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Ethylene bis (*N*-methyl imidazolium) ditribromide: An efficient and reusable reagent for oxidation of thiols and sulfides

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Ethylene bis (*N*-methyl imidazolium) ditribromide (EBMIDTB) is an efficient and selective reagent for the oxidation of thiols to their corresponding symmetrical disulfides and sulfides to sulfoxides under mild reaction conditions in good to excellent yields. Selective oxidation of thiols in the presence of sulfides at room temperature is also achieved with this reagent.

Keywords: Ethylene bis (*N*-methyl imidazolium) ditribromide, thiols, sulfides, oxidation, sulfoxides

INTRODUCTION

Disulfides are important compounds possessing unique and diverse chemistry in the synthetic and biochemical areas. Large disulfide-linked aggregates are prevalent in proteins and many other bioactive molecules [1]. Industrially, disulfides find wide applications as vulcanizing agents for rubbers and elastomers, giving them excellent tensile strength. Numerous reagents and catalysts have been applied to oxidize thiols to disulfides under controlled conditions [2]. Disulfides can also be prepared by electrochemical oxidation of thiols in methanol/sodium methoxide solution under constant current [3].

Most of the described methods for oxidation of thiols to disulfides make use of volatile organic solvents [4] and are promoted by molecular bromine supported on silica gel [4a], anhydrous potassium phosphate [4b], PCC [4c], *N*-phenyltriazolinedione [4d], nitric acid [4e], $I_2/CeCl_3.7H_2O$ [4f], $VO(acac)_2$ [4g], $Bu_3SnOMe/FeCl_3$ [4h], potassium permanganate [4i], $(NH_4)_2S_2O_8$ [4j], trichloroisocyanuric acid [4k], montmorillonite K10 [4l], CsF-Celite [4m], and basic alumina [4n].

Although, many of the reported methods are effective, but suffer from drawbacks such as the use of expensive, rare or toxic reagents and metal oxidants, low yields, long reaction times and high temperature, or the risk of overoxidation of the products. Thus, there is still a demand for simple and efficient oxidative methods that would produce the target disulfides in high yields without above mentioned disadvantages.

On the other hand, selective oxidation of sulfides to the corresponding sulfoxides remains a challenge and is interesting because of the importance of sulfoxides as synthetic intermediates for the construction of various chemically and biologically significant molecules [5], especially for the synthesis of drugs and natural products [6]. There are several reagents available for this key transformation; sulfoxides conventionally prepared using stoichiometric amounts of both organic and inorganic reagents, for example, $FeBr_3$-nitric acid [7a], *N*-halosuccinimides [7b], NBS/*β*-CD/H_2O [7c], *m*-chloroperbenzoic acid [7d], halogens [7e], MnO_2–TMSCl [7f], sodium meta periodate [7g], $(n\text{-}Bu_4N)_2S_2O_8$ [7h], CAN [7i], ozone [7j], $NaClO_2$/ Mn (III) catalyst/Alumina [7k], binuclear manganese complex periodic acid [7l], and $HAuCl_4 \cdot 4H_2O/H_2O_2$ [7m].

Due to importance of sulfoxides in organic synthesis, the introduction of a mild, efficient and selective method to synthesize sulfoxides is still needed.

Tribromide reagents due to their crystalline nature are preferred over some liquid brominating agent such as, liquid bromine, as they are less harmful and can be easily handled and stored, without loss of bromine. These reagents have been recently used as either an oxidant [8] or brominating agent [9] in organic synthesis; for example, tribromides, quinolinium tribromide

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[10a], bipyridinum hydrobromide perbromide [10b], *o*-xylylenebis(triphenylphosphonium tribromide [10c], pyridinium tribromide [10d] and cetyltrimethylammonium tribromide [10e].

EXPERIMENTAL

^{1}H NMR spectra were measured on Bruker Avance DRX 500 MHz and Bruker Avance 400 MHz spectrometers, using deuterated chloroform ($CDCl_3$) as solvent. Melting points were determined on Electro Thermal 9100. Materials were purchased from Fluka and Merck companies. All the products were characterized by ^{1}H NMR, and GC data and also by comparison with authentic samples.

General procedure:

To a solution of thiol or sulfide (1 mmol) in acetonitrile (5 mL), EBMIDTB (0.336 g, 0.5 mmol) was added. The mixture was stirred magnetically at room temperature for the appropriate time (Tables 1, 2). The progress of the reaction was monitored by TLC (eluent: n-hexane/ethyl acetate: 5/1) or GC. The reaction mixture was transferred into a separatory funnel, and washed with water (15 mL) and extracted with dichloromethane (20 mL). Organic layer was dried over anhydrous Na_2SO_4 and solvent was concentrated in a rotary evaporator. The crude product was purified by passing it over a column of silica gel, using a mixture of *n*-hexane and ethyl acetate as the eluent. In order to regenerate the reagent, the aqueous layer was concentrated under vacuum and treated with Br_2 in *n*-hexane. All of the products were known compounds and characterized by comparing melting point and ^{1}H NMR spectra with does reported in the literature. ^{1}H NMR spectra of some products are given below:

1, 2- Diphenyl disulfide ***(2a).***

Yield 97%; mp 60-61 ºC; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.26-7.31 (m, 2H), 7.35–7.40 (m, 4H), 7.57 (d, J = 8.3, 2H), 7.61 (d, J = 8.3, 2H).

1, 2- Di p-tolyl disulfide ***(2c).***

Yield 99%; mp 45-47 ºC; ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.36 (s, 6H), 7.15 (d, J = 8.1, 4H), 7.42 (d, J= 8.1, 4H).

1, 2- Bis(4-bromo phenyl) disulfide ***(2f).***

Yield 90%; mp 92-95 ºC; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.38 (d, J = 8.4, 4H), 7.47 (d, J = 8.4, 4H).

*1, 2- Bis(benzo thiazolyl) disulfide (**2g**).*

Yield 85%; mp 176-178 ºC; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38 (dt, J = 7.7, 1.0, 2H), 7.49 (dt, J = 7.7, 1.0, 2H), 7.79 (d, J = 8.0, 2H), 7.96 (d, J = 8.4, 2H).

1, 2- Dipentyl disulfide ***(2i).***

Yield 85%; ^{1}H NMR (500 MHz, $CDCl_3$): δ 0.95 (t, J = 7.0, 6H), 1.35-1.44 (m, 8H), 1.69-1.75 (m, 4H), 2.72 (t, J = 7.4, 4H).

1, 2- Dicyclohexyl disulfide ***(2j).***

Yield 75%; ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.24-1.39 (m, 10H), 1.64-1.67 (m, 2H), 1.81-1.86 (m, 4H), 2.07–2.09 (m, 4H), 2.69-2.74 (m, 2H).

Dibenzyl sulfoxide ***(4b).***

Yield 98%; mp 134-136 ºC; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.90 (d, J = 13.0, 2H), 3.95 (d, J = 13.0, 2H), 7.30-7.33 (m, 4H), 7.35-7.42 (m, 6H).

Benzyl phenyl sulfoxide ***(4c).***

Yield 96%; mp 122-124 ^{0}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.02 (d, J = 12.4, 1H), 4.12 (d, J = 12.4, 1H), 7.0 (d, J = 7.6, 2H), 7.24-7.33 (m, 4H), 7.38-7.48 (m, 4H).

4-Methyl benzyl phenyl sulfoxide ***(4d).***

Yield 97%; mp 96-98 ºC; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.42 (s, 3H), 4.0 (d, J = 12.6, 1H), 4.11 (d, J= 12.6, 1H), 7.02 (d, J = 7.6, 2H), 7.24-7.31 (m, 7H).

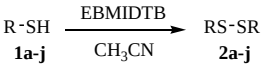
RESULTS AND DISCUSSION

Ethylene bis (*N*-methyl imidazolium) ditribromide is easily prepared from *N*-methylimidazole, 1,2-dibromoethane and bromine and used for bromination of organic compounds [11]. Herein we report its utility for the oxidation of thiols and sulfides to disulfides and sulfoxides, respectively.

Thiols were oxidized in acetonitrile with EBMIDTB to give the corresponding disulfides at room temperature.

In our first experiments, thiophenol was chosen as a model substrate to determine the optimal reaction conditions. When thiophenol was treated with 0.5 mmol EBMIDTB at room temprature in acetonitrile, complete conversion to the corresponding disulfide achieved within 5 min. This reaction was carried out in different solvents such as CH_2Cl_2, THF, dioxane, ethanol, H_2O and under solvent free condition the best results in

Table 1. Oxidation of thiols with EBMIDTB in acetonitrile

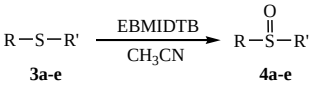


1a-j **2a-j**

Entry	R	Product[a]	Time (min)	Yield %[b]	mp (^{0}C) [Ref]
1	Ph	**2a**	5	97	60-61 [4e]
2	*β*-Naphtyl	**2b**	5	97	143-144 [4e]
3	*p*-Methyl phenyl	**2c**	5	99	45-47 [12]
4	*p*-Methoxy phenyl	**2d**	5	98	42–43 [13]
5	*o*-Methyl phenyl	**2e**	5	60	37-39 [14]
6	*p*-Bromo phenyl	**2f**	5	90	92-95 [4e]
7	Benzo thiazolyl	**2g**	15	85	176-178 [12]
8	Benzyl	**2h**	15	90	71-72 [4e]
9	*n*-Pentyl	**2i**	15	85	Oil [12]
10	Cyclohexyl	**2j**	15	75	Oil [4e]

[a] All the products were identified by comparing of melting point and ^{1}H NMR spectra with those of authentic samples reported in the literature.
[b] Yields refer to isolated products.

Table 2. Oxidation of sulfides with EBMIDTB in acetonitrile.



3a-e **4a-e**

Entry	R	R′	Product[a]	Time (min)	Yield %[b]	mp (^{0}C) [Ref]
1	Phenyl	Me	**4a**	20	95	31-33 [15]
2	Benzyl	Benzyl	**4b**	20	98	134-136 [16]
3	Phenyl	Benzyl	**4c**	20	96	122-124 [17]
4	*p*-Methyl benzyl	Phenyl	**4d**	20	97	96-98 [18]
5	Phenyl	Phenyl	**4e**	90	-	-

[a] All the products were identified by comparing of melting point and ^{1}H NMR spectra with those of authentic samples reported in the literature.
[b] Yields refer to isolated products.

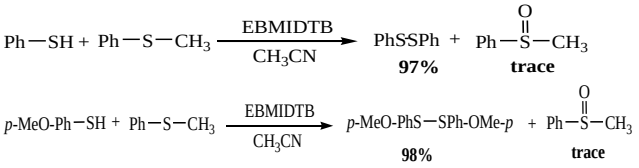
terms of reaction time and yield of the product was obtained, when the reaction conducted in acetonitrile

To show the generality of this procedure, a wide range of aromatic and aliphatic thiols were transformed into the corresponding disulfides by treatment with EBMIDTB in acetonitrile at ambient temperature (Table 1).As it is clear from this table, thiophenol, *β*-thionaphtol, electron donating and electron withdrawing substituted thiophenols were reacted with EBMIDTB at room temperature to give high yield of the expected disulfides (Table 1, entries 1–6).

p-Methyl and *p*-methoxy thiophenol also under reaction conditions formed the expected disulfide in high yields (Table 1, entries 3 and 4). 2-Mercaptobenzothiazol and aliphatic thiols were converted to their corresponding disulfides in good yields in longer reaction times (Table 1, entries 7–10).

Similarly when sulfides were treated with EBMIDTB in acetonitrile at room temperature, a high yield of the corresponding sulfoxides were obtained in 20 min (Table 2, entries 1-4). However, diphenylsulfide under the reaction conditions did not afford any sulfoxides even with higher amount of EBMIDTB and at higher temperature (Table 2, entry 5).

In order to study the selectivity of this method, a mixture of equimolar amounts of thiophenol or *p*-methoxythiophenol and phenylmethylsulfide was treated with 0.5 equivalent of EBMIDTB at room temperature. After only 5 min diphenyl and di(*p*-methoxyphenyl)disulfide were formed in 97-98% yield and a trace amount of phenylmethyl sulfoxide was detected (Scheme 1).



Scheme 1. Selective oxidation of thiol in the presence of sulfide.

Table 4. Comparison of EBMIDTB with other tribromide reagents for the oxidation of thiophenol and methyl phenyl sulfide.

Entry	Substrate	Reagent	Reaction Conditions	Time (min)	Yield%	[Ref]
1	Ph-SH	EBMIDTB	CH_3CN/rt	5	97	-
2	Ph-SH	phenyltrimethylammonium tribromide	Ethyl acetate	1	95	[19a]
3	Ph-SH	*o*-xylylenebis (triphenylphosphonium tribromide)	THF/rt	2	98	[10c]
4	Ph-SH	$\{[K.18\text{-Crown-6}]Br_3\}_n$	CH_3CN/rt	-	100 (conv.)	[19b]
5	Ph-SH	Benzyl trimethyl ammonium tribromide	$NaOH/CH_2Cl_2/H_2O$	-	98	[19c]
6	Ph-SH	Quinolinium tribromide	CH_3CN/rt	10	95	[10a]
7	Ph-SH	bipyridinium hydrobromide perbromide	rt	10	88	[10b]
8	Ph-S-Me	EBMIDTB	CH_3CN/rt	20	95	-
9	Ph-S-Me	*o*-xylylenebis (triphenylphosphonium tribromide)	THF/rt	5	98	[10c]
10	Ph-S-Me	1,1'-(ethane-1,2-diyl)dipyridinium ditribromide	CH_2Cl_2/H_2O	5	87	[19d]
11	Ph-S-Me	phenyltrimethylammonium tribromide	Ethyl acetate	5	86	[19a]
12	Ph-S-Me	Pyridinium tribromide	CH_2Cl_2/ Hydrated silica gel	25	100	[10d]
13	Ph-S-Me	DBU-hydrobromide-perbromide	H_2O_2/H_2O CH_3CN	60	95	[19e]
14	Ph-S-Me	*N*-methylpyrrolidine-2-one hydrotribromide	$CHCl_3$/ H_2O 65 ºC	180	85	[19f]
15	Ph-S-Me	phenyltrimethylammonium tribromide	Pyridine	180	85	[15]
16	Ph-S-Me	Cetyltrimethyl ammonium tribromide	CH_3CN/ H_2O/rt	720	93	[10e]

A good feature of this reagent is that it can be regenerated and reused several times without loss of activity. To regenerate the reagent, after completion of the reaction, the mixture was successively washed with water and CH_2Cl_2. The aqueous layer was concentrated and treated with bromine in *n*-hexane to give ethylene bis (*N*-methyl imidazolium) ditribromide, which was identical in all respects with the parent EBMIDTB. This process repeated for three cycles in the oxidation of thiophenol and the yield of the diphenyldisulfide did not change significantly (Table 3). The decrease in conversion can be attributed to the losses during handling of the small amount of reagent during recycle. These results clearly show the stability of EBMIDTB in the reaction media as well its recovery and recycle without any appreciable decrease in its activity.

Table 3. Recycling studies of oxidation of thiophenol to diphenyldisulfide with EBMIDTB.

Entry	Cycle	Time (min)	Yield %[a]
1	fresh	5	97
2	1	5	94
3	2	7	89
4	3	7	87

[a] Yields refer to isolated products.

However, in order to show the merits and drawbacks of this reagent, some of our results were compared with other tribromide reagents reported in literature (Table 4). As shown in Table 4,thiophenol and methyl phenyl sulfide were oxidized with EBMIDTB very fast at room temperature in excellent yield in mild conditions (Table 4, entries 1 and 8), which is comparable with some reagents (Table 4, entries 2-5 and 9-11), but in other cases gave better results (Table 4, entries 6-7 and 12-16).

In summary, the method described here is very simple and efficient for the oxidation of thiols and sulfides to the corresponding disulfides and sulfoxides using EBMIDTB. This reagent oxidizes thiols almost quantitatively irrespective of the presence of sulfides. This method is applicable to a wide variety of thiols and sulfides, being a useful alternative of the existing methodologies.

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ЕТИЛЕН БИС-(*N*-МЕТИЛ ИМИДАЗОЛ) ДИ-ТРИБРОМИД: ЕФЕКТИВЕН И МНОГОКРАТНО ИЗПОЛЗВАН РЕАГЕНТ ЗА ОКИСЛЕНИЕТО НА ТИОЛИ И СУЛФИДИ

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(Резюме)

Етилен бис-(*N*-метил имидазол) ди-трибромидът (EBMIDTB) е ефективен и селективен реагент за окилението на тиоли до съответните симетрични дисулфиди и на сулфиди до сулфоксиди при меки условия с добри до отлични добиви. С този реагент се постига и селективното окисление на тиоли в присъствие на сулфиди при стайна температура.