

Separation by paper chromatography

1. Performing the exercise

1.1. Preparation of the paper and deposition of the substances.

ATTENTION! - use *Whatman 1 paper for the separation.*

Cut out the blotting paper according to the attached pattern and instructions from the tutor. Mark the lines only with a pencil !!! The end line - 10 cm from the starting line - is the border to which the solution of the developing phase should reach.

Dip the individual tips of the blotting paper once in the appropriate solutions: $J^- / Br^- / SCN^-$ and the tested mixture. After dipping the ends of the paper in the solutions - dry the paper with a stream of warm air in a specially prepared stand.

1.2. Preparation of the developing phase.

Prepare 40 cm³ of the developing phase, which will ensure the separation of the tested mixture according to the following scheme:

butanol	:	acetone	:	NH_3 aq.
3	:	13	:	4

1.3. Developing of the paper chromatogram.

Pour 40 cm³ of the developing mixture into the chromatographic chamber. Hang the dried chromatogram in the chromatographic chamber so that the lower edge of the chromatogram is immersed in the solvent by about 1 cm. Develop the chromatogram until the solvent front reach the top horizontal line.

Take out the chromatogram and dry over a stream of warm air. Spray the chromatogram with the developing solution ($Fe^{3+} : H_2O_2 = 1:1$) (5 cm³ + 5 cm³ made up to 50 cm³ H_2O) and dry again over a stream of warm air. Draw the resulting spots with a pencil and describe.

1.4. Identification reactions of the studied anions without separation.

About 1 cm³ of the mixture for identification pour into a large and tall test tube and dilute with water to 2-3 cm³ - then acidify with 2M H_2SO_4 . Add 1-2 cm³ of chloroform, and then add chlorine water, shake the liquid thoroughly after each portion. The violet color characteristic for J_2 will appear in the organic layer first (if it is in the identification solution!!!). The color fades as more chlorine water is added:



Exercise subsection continued - see second page !!!

If bromide ions are present in the tested mixture, then after the violet color disappears from J_2 , a red-brown color of Br_2 appears, which eventually turns into a yellow color.

ATTENTION ! Pour samples with chloroform into a specially prepared container !!!

1.4.2. About 1 cm^3 of the tested mixture and about 1 cm^3 of Fe^{3+} solution pour into the test tube. If the color of the water layer is blood red, it indicates the presence of SCN^- ions.

2. Elaboration of the results

- Present the observed phenomena in the form of appropriate chemical reactions.
- Calculate the R_f value of the analyzed mixture and standards.
- Determine the composition of the analyzed mixture.
- Attach the obtained chromatogram.
- Make a brief summary of the advantages and disadvantages of detecting ions in a mixture without separation and after separation by paper chromatography.

3. Conclusions

4. The scope of material

- Chromatography - classification, theoretical basis
- Paper chromatography
- Anion analysis

5. Literature

- J. Minczewski, Z. Marczenko. *Chemia analityczna*, t3. PWN W-wa 1976
- M. Weller, T. Overton, J. Rourke, F. Armstrong. *Inorganic chemistry*. Oxford University Press 2018
- C. E. Housecroft, A. G. Sharpe. *Inorganic chemistry*. Pearson 2018
- R. J. Block, E. L. Durrum, G. Zweig, *A Manual of Paper Chromatography and Paper Electrophoresis*. Elsevier 1995

