CHROMATOGRAPHIC SEPARATION AND IDENTIFICATION OF Hg (II) COMPLEXED BY EDTA

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ABSTRACT

A new method for paper chromatography is described for the simultaneous separation and identification of Hg(II) complexed by EDTA (HgY^{2-}), in the pH range of 1-2, in the presence of Cd(II), Pb(II), Bi(II), Fe(III), Zn(II), Mn(II), Co(II), Cu(II) and Ni(II).

RESUMO

É descrito um novo método de cromatografia em papel que permite a separação e identificação simultânea, em pH 1-2, de Hg(II) complexado por EDTA(HgY^{2-}), em presença de Cd(II), Pb(II), Bi(III), Fe(III), Zn(II), Mn(II), Co(II), Cu(II), e Ni(II).

Discussion: The complexing properties of EDTA (sodium salt of ethylenediaminetetraacetic acid) are already known. However, to the authors knowledge, there are no references related to chromatographic migration of complexed ions in different pH values. It was verified, experimentally, that the complexing of metallic ions resulted in a global modification in their behavior, during the chromatographic migration. Due to this fact, experiments have been done by varying the pH in the range of 1-10. The final concentration of the sample was 2.5 mg/ml, an amount of about 2 microliters being applied to the paper. Several chromogenic agents were tested, as dithizone, alizarin, aluminon, hydrogen sulphide and ammonia vapors. The best results were obtained with a concentrated solution of dithizone in carbon tetrachloride.

The results of the separation of the complexed metallic ions with EDTA are satisfactory since, as shown in Table I, Cd(II), Pb(II), Bi(III), Zn(II), Mn(II), Cu(II), Co(II) and Ni(II) have migrated practically with the "front" of the movable phase, while Hg(II) and Fe(III) showed different positions from the mentioned cations in the pH range from 1 to 2. The distinction between the HgY^{2-} and FeY^{-} complexes is not difficult, since their chromatographic behavior and the colors resulting from the reaction with the chromoganic agents are different. In a pH value greater than 2, with the exception of the FeY^{-} complex, which migrates slowly, interference was not observed with the other complexes.

Table 1. Retardation factor (Rf) of the metallic complexes.

Cation	Rf	Cation	Rf
Hg(II)	0.77	Co(II)	0.98
Fe(III)	0.92	Ni(II)	0.98
Cd(II)	0.97	Zn(II)	0.97
Bi(III)	0.93	Mn(II)	0.98
Cu(II)	0.95	Pb(II)	0.99

As suggested by Clark & Lubs, the systems that constituted the movable phases were buffer solutions, in the pH range from 1 to 10.
The practically constant acidity, during the separation, is due to these solutions and by the fact that the samples were complexed in the same pH of the movable phase.

Procedure

a) Development Chamber - Pyrex cylindrical glass container, 6 cm diameter and 24.5 cm of height, saturated with the system that constitutes the movable phase.
b) Sample Preparation - In a test tube, 0.5 ml of the sample (10 mg of M^{n+}/ml) is added and stirred with 1 ml of the buffer solution and 0.5 ml of a 5% solution of EDTA. Stir well and apply the solution in the chromatographic paper. With increasing pH values, insoluble compounds are formed which, however, are dissolved by the excess of the complexing agent, except in the cases of Bi(III), Pb(II) and Fe(III). In these cases, it was utilized the aqueous phase that remained after centrifugation.
c) Application - The samples are applied with cappilary tubes (diameter 0.8 mm), to the bottom part of a chromatographic paper Whatman N.1 or Schleicher N.2043[a], in strips of 20 x 5 cm, 2 cm over the bottom edge of the paper and this region will be in contact with the movable phase contained in the development chamber. Care must be taken not to diffuse the sample (the diameter of the droplet must be less than 3 mm) and this is obtained with a soft and quick touch of the capillary tube on the chromatographic paper, exposed to hot draft air.
d) Development - After the application, the bottom part of the paper is immersed in the movable phase, which is progressively absorbed by this material. When the movable phase reaches the application point, the separation begins.

The movable phase travels 10 cm, measured from the application point, and the time spent is approximately 40 minutes, using the chosen buffer solutions. The paper is then dried in an oven at 80ºC or with hot draft air. During this opertaion, it is possible to locate the position of the colored complexes, e.g. the complexes of cobalt (pink) and iron (yellowish).

After drying, the other complexes are located with chromogenic agents. The determination of the positions is done immersing the paper in a 40 ml volume of the utilized reagent, contained in a Petri plate (diameter = 10 cm) and passing the chromatogram in the opposite direction of migration.

Conclusion

The present work is characterized by the different behaviour of the Hg-EDTA complex, if compared with other complexes tested in the above conditions (pH 1-2). In this range, the HgY^{2-} have shown a Rf value of 0.77, which is smaller than the value of 0.97 shown by the other ions that migrated along with the solvent.

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References

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