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## NOTE

# Synthetic Inorganic Ion Exchangers. XVI. Electrochromatographic Separations of Metal Ions on Zirconium Tungstate-Impregnated Paper

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### Abstract

The electrochromatographic behavior of 25 metal ions on zirconium tungstate-impregnated papers is described. Six background electrolytes were used. On the basis of the differential mobilities of metal ions which depend on the ion-exchange properties of zirconium tungstate and the nature of complex formation with the electrolytes, some important binary and ternary separations have been achieved.

## INTRODUCTION

Electrochromatography is a well-known analytical tool for the separation of metal ions (*1*). Inorganic ion exchangers have recently found remarkable application in analytical separations (*2*). The combination of these two techniques is expected to provide some interesting results. Some studies on electrochromatographic separation using inorganic ion exchanger-impregnated paper have been reported (*3–7*). These papers are highly selective, and excellent separations of metal ions can be achieved. Zirconium tungstate has been explored as an ion exchanger (*8*) but paper impregnated with this exchanger has not been studied. The present work summarizes our studies on electrochromatographic separations of metal ions using zirconium tungstate-impregnated paper. The results were compared with those on Whatman No. 1 paper as blank runs. On the basis of the ion-exchange properties of zirconium tungstate and the migration of metal ions under the electrical potential applied, some important separations are reported.

## EXPERIMENTAL

### Apparatus

Electrochromatographic work was done with an electrophoresis apparatus (Optronics, India, Model EPR 550).

### Reagents

Zirconium oxychloride was supplied by Indian Rare Earths Ltd., India. All other reagents were of analytical grade (E. Merck/B.D.H.). Whatman No. 1 chromatographic paper (40 × 2.5 cm) was used for the electrochromatographic work.

### Preparation of Ion-Exchange Papers

The paper strips (40 × 2.5 cm) were first soaked with 0.1 *M* zirconium oxychloride (in 0.1 *M* $HNO_3$) solution for about 30 sec, then the excess zirconium oxychloride solution was removed with blotting paper and the paper strips were dried at room temperature. The strips were again immersed in a 0.1-*M* sodium tungstate (at pH 3, adjusted with $HNO_3$) solution for about 30 sec. The excess solution was removed with blotting paper and dried at room temperature. After drying, the strips were washed with distilled water and finally dried at room temperature before being used as ion-exchange papers.



### Cation Solutions

In general, the test solution had a metal concentration of 4 mg/mL (chloride/nitrate/sulfate). Bismuth nitrate solution was prepared in dilute nitric acid.

### Detection Reagents

These are listed in Table 1.

### Background Electrolytes

These are listed in Table 2.

TABLE 1

Detection Leagents

| Reagents | Cations detected |
|---|---|
| 1. Yellow ammonium sulfide | $Ag^+$, $Tl^+$, $Pb^{2+}$, $Hg^{2+}$, $Bi^{3+}$, $Pd^{2+}$ |
| 2. Potassium ferrocyanide | $Fe^{3+}$, $UO_2^{2+}$ |
| 3. Rubeanic acid | $Cu^{2+}$ |
| 4. Dithizone | $Cd^{2+}$, $As^{3+}$, $Zn^{2+}$ |
| 5. Alkali and benzidine | $Mn^{2+}$ |
| 6. Benzidine | $Ce^{4+}$, $Au^{3+}$ |
| 7. Potassium iodide | $Pt^{4+}$ |
| 8. Dilute $SnCl_2$ and KI solution | $Rh^{3+}$ |
| 9. Dimethylglyoxime and ammonia | $Ni^{2+}$ |
| 10. 1-Nitroso-2-naphthol | $Co^{2+}$ |
| 11. Hydrogen peroxide | $Ti^{3+}$ |
| 12. Alkaline quinalizarin | $Be^{2+}$, $Mg^{2+}$ |

TABLE 2

Background Electrolytes

| |
|---|
| 1. 0.001 *M* $HNO_3$ |
| 2. 0.05 *M* HCl + 0.09 *M* KCl (1:1) |
| 3. 0.1 *M* $HClO_4$ |
| 4. 0.05 *M* citric acid |
| 5. 0.1 *M* $HNO_3$ + 5% $KNO_3$ (1:1) |
| 6. 0.1 *M* $NH_4Cl$ |



## Procedure

The electrode and electrode vessels were washed with deionized water and dried. The base plate which contains electrode was kept in the horizontal position. Equal volumes of electrolyte were poured into these vessels. The paper strips were soaked with the corresponding electrolyte and the excess solution was removed with blotting paper. These sheets, which act as carriers, were placed in position in the cassette. Then the metal ions were applied separately in the middle of each strip with the help of a fine glass capillary. The cassette was then covered and a potential difference was applied for a fixed period. Then the sheets were dried and the spots were developed with the corresponding detecting agents. The distance of migration (in cm) was measured from the center of the paper to the center of the spots.

TABLE 3

Separations of Metal Ions on Zirconium Tungstate Papers by Electrochromatographic Technique (time: 2 hr)

| Background electrolyte | Electrical potential applied (V) | Separation achieved |
|---|---|---|
| 1. 0.001 *M* $HNO_3$ | 200 | 1. $Ni^{2+}(-5.0)$–$Co^{2+}(-7.2)$ |
| | | 2. $Fe^{3+}(-0.2)$–$Pb^{2+}(-2.0)$ |
| | | 3. $Ag^{+}(0.0)$–$Tl^{+}(-2.6)$ |
| | | 4. $Ag^{+}(0.0)$–$Cu^{2+}(-4.6)$ |
| | | 5. $UO_2^{2+}(-1.0)$–$Mn^{2+}(-4.0)$ |
| | | 6. $UO_2^{2+}(-1.2)$–$Al^{3+}(-5.0)$ |
| | | 7. $UO_2^{2+}(-1.0)$–$Cd^{2+}(-4.2)$ |
| | | 8. $Tl^{+}(-2.1)$–$Ni^{2+}(-5.2)$–$Co^{+2}(-6.7)$ |
| 2. 0.05 *M* HCl + 0.09 *M* KCl (1:1) | 100 | 1. $Pb^{2+}(-0.5)$–$Ni^{2+}(-2.8)$ |
| | | 2. $Pb^{2+}(-0.5)$–$Mn^{2+}(-3.2)$ |
| | | 3. $Pb^{2+}(-0.2)$–$Zn^{2+}(-2.2)$ |
| | | 4. $UO_2^{2+}(-1.8)$–$Be^{2+}(-3.2)$ |
| | | 5. $Pt^{4+}(+1.5)$–$Pd^{2+}(+3.5)$ |
| | | 6. $Fe^{3+}(-0.5)$–$UO_2^{2+}(-2.0)$ |
| | | 7. $Fe^{3+}(-0.5)$–$Cu^{2+}(-2.2)$ |
| 3. 0.1 *M* $HClO_4$ | 100 | 1. $Bi^{3+}(0.0)$–$Cd^{2+}(-2.0)$ |
| | | 2. $Bi^{3+}(0.0)$–$Fe^{3+}(-2.8)$ |
| | | 3. $Bi^{3+}(0.0)$–$Pb^{2+}(-2.2)$ |
| | | 4. $Pb^{2+}(-2.6)$–$Ni^{2+}(-4.0)$ |
| | | 5. $Pb^{2+}(-2.6)$–$Cu^{2+}(-4.0)$ |
| | | 6. $UO_2^{2+}(-3.0)$–$Cu^{2+}(-4.2)$ |
| 4. 0.05 *M* citric acid | 200 | 1. $Pb^{2+}(-1.2)$–$Cu^{2+}(-3.5)$ |
| | | 2. $Au^{3+}(+0.5)$–$UO_2^{2+}(-1.5)$ |
| | | 3. $Fe^{3+}(-0.8)$–$Pd^{2+}(+0.5)$ |



## RESULTS AND DISCUSSION

As a result of the electrochromatographic study of zirconium tungstate ion-exchange papers with the use of a suitable background electrolyte and the appropriate potential, a number of separations has been achieved.

The rates of movement of metal ions which are strongly adsorbed by zirconium tungstate exchangers, e.g., $Bi^{3+}$, $Pb^{2+}$, $Tl^{+}$, and $Fe^{3+}$ (*8*), are comparatively low. Thus these ions can be separated from numerous metal ions. The important separations are summarized in Table 3. Some difficult separations, e.g., $Co^{2+}$–$Ni^{2+}$, $Ag^{+}$–$Tl^{+}$, $UO_2^{2+}$–$Mn^{2+}$, $Cu^{2+}$–$Cd^{2+}$, $Pb^{2+}$–$Cu^{2+}$, $Bi^{3+}$–$Fe^{3+}$, and $Au^{3+}$–$UO_2^{2+}$, are easily achieved. Such separations are not possible on ordinary Whatman paper.

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