

This article was downloaded by:

On: 24 January 2011

Access details: *Access Details: Free Access*

Publisher *Taylor & Francis*

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



## Journal of Liquid Chromatography & Related Technologies

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713597273>

### Chromatography of Some Metal Ions on Silica Gel Thin Layers in Butanol-Formic Acid Media

A. Mohammad<sup>a</sup>; N. Fatima<sup>a</sup>

<sup>a</sup> Chemistry Section Z. H. College of Engineering and Technology, Aligarh Muslim University, Aligarh, U. P., India

**To cite this Article** Mohammad, A. and Fatima, N.(1986) 'Chromatography of Some Metal Ions on Silica Gel Thin Layers in Butanol-Formic Acid Media', Journal of Liquid Chromatography & Related Technologies, 9: 9, 1903 – 1917

**To link to this Article:** DOI: 10.1080/01483918608078751

**URL:** <http://dx.doi.org/10.1080/01483918608078751>

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <http://www.informaworld.com/terms-and-conditions-of-access.pdf>

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

## CHROMATOGRAPHY OF SOME METAL IONS ON SILICA GEL THIN LAYERS IN BUTANOL—FORMIC ACID MEDIA

A. Mohammad and N. Fatima

*Chemistry Section*

*Z. H. College of Engineering and Technology*

*Aligarh Muslim University*

*Aligarh, U. P., India*

### ABSTRACT

Chromatographic behaviour of some metal ions on silica gel thin layers has been studied in butanol - formic acid media. The results have been compared with those obtained in aqueous formic acid medium. Many important and analytically difficult separations have been achieved. Some thin layer chromatographic parameters for the separation of  $\text{VO}^{2+}$  from numerous metal ions using 20.0 M formic acid in butanol have been determined.

### INTRODUCTION

Because of its simplicity and excellent resolving power, silica gel thin layers chromatography has been extensively employed as a universal tool for separating a series of ions and complex mixtures into individual components (1-3). Numerous brands of sorbents and even greater number of mobile phases are being developed to achieve improved

chromatographic performance in terms of selectivity, resolution and reproducibility. Changes in selectivity are possible from a varied choice of mobile phases.

Formic acid (FA) offers numerous advantages as a solvent in chromatography. It forms complexes with many metal ions (4,5), prevents hydrolysis of salts and does not dissolve the ion exchange materials significantly. Its reducing properties do not permit the oxidation of cations during analysis. In 1949, Lacourt and coworkers (6) reported some exciting results of their study on the quantitative separation of aluminium from titanium and iron by paper chromatography in FA medium. Since then, FA has become increasingly popular. So far, many papers have been published on the use of FA as chromatographic solvent (7-13), most of them dealing with the use of FA as eluent for the separations of metal ions by column chromatography using organic resins (7-11). Surprisingly, FA has been used only infrequently as a solvent for chromatographic separations of metal ions on silica gel thin layers (12,13), though the recent findings of Balzer and coworkers (14) point out that the acidic developers such as toluene-ethyl acetate - FA and benzene - acetic acid - methanol are less affected by silica gel properties and give excellent resolution of aflatoxins.

The present work explores the possibility of FA - butanol solvent systems for inorganic thin layer chromatography. As a result, certain very important and difficult separations have been achieved. Butanol was chosen because

it gives clean and useful separations (15). The results of this study are summarized here.

### EXPERIMENTAL

#### Apparatus:

A thin layer chromatography apparatus (Toshniwal, India) for the preparation of silica gel thin layers on 20 x 3.5 cm glass plates was used. The chromatography was performed in 24 x 6 cm glass jars .

#### Reagents:

Silica gel G (SG) from E. Merck and all other chemicals and reagents were of BDH analytical or laboratory reagent grade.

#### Test solutions and detectors:

Solutions, 0.1 M, of chlorides, nitrates, or sulfates of most of the cations were prepared in 0.1 M solutions of the corresponding acids. 1% niobium pentachloride was prepared in 10% tartaric acid according to the method recommended by Sandell (16) and by Fairbrother (17). Solutions of sodium selenite, sodium molybdate and sodium tungstate, 0.1 M, were prepared in water while 0.1 M ceric sulfate was prepared in 3.6 N  $\text{H}_2\text{SO}_4$ . Conventional spot test reagents were used for detection purposes (18).

#### Solvent systems:

The following solvent systems were used in these studies.

1. FA (0.1 M, 0.5 M, 2.0 M, 5.0 M, 10.0 M and 20.0 M) in butanol

2. FA (0.1 M, 0.5 M, 5.0 M, 10.0 M and 20.0 M) in conductivity water
3. Butanol saturated with 5.0 M aqueous FA
4. Butanol saturated with 10.0 M aqueous FA
5. 10.0 M FA in butanol + methyl ethyl ketone (2:1)

#### Preparation of silica gel plates:

The slurry used was prepared by mixing the silica gel with constant shaking for 5 min in conductivity water in the ratio of 1:3. The resultant slurry was used immediately to coat the clean glass plates with the help of an applicator to give a layer of 0.25 mm thickness. These plates were first dried at room temperature and then in an electrically controlled oven at  $100 \pm 5^{\circ}\text{C}$  for 2 hr for complete drying. The plates were kept in closed chamber at room temperature until used.

#### Procedure:

Approximately 5  $\mu\text{l}$  of the test solutions (0.1 M) were applied on each silica gel plate by using a micro-pipette. After drying the spots, development was made in different solvent systems by the ascending technique. The ascent of the solvent was fixed at 10.0 cm in all cases. After development, the plates were dried and the cations were detected using the appropriate spraying reagent.  $R_T$  and  $R_L$  values were measured as usual.

### RESULTS AND DISCUSSION

Thin layer chromatographic studies of metal ions in aqueous FA and non-aqueous FA (FA-butanol) systems offer

some interesting results. A few points are worthy to mention here:

1. The  $R_f$  values for all metal ions studied remain unchanged over the concentration range of 1.0 M to 20.0 M of aqueous FA.  $\text{Mo}^{6+}$  is only the exception which shows decrease in  $R_f$  value with increasing FA concentration. Thus, the most effective concentration range of FA in aqueous medium is 0.1 M to 1.0 M.

2. A reversal trend in mobilities of metal ions is noticed in non-aqueous media containing varying moles of FA in butanol. Most of the metal ions show considerable tailing ( $R_L - R_T > 0.3$ ) in 0.1-2.0 M FA while compact spots were achieved in 5.0-20.0 M FA. Interestingly, 20.0 M FA in butanol was found to be the best solvent to give cleaner, more compact and reproducible spots for all the metal ions.

3.  $\text{Hg}_2^{2+}$  shows considerable tailing in all non-aqueous solvent systems.

4. With increased FA concentration, tailing decreases both in FA-DMW and FA-butanol systems. In sharp contrast to the aqueous FA media, the most appropriate concentration range of FA in butanol was found to be 5.0-20.0 M.

5. A little change in  $R_f$  values has been sometimes observed when mixtures of metal ions were developed.

6. The presence of methyl ethyl ketone in FA-butanol systems improved the resolution of components in mixtures.

7. In FA-butanol systems, a sharp increase in  $R_f$  values for most of cations takes place when the concentra-

tion of FA is increased from 10.0 M to 20.0 M. Therefore, the homogeneity of the mobile phase is an important factor to influence the reproducibility of results.

8. In 20.0 M aqueous FA,  $\text{VO}^{2+}$  shows high mobility and moves with solvent front imposing a restriction on its separation from  $\text{Hg}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{UO}_2^{2+}$ ,  $\text{Se}^{4+}$ ,  $\text{Tl}^+$ ,  $\text{Tl}^{3+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Fe}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Nb}^{5+}$  and  $\text{Ta}^{5+}$ . However, in 20.0 M FA in butanol  $\text{VO}^{2+}$  shows very little mobility while all the metal ions mentioned above move with solvent front leaving behind  $\text{VO}^{2+}$  near the point of application providing an ample opportunity for the separation of  $\text{VO}^{2+}$  from numerous metal ions within 20 min. The clarity and excellent compactness of spots in this solvent give very good separations even if the difference in  $R_f$  values of two components is equal or even less than 0.2 while in aqueous FA media good separations can not be achieved until and unless the difference in  $R_f$  values of two components is more than 0.3.

The results obtained have been summarized in fig. 1 and tables 1- 5. Figure 1 illustrates the dependency of  $R_M$  values of cations on the molar concentration of FA in butanol. Generally,  $R_M$  values for metal ions increase with FA concentration until a maximum is reached at an acid concentration of 10.0 M. However,  $\text{Tl}^+$  and  $\text{Al}^{3+}$  show maximum  $R_M$  values at 5.0 M FA. In case of  $\text{Co}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Cd}^{2+}$  and  $\text{Bi}^{3+}$  a uniform decrease in  $R_M$  values with increase in the acid concentration is observed. Conversely,  $\text{Th}^{4+}$  shows a uniform increase in  $R_M$  value with acid concentration until a

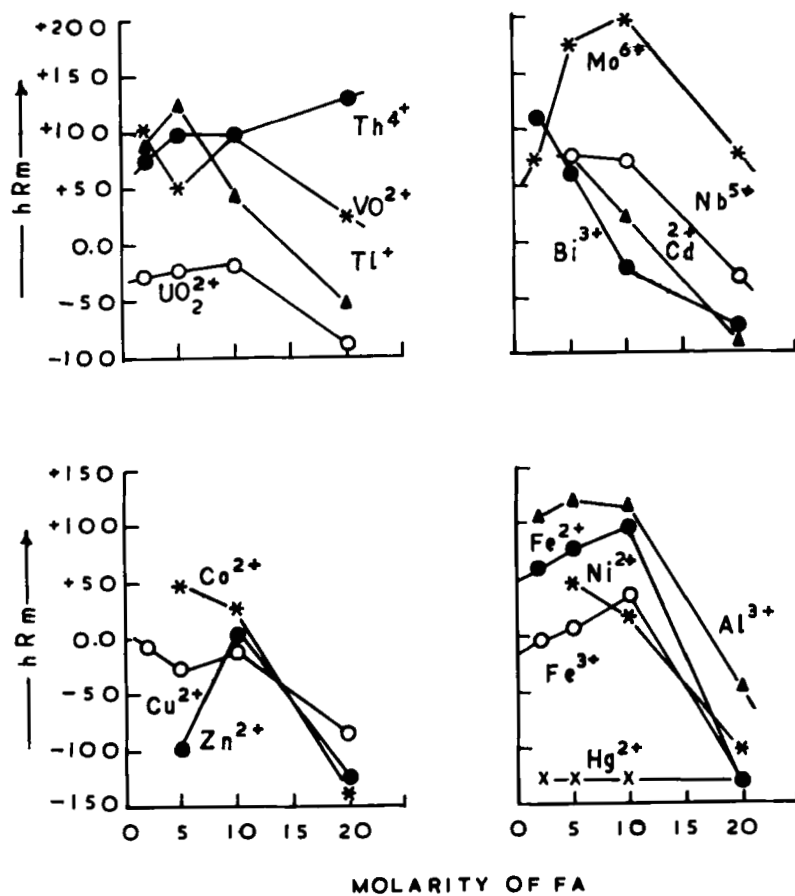


Fig.1 Plots of  $hRm$  Versus Formic Acid Molarity in Butanol



TABLE I

$R_f$  and  $R_i$  values of some Metal Ions on 3G Thin Layers in Acidic Developers Containing Varying Voles of FA

Sl. No.	Metal Ions	0.1 M FA		0.5 M FA		5.0 M FA		10.0 M FA		20.0 M FA	
		$R_f$	$R_i$	$R_f$	$R_i$	$R_f$	$R_i$	$R_f$	$R_i$	$R_f$	$R_i$
1.	Th <sup>4+</sup>	T		T		0.10	ND	0.10	ND	0.05	0.0
2.	Zr <sup>4+</sup>	0.07	-0.07	0.01	-0.01	0.0	0.0	0.0	0.0	0.0	0.0
3.	Hg <sup>2+</sup>	T		T		T		T		T	-
4.	Hg <sup>2+</sup>	0.95	-0.37	0.35	0.0	0.35	-0.05	0.35	-0.05	0.95	-0.30
5.	Bi <sup>3+</sup>	T		T		0.20	-0.10	0.35	-0.30	0.33	+0.07
6.	Cd <sup>2+</sup>	T		T		0.15	+0.20	0.40	+0.52	0.90	+0.02
7.	UO <sub>2</sub>	T		T		0.65	+0.35	0.31	+0.35	0.39	-0.04
8.	Al <sup>3+</sup>	0.0	+0.55	0.05	+0.95	0.05	+0.30	0.07	+0.30	0.73	ND
9.	Se <sup>4+</sup>	T		T		T		0.35	+0.07	0.32	+0.03
10.	Tl <sup>+</sup>	0.05	+0.80	0.07	+0.80	0.05	+0.67	0.26	+0.41	0.75	+0.15
11.	Tl <sup>3+</sup>	0.37	-0.59	0.37	0.0	0.37	0.0	0.35	0.0	ND	-
12.	Ag <sup>+</sup>	T		T		T		0.0	+0.05	0.05	+0.04
13.	Fe <sup>3+</sup>	T		T		0.48	+0.42	0.32	+0.30	0.35	-0.05
14.	Fe <sup>2+</sup>	0.0	+0.20	0.0	+0.53	0.15	+0.77	0.10	+0.84	0.95	-0.05
15.	Cu <sup>2+</sup>	T		T		0.66	+0.29	0.37	+0.55	0.38	+0.07
16.	VQ <sup>2+</sup>	T		-		0.25	+0.70	0.10	+0.35	0.35	+0.57
17.	W <sup>6+</sup>	0.0	ND	0.0	ND	0.0	0.0	0.0	0.0	0.0	0.0
18.	Mo <sup>6+</sup>	0.0	+0.95	0.0	+0.35	0.02	+0.58	0.0	+0.46	0.15	+0.15
19.	Ni <sup>2+</sup>	T		T		0.35	+0.65	0.33	+0.60	0.32	+0.03
20.	Co <sup>2+</sup>	T		T		0.35	+0.65	0.35	+0.60	0.35	0.0
21.	Ce <sup>2+</sup>	T		T		T		0.30	-0.30	ND	-
22.	Zn <sup>2+</sup>	T		T		0.90	+0.02	0.46	+0.50	0.92	+0.03
23.	Pb <sup>2+</sup>	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
24.	Nb <sup>5+</sup>	T		T		0.15	+0.60	0.17	0.71	0.70	+0.16
25.	Ti <sup>4+</sup>	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0

ND = Not detected clearly, T = Tailing

TABLE - 2

Separations achieved Experimentally in Different Solvent Systems containing 0.1 M to 5.0 M FA

Solvent Systems	Separations Achieved, Metal Ions ( $R_L$ - $R_T$ )
0.1 M FA	$Ti^{3+}(1.0-0.90) - UO_2^{2+}(0.40-0.0)$ $Ti^{3+}(1.0-0.95) - Fe^{3+}(0.35-0.0)$ $Ti^{3+}(1.0-0.90)$ from mixture containing $Th^{4+}$ , $Zr^{4+}$ , $Bi^{3+}$ , $UO_2^{2+}$ , $Tl^+$ , $Ag^+$ , $Fe^{2+}$ , $Fe^{3+}$ , $Mo^{6+}$ , $Ce^{4+}$ and $Ti^{4+}$ .
2.0 M FA	$Hg^{2+}(1.0-0.90) - Bi^{3+}(0.55-0.53)$ $Al^{3+}(0.15-0.0) - Zn^{2+}(0.80-0.30)$ $Ti^{4+}(0.0-0.0) - Fe^{2+}(0.60-0.40)$ $Cu^{2+}(0.75-0.40) - Pb^{2+}(0.02-0.0)$ $UO_2^{2+}(0.70-0.55) - Th^{4+}(0.20-0.0)$ $Hg^{2+}(1.0-0.90)$ from mixture containing $Bi^{3+}$ , $Ag^+$ , $Pb^{2+}$ , $Cu^{2+}$ , $Fe^{2+}$ and $Fe^{3+}$ . $Al^{3+}$ from mixture containing $Zn^{2+}$ , $Ni^{2+}$ and $Co^{2+}$ $UO_2^{2+}$ from mixture containing $Th^{4+}$ , $Zn^{2+}$ , $Co^{2+}$ , $Ni^{2+}$ and $Ag^+$ . $Ti^{4+}$ from mixture containing $Ni^{2+}$ , $Co^{2+}$ , $Fe^{3+}$ and $Zn^{2+}$ . $Pb^{2+}$ from mixture containing $Cu^{2+}$ , $Zn^{2+}$ , $Cd^{2+}$ , $Ni^{2+}$ and $Co^{2+}$ .
5.0 M FA	$Fe^{3+}(0.60-0.40) - Al^{3+}(0.02-0.0)$ $Fe^{3+}(0.60-0.35) - Zn^{2+}(1.0-0.80)$ $Mo^{6+}(0.02-0.0) - UO_2^{2+}(0.3-0.2)$ $Hg^{2+}(1.0-0.95) - Cu^{2+}(0.75-0.65)$ $Al^{3+}(0.02-0.0) - Cu^{2+}(0.72-0.60)$ $Ti^{3+}(1.0-0.95) - Tl^+(0.02-0.0)$ $Zr^{4+}(0.02-0.0) - UO_2^{2+}(0.70-0.60)$ $Ti^{4+}(0.0-0.0) - UO_2^{2+}(0.70-0.50)$ $Zr^{4+}(0.02-0.0) - Cu^{2+}(0.72-0.60)$ $Hg^{2+}(1.0-0.95) - Hg_2^{2+}(0.3-0.55)$ $Fe^{3+}(0.55-0.35) - Al(0.02-0.0) - Zn^{2+}(1.0-0.8)$ $UO_2^{2+}(0.75-0.68)$ from mixture containing $Zr^{4+}$ , $Th^{4+}$ , $Co^{3+}$ , $Ni^{2+}$ and $Ti^{4+}$
Butanol saturated with 5.0M aqueous FA	$Mo^{6+}(0.0-0.0) - UO_2^{2+}(0.62-0.45)$ $Hg^{2+}(1.0-0.92) - Cu^{2+}(0.15-0.0)$ $Hg^{2+}(1.0-0.90)$ from mixture containing $Cu^{2+}$ , $Cd^{2+}$ and $Zn^{2+}$

TABLE - 3

Separations Achieved Experimentally in FA - butanol and FA - butanol-H<sub>2</sub>O  
Solvents Containing 10.0M FA

Solvent	Separations Achieved ( $R_L$ - $R_T$ )
10.0 M FA in butanol	$Tl^{3+}$ (0.2-0.0) - $Tl^{3+}$ (0.35-0.80) $Hg_2^{2+}$ (0.60-0.0) - $Hg_2^{2+}$ (1.0-0.95) $VO_2^{2+}$ (0.10-0.0) - $UO_2^{2+}$ (0.5-0.3) $Mo^{6+}$ (0.05-0.0) - $UO_2^{2+}$ (0.42-0.25) $Th^{4+}$ (0.10-0.0) - $UO_2^{2+}$ (0.46-0.30) $Tl^{3+}$ (0.30-0.20) - $Hg_2^{2+}$ (1.0-0.95) $Cu^{2+}$ (0.40-0.25) - $Pb^{2+}$ (0.0-0.0) $Cu^{2+}$ (0.40-0.25) - $Fe^{2+}$ (0.05-0.0) $Cu^{2+}$ (0.40-0.25) - $Al^{3+}$ (0.02-0.0) $Fe^{3+}$ (0.46-0.30) - $Al^{3+}$ (0.02-0.0) $UO_2^{2+}$ (0.50-0.35) - $Fe^{2+}$ (0.05-0.0) $Ce^{4+}$ (0.25-0.15) - $Th^{4+}$ (0.02-0.0) $Ce^{4+}$ (0.25-0.15) - $Tl^{3+}$ (0.23-0.83) $W^{6+}$ (0.0-0.0) - $Fe^{3+}$ (0.4-0.22) $Zr^{4+}$ (0.0-0.0) - $UO_2^{2+}$ (0.60-0.50) $Ce^{4+}$ (0.40-0.22) - $VO_2^{2+}$ (0.05-0.0) $Se^{4+}$ (0.00-0.20) - $Tl^{3+}$ (0.32-0.20) $Bi^{3+}$ (0.70-0.50) - $Hg_2^{2+}$ (1.0-0.95) - $Ag^{+}$ (0.0-0.0) $Se^{4+}$ (0.38-0.20) from mixture containing $Fe^{2+}$ and $Pb^{2+}$ $Tl^{3+}$ (0.30-0.20) from mixture containing $Ag^{+}$ , $Pb^{2+}$ and $Hg_2^{2+}$ $Ti^{4+}$ (0.0-0.0) from mixture containing $Cd^{2+}$ , $Fe^{3+}$ , $Hg_2^{2+}$ , $Se^{4+}$ , $Ce^{4+}$ , $Zn^{2+}$ , $Ni^{2+}$ and $Co^{2+}$
10.0 M FA in butanol +methyl ethyl ketone(2:1)	$Al^{3+}$ (0.0-0.0) - $Fe^{3+}$ (0.60-0.40) $Th^{4+}$ (0.15-0.0) - $UO_2^{2+}$ (0.66-0.50) $VO_2^{2+}$ (0.05-0.0) - $UO_2^{2+}$ (0.63-0.52) $Ta^{5+}$ (0.0-0.0) - $UO_2^{2+}$ (0.70-0.55) $Bi^{3+}$ (1.0-0.90) from mixture containing $Pb^{2+}$ , $Ag^{+}$ and $Fe^{2+}$ $UO_2^{2+}$ (0.70-0.60) from mixture containing $Pb^{2+}$ , $Ag^{+}$ , $Al^{3+}$ , $Th^{4+}$ , $Zr^{4+}$ , $Mo^{6+}$ , $Ni^{2+}$ , $Co^{2+}$ , $W^{6+}$ , $Ti^{4+}$ , $VO_2^{2+}$ , $Se^{4+}$ and $Ce^{4+}$
Butanol saturated with 10.0 M FA	$Hg_2^{2+}$ (1.0-0.95) - $Hg_2^{2+}$ (0.75-0.50) $Hg_2^{2+}$ (1.0-0.95) - $Cd^{2+}$ (0.45-0.10) $Hg_2^{2+}$ (1.0-0.95) - $Cu^{2+}$ (0.45-0.16) $Hg_2^{2+}$ (1.0-0.93) - $Bi^{3+}$ (0.40-0.22) $Hg_2^{2+}$ (1.0-0.92) - $UO_2^{2+}$ (0.60-0.35) $Hg_2^{2+}$ (1.0-0.92) from mixture containing $Cd^{2+}$ , $Zn^{2+}$ , $As^{3+}$ , $Fe^{3+}$ , $Cu^{2+}$ , $Ag^{+}$ , $Pb^{2+}$ , $Co^{2+}$ , $Ni^{2+}$ , $Ce^{4+}$ and $W^{6+}$ $Hg_2^{2+}$ (1.0-0.90) from mixture containing $UO_2^{2+}$ , $VO_2^{2+}$ , $Mo^{6+}$ , $Ti^{4+}$ , $Al^{3+}$ , $Th^{4+}$ , $Se^{4+}$ and $Nb^{5+}$ $Tl^{3+}$ (0.96-0.86) from mixture containing $Zr^{4+}$ , $Cu^{2+}$ , $Th^{4+}$ , $Cd^{2+}$ , Z

TABLE - 4

Separations Achieved Experimentally in 20.0 M FA in Butanol

Solvent	Separations Achieved ( $R_L - R_T$ )
20.0 M FA	$Zn^{4+}(0.0-0.0) - Nb^{5+}(0.85-0.65)$ $Zr^{4+}(0.0-0.0) - UO_2^{2+}(1.0-0.95)$ $Th^{4+}(0.10-0.0) - UO_2^{2+}(0.92-0.80)$ $Ti^{4+}(0.0-0.0) - Al^{3+}(0.60-0.55)$ $Ti^{4+}(0.0-0.0) - UO_2^{2+}(0.95-0.80)$ $Ge^{4+}(1.0-0.85) - Pb^{2+}(0.0-0.0)$ $Mo^{6+}(0.00-0.0) - Fe^{3+}(1.0-0.90)$ $Mo^{6+}(0.00-0.05) - Cu^{2+}(0.90-0.80)$ $Mo^{6+}(0.00-0.05) - UO_2^{2+}(1.0-0.95)$ $Al^{3+}(0.65-0.70) - Fe^{3+}(1.0-0.92)$ $Bi^{3+}(0.05-0.85) - Ag^{+}(0.0-0.0)$ $Pb^{2+}(0.0-0.0) - Cd^{2+}(0.80-0.60)$ $Pb^{2+}(0.0-0.0)$ from mixture containing $Cu^{2+}, Zn^{2+},$ $Cd^{2+}$ and $Bi^{3+}$ $Tl^{+}(0.00-0.80)$ from mixture containing $Ag^{+}$ and $Pb^{2+}$ $Mo^{6+}(0.00-0.05)$ from mixture containing $Fe^{2+}, Fe^{3+},$ $Cu^{2+}, Ni^{2+}, Nb^{5+}$ and $Co^{2+}$ $Ti^{4+}(0.0-0.0)$ from mixture containing $Fe^{2+}, Fe^{3+},$ $Al^{3+}, Co^{2+}, Ni^{2+}$ and $Zn^{2+}$ $Al^{3+}(0.82-0.65)$ from mixture containing $Ni^{2+}$ and $Co^{2+}$ $Bi^{3+}(0.05-0.85)$ from mixture containing $Ag^{+}$ and $Pb^{2+}$ $UO_2^{2+}(0.50-0.30)$ from mixture containing $F^{6+}, Mo^{6+},$ $Cd^{2+}, Zn^{2+}, Cu^{2+}, Hg^{2+}, Hg_2^{2+}, Ag^{+}, Pb^{2+}, Bi^{3+},$ $UO_2^{2+}, Fe^{3+}, Ni^{2+}$ and $Co^{2+}$

maximum is reached at 20.0 M FA. Interestingly, all the metal ions except  $Th^{4+}$  show minimum  $R_M$  value (high mobility) in 20.0 M FA.  $Hg^{2+}$  exhibits a constant low  $R_M$  value at all acid concentrations. The low  $R_M$  value at high acid concentration is because of the presence of large number of  $H^+$  ions which compete with cations in question for the exchange sites and the equilibrium

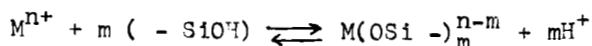


TABLE - 5

$R_s$ ,  $\alpha$  and  $\Delta R_f$  Values for the Separation of  $VO^{2+}$  from Various Metal Ions in 20.0 M FA in Butanol

S.No.	Separation pair	Value of $R_s$	Value of $\alpha$	Value of $\Delta R_f$
1.	$VO^{2+} - Cd^{2+}$	1.95	16.82	0.42
2.	$VO^{2+} - Fe^{2+}$	2.60	37.0	0.65
3.	$VO^{2+} - Fe^{3+}$	2.60	37.0	0.65
4.	$VO^{2+} - Zn^{2+}$	1.77	21.6	0.48
5.	$VO^{2+} - Cu^{2+}$	2.75	13.6	0.55
6.	$VO^{2+} - Ni^{2+}$	2.4	21.6	0.60
7.	$VO^{2+} - Co^{2+}$	2.74	37.0	0.63
8.	$VO^{2+} - Hg^{2+}$	1.81	37.0	0.58
9.	$VO^{2+} - Hg_2^{2+}$	1.81	37.0	0.58
10.	$VO^{2+} - Pb^{2+}$	2.12	-	0.34
11.	$VO^{2+} - Al^{3+}$	1.84	5.0	0.46
12.	$VO^{2+} - UO_2^{2+}$	2.59	15.43	0.57
13.	$VO^{2+} - Ta^{5+}$	2.41	10.51	0.53
14.	$VO^{2+} - Tl^+$	1.59	5.60	0.43
15.	$VO^{2+} - V^{6+}$	2.12	-	0.34
16.	$VO^{2+} - Se^{4+}$	2.40	21.26	0.60
17.	$VO^{2+} - Nb^{5+}$	1.88	4.30	0.49

is shifted to the left resulting high mobility for these cations.  $Th^{4+}$  is strongly adsorbed on silica gel due to some special interactions leading to high  $R_M$ .

The  $R_f$  values of metal ions in FA-butanol solvent systems alongwith the  $R_i$  values ( $R_i = R_f$  in aqueous FA media -  $R_f$  in butanol - FA media) have been tabulated in table 1. It is evident from table 1 that FA - butanol systems of varied FA concentrations are excellent solvents with numerous possibilities of analytically important separations.

Some of those have been actually achieved (Tables 2-4), the most important ones being as follow:  $\text{Hg}_2^{2+}$  -  $\text{Hg}^{2+}$ ,  $\text{UO}_2^{2+}$  -  $\text{Th}^{4+}$ ,  $\text{Bi}^{3+}$  -  $\text{Hg}^{2+}$  -  $\text{Ag}^+$ ,  $\text{Se}^{4+}$  -  $\text{Ti}^+$ ,  $\text{Cu}^{2+}$  -  $\text{Fe}^{2+}$ ,  $\text{Cu}^{2+}$  -  $\text{VO}^{2+}$ ,  $\text{VO}^{2+}$  -  $\text{Nb}^{5+}$ ,  $\text{Al}^{3+}$  -  $\text{Ti}^{4+}$ ,  $\text{Al}^{3+}$  -  $\text{Fe}^{3+}$ ,  $\text{UO}_2^{2+}$  -  $\text{Ti}^{4+}$ ,  $\text{Pb}^{2+}$  -  $\text{Bi}^{3+}$ ,  $\text{Zr}^{4+}$  -  $\text{UO}_2^{2+}$ ,  $\text{Zr}^{4+}$  -  $\text{Nb}^{5+}$  and  $\text{VO}^{2+}$  from mixtures containing  $\text{UO}_2^{2+}$ ,  $\text{Mo}^{6+}$ ,  $\text{W}^{6+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Hg}_2^{2+}$ ,  $\text{Hg}^{2+}$ ,  $\text{Ag}^+$ ,  $\text{Pb}^{2+}$ ,  $\text{Bi}^{3+}$ ,  $\text{Co}^{2+}$  and  $\text{Fe}^{3+}$ .

Table 5 summarizes the values of separation factor ( $\alpha$ ), resolution ( $R_s$ ) and  $\Delta R_f$  ( $R_f$  of metal ion -  $R_f$  of  $\text{VO}^{2+}$ ) for the separation of  $\text{VO}^{2+}$  from numerous metal ions in synthetic mixtures. These parameters on thin layers were calculated by using the following equations:

$$\alpha = \frac{K' \text{ of } \text{VO}^{2+}}{K' \text{ of separating metal ion}} \quad (i)$$

$$\text{where } K' = \frac{1 - R_f}{R_f}$$

Thus,  $\alpha$  is the ratio of capacity factors  $K'_1/K'_2$  for two solutes. Capacity factor is the measure of the degree of retention of a solute compared to the solvent front.

$$R_s = \frac{\Delta X}{\frac{1}{2}(d_1 + d_2)} \quad (ii)$$

where  $\Delta X$  is the distance between the centers of spots of the separating metal ions while  $d_1$  and  $d_2$  are their respective diameters. The two metal ions are just separated when  $R_s = 1$ .

A perusal of the data given in table 5 shows that 20.0 M FA in butanol is a very selective solvent for achieving reliable separations of  $\text{VO}^{2+}$  from many metal ions. The

$R_f$  values are always in reasonable range leaving to well separated separations of  $VO^{2+}$ . In all cases, the  $R_s$  values for the separation of  $VO^{2+}$  are nearly equal to 2.0 indicating well resolved spots. The fairly high value of for the separation of  $VO^{2+}$  from almost all cations shown in table 5 indicates the excellent separation potentially of butanol - FA systems.

#### ACKNOWLEDGEMENT

One of the authors M.F. is highly thankful to CSIR, New Delhi (India) for financial assistance.

#### REFERENCES

1. Brinkman, U.A. Th., De Vries, G. and Kuroda, R., J. Chromatogr., 85, 187, 1973.
2. Fried, B. and Sherma, J., Thin Layer Chromatography: Techniques and Applications, Marcel Dekker, New York, 1982.
3. Touchstone, J.C. and Rogers, D., Thin Layer Chromatography: Quantitative Environmental and Clinical Applications, Wiley Interscience, New York, 1979.
4. Korkisch, J. and Urabay, S., Talanta, 11, 721, 1964.
5. Funk, H., Muller, C. and Paul, A., Z. Chem. 6, 227, 1966.
6. Lacourt, A., Sommereyns, G., Dezeyndt, E., Baruh, J. and Gillard, J., Nature, 163, 999, 1949.
7. Shishkov, D.A. and Koleva, E.G., Talanta, 12, 865, 1965.
8. Qureshi, M., Husain, W. and Israilli, A.H., Talanta, 15, 789, 1968.
9. Qureshi, M. and Husain, W., Talanta, 18, 399, 1971.
10. Qureshi, M. and Husain, K., Anal. Chim. Acta, 57, 387, 1971.
11. Qureshi, M., Varshney, K.G. and Kaushik, R.C., Anal. Chem. 45, 2433, 1973.

12. Qureshi, M., Thakur, J.S. and Qureshi, P.M., J.Liquid Chromatogr., 3, 605 (1980).
13. Fatima, N. and Mohammad, A., Sep. Sci. Technol., 19 429, 1984.
14. Balzer, I., Bogdanic, C. and Pepeljnjak, S., J. Assoc. Anal. Chem., 61, 584, 1978.
15. Qureshi, M., Akhtar, I. and Mathur, K. N., Anal Chem., 39, 1766, 1967.
16. Sandell, E.B., Colorimetric Determination of Traces of Metals, 3rd ed., Interscience, New York, N.Y., 1959, p. 682.
17. Fairbrother, F., The Chemistry of Niobium and Tantalum, Elsevier, Amsterdam, 1967, p. 235.
18. Qureshi, M., Varshney, K. G. and Rajput, R. P. S., Anal. Chem., 47, 1520, 1975.