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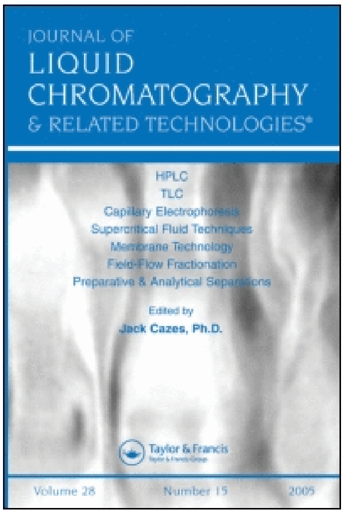

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Synthesis, Ion Exchange Properties and Applications of Thermally Stable Stannic Selenophosphate: Comparison With Other TIN(IV) Based Ion Exchangers

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SYNTHESIS, ION EXCHANGE PROPERTIES AND APPLICATIONS OF THERMALLY STABLE STANNIC SELENOPHOSPHATE: COMPARISON WITH OTHER TIN(IV) BASED ION EXCHANGERS.

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ABSTRACT

Samples of stannic selenophosphate have been synthesized by mixing 0.05m aquous solutions of stannic chloride, sodium selenite and sodium dihydrogen phosphate in different volume ratios at pH = 1. The chemical compositions have been determined. The Tin, Selenium and Phosphorous contents of all these samples have been found to be in the ratio of 1:1:1. Sample with a high ion exchange capacity (1.63 meq/ dry gm) has been studied in detail. pH titrations, IR and X-ray analysis, distribution coefficients of metal ions in different systems were performed. The differential selectivity of metal ions on stannic selenophosphate has been utilized for the important separations. Fourteen quantitative separations of industrial and analytical importance have been achieved on its column. A comparison of the important properties of stannic selenophosphate have been made with stannic tungstoarsenate, stannic molybdoarsenate, stannic arsenophosphate, stannic vanadophosphate, stannic phosphate and stannic selenite. It

was found that stannic selenophosphate is more chemically and thermally stable than other Tin(IV) based ion exchange materials.

INTRODUCTION

The importance of synthetic inorganic ion exchangers has now been well established (1). Besides their numerous analytical applications, they have been found useful in the fields of industry, medicine and biology. Simple salts of the metals which exhibit ion exchange properties have been much studied as compared to double salts. It has been found that heteropolyacid salts of some metals possess ion exchange properties different to the simple salts (2,3,4). The heteropolyacid salts usually show higher ion exchange capacities and are more thermally and chemically stable than simple salts. Stannic Phosphate (5) and Stannic Selenite (6) have been found to exhibit excellent ion exchange properties. We have therefore chosen to synthesize and investigate the properties of stannic selenophosphate as a new ion exchange materials. The results have been compared with stannic selenite, stannic phosphate and also with those of stannic tungstoarsenate (7), stannic molybdoarsenate (8) and stannic arsenophosphate (9).

EXPERIMENTAL

Reagents: Stannic chloride pentahydrate sodium selenite (B.D.H.) and sodium dihydrogen phosphate (E. Merk) were used; other chemical were of reagent grade.

Apparatus: The spectrophotometry, IR studies, pH measurements and the X-ray analysis were performed using Bausch and Lomb

Spectronic-20 spectrophotometer, Perkin-Elmer model 137 spectrophotometer, Elico pH meter model LI- 10 and Philips X-ray diffractometer respectively. A temperature - controlled shaker (SICO) was used for shaking.

Synthesis: Stannic selenophosphate samples were prepared by adding a mixture of aquous sodium dihydrogen phosphate and sodium selenite of 0.05M solutions each to 0.05M aquous solution of stannic chloride in different volume ratio as indicated in Table Ia. The pH was adjusted by adding dilute (5%) hydrochloric acid

Table-Ia Synthesis ion exchange capacity of stannic selenophosphate samples.

Sample	Conditions of synthesis				pH	ion exchange capacity m eq/ dry.g
	SnCl 4.5H$_2$O	Na$_2$SeO$_3$	NaH$_2$PO$_4$	mixing ration v/v		
SSP$_1$	0.05	0.05	0.05	1:1:1	1.0	1.26
SSP$_2$	0.05	0.05	0.05	1:1:2	1.0	1.63
SSP$_3$	0.05	0.05	0.05	1:2:1	1.0	1.28
SSP$_4$	0.05	0.05	0.05	2:1:1	1.0	1.04

Table-Ib Chemical composition of stannic selenophosphate samples.

	Composition			
Samples	Milli-moles of Sn	Milli-moles of Se	Milli-moles of P	Mole ratio of Sn : Se : P
SSP$_1$	1.858	1.9600	1.8380	1.01:1.07:1.0
SSP$_2$	1.937	2.0200	1.9900	1.0:1.05:1.03
SSP$_3$	1.896	2.0000	1.8700	1.01:1.07:1.0
SSP$_4$	-	-	-	-

solution or dilute sodium hydroxide. The gelatinous precipitate so formed, was allowed to settle down for 24 hrs, washed several times with demineralized water and filtered under suction. It was then dried at 40°C. This white hard substance when immersed in water, broke down into smaller particles with slight evolution of heat. To convert it into hydrogen form the material was kept for 2 days in one molar nitric acid solution. It was then washed with demineralized water to remove excess of acid. The conditions or preparation, ion exchange capacity and composition of various samples is reported in Table Ia and Ib.

Ion exchange Capacity: The ion exchange capacity of sample (SSP_2) with different metal ions was determined by column operation. The sample in hydrogen form was placed in the column with glass wool support. The hydrogen ions were eluted with one molar cation solution. The ions released from the exchanger were then determined by titrating the effluent with the standard solution of sodium hydroxide. The ion exchange capacities with different mono and bivalent ions are shown in Table II.

Chemical Stability: A 0.5 g of the material (sample SSP_2) was equilibrated with the 50 ml of the solvent concerned at room temperature (25 ± 2°C) and kept for 24 hrs with occasional shaking. Tin, selenium and phosphate released in the solvent were determined spectrophotometrically using haematoxylin, diaminobenzidine and ammonium molybdate respectively as a colouring reagents (10,11). The results are compared with those of stannic tungstoarsenate and stannic molybdoarsenate (7,8) in Table III.

COMPOSITION: A 0.2 g of the sample was dissolved in hot concentrated hydrochloric acid. The solution was cooled and

Table-II Ion exchange capacity (pH≈6) of stannic selenophosphate (sample SSP_2) at 25 ± 1°C.

Ion	Capacity, meq/g
Li^+	0.86
Na^+	1.34
K^+	1.63
Rb^+	0.86
Mg^{+2}	0.70
Ca^{+2}	0.87
Sr^{+2}	1.10
Ba^{+2}	0.92

selenium was estimated as selenium metal (11) after precipitation with sulphurdioxide solution. From the filtrate, the tin was precipitated using cupferron (11) as precipitating reagent and filtered. The precipitate was then ignited and tin was determined as tin oxide. Lastly the phosphate content in the filtrate was determined titrimetrically (11).

pH-titrations: pH titrations for LiCl - LiOH, NaCl - NaOH and KCl - KOH systems for sample SSP_2 were performed using Topp and Pepper's method (12). For each system, 0.5 g of the exchanger in H^+ form was equilibrated with 50 ml of the solution at room temperature. pH titration curves for these systems are shown in Fig. 1.

Table-III Chemical stability of some Tin(IV) based ion exchangers.

Solvent	Stannic selenophosphate (mg / 50 ml)			Stannic tungstoarsenate (mg / 50 ml			Stannic molybdoarsenate (mg / 50 ml)		
	Sn	Se	P	Sn	W	As	Sn	Mo	As
Water	0.00	0.00	0.00	0.65	1.00	1.80	0.00	6.60	3.80
0.1M Nitric Acid	0.00	0.00	0.00	-	-	-	-	-	-
1.0M Nitric Acid	0.22	0.00	0.00	-	-	-	-	-	-
2.0M Nitric Acid	2.20	1.02	0.00	-	-	-	-	-	-
4.0M Nitric Acid	-	-	-	8.00	0.60	1.00	3.60	20.00	17.50
0.1M Hydrochloric Acid	0.00	0.00	0.00	-	-	-	-	-	-
1.0M Hydrochloric Acid	2.42	1.24	0.80	-	-	-	-	-	-
2.0M Hydrochloric Acid	8.20	6.20	5.10	-	-	-	-	-	-
4.0M Hydrochloric Acid	-	-	-	145.00	11.00	47.50	Dissolved completly		
1.0M Sulphuric Acid	0.00	0.00	0.10	-	-	-	-	-	-
2.0M Sulphuric Acid	0.00	0.22	0.10	-	-	-	-	-	-
4.0M Sulphuric Acid	-	-	-	29.00	0.40	20.00	22.50	62.50	30.00
1.0M Acetic Acid	-	-	-	0.38	0.16	14.0	0.03	10.00	7.50

Thermal treatment: To examine the effect of drying temperature on the ion exchange capacity, the material was heated at different temperatures in a muffle furnace for one hr. The ion exchange capacities for K^+ ion at different temperatures are reported in Table IV.

Table-III (Contd.)

Solvent	Stannic selenophosphate (mg/50 ml)			Stannic tungstoarsenate (mg / 50 ml)			Stannic molybdoarsenate (mg / 50 ml)		
	Sn	Se	P	Sn	W	As	Sn	Mo	As
4.0M Acetic Acid	0.00	0.20	0.00	-	-	-	-	-	-
1.0M Formic Acid	-	-	-	0.38	0.60	5.40	0.25	30.00	8.75
4.0M Formic Acid	0.92	0.10	0.00	-	-	-	-	-	-
1.0M Tartaric Acid	-	-	-	47.50	0.20	0.20	8.75	30.00	15.00
2.0M Tartaric Acid	1.20	0.90	0.81	-	-	-	-	-	-
1.0M Citric Acid	-	-	-	32.50	0.60	0.40	2.88	30.00	8.75
2.0M Citric Acid	0.94	0.57	0.46	-	-	-	-	-	-
0.1M Sodium hydroxide	0.00	6.60	5.10	Dissolved completely					
4.0M Perchloric Acid	2.44	2.70	3.26	-	-	-	-	-	-

<u>IR-Spectrophotometry</u>: IR spectra of sample SSP_2 dried at different temperatures were obtained by potassium bromide disc method Fig. 2.

<u>X-ray diffraction analysis</u>: Nickel-filtered Cu-Kα radiation was used for X-ray diffraction analysis of the sample dried at 40°C.

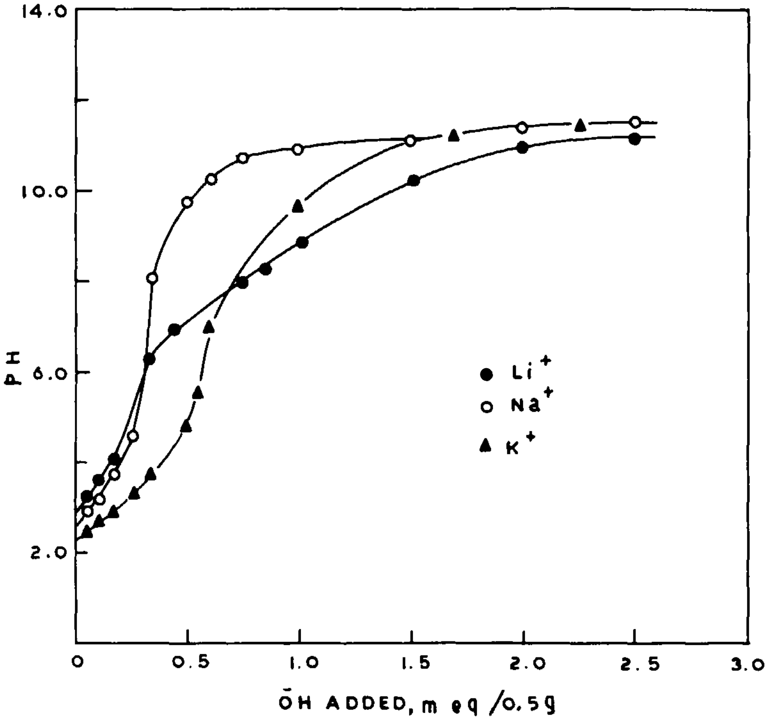

Fig. 1 PH titration curves of Stannicselenophosphate

Distribution Coefficients: Distribution coefficients for different metal ions were determined by a published method (13). The results are given in Table V. Kd values were also compared with some other Tin(IV) based ion exchangers (Table VI,VII).

Quantitative Separations: Important separations of metal ions were achieved on the columns of the exchanger. The column was prepared using 2.0 g of exchanger (150-200 mesh) in H^+ form in a glass tube of internal diameter of 0.6 cm in each case. A mixture of the metal ions solution was poured into the column. The

Table-IV Effect of drying temperatures on exchange capacity of various Tin(IV) based ion exchangers.

Drying temperature °C	Ion exchange capacity (m eq/dry grm)						
	SP	SS	SSP	STA	SMA	SAP	SVP
40	1.35	0.75	1.63	1.18	1.2	1.75	1.70
100	1.62	0.75	1.63	-	0.9	1.65	1.30
200	0.82	0.75	1.0	-	0.6	0.90	1.10
300	0.23	0.75	0.88	0.51	.51	-	0.50
400	0.02	0.75	0.70	-	.35	.20	-
500	-	0.70	0.64	0.31	-	-	-
600	-	0.20	0.38	-	0.28	0.10	-
700	-	-	-	-	0.04	-	-
800	-	-	0.2	0.04	-	0.00	-

SP = Stannic phosphate; SS = Stannic selenite;
SSP = Stannic selenophosphate; STA = Stannic tungstoarsenate;
SMA = Stannic molybdoarsenate; SAP = Stannic arsenophosphate;
SVP = Stannic vanadophosphate.

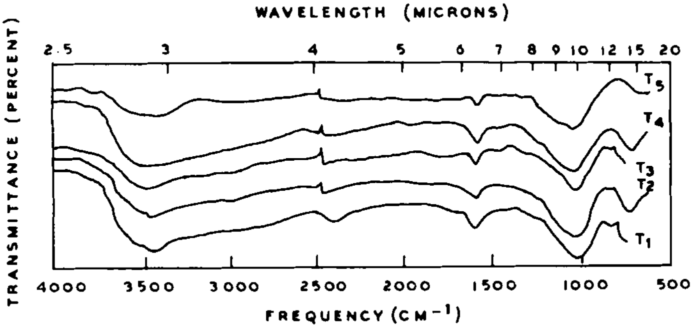

Fig.2 Infrared spectra of Stannic selenophosphate dried at different temperatures
T_1 40°C, T_2 40°C (K^+ FORM), T_3 300°C, T_4 500°C, T_5 800°C

Table-V Distribution coefficients for some cations on stannic selenophosphate (SSP_2) in different medium.

Metal ion	Kd (ml / g)		
	Water (pH = 5.8)	0.01M HNO_3	0.1M HNO_3
Cu^{+2}	940.00	197.00	4.00
Ni^{+2}	1020.00	7.66	0.97
Hg^{+2}	60.00	60.00	28.00
CO^{+2}	2900.00	103.00	37.50
Al^{+3}	2400.00	1566.00	280.00
Fe^{+3}	900.00	150.00	100.00
Pb^{+2}	8500.00	1128.00	105.00
Zn^{+2}	3360.00	73.30	1.20
Cd^{+2}	9900.00	138.00	94.60
Mg^{+2}	920.00	2.00	0.99
Ba^{+2}	1700.00	125.00	1.10
Ca^{+2}	920.00	27.50	1.00
Mn^{+2}	70.00	50.00	1.00
VO^{+2}	3900.00	300.00	14.300
Th^{+4}	4900.00	4750.00	4400.00
UO_2^{+2}	9980.00	9905.00	9900.00
Ce^{+4}	4900.00	1328.00	400.00
Zr^{+4}	9900.00	4070.00	1900.00
La^{+3}	12500.00	12500.00	3050.00
Nd^{+3}	3328.00	800.00	63.60
Pr^{+3}	2150.00	2150.00	1.1
HO^{+3}	1983.00	900.00	66.60
Dy^{+3}	6400.00	3150.00	170.80
Sm^{+3}	6420.00	1525.00	158.00

solution was allowed to flow down through the column. The column was rinsed with a little demineralized water. The metal ions adsorbed in the exchanger were then eluted with appropriate eluting reagent. The flow rate of the effluent was maintained one ml/min. throughout the elution process. The metal ions in 10 ml fractions

Table-VI Comparison of Kd values of metal ions on Tin(IV) based ion exchangers in water (pH≃6).

Metal ion	Kd (ml/ g)				
	SP	SS	SSP	SMA	SVP
Cu^{+2}	51870	C.A.	940	7500	550
Ni^{+2}		1900	1020	1050	60
Hg^{+2}		2260	60	-	-
CO^{+2}	96.70	610	2900	-	-
Al^{+3}		220	2400	740	0
Fe^{+3}	4207	110	900	150	0
Pb^{+2}		640	8500		50
Zn^{+2}	315	1070	3360	10650	1900
Cd^{+2}	-	3870	9900	2150	3900
Mg^{+2}	-	300	920	900	60
Ba^{+2}	-	C.A.	1700	2300	4500
Ca^{+2}	2247	3600	920	1080	300
Mn^{+2}	2090		70	10900	420
VO^{+2}	-	570	3900	-	-
Th^{+4}	-	2400	4900	-	-
UO_2^{+2}	-	C.A.	9980	-	-
Ce^{+4}	-		4900	-	-
Zr^{+4}	-	C.A.	9900	-	-
La^{+3}	-	C.A.	12500	11400	-
Nd^{+3}	-	C.A.	3328		-
Pr^{+3}	-	C.A.	2150	25100	-
HO^{+3}	-		1983	26400	-
Dy^{+3}	-		6400	5000	-
Sm^{+3}	-	C.A.	6420	1660	-

SP = Stannic phosphate; SSP = Stannic selenophosphate;
SMA = Stannic molybdoarsenate; SVP = Stannic vanadophosphate;
SS = Stannic selenite; C.A. = Complete adsorption.

Table-VII Comparison of Kd values of metal ions on Tin(IV) based exchangers in 0.C1M HNO_3 medium.

Metal ion	Kd values (ml/g)				
	SS	SSP	STA	SMA	SVP
Cu^{+2}	950	197	401	510	500
Ni^{+2}	540	7.7	68	80	60
Hg^{+2}	560	60			
CO^{+2}	160	103			
Al^{+3}	440	1566	0.0	240	0
Fe^{+3}	60	150	2.0	70	0
Pb^{+2}	220	1128			160
Zn^{+2}	510	73	0	140	70
Cd^{+2}	680	138	262	140	10
Mg^{+2}	110	2.C	9.0	110	20
Ba^{+2}	600	125	251	440	30
Ca^{+2}	520	27.5	60	110	30
Mn^{+2}		50	116	340	0
VO^{+2}	210	300			
Th^{+4}	960	4750			
UO_2^{+2}	3020	9905			
Ce^{+4}		1328			
Zr^{+4}	C.A.	4070			
La^{+3}	C.A.	12500	850	160	
Nd^{+3}	2250	800	225	800	
Pr^{+3}	3500	2150	408	1160	
HO^{+3}		900	5200	780	
Dy^{+3}		3150	507	1170	
Sm^{+3}	3840	1525	948	780	

of the effluent were collected and determined titrimetrically using 0.002M EDTA solution as a titrant. Fourteen separations of metal ions achieved, have been reported in Table IX. A model elution curve is also shown in Fig. 3.

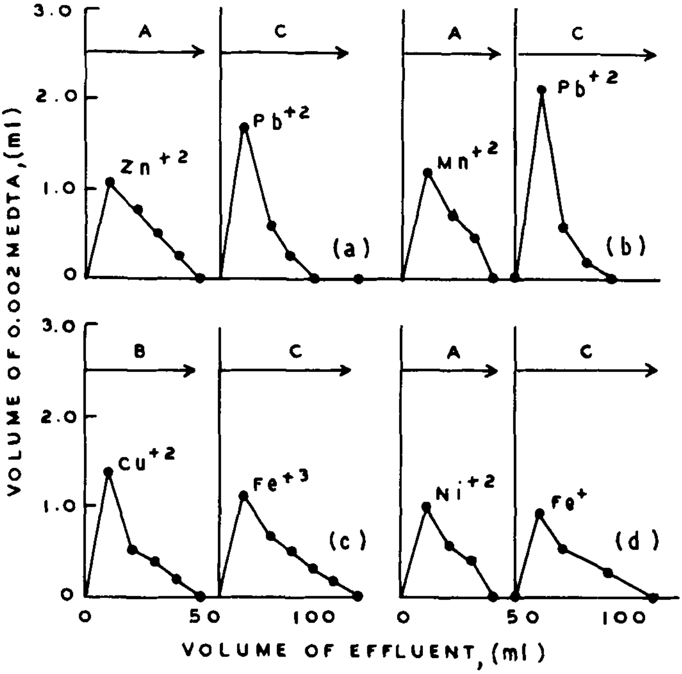

Fig. 3 Separation of (a) Zn^{+2}–Pb^{+2}, (b) Mn^{+2}–Pb^{+2} (c) Cu^{+2}–Fe^{+3} and (d) Ni^{+2}–Fe^{+3} On Stannic selenophosphate columns
Solvent used : A = 0.05M HNO_3 B = 0.1M HNO_3
C = 0.8M HNO_3

DISCUSSION

It is apparent from Tables Ia and Ib that the chemical composition of different samples of stannic selenophosphate prepared at pH = 1 is not altered by varying the mixing volume ratio of stannic chloride, sodium selenite and sodium dihydrogenphosphate solutions. During the preparation of stannic selenophosphate it has been observed that when a mixture of anions solution is added to the stannic chloride solution containing sufficient hydrochloric acid to maintain pH around 1, precipitate appears immediately which settles down and easily filterable. On the other hand when the stannic chloride is added to the anions solution, the precipitation occurs after sometime and do not settle even after keeping it for more than 24 hrs and is difficult to filter. It is found that the ion exchange capacity of the stannic selenophosphate (1.63 meq/g) is much higher as compared to stannic selenite (0.75 meq/g) and stannic phosphate (1.26 meq/g) as shown in Table VIII. It is interesting to note that the exchange capacity for alkali metals is greater than alkaline earths. The ion exchange capacity for alkali metals and also for alkaline earths increases in general with the decrease in hydrated ionic radii as expected.

Table-VIII Comparison of preparation and properties of stannic selenophosphate with other Tin(IV) based ion exchangers.

Ion exchange material	Reagents	mixing ratio	pH	Ion exchange capacity (meq/g)
Sn - Se - P	0.05M $SnCl_4.5H_2O$ + 0.05M Na_2SeO_3 + 0.05M NaH_2PO_4	1:1:2	1.0	1.63
Sn - Se	0.1M $SnCl_4.5H_2O$ + 0.1M Na_2SeO_3	1:1	1.0	0.75
Sn - P	210 gms of $SnCl_4.5H_2O$ + 187 gms $Na_2H_2PO_4$ + 96 gms NaOH	-	-	1.26
Sn - W - AS	0.25M $SnCl_4.5H_2O$ + Na_2WO_4 + 0.25M Na_2ASO_3	2:1:1	1.0	1.06

Sn-Se-P = Stannic selenophosphate; Sn-Se = Stannic selenite
Sn-P = Stannic phosphate; Sn-W-AS = Stannic tungstoarsenate.

A comparison of the ion exchange capacities at different drying temperature of various Tin(IV) based exchangers reveals many interesting features (Table IV). The exchange capacities of stannic selenophosphate, stannic arsenophosphate and stannic vanadophosphate (14) are much higher and the values lie very close with each other when the drying temperature is 40°C. A marked increase in the capacity was observed by Inoue (5) in the case of stannic phosphate when the drying temperature is raised to 100°C. While for other exchangers the capacity either remains the same or slightly decreases at this temperature. It is interesting to note that on increasing the drying temperature successively a significant loss in ion exchange capacity is observed in most of the cases. However, when the drying temperature is raised to 500°C, a remarkable loss in ion exchange capacity is noticed for all the materials except in the case of stannic selenite and stannic selenophosphate. Therefore, stannic selenite and stannic selenophosphate appears to be more thermally stable ion exchange materials. It is also clear from the data that stannic selenophosphate possesses considerable exchange capacity even when drying temperature goes beyond 600°C. The capacity further drops to a negligible value (0.2 meq/g) when the drying temperature is increased to 800°C, due to the formation at respective oxides.

The pH titration curves indicate that adsorption sequence for alkali metals in the acidic range (below pH 6) is $K^+ > Na^+ > Li^+$ and is in accordance with the size of hydrated ionic radii. The same trend is also confirmed by ion exchange capacity data (Table II). In the pH region 6-7.5 Li^+ is more strongly adsorbed than Na^+. If the pH is increased above 7.5, the adsorption sequence becomes $Li^+ > K^+ > Na^+$. This reversal of adsorption sequence of alkali metals on stannic selenophosphate resembles with that of Thorium tungstate and can be explained in the same manner (15).

Stannic selenophosphate is highly stable in water and acidic solutions. It is clear from the Table III that moderately concentrated nitric acid, sulphuric acid and acetic acid do not have considerable effect on the chemical stability of the material. However in concentrated hydrochloric acid and sodium hydroxide the exchanger dissolves significantly and the exchanger is completely soluble in higher concentration of HCl on prolonged heating. It is important to compare the chemical stability of stannic selenophosphate with other ion exchange materials in various solvents (Table III). The order of chemical stability in most of the solvent is stannic selenophosphate $>$ stannic tungstoarsenate $>$ stannic molybdoarsenate. It has also been inferred that stannic selenophosphate is more stable than simple salts, stannic selenite and stannic phosphate.

Stannic selenophosphate has very high affinity for a number of metal ions eg UO_2^{+2}, Th^{+4} and rare earths in water and acid media. The same trend is observed in the case of stannic selenite. A decrease in Kd values with the decrease in pH is noticed in almost all the cases as expected. The increased acid concentration however has pronounced effect on the Kd values for certain metal ions eg Ni^{+2}, Mg^{+2} and Zn^{+2} whereas in the case of UO_2^{+2}, Th^{+4}, Pb^{+2} and La^{+3}, the increased acid concentration do not have significant effect on Kd values since the uptake of these metal ions is very high even in 0.1M nitric acid. The selectivity order of metal ions for various Tin(IV) based ion exchangers in water appears to be as follows:

i) Stannic selenophosphate

$Cd^{+2} > Pb^{+2} > VO^{+2} > Zn^{+2} > Al^{+3} > CO^{+2} > Ni^{+2} > Cu^{+2} > Fe^{+3} > Mn^{+2} > Hg^{+2}$;

$Ba^{+2} > Ca^{+2} > Mg^{+2}$;

$La^{+3} > Sm^{+3} > Dy^{+3} > Nd^{+3} > Pr^{+3} > HO^{+3}$;

ii) Stannic molybdoarsenate

$Mn^{+2} > Zn^{+2} > Cu^{+2} > Cd^{+2} > Ni^{+2} > Fe^{+3} > Al^{+3}$;

$Ba^{+2} > Ca^{+2} > Mg^{+2}$;

$HO^{+3} > Pr^{+3} > La^{+3} > Dy^{+3} > Sm^{+3} > Nd^{+3}$;

iii) Stannic selenite

$Cu^{+2} > Zn^{+2} > CO^{+2} > Ni^{+2}$;

iv) Stannic phosphate

$Cu^{+2} > Fe^{+3} > Ca^{+2} > Mn^{+2} > Zn^{+2} > CO^{+2}$.

The differential selectivity of metal ions in acid solutions has made stannic selenophosphate a useful ion exchange material for many important separations. The practical utility of this material has been demonstrated by achieving a number of important binary separations of the metal ions on the columns of stannic selenophosphate (Table IX).

Separations of Zn from Pb may be utilized for the recovery of Zn from galvanizing wastes. Separation of CO from Fe and Ni from Fe can be used for the determination of Ni and CO in ferromagnetic alloys (CO-Fe and Ni-Fe). Similarly separation of Cu from Fe may also be useful for the estimation of Cu in copper or like chalcopyrite ($CuFeS_2$). The X-ray diffraction analysis however shows that material is amorphous.

The infrared spectra of stannic selenophosphate (Fig. 2) dried at various temperatures show three absorption peaks. A strong and broad peak in the region 3600-3200cm^{-1} presents the inter stitial water, free water and OH groups. Another sharp peak in the region 1700-1500cm^{-1} with a maximum at 1600cm^{-1} is characteristic of inter stitial water. The third peak in the region 1100-900cm^{-1} may be due to HPO_4^{-2} group (16). IR spectra of the samples in H^+ and K^+ forms show no difference at 40°C. It is important to note that the absorption

Table-IX Quantitative separations of metal ions on columns of stannic selenophosphate.

S.No.	Separation achieved	Amount taken (µg)	Amount found (µg)	Eluent	Total elution (ml)
1.	Zn^{+2}	326.5	339.5	0.05M HNO_3	40
	Pb^{+2}	2072.0	2113.4	0.80M HNO_3	50
2.	Mn^{+2}	264.0	269.0	0.05M HNO_3	30
	Pb^{+2}	1243.0	1160.0	0.80M HNO_3	30
3.	Cu^{+2}	330.0	342.0	0.10M HNO_3	40
	Pb^{+2}	1243.0	1181.0	0.80M HNO_3	30
4.	Cu^{+2}	330.0	317.5	0.1M HNO_3	40
	Fe^{+3}	246.0	268.0	0.80M HNO_3	40
5.	Ni^{+2}	270.0	258.2	0.05M HNO_3	30
	Fe^{+3}	246.0	252.0	0.80M HNO_3	50
6.	Mn^{+2}	264.0	242.0	0.05M HNO_3	30
	Fe^{+3}	246.0	240.8	0.80M HNO_3	50
7.	CO^{+2}	590.0	536.9	0.05M HNO_3	40
	Fe^{+3}	246.0	224.0	0.80M HNO_3	40
8.	Zn^{+2}	326.5	319.9	0.05M HNO_3	40
	Fe^{+3}	246.0	213.4	0.80M HNO_3	40
9.	CO^{+2}	590.0	602.0	0.05M HNO_3	40
	VO^{+2}	265.2	270.3	0.50M HNO_3	40
10.	Mg^{+2}	119.0	116.6	0.05M HNO_3	30
	VO^{+2}	265.2	275.4	0.50M HNO_3	40
11.	Ni^{+2}	293.5	305.24	0.05M HNO_3	40
	VO^{+2}	265.2	275.4	0.5M HNO_3	40
12.	Zn^{+2}	326.5	313.4	0.05M HNO_3	40
	VO^{+2}	265.2	273.3	0.5M HNO_3	40
13.	Mn^{+2}	264.0	258.5	0.05M HNO_3	30
	VO^{+2}	265.2	275.3	0.5M HNO_3	40
14.	Mg^{+2}	119.0	114.2	.05M HNO_3	30
	Cu^{+2}	330.0	343.0	0.1M HNO_3	40

peak due to inter stitial water (1600cm^{-1}) persists upto 500°C. However, the peak vanishes completely when the drying temperature is increased to 800°C.

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