

Supplementary Information

**Hydrothermal synthesis of one-dimensional yttrium hydroxide particles by a two-step alkali-addition method**

**Fumiyuki Shiba,<sup>\*a</sup> Tosuke Tamagawa,<sup>a</sup> Takashi Kojima<sup>b</sup> and Yusuke Okawa<sup>a</sup>**

<sup>a</sup> Graduate School of Advanced Integration Science, Chiba University

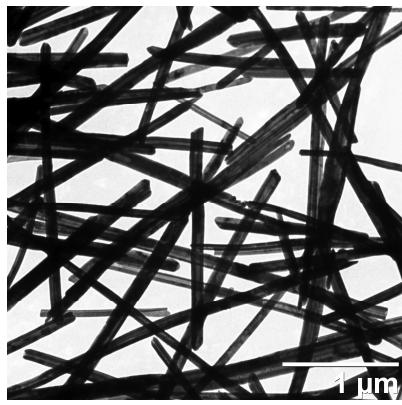
<sup>b</sup> Graduate School of Engineering, Chiba University

1-33 Yoyoicho, Inageku, Chiba 263-8522, Japan

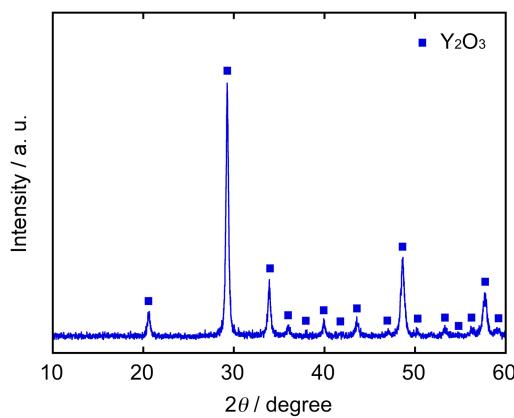
E-mail: shiba@faculty.chiba-u.jp

## 1. Transformation of the 1D $\text{Y(OH)}_3$ particles into 1D $\text{Y}_2\text{O}_3$ particles by calcination

The 1D  $\text{Y(OH)}_3$  particles prepared under the typical condition were calcined to transform them into  $\text{Y}_2\text{O}_3$ . The freeze-dried powder of the 1D  $\text{Y(OH)}_3$  particles were placed in a porcelain crucible and heated to dehydrate in a muffle furnace at 500 °C for 2 h. The TEM image in Fig. S1 shows the 1D shape is fairly maintained even after the dehydration reaction. The XRD pattern (Fig. S2) indicates the  $\text{Y(OH)}_3$  is completely converted to cubic  $\text{Y}_2\text{O}_3$  (JCPDS 71-0049) by the calcination process.



**Fig. S1** TEM image of the 1D  $\text{Y}_2\text{O}_3$  particles calcined at 500 °C for 2 h from the 1D  $\text{Y(OH)}_3$  particles.



**Fig. S2** XRD pattern of the 1D-particles after the calcination process at 500 °C for 2 h. All the peaks are matched with the standard ones of cubic  $\text{Y}_2\text{O}_3$  (JCPDS 71-0049).

## 2. Determination procedures of $\text{Y}^{3+}$ and $\text{Cl}^-$ ions in the supernatant solution

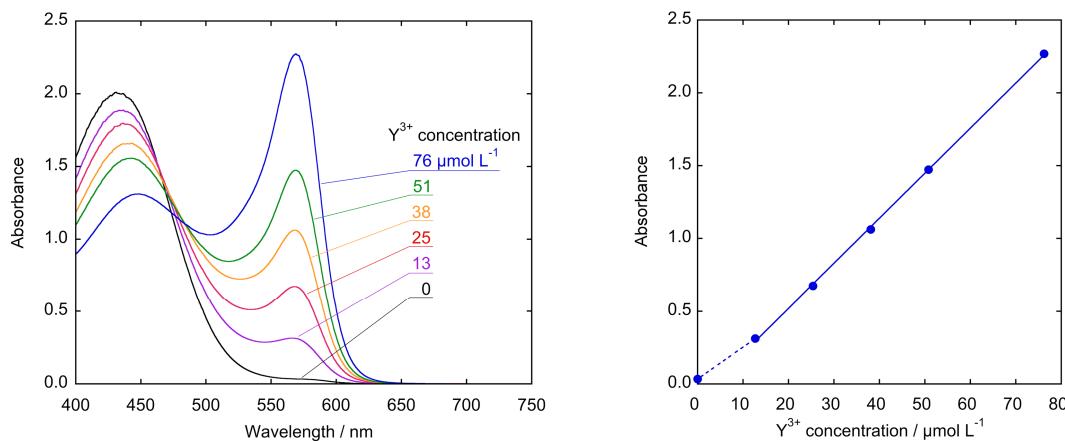
### 2. 1. Sample preparation

For each condition of  $V_{\text{NaOH}}$ , this volume of NaOH solution ( $1 \text{ mol L}^{-1}$ ) was added into 14 mL of  $\text{YCl}_3$  solution ( $0.071 \text{ mol L}^{-1}$ ) at 25 °C under magnetic stirring. After separating the precipitate by centrifuge at 10000 rpm ( $12000 \times g$ ) for 30 min, the supernatant solution was carefully collected from the centrifugal tube.

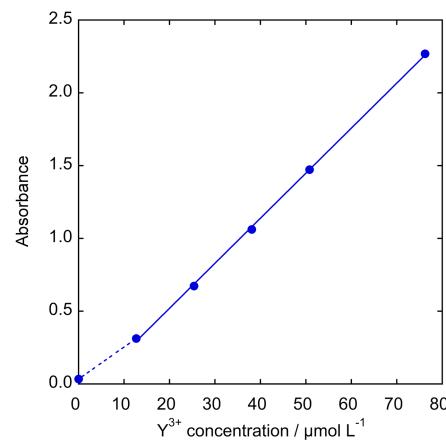
## 2.2. Colorometric determination of $\text{Y}^{3+}$ ion

To determine the  $\text{Y}^{3+}$  ion content, xylenol orange (XO), purchased from Dojindo, was employed as the coloring agent of  $\text{Y}^{3+}$  ion.<sup>S1</sup> Into a volumetric flask (20 mL), 2 mL of acetate buffer (pH 4.6) that contained 0.25 mol L<sup>-1</sup> of  $\text{CH}_3\text{COOH}$  and 0.25 mol L<sup>-1</sup> of  $\text{CH}_3\text{COONa}$ , a certain volume of  $\text{Y}^{3+}$  standard solution or sample one, and 2 mL of XO solution ( $1 \times 10^{-3}$  mol L<sup>-1</sup>) were introduced in this order. After adjusting to 20 mL with distilled water, the absorption spectrum was measured by a spectrophotometer (Shimadzu Multispec-1500).

Since the initial  $\text{YCl}_3$  concentration is as high as 0.071 mol L<sup>-1</sup>, the sample supernatant solution was suitably diluted with distilled water to being applied to determination for the conditions  $V_{\text{NaOH}} = 0 \sim 2$  mL, while it was used as-is for  $V_{\text{NaOH}} \geq 2.5$  mL.



**Fig. S3** Absorption spectra of  $\text{Y}^{3+}$  standard solution colorized with XO in an acetate buffer (pH 4.6).



**Fig. S4** Relationship between  $\text{Y}^{3+}$  concentration and absorbance at 568 nm.

Fig. S3 shows the dependence of absorption spectrum on the  $\text{Y}^{3+}$  concentration. Along with the increase of  $\text{Y}^{3+}$  concentration, absorbance at 568 nm, attributed to  $\text{Y}^{3+}$ -XO complex, is increased, while absorbance at 430 nm, attributed to XO, is decreased. As isobestic point is not found in Fig. S3, more the two kinds of complex species could be formed. However, the

absorbance at 568 nm indicates a sufficient linearity to  $\text{Y}^{3+}$  concentration at least in the range of  $13 \sim 76 \mu\text{mol L}^{-1}$  (Fig. S4).

### 2.3. Potentiometric titration for determination of $\text{Cl}^-$ ion

To determine the  $\text{Cl}^-$  content, potentiometric titration using  $\text{AgNO}_3$  solution was carried out. Into 30 mL of distilled water, 2 mL of an acetate buffer (pH 4.6) that contains 0.25 mol  $\text{L}^{-1}$  of  $\text{CH}_3\text{COOH}$  and 0.25 mol/L of  $\text{CH}_3\text{COONa}$  and 2 mL of sample solution were added, where the acetate buffer was used to avoid possible formation of  $\text{Ag}_2\text{O}$  precipitate in high pH samples. By measuring  $\text{Ag}^+$  potential with a  $\text{Ag}_2\text{S}$ -coated Ag electrode, 0.05 mol  $\text{L}^{-1}$  of  $\text{AgNO}_3$  was introduced drop-by-drop from a bullet to determine the equivalent point of each sample solution.

### Reference

- S1 A. Hrdlička, J. Havel, C. Moreno and M. Valiente, *Anal. Sci.*, 1991, **7**, 925-929.