Evaluation of Measurement Uncertainties for As, Ba, Mn, Sb, V and Zn in Sediment Samples Assayed by Neutron Activation Analysis

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1. Introduction

In analytical chemistry, a measurement uncertainty has been being stressed as an area of concern and diverse efforts have been made to evaluate it. Instrumental Neutron Activation Analysis (INAA) has a big advantage for estimating an uncertainty because of a simple uncertainty budget, especially when a relative method is applied for a quantification [1,2]. In this study, sediment samples were collected and INAA with relative method was employed for a determination of the hazardous elemental contents in the sediment samples. The combined uncertainties for the hazardous elements such as As, Ba, Cr, Mn, Sb, V and Zn were estimated under the given analytical conditions.

2. Experimental

2.1 Sampling and Sample Preparation

Sediment samples were collected from seven locations of the MeeHo stream in Cheongju city during the winter season. Three samples at the sampling points were collected in accordance with the depths, which were surface(0 – 5cm, S), middle(5 -10 cm, M) and lower part(10-15 cm, L). The collected samples were screened by using a sieve of 150 μm in size and dried in an oven for three days at 60 ^{o}C. Finally, analytical samples were prepared by a pulverization with an agate mortar (12-950C, Fisher) and by a re-screening of the samples with a sieve of 75 μm in size.

For an instrumental neutron activation analysis, the prepared samples were divided into two target samples for a detection of the short and long-lived nuclides and they were put in polyethylene vials, which were pre-cleaned using 0.1M nitric acid, for a neutron irradiation. After the irradiation was done, the samples were transferred to new polyethylene vials to be free from impurities in a blank vial. The weights of the samples for the short-lived and long-lived nuclides were about 50 mg and 150 mg, respectively

2.2 Instrumental Neutron Activation Analysis

The NAA #1 and #2 irradiation holes with a Pneumatic Transfer System (PTS) of the HANARO research reactor in Korea were used for the activation of the prepared samples. The thermal neutron fluxes measured at the NAA #1 hole, used for the long-lived nuclides, and the NAA#2 hole, used for the short-lived nuclides, were up to 4.85 x 10^{13}/cm^{2}·sec and 3.20 x 10^{13}/cm^{2}·sec, respectively. For the analyses using short-lived and long-lived nuclides, the samples were irradiated for 4 s and 1 hour, respectively. Al-01%Au and Fe monitors were co-irradiated with the samples 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. For the quantification of the elemental contents in the samples by a relative method, NIST SRM 2711-Montana Soil was chosen as a comparator and analyzed under the same conditions as the sample

3. Results and Discussion

The analytical results of the hazardous elements such as As, Ba, Cr, Mn, Sb, V and Zn from the sampling locations are summarized in Table 1. Concentrations of the hazardous elements such as As, Ba, Cr, Mn, Sb, V and Zn, were 3.76±1.66 mg·kg^{-1}, 881±94 mg·kg^{-1}, 242±142 mg·kg^{-1}, 901±241 mg·kg^{-1}, 1.19±0.51 mg·kg^{-1}, 75.5±7.3 mg·kg^{-1} and 159±41 mg·kg^{-1}, respectively

Table 1. Analytical results of sediment sample(unit : mg/kg)

Element		As	Ba	Cr	Mn	Sb	V	Zn
Location A	S	6.87	782	87.4	778	2.53	62.2	149
	M	6.97	951	93	634	0.67	77.4	116
	L	7.06	980	96.6	609	0.67	68.2	128
Location B	S	6.28	838	74.4	1508	1.73	72.4	169
	M	3.27	913	67.2	1202	0.57	65.1	134
	L	4.02	922	65	925	0.7	68.3	151
Location C	S	5.73	1061	154.5	849	0.42	76.7	125
	M	3.75	1102	152.8	669	0.4	84.5	117
	L	2.82	1017	138.7	829	0.4	81.6	152
Location D	S	4.68	854	68.8	1066	0.62	60.9	184
	M	4.9	864	64.9	703	0.7	84.4	169
	L	3.21	720	54.1	775	0.58	67.9	145
Location E	S	4.25	882	66.7	953	0.9	66.6	145
	M	6.21	950	83.7	927	0.55	75.7	146
	L	3.33	812	72.2	878	0.69	70.3	141
Location F	S	4.23	825	527.2	588	1	71.7	201
	M	7.79	894	574.1	563	1.17	82.9	239
	L	5.19	976	230.9	497	1.2	73.9	260
Location G	S	3.44	903	101.9	620	0.55	67.7	114
	M	3.11	821	95.4	598	0.42	78	102
	L	1.75	823	99.1	699	0.64	81.1	117
Maxium Value		1.75	720	54.1	497	0.4	60.9	102
Minimum Value		7.79	1102	574	1508	2.53	84.5	260
rsd(%)		44.3	10.7	58.7	26.8	42.7	9.6	25.4

Measurement uncertainty for INAA with relative methods can be carried out by the following simplified equation ;

$$W_{sam} = [A_{sam} / (A_{sta})] \times W_{sta}$$

Where, A_{sam} and A_{sta} indicate the initial activities of the concerned nuclides for a sample and standards, respectively. W_{sam} and W_{sta} indicate the mass of the concerned element for a sample and standards, respectively. Therefore, uncertainty sources can be defined in terms of a gamma-ray counting, weighing and the uncertainty of a reference material. Combined uncertainties for As, Ba, Mn, Sb, V and Zn under the applied analytical conditions were estimated with respect to the lowest and highest measured values. The estimated relative combined uncertainties(1s, %) for As, Ba, Mn, Sb, V and Zn are 15.0~45.2, 7.8~8.6, 3.0~3.1, 6.9~31.7, 10.1~12.8 and 2.3~3.8, respectively.

3. Conclusions

Stream sediment samples were analyzed by INAA, for hazardous elements such as As, Ba, Mn, Sb, V and Zn. The combined uncertainties for the hazardous elements were estimated in a simple way. In order to achieve a lower measurement uncertainty, Compton suppression measurement system should be implemented.

REFERENCES

[1] International Atomic Energy Agency, IAEA-TECDOC-1401, Quantifying Uncertainty in Nuclear Analytical Measurements, July, 2004
[2] ISO, Guide to the expression of uncertainty in measurement, 1993.