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Original article

# An efficient and convenient method for the preparation of disulfides from thiols using oxygen as oxidant catalyzed by *tert*-butyl nitrite

Q1 Shan-Li Yi, Mei-Chao Li, Xin-Quan Hu, Wei-Min Mo, Zhen-Lu Shen *

*College of Chemical Engineering, Zhejiang University of Technology, Hangzhou 310014, China*

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ABSTRACT

An efficient and convenient *tert*-butyl nitrite-catalyzed selective aerobic oxidation of thiols has been developed. Under the optimal reaction conditions, a number of thiol derivatives including aromatic thiols, heteroaromatic thiols and aliphatic thiols can be converted into their corresponding disulfides in good to excellent yields.

## 1. Introduction

The development of general and mild protocol for disulfides synthesis has received significant attention because they are indispensable in many important synthetic chemistry [1–3], biochemistry [4] and industrial applications [5]. Various protocols have been developed for the preparation of disulfides. They can be classified as two major strategies. Firstly, a number of different methods have been devised with various sulfur-transfer agents, such as arenesulfonyl chlorides [6], $CS_2$ [7], 1,3-thiazolidinedione [8], potassium 5-methyl-1,3,4-oxadiazole-2-thiolate [9], thiourea [10], S [11], thioacetamide [12] and bunte salts [13]. While, oxidative coupling of thiols with stoichiometric oxidation or catalytic oxidation has become a routine and elegant protocol for the synthesis of disulfides in recent years, primarily because a large number of thiols are easily synthesized or commercially available. Various reagents, such as *N*-phenyltriazolinedione [14], $Fe(NO_3)_3 \cdot 9H_2O/Fe(HSO_4)_3$ [15], 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) [16], tributylammonium halochromates/silica gel [17], Burgess reagent [18], ceric ammonium nitrate (CAN) [19], sulfuryl chloride [20], cetyltrimethylammonium dichromate [21], bromate [22] and $N_2O_4$/PVP [23] have been used as stoichiometric oxidants. Early studies in this field have one or more disadvantages, such as long reaction time, difficult work-up, use of toxic or costly reagents, low yield of product due to over oxidation, etc. Therefore, it is still necessary to develop mild, efficient and environmentally friendly methods to synthesize disulfides from thiols.

In this way, many chemists pay much attention to discover eco-friendly catalytic oxidation in recent years. With the development of green and sustainable chemistry, photocatalytic oxidation of thiols to disulfides appears as an interesting option and some publications have been introduced in this field [24]. Furthermore, some elegant routes with the use of $H_2O_2$ as the green oxidant have been reported [25]. Molecular oxygen is also well known to be an economical, non-toxic, and easy-handle oxidant for the oxidation of thiols into disulfides [26]. These aerobic oxidation of thiol are performed in the presence of Co-salophen [26a], Fe(BTC) [26b], $Au/CeO_2$ [26c], iron(III)–ethylenediaminetetraacetic acid [26d], [hmim]Br [26e], CoPcS@ASMNP [26f], amino acid [26g], $Ag_2O$ nanoparticles/silica [26h], selenium ionic liquid [26i], $Na_2CO_3$/BMIM-$BF_4$[26j], $N_2O_4$/charcoal [26k] and $Fe(CF_3CO_2)_3$ [26l]. Among them, the protocol of $N_2O_4$/charcoal has attracted our attention though it is difficult to impregnate liquid $N_2O_4$ on charcoal. From the practical viewpoint, it is essential to investigate the introduction and application of new member of catalysts in the synthesis of disulfides from thiols. For example, alkyl nitrites, which are valuable reagents in organic synthesis [27], came to our sight. Previously, our group utilized *tert*-butyl nitrite (TBN) as the co-catalyst in the TEMPO or DDQ-catalyzed aerobic oxidation [28]. Herein we report that the TBN/$O_2$ system can proceed well in the disulfides synthesis from thiols. To the best of our knowledge, this work is the first example of conversion of thiols

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* Corresponding author.
*E-mail address:* zhenlushen@zjut.edu.cn (Z.-L. Shen).

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to disulfides catalyzed by TBN with molecular oxygen as the oxidant.

## 2. Experimental

All the chemicals were purchased commercially and used without any further treatment, unless otherwise stated. Benzenethiol (**1a**) was synthesized according to literature [29]. GC analyses were conducted on an Agilent GC6890N system with a flame ionization detector (FID) and an FFAP or OV-17 capillary column. Conversions and selectivities were determined by area normalization. $^1$H NMR and $^{13}$C NMR spectra were carried out on a Bruker Avance III (500 MHz) spectrometer. $CDCl_3$ and DMSO-$d_6$ were used as the solvents with tetramethylsilane (TMS) as the internal standard. GC-MS was performed on Finnigan Trace GC Ultra-Finnigan Trace DSQ instrument. Low resolution mass spectra were recorded in the ESI mode on an Agilent 6210 LC/TOF mass spectrometer. Melting points were measured using Buchi melting pointing M-565.

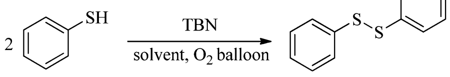
Typical procedure for disulfides (**2a**): A sealed tube (90 mL) equipped with a magnetic stirring bar and an $O_2$ balloon was charged with dichloroethane (DCE, 20 mL), thiophenol (**1a**, 4 mmol, 0.44 g) and TBN (0.16 mmol, 4 mol%, 19.2 μL). Then the tube was placed in an oil bath, which was preheated to 50 °C. The mixture was stirred for 1 h until starting material was completely consumed as monitored by GC and TLC. After removing the solvent, the residue was purified by column chromatography on silica gel to give the desired diphenyl disulfide (**2a**, 90%, 0.394 g) as a white solid.

## 3. Results and discussion

We started to optimize the process with thiophenol (**1a**) as the model substrate (Table 1). In a first set of experiments (entries 1–5), the influence of different solvents was studied. Among the tested solvents, **1a** could be completely oxidized into diphenyl disulfide (**2a**) in excellent selectivity with 5 mol% TBN under atmospheric pressure of $O_2$ within 1 h in chlorobenzene and dichloroethane (entries 1 and 2). When acetonitrile was used as the solvent, the selectivity to **2a** was decreased to 93% (entry 3). In other screened solvents, such as ethanol and *n*-hexane, the reaction was not successful (entries 4 and 5). The low conversion of **1a** in *n*-hexane might be due to the poor solubility of **1a** in *n*-hexane. As a result, dichloroethane (DCE) was chosen as the reaction solvent. Afterward, the effect of temperature was investigated. Results indicated that decreasing temperature from 50 °C to room temperature, the conversion rate of **1a** dropped sharply (entry 7). Perhaps TBN could not be decomposed completely at room temperature, and only partial NO was released which was the active specie in this catalytic oxidation reaction. Later on, the loads of TBN was attempted to be reduced. When the load of TBN was reduced from 5 mol% to 4 mol%, **1a** could also be fully converted to **2a** in 98% selectivity (entry 8). However, decreasing the load of TBN to 3 mol% or 2 mol%, conversion of **1a** was 79% and 58%, respectively (entries 9–10). Just as we have expected, the reaction did not occur in the absence of TBN (entry 11). In addition, under nitrogen atmosphere only 10% conversion of **1a** was obtained (entry 12). On the basis of these experimental data, we concluded that 4 mol% of TBN in DCE at 50 °C was suitable for oxidation of thiols to disulfides under atmospheric pressure of $O_2$.

Using the optimized reaction conditions, the present method could be used to convert a wild range of commercially available thiols into their corresponding disulfides. As shown in Table 2, all

**Table 1**
Oxidative coupling of **1a** into **2a**.[a]



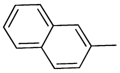
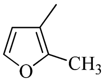
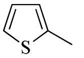
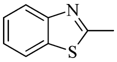
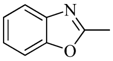
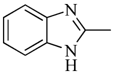
**1a** **2a**

| Entry | Solvent | TBN (mol%) | *T* (°C) [b] | Conv. (%)[c] | Select. (%)[c] |
|---|---|---|---|---|---|
| 1 | PhCl | 5 | 50 | 100 | 98 |
| 2 | DCE | 5 | 50 | 100 | 98 |
| 3 | $CH_3CN$ | 5 | 50 | 100 | 93 |
| 4 | Ethanol | 5 | 50 | 66 | 97 |
| 5 | Hexane | 5 | 50 | 59 | 99 |
| 6 | DCE | 5 | 40 | 80 | 99 |
| 7 | DCE | 5 | r.t. | 52 | 98 |
| 8 | DCE | 4 | 50 | 100 | 98 |
| 9 | DCE | 3 | 50 | 79 | 99 |
| 10 | DCE | 2 | 50 | 58 | 98 |
| 11 | DCE | 0 | 50 | – | – |
| 12[d] | DCE | 5 | 50 | 10 | 98 |

[a] Reaction conditions: **1a** (1 mmol), solvent (5 mL), $O_2$ balloon, reaction time (1 h).
[b] Oil bath temperature.
[c] Determined by GC with area normalization method.
[d] Under nitrogen atmosphere.

**Table 2**
Oxidative coupling of various thiols to disulfides catalyzed by TBN.[a]

$$\underset{\mathbf{1}}{2\,R{-}SH} \xrightarrow[O_2\ \text{balloon, DCE}]{\text{TBN (4 mol\%)}} \underset{\mathbf{2}}{R{-}S{-}S{-}R}$$

| Entry | R | Product | Time (h) | Yield (%)[b] |
|---|---|---|---|---|
| 1 | $C_6H_5$- | **2a** | 1 | 90 |
| 2 | *p*-$ClC_6H_4$- | **2b** | 2.5 | 93 |
| 3 | *m*-$ClC_6H_4$- | **2c** | 2 | 93 |
| 4 | *o*-$ClC_6H_4$- | **2d** | 2 | 96 |
| 5 | *p*-$BrC_6H_4$- | **2e** | 2 | 92 |
| 6 | *p*-$FC_6H_4$- | **2f** | 2 | 91 |
| 7 | *p*-$OCH_3C_6H_4$- | **2g** | 1.5 | 97 |
| 8 | *m*-$OCH_3C_6H_4$- | **2h** | 2 | 93 |
| 9 | *o*-$OCH_3C_6H_4$- | **2i** | 2 | 86 |
| 10 | *p*-$(CH_3)_2CHC_6H_4$– | **2j** | 2 | 84 |
| 11 | *m*-$CH_3C_6H_4$– | **2k** | 2 | 95 |
| 12 |  | **2l** | 2 | 92 |
| 13[c,d] | *o*-$COOHC_6H_4$- | **2m** | 3 | 86 |
| 14[d] |  | **2n** | 4 | 93 |
| 15[d] |  | **2o** | 4 | 81 |
| 16 |  | **2p** | 3 | 95 |
| 17[d] |  | **2q** | 3 | 72 |
| 18[e,f] |  | **2r** | 18 | 88 |
| 19[d] | $C_6H_4CH_2$– | **2s** | 2 | 76 |
| 20[e] | $C_{12}H_{25}$– | **2t** | 6 | 71 |

[a] Reaction conditions: Thiols (4 mmol), TBN (4 mol%), DCE (20 mL), 50 °C, $O_2$ (balloon).
[b] Isolated yield.
[c] Solvent: $CH_3CN$.
[d] TBN (10 mol%).
[e] TBN (30 mol%).
[f] Solvent: $CH_3OH$.

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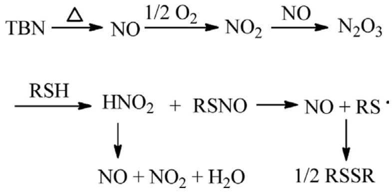

**Scheme 1.** Plausible mechanism of TBN-catalyzed aerobic oxidation of thiols to disulfides.

thiol substrates could be smoothly converted to corresponding disulfides with excellent yields without any evidence for the formation of sulfonic acids under mild conditions in short time. All aromatic thiols were more active and provided better product yields than those of heteroaromatic thiols and aliphatic thiols. The steric effects and electronic effects in aromatic thiols did not play important roles in the isolated yield of the desired products (entries 2–12). In addition, oxidative coupling reaction using thiosalicylic acid (**1m**) as the substrate successfully took place under present catalytic conditions. What is more, heteroaromatic thiols (**1n**–**1r**) also could be successfully converted into desired products (**2n**–**2r**) (entries 13–18), although higher loading of TBN was needed (entries 13–15 and 17). For 2-mercaptobenzimidazole (**1r**), which was difficult to dissolve in most of solvents, the amount of TBN needed to be increased to 30 mol% to complete the reaction (entry 18). Finally, aliphatic thiols also could produce the target products in moderate yields (entries 19 and 20).

According to the literature [30], a plausible reaction mechanism for TBN-catalyzed aerobic oxidation of thiols to disulfides has been proposed (Scheme 1). TBN releases NO under 50 °C, and then NO is easily oxidized by oxygen to form $NO_2$. $NO_2$ reacts with NO to form $N_2O_3$, which reacts with RSH to form unstable RSNO and $HNO_2$. RSSR get from the hemolytic cleavage of the sulfur-nitrogen bond of the RSNO and then coupling of the resultant thiyl radicals.

## 4. Conclusion

In conclusion, we have successfully developed a mild and convenient catalytic approach for the oxidative coupling of thiols to the corresponding disulfides. The significant features of our protocol are metal-free, atom efficiency, using of molecular oxygen as the terminal green oxidant. Furthermore, the isolation of the products is remarkably easy.

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