

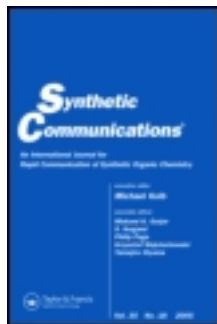
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**OXIDATION OF OXIMES TO KETONES WITH ZEOLITE  
SUPPORTED PERMANGANATE**

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**ABSTRACT** : A variety of aldoximes and ketoximes are oxidised to corresponding aldehydes and ketones in excellent yields by zeolite supported permanganate.

Oximes are extensively used for characterization of carbonyl compounds and in the preparation of amides via the Beckmann rearrangement.<sup>1</sup> Since oximes can be prepared from non- carbonyl compounds,<sup>2</sup> the regeneration of carbonyl compounds from oximes provides an alternative method for preparation of aldehyde and ketone. Since deoximation reaction is important from a synthetic point of view, recently a number of reagents have been reported such as chromium trioxide,<sup>3a</sup> 3-carboxypyridinium chlorochromate,<sup>3b</sup> sodium perborate,<sup>3c</sup> peroxy monosulphate ion,<sup>3d</sup> tert-butyl hydrogenperoxide,<sup>3e</sup> ammonium persulphate-

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\* To whom correspondence should be addressed.

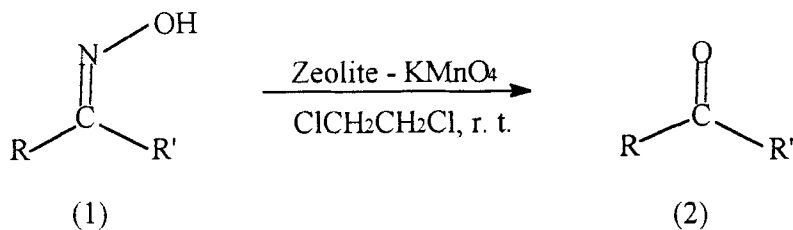
alumina using microwave,<sup>3f</sup> 1-Benzyl-4-aza-1-azoniabicyclo [2.2.2] octane periodate,<sup>3g</sup> Dess-martin periodinane<sup>3h</sup> and micro-wave assisted sodium periodate supported on silica.<sup>3i</sup>

Potassium permanganate is a powerful oxidising agent and it is used widely in organic synthesis but due to its poor solubility in organic solvents<sup>4</sup> it has some limitations in organic synthesis. This problem can be solved by preparation of  $\text{KMnO}_4$ - dicyclohexyl 18-crown-6 stable complex<sup>5</sup> and activation of  $\text{KMnO}_4$  by simple process of impregnation onto inorganic supports such as Linde Molecular Sieves, silica gel and certain clays.<sup>6</sup>

Triphase catalysis system is having importance due to recovery of catalyst and selective chemical conversions. Although polymer is one of the common support in triphase catalysts it has some limitations such as diffusion, high cost, swelling property and mechanical/ chemical instability.<sup>7</sup> Several inorganic supports, such as metal oxides, clays and zeolites have been studied.<sup>8</sup>

Previous deoximation procedures by potassium permanganate requires aqueous  $\text{KMnO}_4$  solution and longer reaction time.<sup>9</sup> Recently Sreekumar *et. al.* reported several oxidation procedures such as oxidation of enamines, alkylarenes and unsaturated alcohol to the corresponding ketones by zeolite supported potassium permanganate.<sup>10</sup>

We now wish to report here the convenient oxidation of ketoxime and aldoxime to ketone and aldehyde by zeolite supported  $\text{KMnO}_4$ . The reaction of oxime (**1**) with zeolite supported  $\text{KMnO}_4$  in 1,2-dichloroethane afforded carbonyl compounds without side product. (**2**) (Scheme)



Scheme

The results of the conversion of various ketoximes and aldoximes to ketones and aldehydes respectively are reported in the Table.

The products of the reaction with zeolite supported potassium permanganate were isolated simply by filtering off the zeolite reagent and evaporating the solvent from the filtrate. Thus the method has advantage in terms of yields, simplicity of the performance, non-aqueous work-up and no side reactions. Under the experimental conditions used sensitive groups such as hydroxy, amino remains unaffected. In addition of this zeolite can be regenerated and reused after activation. Controlled experiments demonstrate that zeolite H-Y does not convert oximes to ketones.

#### EXPERIMENTAL :

The oximes were prepared by a general procedure.<sup>11</sup> The purity of the compounds were checked by TLC. Compounds were further characterized by physical constants.

#### **Zeolite supported potassium permanganate :**

In 1 liter round bottom flask 500 ml of 0.06 M aqueous KMnO<sub>4</sub> (in

Table

Deoxygenation of ketoxime and aldoxime using zeolite supported  $\text{KMnO}_4$ 

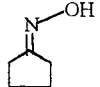

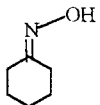

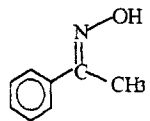
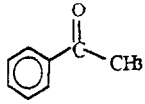
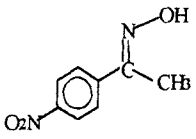
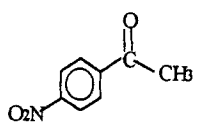
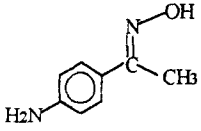
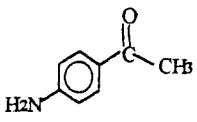
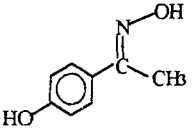
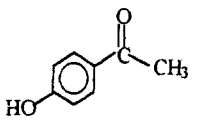
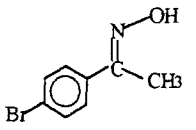
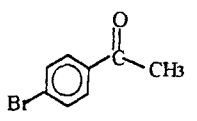
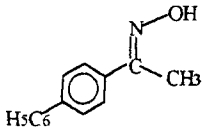
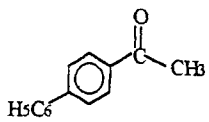
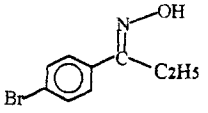
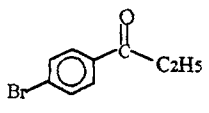
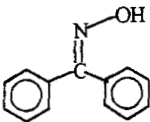
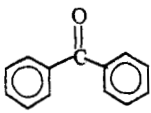
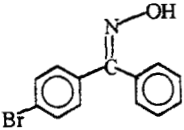
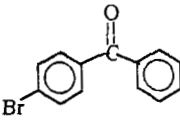
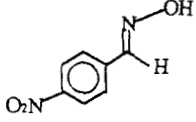
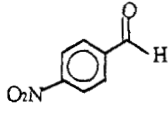
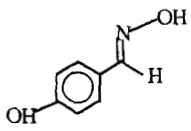
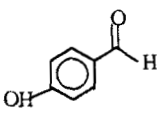
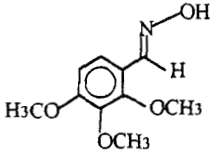
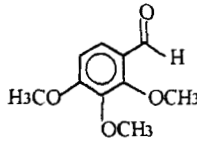
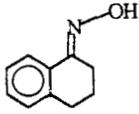
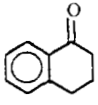
Entry No.	Oximes	Yield in %	Ketones
1.		90	
2.		93	
3.		92	
4.		78	
5.		85	
6.		83	
7.		80	
8.		85	
9.		80	

Table Continued

10.		85	
11.		83	
12.		60	
13.		65	
14.		68	
15.		80	

deionised water) and 20gm of zeolite H-Y stirred at room temperature for 5 minutes and then water was removed on rotary evaporator under reduced pressure. Resultant dark pink colored free flowing zeolite supported  $\text{KMnO}_4$  powder was used for oxidation procedure.

#### General procedure for Oxidation :

Zeolite supported  $\text{KMnO}_4$  ( 1 gm, 1.5 mmole  $\text{KMnO}_4$  content) was added to a solution of oxime (1 mmole) in 1,2-dichloroethane (10 ml) then the mixture

was stirred at room temperature for 30 minutes and filtered immediately through sintered glass funnel, evaporation of solvent under reduced pressure gives product, which was further purified by column chromatography (Silica Gel 60-120). Final product was then characterised by physical constant,  $H^1$ -NMR and IR spectral studies.

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