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Nitric Acid in the Presence of Supported P_2O_5 On Silica Gel Affords an Efficient and Mild System for Oxidation of Organic Compounds Under Solvent-Free Conditions

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*This paper describes an efficient and easy method for oxidation of alcohols **1** and sulfides **2** to their corresponding carbonyl compounds **3** and sulfoxides **4** with nitric acid in the presence of supported P_2O_5 on silica gel under solvent-free conditions in high yields.*

Keywords: is oxidation; nitric acid; solvent-free; sulfoxide

1. INTRODUCTION

The oxidation of organic compounds is one of the most important reactions in modern organic synthesis. For this purpose, several new oxidizing reagents have been prepared [1–8]. Unfortunately, most of them suffer at least from one of the following disadvantages: 1) high cost, 2) long reaction time, 3) instability, 4) dangerous procedures for their preparation, and 5) tedious work-up procedures.

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Sulfoxides play an important role in organic chemistry [9–12]. They have been utilized extensively in carbon-carbon bond forming reactions and as versatile building blocks in organic synthesis [13,14]. The oxidation of sulfides is a very useful route for preparation of sulfoxides and several methods are available for their conversion [15–30]. However, most of the existing methods use expensive, toxic or rare oxidizing reagents that are difficult to prepare. As many of these procedures also suffer from poor selectivity and lack of generality, there is a need for simpler, less expensive, safer, and general methods for the conversion of sulfides to sulfoxides. With that in mind, reactions under solvent-free conditions have received increasing attention in recent years. A potential advantage of these methods over conventional homogenous reactions is that they often provide greater selectivity, proceed with enhanced reaction rates, give cleaner products, and involve simple manipulations [31–36].

2. EXPERIMENTAL

2.1. General Methods

Reported yields refer to isolated pure products after column chromatography. The products were characterized by comparison of their spectral (IR, ^{1}H NMR) and physical data with those of authentic samples [19–30,35]. All ^{1}H NMR spectra were recorded at 300 and 500 MHz in $CDCl_3$ relative to TMS (0.00 ppm) and IR spectra were recorded on Shimadzu 435 IR spectrometer. All reactions were carried out in refluxing acetonitrile.

2.2. Preparation of Reagent [(P_2O_5/Silica Gel) (65%w/w)]

In a mortar, 4.5 g of P_2O_5 (31.69 mmol) and 2.5 g of silica gel (0.063–0.2 mm) were ground for 1 min to form a homogeneous mixture.

2.3. Procedure for Oxidation of Alcohols 1 to Carbonyl Compounds 2

In a mortar, 0.2 g of P_2O_5/silicagel (1 mmol) and the corresponding alcohol (1 mmol) were ground for 30 sec. After that, 0.5 ml of HNO_3 65% was added and the resulting mixture ground with a pestle for the time specified in Table 1. Immediately after adding HNO_3, the release of NO_2 gas was observed. The progress of reaction was monitored by TLC (EtOAc/*n*-hexane = 20/80) until the alcohol had disappeared. When the reaction was completed, the product was extracted with Et_2O

TABLE 1 Oxidation of Alcohols **1** Under Solvent-Free Conditions[a,b]

Entry	Substrate (1)	Time (min)	Yield (%)
1	Benzylalcohol	1.0	95
2	4-Nitrobenzylalcohol	10.0	90
3	3,4-Dimethoxybenzylalcohol	2.0	94
4	1-Phenylethanol	20.0	0
5	4-Methoxybenzylalcohol	1.0	93
6	2-Methoxybenzylalcohol	1.0	95
7	Diphenyl-methanol	6.0	95
8	3-Methoxybenzylalcohol	1.0	93
9	4-Chloroybenzylalcohol	3.0	95
10	2-Chloroybenzylalcohol	5.0	93
11	1,2, Diphenyl-methanol	6.0	95
12	4′-Bromo-1-phenylethanol	5.0	93
13	4′-Chloro-1-phenylethanol	3.0	90
14	Benzoin	20.0	0
15	2,3-Dimethoxybenzylalcohol	3.0	93
16	Cyclohexanol	20.0	0
17	1-Tetralol	4.0	95
18	*n*-Heptanol	20.0	0
19	*n*-Pentanol	20.0	0
20	L-Menthol	20.0	0
21	1-Indanol	3.0	89
22	9-Fluorenol	4.0	88
23	4-*t*-Decylcyclohexanol	20.0	0
24	2-naphthalene-methanol	4.0	92
25	2-Phenylethanol	20.0	0
26	3-Methylcyclohexanol	20.0	0
27	4′-Methyl-1-Phenylethanol	20.0	0

[a]Confirmed by comparison with authentic samples (IR, TLC and NMR) [35]
[b]Yield of product after purification.

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(2×10 ml), dried ($MgSO_4$), and the solvent removed with a rotary evaporator to yield the product with no need of a further purification.

2.4. Typical Procedure for Oxidation of Sulfides 3 to Sulfoxides 4

In a mortar, 0.2 g of P_2O_5/silica gel (65%w/w) (1 mmol) and thioanisole (1 mmol, 0.124 g) were ground for 30 sec. After that 0.5 ml of HNO_3 65% was added and the mixture was ground with a pestle for 4 min. After disappearance of the starting sulfide, monitored by TLC using EtOAc/cyclohexane (2:8), the product was extracted with Et_2O (2×10 ml), filtered through a sintered glass funnel, and dried ($MgSO_4$). The solvent was removed under reduced pressure. The

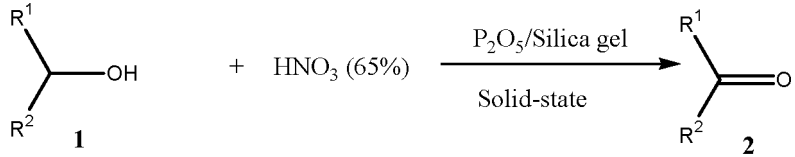
residue was purified by column chromatography using silica gel (EtOAc/cyclohexane, 2:8) to afford methyl phenyl sulfoxide as a colorless oil in 98% yield as revealed from ^{1}H NMR analysis, mp 30–32°C [Lit.[16] mp 32–33°C]. ^{1}H NMR ($CDCl_3$, 300 MHz): $\delta = 2.7$ (s, 3H), 7.48–7.56 (m, 3H), 7.64–7.67 (m, 2H). IR (film): 692, 754, 954, 1046, 1092, 1415, 1446, 1477, 2915, 3000, 3062 cm^{-1}.

3. RESULTS AND DISCUSSION

In continuation of our previous work [33–35], we wish to introduce here an efficient, mild, green, and rapid method for the selective oxidation of alcohols **1** to their corresponding carbonyl compounds. The oxidation is accomplished under solvent-free condition with 65% HNO_3 in the presence of P_2O_5 supported on silica gel, which acts as an efficient and mild oxidizing reagent. This oxidizing system has several advantages. In comparison with a previously reported method [15], it does not require a large excess of reagent and long reaction times. Due to its mildness behavior, no further oxidation to the carboxylic acid was observed.

The P_2O_5/silica gel reagent is stable and can be kept at room temperature for months without losing its activity. Alcohols **1** are mixing with one molar equivalent of P_2O_5/silica gel in a mortar at room temperature for 30 sec, and the oxidation to the corresponding carbonyl compound **2** occurs by grinding the mixture with one molar equivalent of 65% HNO_3. The mixtures were ground for the time specified in Table 1. Notably, this reagent did not oxidize benzoin after 20 min grinding. (Scheme 1 and Table 1.) In comparison to benzylic alcohols, the oxidation of aliphatic alcohols with this reagent did not occur at all suggesting that this method is suitable for the oxidation of benzylic alcohols in the presence of aliphatic alcohols.

In order to evaluate the selectivity of the reagent, the competing reactions shown in Eqs. (1–3) were carried out. When an equimolar amount of 2-phenylethyl alcohol and benzyl alcohol was treated



R^1, R^2 = Alkyl, aryl and H

SCHEME 1

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with 1 only equivalent of the oxidizing reagent, only the benzyl alcohol was oxidized (Eq. (1)). Similarly, treatment of equimolar (1 mmol) amounts of benzyl alcohol and diphenylmethanol with one equivalent of oxidant led to the exclusive oxidation of diphenylmethanol (Eq. (2)). Interestingly, no over oxidation to the corresponding carboxylic acids was observed. When we treated equimolar amounts of (1 mmol) benzyl alcohol and thioanisol with the solid reagent, only the benzyl alcohol was oxidized (Eq. (3)).

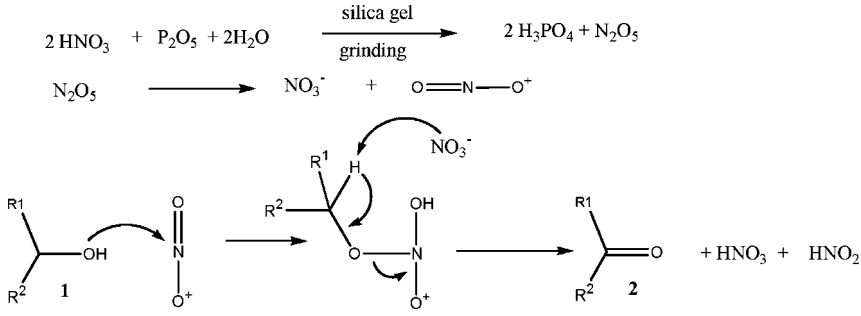
$$\mathrm{PhCH_2CH_2OH + PhCH_2OH \xrightarrow[10\text{min., grinding}]{\text{Solid-state}} \underset{\text{unchanged}}{\mathrm{PhCH_2CH_2OH}} + \underset{95\%}{\mathrm{PhCHO}}} \quad (1)$$

$$\mathrm{PhCHOHPh + PhCH_2OH \xrightarrow[5\,\text{min., grinding}]{\text{Solid-state}} \underset{100\%}{\mathrm{PhCOPh}} + \underset{\text{unchanged}}{\mathrm{PhCH_2OH}}} \quad (2)$$

$$\mathrm{PhSMe + PhCH_2OH \xrightarrow[10\,\text{min., grinding}]{\text{Solid-state}} \underset{\text{unchanged}}{\mathrm{PhSMe}} + \underset{95\%}{\mathrm{PhCHO}}} \quad (3)$$

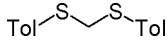
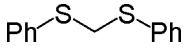
The possible mechanism for the oxidation of alcohols **1** to the corresponding carbonyl compounds **2** using nitric acid in the presence of supported P_2O_5 on silica gel under solvent-free conditions is outlined in Scheme 2.

We also studied the oxidation of sulfides **3** to corresponding sulfoxides **4** with a similar procedure (Table 2, Scheme 3). After extraction with ether followed by solvent evaporation, the product was purified by column chromatography. The sulfoxide products were obtained in excellent yields and short reaction times. This method also offers a simple, general, selective, highly efficient and green route for converting sulfides **3** to the corresponding sulfoxides **4** without over oxidation.



SCHEME 2

TABLE 2 Oxidation of Sulfides **3** to Sulfoxides **4** with 64% HNO_3 in the Presence of P_2O_5/Silca Gel[a,b]

Entry	R_1	R_2	Reaction time (min)	Yield (%)[c]
1	Ph	Me	4	98
2	Ph	*n*-Bu	6	98
3	Ph	$PhCH_2$	6	95
4	$PhCH_2$	Me	6	96
5	$PHCH_2$	*n*-Bu	5	92
6	$PhCH_2$	$PhCH_2$	5	93
7	4-MeC_6H_4	Me	5	95
8	4-ClC_6H_4	Me	6	85
9	C_4H_9	C_4H_9	8	83
10	1-Naphthyl	Me	5	93
11	Pr	Pr	8	82
12	Allyl	Allyl	6	80
13	Ph	CH_2Cl	5	88
14	4-MeC_6H_4	CH_2Cl	5	87
15	1-Naphtyl	$PhCH_2$	5	90
16	1-Naphtyl	CH_2Cl	5	89
17	4-$NO_2C_6H_4$	Me	6	93
18	4-$CHOC_6H_4$	Me	5	97
19	4-$OHCH_2C_6H_4$	Me	7	94
20	4-NCC_6H_4	Me	6	92
21	Ph	CH_2CH_2OH	8	84
22	4-$MeOC_6H_4$	CH_2Cl	5	95
23	$-(CH_2)_4-$	–	8	90
24	$-(CH_2)_3-$	–	8	92
25		–	6	89
26		–	5	80[d]
27		–	6	78[e]
28		–	6	74[f]

[a]Confirmed by comparison with authentic samples [19–30].
[b]Substrate/Oxidant (1:1.2 mmol).
[c]Yield of isolated pure products.
[d]10% disulfoxide.
[e]15% disulfoxide.
[f]12% disulfoxide.

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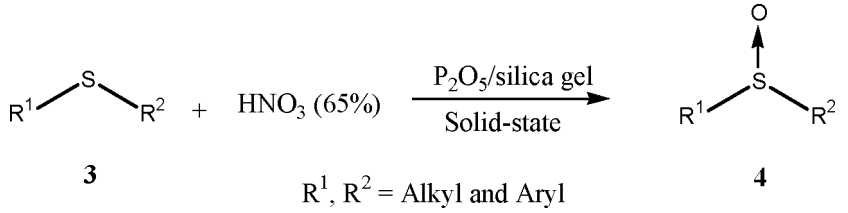


3 **4**

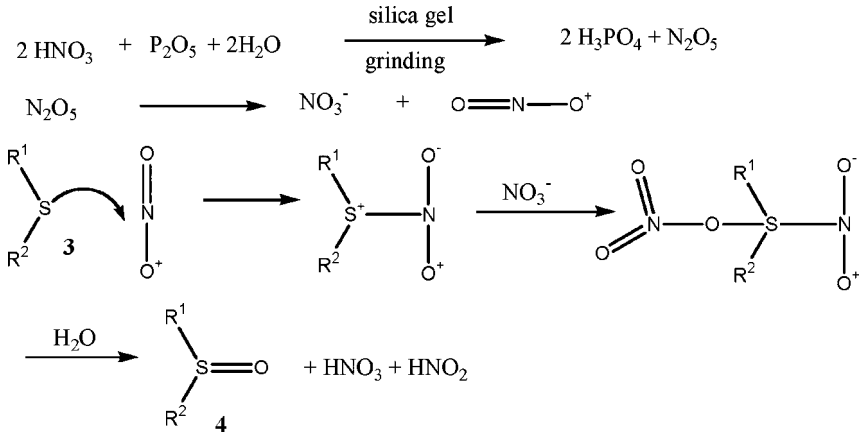
R^1, R^2 = Alkyl and Aryl

SCHEME 3

The generality of the method was examined using alkyl aryl, dialkyl, diaryl, cyclic sulfides and aryl disulfides. It was discovered that a wide variety of sulfides can be selectively oxidized by this inexpensive reagent under mild conditions (Table 2). The rates of the reactions of arylalkyl and diaryl sulfides are not dependent on the substituents on the aromatic ring. The reagent was chemoselective, tolerating various functional groups, such as, methoxy, carbonyl, hydroxy, nitro, nitrile, C=C double bonds, and halides. This method is also an excellent procedure for synthesis of α-chloro sulfoxides from their corresponding sulfides (Table 2, Entries 13, 14). The procedure is also useful for the partial oxidation of formaldehyde *S*,*S*-diphenyl acetal to the corresponding monosulfoxides in good yields (Table 2, Entries 26–28).

A possible mechanism for oxidation of sulfides **1** to the corresponding sulfoxides **2** using nitric acid in the presence of supported P_2O_5 on silica gel under solvent-free conditions is outlined in Scheme 4.

In conclusion, we report an efficient and versatile method for converting of alcohols and sulfides to their corresponding carbonyl



SCHEME 4

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compounds and sulfoxides. We list the following advantages: (a) our reagent is inexpensive and easy to handle, and it can be stored on the bench for months without losing its activity. (b) The procedure is simple and occurs under solvent-free conditions at room temperature. (c) The yields of sulfoxides are high and the reaction times are short. (d) The isolation of the products is straightforward.

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