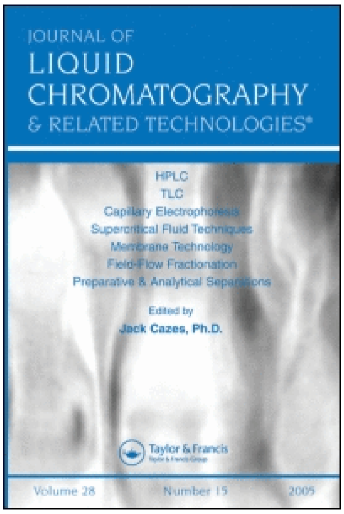

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S. P. Srivastava[a]; Kamlesh[a]; V. K. Gupta[a]
[a] Department of Chemistry, University of Roorkee, Roorkee, U.P., India

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THIN LAYER CHROMATOGRAPHIC SEPARATION OF SOME INORGANIC IONS ON SULPHA DRUG IMPREGNATED LAYERS

S.P.Srivastava, Kamlesh and V.K.Gupta
DEPARTMENT OF CHEMISTRY, UNIVERSITY OF ROORKEE
ROORKEE - 247672, (U.P.) INDIA

ABSTRACT

Binary, ternary, quaternary and hexanary separations schemes for Co(II), Ni(II), Th(II), Fe(II), U(V), V(IV), Cu(II), As(III), Pb(II), Cd(II), Hg(II), Ag(I), Sb(III). Se(IV), Sn(IV), Ti(IV), Mo(VI), Pd(II) and Pt(IV) have been worked out by using sulphaguanidine as an impregnant on silica gel thin layer and by using the solvent system Isopropanol, ethyl acetate, acetic acid, water, DMF in the ratios (60:30:5:10:5) and (60:30:5:10:3). pH Metric studies have been made to establish the formation of complex between sulphaguanidine and the different metal ions to decide the nature of bonding and to determine their stability constants in an attempt to correlate it with chromatographic behaviour.

INTRODUCTION

Sulpha drugs are the N-substituted compounds of sulphanilamide ($H_2NC_6H_4SO_2NH_2$) which are the drugs of proved therapeutic importance. Sulpha drugs are known to form complexes with different metal ions. Gulko and coworkers[1] have reported the complexation of sulphaguanidine with palladium and slso studied the stability of the palladium sulphaguanidine complex. Narang and Gupta [2] synthesised and characterised the Cu(II) chloride complexes with different sulpha drugs namely - sulphanilamide, sulphaguani-

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dine, sulphathiazole, sulphamerazine and sulphapyridine, while Chaturvedi and coworkers [3] reported the sulphaguanidine complexes with few mono and bivalent metal ions. Recently, the remarkable TLC separation of various sulpha drugs was achieved in this laboratory [4] by using different metal salts as impregnants. Reference to literature shows [5] that TLC separation of different inorganic ions has been attampted by many workers but little attention has been paid to impregnation technique for the separation of inorganic ions. Since the chromatographic behaviour has been shown [6-12] to be influenced by the addition of a complexing material either as an impreghant or as a constituent of the developer system, it was considered worthwhile to try different sulpha drugs as impregnants for the TLC separation of metal ions. The different sulpha drugs tried as impregnants were - sulphasomidine, sulphaguanidine, sulphathiazole, sulphaphenazole, sulphamethizole, sulphadimidine and sulphadiazine. From these 0.5 % sulphaguanidine impregnation showed the best separation potentiality. For understanding the role of impregnant, potentiometric studies were carried out to determine the stability constants of the complexes formed between the impregnant and the metal ions. Results of these studies are presented in this paper.

EXPERIMENTAL

The TLC plates (thickness 0.5 mm) were prepared by spreading, by means of a Stahl type applicator, a slurry of 50 g of silica gel (B.D.H.) and 0.50 g of sulphaguanidine in 100 ml of distilled water-alcohol (70:30) mixture and the plates were dried for 24 hours at 60±1°C.

The inorganic ions (0.1 % W/V solution in water) were applied to the layers using glass capillary and the chromatograms were eluted at a constant temperature (30±1°C) with a mixture of Isopropanol-ethyl acetate-DMF-acetic acid-water (60:30:5:5:10), solvent A and (60:30:3:5:10) solvent B. After development the plates were sprayed with the suitable spraying reagent. A freshly prepared solution of $K_4Fe(CN)_6$ (1 % W/V in 2 % HCl) was used as a visualizing reagent in case of Fe, U, V, Cu and Ti while for other metal ions a freshly prepared solution of yellow ammonium sulphide was used.

RESULTS AND DISCUSSION

The hR_f values of different metal ions on silica gel layers and on layers impregnated with 0.5 % sulfaguanidine in solvent systems-Isopropanol-ethyl acetate-DMF-acetic acid-water (60:30:5:5:10) solvent A and (60:30:3:5:10) solvent B, are given under TABLE-1. A perusal of the data given in this table shows tailing to a greater extent (denoted by G in table 1) for metal ions on plain silica gel plate, while all the metal ions are well separated on 0.5% sulphaguanidine impregnated plates. Hence, 0.5 % sulfagunidine was considered to be a suitable impregnant for the separation of metal ions. Based on hR_f values and the analytical, biochemical and industrial importance of the metal ions separated on sulfaguanidine impregnated plate, some binary, ternary, quaternary and hexanary separations of metal ions have been achieved by using solvent systems A and B(tables 2-5). No change in hR_f values has been observed when mixtures of metal ions were developed.

The pH-metric method of Calvin and Bjerum [13,14] was used to determine the stability constants of metal-sulphaguanidine complexes. The values of stability constants thus obtained with metals Fe, V, Cu, Pb, Se, Sn, TABLE - 1

hR_f Values of Metal Ions on Silica Gel Layer Impregnated with 0.5 % Sulfaguanidine

Metal ion		Plain silica gel	hR_f		Detection Limit (ug)
			Solvent A	Solvent B	
Co(II)	(chloride)	51G	51	53	2.5
Ni(II)	(sulphate)	40G	29	40	5.0
Th(II)	(nitrate)	13G	10	13	7.0
Fe(II)	(sulphate)	27G	17	24	2.5
U(V)	(pentaoxide)	85	81	83	4.0
V(IV)	(sulphate)	44	04	23	5.0
Cu(II)	(sulphate)	75	65	67	2.5
As(III)	(oxide)	39	32	34	5.0
Pb(II)	(acetate)	52G	45	48	5.0
Cd(II)	(nitrate)	64	57	60	5.0
Hg(II)	(chloride)	96	90	94	1.0
Ag(I)	(nitrate)	74G	60	72	2.5
Sb(III)	(chloride)	18	11	12	2.5
Se(IV)	(dioxide)	45	41	47	4.0
Sn(IV)	(chloride)	37	24	37	5.0
Ti(IV)	(chloride)	40	21	32	5.0
Mo(VI)	(ammonium)	16	10	14	2.5
Pd(II)	(chloride)	84	84	80	2.0
Pt(IV)	(chloride)	74	74	72	2.5

TABLE - 2

Binary Separation of Metal Ions

Metal ions separated		Solvent system employed
Fe(II)	U(V)	A
Fe(II)	Cu(II)	A
Fe(II)	Mo(VI)	B
U(V)	Se(IV)	A
As(III)	Se(IV)	A
As(III)	V(IV)	A
Cu(II)	Ag(I)	A
Pt(IV)	U(V)	A
Sb(III)	Fe(II)	A

TABLE - 3

Ternary Separation of Metal Ions

Metal ions separated			Solvent system employed
Ag(III)	- Cd(II)	- Hg(II)	B
As(III)	- Pb(II)	- Cd(II)	A
Sb(III)	- Sn(IV)	- Se(IV)	A
Cd(II)	- Cu(II)	- Hg(II)	A
Pb(II)	- Sn(IV)	- Ti(IV)	B
Pd(II)	- Pt(IV)	- Th(II)	A

TABLE - 4

Quarternary Separation of Metal Ions

Metal ions separated				Solvent system employed
Ag(I)	- Cd(II)	- Sn(IV)	- Sb(III)	B
Hg(II)	- Pd(II)	- Pt(IV)	- Ag(I)	A
U(V)	- Ti(IV)	- Th(II)	- V(IV)	A
Pd(II)	- Cu(II)	- Sn(IV)	- Fe(II)	A
Hg(II)	- U(V)	- Ag(I)	- V(V)	A
Hg(II)	- Ag(I)	- Pb(II)	- Sb(III)	A
Se(IV)	- Sn(IV)	- Ti(IV)	- Mo(VI)	B

TABLE - 5

Hexanary Separation of Metal Ions

Metal ions separated						Solvent System employed
Pd(II)	-Pt(IV)	-Cd(II)	-Pd(II)	-Ti(IV)	-Mo(VI)	A
U(V)	-Cu(II)	-Co(II)	-Ni(II)	-Fe(II)	- V(IV)	A
Hg(II)	-Ag(I)	-Se(IV)	-As(III)	-Sn(IV)	-Sb(III)	A

In tables (2-5) solvents A and B stand for :

A - Isopropanol-ethyl acetate-acetic acid-water-DMF (60:30:5:10:5)

B - Isopropanol-ethyl acetate-acetic acid-water-DMF (60:30:5:10:3)

TABLE - 6

Formation Constants of Metal Complexes of Sulphaguanidine

Metal ion	$\log k_1$	$\log k_2$	$\log \Delta hR_f$	$\frac{\log \Delta hR_f}{\log k}$
Fe(II)	6.99	6.78	1.00	0.143
V(IV)	8.75	-	1.60	0.1828
Pb(II)	4.15	-	0.845	0.2036
Cd(II)	2.75⊗	-	0.845	0.307
Se(IV)	5.66	-	0.602	0.106
Sn(IV)	5.27	-	1.1139	0.211
Mo(VI)	4.50	-	0.778	0.1728
Pd(II)	5.42‡	4.38‡	0.00	0.000
Cu(II)	6.77	6.75	1.00	0.1477

⊗The value taken from the work of Chaturvedi and coworkers [15].

‡ The values taken from the work of Gulko and coworkers [1].

and Mo-sulphaguanidine complexes are summarised in the Table 6. The value of stability constant for Cd-sulphaguanidine complex has been taken from the work of Chaturvedi and coworkers [15] while that of Pd-sulphaguanidine complex has been taken from the work of Gulko and coworkers [1].

To find out the relationship, if any, between chromatographic behaviour of different metal ions on sulphaguanidine impregnated plate and the stability constants of their complexes, plots of log k versus $\log \Delta hR_f$ (Fig.1) and of log k versus $\frac{\log \Delta hR_f}{\log k}$ (Fig.2) were drawn. The values of $\log \Delta hR_f$ and $\frac{\log \Delta hR_f}{\log k}$ are also given in Table 6. Although no simple correlation could be obtained between ΔhR_f and the stability constant, it

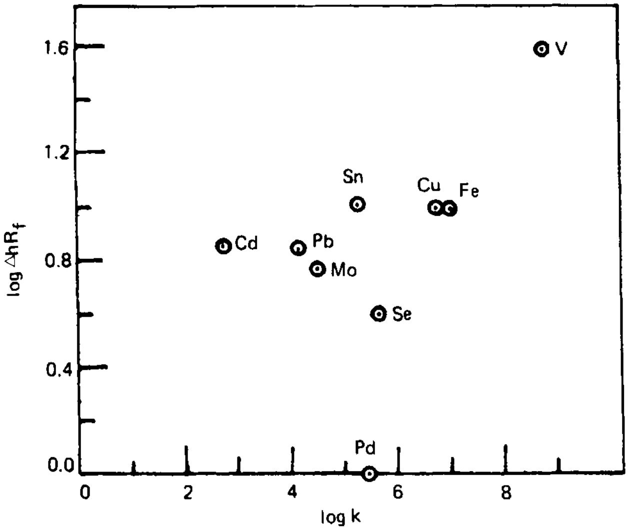

Fig. 1. Relationship between log k and ΔhR_f .

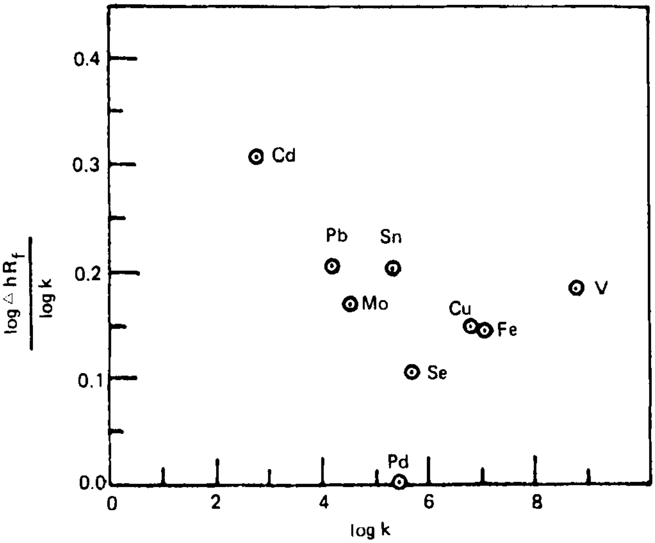

Fig.2. Relationship between log k and $\frac{\log \Delta hR_f}{\log k}$.

can be seen that, in general, ΔhR_f increased with an increase in k suggesting thereby that complex formation between sulphaguanidine and the metal did influence the movement on the sulphaguanidine impregnated plate but it was obviously not the sole factor influencing movement and hence the absence of any correla ion between k and hR_f.

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