CHAPTER-8
Synthesis, Characterization and Ion-Exchange behaviour of Zirconium (IV) Tungstoiodate

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Abstract

Zirconium (iv) Tungstoiodate has been synthesized as a new inorganic cat ion exchanger by adding a mixture of aqueous solution of 0.1 M potassium iodated and O.I.M. sodium tungstate to aqueous solution of O.I.M Zirconium (iv) oxychloride under varying mixing ratios at pH 1. On the basis of high ion exchange capacity, chemical stability and thermal stability the sample SW1-4 has been selected for further studies. The material has been characterized on the basis of chemical composition, FTIR, TGA and XRD. The chemical stability and thermal stability of the exchange material have also been studied, further various ion-exchange studies such as pH titration, Ion-exchange capacities and distribution studies have been performed on this material.

Key words

Zirconium (iv) tungstoiodate; Synthesis; characterization; inorganic ion exchanger.

1. Introduction

Inorganic ion-exchanger materials have established their place in analyticaistry (1-3) due to their resistance to heat and radiation and their different selectivity for metal ions. A large number of such materials have been synthesized earlier, most of which are hydrous oxides and the hetero poly acid-salts of the tetravalent and pentavalent metals. These compounds have shown great promise in analytical chemistry because of
their separation potential of metal ions (4). Every synthetic inorganic ion-exchanger has specific selectivity towards one or two metallic species. They are also highly-stable to a wide range of pH and upto high temperature.

* Since many inorganic exchanger based on tetravalent acid salt have been synthesized and studied for various analytical application. However, zirconium (iv) based inorganic ion-exchangers have received attention due to their excellent ion-exchange behaviour and some important chemical applications (5-12).

In present study, a new cat ion exchanger has been synthesized and characterized i.e. zirconium (iv) tungstioiodate, as per literature no information has been found on this material as ion exchanger.

Chemical and Reagents

Zirconium oxychloride (CDH), potassium iodate (S.D. fine chem.) and sodium tungstate (C.D.H.) were used for the synthesis of ion-exchangers. All other chemicals and reagents were used of analytical reagent grade.

Instrumentation

Spectrophotometer determination were carried out on Bausch and Lomb Spectronic-20 spectrophotometer meter whereas pH measurements were performed using as Elico Model LI-10 pH meter. For distribution studies temperature controlled shaker (Elico) was used.

Synthesis of the Ion-exchange Material

Samples of zirconium (iv) tungstioiodate ion exchanger were synthesized by adding a mixture of 0.1 M potassium iodated and 0.1 M sodium tungstate solution to 0.1 M zirconium (iv) oxychloride solution with continuous stirring. The desired pH was adjusted by adding dilute HNO3 or NaOH solutions. The gel so formed was allowed to
settle down for 24 h at room temperature and filtered under suction. The excess of acid was removed by washing with dematerialized water (DMW) and it was kept in oven at 40 °C for drying. The dried product was then treated with DMW which resulted in cracking of the substance into small particles with slight evolution of heat. The material was converted into the H⁺ form by treating with 1M HNO₃ for 24 hours at room temperature. The material was finally washed with DMW to remove any excess of acid.

**pH. Titration**

pH-titration was performed by the batch process using the toppe and pepper method (13). In this 0.5 g portions of the exchanger in H⁺ form were placed in each of the several 250 ml conical flasks followed by the equitible solution of alkali metal chlorides and their hydroxides in different volume ratios, the final volume being 50 ml to maintain the ionic strength constant. The pH of the solution was recorded after equilibrium and was plotted against the mill equivalents of the OH⁻ ions added.

**Determination of Ion-exchange Capacity (IEC)**

The Na⁺ ion exchange capacity of zirconium (iv) tungstoeniodeate was determined by column method (14), taking 0.5g of exchanger in H⁺ form in a glass column and passing 250 ml solution of sodium acetate (1m) through it, keeping flow rate 8-9 drops/minute. The effluent was collected and titrated with standard NaOH solution to find out the total H⁺ ions eluted.

**Chemical Stability**

To determine the chemical stability of exchanger, 0.5 of exchanger was equilibrated with different solutions of interest for 24 hours at room temperature. The
solution was decanted and analyzed for zirconium, tungstate and iodated using appropriate methods (15-17).

Chemical Composition

The chemical composition of material was determined by dissolving 100 mg portion of exchanger in cone $\text{H}_2\text{SO}_4$ and then diluted upto 100 ml. with distilled water. The molar ratios of zirconium tungstate and iodated were determined using methods (15-17).

Thermal Stability

For determination of thermal stability, 1 grams of exchanger in $\text{H}^+$ form was heated at different temperatures in muffle furnace for 1 hour. The ion exchange capacity of each sample was determined using column method.

Distribution Studies

For distribution studies, 200 mg of exchanger in $\text{H}^+$ form were equilibrated with 20 ml, solution of different metal ions in distilled water and nitric acid. The initial metal ion concentration was so adjusted that it did not exceed 3% of total ion exchange capacity. The metal ion in the solution before and after equilibrium were determined by EDTA titration and the distribution coefficients, $k_d$ were calculated using the given formula.

$$I = \text{initial volumes of EDTA used, } F = \text{final volume of EDTA used, } v = \text{the total volume of the solution (ml), } M = \text{the amount (g) of the exchanger.}$$
2. Results and Discussion

The new cat ion exchanger Zr (iv) tungstioiodate appears to be a promising ion exchange material with good ion exchange capacity, chemical stability and thermal stability. The preparations of various samples of zr (iv) tungstioiodates is given in table-1. It is apparent from the table that the mixing ratio of reagents in the preparation of Zr (iv) tungstioiodate is found to affect the ion-exchange capacity to some extent. The capacity is found higher for the sample 4 which has prepared with mixing ratio of tungstate and iodated simultaneously. The effect of size and charge of the exchanging ingoing ion on the ion exchange capacity of the material was studied for alkali and alkaline earth metal. The sequence shown by exchanger is as follows. And summarized in Table-3.

\[ \text{Li} < \text{Na}^+ < \text{K}^+ < \text{Mg}^{2+} < \text{Ca}^{2+} < \text{Sr}^{2+} < \text{Ba}^{2+} \]

The sequence for alkaline earth metal is in accordance with the hydrated radius. The ion with smaller hydrated radius easily enters the cavities of the exchanger resulting in higher exchange capacity. However, some irregularity that is observed in case of alkali metals is not common with amorphous inorganic ion exchanger and has also been reported by other authors (18-19).

The result of Extents of dissolution of exchange in different solvent is given in Table-3. It is found that material is highly stable in distilled water and stable in moderate concentration of mineral acids. The material was completely dissolved in 2m solution of sodium hydroxide.

From the results of chemical analysis the molar ratio of zirconium, tungstate and iodated was found to be 1:2:1.

The effect of heating on ion-exchange capacity of zr (iv) tungstioiodate (H⁺form) is given in Table-4. It is revealed that ion exchanges capacity decreases from 1.28 to 0.12 meq/ g in the temperature range of 40-600 °C. This is attributed to condensation of structural hydroxyl groups. Further it was found that as heating temperature rises the ion-exchange
capacity decrease (figure-2). The pH titration studies reveal that the material behaves as nonfunctional acid for Na⁺ showing one inflexion point (figure-1).

The distribution behaviour of some of the metal ions have been performed on zirconium (iv) tungstoiodates in distilled water and 0.1m nitric acid. The results are summarized in Table-5. It is reflected from the table that metal ions found affinity in the order as follow in both DMW & 0.1 HNO₃:

\[ \text{Pb}^{2+} > \text{Hg}^{2+} > \text{CO}^{2+} > \text{Cu}^{2+} > \text{Al}^{3+} > \text{Fe}^{3+} > \text{Ca}^{2+} > \text{Mg}^{2+} > \text{Zn}^{2+}. \]

On the basis of these studies, the metrical was found to be highly selective for Pb²⁺ which is polluting ion. The potentiality of the material may be demonstrated by achieving the separation of Pb²⁺ ion from environmental samples.

<table>
<thead>
<tr>
<th>Molar Concentration</th>
<th>Zrocl₂</th>
<th>Na₂WO₄</th>
<th>KIO₃</th>
<th>Ratio</th>
<th>White gelatinous</th>
</tr>
</thead>
<tbody>
<tr>
<td>ZWI-1</td>
<td>0.1</td>
<td>0.10</td>
<td>0.10</td>
<td>1:1:1</td>
<td>1.0  White gelatinous 1.00</td>
</tr>
<tr>
<td>ZWI-2</td>
<td>0.1</td>
<td>0.10</td>
<td>0.10</td>
<td>1:2:1</td>
<td>1.0  White gelatinous 1.20</td>
</tr>
<tr>
<td>ZWI-3</td>
<td>0.1</td>
<td>0.10</td>
<td>0.10</td>
<td>2:1:1</td>
<td>1.0  White gelatinous 1.10</td>
</tr>
<tr>
<td>ZWI-4*</td>
<td>0.1</td>
<td>0.10</td>
<td>0.10</td>
<td>1:2:2</td>
<td>1.0  White gelatinous 1.68</td>
</tr>
<tr>
<td>ZWI-5</td>
<td>0.1</td>
<td>0.10</td>
<td>0.10</td>
<td>1:1:2</td>
<td>1.0  White gelatinous 1.25</td>
</tr>
</tbody>
</table>

*Selected For detailed studies

Table-1: Synthesis and properties of Zr (iv) Tungstoiodate
<table>
<thead>
<tr>
<th>s.N.</th>
<th>Cation</th>
<th>PH</th>
<th>Hydrated radius</th>
<th>Ion-exchange capacity (meq/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Li⁺</td>
<td>6.8</td>
<td>0.340</td>
<td>1.10</td>
</tr>
<tr>
<td>2</td>
<td>Na⁺</td>
<td>6.7</td>
<td>0.276</td>
<td>1.28</td>
</tr>
<tr>
<td>3</td>
<td>K⁺</td>
<td>6.9</td>
<td>0.232</td>
<td>1.30</td>
</tr>
<tr>
<td>4</td>
<td>Mg²⁺</td>
<td>6.6</td>
<td>0.700</td>
<td>0.92</td>
</tr>
<tr>
<td>5</td>
<td>Ca²⁺</td>
<td>6.5</td>
<td>0.630</td>
<td>1.15</td>
</tr>
<tr>
<td>6</td>
<td>Sr²⁺</td>
<td>6.5</td>
<td>0.430</td>
<td>1.18</td>
</tr>
<tr>
<td>7</td>
<td>Ba²⁺</td>
<td>6.4</td>
<td>0.590</td>
<td>1.20</td>
</tr>
</tbody>
</table>

Table 2: Ion-exchange capacity for various cation

<table>
<thead>
<tr>
<th>Amount Released (mg/50 ml)</th>
<th>Zr</th>
<th>WO₄⁻</th>
<th>IO₃⁻</th>
</tr>
</thead>
<tbody>
<tr>
<td>Distilled water</td>
<td>0.00</td>
<td>0.00</td>
<td>0.000</td>
</tr>
<tr>
<td>0.01 M HCl</td>
<td>0.20</td>
<td>0.32</td>
<td>0.40</td>
</tr>
<tr>
<td>0.10 M HCl</td>
<td>0.22</td>
<td>0.36</td>
<td>0.45</td>
</tr>
<tr>
<td>1 M HCl</td>
<td>0.26</td>
<td>0.42</td>
<td>0.48</td>
</tr>
<tr>
<td>0.01 M HNO₃</td>
<td>0.35</td>
<td>0.25</td>
<td>0.23</td>
</tr>
<tr>
<td>1 M HNO₃</td>
<td>0.38</td>
<td>0.185</td>
<td>0.32</td>
</tr>
<tr>
<td>2 M HNO₃</td>
<td>0.40</td>
<td>0.31</td>
<td>0.25</td>
</tr>
<tr>
<td>1 M H₂SO₄</td>
<td>0.45</td>
<td>0.24</td>
<td>0.20</td>
</tr>
<tr>
<td>2 M H₂SO₄</td>
<td>0.32</td>
<td>0.18</td>
<td>0.175</td>
</tr>
<tr>
<td>2 M CH₃COC</td>
<td>0.18</td>
<td>0.15</td>
<td>0.00</td>
</tr>
</tbody>
</table>
Table 3: Chemical Stability of Zr (iii) lungs to date in various solution at Room temperature.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Color</th>
<th>Amount Released (mg/50 ml)</th>
</tr>
</thead>
<tbody>
<tr>
<td>40</td>
<td>White</td>
<td>1.26</td>
</tr>
<tr>
<td>100</td>
<td>Yellowish</td>
<td>1.09</td>
</tr>
<tr>
<td>200</td>
<td>Light brown</td>
<td>0.85</td>
</tr>
<tr>
<td>300</td>
<td>Brown</td>
<td>0.64</td>
</tr>
<tr>
<td>400</td>
<td>Brown</td>
<td>0.48</td>
</tr>
<tr>
<td>500</td>
<td>Brown</td>
<td>0.30</td>
</tr>
<tr>
<td>600</td>
<td>Black</td>
<td>0.12</td>
</tr>
</tbody>
</table>

Table 4: Effect of heating on ion-exchange capacity of Zn (iv) tungstosiodate (H⁺ form)

<table>
<thead>
<tr>
<th>Kd C mg/g</th>
<th>DMW</th>
<th>0.1 M HNO₃</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zn²⁺</td>
<td>10.5</td>
<td>15.0</td>
</tr>
<tr>
<td>Mg⁺</td>
<td>17.8</td>
<td>19.0</td>
</tr>
<tr>
<td>Ca²⁺</td>
<td>15.5</td>
<td>24.8</td>
</tr>
<tr>
<td>Pb²⁺</td>
<td>159.8</td>
<td>162.0</td>
</tr>
</tbody>
</table>
Table-5: Distribution coefficient (kd) of various metal ions on Zr (iv) tungstioxidate (H^+form)

<table>
<thead>
<tr>
<th></th>
<th>DMW</th>
<th>0.1 M HNO3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hg^{2+}</td>
<td>72.9</td>
<td>74.0</td>
</tr>
<tr>
<td>Cu^{2+}</td>
<td>25.5</td>
<td>36.0</td>
</tr>
<tr>
<td>CO^{2+}</td>
<td>32.8</td>
<td>38.0</td>
</tr>
<tr>
<td>Cd^{2+}</td>
<td>12.0</td>
<td>14.5</td>
</tr>
<tr>
<td>Al^{3+}</td>
<td>18.9</td>
<td>26.5</td>
</tr>
<tr>
<td>Fe^{3+}</td>
<td>22.5</td>
<td>33.7</td>
</tr>
</tbody>
</table>

References


NEW METHOD OF SYNTHESIS OF STANNIC (IV) IODO TUNGSTATE AND ITS CHARACTERIZATION AS ION EXCHANGER

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ABSTRACT

A new inorganic ion exchanger Stannic(IV) Iodo Tungstate has been synthesized at different pH values. Amorphous sample prepared at pH 1 having an ion-exchange capacity of 1.25 meq/g was selected for further studies. The material is characterized by using various analytical techniques, like XRD, IR, TGA and SEM. In addition, ion-exchange capacity, chemical stability, distribution coefficient ($K_d$ values) were also studied.

Keywords: Stannic(IV)Iodo Tungstate; Cation exchangers.

INTRODUCTION

Heteropoly acid salts of Tin (IV) and zirconium (IV) are reported in literature having better ion exchange properties than their single salts. Synthetic inorganic ion exchangers are generally produced a gelatinous precipitates rapidly by mixing the elements of group III A to IV A of the periodic table usually at room temperature. The early ion exchangers were largely inorganic in origin. In the recent years, the interest in the inorganic ion exchangers has been revived with the need for high temperature separation of ionic components in radio active wastes. In order to accomplish such operation, high selective exchangers are required which are not only stable at high temperature but also have ion exchange properties unaffected by the acidity at high radiation levels organic ion exchange resins are not suitable for such applications, as changes in capacity and selectively take place on exposure to radiation. Also the degradation takes place at the high temperature.

A large number of inorganic ion exchanger were prepared by mixing phosphoric, arsenic, molybdic, antimoniac and vanadic acids with Zirconium, Tin, Titanium, Cerium, Bismuth, and Tantalum etc. Alberi and Clearfield devoted their studies mainly on the crystalline materials with a view to evaluate their structures based on x-ray studies.

Experimental Section-

Reagent and Chemicals-Stannous chloride, Sodium tungstate and potassium iodate were obtained from CDH (India) and all other reagents and chemicals were of analytical grade.

Instrumentation- pH measurements were performed using an Elico (India) L1-10 model pH meter, FTIR model Shimadzu 820 PC, Phillips X-ray unit, Duprot 900 and 950 thermal analyser and an electric shaking machine were used for IR studies, X-ray diffraction, thermal analysis and shaking respectively. SEM images were obtained by using a JEOl JSM 840, an electron microscope. An electric thermostat oven (Microsl, India) was used for drying the samples.

Synthesis of ion exchange materials-The ion exchange material was synthesized by mixing 1.0 M Stannous chloride solution gradually to a mixture of sodium tungstate in the presence of sulphuric acid (5N), H$_2$O$_2$ (0.3%) and potassium iodate (5N). Flocculating agent was added to reaction mixture. The whole process of synthesis is carried out at pH 1. Reaction time is of 1 hr. The gelatinous precipitate so obtained was allowed to settle down for 24 h at room temperature. The supernatant liquid was removed and the precipitate was washed with distilled water and dried at 40± 1°C in an electric oven. The material was broken down into small granules by placing in distilled water. The granules were converted into H$^+$ form by treating with 1M HNO$_3$ solution for 24h. The material was then washed several times with distilled water to remove excess of acid and finally dried at 40±1°C. Six samples are prepared by using above same method. Results are summarized in table...
Table 1

Conditions of Synthesis and Properties of different samples of Stannic Iodo Arsenate.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Molar Concentration</th>
<th>Mixing Ratio (v/v/v)</th>
<th>pH</th>
<th>Appearance of beads after drying at 40°C</th>
<th>I.E.C. Meq/g</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Sn(M)</td>
<td>IO₂(M)</td>
<td>W₂O₅(M)</td>
<td>1:1:1</td>
<td>1</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1:2:1</td>
<td>1</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>2:1:2</td>
<td>1</td>
</tr>
<tr>
<td>4</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>2:2:1</td>
<td>1</td>
</tr>
<tr>
<td>5</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>3:2:1</td>
<td>1</td>
</tr>
<tr>
<td>6</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>3:1:2</td>
<td>1</td>
</tr>
</tbody>
</table>

**CHARACTERIZATION**

**Determination of ion exchange capacity** - The ion exchange capacity (IEC) was calculated using the following equation:

\[
\text{IEC} = \frac{V \times M}{W_{\text{dry}}}
\]

Where, IEC is the ion exchange capacity (Meq g⁻¹), V the added titrant volume at the equivalent point (ml), M the Molar concentration of the titrant and W_{dry} is the dry Mass of the sample (g).

**pH Titration** - The pH titration of material was performed by the method of toppe and pepper. In this method 0.50 gram of exchanger was equilibrated with varying amounts of 0.1M NaCl and NaOH solutions.

**Thermal Stability** - The ion exchanger sample was dried at different temperature from 40°C to 600°C in a muffle furnace. Thermal stability of the material was evaluated by heating it at various temperatures for one hour in a muffle furnace and the Na⁺ ion exchange capacity in meq g⁻¹ dry exchanger was determined.

**Chemical Composition and chemical stability** -
For the determination of chemical composition of the sample SIT-6, 0.1 gram of exchanger was dissolved in minimum amount of hot concentrated sulphuric acid. The solution was diluted to 100ml with DMW. The amounts of Stannic and Iodate were determined spectrophotometrically using alizarin red-S and pyrogallol as colouring agents respectively. Tungstate was determined titrimetrically using the standard method.

500mg of the sample SIT-5 was shaken with 50ml of the various solutions at 30±2°C for 8 hours. The amounts of Stannic, Iodate and Tungstate released in the solutions were determined spectrophotometrically. The material is found to be quite stable in lower concentration of mineral acid, such as HCl, H₂SO₄ and HNO₃. The sample is quite stable in organic acids.

Chemical stability, especially low solubility, is important for the usefulness of inorganic ion exchange materials. Most Zeolites for example, only work over a limited range pH range (4-9), since they are soluble both in acidic and alkaline media. Of the inorganic ion exchange materials developed so far, those based on pure or mixed chromium or titanium oxides have been shown to be the most stable.

**Distribution Studies** - The distribution studies of various metal ions in distilled water and nitric acid were carried out by the batch equilibrium technique. For this purpose, 0.50 gram of the exchanger in H⁺ form was equilibrated with 20.0 ml of 0.1M solutions of different metal ions.

The mixture was shaken for 6 hours to attain equilibrium. The amount of metal ions left in the solutions was then determined by titrating against 0.01M EDTA solution using appropriate indicators.

The Kd values, were calculated using the following equation

\[ KD = \frac{(1 - F) V}{F x M} \]

where, I and F denote the volume of EDTA solution required before and after equilibrium respectively, V is the solution volume in ml and M is the mass of the exchanger in gram.

**X-Ray Study** - X-ray studies were carried out using a Philips pw-1730 high angle diffractometer (Poland) Cu-Kα (Ni Filter) radiation and it was found that material shows amorphous nature.

**FTIR Spectrum** - FTIR spectrum of sample SIT-5 was obtained by KBr disc method. FTIR spectrum of the prepared sample was obtained by using a FTIR model shimadzu 820 PC.

**Scanning electron micrographs (SEM)** - Surface morphology of the dried exchanger was investigated using SEM.

**Result and Discussion** - The results from the study of the ion exchange capacity of various samples of Stannic Iodo Tungstate are presented in Table 1. The improvement in the ion exchange capacity and chemical stability seem to be much higher for sample SIT-5 and therefore this sample was selected for detailed studies.

The ion exchange capacities for mono- and bivalent metal ions are shown in Table 2 these results indicate that ion exchange selectivity of the material is different for different cat ions. The ion exchange capacities in meq g⁻¹ dry exchanger for various metal ions follow the order:

\[ \text{Li}⁺ > \text{Na}⁺ > \text{K}⁺ > \text{Mg}^{2⁺} > \text{Ca}^{2⁺} > \text{Sr}^{2⁺} \]

Table 2

**IOn Exchange Capacity (Meg g⁻¹ dry exchangers) of Stannic Iodo Tungstate (Sample SIT-5 for various metal ions)**
<table>
<thead>
<tr>
<th>S. No.</th>
<th>Cation</th>
<th>Salt Used</th>
<th>I.E.C. (Meg g⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Li⁺</td>
<td>LiCl</td>
<td>1.40</td>
</tr>
<tr>
<td>2</td>
<td>Na⁺</td>
<td>NaCl</td>
<td>1.30</td>
</tr>
<tr>
<td>3</td>
<td>K⁺</td>
<td>KCl</td>
<td>1.38</td>
</tr>
<tr>
<td>4</td>
<td>Mg²⁺</td>
<td>Mg(NO₃)₂</td>
<td>1.48</td>
</tr>
<tr>
<td>5</td>
<td>Sr²⁺</td>
<td>Sr(NO₃)₂</td>
<td>1.46</td>
</tr>
<tr>
<td>6</td>
<td>Ca²⁺</td>
<td>Ca(NO₃)₂</td>
<td>1.24</td>
</tr>
</tbody>
</table>

In order to examine the effect of heating temperature on the ion exchange capacity, the material was heated at 40, 100, 200, 300, 400, 500 and 600°C and ion exchange capacity for Na⁺ ion was found to be 1.30, 1.05, 0.90, 0.70, 0.30, 0.20 and ~0 meq g⁻¹ dry exchanger respectively. The results are summarized in Table 3.

Table 3

Effect of heating temperature on the ion exchanger capacity of Stannic Iodo Tungstate.

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Heating Temperature</th>
<th>Colour of beads after drying</th>
<th>Ion exchanger Capacity Meq g⁻¹ dry exchanger</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>40</td>
<td>White</td>
<td>1.30</td>
</tr>
<tr>
<td>2</td>
<td>100</td>
<td>White</td>
<td>1.05</td>
</tr>
<tr>
<td>3</td>
<td>200</td>
<td>Brown</td>
<td>0.90</td>
</tr>
<tr>
<td>4</td>
<td>300</td>
<td>Brown</td>
<td>0.70</td>
</tr>
<tr>
<td>5</td>
<td>400</td>
<td>Brown</td>
<td>0.30</td>
</tr>
</tbody>
</table>
The material was found to be fairly stable in lower concentration of mineral acids such as HCl, H₂SO₄, HNO₃ and acidic. The ion exchanger completely dissolved in 2M H₂SO₄ and 2M NaOH solution. The results are shown in Table 4.

### Table 4

**Chemical Stability of Stannic Iodo Tungstate in different Solvents**

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Solution</th>
<th>Sn Released (MG/50ml)</th>
<th>IO₃ released (MG/50ml)</th>
<th>WO₃ Released (Mg/50ml)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>H₂O</td>
<td>0.00</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>2</td>
<td>0.1M HClO₄</td>
<td>0.00</td>
<td>0.24</td>
<td>0.38</td>
</tr>
<tr>
<td>3</td>
<td>1.0 M HClO₄</td>
<td>0.46</td>
<td>0.79</td>
<td>0.90</td>
</tr>
<tr>
<td>4</td>
<td>0.1 M HNO₃</td>
<td>0.00</td>
<td>0.12</td>
<td>0.20</td>
</tr>
<tr>
<td>5</td>
<td>1.0 M HNO₃</td>
<td>0.36</td>
<td>0.72</td>
<td>0.26</td>
</tr>
<tr>
<td>6</td>
<td>0.1 M HCl</td>
<td>0.00</td>
<td>0.00</td>
<td>0.12</td>
</tr>
<tr>
<td>7</td>
<td>1.0 M HCl</td>
<td>1.26</td>
<td>0.57</td>
<td>0.54</td>
</tr>
<tr>
<td>8</td>
<td>1.0 MCH₃COOH</td>
<td>0.00</td>
<td>0.20</td>
<td>0.22</td>
</tr>
<tr>
<td>9</td>
<td>0.1 M NaOH</td>
<td>0.91</td>
<td>2.90</td>
<td>2.05</td>
</tr>
<tr>
<td>10</td>
<td>2.0 M NaOH</td>
<td>Completely Dissolved</td>
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<td></td>
</tr>
</tbody>
</table>

The FTIR of Stannic Iodo Tungstate shows broad bands in the 3180 cm⁻¹ region characteristic of asymmetric and symmetric hydroxo-OH and aqua-OH stretching frequency. A sharp medium band at 1680 cm⁻¹ is attributed to the aqua (H-OH) bending. The spectrum also shows weak band in the region 1000-500 cm⁻¹ indicating the presence of iodate and metal oxide. The FTIR spectra is shown in figure 1.

**Figure - 1 I.R Spectrum of Stannic Iodo Tungstate**
In absence of sharp peaks in the X-ray diffraction pattern of Zirconium (IV) iodide arsenate indicates that the material is amorphous in nature.

In order to explore the potential of this cation exchange material in the separation of metal ions, distribution studies for 10 metal ions were performed. The presence of other electrolytes in metal ion solution strongly affects the Kd value of the metal ion. Kd values change depending on the ionic strength and pH of the electrolyte. The degree of ionization of the ionic groups of the exchanger depends on the acid or base strength of the groups i.e. on their pK value of the groups, the acid groups become predominantly nonionized, and the complex formation capacity, and hence the Kd values thus fall off based on such a study. A proper electrolyte can be selected for the elution of a particular metal ion from the column of the exchanger when a metal ion pair is to be separated using a column of the exchanger, the Kd value of particular metal ion in a particular solvent gives a guideline to separate out distribution coefficient (Kd) calculated for several metal ions in aqueous and various electrolyte media. The distribution studies showed that the material was found to possess exceptional high Kd values for Pb(II). Pb(II) has been considered as a major polluting metal in water. The Table 5 reveals that all the metal ions except Ca²⁺, Al³⁺, Ba²⁺ exhibit exceptionally higher Kd values in 0.02M CH₃COOH media than in distilled water. Similar observation have been made by others.

Table-5

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Metal Ions</th>
<th>DMW</th>
<th>0.01M HNO₃</th>
<th>0.1M HNO₃</th>
<th>1M HNO₃</th>
<th>0.01M HCl</th>
<th>0.1M HCl</th>
<th>1M HCl</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>Al³⁺</td>
<td>9300</td>
<td>4600</td>
<td>3300</td>
<td>2250</td>
<td>3200</td>
<td>1820</td>
<td>1600</td>
</tr>
<tr>
<td>2</td>
<td>Ba²⁺</td>
<td>3016</td>
<td>1100</td>
<td>932</td>
<td>750</td>
<td>1740</td>
<td>1220</td>
<td>700</td>
</tr>
<tr>
<td>3</td>
<td>Ca²⁺</td>
<td>1755</td>
<td>1200</td>
<td>660</td>
<td>640</td>
<td>1050</td>
<td>510</td>
<td>450</td>
</tr>
<tr>
<td>4</td>
<td>Cu²⁺</td>
<td>7950</td>
<td>3800</td>
<td>1540</td>
<td>1050</td>
<td>2100</td>
<td>880</td>
<td>810</td>
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<td></td>
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<td>1650</td>
<td>880</td>
<td>800</td>
<td>1210</td>
<td>820</td>
</tr>
<tr>
<td>---</td>
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<td>---------</td>
<td>------</td>
<td>------</td>
<td>-----</td>
<td>-----</td>
<td>------</td>
<td>-----</td>
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<tr>
<td>6</td>
<td>Ca^{2+}</td>
<td>4500</td>
<td>1224</td>
<td>904</td>
<td>750</td>
<td>1350</td>
<td>1000</td>
<td>900</td>
</tr>
<tr>
<td>7</td>
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<td>4200</td>
<td>2800</td>
<td>1604</td>
<td>1066</td>
<td>2033</td>
<td>1500</td>
<td>1500</td>
</tr>
<tr>
<td>8</td>
<td>Mg^{2+}</td>
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<td>750</td>
<td>1500</td>
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</tr>
<tr>
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<td>Pb^{2+}</td>
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<td>8700</td>
<td>7200</td>
<td>1150</td>
<td>7700</td>
<td>200</td>
<td>1250</td>
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<td>850</td>
<td>520</td>
<td>1220</td>
<td>800</td>
<td>620</td>
</tr>
</tbody>
</table>

REFERENCES