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Oxidation of Alcohols to Carbonyl Compounds with a New Potassium Permanganate Adsorbed on Kieselguhr Reagent

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OXIDATION OF ALCOHOLS TO CARBONYL COMPOUNDS WITH A NEW POTASSIUM PERMANGANATE ADSORBED ON KIESELGUHR REAGENT

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ABSTRACT: A new reagent, potassium permanganate adsorbed on kieselguhr, suitable for the oxidation of alcohols to the corresponding carbonyl compounds is described.

The oxidation of alcohols to the carbonyl compounds with potassium permanganate ($KMnO_4$) is used widely in organic synthesis and a number of procedures have been reported. [1, 2] For this transformation, a new technique of using $KMnO_4$ adsorbed on a solid support has been recently exploited. The solid supports used include molecular sieve, [3] silica gel, [3] montmorillonite clay, [3] $CuSO_4 \cdot 5H_2O$, [4] bentonite [5] and alumina. [6] These supported $KMnO_4$ reagents [7] have achieved good selectivity under mild conditions and convenient isolation of

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the product. Previously, we have found that kieselguhr is a good solid support of chromic acid for the oxidation of primary alcohols to the corresponding aldehydes[8] and now report that $KMnO_4$ adsorbed on kieselguhr is a new reagent suitable for the oxidation of alcohols to the corresponding aldehydes or ketones.

The present $KMnO_4$-kieselguhr reagent not only has the same advantages as other supported $KMnO_4$ reagents described above but also has some added benefits such as shorter reaction time than that of $KMnO_4$-molecular sieve reagent and the oxidation of primary alcohols to aldehydes is more efficient than that of $KMnO_4$-$CuSO_4 \cdot 5H_2O$ or bentonite reagent.

The $KMnO_4$-kieselguhr reagent is prepared by addition of a weighed amount of kieselguhr to a solution of $KMnO_4$ in water and evaporating to dryness. The oxidation is very simple. After contact of the alcohols with $KMnO_4$-kieselguhr reagent the corresponding aldehydes or ketones are obtained by filtration and evaporation of the solvent. Our results are reported in the Table.

Table, Oxidation of Alcohols to Carbonyl Compounds

Substrate	Product [a]	Yield [b] (%)	Bp (lit.) (°C)
$CH_3CH_2CHOHCH_3$	$CH_3CH_2COCH_3$	82	79-81 (79.6) [9]
$(C_6H_5)_2CHOH$	$(C_6H_5)_2CO$	97	47-49 (48.5-49) [d, 9]
$C_6H_5CH_2OH$	C_6H_5CHO	91	179-180 (179) [10]
$C_6H_5CH{=}CHCH_2OH$	$C_6H_5CH{=}CHCHO$	94	240-242 (240-241) [c, 11]
p-$CH_3OC_6H_4CH_2OH$	p-$CH_3OC_6H_4CHO$	86	248 (248) [9]

a. All the aldehydes and ketones have been described previously in the literature and were identified by their infrared spectra or the infrared spectra and Mps of their 2,4-dinitrophenylhydrazones.
b. Yield of isolated product.
c. Mp. of 2,4-DNP.
d. Mp.

EXPERIMENTAL SECTION

Preparation of $KMnO_4$-kieselguhr Reagent: To a solution of $KMnO_4$ (40g) in water (1000ml) kieselguhr (160g) is added with a stirring. After evaporating in vacuo, the resultant is dried to give about 195g $KMnO_4$-kieselguhr reagent.

Oxidation of Benzyl Alcohol to Benzaldehyde. Typical Procedure: The

above reagent (118g) was placed in a flask together with toluene (200ml) and the mixture was magnetically stirred. Then a solution of benzyl alcohol (5.4g, 0.05mol) in toluene (30ml) was added and after 1h at 70°C the solid was filtered off and washed with toluene (3 × 30ml). The combined filtrates were evaporated to give 4.8g (91%) of benzaldehyde, bp. 179-180°C, lit. [10]bp. 179°C.

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