



Trans-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane as a novel and efficient reagent for selective sulfoxidation of sulfides under catalyst-free condition

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ABSTRACT

Application of *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane as an efficient and high-oxygen content reagent in selective oxidation of sulfides into sulfoxides has been successfully explored. The reactions proceeded under mild and catalyst-free conditions in dichloromethane at room temperature to provide the sulfoxides in excellent yields without any significant over-oxidation to sulfones.

1. Introduction

Sulfoxides belong to an important class of organic compounds which play important roles in synthetic organic chemistry [1]. They have also received enormous interests as chiral auxiliaries in organic synthesis [2,3], and as useful building blocks for the synthesis of biologically and medicinally active compounds, and activation of enzymes as well [4,5]. Sulfoxides have also found important applications as therapeutic agents such as cardiotoxic [6], antiulcerative (proton pump inhibitor) [7], antihypertensive [8], as well as psychotropics [9], and vasodilators [10]. Many procedures and synthetic methods have been developed for sulfoxide synthesis [11,12]. Oxidation of sulfides to corresponding sulfoxides is the most simple and straightforward method [4,5,13]. Several types of oxidizing agents and methods have been used for sulfoxidation process in solution and on solid phase [14-16]. A number of oxygen donors such as H₂O₂ [1], *m*-CPBA [17], ozone [18], O₂ and *t*-BuOOH [19], has been reported. Among these, aqueous H₂O₂ is a high atom-efficiency (47%) and most attractive oxidant in the green context as it is environmentally benign (only generates water as the by-product) and safe in operation [20]. However, H₂O₂ oxidizes sulfides rather slowly and therefore various kinds of acids such as AcOH [21], TBHP/*p*-TSA [22], and transition metal (Fe [23,24], Ti [25], Mo [26], V [27,28], W [29], Pt [30], Mn [31], Cu [32], Ru [33], Ta [34], Ce [35], Sc [36], Zr [37], and Cr [38]) based systems have been used to activate hydrogen peroxide. Many of these reagents and catalysts suffer from drawbacks such as long reaction times, low selectivity, low yields of products, and toxicity due to the formation of environmentally harmful waste by-products. Furthermore, many of these catalysts present problems such as over-oxidation into sulfones and unfavorable oxidation of other functional groups present in the sulfides [39]. In order to avoid such drawbacks still there is a great demand for the development of new oxidizing systems.

Recently, Selvam and co-workers reported the novel use of cyclohexylenebishydroperoxide in oxidation of sulfides to sulfoxides [40]. However, low selectivity and oily state of this reagent makes this method practically undesirable.

The objective of the present work was to investigate the capability of *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane as a high-oxygen content oxidation reagent in sulfoxidation reaction. The interest in this protocol is that it accelerates the conversion of various sulfides selectively into sulfoxides with high yields in the absence of any activating catalyst.

2. Experimental

2.1. General procedures and instrumentation

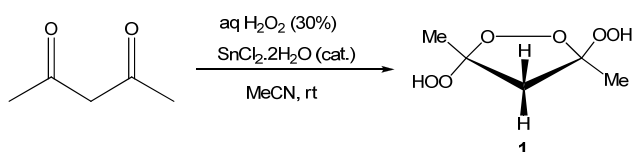
Chemicals were obtained from Merck and Fluka chemical companies. Melting points were measured by a Büchi 530 melting point apparatus. FT-IR spectra were recorded on a Shimadzu 435-U-04 spectrophotometer. ¹H and ¹³C NMR spectra were recorded on a Varian 200 MHz and JEOL FX 90 MHz spectrometer in CDCl₃ solution. All sulfoxides are known and were characterized by comparison of their physical and spectral (¹H NMR, ¹³C NMR and IR) data with those reported in the literature.

Caution: *Trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane as a peroxidic compound is potentially explosive and requires precautions in handling (shields, fume hoods, absence of transition metal salts and heating above room temperature).

2.2. Procedure for preparation of *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane

SnCl₂·2H₂O (45 mg, 0.2 mmol) was added to a stirred solution of acetylacetone (100 mg, 1 mmol) in CH₃CN (5 mL) and stirring of the reaction mixture was continued for 5 min at

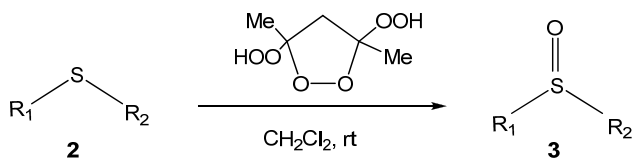
room temperature (Scheme 1). Then, aqueous 30% H₂O₂ (5 mmol) was added to the reaction mixture and was allowed to stir for 12 h at room temperature. After completion of the reaction as monitored by thin layer chromatography (TLC), water (15 mL) was added and the product was extracted with ethylacetate (2×10 mL). The combined organic layer was dried over anhydrous MgSO₄ and evaporated under reduced pressure to give almost a pure white crystalline product, **1**. Yield: 85% (140 mg). M.p.: 98-100 °C; ¹H NMR (200 MHz, CDCl₃) δ (ppm): 8.43 (bs, 2H, OOH), 2.67 (s, 2H, CH₂-4), 1.59 (s, 6H, CH₃). ¹³C NMR (50 MHz, D₂O) δ: 16.5 (CH₃), 50.7 (CH₂-4), 112.7 (C-3, C-5); IR (KBr pellet) ν(cm⁻¹): 3389 (w), 1433, 1380, 1333, 1173, 848, 790, 470. Anal. Calcd. for C₅H₁₀O₆: C 36.14; H 6.02; Found: C 36.08; H 5.87 %.



Scheme 1

2.3. General procedure for oxidation of sulfides to sulfoxides

Trans-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane, **1**, (166 mg, 1 mmol) was added to a solution of sulphide, **2**, (1 mmol) in dichloromethane (5 mL) and the reaction mixture was allowed to stir at room temperature for an appropriate time (Table 1) (Scheme 2). After completion of the reaction as monitored by TLC, saturated aqueous solution of Na₂SO₃ (3 mL) was added to quench the remaining oxidant. Then water (10 mL) was added and the product was extracted with dichloromethane (3 × 5 mL). The combined organic layer was washed with water (2 × 5 mL) and dried over anhydrous MgSO₄. Evaporation of the solvent under reduced pressure gave almost pure product. Chromatography on silica gel provided pure sulfoxide **3**.



Scheme 2

Methyl phenyl sulfoxide, 3a: IR: ν(cm⁻¹) 3056, 2914, 1655, 1583, 1476, 1444, 1090, 1048, 957, 748, 692. ¹H NMR (90 MHz, CDCl₃) δ (ppm): 2.78 (s, 3H, CH₃), 7.48-7.54 (m, 5H, ArH). ¹³C NMR (22.5 MHz, CDCl₃) δ (ppm): 43.8, 123.2, 129.2, 130.8, 145.3. MS (m/z) 141 (M+1).

Diphenyl sulfoxide, 3b: IR: ν(cm⁻¹) 3048, 1580, 1476, 1441, 1087, 1037, 737, 694. ¹H NMR (90 MHz, CDCl₃) δ (ppm): 7.39-7.52 (m, 10H, ArH). MS (m/z) 203 (M+1).

Dibenzyl sulfoxide, 3c: IR: ν(cm⁻¹) 3031, 2958, 1602, 1454, 1032, 775, 699. ¹H NMR (90 MHz, CDCl₃) δ (ppm): 3.98 (s, 4H, CH₂), 7.40-7.42 (m, 10H, ArH). ¹³C NMR (22.5 MHz, CDCl₃) δ (ppm): 57.4, 128.3, 128.9, 130.1, 130.8. MS (m/z) 231 (M+1).

Benzyl phenyl sulfoxide, 3d: IR: ν(cm⁻¹) 3059, 2960, 1600, 1442, 1036, 745, 692. ¹H NMR (90 MHz, CDCl₃) δ (ppm): 3.96 (s, 2H, CH₂), 6.94-7.32 (m, 10H, ArH). ¹³C NMR (22.5 MHz, CDCl₃) δ (ppm): 63.5, 124.3, 128.2, 128.5, 128.8, 129.2, 130.3, 131.2, 142.6; MS (m/z) 217 (M+1).

Ethyl phenyl sulfoxide, 3e: IR: ν(cm⁻¹) 3062, 2921, 1493, 1074, 1032, 775, 700; ¹H NMR (90 MHz, CDCl₃) δ (ppm): 0.98 (t, 3H, CH₃), 2.48-2.91 (m, 2H, CH₂), 7.04-7.44 (m, 5H, ArH). MS (m/z) 155 (M+1).

Di-n-butyl sulfoxide, 3f [22]: IR: ν(cm⁻¹) 2874, 1469, 1381, 1288, 1130, 1288, 1130, 1097, 1024, 772. ¹H NMR (90 MHz, CDCl₃) δ (ppm): 0.84-0.91 (m, 6H, CH₃), 1.55 (m, 8H, CH₂), 2.47-2.63 (m, 4H, CH₂). ¹³C NMR (22.5 MHz, CDCl₃) δ (ppm): 13.3, 21.7, 24.3, 51.6; MS (m/z) 163 (M+1).

1-(4-(Methylsulfinyl)phenyl)ethanone, 3g: IR: ν(cm⁻¹) 1692, 1045. ¹H NMR (90 MHz, CDCl₃) δ (ppm): 2.58 (s, 3H, COCH₃), 2.71 (s, 3H, SOCH₃), 7.61 (d, 2H, ArH), 7.97 (d, 2H, ArH). MS (m/z) 183 (M+1).

Dibenzothiophen-5-oxide, 3h: IR: ν(cm⁻¹) 3055, 1591, 1443, 1067, 1024, 754, 714, 556. ¹H NMR (90 MHz, CDCl₃) δ (ppm): 7.46-7.95 (m, 8H, ArH). ¹³C NMR (22.5 MHz, CDCl₃) δ (ppm): 121.8, 127.2, 129.3, 132.3, 136.8, 145.0. MS (m/z) 201 (M+1).

2-Phenylsulfinyl ethanol (3i): IR: ν(cm⁻¹) 3339, 2973, 1658, 1583, 1460, 1377, 1150, 1031, 723. ¹H NMR (90 MHz, CDCl₃) δ (ppm): 2.95 (t, 2H, SOCH₂), 3.87 (t, 2H, OCH₂), 4.72 (bs, 1H, OH), 7.40 (m, 5H, ArH). ¹³C NMR (22.5 MHz, CDCl₃) δ (ppm): 96.7, 99.0, 131, 133.7, 134.6, 140.5. MS (m/z) 171 (M+1).

Methyl 3-(methylsulfinyl) propanoate, 3j: IR: ν(cm⁻¹) 2855, 1734, 1632, 1461, 1376, 1245, 1017, 722. ¹H NMR (90 MHz, CDCl₃) δ (ppm): 2.55 (s, 3H, SOCH₃), 2.89 (m, 4H, CH₂), 3.66 (s, 3H, OCH₃). MS (m/z) 135 (M+1).

4-Nitrobenzyl phenyl sulfoxide, 3k: IR: ν(cm⁻¹) 1515, 1345, 1026. ¹H NMR (90 MHz, CDCl₃) δ (ppm): 4.08-4.20 (m, 2H, CH₂), 7.23-8.08 (m, 9H, ArH). MS (m/z) 262 (M+1).

Benzyl 4-bromobenzyl sulfoxide, 3l: IR: ν(cm⁻¹) 2855, 1461, 1376, 1245, 1028, 722. ¹H NMR (90 MHz, CDCl₃) δ (ppm): 3.86-3.92 (m, 4H, CH₂), 7.20-7.52 (m, 9H, ArH). MS (m/z) 310 (M+1).

4-Anisyl methyl sulfoxide, 3m: IR: ν(cm⁻¹) 2852, 1460, 1374, 1247, 1045. ¹H NMR (90 MHz, CDCl₃) δ (ppm): 2.68 (s, 3H, SOCH₃), 3.84 (s, 3H, OCH₃), 7.02 (d, 2H, ArH), 7.54 (d, 2H, ArH). ¹³C NMR (22.5 MHz, CDCl₃) δ (ppm): 25.5, 44.1, 55.6, 115.0, 136.8, 162.1. MS (m/z) 171 (M+1).

(3-Methylbut-2-enylsulfinyl)benzene, 3n: IR: ν(cm⁻¹) 2852, 1731, 1630, 1460, 1374, 1247, 1044. ¹H NMR (90 MHz, CDCl₃) δ (ppm): 1.41 (s, 3H, CH₃), 1.71 (s, 3H, CH₃), 3.56 (d, 2H, CH₂), 5.07 (t, 1H, =CH), 7.41-7.62 (m, 5H, ArH). ¹³C NMR (22.5 MHz, CDCl₃) δ (ppm): 18.1, 26.0, 56.8, 111.2, 124.5, 129.0, 131.1, 136.6, 142.3. MS (m/z) 195 (M+1).

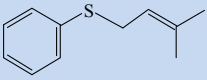
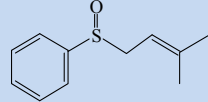
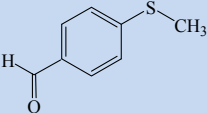
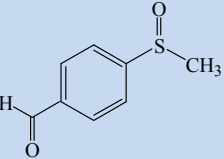
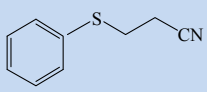
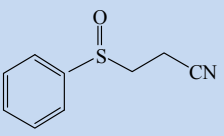
1-(4-(Methylsulfinyl)phenyl)ethanone, 3o: IR: ν(cm⁻¹) 2932, 2855, 1710, 1696, 1424, 1207, 1096, 1042, 773. ¹H NMR (90 MHz, CDCl₃) δ (ppm): 2.82 (s, 3H, CH₃), 7.78-7.96 (m, 4H, ArH), 10.12 (s, 1H, CHO). ¹³C NMR (22.5 MHz, CDCl₃) δ (ppm): 43.8, 124.5, 130.4, 138.3, 152.5, 190.8. MS (m/z) 169 (M+1).

3-(Phenylsulfinyl)propanenitrile, 3p: IR: ν(cm⁻¹) 2978, 2848, 2252, 1455, 1050, 1027, 755. ¹H NMR (90 MHz, CDCl₃) δ (ppm): 2.52-3.05 (m, 4H, CH₂), 7.58-7.60 (m, 5H, ArH). ¹³C NMR (22.5 MHz, CDCl₃) δ (ppm): 9.7, 50.0, 117.2, 123.6, 129.5, 131.7, 141.2. MS (m/z) 180 (M+1).

Table 1. Sulfoxidation of various sulfides using *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane, **1**^a.

Entry	Substrate 2	Product 3 ^b	Time (min)	Yield (%) ^c	Mp (°C)	
					Found	Reported
a			25	96	Colorless oil	Colorless oil [34]
b			20	98	70-72	70 [38]
c			24	96	130-132	135 [34]
d			28	95	120-122	124-126 [34]
e			26	96	142-144	144-146 [38]
f			18	93	32-34	31 [38]
g			40	98	Colorless oil	Colorless oil [29]
h			35	94	194-196	195 [34]
i			30	94	Colorless oil	Colorless oil [42]
j			35	92	Colorless oil	Colorless oil [43]
k			45	94	160-162	161-163 [37]
l			42	95	138-140	139-140 [37]
m			28	95	Colorless oil	Colorless oil [44]

Table 1 (Continued).

Entry	Substrate 2	Product 3 ^b	Time (min)	Yield (%) ^c	Mp (°C)	
					Found	Reported
n			35	93	Colorless oil	Colorless oil [44]
o			45	98	Colorless oil	Colorless oil [35]
p			35	96	Colorless oil	Colorless oil [35]

^a Conditions: Sulfide (1 mmol), Reagent 1 (1 mmol), CH₂Cl₂ (5 mL), Room temperature (rt).

^b The structures of the products were established from their physical properties and spectral (¹H, ¹³C NMR and IR) analysis and compared with the literature.

^c Isolated yield.

3. Results and Discussion

In this ongoing research on the synthesis of *gem*-dihydroperoxides and their use in various transformations [41], we became interested in examining the oxidative ability of *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane for selective conversion of sulfides into corresponding sulfoxides. This reagent was prepared based on our previously reported method [41], as a white crystalline solid in high yield (85%) from the reaction of acetylacetone with aqueous (30%) hydrogen peroxide in acetonitrile at room temperature using a catalytic amount of stannous chloride dehydrate (Scheme 1). As evident from the ¹H NMR spectrum, this compound probably assumes a *trans* geometry. In *trans* isomer, two C(4)-protons of the ring are symmetrically located on either side of the ring and will have similar chemical shifts. However, in *cis* isomer these C(4)-protons experience different chemical environments and expected to undergo a geminal proton-proton coupling with one another to result in two separate sets of doublets in ¹H NMR spectrum. Based on this explanation, a *trans* structure is desirable for 3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane compound since it exhibits a singlet at 2.67 ppm for two C(4)-protons in its ¹H NMR spectrum.

To the best of our knowledge, the present work presents the first example wherein *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane, **1**, has been found to act as an efficient oxidant for selectively converting sulphides, **2**, into the corresponding sulfoxides, **3**, without any detectable over-oxidation to sulfones (Scheme 2). The reactions proceed under mild and catalyst-free conditions at room temperature to yield the products in excellent yields (Table 1).

The effect of solvent and oxidant concentration on the reaction was studied using various solvents such as PhMe, MeOH, H₂O, CH₃CN, AcOH, THF and CH₂Cl₂ and different molar ratios of the oxidant **2**. According to the experimental results, dichloromethane was found to be the solvent of choice in terms of yields (95%) when one equimolar amount of the oxidant **1** is used at room temperature. No improvement in the reaction rate as well as the yield was observed by increasing the amount of the oxidant.

To develop the scope of reaction, a wide range of sulfides carrying different functional groups were subjected to sulfoxidation reaction under the optimized reaction conditions (Table 1). The reactions were conducted with very high

selectivity in relatively short reaction times (18-45 min) to provide the corresponding sulfoxides in excellent yields (92-98%). The over-oxidation of sulfides into sulfones was not observed and functional groups like hydroxy, aldehyde, olefin, nitrite and ester were also tolerated in this procedure. As shown in Table 1, it was observed that sulfides, irrespective of the presence of electron-withdrawing or -releasing groups, were oxidized equally well to sulfoxides.

It is important to note that, *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane operates quite safely in these reactions with no explosion as long as the reactions are conducted at room temperature. Also, this compound tends to smoothly decompose on standing at normal temperatures. However, like many other peroxidic compounds, *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane is expected to be potentially explosive. Therefore, as mentioned in the experimental section, special precautions in handling of this compound are required, particularly heating to higher temperatures should be avoided.

In conclusion, *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane has been newly prepared and utilized as a highly selective and highly active oxidant for oxidation of a wide variety of sulfides to sulfoxides under mild and catalyst-free conditions. The reactions proceeded with no significant over-oxidation to sulfones and also functional groups such as hydroxy, aldehyde, olefin, nitrile and ester remained unaffected. This protocol may be considered as environmentally friendly since no additional catalyst is necessary for the activation and the oxidant is regarded as a non-polluting reagent.

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