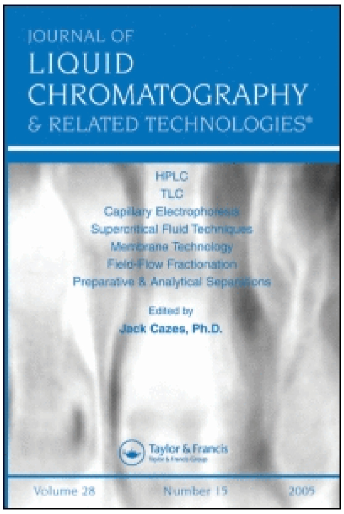

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Studies on Hydrated Stannic Oxide. Part VI. Thin-Layer Chromatography of Some Anions on Hydrated Stannic Oxide: Quantitative Separation of Cr(VI) from Some Ores and Alloys

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STUDIES ON HYDRATED STANNIC OXIDE. PART VI. THIN-LAYER CHROMATOGRAPHY OF SOME ANIONS ON HYDRATED STANNIC OXIDE : QUANTITATIVE SEPARATION OF Cr(VI) FROM SOME ORES AND ALLOYS

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ABSTRACT

The analytical applications of hydrated stannic oxide as an ion-exchanger have been investigated by thin-layer chromatographic (TLC) technique. Binder-free thin-layers of hydrated stannic oxide are useful for some binary separations. Quantitative separation of Cr(VI) and Mo(VI) from some binary mixtures have been achieved. The method is applied to separate microgram-quantities of Cr(VI) quantitatively from several ores and alloys in ammonia buffer (pH 10) system.

INTRODUCTION

In recent years, thin-layer chromatography (TLC) has become popular as a rapid and useful analytical tool in separation science. The most of the reported works were based on silica gel only (I). Qureshi et.al (2-4) studied binder-free thin-layer of tin (IV) - arsenate, antimonate and tungstate. Binder free thin-layer chromatographic studies with thorium-phosphate, antimonate and tungstate have been reported from our laboratory by De et.al. (5-7) recently. In this paper we report the preparation of binder-free thin-layers of hydrated stannic oxide and the results of systematic studies of TLC of anions with the variation of pH in some buffer systems. Some important binary quantitative separations have been achieved. Quantitative separations of Cr(VI) and Mo(VI) from other anions in binary mixtures and Cr(VI) from some ores and alloys have been carried out.

EXPERIMENTAL

Apparatus :- As reported in (7).

Reagents :- Sodium stannate trihydrate was of reagent grade quality (U.S.S.R.). All other reagents were of A.R. grade (B.D.H./ Pfizer/ E. Merck). Other samples are of Durgapur extra low carbon ferro-chrome alloy (Lot No.1627A); DSP alloy steel(A); B.A.S. chrome-ironore (49f) and B.A.S. stainless steel (69a).

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

Hydrated stannic oxide was prepared by adding 0.8M ammonium sulphate to 0.8M sodium stannate solution as described earlier (8). It was powdered and slurried with a little deionised water in a mortar. The thin-layers were prepared over the glass plates as described by De et.al.(7). These plates gave reproducible R_F-values.

Test solution and detection reagents :-

The test solutions in general had anion concentrations of 8-10 mg/ml (Sodium/potassium). Standard spot test reagents were used for the detection (9).

Solvent system :-

1. $0.05M\ HCl + 0.09M\ KCl$ (pH 2)
2. $0.1M\ CH_3COOH + 0.1M\ CH_3COONa$ (pH 4)
3. $0.1M\ CH_3COOH + 0.1M\ CH_3COONa$ (pH 6)
4. $0.1M\ NH_4OH + 0.1M\ NH_4Cl$ (pH 8)
5. $0.1M\ NH_4OH + 0.1M\ NH_4Cl$ (pH 10)

PROCEDURE

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

For quantitative work, stock solutions of Cr(VI) [5250 μg/ml of Cr (VI)] and Mo(VI) [10,000 μg/ml of Mo(VI)] were prepared. Synthetic mixtures containing known amount of Cr(VI) or Mo(VI) was applied with the help of a micropipette on the line of application. A pilot plate was run simultaneously to locate the position of Cr(VI) or Mo(VI) with the help of detecting reagent. The area corresponding to Cr (VI) or Mo(VI) was then scratched off from the working plate and the mass was eluted with 5 ml of IM NaOH. The exchanger was filtered off. The filtrate was then neutralised with dilute mineral acids and Cr(VI) or Mo(VI) were estimated spectrophotometrically (10). When the procedure is applied during selective separation of Cr (VI) in oresand alloys, the pH of the test solution was adjusted freshly to 1.7 just before application.

RESULTS AND DISCUSSION

The results of our TLC studies reveal that the general trend of R_F-values of most of the anions increases with the increase of pH, which indicates ion-exchange process. However, V(V) and W(VI) are retained on the point of application probably due to the formation of insoluble stannic compound, which was also observed in our electrochromatographic and column chromatographic studies (11,8). This permits us separation of V(V) and W(VI) from other anions. But due to incomplete elution of V(V) and W(VI), quantitative estimation were not possible. No spot of thiocyanate was obtained in solvent system (No.1) because of decomposition of thiocyanate ion. Cr(VI) and Mo(VI) showed appreciable high R_F-values which induced us to separate those anions quantitatively from other anions of low R_F values in some binary mixtures. Selective separation of microgram quantities of Cr(VI) in some ores and alloys has been achieved in ammonia buffer (pH = 10) solvent system.

Table-1, 2 and 3 show some binary separations of anions, quantitative separation of Cr (VI) and Mo(VI) from some binary mixtures and selective separation of Cr(VI) from some ores and alloys respectively. Some important separations are Cr(VI)-W(VI), Cr(VI) -

Table-1

Binary Separations Achieved on hydrated stannic oxide Thin-Layer.

Solvent system	Separations achieved (R_T - R_L)	Time (hours)
1. pH 2 (0.05M HCl +0.09M KCl)	W(VI)(0.0-0.0)-$Fe(CN)_6^{3-}$(0.25-0.40)	1.5
	W(VI)(0.0-0.0)-$Fe(CN)_6^{4-}$(0.25-0.48)	
	As(V)(0.0-0.06)-$Fe(CN)_6^{3-}$(0.22-0.45)	
	As(V)(0.0-0.08)-$Fe(CN)_6^{4-}$(0.25-0.40)	
	As(V)(0.0-0.06)-Cr(VI)(0.22-0.38)	
	Mo(VI)(0.0-0.1)-Cr(VI)(0.25-0.45)	
	Mo(VI)(0.0-0.1)-$Fe(CN)_6^{3-}$(0.25-0.45)	
	Mo(VI)(0.0-0.12)-$Fe(CN)_6^{4-}$(0.24-0.43)	
	V(V)(0.0-0.0)-Cr(VI)(0.25-0.40)	
	V(V)(0.0-0.0)-$Fe(CN)_6^{3-}$(0.22-0.42)	
	V(V)(0.0-0.0)-$Fe(CN)_6^{4-}$(0.25-0.42)	
	W(VI)(0.0-0.0)-Cr(VI)(0.25-0.40)	
2. pH 4 (0.1M CH_3COOH +0.1M CH_3COONa)	BO_2^-(0.0-0.1)-Cr(VI)(0.30-0.46)	1.25
	P(V)(0.1-0.2)-$Fe(CN)_6^{3-}$(0.42-0.60)	
	As(V)(0.0-0.15)-SCN^-(0.40-0.55)	
	W(VI)(0.0-0.08)-SCN^-(0.38-0.48)	
	V(V)(0.0-0.05)-SCN^-(0.38-0.50)	
	BO_2^-(0.0-0.15)-SCN^-(0.35-0.48)	
	V(V)(0.0-0.05)-I(V)(0.20-0.30)	
	P(V)(0.1-0.2)-$Fe(CN)_6^{4-}$(0.3-0.42)	
	W(VI)(0.0-0.1)-Mo(VI)(0.2-0.3)	
	V(V)(0.0-0.0)-Mo(VI)(0.21-0.30)	

(continued)

Table-1 (Contd.)

Solvent system	Separations achieved ($R_T - R_L$)	Time (hours)
3. pH 6 (0.1M CH_3COOH +0.1M CH_3COONa)	BO_2^-(0.0-0.1)-$Fe(CN)_6^{3-}$(0.28-0.37) BO_2^-(0.0-0.12)-I^-(0.20-0.28) V(V)(0.0-0.05)-I^-(0.20-0.28) W(VI)(0.0-0.08)-I^-(0.18-0.25)	1.25
4. pH 8 (0.1M NH_4CH + 0.1M NH_4Cl)	W(VI)(0.0-0.1)-I(V)(0.25-0.32)	1.5

Table-2

Quantitative Separation of Cr(VI) and/ Mo(VI) from other anions.

Time - 1.5 hours; Solvent - 0.1M CH_3COOH + 0.1M CH_3COONa (pH=6)

Sl. No.	Mixture taken	Amount of other anion applied (µg)	Amount of Cr(VI) or Mo(VI) Added (µg)	Amount of Cr(VI) or Mo(VI) Recovered (µg)	% error
1	Cr(VI)-V(V)	V(V)(10.00)	10.50	10.10	-3.8
2	Cr(VI)-W(VI)	W(VI)(8.00)	10.50	10.30	-2.0
3	Cr(VI)-BO_2^-	BO_2^-(6.60)	10.50	10.30	-2.0
4*	Cr(VI)-Mo(VI)	Mo(VI)(6.60)	10.50	10.20	-2.86
5	Mo(VI)-W(VI)	W(VI)(20.00)	20.00	20.80	+4.0
6	Mo(VI)-V(V)	V(V)(20.00)	20.00	20.90	+4.5

* Solvent used : 0.05M HCl + 0.09M KCl (pH=2)

Table - 3

Selective Separation of Cr (VI) from some ores and alloys.

Time- 1.5 hours; Solvent - 0.1M NH_4CH +0.1M NH_4Cl (pH=10)

Sl. No.	Sample taken	Composition of Sample, %	Amount of Cr(VI) Added (μg)	Amount of Cr(VI) Recovered (μg)	% error
1	Synthetic chrome iron ore	Cr_2O_3 = 43 FeO = 12	10.00	10.20	+2.0
2	Chrome-iron ore (49f) (B.A.S.)	Cr_2O_3 = 43.1 FeO = 11.6 SiO_2 = 2.84	4.70	4.4	-6.4
3	Synthetic Durgapur Low Carbon Ferro-Chrome Alloy	Cr = 72.5 Fe = 26	6.20	5.95	-3.16
4	Durgapur Extra Low Carbon Ferro-Chrome Alloy [Lot. No.1627A]	C = 0.025 Si = 0.47 Cr = 72.53 Fe = 26.00	5.70	5.60	-2.8
5	Synthetic DSP - Alloy Steel	Mn = 1 Ni =20.5 Cr =24	4.00	3.92	-2.0
6	DSP-Alloy Steel(A) (Heat resistant steel)	C =0.14 Ni=20.41 Mn =1.04 S=0.01 Cr=23.75 Si = 1.30	3.76	3.96	+5.3
7	Synthetic Stainless Steel	Ni = 8.2 Cr =18.4	4.00	4.10	+2.5
8	Stainless Steel (69a) (B.A.S.)	Ni = 8.20 Cr =18.4	4.08	4.20	+2.9

V(V), Cr(VI)-Mo(VI), $[Fe(CN)_6]^{3-}$- P(V), $[Fe(CN)_6]^{3-}$ - As(v), Mo(VI)-W(VI), I(V)-V(V), Mo(VI)-V(V), Cr(VI)-BO_2^-, I(V)-W(VI), SCN^--W(VI) and SCN^--V(V). More clear separations of Cr(VI), Mo(VI), I(V), $[Fe(CN)_6]^{3-}$ and $[Fe(CN)_6]^{4-}$ from V(V) or W(VI) were achieved also in solvent system No.3, 4 and 5.

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