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SYNTHESIS OF SULFOXIDES BY OXIDATION OF THIOETHERS

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CONTENTS

1. INTRODUCTION

The oxidation of a thioether R_1—S—R_2 can yield either the corresponding sulfoxide R_1—SO—R_2 or the corresponding sulfone R_1—SO_2—R_2 or both, depending on the method used. Gentle oxidation to the sulfoxide alone requires highly selective methods which cannot usually be applied generally, whereas complete oxidation to the sulfone is much easier to achieve. Appropriate adjustment of the reaction conditions is usually sufficient.

We present here a review of current methods for obtaining sulfoxides from thioethers, including stereospecific methods.

The oxidation of dibenzyl sulfide to dibenzyl sulfoxide using nitric acid was first described by Märcker in 1865. Since then, interest in this synthetic step has never flagged, and in 1984 and 1985 over 100 papers were published on this topic.

2. OXIDATION OF THIOETHERS USING HYDROGEN PEROXIDE AND ITS DERIVATIVES

2.1. *Oxidation using Hydrogen Peroxide* (H_2O_2)

Hydrogen peroxide (H_2O_2), either alone or associated with various solvents or catalysts, is the most widely used oxidizing agent for oxidizing organic sulfides. The corresponding sulfoxide is obtained according to Eq. (1).

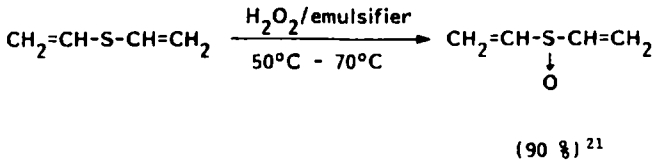
$$R_1\text{-}S\text{-}R_2 + H_2O_2 \longrightarrow R_1\text{-}\underset{\underset{O}{\downarrow}}{S}\text{-}R_2 + H_2O \qquad (1)$$

Oxidation can, however, proceed further to give the corresponding sulfone (Eq. 2).

$$R_1\text{-}S\text{-}R_2 + 2\,H_2O_2 \longrightarrow R_1\text{-}\overset{\overset{O}{\uparrow}}{\underset{\underset{O}{\downarrow}}{S}}\text{-}R_2 + 2\,H_2O \qquad (2)$$

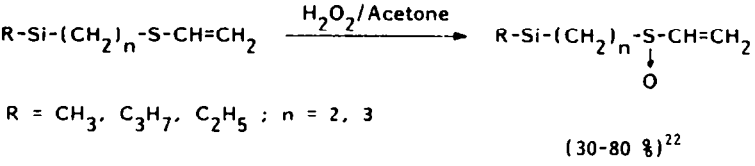
2.1.1. H_2O_2 *in various solvents*

In 1908, Gazdar and Smiles[1] and Hinsberg[2-4] used hydrogen peroxide in acetone or acetic acid to oxidize sulfides to sulfoxides at room temperature. This method has since been widely used to prepare a large number of aliphatic, aromatic, heterocyclic[5-19] and allyl[20] sulfoxides. Addition of an emulsifier improved yields for certain unsaturated sulfides[21] (Scheme 1).



Scheme 1.

Silyl vinyl sulfides[22] have also been oxidized to sulfoxides using hydrogen peroxide (Scheme 2).



Scheme 2.

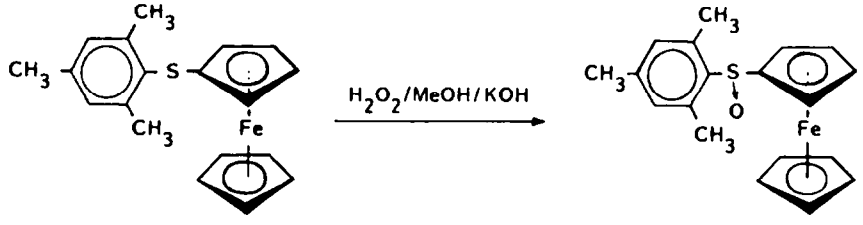
Other solvents have been used[23] including methanol,[24] ethanol,[25] ethyl acetate,[26] dimethylformamide (DMF)[27] and 2-alkoxyethanol.[28]

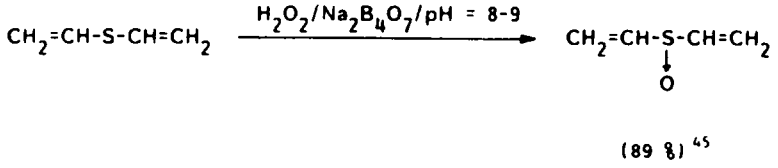
2.1.2. H_2O_2 *in the presence of inorganic acids*

Hydrogen peroxide has often been used in the presence of various inorganic acid catalysts, mainly perchloric,[29–36] hydrochloric[37] and sulfuric acid,[35,36,38–41] but also osmic (H_2OsO_4)[42] and molybdic acid ($H_2Mo_2O_7$)[43] in t-butanol.

2.1.3. H_2O_2 *in basic media*

Oxidation in basic media has been used for the synthesis of certain ferrocenyl sulfoxides[44] and vinyl sulfoxides[45] (Scheme 3).

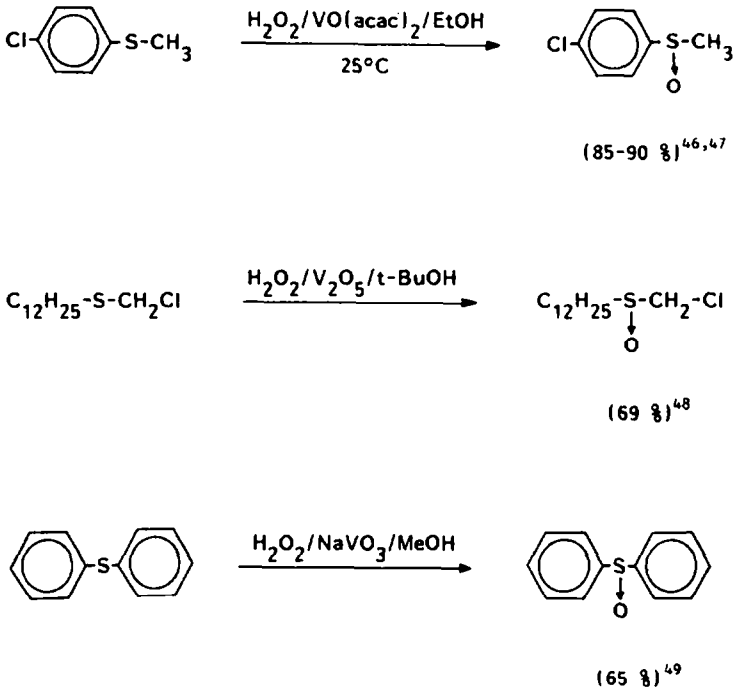




Scheme 3.

2.1.4. H_2O_2 *with other catalysts*

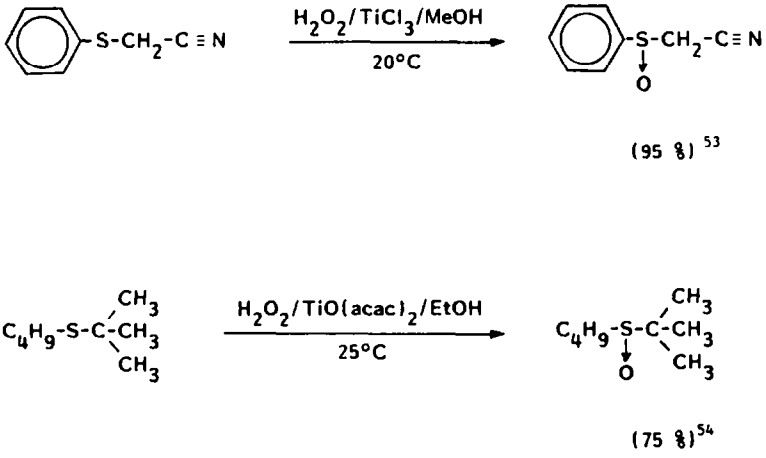
2.1.4.1. *Vanadium salts.* Various sulfides have been selectively oxidized to sulfoxides using hydrogen peroxide in the presence of vanadium salts such as vanadyl diacetylacetonate [VO(acac)$_2$],[46,47] vanadium pentoxide (V_2O_5)[48] and sodium metavanadate ($NaVO_3$)[49] (Scheme 4).



Scheme 4.

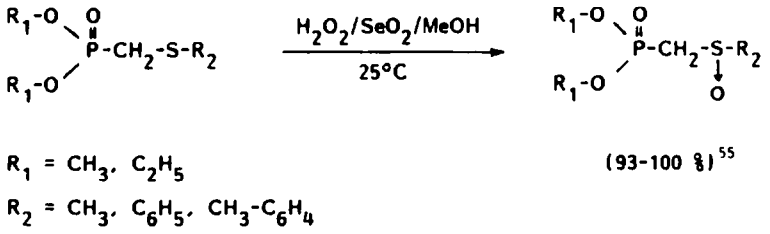
2.1.4.2. *Molybdenum salts.* Molybdenum salts have also been recently used as catalysts in conjunction with H_2O_2,[50,51] especially molybdyldiacetylacetonate [$MoO_2(acac)_2$], hexacarbonyl molybdenum [$Mo(CO)_6$] and molybdenum peroxide (MoO_5) in aqueous hexamethylphosphotriamide (MoO_5, HMPT/H_2O).[51,52]

2.1.4.3. *Titanium salts.* Japanese workers[53] have used titanium trichloride ($TiCl_3$) as a catalyst. The sulfoxides obtained were very pure and the yields were excellent (Scheme 5). Titanyldiacetylacetonate [$TiO(acac)_2$][54] has also been used (Scheme 5).



Scheme 5.

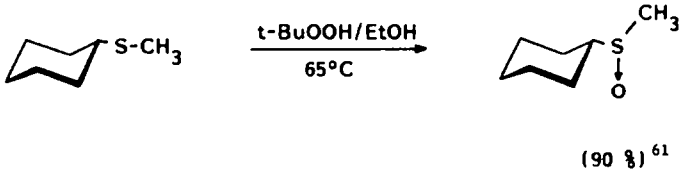
2.1.4.4. *Other catalysts.* Among other catalysts, selenium oxide (SeO_2)[55] (Scheme 6), peroxotungsten complex [$WO(O_2)_2$, HMPT/H_2O],[52] tungstic oxide (WO_3),[56] hexacarbonyltungsten [$W(CO)_6$],[57] Fenton's reagent (Fe^{2+}–H_2O_2)[58] and Milas' reagent [$(Me(O)_x$–H_2O_2][59] have been used.



Scheme 6.

2.2. *Oxidation of Thioethers using Hydroperoxides*

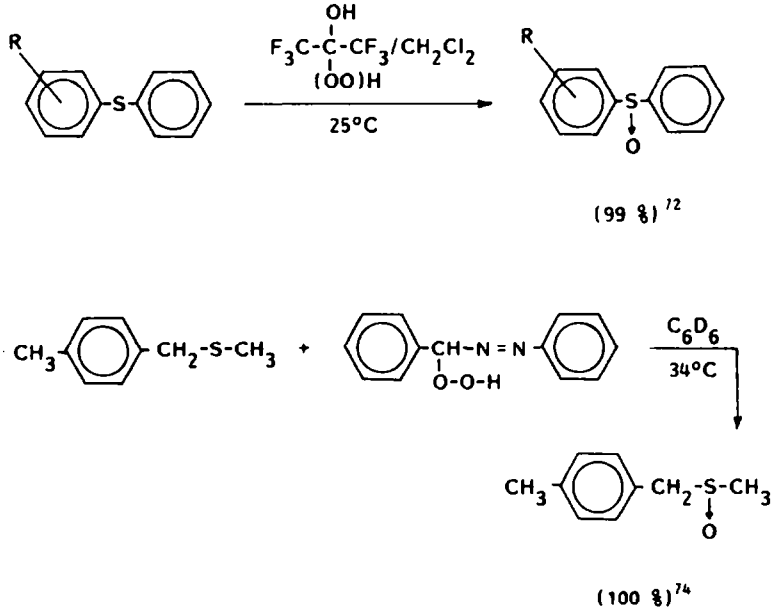
In 1954, Bateman and Hargrave[60,61] oxidized cyclohexyl methyl sulfide using terbutylhydroperoxide (t-BuOOH) and cyclohexenylhydroperoxide (C_6H_4—OOH) in various solvents. High yields of sulfoxide were obtained (Scheme 7).



Scheme 7.

This reaction was subsequently adapted by various workers. Conditions of temperature[62,63] and solvent[64] were modified, and catalysts[65–70] and an organic peroxide[71] added.

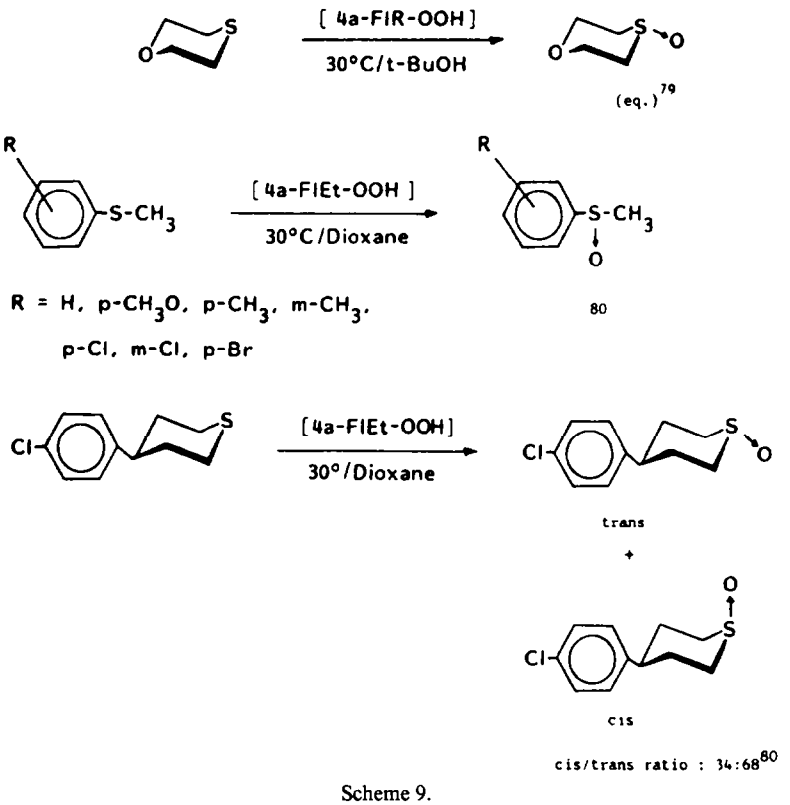
Recently, other hydroperoxides have been used. Ganem *et al.*[72] used 2-hexafluorohydroperoxy-2-propanol (HFHPP) to selectively oxidize diaryl sulfides to sulfoxides (Scheme 8). Baumstark *et al.* have used cyclic[73] and acyclic α-azohydroperoxides[74,75] (Scheme 8).



Scheme 8.

Bruice and co-workers[76–79] and Oae *et al.*[80] have used 4a-hydroperoxylumiflavins (4a-FlR—OOH) (Scheme 9).

Finally, Russian workers[81] have used organic hydrotrioxides (R—O_3H) as selective oxidizing agents to obtain sulfoxides from sulfides.



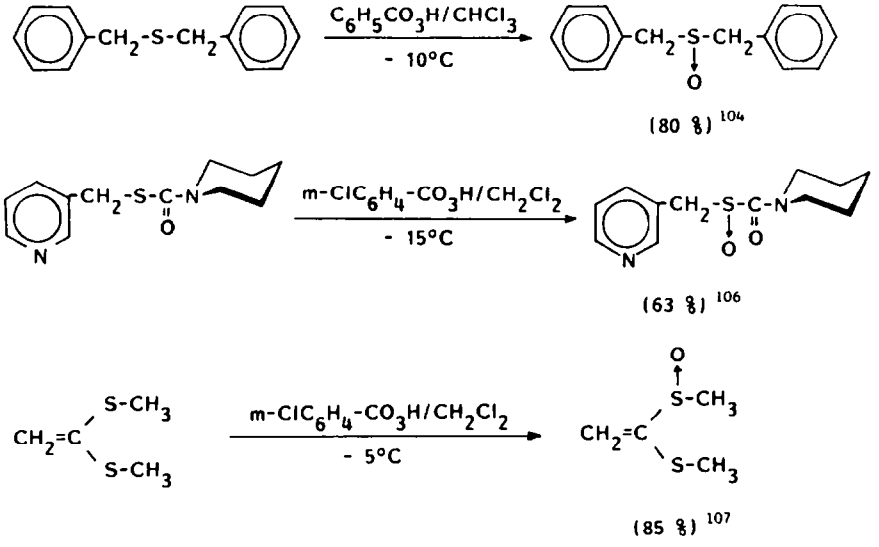
Scheme 9.

2.3. *Organic Peracids and Peroxides*

2.3.1. *Perbenzoic acid and its derivatives*

Lewin[82-85] was the first to describe (in 1928) the oxidation of sulfides to sulfoxides using perbenzoic acid ($C_6H_5CO_3H$) (Scheme 10). This method has since been widely used and the reaction conditions optimized.[86-97]

Metachloroperbenzoic acid (*m*-$ClC_6H_4CO_3H$) is one of the best selective oxidizing agents for oxidizing sulfides to sulfoxides. Ternay *et al.*[98,99] obtained sulfone-free sulfoxides using this reagent. Moreover, it has been used with a wide variety of substrates[100-103] including unstable unsaturated thioethers[104,105] multifunctional heterocyclic thioesters,[106] thioacetals[107] (Scheme 10), disulfides[108] and penicillins,[109] to mention only a few.

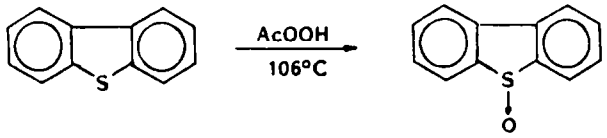


Scheme 10.

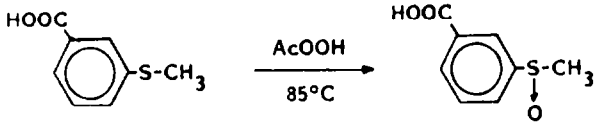
The only organic peroxide to have been successfully used to convert sulfides into sulfoxides is benzoyl peroxide $[(C_6H_5CO)_2O_2]$.[110,111] However, the reaction products include other sulfur-containing compounds such as polysulfides and thiosulfinates.

2.3.2. *Peracetic acid and its derivatives*

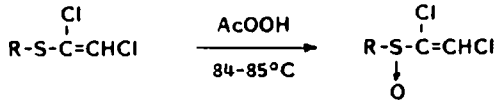
Peracetic acid (AcOOH) has been used to prepare a very large number of sulfoxides.[112,127] Examples of selective oxidation are given in Scheme 11.



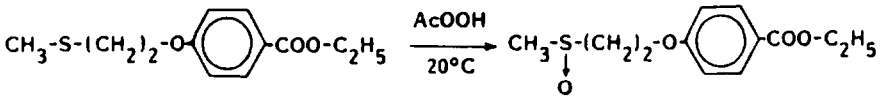
(55,5 %)[114]



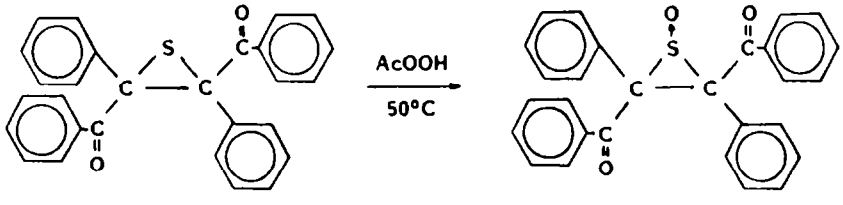
(65 à 80 %)[115]



(76 %)[116]



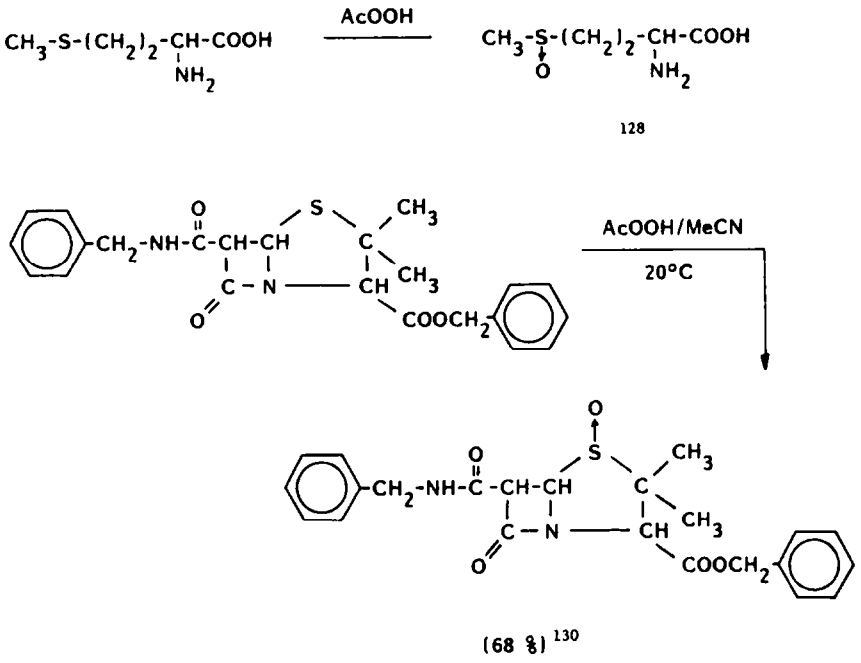
(93,2 %)[118]



(65 %)[120]

Scheme 11.

Methionine S-oxide[128,129] and penicillin S-oxides[130,131] have been obtained using this reagent (Scheme 12).

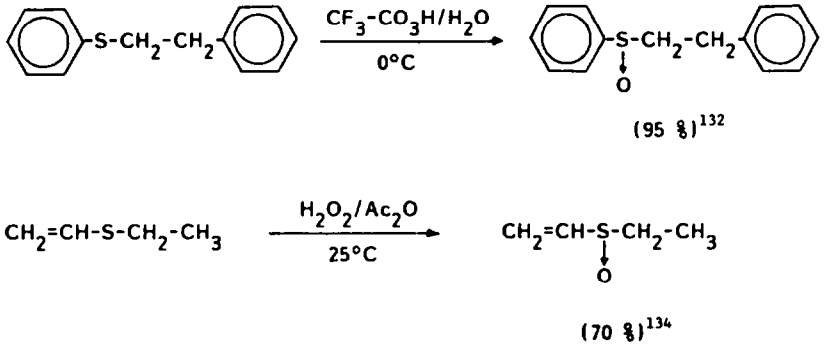


128

(68 %) [130]

Scheme 12.

Pertrifluoroacetic acid (CF_3CO_3H)[132] and hydrogen peroxide in acetic anhydride[133–135] have also been used (Scheme 13).



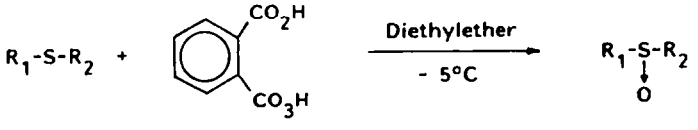
(95 %) [132]

(70 %) [134]

Scheme 13.

2.3.3. *Other organic peracids*

Monoperphthalic acid has been found to be an efficient and convenient reagent for the sulf-oxidation of a variety of sulfides[136] (Scheme 14).



R_1 = C_6H_5-CH_2, n-C_4H_9, t-C_4H_9

R_2 = C_6H_5-CH_2, p-$CH_3C_6H_4$-CH_2, n-C_4H_9, s-C_4H_9, t-C_4H_9

(89-97 %) [136]

Scheme 14.

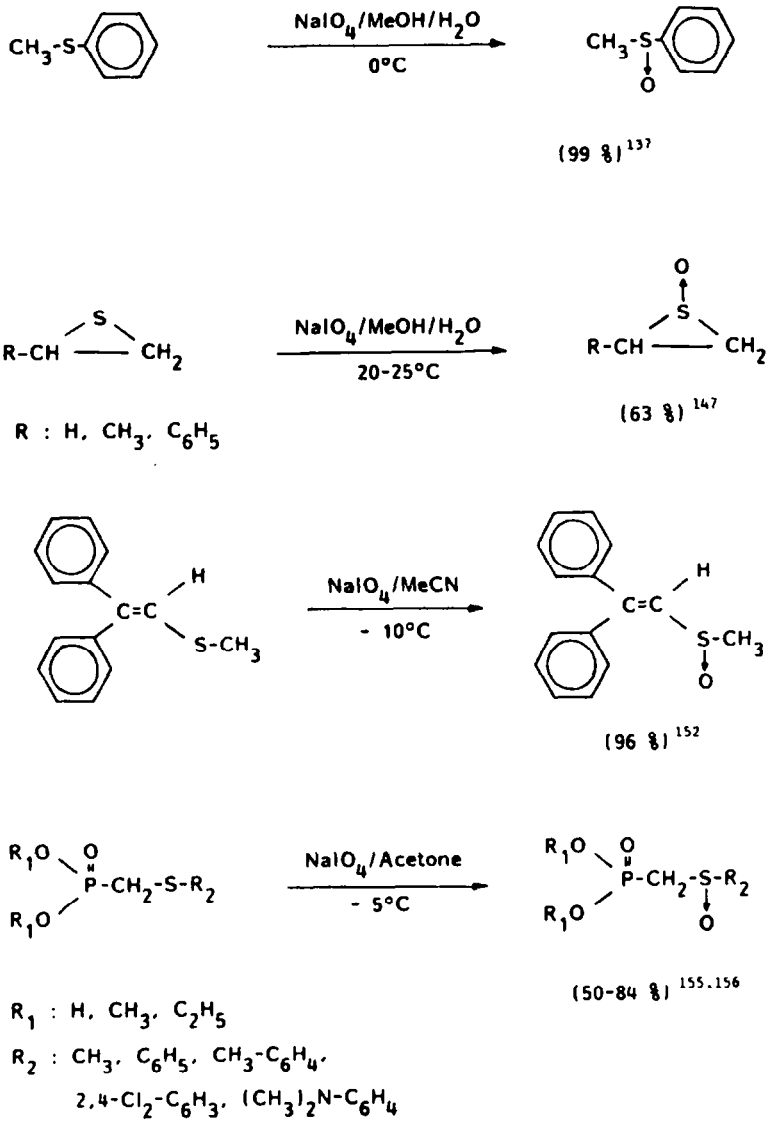
Finally, other peracids such as percamphoric and α-cyclohexyl perpropanoic acids have been used as chiral reagents to obtain optically active sulfoxides. These special applications are dealt with in Section 9.

3. OXIDATION OF THIOETHERS USING HALOGEN DERIVATIVES

3.1. *Halogen Oxide Derivatives*

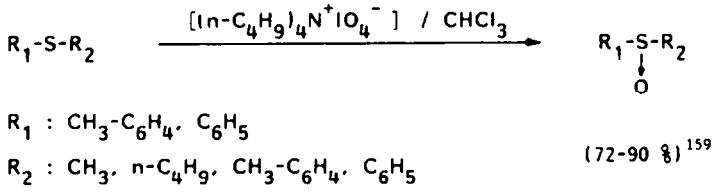
3.1.1. *Sodium metaperiodate*

In 1961, Leonard and Johnson[137,138] selectively prepared sulfoxides by oxidation of thioethers using cold sodium metaperiodate ($NaIO_4$). This reaction (Scheme 15) has remained one of the most widely-used and best methods for this conversion.[139–158]



Scheme 15.

Various modifications have been made to this method. Tetrabutylammonium periodate $[(n\text{-}C_4H_9)_4N^+IO_4^-]$ in chloroform with heating[159] has been used (Scheme 16), while Liu and Tong[160,161] improved selectivity by using sodium metaperiodate on an alumina-based support.

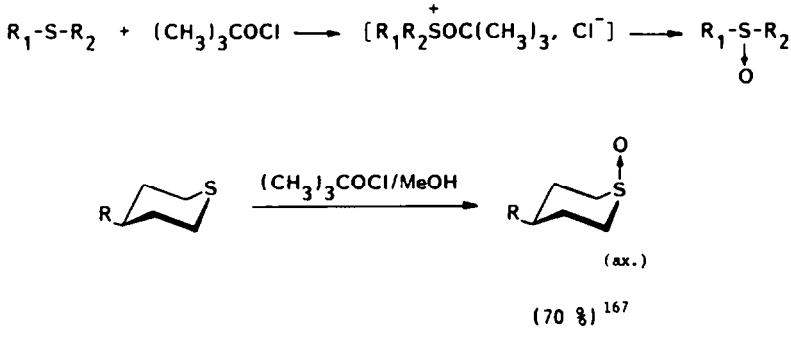


Scheme 16.

This method can also give asymmetric sulfoxides[161] if the solvent used is chiral. Sodium metaperiodate in the presence of bovine serum albumin[162] also gives asymmetric products (see Section 9). Metaperiodate (IO_4^-) on resin[163,164] enables gentle oxidation of sulfides to be carried out by elution.

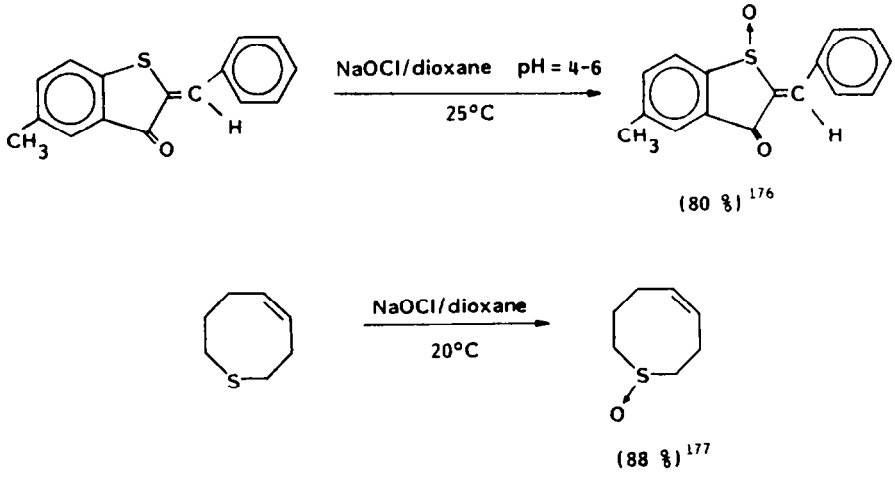
3.1.2. *Hypochlorites*

3.1.2.1. *Terbutyl hypochlorite.* In 1964, Skell and Epstein[165] and Gritter and Carey[166] selectively oxidized certain thioethers using terbutyl hypochlorite [$(CH_3)_3COCl$] at room temperature (Scheme 17). This method has subsequently been widely used.[167-172]



Scheme 17.

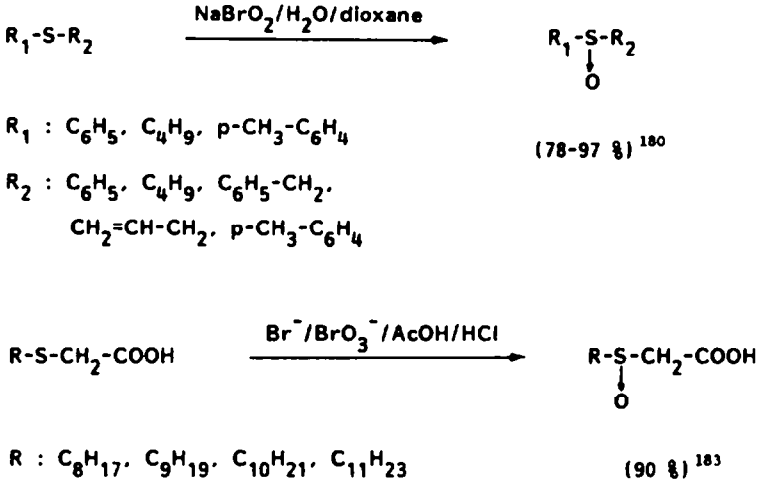
3.1.2.2. *Sodium hypochlorite.* In 1928 Wood and Travis[173] oxidized n-heptylsulfide using sodium hypochlorite (NaOCl), though rather unselectively, since a significant amount of sulfone was obtained along with the required sulfoxide. However, sodium hypochlorite in various solvents has been used successfully to oxidize cyclic sulfides, vinyl sulfides and aryl sulfides to corresponding sulfoxides (Scheme 18).



Scheme 18.

3.1.3. *Bromites and bromates*

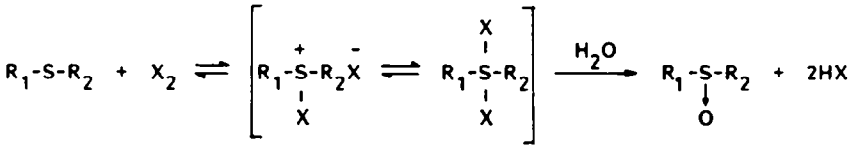
Some sulfides have been oxidized to sulfoxides using bromous acid,[179] bromites (BrO_2^-)[180] and bromate/bromide mixtures (BrO_3^-/Br^-)[181–183] (Scheme 19).



Scheme 19.

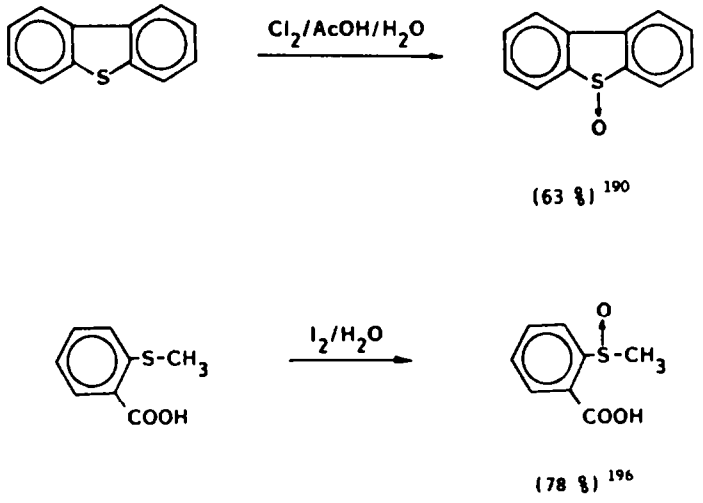
3.2. *Halogens*

Aqueous solutions of halogens have been used since 1907 to oxidize thioethers.[184–187] The reaction involves an intermediate which hydrolyses to give the sulfoxide (Scheme 20).



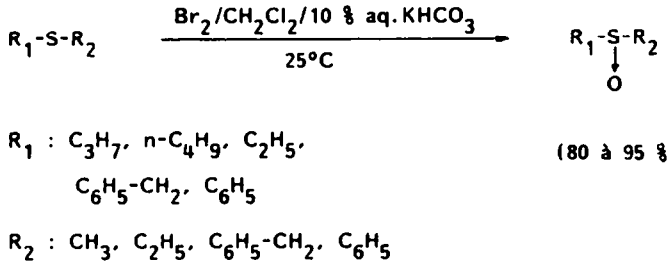
Scheme 20.

This method has been widely used and studied.[184–188] Chlorine,[189–191] bromine[192] and iodine have all been used (Scheme 21).



Scheme 21.

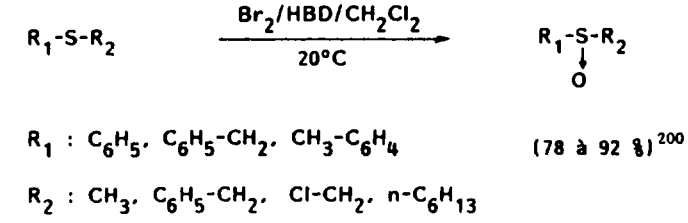
In 1979, Drabowicz *et al.*[197] prepared sulfoxides by oxidation of thioethers using bromine and chlorine in a heterogeneous system consisting of aqueous potassium hydrogen carbonate and dichloromethane (Scheme 22).



Scheme 22.

This highly convenient method has been used to obtain a very wide variety of sulfoxides.[198,199]

Ueno *et al.*[200] have used a novel and highly selective method using bromine in the presence of hexabutyldistannoxane $[(C_4H_9)_3Sn]_2O$ (HBD) in an anhydrous medium (Scheme 23).

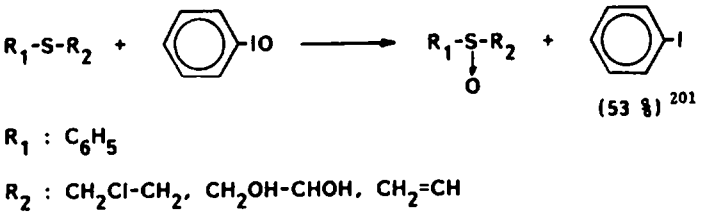


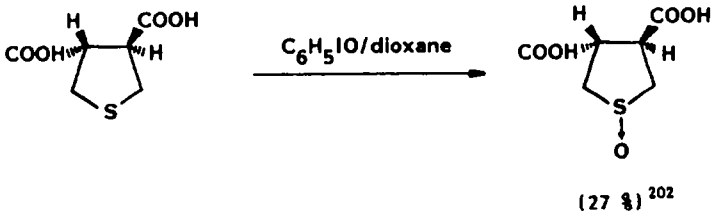
Scheme 23.

3.3. *Iodosobenzene and its Derivatives*

3.3.1. *Iodosobenzene*

Ford-Moore[201] first described the use of iodosobenzene (C_6H_5IO) to selectively oxidize β-hydroxy and vinyl phenyl sulfides in 1949 (Scheme 24). This reagent has since been used to prepare cyclic sulfoxides[202] and in model metalloporphyrin systems[203,204] (see Section 11).





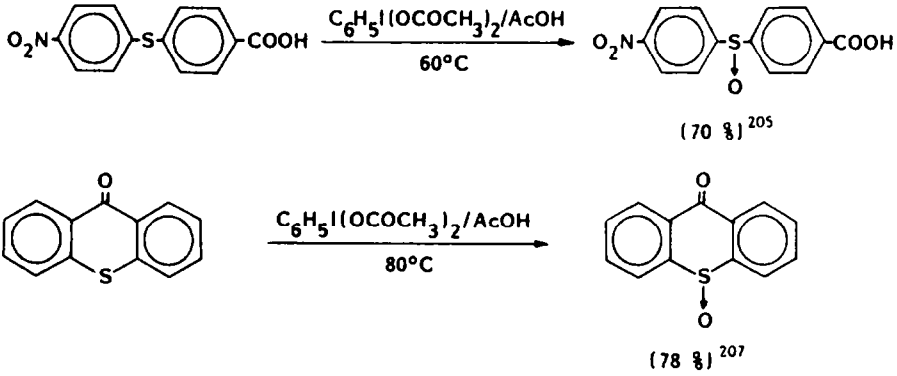
Scheme 24.

3.3.2. *Iodobenzene diacetate (diacetoxyiodobenzene)*

This reagent $[C_6H_5I(OCOCH_3)_2]$[205–207] was used by Szmant and co-workers[205–207] to oxidize *p*-nitrophenylthiobenzoic acid, various aryl sulfides and cyclic sulfides (Scheme 25).

The reaction mechanism and kinetics have been studied by several workers.[208–212]

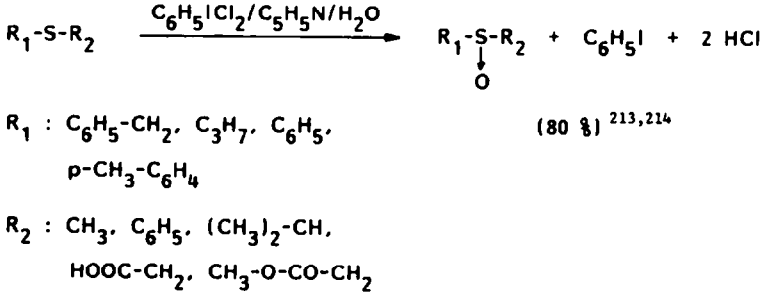
Recently, the sulfoxidation of diphenyl sulfide using a closely-related compound, bis(trifluoroacetoxy)iodobenzene $[C_6H_5I(OCOCF_3)_2]$, was reported.[436]



Scheme 25.

3.3.3. *Iodobenzene dichloride*

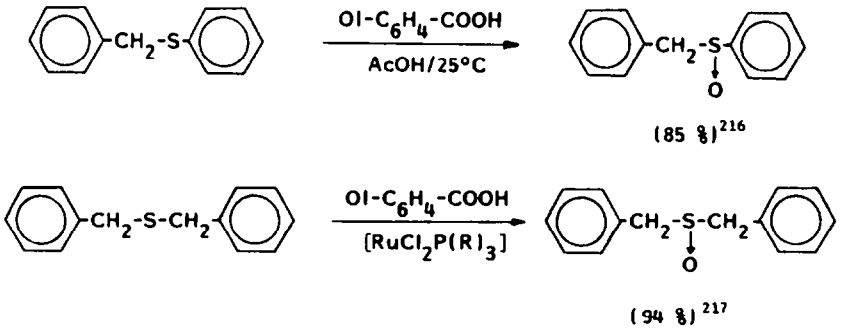
Montanari and co-workers[213–215] have used iodobenzene dichloride ($C_6H_5ICl_2$) in aqueous pyridine to oxidize a wide variety of aliphatic, aromatic and heterocyclic sulfides to their corresponding sulfoxides (Scheme 26).



Scheme 26.

3.3.4. *m-Iodosylbenzoic acid*

Recently, *m*-iodosylbenzoic acid (*m*-OI—C_6H_4—COOH) either alone[216] or in the presence of a catalyst, ruthenium dichlorotriphenylphosphine $[RuCl_2[P(C_6H_5)_3]]$[217] has been used to oxidize aryl sulfides to sulfoxides with excellent yields (Scheme 27).



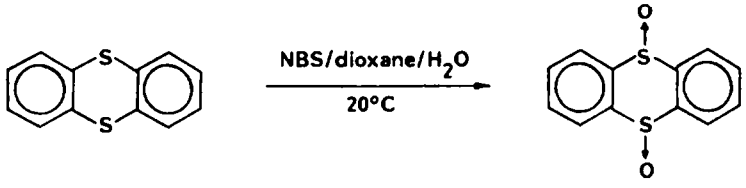
Scheme 27.

3.4. N-*Halogenated Derivatives*

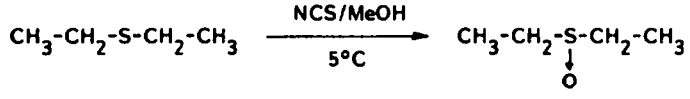
3.4.1. N-*Bromosuccinimide and* N-*chlorosuccinimide*

In 1964, Oae and co-workers[218] oxidized several cyclic sulfides to sulfoxides using N-bromosuccinimide (NBS) (Scheme 28).

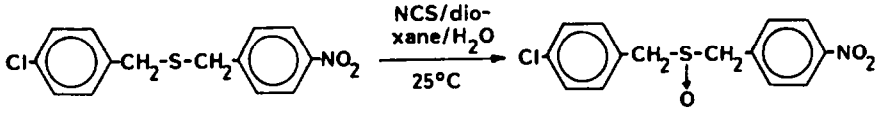
This method was subsequently used by Harville and Reed[219] who used both N-bromosuccinimide (NBS) and N-chlorosuccinimide (NCS), and by Tullen and Smith[220] who obtained excellent results with (NCS) (Scheme 28).



(30 %) [218]



(73 %) [219]

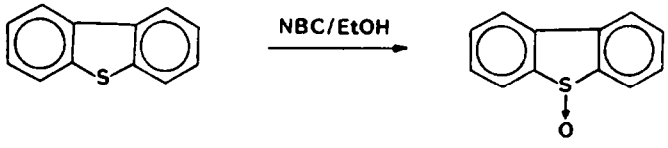


(81 %)[220]

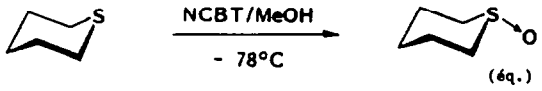
Scheme 28.

3.4.2. *Other* N-*halogenated derivatives*

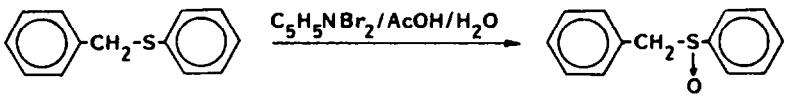
Numerous other N-halogenated reagents have been used to oxidize sulfides to sulfoxides, including N-bromocaprolactam (NBC),[221] N-chlorobenzothiazole (NCBT),[222] N-chloro-6,6-nylon,[223] chloramine B,[224] chloramine T and bromamine T[225–228] and various aromatic N-bromoamines ($ArNBr_2$)[229] such as N-bromopyridine[229] and N-brominated Tröger's base (BT-$2Br_2$)[230] (Scheme 29).



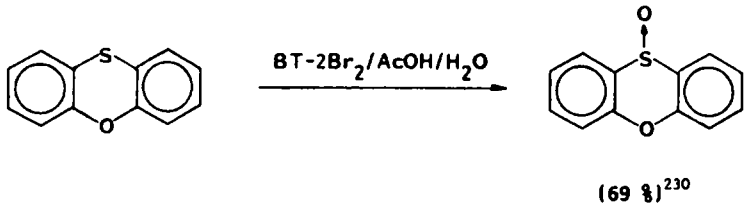
(70 %) [221]



(98 %) [222]



(65 %)[229]

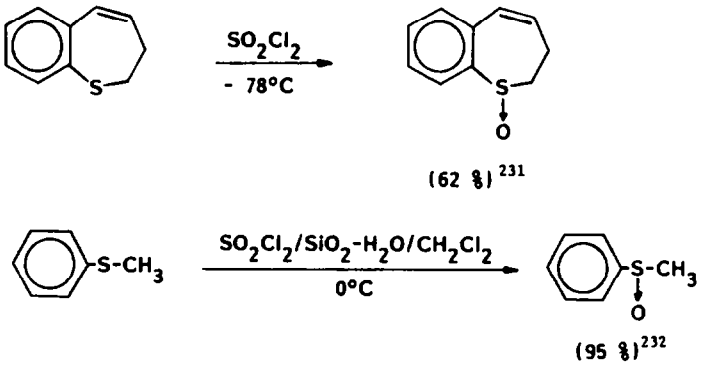


Scheme 29

3.5. *Sulfuryl Chloride (Sulfonyl Chloride)*

Traynelis *et al.*[231] have prepared sulfoxides by oxidation of sulfides using sulfonyl chloride (SO_2Cl_2) at low temperature.

Hojo and co-workers[232,233] improved this method by carrying out the reaction in the presence of silica gel (Scheme 30).

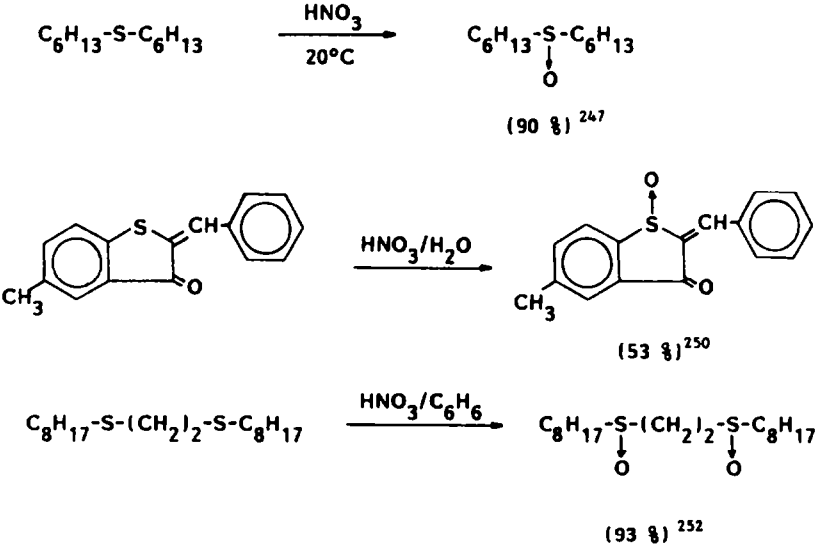


Scheme 30.

4. OXIDATION OF THIOETHERS USING NITROGEN OXIDE DERIVATIVES

4.1. *Nitric Acid*

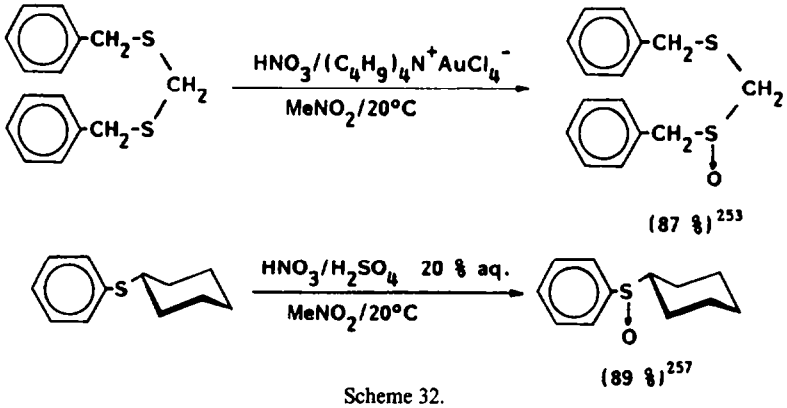
Nitric acid was the very first oxidizing agent used to convert sulfides into sulfoxides. In 1865, Märcker used this reagent to oxidize dibenzylsulfide to dibenzylsulfoxide.[234] This method was subsequently used to prepare a large number of alkyl and aryl sulfoxides[236–252] (Scheme 31).



Scheme 31.

Gasparrini *et al.*[253] have optimized this method using a novel catalyst, tetrabutylammonium tetrachloroaurate $[(C_4H_9)_4N^+AuCl_4^-]$. The synthesis of highly pure multifunctional sulfoxides was made possible by this method[254-256] (Scheme 32).

Recently, Gasparrini *et al.*[257] reported a biphasic process for the sulfoxidation of alkyl and aryl sulfides by nitric and sulfuric acids (Scheme 32).

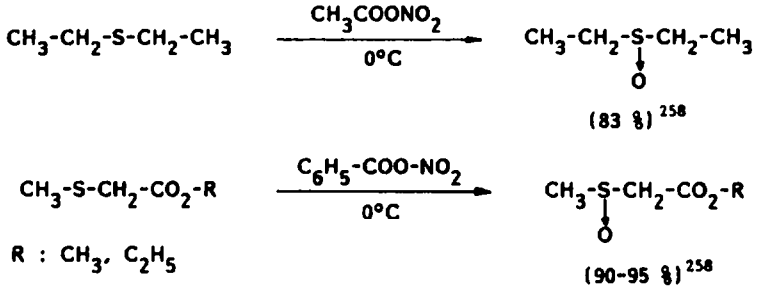


Scheme 32.

4.2. *Nitrates*

4.2.1. *Acyl nitrates*

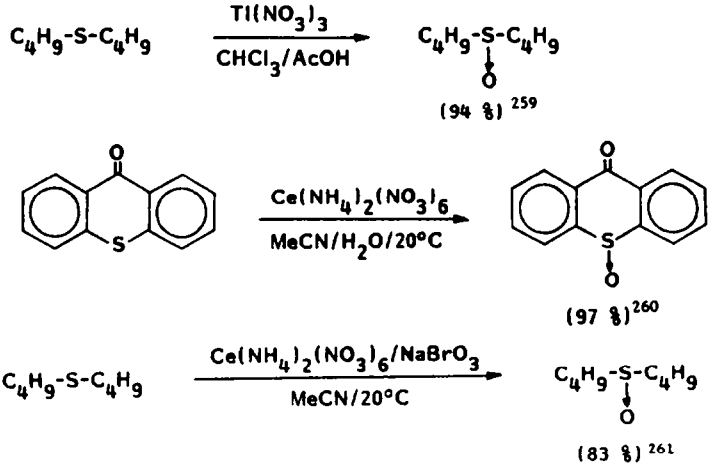
In 1976, Louw *et al.*[258] described the oxidation of alkyl sulfides to sulfoxides using acetyl and benzoyl nitrates at low temperature (Scheme 33). This reaction is highly selective since no sulfone formation occurred even with an excess of oxidizing agent.



Scheme 33.

4.2.2. *Other nitrates*

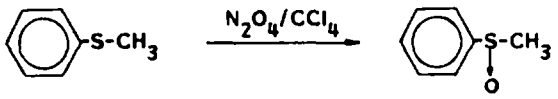
The synthesis of various sulfoxides has been achieved with excellent yields by the oxidation of sulfides using thallium[259] and cerium ammonium[260,261] nitrates (Scheme 34).



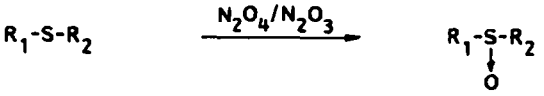
Scheme 34.

4.3. *Dinitrogen Tetroxide*

Pummerer[262] in 1910 was the first to use dinitrogen tetroxide to oxidize organic sulfides. The reaction was subsequently widely used[112,263-273] and underwent a large number of modifications as regards conditions (Scheme 35).

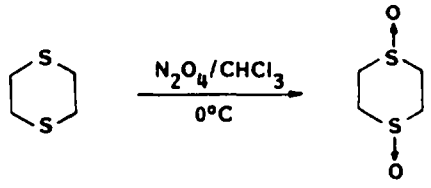


(95 %)[263]

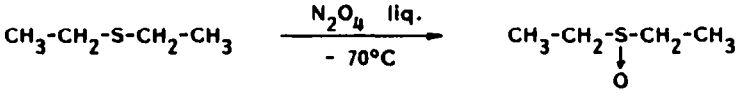


$R_1 = R_2 = CH_3,\ C_2H_5,\ n\text{-}C_3H_7$

(78 %)[264]



(98 %)[267]



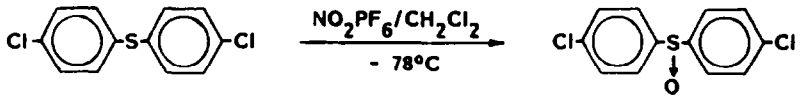
(90 %)[269]

Scheme 35.

4.4. *Other Nitrogen Oxide Derivatives*

In 1979 Olah *et al.*[274] used nitronium hexafluorophosphate (NO_2PF_6) in dichloromethane at low temperature ($-78°$) to oxidize a series of alkyl, aryl and arylalkyl sulfides (Scheme 36).

Recently, Tovrog *et al.*[275] described the oxidation of thioethers using a nitritocobalt complex $[Co(NO_2)_6]^{3-}$ in the presence of a Lewis acid. The reaction is very selective and high yields of sulfoxides are obtained.



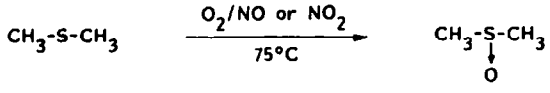
(95 %)[274]

Scheme 36.

5. OXIDATION OF THIOETHERS USING OXYGEN, OZONE AND BY OXYGEN TRANSFER

5.1. *Oxygen*

One method, described by Smedslund[276–279] and subsequently used by other workers,[280,281] involves oxidizing sulfides in the liquid or gas phase using oxygen or air in the presence of nitric oxide or nitrogen dioxide.[282] Sulfoxide yields are generally high (Scheme 37).



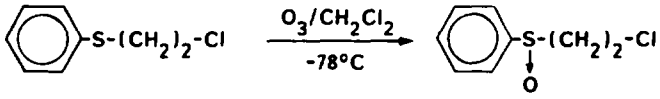
(75-95 %)[279]

Scheme 37.

Bateman and co-workers[283–286] studied the oxidation of thioethers using oxygen at temperatures between 45° and 75° and in the presence of various catalysts. Further modifications have been made to this method,[287–290] and recently catalysts such as ruthenium complexes,[291,292] metal acetylacetonates[293] and vanadium(V) oxide[294] have been used to obtain highly pure aryl sulfoxides.

5.2. *Ozone*

The oxidation of thioethers to sulfoxides with ozone was first described in 1942 by Böhme and Fischer[295] and was subsequently widely used.[296–299] One procedure[300] involves bubbling ozone through a solution of sulfide in methylene chloride at −78° (Scheme 38). High selectivity is achieved in this way.



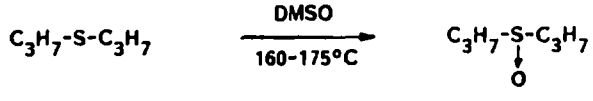
(74 %)[300]

Scheme 38.

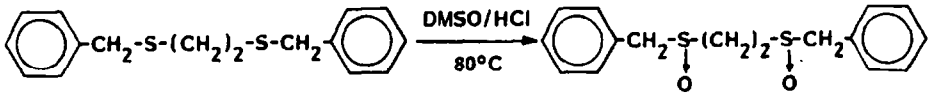
5.3. *Oxidation by Oxygen Transfer*

In 1955, Barnard[301] oxidized a sulfide by oxygen transfer from a sulfoxide labelled with ^{35}S.

This reaction, which was reported independently by Bordwell and Pitt[302] in the same year, was subsequently used by several other workers[303–305] with various modifications (Scheme 39).



(59 %)[303]

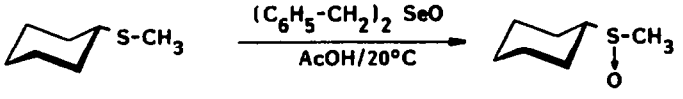


(82 %)[304]

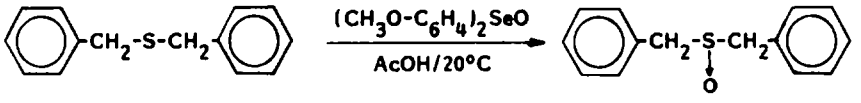
Scheme 39.

Using an analogous reaction, Barnard and Woodbridge[306] in 1959 prepared sulfoxides by oxidation of sulfides by oxygen transfer from a selenoxide (R_1—SeO—R_2) in acetic acid (Scheme 40).

Recently, Ogura *et al.*[307] have shown this oxidation to be highly selective.



(56 %)[306]



(89 %)[307]

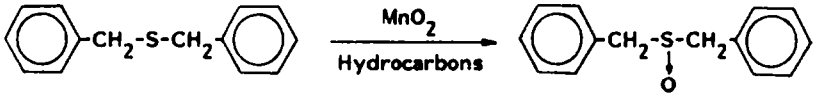
Scheme 40.

6. OXIDATION OF THIOETHERS USING OTHER OXIDIZING AGENTS

6.1. *Oxides, Peroxides and Peracids*

6.1.1. *Inorganic oxides and peroxides*

Oxides of manganese (MnO_2),[308] selenium (SeO_2)[309] and ruthenium (RuO_4)[310] have been used to prepare certain sulfoxides (Scheme 41). However, these reagents are not very selective, and corresponding sulfones are also formed.

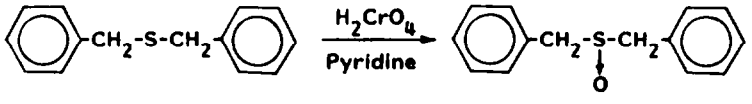


(71 %)[308]

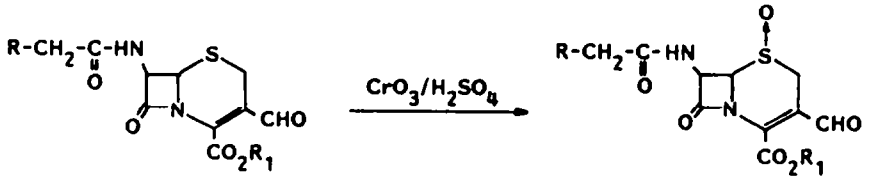
Scheme 41.

6.1.2. *Inorganic acids and peracids and their salts*

6.1.2.1. *Chromic acid and its derivatives.* Knoll[311] in 1926 used nascent chromic acid (H_2CrO_4) to oxidize certain sulfides. The rather low selectivity of this method was improved by carrying out the reaction in pyridine[308,312] (Scheme 42). Similarly, a mixture of chromic anhydride (CrO_3) and sulfuric acid was used to oxidize selectively a cephalosporin to its S-monooxide[313] (Scheme 42).



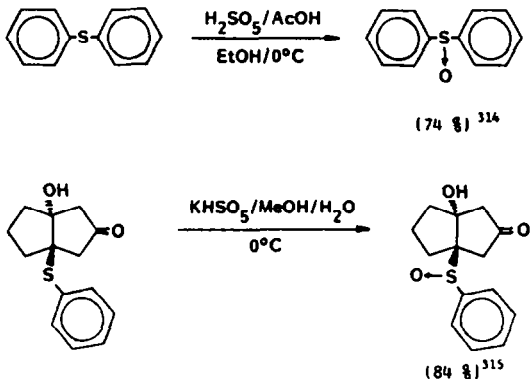
(71 %)[312]



313

Scheme 42.

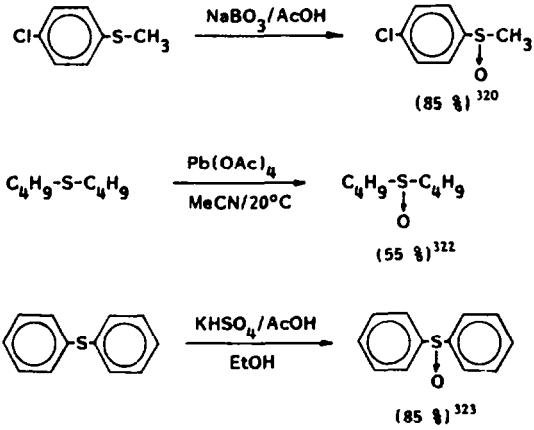
6.1.2.2. *Peroxysulfuric acid and its derivatives.* Peroxymonosulfuric acid (H_2SO_5) (Caro's acid)[314] and its salts[315] have been used to prepare certain sulfoxides (Scheme 43). Peroxydisulfuric acid ($H_2S_2O_8$) salts[316,317] and those of peroxydiphosphoric acid ($H_4P_2O_8$)[318] have also been used.



Scheme 43.

6.2. *Other Inorganic Salts*

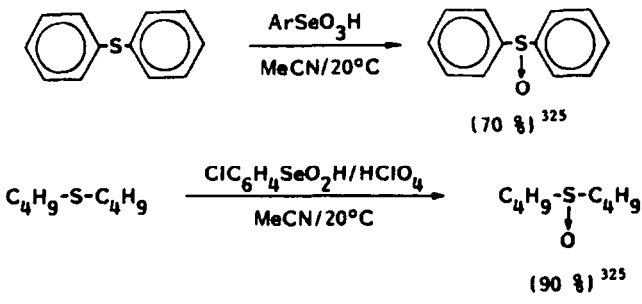
Among others, sodium permanganate ($NaMnO_4$),[319] sodium perborate ($NaBO_3 \cdot nH_2O$),[320] lead tetraacetate [$Pb(AcO)_4$][321,322] and potassium bisulfate ($KHSO_4$)[323] have all been used (Scheme 44).



Scheme 44.

6.3. *Benzene Seleninic and Selenonic Acids*

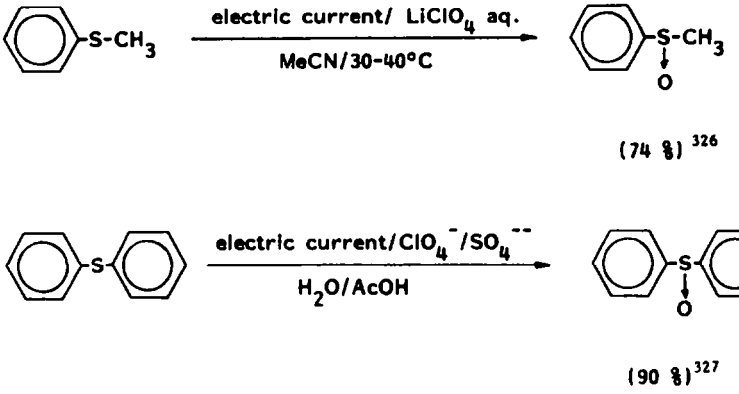
Aryl selenonic acids such as benzene selenonic acid ($C_6H_5SeO_3H$)[324] and its substituted derivatives[325] have been used for the sulfoxidation of various sulfides. Comparable results have been obtained with aryl seleninic acids in the presence of strong acids[325] (Scheme 45).



Scheme 45.

7. ELECTROCHEMICAL OXIDATION OF THIOETHERS

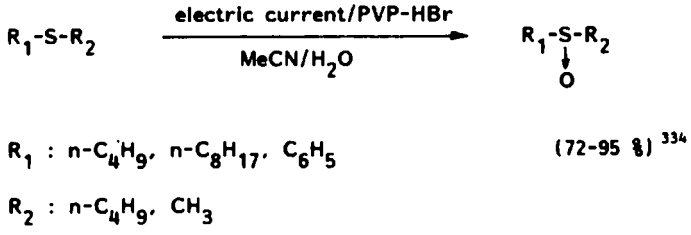
Among the less common methods of oxidation of sulfides is electrolytic oxidation carried out in various electrolyte solutions[326–331] (Scheme 46). In spite of several improvements, this method has remained rather unselective and often yields mixtures of sulfoxides and sulfones.[332,333]



Scheme 46.

A highly selective electrolytic oxidation method has however been recently reported.[334] Electric current passes through a bath of 4-polyvinylpyridine hydrobromide (PVP-HBr) and sulfide.

Only sulfoxide is obtained (Scheme 47).



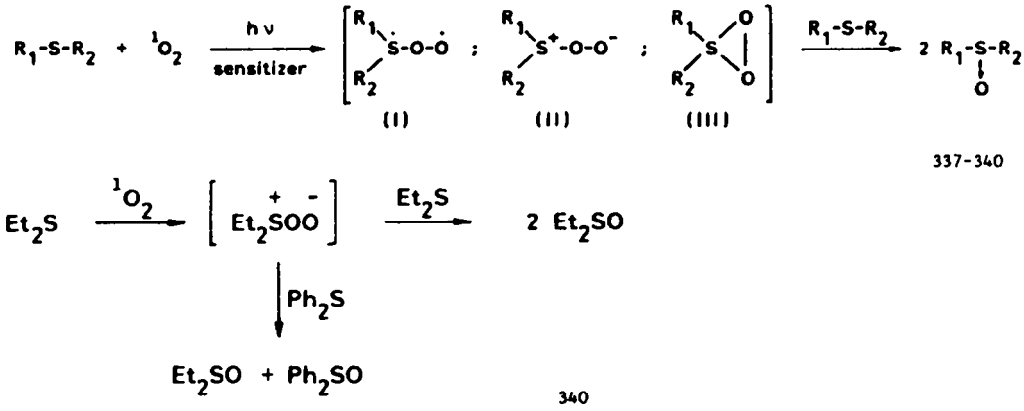
Scheme 47.

8. PHOTOCHEMICAL OXIDATION OF THIOETHERS

Photooxidation of sulfides was first described by Schenck and co-workers[335,336] in 1963. A number of intermediates have been suggested to explain the mechanism of the reaction.

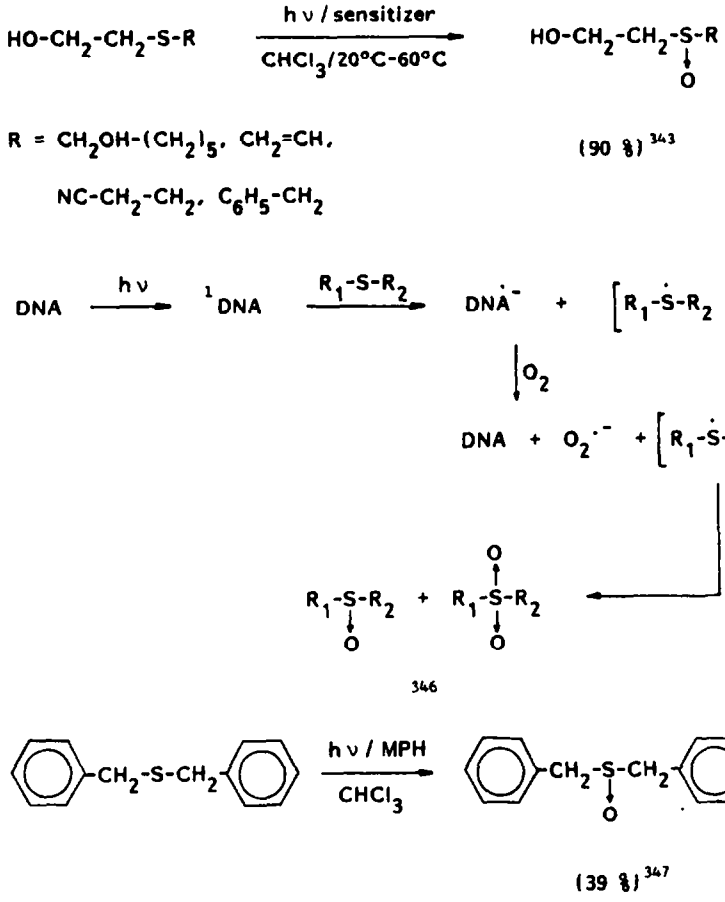
Gollnick[337] has proposed an intermediate diradical of type (I) which reacts with a second sulfide molecule giving two sulfoxide molecules.

Foote and co-workers[338–340] have postulated the intermediate formation of a persulfoxide (II) or a highly reactive cyclic peroxide (III) (Scheme 48).



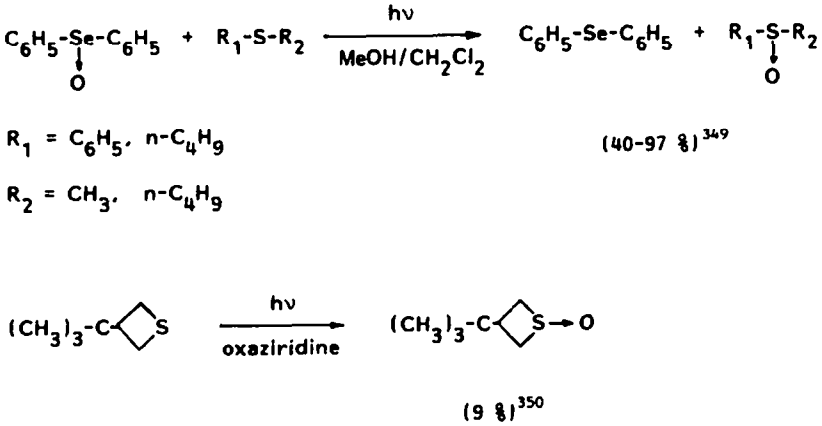
Scheme 48.

A very simple method for the photosynthesis of aliphatic sulfoxides has been proposed, involving UV irradiation of hexane solutions of sulfides.[341,342] Other work describes the photooxidation of sulfides with high yields by irradiation with a mercury vapour lamp of thioether solutions containing photosensitizer such as bengal pink or phloxine,[343,344] methylene blue,[345] 9,10-dinitrile anthracene (DNA),[346] 10-methyl phenothiazine (MPH)[347] and titanium dioxide[348] (Scheme 49).



Scheme 49.

Novel oxidation methods using photochemically-induced oxygen transfer from a selenoxide[349] or certain aromatic N-oxides and oxaziridines[350] have also been described (Scheme 50).



Scheme 50.

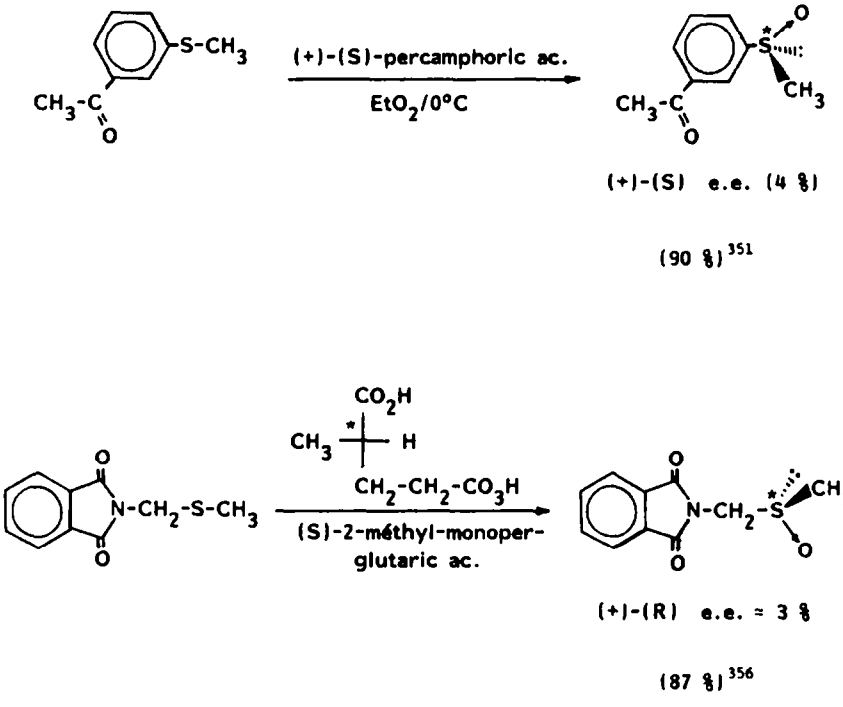
9. SYNTHESIS OF CHIRAL SULFOXIDES BY CHEMICAL OXIDATION

The synthesis of chiral sulfoxides can be achieved by both chemical and microbiological means. However, the optical purity of the products of chemical oxidation is much lower than that of products obtained by microbiological methods.

9.1. *Chiral Organic Peracids*

The first examples of synthesis of chiral sulfoxides by asymmetric induction were described by Balenović *et al.*[351] and Mayr *et al.*[352,353] in 1960. The substrates were alkyl aryl sulfides and the reagent was (+)-(*S*)-percamphoric acid[354,355] (Scheme 51).

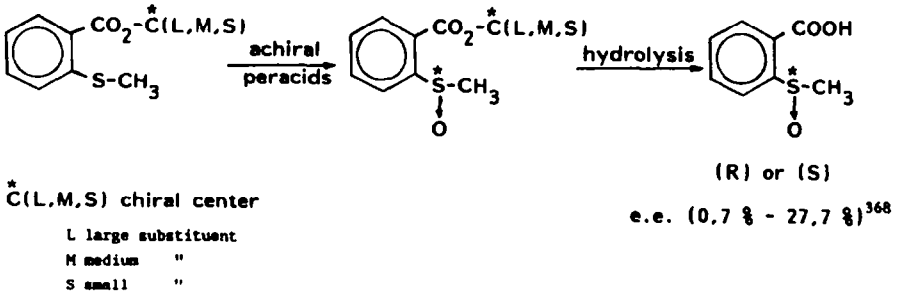
Other chiral mono- and dicarboxylic peracids were subsequently used such as monoperglutaric acids[356] (Scheme 51). (+)-α-cyclohexylperpropionic acid,[357] perhydratropic acid,[358] 1-phenylperpropionic acid[359] and others.[360–363] The mechanism of asymmetric oxidation by these bulky peracids can be understood in terms of Prelog's model.[364]



Scheme 51.

In certain cases, (+)-(*S*)-percamphoric acid by preferential oxidation of one enantiomer of a racemic sulfoxide, can give a mixture of a sulfone and a chiral sulfoxide.[365,366] In this way the final product is enriched in the sulfoxide which is the less easily oxidized by the asymmetric reagent.

A sulfide containing a chiral centre can be oxidized by achiral peracids to give a chiral product[367,368] (Scheme 52).

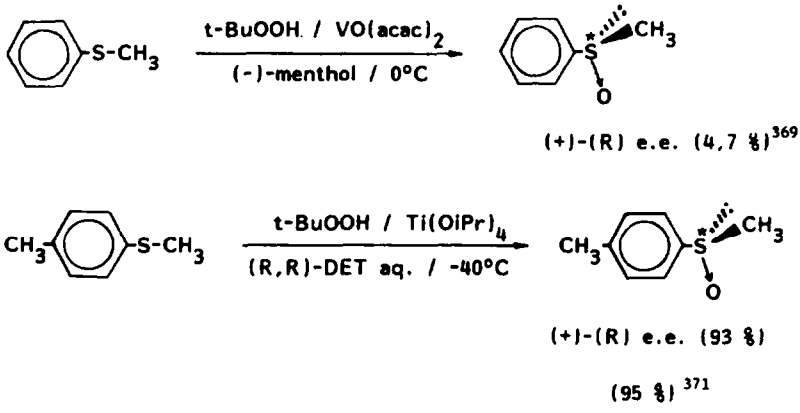


Scheme 52.

9.2. *Oxidation of Thioethers in Chiral Solvents*

Oxidation of sulfides by terbutyl hydroperoxide (t-BuOOH) activated by various catalysts in chiral solvents such as (−)-menthol, (−)-borneol and (−)-octanol can yield a mixture of chiral sulfoxides.[369,370] However, with this method, the enantiomeric excess (e.e.) of the favoured sulfoxide is always small (< 10%) (Scheme 53).

Asymmetric induction is improved if the terbutyl hydroperoxide is associated with Sharpless reagent or titanium tetraisopropoxide [$Ti(OiPr)_4$] in an aqueous solution of (*R*,*R*)-diethyltartrate [(*R*,*R*)-(DET)][370–372,437] (Scheme 53).



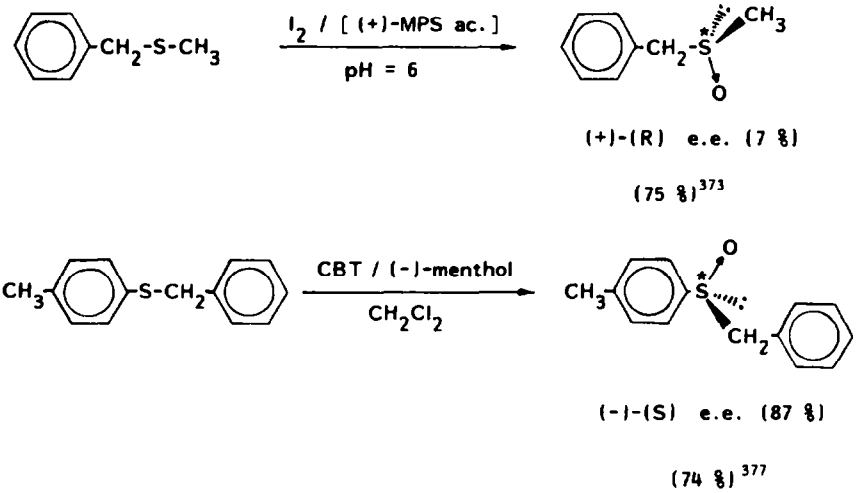
Scheme 53.

Numerous halogenated derivatives have been used for the asymmetric oxidation of sulfides to sulfoxides.

A weakly stereospecific oxidation was reported using iodine in an aqueous solution of (+)-2-methyl-2-phenyl succinic acid [(+)-MPS ac.][373] (Scheme 54).

Similarly, N-bromocaprolactam (NBC),[374,375] N-chloro-6,6-nylon (NCN)[375,376] and N-chloro-succinimide (NCS)[375] in the presence of chiral alcohols such as (−)-2-octanol or (−)-borneol have been used to prepare optically active sulfoxides. Here again, the degree of stereospecificity is always low (1–10% e.e.) (Scheme 54).

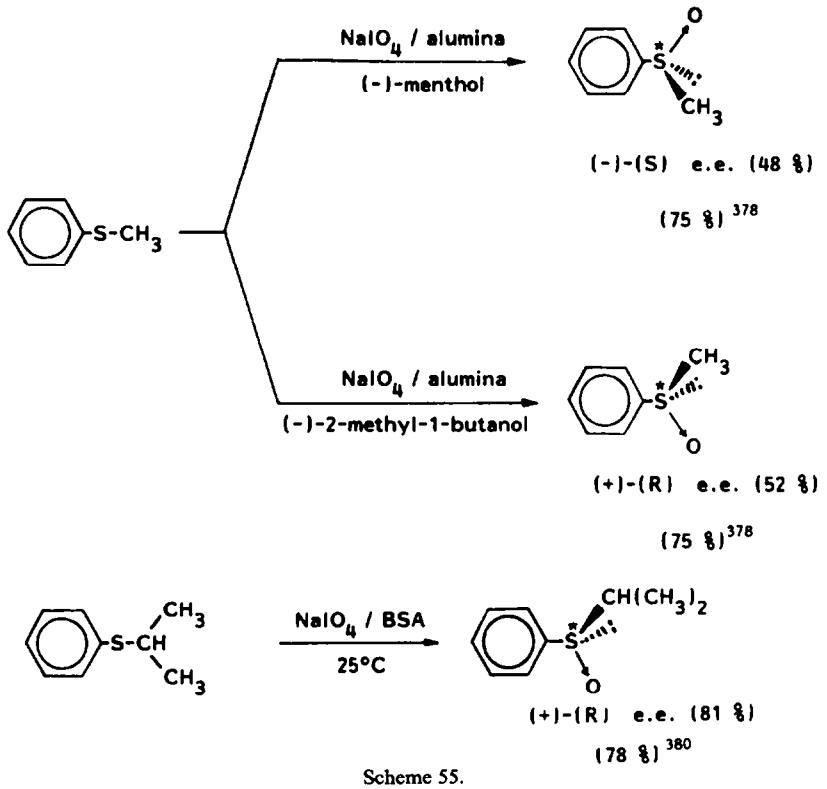
On the other hand, 1-chlorobenzotriazole (CBT)[377] in (−)-menthol has been used to prepare a diaryl sulfoxide of high optical purity (87%) (Scheme 54).



Scheme 54.

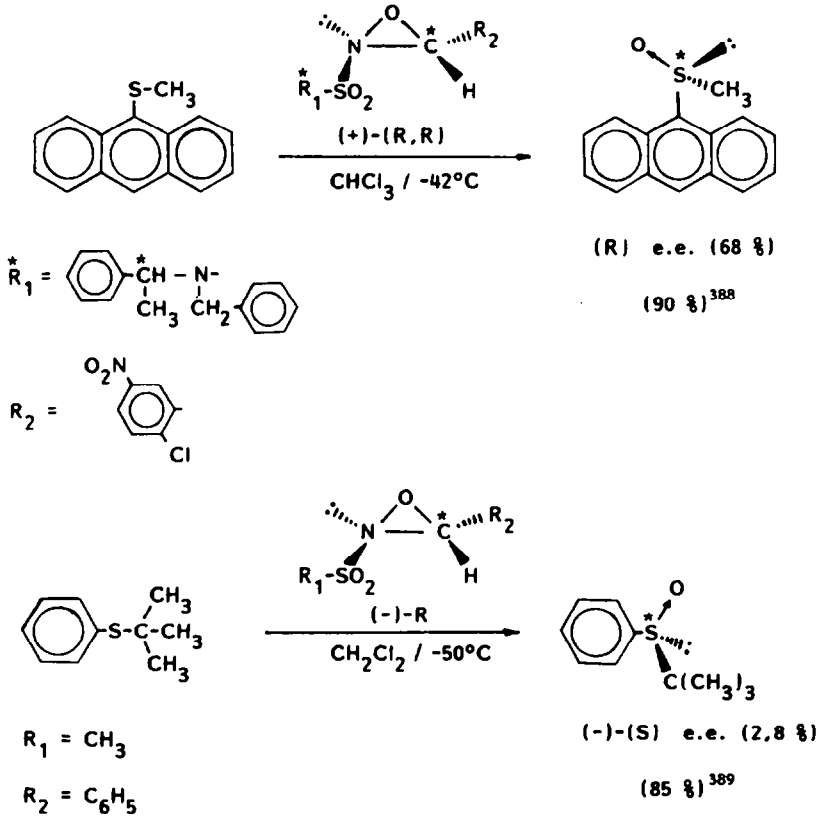
9.3. *Other Methods for Stereoselective Chemical Oxidation of Thioethers*

Sodium *m*-periodate ($NaIO_4$) on an alumina support[160,161,378,379] or in the presence of bovine serum albumin (BSA)[380–384] has been successfully used to prepare chiral sulfoxides (Scheme 55).



Scheme 55.

Davis *et al.*[385–388] and Torre and co-workers[389] have used chiral derivatives of 2-sulfonyl-3-aryloxaziridines (Scheme 56).

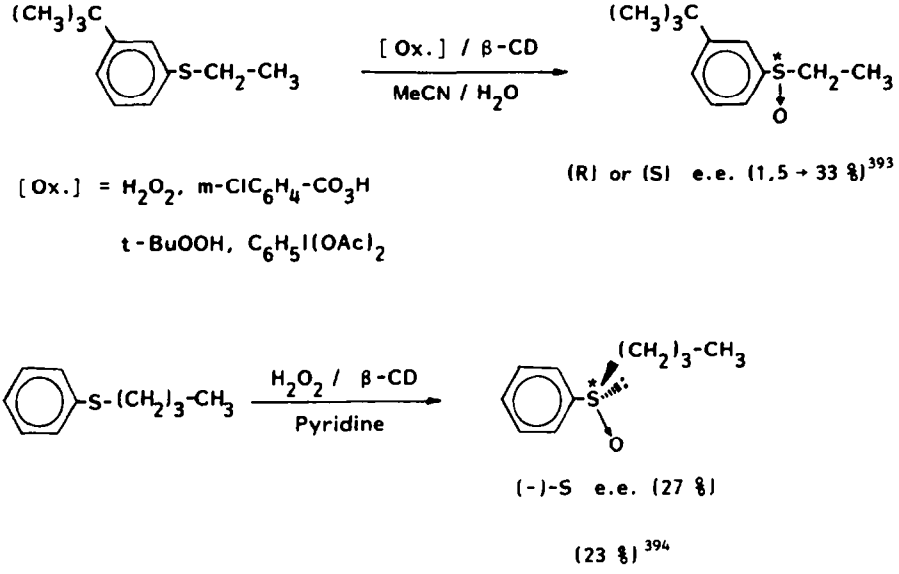


Scheme 56.

The enantioselectivity of this sulfoxidation was highly variable depending on the chiral oxaziridine used and the sulfide oxidized. Enantiomeric excess values for *R* and *S* forms ranged from 1 to 68%.

A chiral derivative of N-chlorocaprolactam [(+)-N-chloro-7-isopropyl-4-methyl-2-oxo-hexamethylene imine][390] and electrochemical oxidation methods[391,392] have also been used to prepare chiral sulfoxides.

Recently, Czarnik[393] and Drabowicz and Mikolajczyk[394] used β-cyclodextrins (β-CD) as "host" molecules to increase the enantioselectivity of the reaction. They used hydrogen peroxide, metachloroperbenzoic acid, terbutyl hydroperoxide and iodosobenzene diacetate as oxidizing agents (Scheme 57).

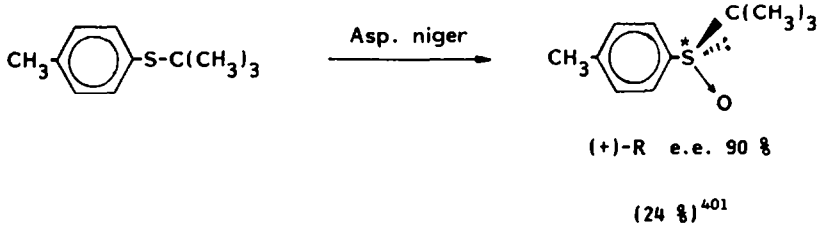


Scheme 57.

10. MICROBIOLOGICAL OXIDATION OF THIOETHERS

Oxidation of biotin to (−)-biotin S-oxide by *Aspergillus niger* was described by Wright *et al.*[395] in 1954. Other fungi such as *Rhizopus nigricans*[396] or *Calonectria decora*[397] were also used to oxidize alkylthiosteroids and sulfur-containing drugs to sulfoxides.

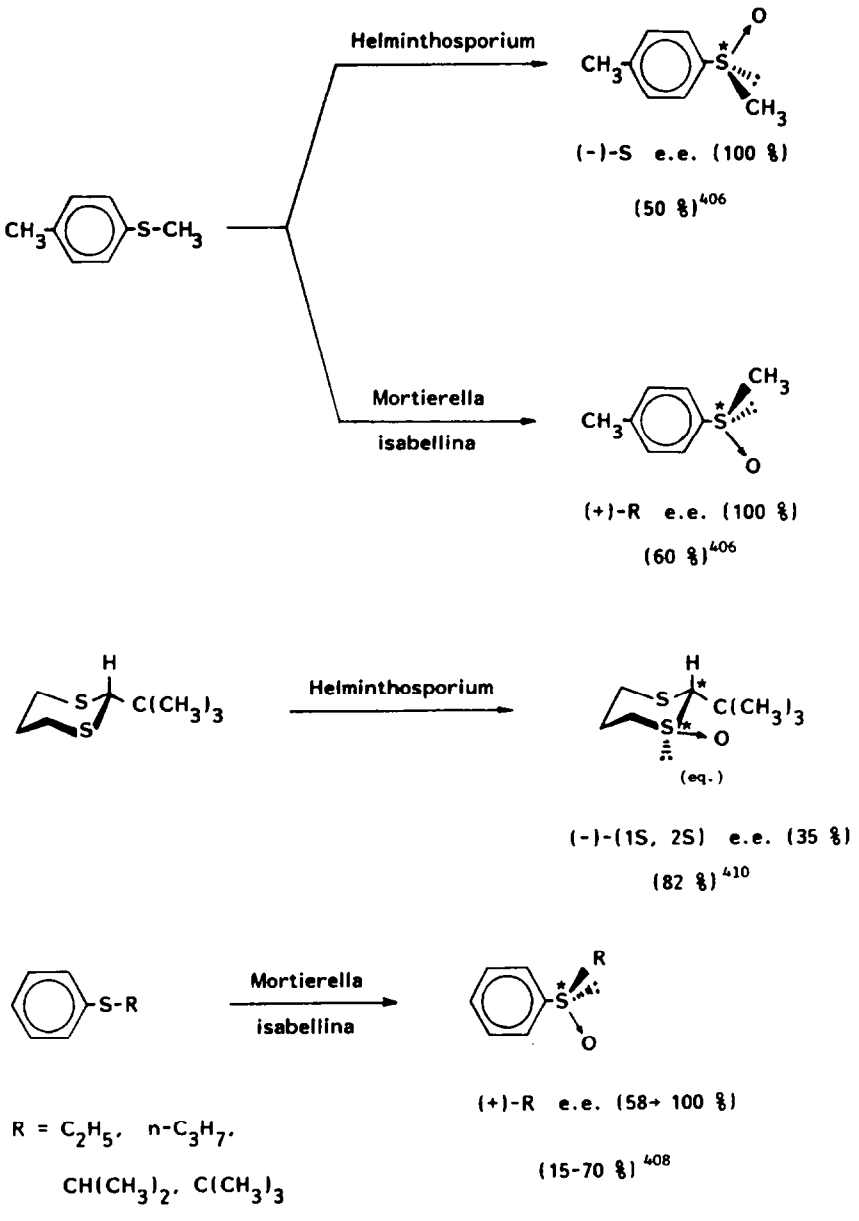
The stereoselectivity of the sulfoxidation of various thioethers by *Aspergillus niger* has been extensively studied by Dodson *et al.*,[399] Auret *et al.*[400–403] and Balenović and co-workers[404] (Scheme 58).



Scheme 58.

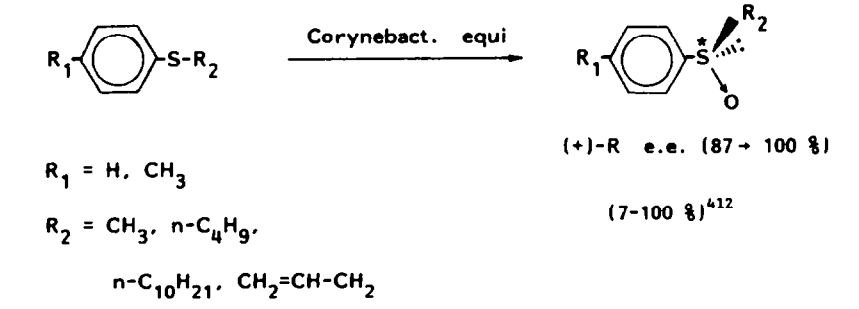
Numerous strains and species of fungi such as *Rhizopus stolonifer*,[399,403] *Rhizopus arrhizus*,[403] *Penicillium notatum*,[405] *Mortierella isabellina*,[406,409] *Helminthosporium*[406,409,410] and others[403,409,411] have also been used to oxidize stereoselectively a large number of sulfides to sulfoxides (Scheme 59).

Thus the two configurations *R* and *S* of *p*-tolyl methyl sulfoxide were isolated with an optical purity of 100% after microbiological oxidation of the corresponding sulfide by two different fungi[406] (Scheme 59).



Scheme 59.

Recently, Ohta *et al.*[412–414] reported the microbial oxidation of aryl alkyl sulfides to optically active sulfoxides by *Corynebacterium equi* (Scheme 60).



Scheme 60.

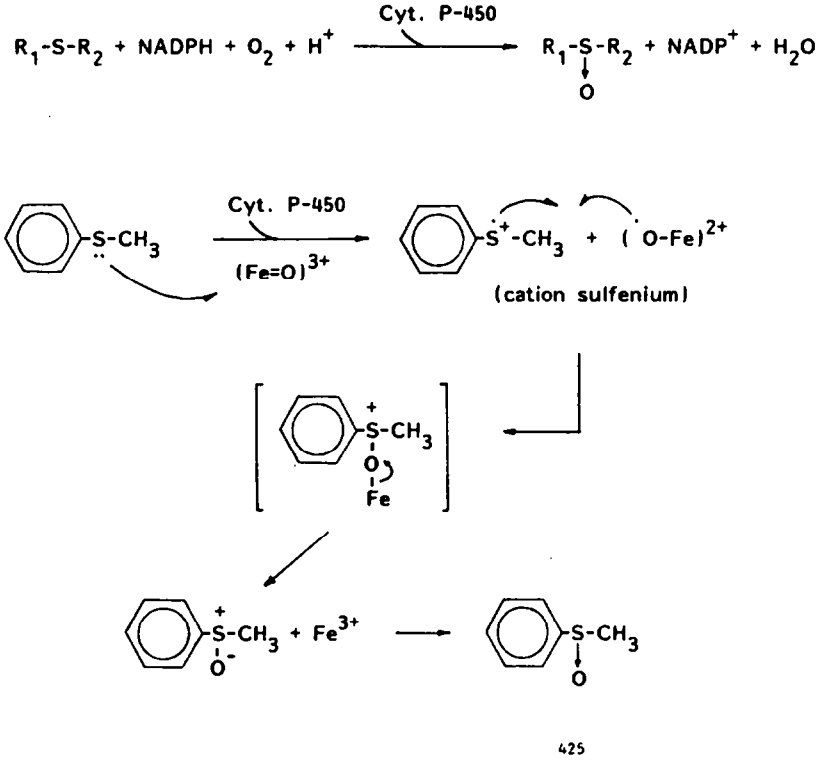
11. ENZYMATIC OXIDATION OF THIOETHERS

In 1957, pharmacological studies[415] showed that chlorpromazine was metabolized to sulfoxide *in vivo* and *in vitro*.

This observation led other workers[416,417] to study the enzymatic oxidation of drugs by guinea pig liver microsomes[416] and rat liver microsomes.[417]

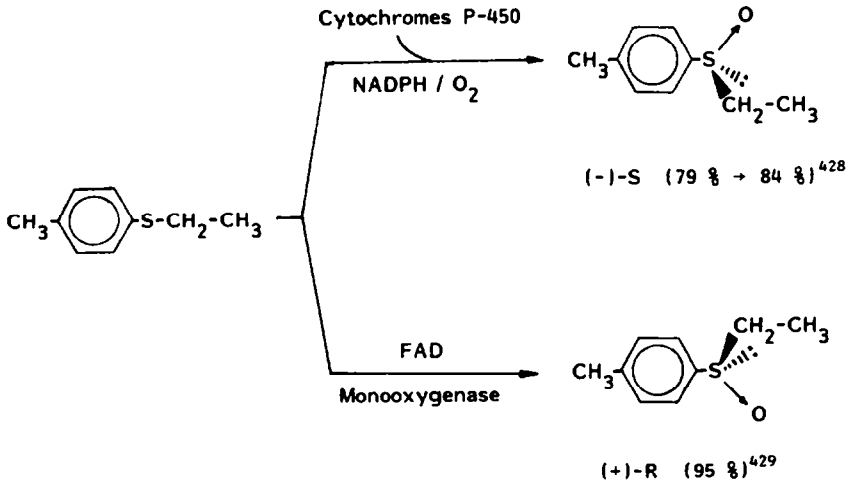
Likewise, Lee *et al.*[418] reported the enzymatic oxidation of α-thiocarboxylic acids to sulfoxides by the microsomal fraction from rat liver homogenates. This system required addition of NADPH and oxygen and was optimal at pH 7.4.

Since then, the enzymatic oxidation of sulfides has been quite extensively studied. Oae and co-workers[419–424] and Watanabe *et al.*[425–427] have synthesized several series of sulfoxides in this way, using a cytochrome P-450 extracted from rabbit liver microsomes with cofactors. The proposed mechanism involves a sulfenium cation (Scheme 61).



425

Scheme 61.

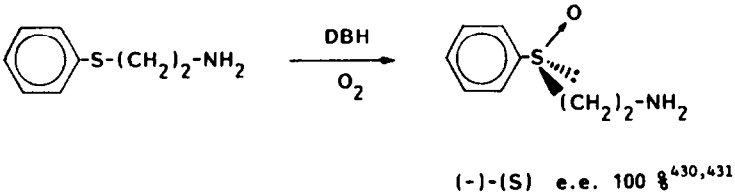


Scheme 62.

The stereoselectivity of the enzymatic oxidation of various diaryl, dialkyl and aryl alkyl sulfides, catalyzed by cytochrome P-450, has been well reported.[421,422,424,428]

In 1982, Waxman and co-workers[428,429] described the chiral sulfoxidation of 4-tolyl ethyl sulfide using two different enzyme systems. The oxidation reaction catalyzed by two cytochrome P-450 isozymes gave preferentially configuration (−)-*S*.[428] On the other hand, flavin adenine dinucleotide (FAD)-containing a monooxygenase purified from pig liver microsomes gave (+)-(*R*)-4-tolyl ethyl sulfoxide with high enantioselectivity (95%)[429] (Scheme 62).

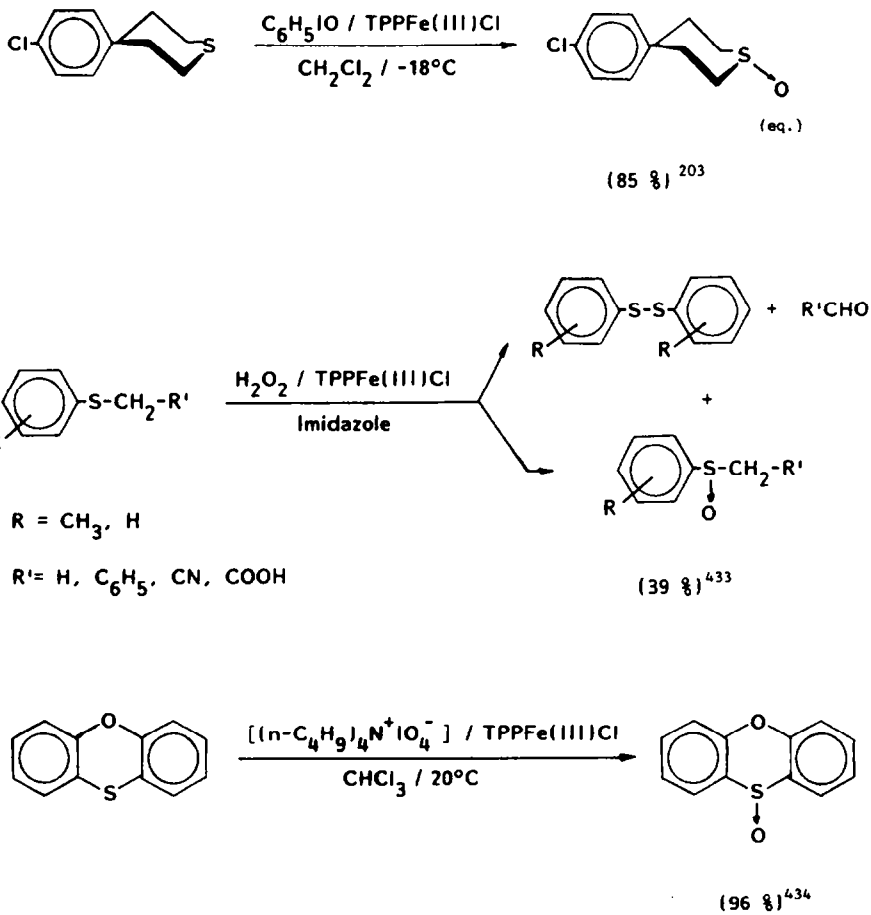
May and Phillips[430,431] report the use of dopamine β-hydroxylase (DBH), a copper-containing monooxygenase to oxidize aryl aminoalkyl sulfides with high enantioselectivity (Scheme 63).



Scheme 63.

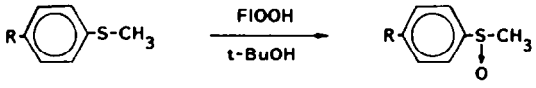
Concurrently, other work in this field has been engaged in setting up chemical systems[203,204,432–434] to model the enzymes able to oxidize sulfides.

One of these, consisting of chloro-5,10,15,20-tetraphenyl porphyrinato iron(III) [TPPFe(III)Cl], which catalyzes the oxidizing action of hydrogen peroxide (H_2O_2),[433] iodosobenzene (C_6H_5IO)[203,204] and tetrabutylammonium periodate [$(n\text{-}C_4H_9)_4N^+IO_4^-$],[434] has been used for the sulfoxidation of various sulfides (Scheme 64).



Scheme 64.

Similarly, 4a-hydroperoxy flavins (4a-FIR—OOH)[79,80] and isoalloxazine hydroperoxide (FIOOH),[435] using an enzyme model system with FAD-containing monooxygenase, have been used to oxidize aryl alkyl sulfides (Scheme 65).



R = H, Cl, CH_3, CH_3O, NH_2, CN

C_6H_5-CO, CH_3-CO-NH

435

Scheme 65.

12. CONCLUSION

The methods used to prepare sulfoxides from sulfides are extremely varied. This probably reflects the absence of any generally applicable method due perhaps to the complexity of the reaction itself.

Recent work focuses on the synthesis of optically active sulfoxides, either by microbiological and enzymatic methods or by asymmetric induction.

Of all the oxidizing agents used, three appear to be particularly useful, namely sodium metaperiodate, metachloroperbenzoic acid and iodosobenzene. These have the advantage of being convenient to work with, reasonably selective for sulfoxide preparation, and likely to give good overall yields. They can also be used in chemical model systems and for asymmetric oxidation in chiral media.

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