagent system that can be used in sodium dodecyl sulfate (SDS) micellar solution in aqueous media for the direct oxidative chlorination of thiol and disulfide derivatives to give the desired sulfonyl chlorides. The oxidation of thiols and disulfides to sulfonic acids with this system is also reported. In most cases, these reactions are highly selective, simple, and clean, affording products in excellent yields and high purity.

Key words: micelles, oxidation, hydrogen peroxide, thiols, solvent systems, amphiphiles

Chemical transformations, as well as other industrial production processes, are experiencing a profound transformation to meet sustainability criteria, moving from old methods to new approaches that have been developed in accordance with green chemistry principles.[1]

Sulfonyl chlorides are precursors with extensive uses in organic synthesis.[2] Generally, sulfonyl chlorides are prepared from the corresponding sulfonic acids[3] and, although other methods are available for this transformation,[4–8] attention is moving toward newer and more selective methods for this purpose. Despite their tremendous success, however, some drawbacks still remain. For example, the use of expensive or less easily available reagents, tedious manipulations in the isolation of the pure products, side reactions, or the use of organic solvents remain problematic. In this respect, the search for processes with environmentally benign reagents and solvents that exhibit a wide range of substrate tolerance under milder reaction conditions still remains a major challenge.

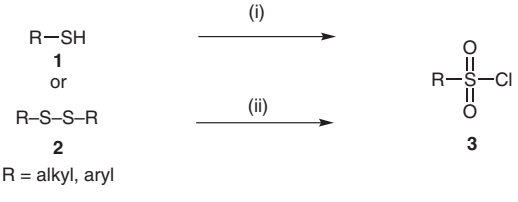
Water is the most abundant, inexpensive, and environmentally friendly solvent, thus the development of organic reactions in aqueous media is one of the practical trends in current chemistry.[9] However, organic reactions in water are often limited in scope due to poor solubility of the organic compounds. A possible new way to improve the solubility of substrates is the use of surface-active compounds that can form micelles.[10]

Surfactants, amphiphilic molecules with a hydrophilic head group and a hydrophobic tail, have been used for washing since 2500 B.C. In aqueous solutions, they self-assemble, i.e., organize spontaneously into aggregated structures, so-called micelles. This concept of structuring is not only widely found in nature and in many everyday applications, but has also recently become of interest for the controlled design of more complex structures in the nanometer size range as well.[11] On the basis of previous reports, it is likely that micelles act as nanoreactors to bring the reactants together to provide a confined reaction environment.[12]

During recent years, aqueous hydrogen peroxide has been one of the most attractive 'green oxidants' for its environmentally benign characteristics; it only produces harmless water as by-product, is safe for storage and operation, inexpensive, readily available, and has a high effective oxygen content.[13]

Phosphoryl chloride (commonly called phosphorus oxychloride) is a reactive chemical reagent. The most important use for this compound is in the manufacture of triarylphosphate esters such as triphenyl phosphate and tricresyl phosphate. These esters have been used for many years as flame retardants and plasticizers for PVC. In the semiconductor industry, $POCl_3$ is used as a safe liquid phosphorus source in diffusion processes. In the laboratory, $POCl_3$ is widely used as a dehydrating agent, for example in the conversion of amides into nitriles. Similarly, certain aryl amides can be cyclized to dihydroisoquinoline derivatives using the Bischler–Napieralski reaction.[14] However, to the best of our knowledge, there are no reports of the application of $POCl_3$ as a promoter in the conversion of thiols into sulfonyl chlorides.

As part of our ongoing efforts to develop environmentally benign processes,[15] we report for the first time a simple and environmentally friendly method for the synthesis of sulfonyl chlorides through the reactions of thiol derivatives with H_2O_2 in the presence of $POCl_3$ in aqueous micellar media, using sodium dodecyl sulfate (SDS). The route used for the synthesis of sulfonyl chlorides is shown in Scheme 1.



Scheme 1 *Reagents and conditions*: (i) H_2O_2 (3 equiv), $POCl_3$ (1 equiv), SDS (1 CMC, 5 mL), H_2O, 25 °C; (ii) H_2O_2 (2 equiv), $POCl_3$ (1 equiv), SDS (1 CMC, 5 mL), H_2O, 25 °C.

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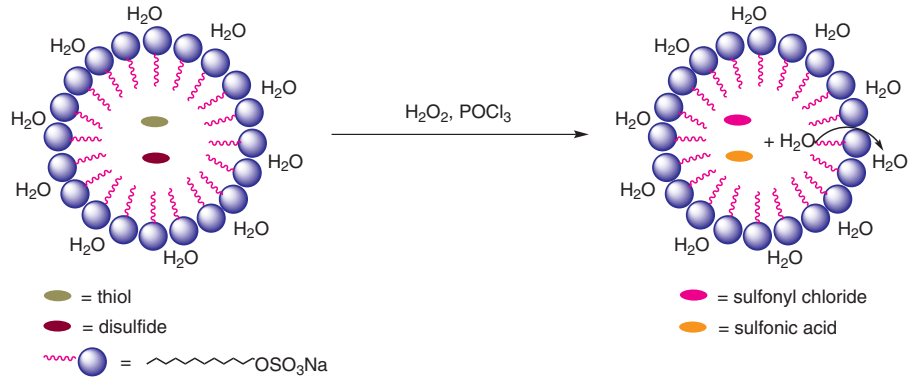


Figure 1 Micelle-promoted synthesis of sulfonyl chlorides and sulfonic acids from thiols and disulfides in water in the presence of H_2O_2 and $POCl_3$

SDS was chosen because it forms micelles in water, can solubilize organic compounds that are otherwise insoluble in water, and has been used successfully in a number of organic reactions as a catalyst.[16]

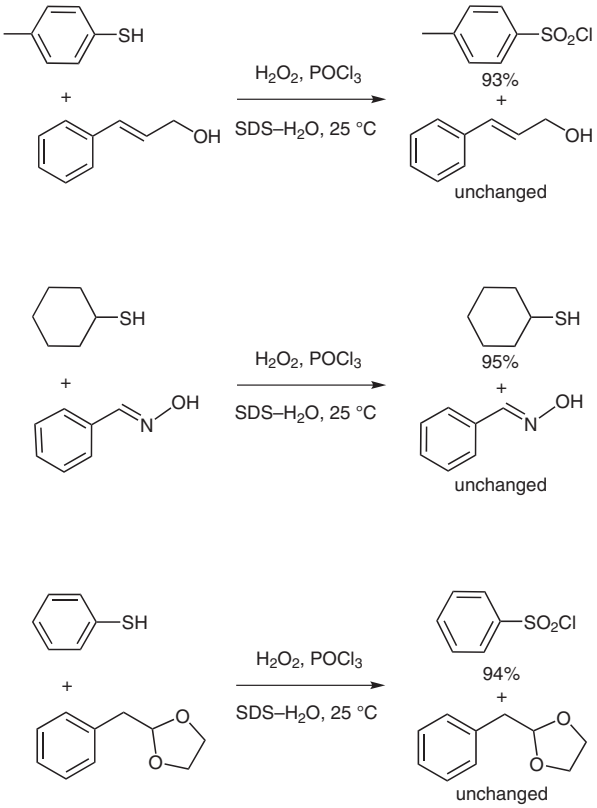
We envisioned that the SDS surfactant micelles (SDS micelle diameter about 4 nm[17]) could act as nanoreactors to induce better solubilization of thiols and disulfides and ultimately allow these compounds to interact more intimately with the H_2O_2 and $POCl_3$. The nonpolar alkyl chains remain in a nonpolar environment and the hydrocarbon chains can dissolve the organic substrate. Thus, there was no requirement to employ water-soluble substrates (Figure 1).

To establish the optimum stoichiometry, when 4-methylphenyl thiol (1 mmol) was allowed to react with 1, 2, and 3 equivalents of H_2O_2 and $POCl_3$ (1 mmol) in the presence of SDS micellar solution (1 CMC, 5 mL) at 25 °C, the yield of sulfonyl chloride obtained was 30, 68, and 95%, respectively. Higher temperatures reduced the reaction time but increased the amount of sulfonic acid contamination, thereby reducing the selectivity of the oxidative chlorination. A control experiment conducted without SDS showed that only a low yield of sulfonyl chloride was obtained under optimized reaction conditions in pure water after five hours.

To check the effect of other surfactants at their critical micelle concentrations (CMC)[18] in water, we studied the effect of different micellar media on the oxidative chlorination of 4-methyphenyl thiol. Micellar solutions of cetyl trimethylammonium bromide (CTAB) as a cationic micelle and Triton-X-100 as a neutral micelle agent were studied for this purpose. The results show that all three micellar media were effective for this reaction, and the desired sulfonyl chloride was produced in quantitative yield within 0.5–2 hours. However, the rate of the reaction was faster in SDS (1 CMC) than with CTAB and Triton-X-100 micellar solutions.

We also studied the use of 37% HCl instead of $POCl_3$ on the oxidative chlorination of 4-methyphenyl thiol under the optimized reaction conditions. Our results show that this reaction remained incomplete and only 55% of the desired product was obtained after two hours.

Having established the optimized reaction conditions, a diverse range of sulfonyl chlorides were synthesized in excellent yields. The results, summarized in Table 1, indicate that aromatic thiols with either electron-donating or electron-withdrawing substituents displayed high reactivity and generated the desired products in excellent yields. In addition to aromatic thiols, aliphatic thiols such as cyclohexanethiol, butanethiol and 1-octanethiol also afforded the corresponding sulfonyl chlorides in excellent yields (Table 1, entries 11–13). However, unfortunately, heterocyclic thiols yielded little or none of the desired product. Furan-2-methanethiol produced a mixture of products that were difficult to separate; after laborious separation, we obtained an unacceptable yield of the desired sulfonyl



Scheme 2 *Reagents and conditions*: molar ratio of substrates to H_2O_2 to $POCl_3$ (1:1:3:1), SDS (1 CMC, 5 mL), H_2O, 25 °C.

chloride, while 2-mercaptobenzimidazole (Table 1, entry 10) did not yield the desired product; this is in accordance with a previous study.[19]

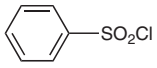
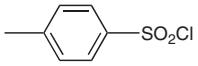
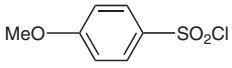
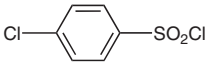
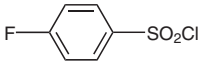
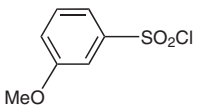
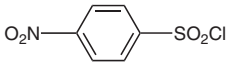
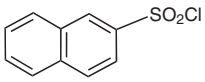
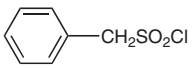
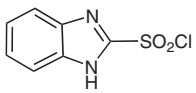
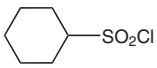
To demonstrate the validity and regioselectivity of this reagent system, we studied the oxidative chlorination of thiols in the presence of an alcohol, a carbon–carbon double bond, an oxime, and an acetal; the results are summarized in Scheme 2. These studies clearly show that this reagent system can be applied for the chemoselective oxidative chlorination of thiols in the presence of the above mentioned functional groups in multifunctional compounds.

Disulfides were also used with similar success to provide the corresponding sulfonyl chlorides (Scheme 1). To optimize the reaction conditions, the reaction of diphenyl disulfide was selected as a model with which to examine the effects of the amounts of H_2O_2 and $POCl_3$ in water in the presence of SDS (1 CMC, 5 mL) at room temperature. The best result (96% yield) was obtained by carrying out the reaction with two equivalents of H_2O_2 in the presence of one equivalent of $POCl_3$ for 30 minutes.

The reactions were repeated with a range of symmetrical disulfides; the results of these studies are collected in Table 1. All reactions resulted in the formation of the corresponding sulfonyl chlorides in excellent yields with high purity. Neither electron-donating nor electron-withdrawing groups on the aromatic ring affected the efficiency of the reaction. For example, 4-methoxybenzenesulfonyl chloride (Table 1, entry 3), and 4-nitrobenzenesulfonyl chloride (Table 1, entry 7) were obtained from the corresponding disulfides in 95 and 93% yields, respectively. The selectivity of the present method is fair-

Table 1 Oxidative Chlorination of Thiol and Disulfide Derivatives

R–S–S–R or R–SH ⟶ R–SO_2Cl

Entry	Product **3**	From thiol[a] Yield (%)[b]	Time (min)	From disulfide[a] Yield (%)[b]	Time (min)	Mp (°C)
1		95	35	96	30	oil[7a]
2		95	30	94	30	71–72[5a]
3		94	35	95	30	40[7a]
4		95	40	92	40	49–50[7a]
5		94	35	94	30	32–33[8b]
6		94	35	93	35	oil[20]
7		95	36	95	32	74–76[7a]
8		94	40	93	30	73–75[5a]
9		93	25	93	25	90–91[7a]
10		0	60	0	60	– (60[18])
11		95	40	96	30	oil[7a]
12	*n*-butyl–SO_2Cl	95	30	93	30	oil[6b]
13	*n*-octyl–SO_2Cl	94	40	95	30	oil[6b]

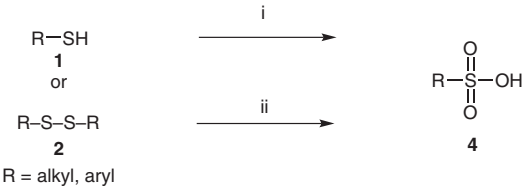
[a] The products were characterized by comparison of their spectroscopic and physical data with authentic samples synthesized by reported procedures.
[b] Isolated yield.

ly wide, as several functionalities remained unaffected under these reaction conditions.

Sulfonic acids and their derivatives are precursors with extensive uses in organic synthesis.[21] They also represent an important class of organic compounds that are used industrially for a range of applications.[22] For example, they are widely used as surfactants, dyes, animal feeds, pesticides, and pharmaceuticals.

The synthetic protocols most frequently reported in the literature for their preparation are based both on the oxidation of different types of sulfur-containing functional groups[23,24] and on the direct sulfonation of aromatic compounds.[25] The oxidation of thiols by many oxidizing agents[26] is one of the classical methods for the preparation of aliphatic and aromatic sulfonic acids.

Following our interest in the use of hydrogen peroxide in organic synthesis,[27] we have also extended the application of the method to prepare different derivatives of sulfonic acids from thiols (Scheme 3).



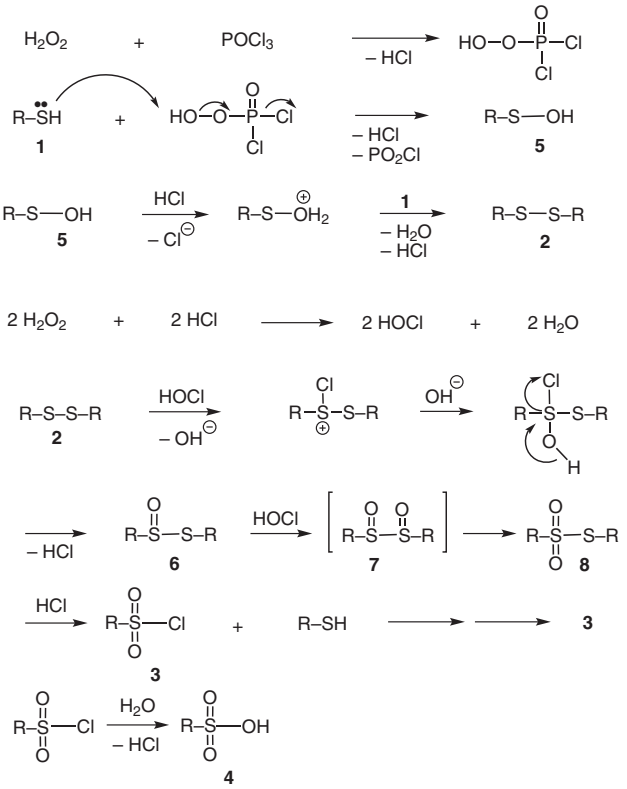
Scheme 3 *Reagents and conditions*: (i) H_2O_2 (5 equiv), $POCl_3$ (1 equiv), SDS (1 CMC, 5 mL), H_2O, 80 °C; (ii) H_2O_2 (4 equiv), $POCl_3$ (1 equiv), SDS (1 CMC, 5 mL), H_2O, 80 °C.

When an optimization of the molar ratio of H_2O_2 to $POCl_3$ was carried out, it was found that a ratio of 5:1 was sufficient for this reaction to proceed in water in the presence of SDS (1 CMC, 5 mL) at 80 °C. At room temperature, the reaction rate was slower and sulfonyl chloride was produced as the major product. At 80 °C, the reaction rate was maximized, and further increases in temperature did not result in any further enhancement. Under these conditions, the product yields could be improved to 91%; increasing the excess H_2O_2 and $POCl_3$ beyond this ratio did not lead to a further increase in conversion or yield. The formation of sulfonyl chlorides and sulfonic acids was performed at pH values of approximately pH 0.9 and 1, respectively, thus it is likely that the increase in the reaction temperature and amount of H_2O_2 are effective factors for the preparation of sulfonic acids.

Having optimized the reaction, a wide range of thiols containing aliphatic, aromatic, benzylic, and heterocyclic thiols could all be oxidized to the corresponding sulfonic acids in excellent yields (Table 2). All of the reactions occurred with complete selectivity for sulfonic acid formation, and no products such as sulfonyl chlorides were detected in the reaction mixtures. Interestingly, the protocol worked efficiently in oxidizing cysteine to afford the corresponding cysteic acid (Table 2, entry 11). The selectivity of the present method is fairly wide, as several functionalities remain unaffected under these reaction conditions.

In a continuation of this work, we also investigated the reaction of a wide variety of structurally diverse disulfides with this system (Scheme 3). When disulfides were reacted under similar reaction conditions, by using four equivalents of 30% H_2O_2, sulfonic acid derivatives were obtained as the sole oxidation products. Moreover, as can be seen from the data presented in Table 2, the chemoselectivity of this process was high and similar to that of the synthesis of sulfonyl chlorides. Therefore, the present protocol can tolerate variations in thiols and disulfides.

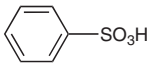
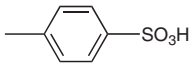
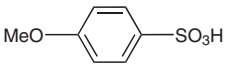
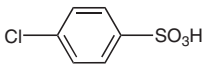
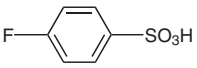
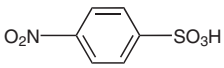
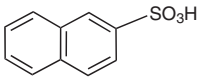
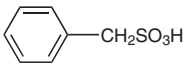
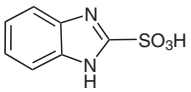
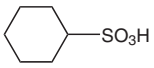
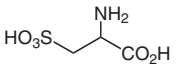
In accordance with the mechanism delineated in our recent paper[19] it may be proposed that the nucleophilic attack of H_2O_2 on $POCl_3$ increases the electrophilicity of the oxygen atoms of the H_2O_2. Nucleophilic attack of the thiol on the activated oxygen atom results in formation of sulfenic acid (**5**), which produces the corresponding symmetric disulfide (**2**) as shown in Scheme 4. Disulfide formation was observed when only one equivalent of H_2O_2 was used. Under these conditions, the successive oxidation of both sulfur atoms of the disulfide molecule by hypochlorous acid produces the intermediate (**7**), which undergoes rapid isomerization to the thiosulfonate (**8**) and can easily furnish sulfonyl chloride (**3**). The sulfonyl chloride (**3**) now has to be converted into the sulfonic acid (**4**). Conversion of **7** into **8** has been well-documented and recognized.[28] The mechanism for this oxidation is depicted in Scheme 4.



Scheme 4 Proposed mechanism for the oxidation of thiols to sulfonic acids with H_2O_2 and $POCl_3$

Table 2 Oxidation of Thiol and Disulfide Derivatives

R–S–S–R or R–SH ⟶ R–SO_3H

Entry	Product **4**	From thiol[a]		From disulfide[a]		Ref.
		Yield (%)[b]	Time (min)	Yield (%)[b]	Time (min)	
1		93	60	94	55	24
2		95	70	94	60	24
3		94	60	95	50	25
4		94	65	92	60	25
5		94	65	92	60	29
6		91	70	93	65	30a
7		94	90	94	70	30a
8		92	65	93	60	30b
9		92	80	91	70	30c
10		92	70	92	65	26
11		94	30	92	30	30d
12	*n*-butyl—SO_3H	95	50	96	50	24
13	*n*-octyl—SO_3H	93	60	95	60	31

[a] The products were characterized by comparison of their spectroscopic data with authentic samples synthesized by reported procedures.
[b] Isolated yields.

In conclusion, a selective oxidative protocol using the $H_2O_2/POCl_3$ reagent system has been found to be very useful for the direct conversion of thiols and disulfides into their corresponding sulfonyl chlorides in excellent yields. This reagent system also represents a practical and convenient synthetic way to oxidize a variety of thiols and disulfides to their respective sulfonic acid derivatives. The simplicity and convenience of this environmentally safe and economical oxidation procedure are attractive, and the fact that the reaction proceeds in excellent yields, and, in particular, can be used in aqueous media, holds promise for further uses. The high reactivity combined with the fact that the reagents are inexpensive and easily available, and that no side products are generated, makes this approach a suitable practical alternative.

Sodium dodecyl sulfate as well as phosphoryl chloride, hydrogen peroxide, and thiol derivatives as substrates are commercial products (Merck) and were used without further purification. Disulfides were prepared according to our previously reported procedure.[32]

Melting points were determined in a capillary tube and are not corrected. ^1H and ^{13}C NMR spectra were recorded with a Bruker-200 NMR spectrometer using TMS as internal standard.

Synthesis of Sulfonyl Chlorides; General Procedure

To a micellar solution of SDS (5 mL, 1 CMC = 8.1×10^{-3} M in H_2O) in a glass reactor, maintained at 25 °C with a thermostatic bath, was added a mixture of thiol (1 mmol) and $POCl_3$ (1 mmol, 0.153 g). Then 30% H_2O_2 (3 mmol, 0.3 mL) was added and the resulting mixture was stirred for the indicated reaction time (Table 1). After completion of the reaction (indicated by TLC), water was removed under vacuum on a rotary evaporator. The reaction mixture was dissolved in EtOH (10 mL) and the surfactant was precipitated by the slow addition of KCl. The resulting precipitate of dodecyl

sulfate was filtered off and the organic phase was concentrated under reduced pressure to give the pure product.

An identical procedure was employed using 30% H_2O_2 (2 mmol, 0.2 mL) and $POCl_3$ (1 mmol, 0.153 g) in the presence SDS (1 CMC, 5 mL) for the oxidative chlorination of disulfides (Table 1). The spectral and physical properties of known products were compared to those reported in the literature. In every case excellent agreement was obtained.

Synthesis of Sulfonic Acids; General Procedure

To a micellar solution of SDS (1 CMC, 5 mL) in H_2O, thiol (1 mmol) and 30% H_2O_2 (5 mmol, 0.5 mL), were added. $POCl_3$ (1 mmol, 0.153 g) was added and the mixture was stirred at 80 °C until the starting material was consumed (see Table 2). The progress of the reaction was monitored by TLC. At the end of the reaction, excess H_2O_2 was deactivated by the addition of saturated aqueous $Na_2S_2O_3$. Subsequently, KCl was added to the reaction mixture, and the resulting precipitate of dodecyl sulfate was filtered off. After removal of water under vacuum on a rotary evaporator, the organic material was extracted with ethanol. The solvent was removed under reduced pressure to give spectroscopically pure product. When necessary, the crude product was recrystallized (EtOAc–*n*-hexane). A similar procedure was applied using 30% H_2O_2 (4 mmol, 0.4 mL) and $POCl_3$ (1 mmol, 0.153 g) in the presence of SDS (1 CMC, 5 mL) for the synthesis of a range of sulfonic acids from the corresponding disulfide derivatives (Table 2).

All the products are known compounds and were easily identified by comparison of their spectroscopic data with those reported. The ^1H and ^{13}C NMR spectra for the sulfonic acids and some sulfonyl chlorides are available as Supporting Information. Accurate melting points were often difficult to determine because of the hygroscopic nature of sulfonic acids.

Supporting Information for this article is available online at http://www.thieme-connect.com/ejournals/toc/synthesis.

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