Chapter-28

Paper Chromatography: Separation of mixtures of ions (Pb²⁺ & Ag⁺) by Paper Chromatographic Technique

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Principle:- Paper chromatography (PC) is a simple technique to separate mixtures of metal ions, amino acids, sugars, dyes, and drugs. A very small amount of sample is required for the analysis. PC has also become a popular technique for the separation of metal cations (basic radicals). In this experiment, the use of PC will be illustrated to separate a mixture of cations (group I and group II in the analytical table) and also identification of the unknown metal ion with the help of their R_f values and colour zones of cations. Compare the R_f values and also colour zones of individual cations with that of their R_f values in known mixture and in unknown.

Calculation the R_f value of each ion by the relation :

 $\mathbf{R}_{f} = \frac{\text{Distance travelled by the centre of solute zone}}{\text{distance travelled by the solvent front}} = \frac{\text{ds}}{\text{dm}}$

Requirements:

(i) Apparatus & chemical required

- * Chromatographic jar
- * Potassium chromate (k₂CrO₄)
- * Measuring cylinder
- * Lead nitrate Pb(NO₃)₂
- * Silver nitrate Ag(NO₃)₂
- * Pipette
- * Spotting capillaries
- * Small test tubes
- * 100ml beaker
- * Whatman No. 41 Filter paper

(ii) Solution required

(1) **Unknown solution**: It can be prepared by dissolving any one or two given unknown nitrate of analytical group I in water. Need to prepare saturated solution of unknown solution.

(2) Detector: 0.25M aqueous solⁿ of potassium chromate K_2CrO_4 , is prepared by dissolving 24.25g K_2CrO_4 in distilled water in 250 ml volumetric flask.

Procedure:

(A) Preparation of solution:

(i) Prepare 1cm³ saturated aq. Solution of Lead nitrate and Silver nitrate by dissolving about 1g crystals in a small test tube.

(ii) Developer: Distilled water

Hang whatman No. 41 filter paper strips in chromatographic jar.

On each stripe draw a line at about 1cm from the bottom and put dot in the side of the line. This end will be the bottom of the strip and development will take place from this end.

Apply the solⁿ of Pb^{2+} and Ag^+ separately on the filter paper with help of a fine capillary through two spot. Use a fresh capillary for each solution.

On the 3rd spot apply unknown solⁿ to predict unknown metal ion. Then dried the filter paper containing 03 spots. Hang the spotted and dried paper strip in the chromatographic jar containing distilled water with the upper end pinned to the steel rod and lower end touching the developer (water). It is observed that the strip is vertical. The Spot should always be above the developer level.

Allow the **developer (water)** to rise along the paper and wait till developer (solvent front) reaches near the upper end of the filter paper.

Remove the paper from the chromatography jar and **mark the solvent front with the help of pen.** Dried the paper to evaporate the developer.

Take potassium chromate (K_2CrO_4) solⁿ as separating solvent in a sprayer and spray it on the filter paper two yellow and one orange red colour developed immediately. Encircle the three coloured zones with pen.

Calculation:

Observe the coloured spots for three different cations.

* First spot Pb²⁺ appeared as yellow.

* Third spot Ag⁺ appeared as orange red.

* Second spot for unknown appeared same as Pb^{2+} or Ag^+ colour.

Measured the distance of colour zones of each solute from the point of application call this distance as (ds). Measured the distance between the solvent front and the starting line and call this distance as (dm).

Calculating the R_f value of each solute by –

$$R_{f} = \frac{\text{Distance travelled by the centre of solute zone}}{\text{Distance travelled by the solvent front}}$$
$$R_{f} = \frac{ds}{dm}$$

Observation Table:

Sample	ds	dm	$R_f = ds/dm$
Pb ²⁺			
Ag^+			
Unknown-1			
OR Unknown-2	·		

Conclusion:

Since the colour of the column and its R_f value of unknown sample is close to Ag^+ or Pb^{2+} therefore, the unknown metal ion will be Ag^+ or Pb^{2+} , lie in Group I & Group II in the analytical table.

Reference Books:

1. Paper Chromatography: A Laboratory Manual, Richard Joseph Block, Raymond Le Strange, Gunter Zweig, Academic Press, 1952.

2. Paper Chromatography and Electrophoresis, Gunter Zweig, John R. Whitaker, Joseph Sherma, Academic Press, 1967.
