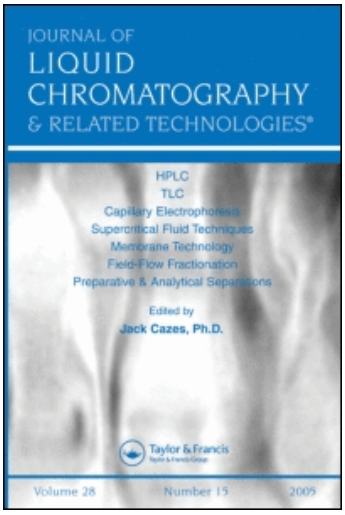

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Synthetic Inorganic Ion-Exchangers XV. Thin-Layer Chromatography of Metal Ions on Thorium Tungstate: Quantitative Separation of Hg(II) from Several Other Metal Ions

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SYNTHETIC INORGANIC ION-EXCHANGERS XV. THIN-LAYER CHROMATOGRAPHY OF METAL IONS ON THORIUM TUNGSTATE: QUANTITATIVE SEPARATION OF Hg(II) FROM SEVERAL OTHER METAL IONS.

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ABSTRACT

The analytical application of Thorium tungstate as an ion exchanger has been explored by thin-layer chromatographic (TLC) technique. Binder-free thin layers of thorium tungstate have been employed for some important binary and ternary separations. Quantitative separation of Hg(II) from the mixture of several other metal ions has been achieved.

INTRODUCTION

In recent years thin-layer chromatography has become popular as an analytical technique for inorganic analysis. The bulk of the reported work is based on silica gel only (1). In continuation of our previous work on thorium phosphate and antimonate (2,3) we report in this paper the preparation of binder-free thin layers of thorium tungstate and its analytical applications. The TLC behaviour in several solvents - nitric acid (at different concentrations), 1,4 - dioxane, butanol, isobutyl

methyl ketone-tetrahydrofuran-nitric acid have been used and several important separations worked out. A quantitative separation scheme of microgramme amounts Hg(II) from the mixture of several other metal ions has been devised.

EXPERIMENTAL

Apparatus:

Thin layers of thorium tungstate were prepared on glass plates (20 x 3 cm.) which were subsequently developed in several solvent systems in glass jars (25 x 7 cm.). Spectrophotometric studies were performed using Electronic Corporation of India Model GS866B Spectrophotometer.

Reagents:

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

Preparation of Ion-exchange materials on thin-layer plates:

Thorium tungstate (Th:W = 1:6.2) in the H^+ form, prepared according to the procedure described earlier (4), was powdered and slurried with a little demineralized water in a mortar. The thin layers were prepared over the glass plates as described in the earlier paper (2).

Test solutions and detection reagents:

The test solutions in general had metal ion concentrations of 4mg/ml (Chloride/Nitrate/Sulphate). Standard spot test reagents were used for detection (5).

Solvent System:

1. HNO_3 solution : (pH 1,2,3).
2. 1,4-Dioxane: 0.1M HNO_3 (100:0, 80:20, 20:80)
3. Butanol

4. Isobutyl methyl ketone : Tetrahydrofuran : 1M HNO_3 (1:8:1).

DMSO and mixed Butanol-6M HNO_3 were avoided as the thin layers split off the plates in these solvents.

PROCEDURE

One or two drops of the test solutions were placed on the plates with thin glass capillaries. After drying the spots development was made in different solvents and the ascent was fixed as 11 cm. for HNO_3 and 8 cm. in case of solvent mixtures. R_T and R_L values were measured as usual after detection. For quantitative work, a stock solution of Hg(II) [4800 µg/ml of Hg(II)] was prepared by dissolving $HgCl_2$ in 0.05M HCl. Synthetic mixtures containing known amount of Hg(II) was applied with the help of a micropipette on the line of application. A pilot plate was run simultaneously to locate the position of Hg(II) by detecting it with the help of dithizone. The area corresponding to Hg was than scratched off from the working plate and the mass was eluted with 1M H_2SO_4. The suspended particles of the exchanger were then filtered off. The filtrate was diluted with 0.05M H_2SO_4 and Hg(II) estimated spectrophotometrically by dithizone method (6).

RESULTS AND DISCUSSION

The results of our TLC studies reveal that whereas several metal ions move on thorium tungstate thin layer in HNO_3 system only a few move in organic solvents. The general trend in R_F values is that the values decrease with increase in pH in HNO_3 system which indicates ion exchange process. Hg(II) and Au(III) show high R_F values in all the solvents studied, presumably due to their anionic nature. In pure butanol and

TABLE-1

Binary and Ternary Separations Achieved on Thorium Tungstate Thin Layer.

Solvent system	Separation achieved ($R_T - R_L$)	Time (hours)
1. pH 1	1. Pb^{2+}(0.0-0.0)-Cu^{2+}(0.50-0.55)	2.5 hrs.
	2. Pb^{2+}(0.0-0.0)-Cd^{2+}(0.65-0.73)	
	3. Pb^{2+}(0.0-0.0)-Zn^{2+}(0.65-0.80)	
	4. Fe^{3+}(0.0-0.20)-Au^{3+}(0.70-0.80)	
	5. Fe^{3+}(0.0-0.25)-Co^{2+}(0.65-0.72)	
	6. Pb^{2+}(0.0-0.0)-Cu^{2+}(0.45-0.55) -Hg^{2+}(0.80-0.85)	
	7. Pb^{2+}(0.0-0.0)-Cu^{2+}(0.50-0.55) -Cd^{2+}(0.65-0.75)	
	8. Ag^{+}(0.0-0.0)-Cu^{2+}(0.50-0.60) -Au^{3+}(0.70-0.80)	
2. pH 2	1. Ag^{+}(0.0-0.0)-Pt^{4+}(0.60-0.70)	2 hrs.
	2. Pb^{2+}(0.0-0.0)-Pd^{2+}(0.65-0.75)	
	3. Mn^{2+}(0.0-0.20)-Bi^{3+}(0.50-0.70)	
3. IBMK:THF: 1M HNO_3 (1:8:1)	1. Pb^{2+}(0.0-0.0)-Bi^{3+}(0.55-0.70)	
	2. Fe^{3+}(0.0-0.0)-Bi^{3+}(0.60-0.70)	
	3. As^{3+}(0.0-0.0)-Bi^{3+}(0.50-0.70)	
	4. Pb^{2+}(0.0-0.0)-Bi^{3+}(0.55-0.65) -Hg^{2+}(0.85-1.00)	

(continued)

TABLE-1(Contd..)

Solvent system	Separation achieved ($R_T - R_L$)	Time (hours)
4. Butanol	1. Cu^{2+}(0.0-0.0)-Hg^{2+}(0.80-0.90)	6 hrs.
	2. Pb^{2+}(0.0-0.0)-Hg^{2+}(0.70-0.90)	
	3. Cd^{2+}(0.0-0.0)-Hg^{2+}(0.70-0.90)	
	4. Zn^{2+}(0.0-0.0)-Hg^{2+}(0.70-0.90)	
	5. Ag^{+}(0.0-0.0)-Hg^{2+}(0.70-0.90)	
	6. Bi^{3+}(0.0-0.20)-Hg^{2+}(0.65-0.95)	
	7. Tl^{+}(0.0-0.0)-Hg^{2+}(0.70-0.85)	
	8. VO_2^{2+}(0.0-0.0)-Au^{3+}(0.80-0.90)	
	9. Ag^{+}(0.0-0.0)-Au^{3+}(0.80-0.90)	
	10. Pt^{4+}(0.0-0.0)-Au^{3+}(0.75-0.85)	
	11. Cd^{2+}(0.0-0.0)-Au^{3+}(0.80-0.85)	
	12. Zn^{2+}(0.0-0.0)-Au^{3+}(0.75-0.85)	
	13. Bi^{3+}(0.0-0.15)-Au^{3+}(0.70-0.85)	
5. 1,4-Dioxane	1. Pb^{2+}(0.0-0.0)-Hg^{2+}(0.95-1.00)	4 hrs.
	2. Bi^{3+}(0.0-0.0)-Hg^{2+}(0.95-1.00)	
	3. Ag^{+}(0.0-0.0)-Hg^{2+}(0.90-0.96)	
	4. Co^{2+}(0.0-0.0)-Hg^{2+}(0.90-1.00)	
	5. Tl^{+}(0.0-0.0)-Hg^{2+}(0.95-1.00)	
	6. Cu^{2+}(0.0-0.0)-Hg^{2+}(0.92-0.96)	
	7. Ni^{2+}(0.0-0.0)-Hg^{2+}(0.90-0.95)	

TABLE-2

Quantitative Separation of Hg(II) from Several Mixtures Solvent : 1,4 Dioxane; Time : 4 hrs.

Sl.No.	Mixture taken	Amount of the other metal ion applied (µg)	Amount of Hg(II) (µg) Added	Found	% error
1.	-	-	13.75	13.25	-3.6%
2.	-	-	8.25	8.37	+1.4%
3.	Cd^{2+}- Hg^{2+}	Cd^{2+}(13.25)	13.75	13.67	-0.6%
4.	Cd^{2+}- Hg^{2+}	Cd (7.95)	8.25	8.12	-1.3%
5.	Cu^{2+}- Hg^{2+}	Cu^{2+}(14.85)	13.75	13.67	-0.6%
6.	Co^{2+}- Hg^{2+}	Co^{2+}(12.75)	13.75	13.0	-5.4%
7.	Co^{2+}- Hg^{2+}	Co^{2+}(7.65)	8.25	8.12	-1.5%
8.	Bi^{3+}- Hg^{2+}	Bi^{3+}(5.68)	5.5	5.5	-
9.	Ni^{2+}- Hg^{2+}	Ni^{2+}(12.40)	11.0	11.25	+2.3%
10.	Zn^{2+}- Hg^{2+}	Zn^{2+}(13.12)	11.0	11.25	+2.3%
11.	Zn^{2+}- Hg^{+}	Zn^{2+}(6.56)	5.5	5.75	+4.5%
12.	Zn^{2+}- Cd^{2+} -Hg^{2+}	Zn^{2+}(6.56) -Cd^{2+}(5.28)	11.0	10.67	-6.1%

1,4 Dioxane all the metal ions except Hg(II) and Au(III) are retained in the base line. This permits quantitative separation of Hg(II) from other metal ions.

Tables 1 and 2 show some binary and ternary separations of metal ions achieved in different solvents.

Some important separations are Pb-Cu, Pb-Cd, Pb-Zn, Ag-Cu-Au, Pb-Cu-Hg, Ag-Pt, UO_2-Au. The separations were found to improve in HNO_3 system.

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