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Summary

Description	Chromatography is an essential physical technique that allows the constituent components of a mixture to be identified, separated, and purified in preparation for qualitative examination. Paper chromatography (PC) is a sort of planar chromatography, which refers to a stationary phase that is a solid, flat surface. In this illustration stationary phase is a particular kind of paper (Whatman quantitative filter paper grade 41). The fundamental idea behind paper chromatography is the differential passage of a mixture's constituent parts through filter paper or chromatography paper. A quick method for separating mixtures of metal ions, amino acids, carbohydrates, colors, and pharmaceuticals is paper chromatography (PC). For this qualitative analysis, only a very small sample is needed. Metal cation separation has seen increased by the use of the PC approach. Here, the experiment shows how PC may be used to separate metal ions (Cu2+ & Fe3+) of different groups of the analytical table based on their colored spots and the retardation factors or retention factors (Rf) values by using different eluting solutions.
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Chapter-31

Innovative Method for the Separation of Group II and Group III Metal ions (Cu^{2+} & Fe^{3+}) by Paper Chromatographic Technique

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Principle:- Chromatography is an essential physical technique that allows the constituent components of a mixture to be identified, separated, and purified in preparation for qualitative examination. Paper chromatography (PC) is a sort of planar chromatography, which refers to a stationary phase that is a solid, flat surface. In this illustration stationary phase is a particular kind of paper (Whatman quantitative filter paper grade 41). The fundamental idea behind paper chromatography is the differential passage of a mixture's constituent parts through filter paper or chromatography paper. A quick method for separating mixtures of metal ions, amino acids, carbohydrates, colors, and pharmaceuticals is paper chromatography (PC). For this qualitative analysis, only a very small sample is needed. Metal cation separation has seen increased by the use of the PC approach. Here, the experiment shows how PC may be used to separate metal ions **(Cu^{2+} & Fe^{3+}) of different groups of the analytical table** based on their colored spots and the retardation factors or retention factors (R_f) values by using different eluting solutions.

Keywords: *qualitative analysis, chromatographic jar, spotting capillaries, colored spots, retention factor, under graduate experiment*

1. PC Experiment : Separation of Group II and Group III Metal ions (Cu^{2+} & Fe^{3+}) by 1(N) $K_4[Fe(CN)_6]$ solution

1.1. Materials and method

i) Experimental

Requirements

A. Apparatus & chemical required

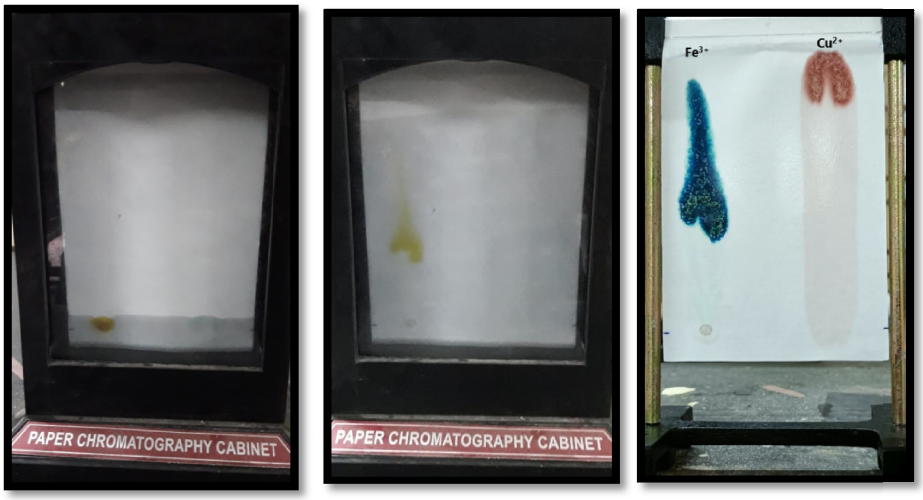
i) Chromatographic jar ii) Spraying bottle iii) 100 ml, 1N aqueous solution of potassium ferrocyanide $K_4[Fe(CN)_6]$ iv) Ferric chloride, $FeCl_3$ v) Copper sulfate pentahydrate $CuSO_4.5H_2O$ vi) Spotting capillaries vii) Small test tubes viii) 100ml Measuring cylinder ix) 10ml, 250ml beaker x) Whatman quantitative filter paper grade 41 xi) Distilled water

(B) Solution required

(i) Metal salts solution: Prepared saturated solution of ferric chloride and copper sulfate by dissolving them in distilled water (1mg/mL**)** in the 10ml beaker.
(ii) Eluting solution: Prepared 100 ml, 1N aqueous solution of potassium ferrocyanide $K_4[Fe(CN)_6]$ in the 250ml beaker.
(C) Developer: 200ml Distilled water in the 250ml beaker.

ii) Procedure

In the chromatographic jar, a strip of Whatman grade 41 filter paper was suspended. Place a dot to the side of the line you drew on this stripe, about 1 centimeter from the bottom. Development will start at this end, which is the bottom of the strip. Using a tiny capillary that passed through two (02) locations, the saturated solutions of Fe^{3+} and Cu^{2+} were applied individually to the filter paper. Every solution was applied to a brand-new capillary. The filter paper with the 02 spots was then allowed to dry outside. The filter paper strip that had been spotted and dried was then suspended once more in the chromatographic jar filled with distilled water, with the lower end coming into contact with the developer (water) and the top end being pinned to the steel rod. The strip is shown to be vertical. Always place the point above the level of the developer. Allow the developer (water) to rise along the filter paper **(Fig. 1a)** and wait for the two metal ions to rise together with the developer (solvent front) to approach the upper end of the filter paper **(Fig. 1b)**. After taking the filter paper out of the chromatography jar, the solvent front was indicated with a pen. The developer was then removed from the paper by drying it. Potassium ferrocyanide 1N aqueous solution was used as an eluting or spraying reagent; it was applied to the dry filter paper using a sprayer. Following the interaction with the aqueous solution of $K_4[Fe(CN)_6]$, prussian blue and reddish-brown spots appeared right away **(Fig. 1c)**, signifying the identification of Fe^{3+} and Cu^{2+} ions, respectively. Pen was used to indicate the colored zones.



(a) **(b)** **(c)**

Figure 1. Separation of metal ions (Fe^{3+} and Cu^{2+}) by paper chromatography by using 1N aqueous $K_4[Fe(CN)_6].3H_2O$ solution

2. Results and Analysis

2.1. Reactions involved during formation of color spots by interaction with solute zone

Ferric chloride solution was mixed with 1N aqueous solution of $K_4[Fe(CN)_6]$ formed Prussian blue or Berlin blue coloration spot, iron(III)hexacyanidoferrate(II), $Fe_4[Fe(CN)_6]_3$ in the Whatman grade 41 filter paper strip.

$$\mathbf{4\ FeCl_3 + 3\ K_4[Fe(CN)_6] \rightarrow Fe_4[Fe(CN)_6]_3 \downarrow + 12KCl}$$

(Prussian blue coloration spot)

$$\mathbf{2CuSO_4 + K_4[Fe(CN)_6] \rightarrow Cu_2[Fe(CN)_6] \downarrow + 2K_2SO_4}$$

(reddish brown coloration spot)

On the other hand, copper sulfate reacted with 1N aqueous solution of $K_4[Fe(CN)_6]$ formed reddish brown coloration spot, cupric ferrocyanide, $Cu_2[Fe(CN)_6]$ in the Whatman grade 41 filter paper strip.

2.2. Data Analysis

Observed the colored spots corresponding to two different cations (Cu^{2+} and Fe^{3+}). Two cations (Cu^{2+} and Fe^{3+}) were identified and separated by comparing their color spots and retention factor values. First spot appeared as prussian blue due to formation of iron(III)hexacyanidoferrate(II), $Fe_4[Fe(CN)_6]_3$ indicated the distance travelled by one solute zone as Fe^{3+} (ds_1) and second spot for Cu^{2+} appeared as reddish brown due to formation of cupric ferrocyanide, $Cu_2[Fe(CN)_6]$

indicated the distance travelled by another solute zone Cu^{2+} (ds_2). Then calculated retardation factors or retention factors (R_f) values **(Table-1).**

Retention Factor (R_f) = Distance travelled by the centre of solute zone in cm (ds_1 or ds_2) / Distance travelled by the solvent front in cm (dm)

Table 1: Separation of metal ions (Cu^{2+} and Fe^{3+}) by paper chromatography

Experiment Name	Solution used (Cation Present)	Eluting Solution	Color of the spot	Distance travelled by solute (ds) (cm)	Distance travelled by solvent (dm) (cm)	R_f value = ds/dm
Separation of metal ions (Cu^{2+} and Fe^{3+}) by paper chromatography	$FeCl_3$ (Fe^{3+} ion)	1N aqueous solution of $K_4[Fe(CN)_6]$	Prussian blue	13.2 (ds_1)	15.8	0.835
	$CuSO_4.5H_2O$ (Cu^{2+} ion)		Reddish brown	14.3 (ds_2)	15.8	0.905

3. Conclusion

Using water as mobile phase (developer), separation of metal ions [(Cu^{2+} & Fe^{3+})] **of different groups of the analytical table** has been done by taking eluting agent like 1N aqueous solution of $K_4[Fe(CN)_6]$ based on their colored spots and the retardation factors or retention factors (R_f) values. Thus, by using this improved technique, make paper chromatography easy to separate two cations [(Cu^{2+} & Fe^{3+})] from two different groups of the analytical table.

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