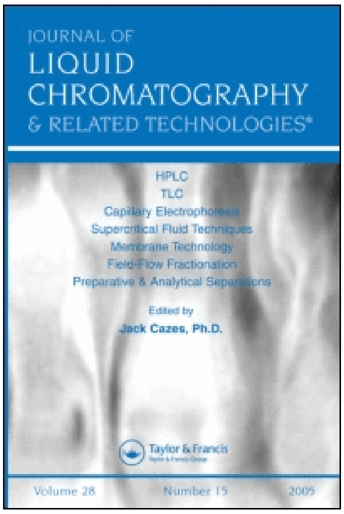

Journal of Liquid Chromatography & Related Technologies
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597273

Synthetic Inorganic Ion-Exchangers. XX. Thin Layer Chromatography of Metal Ions on Lanthanum Antimonate. Quantitative Separation of Hg(II) from Several Metal Ions

Anil K. De[a]; Punyabrata Chakraborty[a]
[a] Department of Chemistry, Visva Bharati, Santiniketan, West Bengal, India

To cite this Article De, Anil K. and Chakraborty, Punyabrata(1981) 'Synthetic Inorganic Ion-Exchangers. XX. Thin Layer Chromatography of Metal Ions on Lanthanum Antimonate. Quantitative Separation of Hg(II) from Several Metal Ions', Journal of Liquid Chromatography & Related Technologies, 4: 12, 2223 — 2228

To link to this Article: DOI: 10.1080/01483918108066855

URL: http://dx.doi.org/10.1080/01483918108066855

JOURNAL OF LIQUID CHROMATOGRAPHY, 4(12), 2223-2228 (1981)

SYNTHETIC INORGANIC ION-EXCHANGERS. XX. THIN LAYER CHROMATOGRAPHY OF METAL IONS ON LANTHANUM ANTIMONATE. QUANTITATIVE SEPARATION OF Hg (II) FROM SEVERAL METAL IONS.

Anil K. De and Punyabrata Chakraborty
Department of Chemistry, Visva Bharati, Santiniketan,
West Bengal, India.

ABSTRACT

The analytical potential of Lanthanum antimonate as an ion exchanger has been explored by thin layer chromatographic (TLC) technique. Binder-free thin layers of Lanthanum antimonate have been explored for several important binary and ternary separations. A TLC method has been developed for quantitative separation of microgram quantities of Hg(II) from several metal ions by using 1,4 dioxane as solvent.

INTRODUCTION

Thin layer chromatography is being used in recent years for inorganic analysis (1). In continuation of our work on TLC studies on thorium phosphate, tungstate, antimonate, ziroconium tungstate (2-4), we report in this paper systematic investigations on TLC behaviour of several metal ions on binder-free thin-layer plates of lanthanum antimonate. Based on studies in HNO_3 (pH-1,2 and 3), butanol, 1,4 dioxane, 1,4 dioxane - HNO_3, some important binary and ternary separations have been achieved. A quantitative method for the separation of Hg(II) from numerous metal ions is recommended.

EXPERIMENTAL

Apparatus

Thin layers of Lanthanum antimonate were prepared on glass plates (20 x 3 cm), which were subsequently developed in several solvent systems in jars (25 x 7 cm). For spectrophotometric studies, Spectrophotometer G.S. 865 B of Electronics Corporation of India, Hyderabad, India was used.

Reagents

Chemicals and solvents used in this work were of analytical grade (B.D.H / E.Merck / Pfizer)

Preparation of the Ion-Exchange Materials on thin layer plates.

The ion exchanger, lanthanum antimonate (Sb:La=4.298) was prepared according to the procedure described in earlier paper (5). Each material was then powdered separately and slurried with a little demineralized water in a mortar. It was then spread over the glass with the help of an applicator. Almost uniformly thin layers (0.1 mm thickness) were obtained. The plates were dried and ready for use. These plates gave reproducible R_F values.

Test Solutions and Detection Reagents

The test solutions in general, had a metal concentration of 4 mg/ml (chloride/nitrate/sulphate) Standard spot test reagents were used for detection (6).

Solvent System

The following solvent systems were used in these studies.

1. HNO_3 solution (pH-1,2,3)
2. Butanol
3. 1,4 Dioxane
4. 1,4 Dioxane: 0.1 M HNO_3 (8:2).

PROCEDURE

One or two drops of the test solution were placed on the plates with thin glass capillaries. The spots were allowed to dry and developed in different solvent systems. In each case the solvent was allowed to rise 11 cm. R_T and R_L values were measured as usual after detection.

For quantitative work, a stock solution of Hg(II) (5.1 mg/ml) was prepared by dissolving $HgCl_2$ in 0.1 M HCl. The known amount of synthetic mixture containing Hg was applied with the help of micro pipette on the line of application. The plates were developed in dioxane system. A pilot plate was run simultaneously to locate the position of Hg by detecting it with yellow ammonium sulphide. The area corresponding to Hg was scratched from the working plate and the mass was extracted with 10 ml 1 M H_2SO_4. The suspended particles of the exchanger were filtered off. The filtrate was collected and Hg(II) was determined spectrophotometrically by dithizone method(8).

RESULTS AND DISCUSSION

The results of our TLC studies reveal that most of the metal ions have appreciable R_F values in nitric acid system. The general trend in R_F values is that these values decrease with increase in

TABLE - I

Binary and Ternary Separations on Lanthanum Antimonate Thin Layer

Solvent system.	Separations achieved R_T - R_L	Time (hours)
0.1M HNO_3	Fe^{3+}(0.0-0-30) - Pt^{4+}(0.72 - 0.90)	2 hrs
	Fe^{3+}(0.0-0.21) - Ni^{2+}(0.62 - 0.85)	
	Fe^{3+}(0.0-0.21) - Au^{3+}(0.78 - 0.95)	
	Ce^{3+}(0.0-0.0) - Au^{3+}(0.85 - 0.94)	
	Ce^{3+}(0.0-0.0) - Hg^{2+}(0.83 - 0.93)	
	Ce^{3+}(0.0-0.0) - Pt^{4+}(0.80 - 0.83)	
	Ce^{3+}(0.0 - 0.0)- Mn^{2+}(0.78 - 0.90)	
	Bi^{3+}(0.0-0.0) - Hg^{2+}(0.89 - 0.96)	
	Bi^{3+}(0.0-0.0) - Au^{3+}(0.82 - 0.95)	
	Bi^{3+}(0.0-0.0) - Ni^{2+}(0.76 - 0.90)	
	Bi^{3+}(0.0-0.0) - Pt^{4+}(0.30 - 1.0)	
	Bi^{3+}(0.0-0.0) - Mn^{2+}(0.71 - 0.90)	
	Cu^{2+}(0.0-0.21) - Au^{3+}(0.68 - 0.84)	
	Cu^{2+}(0.0-0.14) - Hg^{2+}(0.74 - 0.86)	

TABLE 1 (Continued)

Solvent system	Separations achieved R_T - R_L	Time (hours)
$0.01MHNO_3$	Co^{2+}(0.0-0.0) - Pt^{4+}(0.75 - 0.85)	
	Co^{2+}(0.0-0.0) - Ni^{2+}(0.72 - 0.84)	2.5 hrs.
	Co^{2+}(0.0-0.0) - Hg^{2+}(0.85 - 0.92)	
	Co^{2+}(0.0-0.0) - Au^{3+}(0.85 - 0.90)	
	Pb^{2+}(0.0-0.0) - Ni^{2+}(0.80 - 0.89)	
	Pb^{2+}(0.0-0.0) - Pt^{4+}(0.80 - 0.91)	
	Pb^{2+}(0.0-0.0) - Au^{3+}(0.81 - 0.90)	
	Th^{4+}(0.0-0.0) - Hg^{2+}(0.82 - 0.90)	
	Th^{4+}(0.0-0.0) - Pt^{4+}(0.78 - 0.91)	
	Th^{4+}(0.0-0.0) - Au^{3+}(0.80 - 0.92)	
$0.001MHNO_3$	UO_2^{2+}(0.0-0.0) - Hg^{2+}(0.84 - 0.96)	
	UO_2^{2+}(0.0-0.0) - Au^{3+}(0.86 - 0.95)	3 hrs
	UO_2^{2+}(0.0-0.0) - Pt^{4+}(0.78 - 0.98)	
	Ni^{2+}(0.0-0.35) - Hg^{2+}(0.82 - 0.95)	
	Ni^{2+}(0.0-0.25) - Pt^{4+}(0.85 - 0.97)	
Butanol	Zn^{2+}(0.0-0.0) - Au^{3+}(0.71 - 0.85)	10 hrs.
	Al^{3+}(0.0-0.0) - Au^{3+}(0.80 - 0.90)	
	Ni^{2+}(0.0-0.0) - Au^{3+}(0.84 - 0.94)	
	Mn^{2+}(0.0-0.0) - Au^{3+}(0.81 - 0.90)	
	UO_2^{2+}(0.0-0.0) - Ru^{3+}(0.35 - 0.42) -	
	Au^{3+}(0.75 - 0.82)	
	Cu^{2+}(0.0-0.0) - Ru^{3+}(0.26 - 0.35) -	
	Au^{3+}(0.82 - 0.94)	
	Pb^{2+}(0.0-0.0) - Ru^{3+}(0.24 - 0.36) -	
	Au^{3+}(0.81 - 0.92)	
	Bi^{3+}(0.0-0.0) - Ru^{3+}(0.27 - 0.36) -	
	Au^{3+}(0.84 - 0.96)	
1,4 Dioxane :$0.1MHNO_3$ (8:2)	Fe^{3+}(0.0-0.1) - Hg^{2+}(0.85 - 1.0)	8 hrs.
	Pb^{2+}(0.0-0.0) - Hg^{2+}(0.85 - 0.95)	

TABLE - 2

Quantitative Separation of Hg^{2+} from Binary Mixtures

Sl. No.	Mixture taken	Other metal ion added (μg)	Hg^{2+} added (μg)	Hg^{2+} recovered (μg)	Percentage of error
1.	Cu^{2+}-Hg^{2+}	Cu^{2+}(7.8)	10.3	10.4	+ 1.0
2.	Cu^{2+}-Hg^{2+}	Cu^{2+}(3.9)	5.1	5.2	+ 2.0
3.	Pb^{2+}-Hg^{2+}	Pb^{2+}(14.2)	10.3	10.6	+ 3.0
4.	Pb^{2+}-Hg^{2+}	Pb^{2+}(7.1)	5.1	5.4	+ 6.0
5.	Cd^{2+}-Hg^{2+}	Cd^{2+}(14.2)	10.3	10.1	- 2.0
6.	Cd^{2+}-Hg^{2+}	Cd^{2+}(7.1)	5.1	5.2	+ 2.0
7.	Mn^{2+}-Hg^{2+}	Mn^{2+}(7.2)	10.3	10.7	+ 4.0
8.	Mn^{2+}-Hg^{2+}	Mn^{2+}(3.6)	5.1	4.9	- 4.0
9.	Co^{2+}-Hg^{2+}	Co^{2+}(7.6)	10.3	10.0	- 3.0
10.	Co^{2+}-Hg^{2+}	Co^{2+}(3.8)	5.1	5.3	+ 4.0
11.	Zn^{2+}-Hg^{2+}	Zn^{2+}(8.5)	10.3	10.8	+ 5.0
12.	Zn^{2+}-Hg^{2+}	Zn^{2+}(4.3)	5.1	4.9	- 4.0
13.	Fe^{3+}-Hg^{2+}	Fe^{3+}(7.3)	10.3	10.5	+ 2.0
14.	Fe^{3+}-Hg^{2+}	Fe^{3+}(3.7)	5.1	5.2	+ 2.0
15.	Bi^{3+}-Hg^{2+}	Bi^{3+}(13.5)	10.3	10.5	+ 2.0
16.	Bi^{3+}-Hg^{2+}	Bi^{3+}(6.8)	5.1	5.3	+ 4.0
17.	UO_2^{2+}-Hg^{2+}	U^{6+}(14.6)	10.3	10.8	+ 5.0
18.	UO_2^{2+}-Hg^{2+}	U^{6+}(7.3)	5.1	5.3	+ 4.0

pH in HNO_3 system which is a characteristic feature of ion exchange operation. In pure 1,4 dioxane system most of the metal ions except Hg(II) are retained at the base line. This permits quantitative separations of Hg(II) from other metal ions. Tables 1 and 2 show some useful and important binary and ternary separations of metal ions achieved in different solvents.

ACKNOWLEDGEMENT

One of the authors (P.B.C) is grateful to the University Grants Commission, New Delhi for the award of a Junior Research Fellowship.

REFERENCES

1. Brinkman, U.A.T., De, Varies. G and Kuroda, R., J.Chromatog. 85, 187, 1973.
2. De, A.K., Rajput, R.P.S., Das. S.K. and Chowdhury. N.D., J.Liquid Chromatog 2, 117, 1979.
3. De, A.K. and Pal, B.K., Sep. Sci. Tech., 15, 1271, 1980
4. De, A.K., Rajput, R.P.S. and Das. S.K., Sep. Sci, Tech. 14, 735,1979.
5. De, A.K. and Chakraborty, P., Chromatographia (Communicated)
6. Feigl, F., Spot tests in Inorganic Analysis, 5th edition, Elsevier, Amsterdam, 1958.
7. Sandell, E.B., Colorimetric Determination of Traces of Metals, 3rd. edition, Interscience, New York, 1959.