STUDIES ON NIIOBIUM AND TANTALUM

Part III. Tartratoniobates of Alkaline Earth and Other Metals

By N. R. Srinivasan

(Indian Institute of Science, Bangalore)

Received February 1, 1950
(Communicated by Sir C. V. Raman)

INTRODUCTION

The niobates of the alkaline earth and other metals have been found to be insoluble materials. The several niobates of the alkaline earth metals, got by fusion of the metal carbonates or oxides with niobium pentoxide, were insoluble as also the niobates of zinc, magnesium and other heavy metals (Jander, 1931). Pierre Sue (1937) found that, the alkaline earth niobates, obtained as precipitates by the double decomposition of a salt of the metal with a soluble niobate, were impure and colloidal. Balke and Smith (1908) prepared the insoluble metaniobates of the alkaline earth, aluminium, magnesium, cadmium and other metals. Many of the niobates were prepared during the present work and were found to be insoluble and unreactive towards tartaric acid solutions. Hence the tartratoniobates of these metals were sought to be prepared in a different manner.

EXPERIMENTAL

Method of Preparation

Since many of the niobates of the heavy metals were found to be unreactive towards tartaric acid, attempts were made to prepare the compounds by other methods. When a solution of 2-potassium-2-tartrato-2-niobate was treated with soluble salts of metals, characteristic precipitates were obtained as indicated below:

Potassium tartratoniobate Soln. + CaCl₂ Soln. → White ppt.

" " " + Sr(NO₃)₂ " " → "
" " " + Ba(NO₃)₂ " " → "
" " " + CuSO₄ " " → Light green ppt.
" " " + MgCl₂ " " → White ppt.
" " " + ZnCl₂ " " → "
" " " + CdSO₄ " " → "

381
Potassium tartratoniobate Soln. + $\text{Al}_2(\text{SO}_4)_3$ Soln. → White ppt.

$\ldots + \text{PbCl}_2 \ldots \rightarrow \ldots$

$\ldots + \text{Th} (\text{NO}_3)_4 \ldots \rightarrow \ldots$

$\ldots + \text{Cr} (\text{NO}_3)_3 \ldots \rightarrow \text{Green ppt.}$

$\ldots + \text{FeSO}_4 \ldots \rightarrow \text{Dirty white ppt.}$

$\ldots + \text{Co} (\text{SO}_4)_2 \ldots \rightarrow \text{Light orange ppt.}$

In each case the precipitate was filtered and both the precipitate and the filtrate examined. The first case will serve as an illustration. In this case, the filtrate revealed the absence of both niobium and tartrate; the precipitate was found to contain calcium, niobium and tartrate. Hence it is possible that they exist together as a definite compound. The probability that the precipitate might be composed of a mixture of calcium niobate and calcium tartrate was eliminated by the following experiments:

(i) The precipitate was extracted with hot water, in which it was slightly soluble, and the extract answered all the tests for calcium, niobium and tartrate; calcium niobate was insoluble in water.

(ii) The precipitate was insoluble in cold acids, but dissolved to give clear solutions in hot concentrated hydrochloric acid. The solution precipitated niobic acid on dilution. Calcium niobate was insoluble in concentrated acids.

(iii) The precipitate was soluble in cold or hot dilute alkali hydroxides to clear solutions whereas calcium niobate was insoluble even in hot concentrated alkali hydroxides. Therefore it can be concluded that the compound obtained is the tartratoniobate of calcium; and by similar reasoning it can be seen that the characteristic precipitates that are obtained are the tartratoniobates of the various metals.

**Calcium-2-tartrato-2-niobate**

The preparation of this compound was undertaken by the double decomposition of potassium tartratoniobate and calcium chloride or calcium acetate. $\text{K}_2\text{O} \cdot \text{Nb}_2\text{O}_5 \cdot (\text{C}_4\text{H}_4\text{O}_6)_2 \cdot 10 \text{H}_2\text{O}$ was dissolved in warm water and a 10% solution of calcium chloride added drop by drop with vigorous stirring. A white precipitate was formed which settled down quickly; the solution was cooled in ice, filtered, washed with ice-cold water and dried. The analysis presented several difficulties and the final procedure adopted was the following:—