The chemical separation of Nb in presence of Fe

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I. Introduction

Nb-94 is the fission product of uranium and plutonium. It is also produced by neutron activation reaction of stable niobium, which is present in the structural component of nuclear reactor pressure vessel and the cladding nuclear fuel. Because its fission yield is very low, neutron activation is the major source in the formation of Nb-94. Due to its long half-life and relative high inventory in nuclear waste, an accurate radioactivity measurement of Nb is required. Typically, the radioactive concentration of Nb is too low to detect and therefore an individual separation of Nb-94 from co-existing nuclides and matrix element is necessary before a measurement of its radioactivity.

II. Experiment and Results

All chemicals were used without further purification and purchased from commercial sources as follow: Fe standard solution (AccuStandard), Nb standard solution (AccuStandard), hydrochloric acid (HCl, Merck), nitric acid (HNO_3, Merck), and methyl isobutyl ketone (MIBK, Wako Pure Chemical Product). The amount of Fe and Nb was determined by ICP-AES (Horiba Jobin Yvon, Activa M).

A hydroxide precipitation, MIBK extraction, and dissolution in HNO_3 were applied to separate Nb from Fe. For a hydroxide precipitation, a sample was prepared by addition of 10% NH_4OH into a centrifuge tube containing 20 mL of 1 mg/mL Fe standard solution and 2 mL of 1 mg/mL Nb standard solution. To check a pH-dependent separation, a different volume of 10% NH4OH was added. According to ICP-AES results, a hydroxide precipitation is not suitable for a separation of Nb from Fe because Nb is co-precipitated during the formation of iron hydroxide.

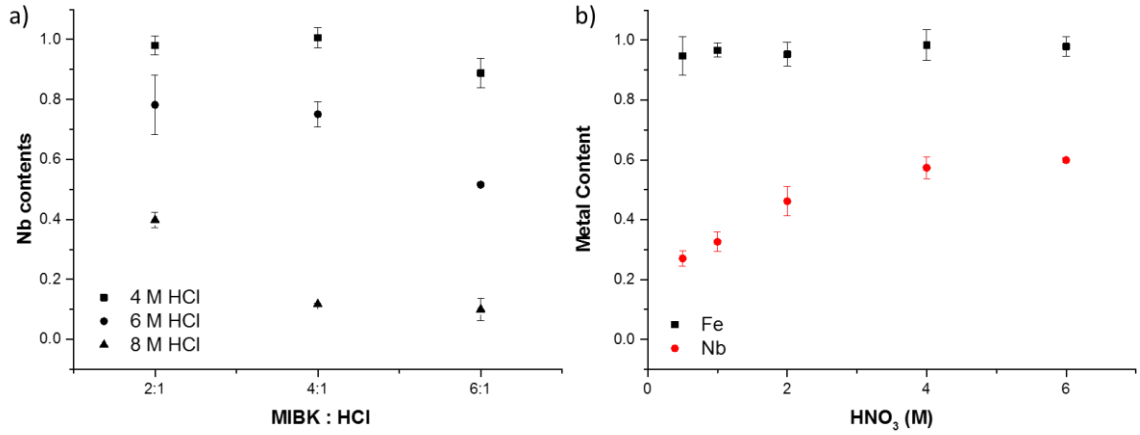

Figure 1 a) Nb contents in 4 M, 6 M, and 8 M HCl after MIBK extraction, b) Fe and Nb contents in a solution at several concentrations of HNO_3

For a MIBK extraction, a sample solution containing Fe and Nb was evaporated to dryness and dissolved in HCl. The concentration of HCl and a volume ratio of MIBK to HCl were adjusted to find the best separation condition. According to Figure 1a, 2MIBK: HCl and 4MIBK: HCl show that the content of Nb is 97.9% and 100% at 4 M HCl, respectively.

For a dissolution in HNO_3, a sample solution was evaporated to dryness and dissolved in several concentrations of HNO_3 to determine a separation condition. Figure 1b showed that the content of Nb was dependent on the concentration of HNO_3 while Fe was independent in a solution. As a result, Nb was isolated from Fe as an insoluble matter due to a different solubility of Fe and Nb in HNO_3. However, there was no complete separation of Nb from Fe in a dissolution condition.

III. CONCLUSIONS

In this work, the separation method of Nb from Fe including a hydroxide precipitation, MIBK extraction, and dissolution in HNO_3 was demonstrated. MIBK extraction showed the best condition for separation of Nb from Fe among three methods.

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