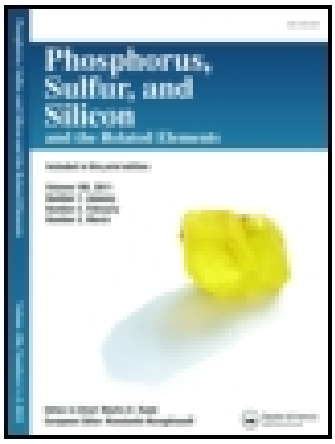

Phosphorus, Sulfur, and Silicon and the Related Elements

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Dess-Martin Periodinane on Silica: Rapid Oxidation of Alcohols using Microwaves

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DESS-MARTIN PERIODINANE ON SILICA: RAPID OXIDATION OF ALCOHOLS USING MICROWAVES

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Adsorbed on silica gel. Dess-Martin periodinane can rapidly oxidize alcohols to the corresponding carbonyl compounds upon exposure to microwave under solvent-free condition.

Keywords: Alcohols; carbonyl compounds; Dess-Martin periodinane; microwave irradiation; solvent-free condition

Oxidation of alcohols to obtain carbonyl compounds or carboxylic acids are important and fundamental transformations in synthetic organic chemistry and several methods have been explored to accomplish this conversion.[1] The most well known is aggressive chromium (VI) oxide[2] and the milder non aqueous variants like dipyridine chromium (VI) oxide,[3] pyridinium chlorochromate,[4] and pyridinium dichromate.[5] In spite of the many other reagents that are available,[6] there is still a demand for mild and selective reagents for the rapid oxidation of alcohols to carbonyl compounds in an eco-friendly manner.

Dess-Martin periodinane (DMP), 1, [1,1,1-triacetoxy-1-dihydro-1,2-benziodoxol-3(1H)-one] has received wide recognition as a mild selective reagent for oxidation of primary and secondary alcohols to the corresponding carbonyl compounds.[7]

The microwave enhanced chemical reactions in general, and on inorganic solid supports in particular, have gained popularity over the usual homogeneous and heterogeneous reactions.[8] In continuation of our investigations on organic reactions in solvent-fee condition,[9] we report a facile, selective, and rapid oxidation of alcohols to carbonyl compounds

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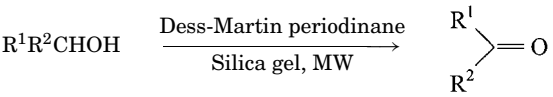
TABLE I Oxidation of Alcohols to Carbonyl Compounds



Entry	Substrate	Time (sec)	Product	Yield[a] (%)
1	Benzyl alcohol	120	Benzaldehyde	77
2	4-Nitrobenzyl alcohol	60	4-Nitrobenzaldehyde	78
3	4-Methylbenzyl alcohol	120	4-Methylbenzaldehyde	86
4	4-Methoxybenzyl alcohol	120	4-Methoxybenzaldehyde	82
5	5-Methyl-2-nitrobenzyl alcohol	120	5-Methyl-2-nitrobenzaldehyde	85
6	Benzhydrol	120	Benzophenone	97
7	Cyclohexanol	60	Cyclohexanone	99

[a]Isolated yield. Melting points for all derivatives agreed with literature values. All compounds identified by their spectroscopic data with those of authentic samples.

using Dess-Martin periodinane supported onto silica gel in solventless system, which is accelarated by exposure to microwave.

Since this reagent is no longer commercially available, it was prepared according to the literature procedure.[10] The reaction is conducted by mixing the supported reagent with alcohol in a beaker. Microwave irradiation of this solvent-free mixture provides an efficient, rapid, selective, and simple method for oxidation of alcohols to carbonyl compounds. To establish the generality of the method a variety of alcohols were efficiently converted to the corresponding aldehydes and ketones in a very short time (Table I). No overoxidation to carboxylic acid was observed.

It is noteworthy that without using silica gel, the reaction does not go to completion and considerable amounts of starting material are recovered unchanged.

In conclusion, oxidation with silica gel supported Dess-Martin periodinane in solventless system under microwave irradiation is a rapid, manipulatively simple selective protocol when compared to the conventional solution phase reaction which uses CH_2Cl_2 as solvent and trifluroacetic acid as cocatalyst. The oxidative use of supported Dess-Martin periodinane reagent under solvent-free conditions will generate newer applications of this and related hypervalent iodine agents in organic synthesis.

EXPERIMENTAL SECTION

All products were known compounds. Yields refer to isolated products. Dess-Martin periodinane was prepared according to referred procedure.[10]

Oxidation of Alcohols to Carbonyl Compound: General Procedure

Dess-Martin periodinane (1.05 g, 1.2 mmol) supported onto silica gel (1 mmol) was mixed with an appropriate alcohol (1 mmol) in a beaker. The reaction mixture was placed in a household microwave oven and irradiated for the specific time (Table I). After the completion of reaction (monitored by TLC, Petrol ether/ethyl acetate 8:2) CH_2Cl_2 (5 mL) was added. The solid was filtered off, and was added to the filtrate sodium hydroxide solution (1.3 M, 10 mL). The organic layer was washed with sodium hydroxide solution (1.3 M, 10 mL) and water (20 mL). The organic layer was dried over Na_2SO_4 and evaporated to dryness to afford the corresponding carbonyl compound (Table I).

(Caution: Although supported DMP under microwave irradiation was safe in our hand, it is advisable to carry out the reaction in an efficient hood.)

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