Separation Science and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

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

A. K. De[a]; R. P. S. Rajput[ab]; S. K. Das[a]
[a] DEPARTMENT OF CHEMISTRY, VISVA-BHARATI SANTINIKETAN, WEST BENGAL, INDIA [b] Chemistry Department, Bareilly College, Bareilly, U.P.

To cite this Article De, A. K. , Rajput, R. P. S. and Das, S. K.(1979) 'Synthetic Inorganic Ion Exchangers. XIII. Thin-Layer Chromatography of Metal Ions on Thorium Antimonate: Quantitative Separation of Hg(II) from Several Metal Ions', Separation Science and Technology, 14: 8, 735 — 739

To link to this Article: DOI: 10.1080/01496397908060233

URL: http://dx.doi.org/10.1080/01496397908060233

SEPARATION SCIENCE AND TECHNOLOGY, 14(8), pp. 735–739, 1979

NOTE

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

---

A. K. DE, R. P. S. RAJPUT,* AND S. K. DAS

DEPARTMENT OF CHEMISTRY
VISVA-BHARATI
SANTINIKETAN 731235, WEST BENGAL, INDIA

Abstract

The analytical potential of thorium(IV) antimonate as an ion exchanger has been explored by the thin-layer chromatographic technique. Binder-free thin layers of thorium antimonate have been prepared, and several important binary and ternary separations have been carried out. It has also been possible to separate microgram quantities of Hg(II) quantitatively from several metal ions in a dioxane solvent system.

INTRODUCTION

Thin-layer chromatography is now being used for inorganic analysis (*1*). In this communication we report the results of systematic studies of thin-layer chromatography of metal ions on binder-free thin layers of thorium antimonate. Both aqueous nitric acid and mixed aqueous-organic solvents containing dimethyl sulfoxide (DMSO) and dioxane have been used. We have been successful in achieving some useful and important binary and ternary separations of metal ions. A quantitative method has been developed for separation of microgram amounts of Hg(II) from various metal ions.

*Permanent address: Chemistry Department, Bareilly College, Bareilly, U.P.

EXPERIMENTAL

Apparatus

Thin layers of thorium antimonate were prepared on glass plates (20 × 3 cm) which were subsequently developed in several solvent systems in glass jars (25 × 7 cm).

Reagents

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

Perparation of the Ion-Exchange Materials and Thin-Layer Plates

The ion exchanger, thorium antimonate, was prepared according to the procedure described earlier (*2*). Each material was then separately powdered and slurried with a little demineralized water in a mortar. It was then spread over the glass plates with the help of an applicator. Almost uniform thin layers (~0.1 mm thickness) were obtained which were ready for use after drying in air. The plates gave reproducible R_F values.

Test Solutions and Detection Reagents

In general, the test solutions had metal ion concentrations of 4 mg/ml (chloride/nitrate/sulfate). Standard spot test reagents were used for detection (*3*).

Solvent Systems

The following solvent systems were used: HNO_3 solution (1 *M*; pH 1, 2, 3, 5); CH_3COOH (0.1 and 1 *M*); DMSO; DMSO:0.1 *M* HNO_3 (8:2, 6:4, 4:6, and 2:8); dioxane; dioxane: 0.1 *M* HNO_3 (8:2, 6:4, 4:6, and 2:8).

Procedure

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

For quantitatives work, a stock solution of Hg(II) (~5500 ppm Hg) was prepared by dissolving $HgCl_2$ in 0.05 *M* HCl. A known amount of the synthetic mixture containing Hg was applied with the help of a micropipette on the line of application. The plates were developed in a dioxane system. A pilot plate was run simultaneously to locate the position of Hg by detecting it with a suitable spraying reagent. The area corresponding to Hg was scratched from the working plate and the mass was eluted with small portions of 0.5 *M* H_2SO_4. The suspended particles of the exchanger were filtered off and the solution was diluted to 20 ml with 0.5 *M* H_2SO_4. Hg(II) was determined spectrophotometrically by the dithizone method (*4*).

RESULTS AND DISCUSSION

The results of our thin layer chromatographic studies reveal that in most of the solvent systems used, several metal ions move up from the point of application and have appreciable R_F values. Among 28 metal ions selected, most of the ions have high R_F values both in aqueous HNO_3 and in mixed systems containing DMSO or dioxane. The R_F values decrease with an increase in pH in HNO_3 systems, which is a characteristic feature of an ion-exchange operation. In pure dioxane, none of the metal ions except Hg(II) moves to the top of the solvent front, which permits quantitative separation of Hg(II) from other metal ions. However, with the addition of HNO_3 to both DMSO and dioxane the metal ions start moving and their R_F values increase with an increased percentage of HNO_3.

Tables 1 and 2 show some useful and important binary and ternary separations of metal ions achieved in different solvent systems. The notable separations include Pb–Cd–Hg, Cu–Cd–Bi, Bi–Hg–Pd, Pb–Hg–Zn, Pd–Pt–Au, Zn–Cd, Ce–La, and Cu–Cd.

The thin layers of thorium antimonate are quite stable and firm, capable of withstanding the solvent systems and the chemical operations.

Acknowledgments

One of the authors (R.P.S.R.) is grateful to UGC (India) for awarding a National Associateship which enabled him to work at Visva-Bharati.

TABLE 1

Separation Achieved on Th(IV) Antimonate Thin-Layer

Solvent system	Separation achieved ($R_T - R_L$)	Time (hr)
0.1 *M* HNO_3	Pb^{2+}(0.00–0.00)–Cd^{2+}(0.30–0.45)–Hg^{2+}(0.58–0.84)	2
	Bi^{3+}(0.00–0.00)–Cd^{2+}(0.35–0.50)–Hg^{2+}(0.65–0.85)	
	Ag^{2+}(0.00–0.00)–Cd^{2+}(0.23–0.41)–Cu^{2+}(0.80–0.90)	
	Bi^{3+}(0.00–0.00)–Cd^{2+}(0.30–0.45)–Cu^{2+}(0.75–0.87)	
	Cd^{2+}(0.25–0.45)–Zn^{2+}(0.55–0.75)	
	Fe^{3+}(0.00–0.00)–Mn^{2+}(0.70–0.80)	
1 *M* HNO_3	Ce^{4+}(0.00–0.00)–La^{3+}(0.60–0.86)–Pr^{3+}(0.62–0.84)–Sm^{3+}(0.60–0.88)	2
0.1 *M* CH_3COOH	Al^{3+}(0.00–0.00)–Mg^{2+}(0.90–0.96)	1
	Cr^{3+}(0.00–0.00)–Zn^{2+}(0.80–0.90)	
	Cr^{3+}(0.00–0.28)–Mn^{2+}(0.70–0.90)	
	Fe^{3+}(0.00–0.00)–Co^{2+}(0.80–0.94)–Ni^{2+}(0.84–0.96)	
1 *M* CH_3COOH	Bi^{3+}(0.00–0.00)–Pd^{2+}(0.20–0.52)–Hg^{2+}(0.70–0.80)	1
	Ag^{+}(0.00–0.00)–Pd^{2+}(0.22–0.52)–Hg^{2+}(0.70–0.88)	
	Pb^{2+}(0.00–0.00)–Pd^{2+}(0.22–0.50)–Hg^{2+}(0.70–0.90)	
DMSO + 0.1 *M* HNO_3 (4:6)	Pb^{2+}(0.00–0.00)–Hg^{2+}(0.25–0.38)–Zn^{2+}(0.65–0.98)	6
	Ag^{+}(0.00–0.00)–Hg^{2+}(0.30–0.40)–Zn^{2+}(0.70–0.90)	
	Bi^{3+}(0.00–0.20)–Hg^{2+}(0.30–0.42)–Zn^{2+}(0.68–0.94)	
	Fe^{3+}(0.00–0.00)–U^{2+}(0.90–1.00)	
	Fe^{2+}(0.00–0.00)–V^{2+}(0.88–1.00)	
Dioxane	Cu^{2+}(0.00–0.00)–Hg^{2+}(0.75–0.85)	4
	Co^{2+}(0.00–0.00)–Hg^{2+}(0.75–0.85)	

TABLE 2

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

No.	Mixture taken	Amount of Hg applied (μg)	Amount of other ion applied (μg)	Amount of Hg found (μg)	Percentage of error
1	Hg^{2+}–Cu^{2+}	5.5	Cu (5.9)	5.35	−2.7
2	Hg^{2+}–Cu^{2+}	13.75	Cu (14.75)	13.55	−1.5
3	Hg^{2+}–Cd^{2+}	5.5	Cd (5.3)	5.30	3.7
4	Hg^{2+}–Cd^{2+}	11.0	Cd (10.6)	11.2	+1.8
5	Hg^{2+}–Pb^{2+}	5.5	Pb (5.1)	5.65	+2.7
6	Hg^{2+}–Pb^{2+}	11.0	Pb (10.2)	11.2	+1.8
7	Hg^{2+}–Bi^{3+}	13.75	Bi (14.25)	13.60	−1.2
8	Hg^{2+}–Zn^{2+}	5.5	Zn (6.6)	5.70	+3.7
9	Hg^{2+}–Zn^{2+}	11.0	Zn (13.2)	10.75	−2.2
10	Hg^{2+}–Zn^{2+}	13.75	Zn (16.5)	13.45	−2.1
11	Hg^{2+}–Co^{2+}	5.5	Co (5.0)	5.72	+4.0
12	Hg^{2+}–Co^{2+}	13.75	Co (12.5)	13.5	−1.8

One of the other authors (S.D.) is grateful to C.S.I.R. (India) for the award of a Senior Fellowship.

REFERENCES

1. U. A. T. Brinkman, G. de Vries, and R. Kuroda, *J. Chromatogr.*, *85*, 187 (1973).
2. A. K. De and S. K. Das, *Chromatographia*, *6*, 350 (1978).
3. F. Feigel, *Spot Tests in Inorganic Analysis*, 5th ed., Elsevier, Amsterdam, 1958.
4. E. B. Sandell, *Colorimetric Determination of Traces of Metals*, 3rd ed., Interscience, New York, 1959.

Received by editor January 26, 1979