

ELECTROPHORETIC SEPARATION OF INORGANIC CATIONS IN BUFFER-ACETYLACETONE

ZAHID HUSSAIN CHOHAN, MUHAMMAD SHAHID GHOURI AND SHAIKAT ALI
Department of Chemistry, Islamia University, Bahawalpur, Pakistan

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Electrophoretic separation of 21 inorganic ions using buffer-acetylacetone system at various pH values [1-8] have been investigated. The results successfully favour the separation/identification of these cations at pH-3 and field strength 10V/cm.

Key words: Electrophoretic separation, Inorganic cation, Buffer-acetylacetone.

Introduction

Many research workers [1-5] have reported the electromigration and separation of metal ions in various electrolyte systems. But, no detailed studies have been made on the mobility and separation of different cations using complexing agent as apart of electrolyte system. Previously, we reported [6] the electromigration of about 20 cations using complexing agents ethylene-diamine (en), diethylamine (dea) and triethylamine (tea) in potassium phthalate buffer system. The present paper presents an account of the electromigration of cations in a mixture of complexing agent acetylacetone (acac) and buffer of various pH values.

Experimental

All the chemicals used were of analytical reagent grade. Whatman 1 (20x20 cm) chromatographic paper was used for the electromigration of cations. A Gelman delux electrophoretic chamber was used for all electrophoresis measurements. The constant voltage (200V) was supplied by a regulated power supply. Eil pH-meter was used for all pH measurements. A microlitre syringe (5 μ l) was used for sample spotting. Ultraviolet lamp was used for the detection of spots wherever required.

Buffer solution of various pH values was prepared by mixing potassium hydrogen phthalate and hydrogen chloride according to the reported method [7].

Sample solution. A solution (0.1M) of various metal ions was made by dissolving their respective chloride salts in distilled water. Antimony (III), bismuth (III), lead (II) and silver (I) were used as their nitrate salts.

Complexing agent. The complexing agent, acetylacetone, in an appropriate amount was added directly into the buffer solution of various pH values to obtain the concentration of 0.05, 0.1 and 0.2M.

General procedure. Buffer solution (400ml) was added in each compartment of electrophoretic chamber. Chromatographic sheet was firstly soaked in the buffer-complexing agent mixture. It was then loaded in the electrophoretic

chamber. The sample solution (1 μ l) was then applied on the sheet with the help of microlitre syringe. The chamber was covered and the constant voltage was supplied for appropriate time. After the requisite time the sheet was removed, dried, zones developed with the specific locating reagent and/or visualised under UV lamp. The colour of the spot was then compared with the known standards and the electrophoretic mobility (μ) was calculated by measuring the path length.

The above method was applied for the separation of cations in steel (alloy steel Mn 12%, C 1%) and Devards' alloy (Cu 50%, Al 45% and Zn 5%) samples. For this purpose steel (2g) and Devards' alloy (2g) were dissolved in conc HNO₃ (10 ml). The mixture was heated to dryness. The residue was cooled, dissolved in distilled water (20 ml), filtered and applied on the chromatographic sheet.

Results and Discussion

Electrophoresis, basically is a technique based on the differential migration of charged particles (cations or anions) in an electric field. Electrophoretic behaviour of cations on ordinary Whatman-1 filter paper using buffer-acetylacetone system have been explored in this work. We have previously reported [6] the use of mixture of buffer complexing agent rather than the use of a routine procedure of preparing complexes first and then subjecting the complexes to electrophoresis in some electrolyte system. Since, the behaviour of migration of cations and the stability of complexes is dependent upon the pH of the medium, hence, in this way we have minimized the expected complications.

In the present studies all experiments were performed at previously selected field strength i.e. 10V/cm. The buffer of pH in the the range 1-8 was employed for the preparation of electrolyte system. At pH-1 of the electrolyte system, electromigration was negligible probably, because, of higher ionic concentration. In the range of pH 2-8, the zones were of diversified shape. In the low pH electrolyte system, the shape of zones were nearly circular. It was found that with the increase in pH, the zones of most of cations elongated

probably, because of the adsorption of cation-acetylacetonate complexes on the filter paper. It was also observed that the slow release and a higher pH value resulted into tailing of the zones. The broadening/elongation of the zone of most of the cations was found to be maximum at pH-6 and above pH-6 the tailing predominates whereas below pH-6 zones become broad. At pH-3, zones of most of cations were found to be circular. Hence, all experiments were run at pH-3 and the results of separation obtained were reasonable. The results of electro-migration are given in Table 1. These values are the average distances (of three or four measurements) of the zone centres from the starting point.

The concentration of acetylacetonate (0.05, 0.1 and 0.2M) prepared in the buffer of appropriate pH was used during the present studies. The concentration of acetylacetonate effects the electromigration of cations as is evident from the data of Table 2. However, the concentration 0.1M at pH-3 has been found to be the most suitable concentration. Most of the zones found at this concentration were of regular shape whereas, at concentration 0.05 and 0.2M, the zones of majority of cations were either broad/elongated or of irregular shape. The critical observations in these studies further lead us to the conclusion that the mixture of inorganic cations can be successfully separated by this method of electrophoresis.

TABLE 1. ELECTROPHORETIC MOBILITY ($\mu \times 10^5$ (cm²V⁻¹S⁻¹) OF CATIONS AT FIELD STRENGTH (10V/cm), TIME (1hr) AND VOL. OF ACETYLACETONE (10ml/lit).

Cations	pH-2	pH-3	pH-4	pH-5	pH-6	pH-7	pH-8
Ag(I)	7.77	10.0	10.55	4.16	4.72	3.88	0.00
Pb(II)	11.16	14.16	7.16	9.16	8.88	7.77	11.11
Hg(II)	6.66	7.50	7.77	8.05	10.55	7.77	8.33
Bi(III)	16.90	15.55	5.20	11.11	11.11	6.11	8.88
Cu(II)	21.10	11.10	3.40	5.55	7.22	6.66	7.22
Cd(II)	18.33	16.10	15.00	10.55	11.38	4.16	5.00
Sb(III)	13.05	12.77	10.00	9.16	8.16	2.77	2.77
Sn(IV)	10.80	1.60	1.60	1.38	0.00	0.00	0.00
Fe(III)	16.66	10.50	7.79	8.88	6.11	6.94	5.00
Cr(III)	15.00	15.50	17.22	12.77	6.11	16.11	3.60
Al(III)	21.60	18.30	18.33	13.37	3.88	2.50	0.00
Zn(II)	21.94	18.88	17.77	13.33	7.50	4.72	3.61
Ni(II)	20.80	18.05	17.50	14.44	14.16	13.33	10.50
Co(II)	20.50	18.08	18.05	14.16	15.00	14.44	10.83
Mn(II)	21.10	19.16	20.80	14.44	15.00	9.44	4.16
Ca(II)	10.00	9.72	9.16	10.55	7.30	0.00	11.11
Ba(II)	20.00	9.72	11.66	9.44	13.61	0.00	0.00
Sr(II)	15.00	12.22	8.88	10.27	23.61	15.00	4.72
Na(I)	11.60	8.33	8.88	10.27	10.00	11.11	4.72
NH ₄ (I)	12.70	0.00	7.77	10.55	23.88	12.50	10.83
Mg(II)	13.30	0.00	0.00	0.00	0.00	0.00	13.61

Using the same optimum conditions some of the mixture of cations have been successfully separated and identified (Table 3). Furthermore, no attempt was made to analyse the electrolyte system after carrying out the experiments. As we have discussed earlier [6] that the main complication experienced in these studies was the location/detection of the zones which could be overcome by the selection of suitable locating procedures. As the work is in progress we hope that we shall be able to report such procedures shortly.

TABLE 2. ELECTROPHORETIC MOBILITY OF CATIONS IN DIFFERENT CONCENTRATION OF ACETYLACETONE AT pH-3.

Cations	Electrophoretic mobility ($\mu \times 10^5$) (cm ² V ⁻¹ S ⁻¹)		
	0.05 ml/lit.	0.1 ml/lit.	0.2 ml/lit.
Ag(I)	0.00	10.00	0.00
Pb(II)	10.27	14.16	0.00
Hg(II)	11.66	7.50	12.70
Bi(III)	0.00	15.55	8.37
Cu(II)	16.10	11.10	11.38
Cd(II)	17.77	16.10	17.50
Sb(III)	25.20	12.77	16.60
Sn(IV)	0.00	1.60	0.00
Fe(II)	12.22	10.50	10.00
Cr(III)	25.00	15.55	24.40
Al(III)	25.50	18.30	25.50
Zn(II)	25.20	18.88	24.10
Ni(II)	25.00	18.05	23.30
Co(II)	25.00	18.08	25.00
Mn(II)	25.50	19.16	25.20
Ca(II)	29.80	9.72	10.50
Ba(II)	7.77	9.72	10.50
Sr(II)	32.50	12.22	35.00
Na(I)	6.66	8.33	7.50
NH ₄ (I)	9.44	0.00	8.80
Mg(II)	26.90	0.00	11.00

TABLE 3. SEPARATION OF MIXTURE OF CATIONS AT OPTIMUM CONDITIONS OF ACETYLACETONE (0.1M), BUFFER pH-3 (POTASSIUM HYDROGEN PHTHALATE + HCl), FIELD STRENGTH (10V/cm) AND TIME (1 hr).

1	Sn	Hg	Pb
2	Fe	Mn	Pb
3	Cu	Cd	Sn
4	Fe	Cr	Mg
5	Fe	Ni	Cu
6	Zn	Cu	Hg
7	Cu	Al	Sn

References

1. A.K. Sen, S.B. Das and U.Ch. Ghosh, J. Ind. Chem. Soc., LXIV, 354 (1987).
2. M. Qureshi and A.H. Israili, Anal. Chim. Acta., 41, 523 (1968).

3. M. Qureshi, K.G. Vershney and F. Khan, *Sep. Sci.*, **6**, 559 (1971).
4. A.K. Sen, U. Ch. Ghosh and R.K. Chatuary, *Chromatographia*, **12**, 237 (1979).
5. D. Gross, *J. Chromatog.*, **10**, 221 (1963).
6. M.S. Ghouri, Z.H. Chohan and A. Sammee, *J. Ind. Chem. Soc.* (In process).
7. L. Meites, *A Handbook of Analytical Chemistry* (McGraw Hill Book Comp., New York, 1963), 1st ed.