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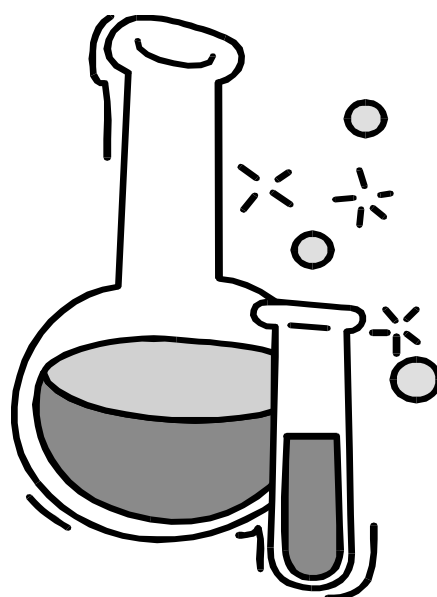
**GOMEL STATE MEDICAL UNIVERSITY**

**Department of general and bioorganic chemistry**

**V. A. Philippova, A. V. Lusenkova, L. V. Chernishova**

# **CHEMISTRY OF CARBONYL COMPOUNDS**

**LABORATORY MANUAL**



**Gomel 2008**

УДК 546.264(075.8)

ББК 28.072

Ф 53

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Ф 53 Chemistry of carbonyl compounds: laboratory manual = Химия карбонильных соединений: метод. рекомендации для лабораторно-практических занятий к курсу по общей химии для студентов лечебного факультета, обучающихся на английском языке / В. А. Филиппова, А. В. Лысенкова, Л. В. Чернышева. — Гомель: Учреждение образования «Гомельский государственный медицинский университет», 2008. — 24 с.  
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Предназначены для организации лабораторного практикума и самостоятельной работы студентов, изучающих курс биоорганической химии на английском языке. Призваны помочь в формировании целостного представления о строении и свойствах карбонильных соединений, рассматривает вопросы биохимических превращений с участием карбонильных соединений и их производных *in vivo* и *in vitro*.

Утверждено и рекомендовано к изданию Центральным учебным научно-методическим советом Учреждения образования «Гомельский государственный медицинский университет» 12 февраля 2008 г., протокол № 2.

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# CHAPTER 1

## ALDEHYDES AND KETONES

**After reading this chapter, you should be able to:**

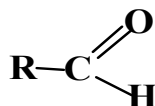
- Define aldehydes and ketones;
- Discuss their classification and nomenclature;
- Describe chemical properties of aldehydes and ketones
- Define the main steps in nucleophilic addition to carbonyl group ( $A_N$ )

### 1.1. General characteristics of aldehydes and ketones

Aldehydes and ketones are carbonyl compounds, containing carbonyl functional  $\text{>C=O}$  group:

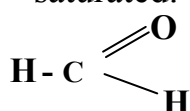
Carbonyl carbon is  $sp^2$ -hybridized thus three atoms attached to it lie in a plane with bond angles  $120^\circ$ . The carbon-oxygen double bond consists of a sigma-bond and a pi-bond. Oxygen is much more electronegative than carbon, therefore the electrons in the  $C=O$  bond are shifted to the oxygen, producing a highly polarized bond.

Aldehydes have at least one hydrogen atom attached to the carbon atom. The remaining group may be another hydrogen atom or any organic group. General formula for aldehydes may be represented as:



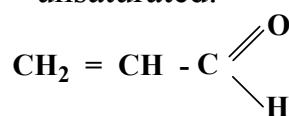
According to the radicals three types of aldehydes are distinguished:

- saturated:



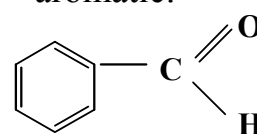
Methanal,  
formaldehyde

- unsaturated:



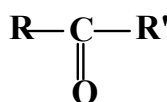
Propenal

- aromatic:



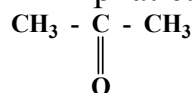
Benzoic aldehyde

In ketones the carbonyl carbon is connected to two radicals. General formula for ketones is:



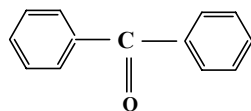
According to the radicals ketones are:

- Aliphatic:



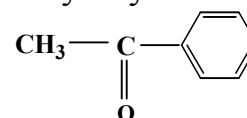
Acetone

- Aromatic:



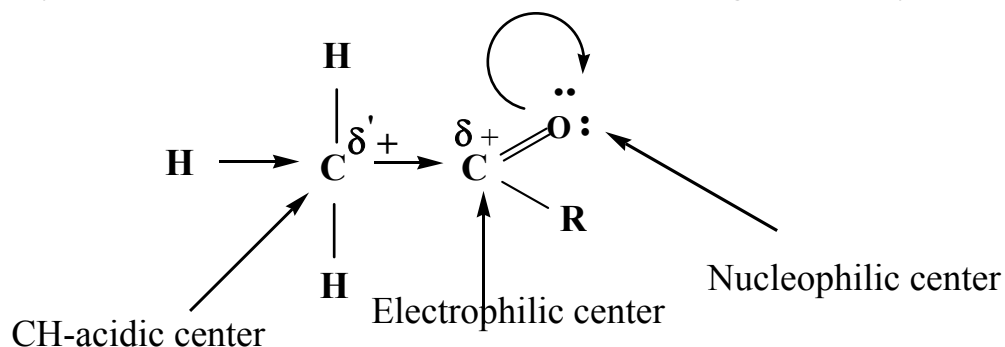
Diphenylketone

- Alkyl aryl ketones:



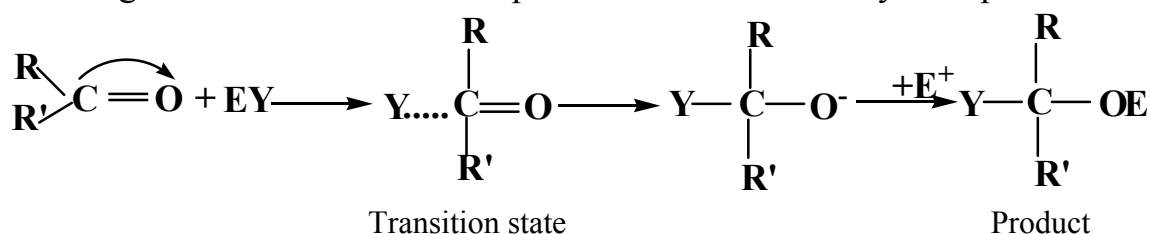
Methylphenylketone

The aldehydes and ketones molecules contain the following reactionary centers:

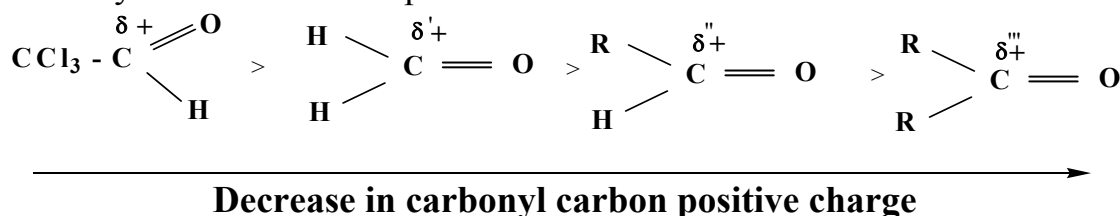


## 1.2. Nucleophilic addition to carbonyl group — $A_N$

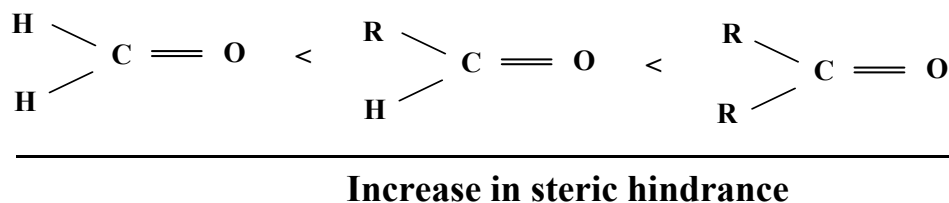
The general scheme for nucleophilic addition to carbonyl compounds is:



Due to electrophilic centers in their molecules, carbonyl compounds attract nucleophiles. The greater is positive charge on carbonyl carbon, the higher is reactivity of substrates toward  $A_N$  reactions. In general, ketones are less reactive than aldehydes toward nucleophiles

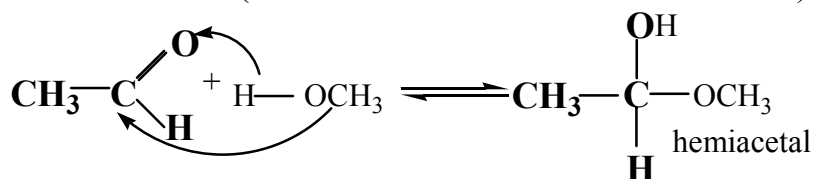


Steric reason for low reactivity of ketones: the carbonyl carbon is more crowded in their molecules than that in aldehydes

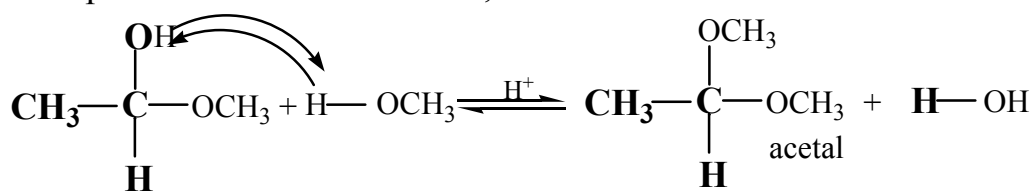


The following reactions occur according to  $A_N$  mechanism:

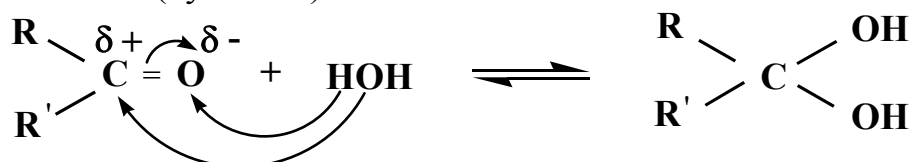
➤ Addition of alcohols (formation of hemiacetals and acetals):



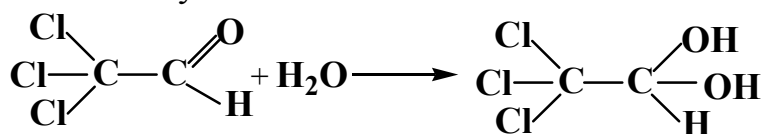
In the presence of excess alcohol, hemiacetals react further to form acetals:



➤ Addition of water (hydration):

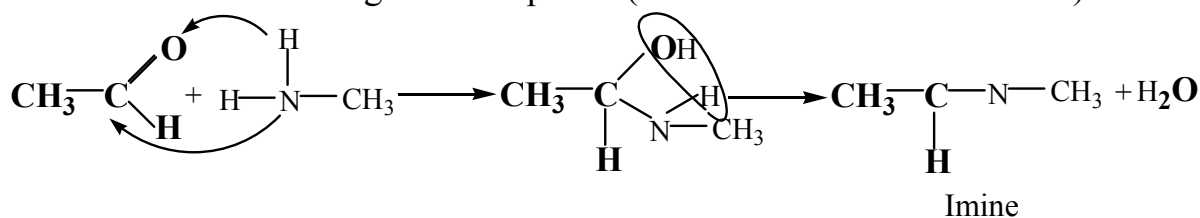


An exception is trichloroacetaldehyde (chloral), which forms a stable crystalline hydrate. Chloral Hydrate is used in medicine as a sedative.



ChloralChloral Hydrate

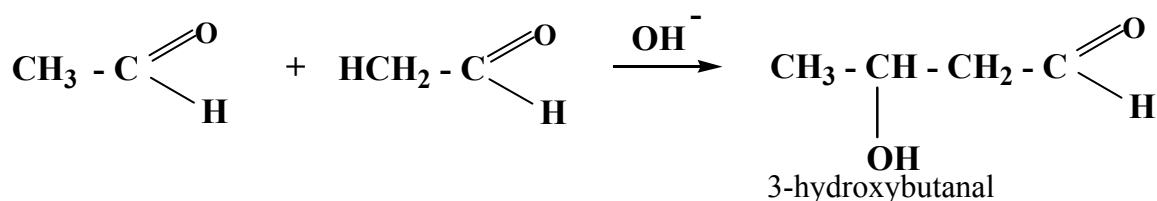
➤ Addition of nitrogen nucleophiles (addition-elimination reaction):



➤ The aldol condensation.

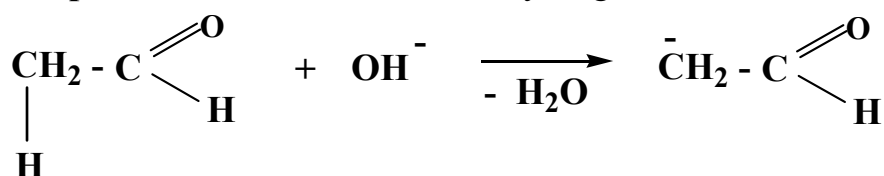
The aldol condensation is an extremely useful carbon-carbon bond-forming reaction which is widespread in nature. It's an important biochemical reaction which takes place in plants' and animals' cells. Only those aldehydes and ketones can be involved in these processes which contain the  $\alpha$ -hydrogen.

The simple example of an aldol condensation is the combination of two acetaldehyde molecules, which occurs when a solution of acetaldehyde is treated with catalytic amounts of aqueous base:



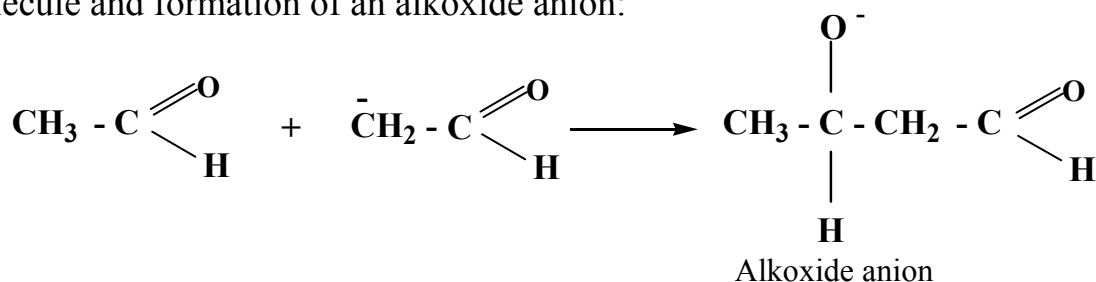
Mechanism of aldol condensation involves three steps:

**Step 1:** The base removes the  $\alpha$ -hydrogen to form an enolate anion:

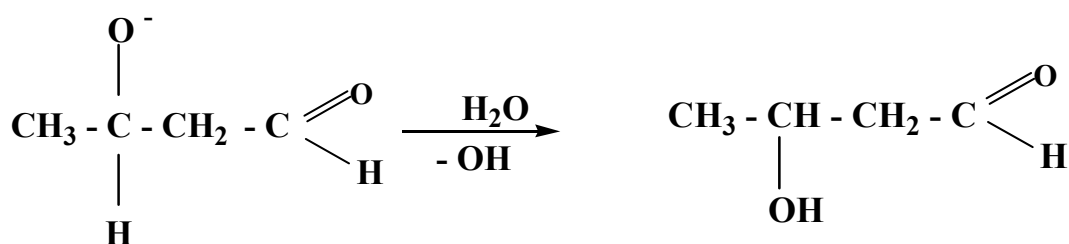


Enolate anion

**Step 2:** Enolate anion attack on a carbonyl carbon of the second aldehydes molecule and formation of an alkoxide anion:



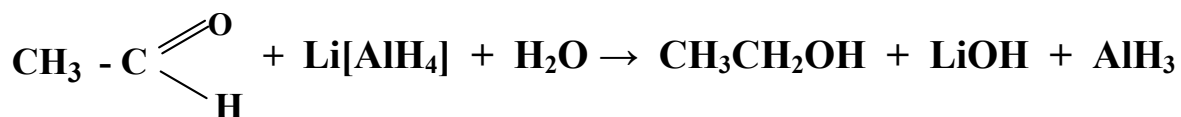
**Step 3:** An alkoxide anion accepts a proton from the solvent thus regenerating the hydroxide ion needed for the first step:



### 1.3. Reduction of carbonyl compounds

Aldehydes and ketones are easily reduced to primary and secondary alcohols respectively. Reduction may be accomplished in many ways, but commonly by metal hydrides. The most common metal hydrides used to reduce carbonyl compounds are lithium aluminum hydride (Li[AlH<sub>4</sub>]) and sodium boron hydride (Na[BH<sub>4</sub>]).

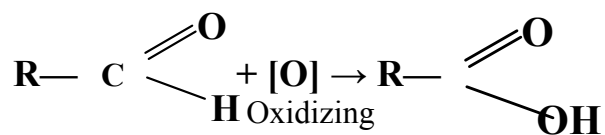
The reaction involves irreversible nucleophilic attack of the hydride (H<sup>-</sup>) at the carbonyl carbon:



In vivo the coenzyme NADH is the main reducing agent in corresponding reactions.

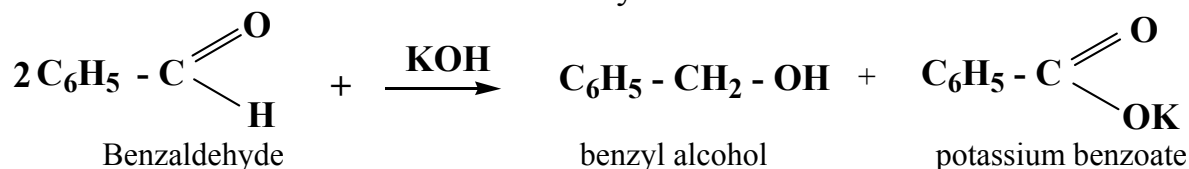
### 1.4. Oxidation of carbonyl compounds

Aldehydes are more easily oxidized than ketones. Oxidation of aldehydes gives acids with the same number of carbon atoms.



### 1.5. Disproportionation of aldehydes (Cannizzarro-Tischenko reaction)

In 1853 the Italian chemist S.Cannizzarro noted that when aldehydes that lack an  $\alpha$ -hydrogen were treated with strong base, a disproportion reaction occurred where half the aldehydes was reduced to corresponding alcohol, and the other half was oxidized to the carboxylic acid:



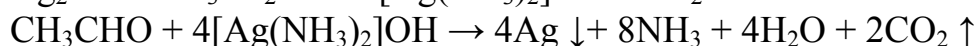
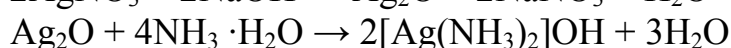
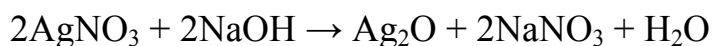
### 1.6. Laboratory work

#### ALDEHYDES AND KETONES

#### **Test 1: FORMALDEHYDE AND ACETONE OXIDATION BY HEAVY METALS OXIDES IN BASIC MEDIUM**

##### *(a) The Tollens' silver mirror test*

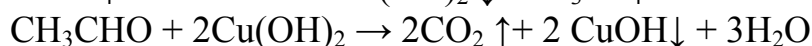
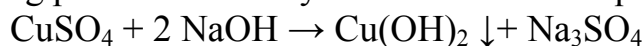
Add 2–3 drops of 5% silver nitrate ( $\text{AgNO}_3$ ) solution into a test tube and treat it by one drop of 10% sodium hydroxide ( $\text{NaOH}$ ) solution. Dissolve the prepared dark-brown precipitate in the excess of aqueous ammonia and treat the prepared solution by 4 drops of 40% formalin. What can you see? Write the equations of fulfilled reactions:



Repeat the experiment using acetone instead of formalin. Can you see any analytical effects? Why?

##### *(b) Oxidation by Copper (II) Hydroxide*

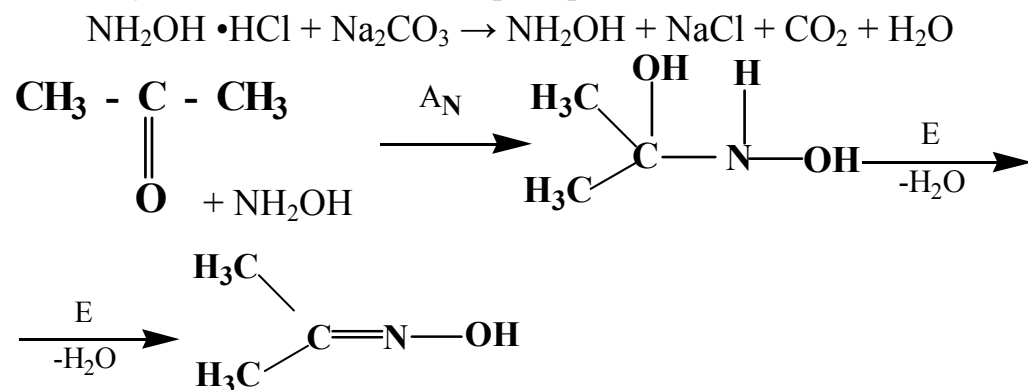
Pour 8–10 drops of 10% sodium hydroxide solution and distilled water into a test tube and add 1–5 drops of 2% copper (II) sulfate  $\text{CuSO}_4$  solution. Treat the prepared precipitate by 3–6 drops of 40% formalin. Heat a mixture up to the boiling point. What can you see? Write the equations for the fulfilled reactions:



Repeat the experiment using acetone instead of formalin. Can you see any analytical effects? Why?

#### **Test 2: ACETONE OXIME PREPARING**

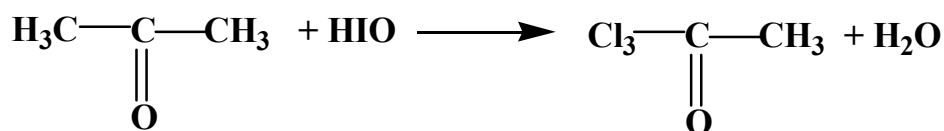
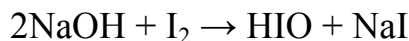
Put some crystals of hydroxylamine hydrochloride  $\text{NH}_2\text{OH} \cdot \text{HCl}$  into a test tube and add some crystals of soda. Dissolve the prepared mixture in 10–25 drops of distilled water. After carbon dioxide gas liberation add 15–20 drops of acetone into a test tube and stir. The reaction is highly exothermic. It results in white crystals of acetone oxime precipitation:



### **Test 3: ACETONE IDENTIFICATION BY IODOPHORME TEST**

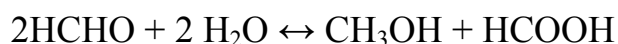
This clinical test is used in biochemical labs for diabetes mellitus diagnosing.

Pour 5 drops of iodine solution into a test tube and treat it by 10% sodium hydroxide solution. When a solution becomes colorless add 1–3 drops of acetone into it and heat a test tube in your hands. What can you see? Write the equations for the fulfilled reactions:



### **Test 4: DISPROPORTIONATION OF FORMALDEHYDE (CANNIZZARRO-TISCHENKO REACTION)**

Pour 2–3 drops of 40% formalin into a test tube and treat it by 1 drop of 0.2% methyl red indicator solution. What can you see? Write the equations for fulfilled reactions:



### **Test 5: OXIDATION OF ETHYL ALCOHOL**

Pour 1 mL of potassium dichromate solution into a test tube and add the equal volume of dilute sulfuric acid. Treat the prepared solution by 1 mL of ethyl alcohol under heating. What can you see? Write the equations for the fulfilled reactions.

#### **1.7. Exercises for the self-control**

1. Write a structural formula for each of the following:



- (a) 4-methylpentanal  
(c) octanone-3  
(e) benzylphenylketone
- (b) penten-2-al  
(d) 1-phenylbutanone-2  
(f) 2,2-dibromohexanal

2. Write an equation for the synthesis of pentanone-2 by

- (a) oxidation of an alcohol  
(b) hydration of an alkyne

3. Write an equation for the reaction of p-bromobenzaldehyde with each of the following reagents. Name the organic product.

- (a) Tollens' reagent  
(c) Methylamine  
(e) Lithium aluminum hydride
- (b) hydroxylamine  
(d) excess methanol, dry HCl  
(f) phenylhydrazine

4. Write an equation for the formation of a hemiacetals from acetaldehyde, ethanol, and  $H^+$ . Show all steps in the reaction mechanism.

5. Write the equation for aldol condensation, which occurs when a solution of ethanal is treated with catalytic amounts of aqueous base. Describe its mechanism.

6. Write an equation for the reaction of ethanal with ethyl amine. Name the product.

7. Write an equation for benzaldehyde disproportionation.

8. Write the equation for propanal reduction.

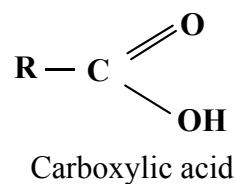
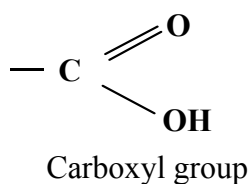
## CHAPTER 2 CARBOXYLIC ACIDS

**After reading this chapter, you should be able to:**

- Define carboxylic acids and their functional derivatives;
- Describe classification and nomenclature of carboxylic acids;
- Discuss chemical properties of carboxylic acids and their functional derivatives;
- Define all steps in Nucleophilic substitution mechanism ( $S_N$ )

### 2.1. Classification of carboxylic acids

Carboxylic acids are the most important organic acids; their functional group is the **carbonyl group**. This name is a contraction of the parts: the carbonyl and hydroxyl groups. General formulas for carboxyl group and carboxylic acid are:



Because of their abundance in nature, carboxylic acids were among the earliest classes of compounds studied by organic chemists. Many acids have common names that indicate the original source of the acid.



Stinging ants are source of formic acid,  $\text{HCOOH}$



The valerian root is a source of valeric acid  $\text{CH}_3(\text{CH}_2)_2\text{COOH}$

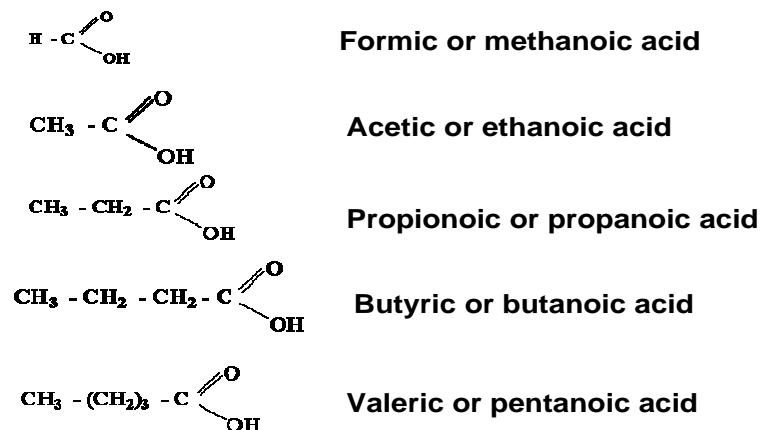
Carboxylic acids are classified by

- 1) a number of carboxyl groups in a molecule:
  - monocarboxylic acids;
  - dicarboxylic acids;
  - tricarboxylic acids
- 2) a nature of radicals adjacent to carboxyl acids:
  - Saturated carboxylic acids;

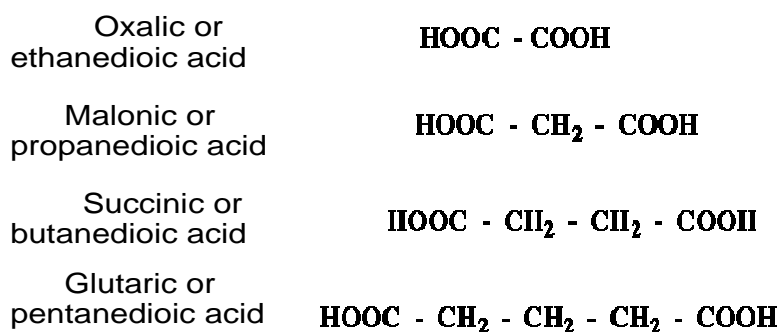
- Unsaturated carboxylic acids;
- Aromatic carboxylic acids/

Let's review the main homologous series of carboxylic acids

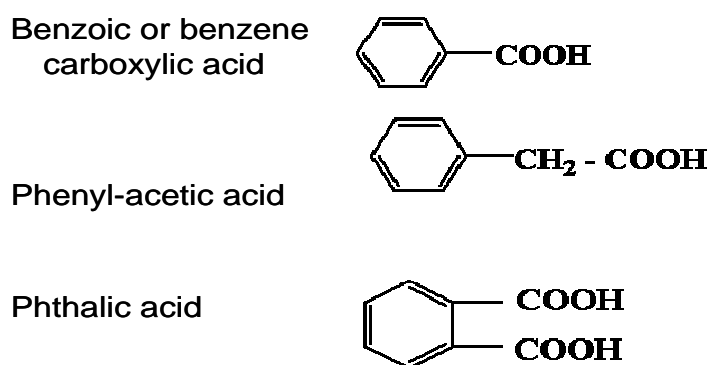
### (a) Saturated Monocarboxylic acids



### (b) Saturated Dicarboxylic Acids



### (a) Aromatic carboxylic acids



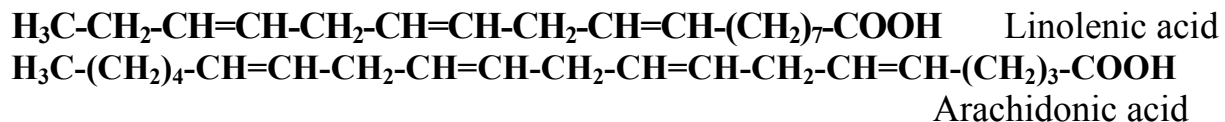
### (d) Unsaturated carboxylic Acid

$\text{H}_2\text{C} = \text{CH} - \text{COOH}$  Acrylic or propenoic acid

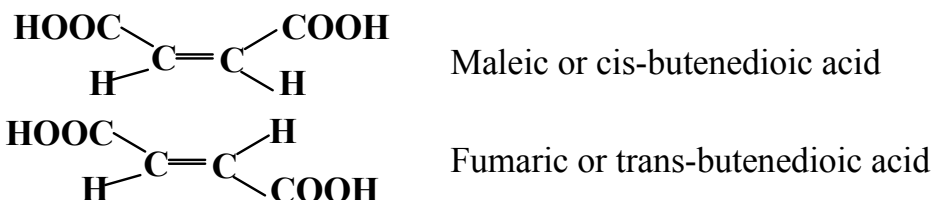
$\text{H}_3\text{C} - \text{CH} = \text{CH} - \text{COOH}$  Crotonic or butane-2-oic acid

$\text{H}_3\text{C} - (\text{CH}_2)_7 - \text{CH} = \text{CH} - (\text{CH}_2)_7 - \text{COOH}$  Oleic acid

$\text{H}_3\text{C} - (\text{CH}_2)_4 - \text{CH} = \text{CH} - \text{CH}_2 - \text{CH} = \text{CH} - (\text{CH}_2)_7 - \text{COOH}$  Linoleic acid

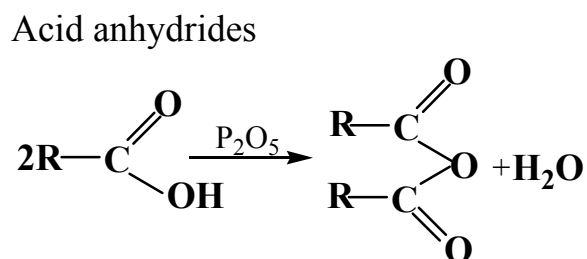
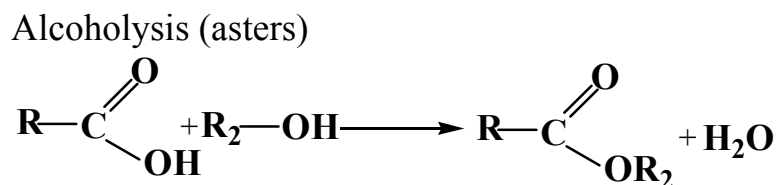
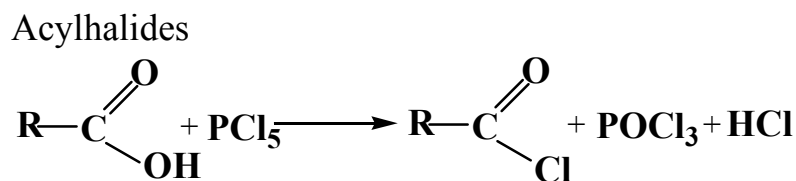
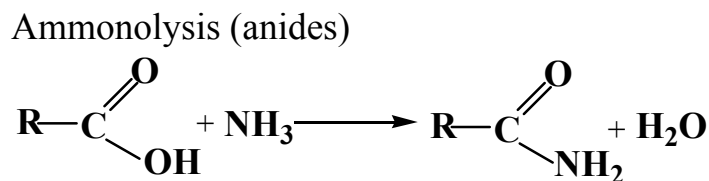
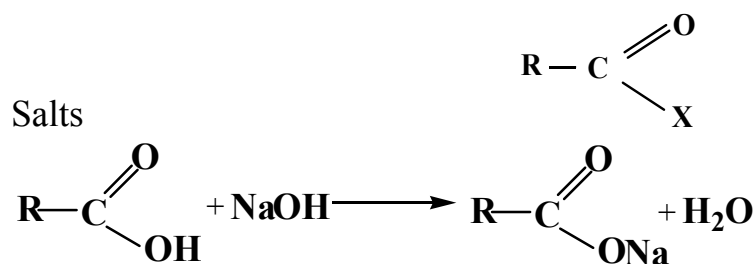


### (e) Unsaturated Dicarboxylic acids



## 2.2. Functional derivatives of carboxylic acids

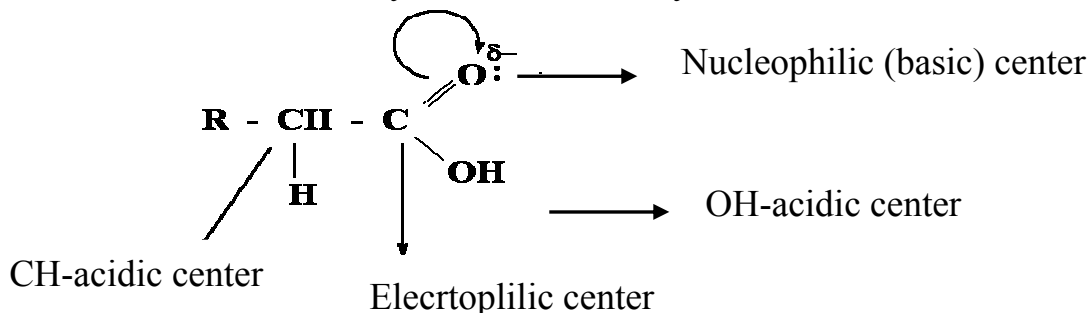
Carboxylic acid derivatives are compounds in which the – OH group of an acid is replaced by other functions:



Esters and amides occur widely in nature. Anhydrides are uncommon in nature. Acyl Halides are strictly creatures of labs.

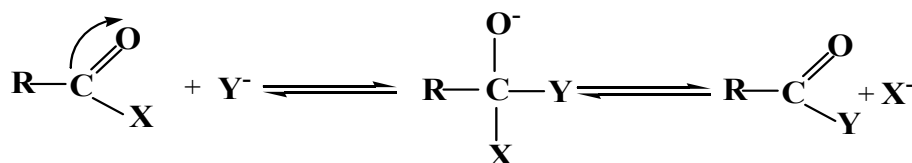
### 2.3. Chemical properties of carboxylic acids - S<sub>N</sub>

Reactionary centers in carboxylic acids



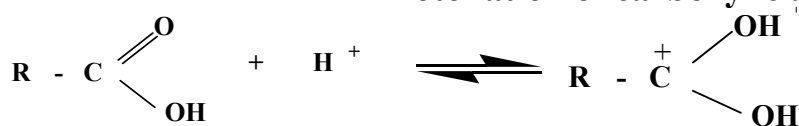
$\pi$ -Bond in carboxylic acids is more stable than that in aldehydes and ketones because it is stabilized through resonance. This is a reason why acids undergo substitution but not addition.

Reactions of **Nucleophilic Substitution** are the typical reactions for carboxylic acids and their derivatives. Their mechanism may be represented by the following scheme:



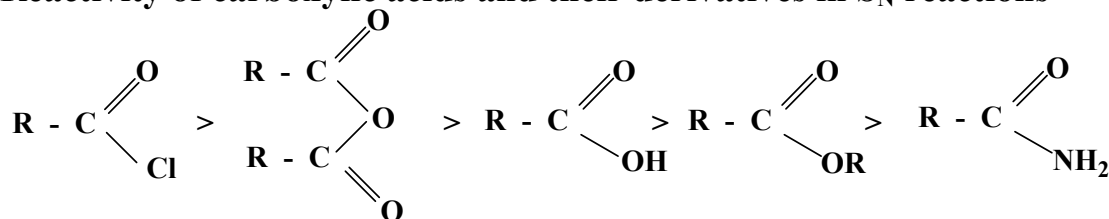
This mechanism is available if X<sup>-</sup> is a good leaving group and Y<sup>-</sup> is strong nucleophile. If not, H<sup>+</sup> ought to be used as a catalyst.

#### Protonation of carboxylic acids



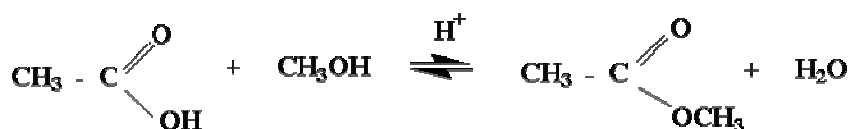
The greater is positive charge on carbonyl carbon, the higher is reactivity of carboxylic acids and their derivatives

#### Reactivity of carboxylic acids and their derivatives in S<sub>N</sub> reactions



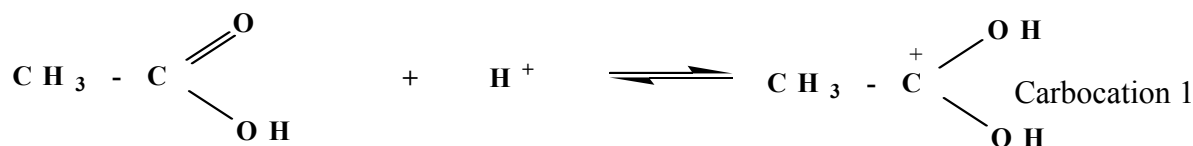
#### S<sub>N</sub> reactions reviewed:

The most important chemical property of carboxylic acids is their interaction with alcohols (esterification reactions).

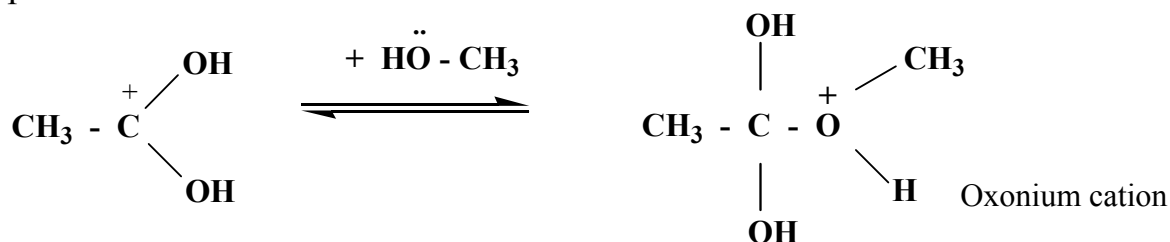


Its mechanism involves 4 steps:

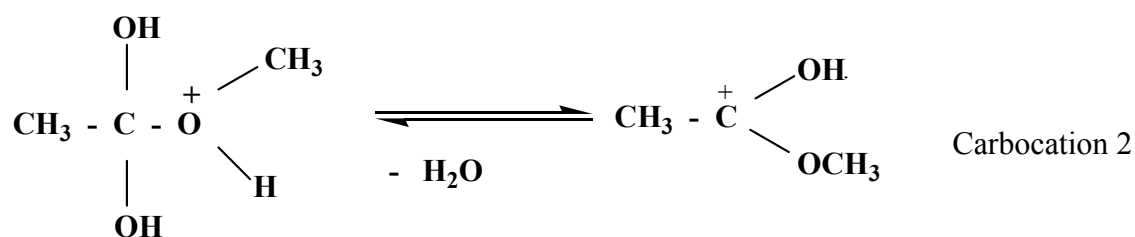
**Step 1. Protonation**



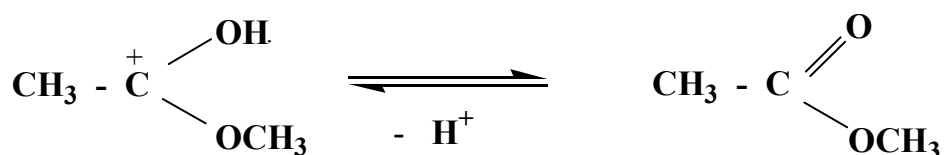
**Step 2.** A nucleophilic attack of water to the electrophilic center of the protonated ester



**Step 3.** Elimination of water; poor leaving OH-group leaves from a substrate as water molecule

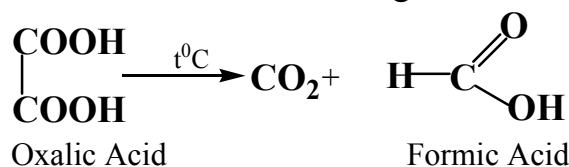


**Step 4.** Deprotonation and regeneration of an acid catalyst

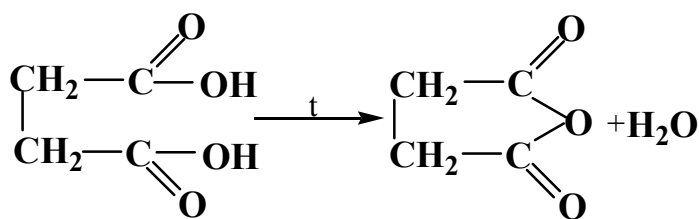


Other important chemical properties of carboxylic acids are:

- **Decarboxylation** of dicarboxylic acids is an essential biochemical process which is energetically favorable because of high stability of  $\text{CO}_2$  molecule. Electron withdrawing substituents on  $\alpha$ -position promote it.



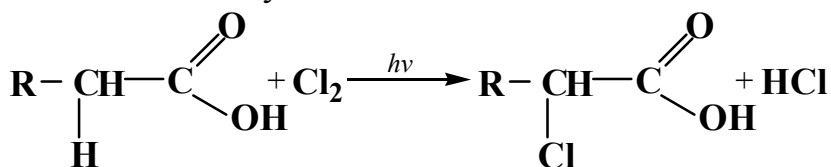
- Succinic and glucaric acids undergo **dehydration and cyclization** under heating. This reactionary pathway is preferable because it results in the formation of five- and six- membered rings. Such rings are practically free of strain and stable.



Succinic Acid

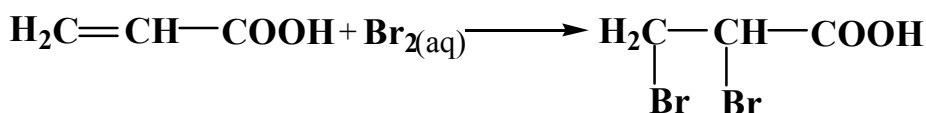
Cyclic Anhydrate

- Saturated carboxylic acids with  $\alpha$ -carbon are active in **halogenation reactions**.



Halide derivatives are used in preparing  $\alpha$ -amino- and  $\alpha$ -hydroxy carboxylic acids.

- For unsaturated acids addition of halogens is their analytical test



## 2.4. Esters and their Hydrolysis

Esters are the most important derivatives of carboxylic acids. Most of them are rather pleasant-smelling substances and are responsible for the flavor and fragrance of many fruits and flowers. Lipids are esters of glycerol and fatty acids. Some esters are applied as medicines. E.g., aspirin is the most popular pain killer in the world:

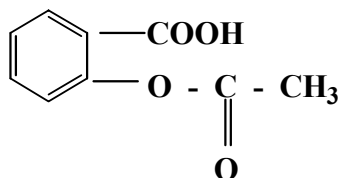


Figure 1 — Aspirine

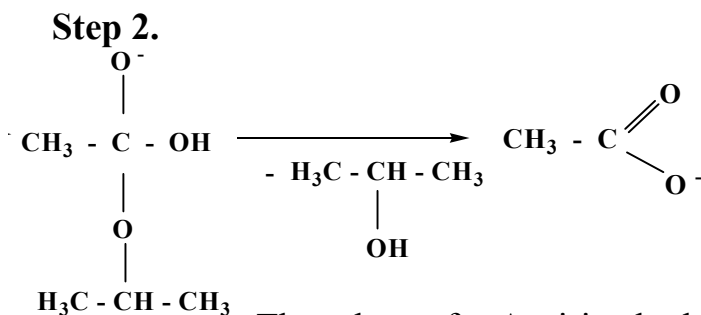


Aspirin has been used to treat toothaches, headaches, arthritis and other pain maladies for 100 years. But only in 1969 Dr. John Vane came to understand how it works. People feel pain when injured cells produce and release prostaglandins by oxidation of arachidonic acid. Aspirin inhibits the production of prostaglandins by injured tissue thus reducing pain and inflammation.

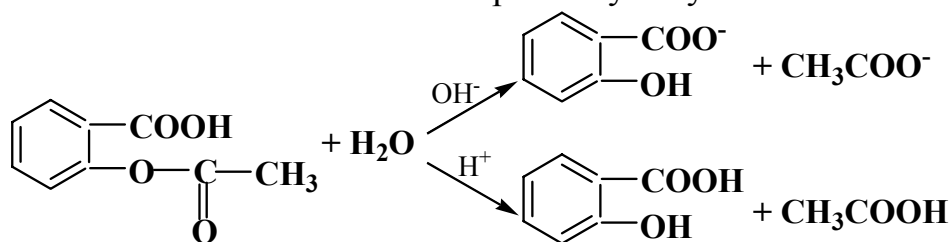
Sir John is credited with discovering how aspirin and similar drugs produce their effects; his work provided a scientific basis for the pain-relieving and anti-inflammatory effects of aspirin and also provided an explanation for how aspirin prevented blood clots and helped prevent heart attacks and strokes. As a result, aspirin is still one of the most common drugs used to treat people with heart disease.







The scheme for Aspirine hydrolysis:



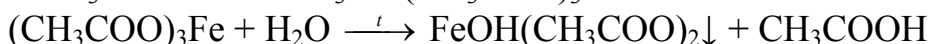
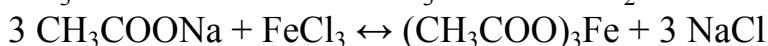
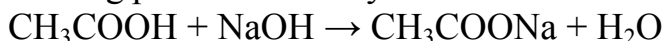
In vivo aspirin undergoes enzymatic hydrolysis.

## 2.5. Laboratory work

### CARBOXYLIC ACIDS

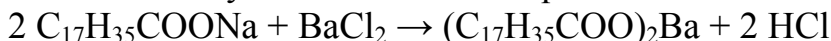
#### Test 1. PREPARING AND HYDROLYSIS OF FERRUM (III) ACETATE

Pour 3 drops of acetic acid and 3 drops of distilled water into a test tube. After determining the pH of a prepared solution add 2–3 drops of 10% NaOH solution to make it neutral. Treat a mixture with 2–3 drops of 1% FeCl<sub>3</sub> solution and heat it up to the boiling point. What can you see? Write the equations of fulfilled reactions:



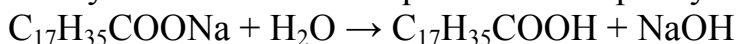
#### Test 2. PREPARING OF INSOLUBLE SALTS OF FATTY ACIDS.

Pour soap's solution into the test tube and treat it with 10% barium chloride solution. What can you see? Write the equation of a fulfilled reaction:



#### Test 3. HYDROLYSIS OF SOAPS

Pour soap's solution into the test tube and add some drops of solution. What can you see? Write the equation for soap's hydrolysis:



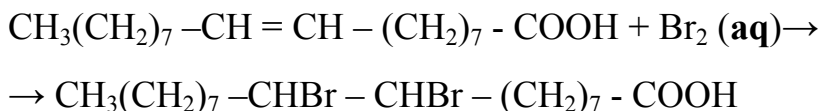
#### Test 4. CHEMICAL PROPERTIES OF OLEIC ACID

##### A. Oleic acid oxidation by potassium permanganate solution

Pour 2 drops of oleic acid into a test tube and add 2 drops of 1% potassium permanganate solution and 1 drop of 5% sodium carbonate solution into it. Stir the mixture. What can you see? Write the equation of a fulfilled reaction.

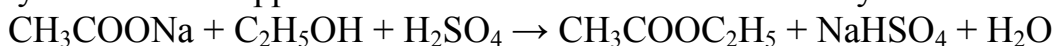
**B. Bromine addition to oleic acid**

Pour 0.5 mL of bromine water into a test tube and add 3–4 drops of oleic acid into it. Stir the mixture. What can you see? Write the equation of a fulfilled reaction.



**Test 5. ETHYL ACETATE PREPARING**

Put some crystals of sodium acetate into a dry test tube and mix them with 0.5 mL of ethyl alcohol. Treat a prepared mixture with 2–5 drops of concentrated sulfuric acid and heat it in the fire of a spirit lamp. In few minutes a pleasant fresh fragrance of ethyl acetate must appear. This reaction is used as a test on ethyl alcohol:



**2.6. Exercises for the self-control**

1. Write a structural formula for each of the following acids.

- |                                  |                                |
|----------------------------------|--------------------------------|
| (a) 4-methylpentanoic acid       | (b) 3-oxobutanoic acid         |
| (c) phenylacetic acid            | (d) cyclobutanecarboxylic acid |
| (e) 2,2-dimethylbutanedioic acid | (f) 1-naphthoic acid           |

2. Write a structural formula for each of the following compounds.

- |                              |                     |
|------------------------------|---------------------|
| (a) sodium 2-chlorobutanoate | (b) calcium acetate |
| (c) Phenyl benzoate          | (d) o-toluamide     |
| (e) propanoic anhydride      | (f) ethyl formate   |

3. In each of the following pairs of acids, which would be expected to be the stronger, and why?

- |   |
|---|
| (a) $\text{ClCH}_2\text{COOH}$ and $\text{BrCH}_2\text{COOH}$                             |
| (b) $\text{C}_6\text{H}_5\text{COOH}$ and $p\text{-CH}_3\text{OC}_6\text{H}_4\text{COOH}$ |
| (c) $\text{CCl}_3\text{COOH}$ and $\text{CF}_3\text{COOH}$                                |

4. Write an equation for:

- |  |
|--|
| (a) hydrolysis of propanoyl chloride                   |
| (b) esterification of 1-pentanol with acetic anhydride |
| (c) ammonolysis of butanoyl bromide                    |
| (d) reaction of benzoyl chloride with methanol         |
| (e) Succinic acid + heat ( $235^\circ\text{C}$ )       |

5. Write out each step in the mechanism for

- |   |
|---|
| (a) esterification of benzoic acid with methanol      |
| (b) esterification of propanoic acid with isopropanol |

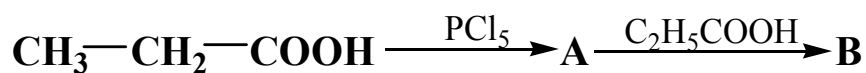
(c) acid-catalyzed hydrolysis of ethylacetate

6. Write out all the steps in the mechanism for

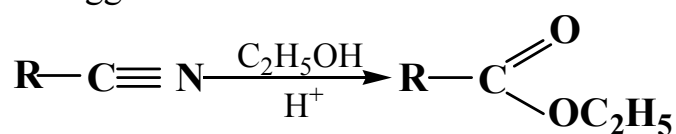
(a) Saponification of  $\text{CH}_3\text{CH}_2\text{COOCH}_3$

(b) Ammonolysis of  $\text{CH}_3\text{CH}_2\text{COOCH}_3$

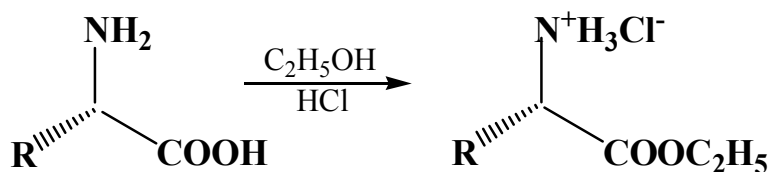
7. Write the equations for the following scheme. Describe the mechanism for the second reaction. Give systematic names for the products.



8. Esters can be made directly from Nitriles by acid-catalyzed reaction with appropriate alcohol. Suggest a mechanism.



9. In making esters of the naturally occurring amino acids (general formula below) it is important to keep them as their hydrochloric salts. What would happen to these compounds if they were neutralized?



## LITERATURE

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2. *Leo J. Malone*. Basic concepts of chemistry. — Fourth education. — 1994.
3. *Tikavy, V.* General chemistry in formulas, definitions, and diagrams. — Ct. Peterburg. — 2000.

## CLOSSARY

**Acidity constant.** ( $K_a$ ) A quantitative measure of the strength of an acid; the greater  $K_a$ , the greater the acid strength.

**Acyl halides.** A carboxylic acid derivative in which the — OH group is replaced by a halogen atom.

**Aldehydes.** Compounds with at least one hydrogen atom bonded to the carbon of a carbonyl group.

**Aldol condensation.** A reaction in which an enolate anion adds to the carbonyl group of an aldehyde or ketone to give an aldol.

**Carbonyl group.** The functional group C=O.

**Carboxylic acid(s).** Compounds with a carboxyl group.

**Ethers.** Compounds with two alkyl or aryl groups bonded to the same oxygen.

**Fatty acids.** Long-chain carboxylic acids obtained from saponification of fats and oils.

**Hemiacetal(s).** A compound with one alkoxy group and one hydroxyl group bonded to the same carbon atom.

**Hydrogen bond.** An attractive interaction that occurs between hydrogen atoms bonded to highly electronegative atoms (O, N, F) and the nonbonding electron pairs on other highly electronegative atoms.

**Nucleophilic acyl substitution.** A substitution reaction in which the hydroxyl group of a carboxylic acid is replaced with another group; also used in reference to conversion of one carboxylic acid derivative to another carbonyl compound.

**Oxidation reaction(s).** A reaction that increases the oxidation state of atoms in a molecule or ion. In organic chemistry this frequently involves reactions in which C—H bonds are replaced by C — O bonds. It is a net decrease in the number of bonds to hydrogen or electropositive elements, or a net increase in the number of bonds to electronegative elements. A net loss of electrons.

**Reaction mechanism.** A step-by-step description of the bond-breaking and bond-making processes that occur when reagents react to form products.

**Reaction rate.** How fast a reaction takes place.

**Reduction.** It is a net increase in the number of bonds to hydrogen or electropositive elements, or a net decrease in the number of bonds to electronegative elements. A net gain of electrons.

**Saponification.** The reaction of fats and oils with aqueous sodium hydroxide to give glycerol and sodium salts of fatty acids (soaps); also used in reference to hydrolysis of any ester.

**Soaps.** The salts (usually sodium) of long-chain fatty acids.

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(на английском языке)

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