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Reversed-Phase Thin-Layer Chromatography of Inorganic Anions on Tri-*n*-Butyl Amine Impregnated Silica Gel-G Layers: Separation of Coexisting I^-, IO_3^- and IO_4^-, $Fe(CN)_6^{4-}$ and $Fe(CN)_4^{3-}$

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Key Words:

Reversed-phase thin-layer chromatography
Tri-*n*-butyl amine impregnation
Inorganic anions
Silica gel-G layers
Separation of anions

Summary

The chromatographic behavior of 24 inorganic anions has been studied on tri-*n*-butyl amine (TBA) impregnated silica gel-G layers using single solvents, two-component non-aqueous mixtures, and aqueous solutions of organic acids as mobile phases. The mechanism of migration is explained in terms of adsorption, precipitation, solubility of sodium or potassium salts of the anions, and the polarity of the mobile phase used. The effect of pk_1 of the complexing acids and that of dielectric constant (∈) of non-aqueous solvents used as mobile phases on the R_F values of anions are discussed. Twenty-percent TBA impregnated silica gel-G layers are found quite effective in the separation of anions. The effect of the addition of DMF to other organic solvents on the R_F values is also discussed. A number of useful binary and ternary separations are achieved, *e.g.*, the separation of coexisting I^-, IO^{3-}, and IO^{4-} and of $Fe(CN)_6^{4-}$ and $Fe(CN)_6^{3-}$ from their mixtures. The R_F values of the anions are found to be in accordance with their lyotropic numbers.

1 Introduction

Since 1995, reversed-phase thin-layer chromatography (RP-TLC) of metal ions has been performed by using different stationary and mobile phases [1–3]. Silica gel admixtures [4–6] and impregnated silica gel layers [7–9] have also been used for the purpose. We have used this technique for the qualitative and quantitative separation of metal ions on TBP and TBA impregnated silica gel-G [10–13] and silica gel G F_{254} layers [14]. RP-TLC of some d-block metal ions on talc layers [15] has also been carried out in this laboratory. In these studies, tri-*n*-butyl amine (TBA) has proved to be a useful impregnating material for the phase reversal, and 20% TBA-impregnated silica gel-G layers were found the most effective for metal ion separations. Lately, there was a resurgence of interest in the normal-phase TLC separation of metal ions [16–27]. However, no such effort was made for the TLC and RP-TLC of anions.

Earlier, *Mohammad et al.* have admirably reviewed the TLC of inorganic anions up to 1995 [28]. During 1995–2002, some studies on normal-phase TLC of inorganic anions have been carried out [29–36]. Later, *Sharma et al.* made an extensive study on normal-phase TLC of anions using silica gel-starch layers [37]. Much earlier, only 2 studies were reported on the reversed-phase column chromatography of anions [38–39]. But, as far as we are aware, the RP-TLC of anions has not yet been attempted and as such, the potential of such a study has not been explored.

It has therefore been decided to perform the RP-TLC of inorganic anions on 20% TBA-impregnated silica gel-G layers in aqueous and non-aqueous mobile phases. As a result, a number of analytically important binary and ternary separations have been achieved.

2 Experimental

2.1 Reagents

TBA from LOBA, silica gel-G from E. Merck (Mumbai, India), and all other chemicals were of AnalaR grade.

2.2 Test Solutions and Detectors

Test solutions, 0.1 м, of anions were prepared by dissolving sodium or potassium salts of anions in distilled water.

Detection was made by the following reagents: saturated solutions of $AgNO_3$ in methanol for Cl^-, Br^-, I^-, AsO_3^{3-}, AsO_4^{3-}, CrO_4^{2-}, $Cr_2O_7^{2-}$, $S_2O_3^{2-}$, and IO_4^-; 1% solution of $FeCl_3$ in 2 м HCl for $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$, SCN^-, and N_3^-; 2% methanolic diphenylamine solution containing 4 м H_2SO_4 for BrO_3^-, NO_2^-, NO_3^-, VO_3^-, MnO_4^-, and IO_3^-; 2% alcoholic solution of pyrogallol for

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MoO_4^{2-} and WO_4^{2-}; aqueous molybdate followed by $SnCl_2$ solution in 4 M HCl for PO_4^{3-}; dilute $ZrCl_4$ solution in HCl and 0.1 M alcoholic alizarin red S for F^-; and ferrous sulfate solution in 30% phosphoric acid for SeO_3^{2-}.

2.3 Mobile Phases

The following 13 mobile phases were used: M_1, methanol; M_2, dimethylformamide (DMF); M_3, diisopropyl ether; M_4, tetrahydrofuran (THF); M_5, 2-propanol; M_6, DMF–methanol (1:1); M_7, DMF–diisopropyl ether (1:1), M_8, DMF–THF (1:1), M_9, DMF–2-propanol (1:1); M_{10}, 0.1 M oxalic acid; M_{11}, 0.1 M tartaric acid; M_{12}, 0.1 M citric acid; and M_{13}, 0.1 M succinic acid.

2.4 Chromatography

The details of apparatus, preparation of chromatoplates, procedure for chromatography, *etc.* are the same as given earlier [13, 37].

3 Results and Discussion

Planar chromatography of 24 inorganic anions have been performed, and their retention behavior on 20% TBA-impregnated silica gel-G layers in aqueous and non-aqueous mobile phases was studied. Tailing was observed only for the following: BrO_3^- (M_2); I^-, BrO_3^-, and $S_2O_3^{2-}$ (M_6); NO_2^- (M_7 and M_9); and PO_4^{3-} (M_{12}). In order to check the reproducibility of the results, 3 sets of each anion were chromatographed in 0.1 M oxalic acid, and it was found that the variation does not exceed 10% of the average R_F value. The R_F values of all these anions are given in **Table 1.**

3.1 Aqueous Organic Acids as Mobile Phases

To study the effect of various organic acids used as the mobile phase on the chromatographic behavior of anions, plots of R_F vs. pk_1 of the acids were drawn **(Figure 1).** A cursory inspection of these plots led to the following conclusions.

Table 1

R_F values of inorganic anions on 20% TBA-impregnated silica gel-G layers (T = tailing; N.D. = not detected).

Inorganic anions	Mobile phases												
	M_1	M_2	M_3	M_4	M_5	M_6	M_7	M_8	M_9	M_{10}	M_{11}	M_{12}	M_{13}
VO_3^-	0.04	0.00	0.00	0.00	0.00	0.04	0.00	0.00	0.00	0.03	0.12	0.95	0.03
AsO_3^{3-}	0.35	0.06	0.00	0.15	0.13	0.25	0.05	0.10	0.12	0.65	0.75	0.45	0.75
AsO_4^{3-}	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.60	0.55	0.98	0.65
N_3^-	0.80	0.85	0.00	0.03	0.00	0.80	0.75	0.81	0.75	N.D.	N.D.	N.D.	0.65
BrO_3^-	0.80	0.75	0.00	0.40	0.78	0.95T	0.87	0.82	0.90	0.98	0.95	0.98	0.95
$S_2O_3^{2-}$	0.80	0.57T	0.00	0.00	0.00	0.85T	0.15	0.75	0.30	0.95	0.95	0.98	0.35
SCN^-	0.82	0.65	0.00	0.75	0.87	0.85	0.80	0.85	0.85	0.85	0.85	0.75	0.85
F^-	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.15	0.07	0.10	0.02
Cl^-	0.80	0.70	0.00	0.15	0.55	0.70	0.05	0.10	0.85	0.95	0.95	0.95	0.95
Br^-	0.80	0.97	0.00	0.00	0.85	0.80	0.10	0.20	0.80	0.95	0.95	0.95	0.96
I^-	0.88	0.75	0.00	0.00	0.72	0.70T	0.05	0.10	0.85	0.68	0.55	0.95	0.80
MnO_4^-	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	N.D.	0.02	0.20	0.00
CrO_4^{2-}	0.80	0.80	0.00	0.00	0.00	0.80	0.00	0.00	0.00	0.96	0.85	0.85	0.82
$Cr_2O_7^{2-}$	0.87	0.82	0.00	0.00	0.75	0.70	0.00	0.70	0.92	0.22	0.20	0.20	0.13
$Fe(CN)_6^{3-}$	090	0.97	0.00	0.10	0.78	0.85	0.85	0.87	0.85	0.95	0.98	0.95	0.96
$Fe(CN)_6^{4-}$	0.82	0.00	0.00	0.00	0.00	0.40	0.00	0.00	0.00	0.99	0.99	0.99	0.99
MoO_4^{2-}	0.92	0.13	0.00	0.00	0.00	0.67	0.05	0.08	0.00	0.98	0.85	0.98	0.98
SeO_3^{2-}	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.95	0.98	0.85	0.92
WO_4^{2-}	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.27	0.55	0.80	0.00
NO_2^-	0.80	0.86	0.00	0.00	0.00	0.67	0.44T	0.55	0.62T	0.95	0.98	0.65	0.90
NO_3^-	0.77	0.80	0.00	0.00	0.00	0.82	0.70	0.90	0.75	0.99	0.92	0.90	0.90
PO_4^{3-}	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.42	0.55	0.75T	0.50
IO_3^-	0.50	0.13	0.00	0.00	0.00	0.15	0.00	0.00	0.00	0.98	0.90	0.75	0.80
IO_4^-	0.00	0.00	0.00	0.15	0.00	0.00	0.00	0.00	0.05	0.82	0.80	0.80	0.80

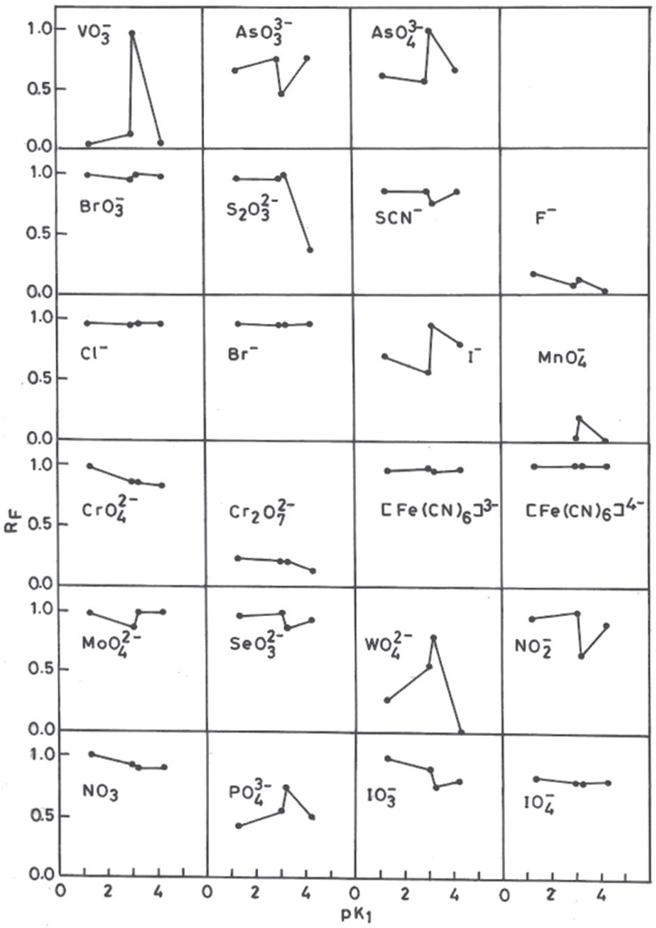

Figure 1

Plots of R_F vs. pk$_1$ of acids.

(1) The anions, such as BrO_3^-, SCN^-, Cl^-, Br^-, CrO_4^{2-}, $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$, MoO_4^{2-}, SeO_3^{2-}, NO_3^-, IO_3^-, and IO_4^-, give higher R_F irrespective of the mobile phases used. It may be due to the higher solubility of the salts of these ions. Lesser adsorption of these anions on TBA-impregnated layers may be another reason for their higher R_F.

(2) In citric acid media, the anions, *viz.*, VO_3^-, AsO_4^{3-}, PO_4^{3-}, WO_4^{2-}, and I^-, give much higher R_F values as compared to the values reported for other mobile phases. It may be due to the higher solubility of these anions.

(3) In oxalic acid media, MnO_4^- escapes detection probably due to its reduction.

(4) F^- gives low R_F in all the aqueous mobile phases used in comparison to other anions of its class, such as Cl^-, Br^-, and I^-. It is due to the very low solubility of fluoride as compared to other sodium halides (solubilities of NaF, NaCl, NaBr, and NaI are 4.17, 36, 90.5, and 178.7 g per 100 g of water at 20°C, respectively).

(5) For all the mobile phases, $Cr_2O_7^{2-}$ gives lower R_F as compared to CrO_4^2. It may be due to the large size of $Cr_2O_7^{2-}$ which led to its strong adsorption on TBA-impregnated layers, causing lower R_F. Another reason for it may be the low solubility of potassium dichromate as compared to potassium chromate (the solubilities of K_2CrO_4 and $K_2Cr_2O_7$ are 61.7 and 13.1 g per 100 g of water at 20°C, respectively.)

3.2 Non-Aqueous Organic Solvents as Mobile Phases

In non-aqueous mobile phases, various anions give double spots, one being at the point of application (R_F = 0.00), and the other has significant R_F value. These anions (mobile phase given in parenthesis) are given below:

BrO_3^- (M_1); Cl^- and $Fe(CN)_6^{3-}$ (M_2); BrO_3^- (M_4); BrO_3^-, $Cr_2O_7^{2-}$, and $Fe(CN)_6^{3-}$ (M_5); BrO_3^- $S_2O_3^{2-}$, $Cr_2O_7^{2-}$ and $Fe(CN)_6^{3-}$ (M_6); BrO_3^- and $Fe(CN)_6^{3-}$ (M_7); AsO_4^{3-}, BrO_3^-, $S_2O_3^{2-}$, $Cr_2O_7^{2-}$, and $Fe(CN)_6^{3-}$ (M_8); BrO_3^-, Cl^-, Br^-, I^-, $Cr_2O_7^{2-}$, and $Fe(CN)_6^{3-}$ (M_9). The double spots may be due to these anions being partially soluble in the given mobile phase. The insoluble part remaining at the point of application resulted in zero R_F.

(a) To study the effect of the polarity of various non-aqueous solvents on the migration behavior of anions, plots of R_F vs. dielectric constant (DEC) of these solvents used as mobile phases were drawn **(Figure 2).** In the case of anions having double spots, the one suggesting a significant R_F value has been taken into consideration. The salient features are the following.

(1) All the anions exhibit zero migration in diisopropyl ether which may be due to the very low polarity

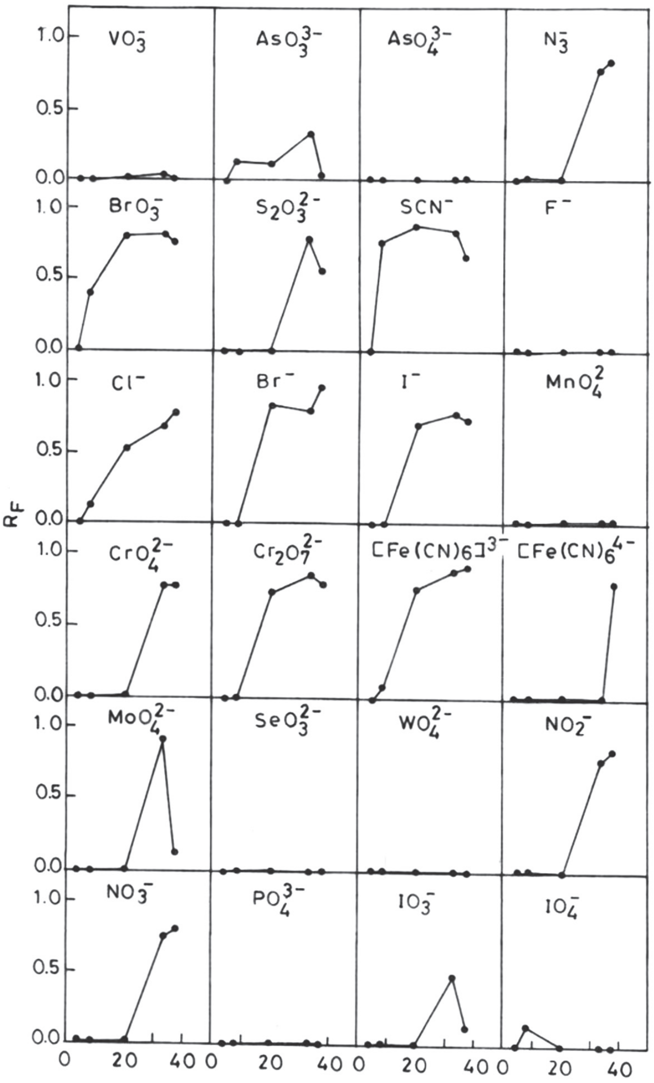

Figure 2

Plots of R_F vs. dielectric constant (∈) of mobile phases.

of the mobile phase. $\in$ for diisopropyl ether is 3.39, whereas for MnO_4^-, precipitation mechanism holds good (**Table 2**).

(2) In THF media, only SCN^- and BrO_3^- show significant movement whereas all the other anions give zero or very low R_F. The zero R_F for MnO_4^-, F^-, and MoO_4^{2-} is due to precipitation (Table 2). Further the movement of SCN^- is greater than that of BrO_3^-. This is because of the lesser adsorption of SCN^- on these payers.

(3) The anions, such as VO_3^-, AsO_4^{3-}, F^-, MnO_4^-, SeO_3^{2-}, WO_4^{2-}, and PO_4^{3-}, give zero R_F irrespective of the mobile phase used. It may probably be due to low solubility or strong adsorption of these anions on TBA-impregnated layers (the solubility of NaF in methanol at 20°C is 0.42 g per 100 g). In the case of MnO_4^-, it is due to the precipitation mechanism (Table 2).

(4) The case of Cl^- and $Fe(CN)_6^{3-}$ is quite interesting as their R_F values increase with an increase in the dielectric constant ($\in$) of the mobile phase used. This is probably due to an increase in the polar nature of the mobile phase concerned.

(5) In methanol, most of the anions such as AsO_3^{3-}, N_3^-, BrO_3^-, $S_2O_3^{2-}$, SCN, CrO_4^{2-}, MoO_4^{2-}, NO_2^-, NO_3^-, and IO_3^- give higher R_F, which can be attributed to the relatively higher polarity of methanol. The dielectric constant of the various non-aqueous solvents used as mobile phases lies in the following order:

DMF > methanol > 2-propanol > THF > diisopropyl ether.

(b) The anions that have R_F values zero or less than 0.05 do so, owing to the following: (1) precipitation, (2) strong adsorption, and (3) very low solubility of sodium or potassium salt of the anion in the given mobile phase.

On mixing solution of an anion with TBA followed by the addition of different mobile phases, it was found that for VO_3^-, MnO_4^-, F^-, MoO_4^{2-}, and IO_3^-, a precipitate was obtained. In such cases, the zero or very low R_F values on TBA-impregnated layers are therefore due to the precipitation mechanism (Table 2).

(c) To study the effect of the addition of DMF to other organic solvents used as the mobile phase on the retention behavior of anions, plots of R_F vs. anions in single mobile phase and admixtures of various mobile phases with DMF are given **(Figures 3–6).** From these plots, the following conclusions are drawn.

(1) It is evident that almost all the anions exhibit similar trend in methanol and methanol–DMF as the DEC for both is not much different. The only exceptions are $Fe(CN)_6^{4-}$ and IO_3^-, which exhibit higher R_F in methanol than in methanol–DMF (1:1) media (ΔR_F is 0.44 for $Fe(CN)_6^{4-}$ and is 0.35 for IO_3^-). This difference in R_F may be due to the higher adsorption of these ions in DMF–methanol media.

(2) In diisopropyl ether and diisopropyl ether–DMF (1:1) media, the anions such as N_3^-, BrO_3^-, SCN^-, $Fe(CN)_6^{3-}$, NO_2^-, and NO_3^- show a remarkable increase in their R_F value, when we add DMF into diisopropyl ether. This is due to the presence of DMF which has a much higher dielectric constant than that of diisopropyl ether (DEC values for DMF and diisopropyl ether are 36.71 and 3.59, respectively).

(3) In THF–DMF (1:1) media, the anions AsO_4^{3-}, N_3^-, BrO_3^-, $S_2O_3^{2-}$, Br^-, I^-, $Cr_2O_7^{2-}$, $Fe(CN)_6^{3-}$, NO_2^-, and NO_3^- give higher R_F values as compared to those taken in THF media. The higher R_F values for these ions may be attributed to the much higher DEC of DMF as compared to THF (the DEC of THF is 7.58).

Table 2

Precipitation of anions in the mixture of the impregnated material and the mobile phase used.

Mobile phase	Anion + TBA + mobile phase	
	Anion which precipitate	Anions which do not precipitate
M_1	MnO_4^-	VO_3^-, AsO_4^{3-}, F^-, WO_4^{2-}, PO_4^{3-}, SeO_3^{2-}, IO_4^{4-}
M_2	Mn	VO_3^-, AsO_3^{3-}, AsO_4^{3-}, F^-, $Fe(CN)_6^{4-}$, WO_4^{2-}, SeO_3^{2-}, IO_4^-, PO_4^{3-}
M_3	MnO_4^-	VO_3^-, AsO_3^{3-}, AsO_4^{3-}, N_3^-, BrO_3^-, $S_2O_3^{2-}$, SCN^-, F^-, Cl^-, Br^-, I^-, CrO_4^{2-}, $Cr_2O_7^{2-}$, $Fe(CN)_6^{4-}$, MoO_4^{2-}, SeO_3^{2-}, WO_4^{2-}, NO_2^-, NO_3^-, PO_4^{3-}, IO_3^-, IO_4^-
M_4	MnO_4^-, F^-, MoO_4^{2-}	AsO_4^{3-}, VO_3^-, N_3^-, $S_2O_3^-$, Cl^-, Br^-, I^-, CrO_4^{2-}, $Cr_2O_7^{2-}$, $Fe(CN)_6^{2-}$, SeO_3^{2-}, WO_4^{2-}, NO_2^-, NO_3^-, $PO4_3^-$, IO_3^-
M_5	MnO_4^-, IO_3^-	MoO_4^{2-}, $S_2O_3^{2-}$, VO_3^-, AsO_4^{3-}, N_3^-, F^-, CrO_4^{2-}
M_6	MnO_4^-	VO_3^-, F^-, SeO_3^{2-}, WO_4^{2-}, PO_4^{3-}, PO_4^{3-}, IO_4^-
M_7	MnO_4^-	VO_3^-, AsO_3^{3-}, AsO_4^{3-}, F^-, CrO_4^{2-}, CrO_4^{2-}, $Cr_2O_7^{2-}$ $Fe(CN)_6^{4-}$, SeO_3^{2-}, WO_4^{2-}, PO_4^{3-}, IO_3^-, IO_4^-
M_8	MnO_4^-, F^-, MoO_4^{2-}	VO_3^-, CrO_4^{2-}, $Fe(CN)_6^{4-}$, SeO_3^{2-}, WO_4^{2-}, PO_4^{3-}, IO_3^-
M_9	MnO_4^-, IO_3^-	VO_3^-, AsO_4^{3-}, F^-, CrO_4^{2-}, $Fe(CN)_6^{4-}$, MoO_4^{2-}, WO_4^{2-}, SeO_3^{2-}, PO_4^{3-}, IO_4^-
M_{10}	MnO_4^-, IO_3^-	VO_3^-
M_{13}	VO_3^-, MnO_4^-	F^-, WO_4^{2-}

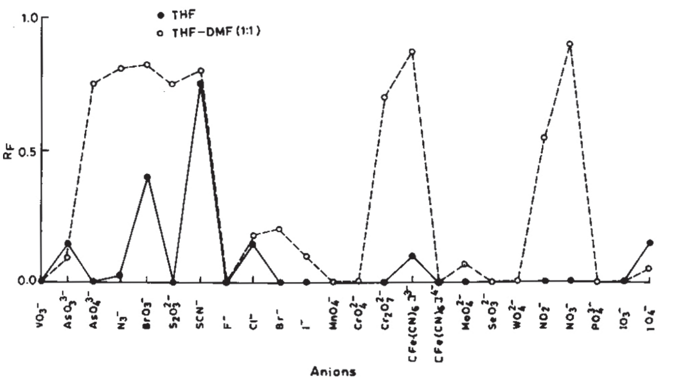

Figure 3

Plot of R_F vs. anions in THF and THF–DMF (1:1).

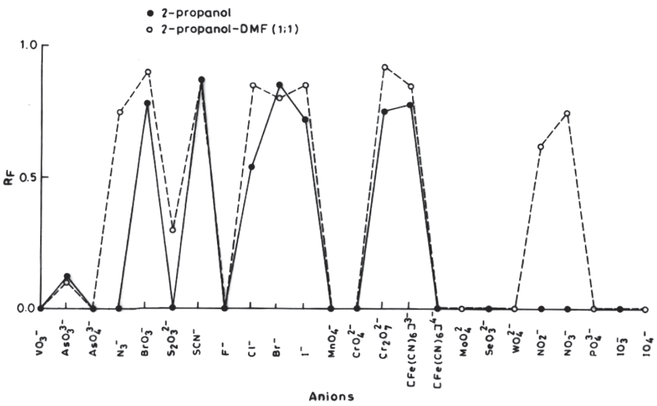

Figure 5

Plot of R_F vs. anions in 2-propanol and 2-proponol–DMF (1:1).

(4) In 2-propanol and 2-propanol–DMF (1:1) media, the majority of the anions exhibit a similar behavior. The only exceptions are N_3^- and NO_3^-, which give unusually much higher R_F in 2-propanol–DMF (1:1) media. This may be due to the lesser adsorption of these ions on the impregnated silica gel-G layers in this media.

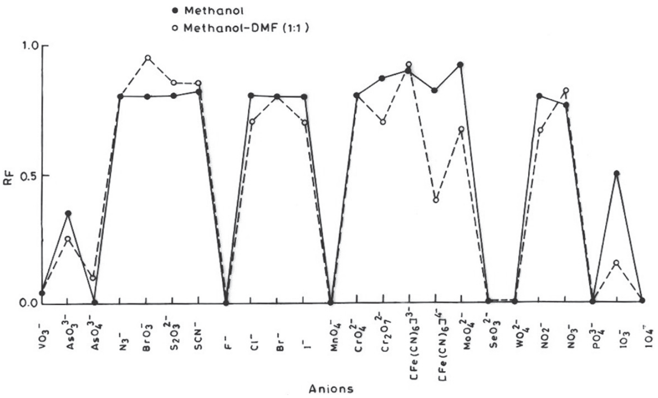

Figure 4

Plot of R_F vs. anions in methanol and methanol–DMF (1:1).

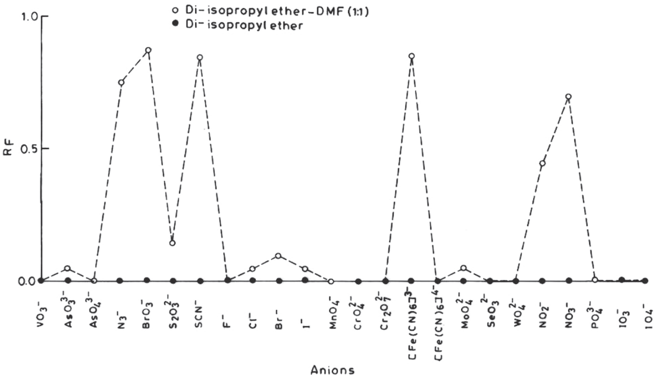

Figure 6

Plot of R_F vs. anions in diisopropyl ether and diisopropyl ether–DMF (1:1).

From the above discussion, it is evident that the R_F values of some anions increase when DMF is mixed with solvents of low DEC (<7.58). For solvents of higher DEC, the addition of DMF does not result in significant increase in the R_F for most of the anions.

On the basis of the retention behavior of anions, a number of analytically important binary and ternary separations are possible.

Table 3

Separations achieved on 20% TBA-impregnated silica gel-G layers.

Mobile phase	Separation achieved
	Anion (R_F)
M_1	AsO_3^{3-} (0.30) – AsO_4^{3-} (0.00), WO_4^{2-} (0.00) – MoO_4^{2-} (0.90) or CrO_4^{2-} or Cr_2O_7 (0.80) or CrO_4^{2-} (0.78)
M_2	$Fe(CN)_6^{3-}$ (0.95) – $Fe(CN)_6^{4-}$ (0.00)
M_4	IO_3^- (0.00) – IO_4^- (0.18), SCN^- (0.70) – AsO_3^{3-} (0.12) or AsO_4^3 or CrO_4^{2-} (0.00) or $Cr_2O_7^{2-}$ or $Fe(CN)_6^{3-}$ or $Fe(CN)_6^{4-}$ (0.00) or MoO_4^{2-} (0.00) or WO_4^{2-} (0.00) or PO_4^{3-} (0.00)
M_5	CrO_4 (0.00) – $Cr_2O_7^{2-}$ (0.72) F^- (0.00) – Cl^- (0.52) or Br^- (0.81) or I^- (0.69)
M_8	NO_2^- (0.50) – NO_3^- (0.91)
M_{10}	PO_4^{3-} (0.38) – NO_3^- (0.90) or NO_2^- (0.91), WO_4^{2-} (0.27) – MoO_4^{2-} (0.95) F^- (0.12) – Cl^- (0.92) or Br^- (0.94) or I^- (0.72), $Cr_2O_7^{2-}$ (0.18) – CrO_4^{2-} (0.90)
M_{12}	AsO_3^{3-} (0.40) – AsO_4^{3-} (0.96), CrO_4^{2-} (0.81) or AsO_4^{3-} (0.92) – AsO_3^{3-} (0.38)
M_{13}	WO_4^{2-} (0.00) – PO_4^{3-} (0.44) – SeO_3^{2-} (0.88), $S_2O_3^{2-}$ (0.32) – BrO_3^- (0.92)

Table 4

Comparison of the efficiency of separation of ternary mixture of iodide, iodate, and periodate and binary mixtures of hexacynoferrate (II) and hexacynoferrate (III) developed with different mobile phases on alumina, silica gel, and TBP-impregnated silica gel-G layers.

Stationary phase	Sample	Mobile phase	Separations (R_F)	Reference
20% TBA–impregnated silica gel-G layers	$I^- – IO_3^- – IO_4^-$	M_1	I^- (0.88) – IO_3^- (0.48) – IO_4^- (0.00)	This study
Alumina layers	– do –	100 nM aqueous sodium dodecyl sulphate (SDS)	I^- (0.92) – IO_3^- (0.25) – IO_4^- (0.00)	Ref. 36
– do –	– do –	100 nM Triton X-100	I^- (0.82) – IO_3^- (0.10) – IO_4^- (0.00)	Ref. 36
– do –	– do –	100 nM CTAB a)	I^- (0.95) – IO_3^- (0.22) – IO_4^- (0.00)	Ref. 36
20% TBA impregnated silica gel-G layers	$Fe(CN)_6^{3-}$ – $Fe (CN)_6^{4-}$	M_2	$Fe(CN)_6^{3-}$ (0.98) – $Fe (CN)_6^{4-}$ (0.00)	This study
Silica gel layers	– do –	Acetonitrile–water (9:1)	$Fe(CN)_6^{3-}$ (0.75) – $Fe(CN)_6^{4-}$ (0.05)	Ref. 34
– do –	– do –	Acetone–water (8:2)	$Fe(CN)_6^{3-}$ (0.90) – $Fe (CN)_6^{4-}$ (0.05)	Ref. 34

a) CTAB: *N*-Cetyl–*N*,*N*,*N*-trimethylammonium bromide
– do –: details as given above

Some of them have actually been realized and are given in **Table 3.** The separation of co-existing I^-, IO_3^- and IO_4^- is somewhat difficult as these ions are interconvertible. In an alkaline medium, iodine may react with OH^- ions to give I^- and IO_3^- ions. On the other hand, in dilute aqueous acid solutions (0.1 to 2.0 M HCl), IO_3^- can be reduced to iodine. However, we have been able to achieve the ternary separation of these ions. Further, it is observed that the separation of coexisting I^-, IO_3^-, and IO_4^- **(Table 4)** is more efficient than the one observed on plain alumina layers [40] (Table 4). The R_F values are indicative of better separations on 20% TBA-impregnated silica gel-G layers. The same is true for the separation of $Fe(CN)_6^{4-}$ and $Fe(CN)_6^{3-}$ on silica gel [34] and 20% TBA-impregnated silica gel-G layers (Table 4). The R_F values of these anions decrease in the order $Fe(CN)_6^{3-} > Fe(CN)_6^{4-}$, analogous with the results obtained on silica gel layers with acetone–water [34] and microcrystalline cellulose layers with acetone–ethyl acetate–water [40] mobile phases.

Table 5

R_F values and lyotropic numbers (*N*) of some anions.

Anion	R_F a)	*N*
F^-	0.05	4.8
BrO_3^-	0.88	9.7
Cl^-	0.90	10.0
NO_2^-	0.90	10.1
Br^-	0.92	11.5
NO_3^-	0.95	11.6
I^-	0.95	12.5
SCN^-	0.95	13.0
SCN^-	0.95	13.3

a) Values obtained with 0.1 M NaCl as the mobile phase

It is interesting to correlate the R_F values of anions with their lyotropic numbers. For this, the R_F values of some anions (whose lyotropic numbers are known) in 0.1 M NaCl as the mobile phase are determined **(Table 5).** Evidently, these values are found in accordance with the order of lyotropic numbers [41] in colloid chemistry rather than with hydrated ionic radii [42]. It is well known that the lyotropic numbers are derived from the concentrations of the sodium salts of the different anions required to flocculate agar. The lyotropic numbers for other anions are not known. It is expected that for these anions, too, the values would also be in accordance with the order of their lyotropic numbers. The lyotropic number is a measure of the extent of dehydration of the gel which in turn depends upon the hydration of anion. On this basis, hydration of the anion should increase with decreasing the lyotropic number of the anion. Thus, anions with low lyotropic numbers can be referred to as hydrophilic anions, whereas those with high lyotropic number are hydrophobic anions. The order of R_F values should be comparable with the strength of hydration.

4 Conclusion

From the above discussion, it is clear that in aqueous mobile phases, the majority of anions give higher R_F values, and the solubility of sodium or potassium salts of the anions plays a significant role, whereas for non-aqueous mobile phases, the R_F value of some of these anions increases with an increase in the polarity of the mobile phase used. Increase in R_F values of some anions is noticed when DMF is mixed with solvents of low DEC

(<7.58). All the anions give zero R_F in diisopropyl ether media due to its low DEC (3.39). 20% TBA-impregnated silica gel-G layers are found very effective for binary and ternary separations of anions. A correlation between the retention and lyotropic number of anions is observed. The R_F values are found to be in accordance with lyotropic numbers.

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