Comparison of ^{55}Fe Recovery Results by Gravimetric and Spectrometric Analysis

Hyeon Jeong Oh*, Jung Bo Yoo, Hong Joo Ahn, and Kwang Yong Jee
Nuclear Chemistry Research Division, Korea Atomic Energy Research Institute, 111 Daedeok-daero-989,
Yuseong-gu, Daejeon, Republic of Korea
*ohj@kaeri.re.kr

1. Introduction

According to the Notice No. 2015-4 from the Nuclear Safety and Security Commision (NSSC), it should be know the clarify radioactivities of gross α as well as over 10 radionuclides such as ^{3}H, ^{14}C, ^{129}I, ^{60}Co, ^{55}Fe, ^{59,63}Ni, ^{90}Sr, ^{94}Nb, ^{99}Tc and ^{137}Cs. Among them, ^{55}Fe, ^{59,63}Ni, ^{94}Nb, ^{99}Tc nuclides have to solely separate because of their low energy beta- and X-ray emitting radiochemical characteristics. What if it does not separate those nuclides individually, detecting signals show the overlapping circumstance. For that reason, development of chemical separation process is an important issue. In fact, KAERI (Korea Atomic Energy Research Institute) has investigated the analytical method regarding sequential chemical separation procedure for those radionuclides from radioactive waste samples [1].

To acquire the accurate results, it is essential to manage the determination techniques of individual radionuclides. There are two representative ways to determine recovery results. The first one is a gravimetric analysis. Gravimetric analysis is the process of producing and weighing a compound or element in as pure form as possible after some form of chemical treatment has been carried out on the substances to examined. It is one of the most accurate and precise analytical skills of macro quantitative method. Another one is ICP-AES (Inductive Coupled Plasma-Atomic Emission Spectrometry) analysis. It is very rapid (within 1 ~ 2min) simultaneous multi-element analysis. ICP-AES has good precision (0.5~1%) and accuracy as well as good reliability and inherent safety of modern instrumentation.

Herein, we have focused on comparing the recovery results of ^{55}Fe by gravimetric method and spectrometric method. To compare the recovery results based on the measured techniques, we separated the ^{55}Fe nuclide from the synthetic waste samples. The gravimetric analytical results are in good agreement with ICP-AES analysis.

2. Experimental section

2.1 Reagents

All the chemicals were purchased from commercial suppliers with analytical reagent grade. The Fe standard solution with 10,000 mg/L was purchased from Accustandard, USA. HF (48%), ammonia solution (25%) were purchased from MERCK (Germany).

2.2 Separation process

Radioactive waste sample solutions were prepared according to the previously procedure. In brief, from 1.0 mL to 5.0 mL of Fe carrier solutions (10 mg/mL) was added into a 50 mL plastic beaker. To evaporate the acidic solutions, the beaker was placed on the hot plate for 3 h. And the dried product was dissolved in 2 mL of 1 M HNO_3. After adjusting its pH to 4.0 ~ 5.0 with 10% NH_4OH and diluting this solution by adding deionized water. The diluting solution were passed through the anion exchange resin column. 100 mL of 0.1 M NH_4-oxalate solution (pH 4.0 ~ 5.0) and 25 mL of 4 M HF solution were sequentially poured into the column. After that, Fe nuclide was collected in a 200 mL plastic beaker with 65 mL of saturated boric acid by adding the 60 mL of 4 M HF solution [2]. This solution with Fe nuclide not only was measured with an ICP-AES but also a gravimetric method. To gain a precipitate, 25% NH_4OH solution was added into the 200 mL beaker. The brown precipitate was made as an amorphous

$Fe(OH)_3$ phase. It was centrifuged at 4,000 rpm for 5 min. The precipitate was transferred to a 15 mL of alumina crucible and evaporated on a hot plate with 100°C for 3 h. The dried precipitate was annealed at 800°C for 30 min to get a crystalline Fe_2O_3[3].

3. Results and discussion

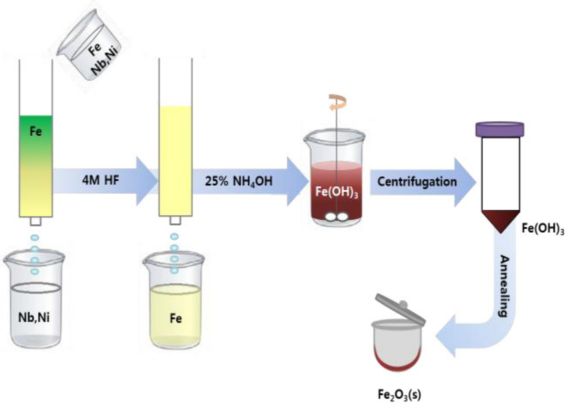

Fig. 1. Schematic illustration of ^{55}Fe chemical separation process. Bio Rad AG 1×8 (100~200 mesh) anion exchange resin was packed in the Econo-Pac Disposable Chromatography Column (20 mL, Bio Rad, U.S.A.).

A separation process of ^{55}Fe is shown in Fig. 1. When the solution with those nuclides were passed through the anionic exchange resin column, two nuclides (Fe, Nb) were placed in the column with green color which is correspond to Fe element. To collect the Fe nuclide, 4 M HF was put into the column. Fe-oxalate anion bonds were break by adding HF, the effluent was collected in a 200 mL beaker. The amount of Fe standard solutions were changed from 10 mg to 20 mg, 30 mg, 40 mg and 50 mg, respectively. In the case of gravimetric analysis, the chemical yields were 95.7±3%, 93.2±1%, 89.7±6%, 87.2±1% and 84.8±8%. When the ICP-AES was used as a tool, the chemical yields were 92.6±1%, 94.7±3%, 88.0±3%, 83.9±6% and 81.5±2%. When the amount of Fe carrier was increased, the chemical yields were decreased. This results indicate that the resin has its own capacity. In this case, when the amount of anion exchange resin was 10 mL, the maximum ability to capture the Fe moiety is about 20 mg.

Table 1. Calculated chemical Yields for ^{55}Fe samples measured by gravimetric and ICP-AES analysis. The amount of Fe standard solutions were changed from 10 mg to 20 mg, 30 mg, 40 mg and 50 mg, respectively

	Gravimetric Method	**ICP-AES Analysis**
10 mg	95.7±3%	92.6±1%
20 mg	93.2±1%	94.7±3%
30 mg	89.7±6%	88.0±3%
40 mg	87.2±1%	83.9±6%
50 mg	84.8±8%	81.5±2%

4. Conclusion

We have carried out comparison of the recovery results of ^{55}Fe by gravimetric method spectrometric (ICP-AES) analysis. To define the recovery result above two techniques, we separated the ^{55}Fe nuclide from the synthetic waste samples by an extraction chromatography. Based on the above results, the gravimetric analytical results are well matched with spectrometric results. To verify those recovery results, further study is still required.

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