



Scholars Research Library

Der Pharma Chemica, 2011, 3 (6):474-482
(<http://derpharmachemica.com/archive.html>)



ISSN 0975-413X
CODEN (USA): PCHHAX

Chromatographic Behavior of Metal Cations on Addition of Various Additives in Aq. BAC Mobile Phase

Sarang S. Dhote¹, Lata Deshmukh² and L. Paliwal²

¹Post Graduate Department of Chemistry, Hislop College, Civil Lines, Nagpur(M.S.) India

²Post Graduate Teaching Department of Chemistry, Rashtrasant Tukdoji Maharaj Nagpur University Campus, Nagpur (M.S.) India

ABSTRACT

A TLC method has been developed for the separation of various metal ions on silica gel layers with mixed organic solvents containing cationic surfactant BAC. The separation of various metal ions, under a range of different concentration of various additives was examined. Cd^{2+} , Zn^{2+} , Co^{2+} , and Cu^{2+} were successfully separated from binary mixture.

Keywords: TLC, BAC, Zn^{2+} , Silica gel, Cationic Surfactant.

INTRODUCTION

Thin layer chromatography (TLC) is considered to be superior to other chromatographic techniques, because of its simplicity and relatively low cost. Success in TLC depends to great extent upon the proper selection of the mobile phase. The separation possibilities in TLC are greatly enhanced when chromatoplates are developed with mixed solvent systems. TLC has been successfully utilized for various purposes, such as the separation of metal ions from a water sample [1], the characterization of the mobility of metal [2]. The use of aqueous surfactant solution as a mobile phase in TLC was pioneered by Armstrong and Terrill [3]. Using a surfactant as the mobile phase gained popularity and became more widely applied due to its operational simplicity, cost effectiveness, relative non-toxicity and enhanced separation efficiency [4-8]. The use of silica gel and an alumina layer with surfactant –mediated mobile phase systems [9-14] has been used to separate various inorganic species. In the present study a new mobile phase solvent system was developed, implanting a cationic surfactants benzalkonium chloride (BAC), when employed as a mobile phase, BAC may be used to separate various metal ions under a range of different conditions.

We herein describe the continuation of our earlier work on TLC [1and2], and discuss our systematic study of the separation and selectivity of mobile phases for different metal ions using TLC with silica gel 'G' in thin layers.

MATERIALS AND METHODS

Chemicals and Reagents: Silica gel 'G' and benzalkonium chloride (BAC) were obtained from Merk, dimethylglyoxime, dithizone, potassium ferrocyanide, carbon tetrachloride, methanol, ethanol, propanol, butanol, NaCl glacial acetic acid, and HCl were obtained from SD Fine India. All other chemicals were of analytical reagent grade.

Metal ion Studied: Zn^{2+} , Cd^{2+} , Hg^{2+} , Fe^{3+} , Ni^{2+} , Co^{2+} , Cu^{2+} , Bi^{3+} , U^{6+} , V^{5+} , Zr^{4+} , Sb^{3+} , Pb^{2+} , Ag^+ , Th^{4+} , Mo^{6+} , As^{3+} , and W^{6+} .

Test Solution: TLC was performed using a standard aqueous solution (1%) of the chloride, nitrate or sulphate salts of the metal ions listed.

Detection: Fe^{3+} , Cu^{2+} , U^{6+} , V^{5+} , Zr^{4+} , and Th^{4+} were detected using 1% aqueous potassium ferrocyanide; Zn^{2+} , Cd^{2+} , Hg^{2+} , Bi^{3+} , Sb^{3+} , Pb^{2+} , Ag^+ , Mo^{6+} , As^{3+} , and W^{6+} using 0.5% dithizone in carbon tetrachloride; and Ni^{2+} , and Co^{2+} using a 1% solution of alcoholic dimethylglyoxime.

Stationary Phase: Silica gel 'G' was used as the stationary phase.

Mobile Phase: The various solvent systems that were used as the mobile phases are listed in Table No.1

Table 1: Mobile phase systems

S.No.	Symbol	Compositions
A.1	H1	Pure water mobile phase system
A.2	B 1	3% aq. BAC at pH 3.5
B	Addition of Alkanols	
B.1	M1	3% aq. BAC + Methanol [9:1, V/V], at pH 3.5
B.2	M2	3% aq. BAC + Methanol [8:2, V/V], at pH 3.5
B.3	M3	3% aq. BAC + Methanol [7:3, V/V], at pH 3.5
B.4	M4	3% aq. BAC + Ethanol [9:1, V/V], at pH 3.5
B.5	M5	3% aq. BAC + Ethanol [8:2, V/V], at pH 3.5
B.6	M6	3% aq. BAC + Ethanol [7:3, V/V], at pH 3.5
B.7	M7	3% aq. BAC + Propanol [9:1, V/V], at pH 3.5
B.8	M8	3% aq. BAC + Propanol [8:2, V/V], at pH 3.5
B.9	M9	3% aq. BAC + Butanol [9:1, V/V], at pH 3.5
B.10	M10	3% aq. BAC + Butanol [8:2, V/V], at pH 3.5
C	Addition of Electrolyte	
C.1	M11	3% aq. BAC + 1ml Acetic acid at pH3.5
C.2	M12	3% aq. BAC +3ml Acetic acid at pH3.5
C.3	M13	3% aq. BAC + 1gm NaCl at pH 3.5
C.4	M14	3% aq. BAC + 3gm NaCl at pH 3.5

Thin - layer chromatography

A. Preparation of Plates: The TLC plates were prepared by mixing silica gel with demineralised water in ratios of 1:2 by weight with constant shaking to obtain homogeneous slurry. The slurry was then immediately applied to the glass plates using the dipping method. The plates were allowed to dry overnight at room temperature and were used following day in TLC.

B. Procedure: Test solutions were spotted onto thin- layer plates with the help of a micropipette positioned about 1.0 cm above the lower edge of the TLC plates. The spots were air dried and the plates were then developed with the given mobile phase using by the one dimensional ascending technique in glass jars. The development distance was fixed at 10 cm in all cases. Following development, the plates were again air dried and the spots of the cations were visualized as coloring spots using the appropriate spraying reagent. R_F values were then calculated.

C. Separation: For the separation, the metal ions to be separated were mixed in equal amounts. A test solution of the resultant mixture was spotted onto the activated TLC plate, and was then air dried. The plates were developed to a distance of 10 cm. The spots were detected and the separated metal cations were identified by their R_F values.

D. Limits of Detection: The limits of detection of the metal cations were determined by spotting different amounts of metal ion onto the TLC plates, developing the plates using the method describe above, and then detecting the spots. This method was repeated with a successive decrease in the amount of metal ion used until spots were not detected. The minimum detectable amount on the TLC plates was taken as the limit of detection.

RESULTS AND DISCUSSION

The results of the study describe herein are summarized in Table2 to 8 and in Figures 1 and 2. The mobility of 18 metal cations was examined on silica gel layers, using aqueous solutions of BAC in order to optimize the experimental conditions used. The effect of addition of methanol, ethanol, propanol, butanol, acetic acid and NaCl at various range of concentration on mobility of metal ion was examined, at pH3.5.

Table 2. R_F values of metal cations in Pure water and 3% BAC at 3.5pH mobile system

METAL	H1	B1	METAL	H1	B1
Cu ²⁺	0.15T	0.26	Co ²⁺	0.62T	0.53
Zn ²⁺	0.27T	0.26	Hg ²⁺	0.92T	0.85
Pb ²⁺	0	0	Ag ⁺	0.53T	0.09
Fe ³⁺	0.07	0.26	Zr ⁴⁺	0.89T	0.39
Ni ²⁺	0.52	0.58	V ⁵⁺	0.35T	0.38
Sb ³⁺	0.91T	0.66	Cd ²⁺	0.31T	0.53
As ³⁺	0.87	0	Bi ³⁺	0.62T	0.52
U ⁺⁶	0.15	0.3	W ⁶⁺	0	0
Th ⁴⁺	0.13	0	Mo ⁶⁺	0	0

A.1. Pure water mobile phase system and 3% aq. BAC mobile phase system at pH3.5:

For the study the effect of addition of various alcohols and electrolytes in 3% aq. BAC at pH3.5, we were taking pure water and 3% aq. BAC at pH 3.5 mobile phase system from where we get R_F

values of metal cations which were used as reference for other system. The R_F values of the metal cations obtained under these varying concentrations are listed in table 2.

T- Tailed spot

From the data shown in Table 2, following conclusion may be drawn:

1. In pure water mobile system all metal cations gave tailed spot, whereas in B1 mobile system metal cations gave compact spot with higher mobilities as compared to H1 mobile phase.
2. Ag^+ , Th^{4+} and As^{3+} shows little mobilities in H1 but as soon as there were addition of BAC them mobilities get decreases.
3. W^{6+} and Mo^{6+} did not show any mobility in H1 as well as in B1.

A.2.Effect of Addition of Alkanols:

In order to investigate the mobility of the metal cations on the silica layer by using a mobile phase system containing 3% aq.BAC and -alcohol (methanol, ethanol, butanol and propanol) mixture at pH 3.5 was also used. A better chromatographic performance was observed as a result, in terms of the differential migration of metal ions in their mobile phases over an aqueous mobile phase system. We used different ratio of concentrations of 3% aq. BAC surfactant to alcohols (9:1, 8:2, 7:3) (V/V) at pH 3.5.

A.2.1 Methanol system [M1-M3]:

Here we used different ratios of concentration of 3% aq. BAC to methanol [9:1, 8:2 & 7:3, V/V] at pH 3.5. The R_F values of the metal cations obtained under these varying concentrations are listed in table 3.

Table 3. R_F values of metal cations in 3% BAC with methanol addition system at 3.5pH

METAL	M1	M2	M3	METAL	M1	M2	M3
Cu^{2+}	0.11	0.11	0.08	Co^{2+}	0.33	0.3	0.29
Zn^{2+}	0.14	0.15	0.16	Hg^{2+}	0.7	0.77	0.76
Pb^{2+}	0	0	0	Ag^+	0	0	0
Fe^{3+}	0.06	0.06	0.11	Zr^{4+}	0.24	0.6	0.3
Ni^{2+}	0.34	0.3	0.22	V^{5+}	0.28	0.32	0.41
Sb^{3+}	0.77	0.79	0.75	Cd^{2+}	0.23	0.27	0.3
As^{3+}	0	0	0	Bi^{3+}	0.33	0.33	0.33
U^{+6}	0.1	0.08	0.08	W^{6+}	0	0	0
Th^{4+}	0	0	0	Mo^{6+}	0	0	0

From the data shown in Table 3, following conclusion may be drawn:

1. Higher mobilities of Sb^{3+} ($R_F \approx 0.79$) and Hg^{2+} ($R_F \approx 0.76$), but lower mobilities of Cu^{2+} , Zn^{2+} , Fe^{3+} and U^{6+} were observed. The same mobilities of Cu^{2+} , Zn^{2+} , U^{6+} , Co^{2+} , Hg^{2+} and Bi^{3+} were observed for all concentration of methanol. Intermediate mobilities were observed for V^{5+} and Zr^{4+} were observed.
2. Good separations were observed of $Cu^{2+} - Co^{2+}$ and $Zn^{2+} - Cd^{2+}$ from their binary mixtures in (at a ratio of 8:2, 3% BAC: methanol) at pH 3.5.
3. In all the systems (M1-M3), Pb^{2+} , As^{3+} , W^{6+} , Mo^{6+} , Ag^+ and Th^{4+} showed no mobility.

A.2.2.Ethanol system [M4-M6]:

Here we used different ratios of concentration of 3% aq. BAC to ethanol [9:1, 8:2 & 7:3, V/V] at pH 3.5. The R_F values of the metal cations obtained under these varying concentrations are listed in table 4.

Table 4. R_F values of metal cations in 3% BAC with ethanol addition system at 3.5pH

METAL	M4	M5	M6	METAL	M4	M5	M6
Cu ²⁺	0.15	0.27	0.22	Co ²⁺	0.53	0.36	0.35
Zn ²⁺	0.22	0.35	0.22	Hg ²⁺	0.92	0.94	0.88
Pb ²⁺	0	0	0	Ag ⁺	0	0	0
Fe ³⁺	0.13	0.18	0.08	Zr ⁴⁺	0.31	0.33	0.41
Ni ²⁺	0.5	0.36	0.35	V ⁵⁺	0.22	0.3	0.41
Sb ³⁺	0.98	0.88	0.88	Cd ²⁺	0.33	0.24	0.27
As ³⁺	0	0	0	Bi ³⁺	0.35	0.46	0.51
U ⁺⁶	0.07	0.1	0.16	W ⁶⁺	0	0	0
Th ⁴⁺	0	0	0	Mo ⁶⁺	0	0	0

From the data shown in Table 4, following conclusion may be drawn:

1. The same mobilities of Cu²⁺, Zn²⁺, Fe³⁺, Hg²⁺ and Cd²⁺ were observed for all concentration of ethanol. Higher mobilities of Sb³⁺ ($R_F \approx 0.98$ in 9:1) and Hg²⁺ ($R_F \approx 0.92$ in 9:1 and 0.94 in 8:2) were observed.
2. For some metal ions such as, Co²⁺, Zr⁴⁺, V⁵⁺ and Bi³⁺, mobility increases with increasing concentration of ethanol in aq. 3% BAC at pH 3.5.
3. Cd²⁺ and Sb³⁺, mobility decreased with increasing concentration of ethanol in aq. 3% BAC at pH 3.5.

A.2.3.Propanol system [M7-M8]:

Here we used different ratios of concentration of 3% aq. BAC to propanol [9:1 & 8:2 V/V] at pH 3.5. The R_F values of the metal cations obtained under these varying concentrations are listed in table 5.

Table 5. R_F values of metal cations in 3% BAC with propanol addition system at 3.5pH

METAL	M7	M8	METAL	M7	M8
Cu ²⁺	0.22	0.48	Co ²⁺	0.71	0.75
Zn ²⁺	0.33	0.57	Hg ²⁺	0.9	0.88
Pb ²⁺	0	0	Ag ⁺	0	0
Fe ³⁺	0.07	0.07	Zr ⁴⁺	0.15	0.34
Ni ²⁺	0.69	0.6	V ⁵⁺	0.15	0.23
Sb ³⁺	0.61	0.4	Cd ²⁺	0.86	0.87
As ³⁺	0	0	Bi ³⁺	0.42	0.45
U ⁺⁶	0.11	0.16	W ⁶⁺	0	0
Th ⁴⁺	0	0	Mo ⁶⁺	0	0

From the data shown in Table 5, following conclusion may be drawn:

1. For some metal ions such as, Cu^{2+} , Zn^{2+} , Zr^{4+} , V^{5+} and Bi^{3+} mobilities increased with the increasing concentration of propanol in 3% aq. BAC at pH3.5.
2. Higher mobility of Cd^{2+} ($R_F \approx 0.86$ in M7 and $R_F \approx 0.87$ in M8); Hg^{2+} ($R_F \approx 0.9$ in M7 and $R_F \approx 0.88$ in M8) were obtained. Mid mobilities of Ni^{2+} ($R_F \approx 0.69$ and 0.6 in M7 and in M8 resp.) were observed.
3. Middle mobilities were obtained in Ni^{2+} in both M7 and M8.

A.2.3. Butanol system [M9-M10]:

Here we used different ratios of concentration of 3% aq. BAC to butanol [9:1 & 8:2 V/V] at pH 3.5. The R_F values of the metal cations obtained under these varying concentrations are listed in table 6.

Table 6. R_F values of metal cations in 3% BAC with butanol addition system at 3.5pH

METAL	M7	M8	METAL	M7	M8
Cu^{2+}	0.11	0.28	Co^{2+}	0.41	0.25
Zn^{2+}	0.17	0.14	Hg^{2+}	0.86	0.9
Pb^{2+}	0	0	Ag^+	0	0
Fe^{3+}	0.05	0.14	Zr^{4+}	0.22	0.58
Ni^{2+}	0.38	0.25	V^{5+}	0.32	0.5
Sb^{3+}	0.35	0.5	Cd^{2+}	0.24	0.31
As^{3+}	0	0	Bi^{3+}	0.4	0.66
U^{6+}	0.08	0.24	W^{6+}	0	0
Th^{4+}	0	0	Mo^{6+}	0	0

From the data shown in Table 6, following conclusion may be drawn:

1. Maximum time required for separation of all metal cations as compared to other mobile phase system.
2. Increasing mobility were observed in Cu^{2+} , Fe^{3+} , Zr^{4+} , V^{5+} , Sb^{3+} , U^{6+} , V^{5+} , Cd^{2+} and Bi^{3+} with increasing concentration of butanol..
3. No mobility were observed in case of Pb^{2+} , As^{3+} , W^{6+} , Mo^{6+} , Ag^+ and Th^{4+} in all alcohol added mobile phase system.

From all Table 2-6 it was seen that, there were general trend of decreasing mobilities in some metal ions in alcohol concentration. The retention mechanism is connected with the adsorption of surfactant on the surface of the stationary phase. As long as the surface of the silica gel layer is saturated by the adsorbed surfactant that is there are no free silanol groups on the layer for the interaction, metal ions move faster showing high R_F value. The addition of alcohol to the mobile phase leads to the reduction in the adsorbed amount of surfactant on the stationary phase which causes an increasing in retention of solute and change in selectivity. Thus in presence of alcohol the silica layers are not completely saturated by adsorbed surfactant and there are free silanol groups, the interaction between them and the metal cations affects the retention pattern. In these cases the nature and the polarity of alcohol play an important role in the separation.

A.3. Effect of Addition of Electrolyte:

In order to investigate the mobility of the metal cations on the silica layer by using a mobile phase system containing BAC, a-water and –a weak and strong electrolyte (acetic acid and NaCl resp.) mixture at pH 3.5 was also used. A better chromatographic performance was observed as a result,

in terms of the differential migration of metal ions in their mobile phases over an aqueous mobile phase system. We used different concentrations of electrolytes in a 3% aq. BAC surfactant at pH 3.5.

A.3.1. Weak Electrolyte Acetic acid addition system [M11-M12]:

The effect of the addition of acetic acid to a 3% BAC mobile system at pH 3.5 was also investigated in the current study. The separation of various metal cations was carried out at various concentrations in 3% BAC at pH 3.5, specifically, 1ml and 3ml acetic acid in 100ml of 3% BAC at pH 3.5 (M11-M12). The R_F values for the various metal cations are listed in Table 7.

Table 7. R_F values of metal cations in 3% BAC with acetic acid addition system at 3.5pH

METAL	M11	M12	METAL	M11	M12
Cu ²⁺	0.86	0.92	Co ²⁺	0.86	0.92
Zn ²⁺	0.86	0.86	Hg ²⁺	0.97	0.95
Pb ²⁺	0	0	Ag ⁺	0	0
Fe ³⁺	0.19	0.28	Zr ⁴⁺	0.22	0.22
Ni ²⁺	0.86	0.92	V ⁵⁺	0.3	0.3
Sb ³⁺	0.6	0.69	Cd ²⁺	0.87	0.88
As ³⁺	0	0	Bi ³⁺	0.66	0.65
U ⁶⁺	0.51	0.59	W ⁶⁺	0	0
Th ⁴⁺	0	0	Mo ⁶⁺	0	0

From the data shown in Table 7 the following conclusions may be drawn:

1. Very good results were obtained for the mobile phase of acetic acid in 3% BAC compared with that of pure water.
2. Higher mobilities were obtained for the Cu²⁺, Zn²⁺, Ni²⁺, Co²⁺, Hg²⁺, Cd²⁺, Bi³⁺, U⁶⁺ and Sb³⁺ metal cations for the mobile phase of acetic acid.
3. For some metal cations, such as Cu²⁺, Zn²⁺, Fe³⁺, Ni²⁺, Sb³⁺, U⁶⁺ and Co²⁺, mobility increased with the increasing concentration of acetic acid in 3% BAC at pH 3.5. Higher mobilities of Cu²⁺, Zn²⁺, Fe³⁺, Ni²⁺, Sb³⁺, U⁶⁺, Co²⁺ and Cd²⁺ were observed in M11 and M12 compared to a mobile system of pure water; whereas a lower mobility of Zr⁴⁺ was detected in M11 and M12 as compared to that found for a mobile phase of pure water. The results were obtained relatively quickly.
4. The mobilities of the metals Ni²⁺, Sb³⁺, Hg²⁺, Cd²⁺ and Bi³⁺, remained the same in both the mobile phases of acetic acid and pure water.

A.3.2. Strong Electrolyte NaCl addition system [M13-M14]:

The effect of the addition of NaCl to a 3% BAC mobile system at pH 3.5 was also investigated in the current study. The separation of various metal cations was carried out at various concentrations in 3% BAC at pH 3.5, specifically, 1gm and 3gm NaCl in 100ml of 3% BAC at pH 3.5 (M13-M14). The R_F values for the various metal cations are listed in Table 8.

From the data shown in Table 8 the following conclusions may be drawn:

1. Cd²⁺ and Hg²⁺ show maximum mobility in M14 mobile phase.
2. For some metal cations such as Cu²⁺, Pb²⁺, Fe³⁺, Ni²⁺, Sb³⁺, U⁶⁺, Co²⁺, V⁵⁺ nearly same mobility obtained in all NaCl added mobile phase systems.
3. As³⁺, Th⁴⁺, Ag⁺, W⁶⁺ and Mo⁶⁺ did not show any mobility in all NaCl added mobile phase systems.

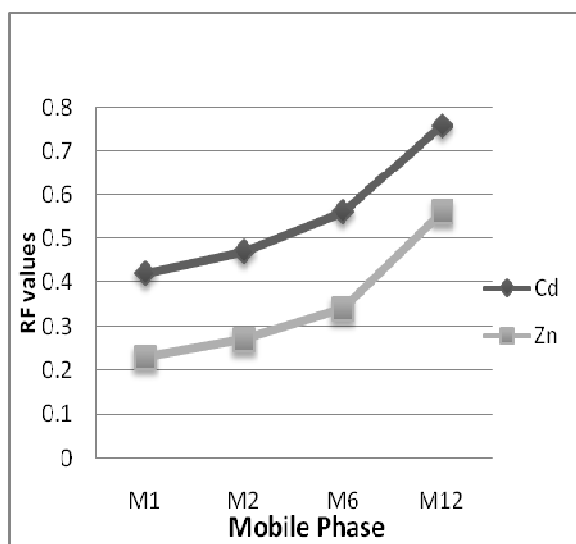
Table 8. R_F values of metal cations in 3% BAC with NaCl addition system at 3.5pH

METAL	M13	M14	METAL	M13	M14
Cu ²⁺	0.12	0.11	Co ²⁺	0.8	0.83
Zn ²⁺	0.14	0.05	Hg ²⁺	0.94	0.94
Pb ²⁺	0.01	0.01	Ag ⁺	0	0
Fe ³⁺	0.12	0.12	Zr ⁴⁺	0.37	0.23
Ni ²⁺	0.8	0.83	V ⁵⁺	0.35	0.31
Sb ³⁺	0.6	0.66	Cd ²⁺	0.72	0.93
As ³⁺	0	0	Bi ³⁺	0.51	0.46
U ⁶⁺	0.05	0.01	W ⁶⁺	0	0
Th ⁴⁺	0	0	Mo ⁶⁺	0	0

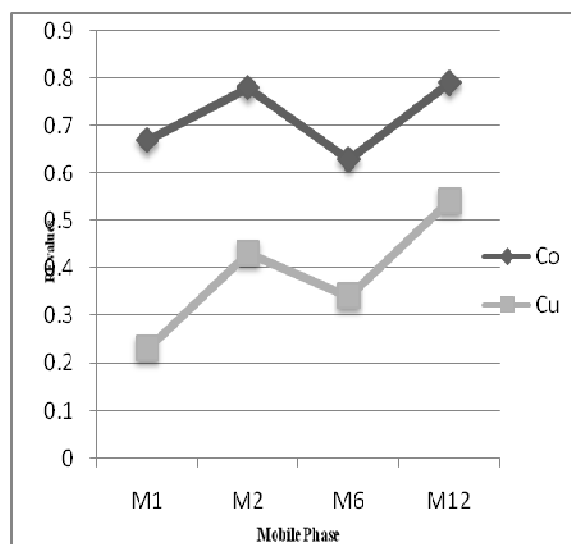
From all Table 7-8 it was seen that, in all acetic acid added mobile phase system, all metal cations shows good mobilities and compact spots were obtained as compared to strong electrolyte like NaCl added system. Some metal cations like Cu²⁺, Zn²⁺ and U⁶⁺ shows decreasing mobilities as soon there was addition of NaCl in 3% aq. BAC at 3.5pH. From these result it is clear that instead of strong electrolyte like NaCl, weak electrolyte like acetic acid is best additives in 3% aq. BAC mobile phase for separation of metal cations.

A. Binary mixture separations:

From all above conclusions, by using some mobile phase we were able to separate metal cations from there binary mixtures, in this section we were taken two binary mixtures that is Cd²⁺ - Zn²⁺ and Co²⁺ - Cu²⁺. Mobile systems M1, M2, M6 and M12 used for separation of this binary mixtures.



(1)



(2)

Fig. (1) and (2) Separation of cations from there binary mixtures

From Fig. 1 and 2 it was clear that by using 3% BAC at pH 3.5 with various additives we were able to separate various metal cations from there binary mixtures.

Applications

The proposed method was applied for identification of various heavy metal cations in industrial waste water, river and sea water samples as well as in metal sulphides ores after separation on silica layer. The result shown in Fig. 10 and 11 clearly demonstrated that Cd²⁺, Zn²⁺, Co²⁺ and Cu²⁺ can be separated on silica gel TLC plates developed with M1, M2, M6 and M12.

Acknowledgement

The authors would like to thank the principal and head of the Department of Chemistry, Hislop College Nagpur, and M.S. India for the provision of the research facilities used in our study.

REFERENCES

- [1].L.Deshmukh, R. B.Kharat, *Intern J. Environ, Anal Chem.***1988**, Vol.36, 1-6.
- [2].L. Deshmukh, R. B. Kharat; *Journal of Liquid Chromatography*, 1989, 12(6), 937-947.
- [3].D.Armstrong, R. Q. W. Terrill, *Anal. Chem.*, **1979**, 51, 2160-2163.
- [4].M. F. Borgerding, Williams (Jr), R. L.; Hinze, W. L.; Quina, *New J. Liq. Chromatography*, **1989**,12, 1367-1406.
- [5].A.S. Kord, M. G. Khaledi, *Anal.Chem.*,**1992**, 64,1901-1907.
- [6].A.Mohammad, Nahed Jabeen; *Acta. Chromatographia*, **2003**, 13, 135-153.
- [7].M. G.K haledi, J. K.Strasters, A.H. Rodgers, E. D. Breyer, *Anal. Chem.*, **1990**, 62,130-136.
- [8].A. Mohammad, V.J. Agrawal, *J. Quimica Analytica*, **2000**,254, 20-25.
- [9].A. Mohammad, Y. H. Sirwal, *J. Planar Chromatography-Mod.TLC*, **2002**,15, 107-115.
- [10]. A Mohammad, Eram Iraqi, and Iftkhar Alam Khan, *J. of Surfactant and Detergent*, **1999**, Vol.2; No.4, 523-529.
- [11]. A. Mohammad, V. Agrawal, Nahed Jabeen. *Chromatography*, **2003**, 24, 2 1-7.
- [12]. C. G.Yeole, V. M.Shinde, *Analyst*, **1983**,104, 1102-1107.
- [13]. T. Okada, *Anal Chem.*, **1988**, 60:2116.
- [14]. J. G.Dorsey, M.T.De Etchegaray, J.S.Landy, *Anal. Chem.*; **1983**,55, 924-928.