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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+ & Co2+) 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-32

Innovative Method for the Separation of Group II and Group IV Metal ions (Cu^{2+} & Co^{2+}) 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+} & Co^{2+}) 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 IV Metal ions (Cu^{2+} & Co^{2+}) by 10% NH_4OH solution

1.1. Materials and method

i) Experimental

Requirements

A. Apparatus & chemical required

i) Chromatographic jar ii) 10% NH_4OH Solution iii) Measuring cylinder iv) Copper sulphate $CuSO_4.5H_2O$ v) Cobalt nitrate $Co(NO_3)_2.6H_2O$ vi) Spotting capillaries vii) Small test tubes viii) 10ml, 100ml, 250ml beaker ix) Whatman quantitative filter paper grade 41 x) Distilled water xi) FT-IR spectrophotometer

(B) Solution required

(i) Metal salts solution: Prepared saturated solution of copper (II) sulfate and cobalt (II) nitrate by dissolving them in distilled water (1mg/mL**)** in the 10ml beaker.

(ii) Detector: Prepared 10% NH_4OH solution in distilled water in the 100ml beaker.

(C) Developer: Taken 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. Cu^{2+} and Co^{2+} saturated solutions were administered individually to the filter paper through two (02) locations using a tiny capillary. For every remedy, a brand-new capillary was used. 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. Developer (water) is allowed to rise along the filter paper **(Fig. 1a)**; two metal ions rise along the filter paper as the developer (solvent front) approaches the upper end of the 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. 10% NH_4OH solution was used in a sprayer as a separating solvent or spraying reagent, and the dried filter paper was covered with it. As soon as 10% NH_4OH was added, green and blue colored spots appeared **(Fig. 1c)**, signifying the presence of Co^{2+} and Cu^{2+} ions, respectively.

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

Figure 1. Separation of metal ions (Cu^{2+} and Co^{2+}) by paper chromatography by using 10% NH_4OH solution

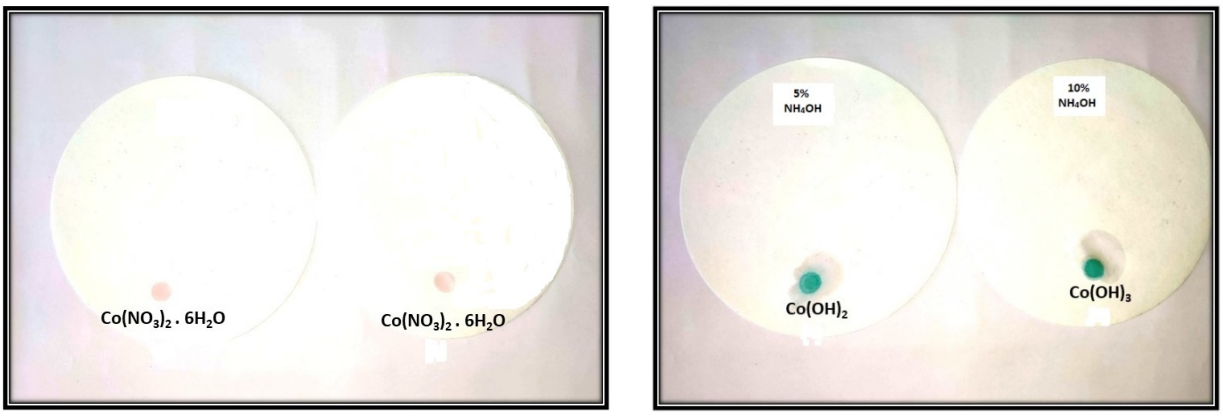
2. Results and Analysis

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

When copper salt reacts with 10% NH_4OH solution gives metal (II) hydroxide, $Cu(OH)_2$, which results of blue colored spot in the Whatman grade 41 filter paper. When Co(II) nitrate reacts with 10% NH_4OH solution gives Co(III) hydroxide, $Co(OH)_3$, results green colored spots in the Whatman grade 41 filter paper instead of bluish green $Co(OH)_2$, which obtained by the reaction of Co(II) nitrate with 5% NH_4OH solution. The cobalt(II) ion is more stable because it cannot quickly oxidize to the far less stable cobalt(III) state. All cobalt(II) salts are therefore stable, however cobalt(II) is readily converted to cobalt(III) in basic solutions (50 ml 10% NH_4OH). With increasing concentration of NH_4OH solution (5% to 10%), basic nature of the reaction medium increases and with increasing concentration of basic medium, air oxidation of cobalt(II) to cobalt(III) takes place relatively easily. Thus, Co(II) hydroxide (bluish green) oxidizes to Co(III) hydroxide results green colored spot. That's why after eluting with 10% ammonium hydroxide solution bluish green coloured of $Co(OH)_2$ immediately converted into green coloured $Co(OH)_3$. It was further confirmed by the spot analysis of cobalt(II) nitrate solution with 5% and 10% NH_4OH solution individually in the Whatman grade 41 filter paper **(Fig.2.)**. It was observed that during spot test of cobalt (II) nitrate solution with 5% NH_4OH, bluish green coloured developed **(Fig.2a Left)** and with 10% NH_4OH the green colour developed immediately **(Fig.2b Right)**.

$$\mathbf{Co^{2+} + 10\%\ NH_4OH \rightarrow Co(OH)_3\,(s)\downarrow}$$
(Green coloration spot)

$$\mathbf{Cu^{2+} + 10\%\ NH_4OH \rightarrow Cu(OH)_2(s)\downarrow}$$
(Blue coloration spot)



(a) **(b)**

Figure 2. Spot analysis of cobalt(II) nitrate in the Whatman grade 41 filter paper (a) before addition of NH_4OH (b) after addition of 5% and 10% NH_4OH solution

2.2. FT-IR Analysis of Green colored spot

The green spot underwent FT-IR analysis using a Bruker Alpha II model spectrometer, operating in the 4000–400cm^{-1} range. The hydroxyl assembly in the Co(III) hydroxide typically experiences stretching and bending vibrations in the IR region. About 1638 cm^{-1}, a bending vibration is seen, and within 3490-3499 cm^{-1} is often where the tough broad band for the -OH stretching vibration occurs. These vibrations indicate that the cobalt(III)hydroxide containing –OH group. For a Co-O bond, the average range of an IR frequency is 400–600cm^{-1}. However, in the green area, the Co-O bond's IR band is located at **646 cm^{-1} (Figure 3).** In the higher oxidation state of cobalt atom, such (+3) or (+4), there is usually a (+ve) shift in the i.r. frequency of the cobalt-oxygen bond (Co-O). Metal-oxygen bond gets stronger in higher oxidation states, which raises the vibrational frequency. The presence of cobalt in the (+3) state in cobalt(III)hydroxide, is therefore suggested by the observed i.r. frequency of the cobalt-oxygen linkage, resulted green color spot.

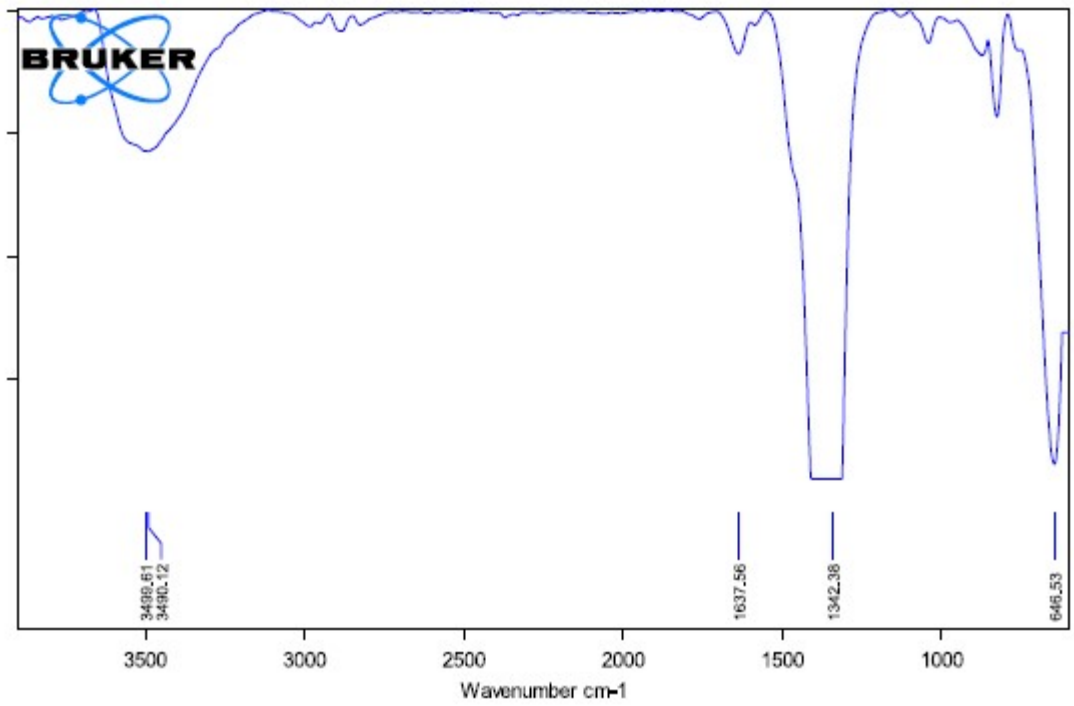


Figure 3. IR spectrum of green ppt. of $Co(OH)_3$

2.3. Data Analysis

Two cations (Cu^{2+} and Co^{2+}) were identified and separated by comparing their color spots and retention factor values. Observed the coloured spots corresponding to two different cations (Cu^{2+}

and Co^{2+}). First spot appeared as green due to formation of cobalt (III) hydroxide $Co(OH)_3$ indicated the distance travelled by one solute zone, Co^{2+} (ds_1), where in basic medium it is readily oxidized into Co^{3+} and second spot Cu^{2+} appeared as blue due to formation of copper (II) hydroxide $Cu(OH)_2$ indicated the distance travelled by the 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 Co^{2+}) 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 Co^{2+}) by paper chromatography	$Co(NO_3)_2.6H_2O$ (Co^{2+} ion)	10% NH_4OH solution	Green	12.2 (ds_1)	14.5	0.8413
	$CuSO_4.5H_2O$ (Cu^{2+} ion)		Blue	13.4 (ds_2)	14.5	0.9241

3. Conclusion

Using water as mobile phase (developer), separation of metal ions [(Cu^{2+} & Co^{2+})] **of different groups of the analytical table** has been done by taking eluting agent like 10% NH_4OH 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 [(Cu^{2+} & Co^{2+})] from two different groups of the analytical table.

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