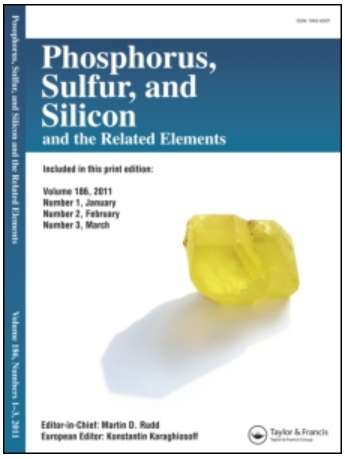

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A New Application of *N,N′*-dibromo-*N,N′*–1,2-ethanediylbis(*p*-toluenesulfonamide) as Selective and Efficient Reagent for the Oxidation of Various Thiols to Disulfides

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An efficient method for oxidation of thiol to their corresponding disulfides in high yields with N,N′*-Dibromo-*N,N′*–1,2-ethanediyl bis (*p*-toluensulphonamide) in dichloromethane at room temperature is described.*

Keywords BNBTS; Disulfide; Oxidation; Thiol

INTRODUCTION

Disulfides are an important compounds from both biological and synthetic points of views.[1,2] Disulfides are also important intermediates with a great deal of application in organic synthesis.[3–5] The transformation of thiols to disulfides has been studied employing various oxidants.[6–9] However, some of these reagents suffer from one or more of the following disadvantages: availability of the reagents, toxicity, tedious work-up, high cost, preparation, and stability of the reagent . As a result, there is still a need for introducing readily available, safe, stable, and in a cheap reagents for the oxidation of thiols to disulfides.

Extending our work on the use of N-halosolulfonamid, in organic synthesis.[10–14] We now report an efficient and convenient method for the oxidative coupling of thiols to their corresponding disulfides by using a new, cheap, and easily made reagent *N,N′*-Dibromo-*N,N′*–1,2-ethanediylbis(*p*-toluenesulfonamide), [BNBTS] (**2**) that was prepared

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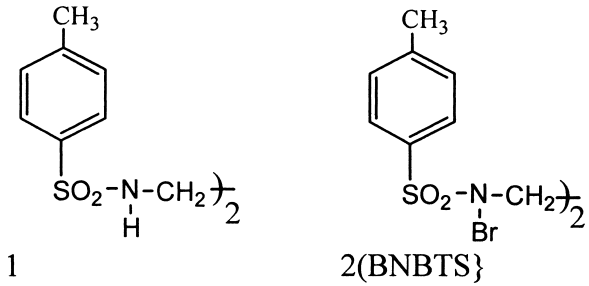
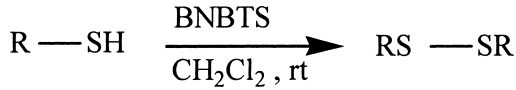


FIGURE 1

from *N,N′*–1,2-ethanediylbis(*p*-toluenesulfonamide) [BNHTS] (1) (Figure 1).

Different kinds of thiols were subjected to oxidation reaction in the presence of BNBTS in CH_2Cl_2 at room temperature (Scheme 1).



SCHEME 1

The results of this study are summarized in Table I. As shown in the Table I, a variety of aliphatic (cyclic and acyclic), aromatic thiols are converted into symmetrical disulfides with high yields. In case of aromatic thiols bromination of aromatic ring was not observed; also selectivity of the present method is evident by the oxidation of 2-mercaptoethanol (entry 11) where only mercaptan functionality is converted to the disulfides.

TABLE I Oxidation of Thiols to Disulfides with BNBTS in Dichloromethane at Room Temperature

Entry	R	Time (h)	Mol ratio	Yield (%)
1	Ph	1.5	1	93
2	4-MeC_6H_4	1.5	1	94
3	4-ClC_6H_4	2	1	95
4	2-MeC_6H_4	1.7	1	92
5	2-Naphthyl	1.8	1	94
6	$PhCH_2$	2.5	1	93
7	Cyclohexyl	3	1	91
8	n-Ethyl	3	1	92
9	n-Buthyl	3	1	90
10	n-Octyl	3.2	1	94
11	$HOCH_2CH_2$	3.5	1	90
12	$HOOCCH_2CH_2$	3.8	1	93

CONCLUSION

This article describes a facile synthesis of disulfides using BNBTS that is cheap, stable, and an easily handled oxidizing agent in comparison to most oxidants usually employed for this transformation. Also the recovered starting martial (**1**) was rebrominated and used many times without reducing the yield. The method offers several other advantages including simplicity of the reaction conditions, application to alkyl and aryl thiol, selectivity, and ease of isolation of the products.

EXPERIMENTAL

All products are known compounds and were characterized by comparison of their spectral data (^{1}H- NMR and IR) and their physical properties with those reported in the literature.

GENERAL PROCEDURE FOR OXIDATION OF THIOLS

To a solution of thiol (1 mmol) in dichloromethane (10 mL) was added BNBTS (1 mmol) and the mixture was stirred vigorously and magnetically at room temperature for the indicated time according to Table I. The reaction was monitored by thin layer chromatography (TLC). After completion of the reaction, the insoluble sulfonamide (**1**) was removed by filtration and washed with dichloromethan (10 mL). Evaporation of the solvent followed by recrystallization or chromatography on silica gel afforded pure disulfide in 90–95% yields (Table I).

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