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NOTE

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Abstract

Thin-layer chromatography of 23 metal ions in 10 aqueous and mixed solvent systems has been performed on zirconium tungstate ion-exchange material. Several important binary and ternary separations have been achieved. Quantitative separation of Hg(II) from Pb^{2+}, Zn^{2+}, Cd^{2+}, Bi^{3+}, Co^{2+}, and mixtures has been achieved using 1,4-dioxane as solvent.

INTRODUCTION

In continuation of our work on TLC studies on thorium phosphate, tungstate, and antimonate (*1–3*), in this paper we report systematic investigations on TLC behavior of several metal ions on binder-free thin-layer plates of zirconium tungstate. Based on studies in HNO_3, butanol, dioxane, isobutyl methyl ketone–tetrahydrofuran–HNO_3, some important binary and ternary separations have been achieved. A quantitative method for the separation of Hg(II) from numerous metal ions has been devised.

EXPERIMENTAL

Apparatus

Zirconium tungstate thin layers were prepared on glass plates (20 × 3 cm) which were subsequently developed in several solvent systems in glass jars. For spectrophotometric studies, EC Model GS 866 B India was used.

Reagents

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

Preparation of Ion-Exchange Materials on Thin-Layer Plates

The ion-exchanger, zirconium tungstate (Zr: W = 1: 1.12) in the H^+ form, was prepared according to the procedure described earlier (*4*). Each material was then powdered separately and slurried with a little demineralized water in a mortar. It was then spread over the glass plate 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/sulfate). Standard spot test reagents were used for detection (*5*).

Solvent Systems

1. 0.1 *M* HNO_3
2. 0.01 *M* HNO_3
3. 0.001 *M* HNO_3
4. 0.0001 *M* HNO_3
5. Butanol:6 *M* HNO_3 (1:1)
6. Butanol
7. Dioxane: 0.1 *M* HNO_3 (2:8)
8. Dioxane: 0.1 *M* HNO_3 (8:2)
9. Dioxane
10. IBMK:THF:1 *M* HNO_3 (1:8:1)

Procedure

For qualitative studies, one or two drops of the test solutions were placed on plates with thin glass capillaries. After drying the spots, development was made in different solvent systems. The ascent was fixed as 11 cm in HNO_3 and the dioxane–HNO_3 mixture, and for other cases the ascent was fixed at 8 cm. R_T and R_L values were measured after development of the spots (Table 1).

For quantitative work, a stock solution of mercuric nitrate (6500 μg/mL) was prepared and standardized (*6*). Synthetic mixtures of Hg(II) and other ions were applied with the help of a micropipette on the line of application. The plates were developed in 1,4-dioxane solution. A pilot plate was run simultaneously to locate the position of Hg(II) by detecting it with dithizone. The area corresponding to Hg(II) was scratched (*7*) from the working plate. The mass was eluted with 1 *M* H_2SO_4 and filtered. The filtrate was then diluted with 0.05 *M* H_2SO_4, and the amount of Hg(II) was measured spectrophotometrically by the dithizone method (*8*) (Table 2).

RESULTS AND DISCUSSION

The results of TLC studies reveal that most of the metal ions have appreciable R_F values in HNO_3, BuOH–HNO_3, dioxane–HNO_3, and IBMK–THF–HNO_3 mixtures. The general trend is decrease in R_F with increasing pH of the solution, which is the characteristic feature of an ion-exchange process. In pure butanol all the metal ions except Sb(V) are retained in the base line, which permits several binary separations of Sb(V) from other metal ions. Table 1 summarizes some important binary and ternary separations that are actually achieved with different solvents. The

TABLE 1

Binary and Ternary Separations Achieved on Zirconium Tungstate Thin Layer (the numbers in parentheses gives the $R_T - R_L$ values)

1. *Nitric acid 0.01 or 0.1 M*: Time, 1.5 hr; Ag^+, Tl^+, Pb^{2+}, Bi^{3+}(0.0) from Fe^{3+} (0.55–0.70), Au^{3+}(0.5–0.6), Cd^{2+}(0.7–0.8), Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}(0.75–0.95)
2. *1-Butanol*: Time, 8 hr; Au^{3+}, Zn^{2+}, Cd^{2+}, Tl^+, Bi^{3+}, UO^{2+}(0.0) from Sb^{2+} (0.8–0.95)
3. *1,4-Dioxane*: Time, 6 hr; Cu^{2+}, Zn^{2+}, Cd^{2+}, As^{3+}, Pb^{2+}(0.0) from Hg^{2+}, Sb^{5+} (0.95–1.00)
4. *1,4-Dioxane:0.1 M HNO_3 (8:2)*: Time, 3 hr; Ag^+(0.0) from Ni^{2+}(0.45–0.65), Au^{3+}(0.56–0.65)–Hg^{2+}(0.70–0.85); Pb^{2+}(0.0) from Cd^{2+}(0.30–0.40)–Hg^{2+} (0.70–0.85)
5. *1,4-Dioxane:0.1 M HNO_3 (2:8)*: Time, 2.0 hr; Ag^+(0.0) from Ni^{2+}(0.70–0.85); Bi^{3+}(0.0–0.1) from Hg^{2+}(0.50–0.60); Au^{3+}(0.35–0.50) from Mn^{2+}(0.70–0.80); Pb^{2+}(0.0) from Au^{3+}(0.40–0.50)–Cu^{2+}(0.70–0.85)
6. *IBMK:THF:1 M HNO_3 (1:8:1)*: Time, 8 hr; UO^{2+}(0.45–0.50) from Au^{3+} (0.95–1.00); As^{3+}(0.0) from Bi^{3+}(0.35–0.50), Bi^{3+}(0.35–0.50)–Sb^{5+}(0.90–0.95); Pb^{2+}(0.0) from Bi^{3+}(0.30–0.40)–Cu^{2+}(0.70–0.85); Ni^{2+}(0.20–0.30) from Mn^{2+} (0.65–0.75)–Co^{2+}(0.80–0.85); Rh^{3+}(0.0) from Pd^{2+}(0.60–0.80)–Au^{3+}(0.90–0.95)

TABLE 2

Quantitative Separation of Hg^{2+} from the Mixture of Other Metal Ions on Zirconium Tungstate Thin Layer

	Mixture taken	Amount of other metal ions applied (μg)	Amount of Hg^{2+} (μg) Added	Found	% error
	—	—	6.5	6.75	+3.84
	—	—	13.0	12.50	−3.85
1.	Hg^{2+}–Pb^{2+}	Pb^{2+}(5.1)	6.5	7.00	+7.75
2.	Hg^{2+}–Pb^{2+}	Pb^{2+}(10.2)	13.0	13.00	—
3.	Hg^{2+}–Zn^{2+}	Zn^{2+}(6.57)	6.5	6.37	−2.0
4.	Hg^{2+}–Zn^{2+}	Zn^{2+}(13.14)	13.0	13.00	—
5.	Hg^{2+}–Cd^{2+}	Cd^{2+}(5.3)	6.5	6.75	+3.84
6.	Hg^{2+}–Cd^{2+}	Cd^{2+}(10.6)	13.0	12.25	−5.77
7.	Hg^{2+}–Co^{2+}	Co^{2+}(10.2)	13.0	12.75	−1.95
8.	Hg^{2+}–Cu^{2+}	Cu^{2+}(5.94)	6.5	6.75	+3.84
9.	Hg^{2+}–Cu^{2+}	Cu^{2+}(11.88)	13.0	12.25	−5.77
10.	Hg^{2+}–Bi^{3+}	Bi^{3+}(11.35)	13.0	12.50	−3.85
11.	Hg^{2+}–Zn^{2+}–Cd^{2+}	Zn^{2+}(3.24)–Cd^{2+}(2.65)	6.5	6.25	−3.84
12.	Hg^{2+}–Zn^{2+}–Cd^{2+}	Zn^{2+}(6.48)–Cd^{2+}(5.3)	13.0	13.37	+2.8
13.	Hg^{2+}–Zn^{2+}–Cd^{2+}–Cu^{2+}–Bi^{3+}	Zn^{2+}(1.62)–Cd^{2+}(1.32)–Cu^{2+}(1.48)–Bi^{3+}(1.42)	6.5	7.0	+7.7
14.	Hg^{2+}–Zn^{2+}–Cd^{2+}–Cu^{2+}–Bi^{3+}	Zn^{2+}(3.24)–Cd^{2+}(2.64)–Cu^{2+}(2.96)–Bi^{3+}(2.84)	13.0	12.15	−6.54

noteworthy separations are Ag–Cu–Au, Pb–Cd, Pb–Cu, Bi–Fe, Zn–Hg, Tl–Pb–Cu, As–Pb–Bi, Tl–Pb–Hg, As–Sb–Bi, Ni–Mn–Co, Rh–Pd–Au, Ru–Au, and Ag–Au–Hg.

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Note Added in Proof. Table 2 shows the quantitative separation of Hg(II) from various mixtures using 1,4-dioxane as the solvent.