Separation Science and Technology
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713708471

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To cite this Article Qureshi, Moshin and Kaushik, R. C.(1982) 'Synthesis and Analytical Applications of Stannic Vanadotungstate', Separation Science and Technology, 17: 5, 739 — 744
To link to this Article: DOI: 10.1080/01496398208068564
URL: http://dx.doi.org/10.1080/01496398208068564

SEPARATION SCIENCE AND TECHNOLOGY, 17(5), pp. 739–744, 1982

NOTE

Synthesis and Analytical Applications of Stannic Vanadotungstate

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Abstract

A new inorganic ion exchanger stannic vanadotungstate has been prepared by treating 0.25 *M* Sn(IV) chloride with a 1:1 ratio of 0.25 *M* sodium tungstate and 0.25 *M* sodium vanadate until the three components were in the ratio of 2:1:1 by volume at different pH values (0, 1, and 2). It may be used for the binary separation of Al^{3+}–Ga^{3+} and Al^{3+}–Fe^{3+}.

INTRODUCTION

Many amorphous and crystalline inorganic ion-exchange materials have been synthesized during the last decade (*1–3*). They have been used to study the distribution coefficients (K_d values) in different solvent systems in the hope of finding special selectivities.

Niobium (*4*), aluminum (*5*), and stannic antimonates (*6*) were used to separate metal ions from interfering elements. Similarly, heteropoly acid salts such as titanium phosphosilicate (*7*) was used as a cation exchanger. Qureshi and Kaushik (*8–10*) synthesized crystalline stannic vanadophosphate and studied its ion-exchange behavior.

No such studies have been reported on tin(IV) vanadophosphate. It was therefore decided to synthesize the material to study its analytical utility and to determine its ion-exchange characteristics.

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EXPERIMENTAL AND RESULTS

Apparatus

Titrimetric and spectrophotometric studies were carried out with an Elico pH meter and a Bausch and Lomb Spectronic 20 Colorimeter, respectively. An electric temperature-controlled SICO shaker, Perkin-Elmer IR spectrophotometer and Phillips x-ray unit were used for K_d estimation, IR, and x-ray studies, respectively.

Reagents

Tin(IV) chloride pentahydrate (pure, Poland), sodium arsenate heptahydrate (Riedel, G.F.R.), and sodium metavanadate (B.D.H., England). All other reagents were of analytical grade.

Method of Preparation

0.25 *M* Sn(IV) chloride was added to a 1:1 mixture of 0.25 *M* sodium tungstate and 0.25 *M* sodium vanadate until the three components were in the ratio of 2:1:1 by volume (Table 1). Ammonia solution (*d* 0.88) was used for adjusting the pH wherever necessary. The precipitate obtained was allowed to stand for 24 h at room temperature and divided into two parts. One part was filtered off and washed with acid, and the gel dried at 40°C. The other part was refluxed in the mother liquor for 24 h and the precipitate dried at the same temperature. The dried materials were converted into the hydrogen form as described earlier (*10*).

TABLE 1

Synthesis, Chemical Composition, and Ion-Exchange Capacity of Stannic Vanadotungstate Samples

				Composition		
Sample no.	Sample[a]	pH	IEC (meg/g)	Sn	V	W
1	SWV (UR)	1	1.0	2	1	1
2	SWV (R)	1	0.8	6	2	3
3	SWV (UR)	2	0.8	6	3	2
4	SWV (R)	2	0.8	5	2	2

[a] UR = unrefluxed; R = refluxed; IEC = ion exchange capacity.

Chemical Composition

200 mg of the exchanger was dissolved in concentrated hydrochloric acid. Tungsten and vanadium were separated and determined as described earlier (*8, 10, 11*).

IR Spectra

IR absorption spectra were obtained by the KBr technique.

Heat Treatment

The ion-exchange material was dried at different temperatures and its ion-exchange capacity and distribution coefficient (K_d) were studied. The results are shown in Tables 2, 3, and 4.

Chemical Stability

A 500-mg sample was shaken in a conical flask with 50 mL of the concerned solution in the temperature-controlled shaker for 6 h until equilibrium was attained. Tin, vanadium, and tungsten were determined spectrophotometrically after filtration. The results are given in Table 5.

TABLE 2

Distribution Coefficient of Cations on Stannic Vanadotungstate (Sample 1)

Cations/system	Water	0.1 *M* NH_4NO_3	0.1 *M* HNO_3	0.01 *M* HNO_3	0.01 *M* CH_3COOH
Mg^{2+}	4400	20.0	20.0	150	429
Ca^{2+}	9900	600	0.0	400	566
Sr^{2+}	4500	360	68	2680	543
Ba^{2+}	4500	820	130	240	666
Zn^{2+}	1900	166	54	166	1900
Cd^{2+}	3900	300	0.0	3900	3900
Mn^{2+}	4300	193	42	340	4300
Pb^{2+}	820	4500	820	4500	4500
In^{3+}	1900	2400	900	2400	2400
Cu^{2+}	3800	4800	4800	53	4800
Ni^{2+}	3650	275	3650	66	3650
Fe^{3+}	213	2750	2250	20	3650
Al^{3+}	1600	1600	1600	0	360
Ga^{3+}	150	40	400	150	1365

TABLE 3

Distribution Coefficient of Cations on Tin(IV) Vanadotungstate Dried at Different Temperatures

Cations	Distilled water temperature (°C)		
	40	200	500
Mg^{2+}	4400	250	11
Ca^{2+}	9900	650	150
Sr^{2+}	4500	1110	30
Pa^{2+}	4500	1260	17
Zn^{2+}	1900	1676	15
Cd^{2+}	3900	437	38
Cu^{2+}	3800	1650	27
Ni^{2+}	3650	100	36
Mn^{2+}	4300	1330	10
Fe^{3+}	213	608	9
Al^{3+}	1600	718	14

Column Operation

A 30 × 0.3 (i.d.) cm glass column was used and 2 g of tin(IV) vanadotungstate was placed in the column with a glass wool support. The column was washed with demineralized water. The rate of flow was 1 mL/min. The effluent was titrated with EDTA.

Distribution Coefficient Studies

The distribution studies of cations were made in systems in which the exchanger was found to be more stable. Loading of cations for the system was less than 5% of the ion-exchange capacity of the material used.

TABLE 4

Separation Achieved on the Column

Cation	Eluent	Amount taken (μg)	Amount found (μg)	Error (%)
Ga^{3+}	1% HNO_3	880	850	−4.5
Al^{3+}	1% NH_4NO_3 + 0.001 *M* HNO_3	1000	990	−1.0
Ga^{3+}	1% HNO_3	880	860	−2.2
Al^{3+}	1% NH_4NO_3 + 0.001 *M* HNO_3	1000	1000	0.0

TABLE 5

Chemical Stability of Stannic Vanadotungstate (Sample 1)

Solvent	Solubility (mg/50 mL)		
	Sn	V	W
Distilled water	0.0	0.0	0.0
0.1 *M* Ammonium nitrate	0.0	0.6	0.0
4 *M* Nitric acid	7.0	0.4	0.0
Sodium hydroxide		Hydrolysis	
2 *M* Acetic acid	0.38	14	0.0

Separation Achieved

Al^{3+} (1000 μg) was separated from Ga^{3+} (880 μg) and Fe^{3+} (700 μg) on a stannic vanadotungstate column. Fe^{3+} and GA^{3+} were first eluted with 1% HNO_3, and Ga^{3+} was eluted with 1% HN_4NO_3 in 0.001 *M* HNO_3 (Table 4).

DISCUSSION

In a series of heteropolyacid salts, the Sn(IV) vanadotungstate can be used as an amorphous monoprotic weak acidic ion-exchange material.

The distribution coefficients of metal ions were determined on the exchanger samples heated to 40, 200, and 400°C. It was observed that the ion-exchange capacity decreases as the drying temperature of the material increases. (Results omitted to save space.) The IR spectra of stannic vanadotungstate have five characteristics peaks at 500, 900, 1400, and 1800 cm^{-1}. These peaks are strong and sharp while the one observed at 3600 cm^{-1} is strong but broad. These peaks correspond to SnO, VO, WO_3, OH, and free water, respectively.

Because the K_d values of Fe^{3+} and Ga^{3+} are much smaller than Al^{3+} (Table 3), the binary separations of Al^{3+} from Ga^{3+} and Fe^{3+} were quantitatively achieved (Table 5).

Acknowledgments

The authors are grateful to CSIR (India) for the financial assistance and to the Director of SPRC for library facilities.

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Received by editor August 15, 1981