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Mohsin Qureshi^a; J. P. Rawat^a

^a THE Z. H. COLLEGE OF ENGINEERING AND TECHNOLOGY AND CHEMICAL LABORATORIES ALIGARH MUSLIM UNIVERSITY ALIGARH, U.P., INDIA

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NOTE

Paper Chromatographic and Electrochromatographic Separation of EDTA Complexes of Metal Ions

MOHSIN QURESHI and J. P. RAWAT

THE Z. H. COLLEGE OF ENGINEERING AND TECHNOLOGY AND CHEMICAL LABORATORIES
ALIGARH MUSLIM UNIVERSITY
ALIGARH, U.P., INDIA

Summary

R_F values of EDTA complexes of some common metal ions in seven solvent systems are given. The separation of Al and Be EDTA complexes has been achieved in different ratios by paper chromatography. Paper electrophoresis is also performed in four solvent systems to study the separation of EDTA complexes of metal ions.

INTRODUCTION

In recent years a number of new aminopolycarboxylic acids which have the special property of forming water-soluble chelates have come into widespread use, the most important of which are the alkali metal salts of EDTA. However, the use of EDTA in chromatography has not been studied systematically. Pickering studied the role of complex formation in paper chromatography using alcohol-aqueous EDTA solvent systems (1). He found that the pH of a solution and the concentration of EDTA is an important factor in determining the R_F values of an ion. Elbeigh used a antipyrine-EDTA-dioxane solvent system to separate numerous metal ions (2). Hill-Cottingham studied the separation of complexes of Fe with EDTA and with other aminopolycarboxylic acids in phenol-water and in butanol-acetic acid water solvent systems (3). No effort has been made to separate the EDTA complexes of metal ions. The present work was therefore undertaken

to study the separation of EDTA complexes of metal ions, and to study the effect of EDTA concentration on R_F values.

EXPERIMENTAL

Apparatus and Reagents

Whatman No. 1 filter paper strips 3×20 cm were used. Development was performed in 25×5 cm glass jars using the ascending technique. EDTA complexes were prepared by reported methods (4-6) and were dissolved in deionized water. All other chemicals were of reagent grade.

Detectors

Iron and copper were detected by $HCl + K_4Fe(CN)_6$; aluminum and beryllium by aluminon, cobalt by α -nitroso- β -naphthol, nickel by first keeping over HCl and then dipping in dimethylglyoxime and then over ammonia; manganese by ammonical silver nitrate; zinc by dithizone; chromium by heating the paper in an oven to $90^\circ C$; silver, lead and bismuth by H_2S ; calcium and strontium by alizarine Red-S; barium by sodium rhodizonate; magnesium by megnesone; and cadmium by cadion 2-B.

Procedure

One or two spots of nearly 0.1 M solutions of EDTA complexes of metal ions were applied on the paper. Conditioning was usually done for $1/2$ hr and the developer was allowed to ascend 16 cm in all cases.

RESULTS

Numerous solvent systems were tried to achieve the separation of aluminum and beryllium EDTA complexes. Beryllium was separated from Th, Ni, Zr, Ce, Ag, Co, Al, Ca, Sr, Zn, Ba, Cd, Pb, Cu, Mg, and Bi in water saturated with *n*-butanol + ammonia (40:1), in which Be has zero R_F value and all others move. Various ratios of complexes of Al and Be were tried and it was found that these complexes can be separated if present in the ratio 1:20 and 20:1. Various other solvent systems of such type, where there was no fear

TABLE 1
R_F Values of EDTA Complexes

Solvent systems	Complexes of					
	Al	Fe	Cu	Co	Mn	Zn
1. Alcohol + dioxane + acetone + water (1:1:1:1)	0.42	0.44	0.20	0.18	0.3T ^a	0.23
2. Alcohol + dioxane + water (1:1:1)	0.18	0.56	0.46	0.50	0.46T ^a	0.51
3. Dioxane + water						
(a) (1:1)	0.47T ^a	0.80	0.75	0.85	0.43	0.76
(b) (2:1)	0.63	0.45	0.34	0.38	0.23T ^a	0.22
4. 33% Methylamine	0.68	0.02	0.14	0.68T ^a	0.00	0.84
5. Tetrahydrofuran + dioxane + water (1:1:1)	0.19	0.34	0.40	0.36	—	—
6. Pyridine + water						
(a) (2:1)	0.35	0.40	0.59	0.65	0.22T ^a	0.40
(b) (3:2)	0.62	0.72	0.78	0.72	0.40T ^a	0.83
(c) (1:1)	0.50	0.37	0.75	0.77	—	0.76
7. Tetrahydrofuran + water (2:1)	0.26	0.47	0.33	0.30	0.26T	0.30T ^a

^a T = Trailing.

of breaking the complexes, were tried to study the separation of Al, Fe, Cu, Co, Mn, and Zn. Useful results are given in Table 1.

Paper electrophoresis was also performed in some solvent systems to study the separation of EDTA complexes of metal ions. The results are summarized in Table 2.

DISCUSSION

The separation of EDTA complexes of metal ions is important since EDTA is extensively used as a complexing agent. The paper chromatography of EDTA complexes proved very difficult and posed many problems owing to the following reasons:

(a) EDTA complexes are very stable and therefore very difficult to detect. Special methods had to be developed for the detection of these complexes.

(b) EDTA complexes are not stable in acidic media, hence either neutral or alkaline media were used.

(c) EDTA complexes are soluble only in waterlike systems, hence any nonpolar solvents cannot be used or can be used only in mixtures with water.

TABLE 2
Migration of EDTA Complexes

Electrolyte	Complexes of						Time of passing current (hr)
	Al (cm)	Fe (cm)	Cu (cm)	Co (cm)	Mn (cm)	Zn (cm)	
Water	+3	+3	+3	+3	+3	+3	4.5
1% ammonium carbonate	+9.5	+10.5	+9.0	+8.5	zero	+8.5 -2.5	6.0
1% Sodium- potassium tartrate	+7.4	+6.0	+6.5	+7.0	0.0	+6.5 +6.0	4
5% acetic acid + 2% diox- ane (1:1)	+12.5	+0.50	+0.70	0.00	+12.0 -12.0	0.00 -8.0	4.5

The results show that some important and selective separations can be developed in addition to these described here. Separation of Be from numerous metal ions is an important separation. From electrophoresis studies it appears that Mn can be separated from, Al, Zn, Co, Cu, and Fe.

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