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Cation Chromatography on Stannic Tungstate Thin Layers in DMSO-Acid and Aqueous DMSO Systems: Quantitative Separation of Gold from Numerous Metal Ions

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NOTE

Cation Chromatography on Stannic Tungstate Thin Layers in DMSO–Acid and Aqueous DMSO Systems: Quantitative Separation of Gold from Numerous Metal Ions

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Abstract

Thin-layer chromatography of metal ions has been performed on stannic tungstate using solvent systems containing dimethyl sulfoxide. Such binary separations as Fe–Al, La–Pr, La–Nd, Cr–Al, Tl–In, Fe–Mn, and Cr–Mn have been achieved. Gold (100 to 240 μg) has been separated quantitatively from vanadium, chromium, cerium, iron, cobalt, nickel, bismuth, manganese, titanium, and thorium in DMSO: water (1:1), and the method has been found to be precise and accurate.

INTRODUCTION

Thin-layer chromatography was first applied to separate inorganic cations by Seiler and co-workers (*1–5*). A review by Brinkman (*6*), however, showed that most of these separations were effected on thin layers of silica gel. Some studies on thin layers of other inorganic ion exchangers have been reported (*7–21*), but in most of them the use of binders such as silica gel, starch, or cellulose makes it difficult to have a clear interpretation of the mechanism. In these laboratories we have been able to prepare binder-free layers of the inorganic ion exchange materials stannic antimonate (*22*) and stannic arsenate (*23*). These layers have been used successfully for the quantitative separation of UO_2^{2+} from numerous metal ions and for the separation of Hg^{2+} from Cd^{2+} and Zn^{2+}. It is essential to search for thin layers which may be selective for certain metal ions. With this end in view, stannic tungstate thin layers were tried because this material has previously been found to be quite stable in acids and to have good ion exchange properties (*24*). We have used this inorganic ion exchanger in paper (*25*) and electrochromatography (*26*) to achieve various useful separations of inorganic cations. The present work is in continuation of our attempts to prepare thin layers without a binder and to use them for the quantitative separation of metal ions. Dimethyl sulfoxide (DMSO) has been used as a developer because it has earlier shown a great potential for separations (*22*). As a result, many useful separations have been achieved. Gold has been separated quantitatively from numerous metal ions in a solvent system containing DMSO.

EXPERIMENTAL

Apparatus

Thin layers of stannic tungstate were prepared on glass plates (15 × 3 cm) and the plates were developed in glass jars (20 × 5 cm). A Bausch and Lomb spectronic-20 was used for spectrophotometric studies.

Reagents

Analar grade reagents were used.

Preparation of Ion Exchange Material and Thin-Layer Plates

Stannic tungstate beads in the H^+ form were prepared according to the procedure reported earlier (*24*) by mixing 0.2 *M* solutions of stannic chloride and sodium tungstate solutions in a molar ratio of 2:1. They were powdered and slurried with a little demineralized water in a mortar. The slurry was then spread over the glass plates with the help of an applicator to prepare thin layers of 0.1 mm thickness. The plates were dried in air for use.

Test Solutions and Detection Reagents

The test solutions were generally 0.1 *M* in metal chlorides or nitrates. Conventional spot test reagents were used for detection purposes (*27*).

Procedure

For Qualitative Work

One or two drops of the test solutions were placed on plates with glass capillaries. After the spots had dried, development was made in different solvent systems and the solvent was allowed to rise 10 cm in all the cases. R_T and R_L values were measured as usual after detection.

For Quantitative Work

A stock solution of gold chloride (~10,000 ppm Au) was prepared in 4 *M* HCl by dissolving auric chloride. A standard spectrophotometric method (*28*) using $SnCl_2$ solution as the coloring reagent was set up for the quantitative determination of gold. Known amounts of gold along with other metal ions were spotted with the help of a micropipette, and the development was performed in a DMSO:water (1:1) solvent system. A pilot plate was run simultaneously in order to locate the exact position of the spot on the working plate. The area where gold was located was scratched, and the mass was eluted with distilled water. The exchanger was separated from the solution by filtration and washed with distilled water 4 or 5 times to ensure complete elution of the gold. The filtrate and the washings were collected in a 25-ml measuring flask. A $SnCl_2$ solution (2 ml) was added to develop the color. This was then made up to 25 ml with distilled water, and the gold was determined spectrophotometrically.

RESULTS

The following solvent systems were used in these studies.

(1) HNO_3: DMSO (6:4, 5:5, 4:6, 3:7, 2:8, 1:9)
(2) HCl: DMSO (6:4, 5:5, 4:6, 3:7, 2:8, 1:9)
(3) DMSO: water (1:1)
(4) HNO_3: water (1:1)

Table 1 summarizes the separations achieved on stannic tungstate thin layers, while Tables 2 and 3 show the quantitative results.

TABLE 1

Separations Actually Achieved on a Stannic Tungstate Thin Layer in Different Solvent Systems

Solvent systems	Metal ion separation ($R_T - R_L$)	Time required (hr)
HNO_3:DMSO (6:4)	Cu (0)–Cd (0.36–0.40)	
	Fe (0)–Al (0.30–0.56)	
	La (0)–Pr (0.80–1.0)	
	La (0)–Nd (0.78–1.0)	
	La (0)–Sm (0.76–1.0)	
	Fe (0)–Au (0.90–1.0)	6
HCl:DMSO (6:4)	Mn (0)–Zn (0.16–0.30)	
	Cr (0)–Al (0.30–0.50)	
	Pt (0.30–0.40)–Au (0.62–1.0)	
	Pd (0.30–0.40)–Au (0.65–1.0)	
	Hg (0.25–0.40)–Au (0.63–1.0)	6
HNO_3:water (1:1)	Cr (0.0)–Al (0.85–1.0)	
	Tl (0.0)–In (0.32–0.55)	
	Fe (0–0.35)–Mn (0.70)–1.0)	
	Cr (0.0)–Mn (0.72–1.0)	2

TABLE 2

Quantitative Separation of Gold in Binary Mixtures

Sample no.	Mixture	Amount of gold applied (μg)	Amount of gold recovered (μg)	Percentage error
1	Au–V	100	105	5
2	Au–Cr	100	102	2
3	Au–Ce	100	105	5
4	Au–Fe	100	105	5
5	Au–Co	100	100	0
6	Au–Ni	100	102	2
7	Au–Bi	100	105	5
8	Au–Mn	100	102	2
9	Au–Ti	100	102	2
10	Au–Th	100	105	5

TABLE 3

Quantitative Separation of Gold from Synthetic Mixtures

Sample no.	Gold applied (μg)	Gold recovered (μg)	Percentage error
1	240	245	+2.1
2	240	240	0.0
3	240	225	−6.25
4	240	225	−6.25
5	160	150	−6.25
6	160	155	−3.1
7	160	150	−6.25
8	160	150	−6.25

DISCUSSION

The present study reveals that stannic tungstate thin layers are highly selective for metal ions, a behavior similar to stannic antimonate and stannic arsenate layers. Almost all metals have zero R_F values in solvent systems containing HNO_3 : DMSO in different volume ratios. In some cases a little movement of metal ions is observed which diminishes with an increase of DMSO in the system, and finally in the solvent system HNO_3 : DMSO (1 : 9) all metals remain at the point of application after development. However, Au^{3+} shows a little movement ($R_F = 0.08$–0.23) with a change of acid in the solvent system. When we use HCl instead of HNO_3, we observe that the metal ions in general show an increased tendency to move. This is possibly due to the formation of negative chlorocomplexes of metal ions which may have greater R_F values on a cation exchanger such as stannic tungstate. Gold is singled out to show higher R_F values than other metal ions, although there is some tailing ($R_F =$ 0.62–1.00). However, if we withdraw acid from the solvent system completely and use only the DMSO : water (1 : 1) system, only gold and platinum show R_F values, the other ions being stopped at the starting point. Platinum shows a double spot ($R_F = 0$, 0.68–0.75). This may be because most of the transition metals are strongly bonded to six dimethyl sulfoxide molecules, making the exchange reaction difficult due to steric reasons (*29*). The presence of the anionic species of Au and Pt in acidic solutions may be the cause of a greater movement of these metals on stannic tungstate layers. This behavior of stannic tungstate suggests the possibility of a quantitative separation of gold from other metal ions. In practice, gold has been separated from some binary mixtures containing different amounts of gold, and it has been noticed that the method works well within an error range of 5% (Table 2). To test the validity of the method and its application in a wider field, some synthetic mixtures were prepared by taking Fe, V, Cr, Ti, Ni, Cu, Ce, and Co along with Au and were applied on the thin layer plates. The results obtained in this manner also show great accuracy and precision (Table 3). When the HNO_3: water (1: 1) system was used for the development of plates, a larger number of cations show a R_F value $\approx$ 1; viz., Be, Al, Mn, Co, Ni, Cu, Zn, rare earths, Au, and Hg. We also obtained a number of important separations such as Cr–Al, Tl–In, Fe–Mn, and Cr–Mn. Thus the most useful solvent systems in these studies are HNO_3:DMSO (6:4), HCl:DMSO (6:4), and HNO_3: water (1:1) which give important binary separations of metal ions. Another striking feature of this study is that HNO_3: DMSO (6: 4) is able to differentiate between movements of lanthanides, thus resulting in their separations. For example, La can be separated from Pr, Nd, and Sm by using this solvent system. DMSO systems generally take larger time for development than water systems because they are highly viscous liquids (viscosity = 2.473 cP at 20°C) with a low freezing point (18.2°C).

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