

Identification of Toxic Metals and TLC Separation by Using Aq. Humic Acid

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ABSTRACT

Thin layer chromatographic method is used for the separation and identification of metal ions. The separations were performed on thin layer of silica gel-G using aqueous humic acid as mobile phase. The chromatographic behavior of metal ions in surfactant mixed solvents was generally studied by using the thin layer of Silica Gel-G. Effect of concentrations, effect of pH. Effect of adsorbent effect on the R_f value of different metal cations were studied by using Aq. Humic acid as a mobile phase.

Key words: separation, TLC, aqueous, pH, adsorbent, metal ions

I. INTRODUCTION

The distribution of metals in the environment is governed by the properties of the metal and influences of environmental factors[1]. In generic term Heavy metals for metallic elements having an atomic weight higher than 40.04. The entry of Heavy metals in environment by natural and anthropogenic means. Like mining natural weathering of the earth's crust soil erosion, urban runoff, industrial wastage. Thin layer chromatography is a versatile technique for the identification and separation of organic and in-organic compounds [3,4]. The main factor which influences TLC separations is the coordination properties of solvent. Heavy metals have received considerable attention from analysts, because of their physical and environmental importance [5, 6]. Some of the metals like, T, As, Cr, Hg Tl, Cd, Ni, Zn, Pb, Cu, Fe, Mn, Co are harmful and toxic to human health. These metal ions can form complexes with bio-ligands containing Oxygen, Nitrogen and Sulphur atoms [7], which causes many problems by their redox processes in living organisms.

There are different analytical techniques of detection and separation of chromium including graphite furnace atomic-absorption spectroscopy 1891 atomic emission spectroscopy [10], normal phase and reverse phase thin layer chromatography [11,13] ion exchange chromatography (14,15] precipitation floatation [16].

This paper deals with the rapid separation of heavy metal ions present in three, as well as four component mixtures on non-impregnated silica gel G coated plates, using aqueous solution of Humic acid as a mobile phase. Humic acid is a principal component of humic substances, which are the major organic constituents of soil (humus), peat, coal, many upland streams, dystrophic lakes, and ocean water 1171 Generally biodegradation of dead organic matter produces humic acid. It is not a single acid but it is a complex mixture of many different

acids which containing phenolate and carboxyl groups so that the mixture behaves functionally as a dibasic acid or, sometimes as a tribasic acid The structure of Humic acid is as follows.

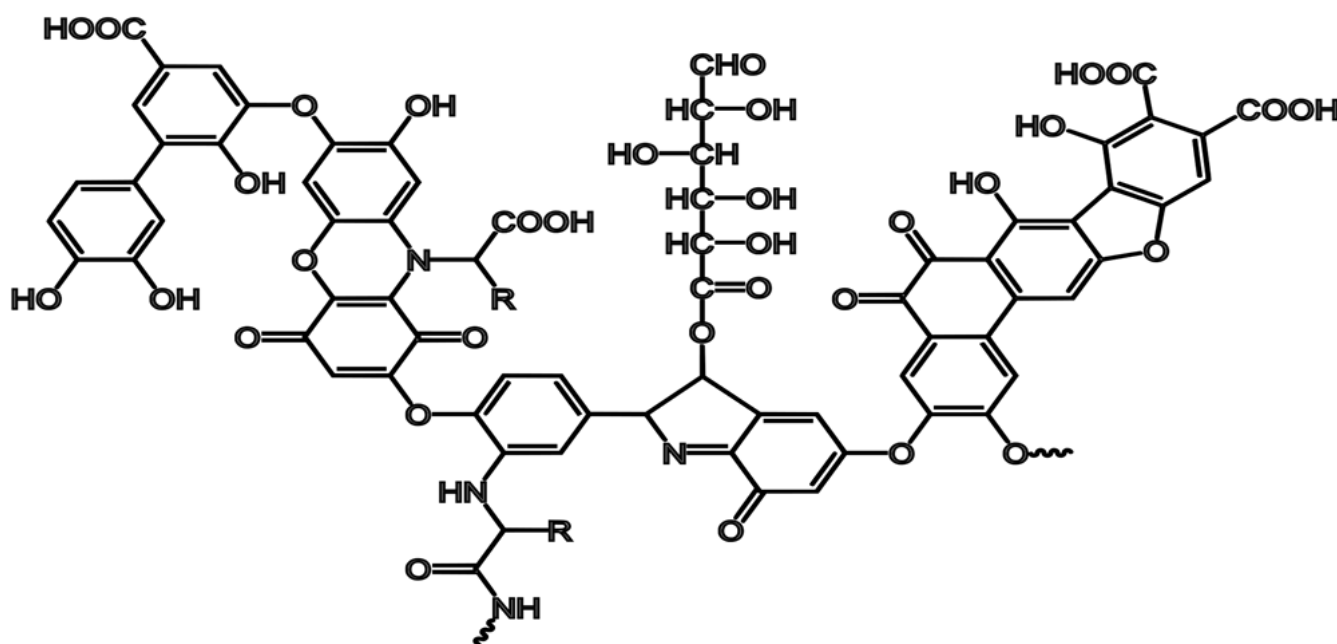


Fig. 1 Structure of Humic acid

II. EXPERIMENTAL SECTION

Apparatus

Glass plates of 4 x 20 cm size (coated with silica gel 'G'), 20 x 25 cm glass jars for the development of glass plates, glass sprayer for spraying reagents and El pH meter.

Chemicals and Reagents:

Humic acid, silica gel-G, HCl and sodium hydroxide

Metal ion studied:

Ni (II). Me (VI)Cr (VI). Cr (III) Co (II), Zn (II), Ca (II), and Fe(III)

Stock Solutions:

Stock solutions of 1% of following salts were prepared in the 0.1 M HCl.

- Potassium salt of Cr (VI)
- Chloride of Zn (II), Cr (III), Fe (III) and Ni (II)
- Sulphate of Cu (II)
- Trioxide of Mo (VI)
- Nitrate of Co (II)
- The mobile phase was prepared in double distilled water

Detection Reagents:

For the detection of various cations, below reagents were used

- 0.05 % Dithiozone in carbon tetrachloride.
- Saturated alcoholic AgNO₃

- Saturated alcoholic alizarin red
- 1 % Alcoholic solution of DMG
- 1 % Aqueous potassium ferrocyanide.
- Stationary phase Silica gel – G

Thin-layer chromatography (TLC)

- **Preparation of plates**

Slurry was prepared by mixing silica gel 'G' in double distilled water in the ratio of 1:2 with constant steering for about 10 minutes. Then by using dipping method it was immediately applied to the glass plate 1211 and then plates were dried over night at room temperature.

- **Running of TLC plates**

The test solutions were spotted on the uniformly coated silica gel-G plates using fine glass capillaries and they were blow-dried with hot air. The aqueous humic acid of varying concentration was adjusted to the desired pH using sodium hydroxide and hydrochloric acid solution. The plates were developed for about 15 min in the glass jar which containing 15 ml aqueous humic acid solution At least 2 -3 ml of mobile phase was required to run the sample per plate.

- **Development of TLC plates**

The plates were dried and different metal cations were detected by spraying various detecting reagent, which are saturated alcoholic alizarin red, saturated alcoholic silver nitrate, dimethylglyoxime, potassium ferrocyanide and dithiozone in carbon tetra chloride. The R_f values of metal cations were measured in triplicate for each set of determinations. Various experiments were carried out to study the mobile phase to (0.005M – 0.1M), pH (1.0-7.0) and time (5 - 20 min) for the R_f values of the individual cations.

III. RESULTS AND DISCUSSION:

Effect of pH

Various experiments were carried out at different run time different pH and at different concentration of aqueous humic acid for determining optimum conditions for separation of the metal ions. The R_f values have been measured at pH 4.0 and 0.01% concentration of aqueous Humic acid. It was observed that metal ions shows a little difference in the R_f value of pH 3.5 but as the pH was increased To 4 there was maximum difference in the R_f value of different metal ions. The clear separation was observed only at pH 4.0 Thus separation measurement has Carried out at pH 4.0, which has been considered as optimum separating pH in aqueous Humic acid media.

Table 1. Effect of pH on the R_f value of metal ions

Metal ions	pH of Humic acid										
	1	2	2.5	3	3.5	4	4.5	5	5.5	6	7
Cr (IV)	0.97	0.96	0.97	0.98	0.98	0.98	0.97	0.97	0.96	0.90	0.90
								DS	DS	DS	DS
Cr (III)	0.88	0.88	0.86	0.74	0.75	0.76	0.75	0.75	0.75	0.72	0.92
Co (II)	0.78	0.75	0.76	0.72	0.70	0.56	0.46	0.45	0.45	0.46	0.46

Ni (II)	0.89	0.87	0.86	0.86	0.84 Spr	0.64 spr	0.52 Spr	0.51 spr	0.52 Spr	0.53	0.53
Zn (II)	0.74	0.70	0.69	0.45	0.15 T	0.15 T	0.15 T	0.16 T	0.18 T	0.17 T	0.17 T
Cu (II)	0.73	0.70 spr	0.44 spr	0.43	0.36	0.15	0.14	0.14	0.15	0.14	0.13
Fe (III)	0.75	0.72 spr	0.45 Spr	0.44	0.31	0.12	0.11	0.12	0.11	0.10	0.10
Mo (VI)	0.48	0.48	0.45	0.44	0.36 T	0.15	0.15 T	0.14 T	0.14 T	0.15	0.15

Notation : T- Tailing, Spr – Spreading, DS – Double spot

IV. CONCLUSION:

Humic acid was found to be good mobile phase for separation of metal ions. Using the above mentioned optimum separating conditions, of pH aqueous humic acid 4.0, qualitative separation of various binary mixtures, ternary mixtures, five quaternary mixtures of metal ions have been carried out.

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