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Thin-Layer Chromatography of Metal Ions in Formic Acid Medium on Impregnated and Unimpregnated Silica Gel G: Semiquantitative Determination of Fe^{3}, Cd^{2}, Th^{4}, Al^{3}, Zn^{2}, $UO^{2}{}_{2}$, VO^{2}, Ce^{4}, and Ni^{2}

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Thin-Layer Chromatography of Metal Ions in Formic Acid Medium on Impregnated and Unimpregnated Silica Gel G: Semiquantitative Determination of Fe^{3+}, Cd^{2+}, Th^{4+}, Al^{3+}, Zn^{2+}, UO_2^{2+}, VO^{2+}, Ce^{4+}, and Ni^{2+}

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Abstract

The adsorption behavior of metal ions in the formic acid–sodium formate system using unimpregnated as well as impregnated silica gel G as thin layers has been studied. A remarkable result of this study is the dramatic selectivity of impregnated silica gel G thin layers when compared to the corresponding unimpregnated silica gel G thin layers. This impregnation effect provides us with a new adsorbent phase which is sufficiently stable in the formic acid–sodium formate medium. Some important ternary separations have been achieved on silica gel G thin layers. Al^{3+}, Ni^{2+}, Fe^{3+}, and Pb^{2+} have been qualitatively separated from mixtures of other ions. Cu^{2+} has been selectively separated on impregnated silica gel G thin layers. The semiquantitative determination of nine metal ions on impregnated silica gel G layers has also been attempted.

INTRODUCTION

Thin-layer chromatography (TLC) is more efficient than column and paper chromatography for the separation of metal ions because it gives faster and more accurate results. TLC has been widely used in organic analysis but little attention has been paid for its use in inorganic chemistry.

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Brinkman (*1*) has nicely documented the work done on unimpregnated silica gel G (SG) as well as impregnated SG with organic solvents. A few studies have also been reported (*2–13*) on the use of inorganic ion-exchange materials. As far as we are aware, not a single reference is available on the use of SG impregnated with inorganic materials as thin layers for the separation of metal ions. During our previous studies (*14–17*) on inorganic materials as thin layers, we came across four main difficulties: (a) a large quantity of ion-exchange material was needed to prepare thin layers; (b) the synthesis of the ion exchanger and the preparation of slurry for thin layers was a very time consuming and tedious job; (c) the time needed for the development of chromatographic plate is longer; and (d) there is a lack of reproducibility and unstability of ion-exchange materials in solvent systems. Our recent findings on inorganic ion exchangers (*18*) revealed that the antimonates of Pb^{2+} and Hg_2^{2+} are more stable in formic acid than in demineralized water (DMW). This shows that formic acid has high analytical potential.

Surprisingly, very little work appears to have been done on the adsorption of cations in formic acid medium (*19–25*). Most of the work done on TLC till now is qualitative, and a detailed study on the quantitative or semiquantitative determination of metal ions by TLC is lacking.

It was therefore decided to develop a technique for the preparation of highly stable thin layers of SG mixed with sodium molybdate and to explore its analytical potential for the selective detection, separation, and semiquantitative determination of some metal ions in formic acid medium.

The proposed thin-layer chromatographic method is very fast and more selective than those reported earlier (*2–17*). The time required for the development of chromatographic plates is surprisingly short, i.e., 12 to 15 min. At the same time, this method is very inexpensive because it is based on the use of a larger quantity of commercially available SG impregnated with a small quantity of sodium molybdate.

EXPERIMENTAL

Apparatus

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

Reagents

SG (E. Merck) and sodium molybdate bihydrate (Veb Jena Phar, Germany) were used. All other reagents were of Analar grade.

Test Solutions and Detectors

The test solutions were generally 0.1 *M* in the metal chloride or nitrate. Conventional spot test reagents were used for detection purposes (*26*).

Preparation of Thin-Layer Plates

The SG plates were prepared by mixing SG with conductivity water in a ratio of 1:3 with constant shaking for 5 min. The resultant slurry was used immediately to coat the clean glass plates with the help of an applicator to give a layer 0.25 mm thick. The plates were first dried at room temperature and then kept in an electrically controlled oven at 100 ± 5°C for 2 h. The plates were stored in an oven at room temperature.

To prepare impregnated SG thin layers, a slurry was made by mixing an aqueous solution of 0.3 *M* sodium molybdate with SG in a ratio of 3:1. Thin layers were prepared by using this slurry under the same experimental conditions as cited above for pure SG plates.

Solvent Systems

The following solvent systems were used in these studies:

Demineralized water (DMW)
1.0 *M* HCOONa
1.0 *M* HCOONa + 1.0 *M* HCOOH (9:1)
1.0 *M* HCOONa + 1.0 *M* HCOOH (7:3)
1.0 *M* HCOONa + 1.0 *M* HCOOH (6:4)
1.0 *M* HCOONa + 1.0 *M* HCOOH (1:1)
1.0 *M* HCOONa + 1.0 *M* HCOOH (4:6)
1.0 *M* HCOONa + 1.0 *M* HCOOH (3:7)
1.0 *M* HCOONa + 1.0 *M* HCOOH (1:9)
1.0 *M* HCOOH

Procedure

Qualitative chromatography was performed as in the ascending technique with a solvent ascent of 10 cm in all cases.

For semiquantitative determination, various known amounts of 0.1 *M* metal salt solution were spotted with the help of a lambda pipette at the line of application, and the development was performed in the chosen solvent systems. After detecting the spot, the area of the spots was traced on tracing paper and the spot area was measured planimetrically.

RESULTS AND DISCUSSION

The results obtained have been summarized in Figs. 1–9. Table 1 shows the selective detection and separation of Cu^{2+} from numerous metal ions on impregnated SG. Such a separation is not possible when we use pure SG. Table 2 summarizes some separations achieved on pure SG thin layers.

The main interesting features of this study are:

(1) Selection of a new solvent system in which most of the inorganic salts are highly stable

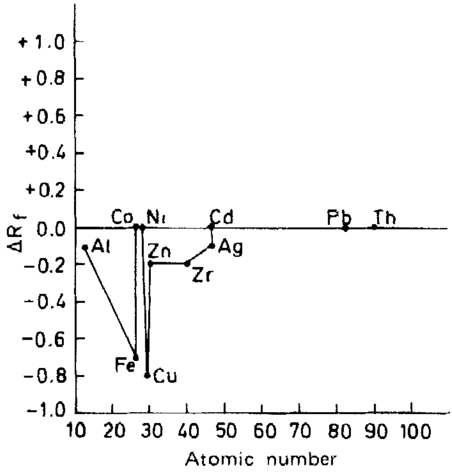

FIG. 1. Plot of ΔR_f vs atomic number in demineralized water.

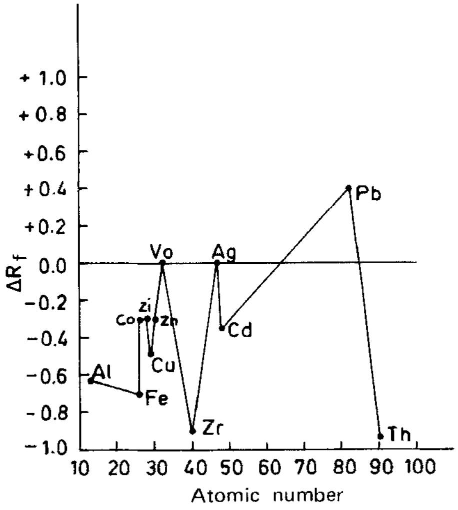

FIG. 2. Plot of ΔR_f vs atomic number in 1 *M* HCOOH. pH = 1.3.

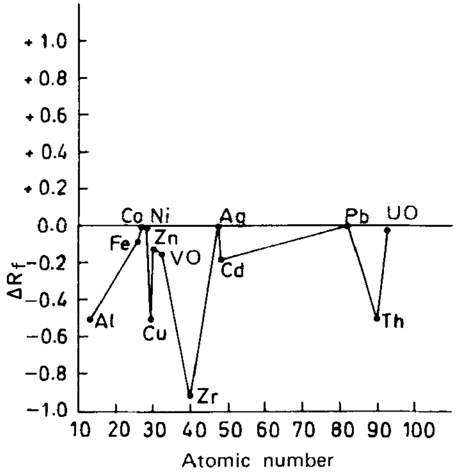

FIG. 3. Plot of ΔR_f vs atomic number in 1 *M* HCOOH + 1 *M* HCOONa (1:1). pH = 3.2.

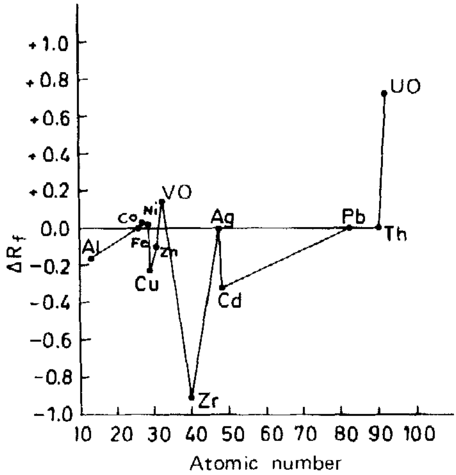

FIG. 4. Plot of ΔR_f vs atomic number in 1 *M* HCOONa. pH = 6.2.

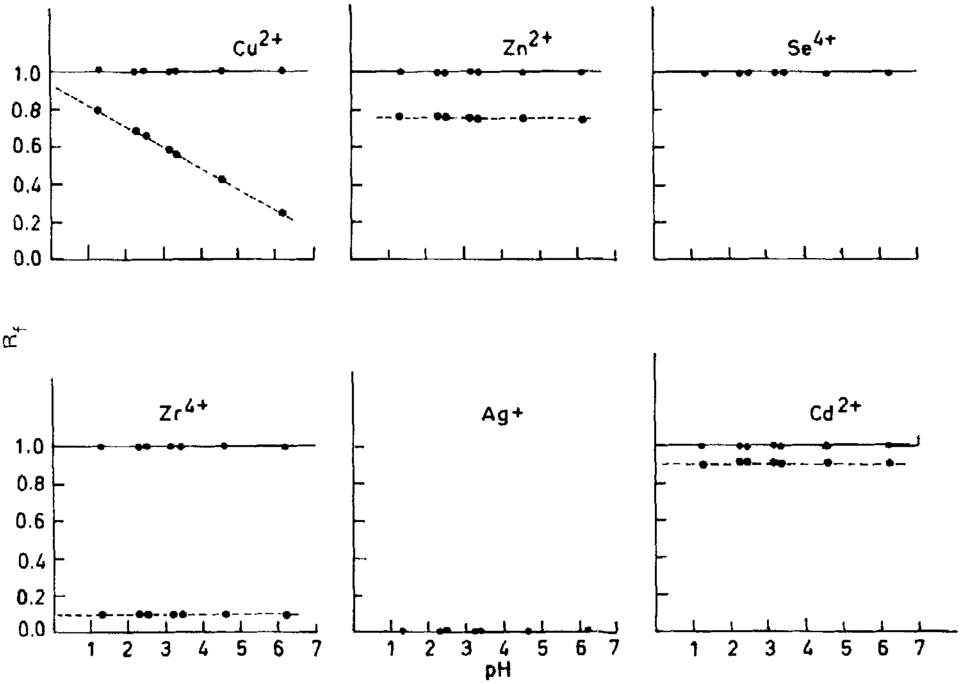

FIG. 5. Plots of R_f vs pH. (——) Unimpregnated silica gel thin layers. (- -) Impregnated silica gel thin layers.

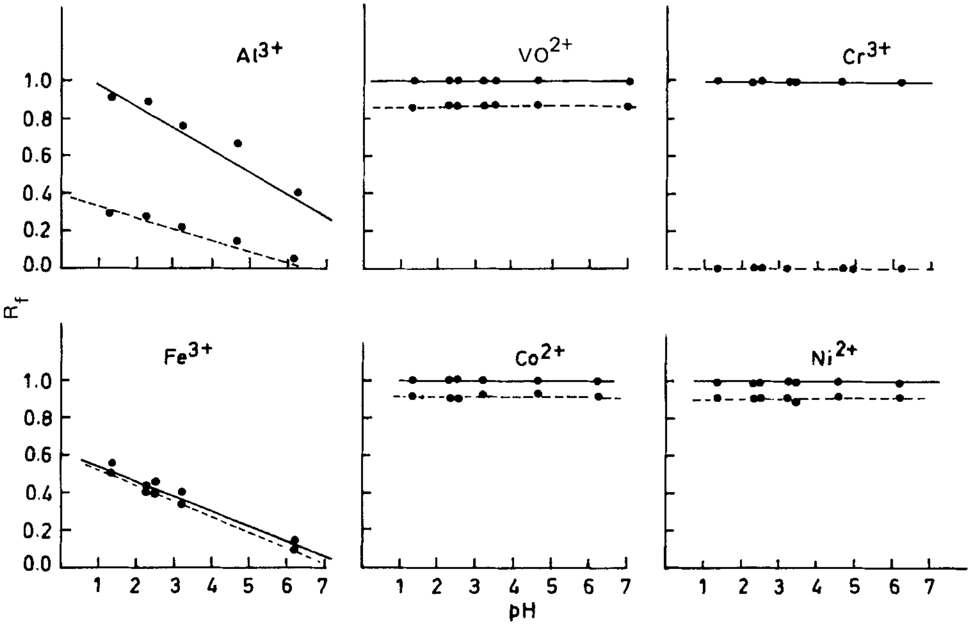

FIG. 6. Plots of R_f vs pH. (——) Unimpregnated silica gel thin layers. (- -) Impregnated silica gel thin layers.

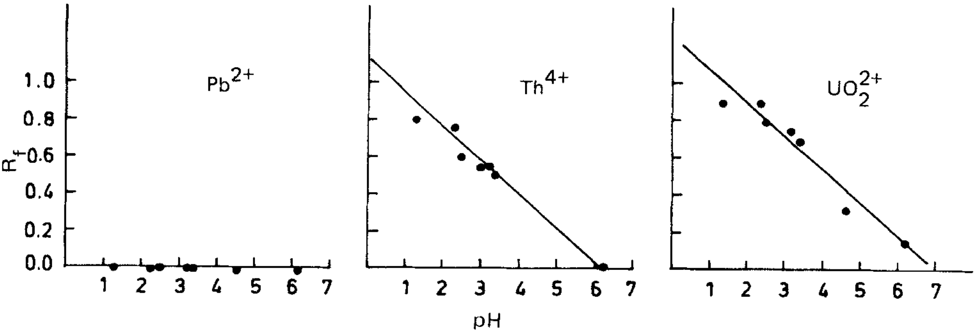

FIG. 7. Plots of R_f vs pH. (——) Unimpregnated silica gel thin layers. (- -) Impregnated silica gel thin layers.

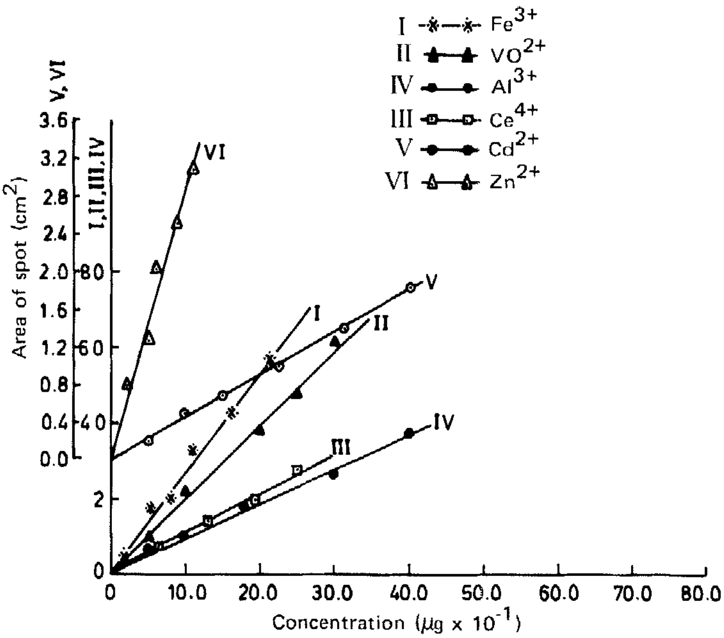

FIG. 8. Plots of spot area vs concentration of metal ions.

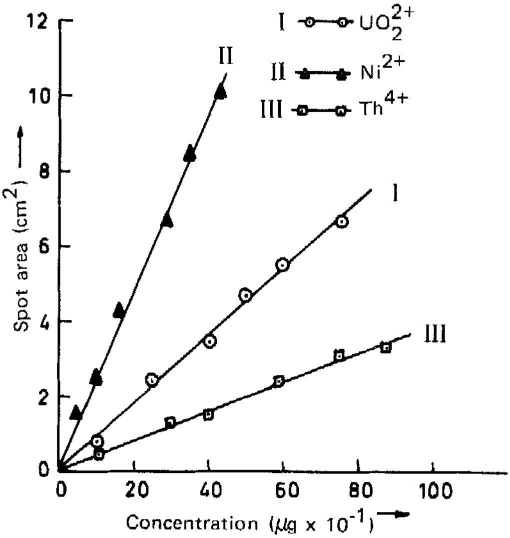

FIG. 9. Plots of spot area vs concentration of metal ions.

TABLE 1
Separation of Cu^{2+} from Zn^{2+}, Cd^{2+}, Pb^{2+}, Co^{2+}, Ni^{2+}, Cr^{3+}, Al^{3+}, Fe^{3+}, and Zr^{4+} on Impregnated Silica Gel G Thin Layers

Solvent system	Separations achieved
1 *M* HCOO Na	Cu^{2+}(0.30)-Zn^{2+}(0.68) Cu^{2+}(0.30)-Cd^{2+}(0.85) Cu^{2+}(0.30)-Pb^{2+}(0.00) Cu^{2+}(0.30)-Co^{2+}(0.84) Cu^{2+}(0.30)-Ni^{2+}(0.84) Cu^{2+}(0.30)-Cr^{3+}(0.00) Cu^{2+}(0.30)-Al^{3+}(0.02) Cu^{2+}(0.30)-Fe^{3+}(0.08) Cu^{2+}(0.30)-Zr^{4+}(1.00)

(2) Utilization of silica gel G mixed with 0.3 *M* aqueous sodium molybdate solution (termed impregnated silica gel in this paper) as a highly selective adsorption medium for TLC
(3) Selective separation of Cu^{2+} from numerous metal ions on impregnated silica gel
(4) Semiquantitative determination of metal ions on impregnated thin layers by measuring the spot area

Formic acid has been chosen as the solvent system for TLC because of the following properties:

(a) It does not deform thin layers
(b) It provides compact planetary spots on thin-layer plates for cations
(c) It does not permit the oxidation of cations during analysis
(d) It is sufficiently acidic [$Ka(H_2O)$ at 25°C = 1.77×10^{-4}] to prevent hydrolysis of salts, and at the same time it is less acidic than necessary to dissolve the ion-exchange materials significantly

To choose the most stable and selective adsorption phase, the R_f values of metal ions were determined under different experimental conditions by using:

(i) Pure SG thin layers and DMW as developer
(ii) Pure SG thin layers and aqueous solutions of sodium molybdates, stannic chloride, sodium arsenate, phosphoric acid, lead nitrate, and mercurous nitrate as developers

TABLE 2
Separations Achieved in Formic Acid-Sodium Formate Systems on Pure Silica Gel G Plates

Solvent systems	Separations achieved
1 *M* HCOOH + 1 *M* HCOONa (4:6)	Pb^{2+}(0.00)-Cd^{2+}(0.85)-UO_2^{2+}(0.7)
	Pb^{2+}(0.00)-Cu^{2+}(0.84)-UO_2^{2+}(0.7)
	Pb^{2+}(0.00)-Zn^{2+}(0.98)-UO_2^{2+}(0.7)
	Pb^{2+}(0.00)-Ni^{2+}(1.00)-UO_2^{2+}(0.7)
	Pb^{2+}(0.00)-Co^{2+}(1.00)-UO_2^{2+}(0.7)
	Pb^{2+}(0.00)-Se^{4+}(1.00)-UO_2^{2+}(0.7)
	Pb^{2+}(0.00)-VO^{2+}(1.00)-UO_2^{2+}(0.7)
1 *M* HCOOH + 1 *M* HCOONa (6:4)	Pb^{2+}(0.00)-Cd^{2+}(0.85)-Fe^{3+}(0.55)
	Pb^{2+}(0.00)-Cu^{2+}(0.84)-Fe^{3+}(0.55)
	Pb^{2+}(0.00)-Zn^{2+}(0.98)-Fe^{3+}(0.55)
	Pb^{2+}(0.00)-Ni^{2+}(1.00)-Fe^{3+}(0.55)
	Pb^{2+}(0.00)-Co^{2+}(1.00)-Fe^{3+}(0.55)
	Pb^{2+}(0.00)-Se^{4+}(1.00)-Fe^{3+}(0.55)
	Pb^{2+}(0.00)-VO^{2+}(1.00)-Fe^{3+}(0.55)
1 *M* HCOOH + 1 *M* HCOONa (4:6)	Ag^{+}(0.00)-Cd^{2+}(0.85)-UO^{2+}(0.7)
	Ag^{+}(0.00)-Cu^{2+}(0.84)-UO^{2+}(0.7)
	Ag^{+}(0.00)-Zn^{2+}(0.98)-UO^{2+}(0.7)
	Ag^{+}(0.00)-Ni^{2+}(1.00)-UO^{2+}(0.7)
	Ag^{+}(0.00)-Co^{2+}(1.00)-UO^{2+}(0.7)
	Ag^{+}(0.00)-Se^{4+}(1.00)-UO^{2+}(0.7)
	Ag^{+}(0.00)-VO^{2+}(1.00)-UO^{2+}(0.7)
1 *M* HCOOH + 1 *M* HCOONa (6:4)	Ag^{+}(0.00)-Cd^{2+}(0.85)-Fe^{3+}(0.55)
	Ag^{+}(0.00)-Cu^{2+}(0.84)-Fe^{3+}(0.55)
	Ag^{+}(0.00)-Zn^{2+}(0.98)-Fe^{3+}(0.55)
	Ag^{+}(0.00)-Ni^{2+}(1.00)-Fe^{3+}(0.55)
	Ag^{+}(0.00)-Co^{2+}(1.00)-Fe^{3+}(0.55)
	Ag^{+}(0.00)-Se^{4+}(1.00)-Fe^{3+}(0.55)
	Ag^{+}(0.00)-VO^{2+}(1.00)-Fe^{3+}(0.55)
1 *M* HCOOH + 1 *M* HCOONa (1:1)	Ag^{+}(0.00)-Cd^{2+}(0.85)-Th^{4+}(0.65)
	Ag^{+}(0.00)-Cu^{2+}(0.84)-Th^{4+}(0.65)
	Ag^{+}(0.00)-Zn^{2+}(0.98)-Th^{4+}(0.65)
	Ag^{+}(0.00)-Ni^{2+}(1.00)-Th^{4+}(0.65)
	Ag^{+}(0.00)-Co^{2+}(1.00)-Th^{4+}(0.65)
	Ag^{+}(0.00)-Se^{4+}(1.00)-Th^{4+}(0.65)
	Ag^{+}(0.00)-VO^{2+}(1.00)-Th^{4+}(0.65)
	Pb^{2+}(0.00)-Cd^{2+}(0.85)-Th^{4+}(0.65)
	Pb^{2+}(0.00)-Cu^{2+}(0.84)-Th^{4+}(0.65)
	Pb^{2+}(0.00)-Zn^{2+}(0.98)-Th^{4+}(0.65)
	Pb^{2+}(0.00)-Ni^{2+}(1.00)-Th^{4+}(0.65)
	Pb^{2+}(0.00)-Co^{2+}(1.00)-Th^{4+}(0.65)
	Pb^{2+}(0.00)-Se^{4+}(1.00)-Th^{4+}(0.65)
	Pb^{2+}(0.00)-VO^{2+}(1.00)-Th^{4+}(0.65)
	Pb^{2+}(0.00)-Cd^{2+}(0.85)-Al^{3+}(0.45)
	Pb^{2+}(0.00)-Cu^{2+}(0.84)-Al^{3+}(0.45)

(continued)

TABLE 2 *(continued)*

Solvent systems	Separations achieved
1 M HCOOH + 1 M HCOONa (1:1)	Pb^{2+}(0.00)-Zn^{2+}(0.98)-Al^{3+}(0.45)
	Pb^{2+}(0.00)-Ni^{2+}(1.00)-Al^{3+}(0.45)
	Pb^{2+}(0.00)-Co^{2+}(1.00)-Al^{3+}(0.45)
	Pb^{2+}(0.00)-Se^{4+}(1.00)-Al^{3+}(0.45)
	Pb^{2+}(0.00)-Vo^{2+}(1.00)-Al^{3+}(0.45)
	Ag^{+}(0.00)-Cd^{2+}(0.85)-Al^{3+}(0.45)
	Ag^{+}(0.00)-Cu^{2+}(0.84)-Al^{3+}(0.45)
	Ag^{+}(0.00)-Zn^{2+}(0.98)-Al^{3+}(0.45)
	Ag^{+}(0.00)-Ni^{2+}(1.00)-Al^{3+}(0.45)
	Ag^{+}(0.00)-Co^{2+}(1.00)-Al^{3+}(0.45)
	Ag^{+}(0.00)-Se^{4+}(1.00)-Al^{3+}(0.45)
	Ag^{+}(0.00)-VO^{2+}(1.00)-Al^{3+}(0.45)
1 M HCOOH	Pb^{2+}(0.00)-A mix of VO^{2+}, Cr^{3+}, Fe^{3+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Zr^{4+}, Cd^{2+}, Th^{4+}, and UO_2^{2+}
1 M HCOONa	Al^{3+}(0.38)-A mix of VO^{2+}, Cr^{3+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Zr^{4+}, Cd^{2+}, Fe^{3+}, and Th^{4+}
	Fe^{3+}(0.15)-A mix of Vo^{2+}, Cr^{3+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Zr^{4+}, Cd^{2+}, and Th^{4+}
	Ni^{2+}(1.00)-A mix of Al^{3+}, Fe^{3+}, Th^{4+}, UO_2^{2+}, and Pb^{2+}

(iii) SG thin layers impregnated with aqueous salt solutions mentioned in (ii) and DMW as developer

(iv) SG mixed with aqueous solutions of the salts mentioned in (ii) as thin layers after drying at 100 ± 5°C with DMW as developer

The R_f values of metal ions remained unaltered in Cases (i), (ii), and (iii). In Case (iv), an alteration of R_f value was only obtained when sodium molybdate was taken as a mixer with SG, while for other salts as the mixer, the R_f values of metal ions remained the same as in Cases (i), (ii), and (iii). It appears that a new adsorption phase, most probably sodium molybdosilicate, is formed in Case (iv) when sodium molybdate is mixed with SG, and that this phase is responsible for the high selectivity and stability in formic acid medium. Further, the appropriate concentration of the sodium molybdate was found to be 0.3 M to obtain compact spots for the metal ions. Tailing starts at higher concentrations, while at lower concentrations the impregnation effect is insignificant.

Figures 1–4 show that the selectivity of SG is enhanced on impregnation with sodium molybdate. The results obtained on pure SG and impregnated

SG plates in DMW, 1.0 *M* HCOOH, 1.0 *M* HCOONa, and 1.0 *M* HCOOH + 1.0 *M* HCOONa (1:1) have been compared, and the differences in the R_f values ($\Delta R_f = R_f$ on impregnated plates $-$ R_f on unimpregnated plates) have been plotted (Fig. 1). In DMW most of the cations have negative ΔR_f values, showing the enhanced selectivity of impregnated SG. In the case of the 1.0 *M* HCOOH (pH = 1.3) solvent system, the ΔR_f value is more negative than in DMW for most of the cations except for Cu^{2+}, Ag^+, and Pb^{2+} which have more positive ΔR_f values than in DMW. For Fe^{3+} there is no change in ΔR_f value. Most of the cations, except VO^{2+}, Zr^{4+}, and Ag^+ have more positive ΔR_f values in the 1.0 *M* HCOOH + 1.0 *M* HCOONa (1:1, pH = 3.2) solvent system in comparison to the 1.0 *M* HCOOH (pH = 1.3) system while Pb^{2+} has a more negative ΔR_f value. Furthermore, in the case 1.0 *M* HCOONa (pH = 6.2), the ΔR_f values of most of the cations become more positive than in 1.0 *M* HCOOH and 1.0 *M* HCOOH + 1.0 *M* HCOONa (1:1) solvent systems. It shows that the selectivity of the adsorbent is reduced when the pH of the solvent system increases.

The effect of pH on R_f values of metal ions on unimpregnated and impregnated thin layers is summarized in Figs. 5–7. On the basis of the dependence of the mobility of metal ions on the pH of the solvent system, they can be grouped into three classes when unimpregnated thin layers are used.

(a) Metal ions for which the R_f value is equal to zero and whose mobility is independent of pH (Pb^{2+} and Ag^+)
(b) Metal ions having an R_f value equal to 1 at all pH values (VO^{2+}, Cr^{3+}, Ni^{2+}, Co^{2+}, Zn^{2+}, Cd^{2+}, Cu^{2+}, Se^{4+}, and Zr^{4+})
(c) Metal ions for which the R_f value decreases with an increase in pH (Al^{3+}, Fe^{3+}, UO_2^{2+}, and Th^{4+})

A similar trend for the dependence of R_f values on the pH of the solvent system was observed on impregnated thin layers for all metal ions as classified above except for Cu^{2+} (Figs. 5–7). It is interesting to note from Figs. 1–4 that there is a remarkable enhancement in the selectivity of metal ions on impregnated layers. The selectivity of the ion-exchange process depends upon (a) the ion-exchange material used and (b) the solvent medium of exchange. It is apparent that the low R_f values on impregnated layers are due to strong adsorption of metal ions on the silica gel molybdosilicate phase (impregnated silica gel), thus showing its high selectivity.

Among the cations grouped in Class (b), Cu^{2+} showed a unique behavior on impregnated layers at different pH values (Figs. 5–7). The R_f value for Cu^{2+} decreases with an increase in the pH of the solvent system. The R_f

value for Cu^{2+} is at a maximum at pH 1.3 and at a minimum at pH 6.2. It is difficult to explain why Cu^{2+} has a lower R_f value than Ni^{2+} and Co^{2+}. Cu^{2+}, Ni^{2+}, and Co^{2+} have almost the same electronegativity. Further, Co, Ni, and Cu all have nonspherical distribution of the *d*-electrons and hence are more shielded.

It appears that Cu^{2+} is precipitated at pH 6 as copper molybdosilicate. However, Ni^{2+} and Co^{2+} are precipitated at higher pH ($\approx$ 8), and hence they are still in the form of ions. This behavior is analogous to the precipitation of these ions as hydrous oxides (*27*).

This unique behavior of Cu^{2+} on impregnated layers leads to very important separations of Cu^{2+} from Zn^{2+}, Cd^{2+}, Pb^{2+}, Co^{2+}, Ni^{2+}, Cr^{3+}, Fe^{3+}, Al^{3+}, and Zr^{4+} at pH 6.2 (Table 1). Such separations are not possible if pure SG is used as the adsorption medium. Exceptions are Pb^{2+}, Al^{3+}, and Fe^{3+}.

There are three main methods for the quantitative determination of compounds on a chromatoplate:

(a) Elution and chemical estimation
(b) Measurement of optical density
(c) Measurement of spot area

The spot area method is the simplest, most rapid, and most inexpensive although it is less accurate. The relationship between the logarithm of the amount of the substance in the spot and the area of the spot is usually linear. However, other quantitative relationships have also been described, e.g., between the logarithm of the quantity and the logarithm of the length of the spot, between the length of the spot and the logarithm of the amount (*28*), and between the area of the spot and the amount (*29*). This is because the absolute amount of the chromatographed substance does not itself decide the definite shape and size of the spot, but also the volume of solution applied, the diameter of the spot at the start, and a series of other factors.

Therefore, it is necessary to assess the relationship experimentally by means of a calibration chromatogram before a quantitative determination is made. The quantitative determination of Zn^{2+}, Fe^{3+}, UO_2^{2+}, Th^{4+}, Ni^{2+}, VO^{2+}, Al^{3+}, Ce^{4+}, and Cd^{2+} on impregnated SG was obtained by plotting spot areas versus concentration of metal ions (Figs. 8 and 9). The planetary spots were drawn on tracing paper directly from thin-layer chromatograms and then the areas of the spots were calculated. A linear relation is obtained between the amount of the sample spotted and the area of the planetary spot. The linearity is maintained in a limited concentration range, e.g., 5–220 μg for Fe^{3+}, 5–300 μg for VO^{2+}, 5–400 μg for Al^{3+}, 5–250 μg for Ce^{4+}, 2–120 μg for Zn^{2+}, 5–400 μg for Cd^{2+}, 5–750 μg for UO_2^{2+}, 5–900 μg for Th^{4+}, and 5–500 μg for Ni^{2+}. Thus, there is a narrow concentration range for Zn^{2+}, and a wide concentration range for Th^{4+}. When the procedure is applied to higher concentrations of metal ions, a positive deviation from the linear law is observed. The accuracy and precision falls within a 4–5% limit for all the metal ions determined. The method is less accurate for quantitative determination but is quite accurate for semiquantitative determination.

Seher (*29*) has determined the quantity of α-locophenol in lipid mixtures from a graph of spot areas versus weight of material, but Purdy and Truter (*30*) found a linear relationship between the square root of the spot area and the logarithm of the weight for several compounds.

On the basis of the R_f value difference on pure SG thin layers, a number of separations (ternary and one from numerous metal ions) have been achieved (Table 2). Pb^{2+}, Al^{3+}, Fe^{3+}, and Ni^{2+} have been separated from mixtures with other metal ions. Al^{3+}, Fe^{3+}, and Ni^{2+} are important constituents of alloy, while Pb^{2+} is highly toxic and an important pollutant.

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