Synthesis and Use of Aluminum Antimonate as an Ion-Exchanger

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Summary
A new inorganic ion-exchanger, aluminum antimonate, is described. It is the most stable of the antimonates so far studied as ion-exchangers. Exchange capacity and Kd values have been determined, and the effect of heat, stability etc., determined. Important binary and ternary separations have been achieved. The material is a weak cation-exchanger.

Introduction
Work on inorganic ion-exchangers has recently been reviewed by Walton [1]. In earlier papers from these laboratories studies on ion-exchangers prepared from metals in oxidation states four and five have been reported. Studies of aluminum salts did not receive much attention. Aluminum triphosphate [2] has been prepared and can be used as a selective ion-exchanger for $K^+$ within a limited pH range. An aluminum vanadate membrane has been prepared [3] having an ion-exchange capability and operating efficiently up to 900 °C.

Antimonic acid has large adsorption capacity compared with many other hydrous metal oxides [4]. Thermally and chemically stable tin antimonate has been synthesized [5]. The antimonates of Ti, Zr, Ta, Cr and Ce have also been reported [6-10]. Aluminum antimonate has not been studied as an ion-exchanger so far therefore we decided to investigate this material. This report summarizes the preparation and properties of aluminum antimonate, and its use in column operation for separation purposes.

Experimental
Reagents: Aluminum nitrate (AnalR, B.D.H., India) and antimony pentachloride B.D.H. (England) were used. Antimony pentachloride solution was diluted with 4 mol dm$^{-3}$ HCl to the desired concentration. All other reagents were of AnalR grade.

Apparatus: A temperature controlled SICO shaker, Bausch and Lomb spectronic 20 and 'ELICO' pH meter model Li-10, were used.

Synthesis procedure: Four samples of aluminum antimonate were prepared by mixing 0.1 mol dm$^{-3}$ aluminum nitrate and 0.1 mol dm$^{-3}$ SbCl$_5$ in the volume ratio 3:1.

The aluminum nitrate was added to the SbCl$_5$ and the pH adjusted with 0.1 mol dm$^{-3}$ NaOH. Sample 5 was prepared by refluxing the mother liquor of sample 1 for 24 hours. The precipitates were allowed to settle for 24 hours at room temperature and then washed by decantation. The residues were dried at 40 °C. The dried products broke down into small particles when immersed in water. They were converted to the hydrogen form by repeated treatment with 1.0 mol dm$^{-3}$ HNO$_3$ and finally repeatedly washed with demineralized water to remove excess acid. All the samples were again dried at 40 °C. The sieve fraction 100-200 mesh was used for distribution studies. The conditions of preparation are summarized in Table I.

<table>
<thead>
<tr>
<th>Sample</th>
<th>pH of final solution</th>
<th>Analysis</th>
<th>Ion-exchange capacity meq/g for Na$^+$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Negative</td>
<td>4.2:1</td>
<td>1.14</td>
</tr>
<tr>
<td>2.</td>
<td>0</td>
<td>4.3:1</td>
<td>0.67</td>
</tr>
<tr>
<td>3.</td>
<td>1</td>
<td>5:1</td>
<td>0.40</td>
</tr>
<tr>
<td>4.</td>
<td>4</td>
<td>5:1</td>
<td>0.12</td>
</tr>
<tr>
<td>5.</td>
<td>Negative</td>
<td>4.2:1</td>
<td>1.14</td>
</tr>
</tbody>
</table>

Results and Discussion
All the samples were white. They were apparently stable in water and dilute acids.

Ion-exchange capacity: The exchange capacity of this material (sample No. 1) was determined by taking 1 g, in the H$^+$ form (converted according to the procedure given in the synthesis) in a column and passing 1.0 mol dm$^{-3}$ solution of different mono and divalent ions over it. The liberated H$^+$ was titrated with standard NaOH solution. The effluent collected in each case was 400 cm$^3$ on the basis of elution curve, which was similar to that obtained with stannic molybdate [11]. Using 1 mol dm$^{-3}$NaNO$_3$ as the eluent the release of hydrogen ions falls sharply after 25 cm$^3$ of effluent and finally becomes insignificant after 400 cm$^3$. Hence it was assumed that exchange is nearly complete after passing 400 cm$^3$ of electrolyte through the column. The ion-exchange capacities were 0.77, 1.14, 1.17, 1.52, 1.28, 1.25 and 1.25 meq/dry gram of aluminum antimonate for Li$^+$, Na$^+$, K$^+$, Mg$^{2+}$, Ca$^{2+}$, Sr$^{2+}$ and Ba$^{2+}$ respectively.

Aluminum antimonate behaves as a weak cation-exchanger. The exchange capacity varies with the metal ion. It is nearly the same for Ca$^{2+}$, Sr$^{2+}$ and Ba$^{2+}$ (1.25 meq/g) and for Na$^+$ and K$^+$ (1.15 meq/g); but much less (0.77 meq/g) for Li$^+$ and much higher (1.52 meq/g) for Mg$^{2+}$. This
Fig. 1a
- Separation of Th$^{4+}$–UO$_2^{2+}$ and Ni$^{2+}$–Cu$^{2+}$–Ag$^{+}$ on aluminum antimonate

Fig. 1b
- Separation of Mg$^{2+}$–Ca$^{2+}$ Mg$^{2+}$–Sr$^{2+}$ and Mo$^{6+}$ Ti$^{4+}$–W$^{6+}$ on aluminum antimonate