

Progress in Chemical and Biochemical Research

Journal homepage: www.pcbiochemres.com



Review Article

Practical Approach for the Identification of Functional Groups in Organic Compounds

Ayman Y. El-Khateeb

Agricultural Chemistry Department, Faculty of Agriculture, Mansoura University, Mansoura, Egypt.

GRAPHICAL ABSTRACT ABSTRACT



ARTICLE INFO

Article history:

Submitted: 2020-03-19 Revised: 2020-04-21 Accepted: 2020-04-23 Published: 2020-05-10

Manuscript ID: PCBR-2003-1086

KEYWORDS

Organic chemistry, Alcohols,

Aldehydes, Ketones,

Organic acids

* Corresponding author: **Ayman Y. El-Khateeb** (E-mail: aymanco@mans.edu.eg); Tel: +20502245274; Fax: +20502221688 ORCID link: orcid.org/0000-0003-4014-524X.

DOI: 10.33945/SAMI/PCBR.2020.2.9 © 2020 by SPC (Sami Publishing Company)

Organic reactions are chemical reactions involving organic compounds. The basic organic chemistry reaction types are addition reactions, elimination reactions, substitution reactions, pericyclic reactions, rearrangement reactions, photochemical reactions and redox reactions. In organic synthesis, organic reactions are used in the construction of new organic molecules. The production of many manmade chemicals such as the production of pharmaceuticals drugs, plastics, food additives, fabrics depend on organic reactions. Factors governing organic reactions are essentially the same as that of any chemical reaction. Factors specific to organic reactions are those that determine the stability of reactants and products such as conjugation, hyperconjugation and aromaticity and the presence and stability of reactive intermediates such as free radicals, carbocations and carbanions. An organic compound may consist of many isomers. Selectivity in terms of regioselectivity, diastereoselectivity and enantioselectivity is therefore an important criterion for many organic reactions. There is no limit to the number of possible organic reactions and mechanisms. However, certain general patterns are observed that can be used to describe many common or useful reactions. Each reaction has a stepwise reaction mechanism that explains how it happens, although this detailed description of steps is not always clear from a list of reactants alone. Organic reactions can be organized into several basic types. Some reactions fit into more than one category. For example, some substitution reactions follow an additionelimination pathway. This overview isn't intended to include every single organic reaction. Rather, it is intended to cover the basic reactions.

© 2020 by SPC (Sami Publishing Company)



1. INTRODUCTION

For many college students, the phrase "organic chemistry" evokes angst and worry, as the subject can seem daunting to learn, let alone to master. For any chemistry major or pre-med student, this investigation is a requirement, either for a degree or for admittance into medical school or other graduate study; however, it need not be the torturous experience it is rumored to be. It can be survived, if not enjoyed, with the right approach [1].

Typically, organic chemistry is taught over the course to introduce key concepts and a few reactions and the elaborates on these concepts and introduces more many reactions that often seems unmanageable. Indeed, the number of reactions discussed would be overwhelming to any student who tried simply to memorize them; however, to the student who understands the underlying mechanisms and principles, the number of reactions covered by the class will seem a lot more manageable. A great deal of organic chemistry can be explained by understanding these concepts and how they are related, and thereby a lot of reactions introduced will make more sense [2].

Consider, as an example, the addition of a Grignard reagent to a ketone. Here, a nucleophile (the Grignard reagent) attacks an electrophile (a positively charged nucleus, i.e. the carbon of the ketone group) to form a carbon-carbon bond. Essentially, this reaction involves an electrostatic attraction between a partially positively-charged carbon (the carbonyl) and a partially negativelycharged carbon (the Grignard). Now, consider the reaction of a Grignard reagent with a nitrile, aldehyde, and ester. These three reactions can be explained by the exact same concepts as the Grignard / ketone reaction! Thus, instead of trying to memorize four reactions, a student, with the proper understanding, can apply one mechanism to predict (without much memorization) the outcome of all of these reactions. Even more good news: other classes of organic chemistry reactions lend themselves to the same process [3].

Consider another example: the dreaded Sn1, Sn2, E1 and E2 debate. These are typically the first reactions discussed in any organic chemistry; however, the principles behind these reactions

(nucleophile, good leaving group, steric hindrance) can be applied to the whole investigation, explaining many reactions later on: for instance, the Sn1 and E1 reactions proceed through a carbocation intermediate. The stability of carbocation intermediates drives many of the organic reactions that are covered in both semesters. Understanding this principle can be as helpful in learning about E1 reactions as it can be in learning about allylic cation reactions [4].

So, to conclude with some practical advice: avoid memorization! Use your time instead to learn and understand the underlying concepts and reaction mechanisms demonstrated by each type of reaction. You can start doing this by taking Varsity Tutors' free organic chemistry Practice Tests which will give you a cross-section of topics from organic chemistry. Once you realize which topics you need to work on learning, you can focus on understanding the underlying concepts that explain them, and not just memorizing chemical reactions. This will give you a deeper understanding of the material and help you survive, if not thrive, in organic chemistry [5].

The study of practical organic chemistry aims to acquire the necessary expertise to qualify for the identification of organic compounds. This requires many operations from extraction of organic compounds and purification of organic compounds Purification and then conduct many tests that lead to the identification of the organic compound and knowledge of its composition The chemical and therefore the chemical symbols of various organic compounds. The most important tests to be followed when distinguishing different organic compounds can be summarized as follows [6].

2. PHYSICAL CONSTANTS FOR ORGANIC COMPOUNDS

Physical properties of organic compounds typically of interest include both quantitative and qualitative features. Quantitative information includes melting point, boiling point, and index of refraction. Qualitative properties include odor, consistency, solubility, and color [6].

2.1. Melting and boiling properties:

Organic compounds typically melt and many boil. In contrast, while inorganic materials generally can be melted, many do not boil, tending instead to degrade. In earlier times, the melting point (m.p.) and boiling point (b.p.) provided crucial information on the purity and identity of organic compounds. The melting and boiling points correlate with the polarity of the molecules and their molecular weight. Some organic compounds, especially symmetrical ones, sublime, that is they evaporate without melting. A well-known example of a sublimable organic compound dichlorobenzene, the odiferous constituent of modern mothballs. Organic compounds are usually not very stable at temperatures above 300°C, although some exceptions exist [6].

2.2. Solid state properties:

Various specialized properties of molecular crystals and organic polymers with conjugated systems are of interest depending on applications, e.g. thermo-mechanical and electro-mechanical such as piezoelectricity, electrical conductivity (see conductive polymers and organic semiconductors), and electro-optical (e.g. non-linear optics) properties. For historical reasons, such properties are mainly the subjects of the areas of polymer science and materials science [6].

2.3. Detection of the elements:

Qualitative analysis of the various elements involved in the composition of the organic compound is carried out. Carbon, hydrogen, oxygen, nitrogen, sulfur, phosphorus and halogens are usually detected. The quantitative analysis of the percentages of the various elements involved in the composition of the organic compound is estimated so that the first symbol of the organic compound is obtained Empirical formula and thus obtain the molecular formula after estimating the molecular weight of the organic compound molecular weight [7].

2.4. Preparation of organic derivatives:

Finally, the organic compound is confirmed by the preparation of a derivative of the compound that can be obtained in large quantities in a pure form and conducts the aforementioned tests. If its properties and test results match with what is recorded in the special references, this is a proof and confirmation of the truth of the organic compound and how it is widely used [7].

Extraction of organic compounds is one of the most important steps to obtain the organic compound alone. It is then carried out the rest of the procedures of purification and descriptive analysis and quantitative analysis and various tests. Organic compounds are extracted by appropriate solvents. The amount of material extracted is subject to a law called the distribution law or partition law. This law provides that if there is a system consisting of two layers of liquid unmixed or poorly mixed, and then add to these layers some amount of a third mixable material in both layers, it distributes itself so that the proportion of its concentration in one layer to the other constant at a certain temperature [8].

2.5. Purification of organic liquids:

Purification of organic compounds is one of the most important steps to be taken before analyzing these compounds. Impurities often result in different chemical analysis results. The organic material is rarely obtained in a naturally pure state, so purification of the organic material is carried out because of the impurities associated with it [9].

The liquid organic matter is usually purified by distillation. The scientific basis for this process is the difference in the boiling point between the organic liquid to be purified and the impurities mixed with it. The distillation method also depends on the nature of the liquid and its tolerance to temperatures [9].

2.6. Purification of organic solids:

Solid organic materials are purified according to their nature in one of the following ways. Crystallization, the scientific basis in this process is that the degree of solubility of the solid organic material to be purified in a solvent or a mixture of solvents varies at the boiling point of this solvent. To identify the organic compound by descriptive analysis, two types of tests are performed: initial tests of color, odor and combustion. A descriptive analysis is carried out which includes the detection

of the elements involved in the composition of the organic compound. The results of the descriptive analysis refer to the different elements involved in the synthesis of organic matter. This is not sufficient to infer the chemical symbols of the organic compounds. Therefore, it is necessary to estimate the quantities or percentages of each component of the organic matter. Organic matter to reach the molecular formula of the organic compound under study [9].

Sublimation, is the transition of a substance directly from the solid to the gas phase. Sublimation is an endothermic process that occurs at temperatures and pressures below a substance's triple point in its phase diagram, which corresponds to the lowest pressure at which the substance can exist as a liquid. The reverse process of sublimation is deposition or desublimation, in which a substance passes directly from a gas to a solid phase. Sublimation has also been used as a generic term to describe a solid-to-gas transition (sublimation) followed by a gas-to-solid transition (deposition). While a transition from liquid to gas is described as evaporation if it occurs below the boiling point of the liquid, and as boiling if it occurs at the boiling point, there is no such distinction within the solidto-gas transition, which is always described as sublimation [9].

Sublimation is a technique used by chemists to purify compounds. A solid is typically placed in a sublimation apparatus and heated under vacuum. Under this reduced pressure, the solid volatilizes and condenses as a purified compound on a cooled surface (cold finger), leaving a non-volatile residue of impurities behind. Once heating ceases and the vacuum is removed, the purified compound may be collected from the cooling surface. For even higher purification efficiencies, a temperature gradient is applied, which also allows for the separation of different fractions. Typical setups use an evacuated glass tube that is heated gradually in a controlled manner. The material flow is from the hot end, where the initial material is placed, to the cold end that is connected to a pump stand. By controlling temperatures along the length of the tube, the operator can control the zones of re-condensation, with very volatile compounds being pumped out of the system completely (or caught by a separate cold

trap), moderately volatile compounds recondensing along the tube according to their different volatilities, and non-volatile compounds remaining in the hot end. Vacuum sublimation of this type is also the method of choice for purification of organic compounds for use in the organic electronics industry, where very high purities (often > 99.99%) are needed to satisfy the standards for consumer electronics and other applications [9].

2.7. Organic compounds solubility:

organic compounds Neutral tend hydrophobic; that is, they are less soluble in water than in organic solvents. Exceptions include organic compounds that contain ionizable (which can be converted in ions) groups as well as low molecular weight alcohols, amines, and carboxylic acids where hydrogen bonding occurs. Organic compounds tend to dissolve in organic solvents. Solvents can be either pure substances like ether or ethyl alcohol, or mixtures, such as the paraffinic solvents such as the various petroleum ethers and white spirits, or the range of pure or mixed aromatic solvents obtained from petroleum or tar fractions by physical separation or by chemical conversion. Solubility in the different solvents depends upon the solvent type and on the functional groups if present in the solution [10].

Solubility can sometimes provide a surprisingly useful amount of information. First, you will test your unknown's solubility in water. Compounds with 4 carbons or less will easily dissolve in water, whereas compounds with 8 carbons or more will be insoluble. Compounds containing 5 to 7 carbons may or may not dissolve (often they will display "partial" solubility). If your compound dissolves in water, you will also want to check the pH of the solution. Amines will typically be basic, and carboxylic acids will typically be acidic. Most other compounds will be neutral. Compounds that are insoluble in water should then be subjected to a solubility test in 5% HCl. Typically, only amines will be soluble in HCl because they form water soluble hydrochloride salts when they react with HCl. Compounds that are not soluble in HCl, should be subjected to testing in basic solutions (5% NaOH and 5% NaHCO₃).

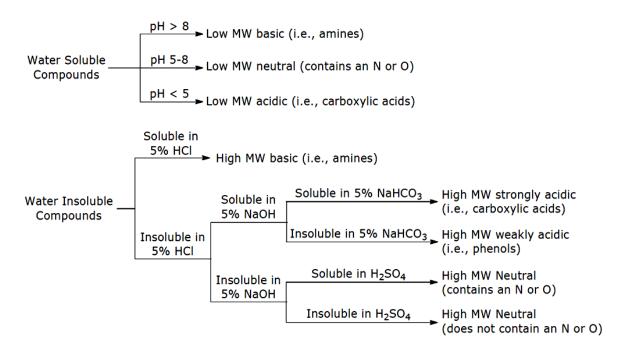


Fig. 1: Flow chart of solubility tests

Both strong and weak acids (Carboxylic acids and phenols) will be deprotonated by NaOH to form water soluble alkoxides. Only strong acids like carboxylic acids will react with NaHCO₃. Compounds that are not soluble in base should then be reacted with a very strong acid, sulfuric acid (note that in the case of sulfuric acid, "solubility" is also indicated by any type of reaction such as heat, gas generation, or a color change). Compounds that cannot become protonated by sulfuric acid at all (i.e., alkanes, alkyl halides, and aromatic carbons) will still remain insoluble. These solubility tests are summarized in the flow chart below **Fig.1** [10].

There are two main types of solubility: natural solubility and chemical solubility, both of which can be illustrated as follows: Natural solubility, In the natural solubility there is a disappearance of the molecules of the organic compound in the solvent without any change in the molecule. In other words, there is no chemical reaction between the solvent and solute molecules, and each retains its natural and chemical properties, and can be separated from each other. Chemical Solubility, In the chemical solubility there is an interaction between the solute

molecules, i.e., the organic compound with the solvent molecules. Both solute and solvent cannot be separated or recovered thereafter [11].

3. DETECTION OF FUNCTIONAL GROUPS

The concept of functional groups is central in organic chemistry, both as a means to classify structures and for predicting properties. A functional group is a molecular module, and the reactivity of that functional group is assumed, within limits, to be the same in a variety of molecules. Functional groups can have decisive influence on the chemical and physical properties of organic compounds. Molecules are classified on the basis of their functional groups. Alcohols, for example, all have the subunit C-O-H. All alcohols tend to be somewhat hydrophilic, usually form esters, and usually can be converted to the corresponding halides. Most functional groups feature heteroatoms (atoms other than C and H). Organic compounds are classified according to functional groups, alcohols, carboxylic acids, amines, etc. [11].

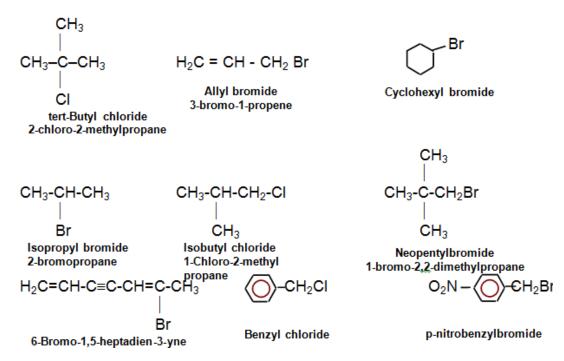


Fig. 2: Chemical structures of some alkyl halides

3.1. Alkyl halides:

Alkyl halides are organic compounds containing carbon–halogen bond. The polarizability of carbon–halogen bond greatly influences the chemical reactivity of alkyl halides. Alkyl halides undergo nucleophilic substitution reaction. Haloalkanes or commonly known as alkyl halides are organic compounds containing halogen atom directly bonded to carbon atom. They are derived from alkanes or alkenes. Halides are important class of organic compounds being precursor in many organic syntheses. They find many industrial applications such as flame retardants, organic solvents and propellants. Chemical structures of some alkyl halides are represented as shown in Fig. 2.

3.1.1. Belistein Wire Test for Halogen Detection

This test is summarized that when the organic matter containing a halogen is heated in contact with a copper wire in a benzene flame, copper halide is formed which gives the flame a bluish green or green color as a result of volatilization. Reaction procedure: The copper wire is heated by

direct flame for cleaning until the flame becomes colorless, dipping the tip of the hot wire into the organic material and then displaying the flame again. If a bluish green color appears, there is a possibility of halogens in the organic matter [11].

3.1.2. Ethanolic silver nitrate test:

Alkyl halide is added to 1 ml of about 1 ml Alcohol silver nitrate solution $AgNO_3$ and the precipitate is either white or yellowish white or yellow depending on the type of halogen in the organic matter under study.

3.2. Aromatic compounds:

Aromatic compounds are substances that consist of one or more rings that contain alternating single and double bonds in its chemical structure. In real life, many aromatic compounds have an odor, however, there are some compounds that are chemically aromatic, but do not have a distinct smell. For example, benzene is an aromatic compound. The chemical structure of benzene, as illustrated here, contains a hexagon ring with alternating double bonds. Chemical structures of some aromatic compounds are shown in Fig.3.

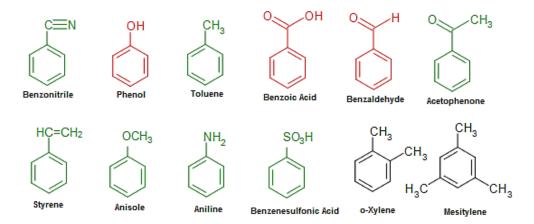


Fig. 3: Chemical structures of some aromatic compounds

3.2.1. Bromine water test:

Reaction procedure: Add to the drops of diluted phenol about 1 ml of bromine water solution is noticed the disappearance of bromine color and consists of a white precipitate of the compound of the bromine phenol Tribromo phenol (replacement reaction) [11].

3.2.2. Ferric chloride FeCl₃ test:

Reaction procedure: 1 ml of diluted phenol solution is added to about 1 ml of ferric chloride FeCl₃ (yellow color) shows a violet color disappears by adding acetic acid or mineral acid such as HCl as a positive result of this test [11].

3.2.3. Aluminum chloride in chloroform AlCl₃/CHCl₃ test:

Aromatic compounds are given special and distinctive colors when interacting with a mixture of aluminum chloride and chloroform. Reaction procedure: Add 2 ml chloroform in a test tube to 0.5 g solid aluminum chloride. In another tube, apply 2 ml of organic matter. Then add the mixture of the first tube to the second with a note of the reaction procedure on the inner wall of the tube if the color of the solution to orange or red indicates the presence of the aromatic compound [11].

3.2.4. Interaction with halogens:

Bromine reacts by substituting with benzene and the product is Bromo benzene with the rise of HBr gas. Reaction procedure: Add to 1 ml benzene a few drops of bromine. Note that the reaction starts first and then the liquid is heated and the reaction increases with the rise of hydrogen bromide gas HBr, which is a withdrawal of ammonium bromide NH₄Br when exposing a leg moistened with ammonium hydroxide solution NH₄OH to the test tube nozzle [11].

3.2.5. Interaction with concentrated nitric acid:

Aromatic compounds are relatively easy to react with concentrated nitric acid. The reaction is that the nitro group (NO_2) replaces a hydrogen atom in the aromatic compound. This process is called nitrification. Where gasoline is given when it interacts with concentrated nitric acid in the presence of concentrated sulfuric acid compound nitrobenzene. Reaction procedure: Benzene is gradually added to a mixture of concentrated nitric acid and concentrated sulfuric acid in a ratio of (1:1) with a good shaking of the mixture and heating in a conical flask with the use of an inverter condenser. The liquid is then poured into a glass of cold water and nitrobenzene is separated in the form of yellow, heavy oil [11].

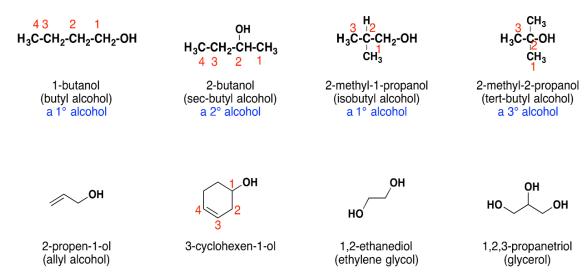


Fig. 4: Chemical structures of some alcohols

3.3. Alcohols:

Alcohol is an organic compound that carries at least one hydroxyl functional group (-OH) bound to a saturated carbon atom. The term alcohol originally referred the primary alcohol ethanol (ethyl alcohol), which is used as a drug and is the main alcohol present in alcoholic beverages. An important class of alcohols, of which methanol and ethanol are the simplest members, includes all compounds for which the general formula is C_nH_{2n+1}OH. Simple monoalcohols that are the subject of this article include primary (RCH₂OH), secondary (R₂CHOH) and tertiary (R₃COH) alcohols. Chemical structures of some alcohols are shown in Fig.4.

3.3.1. Solubility test:

Most alcohols dissolve in ether, and alcohols with the least 5 carbon atoms dissolve in water. All alcohols are weak bases, so they dissolve in concentrated sulfuric acid H_2SO_4 , forming dissolved salts in water [11].

3.3.2. Oxidation test with chromic acid:

The first alcohol is oxidized by chromic acid to the corresponding aldehyde and then to the corresponding acid, while the second alcohol is oxidized to the corresponding ketone and does not oxidize any more, while the third alcohol does not have any oxidation, The oxidation and reduction for alcohols are summarized in the flow chart below

Fig. 5. Reaction procedure: Add to 1 ml of alcohol a few drops of chromic acid and notice the extent of change in color, where when the color of reddishorange chromic acid to green indicates the oxidation of the first or second alcohol **Fig. 6** [12].

3.3.3. Zinc chloride test in HCl (Lucas test)

Scientific basis: It is a distinctive test for all three alcohols based on reaction speed. Where the fastest possible in the case of the third alcohol, then followed by the second alcohol, which takes 5-10 minutes, then be the slowest possible in the first alcohol, where it takes several hours to form an alkyl halide. Reaction procedure: Add to 1 ml alcohol about 1 ml Lucas solution with good shaking and wait. The alkyl halide is insoluble in water and appears as a separating surface [12].

3.3.4. Potassium permanganate test (Bayer test)

Scientific basis: It is a distinctive test between the first and second alcohols based on reaction speed. The second alcohol, which is easier to oxidize by neutral permanganate, is as fast as possible in 1-2 minutes, the first alcohol slowly oxidizes and takes about 3 minutes. The first alcohol with the long chain takes longer. Reaction procedure: Add 2% potassium permanganate solution to about 2 ml of alcohol until the violet color of the permanganate appears. If the violet color of the permanganate does not disappear immediately wait 5 minutes shaking [12].

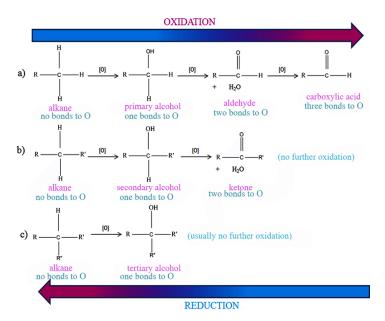


Fig. 5: Flow chart of oxidation and reduction for alcohols

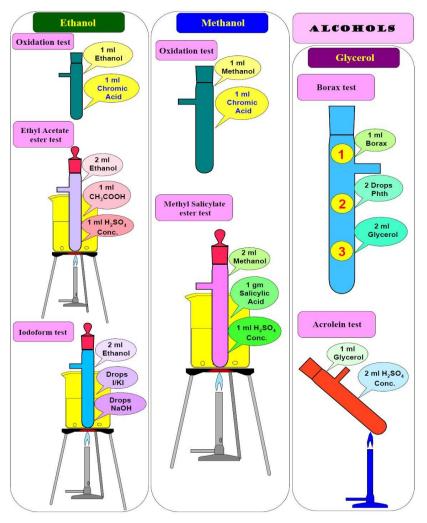


Fig. 6: Some alcohols detection tests

3.3.5. Periodic (HIO₄) test:

Scientific basis: Periodic acid reacts with glycols, alpha hydroxy acids, alpha hydroxy aldehydes and alpha hydroxy ketones. Two adjacent hydroxide groups, one adjacent hydroxide group and one aldehyde group, or one adjacent hydroxide group and a ketone group are required for the success of this test. The pyridic acid breaks the bond and produces aldehydes and acids, while the acid is reduced to the HIO₃ iodate, which is tested by aqueous silver nitrate solution AgNO3 and a precipitate is a direct test of the test. Reaction procedure: Take 3 ml of pyridic acid in a test tube and add one drop of concentrated nitric acid, then add drops of organic matter with shaking for 15 minutes, then add the solution of silver nitrate water and form a precipitate directly as a positive result of this test [12].

3.3.6. Methyl salicylate ester test

Scientific basis: Methyl alcohol reacts with salicylic acid in the presence of concentrated sulfuric acid (which removes or absorbs the water from the reaction and thus prevents the reverse reaction) and produces a methyl salicylate ester that smells like camphor. Reaction procedure: Add to 2 ml of methyl alcohol in a test tube about 1 ml of concentrated sulfuric acid.

Then add about 1 gram of salicylic acid with good shaking. Heat in a water bath for 2 minutes and note that the nozzle of the tube is blocked with a cork plug. Then open the nozzle of the tube after the expiration of the heating period and notice the appearance of the smell of ester methyl salicylate, which resembles the smell of camphor **Fig. 6** [13].

3.3.7. Ethyl acetate ester test:

Scientific basis: Ethyl alcohol reacts with acetic acid in the presence of concentrated sulfuric acid (which removes or absorbs the resulting water from the reaction from the reaction medium and thus prevents the reverse reaction) and produces an ethyl acetate ester, which smells like a dirty

apple. Reaction procedure: Add to 2 ml of ethyl alcohol in a test tube about 1 ml of dilute acetic acid, then add about 1 ml of concentrated sulfuric acid with good shaking. Heat in a water bath for 2 minutes and note that the nozzle of the tube is blocked with a cork plug.

Then open the nozzle of the tube after the expiration of the heating period is noticed the appearance of the smell of ethyl acetate ester, which resembles the smell of sour apple **Fig. 6** [14].

3.3.8. Iodoform test:

Scientific basis: This test is successful with compounds containing a methyl group associated with the carbonyl group CH₃-CO-, or compounds that are oxidized under experimental conditions and produce this composition. It therefore works with ethyl alcohol, which is oxidized to acetaldehyde, as well as with acetaldehyde, acetone and acetophenone. It does not work with formaldehyde or benzaldehyde. Ethyl alcohol reacts with halogens in the presence of an alkaline medium of NaOH or with NaOI or NaOI or NaOCl and NaOBr to produce haloforms depending on the type of halogen, where they are called iodoform chloroform bromoform, respectively. Iodoform (CHI₃) is one of the most common haloform compounds. Ethyl alcohol reacts with iodine in the presence of sodium hydroxide and the iodoform is formed as yellow crystals.

Reaction procedure: Add 2 ml of ethanol drops of potassium iodide iodide solution (I / KI) and then add drops of NaOH point by point with shaking until a pale yellow color is formed. Heat in a water bath for 2 minutes while maintaining the pale yellow color by adding drops of iodine whenever the color is removed. Yellow crystals of iodoform are evident by cooling the tube with the appearance of the odor of iodoform, which is similar to the antiseptic Fig. 6 [14].

3.3.9. Acrolein:

Scientific basis: This test is used to detect glycerol, where two molecules of water are removed from each glycerol molecule when treated with some dehydrated or voracious substances for water absorption such as concentrated sulfuric acid or potassium sulfate with heating to produce the acrolein compound, an unsaturated aldehyde characterized by the rise of its strong and permeable vapors. Reaction procedure: Add to 1 ml of glycerol about 1 ml of concentrated sulfuric acid (or about half a gram of solid potassium sulfate) with heating and note the emergence of the smell of acrolein permeable Fig. 6 [14].

3.3.10. Borax test:

Scientific basis: The borax solution Na₂B₄O₇ has an alkaline effect as it results from the interaction of a strong base with a weak acid. Therefore, borax solution contains ions $(B_4O_7^{-2})$ as well as molecules of boric acid (H₃BO₃), which reacts with alcohol and is an ester. Three groups of hydroxide from each two molecules of polyhydroxide alcohol (such as trihydroxide glycerol) are involved in this reaction, resulting in constant cyclic esters. The free hydrogen ions are unique in the solution, and the medium becomes acidic in the scarlet red color with the index of phenol. When the solution is heated, the ester ring formed breaks down and the hydrogen ions return to their first place in the equivalence supplement. The medium then returns to the alkaline state again and the scarlet red color appears with *phth* indicator.

Reaction procedure: Note: In this test, the order of addition of solutions should be strictly adhered to as follows: Place 1 ml borax solution into the test tube. Two drops of *phth* indicator are added with shaking, showing a crimson red color. Add 1 ml of glycerol with shaking and note the disappearance of scarlet red color. The solution is lightly heated and scarlet red color is observed, but with less intensity. When the solution is cooled, the color disappears again Fig. 6 [14].

3.4. Aldehydes and Ketones:

Aldehydes and ketones contain the carbonyl group. Aldehydes are considered the most important functional group. They are often called

the formyl or methanoyl group. Aldehydes derive their name from the dehydration of alcohols. Aldehydes contain the carbonyl group bonded to at least one hydrogen atom. Ketones contain the carbonyl group bonded to two carbon atoms. Aldehydes and ketones are organic compounds which incorporate a carbonyl functional group, C=O. The carbon atom of this group has two remaining bonds that may be occupied by hydrogen, alkyl or aryl substituents. If at least one of these substituents is hydrogen, the compound is an aldehyde. If neither is hydrogen, the compound is a ketone.

3.4.1. Schiff's test:

Scientific basis: The Schiff's test works with aldehydes and does not work with ketones, where aldehydes are oxidized to the corresponding acids while ketones are not oxidized. Preparation of Schiff's Solution: Schiff's solution consists of a dyed foxin pigment reduced by SO₂. This solution is prepared by dissolving 0.2 grams of acid dye (Para -Rosaniline hydrochloride) in 20 ml of a saturated solution of SO₂ and leave for a few hours and then complete the solution to 200 ml with distilled water. The former SO₂ solution can be replaced with 2 g of N₂S and 2 ml of concentrated HCl. It is noted that heating or the addition of alkalis or alkaline salts react with SO₂ and is formed a violet red color. The addition of aldehyde to the solution reacts with SO₂ and is given a purple red color, so the appearance of this color is evidence of the success of the test. Reaction procedure: Add to the aldehyde drops of about 2 ml of a solution of the shave with a good shaking shows red violet in no more than 3-4 minutes Fig. 10 [15].

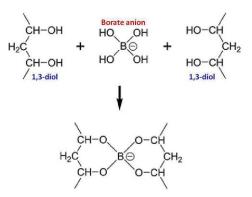


Fig 7: The reaction involved Borax test

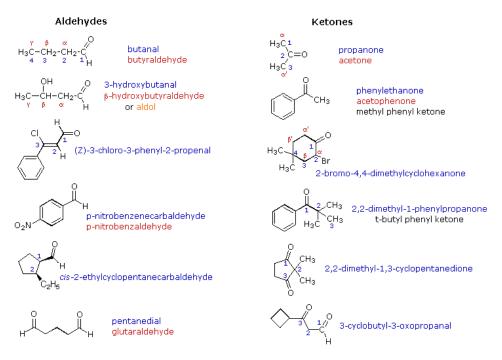


Fig. 8: Chemical structures of some aldehydes and ketones

3.4.2. Fehling's test:

Scientific basis: Fehling test passes with aldehydes and does not work with ketones because ketones do not oxidize. Preparation of the Fehling reagent: The Fehling reagent consists of a mixture of two solutions Fehling A, Fehling B as follows: Fehling A solution consists of 6.9% copper sulfate solution. Fehling B solution is composed of 12% NaOH and Rochelle salt (sodium and potassium tartrate) 34.6% in the same solution; in other words, add 12 grams of NaOH to 34.6 grams of Rochelle salt and supplement the solution with distilled water to 100 ml. Reaction procedure: Add to 2 ml of aldehyde about 4 ml Fehling reagent (2 ml Fehling A + 2 ml Fehling B) and then heating in a water bath for 3 minutes and a precipitate of copper oxide color ranging from orange to red depending on the concentration of aldehyde as a positive result of this test [15].

3.4.3. Benedict's test:

Scientific basis: Benedict's test works with aldehydes and does not work with ketones.

Benedict's solution is a modification of Fehling's solution, as it does not need to be confused with two solutions. Therefore, Benedict's test is preferable to Fehling's test for the following reasons: Benedict's reagent consists of one solution, while Fehling consists of two solutions. Therefore, any error in the percentage of their addition causes a test error.

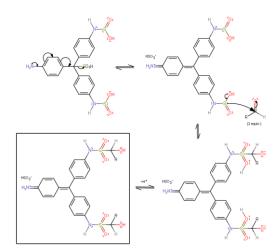


Fig. 9: The reaction involved Schiff's test

Benedict's reagent is a constant solution where it is not required to be newly prepared, unlike Fehling. Benedict's reagent is sensitive to very low concentrations of aldehydes unlike Fehling. Benedict's test works with aliphatic aldehydes but is not sensitive to aromatic aldehydes and is therefore used to distinguish them. Preparation of Benedict's solution: Benedict's solution consists of 173 grams of sodium citrate and 17.3 grams of copper sulphate CuSO₄ and 100 grams of sodium carbonate Na₂CO₃ anhydrous, and this mixture is dissolved in distilled water and then supplemented with distilled water to 1 liter, the solution must be completely clear. Reaction procedure: Add to 1 ml of aldehyde about 2 ml of Benedict's solution and then heating in a water bath for 3 minutes to note the formation of a precipitate of copper oxide color ranging from orange to red depending on the

concentration of aldehyde as a positive result of this test **Fig.10** [16].

3.4.4. Tollen's test:

Scientific basis: This test works with aldehydes and does not work with ketones. It shall be as follows: Reaction procedure: Preparation of Tollen solution: Tollen solution is a solution of ammonia silver hydroxide which is prepared by adding drops of NaOH solution to about 1 ml of silver nitrate solution AgNO3 in a test tube. All the precipitates formed only with no increase in ammonium hydroxide solution, we obtain a solution of Ag(NH₃)₂OH which is ionized to Ag(NH₃)⁺², OH⁻. Add to 1 mL aldehyde about 3 mL of a tolene solution and then heat in a water bath. A black precipitate or shiny silver mirror is observed on the inner wall of the individual silver tube as a positive result of this test Fig.10 [17].

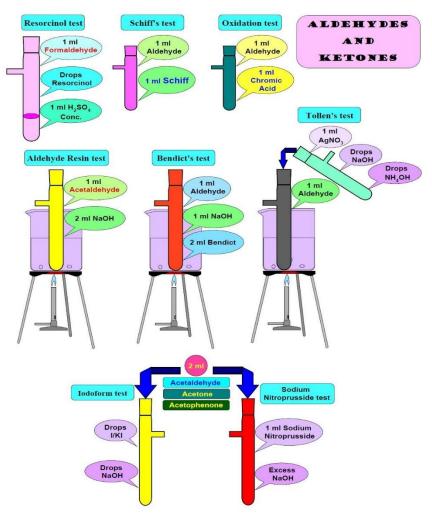


Fig 10. Some aldehydes and ketones detection tests

Fig. 11: The reaction involved Tollen's test

3.4.5. Resorcinol test:

Scientific basis: This test passes with formaldehyde only, as it is only for formaldehyde and other aldehydes and ketones. Reaction procedure: Add drops of resorcinol solution (0.5%) to about 1 ml of formaldehyde with good shaking and then add about 2 ml concentrated sulfuric acid $\rm H_2SO_4$ on the inner wall of the tube with great caution note that the tube is violet red at the surface of separation as a result Positive for this test Fig.10 [18].

3.4.6. Aldehyde resin test:

Scientific basis: This test only works with acetaldehyde. It is unique to acetaldehyde only from aldehydes and ketones. Reaction procedure: Add to 1 ml acetaldehyde about 2 ml NaOH 5% and then heat until boiling for two minutes, it is noted that the yellow color becomes viscous textures after cooling Fig.10 [19].

3.4.7. Cannizaro's test:

Scientific basis: This test only works with benzaldehyde. It is unique to benzaldehyde only from aldehydes and ketones. Benzaldehyde is not given a resin product when reacting with NaOH solution, as in the case of acetaldehyde. However, benzaldehyde reacts with NaOH with boiling and produces salt of sodium benzoate and benzyl alcohol. The solution is acidified after being cooled

with concentrated HCl acid, which reacts with sodium benzoate salt to form bezoic acid, which is separated into a white crystalline precipitate when the solution is cooled in ice. Reaction procedure: Add to 1 ml of benzaldehyde about 2 ml of 5% NaOH solution and heat until boiling for 2 minutes. The solution is then cooled and acidified with concentrated HCl acid. A white crystalline precipitate from benzoic acid is observed when the solution is cooled in ice [20].

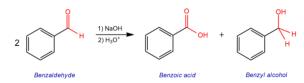


Fig. 12: The reaction involved Cannizaro's test

3.4.8. Sodium nitroprusside test:

Scientific basis: This test works with acetaldehyde, acetone and acetophenone. It does not work with formaldehyde or benzaldehyde. Add about 1 ml of sodium nitroprusside solution (newly prepared) to drops of acetaldehyde, acetone or acetophenone, then add an excess of 5% NaOH about 2-3 ml with good shaking, the discoloration of the solution in red is observed as a positive result of this test Fig.10 [21].

Fig. 13: The reaction involved sodium nitroprusside test

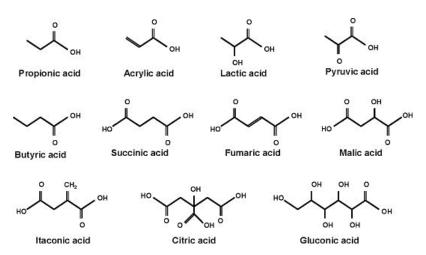


Fig. 14: Chemical structures of some organic acids

3.5. Organic acids and their salts and anhydrides:

Organic acids contain an active peripheral group known as the carboxyl group (-COOH). The organic acids are divided into two main sections, aliphatic and aromatic acids, each of which is also divided into different sections depending on the number of carboxyl groups present in the molecule to: monobasal, dibasic and basal acids. For the type and number of other active groups represented by the acid molecule (e.g. hydroxyl acids and amino acids). Fig.14 showed the chemical structures of some organic acids.

3.5.1. Preparation of neutral acid solution:

A neutral solution of organic acid can be prepared as follows: The ammonium hydroxide solution is added to the organic acid solution in water or directly to the acid until the solution is completely alkali (ammonia is proven with stirring or shaking). The resulting solution is heated in a beaker to a boiling point to expel excess ammonia and the remaining solution contains neutral ammonium salt of organic acid. Note: In the case of tests on organic acid salt solution does not have the previous draw, as the solution of organic acid salt is basically a

neutral solution and all tests are carried out directly [22].

The following are the most important tests to detect organic acids and their salts: Melting Test, all organic acids are colorless crystalline solids with the exception of formic acid and acetic liquids. They are the only volatile acids with a distinct pungent odor. Organic aliphatic acids such as formic, acetic, oxalic, citric and tartaric are soluble in cold water. While aromatic organic acids such as benzoic and salicylic do not dissolve in cold water and dissolve a little in hot water and dissolve in the ether because of the presence of benzene ring in its composition. All organic acids are dissolved in NaOH 5% chemical soluble forming sodium salts of organic acid. All organic acids interact with sodium bicarbonate NaHCO₃ or sodium carbonate Na₂CO₃, forming sodium salt of organic acid and carbon dioxide gas rising CO₂ and gas flux accompanying the occurrence of evidence of the presence of free carboxyl group. CO₂ gas mounting. Esters are sweet scents that resemble fruits, do not dissolve in water but dissolve in ether, as well as dissolve in concentrated sulfuric acid and do not dissolve in other scouts [23].

3.5.3. Mercuric chloride HgCl₂ test:

This test works with neutral formic acid solution or sodium formate salt only. A little HgCl2 is added to an ample amount of neutral formic solution of formic acid or formate salt directly and then heated in a water bath A white precipitate of mercury chloride is formed [24].

$$\begin{array}{cccccc} HCOOH & + & 2HgCl_2 & \longrightarrow & Hg_2Cl_2 \downarrow & + & 2HCl + CO_2 \uparrow \\ Formic acid & Mercuric & Mercurous chloride & & & & & \\ & & & & & & & & & \\ & & & & & & & & \\ CH_3COOH & & & & & & & \\ & & & & & & & & \\ & & & & & & & \\ & & & & & & & \\ & & & & & & & \\ & & & & & & & \\ & & & & & & & \\ & & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & \\ & & & & \\ & & &$$

3.5.4. Calcium chloride CaCl₂ test:

This test works only with neutral solutions. It is used to distinguish between oxalic, tartaric and citric acids and their salts. Calcium chloride solution is added directly to the neutral solution of acid or salt solution. In the case of oxalic acid and salt, a white precipitate of calcium oxalate is formed that does not dissolve in acetic acid and dissolve in

$$R\text{-COOH} + \text{NaOH} \rightarrow \text{R-COONa} + \text{H}_2\text{O}$$

$$R\text{-COOH} + \text{NaHCO}_3 \rightarrow \text{R-COONa} + \text{H}_2\text{O} + \text{CO}_2\uparrow$$

3.5.2. Ferric chloride FeCl₃ test:

This test is only for neutral solutions. Reaction procedure: Add about 1 ml of ferric chloride (FeCl₃) to 2 ml of neutral acid solution of organic acid or salt solution directly and note one of the following results: In the case of formic acid, sodium fermate salt, acetic acid and sodium acetate salt, a red color of ferric or acetate formate appears. In the case of benzoic acid and sodium benzoate salt, a cold skin precipitate of ferric benzoate is formed. In the case of salicylic acid and salicylate, a red-purple color appears. In the case of negative result, no change in the color of the solution of ferric yellow chloride indicated by oxalic acids, tartaric or citric or their salts Fig.15 [24].

mineral acids such as diluted HCl acid. In the case of tartaric acid and salt, a cold white precipitate of calcium tartrate is formed, but after very severe shaking. In the case of citric acid and salt does not form a precipitate only after heating (a white precipitate of calcium citrate) and requires a concentrated solution of neutral solution of citric acid or salt. In the case of formic, acetic, benzoic and salicylic acids, as well as their salts, the result is negative as no precipitate is formed either cold or after heating. Note: The neutral solution should be cooled before the test. It also needs concentrated solutions of acids or their salts Fig.15 [25].

3.5.5. Fenton's test:

This test works only with neutral tartaric acid and sodium tartrate salt directly. Two drops of saturated solution of newly prepared ferrous sulfate are added to 2 mL of diluted solution of neutralizing tartrate.

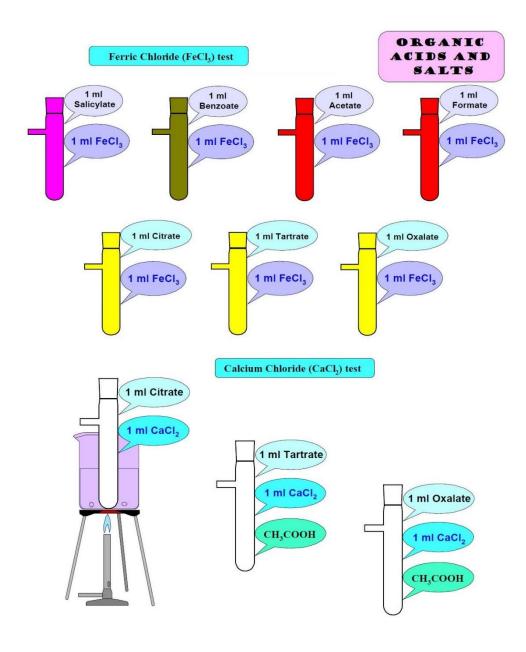


Fig. 15. Some organic acids and/or salts detection tests

Scientific basis: The success of the Fenton test with tartaric acid and its salt is solely due to the oxidation of tartaric acid to dihydric malic acid, which is given a green color with ferric salts (after the ferrous sulfate oxidation to ferric sulphate by H_2O_2) and changes to violet by alkali by adding NaOH. Add drops of H_2O_2 solution (5%) point by point until the solution becomes dark green. An excess of NaOH solution (5%) is added and the solution is observed to be purple. As a positive result of this test [26].

3.5.6. Cobalt nitrate Co(NO₃)₂ test:

This test only works with tartaric acid and salt. Add 1 ml of cobalt nitrate to 1 ml of tartaric solution or salt of sodium tartrate, then add an increase of NaOH solution (about 3 ml) and then heating [26].

3.5.7. Beta-Naphthol test:

This test only works with tartaric acid and salt. Add to 0.5 grams of tartaric solution or sodium tartrate salt about 0.5 grams of solid ß-naphthol butanafthul add about 1 ml concentrated sulfuric acid, and heat in a water bath for a minute with shaking the test tube, then cool the tube is observed discoloration of the solution in green [27].

3.5.8. Denige's test:

This test is unique to citric acid and sodium citrate salt only. Denige's solution is prepared by dissolving 50 grams of red mercury oxide in 1 liter of dilute sulfuric acid, thereby obtaining a solution of HgSO₄ (Denige's solution). Reaction procedure: Add to 2 ml of the solution of citric acid or sodium citrate salt about 1 ml of Denige's solution and then heat to a boiling point. Add a solution of potassium permanganate (2%), point by point, and note the demise of the distinctive violet color of the permanganate and the formation of a white precipitate of double mercury salt for both sulfuric acid and acetone bicarboxyl [27].

$$\begin{array}{ccc} 5HCOOH + 2KMnO_4 + 3H_2SO_4 & \longrightarrow & K_2SO_4 + 2MnSO_4 + 5CO_2 \uparrow + 8H_2O \\ \hline & CH_3COOH & \xrightarrow{KMnO_4} & Does not discharge pink colour of KMnO_4 solution \\ \hline & Acetic acid & \end{array}$$

3.5.9. Heating test with soda lime (NaOH/CaO):

This test works with aromatic organic acids and their salts only such as benzoic acid and salicylic acid. Sodium lime removes or removes the decarboxylation group from the acid. Mix about 0.2 grams of aromatic acid or salt with about 1 gram of sodium lime (NaOH/CaO) in a dry test tube and then heat is observed: In the case of benzoic acid and salt of sodium benzoate it is noted that gasoline vapors with a distinct smell, which ignite at the nozzle of the tube when it is exposed to flame, are observed. In the case of salicylic acid and sodium salicylate salts, the rise of phenol fumes with a characteristic odor is observed [28].

3.5.10. Methyl salicylate ester test

Add to 2 ml of methyl alcohol in a test tube about 1 ml of concentrated sulfuric acid, then add about 1

gram of salicylic acid with good shaking. Heat in a water bath for 2 minutes and note that the nozzle of the tube is blocked with a cork plug. Then open the nozzle of the tube after the expiration of the heating period and notice the appearance of the smell of ester methyl salicylate, which resembles the smell of camphor [28].

3.5.11. Ethyl acetate ester test:

Add to 2 ml of ethyl alcohol in a test tube about 1 ml of dilute acetic acid, then add about 1 ml of concentrated sulfuric acid with good shaking. Heat in a water bath for 2 minutes and note that the nozzle of the tube is blocked with a cork plug. Then open the nozzle of the tube after the expiration of the heating period is noticed the appearance of the smell of ethyl acetate ester, which resembles the smell of sour apple [28].

3.5.12. Ethyl bezoate ester test:

Benzoic acid interacts with ethyl alcohol in the presence of concentrated sulfuric acid (to prevent adverse reaction) and produces ethyl benzoate distinctive aromatic Reaction procedure: Add to 1 gram of benzoic acid about 2 ml of ethyl alcohol in a test tube, then add about 1 ml of concentrated sulfuric acid with good shaking. It is heated in a water bath for two minutes, noting that the tube nozzle is blocked with a cork plug. Then the nozzle of the tube opens after the heating period has ended, and a distinctive ethyl benzoate ester is observed. In the event that the smell is not clear, the resulting solution is poured into a glass containing a dilute solution of sodium carbonate, and a distinct aromatic odor of ethyl benzoate ester is observed [28].

Fig. 16. The reaction involved ethyl benzoate ester test

3.6. Identifying an Unknown Compound by Solubility and Functional Group Tests:

Organic qualitative analysis is an exercise in spectroscopy. Nuclear magnetic resonance spectroscopy and infrared spectroscopy are the major spectroscopic techniques used by organic chemists. However, much insight can be gained from using simple qualitative tests to determine the

identity of unknowns. Structures of unknown compounds can be determined by comparing physical properties, performing functional group tests, and checking melting points of derivatives against those of known compounds reported in the literature. Solubility properties and chemical reactivity become apparent during these qualitative tests. Fig.17 and 18 showed the detection of unknown liquid and solid organic compounds.

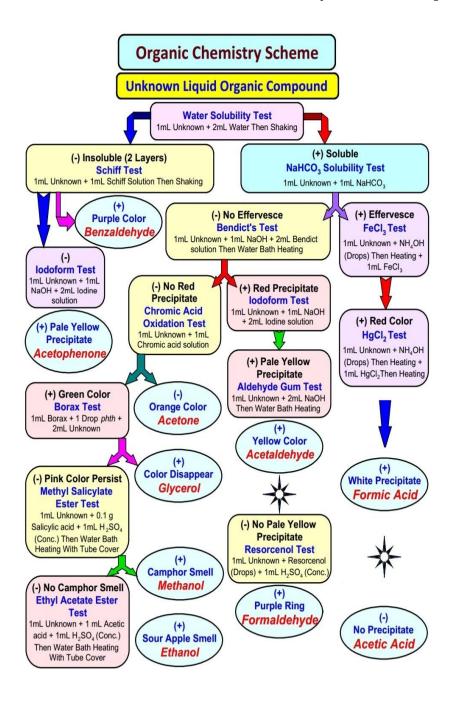


Fig. 17. Detection of unknown liquid organic compounds

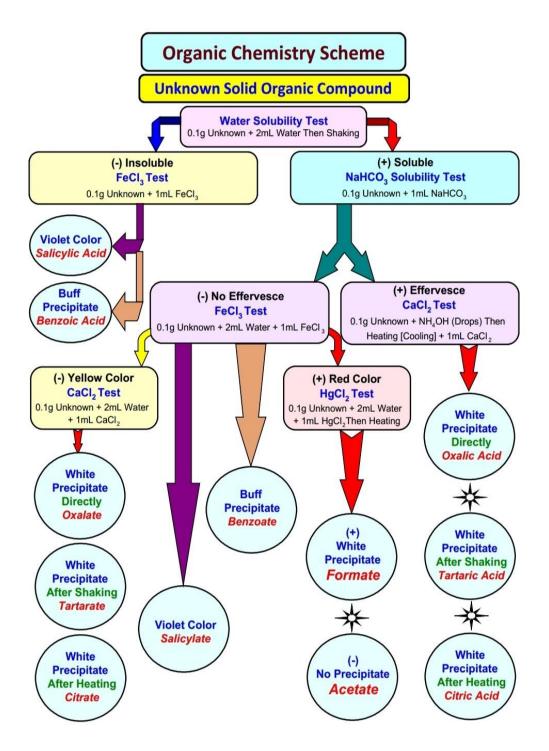


Fig. 18. Detection of unknown solid organic compounds

CONCLUSION

To conclude this investigation, it is important to emphasize a logical point about the determination of reaction mechanisms. A proposed mechanism can never really be proven; rather, it is a case of alternative mechanisms being eliminated. Having in mind a mechanism that explains all the facts does not constitute proof that the mechanism is correct. That conclusion is possible only when all alternatives have been excluded. A key stage in a mechanistic investigation then is the enumeration of the various possible mechanisms and the design of experiments that distinguish between them. The principal basis for enumerating mechanistic possibilities is accumulated chemical experience with related systems and the inherent structural features of the system. A chemist approaching a

mechanistic study must cast as broad as possible vision on the problem so as not to exclude possibilities. Organic chemistry provides fuels and industrial chemicals from petroleum, coal and biological materials. It supplies plastics, dyes, fibers, pharmaceuticals, detergents, rubber and paints. Chemical engineering is largely organic chemical engineering, carrying out organic reactions on an industrial scale.

REFERENCES

- 1. Adlard, E. R. (2019). E. Hywel Evans and Mike. E. Foulkes: Analytical Chemistry. A Practical Approach.
- Brown, T. L., LeMay, H. E., & Wilson, R. (1988). Chemistry: The central science (No. QD 31.2. B76 1988). Englewood Cliffs, NJ: Prentice Hall.
- Brown, W. H., Poon, T., & Poon, T. (2014). Introduction to organic chemistry. John Wiley & Sons.
- 4. Connor, R. (1937). Laboratory Practice of Organic Chemistry (Robertson, G. Ross).
- Crowe, J., & Bradshaw, T. (2014). Chemistry for the biosciences: the essential concepts. Oxford University Press.
- Dorr, D. Q., Denniston, K. J., & Topping, J. J. (2017). General, organic, and biochemistry. McGraw-Hill.
- 7. Erduran, S., & Kaya, E. (2019). Transforming Teacher Education Through the Epistemic Core of Chemistry. Springer.
- 8. Haynes, W. M. (2014). CRC handbook of chemistry and physics. CRC press.
- 9. Holum, J. R. (1975). Organic chemistry: a brief course (No. QD453. 2. H64I 1975.). Wiley.
- 10. Jacobs, D. S., Kasten, B. L., Demott, W. R., & Wolfson, W. L. (1996). Laboratory test handbook. Lexi-Comp Incorporated.
- 11. Katz, M. (1998). Organic Chemistry, (Bruice, Paula Y.).
- 12. Lahann, J. (Ed.). (2009). Click chemistry for biotechnology and materials science. John Wiley & Sons
- 13. Leicester, H. M., & Klickstein, H. S. (Eds.). (1952). A Source Book in Chemistry, 1400-1900 (Vol. 5). Harvard University Press.

- 14. Leonard, J., Lygo, B., & Procter, G. (2013). Advanced practical organic chemistry. CRC press.
- 15. Nadendla, R. R. (2007). Principles of organic medicinal chemistry. New Age International.
- Nelson, W. M. (2003). Green solvents for chemistry: perspectives and practice. Oxford University Press.
- 17. Ouellette, R. J., & Rawn, J. D. (2014). Organic chemistry: structure, mechanism, and synthesis. Elsevier.
- 18. Petrucci, R. H., Cunningham, C. M., & Moore, T. E. (1989). General chemistry (pp. 136-146). New York: Macmillan.
- 19. Petrucci, R. H., Harwood, W. S., & Herring, F. G. (1993). General chemistry. Prentice Hall.
- 20. Reger, D. L., Goode, S. R., & Ball, D. W. (2009). Chemistry: principles and practice. Cengage Learning.
- 21. Schultz, M., Schmid, S., & Lawrie, G. A. (2019). Research and Practice in Chemistry Education. Springer.
- 22.A. babaei, A. Taheri, Direct Electrochemistry and Electrocatalysis of Myoglobin Immobilized on a Novel Chitosan-Nickel Hydroxide Nanoparticles-Carbon Nanotubes Biocomposite Modified Glassy Carbon Electrode, Anal Bioanal Electrochem, 4(2012) 342 56.
- 23.m. Afrasiabi, Z. Rezayati-zad, s. Kianipour, A. babaei, A. Taheri, A Sensor for Determination of Tramadol in Pharmaceutical Preparations and Biological Fluids Based on Multi-Walled Carbon Nanotubes-Modified Glassy Carbon Electrode, JChemSocpak, 35(2013) 1106-12.
- 24. Smith, J. G. (2010). General, Organic & Biological Chemistry. McGraw-Hill.

- 25. Smith, M. B. (2019). March's advanced organic chemistry: reactions, mechanisms, and structure. John Wiley & Sons.
- 26. Surrey, A. R. (2013). Name reactions in organic chemistry. Elsevier.
- 27. Thorpe, G. S. (2007). CliffsAP 5 chemistry practice exams. Houghton Mifflin Harcourt.
- 28. Toda, F., & Bishop, R. (Eds.). (2004). Separations and reactions in organic supramolecular chemistry. New York: Wiley.

HOW TO CITE THIS ARTICLE

Ayman Y. El-Khateeb Practical Approach for the Identification of Functional Groups in Organic Compounds, Prog. Chem. Biochem. Res. 2020, 3(2), 147.168

DOI: 10.33945/SAMI/PCBR.2020.2.9

URL: http://www.pcbiochemres.com/article 107327.html

