Asian Journal of Chemistry; Vol. 23, No. 1 (2011), 23-25

ASIAN JOURNAL OF CHEMISTRY

www.asianjournalofchemistry.co.in

# Reversed Phase Paper Chromatographic Separation of Lead(II) and Bismuth(III)

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(*Received*: 27 January 2009; *Accepted*: 18 August 2010) AJC-8977

Reversed phase paper chromatographic separation of lead and bismuth have been carried out on Whatman no.1 filter paper impregnated with *n*-octylaniline as a stationary phase and using organic complexing agents such as sodium acetate, sodium malonate, sodium succinate, sodium citrate and sodium salicylate solutions as a mobile phase. Various experiments were carried out to study the effect of pH (4.0, 7.0 and 9.0), concentration range of mobile phase (0.01-0.1 M) and concentration of stationary phase (0.1-0.5 % using chloroform as a diluent) on the $R_f$ values of individual cations Optium concentration of *n*-octylaniline, mobile phase and pH were determined. The proposed method is applicable for separation and detection of lead and bismuth in various samples.

**Key Words: *n*-Octylaniline, N-*n*-Octylaniline, Lead, Bismuth.**

## INTRODUCTION

The reversed phase paper chromatography has become a popular separation technique due to simplicity and better resolution. Earlier, a number of studies have been reported on separation of different metal ions using various mobile and stationary phases[1-8]. The drawbacks like requirements of high concentration of mobile phase or developing solvent, long separation time, *etc*. The use of *n*-octylaniline as an extractant for some metal was also reported[9-16].

In this work, reversed phase paper chromatographic separation of lead and bismuth has been carried out. The effect of parameters *viz*., concentration of stationary and mobile phases and pH on $R_f$ values have been studied.

## EXPERIMENTAL

All solutions have been prepared from analytical reagents. Digital pH meter control dynamic was used for pH measurement and a reagents solution was equilibrated using griffin flask shaker. The stock solution of lead(II) was prepared by dissolving 3.99 g of A.R. grade lead nitrate in distilled water containing 0.5 mL of nitric acid and diluted to 100 mL using distilled water. The solution was standardized by known method[17]. The concentration of metal ion was found to be 2.50 mg/mL. The stock solution of bismuth(III) was prepared by dissolving 5.80 g of A.R. grade bismuth nitrate in distilled water containing 0.5 mL of nitric acid and diluted to 100 mL using distilled water. The solution was standardized by known method[17]. The concentration of metal ion was found to be 2.50 mg/mL. *n*-octylaniline and N-*n*-octylaniline was prepared according to the method given by Garlund *et al*[18].

Whatman paper no. 1 strips (15 × 4) were sprayed using *n*-octylaniline and N-*n*-octylaniline in chloroform which was equilibrated with weak acid having different concentration and pH. The desired metal ion solutions (150-250 μg) were spotted with microcapillaries. The spot was developed by ascending technique for 10 cm in glass jar (21.5 cm × 5.2 cm) containing aqueous solution of either sodium acetate, sodium malonate, sodium succinate, sodium citrate and sodium salicylate of required pH and required concentration. It requires 30-45 min development time. After development, paper was dried, cations were detected by spraying colour forming reagents and $R_f$ values were calculated for individual cations by usual procedure.

Lead(II) were detected as yellow colour spot with 0.2 % aqueous potassium chromate solution[17] and bismuth(III) were detected as brownish colour spot with 0.2 % aqueous solution of potassium iodide[17].

## RESULTS AND DISCUSSION

The experiments are carried out to study the effect of concentration of mobile phase (0.01-1.0 M) on values of individual cations. The best results are obtained with sodium salicylate (0.01 M at all pH), with sodium citrate (0.03-0.10 M), at pH 7.0, with sodium acetate (0.05-0.10 M at pH 7.0) with sodium succinate (0.05-0.10 M at pH 4.0) and with sodium malonate (0.05 M) at pH 4.0 using 0.1 % *n*-octylaniline for lead(II). N-*n*-octylaniline also tried as stationary phase for lead(II) gaves best results with sodium salicylate (0.01 M at pH 4.0 and 7.0, 0.07 M at all pH), sodium citrate (0.07 M at pH 7.0), sodium acetate (0.10 M at pH 7.0), sodium succinate (0.05 M at pH 4.0-9.0) and sodium malonate (0.03 M at pH 4.0-9.0).

The *n*-octylaniline concentration and N-*n*-octylaniline concentration are also varied from 0.1-1.0 % for lead(II) at different concentrations of sodium salicylate, sodium citrate, sodium acetate, sodium succinate and sodium malonate at pH 4.0, 7.0 and 9.0. Lead(II) has not extracted only at 0.3 % *n*-octylaniline, pH 4.0 when 0.07 M sodium citrate is used as mobile phase 0.01-0.03 M sodium malonate at pH 7.0, 0.05, 0.07 and 0.10 M sodium malonate at pH 4.0. Lead has not extracted at pH 7.0-9.0 at 0.05-0.10 M sodium citrate, respectively, pH 7.0 at 0.03 M sodium malonate with 0.3 % N-*n*-octylaniline. At all other ranges studied for weak acid, pH and reagent concentration lead(II) has been extracted (Table-1).

The experiments are also carried out to study the effect of mobile phase concentration *i.e.,* sodium salicylate, sodium citrate, sodium acetate, sodium succinate, sodium malonate (0.01-0.10 M) at pH (4.0, 7.0 and 9.0) and *n*-octylaniline, N-*n*-octylaniline concentration (0.1-1.0 %) on $R_f$ values of bismuth(III) (Table-2).

Bismuth(III) is not extracted when sodium citrate (0.05 M) as a mobile phase at pH 9.0 when 0.1 % N-*n*-octylaniline is stationary phase. 0.3 % N-*n*-octylaniline is used as stationary phase and 0.03 M sodium citrate is mobile phase. Bismuth(III) is not extracted at pH 4.0-9.0, 1.0 M sodium succinate at pH 9.0 also not gave extraction of bismuth(III) at 0.3 % N-*n*-octylaniline as a stationary phase. At all other conditions of

TABLE -1
EFFECT OF pH AND CONCENTRATION OF STATIONARY PHASE ON $R_f$ VALUES WITH DIFFERENT MOBILE PHASES

| Metal ion | Stationary phase | Conc. (M) | Mobile phase | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Na-acetate at pH | | | Na-malonate at pH | | | Na-salicylate at pH | | |
| | | | 4 | 7 | 9 | 4 | 7 | 9 | 4 | 7 | 9 |
| Pb(II) | *n*-OA 0.1 % | 0.01 | 0.79 | 0.78 | 0.77 | 0.64 | – | – | 0.70 | 0.63 | 0.68 |
| | | 0.03 | 0.79 | 0.81 | 0.81 | 0.49 | 0.27 | 0.25 | 0.59 | 0.65 | 0.62 |
| | | 0.05 | 0.88 | 0.87 | 0.84 | 0.74 | 0.22 | 0.28 | 0.54 | 0.55 | 0.53 |
| | | 0.07 | 0.82 | 0.85 | 0.87 | 0.10 | 0.10 | 0.13 | 0.52 | 0.56 | 0.56 |
| | | 0.10 | 0.81 | 0.88 | 0.86 | 0.17 | 0.13 | 0.13 | 0.49 | 0.47 | 0.50 |
| | *n*-OA 0.3 % | 0.01 | 0.77 | 0.74 | 0.75 | 0.74 | – | 0.12 | 0.68 | 0.60 | 0.63 |
| | | 0.03 | 0.74 | 0.76 | 0.72 | 0.69 | – | 0.67 | 0.58 | 0.50 | 0.51 |
| | | 0.05 | 0.78 | 0.80 | 0.81 | – | 0.10 | 0.14 | 0.47 | 0.39 | 0.30 |
| | | 0.07 | 0.82 | 0.83 | 0.81 | – | 0.10 | 0.09 | 0.50 | 0.39 | – |
| | | 0.10 | 0.83 | 0.86 | 0.94 | – | 0.07 | 0.05 | 0.40 | 0.36 | 0.38 |
| | *n*-OA 0.5 % | 0.01 | 0.77 | 0.81 | 0.57 | 0.77 | 0.45 | – | 0.36 | 0.23 | 0.32 |
| | | 0.03 | 0.72 | 0.81 | 0.60 | 0.56 | 0.40 | 0.30 | 0.43 | 0.36 | 0.32 |
| | | 0.05 | 0.74 | 0.69 | 0.38 | 0.63 | 0.47 | 0.44 | 0.43 | 0.27 | 0.25 |
| | | 0.07 | 0.72 | 0.64 | 0.64 | 0.16 | 0.40 | 0.42 | 0.32 | 0.25 | 0.29 |
| | | 0.10 | 0.81 | 0.68 | 0.24 | 0.68 | 0.45 | 0.36 | 0.31 | 0.27 | 0.22 |
| Bi(III) | *n*-OA 0.1 % | 0.01 | 0.79 | 0.80 | 0.79 | 0.80 | 0.80 | 0.80 | 0.76 | 0.80 | 0.75 |
| | | 0.03 | 0.84 | 0.81 | 0.85 | 0.82 | 0.84 | 0.81 | 0.74 | 0.78 | 0.80 |
| | | 0.05 | 0.91 | 0.91 | 0.85 | 0.73 | 0.80 | 0.73 | 0.75 | 0.73 | 0.82 |
| | | 0.07 | 0.73 | 0.79 | 0.78 | 0.88 | 0.77 | 0.83 | 0.80 | 0.80 | 0.73 |
| | | 0.10 | 0.84 | 0.85 | 0.78 | 0.75 | 0.70 | 0.80 | 0.80 | 0.73 | 0.74 |
| | *n*-OA 0.3 % | 0.01 | 0.87 | 0.85 | 0.82 | 0.84 | 0.81 | 0.62 | 0.67 | 0.74 | 0.68 |
| | | 0.03 | 0.82 | 0.80 | 0.80 | 0.80 | 0.83 | 0.78 | 0.77 | 0.80 | 0.82 |
| | | 0.05 | 0.82 | 0.78 | 0.75 | 0.78 | 0.69 | 0.68 | 0.74 | 0.66 | 0.72 |
| | | 0.07 | 0.90 | 0.83 | 0.81 | 0.85 | 0.78 | 0.78 | 0.73 | 0.65 | – |
| | | 0.10 | 0.85 | 0.80 | 0.83 | 0.80 | 0.66 | 0.82 | 0.68 | 0.61 | 0.38 |
| | *n*-OA 0.5 % | 0.01 | 0.62 | 0.66 | 0.50 | 0.36 | 0.42 | 0.37 | 0.76 | 0.73 | 0.69 |
| | | 0.03 | 0.53 | 0.53 | 0.63 | 0.51 | 0.65 | 0.60 | 0.68 | 0.60 | 0.60 |
| | | 0.05 | 0.71 | 0.34 | 0.51 | 0.61 | 0.64 | 0.59 | 0.71 | 0.76 | 0.73 |
| | | 0.07 | 0.65 | 0.51 | 0.50 | 0.61 | 0.59 | 0.59 | 0.74 | 0.73 | 0.73 |
| | | 0.10 | 0.45 | – | – | 0.73 | 0.61 | 0.61 | 0.68 | 0.66 | 0.70 |

TABLE-2
CONDITIONS FOR THE SEPARATION OF BINARY MIXTURES

| Mixture | Stationary phase | | Mobile phase | pH | Time required (min) | $R_f$ values | |
|---|---|---|---|---|---|---|---|
| | Reagent | % (V/V) | | | | | |
| Pb, Bi | n-OA | 0.5 | 0.01 M Sodium malonate | 4.0 | 45 | Pb(II) 0.77 | Bi(III) 0.36 |
| Pb, Bi | n-OA | 0.5 | 0.01 M Sodium salicylate | 7.0 | 45 | Pb(II) 0.23 | Bi(III) 0.73 |
| Pb, Bi | n-OA | 0.5 | 0.01 M Sodium citrate | 4.0 | 45 | Pb(II) 0.90 | Bi(III) 0.40 |
| Pb, Bi | N-n-OA | 0.5 | 0.01 M Sodium salicylate | 9.0 | 45 | Pb(II) 0.17 | Bi(III) 0.71 |
| Pb, Bi | N-n-OA | 0.5 | 0.10 M Sodium succinate | 4.0 | 45 | Pb(II) 0.89 | Bi(III) 0.29 |
| Pb, Bi | N-n-OA | 0.5 | 0.07 M Sodium malonate | 9.0 | 45 | Pb(II) 0.09 | Bi(III) 0.69 |

weak acids *n*-octylaniline concentration and N-*n*-octylaniline at pH 4.0, 7.0 and 9.0 extraction of bismuth(III) is satisfactory (Table-1).

## ACKNOWLEDGEMENTS

Thanks are due to U.G.C. for providing a Minor Research Project scheme to one of the authors (BKU). The authors are also thankful to Management, Pravara Rural Education Society and Principal, P.V.P. College, Pravaranagar for providing necessary facilities in department.

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