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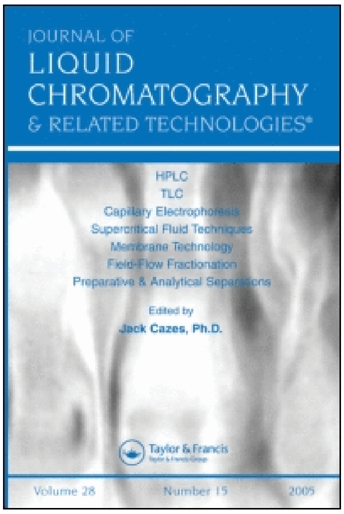

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Pritam Singh Thind[a]; Harbans Singh[a]
[a] Department of Chemistry, Guru Nanak Dev University, Amritsar, Pb, India

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STUDIES ON ENVIRONMENTAL POLLUTANTS: SELECTIVE SEPARATION AND RECOVERY OF Pb(II) ON FERRIC PHOSPHATE COLUMNS

Pritam Singh Thind and Harbans Singh

Department of Chemistry
Guru Nanak Dev University
Amritsar (Pb) India

ABSTRACT

A new method is described for the recovery and selective separation of Pb(II). The following mixtures have been separated: Hg(II)-Pb(II), ZrO(II)-Pb(II), Cd(II)-Pb(II), Cu(II)-Pb(II), Mn(II)-Pb(II), Zn(II)-Pb(II), Sr(II)-Pb(II) and Ni(II)-Pb(II), on ferric phosphate columns. Cu(II), Cd(II), Ni(II), Hg(II), Mn(II), Zn(II), Sr(II) and ZrO(II) were eluted with 0.01M-NH_4NO_3 and Pb(II) with a mixture of 0.1M-HNO_3 + 1.0M-NH_4NO_3 solution. Proposed studies can be applied to pollution analysis and alloy analysis.

INTRODUCTION

Lead poisoning has long been known and has been exhaustively studied. Although fatal cases of plumbism are now relatively rare, lead and its compounds still constituted one of the most important industrial hazards. Serious lead intoxication is most frequently encountered in persons who have inhaled the vapours, fumes or dust of lead and lead containing compounds. For example, the process of sanding and spraying lead paints are extremely hazardous with adequate protection. The allowable limits of lead in potable water (1) and air (2) as fixed by U.S. Public Health Service is 0.2-2 µg/l and 0.15 mg./cubic meter of air respectively. The recovery and selective separation of lead in water and in other various biological samples is necessary with the view point of pollution studies. In continuation of our work on analytical applications of ion exchange materials (3-5), the present communication extends our studies to the application of ferric phosphate, an inorganic ion exchanger, for the selective separation and recovery of lead.

EXPERIMENTAL

Apparatus:

A glass column (i.d. 1.3cm) was used for column operation. 1.0g. of ferric phosphate (H^+-form) was taken in the column and flow rate was initially maintained at 1.5ml/minute.

For pH measurements an ELICO pH meter model LI-10 was used.

Reagents and Chemicals:

Ferric nitrate (BDH) and ammonium dihydrogen phosphate (BDH) were used. All other chemicals were of analytical grade.

Synthesis of Ferric phosphate:

Ferric phosphate was prepared by adding 0.1M aqueous solution of ammonium dihydrogen phosphate to 0.1M-ferric nitrate solution as described earlier(4). The dried product was converted into the H^+-form by treating with 0.1M nitric acid for 24 h with ocassional shaking and intermittant changing of the acid. The product thus formed was dried at 40°C in a temperature controlled ovan.

Distribution Studies:

The distribution studies were carried out for 19 metal ions by batch process in the usual manner(4) after attaining equilibrium by shaking the metal ion solution with exchanger beads for 6 hours at room temperature($30^{+}-1^{\circ}C$). The distribution coefficients (K_d) for metal ions were calculated by using the following expression:

$$Kd = \frac{I-F}{F} \times \frac{20}{0.2}$$

where I is the volume of EDTA consumed by the original solution and F is the volume of EDTA consumed after equilibrium. The total volume of solution was 20 ml and the amount of exchanger used was 0.2g.

Elution behaviour:

From preliminary elution studies of Pb(II),Cu(II), Ni(II),Cd(II),Hg(II),Zn(II),Mn(II),Sr(II) and Zrc(II), it is observed that Pb(II) remains strongly adsorbed on ferric phosphate (H^{+}-form) bed in 0.01M-NH_4NO_3 where as Cu(II), Ni(II), Cd(II),Hg(II), Zn(II), Mn(II), Sr(II) and Zrc(II) percolate through the exchanger bed and quantitatively collected in the effluent. The flow rate is kept at 1.5ml/minute. After removal of Cu(II), Ni(II),Cd(II),Hg(II),Zn(II), Mn(II),Sr(II), and Zrc(II), Pb(II) can be eluted quantitatively with a mixture of 0.1M-HNO_3+1.0M-NH_4NO_3(1:1) solution.

Procedure:

The ion exchanger column (H^{+}-form) was washed with demineralized water before passing the test solution. A known volume of test solution(or synthetic mixture) was taken accurately and was recycled thrice through the exchanger bed at the flow rate of 1.5ml/minute. The column was then washed with 0.01M-NH_4NO_3. Cu(II),Ni(II),Cd(II),Hg(II),Zn(II),Mn(II), Sr(II) and Zrc(II) were percolated out with the effluent and washing liquid, the adsorbed Pb(II) was then eluted quantitatively with a mixture of 0.1M-HNO_3+ 1.0M-NH_4NO_3(1:1)solution.

RESULTS AND DISCUSSION

The distribution coefficients (K_d) of the cations are reported in TABLE-I. It is obvious from the results that the K_d values of Cu(II), Ni(II), Cd(II), Hg(II), Zn(II), Mn(II), Sr(II) and ZrO(II) are approximately ten times less than Pb(II) hence Pb(II) can be separated from these metals.

Recovery of Pb(II):

TABLE-II shows the successful recoveries of Pb(II) from solutions containing Cu(II), Ni(II), Cd(II), Hg(II), Zn(II), Mn(II), Sr(II) and ZrO(II). In the presence of five fold excess of these metal ions, all of them could be eluted with 0.01M-NH_4NO_3 except Pb(II). The retained Pb(II). The retained Pb(II) was easily eluted with a mixture of 0.1M-HNO_3 + 1.0M-NH_4NO_3(1:1) solution. This shows the importance of ferric phosphate in the recovery of Pb(II).

Column Separation:

Unlike Pb(II) which was retained by the exchanger, Cu(II), Ni(II), Cd(II), Hg(II), Zn(II), Mn(II), Mn(II), Sr(II) and ZrO(II) passed out unsorbed. Quantitative separations of these metal ions from Pb(II) have been successfully achieved. TABLE-III shows a list of separations successfully achieved on a column of ferric phosphate. The order and the eluents are presented in Figures 1-8.

The minimum number of theoretical plates (N) necessary for separation as tabulated in TABLE-III were calculated using equation

$$N \geqslant 2\pi \left[\frac{\dfrac{K_{d,Pb} + a}{K_{d,M} + a} + 1}{\dfrac{K_{d,Pb} + a}{K_{d,M} + a} - 1} \right]^2$$

where $K_{d,Pb}$ and $K_{d,M}$ are distribution coefficients of lead

TABLE-I

Distribution coefficients of Metal Ions on Ferric Phosphate in Water (pH ≅ 5-6)

Metal ions	K_d	Metal ions	K_d	Metal ions	K_d
Mg(II)	18.2	Ca(II)	14.3	Cd(II)	12.5
Zn(II)	11.3	Sr(II)	9.0	Hg(II)	75.0
Mn(II)	9.8	Ba(II)	20.8	Nd(III)	46.4
Ni(II)	15.5	Cu(II)	6.6	Mo(II)	52.0
Co(II)	53.0	Pb(II)	130.0	Al(III)	100.0
ZrO(II)	9.8	La(III)	52.2	Cr(III)	54.6
Th(IV)	T.A.				

Note: T.A. = Totally Adsorbed.

TABLE-II

Recovery of Pb(II) From Solutions Containing 5,000µg Each of Cu(II), Ni(II), Cd(II), Hg(II), Zn(II), Mn(II), Sr(II) and ZrO(II)

Recovery of	Eluents	Volume of effluent (ml)	Amount of Pb(II) loaded (µg)	Recovered (µg)	Error%
Pb(II) from Impurities	Impurities-0.01M-NH_4NO_3	50	-	-	-
	Pb(II)-0.1M-HNO_3+1.0M-NH_4NO_3	10	1,000	1,000	0.0
Pb(II) from Impurities	Impurities-0.01M-NH_4NO_3	60	-	-	-
	Pb(II)-0.1M-HNO_3+1.0M-NH_4NO_3	45	10,000	10,000	0.0

TABLE III

Separation of Pb(II) From the Solutions Containing Cu(II), Ni(II) Cd(II) Mg(II), Zn(II) Mn(II), Sr(II) and ZrO(II) on Ferric Phosphate (H^+-Form) Columns.

Separation	Eluents	Vol. of Effluent (ml)	Amount of cation Loaded (μg)	Amount of cation Recovered (μg)	Error %	N^a	h^b
Cu(II)-Pb(II)	Cu(II)-0.01M-NH_4NO_3	35	5883	5851	-0.54	7.95	1.01
	Pb(II)-0.1M-HNO_3+1.0M-NH_4NO_3	50	7251	7251	0.0		
Ni(II)-Pb(II)	Ni(II)-0.01M-NH_4NO_3	20	2348	2348	0.0	10.20	1.01
	Pb(II)-0.1M-HNO_3+1.0M-NH_4NO_3	50	7251	7251	0.0		
Zn(II)-Pb(II)	Zn(II)-0.01M-NH_4NO_3	30	5393	5393	0.0	9.0	0.253
	Pb(II)-0.1M-HNO_3+1.0M-NH_4NO_3	50	7251	7251	0.0		
Cd(II)-Pb(II)	Cd(II)-0.01M-NH_4NO_3	25	4720	4777	+1.10	9.30	1.01
	Pb(II)-0.1M-HNO_3+1.0M-NH_4NO_3	50	7251	7251	0.0		
Mg(II)-Pb(II)	Mg(II)-0.01M-NH_4NO_3	25	10,932	10,932	0.0	86.0	1.01
	Pb(II)-0.1M-HNO_3+1.0M-NH_4NO_3	50	7251	7251	0.0		
Mn(II)-Pb(II)	Mn(II)-0.01M-NH_4NO_3	30	1705	1705	0.0	8.60	1.01
	Pb(II)-0.1M-HNO_3+1.0M-NH_4NO_3	50	7251	7251	0.0		
Sr(II)-Pb(II)	Sr(II)-0.01M-NH_4NO_3	25	4381	4381	0.0	8.30	1.01
	Pb(II)-0.1M-HNO_3+1.0M-NH_4NO_3	50	7251	7251	0.0		
ZrO(II)-Pb(II)	ZrO(II)-0.01M-NH_4NO_3	25	2144	2144	0.0	8.60	1.01
	Pb(II)-0.1M-HNO_3+1.0M-NH_4NO_3	50	7251	7251	0.0		

Note: a = Minimum number of Theoretical plates.
b = Height equivalent to one theoretical plate.

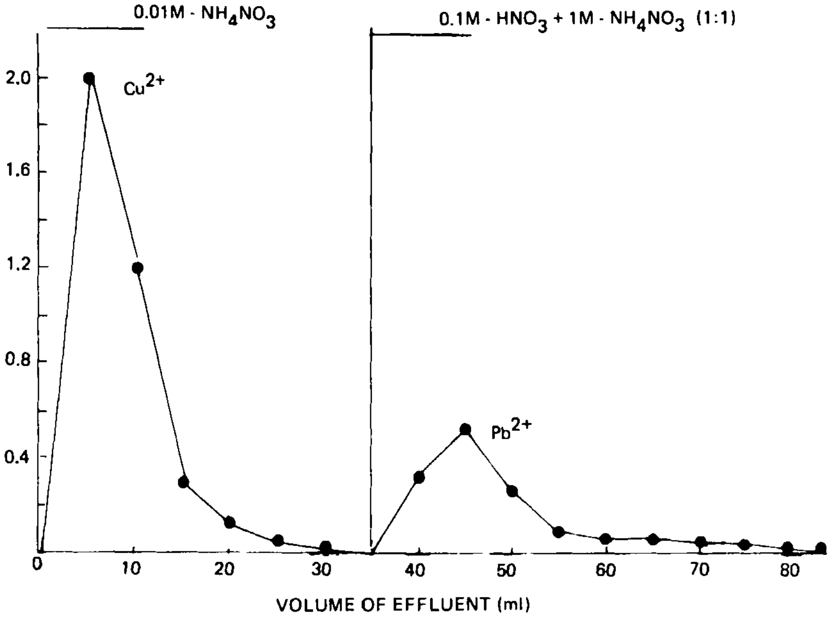

FIG. 1 SEPARATION OF Cu^{2+} FROM Pb^{2+}

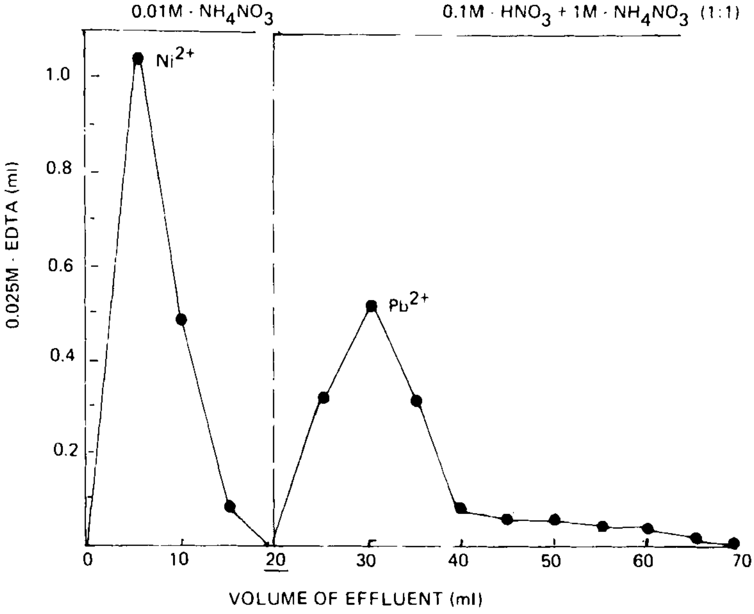

FIG. 2 SEPARATION OF Ni^{2+} FROM Pb^{2+}

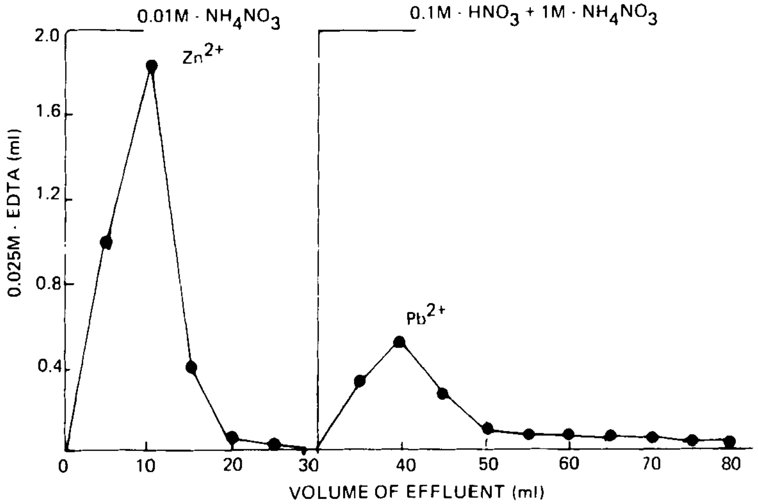

FIG. 3 SEPARATION OF Zn^{2+} FROM Pb^{2+}

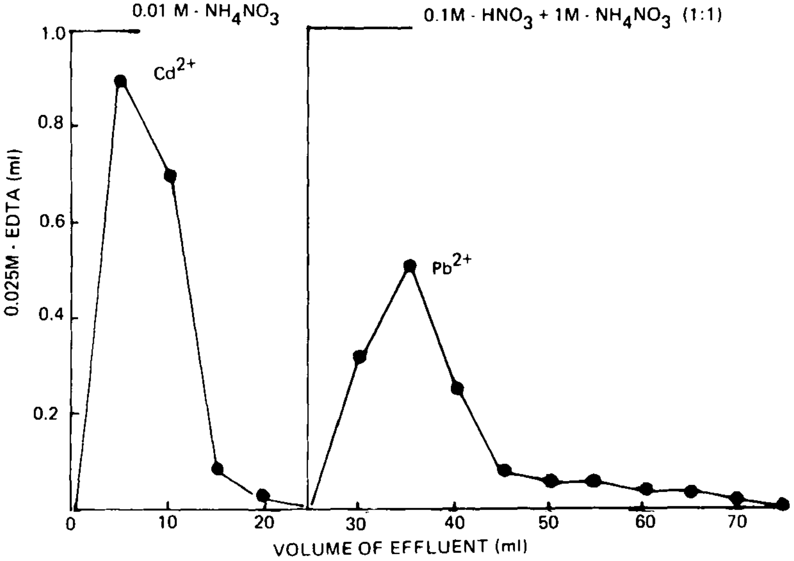

FIG. 4 SEPARATION OF Cd^{2+} FROM Pb^{2+}

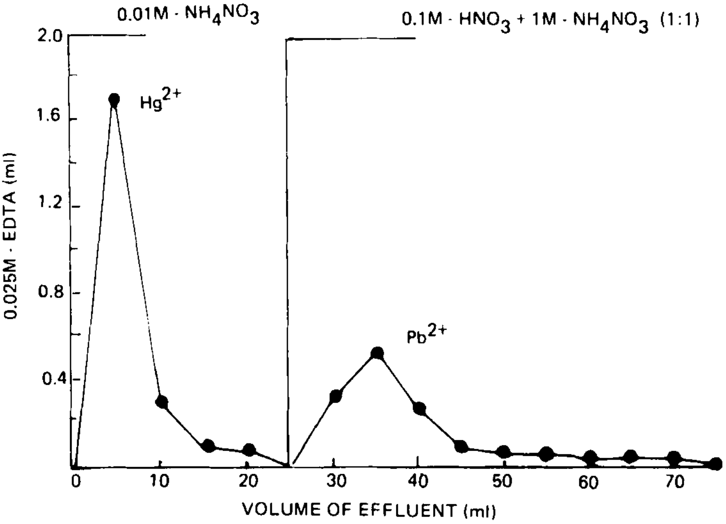

FIG. 5 SEPARATION OF Hg^{2+} FROM Pb^{2+}

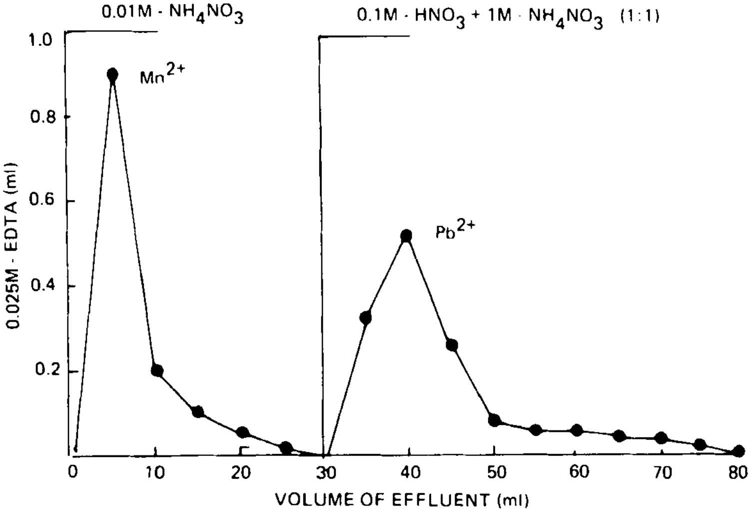

FIG. 6 SEPARATION OF Mn^{2+} FROM Pb^{2+}

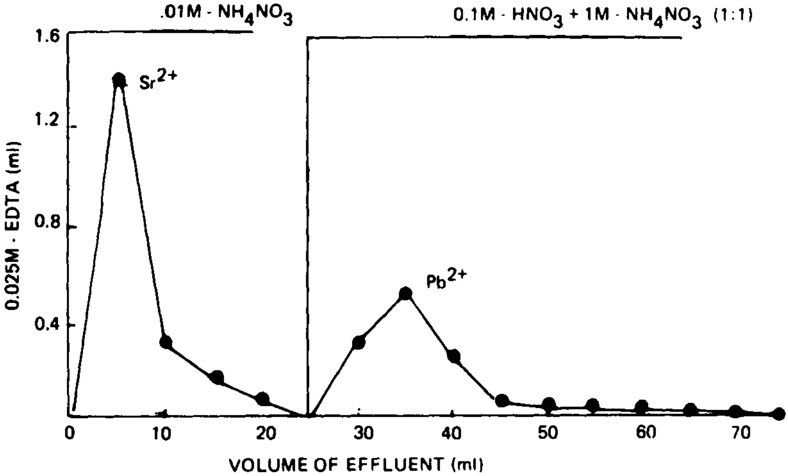

FIG. 7 SEPARATION OF Sr^{2+} FROM Pb^{2+}

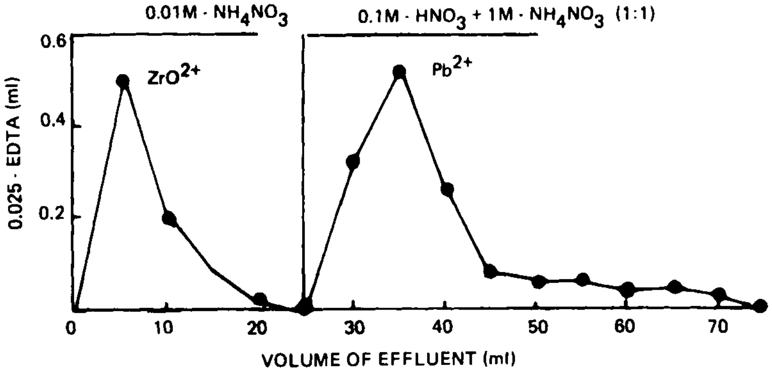

FIG. 8 SEPARATION OF ZrO^{2+} FROM Pb^{2+}

and the other metal separated respectively. a is the Void fraction (~0.4). The values of height equivalent to one theoretical plate (h) as reported in, TABLE-III, can be determined from the data obtained from an elution curve

$$h = \frac{L\,b^2}{8\,v_{max}^2}$$

where L is the length of the ion exchange column (cm), v_{max} is the eluent volume at peak (ml) and b is the peak width (ml) at a height of 0.368 C_{max}.

Application to Pollution Studies:

The main sources of lead pollution are the industries manufacturing storage batteries, cable covering, paints and gasoline. Becuase Hg(II), Cu(II), Zn(II), Cd(II) and Ni(II) that react with dilhizone are usually present in polluted samples. Hence, the proposed method of selective separation of Pb(II) followed by determination of lead by dilhizone is the better method compared to other time consuming methods.

Application of Analysis of non-ferrous Alloys:

Lead is present as a major constituent of the following alloys: white metal bearing alloys, solders, type metals, leaded brasses and bronzes, various zinc-base die casting alloys and many others. The method of decomposition applied depends, of course, on the composition of the alloy to be analyzed. For this reason, no universally applicable procedure can be cited. Usually gravimetric methods are used for the analysis of alloys but these methods are time consuming. The proposed method is simple and rapid for the estimation of lead in various alloys as these alloys contain Cu,Ni,Cd,Zn,Mn as main constituents that can be selectively separated from Pb(II).

This method can also be used for the concentration of traces of lead in food products e.g. tea, coffee, sugar and butter. The final determination is made with dilhizone spectrophotometrically.

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