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ORIGINAL PAPER

One-pot conversion of alkyl halides to organic disulfides (disulfanes) using thiourea and hexamethyldisilazane (HMDS) in DMSO

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Abstract A practical method to synthesize symmetric disulfides from alkyl halides has been developed. Using this procedure primary, secondary, benzylic and allylic disulfides were prepared by treating the corresponding alkyl halides with thiourea and HMDS in DMSO at 50 °C within 10–24 h in high yields.

Keywords Disulfide · Alkyl halide · HMDS · DMSO · Thiourea

Introduction

Organic disulfides or disulfanes are among organosulfur compounds which have important potentials in biology, industry and synthetic organic chemistry [1, 2]. They have been used as anti-bacterial [3–7], anti-malarial [8, 9] and enzyme modulator reagents [10]. They have been also utilized in developing drug delivery systems [11]. Disulfides can form self-assembled monolayers on metal surfaces which are necessary for the construction of an electrochemical biosensor [12–16]. They have potentials in optical data processing and communication [17]. Synthetically, disulfides are key intermediates in a wide variety of organic synthetic routes.

Disulfides have been synthesized by employing different strategies using structurally diverse substrates [18, 19]. The main route for the preparation of symmetric disulfides is oxidation of thiols which has been carried out with many oxidizing agents [20–27]. Alternatively, the symmetrical disulfides can be obtained by treating sulfonyl chlorides with reducing reagents [28], and unsaturated hydrocarbons with sulfur monochloride [29–31]. In addition, they can be prepared by starting from Bunte salts [32–36], organic thiocyanates [37–39] or alcohols [40–43].

As alkyl halides are precursors of thiols, their conversion to disulfides seems to be more appropriate from organic synthesis point of view. In this line, symmetrical organic disulfides have been prepared by treating alkyl halides with disulfide anion [44–49], Na_2S/C_2Cl_6 [50], Na_2S/CCl_4 [50], S_8/NaOH [51, 52], $S_8/NaBH_4$ [53], tetrathiotungstate or tetrathiomolybdate complexes [54, 55]. Furthermore, symmetric disulfides can be achieved when alkyl halides are reacted with thiourea in the presence of a base and a suitable oxidizing reagent (Scheme 1) [56–60].

According to this strategy, a disulfide is produced through oxidation of the corresponding thiol in situ generated from the reaction of an alkyl halide and thiourea. So far, many oxidizing reagents have been introduced for conversion of thiols to disulfides [18, 19]. However, the selection of an oxidant is a major challenge for developing this strategy since thiol over-oxidation [61–63], thiourea degradation [64–66], and formation of undesired thioethers [67–69] can take place by employment of inappropriate oxidants.

Results and discussion

Dimethyl sulfoxide (DMSO) has been previously applied for oxidizing thiols to disulfides [70]. The oxidative coupling of thiols by DMSO suffers from difficulties

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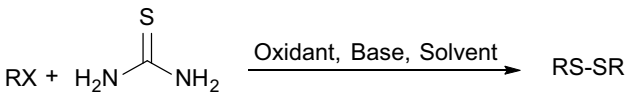
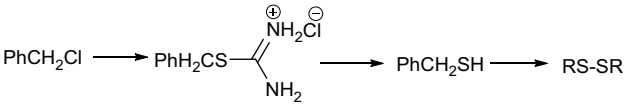


Scheme 1 Disulfide formation from alkyl halide and thiourea in the presence of a suitable oxidant

such as low yields and long reaction times. This problem is circumvented by utilizing DMSO in combination with oxophile reagents [71, 72]. Hexamethyldisilazane (HMDS) is a stable, commercially available, cheap and mild oxophile silylating reagent. This reagent and ammonia as its by-product are mild basic reagents. Considering these, we are interested in developing the one-pot synthesis of symmetrical disulfides from their corresponding organic halides using thiourea and HMDS in DMSO. To establish an efficient procedure, we studied the conversion of benzyl chloride into dibenzyl disulfide under various conditions. After several attempts, the expected disulfide (1) was produced in 93 % yield within 10 h by reacting benzyl chloride (2 mmol), thiourea (2.2 mmol) and HMDS (3 mmol) in wet DMSO (2 mL DMSO + 0.05 mL H_2O) at 50 °C. This reaction can be divided into three steps as below.



It is a tandem reaction which is started with a nucleophilic attack of thiourea on benzyl chloride to generate the *S*-benzylisothiouronium salt. Next, benzyl mercaptan is produced through the hydrolysis of the intermediate salt and undergoes fast oxidation with DMSO subsequently. Under reaction conditions, the starting halide was gradually consumed within 3 h. Work-up of the reaction immediately after consumption of the starting halide (3 h) gave the desired disulfide in 61 % yield whereas the work-up of the same reaction after 10 h resulted benzyl disulfide in 93 % yield due to completion of the *S*-benzylisothiouronium salt hydrolysis process. The thiol intermediate was not observed during reaction leading to the conclusion that the oxidation of thiol must be very fast. This reaction was scaled up to 30 mmol (3.797 g) of benzyl chloride without significant change of yield. The scope and limitation of this procedure was then investigated by treating a range of alkyl halides to the reaction. The results have been tabulated in Table 1.

Primary, benzylic and allylic halides (entries 1–14) reacted with thiourea cleanly to produce the corresponding *S*-alkylisothiouronium salt within 2–8 h. It was observed that a primary halide having a shorter alkyl chain reacted with thiourea much rapidly than that having a longer alkyl group. For example, the reaction of *n*-propyl bromide with thiourea was completed within 4 h whereas *n*-octyl bromide reacted with thiourea within 8 h under same conditions. The reaction of 2-bromopropane, bromocyclohexane, bromocyclopentane as secondary halides (entries 15–17) with thiourea proceeded more slowly than previously mentioned halides. However, the corresponding disulfides were obtained in good to excellent yields after 24 h. The reaction of *tert*-butyl bromide as a tertiary substrate was not desirable and produced the corresponding disulfide only in 23 % yield after 72 h.

In accordance with our knowledge and previous reports [55, 56] a mechanism for explaining this conversion has been proposed in Scheme 2.

In this reaction pathway, HMDS plays a dual role. It acts as the required base to produce the thiol moiety from the corresponding *S*-alkylisothiouronium salt. In addition, it plays a key role in oxidation of the in situ generated thiol to disulfide by DMSO. Similarly, DMSO plays both solvent reaction and oxidizing reagent roles in the reaction. The oxidative coupling of thiol moiety by DMSO/HMDS produces NH_3 as by-product which is needed for producing thiol moiety from *S*-alkylisothiouronium salt.

Conclusion

In conclusion, an efficient, versatile and high-yielding protocol for one-pot preparation of disulfides from alkyl halides using cheap, easy handling and readily available thiourea, HMDS and DMSO under mild conditions was developed. As disulfides precursors including thiols, Bunte salts and thiocyanates are synthesized from alkyl halides, this procedure provides a shorter synthetic route to disulfides in the laboratory. This route was also applicable for very convenient large-scale operation.

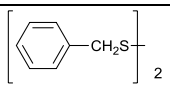
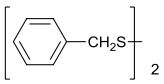
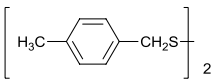
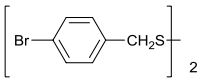
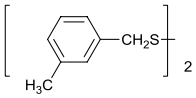
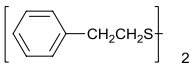
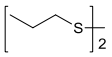
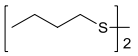
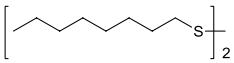
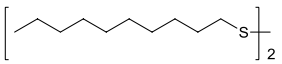
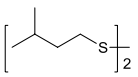
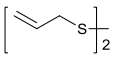
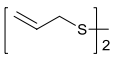
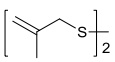
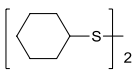
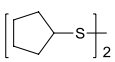
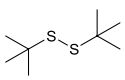
Experimental

General procedure

An alkyl halide (2 mmol) and HMDS (3 mmol) were added to a solution of thiourea (2.2 mmol) in wet DMSO (2 mL DMSO + 0.05 mL H_2O). The mixture was stirred magnetically at 50 °C for 10–24 h. Then, the mixture was diluted with water (2 mL) and extracted with 1:1 EtOAc/hexane (3 × 2 mL). The upper layers were decanted, combined, and concentrated. The crude product was purified by silica gel chromatography using low-boiling petroleum ether as eluent to provide the desired disulfide in high yield.

Table 1 Conversion of various alkyl halides into disulfides

Rx + Thiourea + HMDS $\xrightarrow{\text{wet DMSO}}$ RS-SR

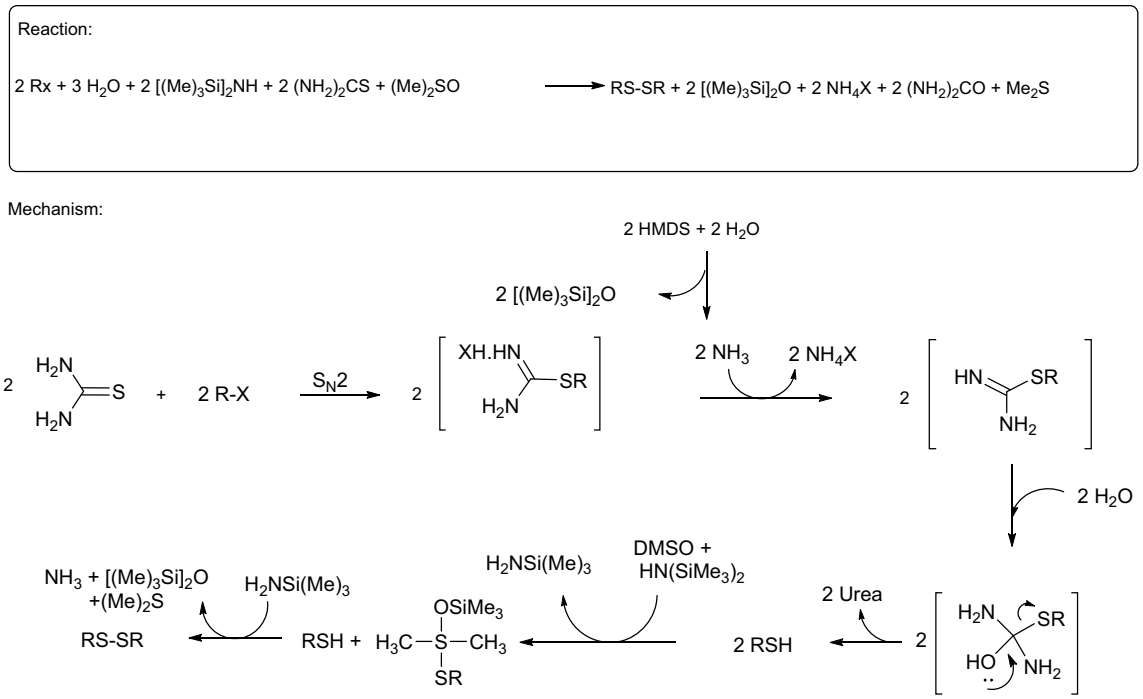
Entry	R-X	RS-SR		Yield (%)[b]	[Ref]
1	$PhCH_2Cl$		**(1)**	93	[52]
2	$PhCH_2Br$		**(1)**	90	[52]
3	*4*-$CH_3C_6H_4CH_2Cl$		**(2)**	89	[73]
4	*4*-$BrC_6H_4CH_2Br$		**(3)**	86	[53]
5	*3*-$CH_3C_6H_4CH_2Cl$		**(4)**	90	[74]
6	$PhCH_2CH_2Br$		**(5)**	91	[75]
7	*n*-C_3H_7Br		**(6)**	89	[76]
8	*n*-C_4H_9I		**(7)**	86	[77]
9	*n*-$C_8H_{17}Br$		**(8)**	85	[78]
10	*n*-$C_{10}H_{21}I$		**(9)**	87	[79]
11	1-Bromo-3-methylbutane		**(10)**	87	[50]
12	Allyl bromide		**(11)**	90	[80]
13	Allyl chloride		**(11)**	89	[80]
14	3-Chloro-2-methyl-1-propene		**(12)**	93	[81]
15[b]	2-Bromopropane		**(13)**	83	[82]
16[b]	Bromocyclohexane		**(14)**	89	[83]
17[b]	Bromocyclopentane		**(15)**	86	[84]
18[c]	*tert*-Butyl bromide		**(16)**	23	[85]

Reaction conditions RX (2 mmol), thiourea (2.2 mmol), HMDS (3 mmol), DMSO (2 mL), H_2O (0.04 mL), 50 °C, 10 h

[a] Isolated yields

[b] The reaction was coducted for 24 h

[c] The reaction was coducted for 72 h



Scheme 2 A proposed reaction and mechanism for preparing disulfides from the reaction of alkyl halides, thiourea, and HMDS in DMSO

Bis(4-methylbenzyl) disulfide (2)

Colorless oil; ^{1}HNMR (250 MHz, $CDCl_3$): δ 7.05 (s, 8H), 3.52 (s, 4H), 2.25 (s, 6H); ^{13}CNMR (62.5 MHz, $CDCl_3$): δ 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 (4)

Colorless oil; ^{1}HNMR (250 MHz, $CDCl_3$): ¤ 7.35 (d, J = 8.3 Hz, 4H),7.00 (d, J = 8.3 Hz, 4H), 3.46 (s, 4H); ^{13}CNMR (62.5 MHz, $CDCl_3$): ¤ 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 %.

Dioctyl disulfide (8)

Colorless oil; ^{1}HNMR (250 MHz, $CDCl_3$): δ 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); δ ^{13}CNMR (62.5 MHz, $CDCl_3$): 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, 66.07; H, 11.81; S, 22.12 %.

Didecyl disulfide (9)

Colorless oil; ^{1}HNMR (250 MHz, $CDCl_3$): δ 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}CNMR (62.5 MHz, $CDCl_3$): δ 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, 69.40; H, 12.23; S, 18.37 %.

Bis(2-methyl-2-propenyl) disulfide (12)

Colorless oil; ^{1}HNMR (250 MHz, $CDCl_3$): δ 4.89–4.80 (m, 4H), 3.22 (s, 4H), 1.76 (s, 6H); ^{13}CNMR (62.5 MHz, $CDCl_3$): δ 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, 55.03; H, 8.06; S, 36.91 %.

Dicyclohexyl disulfide (14)

Colorless oil; ^{1}H NMR (250 MHz, $CDCl_3$): δ 2.69–2.61 (m, 2H) 2.03–1.97 (m, 4H) 1.80–1.72 (m, 4H) 1.63–1.55 (m, 2H) 1.34–1.14 (m, 10H); ^{13}C NMR (62.5 MHz, $CDCl_3$): 49.9, 32.8, 26.1, 25.7; Anal. calcd for $C_{12}H_{22}S_2$: C, 62.55; H, 9.62; S, 27.83. Found: C, 62.70; H, 9.55; S, 27.75 %.

Dicyclopentyl disulfide (15)

Colorless oil: ^{1}HNMR (250 MHz, $CDCl_3$): δ 3.45–3.39 (m, 2H), 1.97–1.94 (m, 4H), 1.69–1.51 (m, 12H); ^{13}CNMR (62.5 MHz, $CDCl_3$): δ 50.7, 32.9, 24.7; Anal. calcd for $C_{10}H_{18}S_2$: C, 59.35; H, 8.97; S, 31.68 %. Found: C, 59.51; H, 8.99; S, 31.50 %.

Typical scale-up procedure

To a solution of thiourea (33 mmol) in wet DMSO (30 mL DMSO + 0.6 mL H_2O) benzyl chloride (30 mmol, 3.45 mL) and HMDS (45 mmol, 9.43 mL) were added, respectively. The mixture was stirred magnetically at 50 °C. The starting halide was completely consumed within 3 h. However, the stirring was continued for 10 h under such conditions to ensure the reaction completion. Then, the mixture was diluted with water (10 mL) and extracted with 1:1 EtOAc/hexane (3 × 10 mL). The upper layers were decanted, combined, dried over Na_2SO_4, filtered and concentrated to yield the crude product, which was further purified by silica gel chromatography, using low-boiling petroleum ether as eluent to provide the dibenzyl disulfide in 93 % (3.437 g) yield.

Dibenzyl disulfide (1)

White crystal powder, M.p. 68–70 °C (Lit [52]. 69–70 °C); ^{1}HNMR (400 MHz, $CDCl_3$): δ 7.34–7.26 (m, 10H), 3.61 (s, 4H); ^{13}CNMR (100 MHz, $CDCl_3$): δ 138.6, 130.7, 129.7, 128.7, 44.4; Anal. calcd for $C_{14}H_{14}S_2$: C, 68.25; H, 5.73; S, 26.02 %. Found: C, 68.39; H, 5.76; S, 25.85 %.

Acknowledgments We gratefully acknowledge the support of this study by Persian Gulf University Research Council.

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