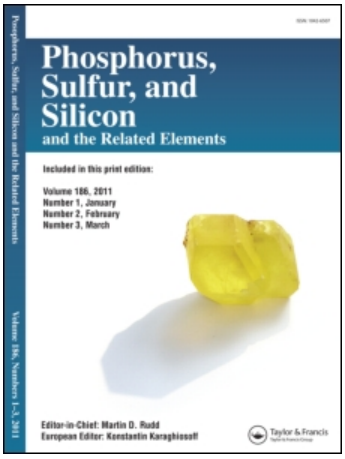

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Melamine Hydrogen Peroxide (MHP): Novel and Efficient Reagent for the Chemo- and Homoselective and Transition Metal-Free Oxidation of Thiols and Sulfides

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MELAMINE HYDROGEN PEROXIDE (MHP): NOVEL AND EFFICIENT REAGENT FOR THE CHEMO- AND HOMOSELECTIVE AND TRANSITION METAL–FREE OXIDATION OF THIOLS AND SULFIDES

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Melamine hydrogen peroxide (MHP) as a novel hydrogen peroxide adduct was easily prepared. Both chemo- and homoselective oxidation of thiols and sulfides to their corresponding disulfides and sulfoxides occurred by using MHP in moderate to excellent yields. $AlCl_3$ acts as a suitable activator in the oxidation of sulfides with MHP.

Keywords Disulfides; homoselective oxidation; melamine hydrogen peroxide; sulfoxides; transition metal–free oxidation

INTRODUCTION

Concentrated H_2O_2 is very dangerous to handle and not readily available. Hence this reagent is now replaced by more stable and safe complexes.[1] Organic addition compounds with hydrogen peroxide were prepared first by Tanatar.[2] These compounds are formed for example, with urea, urethane, succinimide, and erythritol. Krepelka and Buksa[3a] also prepared addition compounds of hydrogen peroxide with hexamethylene tetramine, diacetylhydrazine, quininesulfate, and aminoacetic acid. Of these compounds, the addition compound of urea with hydrogen peroxide is well known, is commercially available, and is important in industry.[3]

Oxidative coupling of thiols to disulfides is of practical importance in synthetic chemistry and biochemistry. Disulfides have found industrial applications as vulcanizing agents and are important synthetic intermediates with many applications in organic synthesis.[4] Disulfides are also essential moieties of biologically active compounds for peptide and protein stabilization.[5] The most important protocol for the preparation of disulfides is the oxidative coupling of thiols, and variety of reagents have been reported for this purpose.[6]

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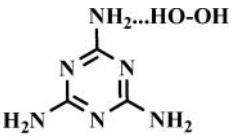
Organic sulfoxides are useful synthetic intermediates for the construction of various chemically and biologically active molecules. They often play an important role as therapeutic agents such as antiulcer (proton pump inhibitor), antibacterial, antifungal, anti-atherosclerotic, anthelmintic, antihypertensive, and cardiotonic agents, as well as psychotonics and vasodilators. The oxidation of sulfides is the most straightforward method for the synthesis of sulfoxides. There are a lot of reagents available for the oxidation of sulfides, but many of them cause over-oxidation to sulfones. Therefore, the conditions of the reaction, that is, time, temperature, and the relative amount of oxidants, must be controlled to avoid forming side products of the oxidation.[7]

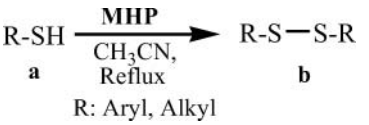
In continuation of our studies on the application of hydrogen peroxide adducts,[6a,6bb,8] we found that melamine, which is widely used as a fire retarder in polymers,[3] also forms an addition compound with hydrogen peroxide. More studies on this subject have shown us that the thermal behavior of melamine hydrogen peroxide was discussed previously by Nagaishi et al.,[3] but we did not find any reports on its application in organic functional group transformations. Therefore, we decided to apply this reagent for this purpose. On the basis of our previous investigations in the oxidation of thiols[9] and sulfides,[10] in this article we report melamine hydrogen peroxide [MHP, (**I**)] as a novel, efficient, and bench top reagent for the chemo- and homoselective oxidation of thiols and sulfides to their corresponding disulfides and sulfoxides.



I: MHP

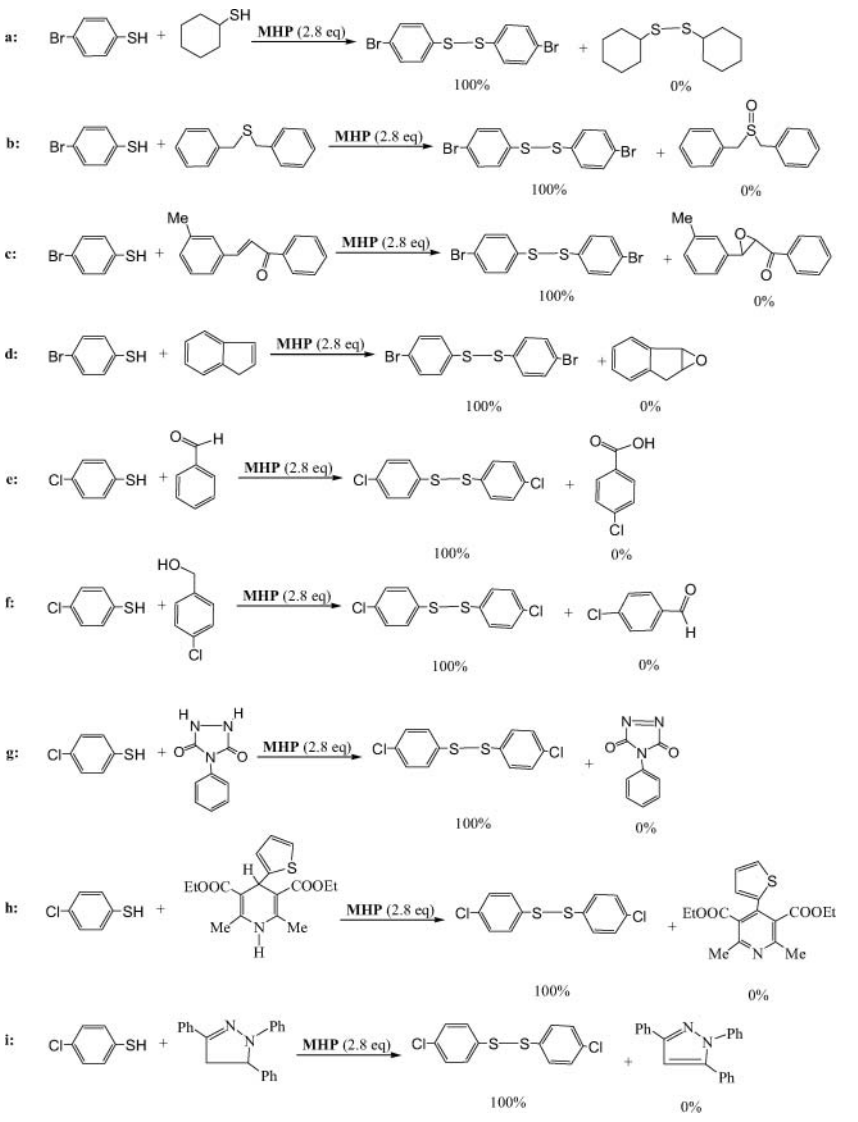
RESULTS AND DISCUSSION

Aromatic thiols were oxidized with MHP (**I**) in acetonitrile under reflux conditions and give the corresponding disulfides in moderate to excellent yields (Scheme 1 and Table I).



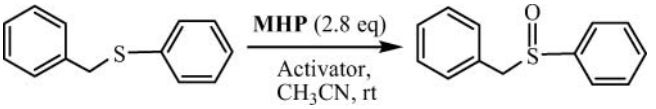
Scheme 1

The results shown in Table I indicate that the method is applicable for the oxidative coupling of aromatic thiols and is not suitable for aliphatic analogs (entries 10–12). For showing the chemoselectivity of the described system, some competitive reactions were designed. A mixture of equal amounts of aromatic thiols and aliphatic ones was subjected to oxidation in the presence of MHP under the optimized reaction conditions. Furthermore, we observed the excellent chemoselectivity between aromatic thiols and other oxidizable organic functional groups (Scheme 2).



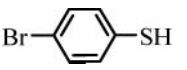
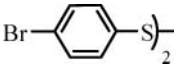
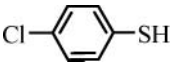
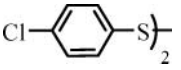
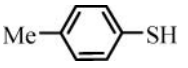
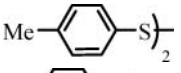
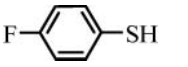
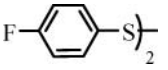
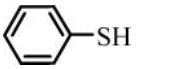
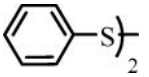
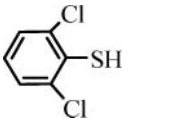
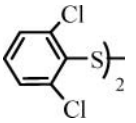
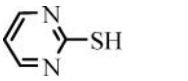
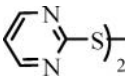
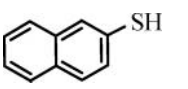
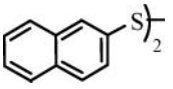
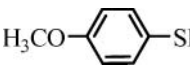
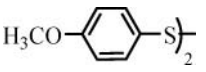
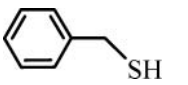
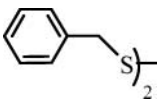
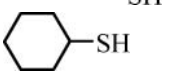
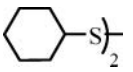
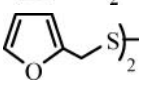
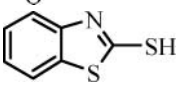
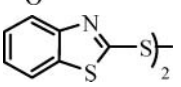
Scheme 2

Since MHP could not oxidize the sulfides alone (Scheme 2, Entry b), we decided to find a suitable activator for the activation of MHP in the oxidation of sulfides. For this purpose, we studied a number of various activators. The results are presented in Scheme 3 and Table II.



Scheme 3

Table I Coupling of thiols (**a**) to their corresponding disulfides (**b**) using MHP (**I**) in acetonitrile under reflux conditions

Entry	Substrate (a)	Product (b)	Reagent/Substrate (mmol)	Time (h)	Yield (%)[i]
1			2.2	0.08	82
2			2.2	0.25	91
3			2.2	1	75
4			2.9	0.25	99
5			2.2	0.25	88
6			2.9	0.17	71
7			2.9	0.5	94
8			2.2	0.17	85
9			1.4	2	59[ii]
10			11.5	20	19
11			11.5	24	48
12			1.4	1	—
13			1.4	1	Sluggish

[i]Isolated yields.
[ii]41% Undesired product.

Among the activators that have been used (Table II), we selected $AlCl_3$ as it was cheap and commercially available due to the best reaction conditions. In the next step, we checked the effect of various solvents on the progress of reactions. As illustrated in Scheme 4 and Table III, we have found that acetonitrile is the best solvent for the abovementioned reactions.

Scheme 4

Table II Oxidation of Bn-S-Ph using MHP (**I**) in the presence of various activators in acetonitrile under ambient conditions

Entry	Activator	Activator (mmol)	Time (h)	Yield (%)[a]
1	—	—	17	—
2	$ZrCl_4$[7e]	3	2	100
3	WCl_6[8a]	2	2	100
4	$AlCl_3$[8a]	2	0.33	100
5	$Al(HSO_4)_3$[11]	4	2.5	100
6	$FeCl_3$	4	Immed.	—[b]
7	Silica Chloride[11]	0.4(*g*)	2.5	trace
8	$NaHSO_4$[11]	4	3.5	80
9	DCC[12]	6	1.5	100
10	$CaCl_2.2H_2O$	4	2	70
11	$ZnCl_2$	4	0.83	100
12	ZnO	12	3	—
13	MgO	12	3	—
14	Silica sulfuric acid[11]	0.1(*g*)	1.5	—
15	Poly-SO_3H[11]	0.4(*g*)	2	—
16	Succinic anhydride	6	0.5	100[c]
17	Maleic anhydride	6	0.25	100[c]
18	Phtalic anhydride	6	0.33	100[c]
19	KBr	8	2	—

[a]Conversion.
[b]Mixture of products.
[c]With the side product.

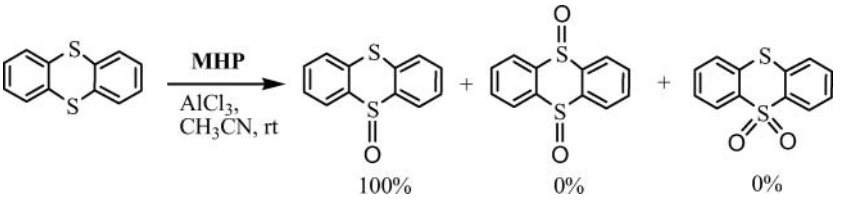
Table III Solvent effect in the oxidation of sulfides with MHP in the presence of $AlCl_3$ as an activator

Entry	Solvent	Time (h)	Yield (%)[a]
1	CH_2Cl_2	2	Trace
2	$CHCl_3$	2	Trace
3	EtOAC	0.5	—[b]
4	Acetone	2	—
5	*n*-Hexane	2.5	—
6	Acetic anhydride	0.17	—[b]
7	CH_3CN	0.33	100

[a]Conversion.
[b]Mixture of products.

For studying the scope and limitations of the described procedure, the oxidation of other sulfides was also studied. A good range of substrates such as aryl alkyl, diaryl, and dialkyl sulfides has been selectively oxidized to their corresponding sulfoxides (Table IV).

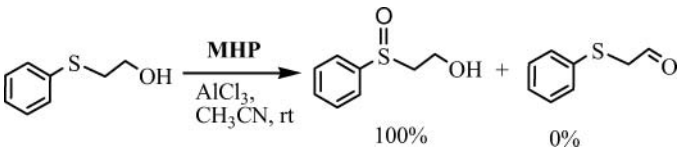
An excellent homoselectivity was observed in the oxidation of thianthrene. We wish to introduce the term homoselectivity[10c] for the first time in this article. We think that this keyword is suitable for application in special reactions so that a substrate that has two or more identical functional groups and one of them reacts in the course of the reaction selectively and to produce only one product. When the thianthrene was subjected to the oxidation (entry 4), homoselective monosulfoxidation took place, and surprisingly further oxidation to disulfoxidation or monosulfonation did not occur (Scheme 5).



Scheme 5

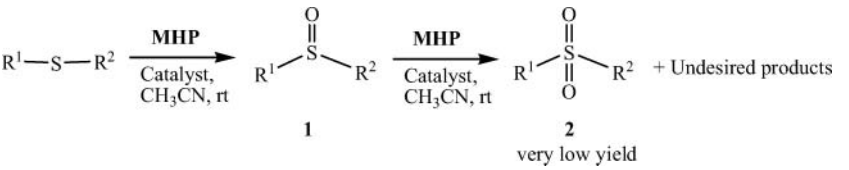
For demonstration of this fact, the ^{13}C NMR of the substrate and obtained product has been shown in Figures 1 and 2. As can be seen, the obtaining product has only one plane of symmetry and indicates six distinguished peaks.

Also, as is show in the entry 8 and Scheme 6, there is a chemoselectivity in the oxidation of **8c**. In this substrate, the functional group of sulfide was converted to the sulfoxide, while the alcoholic hydroxy group did not react with reagents and was intact.



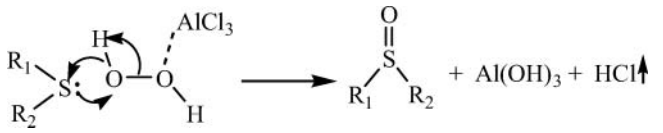
Scheme 6

Unfortunately, several attempts for the further oxidation of sulfoxides to their corresponding sulfones using the described system have failed, and the desired sulfones (**2**) were produced in low yield with some unidentified byproducts (Scheme 7). Therefore, this reagent system is not practical and suitable for this purpose.



Scheme 7

A plausible mechanism for the oxidation of sulfides to their corresponding sulfoxides using MHP/$AlCl_3$ was shown in the Scheme 8 based on the literature[7c,8a] and our observations in the course of the reaction.



Scheme 8

Although very recently, aqueous H_2O_2 has been reported for the oxidation of sulfides into the corresponding sulfoxides/sulfones in the presence of L-proline,[14] silica sulfuric acid,[15] and 10,10′-linked bisflavinium perchlorates,[16] but MHP in the same as other analogs such as PVP-H_2O_2,[6a] DABCO-DNOHP,[6b,8b] UHP,[8] sodium perborate[13] was used as a suitable "dry carrier" of the hazardous and unstable hydrogen peroxide, due to easy preparation, easy handling, safety, and stability at room temperature.

As shown in Table V, various systems for the oxidation of diphenylsulfide (**6c**) to diphenylsulfoxide (**6d**) suffer at least from one of the following disadvantages: long reaction times, using transition metal, low selectivity between sulfoxides and sulfones, expensiveness of reagents and catalyst, tedious workup procedure, low yields of products, etc., whereas the MHP/$AlCl_3$ system does not have the abovementioned disadvantages.

In conclusion, we think that the ability of MHP to generate oxidative species via in situ releasing of H_2O_2 in the reaction media will make it a useful reagent in organic functional group transformations.

EXPERIMENTAL

Chemicals were purchased from Fluka, Merck, and Aldrich chemical companies. Yields refer to isolated pure products. The oxidation products were characterized by comparison of their spectral (IR and ^1H NMR) and physical data with those authentic samples, which were produced by other reported procedures.[14–16,30]

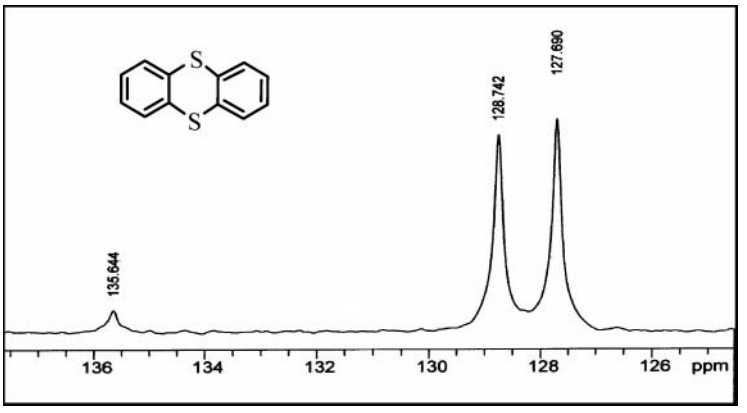
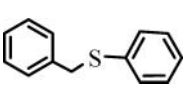
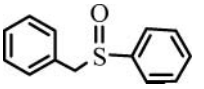
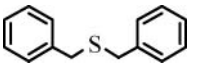
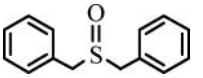
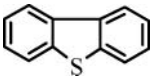
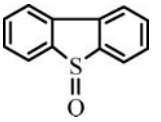
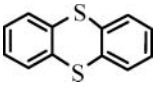
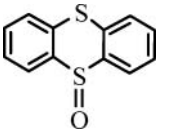
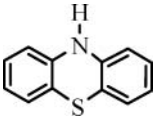
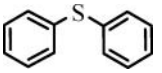
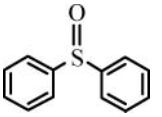
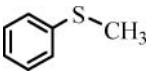
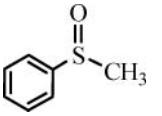
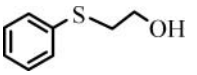
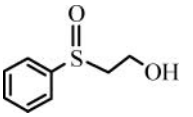
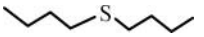
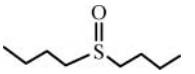
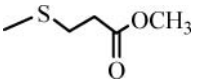
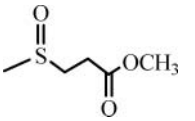


Figure 1 The ^{13}C NMR of thianthrene (Table II, entry 4).

Table IV Chemoselective oxidation of sulfides (**c**) to their corresponding sulfoxide (**d**) using MHP (**I**) in the presence of $AlCl_3$ (**II**) as an activator in CH_3CN at room temperature

Entry	Substrate (c)	Product (d)	Reagent/Substrate(mmol) I	Reagent/Substrate(mmol) II	Time (h)	Yield (%)[a]
1			1.78	1.25	0.33	84
2			1.71	1.2	1.25	92
3			1.71	1.2	0.67	92
4			1.42	1	1.25	90
5		—	1.71	1.2	2	—[b]
6			1.71	1.2	1.5	97
7			1.71	1.2	0.67	99
8			1.71	1.2	0.67	52
9			1.57	1.1	Immed.	97
10			1.57	1.1	0.67	82

[a]Isolated yields.
[b]Undesired products.

Preparation of MHP (I)

In a round-bottomed flask (100 mL) equipped with a magnetic stirrer, a suspension of melamine (12.6 g, 10 mmol) and H_2O_2 (30%, 10 mL) was stirred at 60°C for 30 min. Then reaction mixture was cooled and transferred to a crystallizing dish for slow

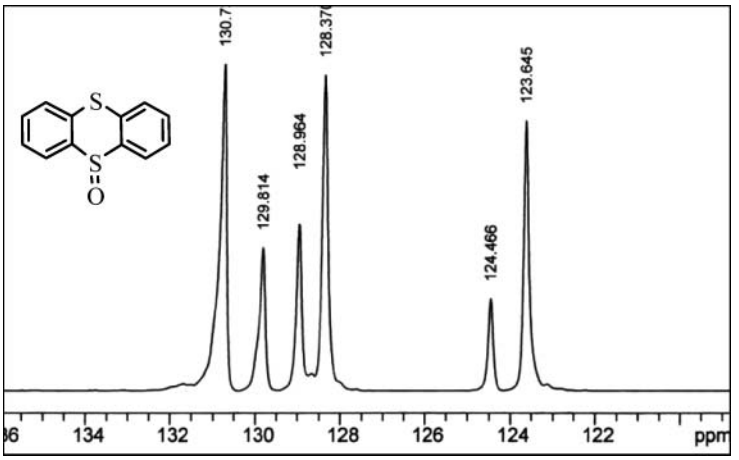


Figure 2 The ^{13}C NMR resulting from oxidation of thianthrene (Table II, entry 4).

evaporation of water under a mild air flow. After 3 days, H_2O_2-melamine as a white solid was obtained.[3]

General Procedure for the Oxidative Coupling of Thiols

A suspension of thiol (1.425 mmol) and MHP (**I**) in CH_3CN (6 mL) was stirred under reflux condition for the specified time (for time and molar ratio see Table I). The progress

Table V Various systems for the oxidation of diphenylsulfide (**6c**)

Entry	Oxidizing systems	Time (h)	Yields[(Ref.)]
1	H_2O_2-Melamine/$AlCl_3$/acetonitrile[a]	1.5	97
2	H_2O_2/ manganese (III) Schiff-base complex	0.5	91[17]
3	H_2O_2/hexafluoro-2-propanol	0.08	99[18]
4	H_2O_2/dodecyl hydrogen sulfate	2	95[6d]
5	H_2O_2/Na_2WO_4, $C_6H_5PO_3H_2$ and PTC	2	96[19]
6	H_2O_2/$ZrCl_4$	1	96[7e]
7	H_2O_2/Silica sulfuric acid	3	95[15]
8	H_2O_2/Phenole	0.07	99[20]
9	H_2O_2/$TiCl_3$	0.3	100[21]
10	H_2O_2 or *t*-BuOOH/titanium derivatives supported on silica[b]	24	95[22]
11	$NaBrO_3$/$Mg(HSO_4)_2$	24	45[23]
12	H_2O_2/ silica-based tungstate interphase catalyst	6.5	80[7c]
13	$NaClO_2$/manganese(III) acetylacetonate	0.7	95[24]
14	$Ce(BrO_3)_3$	3	99[25]
15	Silica sulfuric acid/$NaBrO_3$	4	75[26]
16	$NaIO_4$/wet SiO_2	2	85[27]
17	Trichloroisocyanuric acid/C_5H_5N/H_2O	0.5	85[28]
18	$KMnO_4/CuSO_4$. $5H_2O$[c]	10	[29]—

[a]Our obtained result by using the described procedure in this article.
[b]2% Sulfone.
[c]92% Sulfone.

of the reaction was monitored by TLC. After completion the reaction, the suspension was cooled. Then CH_3CN was evaporated, and the residue was dissolved in boiling chloroform (10 mL) and filtered. The crude products were obtained after evaporation of solvent.

General Procedure for the Oxidation of Sulfides

A mixture of sulfides (1.425 mmol), MHP (**I**), and $AlCl_3$ in acetonitrile (6 mL) was vigorously stirred at room temperature (for time and molar ratio see Table I). The progress of the reaction was monitored by TLC. When the reaction was completed, CH_3CN was evaporated. The residue was dissolved in boiling chloroform (10 mL) and filtered off. $CHCl_3$ was removed by simple distillation, and crude products were obtained with high purity.

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