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Thin-Layer Chromatographic Separation of Tropolone Chelates

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NOTE

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Abstract

Speedy microanalytical separation of tropolone chelates of various metal ions have been made on silica gel "G" support.

The separation of metal chelates by thin-layer chromatography (TLC) has a large analytical scope so far as the reproducibility and speed of the separations are concerned. Separation based on metal chelates affords several advantages over the procedure dealing directly with free cations.

The presence of hydroxyl and carbonyl groups in favorable positions in the tropolone molecule makes it an excellent bidentate chelating agent. Tropolone (I) has been found to form a large number of chelates with metal ions. On account of the extractibility of a large number of metal tropolone chelates, tropolone cannot be used as a selective reagent. In the present study, ions known to form tropolone chelates have been grouped while keeping in view, as far as possible, those which present group

similarities, are present together in ores or materials of industrial importance, or those which form extractable chelates under similar conditions. The constitutions of such groups of ions have been separated into individual ions using TLC. The groups of ions investigated were

1. Ruthenium(III), rhodium(III), and palladium(II).
2. Titanium(IV), iron(III), and vanadium(V).
3. Nickel(II), cobalt(II), and uranium(VI).
4. Uranium(VI), lead(II), and iron(III).

EXPERIMENTAL

Metal Solutions. 1000 ppm solutions of Ru(III), Rh(III), Pd(II), Ti(IV), Fe(III), V(V), Ni(II), Co(II), U(VI), Cu(II), Cd(II), and Pb(II) were prepared by taking their analytical grade reagent.

Tropolone Solution. 0.02 M tropolone (Aldrich, U.S.A.) solution in distilled water was used.

Glass Plates. Those coated with silica gel "G" were employed.

Visualizing Agent. 0.05 M aqueous potassium thiocarbonate (PTC) was employed.

The solvents used were distilled before use.

Preparation of Metal-Tropolone Chelates

Suitable aliquots of solutions containing 300 to 600 μg of a metal ion were taken. To this was added 5.0 ml of $M/50$ tropolone solution, followed by 5 ml of sodium acetate-acetic acid buffer of pH 5 and diluted to 15 ml. The chelate so formed was extracted into chloroform. The chloroform phase was separated. The conditions for chelate formation for a particular metal ion are listed in Table 1.

Separation Procedure

By means of a microsyringe, 2 to 4 μl of the chloroform extract of the metal-tropolone chelates was brought separately to one end of the base line, and on the other end of the base line 2 to 4 μl of each of the respective metal chelates was applied at the same spot so as to allow mixing of the metal chelates in known concentrations. The jar containing the eluents (chloroform-benzene) was allowed to saturate with the vapors for 5 to 10 min. The spotted plate was kept in the jar and a chromatogram was run.

TABLE 1

Metal ions	pH range	Extracting solvent	Color of the extracted chelate	Other conditions needed for complete extraction
Ru(III)	4-6	Chloroform	Yellow	On heating for 10 min at 80°C
Rh(III)	5-6.5	Chloroform and pyridine	Red	On heating for 5 min
Pd(II)	4-9	Chloroform	Reddish-brown	On heating at 80°C for 10 min
Ti(IV)	4-8 <i>NHCl</i>	Chloroform, MIBK, isobutyl alcohol, isoamyl alcohol	Yellow	High chloride ion concentration
Fe(III)	1-9	Chloroform	Red	—
V(V)	4-7 <i>NHCl</i>	Chloroform, carbon tetrachloride	Blue	—
Ni(II)	6-9	Chloroform	Reddish-brown	Pyridine
Co(II)	5-9	Chloroform	Yellow	Pyridine
U(VI)	4.5-7	Chloroform	Yellowish-orange	Pyridine
Cu(II)	5-8.5	Chloroform, benzene, MIBK	Green	—
Pb(II)	6-9	Chloroform	Yellow	—
Cd(II)	6-8	Chloroform	Yellow	—

TABLE 2
Results of TLC Separation

Metal ion	Eluents	Spray reagent	Color of the spot	$R_f \times 100$	Limits of identification (μg)
Ru(III)	Chloroform: benzene (3:1)	Tropolone	Yellow	86.0	0.12
Rh(III)	Chloroform: benzene (3:1)	Tropolone	Red	38.0	0.24
Pd(III)	Chloroform: benzene (3:1)	Tropolone	Reddish-brown	64.0	0.38

The plate was taken out when the solvent front had moved 8 to 10 cm. The results of the separation are given in Table 2.

Separation of Ti(IV), Fe(III), and V(V)

The separation procedure was the same as for the previous separation.

The position of the spots on the chromatoplate was distinct, and no spray reagent was necessary because the metal chelates had characteristic colors. The results of the TLC separation are given in Table 3.

Separation of Ni(II), Co(II), and U(VI)

The separation procedure followed was the same as for the first separation except that chloroform:pyridine:MIBK (15:2:5, v/v) was employed as the solvent system. The development time for clean separation was 10 min. The separation results are given in Table 4.

Separation of Cu(II), Fe(III), and Cd(II)

The separation procedure adopted was the same as that given for the previous cases except that the solvent system employed was chloroform:

TABLE 3
Results of Separation

Metal ion	Eluents	Color of the spot	$R_F \times 100$	Limits of identification (μg)
Ti(IV)	Chloroform: butanol (5: 2)	Yellow	57.0	0.20
V(V)	Chloroform: butanol (5: 2)	Bluish-black	0	0.10
Fe(III)	Chloroform: butanol (5: 2)	Red	88.0	0.08

TABLE 4
Results of Separation

Metal ion	Eluents	Spray reagent	Color of the spot	$R_F \times 100$	Limits of identification (μg)
Ni(II)	Chloroform: pyridine: MIBK (15: 2: 5)	Tropolone	Reddish-brown	76.0	0.50
Co(II)	Chloroform: pyridine: MIBK (15: 2: 5)	Tropolone	Yellow	88.0	0.25
U(VI)	Chloroform: pyridine: MIBK (15: 2: 5)	Tropolone	Orange	0	0.35

TABLE 5
Results of Separation

Metal ion	Eluents	Spray reagent	Color of the spot	$R_f \times 100$	Limits of identification (μg)
Cu(II)	Chloroform: MIBK (3:1)	PTC	Black	75.0	0.25
Fe(III)	Chloroform: MIBK (3:1)	—	Red	45.0	0.45
Cd(II)	Chloroform: MIBK (3:1)	PTC	Yellow	0	1.65

MIBK (3:1, v/v). The development time for clean separation was 15 min. The separation results are given in Table 5.

Detection. Detection of cadmium and copper was made by spraying with aqueous potassium thiocarbonate reagent. In the case of iron there was no need for spraying because the color (red) was distinct.

Separation of Pb(II), U(VI), and Fe(III)

The procedure followed for the separation of these constituents was the same as that applied for other cases except the solvent system employed was pyridine. The development time was 10 min. The results of the separation are tabulated in Table 6.

TABLE 6

Metal ion	Eluent	Spray reagent	Color of the separation	$R_f \times 100$	Limits of identification (μg)
Pb(II)	Pyridine	PTC	Pinkish-red	76.0	3.80
U(VI)	Pyridine	—	Yellow	57.0	0.36
Fe(III)	Pyridine	—	Red	92.0	0.04

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