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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 of group III (Fe3+ & Cr3+) 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-33

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

Arijit Das
Department of Chemistry, Bir Bikram Memorial College, Agartala, Tripura, India
Email: arijitdas78chem@gmail.com

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 **of group III (Fe^{3+} & Cr^{3+}) 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 III Metal ions (Fe^{3+} and Cr^{3+}) by 1(N) $K_4[Fe(CN)_6]$ Solution

Materials and method

Experimental

Required chemicals and apparatus

(i) ChromatographyJar, (ii) Measuring cylinder, (iii) Capillary, (iv) Tiny test tube, (v) Beakers (10mL, 100mL, and 500mL), (vi) Grade 41 Whatman quantitative filter paper, (vii) Chromium (III) oxide, (viii) Ferric chloride, (ix) 1(N) $K_4[Fe(CN)_6]$ solution.

Required solution

(i) Solution of metal salts/oxide: To make a saturated solution, metal salts/oxide were dissolved in 1 mg/mL of distilled water in a 10 mL beaker. Metal salts/oxide: $FeCl_3$ & Cr_2O_3

(ii)Eluting agents used: 100ml 1(N) $K_4[Fe(CN)_6]$ solution was prepared in a 250 mL beaker with distilled water

Green developer

500 mL distilled water is used as green developer.

Procedure

A strip of Whatman 41 grade filter paper was carefully positioned inside the chromatographic jar, with a dot placed approximately 0.5 centimeters from the bottom as the starting point for development. Using fresh capillaries for each application, saturated solutions of metal salts/oxides were individually deposited onto two locations at the top of the chromatographic paper. Subsequently, the paper was left exposed to air to allow the spotted areas to dry.Once dry, the chromatographic paper strip was placed back into the chromatography jar filled with a green solvent (distilled water), with the bottom end submerged in the solvent and the upper end secured to a steel bar. The solvent (green developer) was allowed to ascend through the paper strip until it reached the uppermost portion. The movement of the solvent front was then marked using a pen upon removal of the chromatographic paper from the jar. Afterwards, the chromatographic paper strip was dried to remove any remaining developer. Eluting agents, as specified previously, were then applied over the dry filter paper using a sprayer. In the first paper chromatography experiment, the reaction with 1(N) $K_4[Fe(CN)_6]$ produced one Prussian blue spot and one light brown spot, indicating the presence of Fe^{3+} and Cr^{3+}ions, respectively. Each distinct zone of color was carefully outlined with a pencil for reference.

Results and Analysis

Reactions Involved During Formation of Color Spots by Interaction with Solute Zone

In the chromatographic filter paper strip, berlin blue or Prussian blue colored spot appeared (Kawatake et al., 2012) due to the formation of $Fe_4[Fe(CN)_6]_3$, iron(III) hexacyanidoferrate(II), when metal salt, $FeCl_3$ was combined with eluting solvent 1(N) $K_4[Fe(CN)_6]$.

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

(Prussian blue spot)

$$2Cr^{3+} + K_4[Fe(CN)_6] \rightarrow \{Cr[Fe(CN)_5OH]\}^{-1} \downarrow + 2\ CN^-$$

(Brown spot)

Conversely, in the filter paper strip, Cr^{3+} ion combined with a 1(N) aqueous solution of $K_4[Fe(CN)_6]$ to generate $\{Cr[Fe(CN)_5OH]\}^{-1}$, a light brown coloring spot.

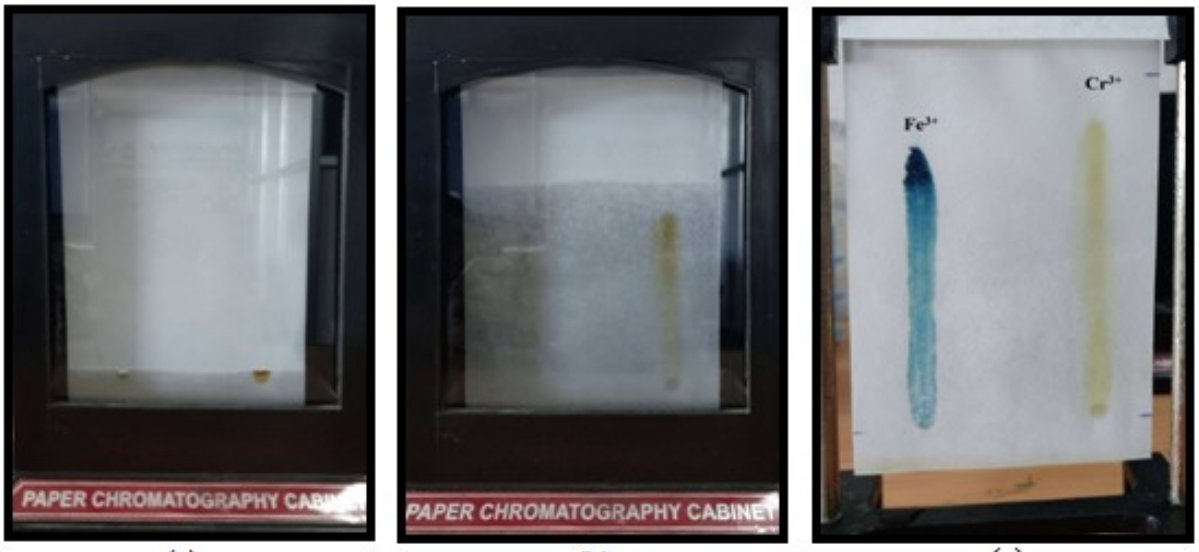

Figure 1. Separation of transition metal ions (Fe^{3+} and Cr^{3+}) by PC

Data Analysis

By comparing the retention factor values and observing the color spots of the two cations, (Fe^{3+} and Cr^{3+}), clear distinctions between them were made. The first spot, appearing as prussian blue, was attributed to the formation of $Fe_4[Fe(CN)_6]_3$. The distance traveled by the solute zone of Fe^{3+} (ds_1) was determined by the reaction between $FeCl_3$ and an aqueous solution of 1 (N) $K_4[Fe(CN)_6]$. The second spot, representing Cr^{3+}, emerged as light brown due to the formation of $\{Cr[Fe(CN)_5OH]\}$-1, formed from the reaction of Cr^{3+} in Cr_2O_3 with an aqueous solution of 1 (N) $K_4[Fe(CN)_6]$, indicating the distance traveled by another solute zone (ds_2). Retention factors (R_f), or retardation factors, were then calculated (see Table-1). By comparing the color spots and R_f values, the two cations (Fe^{3+} and Cr^{3+}) were successfully identified and distinguished from one another.

Retention Factor (R_f) = $\frac{\text{Distance travelled by the centre of solute zone in cm } (ds_1 \text{ or } ds_2)}{\text{Distance travelled by the solvent front in cm (dm)}}$

Table 1: Separation of metal ions (Fe^{3+} and Cr^{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 (Fe^{3+} and Cr^{3+}) by paper chromatography	$FeCl_3$ (Fe^{3+})	1(N) solution of $K_4[Fe(CN)_6].3H_2O$ (aq)	Prussian blue	10.7 (ds_1)	13.5	0.79
	Cr_2O_3 (Cr^{3+})		Light brown	11.8 (ds_2)	13.5	0.87

3. Conclusion

Using water as mobile phase (developer), separation of metal ions [(Fe^{3+} & Cr^{3+})] **of group III of the analytical table** has been done by taking eluting agent like 1(N) $K_4[Fe(CN)_6]$ solution 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 [(Fe^{3+} & Cr^{3+})] from the same group of the analytical table.

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