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Specific Separation of Gallium, Gold, and Magnesium, and Separation of Be-AI-Ga on Stannic Molybdate Papers

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NOTE

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Summary

Chromatography of 47 cations in 11 solvent systems has been performed on stannic molybdate ion exchange papers. Some difficult separations, e.g., Rb from Cs, Al–Be–Ga, and Tl–In, and specific separations of Ga, Au and Mg from numerous metal ions, have been achieved. R_F values of some common metal ions on these papers have been compared with R_F values of these cations on stannic tungstate and stannic phosphate papers. The preparation of papers is easy and the results are reproducible.

INTRODUCTION

Papers impregnated with ion exchangers combine the advantages of ion exchange, adsorption, and partition. Such papers are therefore particularly useful in difficult separations. The separations achieved in this manner are rapid, selective, and clean. With the discovery of inorganic ion exchangers the value of such papers has increased considerably. Now it is possible to prepare the papers very easily in the

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laboratory (*1–4*), and the concentration as well as the nature of the ion exchanger can be varied at will. For these reasons a systematic study of such papers needs particular attention. Lederer et al. (*5*) have described some interesting studies on zirconium phosphate papers showing the effect of pH, nature of the acid used in the eluent, concentration of the exchanger on the paper, etc. Recently a systematic study was reported on stannic phosphate and stannic tungstate papers (*6*). This study showed that these papers are very selective, difficult separations can be achieved very easily, and the time required to achieve clean separations is very short, i.e., about 40 min. However, no such study has been reported on stannic molybdate papers. The present chapter deals with the chromatography of numerous metal ions on these papers.

EXPERIMENTAL

Apparatus

Chromatography was performed on Whatman No. 1 paper strips of size 14 × 3 cm using 20 × 5 cm glass jars.

Reagents

Chemicals and solvents were either E. Merck (Darmstadt) or B.D.H. Analar grade. Stannic chloride pentahydrate (Poland) and sodium molybdate Riedel (Germany) were used.

Preparation of Ion Exchange Papers

Aqueous solutions of stannic chloride (0.25 M) and sodium molybdate (0.25 M) were prepared. Paper strips of required size were first impregnated in stannic chloride solution for 3–5 sec. The excess of the reagent was removed by placing the strips over a filter paper sheet and allowed to dry for 15 min at room temperature. The strips were then dipped into sodium molybdate solution for 5 sec, the excess solution drained off, and the strips were placed over a filter sheet. These strips were dried at room temperature and then washed with distilled water in order to remove the excess of the reagents. Finally these were dried at room temperature and used as such for chromatography.

Test Solutions

Cation solutions were prepared and detected as earlier (*8*).

Procedure

One or 2 spots of test solutions were spotted with the help of thin glass capilaries. The paper was conditioned for 15 min and the solvent was then allowed to ascend (always 11 cm). The front limit (R_L) and the rear limit (R_T) were measured.

RESULTS

Forty-seven metal ions; Te(IV), W(VI), Ir(IV), Au(III), Ru(III), Ba(II), Sr(II), K(I), Rb(I), Cs(I), Mg(II), Fe(II), As(III), Co(II), Ni(II), Ag(I), Pb(II), Hg_2(II), Hg(II), Tl(I), Bi(III), Cd(II), Pd(II), Sb(III), Fe(III), V(IV), Cu(II), UO_2(II), Cr(III), Mn(II), Zn(II), Y(III), Zr(IV), Th(IV), Ce(III); Ce(IV), La(III), In(III), Nb(V), Ca(II), Hf(IV), Ti(IV), Al(III), Be(II), Ga(III), Pt(IV), and Se(IV) have been chromatographed in the following 11 solvent systems.

1. 0.1 *N* Nitric acid
2. Acetone + acetic acid + *n*-butanol + 4 *M* HCl (1:1:1:1)
3. *n*-Butanol + HCl (7:3)
4. Ethyl methyl ketone + acetone + 50% HCl (7:3:1)
5. Acetone + acetic acid + *n*-butanol + 1 *M* NaCl (1:1:1:1)
6. Acetone + acetic acid + *n*-butanol + 4 *M* HNO_3 (1:1:1:1)
7. 0.5 *M* HCl + KCl saturated (1:1)
8. Ethyl methyl ketone + acetone + 50% HCl (6:3:1)
9. 1 *M* H_3PO_4 + 1 *M* HCl (1:1)
10. Ethyl methyl ketone + acetone + 50% HCl (3:2:5)
11. Ethyl methyl ketone + acetone + 50% HCl (1:6:1)

By using these solvents a number of interesting separations are possible. R_L and R_T are given in parentheses.

1. Separation of Au from numerous metal ions using acetone + acetic acid + *n*-butanol + 4 *M* HCl (1:1:1:1) as developer. This separation is rapid and almost specific. Probably this is the best separation of Au yet reported. Even Sb and U, which interfere in the method of Kember (*7*), do not interfere in this case. ΔR_F is greater than 0.40.

2. Separation of Mg from numerous metal ions. Mg can be separated from Ca, Sr, Ba, K, Rb, Cs, Y, Zr, Hf, Ce(III), Ti, La, Ni, Co, W, Te, Tl, Al, Cr, Hg(I), UO_2, Mn, and Cu using ethyl methyl ketone +

TABLE 1

Comparative R_F Values of Some Metal Ions on Stannic Molybdate, Stannic Phosphate, and Stannic Tungstate Papers

		R_F				R_F	
Metal ion	Solvent system	Stannic molybdate	Stannic phosphate	Metal ion	Solvent system	Stannic molybdate	Stannic tungstate
Pb	*n*-Butanol + HCl (7:3)	0.12	0.02	Hg(I)	0.5 *N* HCl + KCl satd. (1:1)	0.00	0.00
Cu		0.44	0.44	Hg(II)		0.92	0.90
Fe		0.95	0.90	Ce(III)		0.87	0.82
Zn		0.74	0.77	Ce(IV)		0.00	0.03
Th		0.04	0.01	Pt		0.81	0.80
Al		0.12	0.07	Au		0.43	0.40
Be		0.40	0.38	Ir		0.07	0.00
UO_2		0.45	0.48	Y		0.90	0.82
Ni		0.14	0.13				
Mn		0.17	0.21				

acetone + 50% HCl (3:2:5) solvent system. The separation of Mg from Ca is particularly useful. Mg moves to the solvent front while all others have R_F values less than 0.70.

3. Separation of Rb from Cs. This separation is very neatly achieved using ethyl methyl ketone + acetone + 50% HCl (3:2:5) as developer. This is one of the most difficult separations and of all the separations reported in the literature this appears to be the best. The ΔR_F is 0.50 which ensures a clean separation.

4. Al(0.18–0.06)–Be(0.45–0.35)–Ga(1.00–0.91). This separation is easily achieved in 3 hr using *n*-butanol + HCl (7:3) as mobile phase.

5. Ga(0.90–0.75)–Be(0.23–0.03), or Al(0.00), or Tl(0.00)–In(0.72–0.40). This separation is achieved in 45 min using ethyl methyl ketone + acetone + 50% HCl (1:6:1) as solvent.

DISCUSSION

A number of interesting points emerge from this study:

1. The R_F values of metal ions are almost the same for the same

developer whether the ion exchanger is stannic molybdate or stannic phosphate or stannic tungstate (*6*). This is clear from the R_F values given in Table 1.

2. The papers show excellent selectivity when developed with mixed solvents. Any solvent is efficient enough to give some interesting separations.

3. The results are reproducible and the preparation is easy and is not a difficult technique.

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