

Formal report exp 9

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Laboratory APPLICATION OF DIFFERENT KINDS OF TEST TO CLASSIFY

HYDROXY- AND CARBONYL-CONTAINING COMPOUNDS Jane Catherine SP.

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Laboratory ABSTRACT Hydroxyl group refers to a functional group containing OH- when it is a substituent in an organic compound. It is also known as the characteristic functional group of alcohols and phenols.

On the other hand, carbonyl group refers to a divalent chemical unit consisting of a carbon and an oxygen atom connected by a double bond. It is known as the characteristic functional group of aldehydes and ketones instead. In this experiment, hydroxyl- or carbonyl- containing samples were given to the group for examination. The samples were analyzed through different tests namely the involvement of the solubility of alcohols in water, the Lucas Test, the Chromic Acid Test or also known as Jones Oxidation, the 2, 4-Dinitrophenylhydrazine (2, 4-DNP) Test, the Fehling's Test, the Tollens' Silver Mirror Test, and the Iodoform Test.

The solubility of alcohols in water test showed that the sample, benzyl alcohol was immiscible while ethanol was the most miscible from all the other compounds used. While in Lucas Test which was used to differentiated the primary, secondary, and tertiary alcohols had turned tert-butyl alcohol into a cloudy solution afterwards. In Chromic Acid Test which was a test for oxidizable compounds or any compounds that possess reducing property would yield to a blue green solution if it reacted positively. This was seen in all the sample used in this test except for acetone.

Whereas Dinitrophenylhydrazone (2, 4-DNP) Test was performed to test for aldehydes and ketones which would result to a yellow orange precipitate if it was positively reacted. All the compounds subjected to this test namely n-butylaldehyde, benzaldehyde and acetone gave a positive result. Fehling's Test and Tollens' Silver Mirror Test were used to tests for aldehydes. In Fehling Test, both the n-butylaldehyde and benzaldehyde gave a positive result which was a brick red precipitate but acetone gave a negative result which was only a blue solution.

While the Tollens' Silver Mirror Test had shown that both n-butylaldehyde and benzaldehyde gave a positive result which was a silver mirror and then again acetone gave a negative result which was the absence of a silver mirror. Lastly Iodoform test was performed and was known as a test for methyl carbinol and methyl carbonyl groups. Both acetone and isopropyl alcohol resulted to a positive outcome in this test which was formation of yellow precipitate but n-butylaldehyde on the other hand yield to a negative result which was a yellow solution containing black precipitate.

INTRODUCTION

In organic chemistry, classification of test was tests that categorize a substance into one of several classes. They were used to detect functional groups and other structural features. Alcohol were derivatives of hydrocarbons in which one or more of the hydrogen atoms have been replaced by a hydroxyl (-OH) functional group. Hydrocarbons are compounds which contain hydrogen (H) and carbon (C) only. The hydroxyl group imparts particular properties to the radical to which it is attached. [1] Figure 1.

Alcohol Alcohols are classified into three categories: primary (1°), secondary (2°) and tertiary (3°).

This classification is based on the number of carbon-containing groups (R for an alkyl or an aromatic group) attached to the carbon bearing the hydroxyl group. If the carbon bearing the OH has one R group, the molecule is a primary alcohol. If two R groups are attached, it is then a secondary alcohol. If three R groups are attached, then the alcohol is tertiary[1][4] Figure 2.

Three alcohol groups There are other molecules that contain an -OH group. Even though water (H_2O) contains OH, it is not considered as an alcohol because alcohols were defined as organic compounds that have little or no ionization of the hydrogen. Other organic compounds that contain -OH groups but are not alcohols are phenol ($\text{C}_6\text{H}_5\text{OH}$) and acetic acid (CH_3COOH). These compounds are not alcohols because they are acidic. The term alcohol, then, is another representation of a type of electronic structure in the molecules of substances. [3] [4] Phenols are aromatic compounds in which a hydroxide group is directly bonded to an aromatic ring system. They are very weak acids, and like alcohols, form ethers and esters. The main phenols are phenol itself, cresol, resorcinol, pyrogallol, and picric acid.

Phenol itself ($\text{C}_6\text{H}_5\text{OH}$), also known as carbolic acid, is a white, hygroscopic crystalline solid, isolable from coal tar, but made by acid hydrolysis of cumene hydroperoxide, or by fusion of sodium benzenesulfonate with sodium hydroxide. Formerly used as an antiseptic, phenol has more latterly been used to make bakelite and other resins, plastics, dyes, detergents, and drugs. [4] [15] The hydroxyl- containing compounds used in the experiment

were ethanol, n-butyl alcohol, sec-butyl alcohol, tert-butyl alcohol, isopropyl alcohol, and benzyl alcohol.

Ethanol also known as ethyl alcohol is a clear, colorless liquid with a characteristic, agreeable odor. In dilute aqueous solution, it has a somewhat sweet flavor, but in more concentrated solutions it has a burning taste. Its low freezing point has made it useful as the fluid in thermometers for temperatures below -40°C , the freezing point of mercury, and for other low-temperature purposes, such as for antifreeze in automobile radiators.

Ethanol is miscible in all proportions with water and with most organic solvents. It is useful as a solvent for many substances and in making perfumes, paints, lacquer, and explosives. 15] Figure 3. Structure of Ethanol n-butyl alcohol also known as n-butanol, 1-Butanol or 1-butyl alcohol is a four carbon straight chain alcohol. It is a volatile, clear liquid with a strong alcoholic odor, and is miscible with water. It is a highly refractive compound which corrodes some plastics, and rubbers. It is miscible with many organic solvents, and incompatible with strong oxidizers. It is also used as a direct solvent and as an intermediate in the manufacture of other organic chemicals. [7] Figure 4. Structure of n-butyl alcohol

Sec-butyl alcohol, a four carbon secondary alcohol, is a volatile, clear liquid with a strong alcoholic odor with a water solubility of 12. 5%. This substance is most hazardous when peroxide levels are concentrated by distillation or evaporation. It is a highly refractive compound which corrodes some plastics, and rubbers. It is miscible with many organic solvents, and incompatible with strong oxidizers. It is flammable strongly with a luminous flame. It is used as

a direct solvent and as an intermediate in the manufacture of other organic chemicals. [8] Figure 5.

Structure of Sec-butyl alcohol Tert-butyl alcohol is a clear, noncorrosive liquid. It is miscible with water as well as most common organic solvents. The sterically hindered tertiary butyl group imparts stability compared to primary and secondary alcohols. As a result, the solubility and oxidative stability characteristics provide many industrial applications as a reaction and process solvent and chemical intermediate. It is used as a non-reactive solvent for chemical reactions, a non-surfactant compatibilizer for many solvent blends, and a non-corrosive solvent.

It is used in free radical polymerizations to dissolve monomers. TBA is a main raw material of tert-butyl functional group in organic synthesis. [9] Figure 6.

Structure of Tert-butyl alcohol Isopropyl alcohol also known as propan-2-ol, 2-propanol is a common name for a chemical compound with the molecular formula C_3H_8O . It is a colorless, flammable chemical compound with a strong odor. It is the simplest example of a secondary alcohol, where the alcohol carbon is attached to two other carbons. Being a secondary alcohol, isopropyl alcohol can be oxidized to acetone, which is the corresponding ketone.

Isopropyl alcohol dissolves a wide range of non-polar compounds. It is also relatively non-toxic and evaporates quickly. Thus it is used widely as a solvent and as a cleaning fluid, especially for dissolving lipophilic contaminants such as oil. [10] Figure 7. Structure of Isopropyl alcohol Benzyl alcohol ($C_6H_5CH_2OH$) is a colorless liquid with a mild pleasant aromatic odor.

It is a useful solvent due to its polarity, low toxicity, and low vapor pressure. Benzyl alcohol is partially soluble in water (4 g/100 mL) and completely miscible in alcohols and diethyl ether.

Like most alcohols, it reacts with carboxylic acids to form esters. Benzyl alcohol is used as a general solvent for inks, paints, lacquers, and epoxy resin coatings. It is also a precursor to a variety of esters, used in the soap, perfume, and flavor industries. It is often added to intravenous medication solutions as a preservative due to its bacteriostatic and antipruritic properties. [15] Figure 8. Structure of Benzyl alcohol Carbonyl group is a divalent chemical unit consisting of a carbon and an oxygen atom connected by a double bond.

The group is a constituent of carboxylic acids, esters, anhydrides, acyl halides, amides, and quinones, and it is the characteristic functional group of aldehydes and ketones. Carboxylic acid and their derivatives, aldehydes, ketones, and quinones are also known collectively as carbonyl compounds. Aldehydes and ketones contain carbonyl groups attached to alkyl or aryl groups and a hydrogen atom or both. These groups have little effect on the electron distribution in the carbonyl group; thus, the properties of aldehydes and ketones are determined by the behavior of the carbonyl group.

In carboxylic acids and their derivatives, the carbonyl group is attached to one of the halogen atoms or to groups containing atoms such as oxygen, nitrogen, or sulfur. These atoms do affect the carbonyl group, forming a new functional group with distinctive properties. Figure 9. Carbonyl Group An aldehyde is an organic compound containing a terminal carbonyl group. This

functional group, called an aldehyde group, consists of a carbon atom bonded to a hydrogen atom with a single covalent bond and an oxygen atom with a double bond.

Thus the chemical formula for an aldehyde functional group is -CH=O , and the general formula for an aldehyde is R-CH=O . The aldehyde group is occasionally called the formyl or methanoyl group. The word aldehyde is a combination of parts of the words alcohol and dehydrogenated, because the first aldehyde was prepared by removing two hydrogen atoms (dehydrogenation) from ethanol. Molecules that contain an aldehyde group can be converted to alcohols by the addition of two hydrogen atoms to the central carbon oxygen double bond (reduction).

Organic acids are the result of the introduction of one oxygen atom to the carbonyl group (oxidation). Aldehydes are very easy to detect by smell. Some are very fragrant, and others have a smell resembling that of rotten fruit. [15] On the other hand, Ketone features a carbonyl group (C=O) bonded to two other carbon atoms. They differ from aldehydes in that the carbonyl is placed between two carbons rather than at the end of a carbon skeleton. They are also distinct from other functional groups, such as carboxylic acids, esters and amides, which have a carbonyl group bonded to a hetero atom.

Ketone compounds have important physiological properties. They are found in several sugars and in compounds for medicinal use, including natural and synthetic steroid hormones. [15] The difference between aldehydes and ketones is in the groups that are attached to the carbonyl carbon atom. In

the case of an aldehyde, there is always at least one H atom attached to the carbonyl carbon atom. An aldehyde has one R group attached. R stands for any other organic chain or group. In the case of ketones, there are no H atoms attached to the carbonyl carbon. The ketone has two R groups attached. [2] [15] Figure 10.

Structure of Aldehyde and Ketone Some of the carbonyl-containing compounds used in the experiment were benzaldehyde, n-butanaldehyde, acetaldehyde, acetone and acetophenone. Benzaldehyde ($\text{C}_6\text{H}_5\text{CHO}$) also known as benzenecarbonal is a colorless liquid aldehyde with a characteristic almond odor. It boils at 180°C , is soluble in ethanol, but is insoluble in water. It is formed by partial oxidation of benzyl alcohol, and on oxidation forms benzoic acid. It is called oil of bitter almond, since it is formed when amygdalin, a glucoside present in the kernels of bitter almonds and in apricot pits, is hydrolyzed, e. . , by crushing the kernels or pits and boiling them in water; glucose and hydrogen cyanide (a poisonous gas) are also formed. It is also prepared by oxidation of toluene or benzyl chloride or by treating benzal chloride with an alkali. Benzaldehyde is used in the preparation of certain aniline dyes and of other products, including perfumes and flavorings. [13] Figure 11. **Structure of Benzaldehyde** Acetaldehyde (CH_3CHO) also known as ethanol is a colorless liquid aldehyde, sometimes simply called aldehyde. It is soluble in water and ethanol.

Acetaldehyde is made commercially by the oxidation of ethylene with a palladium catalyst. It is used as a reducing agent (e. g. , for silvering mirrors), in the manufacture of synthetic resins and dyestuffs, and as a

preservative. [11] Figure 12. Structure of Acetaldehyde n-butyraldehyde ($\text{CH}_3(\text{CH}_2)_2\text{CHO}$) also known as butanal is an aldehyde derivative of butane. It is a colorless flammable liquid that smells like sweaty feet. It is miscible with most organic solvents. n-butyraldehyde is used as an intermediate in the manufacturing plasticizers, alcohols, solvents and polymers.

It is also used as an intermediate to make pharmaceuticals, agrochemicals, antioxidants, rubber accelerators, textile auxiliaries, perfumery and flavors.

[12] Figure 13. Structure of N-butyraldehyde Acetone ($(\text{CH}_3)_2\text{CO}$) also known as propanone is colorless, mobile, flammable liquid with a characteristic sweetish smell is the simplest example of the ketones. Acetone is miscible with water and serves as an important solvent in its own right, typically as the solvent of choice for cleaning purposes in the laboratory. [6] Figure 14. Structure of Acetone Acetophenone ($\text{C}_6\text{H}_5\text{C}(\text{O})\text{CH}_3$) is the simplest aromatic ketone.

This colorless, viscous liquid is a precursor to useful resins and fragrances. It can be obtained by a variety of methods. In industry, acetophenone is recovered as a by-product of the oxidation of ethylbenzene, which mainly gives ethylbenzene hydroperoxide for use in the production of propylene oxide. [5] Figure 15. Structure of Acetophenone The hydroxyl- and carbonyl-containing compounds were analyzed by utilization of different tests such as testing the solubility of alcohols in water, Lucas Test, Chromic Acid Test (Jones Oxidation), 2, 4-Dinitrophenylhydrazone Test, Fehling's Test, Tollens' Silver Mirror Test, and Iodoform Test.

Most organic compounds were not soluble in water with the exception of low molecular-weight amines and oxygen-containing compounds like alcohols, carboxylic acids, aldehydes, and ketones. Low molecular-weight compounds are generally limited to those with fewer than five carbon atoms. [14] Lucas Test often provides classification information for alcohols, as well as a probe for the existence of the hydroxyl group. Substrates that easily give rise to cationic character at the carbon bearing the hydroxyl group undergo this test readily; primary alcohols do not give a positive result.

Since the Lucas Test depends on the appearance of the alkyl chloride as a second liquid phase, it is normally applicable only to alcohols that are soluble in the reagent. This limits the test in general to monofunctional alcohols lower than hexyl and certain polyfunctional molecules. [4] Chromic Acid Test also called Jones Oxidation detects the presence of a hydroxyl substituent that is on a carbon bearing at least one hydrogen, and therefore oxidizable. It is detected by the appearance of Cr^{+3} ion. This test can be used to differentiate aldehydes and ketones.

A positive result would show green or blue-green solution. [4] 2, 4-Dinitrophenylhydrazone Test can be used to qualitatively detect the carbonyl functionality of a ketone or aldehyde functional group. Ketones and Aldehydes would form yellow to orange precipitate after undergoing in this test. [4] Fehling's Test and Tollens' Silver Mirror Test are used to detect aldehydes. However, Fehling's solution can only be used to test for aliphatic aldehydes, whereas Tollens' reagent can be used to test for both aliphatic and aromatic aldehydes.

A positive result in Fehling's Test would give a brick red precipitate while in Tollens' Silver Mirror, it is the formation of silver mirror. [4] Iodoform Test is a test for methyl carbinol and methyl carbonyl group. A positive result would yield to yellow crystals or precipitate. Its mechanism occurs through a series of enolate anions which are iodinated. [4] The objectives of the experiment were to distinguish whether a compound was a hydroxyl- or carbonyl-containing, to differentiate the three types of alcohols, to differentiate aldehydes from ketones and to explain the mechanisms involved in the differentiating tests.

EXPERIMENTAL A. Compounds Tested * Ethanol * n-butyl alcohol * Sec-butyl alcohol * Tert-butyl alcohol * Benzyl alcohol * n- butyraldehyde *

Benzaldehyde * Acetone * Acetophenone * Isopropyl alcohol * Acetaldehyde * Lucas reagent * Chromic acid reagent * 95% ethanol * Fehling's A and B * Tollen's reagent * 5% NaOCl solution * Iodoform test reagent * 2, 4-

dinitrophenylhydrazine B. Procedure 1. Testing the solubility of alcohols in water The samples involved in the experiment were ethanol, n-butyl alcohol, sec-butyl alcohol, tert-butyl alcohol, and benzyl alcohol.

Five test tubes were labeled with each of the alcohol samples. With the aid of a Pasteur pipette, 10 drops from each of the samples were taken then placed into the appropriate test tube. To the tube containing ethanol, 1-ml of water was then added drop wise to the tube containing alcohol and the mixture was shaken thoroughly after each addition. If cloudiness resulted, 0. 25-ml of water at a time was added continuously with vigorous shaking until a homogeneous dispersion results. The total volume of water added was

noted. If cloudiness resulted after the addition of 2. -ml of water, the alcohol is said to be immiscible in water but if there was no cloudiness then it is miscible to water. The results were noted down. The same procedure was performed on the test tubes containing n-butyl alcohol, sec-butyl alcohol, tert-butyl alcohol, and benzyl alcohol. 2. Using the Lucas Test This test was performed on n-butyl alcohol, sec-butyl alcohol, tert-butyl alcohol and isopropyl alcohol. Lucas reagent was prepared by dissolving 16 g of anhydrous zinc chloride in 10-ml of concentrated hydrochloric acid. The mixture was then allowed to cool.

The Lucas Reagent was already prepared beforehand. 50-mg or 2-3 drops of the sample was added to 1-ml of the reagent in a test tube and the mixture was shaken vigorously for a few seconds. The mixture was allowed to stand at room temperature. The rate of formation of the cloudy suspension or the formation of two layers was observed. 3. Using the Chromic Acid Test / Jones Oxidation This test was performed on n-butyl alcohol, sec-butyl alcohol, tert-butyl alcohol, benzaldehyde and acetone. 1 drop of liquid or a small amount of the solid sample was dissolved in 1-ml of acetone in a small vial or test tube. drops of 10% aqueous Potassium chromate solution and 5 drops of 6M sulphuric acid were added into the mixture. 4. Using the 2, 4-DNP Test This test was performed on acetone, n-butyraldehyde and benzaldehyde. The reagent was prepared by slowly adding a solution of 3 g of 2, 4-dinitrophenylhydrazine in 15-ml of concentrated sulphuric acid, while stirring to a mixture of 20-ml of water and 70-ml of 95% ethanol. The solution was then stirred and filtered. This reagent was already prepared beforehand. A

drop of a liquid sample was placed into a small sample. 5 drops of 95% ethanol was added and well shaken.

Afterwards, 3 drops of 2, 4-DNP was added and if no yellow or orange precipitate formed, the solution was allowed to stand for at least 15 minutes.

5. Using the Fehling's Test This test was performed on acetone, n-butyraldehyde, and benzaldehyde. Fehling's reagent was prepared by mixing equal amounts of Fehling's A and Fehling's B. Fehling's A was prepared by dissolving 7 g of hydrated copper (II) sulfate in 100-ml of water. Fehling's B was prepared by mixing 35 g of Potassium sodium tartrate and 10 g of Sodium hydroxide in 100-ml water. Then, 1-ml of freshly prepared Fehling's reagent was placed into each test tube. drops of the sample to be tested was added in to the tube. The tubes were then placed in a beaker of boiling water and changes within 10-15 minutes were observed. 6. Using the Tollens' Silver Mirror Test This test was performed on benzaldehyde, acetone and n-butyraldehyde. The reagent was prepared by adding 2 drops of 5% Sodium hydroxide solution to 2-ml of 5% Silver nitrate solution and mixing thoroughly. Next, only enough 2% ammonium hydroxide (concentrated ammonium hydroxide is 28%) was added drop by drop and with stirring to dissolve the precipitate.

Adding excess ammonia will cause discrepancies on the result of the test. Then, four test tubes with 1-ml of freshly prepared Tollens' reagent were prepared. Two drops each of the samples were then added. The mixture was shaken and allowed to stand for 10 minutes. If no reaction has occurred, the test tube was placed in a beaker of warm water (35-50 oC) for 5 minutes.

Observations were recorded. It was noted that if Tollens' reagent is left unused for a period of time, it may form explosive silver. This was avoided by neutralizing unused reagent with a little nitric acid and discarded afterwards. . Using the Iodoform Test This test was performed on acetone, n-butyraldehyde and isopropyl alcohol. 2 drops of each sample was placed into its own small vial or test tube. 20 drops of fresh chlorine bleach (5% Sodium hypochlorite) was slowly added while shaking to each test tube and then, mixed. The formation of a yellow precipitate was noted. RESULTS AND DISCUSSION 1. Solubility of Alcohols in Water In the experiment, five compounds were tested to determine the presence of the -OH, hydroxyl group through solubility of the sample in water.

The presence of an -OH group was indicated by the miscibility of the substance. This follows the general rule in solubility that "like dissolves like". Meaning, a polar solute will dissolve in a polar solvent and a non polar solute will be insoluble in a polar solvent. [14] Going back to the experiment, it was observed that alcohol was soluble in water but as the number of carbon atoms in the carbon chain of the alcohol increased, the solubility of the alcohol sample decreased. It was also observed that branching of the compound increased its solubility in water.

Branching will increase solubility since more branching will reduce the size of the molecule and make it easier to solvate the molecules with the solvent. [14] The results of the experiment show that the solubility of alcohols in water depends on the balance between the strength of the hydrogen bonds formed between water and the hydroxyl group, and the strength of the Van

der Waals forces between the hydrocarbon chains of the alcohol. Alcohol| Condensed Structural Formula| Amount of Water (in ml) needed to produce a homogeneous dispersion| Solubility in Water| Ethanol| $\text{CH}_3\text{CH}_2\text{OH}$ | 0. ml| Most Miscible| n-butyl alcohol| $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$ | 2. 0 ml| Miscible| Sec-butyl alcohol| | 1. 4 ml| Miscible| Tert-butyl alcohol| | 0. 5 ml| Miscible| Benzyl alcohol| | More than 2. 0 ml| Immiscible| Table 1. Solubility of alcohols in water The table above showed that ethanol, n-butyl alcohol, sec-butyl alcohol, and tert-butyl alcohol were all miscible with water. Only benzyl alcohol had exhibited immiscibility with water. As stated, all alcohols were soluble in water except under C6. Hence, ethanol, n-butyl alcohol, sec-butyl alcohol, and tert-butyl alcohol are all miscible with water.

Ethanol has two carbon atoms, while the other three all have four carbons since they are all derivatives of the alcohol, butanol. Benzyl alcohol was immiscible with water because it is an aromatic alcohol. Ethanol was the most miscible alcohol followed by tert-butyl alcohol, sec-butyl alcohol, and n-butyl alcohol. Ethanol exhibited fastest solubility because it has only two carbon atoms as compared to the butanol derivatives having four carbon atoms. Tert-butyl alcohol was the most miscible among the butanol derivatives because it has the most branching substituents present. 2. Lucas Test

The four types of alcohols namely n-butyl alcohol, sec-butyl alcohol, tert-butyl alcohol and isopropyl alcohol were differentiated from each other by way of the Lucas Test. Lucas Test differentiates primary, secondary, and tertiary alcohols. Reagents used include anhydrous ZnCl_2 and HCl . Positive

result was based on its turbidity or alkyl chloride formation and its rate of the reaction. Tertiary alcohols formed the second layer in less than a minute. Secondary alcohols required 5-10 minutes before formation of second layer while primary alcohols were usually unreactive.

Substance	Condensed Structural Formula	Reaction
n-butyl alcohol	$\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$	Clear solution(+)
Sec-butyl alcohol		Clear solution(+)
Tert-butyl alcohol		Turbid (+++) / Cloudy solution and formation of two layers
Isopropyl Alcohol		Clear solution(+)

Table 2. Lucas Test

Based on Table 2, it was only tert-butyl alcohol which had immediately formed two layers or a cloudy solution; hence, it was known to be a tertiary alcohol. Sec-butyl alcohol and Isopropyl alcohol when subjected to Lucas test resulted to a clear solution although theoretically, a secondary alcohol dissolves to give a clear solution then form chlorides which would yield to a cloudy solution within five minutes. n-butyl alcohol was considered as a primary alcohol. It was unreactive but eventually would react after long period of time. Generally, the order of reactivity of the alcohols toward Lucas reagent was 3° ; 2° ; 1° because the reaction rate was much faster when the carbocation intermediate was more stabilized by a greater number of electron donating alkyl group bonded to the positive carbon atom. This means that the greater the alkyl groups present in a compound, the faster its reaction would be with the Lucas solution. [1] Figure 16.

Reaction in Lucas Test 3. Chromic Acid Test (Jones Oxidation) This test was performed on n-butyl alcohol, sec-butyl alcohol, tert-butyl alcohol, benzaldehyde and acetone. The chromic acid test classifies the three types of alcohols by oxidizing the alcohol. The test was also used to be able to

distinguish aldehydes from ketones. Since primary and secondary alcohols were also oxidized by the chromic acid reagent, this test was not useful for distinguishing aldehydes unless a positive identification of a carbonyl group has been obtained from the 2, 4-DNP test.

Chromic acid has an orange-red color due to the presence of Cr^{+6} ions, upon oxidation of the aldehyde, the chromium was reduced to Cr^{+3} , which had a green color. A positive result was indicated by a green precipitate due to chromous sulfate, $\text{Cr}^{+3}(\text{SO}_4)_2 \cdot x\text{H}_2\text{O}$. [1] From the results, it was noted that the formation of an opaque blue-green suspension within 2-3 seconds, accompanied by disappearance of the orange color of the reagent, indicates a primary or secondary alcohol. A primary alcohol oxidizes readily, first to an aldehyde, then to a carboxylic acid.

These two oxidation steps made sense because the primary alcohol functional group has two C-H bonds that can be broken; secondary alcohols were oxidized to ketones, a secondary alcohol only has one C-H bond that can be broken, so it can only oxidize once, to a ketone; a tertiary alcohol has no C-H bond that can be broken, so it was not oxidized, no matter how strong the oxidizing agent was. During the oxidation, the orange-red color of the chromic acid changed to a blue-green solution. Figure 17. Oxidation of the three types of Alcohols The results also show that aldehydes gave the same result but reacted more slowly.

With aliphatic aldehydes, the solution turned cloudy in about 5 seconds, and the opaque blue-green suspension formed within 30 seconds; aromatic aldehydes required 30-90 seconds or longer before a suspension formed.

The generation of some other dark color, particularly with the liquid remaining orange, was considered a negative test. It was concluded that alcohols and aldehydes are oxidized by chromic acid but ketones were not because they don't have a hydrogen atom attached to their carbonyl group that can be used for oxidation. Figure 18. Oxidation of Aldehyde Substance| Condensed Structural Formula| Reaction| -butyl alcohol| $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$ | Blue green solution(+)| Sec-butyl alcohol| | Blue green solution(+)| Tert-butyl alcohol| | Blue green solution (+)| n-butyraldehyde| | Blue greenSolution (+)| Benzaldehyde| | Blue green solution(+)| Acetone| | Green solution(-)| Table 3.

Reactions to the Chromic Acid Test It was observed that all the compounds tested gave a visible positive result, a blue green solution, except for acetone which had yielded to a green solution. 4. 2, 4-dinitrophenylhydrazine Test This test was performed on acetone, n-butyraldehyde and benzaldehyde.

The 2, 4-dinitrophenylhydrazine (2, 4-DNP) test determined the presence of a carbonyl group in the sample compound. The test used an organic reactant, 2, 4-dinitrophenylhydrazine, to distinguish the carbonyl compounds, aldehydes and ketones, from the non-carbonyl compounds, alcohols. The 2, 4-dinitrophenylhydrazine reagent was a translucent yellow solution. When this reagent was subjected in the presence of a carbonyl compound, a yellow colored precipitate would form while in the presence of an alcohol, the solution would remain translucent yellow with no precipitate formed.

The reaction of 2, 4-DNP with an aldehyde or ketone was a condensation reaction. Under less acidic conditions, in this type of reaction, a nucleophile donates a pair of electrons toward the carbonyl carbon forming a single bond to it. [2] At the same time the double bond between the carbonyl carbon and oxygen becomes a single bond as one bonding pair of electrons in the double bond moves to become an unshared pair on the oxygen. The oxygen now has one bond to it and it holds three pairs of unshared electrons, so it has a negative charge.

Consequently, the oxygen picks up a proton from somewhere and becomes an -OH group. The proton from the acid attaches itself to one of the unshared pairs of electrons on the oxygen. The carbonyl group now has a +1 charge and is very inviting to even a weak nucleophile. So, the nucleophile attacks the carbonyl carbon forming a bond and the doubly bonded oxygen of the carbonyl becomes an -OH, as before. [1] Figure 19. Nucleophilic addition of 2, 4-DNP to Acetone. As seen just below, this product is not usually the one that was isolated.

Rather this product undergoes an elimination reaction in which the -OH was removed from the carbon to which it is attached and the hydrogen was removed from the nitrogen immediately to the right, resulting in a double bond between the nitrogen and carbon and a molecule of water. The final product was known as a 2, 4-dinitrophenylhydrazone. That is why this reaction was also considered as an elimination reaction. Figure 20.

Elimination reaction of DNP Figure 21. Reaction of 2, 4-DNP with a Carbonyl

group	Substance	Condensed Structural Formula	Reaction	n-butyraldehyde
				Yellow- orange precipitate(+)

Benzaldehyde | Yellow -orange precipitate(+)| Acetone | Yellow -orange precipitate(+)|

Table 4. Reactions to the 2, 4- DNP Test As shown on table 4, it was observed that there was a formation of a yellow – orange precipitate in all the compounds used. This would then indicate a presence of either an aldehyde or a ketone.

5. Fehling's Test Fehling's test differentiated aldehydes and ketones. It was based upon the ability of the aldehyde group to reduce the Cu^{+2} ion of $\text{Cu}(\text{OH})_2$, a blue color, to the Cu^{+} ion of Cu_2O , a dark red color, in the presence of a base.

Fehling's solution contains copper (II) ions complex with tartrate ions in sodium hydroxide solution. Complexion of the copper (II) ions with tartrate ions prevents precipitation of copper (II) hydroxide. Aldehydes reduce the complex copper (II) ion to copper (I) oxide, changing the color of the solution to brick red or dark green. Because the solution is alkaline, the aldehyde itself is oxidized to a salt of the corresponding carboxylic acid. [2] In short it involved a redox reaction wherein aldehyde was oxidized to carboxylic acid and ketones did not undergo oxidation. Copper was reduced from Cu^{2+} to Cu^{+} . Figure 22.

Oxidation of aldehyde to carboxylic acid through Fehling's test It was a test for aldehydes. Reagents include CuSO_4 , NaOH . A positive result is the formation of brick red precipitate (Cu_2O /cuprous oxide). This test was performed on acetone, n-butyraldehyde, and benzaldehyde.

Substance	Condensed Structural Formula	Reaction	n-butyraldehyde
			Brick red

precipitate (+)| Benzaldehyde| | Brick red precipitate(+)| Acetone| | Clear blue solution(-)| Table 5. Reactions to the Fehling's Test As shown in Table 5, n-butyraldehyde and benzaldehyde exhibited positive result while acetone exhibited an absence of brick red precipitate.

It can be concluded that n-butyraldehyde and benzaldehyde were both aldehyde. 6. Tollens' Silver Mirror Test Tollens' silver mirror test was a test for aldehydes. Tollen's reagent was an ammoniacal solution of silver ion prepared by dissolving silver oxide in ammonia. The preparation of the reagent is based on the formation of a silver diamine complex that is water soluble in basic solution. In this reaction, the aldehyde was oxidized to a carboxylic acid while the Ag^+ was reduced to silver metal, which deposited as a thin film on the inner surface of the glass.

The generic reaction was as follows and was specific for aldehydes. [16]

Figure 23. Oxidation of aldehyde with Tollen's reagent Substance|

Condensed Structural Formula	Reaction
n-butyraldehyde	Silver Mirror(+)
Benzaldehyde	Silver Mirror(+)
Acetone	Clear grayish-black solution (-)

Table 6. Reactions to Tollen's Silver Mirror Test This test was performed on benzaldehyde, acetone and n-butyraldehyde. Based on the results seen in table 6, it was concluded that the aldehyde samples produced a silver mirror on the inner surface of the test tube since aldehydes were easily oxidized.

The ketones sample, acetone, on the other hand, didn't form this mirror image because of its inability to oxidize due to the lack of a hydrogen atom attached to its carbonyl group that could be used for oxidation. 7. Iodoform Test Iodoform test was a test for methyl carbinol, secondary alcohol with

adjacent methyl group, and methyl carbonyl. Methyl ketones, but not other ketones, were oxidized by iodine in aqueous sodium hydroxide. The ketone was oxidized to a carboxylic acid which yellow iodoform/ precipitate would be formed. It was the yellow precipitate formed would be the basis of a positive result.

Acetaldehyde, but not other aldehydes, would yield to a positive result in this test owing to its structural similarity to methyl ketones. It was also true that ethanol would be oxidized to acetaldehyde and secondary alcohols that could be oxidized to methyl ketones given this test. [2] Figure 24. Oxidation of a methyl ketone Substance| Condensed Structural Formula| Reaction| N-butyraldehyde| | Yellow solution with black precipitate (-)| Acetone| | Yellow precipitate| Isopropyl alcohol| | Yellow precipitate| Table 7. Reaction to the Iodoform Test

This test was performed on acetone, n- butyraldehyde and isopropyl alcohol. Based on table 7, the results indicate that the methyl ketones of isopropyl alcohol and acetone were oxidized by iodine to carboxylic acids because the compounds formed a yellow precipitate while n-butyraldehyde didn't. It was then concluded that compounds with a methyl group next to the carbonyl group would give a positive result in the iodoform test, ethanol and secondary alcohols with the methyl group attached to the same carbon as the OH- group would also give a positive result.

During the experiment, the compounds acetaldehyde and acetophenone were not available. This was the reason why results of these compounds in different tests were not observed but based from different informations

which were gathered from different sources. Acetophenone would give a positive result in the following test namely 2, 4 DNP test and Iodoform test. While acetaldehyde would give a positive result in the following test namely Chromic Acid test, 2, 4 DNP test, Fehling's test and as well as Tollens' Silver Mirror test. REFERENCES: From books: [1]Lehman, John W(2009).

Operational Organic Chemistry: A Problem-Solving Approach to the Laboratory Course. Upper Saddle River, New Jersey: Pearson Prentice.

[2]Martin, Stephen F(2011). Organic Chemistry Laboratory Experiments: Miniscale and Microscale. Brooks/Cole Cengage Learning. [3]McMurry, John(2010). Foundations of Organic Chemistry, Philippine Edition. Cengage Learning. [4]Shriner, Ralph Lloyd (1980). Systematic Identification of Organic Compound: A Laboratory Manual (6th Ed.). John Wiley ; Sons, Inc. New York: Van Hoffmann Press. From Websites: [5]Acetophenone. www.chemicaland21.com/industrialchem/solalc/ACETOPHENONE.htm 09/09/11 [6]Acetone. www.chemicaland21.com/industrialchem/solalc/ACETONE.htm 09/09/11 [7]n-butyl alcohol. www.chemicaland21.com/industrialchem/solalc/NBUTYLALCOHOL.htm 09/09/11 [8]Sec-butyl alcohol. www.chemicaland21.com/industrialchem/solalc/2-BUTANOL.htm 09/09/11 [9]Tert-butyl alcohol. www.chemicaland21.com/industrialchem/solalc/TERTBUTYL%20ALCOHOL 09/10/11 [10]Isopropyl alcohol. www.chemicaland21.com/industrialchem/solalc/ISOPROPANOL.htm 09/10/11 [11]Acetaldehyde. www.ntp.niehs.nih.gov/ntp/roc/eleventh/profiles/s001acet.df 09/10/11 [12]n-butyraldehyde. www.chemicaland21.com/industrialchem/organic/N-BUTYRALDEHYDE.htm 09/10/11 [13]Benzaldehyde. www.chemicaland21.com/industrialchem/organic/BENZALDEHYDE.htm 09/10/11

com/specialtychem/perchem/BENZALDEHYDE. htm 09/10/11 [14]Solubility of Things. www. solubilityofthings. com/water/alcohols 09/09/11 [15]Alcohols, Aldehydes and Ketones. www. ipfw. edu/chem/112/kimble/3-Alcohol %20Aldehyde%20Ketones. pdf 09/10/11 From scientific journals: [16]Ennis, J. L. and E. S. Shanley. " Silver Nitrides. " Journal of Chemical Education(1991): 68, A6. " Silver Nitrides. " Journal of Chemical Education (1991): 68, A6.