相轉移環保氧化反應

Organic-Solvent-Free Phase-Transfer Oxidation of Alcohols Using Hydrogen Peroxide

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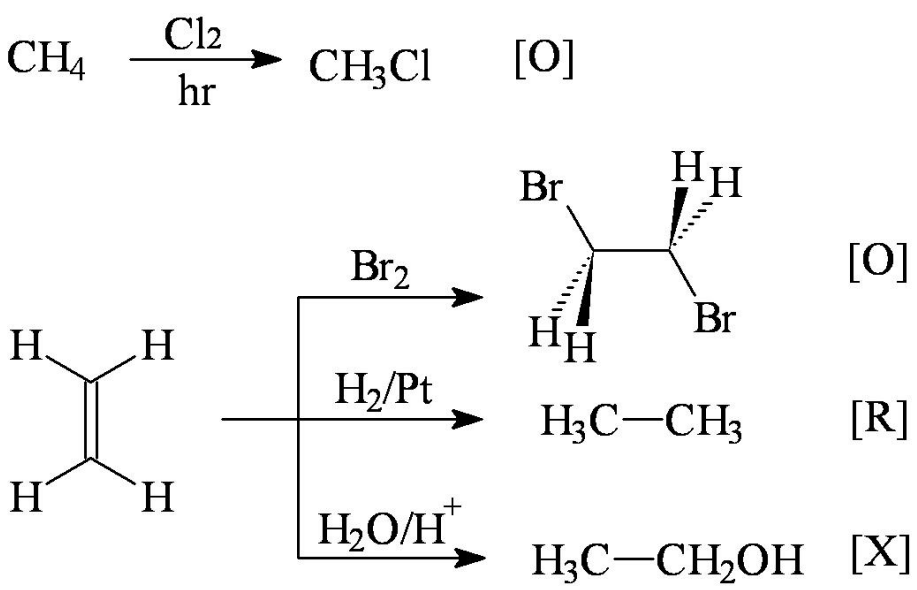
原　理

1. Oxidation:

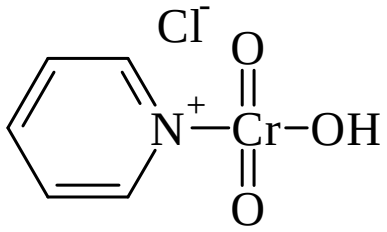
add “O”, de “H”, Oxid.# ↑, electron density on C ↓



2. Oxidant: (oxidize others)

(1) high valence metal : $Na_2Cr_2O_7$; $KMnO_4$ …(pollution)

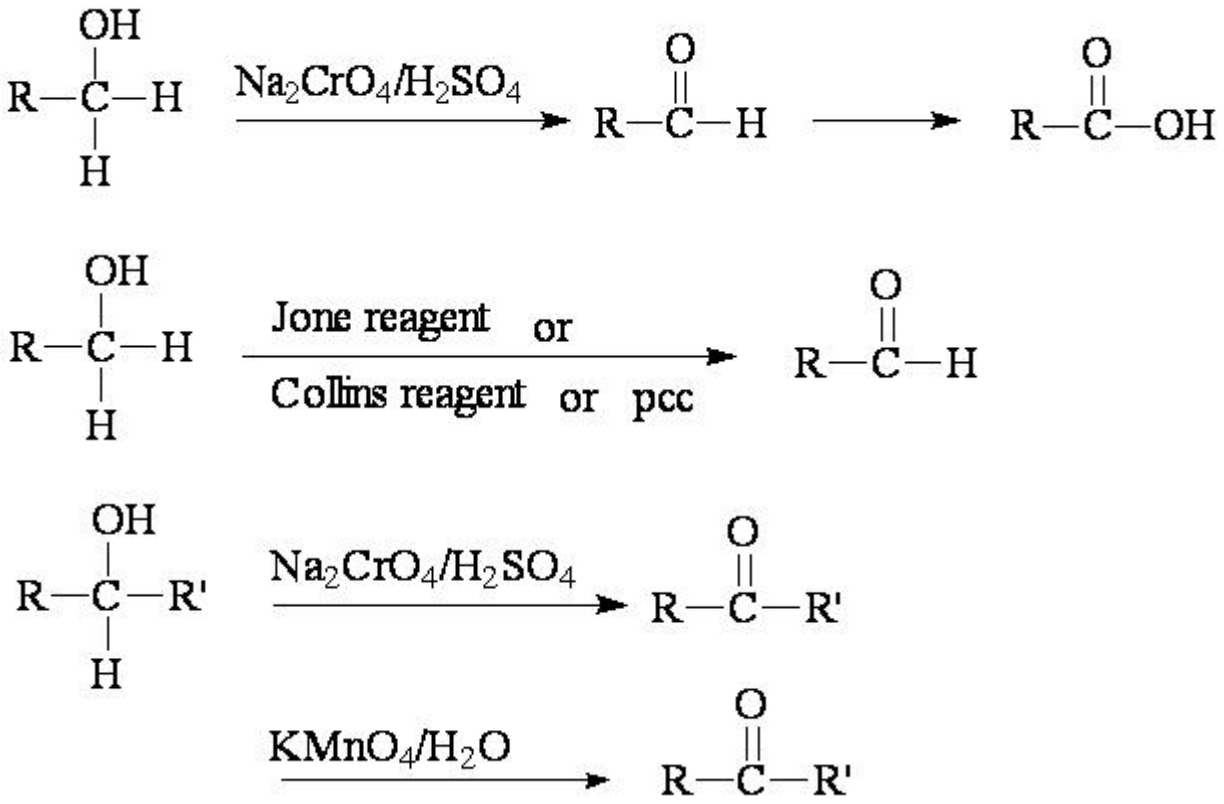
Pyridinium **C**hloro**C**hromate:



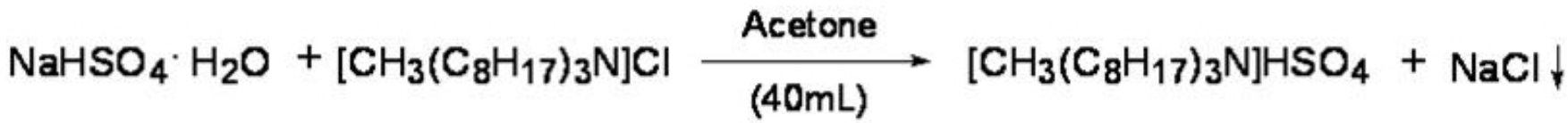
(2) O_2 , O_3

(3) NaOCl , H_2SO_4 , H_2O_2 ,HNO_3

3. Oxidize Alcohols to Aldehydes , Ketones and Acids



1. Preparation of the Phase-Transfer Catalyst $[CH_3(C_8H_{17})_3N]HSO_4$:



MW:	138	404.15	483.65	58.5
密度:		0.88g/mL		
用量:	13.8g	45.93mL		
mol數:	0.1	0.1		

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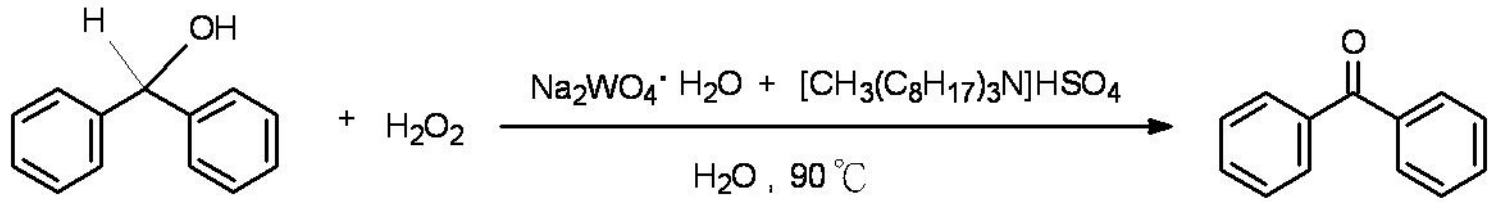
Reaction equation:



2.5 mL dist. H_2O + 0.75 mL H_2O_2 + 0.05 g $Na_2WO_4 \cdot H_2O$ + 1.15 g benzhydrol in 25mL R.B. flask

↓

add 0.075 mL (2 drops) $[CH_3(C_8H_{17})_3N]HSO_4$

↓

stir for 2 min

↓

gentle reflux for 30min

↓

add 5 mL saturated $NaCl_{(aq)}$

↓

transfer the mixture to a separatory funnel

↓

extract the product with 5 mL CH_2Cl_2

↓(collect the organic layer)

dry with $MgSO_{4(anhy)}$

↓

filter and collect the filtrate

↓

concentration

↓

the oily product + 3 mL hexane

↓

swirl in ice bath until white solid appears

↓

collect the solid (wash it with 1mL hexane)

↓

suction to dry

↓

weight

TLC 測 試

↓

TLC test [elute: hexane/ CH_2Cl_2 (1:1)]

prod: benzophenone Rf=0.35 , s.m.: benzhydrol Rf=0.15

↓

calculate the % yield (result report)

Check out

1. 繳交產物並告知產物淨重。

2. 實驗問題：1，2


The End !
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