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NOTE

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Abstract

The thin-layer chromatography of 48 metal ions has been performed in 29 DMSO–HCl solvent systems. The HCl concentration varies from 1 to 6 *M* and the ratios of DMSO–HCl by volume are 1 : 9, 3 : 7, 5 : 5, 7 : 3, and 9 : 1. A number of interesting separations have been achieved: e.g., Sn^{4+}–Sn^{2+}, Ti^{4+}–Fe^{3+}, Zr^{4+}–UO_2^{2+}, Zr^{4+}–Th^{4+}, Zr^{4+}–La^{3+}, Sn^{2+}–Sb^{3+}, and W^{6+}–Mo^{6+}–UO_2^{2+}.

INTRODUCTION

Silica gel-G has been used extensively for the separation of organic and inorganic substances (*1*). A number of nonaqueous systems have been used as eluants. A search of the literature reveals that no work has been reported on the thin-layer chromatography of cations of silica gel-G plates using DMSO systems. Our earlier studies have pointed to the excellent solvating properties of DMSO (*2–5*). It is therefore surprising that this solvent has been neglected as an eluant for silica gel-G plates. The present work was therefore undertaken and a number of interesting results have been obtained which are summarized in this note.

EXPERIMENTAL

Apparatus

A thin-layer chromatography apparatus (Toshniwal, India) for the preparation of silica gel-G layers on 20 × 3 cm glass plates was used. The plates were developed in glass jars (20 × 6 cm).

Reagents

Silica gel-G (B.D.H., England) and DMSO (B.D.H., England) were used. All other chemicals were of AnalaR grade.

Test Solutions and Detectors

Decimolar solutions of metal ions containing a little acid were used for qualitative studies. Conventional spot test reagents were used for detection purposes (*6*).

Preparation of Silica Gel-G Plates

The slurry used was prepared by mixing the silica gel-G with constant shaking for 5 min in conductivity water in the ratio of 1: 3. This slurry was used immediately to coat the clean glass plates with the help of an applicator to give a layer of 0.15 mm thickness for the studies. The plates were first dried at room temperature and then in an electrically controlled oven at 100 ± 5°C for 2 h. These were then stored in an oven at room temperature.

Procedure

The cation solutions were spotted on the plates with thin glass capillaries. After drying the spots, plates were developed in the chosen solvent system by the ascending technique. The solvent was allowed to ascend 11 cm from the starting line on the plate in all cases. R_T and R_L values were then measured as usual.

Solvent Systems

In all, 29 solvent systems were used. The compositions of the systems used are given in Table 1.

TABLE 1

Composition of the Solvents Studied

Solvent	Composition	Mole fraction of DMSO	Mole fraction of H_2O	Mole fraction of HCl
S_1	Pure DMSO	1.0	—	—
S_2	1 M HCl	—	.93	.05
S_3	2 M HCl	—	.88	.11
S_4	4 M HCl	—	.77	.23
S_5	6 M HCl	—	.66	.34
S_6	DMSO + 1 M HCl (1: 9)	.03	.92	.05
S_7	DMSO + 1 M HCl (3: 7)	.10	.85	.05
S_8	DMSO + 1 M HCl (5: 5)	.21	.75	.04
S_9	DMSO + 1 M HCl (7: 3)	.38	.59	.03
S_{10}	DMSO + 1 M HCl (9: 1)	.70	.28	.02
S_{11}	DMSO + 2 M HCl (1: 9)	.03	.87	.10
S_{12}	DMSO + 2 M HCl (3: 7)	.10	.80	.10
S_{13}	DMSO + 2 M HCl (5: 5)	.21	.70	.08
S_{14}	DMSO + 2 M HCl (7: 3)	.39	.54	.07
S_{15}	DMSO + 2 M HCl (9: 1)	.71	.26	.03
S_{16}	DMSO + 4 M HCl (1: 9)	.03	.74	.23
S_{17}	DMSO + 4 M HCl (3: 7)	.11	.68	.21
S_{18}	DMSO + 4 M HCl (5: 5)	.23	.59	.18
S_{19}	DMSO + 4 M HCl (7: 3)	.41	.45	.14
S_{20}	DMSO + 4 M HCl (9: 1)	.73	.21	.06
S_{21}	DMSO + 6 M HCl (1: 9)	.03	.59	.37
S_{22}	DMSO + 6 M HCl (3: 7)	.12	.54	.34
S_{23}	DMSO + 6 M HCl (5: 5)	.24	.46	.29
S_{24}	DMSO + 6 M HCl (7: 3)	.43	.35	.22
S_{25}	DMSO + 6 M HCl (9: 1)	.74	.16	.10
S_{26}	H_2O + 1 M HCl (1: 1)	—	.97	.03
S_{27}	H_2O + 2 M HCl (1: 1)	—	.95	.05
S_{28}	H_2O + 4 M HCl (1: 1)	—	.89	.11
S_{29}	H_2O + 6 M HCl (1: 1)	—	.83	.17

RESULTS

The plots given in the figures are only for those metal ions for which $R_L - R_T$ is less than 0.3. A number of binary and ternary separations of analytical importance have been achieved experimentally as summarized in Table 2.

DISCUSSION

The more important features of this study are as follows:

TABLE 2

Separations Achieved Experimentally

Solvent system	Separations achieved, metal ion ($R_T - R_L$)	Time
DMSO + 1 M HCl (1: 9)	W^{6+}(0.00–0.00)–Mo^{6+}(0.38–0.63)	1 h, 15 min
	W^{6+}(0.00–0.00)–Mo^{6+}(0.36–0.66)–UO_2^{2+}(0.91–1.00)	
	Ag^{+}(0.00–0.00)–Sb^{3+}(0.14–0.49)–Cd^{2+}(0.90–1.00)	
	Ag^{+}(0.00–0.00)–Sb^{3+}(0.25–0.70)–UO_2^{2+}(0.90–1.00)	
	Ag^{+}(0.00–0.00)–Sb^{3+}(0.26–0.62)–Hg^{2+}(0.90–1.00)	
	Ag^{+}(0.00–0.00)–Sb^{3+}(0.25–0.67)–Tl^{+}(0.92–1.00)	
	Ag^{+}(0.00–0.00)–Sb^{3+}(0.26–0.64)–Cu^{2+}(0.95–1.00)	
	Sn^{2+}(0.90–1.00)–Sb^{3+}(0.45–0.74)	
	W^{6+}(0.00–0.00)–Fe^{3+}(0.85–1.00)	
	W^{6+}(0.00–0.00)–Cr^{3+}(0.90–1.00)	
	W^{6+}(0.00–0.00)–VO^{2+}(0.90–1.00)	
DMSO + 1 M HCl (3: 7)	Zr^{4+}(0.00–0.22)–La^{3+}(0.51–0.96)	2 h, 15 min
	Zr^{4+}(0.00–0.23)–Th^{4+}(0.90–1.00)	
	Zr^{4+}(0.00–0.25)–UO_2^{2+}(0.78–1.00)	
	Sb^{3+}(0.00–0.20)–Bi^{3+}(0.62–0.90)	
	Sb^{3+}(0.00–0.23)–Pd^{2+}(0.87–1.00)	
DMSO + 1 M HCl (5: 5)	Pb^{2+}(0.00–0.30)–Bi^{3+}(0.85–0.98)	3 h, 30 min
	Pb^{2+}(0.00–0.25)–Hg^{2+}(0.83–0.94)	
	Pb^{2+}(0.00–0.30)–UO_2^{2+}(0.85–0.97)	
DMSO + 2 M HCl (1: 9)	Ti^{4+}(0.00–0.37)–Fe^{3+}(0.90–1.00)	1 h, 30 min
DMSO + 4 M HCl	Sn^{4+}(0.00–0.08)–Sn^{2+}(0.90–1.00)	45 min
Pure DMSO	Tl^{+}(0.00–0.22)–Pd^{2+}(0.85–1.00)	3 h, 30 min
1 M HCl	Hg_2^{2+}(0.00–0.05)–Hg^{2+}(0.93–1.00)	40 min
	Cd^{2+}(0.91–1.00)–Hg_2^{2+}(0.00–0.05)	
	Hg_2^{2+}(0.00–0.05)–Zn^{2+}(0.52–0.98)	

(1) Only three ions (Ag^{+}, Sb^{3+}, and W^{6+}) have R_F values lower than 0.5 in solvent S_1 and therefore they can be separated from numerous metal ions. This system also shows many other interesting possibilities of separations such as those of Mo^{6+}–W^{6+}, Mo^{6+}–Pd^{2+}, and, in fact, the separation of Mo^{6+} from almost all other ions (Fig. 1).

(2) As the mole fraction of DMSO is increased, the number of cations with R_F values less than 0.5 increases until, in solvent S_{10}, Ti^{4+}, Se^{4+}, Y^{3+}, Te^{4+}, and Pt^{4+} have R_F values less than 0.1 (Fig. 2). This shows the possibility of separating numerous metal ions from

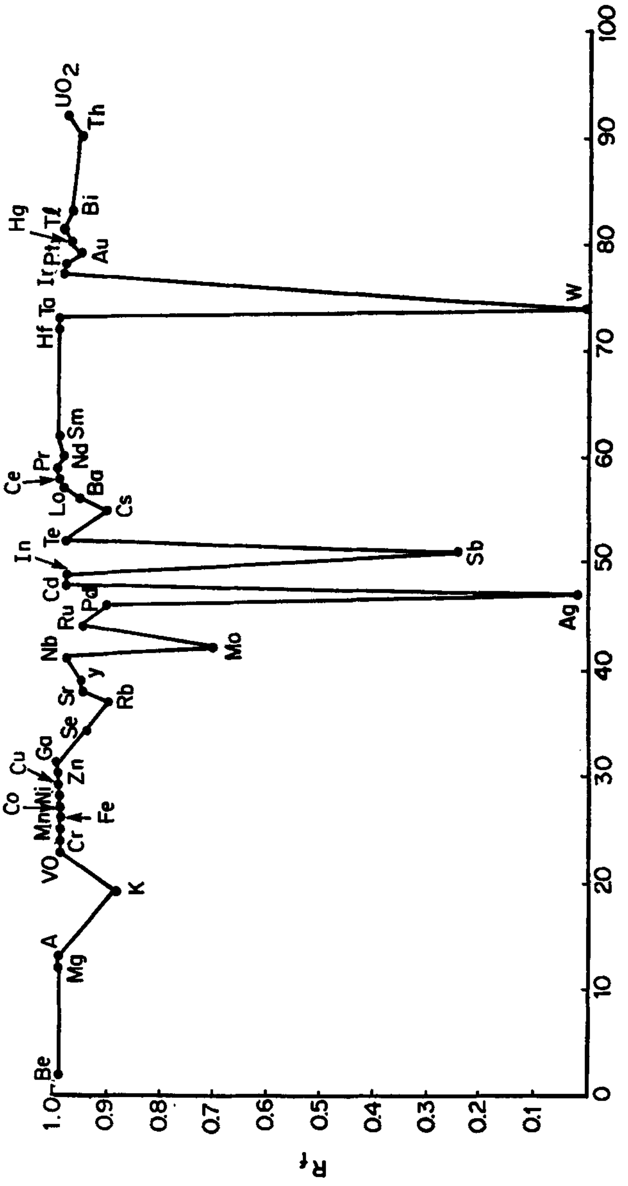

FIG. 1. Plot of R_F vs atomic number in DMSO + 1 M HCl (1:9).

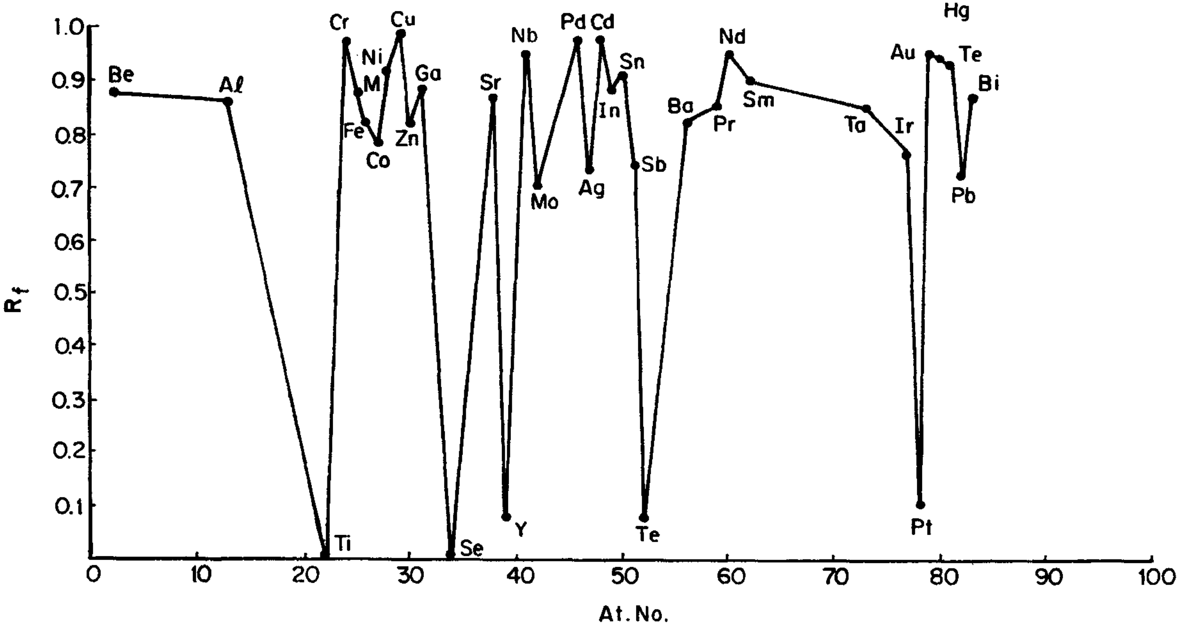

FIG. 2. Plot of R_F vs atomic number in DMSO + 1 M HCl (9:1).

one ion: e.g.,

Ti^{4+} from Be^{2+}, Al^{3+}, Cr^{3+}, Mn^{2+}, Fe^{3+}, Cu^{2+}, Co^{2+}, Zn^{2+}, Nb^{4+}, Mo^{4+}, Pd^{2+}, Cd^{2+}, Ta^{4+}
Y^{3+} from Sr^{2+}, Ba^{2+}, Be^{2+}
Pt^{4+} from Ir^{3+}, Au^{3+}, Cu^{2+}

(3) The R_F values of some of the metal ions in DMSO–1 *M* HCl systems in the ratios of 1: 9, 3: 7, 5: 5, 7: 3, 9: 1, and pure DMSO have been plotted in Figs. 3A and 3B. The metal ions can be divided into the following groups in accordance with their chromatographic behavior:

(A) Ag^+, Sn^{4+}, Sb^{3+}

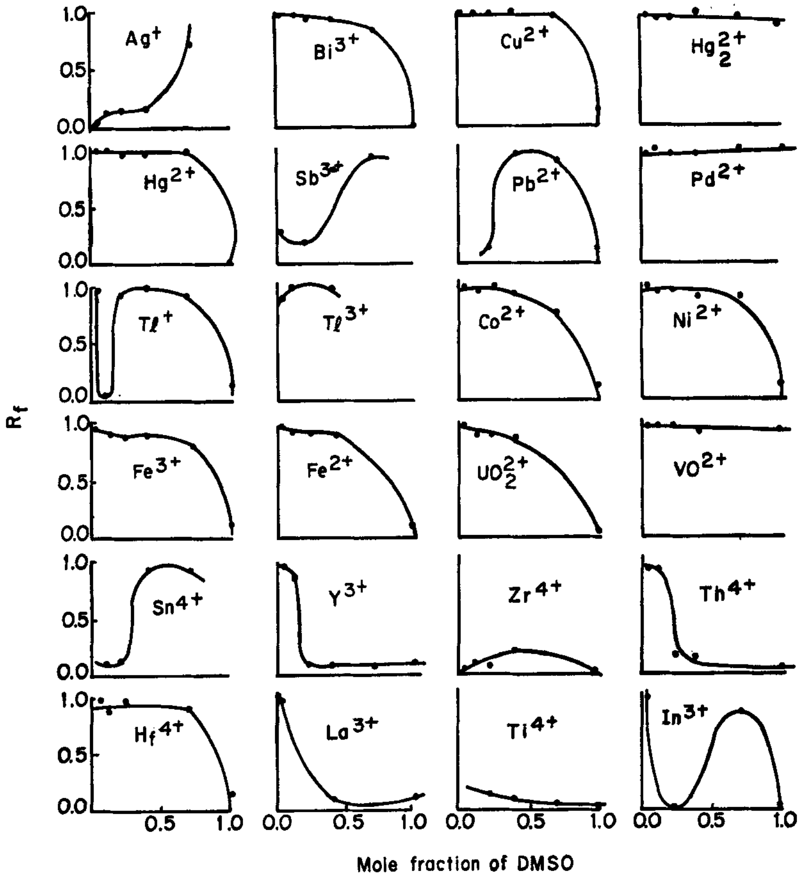

FIG. 3A. Plots of R_F vs mole fraction of DMSO.

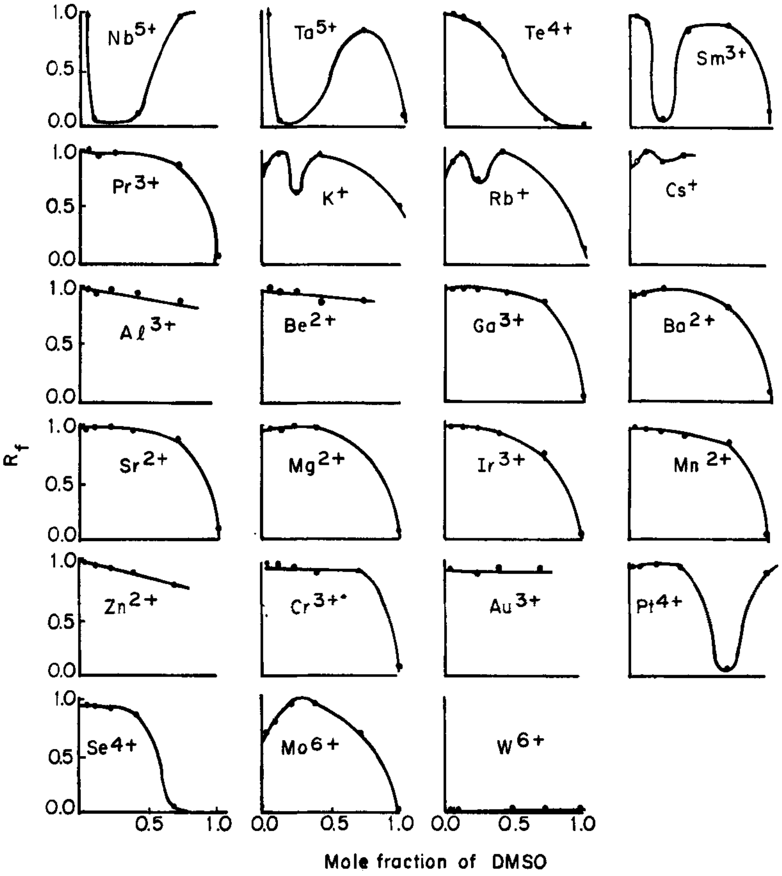

FIG. 3B. Plots of R_F vs mole fraction of DMSO.

(B) Bi^{3+}, Cu^{2+}, Hg^{2+}, Co^{2+}, Ni^{2+}, Ga^{3+}, Ba^{2+}, Sr^{2+}, Mg^{2+}, Ir^{3+}, Mn^{2+}, Cr^{3+}, Fe^{2+}, Pr^{3+}, $UO_2{}^{2+}$, Fe^{3+}, Hf^{4+}

(C) Y^{3+}, La^{3+}, Th^{4+}

(D) Zr^{4+}, Ti^{4+}, W^{4+}

(E) $Hg_2{}^{2+}$, Pd^{2+}, VO^{2+}, Au^{3+}, Be^{2+}, Al^{3+}, Zn^{2+}

In Group (A) the R_F value increases with an increase in the DMSO mole fraction because DMSO solvates the cation. In Group (B) there is very little change in R_F value with an increase in the DMSO mole fraction up to a certain point and then the R_F value suddenly changes to zero in pure DMSO. This may be due to the fact that the role of HCl predominates and, in the absence of HCl, the R_F value is very low. In the presence of

HCl, the DMSO mole fraction does not affect the R_F value. In Group (C) the R_F values are very high in almost pure HCl (mole fraction 0.97). However, as soon as the DMSO mole fraction becomes significant, i.e., 0.3, the R_F value falls sharply because these ions are not solvated by DMSO. The ions in Group (D) have almost a zero R_F owing to their high charge and low solvation by DMSO. In Group (E) the R_F values remain almost constant irrespective of the increasing DMSO mole fraction. The high R_F values may be due to the solvation of cations even at a DMSO mole fraction of the order of 0.03.

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