

Practical Organic Chemistry

Methods of Purification

- 1) Sublimation 2
- 2) Crystallisation 2
- 3) Differential extraction 2
- 4) a) Distillation 3
- b) Fractional distillation -
- c) Under reduced pressure -
- d) Steam distillation -
- 5) Chromatography -
 - i) adsorption -
 - a) Column 4
 - b) Thin layer 5
 - ii) Partition -
 - a) Paper 6

Qualitative Analysis

- 1) Detection of C, H 7
- 2) Lassaigne's test 8
 - a) Test for N 8
 - b) Test for S 9
 - c) Test for X 10
 - d) Test for P 11

Quantitative analysis

- 1) C & H 11
- 2) Nitrogen
 - a) dumas method 12
 - b) Kjeldahl's method 13
- 3) Halogens
 - *) Carius method 15
- 4) Sulphur 16
- 5) Phosphorus 16

Methods of Purification

1) Sublimation :

- * Separates sublimable compounds
- * directly Solid to Vapour.

2) Crystallisation :

- * Mostly used for purification of Solid Organic Compounds.
- * Based on diff in Solubilities of Compounds & impurities.

3) Differential extraction :

- * Organic Comp in aq medium is separated by shaking it with an organic solvent.
- * They must be immiscible so that they form two distinct layers.
- * Later removed by evaporation / distillation

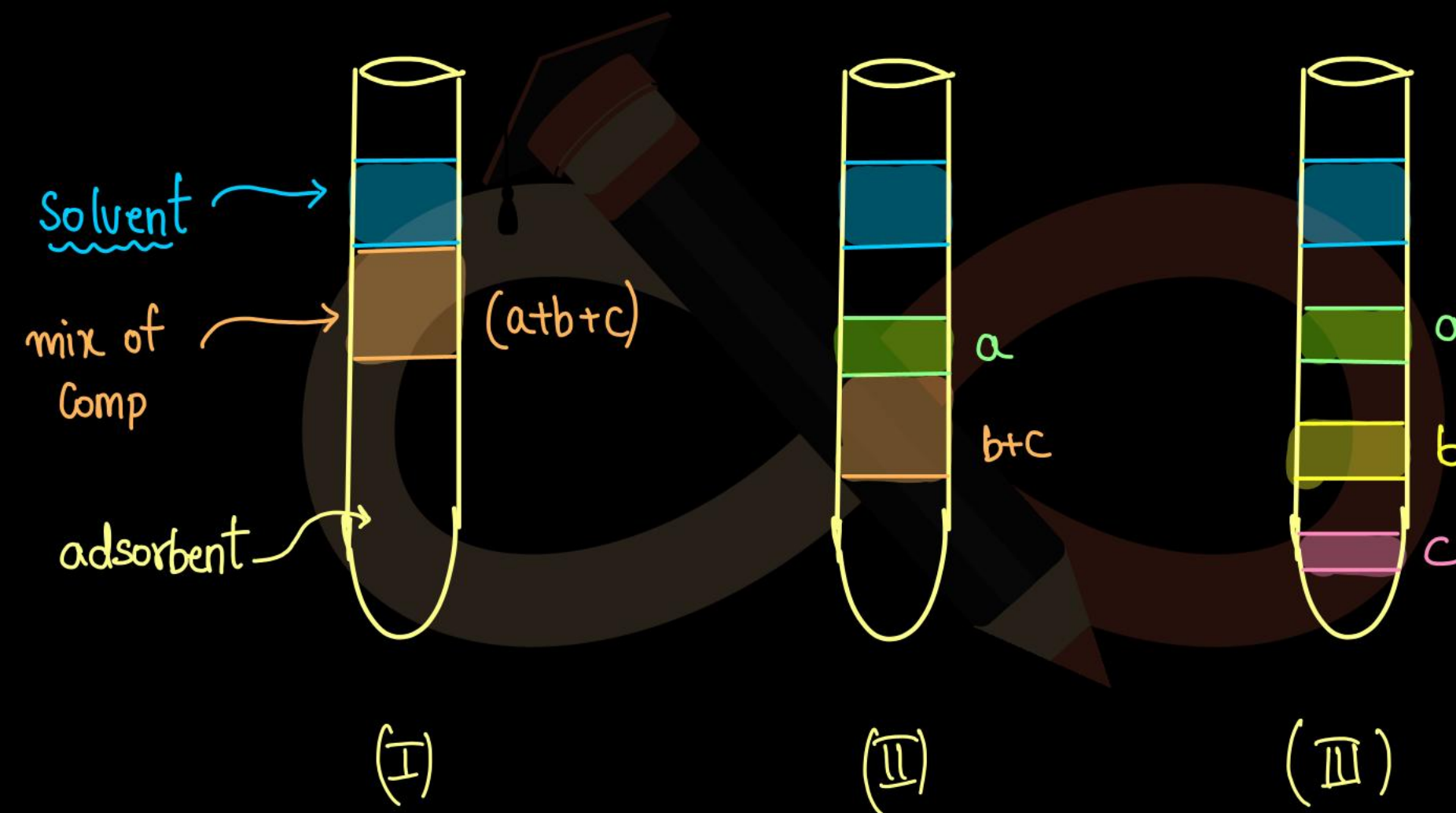
4) Distillation volatile liq from non-volatile impurities

a) <u>Distillation</u> :-	* Used to separate i) liq's having suff diff in their BP.	Chloroform & Aniline (334 K) (457 K)
b) <u>Fractional distillation</u> :	* Used when diff in T_b is not much. * fractional columns (theoretical plates) are used	Crude oil in petroleum industry
c) <u>Distillation under reduced pressure</u>	* Used to purify liquids having very High BP.	Glycerol from spent-lye in Soap industry
d) <u>Steam distillation</u>	* Used to separate subs which are steam volatile & immiscible with water	Aniline from aniline-water mixture.

5) chromatography :-

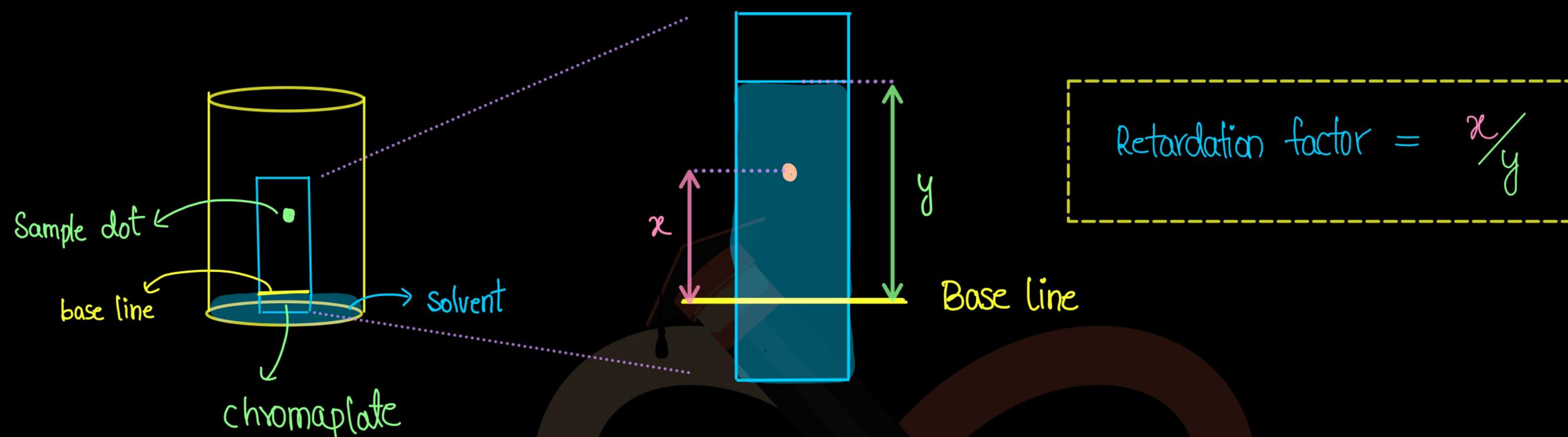
i) Adsorption chromatography :- *) Commonly used adsorbents are Silica gel & Alumina

a) Column chromatography :-



Different stages of Separation

b) Thin Layer chromatography (TLC) :



* If the spot is not visible, appropriate reagent can be sprayed

Ex:- amino acids may be detected by spraying ninhydrin solution

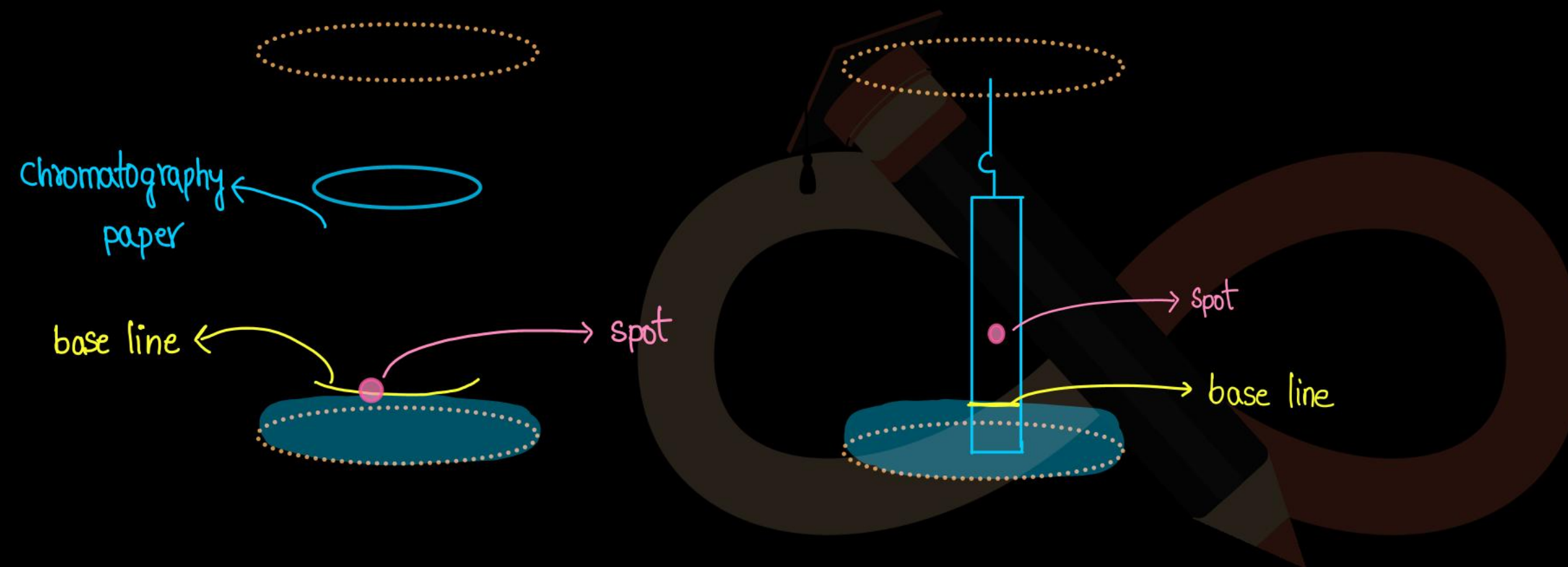
ii) Place the plate in jar containing Iodine crystals.

* They can also be spotted under UV.

ii) Partition chromatography

* Based on cont. differential partitioning of comp.

* Paper chromatography:

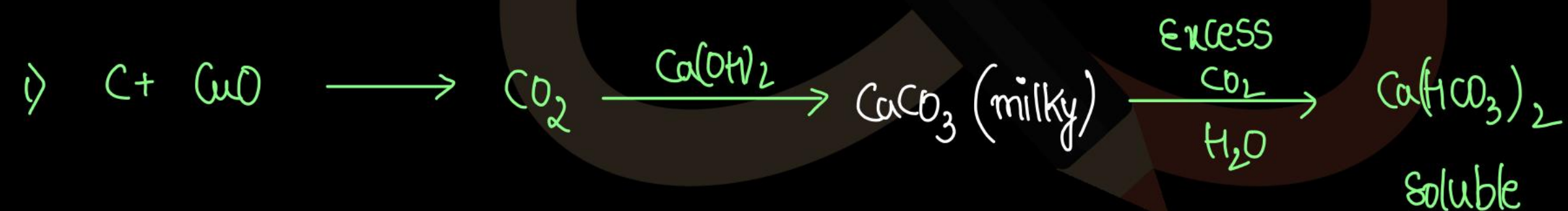
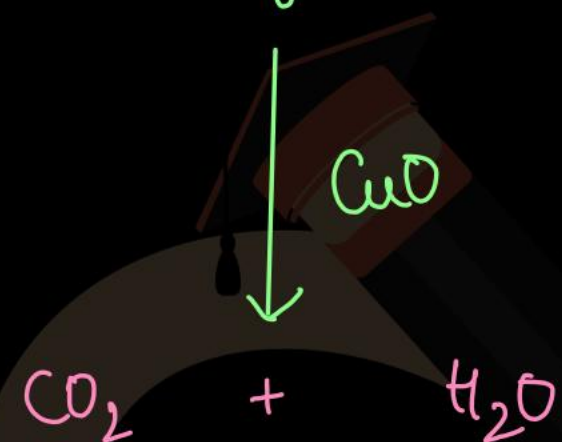


* Paper strip $\rightarrow$ chromatogram.

Qualitative Analysis :-

1) Liebig's method

O.C containing H & C



2) Lassaigne's test : (N, S, P, X)

* The elements present in the compound are converted from covalent to ionic by fusion with Na



Extracted
by boiling with
distilled water

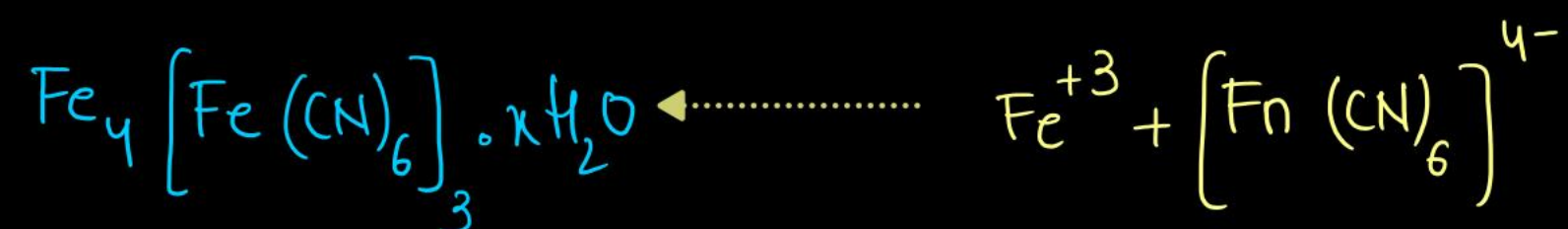


Sodium fusion
extract

A) Test for N

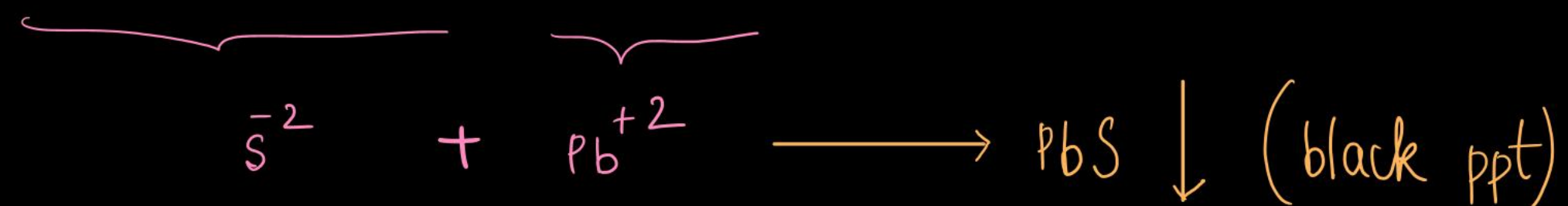
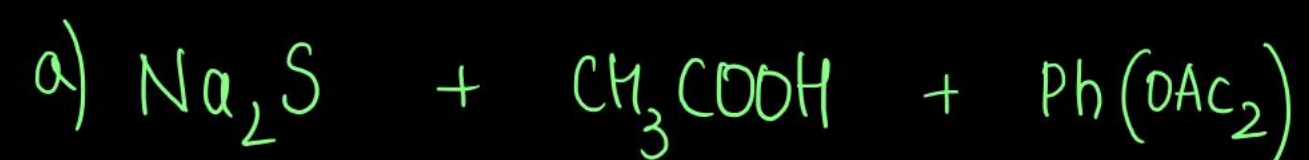


Conc H_2SO_4 $\rightarrow$ oxidises few
 H^+ Fe^{+2} to Fe^{+3}

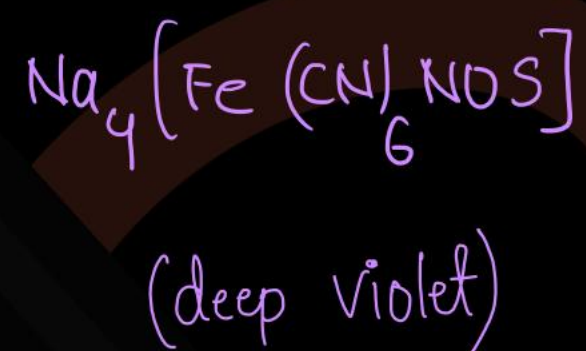


(Prussian Blue)

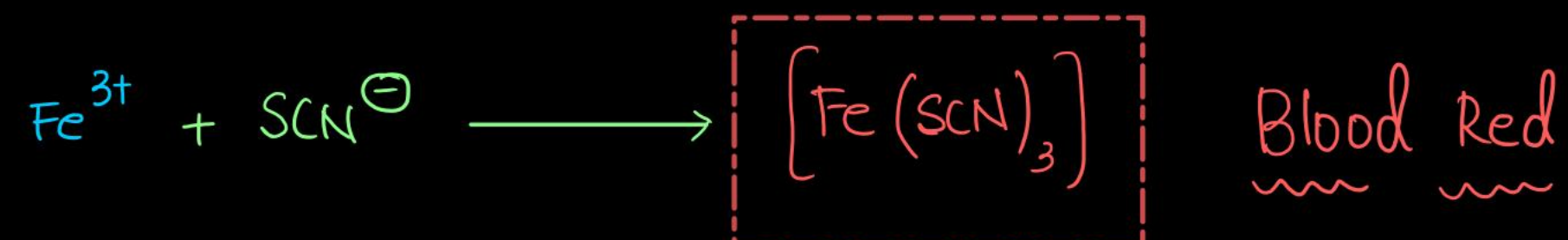
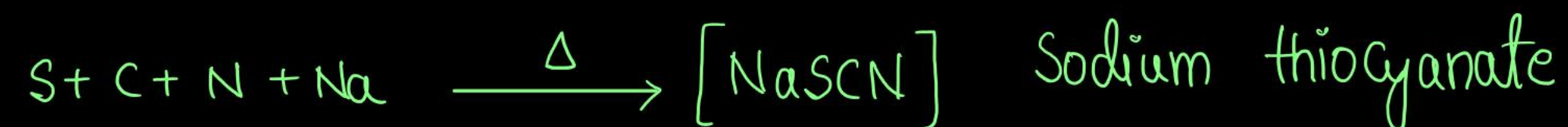
(B) Test for Sulphur :



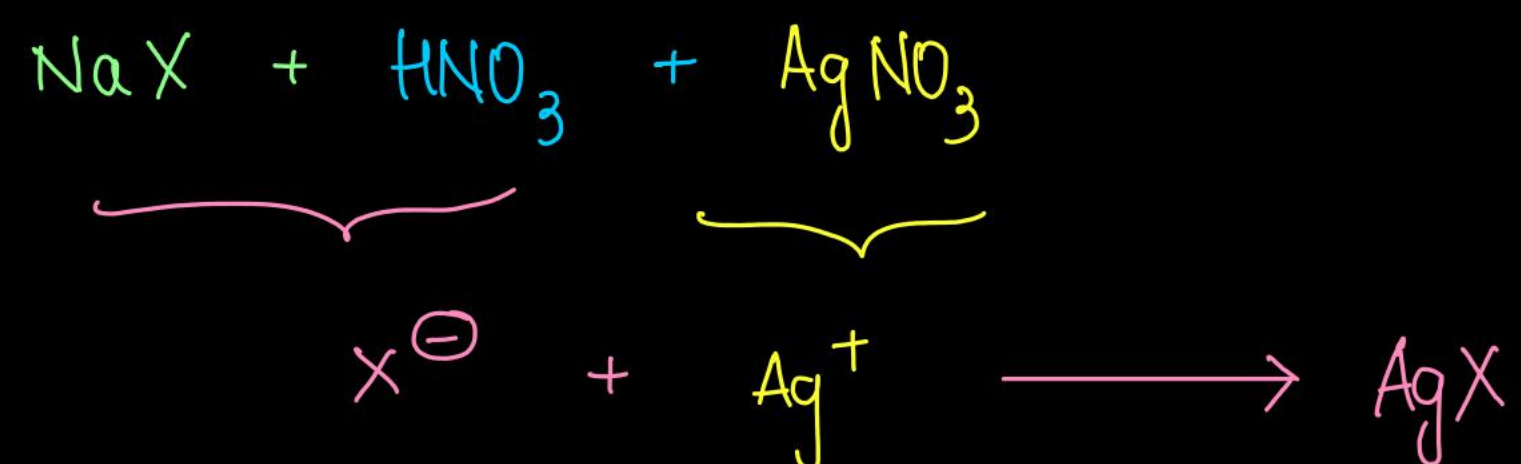
b) Sodium Nitroprusside test :-



* If S & N both are present,



c) Test of X :-



AgCl (white)> Soluble in NH_4OH

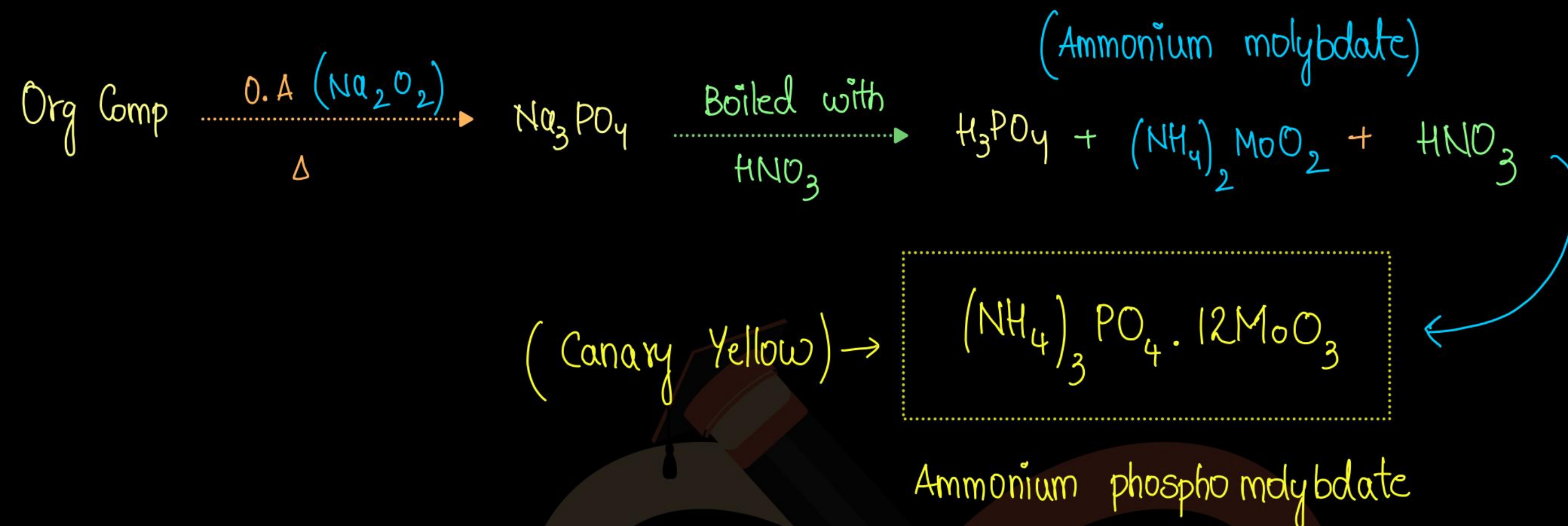
AgBr (Pale Yellow)> Sparingly soluble

AgI (Dark Yellow)> Insoluble

* If N (or) S is also present in the Comp, the sodium fusion extract is first boiled with Conc. HNO_3 to decompose Cyanide / Sulphide.

Because they would interfere with AgNO_3 .

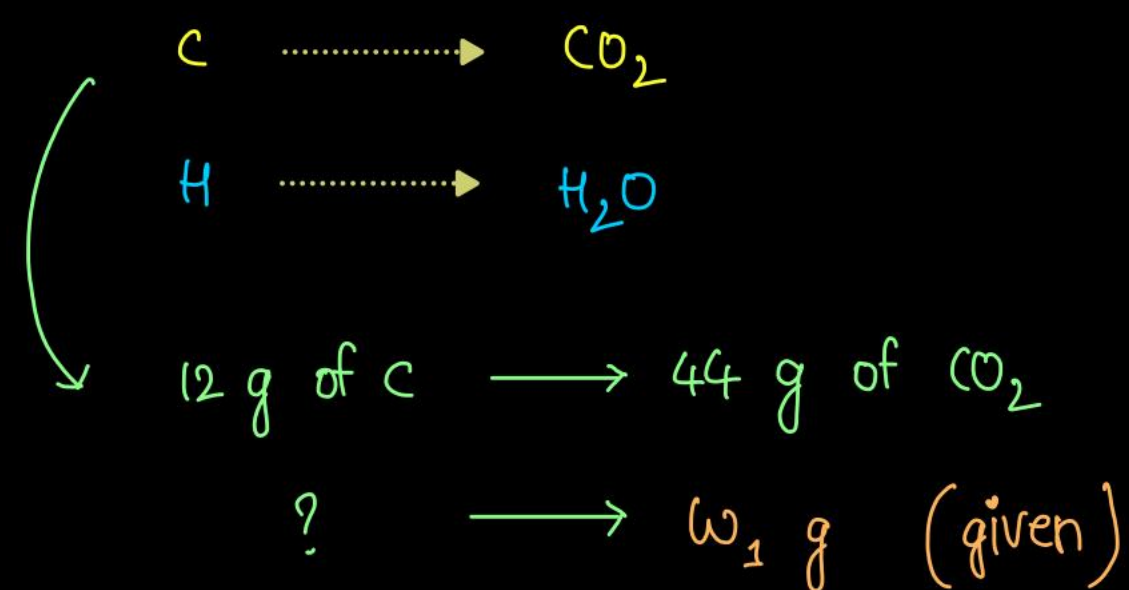
d) Test for Phosphorous:



Quantitative Analysis :-

1) Liebig's method :

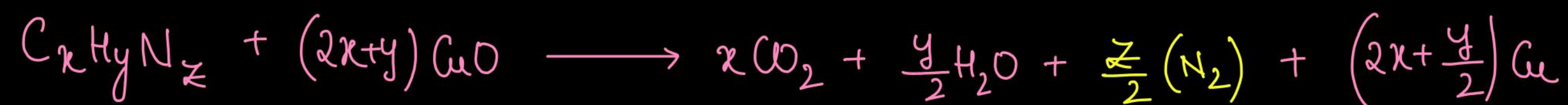
mass of O.C = W gm



$$\Rightarrow \% \text{ C} = \left(\frac{12}{44} \times \frac{W_1}{W} \right)$$

$$\% \text{ H} = \left(\frac{2}{18} \times \frac{W_2}{W} \right)$$

2) Duma's method (for N)



(Copper quaze)

$$\frac{(P-P_1)V_1}{T_1}$$

=

$$\frac{P_2V_2}{T_2}$$

At STP

$$\% N = \frac{28}{224} \times \frac{V_2}{V_1}$$

P = Pressure of moist gas

P₁ = aq. Tension

V₁ = Volume

T₁ = Temp

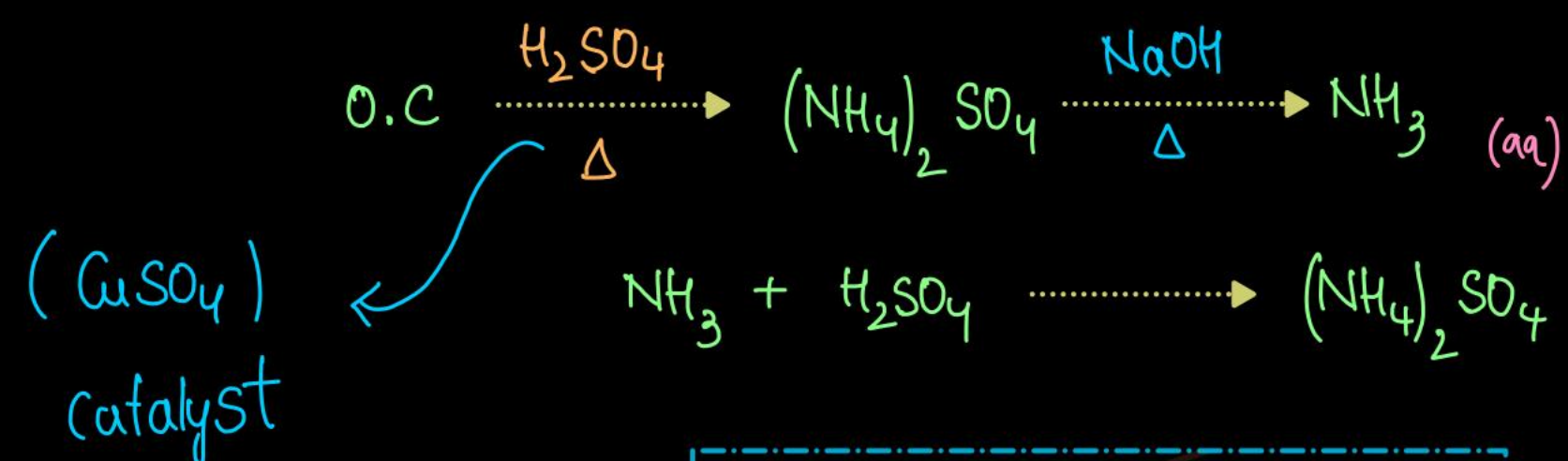
P₂ = 1 atm

= 760 mm of Hg

T₂ = 273 K

V₂ = Volume of N₂ collected at STP.

3) Kjeldahl's method :



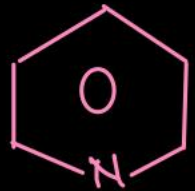
$$\% \text{N} = \frac{1.4 \times N \times V_{(\text{ml})}}{W}$$

* MI :

This method is not applicable for

i) -NO₂ (nitro comp)

ii) -N=N- (azo comp)

iii)  (Nitrogen in ring)

Q) 0.16 g of an Org Comp. gave by Duma's method 46 cc of N_2 measured over H_2O at $15^\circ C$ and at a pressure of 755 mm of Hg. find % N ? (aq. tension of H_2O = 15 mm of Hg)

Sol:-

$$\begin{aligned} V_1 &= 466 \\ P - P_1 &= 755 - 15 \\ &= 740 \\ T_1 &= 288 \end{aligned}$$

$$\frac{P_1 V_1}{T_1} = \frac{P_2 V_2}{T_2}$$

$$V_2 = \left(\frac{28}{224} \times \frac{V_2}{0.16} \right)$$

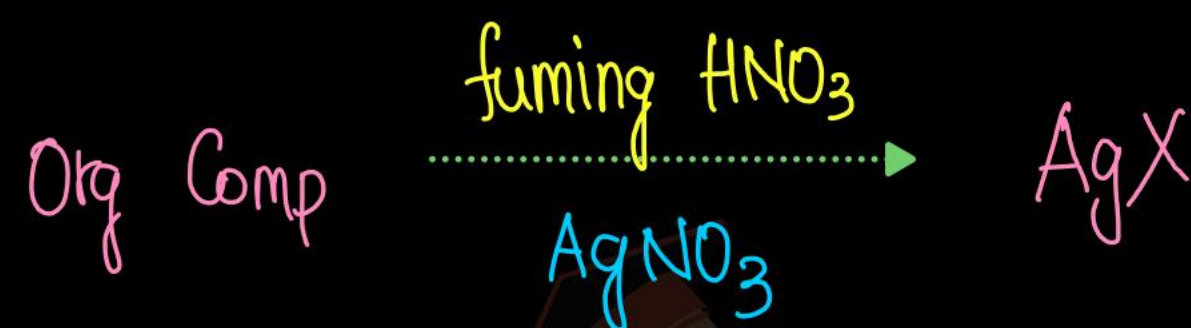
Q) During estimation of N present in an Org. Comp by Kjeldhal's method, the ammonia evolved from 0.5 gm of Comp neutralises 10 ml of 1M H_2SO_4 , find % N ?

Sol:-

$$\% N = \frac{1.4}{(0.5)} \times (2) \times 1 \times (10 \text{ ml}) \quad (M=nf)$$

④ Carius Method for (Cl, Br, I, S, P)

→ for X :



AgCl (white)

.....→ Soluble in NH₄OH

$$\% \text{ Cl} = \frac{35.5}{143.5} \times \frac{w_1}{w} \times 100$$

AgBr (Pale Yellow)

.....→ Sparingly soluble

$$\% \text{ Br} = \frac{80}{188} \times \frac{w_1}{w} \times 100$$

AgI (Dark Yellow)

.....→ Insoluble

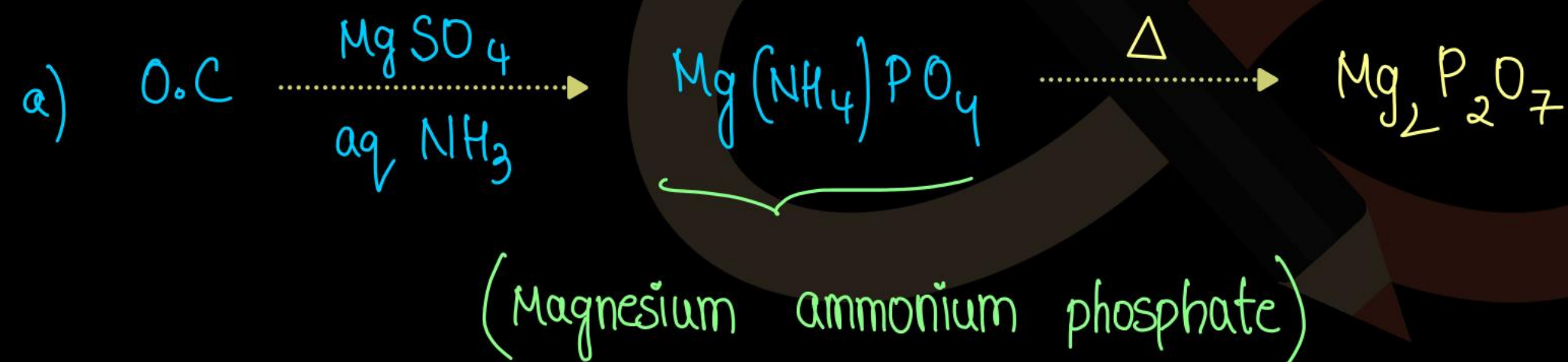
$$\% \text{ I} = \frac{127}{235} \times \frac{w_1}{w} \times 100$$

⑤ for S :-



$$\% \text{S} = \frac{32}{233} \times \frac{w_1}{w} \times 100$$

⑥ for P :-



$$\% \text{N} = \frac{62}{222} \times \frac{w_1}{w} \times 100$$



$$\% \text{P} = \frac{31}{1877} \times \frac{w_1}{w} \times 100$$

