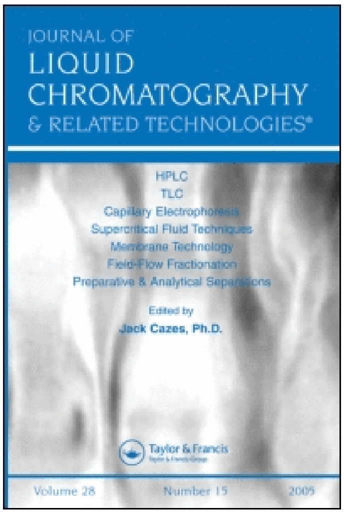

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SYNTHETIC INORGANIC ION EXCHANGERS. XIV THIN-LAYER CHROMATOGRAPHY OF METAL IONS ON THORIUM PHOSPHATE : QUANTITATIVE SEPARATION OF Au (III) FROM SEVERAL METAL IONS.

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ABSTRACT

The analytical potential of thorium phosphate as an ion exchanger has been explored by thin layer chromatographic (TLC) technique. Binder-free thin layers of thorium phosphate have been explored for several important binary and ternary separations. A TLC method has been developed for quantitative separation of microgram-quantities of Au(III) from several metal ions in pure butanol system.

INTRODUCTION

Thin layer chromatography is being used in recent years for inorganic analysis (I). Most of the reported work is based on silica gel only. Synthetic inorganic ion exchangers have not been properly explored for TLC technique. In most cases some binder, such as silica gel, starch or cellulose is used which obviously does not give the correct picture of the mechanism involved. Recently Qureshi and Co-workers (2-4) have prepared a few binder-free thin layers of inorganic exchangers - Sn(IV)-arsenate-antimonate-tungstate- and achieved some useful qualitative and quantitative separations of metal ions on the thin layers of these exchangers.

In continuation of our earlier work on TLC of thorium antimonate (5), we report, in this paper, the results of systematic studies of thin layer chromatography of metal ions on binder-free thin layers of thorium phosphate. Aqueous nitric acid, acetic acid and butanol systems have been used. Some useful and important binary and ternary separations of metal ions have been achieved

A quantitative method has been developed for separation of microgram amounts of Au(III) from various metal ions.

EXPERIMENTAL

Apparatus

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

Reagents

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

Preparation of the Ion Exchange Materials on thin layer plates.

The ion exchanger, thorium phosphate ($PO_4^{3-}:Th^{4+}=1.9$) was prepared according to the procedure described earlier (6). Each material was then separately powdered and slurried with a little demineralised water in a mortar. It was then spread over the glass plates with the help of an applicator: almost uniformly thin layers (~0.1 mm. thickness) were obtained, which were ready for use after drying in air. These plates gave reproducible R_F values.

Test solutions and Detection Reagents

The test solutions in general had metal ion concentrations of 4 mg/ml (chloride/nitrate/sulphate). Standard spot test reagents were used for detection (7).

Solvent systems

The following solvent systems were used in these studies.

1. HNO_3 solution (pH 1, 2, 3, 4, 5)
2. CH_3COOH (0.1M and 1M)
3. Butanol.

It was found that, in presence of DMSO and mixed butanol-HNO_3, the thin layers tended to split off the plates. Hence these solvents were avoided in our work.

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 Au(III) (~6600 ppm Au) was prepared by dissolving $HAuCl_4$ in 0.1M HCl. The known amount of synthetic mixture containing Au was applied with the help of a micro pipette on the line of application. The plates were developed in butanol system. A pilot plate was run simultaneously to locate the position of Au by detecting it with benzidine. The area corresponding to Au was scratched from the working plate and the mass was extracted with 5 ml. saturated NH_4Cl containing 0.5 ml. 6 M HCl. The suspended particles of the exchanger were filtered off and washed with 7.5 ml. water. The filtrate and the washings were collected and Au (III) was determined spectrophotometrically by Rhodamine B 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 and acetic acid systems. The general trend in R_F values is that these values

TABLE -1

Binary separations achieved on Th(IV) Phosphate Thin-layer.

Solvent System	Separations achieved R_L-R_T	Time (hours)
0.1M HNO_3	Pb^{2+}(0.0-0.2)-Co^{2+}(0.85-0.94)	2
	Pb^{2+}(0.0-0.2)-Zn^{2+}(0.85-1.0)	
	Pb^{2+}(0.0-0.15)-Hg^{2+}(0.75-0.9)	
	Pt^{4+}(0.1-0.2)-Cu^{2+}(0.94-0.96)	
	Pt^{4+}(0.15-0.23)-Hg^{2+}(0.8-0.9)	
	Bi^{3+}(0.0-0.07)-Hg^{2+}(0.95-1.0)	
	Pd^{2+}(0.25-0.5)-Hg^{2+}(0.90-1.0)	
	Fe^{3+}(0.0-0.0) - Zn^{2+}(0.84-1.0)	
	Fe^{3+}(0.0-0.0) - Ni^{2+}(0.92-0.95)	
	Fe^{3+}(0.0-0.0) - Co^{2+}(0.8-0.91)	
	Fe^{3+}(0.0-0.0) - Mn^{2+}(0.87-0.91)	
	Uo_2^{2+}(0.0-0.0) - Au^{3+}(0.56-0.82)	
0.01M HNO_3	VO^{2+}(0.0-0.2)-Cu^{2+}(0.52-0.71)	2.5
	Ag^{2+}(0.0-0.22) - Cu^{2+}(0.55-0.73)	
	Ag^{+}(0.0-0.25) - Au^{3+}(0.59-0.63)	
	Cd^{2+}(0.0-0.15) - Cu^{2+}(0.58-0.73)	
	Cd^{2+}(0.0-0.3) - Zn^{2+}(0.7-0.88)	
0.001M HNO_3	Ag^{+}(0.0-0.0) - Hg^{2+}(0.65-0.72)	3
	Ag^{+}(0.0-0.0) - Pd^{2+}(0.25-0.51)	
0.0001M HNO_3	Cu^{2+}(0.0-0.1)- Pd^{2+}(0.56-0.81)	3.5
	VO^{2+}(0.0-0.0)- Pd^{2+}(0.61-0.8)	
	VO_2^{2+}(0.0-0.0) - Pd^{2+}(0.6-0.78)	
	Ni^{2+}(0.0-0.2)- Pd^{2+}(0.62-0.85)	
0.1M CH_3COOH	Ag^{+}(0.0-0.2)- Au^{3+}(0.59-0.65)	3

TABLE -2

Ternary Separations achieved on Th(IV) Phosphate Thin-layer.

Solvent system	Separations achieved R_L - R_T	Time (Hours)
0.1M HNO_3	Pb^{2+}(0.0-0.13)-Pd^{2+}(0.21-0.48) -Hg^{2+}(0.93-0.98)	2
	Bi^{3+}(0.0-0.07)-Cd^{2+}(0.23-0.3) -Hg^{2+}(0.9-1.0)	
	Bi^{3+}(0.0-0.07)-Cd^{2+}(0.21-0.23) -Cu^{2+}(0.9-1.0)	
	Fe^{3+}(0.0-0.1)-VO^{2+}(0.2-0.5) -Ni^{2+}(0.89-0.95)	
0.01M HNO_3	Ag^{+}(0.0-0.1)-Pd^{2+}(0.2-0.25) -Zn^{2+}(0.8-1.0)	2.5
0.0001M HNO_3	Bi^{3+}(0.0-0.0) - Pt^{4+}(0.3-0.35) -Pd^{2+}(0.6-0.71)	3.5
	Ag^{+}(0.0-0.0)-Au^{3+}(0.45-0.55) Pd^{2+}(0.7-0.85)	

decrease with increasein pH in HNO_3 systems which is a characteristic feature of ion exchange operation. In pure butanol all the metal ions except Au(III) are retained at the base line. This permits quantitative separation of Au(III) from other metal ions.

Tables 1,2 and 3 show some useful and important binary and ternary separations of metal ions achieved in different solvents. Some important separations are Cu-Ag, Cu-Cd, Zn-Cd; Pb-Pd-Hg, Bi-Cd-Hg, Cu-Cd-Bi and Ag-Au-Pd. The separations were found to be better from HNO_3 than from CH_3COOH system.

TABLE -3

Quantitative Separation of Au^{3+} from binary mixtures.

(Au^{3+} added = 13.38 μg in each experiment)

Sl. No.	Mixture taken	Amount of the other metal ion applied	Amount of Au^{3+} recovered μg	Percentage of error
1.	Pt^{4}-Au^{3+}	Pt(8)	13.6	+1.6
2.	Bi^{3+}-Au^{3+}	Bi(10)	13.0	-4.6
3.	Fe^{3+}-Au^{3+}	Fe(13.3)	13.0	-4.6
4.	Pb^{2+}-Au^{3+}	Pb(10.2)	13.3	-0.6
5.	Hg^{2+}-Au^{3+}	Hg(10.8)	13.9	+3.8
6.	Pd^{2+}-Au^{3+}	Pd(8)	12.96	-3.4
7.	Zn^{2+}-Au^{3+}	Zn(13.4)	12.9	-3.4
8.	Ni^{2+}-Au^{3+}	Ni(21.6)	13.0	-4.6
9.	Cu^{2+}-Au^{3+}	Cu(11.8)	13.6	+1.6
10.	Co^{2+}-Au^{3+}	Co(10)	13.8	+3.1
11.	Cd^{2+}-Au^{3+}	Cd(10.6)	13.5	+0.98
12.	Mn^{2+}-Au^{3+}	Mn(21.8)	13.8	+3.1

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