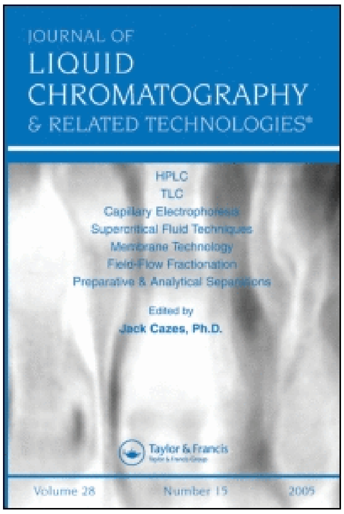

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TLC Separation of Transition Metal Ions and Their Quantitative Estimation by Atomic Absorption Spectroscopy

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TLC SEPARATION OF TRANSITION METAL IONS AND THEIR QUANTITATIVE ESTIMATION BY ATOMIC ABSORPTION SPECTROSCOPY

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ABSTRACT

Separation of an eight component mixture of transition metal ions has been studied on plain and impregnated silica gel layers. The solvent systems used were pyridine-benzene-acetic acid-water (6:5:8:4,v/v) on 1,10-phenanthroline (1%), n-butanol-benzene-formic acid (5:10:9,v/v) on DMG (1%) and pyridine-benzene-acetic acid-water (5:5:4:1,v/v) on EDTA (2%) impregnated silica gel layers. ß-naphthol (0.1% in methanol) has been developed as a new locating reagent. The percentage recovery of these metal ions in quantitative estimation by AAS ranged between 65.2 to 89.9.

INTRODUCTION

Separation and identification of transition metal ions is of great importance in chemical, biochemical, biological, industrial and environmental sciences. The transition metal ions are found in various alloys in solid state and in the form of solution in various parts of the body and a cross section of these metals exists as free ions and their complexes in the blood stream. Various transition metal ions are toxic and produce various types of diseases in the body (1,2). TLC has long been used for the separation and identification of metal ions on the various adsorbent layers (3). Impregnation of TLC layers has been used for the better and reproducible separation of various compounds (4-8) but there are only few reports available on the separation of transition metal ions on impregnated layers (3). In view of the importance of metal ions and practical utility of impregnated TLC plates, attempts were made to separate and identify Cr(III), Mn(II), Fe(II), Co(II), Ni(II), Cu(II), Zn(II) and Cd(II) metal ions on different impregnated silica gel lagers using three new solvent systems. The present paper describes the TLC separation and quantitative estimation by atomic absorbtion spectroscopy of these metal ions. In addition to this, ß-napthol (0.1% in methanol) was developed as a new locating reagent for these metal ions.

EXPERIMENTAL

The metal salts were obtained from E. Merck, India,. Silica gel G (size, 10-40 μ) with calcium sulphate (13%) as binder, impurities of chloride, iron and lead (0.02% each), and showing a pH 7.0 in a 10% aq. suspension, was from E. Merck India. All other reagents and solvents used were of A.R. grade and purchased from E. Merck, India. Perkin Elmer (U.S.A.) Atomic Absorbtion Spectrophotometer model 3100 was used.

Preparation of TLC plates and solution of metal ions:

TLC plates (20cm x 20cm x0.5mm) coated with plain slica gel G were prepared by spreading a slurry of silica gel G (50g) in double distilled water (100 mL) with the help of Stahl type applicator. Impregnated silica gel layers were prepared by spreading a slurry of silica gel G (50 g) in 70%

methanol (100 mL), containing 1,10-phenanthroline (1%), DMG (1%) and EDTA (2%) respectively. The plates were dried overnight at 60±2°C in an oven. The solutions of different metal salts (10^{-3} M) were prepared in double distilled water.

Development of Chromatograms:

The solutions of metal ions were applied on plain and impregnated silica gel plates at 50 ng level using a 25 μL Hamilton syringe. The chromatograms were developed upto 10 cm at 25 ± 2°C in pyridine-benzene-acetic acid-water (6:5:8:;4,v/v), n-butanol-banzene-formic acid (5:10:9,v/v) and pyridine-benzene-acetic acid-water (5:5:4:1,v/v) solvent systens in paper lined rectangular glass chambers which were pre-equilibrated with the solvent systems for 10-20 minutes. The plates were then dried in an oven at 60 ± 2°C for 30 minutes. The plates were sprayed with ß-napthol (0.1% in methanol) and further heated for 20 minutes. The spots of these metal ions were located as redish-yellow in color.

Quantitative Estimation by AAS:

The standard solutions of each metal ions (1 ppm to 4 ppm) were prepared in double distilled water for the calibration of atomic absorption spectrophotometer. The studied metal ions separated in TLC were erased along with silica gel G from the plates. These metal ions were extracted from silica gel G in double distilled water (10 mL) individually and separately. The concentration of each metal ion was determined by atomic absorbtion spectrophotometer.

RESULTS AND DISCUSSION

The hR_f values of metal ions on plain and various impregnated silica gel layers are given in table-1. The results are an average of atleast three identical runs with a standard deviation of ± 0.40 to ± 0.50 on plain and impregnated layers respectively. The resolution of these metal ions was calculated and confirmed by usual method (4). In order to optimize the separation conditions, variation in the concentrations of impregnating reagents and in the

TABLE 1

hR_f Values of Transition Metal Ions on Plain and Impregnated Silica Gel Layers

Sl. No.	Metal Ion	A			B	C	D
		I	II	III	I	II	III
1	Cr (III)	38	88	13	24	37	43
2	Mn (II)	54T	82T	41T	28	34	57
3	Fe (II)	47	77	20	37	30	50
4	Co (II)	90	66	83	57	68	21
5	Ni (II)	50T	54T	67T	61	53	28
6	Cu (II)	47T	60T	56	68	47	47
7	Zn (II)	50T	64T	58T	33	56	24
8	Cd (II)	54	75	61	43	64	38

T : Tailing
A : Plain silica gel layers, B: 1,10-phenanthroline (1%) impregnated silica gel layer, C: DMG (1%) impregnated silica gel layer, and D: EDTA (2%) silica gel layer.

Solvent systems :

I : Pyridine-benzene-acetic acid-water (6:5:8:4,v/v)
II : n-Butanol-benzene formic acid (5:10:9,v/v)
III: Pyridine-benzene-acetic acid-water (5:5:4:1,v/v)

Room temperature : 25 ± 2^0C.
Solvent Front : 10 cm.
Detection : ß-Napthol (0.1% in methanol).

composition of solvent systems was carried out. As a result of extensive experimentation the best solvent systems and impregnating reagents were selected and reported herewith.

It is clear from table-1 that only four to five metal ions got separated from an eight component mixture, with broad spots, on plain silica gel plates. Contrary to this, all the eight metal ions got separated on impregnated silica gel layers with compact spots. It is a well known fact that the impregnating reagent selected e.g, 1,10-phenanthroline, DMG and EDTA are very good chelating agents and have a very good tendency to form chelates with transition metal ions immediately. Therefore, the transition metal ions should form chelates with the reported impregnating reagents on impregnated silica gel layers and got separated, due to the different migration behaviour of these chelates on the impregnated plates than on the plain plates. Thus it must be the combined effect of partition and adsorption phenomenon of these chelates which should be responsible for the resolution of these metal ions. This fact was verified by preparing the chelates of these metal ions with the reported impregnating reagents and separating them on plain silica gel plates using the same solvent systems. The results were in good agreement with those on impregnated silica gel layers.

The results of the quantitative estimation of these metal ions on impregnated TLC plate are given in table-2. It is clear from table-2 that percentage recovery of these metal ions varies between 62.2 to 89.9. It is interesting to note that none of the metal ion was hundred percent recovered. This fact suggests that some amount of the metal ions diffuse on TLC plates and remains adsorbed on the silica gel layer even after extraction. The difference in the recovery of transition metal ions may be due to the difference in their diffusion and adsorption capacities on silica gel layers. This observation is verified by the fact that the percentage recovery of these metal ions is greater on EDTA impregnated plates

TABLE 2

Quantitative Estimation of Transition Metal Ions on Impregnated Silica Gel Layers by Atomic Absorbtion Spectrophotometer.

Sl. No.	Metal Ion	λmax (nm)	E	B		C		D	
				F	G	F	G	F	G
1	Cr (III)	357.9	10	6.52	65.2	6.62	66.2	6.91	69.1
2	Mn (II)	279.5	10T	7.67	76.7	8.02	80.2	8.35	83.5
3	Fe (II)	248.3	10	8.38	83.8	8.63	86.3	8.99	89.9
4	Co (II)	240.7	10	6.70	67.0	7.02	70.2	7.45	74.5
5	Ni (II)	232.0	10	8.15	81.5	8.67	86.7	8.84	88.4
6	Cu (II)	324.8	10	7.17	71.7	7.67	76.7	7.90	79.0
7	Zn (II)	213.9	10	7.83	78.3	8.01	80.1	8.30	83.0
8	Cd (II)	228.8	10	6.32	63.2	6.92	69.2	7.10	71.0

B : 1, 10-Phenanthroline (1%) impregnated silica gel layer.
C : DMG (1%) impregnated silica gel layer.
D : EDTA (2%) impregnated silica gel layer.
E : Amount of transition metal ions spotted in μg.
F : Amount of transition metal ions recovered in μg.
G : Percentage of transition metal ions recovered .

in comparison to DMG and 1,10-phenanthoraline impregnated plates. EDTA is a stronger chelating ligand than DMG and 1,10-phenanthroline which form chelates with transition metal ions quickly leaving very poor amount for diffusion and adsorption on silica gel layers and therefore gives greater percentage recovery.

Thus, the reported chromatographic system can be considered as sucessful, reliable and reproducible method for the separation and identification of reported transition metal ions and can be used for the separation of these metal ions from unknown samples.

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