# Aldehyde, ketone tests and preparation of derivatives

**Results:**

**I. Aldehyde and ketone testes and preparation of derivatives**

**2, 4 DINIROPHENYL HYDRAZINE**

**Observations**

2-butanone

Solid orange precipitate at bottom

Benzaldehyde

mp of derivative: 217-220°C dark yellow precipitate forms.

**BISULFITE ADDITION TEST**

Acetophenone

Cloudy , off-white solution (no reaction)

2-butanone

Clear solution and no colour change(no reaction)

Benzaldehyde

White precipitation forming after few minutes from clear solution. It appears like white solid crystals.

Trans-cinnamaldehyde

Thick white yellow particles floating (precipitate) on top and clear solution at bottom.

## IODOFORM TEST

Acetophenone

Solution turned to yellow, yellow precipitate form

n-butyraldehyde

Forms 2 layers: top layer creamy and bottom layer lime yellow(no reaction)

2, 4-pentanedione

3 layers: top layer lime yellow, middle layer foggy and bottom layer orange-yellow precipitate.

## OXIDATION OF ALDENHYDES

2-butanone

Orange red colour precipitate turns to green after long time. Takes long time to react.

n-butyraldehyde

Bottom greenish brown colour precipitate and top layer brown. Precipitate after 1 minute

Benzaldehyde

Dark greenish brown precipitate and liquid is greenish colour forms immediately after adding CrO3.

## II. Alcohol tests and preparation of derivatives

SODIUM TEST

Observations

1-butanol

Litmus test pH of 9-10 is observed forms dark blue colour. Bubbles forms after adding sodium in solution. 2 layers are seen which are clear after adding ether.

## LUCAS TEST

1-butanol

2 layers: top layer light orange and bottom layer clear solution

2-pentanol (sec-amyl alcohol)

2 layers: top layer light clear and bottom is off-white. Observed after 3 minute.

1-propanol

Clear solution. Observed after 2 minutes.

t-butyl alcohol(2-methyl-2-propanol)

Reaction occurs as soon as reagent is added. 3 layers: top clear, middle layer off-white and bottom off-white. Observed in 2 seconds.

## URETHANE DERIVATIVE

1-heptanol

Mp: 25-30 °C white crystals forms after cooling, white precipitate

## III. Solubility tests

Solubility In Water

Solubility in Cyclohexane

methanol

Soluble

Insoluble

Ethanol

Soluble

Soluble

Diethyl ether

Insoluble

Soluble

Cyclohexanol

Insoluble

Soluble

Acetone

Soluble

Soluble

2-butanone (ethyl-methyl ketone)

Insoluble

Soluble

Cyclohexanone

Insoluble

Soluble

## Introduction:

Identifying and classifying of the unknown molecules is significant part of organic chemistry. The purpose of this lab is to identify functional groups which are present in alcohol, ether, aldehyde and ketone. This lab helps in gaining knowledge about chemical and physical properties of all this four structures. Distinguish them using function group and solubility tests. In this experiment several test such as physical properties including melting point and index of refraction with literature values to compare. Furthermore, identification can be acquired by crystalline derivative and comparing its derivative melting point with original to find the original carbonyl functional group present. Lucas test is used for determining if alcohol is primary, secondary or tertiary. This depends on the substitution reactions in the presence of acid but, the rate at which it reacts depends on the structure of alcohol. Aldehyde and ketone reacts with 2, 4-dinitrophenylhydrazine to form yellow-orange precipitate but, does not react with alcohol. In addition, iodoform test use to recognize methyl ketone by forming precipitation. In

this experiment it is necessary to give all attention to what reaction is being form so that accurate results can be obtained.

**Discussion:**

2, 4-dinitrophenylhydrazine reagent helps to identify carbonyl groups associated with ketone and aldehyde. When this reagent is added solid yellow-orange precipitate is observed in benzaldehyde and 2-butanone which indicated positive test. Derivatives of this can also, be use to identify the compound. In the experiment benzaldehyde melting point is 217-220°C but, original is 237 °C which is close enough (Caroly and David(1999)). This can be due to error caused by taking too much of benzaldehyde to measure its melting point or some impurities in crystals. This test is also called brady's test.

+ 2, 4-dinitrophenylhydrazine –à 2, 4-dinitrophenylhydrazone (precipitate)

+ 2, 4-dinitrophenylhydrazine –à 2, 4-dinitrophenylhydrazone (precipitate)

**benzaldehyde**

Nitrogen gets attach to carbonyl group after reagent is added therefore, dehydration in reaction results. 2, 4-dinitrophenylhydrazine does not react with alcohol therefore, it is good reagent for carbonyl group.

Sodium bisulfite addition test is only positive with aldehydes and methyl ketones. This reaction does not occur with stearically hindered ketones but, it is best indication for aldehyde. In this experiment no reaction occurred in acetophenone and 2-butonone when bisulfite is added to it. This test is

positive when white precipitate is formed which is observed when it is added in benzaldehyde and trans-cinnamaldehyde.

+ NaHSO3 (sodium bisulfite) —à white precipitate

**Benzaldehyde**

Iodoform test is use to distinguish ketones from methyl ketones, when the test is positive it forms iodoform as yellow precipitate. Acetaldehyde and alcohols with hydroxyl group at its second place can also give positive and alcohol can oxidize to methyl ketone with iodoform reaction as I2 is oxidizing agent. It is observed that acetophenone and 2, 4-pentanedione both reacts as it form yellow precipitate whereas, n-butyraldehyde did not. As, n-butyraldehyde did not contain methyl group it did not react. On the other hand, acetophenone and 2, 4-pentanedione contains methyl group which helps replace a hydrogen atom next to carbonyl group with iodine further, cleaves the iodinated compound to iodoform and carboxylic acid (yellow precipitation). This classifies them as methyl ketones.

NaOH and I2 -à + iodoform

2, 4-pentanedione

Oxidation of aldehyde both n-butyraldehyde and benzaldehyde reacts and gives greenish precipitation when reacts with chromic reagent. Therefore, positive sign is it gives greenish bluish precipitate which represents that oxidation or reduction has occurred. Aldehydes are oxidized easily due to chromic acid and forms carboxylic acid whereas, ketones. Chromate reagent replaces hydrogen atom and adds oxygen resulting carboxylic acid. 2-

butanone is not able to react because it is ketone and it lacks hydrogen atom next to carbonyl group.

C3H7CHO(n-butyraldehyde ) + CrO3(chromic acid) –à n-butyric acid

(benzaldehyde) + CrO3 (chromic acid)à Benzoic acid

When sodium is reacted with 1-butanol hydrogen gas is release because alcohols are weakly acidic so went they react with alkali metal gas is produce. So, when tested with litmus paper solution found to be basic.

(l) +Na(s) à CH3(CH2)3ONa(l) + H-H(g)

In lucas test t-butyl alcohol reacted immediately with lucas reagent (HCl and ZnCl2) because it is tertiary alcohol. It forms stable carbocation and forms chlorinated alkane after attracting negative chlorine. Positive test indicates when turbidity due to formation of insoluble alkyl chloride is mark. 1-propanol and 1- butanol does not react with lucas reagent but, 2-pentanol reacts very slowly and reaction is observed after long time. So, lucas test helps determine rate and which is tertiary or secondary alcohol.

(CH3)3C-OH + ZnCl2/HCl à H2O + (CH3)3C-Cl

Urethane derivative reacts with 1-heptanol which results in phenyl urethane as white precipitate. Phenyl isocynate reacts with alcohol.

+ —à N-phenyl urethane derivative

Melting point of this is 25-30 °C but original is 60°C (Caroly and David(1999)) there is a lot of difference; this can be due to error performed during experiment.

Solubility test concludes that alcohol with less than six carbons are soluble in water, rest of them due to increasing number of carbon it is insoluble in water.

In this experiment almost all results were equivalent to expect results.

**Questions:**

**1)**

a. Certain are like hydroquinone, whose tautomer is ketone and it is oxidizable. Uncertain is if there are other things attach to this molecules may be these tests were not able to detect it example cyano or nitro groups.

b. Catechol

c. IR will identify all other functional groups which were unable to detect by qualitative tests. NMR will give information about isomers example between hydroquinone and catechol.

**2) Chromic acid forms a chromate ester that will decompose to carnonyl compound and chromium in a lower oxidation state.**

C4H9CH(CH3)OH + HO- -> C4H9CH(CH3)O- + H2O

C4H9CH(CH3)O- + I-I -> C4H9CH(CH3)OI + I-

C4H9CH(CH3)OI + HO- -> C4H9C(CH3)= O + I- + H2O

**Reference:**

Carolyn Oconnell and David Dollimore. 1999. Determination of melting point using derivative. Instrumentation science and technology. Volume 27 Issue 1. pp. 13-21

**Experiment 7**

Identifying and classifying organic functional groups: alcohols, ethers, aldehyde and ketones