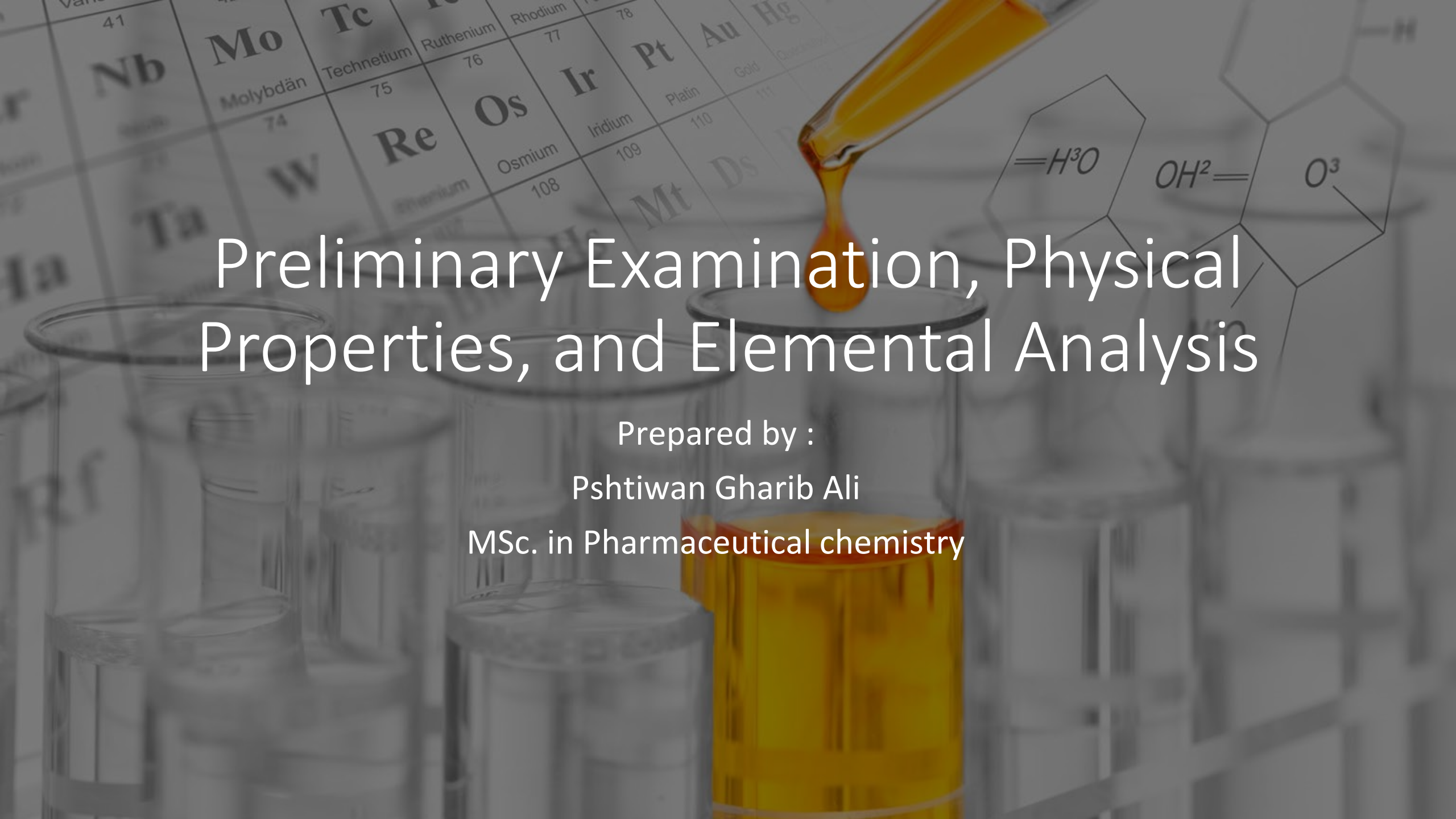

Preliminary Examination, Physical Properties, and Elemental Analysis

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MSc. in Pharmaceutical chemistry

PHYSICAL STATE

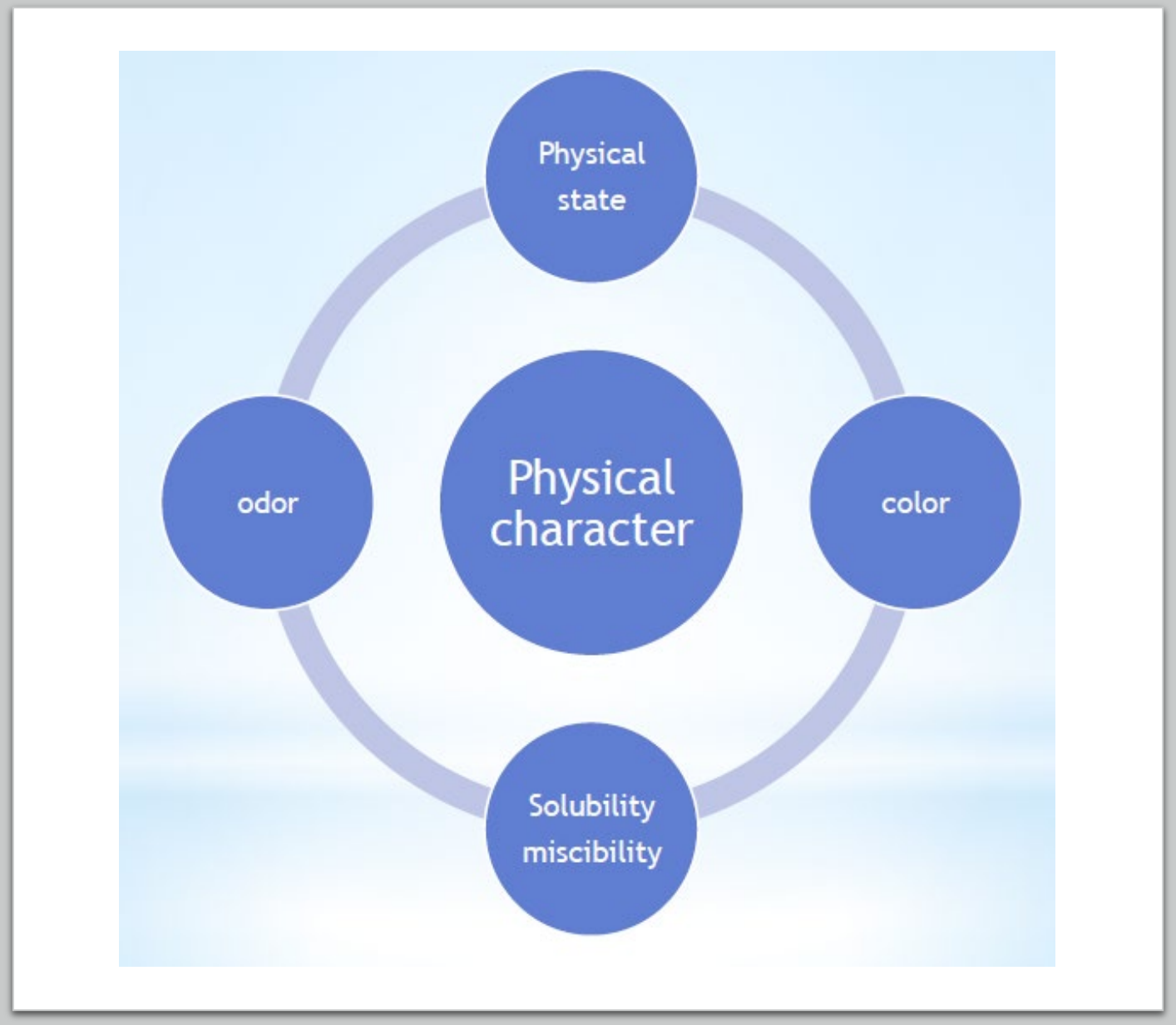
COLOR

ODOR

IGNITION TEST

Acid or Base character

ELEMENTAL ANALYSIS



PHYSICAL STATE

Among the 16 million that have been made, there are all kinds of molecules with amazingly varied properties. What do they look like?

1. *Solid:*

A- Crystalline it has a definite shape may be : *Needles*, *Prisms*, *Plates*,

Microcrystalline.

B- Powder may be *Fine* or *Coarse.*

C-Amorphous (has no definite shape)

2. Liquid:

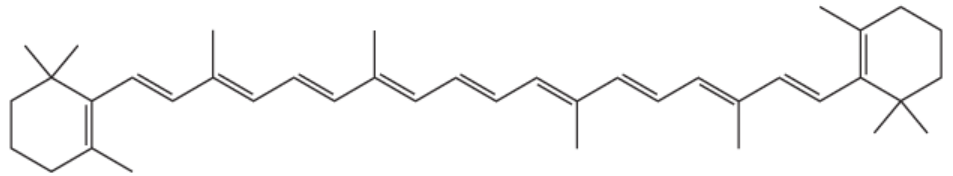
A- Mobile (such as : methanol, ethanol)

B- Viscous (such as lactic acid, glycerol).

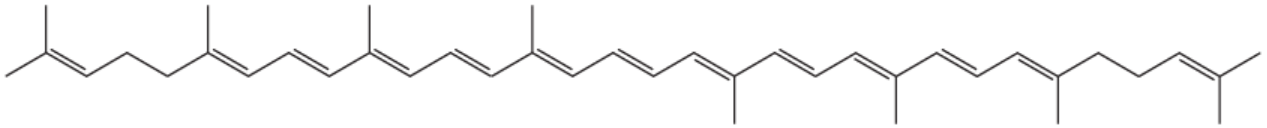
3. *Gas.*

Color

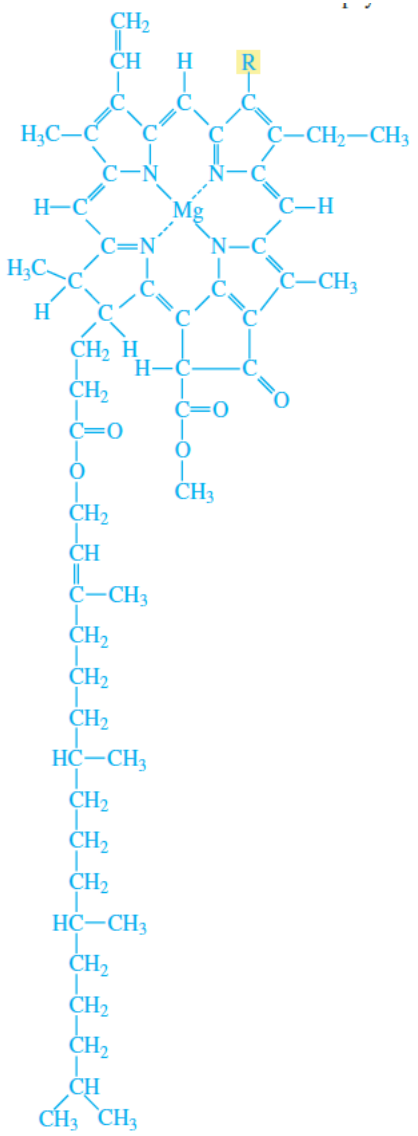
If a compound has enough conjugated double bonds, it will absorb visible light (light with wavelengths > 400 nm) and the compound will be colored. For example, beta-carotene, a precursor of vitamin A with a λmax = 455 nm, is an orange substance found in carrots, apricots, and the feathers of flamingos. Lycopene with a λmax = 474 nm—found in tomatoes, watermelon, and pink grapefruit—is red.



β-carotene
λ_{max} = 455 nm



lycopene
λ_{max} = 474 nm



Chlorophyll *a* and *b* are highly conjugated compounds that absorb visible light, causing green light to be reflected from the surface tissues of plants.

When a molecule absorbs light, all the non-absorbed (reflected) light combines to produce the complement of the absorbed color.

Wavelength Absorbed (nm)	Color Absorbed	Color Observed
400	Violet	Yellow-green
425	Blue-violet	Yellow
450	Blue	Orange
490	Blue-green	Red
510	Green	Purple
530	Yellow-green	Violet
550	Yellow	Blue-violet
590	Orange	Blue
640	Red	Blue-green
730	Purple	Green

Colour

Many liquids and solids are definitely colored because of the presence of chromophoric groups in the molecule. Many nitro compounds, quinones, azo compounds, stable carbocations and carbanions, and compounds with extended conjugated systems are colored.

If an unknown compound is a stable, colorless liquid or a white crystalline solid, this information is valuable because it excludes chromophoric functional groups as well as many groups that would become chromophores by oxidation.

Colour

The region of the molecule responsible for the absorption (the conjugated π system) is called the chromophore, while the groups attached to the chromophore are called auxochromes.

The color of some compounds is due to impurities; frequently these are produced by the slow oxidation of the compound by oxygen in the air. Aniline, for example, is usually reddish brown, but a freshly distilled sample is colorless.

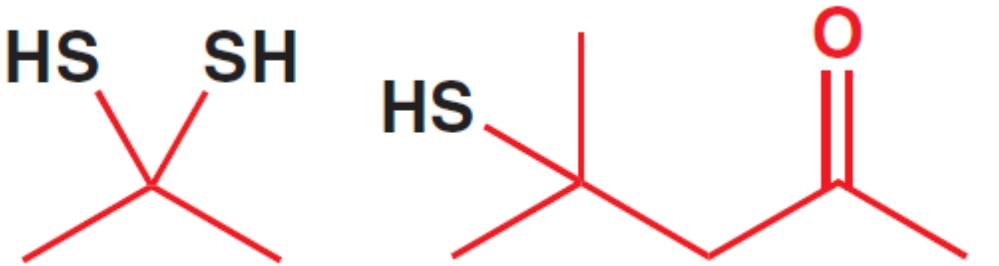
Some colourless solids and liquids

- Carbohydrates
- Simple phenols
- High M.W. esters
- Aldehyde
- Amide
- Aromatic acids, their amides
- Sulphonic acid
- Carboxylic acid
- Alcohols
- Hydrocarbons
- Low M.W ethers
- Simple amines
- Simple ketones
- Anilides

Some colored compounds

Compound	Color
p-Nitrotoluene	Lemon
o-Nitrophenol, iodoform	Yellow
o-Nitroaniline	Orange
m-Nitroaniline	Golden yellow
p-Nitroaniline	Pale yellow
o-Nitrobenzoic acid	Colorless
Resorcinol	Light pink
p-Cresol	Dark pink
m-Cresol	Dark pink
β-Naphthol	Chacolate
Sulfanilic acid	Grey
Aniline hydrochloride	Colorless but becomes black on keeping
Picric acid	Yellow
Azobenzene	Orange
p-Benzoquinone	Yellow

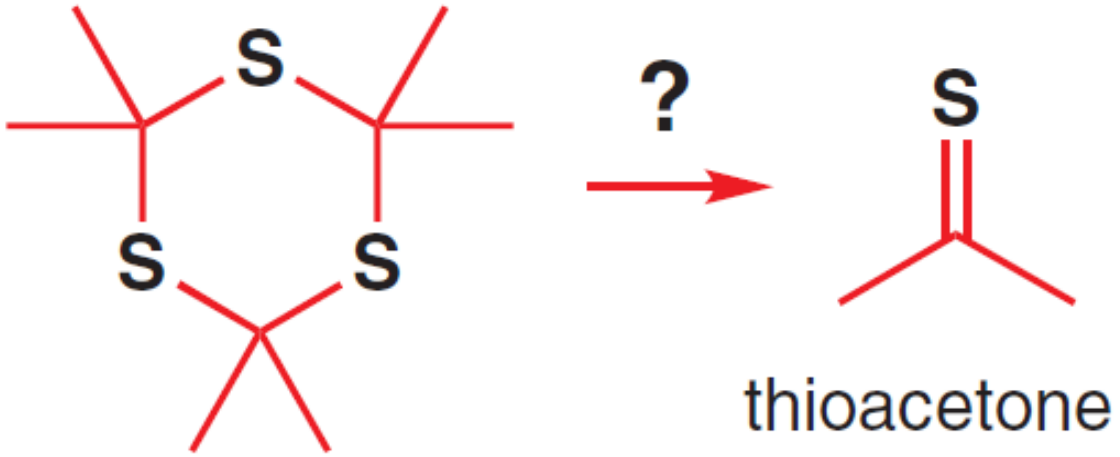
Odour



propane-dithiol

4-methyl-4-sulfanylpentan-2-one

two candidates for
the worst smell in the world
(no-one wants to find the winner)



thioacetone

Odor

Odour	Compounds
Of spirit like	Ethanol or methanol
Pleasant (fruity)	Acetone, ether
Of a vinegar	Acetic acid
Of bitter almond type	Benzaldehyde, nitrobenzene
Foul (very unpleasant)	Isocyanide
Pungent (Strong)	Aldehyde
Very pungent	Formic acid, acid halides, thioacids
Of mice	Acetamide
Of fish or resembling ammonia	Low aliphatic amines, benzylamine
Of aniline	Aromatic amine
Of garlic	Thioalcohols
Phenyl type	Naphthalene
Pleasant with a feeling of sweetness at the throat	Carbon tetrachloride, Chloroform
Of carbolic acid	Phenol

Ignition test

Heat the sample gently over a low flame, behind a safety shield. Heat the sample until ignition has occurred .

Note (1) the flammability and nature of the flame (is the compound explosive?); (2) whether the compound is a solid, whether it melts, and the manner of its melting; (3) the odor of the gases or vapors evolved *(caution!);* and (4) the residue left after ignition.

Ignition test

A small amount of the sample is placed on a spatula and burnt on a naked flame

Observation	Inference
Compound burns with a smoky flame (yellow, sooty flame)	Aromatic compounds, Highly unsaturated aliphatic and higher saturated compounds
Burns with a clear flame (yellow) but much less sooty	Saturated aliphatic compounds
Oxygen content of the compound increases the flame becomes more and more clear (blue)	Low MW. Of alcohols
Compounds chars with burnt sugar smell	Carbohydrates, tartarates, and some citrates
Ammonia gas is evolved	Nitrogenous compounds
Sublimes with burnt sugar smell	Oxamide
Compound chars without melting	Uric acid, sulphonic , starch
Some residue is left on heating for longer period	Metallic salts, bisulphite compounds of aldehydes and ketones.

Acid or Base character

A few milligrams of the compound are dissolved in a suitable solvent like water, ethanol or dioxane and tested with litmus paper.

A change in color from blue to red indicates that the substance is acidic and red to blue indicates a basic substance .

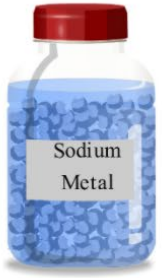
On the other hand, if there is no charge then the compound is neutral such as aldehydes ,ketones ,esters or hydrocarbons.

Elemental analysis

The elements usually detected are N,S and the halogens(F,Cl,Br,I) whereas C and H are always assumed to be present. The presence of oxygen as a part of functional group will become apparent in second course by Functional group tests.

For the detection of elements, the organic substance is first decomposed by fusion with sodium metal to prepare an extract, called the sodium fusion extract (Lassign’s extract).

Materials Required



Sodium metal

Distilled water

Fusion tube
(ignition tube)

Test tubes

Bunsen burner

Tongs

Small spatula

Wire gauze

China dish

Forceps

Funnel

Tripod stand

Filter paper

Organic compound

Glass rod

Procedure

1- Freshly cut a piece of sodium metal about a quarter size of a pea and dry in the folds of a filter paper (sodium is dangerous, therefore, handle it carefully). Use a sharp knife to cut sodium. Never throw scraps of sodium in water.

2- Place the sodium piece in an ignition tube held with a pair of tongs.

3- Heat the lower part of the tube on a flame until sodium melts.

4-Remove the tube from the flame and rapidly add a small amount (0.2 g of the solid or 4 drops of the liquid) of the sample directly over the melted sodium. Be careful not to allow the sample to touch the sides of the hot fusion tube during the addition Again heat the tube to redness, a brisk reaction is observed.

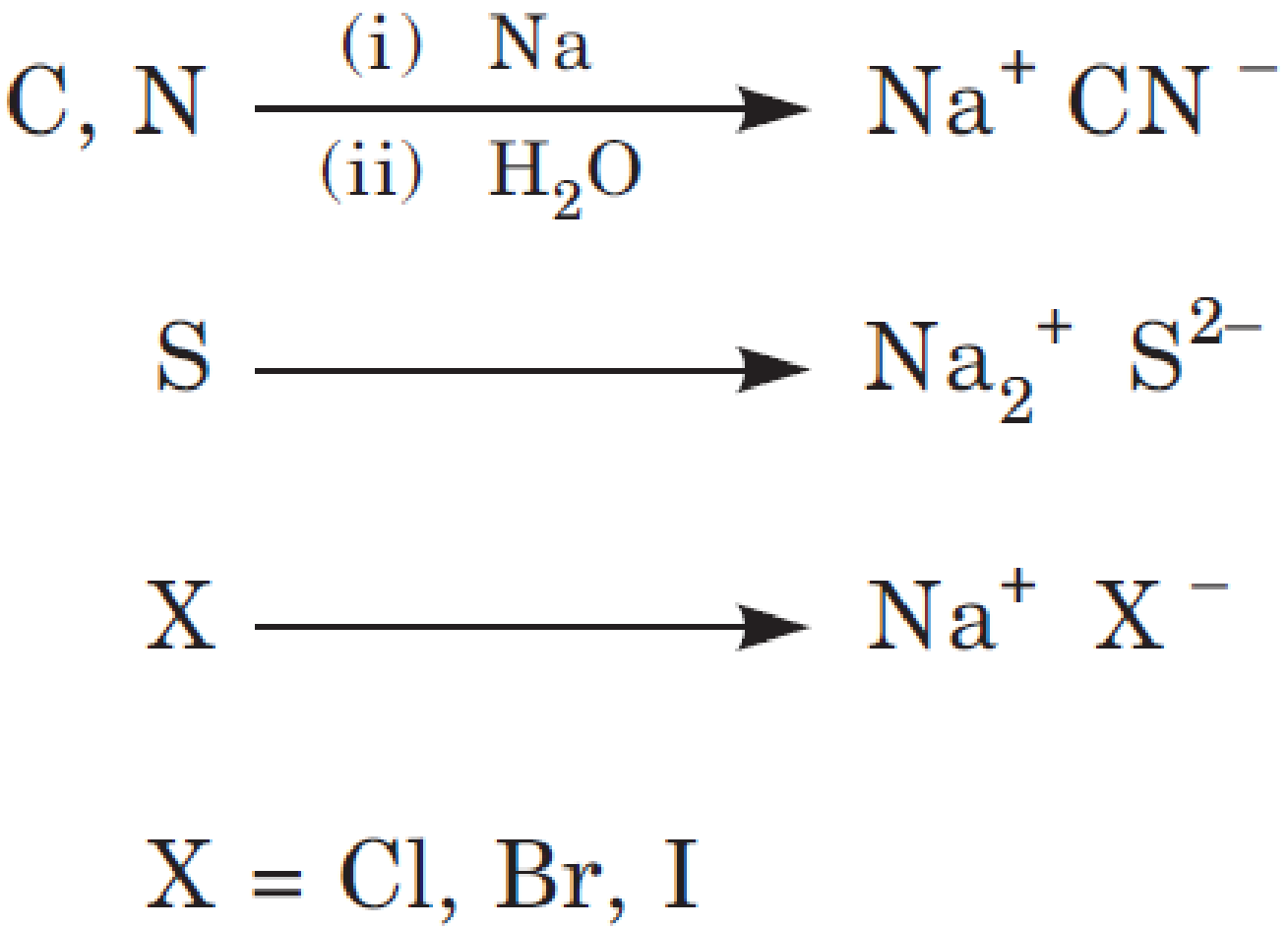
Procedure

5-Remove the tube from the flame, add another small portion of the unknown sample and heat the tube again till it is red hot.

6-Then immediately immerse the tube in 15 ml of distilled water taken in a beaker, and crush into small pieces with a glass rod or shake the covered beaker (covered with petridish).

7-This will hydrolyze sodium and dissolve the ions in water. Boil the mixture for 5 minutes and filter hot to remove the glass splinters. The alkaline filtrate so obtained is usually called the sodium fusion extract. This solution is used in subsequent tests for elemental analysis.

Chemical reaction



Test for Nitrogen

- Take 0.5 ml of sodium fusion extract in a test tube and add 2 drops of freshly prepared saturated ferrous sulfate solution. A green precipitate should be obtained. In case no such precipitate is obtained, add a few drops of sodium hydroxide solution.
- At this stage a green precipitate should appear. The green precipitate is due to the formation of Fe(OH)2 and not due to the presence of nitrogen. Ferrous hydroxide reacts with sodium cyanide to form sodium ferrocyanide. Boil the mixture for 10 sec. and acidify with dil. sulfuric acid, while shaking till a clear solution is obtained. A Prussian blue color indicates the presence of nitrogen.

Chemical reactions:

$$FeSO_4 + 2NaOH \longrightarrow Fe(OH)_2 + Na_2SO_4$$

$$Fe(OH)_2 + 6NaCN \longrightarrow \underset{\text{Sodium ferrocyanide}}{Na_4\left[Fe(CN)_6\right]} + 2NaSO_4$$

$$3Na_4\left[Fe(CN)_6\right] + 2\left[Fe_2(SO)_4\right]_3 \longrightarrow \underset{\text{Ferri-ferrocyanide}}{Fe_4\left[Fe(CN)_6\right]_3} + 6Na_2SO_4$$

Test for Halogen

Acidify 0.5 ml of the sodium fusion extract with dil. nitric acid and boil. Add several drops of silver nitrate solution. If halogens are present a flocculent white or yellow precipitate which darkens on exposure to light and is soluble in ammonium hydroxide indicates the presence of halogens.

The acidification of the sodium fusion extract is necessary before adding silver nitrate solution to prevent the precipitation of silver hydroxide or silver oxide.

Chemical reaction:

$$Na^+X^- + AgNO_3 \xrightarrow{\text{dil. } HNO_3} AgX + NaNO_3$$

Test for sulfur

In a test tube acidify 0.5 ml of sodium extract with dil. acetic acid. In acid any sulfide ion present is converted to hydrogen sulfide gas.

Add 1–2 drops of lead acetate solution (preferably saturated solution). The appearance of a black precipitate due to the formation of lead sulfide indicates sulfur.

$$Na_2S + 2CH_3COOH \longrightarrow H_2S + 2CH_3COONa$$

$$Pb(CH_3COO)_2 + H_2S \longrightarrow PbS + 2CH_3COOH$$

Lead acetate

(Black ppt)

Lead sulphide

✓ **Test for S:**

Acidify 0.5 mL of the test solution with dilute **Acetic Acid** and add a few drops of **Lead Acetate** solution. A black precipitate of **PbS** indicates presence of **S**

$$Na_2S + Pb(CH_3COO)_2 \rightarrow PbS + 2CH_3COONa$$

✓ **Test for N:**

To 0.5 mL of the test solution add few crystal of **ferrous ammonium sulphate** $(NH_4)_2Fe(SO_4)_2$. Heat to boiling and then add immediately 2-3 drops concentrated **sulphuric acid** to get **blue color** solution indicates presence of **N**.

$$Fe_4[Fe(CN)_6]_3$$

✓ **Test for Halogen:**

To 0.5 mL of the test solution, add dilute **nitric acid** HNO_3 to produce a distinct **acidity**. Boil gently to expel any HCN or H_2S **(fume hood)** that may be present. Add **AgNO3** solution to get ppt indicate of halogens.

AgCl white ppt, AgBr pale yellow ppt, AgI yellow ppt

SAFETY ALERT

1. Sodium fusion involves heating sodium metal or a sodium-lead alloy to a high temperature and then adding the organic compound. Use extreme care when performing both the heating and addition.
2. Perform the sodium fusion in the hood if possible.
3. Use a Pyrex™ test tube and check it for cracks or other imperfections before performing the sodium fusion.
4. If pure sodium metal is used, hydrolyze the residue very carefully as directed, because any excess metal reacts *vigorously* with alcohol or water.
5. Be careful when handling the test tube after the fusion is complete; remember that it may still be hot.
6. *Throughout this procedure, point the mouth of the test tube away from yourself and your neighbors;* the organic material may burst into flame when it contacts the hot metal, or it may react so violently that hot materials are splattered from the test tube.

Thank you