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# Distinct Catalytic Effect of Micellar Solution of Sodium Dodecyl Sulfate (SDS) for One-Pot Conversion of Alkyl Halides to Disulfides via an Odourless Process Using Thiourea and $MnO_2$

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A novel one-pot odourless synthesis of symmetrical disulfides from their corresponding halides in aqueous media using thiourea and $MnO_2$ in the presence of $NaHCO_3$ or $Na_2CO_3$ catalyzed by micellar solution of sodium dodecyl sulfate (SDS) is described. By this method, primary, allylic and benzylic halides were converted into their corresponding disulfides in high yields.

Sulfur–sulfur bonds are found in many biologically active compounds. For example, S–S bonds between cysteine residues in biologically active peptides and peptide mimetics have a crucial effect on their structure and therefore in their biological activity.[1–4]

Moreover, new approaches toward disulfides formations are interesting, because of their application in the preparation of a variety of self-assembled monolayers from organosulfur compounds,[5] monomolecular layers on certain metal surfaces,[6–9] and as donor–acceptor molecules in optical data processing and communication.[10] Disulfides are also important materials in the preparation of other organic compounds. Some examples are insertion of alkynes into sulfur–sulfur bonds of disulfides, electrophilic addition of disulfides to alkenes, hydroxysulfenylation of alkenes with disulfides,[11] and addition of disulfides to allenes.[12] In addition, utilizing disulfides as sulfenylating agents in the synthesis of 3-(arylthio)indoles,[13,14] which have interesting biological effects, in thia-Michael addition reactions,[15–18] and in the ring opening of epoxides are the other examples.[19]

The most familiar method for the generation of S–S bonds is the direct oxidation of thiols to their corresponding disulfides using different oxidants.[20] Along this line, we have also reported protocols for disulfide formation via oxidative coupling of thiols.[21–24] The oxidative protocols may encounter problems such as a) over-oxidation of SH moiety, b) usually the desired thiols are not commercially available, and c) lower molecular weight thiols are toxic having such a strong foul smell that their handling is not so easy and is extremely unpleasant, especially for large-scale operations.

Disulfides can also be prepared via alternative methods, within which the conversion of alkyl halides into disulfides is regarded as more suitable.[25] To the best of our knowledge, there are only a few reports on this topic in the literature. For example, conversion of alkyl halides to disulfides using alkali metal disulfides,[26–30] and sulfurated borohydride exchange resin have been reported.[31] Disulfides formation by alkaline hydrolysis of *S*-alkylisothiouronium salts by oxygen is also reported in the literature.[32] Also, low to moderate yields of disulfides have been obtained by a time consuming oxidative method of *S*-alkylisothiouronium salts using basic hydrogen peroxide.[33] Therefore, new investigations for the development of disulfide synthetic methods from alkyl halides are still of interests and a necessity. On the other hand, organic synthesis developing in water has attracted a great deal of attention at the present time because of its low cost, safety, and environmentally benign nature. Conversely, the poor solubility of hydrophobic organo-reactants in water limits its general application as a media in organic reactions. The use of surface-active reagents improves the solubility and reactivity of organic compounds in water via micellar solution formation. In this regard, we have developed and reported some organic reactions in micellar solutions in recent years.[34–37] Very recently we have reported one-pot and odourless procedures for C–S bond formation via thia-Michael addition reaction using alkyl halides, thiourea, and electron-deficient olefins in SDS micellar solution[38] and one-pot odourless thia-etherification of aryl halides using thiourea and different alkyl bromides catalyzed by copper(I) iodide in water and poly(ethylene glycol).

Herein, we report a novel procedure for a one-pot, odourless, and efficient preparation of disulfides catalyzed by SDS micellar solution from alkyl halides, thiourea, and $MnO_2$ in the presence of a mild base.

## Results and Discussion

Reaction of organic halides with thiourea followed by basic hydrolysis is a well-known process for the preparation of thiols. On the other hand, one of the generally accepted routes for the preparation of symmetric disulfides is oxidation of thiols. In this article, we have introduced a one-pot odourless method for the preparation of disulfides using alkyl halides and thiourea in the presence of a base using an oxidizing agent in water (Scheme 1). First, the reaction of *n*-octyl bromide (4 mmol), thiourea (6 mmol), $MnO_2$ (4 mmol), and $NaHCO_3$ (6 mmol) in water at room temperature and at 80 °C was studied (Table 1).

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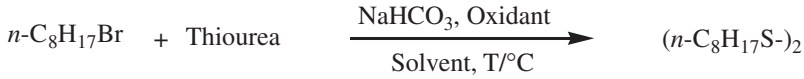
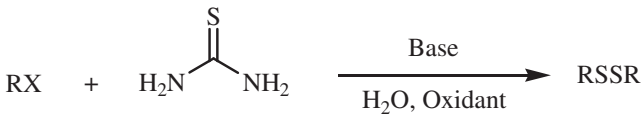
**Table 1.** The Effect of SDS, Temperature, and Oxidizing Agent on the Preparation of *n*-Octyl Disulfide from *n*-Octyl Bromide and Thiourea in Water in the Presence of $NaHCO_3$[a)]




| Entry | Solvent | Oxidant | $T$/°C | Time/h | Isolated yield/% |
|---|---|---|---|---|---|
| 1 | Water | $MnO_2$ | rt | 24 | 0 |
| 2 | Water | $MnO_2$ | 80 | 24 | 0 |
| 3 | SDS micellar solution | $MnO_2$ | rt | 24 | 0 |
| 4 | SDS micellar solution | $MnO_2$ | 80 | 8 | 85 |
| 5 | SDS micellar solution | $BaMnO_4$ | rt | 24 | 0 |
| 6 | SDS micellar solution | $BaMnO_4$ | 80 | 8 | 83 |
| 7 | SDS micellar solution | $KMnO_4$ | rt | 24 | 0 |
| 8 | SDS micellar solution | $KMnO_4$ | 80 | 12 | 65 |

a) Reaction conditions: *n*-octyl bromide (4 mmol), thiourea (6 mmol), $NaHCO_3$ (6 mmol), oxidizing agent (4 mmol), $H_2O$ (1.2 mL for 80 °C and 2 mL for room temperature), and SDS micellar solution (1.2 mL, 0.2 M for 80 °C and 2 mL, 0.2 M for room temperature).




**Scheme 1.** Generation of disulfides from alkyl halides using thiourea, a base, and an oxidizing agent.

GC analysis of the reaction mixture after 24 h, revealed the presence of an almost unreacted *n*-octyl bromide using *n*-octane as an internal standard.

Regarding our past experience,[34–37] we decided to study the similar reaction in sodium dodecyl sulfate (SDS) micellar solution at room temperature and at 80 °C. The reaction at room temperature was failure. However, the reaction at 80 °C proceeded to completion within 8 h and the desired disulfide was isolated in 85% yield. In addition, the effect of different oxidants such as $KMnO_4$, $BaMnO_4$,[39,40] $H_2O_2$, $Na_2S_2O_8$, oxone, $KIO_4$, NaClO, $I_2$, and $Br_2$ upon similar reaction in SDS micellar solution was studied. We observed that $BaMnO_4$ and $MnO_2$ were both suitable oxidants for this reaction. Moreover, the reaction in the presence of $KMnO_4$ was not a clean reaction and proceeded with a lower yield. However, in the presence of the other oxidants, the reaction did not progress at all and *n*-octyl bromide remained unreacted in the reaction mixture after a long period of time. We believe that thiourea is completely destroyed in the presence of these oxidants. Consequently, the generation of *S*-alkylisothiouronium salt which is the precursor for the formation of thiol moiety is disturbed.

With these results in hand, we selected $MnO_2$ as the oxidizing agent for the preparation of disulfides in SDS micellar solution at 80 °C (Table 1, Entry 4) and the general applicability of the method for the preparation of various symmetric disulfides from the corresponding alkyl halides was studied. The results of this study are tabulated in Table 2.

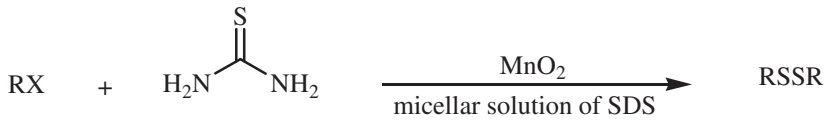
Conclusively, as is evident from the results tabulated in Table 2, this method is easily applicable for the odourless one-pot preparation of disulfides from their corresponding primary aliphatic and benzylic halides in good to excellent yields at 80 °C (Table 2, Entries 1–11). By this protocol the reaction of cyclohexyl bromide and *tert*-butyl bromide completely failed and the unreacted halides were recovered from the reaction mixture after 24 h.

In comparison with aliphatic halides, benzylic and allylic halides were successfully converted to their corresponding disulfides at room temperature using thiourea, $MnO_2$, and $Na_2CO_3$ instead of $NaHCO_3$ in SDS micellar solution. For optimization of the reaction conditions, we studied the conversion of allyl chloride to its corresponding disulfide using thiourea, $MnO_2$, and $Na_2CO_3$ in SDS micellar media at room temperature. We found that the optimized molar ratio of allyl chloride/thiourea/$MnO_2$/$Na_2CO_3$ was 4 mmol/6 mmol/4 mmol/6 mmol in SDS micellar solution (2 mL, 0.2 M). The reaction proceeded well at room temperature and the desired disulfide was isolated in 82% yield after 15 h. The same reaction in water and in the absence of SDS produced only 30% of the desired product after 24 h. This obviously shows the significant catalytic role of SDS in this reaction. Then the above optimized conditions were applied to the preparation of benzylic and allylic disulfides from their corresponding halides. The results of this study are shown in Table 2 (Entries 8–15). As the results show, this method is general and applicable for one-pot conversion of benzylic and allylic halides to their corresponding disulfides in high yields.

A general pathway for the production of symmetric disulfides from halides and thiourea in the presence of an oxidant and a base is postulated as presented in Scheme 2.

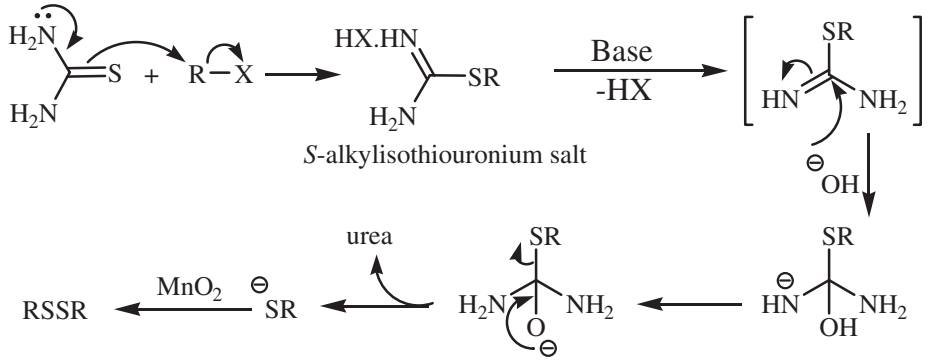
The role of SDS micellar solution in these reactions may be explained as follows. The poor solubility of organic halides is the main limitation for the formation of their corresponding *S*-alkylisothiouronium salts in water (Scheme 2). Sodium dodecyl sulfate with polar and non-polar heads orient themselves into micellar droplets with a hydrophobic solvent-like interior and a polar hydrophilic outer surface. Hydrophobic organic halides and thiourea molecules are localized inside the hydrophobic interior core of the micelle where efficient collisions between alkyl halides and thiourea molecules can occur easier to produce the corresponding *S*-alkylisothiouronium molecules. Hydrolysis of *S*-alkylisothiouronium salts producing the thiolate anion moieties which are amphiphilic molecules containing both non-polar hydrocarbon tail and polar head group. The non-polar tail dissolves in the hydrophobic core of

**Table 2.** One-Pot Conversion of Alkyl Halides to Their Corresponding Symmetric Disulfides in the Presence of Thiourea and $MnO_2$ Catalyzed by SDS Micellar Solution




| Entry | R–X | Product | Time/h | Isolated yield/% |
|---|---|---|---|---|
| 1 | $n$-$C_{10}H_{21}I$ | ($n$-$C_{10}H_{21}S$–)$_2$ (**1**) | 4 | 90[a] |
| 2 | $n$-$C_8H_{17}I$ | ($n$-$C_8H_{17}S$–)$_2$ (**2**) | 3.5 | 87[a] |
| 3 | $n$-$C_8H_{17}Br$ | ($n$-$C_8H_{17}S$–)$_2$ (**2**) | 8 | 85[a] |
| 4 | $n$-$C_4H_9I$ | ($n$-$C_4H_9S$–)$_2$ (**3**) | 2 | 89[a] |
| 5 | $n$-$C_4H_9Br$ | ($n$-$C_4H_9S$–)$_2$ (**3**) | 5 | 87[a] |
| 6 | $n$-$C_3H_7I$ | ($n$-$C_3H_7S$–)$_2$ (**4**) | 2 | 85[a] |
| 7 | $C_2H_5Br$ | ($C_2H_5S$–)$_2$ (**5**) | 3 | 80[a,c] |
| 8 | $PhCH_2Br$ | ($PhCH_2S$–)$_2$ (**6**) | 0.5[a] (4)[b] | 87 |
| 9 | $PhCH_2Cl$ | ($PhCH_2S$–)$_2$ (**6**) | 0.5[a] (10)[b] | 85 |
| 10 | 4-$CH_3C_6H_4CH_2Cl$ | (4-$CH_3C_6H_4CH_2S$–)$_2$ (**7**) | 0.5[a] (8)[b] | 87 |
| 11 | 4-$BrC_6H_4CH_2Br$ | (4-$BrC_6H_4CH_2S$–)$_2$ (**8**) | 0.5[a] (6)[b] | 88 |
| 12 | $PhCH_2CH_2Br$ | ($PhCH_2CH_2S$–)$_2$ (**9**) | 6 | 88[b] |
| 13 | $CH_2$=$CHCH_2Br$ | ($CH_2$=$CHCH_2S$–)$_2$ (**10**) | 6 | 87[b] |
| 14 | $CH_2$=$CHCH_2Cl$ | ($CH_2$=$CHCH_2S$–)$_2$ (**10**) | 15 | 82[b] |
| 15 | $CH_2$=$C(CH_3)CH_2Cl$ | ($CH_2$=$C(CH_3)CH_2S$–)$_2$ (**11**) | 15 | 85[b] |

a) The reaction was carried out at 80 °C using alkyl halide (4 mmol), thiourea (6 mmol), $MnO_2$ (4 mmol), $NaHCO_3$ (6 mmol), and SDS micellar solution (1.2 mL, 0.2 M). b) The reaction was carried out at room temperature using alkyl halide (4 mmol), thiourea (6 mmol), $MnO_2$ (4 mmol), $Na_2CO_3$ (6 mmol), and SDS micellar solution (2 mL, 0.2 M). c) The reaction was carried out in a sealed tube.




**Scheme 2.** A proposed pathway for the preparation of disulfides from alkyl halides.

the micelle droplet, while the polar head group which carries $-S^-$ moiety is located at the micellar surface droplet which is easily exposed to the oxidant and can be oxidized effortlessly by $MnO_2$ affording the formation of symmetric disulfide compounds.

## Conclusion

We have described herein an efficient process for one-pot odourless synthesis of disulfides from alkyl halides. This method is important because it provides a simple and odourless route to primary, benzylic and allylic disulfides from their corresponding organic halides in good to excellent yields. Moreover, this method is conducted under environmentally benign surroundings and does not require harsh basic conditions and powerful oxidizing agents. Lack of use of foul-smelling thiols is the main advantage of this protocol which is highly suitable for scaled-up reactions. Moreover, in this article another useful application of micellar solution as media and also important catalytic activity in organic synthesis is presented. The procedure can be easily scaled-up for large-scale operation without any difficulties.

## Experimental

$MnO_2$ (sample No. 63548) was purchased from Fluka Chemical Co. and $BaMnO_4$ was prepared according to our reported procedure.[40,41] The other chemicals were purchased from Merck, Fluka and Acros Chemical Companies. IR spectra were run on a Shimadzu FT-IR 8300 spectrophotometer. $^1$H NMR and $^{13}$C NMR spectra were recorded on a Bruker Avance DPX instrument ($^1$H NMR 250 MHz, $^{13}$C NMR 62.5 MHz) in $CDCl_3$. Chemical shifts are reported in ppm ($\delta$) downfield from TMS. Coupling constants ($J$) are in Hertz. Mass spectra were recorded on a Shimadzu GC-MS QP 1000 EX instrument. Elemental analyses were run on a Thermo

Finnigan Flash EA-1112 series. Thin-layer chromatography was carried out on silica gel 254 analytical sheets obtained from Fluka. Column chromatography was performed on Merck Kieselgel (70–230 mesh).

**General Procedure for One-Pot Preparation of Symmetric Disulfides from Alkyl Halides in SDS Micellar Solution at 80 °C.** To a micellar solution of SDS (1.2 mL, 0.2 M), alkyl halide (4 mmol), thiourea (6 mmol), $MnO_2$ (4 mmol), and $NaHCO_3$ (6 mmol) were added. The resulting mixture was stirred magnetically at 80 °C while being monitored by GC until the alkyl halide was consumed. After completion of the reaction, the mixture was directly extracted three times with low-boiling petroleum ether (3 × 3 mL). The organic layer extracts were combined and dried over $Na_2SO_4$, filtered and concentrated to yield the crude product, which was further purified by silica gel column chromatography, using petroleum ether to provide the desired product in good to excellent yields (Table 2, Entries 1–11).

**Typical Physical Data for Some of the Reported Compounds: Didecyl Disulfide (1);** Colorless oil; $^1$H NMR (250 MHz, $CDCl_3$): $\delta$ 2.63 (t, $J = 7.3$ Hz, 4H), 1.76–1.61 (m, 4H), 1.21 (broad band, 28H), 0.84–0.79 (m, 6H); $^{13}$C NMR (62.5 MHz, $CDCl_3$): $\delta$ 39.2, 31.9, 29.6, 29.5, 29.3, 29.3, 29.2, 28.5, 22.7, 14.1; Anal. Calcd for $C_{20}H_{42}S_2$: C, 69.29; H, 12.21; S, 18.50%. Found: C, 69.20; H, 12.24; S, 18.56%.

**Dioctyl Disulfide (2);**[42] Colorless oil; $^1$H NMR (250 MHz, $CDCl_3$): $\delta$ 2.61 (t, $J = 7.3$ Hz, 4H), 1.66–1.54 (m, 4H), 1.30–1.21 (broad band, 20H), 0.84–0.79 (m, 6H); $^{13}$C NMR (62.5 MHz, $CDCl_3$): $\delta$ 39.2, 31.8, 29.2, 29.2, 29.1, 28.5, 22.6, 14.1; Anal. Calcd for $C_{16}H_{34}S_2$: C, 66.14; H, 11.79; S, 22.07%. Found: C, 66.21; H, 11.71; S, 22.08%.

**Typical Large-Scale Procedure for One-Pot Preparation of Dibutyl Disulfide from *n*-Butyl Bromide in SDS Micellar Solution at 80 °C (Table 2, Entry 5).** To a micellar solution of SDS (12 mL, 0.2 M), thiourea (60 mmol, 4.57 g), *n*-butyl bromide (40 mmol, 5.48 g), $MnO_2$ (40 mmol, 3.48 g), and $NaHCO_3$ (60 mmol, 5.04 g) were added. The mixture was stirred magnetically at 80 °C and the progress of the reaction was monitored by GC until *n*-butyl bromide was consumed. After completion of the reaction (5 h), the mixture was extracted with petroleum ether (3 × 10 mL). The organic layers were combined together and dried over $Na_2SO_4$, filtered and concentrated to yield the crude product. The resulting crude product was further purified by silica gel column chromatography eluted with low-boiling petroleum ether to provide the desired product in 3.10 g (87% yield).

**General Procedure for One-Pot Preparation of Benzylic and Allylic Disulfides from Their Corresponding Halides in SDS Micellar Solution at Room Temperature.** To a micellar solution of SDS (2 mL, 0.2 M), thiourea (6 mmol), benzyl or an allyl halide (4 mmol), $MnO_2$ (4 mmol), and $Na_2CO_3$ (6 mmol) were added. The mixture was stirred magnetically at room temperature and the progress of the reaction was monitored by TLC until the halide was consumed (10 h). After completion of the reaction, the mixture was directly extracted with petroleum ether (3 × 3 mL). The organic layers were decanted and combined together, dried over $Na_2SO_4$, filtered and concentrated to yield the crude product. Further purification of the crude product was performed by silica gel column chromatography using light petroleum ether as an eluent to provide the pure desired product in good to excellent yields (Table 2, Entries 8–15).

**Typical Physical Data for Some of the Reported Compounds: Bis(4-methylbenzyl) Disulfide (7);** Colorless oil; $^1$H NMR (250 MHz, $CDCl_3$): $\delta$ 7.05 (s, 8H), 3.52 (s, 4H), 2.25 (s, 6H); $^{13}$C NMR (62.5 MHz, $CDCl_3$): $\delta$ 137.2, 134.4, 129.4, 129.3, 42.7, 21.3; Anal. Calcd for $C_{16}H_{18}S_2$: C, 70.02; H, 6.61; S, 23.37%. Found: C, 70.09; H, 6.58; S, 23.33%.

**Bis(4-bromobenzyl) Disulfide (8);**[30] Colorless oil; $^1$H NMR (250 MHz, $CDCl_3$): $\delta$ 7.35 (d, $J = 8.3$ Hz, 4H), 7.00 (d, $J = 8.3$ Hz, 4H), 3.46 (s, 4H); $^{13}$C NMR (62.5 MHz, $CDCl_3$): $\delta$ 136.3, 131.7, 131.2, 121.5, 42.5; Anal. Calcd for $C_{14}H_{12}Br_2S_2$: C, 41.60; H, 2.99; S, 15.87%. Found: C, 41.66; H, 2.90; S, 15.93%.

**Bis(2-methyl-2-propenyl) Disulfide (11);** Colorless oil; $^1$H NMR (250 MHz, $CDCl_3$): $\delta$ 4.89–4.80 (m, 4H), 3.22 (s, 4H), 1.76 (s, 6H); $^{13}$C NMR (62.5 MHz, $CDCl_3$): $\delta$ 140.7, 114.9, 46.4, 20.9; Anal. Calcd for $C_8H_{14}S_2$: C, 55.12; H, 8.09; S, 36.79%. Found: C, 55.16; H, 8.01; S, 36.83%.

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