SHORT PAPER

J. Chem. Research (S),
2001, 39–40

Potassium ferrate supported onto montmorillonite K-10: a mild, efficient and inexpensive reagent for oxidative deprotection of trimethylsilyl ethers in non-aqueous conditions

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Potassium ferrate supported onto montmorillonite K-10 was found to deprotect trimethylsilyl ethers oxidatively to the corresponding carbonyl compounds in non-aqueous conditions.

Keywords: potassium ferrate, Montmorillonite

The protection of certain functional groups and their subsequent deprotection constitute important steps in the synthesis of polyfunctional molecules including natural products. The trimethylsilyl group is one of the most useful protecting groups for alcohols in a multi-step organic synthesis.[1]

Direct oxidation of trimethylsilyl ethers to the corresponding carbonyl compounds has attracted much attention in recent years and many reagents including chromium compounds have been introduced.[2] The drawbacks with such oxidants in multistage synthesis despite their power, is their lack of selectivity. The degradation of unsaturated substrates are often an unavoidable side reaction.

Furthermore chromium based reagents are inherently toxic[3] and handling their complexes are potentially dangerous (ignition).[4] Therefore the introduction of new methods, inexpensive and eco-friendly reagents is still in demand.

Potassium ferrate (K_2FeO_4) is a six-valent iron compound which can be easily and economically prepared by oxidizing ferric nitrate with sodium hypochlorite and subsequent treatment with potassium hydroxide.[5] It has been used recently by Laszlo and Delaude for oxidizing organic substrate in non-aqueous media.[6]

Heterogeneous supports and catalysts such as the commercially available montmorillonite K-10, clay have been widely employed in organic synthetic methodologiy. [7]

In this communication we wish to extend the versatility of potassium ferrate supported on montmorillonite K-10 to the oxidative deprotection of trimethylsilyl ethers which leads to the corresponding carbonyl compounds under non-aqueous conditions.

Potassium ferrate (VI) is a black-purple powder. It was mixed with montmorillonite K-10 and refluxed with trimethylsilylbenzyl ether in acetonitrile. Clean and selective reaction occurred and benzaldehyde was obtained in almost quantitative yield. It is noteworthy that in the absence of the clay, the above reaction was sluggish and considerable amounts of trimethylsilylbenzyl ether and benzylalcohol along with benzaldehyde were obtained. The double bonds are not liable to cleavage by this method as trimethylsilylcinnamyl ether gave cinnamaldehyde in high yield.

To assess the generality of the method, a variety of trimethylsilyl ethers were converted to the corresponding carbonyl compounds (Table 1)

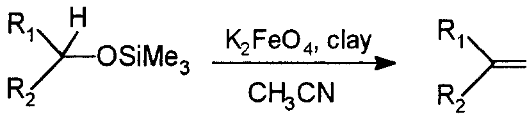
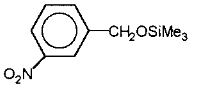
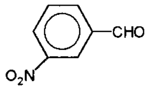
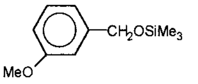
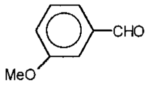
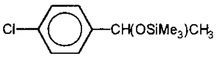
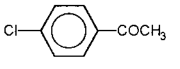
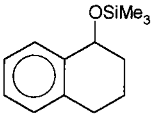
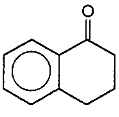


Table 1 Oxidative deprotection of trimethylsilyl ethers with montmorillonite K-10 supported K_2FeO_4

Substrate	t/h	Product	[a]Yield/%
$PhCH_2OSiMe_3$	3	$PhCHO$	95
$PhCH_2CH_2\ OSiMe_3$	2.5	$PhCH_2CHO$	92
$Ph(CH_2)_3\ OSiMe_3$	11	$Ph(CH_2)_2CHO$	85
$PhCH(OSiMe_3)COPh$	16	$PhCOCOPh$	80
	2		95
	8		95
	10		95
	10		80

a Yield refer to isolated product.

In conclusion this methodology affords an attractive, efficient, low cost and eco-friendly method for the direct oxidative cleavage of trimethylsilyl ethers to the corresponding carbonyl compounds.

Experimental

All compounds used are known, their physical and spectroscopic data were compared with those of authentic samples and found to be identical. K_2FeO_4 was prepared according to the modified procedure.[6] Trimethylsilyl ethers were prepared by reported procedure.[8]

Oxidative deprotection of trimethylsilyl ethers- General procedure: A suspension of trimethylsilyl ether (2 mmol), K_2FeO_4 (4 mmol) and

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† This is a Short Paper, there is therefore no corresponding material in *J Chem. Research (M).*

montmorillonite K-10 in CH_3CN (20 ml) was prepared in a flask (50 ml). This mixture was refluxed for indicated time (Table 1). The progress of reaction was monitored by TLC. The mixture was filtered, washed with CH_3CN (10 ml). The solvent was evaporated to dryness under reduced pressure. The crude product was passed through silica gel pad using suitable solvent to yield the corresponding carbonyl compound (Table 1).

The financial support from research council of Mazandaran University is gratefully acknowledged.

Received 4 Novemer 2000; accepted 18 December 2000
Paper 00/597

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