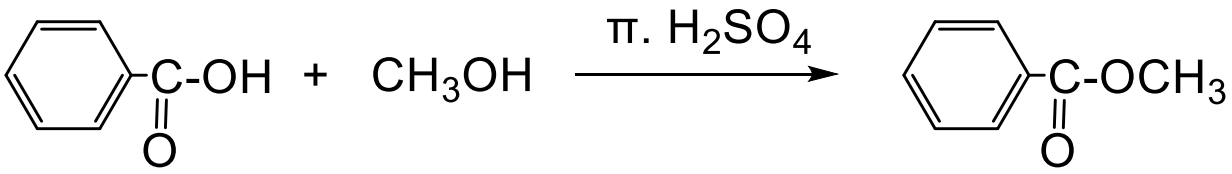
Synthesis of benzoic acid methylester



Reagents:

PhCOOH: 6 g (0,05 mol)

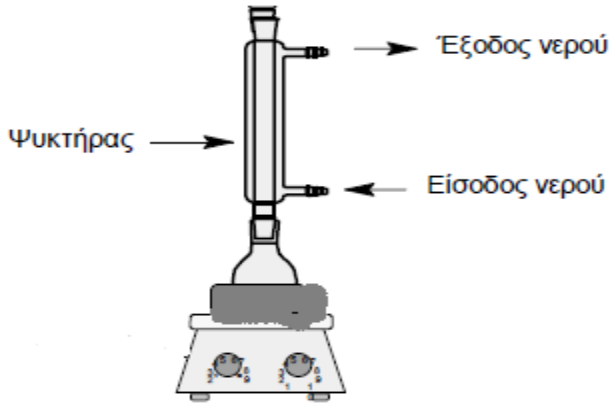
CH_3OH: 20 ml (15,8 g, 0,49 mol)

H_2SO_4: 1 ml

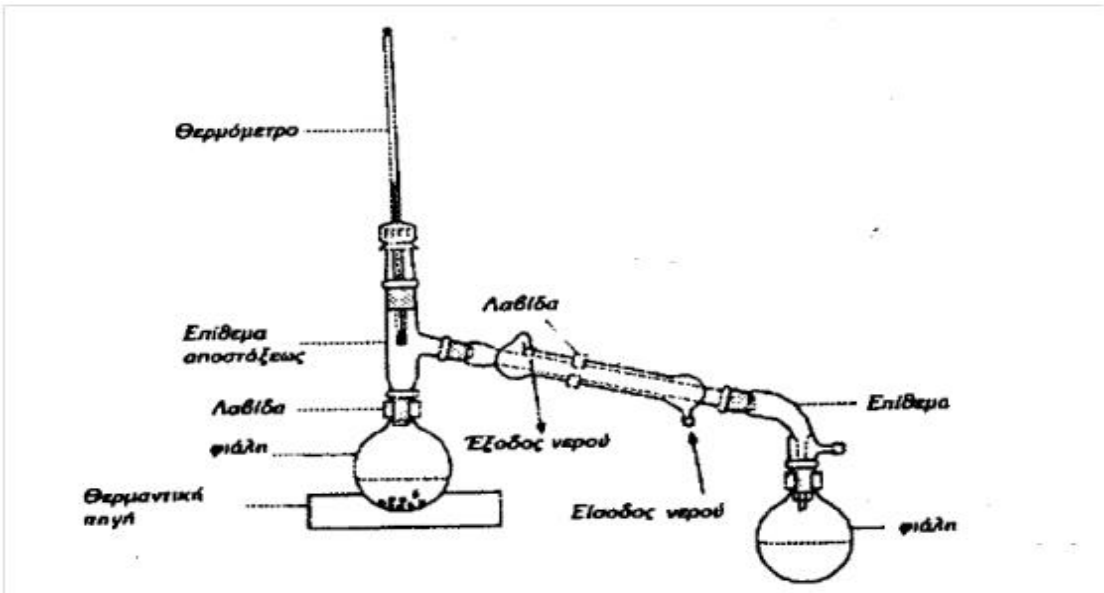
Na_2CO_3 (5% w/v): 40 ml

Hexane/Ethyl Acetate (7:3)

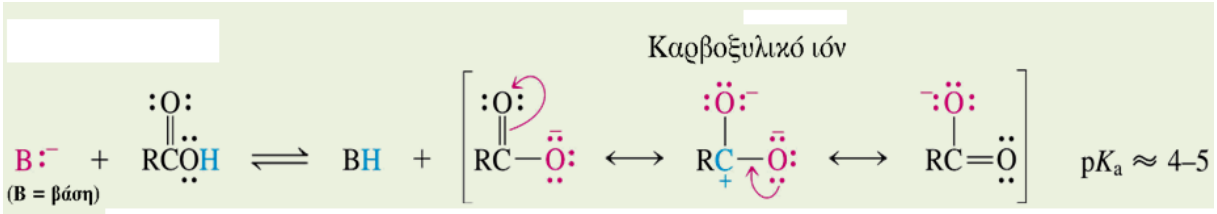
Reflux



Απόσταξη
Κλασματική

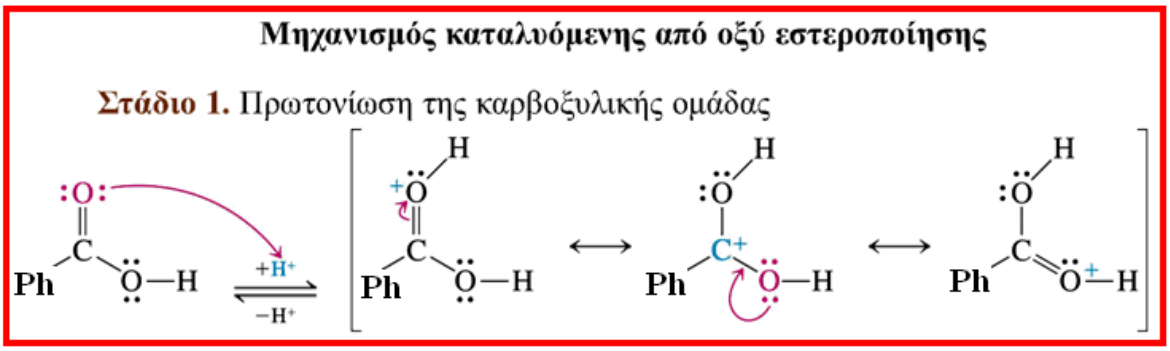


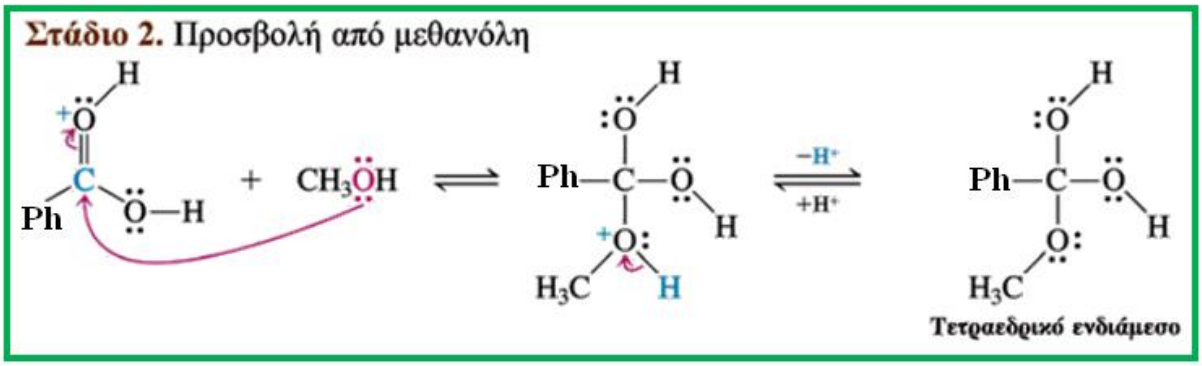
In the presence of Base:

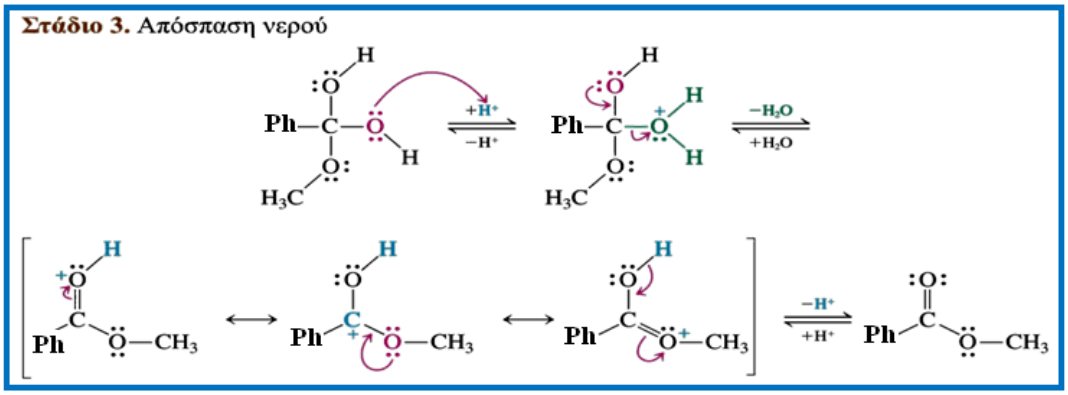


Not possible the attack from the MeOH.

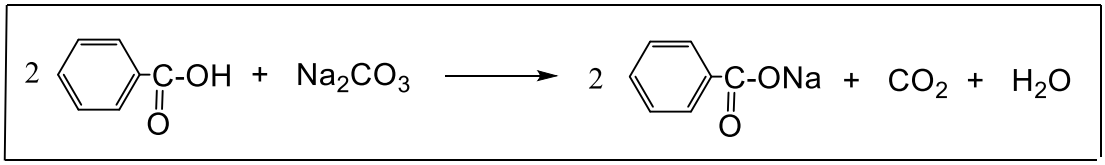
In the presence of acid



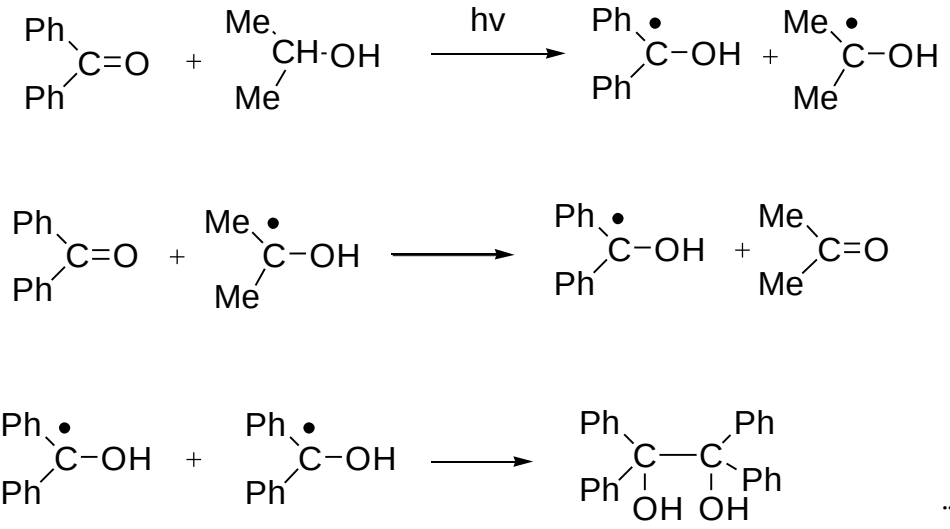




Removement of excess of PhCOOH by Na_2CO_3

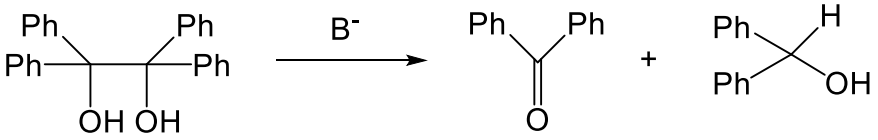


PHOTOCHEMICAL REDUCTION OF BENZOPHENONE TO BENZOPINACOL

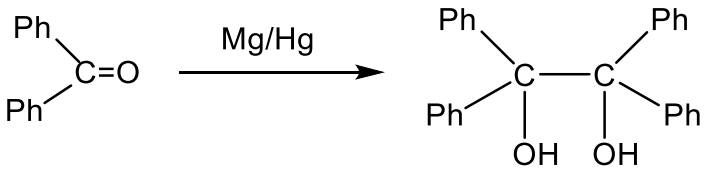


Αντιδραστήρια:
Benzophenone: 1 g (0,0055 mol)
Isopropanol: ~10 ml
gl. CH_3COOH: 1-2 σταγόνες.

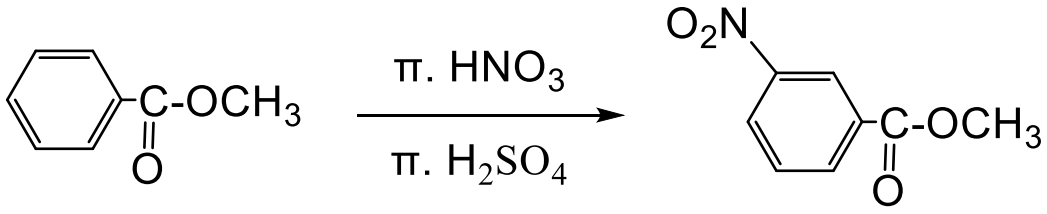
Remarks:
- CH_3COOH : For the removement of base traces (from the glass):



- No air (O_2 is reacted we the radicals formed).
- With UV lamp (Hg) the reaction is completed in ~ 1 h.
- Other reaction for this transformation:



Nitration of methyl benzoate

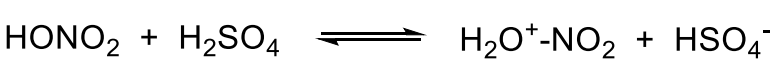


Reagents:
PhCOOMe: 3 ml (3,28 g, 0,024 mol)
HNO_3: 2 ml
H_2SO_4: 6 ml + 2 ml

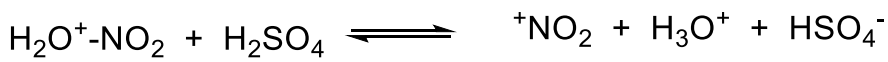
PhCOOMe H_2SO_4 in ice-bath.
Cold solution H_2SO_4 - HNO_3 (**Nitration Acid**).
The reaction is performed under stirring at **5-20°C**.

Reaction Mechanism

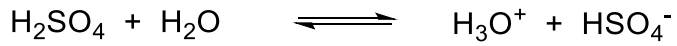
Σχηματισμός ηλεκτρονιοφίλου

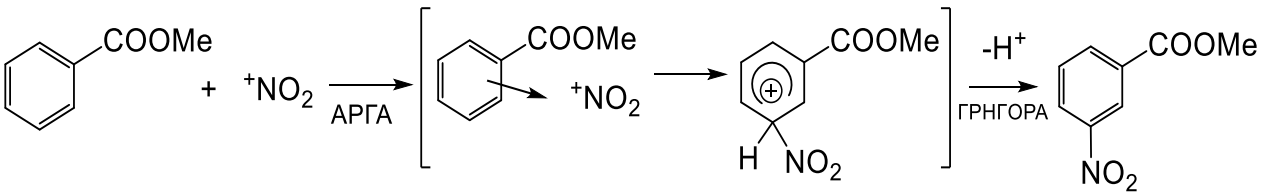


Αργό στάδιο χωρίς H_2SO_4



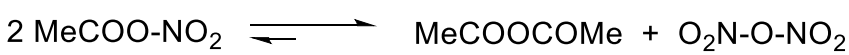
Dilute solution of H_2SO_4 : **Low concentration of $^+NO_2$.**



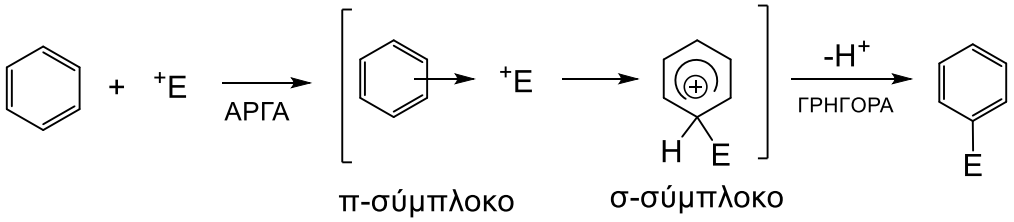


Nitration also with HF, BF_3, $HClO_4$ in the place of H_2SO_4 .

Νίτρωση με ατμίζον HNO_3 και οξικό ανυδρίτη:

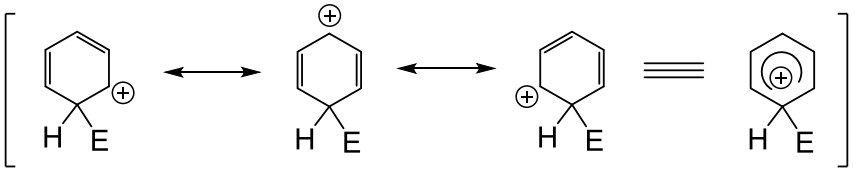


AROMATIC ELECTROPHILIC SUBSTITUTION



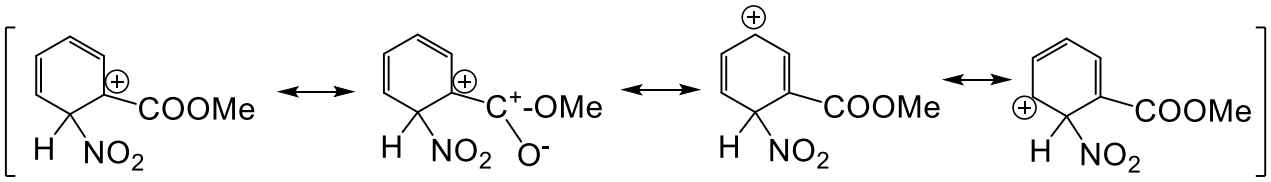
π-σύμπλοκο σ-σύμπλοκο

σ-σύμπλοκο:

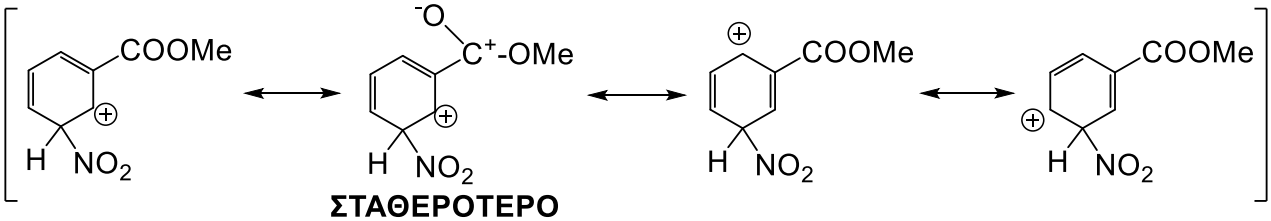


EXPLANATION for *μ*-orientation:

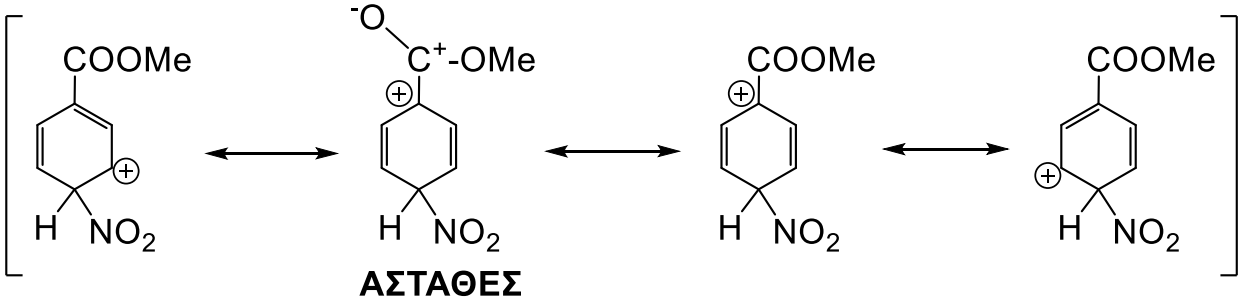
Possible intermediates:



ΑΣΤΑΘΕΣ



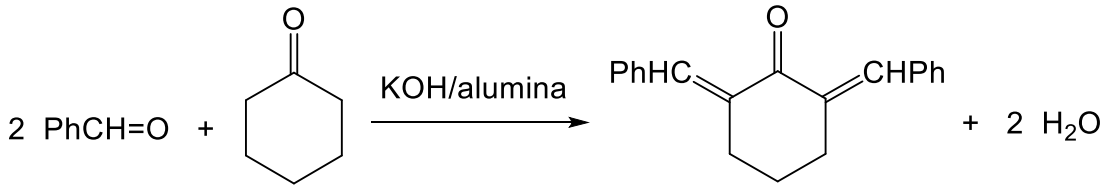
ΣΤΑΘΕΡΟΤΕΡΟ



ΑΣΤΑΘΕΣ

Aldol Condensation.
Preparation of dibenzalcyclohexanone with the catalystκαταλύτη KOH supported on Al_2O_3

Aldol Condensation benzaldehyde - cyclohexanone



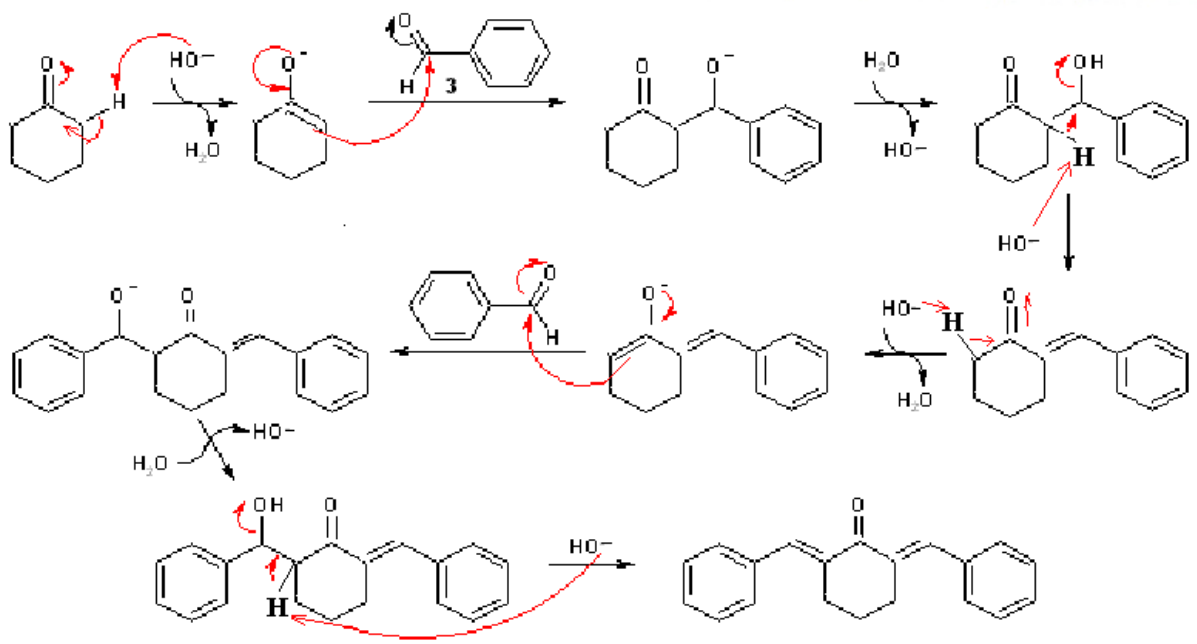
Reagents:

Alumina/KOH: 1 g

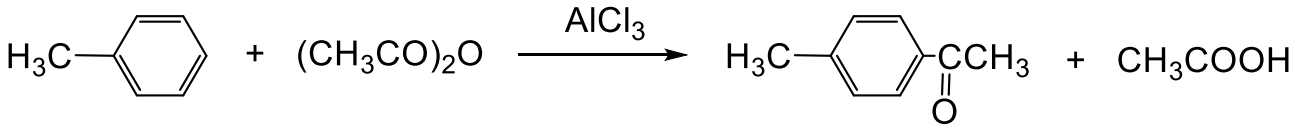
PhCH=O: 0.5 mL (d=1.04)

Cyclohexanone: 0.2 mL (d=0.95)

EtOH: 6 mL

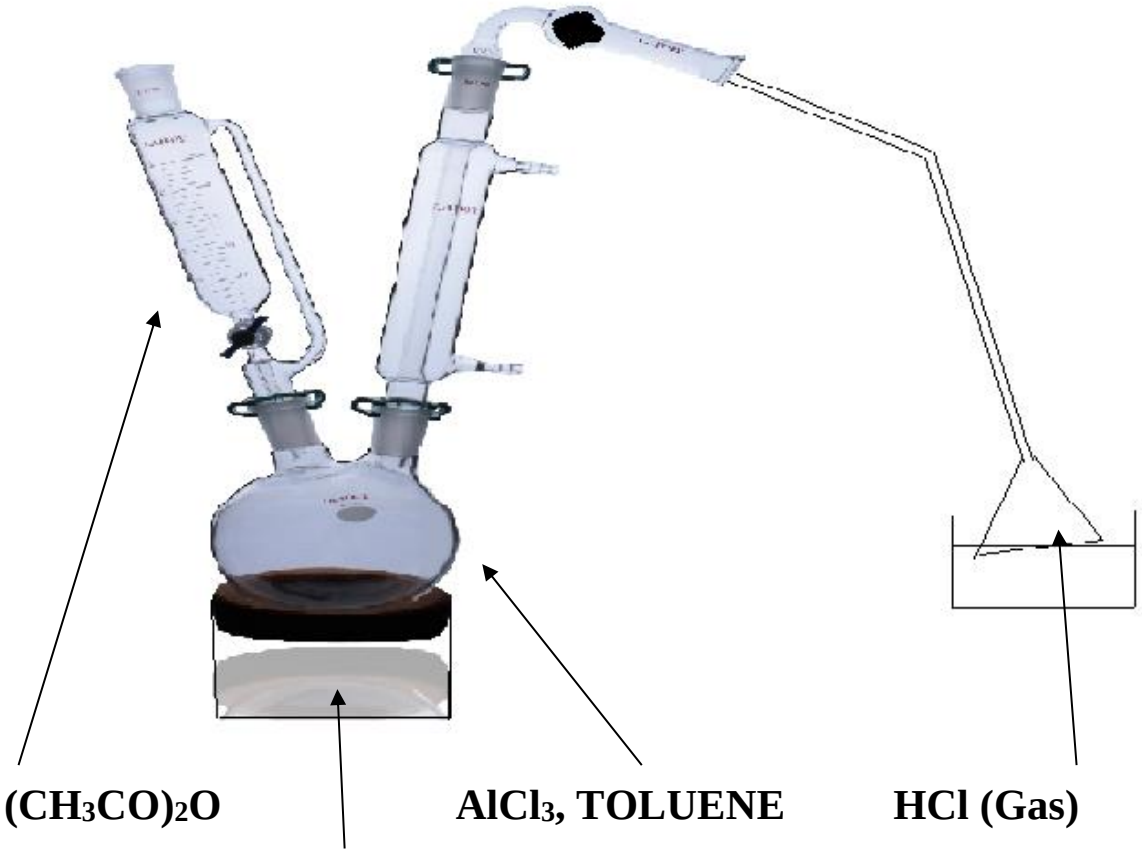


Friedel-Crafts Reaction
Preparation of p-methylacetophenone

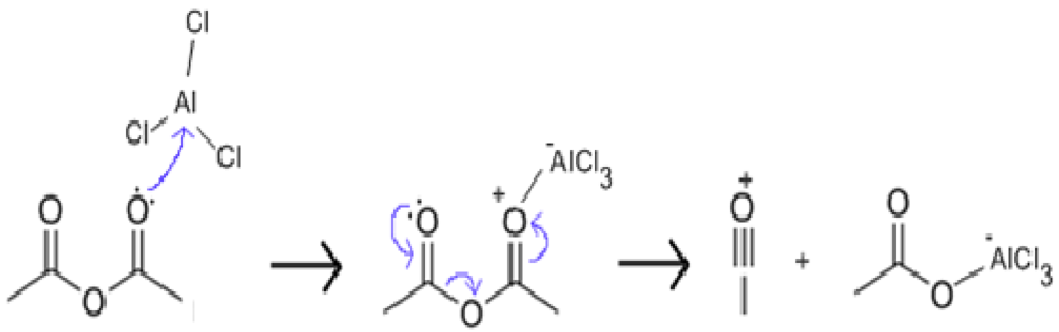


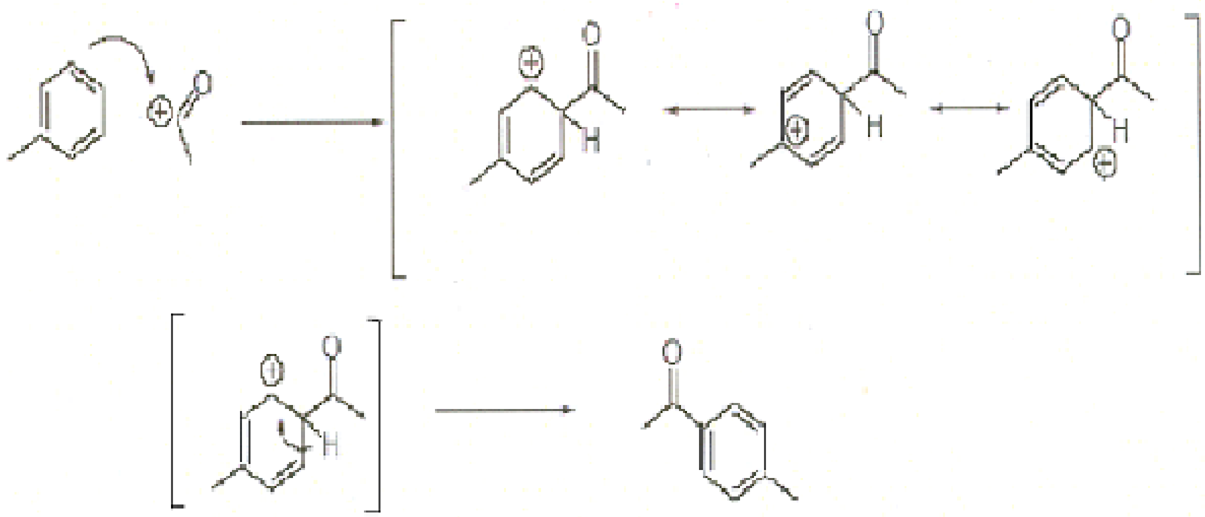
Reagents:

Toluene: 30 ml (25,5 g, 0,28 mol)
Acetic Anhydride: 5 ml (5,4 g, 0,053 mol)
$AlCl_3$: 15,5 g (0,18 mol)
HCl (πυκνό): 40 ml
NaOH 10%: 30 ml

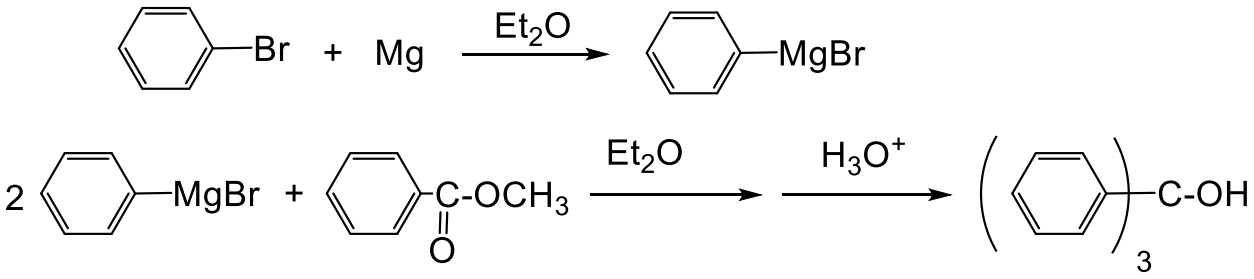


FRIEDEL-CRAFTS REACTION





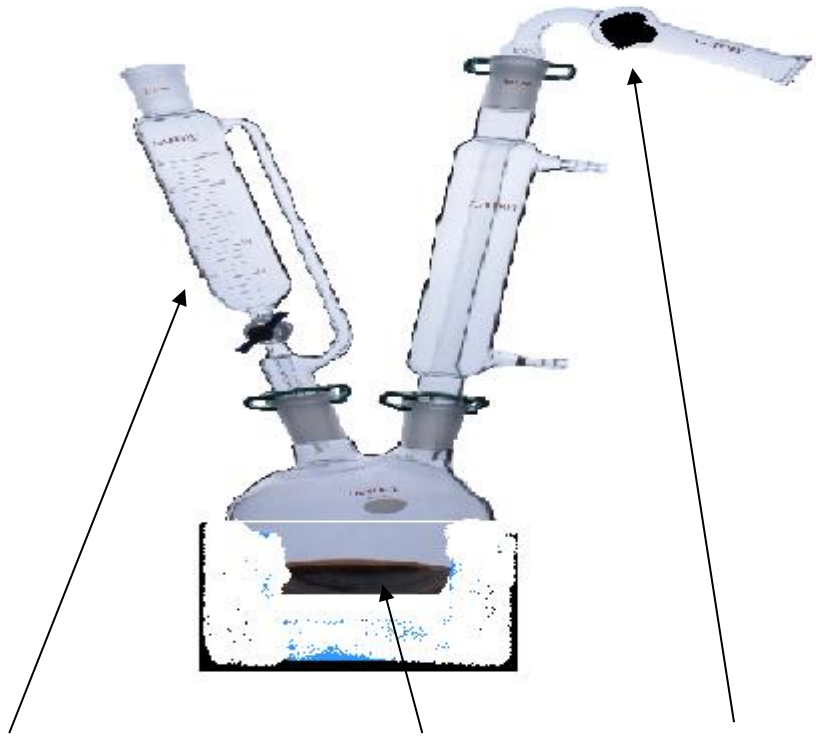
Grignard Reaction. Synthesis of Trophenylcarbinol



Reagents:

Mg: 1,1 g (0,038 mol)
Et_2O, absolute: 45 ml
Bromobenzene, dry: 3,9 ml (5,85 g, 0,037 mol)
PhCOOMe: 2,3 ml (2,5 g, 0,0185 mol)
HCl 10%: 20 ml

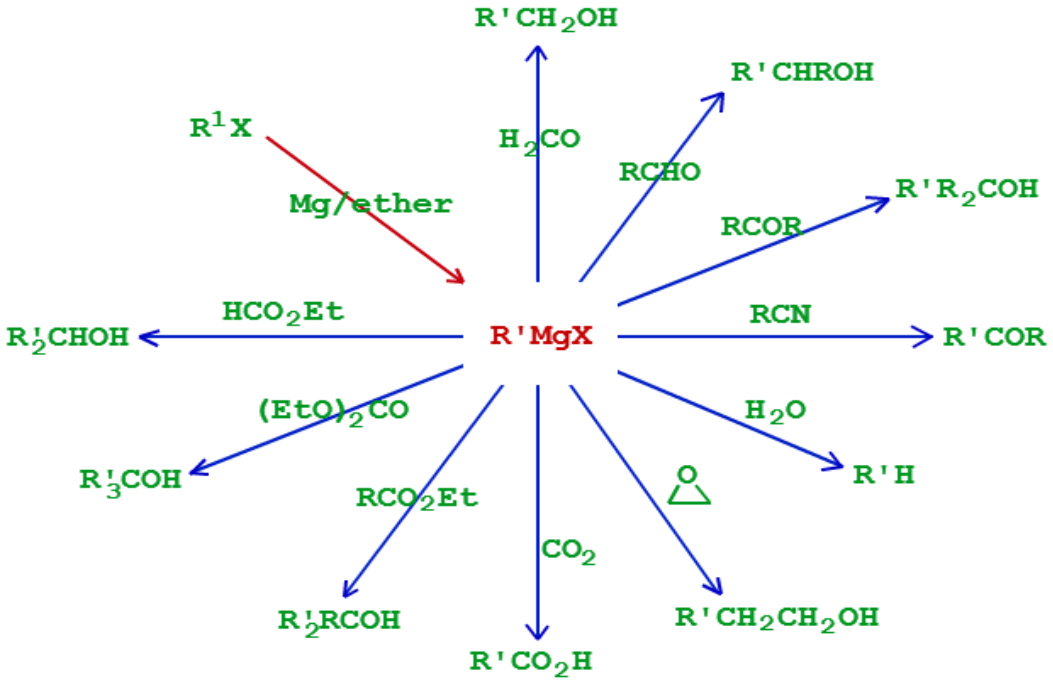
ATTENTIO!!! DRY EQUIPMENTS



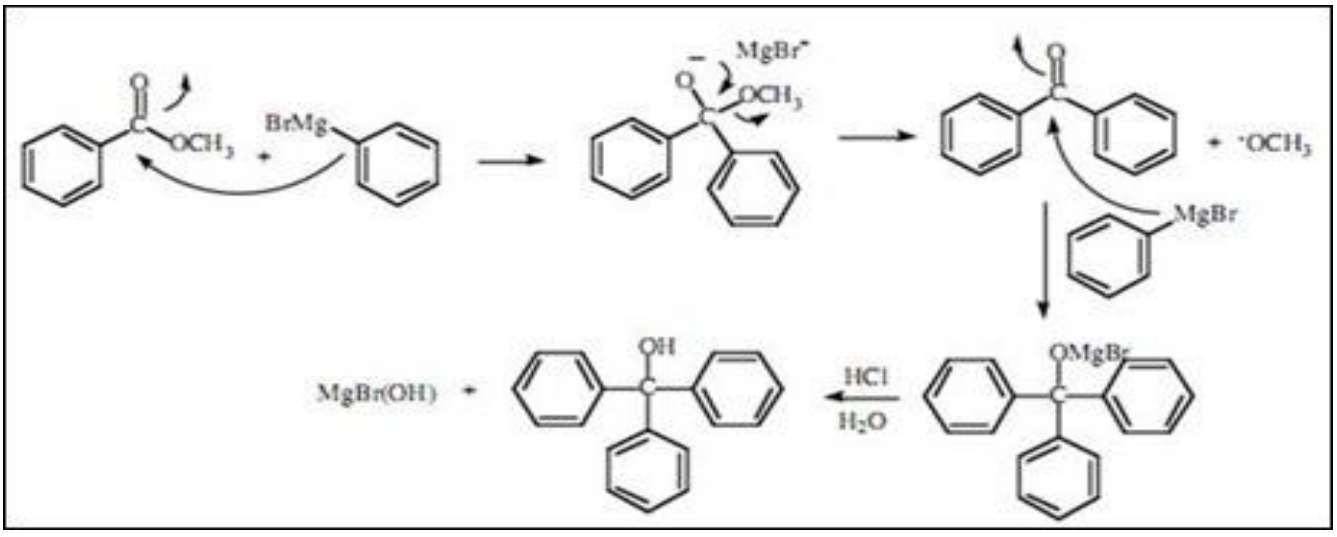
1º PhBr, Et_2O **Mg** **$CaCl_2$**

2º PhCOOMe

REACTIONS OF ORGANOMAGNESIUM COMPOUNDS



GRIGNARD REACTION MECHANISM



BYPRODUCTS: