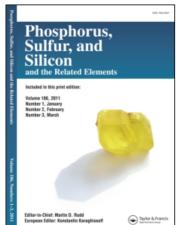
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## Phosphorus, Sulfur, and Silicon and the Related Elements

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# Potassium Ferrate Supported on Silica Gel: A Mild, Efficient, and Inexpensive Reagent for Oxidative Deprotection of Tetrahydropyranyl Ethers in Nonaqueous Conditions

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### POTASSIUM FERRATE SUPPORTED ON SILICA GEL: A MILD, EFFICIENT, AND INEXPENSIVE REAGENT FOR OXIDATIVE DEPROTECTION OF TETRAHYDROPYRANYL ETHERS IN NONAQUEOUS CONDITIONS

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Primary and secondary tetrahydropyranyl ethers are efficiently converted to the corresponding carbonyl compounds using potassium ferrate supported onto silica gel in nonaqueous conditions.

Keywords: Deprotection of tetrahydropyranyl ethers; carbonyl compounds; potassium ferrate

The protection of hydroxy groups with 2,3-dihydro-4H-pyran (DHP) is one of the most widely used methods which has a broad field of application in organic synthesis.<sup>1</sup>

Although considerable efforts have been made in the development of their removal,<sup>2</sup> there are only a few reports dealing with the direct oxidation of tetrahydropyranyl ethers to the corresponding carbonyl compounds.<sup>3</sup> Consequently, there is a need to develop and introduce new methods and reagents for such a transformation.

The use of a supported reagent<sup>4</sup> has attracted much attention because of improved selectivity, reactivity, and associated ease of manipulation. Potassium ferrate  $(K_2FeO_4)$ , a six-valent iron compound, can be easily prepared by oxidizing ferric nitrate with sodium hypochlorite and subsequent treatment with potassium hydroxide.<sup>5</sup> It has been recently used as a versatile oxidizing agent.<sup>6</sup>

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In this article, we wish to report that economically prepared potassium ferrate supported onto silica gel can easily and efficiently oxidatively deprotect THP ethers to the corresponding carbonyl compounds.

In continued investigations on organic reactions utilizing reagents<sup>7</sup> adsorbed on inorganic supporting material, we prepared and mixed potassium ferrate with silica gel and caused the reaction of this iron-supported reagent in acetonitrile with tetrahydropyranyl ethers, and observed the efficient and high yielding conversion of THP ethers to the corresponding carbonyl compounds. In the absence of silica gel, the reaction is sluggish and considerable starting material and deprotected alcohol are maintained even for an extended period of time.

To assess the generality of this oxidative deprotection method, a variety of tetrahydropyranyl ethers were caused to react under these conditions giving the corresponding carbonyl compounds in good-to-excellent yields (Table I).

It is noteworthy to mention that primary tetrahydropyranyl ethers did not undergo oxidation to the corresponding carboxylic acid.

In conclusion, silica gel-supported potassium ferrate represents a mild, efficient, and inexpensive reagent for the one-pot oxidative deprotection of THP ethers to the corresponding carbonyl compounds in nonaqueous condition.

#### **EXPERIMENTAL**

THP ethers were prepared according to known procedure. <sup>1e</sup> All oxidation products were known and identified by comparison of their physical data with those given in the literature. Yields refer to isolated products. Potassium ferrate was prepared according to a modified procedure. <sup>6</sup>

# Oxidative Deprotetion of THP Ether: General Procedure

In a flask (50 ml), a solution of 2 mmol THP ether in  $CH_3CN$  (20 ml) was prepared. To this solution, freshly prepared potassium ferrate (4 mmol) and silica gel (1.0 g) was added. This reaction mixture was refluxed

$$\begin{array}{c} R \\ \\ R_2 \end{array} \rightarrow \begin{array}{c} O \text{ THP} & \begin{array}{c} Potassium \text{ ferrate} \\ \hline silica \text{ gel} \end{array} & \begin{array}{c} R_1 \\ R_2 \end{array} \rightarrow \begin{array}{c} O \end{array}$$

FIGURE 1

Entry	Substrate	Time (h)	Products	Yield <sup>a</sup> %
1	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> OTHP	2	C <sub>6</sub> H <sub>5</sub> CHO	92
2	$C_6H_5CH_2CH_2OTHP$	3	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> CHO	85
3	(C <sub>6</sub> H <sub>5</sub> ) <sub>2</sub> CHOTHP	14	$(C_6H_5)_2CO$	92
4	p-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> OTHP	7	p-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> CHO	81
5	C <sub>6</sub> H <sub>5</sub> (CH <sub>2</sub> ) <sub>3</sub> OTHP	7	C <sub>6</sub> H <sub>5</sub> (CH <sub>2</sub> ) <sub>2</sub> CHO	94
6	m-OCH <sub>3</sub> C <sub>6</sub> H <sub>4</sub> CH <sub>2</sub> OTHP	5	m-OCH <sub>3</sub> C <sub>6</sub> H <sub>4</sub> CHO	90
7	p-ClC <sub>6</sub> H <sub>4</sub> CH(CH <sub>3</sub> )OTHP	11	p-ClC <sub>6</sub> H <sub>4</sub> COCH <sub>3</sub>	88
8	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>7</sub> OTHP	6	CH <sub>3</sub> (CH <sub>2</sub> ) <sub>6</sub> CHO	80

**TABLE I** Oxidative Deprotection of THP Ethers Supported on Silica Gel in Nonaqueous Conditions

for the indicated time (Table I). The progress of reaction is monitored by TLC. The mixture was filtered, washed with CH<sub>3</sub>CN, and the filtrate was evaporated to dryness under reduced pressure. The crude was passed through a silica gel pad with appropriate eluent. Pure carbonyl compounds were obtained in yields of 80%–94% (Table I).

### **ACKNOWLEDGMENT**

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<sup>&</sup>lt;sup>a</sup>Yields refer to isolated products which were identified by melting point and the spectra characteristics with the corresponding authentic sample.

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