A Selective Separation of Tl (III) from Some Metal Ions on Sodium Molybdate Impregnated Silica Gel Layers in Formic Acid-Butanol System

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A selective separation of Tl (III) from a number of metal ions has been achieved on silica gel thin layers impregnated with 0.3 M aqueous solution of sodium molybdate in formic acid-butanol solvent systems. Thallium (III) (0.4-1.0 mg) has been successfully separated from Cd, Al, Ni, Fe, Ag and Pb.

The selective separation of Tl (III) is important because of its association in minute quantities with iron, zinc, lead, copper and managanese in certain rocks and minerals. Our recent thin layers chromatographic studies\(^1\) showed that silica gel layers impregnated with sodium molybdate behave differently than plain silica gel layers and offer attractive possibilities for the selective separation of metal ions from aqueous formic acid systems. Formic acid (FA), being sufficiently acidic, prevents hydrolysis of salts and is capable of forming complexes with many metals.\(^3\) Keeping this in views, a method has been developed for the selective separation of Tl (III) on sodium molybdate impregnated silica gel layers with FA-butanol solvent systems.

Silica gel thin layers were prepared on glass plates (20 × 3.5 cm) by using a thin-layer chromatography apparatus (Toshniwal, India), and the plates were developed in glass jars (24 × 6 cm).

Silica gel (BDH), sodium molybdate dihydrate (Veb Jena Phar, Germany), formic acid (Merck) and n-butanol (BDH) were used as such. All other reagents were of AR grade.

The test solutions were ~ 0.1 M in the metal chloride or nitrate. Conventional spot test reagents were used for the detection of metal ions.

The solvent system concentrations used were 0.1 M, 0.5 M, 2.0 M, 5.0M and 10.0 M formic acid in butanol.

The impregnated silica gel plates were prepared by mixing pure silica gel with aqueous solution of sodium molybdate (0.3 M) in 1:3 ratio with constant shaking for 5 min. The slurry so obtained was spread over glass plates to get a layer of 0.25 mm thickness. The plates were air-dried, and then activated at 100 ± 2°C for 2 hr. The plates were kept in a closed chamber until used.

**Procedure**

One or two drops of the cation solutions were spotted on the plates with the help of thin glass capillaries. The spots were allowed to dry and then the plates were developed in the chosen solvent system by the ascending technique. The ascent of the solvent was fixed at 10 cm in all cases. After development, the plates were dried and the cation were detected with appropriate colouring reagents.

Impregnated silica gel layers show unusual selectivity towards metal ions, most of which are strongly adsorbed and remain near the point of application. The metal ions Th\(^{4+}\), Zr\(^{4+}\), Hg\(^{2+}\), Hg\(^{2+}\), Bi\(^{3+}\), Cd\(^{2+}\), UO\(^{2+}\), Al\(^{3+}\), Tl\(^{+}\), Ag\(^{+}\), Hg\(^{2+}\), Fe\(^{2+}\), VO\(^{2+}\), W\(^{6+}\), Ni\(^{2+}\), Co\(^{2+}\), Ce\(^{4+}\), Zn\(^{2+}\), Pb\(^{2+}\), Cu\(^{2+}\), Ti\(^{4+}\), Nb\(^{5+}\), and Ta\(^{5+}\) do not show any mobility while Tl\(^{3+}\) moves to give \(R_F\) value 0.8-0.85 over the entire range of formic acid concentrations (0.1-10.0 M). This weaker interaction of Tl\(^{3+}\) with the sorbent phase leads to its selective separation from all the above mentioned metal ions. Thallium (III) produces fine streak at low loading (10 µg) while at higher loading (100 µg-l mg) it gives good triangular spot. A slight decrease in the \(R_F\) of Tl\(^{3+}\) is observed when it is chromatographed as synthetic mixture with other metal ions. Several qualitative separations of Tl\(^{3+}\) (\(R_F=0.65-0.75\)) from Ni\(^{2+}\), Co\(^{2+}\), Fe\(^{3+}\), Zn\(^{2+}\), Zr\(^{4+}\), Bi\(^{3+}\), Al\(^{3+}\), Tl\(^{+}\), W\(^{6+}\), Pb\(^{2+}\), Ti\(^{4+}\), and Cu\(^{2+}\) were experimentally achieved. We have successfully separated Tl\(^{3+}\) (upto 400 µg) from Ni\(^{2+}\) (10-60 µg), Bi\(^{3+}\) (50-210 µg) UO\(^{2+}\) (50-240 µg); upto 600 µg from Cd\(^{2+}\) (50-350 µg), Al\(^{3+}\) (10-81 µg) and upto 1 mg from Zn\(^{2+}\) (65.4 µg), Fe\(^{3+}\) (56 µg), Ag\(^{+}\) (108.2 µg) and Pb\(^{2+}\) (210.0 µg) using formic acid (1.0 M) in butanol as mobile phase.

Substitution of butanol by methanol leads to the formation of tailed spots and all metals except Al\(^{3+}\) show considerable tailing over the entire
range of formic acid concentration (0.1-10.0 \textit{M} in MeOH). The presence of 5-90\% acetone in formic acid (10.0 \textit{M} or 1.0 \textit{M}) in butanol does not alter the retention sequence of metal ions.

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References