

Analytical procedure for swipe samples

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1. Introduction

The analytical method for a swipe sample was introduced as one of the important tools for the safeguards system based on the program 93+2 of the IAEA. Its aim is to find undeclared nuclear activities by analyzing isotopic fingerprints of trace amounts of uranium and/or plutonium in swipe samples taken from nuclear facilities [1]. In particular, the uranium isotopic ratio of the individual particles in the samples reflects their operation history. TIMS combined with a fission track method has been used for the isotopic ratio determination of individual uranium particles. The swipe samples were covered with a sheet of Lexan track detector and then irradiated with thermal neutrons. The particle containing fissile radionuclides can be detected by analyzing the fission tracks created on the Lexan track detector. And then, the individual particles were loaded onto a rhenium filament for a determination of the isotopic ratio by using TIMS.

The nuclear chemistry research division (NCRD) in KAERI established the analytical procedure to determine the uranium and plutonium contents in swipe samples.

2. Methods and Results

In this section, several steps of the analytical method are described and the cotton swipe and necessary documents are shown in Fig. 1.



Figure 1. Cotton swipe samples.

2.1 γ spectrometry

The first action taken in the NCRD laboratory is to screen the swipe samples for γ activity by using a γ -spectrometer. This is a low-background γ -spectrometer with a coaxial Ge detector and Pb shielding. Provided that enough counts are collected, the spectrum is evaluated and a radioactivity is reported in Bq/sample

for the radioisotopes such as Am, Cs, U by using the measured detector efficiency.

2.2 Alpha Track Method

The swipe samples were placed in close contact with a piece of LR-115 detector cut to the same size of the swipe sample and fixed with tape. The prepared samples were contacted for one day. After being contacted, the detector was removed and etched in a 2.5M NaOH solution for 160 minutes at 40 °C in a shaking water bath. The alpha tracks formed on the detector were observed with an optical microscope as shown in figure 2. The alpha track method can detect U, Pu, Am, Th. Especially, 10ng of enrichment uranium (> 20% ^{235}U) was detected by this method

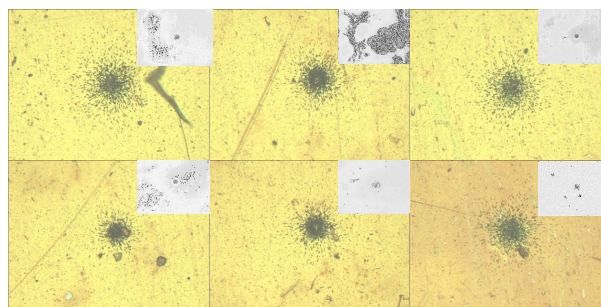


Figure 2. The alpha track images of uranium particles in the swipe sample.

2.3 Ashing of swipe samples

After a fission track test, the swipe sample was placed into a porcelain crucible and heated up to 600 °C for 3 hours in a muffle furnace. The sample was kept at 600 °C for 4 hours before a cooling down.

2.4 Fission track

The ashing products of the swipe samples were dispersed onto a Lexan plate by using the Collodion solution and placed in close contact with another piece of Lexan detector of the same size by fixing it with a plastic tape. The prepared samples were irradiated with thermal neutron in the HANARO reactor. After being irradiated, the Lexan detector was removed and then etched in a 6.25M NaOH solution for 10 minutes at 60 °C in a shaking water bath. The fission tracks formed on the detector observed by an optical microscope are shown in Figures 3 and 4.

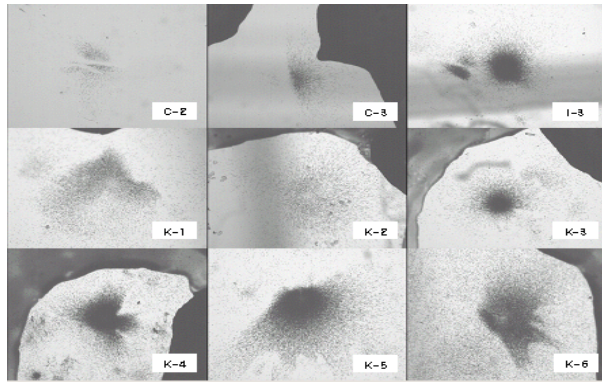


Figure 3. The fission track images of uranium particles in the swipe sample.

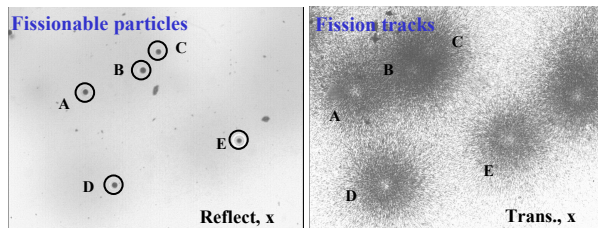


Figure 4. The fission track images as a mirror image for the uranium particles in the swipe sample.

2.5 Resin bead

After alpha or fission track tests, an interesting region in the swipe sample was cut and treated with HNO_3 for a dissolution. The resin bead was applied to that solution for a U and Pu separation and transferred to TIMS for an isotopic measurement.

2.4 TIMS

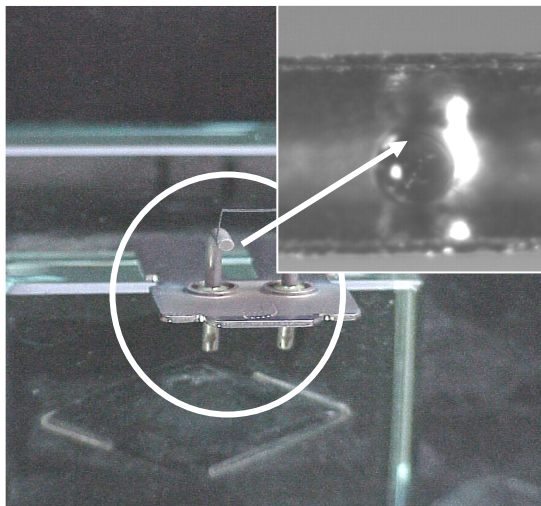


Figure 3. The resin bead on a Re filament for TIMS

A resin bead technique was developed for an isotopic ratio measurement by TMS. Figure 5 shows a resin bead fixed on the Re filament of the TIMS.

3. Conclusion

Analytical method for swipe samples by using γ -spectrometry, an alpha track method, a fission track method and a resin bead technique has been established in NCRD at KAERI. This method was applied to a swipe sample for a screening of swipe samples.

For the development of this method, we synthesized the standard uranium-containing SiO_2 particles [2], and they were utilized throughout this work. A homemade computer program (UFT-DB v.2) was used for the screening of the swipe samples. In addition to the fission track analysis, an alpha track technique was also applied for the screening of the swipe samples, whose theoretical evaluation was discussed in a previous work [3].

As a conclusion, the analytical procedure developed in NCRD by using the alpha track technique combined with other techniques such as a γ -spectrometer, fission track analysis and resin bead technique can be useful for a screening of swipe samples. This method can be applied to the micro analysis of a swipe sample by using TIMS for a determination of the isotopic ratio of fissile materials for safeguards monitoring.

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