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THEORETICAL AND EXPERIMENTAL RESEARCH METHODS IN
CHEMISTRY SEPARATION AND IDENTIFICATION OF METALS (NI(II),
CU(II), PB(II), FE(III), CO(II), CD(II)) BY PAPER AND THIN-LAYER
CHROMATOGRAPHY

Аннотация: В данном исследовании изучается применение хроматографических методов для разделения и идентификации катионов металлов Fe(III), Ni(II), Co(II), Cu(II), Cd(II) и Pb(II). Объединяя бумажную хроматографию и тонкослойную хроматографию, анализ оценивает эффективность этих методов в строгих аналитических рамках. Тщательный обзор хроматографических принципов предшествует экспериментальному подходу, в котором подробно описываются оптимальные условия разделения, выбор подвижных и неподвижных фаз, а также выявляющих реагентов. Полученные результаты подтверждают надежность и селективность этих методов идентификации металлических частиц, подчеркивая их актуальность для аналитического и экологического контроля.

Ключевые слова: Хроматография, Металлы, Анализ, Разделение, Идентификация

Annotation: *This study examines the application of chromatographic methods for the separation and identification of metal cations Fe (III), Ni(II), Co(II), Cu(II), Cd(II) and Pb(II). Combining paper chromatography and thin-layer chromatography, the analysis considers the effectiveness of the methods*

used in the analysis analytical frames. A thorough review precedes the experimentally possible, and the ship can be operated Optimal listening, continuous and non-current transmission phases, as well as the detection reagents. The results obtained demonstrate the selectivity of these methods for the identification of metal species, emphasizing their Current analytical and ecological control.

Keywords: *Chromatography, Metals, Analysis, Separation, Identification*

INTRODUCTION

Chromatography (from the Greek chroma, chromatos – color, paint).

Chromatography is defined as a "method of analysis" in which a solvent or gas flow promotes the separation of substances by differential migration from a narrow initial zone to a porous sorbent medium. Gas chromatography and solution (liquid) chromatography are the main subdivisions of chromatography, and paper chromatography is a subdivision of solution chromatography based on the shape of the sorption system. The solvent in paper chromatography is usually organic in nature, and the stationary phase is a sheet of paper containing water or some other polar liquid. Reversed phase paper chromatography uses a fixed non-polar sorbent phase, usually produced by impregnating paper, in combination with a polar solvent.

Paper chromatography may have its origins in the description of Pliny (23-79 AD) using papyrus impregnated with an extract walnuts, for the determination of iron sulfate. In the nineteenth century, dyeing industry, solutions in vats were checked for purity by applying stains to paper or fabric and observing the number of concentric rings formed when the liquid spreads [3]. This is one of the method of spot testing color mixtures on printing, blotting and filter paper was studied by Runge and Reed, who independently published the results before 1900.

In the practice of chromatography, it is important to use substances in a precise quantity, clean materials to achieve a good analytical result.

Heavy metals have a strong toxic effect on humans and warm-blooded animals, as well as on the inhabitants of the aquatic environment (plankton, crustaceans, fish).

The rapid development of methods for determining toxic substances in the environment has raised the study of the processes of transformation of substances and hygienic assessment of the quality of the environment for a qualitatively new level. Very high requirements must be imposed on the quality of control, its reliability and accuracy. The reliability of the method depends on mainly on the physicochemical properties of the substances to be determined, the correct choice of method and its characteristics. For a more correct Methods for determining the degree of pollution of environmental objects must be sufficiently sensitive and selective.

In some cases, it is highly desirable to control the content of poisonous substances.

1. **SEPARATION AND IDENTIFICATION of Fe(III), Ni(II), Co(II), Cu(II) cations**

Reagents and equipment

Standard aqueous solutions of Fe (III), Ni (II), Cu (II) with a concentration of each component of 10 mg/ml

Mobile phase: a mixture of n-butanol, acetone, end. HCl and Water (4:3:2:1)

Developers: 1. Saturated acetone solution of ammonium rhodanide;

2. Dimethylglyoxime in a 10% solution of ammonium hydroxide, 1% solution.

(Mobile phase: ethanol-diluted by half the end. HCl (4:1), 50 mL total volume

Mobile phase: acetone-3H HCl mixture (9:1), 50 ml total volume)

(Developer 1: 10% potassium hexacyanoferrate (II) solution

Developer 2: 25% ammonia solution)

Chromatographic plates with an unmodified silica gel layer (Silufol, Sorbfil, Merck)

Glass capillaries / microsyringe, Tweezers

Chromatographic chamber/desiccator or cylinder with a lapped cover, spray bottle

Electric stove / drying cabinet

Protective equipment: latex gloves

The separation of iron (III), cobalt (II), and nickel(II) ions is based on their ability to form complex ions with chloride ions of different stability and on the different mobility of these ions in the mobile-stationary solvent system.

Complex iron ions $[\text{FeCl}_4]$ move almost along with the solvent front. Behind them are cobalt ions and then nickel ions.

The iron zone on the chromatogram is cut out and, after extraction, its photometric content in the form of iron rhodamine is determined.

Performance of work. Attention! Chromatography paper should be grasped with your hands only by the corners of the square.

A strip no more than 1 cm wide ("tail") is cut out on a standard sheet of chromatographic paper and shortened by 1.5 cm (Fig. 3.13). A sample of the tested solution of 8-10 μl in 2-3 steps is applied to the center of the sheet at the base of the "tail" using a microsyringe. After each application, the stain is allowed to dry. The diameter of the spot on the sheet should not exceed 3 mm. 10-15 ml of mobile solvent is poured into the bottom of the Petri dish. A square sheet of chromatographic paper with a fineness is placed on a Petri dish, dipping the "tail" (without bending its base) into the solvent and covered with the same Petri dish.

The solvent rises on the sheet along the "tail" and moves radially on the paper. The movement of the zones of the separated substances also occurs radially. The zones take the form of expanded arcs. When the solvent on the paper has traveled $\frac{2}{3}$ of the way to the walls of the Petri dish, the development of the chromatogram is stopped, the chromatogram is removed and dried in a box under

traction. To develop the chromatogram, it is sprayed from a spray bottle with a saturated acetone solution of ammonium rhodanide. The zone of iron (III) is colored red-brown, and cobalt (II) is colored blue. After drying the chromatogram, measure the Rf for Fe³⁺ and Co²⁺ and use a brush to moisten the area of paper between the cobalt (II) zone and the starting line with an ammonia solution of dimethylglyoxime (closer to the cobalt zone, trying not to touch its blue zone). A zone of nickel (II) appears, colored crimson.



Figure 1: Cobalt appearance when drying paper from a petri dish

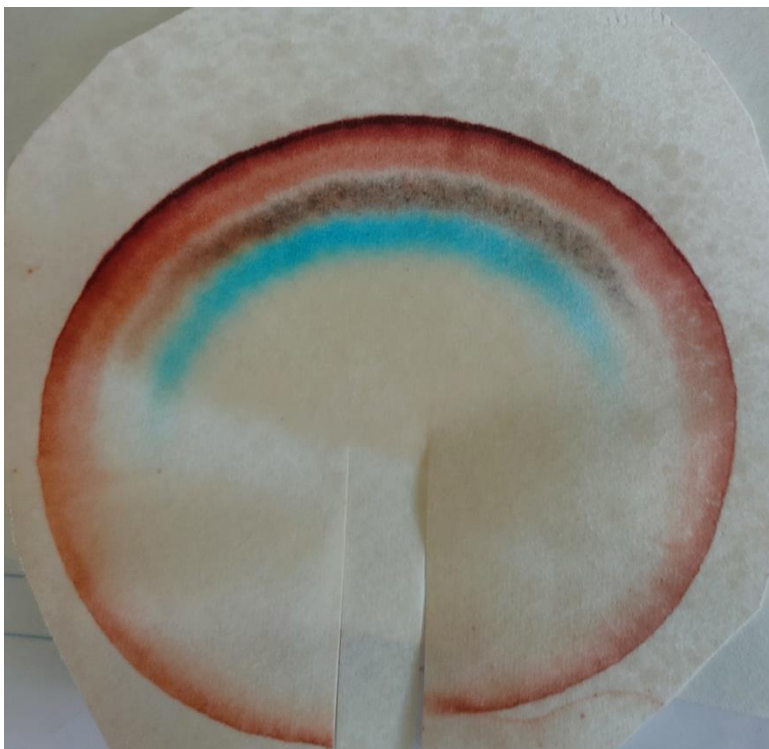


Figure 2: the result of chromatographic concentration of the solution (CuII, CoII, FeIII)

Calculation:

$$R_f = \frac{l}{L}$$

$$R_f(Fe) = \frac{4}{4,2} = 0,952$$

$$R_f(Cu) = \frac{3}{4,2} = 0,71$$

$$R_f(Co) = \frac{2,5}{4,2} = 0,59$$

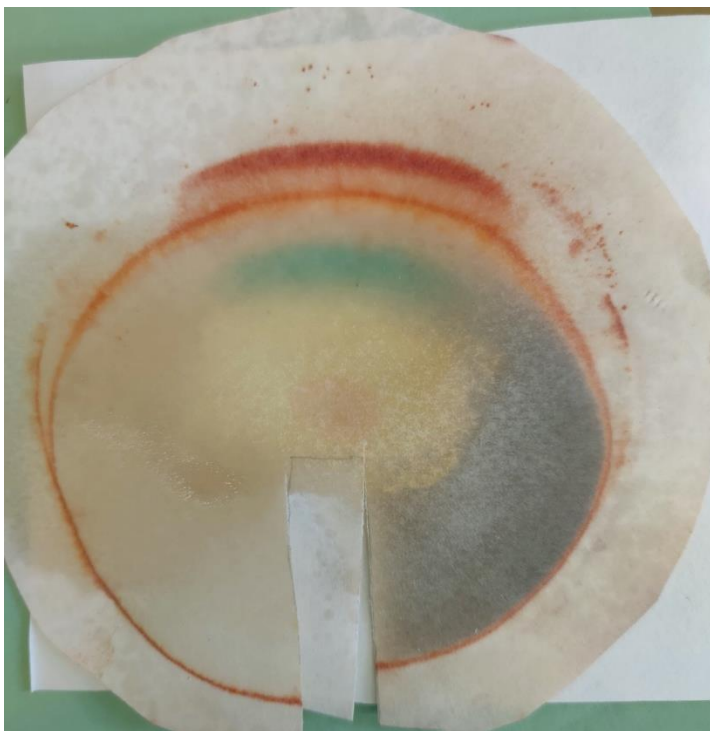


Figure 3: Result of chromatographic solution of the problem

$$R_f = \frac{l}{L}$$

$$R_f(Fe) = \frac{5}{5,5} = 0,95$$

$$R_f(Co) = \frac{3,3}{5,5} = 0,6$$

$$R_f(Co) = \frac{1}{4,2} = 0,182$$

1. **SEPARATION AND IDENTIFICATION of cations Cd(II), Pb(II), Cu(II), Co(II), using Merck paper for chromatograms**

Reagents and equipment

0.1M standard solutions of Cd(II), Pb(II), Cu(II), Co(II)

Preparation of the solution:

$$m(\text{Cd}(\text{NO}_3)_2) = \frac{M(\text{Cd}(\text{NO}_3)_2) \cdot C \cdot V_{\text{flask}}}{1000} = \frac{236,4 \cdot 0,125}{1000} = 0,5910\text{r}$$

$$m(\text{CuCl}_2) = \frac{M(\text{CuCl}_2) \cdot C \cdot V_{\text{flask}}}{1000} = \frac{135,0 \cdot 0,125}{1000} = 0,3375\text{r}$$

$$m(\text{Pb}(\text{NO}_3)_2) = \frac{M(\text{Pb}(\text{NO}_3)_2) \cdot C \cdot V_{\text{flask}}}{1000} = \frac{331,0 \cdot 0,125}{1000} = 0,5910\text{r}$$

$$m(\text{CoCl}_2) = \frac{M(\text{CoCl}_2) \cdot C \cdot V_{\text{flask}}}{1000} = \frac{130,0 \cdot 0,125}{1000} = 0,3250\text{r}$$

Mobile phase: mixture of 100 ml n-butanol, 20 ml 1.5M HCl, 0.5 ml acetylacetone

Developers: 1. potassium hexacyanoferrate (II) (brown-red with copper),
2. sodium sulfide for cadmium (yellow), 3. Potassium iodide for lead (yellow)

Chromatographic plates with an unmodified silica gel layer (Silufol, Sorbfil, Merck)

Glass capillaries / microsyringe, Tweezers

Chromatographic chamber/desiccator or cylinder with a lapped cover, spray bottle

Electric stove / drying cabinet

Protective equipment: latex gloves

Method

1. Prepare a desiccator/chamber for chromatography.
2. Without disturbing the sorbent layer, draw the start line and finish lines with a simple pencil on the plate at a distance of 1 cm from the edge.
3. The analyzed sample must be applied quantitatively, while the diameter of the spot should not exceed 2-5 mm, the center of the spot should be on the start line.

Take the plates with tweezers carefully by the edges, without grabbing the central part!

Use a microsyringe/capillary to apply a solution containing a mixture of amino acids to the starting line of the sample to be analyzed. Next to it, apply 0.1M standard solutions

Cd(II), Pb(II), Cu(II).

4. Dry the plate and reapply the analyzed sample to the start line and dry the plate again.

5. Place the plate with the applied sample and witnesses vertically in the chamber/desiccator so that it is immersed in the mobile phase by no more than 5 mm.

In this case, the applied sample should be above the solvent layer.

6. To carry out chromatography.

7. Carefully!! with tweezers to deliver the plate from the chamber/desiccator when the front of the PF

will pass the specified distance and the components will be separated.

8. Dry the plate over the tile or in a drying cabinet (do not put it on the tile!).

9. Spray the plate with a solution from a spray bottle (under the draft)

Then dry the plate further, heating until spots appear on the chromatogram.

10. After chromatography, compare the position of the stains of the tested mixture and individual substances, then draw conclusions about the presence or absence of certain components in the analyzed sample. To identify the components, compare the calculated R_f values for the components of the mixture and the individual substances.



Figure 4: Qualitative Analysis of Metals Using Merck Paper for Chromatograms

Calculation of R_f :

$$R_f(FeIII) = \frac{1}{9} = 0,11$$

$$R_f(Ni(II)) = \frac{0,1}{9} = 0,01$$

$$R_f(Pb(II)) = \frac{0,1}{9} = 0,01$$

$$R_f(Co(II)) = \frac{0,2}{9} = 0,02$$

CONCLUSION

In the process of passing the introductory practice, the techniques of work in the analytical laboratory, preparation of solutions, work with chromatographic paper and equipment, selection of optimal conditions for chromatographing of simulated mixtures were mastered.

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