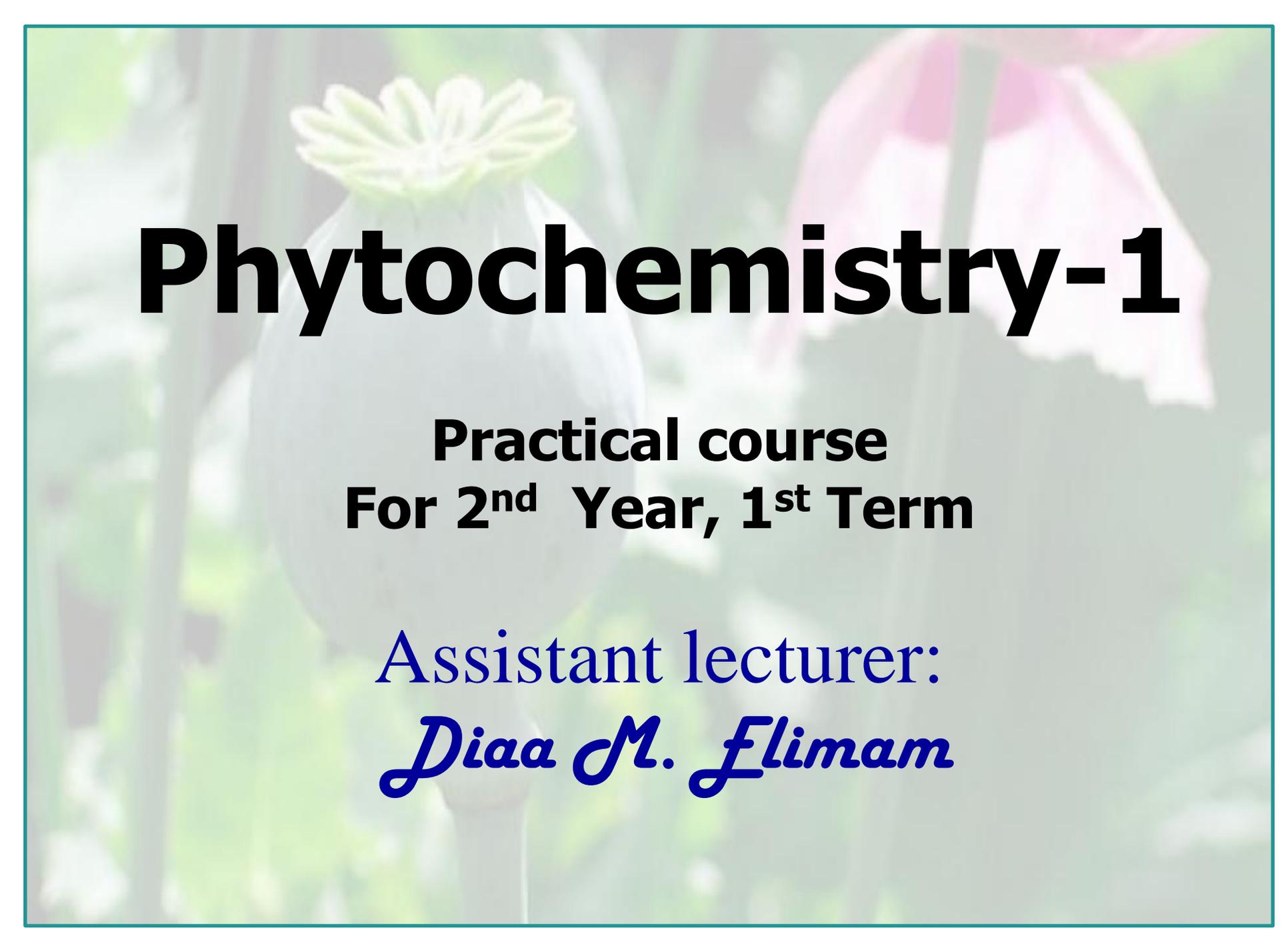


بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ

The background of the slide features a soft-focus photograph of several flowers. In the foreground, a light-colored flower with many small petals is visible. To the right, a pink flower is partially seen. The overall background is a mix of green and light colors, suggesting a garden or field setting.

# Phytochemistry-1

**Practical course  
For 2<sup>nd</sup> Year, 1<sup>st</sup> Term**

Assistant lecturer:

*Diaa M. Flimam*

# In this course you will study:

## 1- Alkaloids

Date	Subject
26/9	Orientation / Introduction (alkaloids)
3/10	(Quinine, Quindine, Caffeine, Theophylline).
10/10	(dil.Ephedrine, Papverine, Emetine, Eserine).
17/10	(Ergot, Atropine, Pilocarpine).
24/10	(Brucine, Strychnine, Colchicine).
31/10	(General scheme for identification of unknown alkaloid).
7/11	Midterm

## 2- Volatile Oil

Date	Subject
14/11	Introduction / Preparation of volatile oils / Determination of Phenolics (Eugenol in clove oil)
21/11	Determination of oxides (Cineol in Eucalyptus) & peroxides (Ascaridol in Chenopodium).
28/11	Determination of Cinnamaldehyde in cinnamon oil
5/12	Quantitative estimation of <u>N</u> & <u>S</u> volatile constituents (allyl-isothiocyanate in mustard oil).
12/12	Final Practical Exam + Sheet Exam

# **Alkaloids**

## **SEC-1**

**(Introduction) - (17 slides)**

# Alkaloids

```
graph TD; A[Alkaloids] --> B[Introduction]; B --> C[General Physical Properties]; C --> D[General Chemical Properties]; D --> E[Action of Acids & Alkalis];
```

**Introduction**

**General Physical Properties**

**General Chemical Properties**

**Action of Acids & Alkalis**

# The term alkaloid = alkali-like

Slide  
2/17

## Definition:

They are **basic, nitrogenous** compounds of **plant** origin, having **complex** molecular structures and marked **pharmacological activities** on man or animal.

## Note:

Many widely distributed bases of plant origin **are not classified** as alkaloid but they are classified as biological-amines or proto-alkaloids such as

\* **Methyl-amine**

\* **Trimethyl-amine**

\* **Cholines**

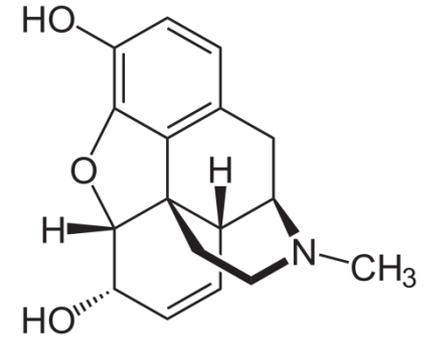
\* **Betaines**

## Example of typical alkaloids:

### Morphine

Basic, N atom is in heterocyclic ring = complex,  
From plant origin & has marked pharmacological  
activity.

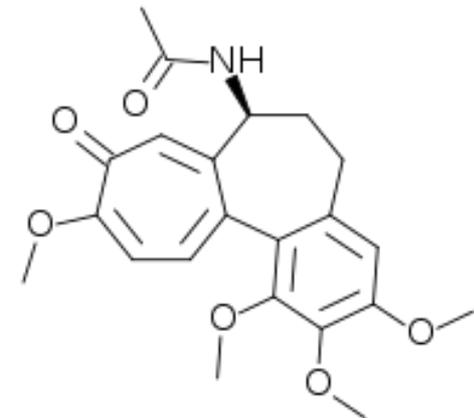
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## Exception for alkaloids:

### Colchicine

It is not basic, and its nitrogen atom is not  
incorporated in a heterocyclic ring but classified as  
alkaloid, because of its pharmacological activity &  
limited distribution in the plant world.



# General Physical Properties

Slide  
4/17

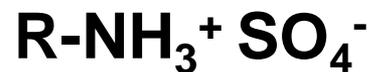
Alkaloids may occur  
either as

**Free base**



Soluble in organic solvents

**Salt with acid**



Salt = Soluble in water

# Alkaloid

## Alkaloid base

Soluble in organic solvents

Insol. or sparingly soluble in water

### Exceptions

Bases insoluble or Sparingly soluble in org. solvent (Ether)

Bases soluble in water

**Morphine** (1:5000)  
**Pilocarpine** (insol.)  
**Narceine** (insol.)

**Caffeine, Pilocarpine.**  
**Colchicine** (sol. In acidic, neutral or alkaline water)  
**Berberine** (4<sup>ry</sup> alkaloid-bases)

## Alkaloid salt

Insol. or sparingly soluble in org. solvents, Less sol. in alcohol

Soluble in water

### Exceptions

Salts dissolve in org. solvents (chloroform)

Salts less or sparingly soluble in water

**Lobeline-HCl**  
**Strychnine-NO<sub>3</sub>**

**Quinine-SO<sub>4</sub>**  
**(1:1000)**

# General Chemical Properties

```
graph TD; A[General Chemical Properties] --- B[A. Precipitation by certain reagents]; A --- C[B. Color reactions with certain reagents]
```

**A. Precipitation by  
certain reagents**

**B. Color reactions  
with certain reagents**

## A. Precipitation by certain reagents

Slide  
7/17

Many alkaloids in small quantities may form ppt or turbidity with certain reagents.

**Negative response, (i.e. no precipitate or turbidity):**

**= Absence of alkaloids.**

**Positive response, (i.e. ppt or turbidity):**

**= May or may not be due to alkaloid, Need confirmation.**

**E.g. Protein, ammonium salts may give Positive response to these reagents**

# Common precipitating reagent, 9 reagents

Slide  
8/17

## 1-Mayer,s reagent:

**Potassium Mercuric Iodide** solution ( $\text{KHgI}_3$ ) in acidic pH.

Precipitate all alkaloids **except:**

- Purine base alkaloids (Caffeine, Theophylline, Theobromine).
- Colchicine.
- Methyl-ergometrine.
- dil. Ephedrine. (Note: Conc. Ephedrine produces ppt).

Some alkaloids need care (drops of reagent), as excess reagent may dissolve ppt (gives false negative)

- Atropine
- Pilocarpine

**2-Wagner,s reagent:**

Potassium tri-Iodide, ( $KI/I_2 = KI_3$ ).

**3-Kraut,s reagent:**

Potassium. Bismuth Iodide, ( $KBiI_4$ ) (modified Dragendroff,s reagent).

**4-Marme,s reagent:**

Potassium Cadmium Iodide ( $KCdI_3$ ).

**5-Hager,s reagent:**

Saturated solution of picric acid.

**6-Mercuric chloride**

1%  $HgCl_2$  / (dps of HCl).

**7-Gold chloride**

1%  $AuCl_2$  / (dps of HCl).

**8-Platinic chloride**

1%  $PtCl_2$  / (dps of HCl).

**9-Tannic acid solution.**

Most alkaloids ppt by tannin, neutral solution is recommended.

# Mechanism for precipitation of alkaloids

Slide  
10/17

1-Through formation of **insoluble complex additive** compounds:

**(Mayer's, Marme's & Kraut's reagent).**

2-Through **halogenations** in which **high molecular weight** compounds of **low solubility** are formed, giving ppt:

**(Wagner,s reagent).**

3- Through formation of **salts that precipitate** from their solution:

**(Picric acid = Hagars reagent).**

4- Through combination with **high molecular weight oxygenated** acids

(formation of **insoluble salts in oxygenated form**)

# Microcrystal test.....?

## Principle:

**Specific** alkaloid + **specific** reagent = **Specific** crystals  
→ (tested under microscope).

## Why do Crystals form ?

- \* Alkaloids form **double salts** with compounds of **Mercury, Gold, Platinum** and other **heavy metals** in reagents.
- \* The double salts are usually obtained as **precipitate** and many of them are **micro-crystallographically** characteristic.

## B. Color reactions with certain reagents

Color reagents are **very sensitive**

They depend on dehydration or oxidation of the alkaloids to produce characteristic color.

Most of these reagents consist of:

**C.H<sub>2</sub>SO<sub>4</sub>** + compounds such as **Selenic acid,**  
**Formaldehyde ...etc.**

# Common coloring reagents

Slide  
13/17

## 1-Froehd's reagent

Sodium Molybdate + C.  $\text{H}_2\text{SO}_4$  = (Sulfo-molybdic acid)

## 2-Marqui's reagent:

Formaldehyde + C.  $\text{H}_2\text{SO}_4$

## 3-Mandalin's reagent:

Amm. Vanadate + C.  $\text{H}_2\text{SO}_4$  = (Sulfovanadic acid)

## 4-Erdman's reagent:

C. Nitric acid + C.  $\text{H}_2\text{SO}_4$

## 5-Meck's reagent:

Selenious acid + C.  $\text{H}_2\text{SO}_4$

## 6-Van Urk's reagent (Ehrlich reagent):

P-Di-methyl-amino-bezaldehyde (DMAB) + C.  $H_2SO_4$  + traces of  $FeCl_3$

## 7- Dragendorff's reagent:

Potassium Bismuth Iodide, (widely used for spraying paper chromatogram of many alkaloids).

## 8-Conc nitric acid

C.  $HNO_3$

## 9-Conc sulfuric acid

C.  $H_2SO_4$

# Under standardized conditions:

The intensity of the Color is in linear proportion to the Conc. → May be used in quantitative determination of that group of alkaloids.

e.g.

1-The blue color formed by Ergot alkaloids with Van Urk's reagent.

2-The violet color formed by Atropine in vitali's test.

**Conc. acids → It give color with alkaloids as a result of:**

**1-Dehydration: to give anhydro or apo-alkaloid.**

Production of apo-morphine from morphine and apo-atropine from atropine

**2-Hydrolysis:**

For ester alkaloids such as cocaine and atropine

Atropine + H<sub>2</sub>O + H<sup>+</sup> → Tropine + Tropic acid

Cocaine + H<sub>2</sub>O + H<sup>+</sup> → Ecgonine + benzoic acid + methyl alcohol

**3-Elimination of methoxy groups:**

Leaving a residue of hydroxyl base.

Codeine, Quinine, and papaverine.

**dil. acids → Formation of Alkaloid salt**

**For preparation of alkaloid salts: cold dilute acid solutions are used.**

## Action of alkalis

**1- Cold dil. alkali + alkaloid salt →  
free alkaloid base.**

**2- Hot dil. alkali + alkaloid salt →  
hydrolysis of ester alkaloid.**

**3- Heating with dry alkali (very Conc) + alkaloidal salt →  
decomposition & production of simple bases  
such as pyridine.**

# NEEDED TOOLS

- 1) White lab Coat.
- 2) Beaker 250 ml.
- 3) Test tubes.
- 4) Rack for test tubes.
- 5) Test tubes holder
- 6) Porcelain dish.
- 7) Plastic droppers.
- 8) Lab goggles (نظارة واقية).
- 9) Lab towel (فوطة المعمل).
- 10) Lab guide (شرح العملي).



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3, 4



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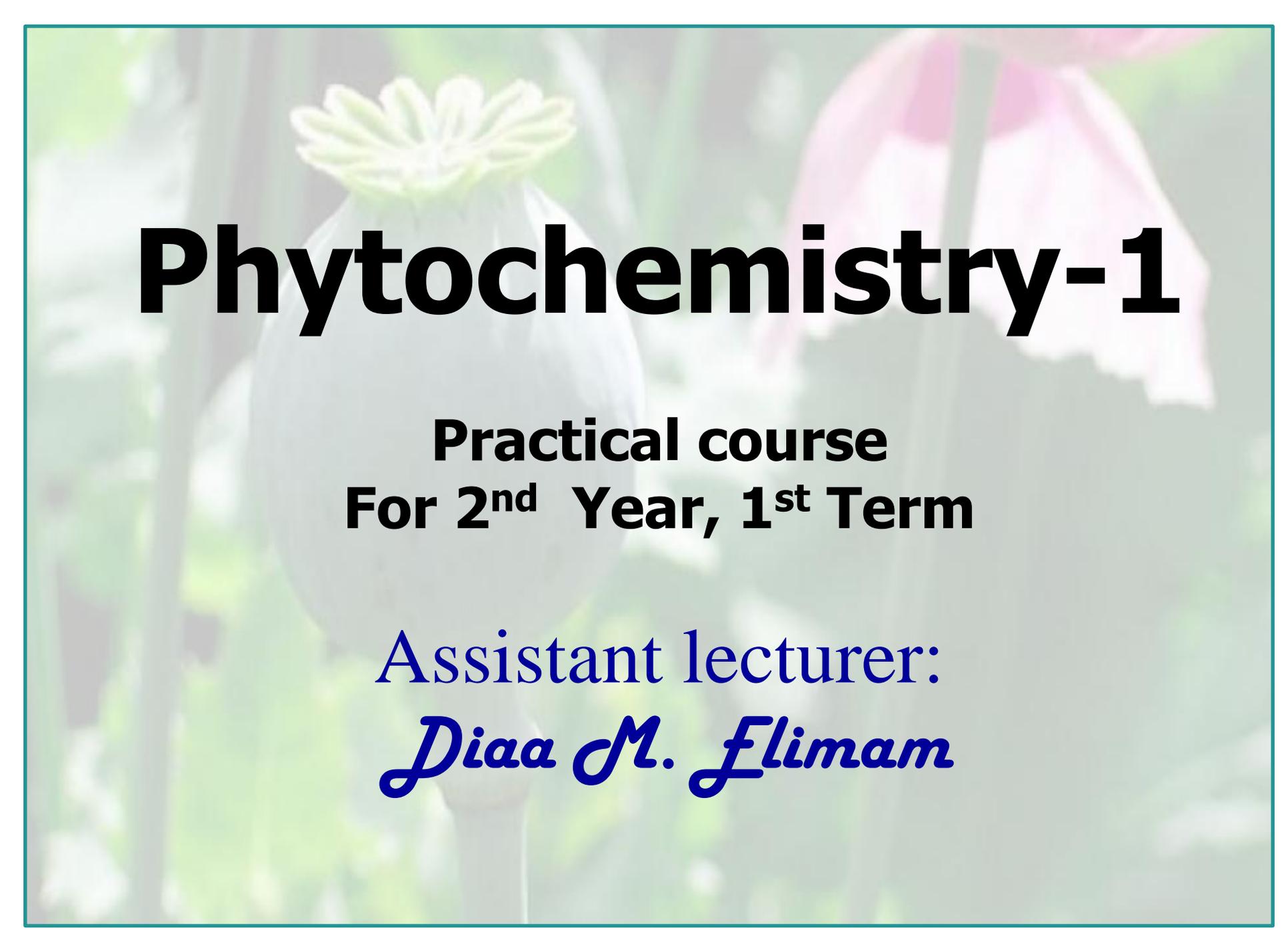


9



Thank you

بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ

The background of the slide features a soft-focus photograph of several flowers. On the left, a light-colored flower with many small petals is visible. On the right, a pink flower with a white center is partially shown. The overall background is a mix of green and light colors, suggesting a garden or field setting.

# Phytochemistry-1

**Practical course  
For 2<sup>nd</sup> Year, 1<sup>st</sup> Term**

Assistant lecturer:

*Diaa M. Flimam*

# **Alkaloids**

## **SEC-2**

**(Quin, Quinid, Caff, Theo) - (11 slides)**

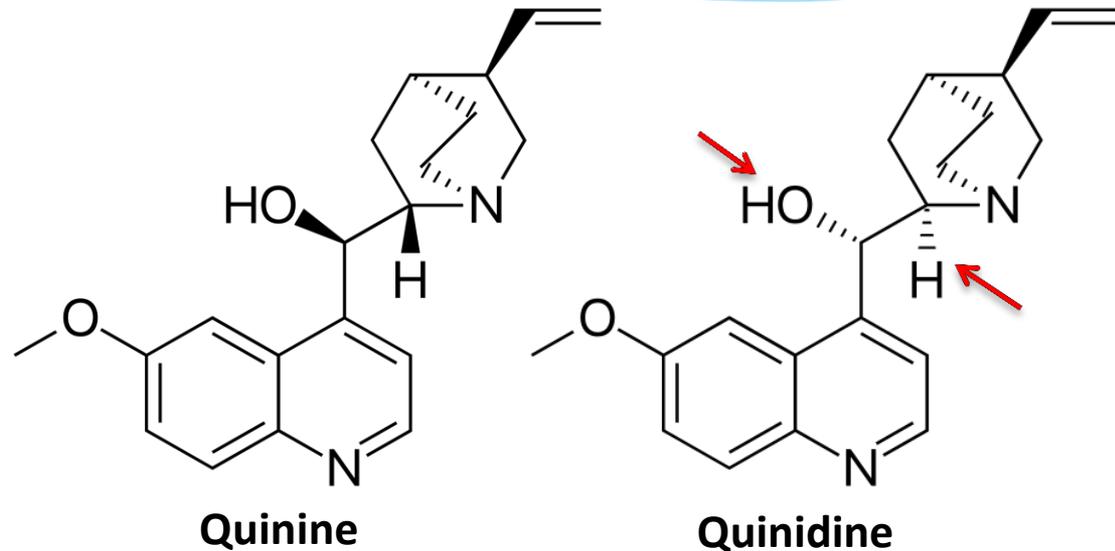
# Qualitative estimation of alkaloids

**Quinine / Quinidine**

**Caffeine / Theophylline**

# Quinine / Quinidine

- \* Nucleus ?
- \* Natural source ?
- \* Pharmacological effects?
- \* Uses / abuses ?



Quinoline nucleus

Cinchona bark

**Quinine : Anti-malarial**

**Quinidine : Anti-arrhythmic**

# Quinine & Quinidine

3

## 1. Mayer's test:

**1ml Alkaloid + 1-2 dps. Mayer's reagent (or maximum till eq. vol)**

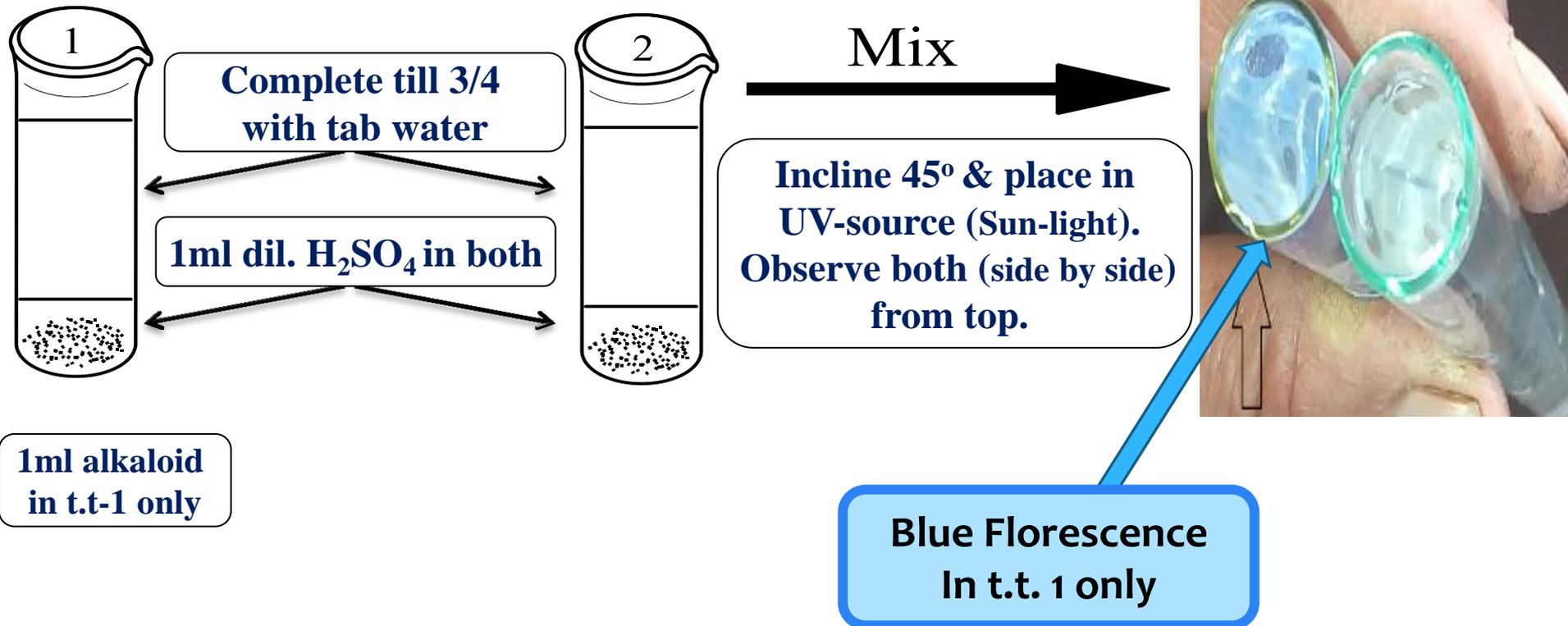
**→ +Ve (Turbidity, or White to yellowish-white ppt).**



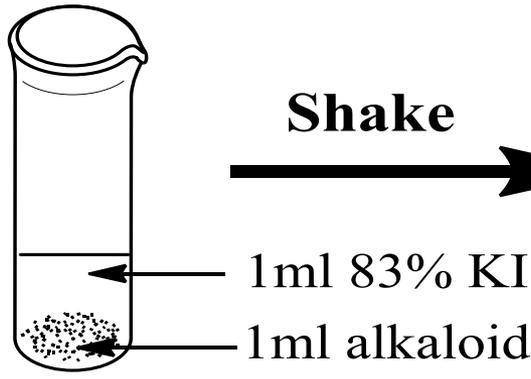
# Quinine & Quinidine

4

## 2. $H_2SO_4$ test = Florescence test



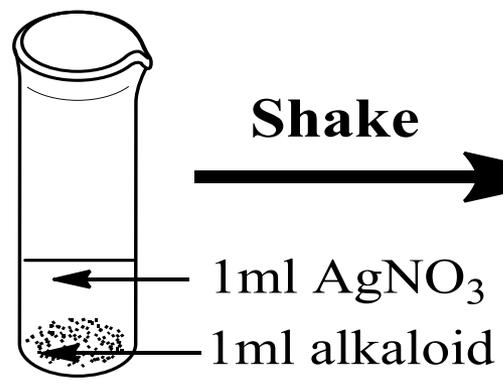
### 3. 83% KI test:



Quinine = -ve

Quinidine = +ve  
(white ppt)

### 4. AgNO<sub>3</sub> test:



Quinine = -ve

Quinidine = +ve  
(white ppt)

It is not used as:  
**"Quinine-HCl"**  
will give a white ppt, which is due to the Cl<sup>-</sup> anion (AgCl = false positive).

## 5. Thalleioquine test:

6

**Mix**

0.5 ml dil  $\text{NH}_4\text{OH}$   
3 drops  $\text{Br}_2$  water  
2 ml alkaloid

**+CHCl<sub>3</sub>**

**Shake**

Emerald green color

Emerald green color in  $\text{CHCl}_3$  layer

## 6. Rosequine = Erythroquine test:

**Mix**

K-ferrocyanide  
dp dil.  $\text{NH}_4\text{OH}$   
 $\text{Br}_2$ -water (dropwise till yellow sol).  
2 ml alkaloid

**+CHCl<sub>3</sub>**

**Shake**

Red color

Red color in chloroform layer

<b>Test</b>	<b>Quinine</b>	<b>Quinidine</b>
<b>1. Mayer's</b>	Yellowish white ppt.	
<b>2. H<sub>2</sub>SO<sub>4</sub> test: Florescence test</b>	Blue Florescence	
<b>3. Thalleiquine</b>	Emerald Green color	
<b>4. Rosequine = Erythroquine</b>	Rose red color	
<b>5. 83% KI test</b>	- ve	White ppt. Sol. in excess KI
<b>6. AgNO<sub>3</sub> test</b>	- ve (may give false +ve if Quinine-HCl is used)	white ppt.

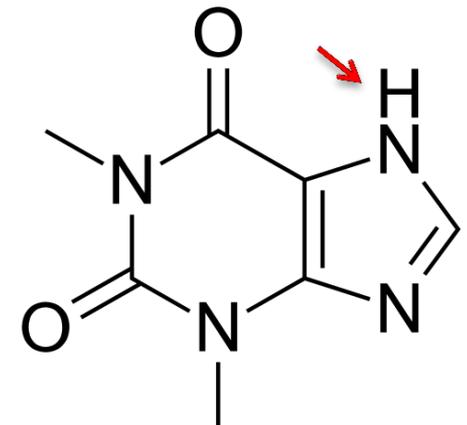
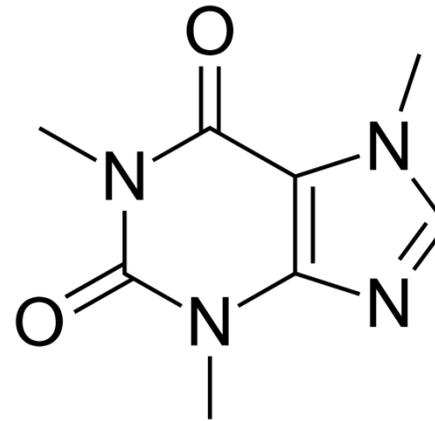
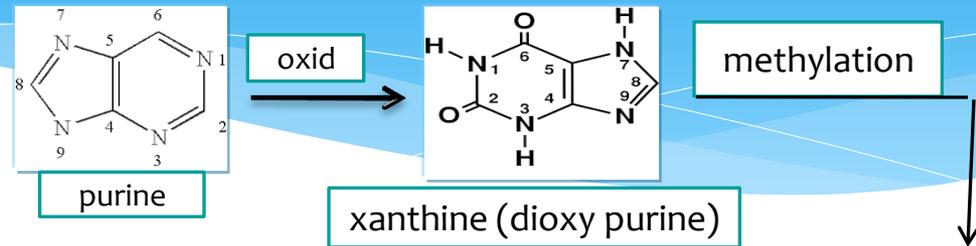
# Caffeine / Theophylline

\* Nucleus ?

\* Natural source ?

\* Pharmacological effects?

\* Uses / abuses ?



Xanthine nucleus

Camellia sinensis, Coffea arabica

Caff: CNS stimulant

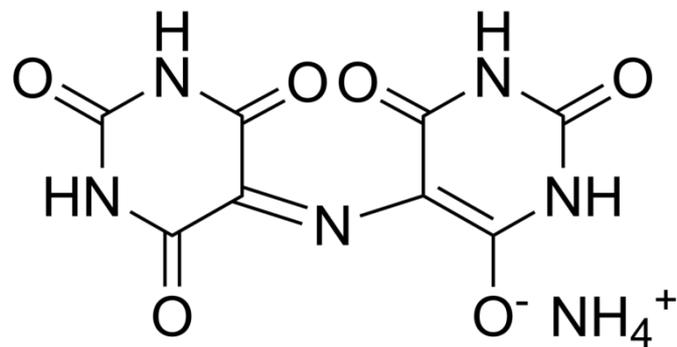
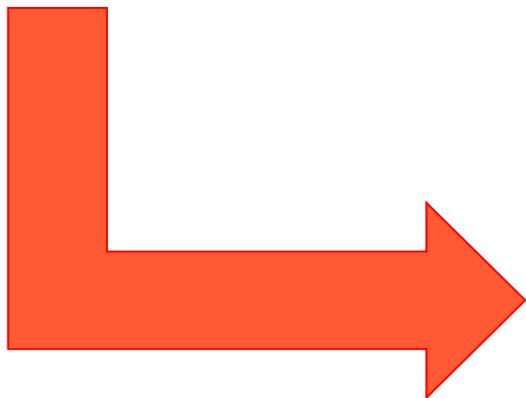
Theo: Bronchodilator

# Caffeine & Theophylline

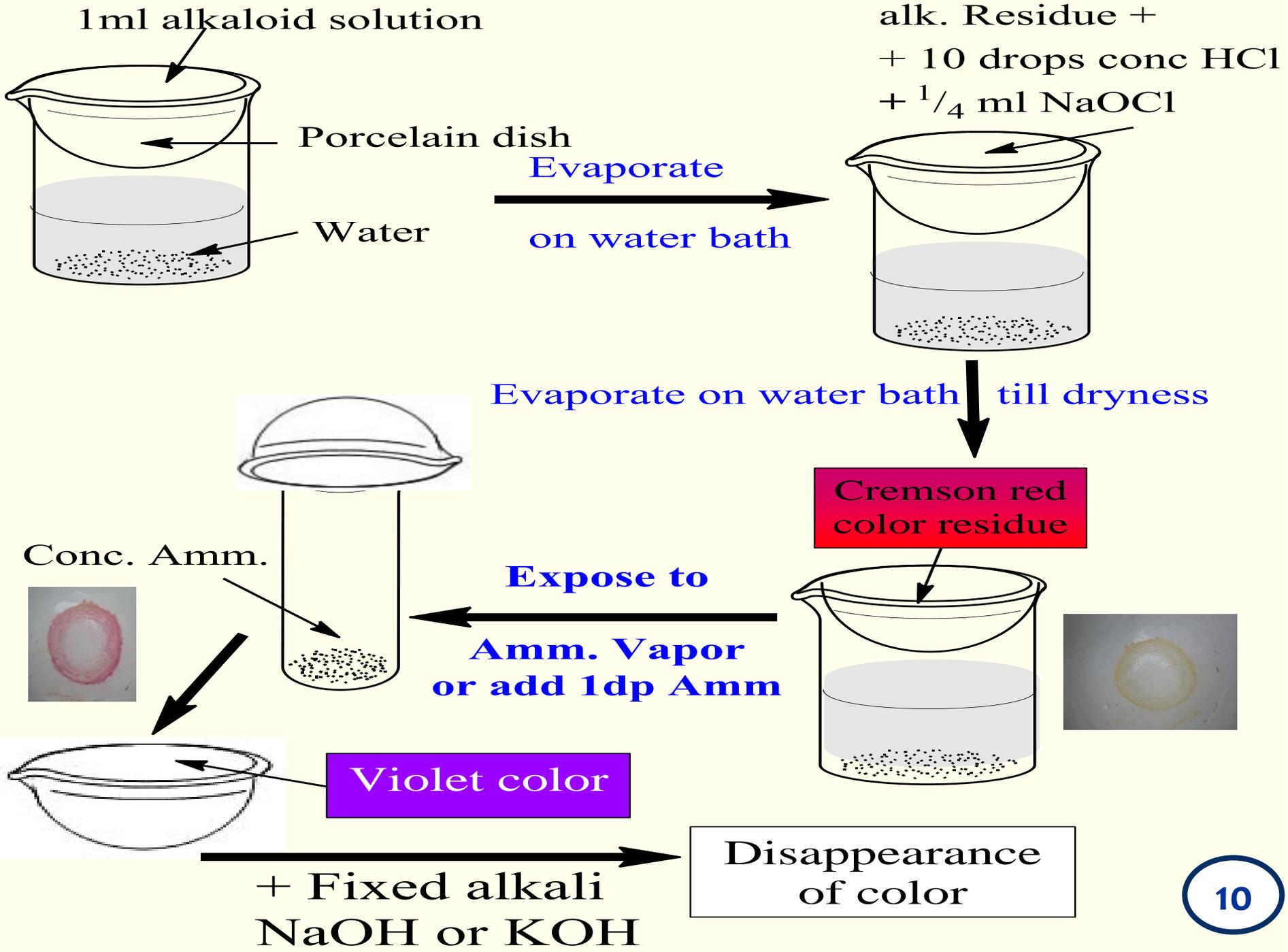
1. Mayer's test:

**-ve: No ppt (Clear transparent solution).**

2. Murexide test:



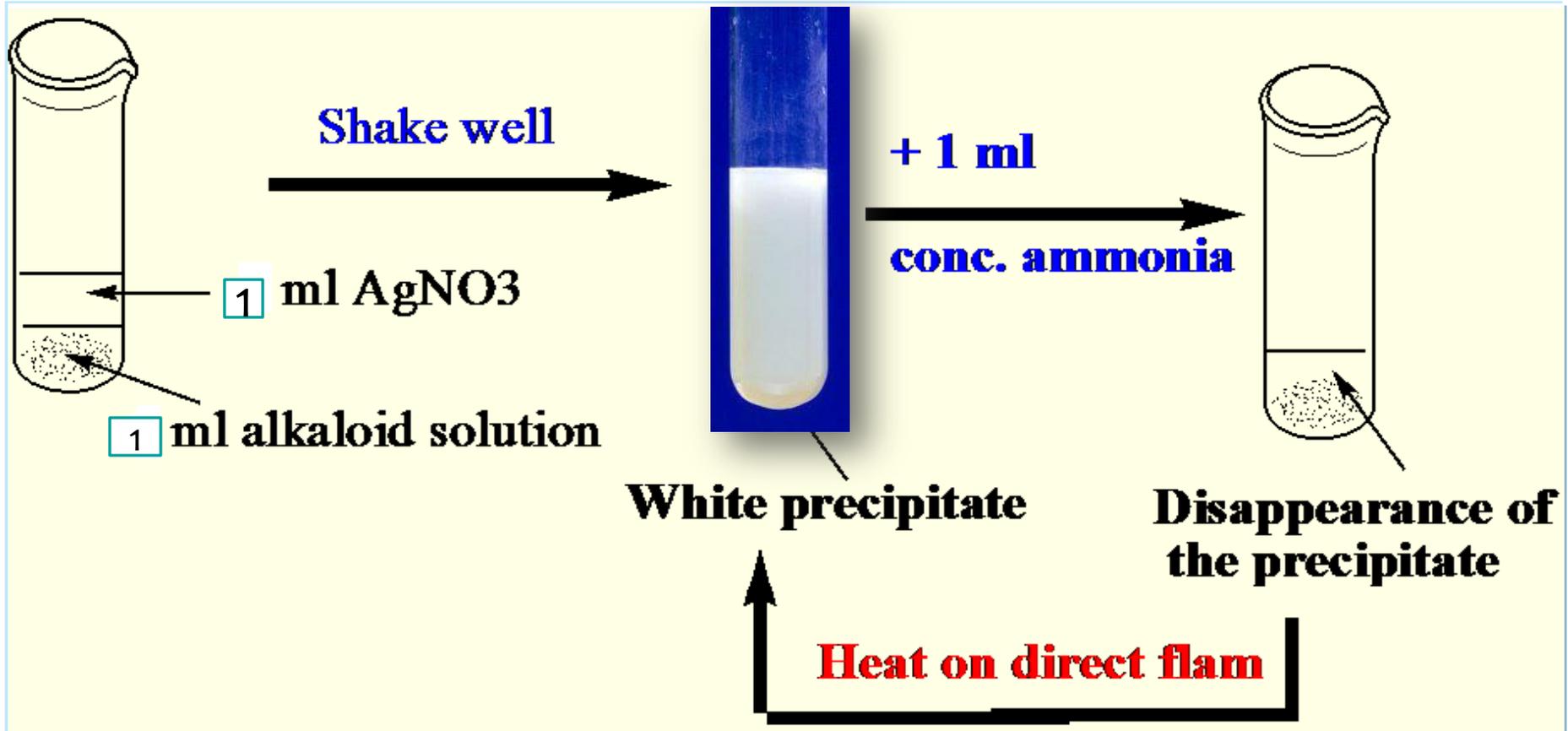
**Murexide**  
**(Amm. pupurate)**



# For Theophylline only

11

**AgNO<sub>3</sub> test: Specific test for Theophylline**



# NEEDED TOOLS

- 1) White lab Coat.
- 2) Beaker 250 ml.
- 3) Test tubes.
- 4) Rack for test tubes.
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3, 4



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That's all for today ...

**Thanks**