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NOTE

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Summary

Interesting separations of metal ions were observed by using the technique of reverse phase partition chromatography on 2-thenoyltrifluoroacetone—impregnated paper. Ketone–hydrochloric acid mixtures in different proportions were used as the developing solvent for the separations of different metal ion mixtures. The R_F values of metal ions separated are given and the superiority of separation over that of ordinary filter paper is discussed.

INTRODUCTION

Berg (*1*, *2*) and Strassner (*3*) separated a number of binary metal ion mixtures using 2-thenoyltrifluoroacetone (TTA) in dioxane on ordinary filter paper. Filter paper pre-treated with cellulose acetate and impregnated with TTA has been used by Kawamura, Fujimoto, and Izawa (*4*) for a few typical binary and ternary mixtures of metal ions, with acetate buffer as the developing agent, and radioactive tracers. Synergism in the reversed phase partition chromatography of americium, cerium, and lanthanum have been studied by Cvjeticanin (*5*) who used a mixture of trioctylamine and TTA-impregnated paper. The reagent TTA has also been extensively used in the authors' (*6*) laboratory for solvent extraction of metal ions. The present paper, for the first time, reports the results of systematic studies on reverse phase partition chromatography (RPPC) using TTA-impregnated filter paper (Whatman No. 1). Excellent separations of complex metal ion mixtures containing three to six metal ions were achieved in 1 to 2 hr by using mixtures of hydrochloric acid and such ketones as acetone and methyl isobutyl ketone. Beautiful and sharply defined spots were obtained after development in each run. Such spots were never observed on ordinary papers in the absence of TTA.

EXPERIMENTAL PROCEDURE

In order to impregnate filter papers (Whatman No. 1) with TTA, a 0.10-*M* solution of TTA (Columbia Organic Chemicals, Columbia, S. C.) in benzene was first prepared. Paper strips (25 × 2.5 cm) were then allowed to be in contact with TTA solution by the ascending technique until it reached the upper end. The excess of TTA solution was removed by hanging the strips vertically for 1 min and then pressing them between filter papers.

Fine glass capillaries were used to apply test solutions (containing 1 mg of each metal ion per milliliter of the solution) on the TTA-impregnated air-dried filter papers.

All the chromatographic separations were carried out in 30 × 5 cm glass jars by the technique of ascending paper chromatography and the solvent mixtures were allowed to be in contact with the TTA impregnated paper strips for about 1 to 2 hr. The different metal ions separated were developed as colored zones by spraying with suitable reagents as follows:

Trisodium pentacyanoamminoferrate + rubeanic acid: Mn (light blue), Fe(III) (deep blue), Co(II) (yellowish brown), Ni(II) (blue), Cu(II) (apple green), Zn(II) (red).

Alizarin Red S: La(III) (brown), Ce(IV) (violet), Zr(IV) (red), Th(IV) (reddish violet), In(III) (violet), Al(III) (reddish violet).

KI + $SnCl_2$: Pt(II) (yellow to brownish yellow), Pd(II) (pink to dark purple).

$K_4[Fe(CN)_6]$ + CH_3COOH: U(VI) (light brown), Mo(VI) (deep brown), V(V) (yellow).

KI (aqueous): Tl(I) (yellow).

Oxine: Nb(V) (pink), Ta(V) (brown).

NH_4SCN + $SnCl_2$: Cr(III) (red), W(VI) (red).

Rhodamine B: Ga(III) (red).

Dithizone: Zn(II) (red), Cd(II) (purple), Hg(II) (pink), As(III) (yellow), Sb(III) (red), Bi(III) (purple).

RESULTS AND DISCUSSIONS

The separations of the metal ions are summarized in Table 1. The separated metal ions have been arranged according to their increased

TABLE 1

R_F Values of Cations on TTA-Impregnated Paper

Solvent system	Separations achieved, metal ions
Acetone:HCl (6 N) = 9.0:1.0 (T = 1 hr)	Mn(0.32), Fe(1.0), Co(0.64), Ni(0.06), Cu(0.85), Zn(0.93)
Acetone:HCl (6 N) = 8.0:2.0 (T = 1 hr)	Mn(0.40), Fe(1.0), Co(0.63), Ni(0.20), Cu(0.83), Zn(0.95)
MIBK:acetone:HCl (6 N) = 1.0:8.0:1.0 (T = 1 hr)	Mn(0.08), Fe(1.0), Co(0.34), Ni(0.03), Cu(0.46), Zn(0.73)
Acetone:HCl (6 N) = 9.0:1.0 (T = 1 hr)	Zn(0.71), Cd(0.83), Hg(0.93)
Acetone:HCl (6 N) = 8.0:2.0 (T = 1 hr)	Zn(0.80), Cd(0.88), Hg(0.95)
Acetone:HCl (12 N) = 9.0:1.0 (T = 1 hr)	As(0.90), Sb(1.0), Bi(0.82)
MIBK:acetone:HCl (6 N) = 1.0:8.0:1.0 (T = 1 hr)	Al(0.08), Cr(1.0), Zr(0.64)
Acetone:HCl (12 N) = 7.5:2.5 (T = 1 hr)	Cu(0.80), Pd(0.74), Ag(1.0), Pt(0.90)
Acetone:HCl (12 N) = 9.0:1.0 (T = 1 hr)	Nb(0.75), La(0.90), Ce(0.00)
Acetone:HCl (12 N) = 8.0:2.0 (T = 1 hr)	Nb(0.66), La(;.90), Ce(0.00)
Acetone:HCl (12 N) = 7.0:3.0 (T = 1 hr)	Nb(0.70), La(0.90), Ce(0.00)
Acetone:HCl (12 N) = 6.0:4.0 (T = 1 hr)	Nb(0.78), La(1.0), Ce(0.21)
Acetone:HCl (12 N) = 7.5:2.5 (T = 2 hr)	Zr(0.63), Ce(0.07), Th(0.97)
Acetone:HCl (6 N) = 7.5:2.5 (T = 1.5 hr)	Cr(0.90), Mo(1.0), W(0.75), U(0.83)
Acetone:HCl (6 N) = 8.0:2.0 (T = 1.5 hr)	Al(0.20), Ga(1.0), In(0.90), Tl(0.40)
Acetone:HCl (12 N) = 8.0:2.0 (T = 1.5 hr)	V(0.12), Cr(0.73), Fe(1.0), W(0.40)

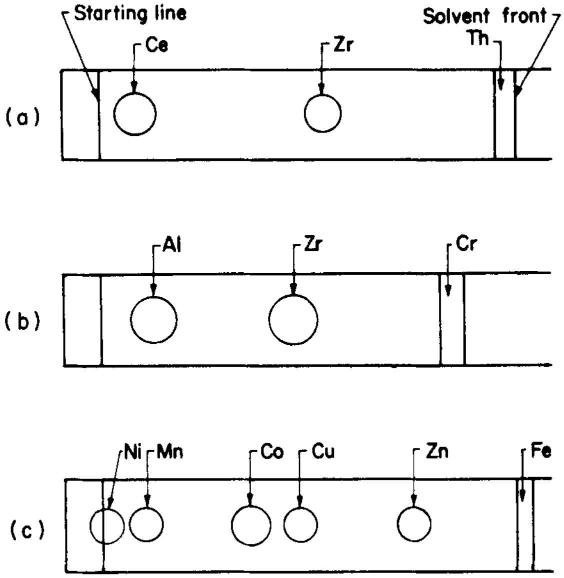

FIG. 1. Separation of metal ions on TTA-impregnated paper. Solvent systems: (a) and (b); acetone:HCl (12 *N*) = 7.5:2.5 (v/v). (c) MIBK: acetone:HCl (6 *N*) = 1.0:8.0:1.0.

atomic weights instead of by their R_F values. Eleven metal ion mixtures containing three to six metal ions have been tried. Since at low pH TTA forms chelates with most of the metal ions, 1.2 to 0.6 *N* over-all acidity of the developing solvent was sufficient for the separation of metal ions. The important results of analytical interest are given below.

1. At higher acidity, 3*d*-transition metal ions, viz., Mn(II), Co(II), Ni(II), Cu(II), and Zn(II), have higher R_F values.

2. Increased R_F values are obtained with acetone-hydrochloric acid as developing solvent rather than acetone–methyl isobutyl ketone and hydrochloric acid (Fig. 1). This fact shows that polar solvent like acetone increases the ability of TTA for metal extraction.

3. At the identical solvent composition, Fe(III) and Cr(III) have identical R_F values (equal to unity).

4. In the different separation schemes, Ag(I), Nb(V), La(III), Mo(VI), Ga(III), Fe(III), Cr(III), and Sb(III) move along with the solvent fronts and have R_F values equal to 1.0.

5. The most significant separations are: (a) Mn–Fe–Co–Ni–Cu–Zn; (b) Al–Ga–In–Tl(I); (c) V–W–Fe–Cr; (d) V–Nb–Ta; and (e) Zr–Ce–Th.

All the common metal ion mixtures were quickly and conveniently separated using this RPPC technique. For comparison, several runs were made using ordinary filter paper and identical solvent compositions. In most cases, poorer separations were achieved with much longer periods of development. Furthermore, wider bands rather than well-defined spots were obtained with ordinary paper chromatographic separations. The RPPC technique on TTA-impregnated paper thus provides a rapid, clean-cut method of separation for many complex metal ion mixtures. Other diluents, such as alcohols, esters, and ethers, may also be tried for similar separations.

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