Application of Neutron Activation Analysis for the Homogeneity Test of a Candidate Soil Reference Material

J. H. Moon[a*], S. H. Kim[a], Y. S. Chung[a], K. H. Cho[b]
[a] *Korea Atomic Energy Research Institute, 150-1 Deokjin-dong, Yuseong-gu, Daejeon, Korea*
[b]*Korea Research Institute of Standards and Science, 267 Gajeong-ro, Yuseong-gu, Daejeon, Korea*
[*]*Corresponding author: jhmoon1@kaeri.re.kr*

1. Introduction

A variety of certified reference materials(CRMs) has been developed by standard institutes like NIST(USA), IRMM(EU), KRISS(Korea) and has been supplied for quality control with respect to industrial production as well as research and development. ISO Guide 35 describes the certification of reference materials in terms of general and statistical principles. In order to produce CRMs, a homogeneity study is indispensible as it is stated in ISO guide 35. This work performed a homogeneity test for a candidate soil reference material by using Neutron Activation Analysis(NAA). Five elements such as As, Co, Fe, Na and Th, which have highly precise gamma-ray counting statistics were chosen to fulfill the homogeneity test through a preliminary experiment. Measured results for the five elements were evaluated in a statistical way.

2. Experiments

2.1 Sampling and Sample Preparation

Sampling and sample preparation were carried out by the Korea Research Institute of Standards and Science(KRISS). Samples were collected from a tailing dump of a mining area placed in Woongok, Cheongyang-Eup, Chungnam. The sampling site is shown in Fig. 1.

Fig. 1. Tailing dumps for sampling of a candidate soil reference materials

Approximately, 200 kg of sample soil was collected by using plastic shovel and the collected samples were naturally dried for ten days. Then, it was screened through a 2mm diameter sieve and dried in an oven at 55^{o}C for 22 hrs. The dried sample was pulverized to create particles that are smaller than 70 μm in diameter using air jet mill(Hosoga Alpine, 100AFG, German) and blended for 10 hrs using a V-mixer. Finally, 50 grams of the homogenized sample was put into a brown glass bottle. Ten bottles of samples for this work were provided from KRISS, shown in Fig.2 and three replicates were prepared in a bottle for NAA.

Fig.2. Sample bottles provided from KRISS

2.2 Neutron Activation Analysis

The NAA #1 irradiation holes with a Pneumatic Transfer System(PTS) of the HANARO research reactor was used for the activation of the prepared samples. For the analysis of As and Na using medium lived nuclide, samples were irradiated for a minute, decayed for two days and measured for 700 s. Three Fe monitors were co-irradiated with a sample to monitor the thermal neutron flux during a sample's irradiation. For the measurement of the gamma-rays from the interesting nuclides, a HPGe detector (EG & G ORTEC, 25% relative efficiency) coupled to a 16K-Multichannel Analyzer was used. Fig. 3 shows a measured gamma-ray spectra for the medium lived nuclides

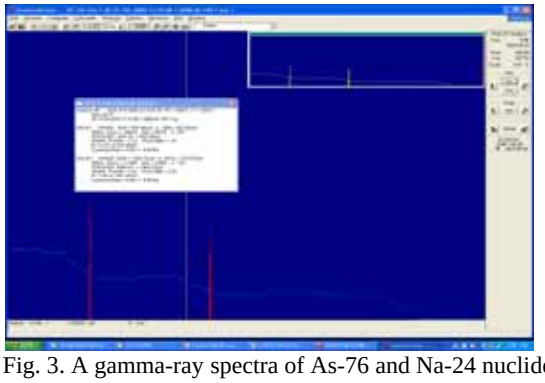

Fig. 3. A gamma-ray spectra of As-76 and Na-24 nuclides

For the analysis of Co, Fe and Th using long lived nuclides, samples were irradiated for 30 minutes, decayed for 20 days and measured for 10000 s. The same gamma-ray spectrometer was used with the medium lived nuclides. For the quantification of the elemental contents in the samples by a relative method, two NIST SRMs(2710-Montana Soil and 8704-Buffalo River Sediment) were analyzed under the same conditions as the samples.

3. Results and Discussion

As described in ISO guide 35, two kinds of homogeneity studies are necessary. One is to determine between-unit variation(between-bottle homogeneity study) and the other is to determine minimum sample intake(within-bottle homogeneity study). The summary of this work is shown in Table 1 and 2.

Table 1. Summary of analytical results.

Element	Mean Value (mg/kg)	Overall SD (RSD,%)	SD by between-bottle (RSD,%)
As	3328	60(1.80)	38(1.14)
Na	1887	37(1.96)	19(1.01)
Co	5.193	0.16(3.08)	0.105(2.02)
Fe	40788	889(2.18)	685(1.68)
Th	7.992	0.235(2.94)	0.158(1.98)

Table 2. Aanalytical results of NIST SRMs.

Element	Certi. Value	±	Unc. (2s)	This work Mean	±	SD	RSD(%)	SRMs
As	626	±	38	645	±	8	3.04	2710-Soil
Na	11400	±	600	11367	±	174	0.29	
Co	13.57	±	0.43	13.66	±	0.94	0.66	8704-Sediment
Fe	39700	±	1000	40781	±	2657	2.72	
Th	9.07	±	0.16	9.79	±	0.68	7.94	

The basic model for homogeneity testing is a one-way ANOVA, especially for between-bottle homogeneity test. In this case, degrees of freedom for between- and within-bottle are 9 and 20, respectively and the critical F-value is 2.393. The results of a one way ANOVA test for the five elements are indicated in Table 3. This result says that between-bottle homogeneity is not statistically significant with within-bottle homogeneity.

Table3. One-way ANOVA test results

Element	F ratio
As	1.28
Na	0.68
Co	1.47
Fe	2.30
Th	1.60

4. Conclusion

NAA has been used to certify elemental contents of reference materials. Contrary to XRF technique, it is affirmed that NAA is applicable to the homogeneity test of trace elements as well as major elements in candidate reference materials. Application works using NAA will be expanded in the QA/QC process.

REFERENCES

[1] International Standard Organization, ISO Guide 35-Certification of Reference Materials.
[2] R. L. Paul, E. A. Mackey, Rolf Zeisler, R. O. Spatz, B. E. Tomlin, Determination of Elements in SRM Soil 2709a by Neutron Activation Analysis, Vol.282, p.945, 2009.