

Hydrolysis of Antimony(III)-Hydrochloric Acid Solution at 25°C*¹

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The system Sb(III)-HCl-H₂O has been studied by equilibrating mixtures of antimony trioxide and hydrochloric acid solution (<6 kmol/m³) at 25°C for 30 days. X-ray diffraction analysis of the solids showed the existence of Sb₂O₃ (HCl < 0.1 kmol/m³) and Sb₄O₅Cl₂ (HCl > 0.1 kmol/m³) as the final solid phases. The solubility for both compounds was also determined as a function of HCl concentration. The hydrolysis of antimony(III)-hydrochloric acid solution (Sb(III) = 140 mg/L, HCl = 0.1 and 1.0 kmol/m³) by neutralizing with NaOH solution was then studied in a pH range of 1.1 to 12.3 at 25°C. The amorphous Sb₈O₁₀(OH)₂Cl₂ (spherical, particle size: 0.05–0.2 μm), crystalline Sb₈O₁₀(OH)₂Cl₂ (orthorhombic, rectangular plate in 1 μm thick layer) and crystalline Sb₂O₃ (orthorhombic) were found to be a solid phase depending on pH and hydrolysis time. The particle morphology of Sb₂O₃ was affected strongly by pH. The minimum solubility of crystalline Sb₈O₁₀(OH)₂Cl₂ (pH 2–3) and Sb₂O₃ (pH 3–11) was at a level of 10 mg/L in Sb(III).

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1. Introduction

Antimony has been known as one of metals since ancient times, but its use was limited to alloys for decorative pots, bells and printer's types. In recent years, dramatic growth of car industry gave rise to expansion of demand of antimony as an alloying element of lead alloy for battery grids, particularly for positive pole electrode material. Currently, antimony attracts attention as a stuff element of III–V semiconducting materials such as InSb, AlSb and GaSb, and as an alloying ingredient in thermoelectric alloys.

Antimony trioxide has been used in various industries of ceramics, glass, pigments, catalysts, resistors, plastics and textiles, and is specially in great demand in flame-retarding formulations for plastics, rubber and fibers. Commercial users request various grades of antimony trioxide based on the relative tinting strength of their product. Most of crude antimony trioxide has been produced by roasting of antimony sulfide (Sb₂S₃) ore in oxygen-enriched air. Metallic antimony has been supplied to vaporization-oxidation process to produce a high-grade antimony trioxide. The characteristics of the oxide such as crystal morphology and particle size are sensitively affected by oxidation temperature, residence time and cooling rate.

In general, the careful hydrolytic precipitation processes may yield a very fine powder of metal oxides. Hence, a hydrometallurgical process, which consists of (1) hydrochloric acid leaching of crude Sb₂S₃ or Sb₂O₃, (2) hydrolysis to produce Sb₄O₅Cl₂ precipitate, (3) hydrochloric acid leaching of Sb₄O₅Cl₂, (4) Sb₂O₃ precipitation by hydrolysis, can be considered to be capable of producing a fine powder of high-grade antimony trioxide. For better understanding and development of this method, detailed data on the system Sb(III)-HCl-H₂O and the hydrolysis of antimony(III)-hydrochloric acid solution are required.

Furthermore, the data of the hydrolysis of antimony species will provide fundamental knowledge for develop-

ment of processes involving pH adjustment and coprecipitation with metal hydroxides to remove antimony species from aqueous solutions at extremely low level of concentration.^{1–5)}

In this work, the equilibrium of Sb(III)-HCl-H₂O system has been studied to determine the stability and solubility for Sb₂O₃ and Sb₄O₅Cl₂ at 25°C and then the hydrolysis of antimony(III)-hydrochloric acid solution by neutralizing with NaOH solution was studied, paying particular attention to the effect of pH and chloride ion concentration on the particle shape and size of produced Sb₂O₃ and its solubility at 25°C.

2. Experimental

2.1 Equilibrium of Sb(III)-HCl-H₂O system

A quantity of 25 mL of hydrochloric acid solutions of various concentrations (0 to 6 kmol/m³) was charged in 100 mL sealable glass bottles. Antimony trioxide, Sb₂O₃ (Aldrich Chemical Co., Gold Label, orthorhombic) was dissolved in the solutions up to the saturation and then an excess of Sb₂O₃ (0.25 to 7.0 g in total) was added to produce a fine suspension. The bottles were tightly capped and shaken in an air bath at 25°C for 30 days to achieve equilibrium between the solid and liquid phases. The suspensions of precipitate were filtered with a membrane filter (pore size 0.65 μm) and the filtrates were submitted to chemical analysis for antimony by the inductively coupled plasma atomic emission spectroscopy (ICP-AES) method. For the solutions of low initial concentration of HCl in which very fine particles were in suspension, centrifugal separation was employed to eliminate by the suspended particles and the supernatant solutions were analyzed for antimony. The solid phase was submitted to X-ray diffraction (XRD) analysis. Selected solid samples were also examined by scanning electron microscopy (SEM).

2.2 Hydrolysis of Antimony(III) in the presence of chloride ion

In the hydrolysis experiment, the starting solution of antimony(III)-hydrochloric acid ([Sb(III)]₀ = 140 mg/L, [HCl]₀ = 0.1 and 1.0 kmol/m³) was prepared by dilution of

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the filtrate ($[\text{Sb(III)}] = 5.67 \text{ g/L}$, $[\text{HCl}] = 3.96 \text{ kmol/m}^3$) obtained in the previous series of equilibrium experiment with distilled water and 36% HCl solution. A quantity of 50 mL of the solution was charged in a three-necked flask of 100 mL capacity which was placed in a water bath at 25°C and stirred with a two-bladed glass impeller. The solution pH was shifted first to 1.3 with sodium hydroxide tablets and then adjusted to the individual starting value with NaOH solution. After appropriate holding-times, the solutions with precipitate were filtered with a membrane filter (pore size 0.65 μm) and the filtrates were analyzed for Sb by ICP-AES method. The precipitate from each run was submitted to X-ray diffraction and SEM observation. The groups of the solutions submitted to the hydrolysis procedures are denoted by their chloride concentrations, 0.1 or 1.0 kmol/m^3 in Figs. 3 to 12.

3. Results and Discussion

3.1 Solubility and stability region of antimony(III) compounds in hydrochloric acid solution

The preliminary experiments were carried out to determine the equilibration conditions for Sb(III)-HCl-H₂O system at HCl = 0, 0.1 and 1.0 kmol/m^3 by analysis of the solutions at interval of 7 days. It was confirmed that, in 1.0 kmol/m^3 HCl solution, Sb₂O₃ reacts with HCl to form solid Sb₄O₅Cl₂ at a fairly rapid reaction rate and that equilibrium was reached in one or two weeks in the most cases. In all the subsequent experiments, the shaking to establish equilibrium was continued for 30 days.

In order to identify the solid phase in equilibrium with HCl solution (0 to 6 kmol/m^3 HCl), the solid products were submitted to XRD analysis. The typical XRD patterns of the solid phase are shown in Fig. 1, indicating that Sb₂O₃ (JCPDS No. 11-0-689, orthorhombic) is a stable solid phase in the solution of 0 to 0.1 kmol/m^3 HCl and Sb₄O₅Cl₂ (JCPDS No. 30-0091, monoclinic) is stable at higher HCl concentration up to 5.6 kmol/m^3 in this study. It is worthy to note that the main XRD peaks for the solid produced in the solution of 0.12 kmol/m^3 HCl can be assigned to Sb₄O₅Cl₂ instead of Sb₂O₃.

The weak, undefined peaks at higher peak-angles in Fig. 1 are still under discussion.

The Sb(III) oxychlorides, SbOCl, was not detected at HCl concentration below 6 kmol/m^3 . The concentration of free hydrochloric acid was calculated by subtracting the stoichiometric amount of HCl for the reaction ($2\text{Sb}_2\text{O}_3 + 2\text{HCl} = \text{Sb}_4\text{O}_5\text{Cl}_2 + \text{H}_2\text{O}$) from the initial amount of HCl in the solution, and is here presented as a HCl concentration of solutions in equilibrium with Sb₄O₅Cl₂.

Figure 2 shows the Sb(III) concentration of the HCl solutions in equilibrium with solid Sb(III) compounds as a function of concentration of free HCl at 25°C. In this figure, the Sb(III) compound of the solid phase identified by XRD analysis are also presented: Sb₂O₃ (A) at HCl concentration below 0.1 kmol/m^3 and Sb₄O₅Cl₂ (B) above 0.12 kmol/m^3 HCl. The observed solubility of Sb₂O₃ in water was 6.4 mg/L in Sb(III), which is very close to the value of 7.1 mg/L reported by Gayer and Garrett in 1952.⁶⁾ The solubility of Sb₂O₃ in solutions containing HCl has a minimum value of

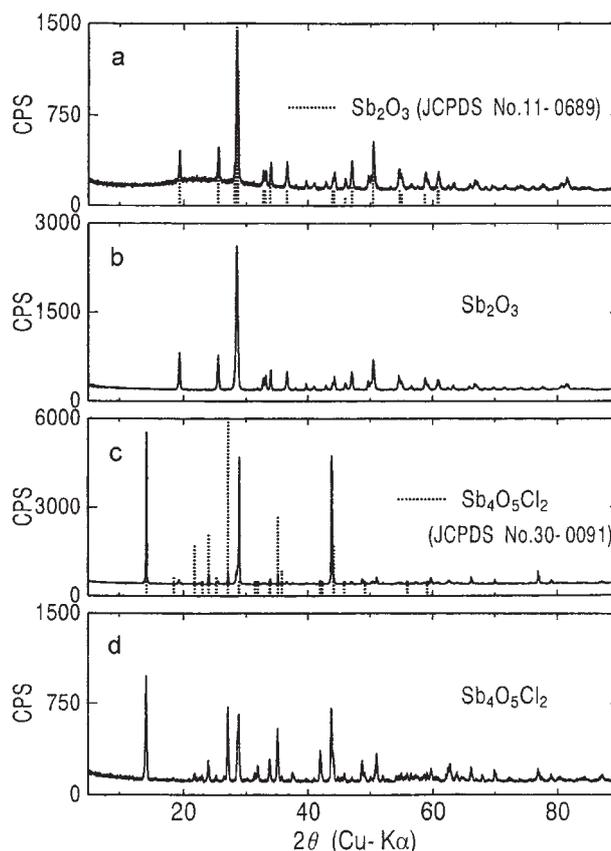


Fig. 1 X-ray diffraction patterns of Sb(III) compounds produced by equilibrating mixtures of Sb₂O₃ and HCl solution at 25°C for 30 days. HCl concentration (kmol/m^3): a 0 (water), b 0.1, c 0.12, d 5.6.

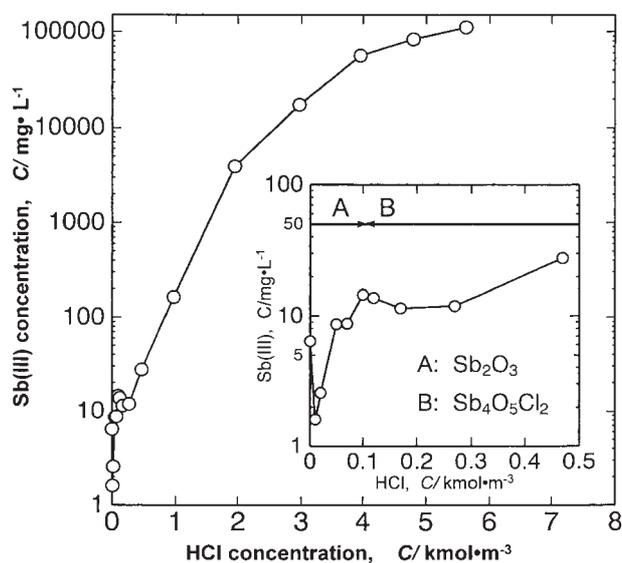


Fig. 2 Sb(III) concentration in the solution after equilibrating mixtures of Sb₂O₃ and HCl solution at 25°C for 30 days.

1.6 mg/L in Sb(III) at 0.01 M HCl, whereas the solubility of crystalline Sb₄O₅Cl₂ increases from 15 to 110 000 mg/L Sb(III) as the HCl concentration increases from 0.12 to 5.6 kmol/m^3 . It is noted that the difference between solubilities for Sb₂O₃ and Sb₄O₅Cl₂ is sufficiently large to recover antimony as Sb₂O₃ from the Sb(III)-HCl solution by hydrolysis process.

3.2 Hydrolysis of antimony(III)-hydrochloric acid solution

As stated in the previous section, Sb(III) solubility is fairly larger in HCl solution in contact with $\text{Sb}_4\text{O}_5\text{Cl}_2$ than in very diluted HCl solution where Sb_2O_3 is a solid equilibrium phase.

Then Sb_2O_3 is expected to be precipitated out by hydrolysis of Sb(III)-bearing HCl solution ($\text{HCl} > 0.12 \text{ kmol/m}^3$). As some entities of dissolved Sb(III) in such chloride solutions are likely to form stable chloro or oxychloro complex ions, the terminal concentration of Sb(III) is considered to be strongly affected by the concentration of chloride ion in the solution. Here, hydrolysis of Sb(III) ions in the solution initially containing 140 mg/L Sb(III) and 0.1 or 1.0 kmol/m^3 in HCl was conducted at 25°C by adjusting pH to individual starting values with NaOH solution and holding for desired period of time. The XRD patterns and morphology of the hydrolysis products and the terminal Sb(III) concentration of the aqueous phase were determined.

3.2.1 Formation of $\text{Sb}_8\text{O}_{10}(\text{OH})_2\text{Cl}_2$

The progress of hydrolysis of antimony(III) in the presence of 1.0 kmol/m^3 chloride ($[\text{HCl}]_0 = 1.0 \text{ kmol/m}^3$) at the starting pH of 2.0 is shown in Fig. 3, presenting the variation in pH and antimony(III) concentration of solution with time. The pH of solution stayed unchanged during the hydrolysis for 48 hours. The antimony(III) concentration of solution dropped to 50 mg/L immediately after the pH adjustment with NaOH solution to the set value 2.0, and stayed there over the first 12 hours. It is also observed that in the period of holding time over 12 hours, the Sb concentration decreased gradually toward a terminal value of 9 mg/L.

The precipitate samples were washed, dried and then submitted to XRD analysis and SEM observation. Figure 4 presents the XRD patterns of the solid products which were sampled at 10 minutes, 12 and 24 hours of hydrolysis time. The XRD data presented here reveals that the precipitates were of amorphous state at the beginning stage of hydrolysis and then converted into crystalline $\text{Sb}_8\text{O}_{10}(\text{OH})_2\text{Cl}_2$ (orthorhombic) which is reported in the JCPDS Index (No. 11-38).

The SEM images for four precipitates which were sampled at different times are compared in Fig. 5. The amorphous precipitates sampled at 10 minutes and 12 hours of holding time consist of spherical particles which are about 0.2 μm in

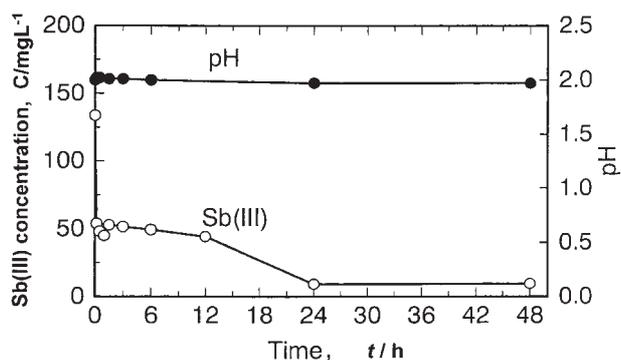


Fig. 3 Change in pH and Sb(III) concentration with time during hydrolysis in the presence of 1 kmol/m^3 of chloride at the initial pH 2.0 at 25°C.

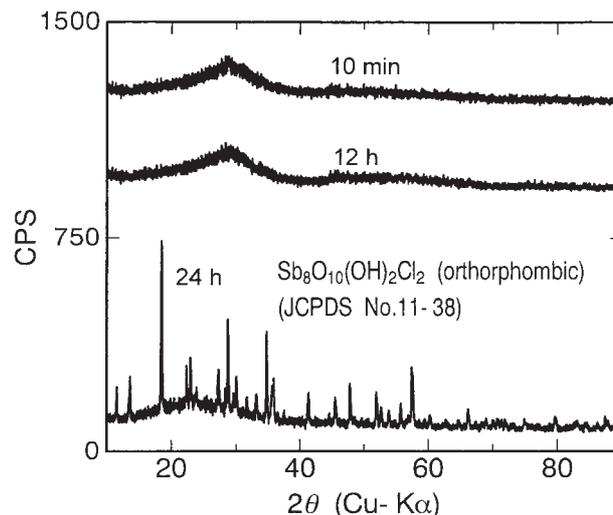


Fig. 4 Change in X-ray diffraction patterns of the precipitates with time during hydrolysis of Sb(III) in the presence of 1 kmol/m^3 of chloride at the initial pH 2.0 at 25°C.

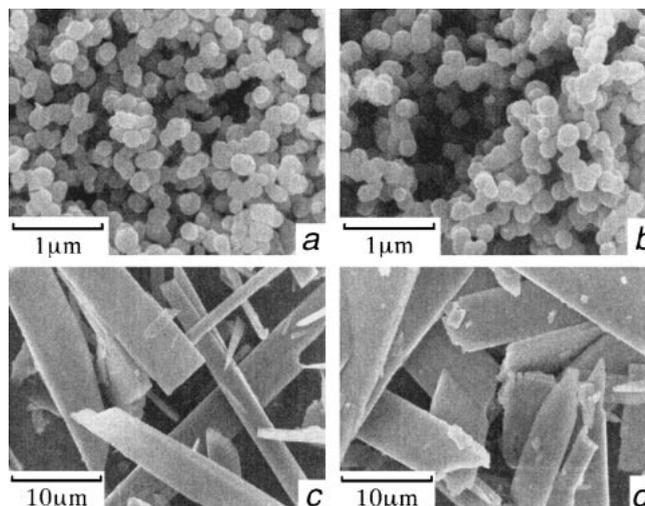


Fig. 5 Change in the particle morphology with time during hydrolysis of Sb(III) in the presence of 1 kmol/m^3 of chloride at the initial pH 2.0 at 25°C. Time: a 10 min, b 12 h, c 24 h, d 48 h.

diameter. On the other hand, the precipitate sampled at 24 hours, which was identified as $\text{Sb}_8\text{O}_{10}(\text{OH})_2\text{Cl}_2$ based on XRD data, shows a rectangular plate form without small spherical particles present. The precipitates after 48 hours show that further hydrolysis caused no more change in the shape and size of the particles.

Summarizing the data presented in Figs. 3 to 5, it is noted that, in the hydrolysis of antimony(III) at pH around 2, the antimony(III) compound with XRD patterns typical to amorphous materials is firstly precipitated in the form of small spherical particles and the antimony(III) concentration of solution stays unchanged as long as the spherical form is kept. The further hydrolysis is accompanied by the formation of crystalline $\text{Sb}_8\text{O}_{10}(\text{OH})_2\text{Cl}_2$ and a decrease in the antimony(III) concentration approaching its solubility value.

The chemical analysis for antimony by iodometry and Energy Dispersive X-ray Spectrometer (EDS) analysis for

chlorine were carried out to determine a chemical formula for the X-ray amorphous antimony(III) compound produced at the early stage of hydrolysis, and gave 75.3 mass% Sb; 5.7 mass% Cl, approximately corresponding to the theoretical values (78.62 mass% Sb; 5.72 mass% Cl) for $\text{Sb}_8\text{O}_{10}(\text{OH})_2\text{Cl}_2$. Hence, the hydrolysis reaction of antimony(III) in the presence of chloride to form $\text{Sb}_8\text{O}_{10}(\text{OH})_2\text{Cl}_2$ at pH around 2 can be represented by



3.2.2 Formation of Sb_2O_3

In solutions of a very low concentration of HCl, *i.e.* of low acidity, and alkaline chloride solutions, hydrolysis behavior of Sb(III) was determined. According to the results described in section 3.1, the probable hydrolysis product is Sb_2O_3 under these conditions.

Figure 6 shows the progress of hydrolysis of antimony(III) in the presence of 1.0 kmol/m^3 chloride, $[\text{HCl}]_0 = 1.0 \text{ kmol/m}^3$, at the starting pH of 4.5 without further pH control. The pH increases from 4.5 to 4.7 for the first 2 hours and gradually decreased toward a terminal value of 3.5. The antimony(III) concentration of solution decreases rapidly down to 85 mg/L when the neutralization starts and, after staying there for about 30 minutes, decreased with time to 19 mg/L.

The precipitates sampled at holding times of 10 minutes, 3 and 6 hours were submitted to XRD analysis and morphology observation with SEM. As shown in Fig. 7, the precipitate sampled at 10 minutes showed typical spectrum for amorphous materials and the XRD patterns of the precipitates sampled at 3 and 6 hours were matched with that in JCPDS Index No. 11-0-689 for Sb_2O_3 (orthorhombic). Chemical analysis revealed that the solid phase sampled at 10 min of holding time had a composition corresponding to $\text{Sb}_8\text{O}_{10}(\text{OH})_2\text{Cl}_2$.

SEM images of the precipitate particles which were submitted to XRD analysis are presented in Fig. 8. The precipitates showing XRD patterns for amorphous materials consist of spherical grains of size of about $0.2 \mu\text{m}$. However, the orthorhombic Sb_2O_3 particles have completely different shapes. The particles sampled at 3 hours of holding time have spherical shape of about $1 \mu\text{m}$ in diameter, while that at 6 hours turns to be blocks of stacked layers of planes with sides of about $4 \mu\text{m}$ in length. Such change in particle morphology

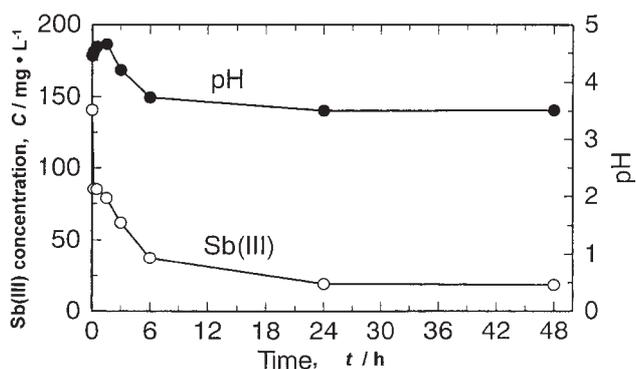


Fig. 6 Change in pH and Sb(III) concentration with time during hydrolysis in the presence of 1 kmol/m^3 at the initial pH 4.5 at 25°C.

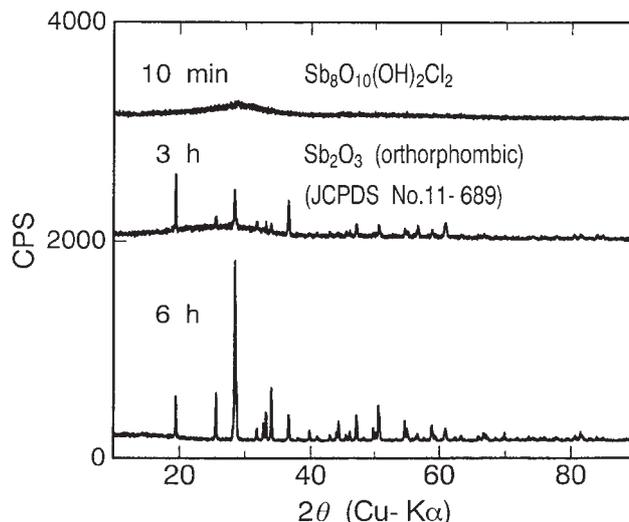


Fig. 7 Change in X-ray diffraction patterns of the precipitate with time during hydrolysis of Sb(III) in the presence of 1 kmol/m^3 of chloride at the initial pH 4.5 at 25°C.

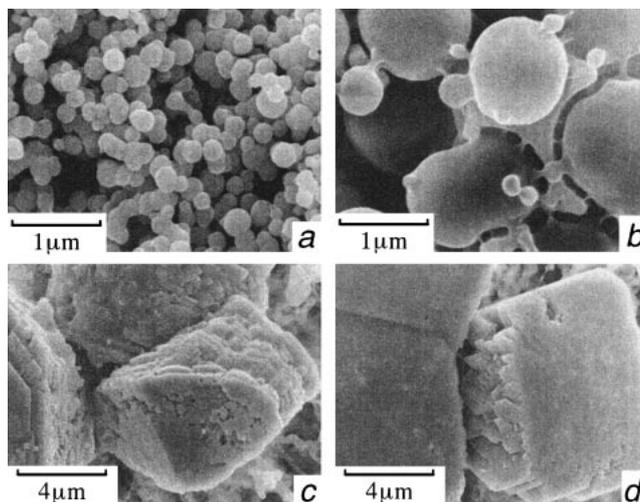


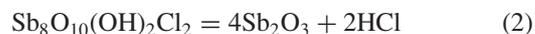
Fig. 8 Change in the particle morphology with time during hydrolysis of Sb(III) in the presence of 1 kmol/m^3 at the initial pH 4.5 at 25°C. Time: a 10 min, b 3 h, c 6 h, d 24 h.

with holding time is in excellent correspondence with change in the intensity ratio of main XRD peaks of Sb_2O_3 samples.

Based on these results, in the course of hydrolysis of antimony(III) in the presence of chloride in solution to form Sb_2O_3 as a final solid phase, it is considered that $\text{Sb}_8\text{O}_{10}(\text{OH})_2\text{Cl}_2$ (amorphous) is precipitated first according to the reaction (1) in which hydrogen ion is consumed



and then $\text{Sb}_8\text{O}_{10}(\text{OH})_2\text{Cl}_2$ is converted to Sb_2O_3 (orthorhombic) according to the reaction (2) in which hydrogen ion is liberated.



3.3 Effect of pH on the hydrolysis

Solution pH is one of the main factors governing the precipitation of various compounds from aqueous solutions

by hydrolysis. The typical XRD patterns and SEM images of the precipitates produced by 3-hour hydrolysis of antimony(III) in the presence of 1.0 kmol/m^3 chloride, $[\text{HCl}]_0 = 1.0 \text{ kmol/m}^3$, at different starting pH are compared in Figs. 9 and 10, respectively. For the precipitates produced at pH 1.5

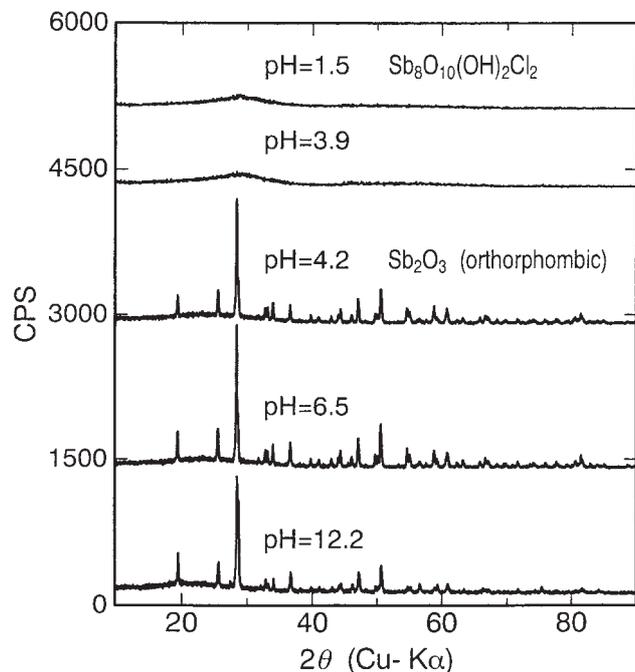


Fig. 9 X-ray diffraction patterns of the precipitates produced at different pHs by 3-hour hydrolysis of Sb(III) in the presence of 1 kmol/m^3 at 25°C .

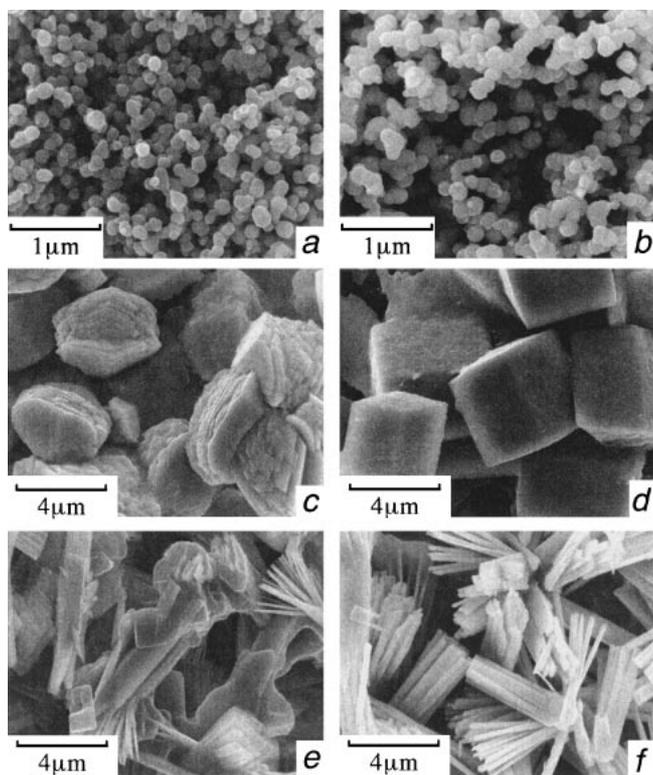


Fig. 10 SEM images of the precipitates produced at different pHs by 3-hour hydrolysis Sb(III) in the presence of 1 kmol/m^3 at 25°C . pH: a 1.5, b 3.9, c 4.2, d 6.5, e 7.7, f 12.2.

and 3.9, the XRD patterns without sharp and intense peaks were observed. Chemical analysis for antimony and EDS analysis for chlorine indicated these solids to be $\text{Sb}_8\text{O}_{10}(\text{OH})_2\text{Cl}_2$. The particles of both amorphous precipitates consist of fine spherical grains (Figs. 10a and b).

In the XRD patterns for the precipitates produced at pH 4.2, 6.5 and 12.2, the peaks for Sb_2O_3 (orthorhombic) were observed to have the same peak angles and the similar peak intensities. Also the SEM images show that these Sb_2O_3 precipitates consist of grains with well developed shape and, however, the shapes of their particles are significantly changing with pH. It can be seen that a rise in pH leads to preferential growth of some particular crystal planes which results in the morphology presented in Figs. 10e and f. The mechanisms for their crystal growth are not clear at this moment. From the above, it is noted that in 3-hour hydrolysis of antimony(III) in the presence of 1.0 kmol/m^3 chloride, the reaction product is $\text{Sb}_8\text{O}_{10}(\text{OH})_2\text{Cl}_2$ (amorphous) at $\text{pH} < 3.9$ and Sb_2O_3 (orthorhombic) at $\text{pH} > 4.2$.

Figure 11 shows the concentration of antimony(III) remaining in the solutions after 3, 24 and 48-hour hydrolysis at different pH and the solid phase identified by XRD: \circ stands for $\text{Sb}_8\text{O}_{10}(\text{OH})_2\text{Cl}_2$ (amorphous), \square for Sb_2O_3 (orthorhombic) and \bullet for $\text{Sb}_8\text{O}_{10}(\text{OH})_2\text{Cl}_2$ (orthorhombic). In this figure, the thermodynamically predicted solubility of Sb_2O_3 (orthorhombic) based on the standard free energy data listed in NBS Technical Note is also shown as a function of pH by a dotted line. This predicts that Sb_2O_3 is an amphoteric oxide which dissolves in water. In solutions of pH between 2 to 11, the undissociated antimonious acid (HSbO_2) is considered to be the most probable entity and the solubility is equal to 7 mg/L in Sb(III), while the formation of antimonyl ion (SbO^+) in a pH range below 2 and antimonite ion (SbO_2^-) in high pH zone above 11 are responsible for higher solubility of Sb(III). For 3-hour hydrolysis, the

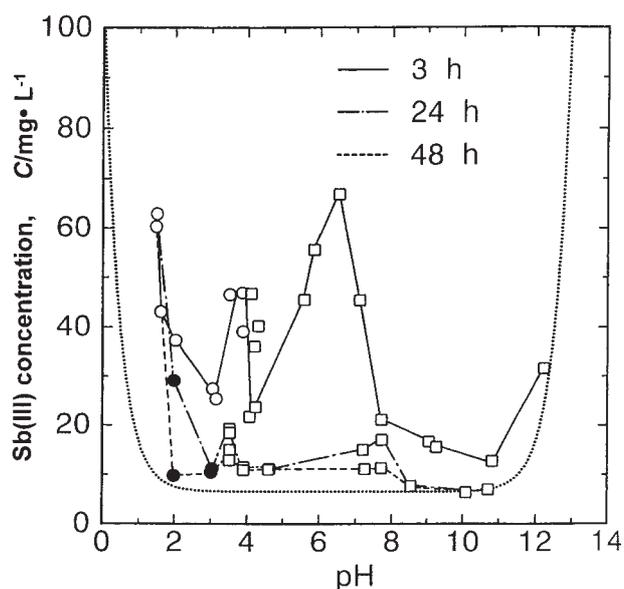


Fig. 11 Sb(III) concentration of the solution at different pHs after 3-, 24- and 48-hour hydrolysis in the presence of 1 kmol/m^3 of chloride at 25°C . \circ $\text{Sb}_8\text{O}_{10}(\text{OH})_2\text{Cl}_2$ (amorphous), \bullet $\text{Sb}_8\text{O}_{10}(\text{OH})_2\text{Cl}_2$ (orthorhombic), \square Sb_2O_3 (orthorhombic).

concentration of antimony(III) remaining in the solution decreased with a rise of pH to around 25 mg/L at pH 3.0 and then increases to 48 mg/L with a rise in pH toward an invariant point where a reaction product changes from $\text{Sb}_8\text{O}_{10}(\text{OH})_2\text{Cl}_2$ (amorphous) to Sb_2O_3 (orthorhombic). At pH around 4.2, the Sb(III) concentration falls from 48 mg/L down to 21 mg/L owing to the formation of Sb_2O_3 but increases again with a rise in pH due to the slower precipitation of Sb_2O_3 of cubic shape with smooth surface. As the particle shape changes from cube to thin plate, the residual antimony(III) concentration decreases over a pH range 6.5 to 11.0. The residual antimony(III) concentration at pH above 11 where SbO_2^- is a dominant species lies on the predicted solubility of Sb_2O_3 .

At pH 1.5, amorphous $\text{Sb}_8\text{O}_{10}(\text{OH})_2\text{Cl}_2$ is still detected and the concentration of antimony(III) in the solution was as high as 60 mg/L even after 48-hour hydrolysis. In a pH range of 2.0 to 3.5, the hydrolysis product was orthorhombic $\text{Sb}_8\text{O}_{10}(\text{OH})_2\text{Cl}_2$ after 24 and 48 hours and the invariant point was observed at pH 3.5. At pH exceeding 3.5, orthorhombic Sb_2O_3 is a dominant solid species and the antimony(III) concentration approached a level of the thermodynamically predicted solubility after 24 hours of holding time.

The effect of the presence of chloride on the hydrolysis reaction is demonstrated in Fig. 12. Here, the observed

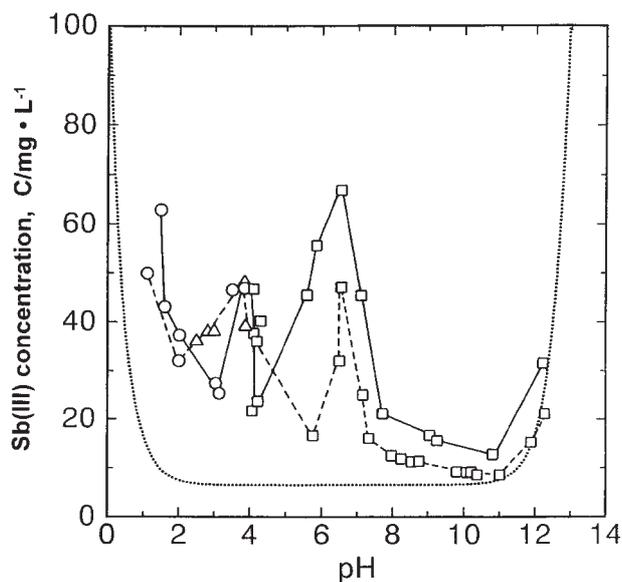


Fig. 12 Sb(III) concentration of the solution at different pHs after 3-hour hydrolysis in the presence of 0.1 kmol/m³ and 1 kmol/m³ of chloride at 25°C. ○ $\text{Sb}_8\text{O}_{10}(\text{OH})_2\text{Cl}_2$ (amorphous), ● $\text{Sb}_8\text{O}_{10}(\text{OH})_2\text{Cl}_2$ (orthorhombic), □ Sb_2O_3 (orthorhombic). Chloride concentration (kmol/m³): --- 0.1, — 1.

Sb(III) concentrations are compared at two levels of chloride concentrations, 0.1 and 1.0 kmol/m³ for 3-hour hydrolysis. In a low pH range, *i.e.* acidic zone, the effect of the chloride concentration is not very clear, but in alkaline solutions, the presence of chloride at higher concentration give rise to stabilization of dissolved antimony entities in the solution, leading to higher concentration of antimony in the solution of higher chloride concentration.

4. Summary

Equilibrium of Sb(III)-HCl-H₂O system and hydrolysis of Sb(III)-HCl solution have been investigated.

- (1) Solid phase in Sb(III)-HCl-H₂O system at 25°C was identified by XRD method in HCl concentration range up to 6 kmol/m³. Two solid antimony compounds, Sb_2O_3 (HCl < 0.1 kmol/m³) and $\text{Sb}_4\text{O}_5\text{Cl}_2$ (HCl > 0.12 kmol/m³) are detected to be the stable solid phase.
- (2) The observed solubility of Sb_2O_3 was 6.4 mg/L in water and has a minimum value of 1.6 mg/L at HCl concentration 0.01 kmol/m³. The concentration of Sb(III) of the solution in contact with $\text{Sb}_4\text{O}_5\text{Cl}_2$ increases from 15 mg/L at 0.12 M HCl to 110000 mg/L at 5.6 kmol/m³ HCl.
- (3) Hydrolysis of Sb(III) species in the presence of chloride over a pH range of 1.1 to 12.3 at 25°C has been studied. In the process of hydrolysis at pH around 2, amorphous particles turns to crystalline $\text{Sb}_8\text{O}_{10}(\text{OH})_2\text{Cl}_2$ without detectable pH change. At pH around 4.5, the first precipitate, $\text{Sb}_8\text{O}_{10}(\text{OH})_2\text{Cl}_2$, is converted into Sb_2O_3 .
- (4) Through the hydrolysis reactions, the Sb(III) concentration of the solution approached the thermodynamically predicted solubility at any pH. However, at pH around 4 and 6.5, the hydrolysis reaction is found to be very slow. The presence of chloride trends to retard reaction of hydrolysis in alkaline solutions.

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