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대기분진의 원소분석에 대한 k_0-NAA법의 비교

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Comparative Analysis of Elemental Components in Airborne Particulate Matter by k_0-NAA Methods

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Abstract : A comparison of the analytical data obtained by three k_0-NAA software programs was carried out using both the airborne particulate matter collected from an urban site and the certified reference materials of the air filter and urban dust to evaluate the performance of the analysis. The individual k_0-NAA standardization methods of three countries, Korea, China and Vietnam which had been modified from the well established k_0-program were used for the comparative analysis. The measured concentrations of 30 elements from the two kinds of air samples based on this software were in agreement with each other within about 20% analytical error except for a few elements. By contrast, the results of China and Vietnam were moderately higher than that of Korea due to a systematic error associated with the detection efficiency, gamma peak analysis and geometric effect.

Key words : Neutron Activation Analysis, Airborne Particulate Matter, Trace Analysis, k_0-Standardization Method, Data Comparison, Certified Reference Material

1. Introduction

Instrumental neutron activation analysis (INAA) for the determination of an element in a sample of different matrices is usually performed by one of the absolute, relative and comparator method with and without a standards and certified reference materials. To improve the disadvantages of three quantitative methods, an advanced INAA based on well-known k_0-standardization method (k_0-NAA) has been developed. The advantages of k_0-NAA are experimental simplicity, high accuracy, excellent flexibility with respect to the irradiation and counting conditions, and its suitability for computerization.[1-4] In recent years, the k_0-method has been regarded as a reliable standardization method of an INAA.[5] Consequently, the k_0-NAA requires the calibration of all of the γ-ray spectrometers and irradiation facilities in order to obtain the essential k_0-parameters of the gamma peak detection efficiency and reactor neutron spectrum (i.e. α and f-

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factor, cadmium ratio, neutron flux) which vary depending on the irradiation site in the reactor and the detection system used. In practice, this method is also applicable to a simultaneous multi-element analysis of an unknown sample. This method does not require further preparation of the reference material for each element under the same conditions of neutron irradiation and gamma-ray spectrometric measurement. In view of the advantages of k_0-NAA, many laboratories have attempted the application of this method and the development of a relevant software to improve the reliability, convenience and accuracy of this technique analyzing different types of samples. Recent advances and applications of the k_0-NAA technique have been published elsewhere by other authors.[6-8] Firstly, the feasibility test for the application of k_0-NAA was carried out and the experimental k_0-parameters for a well thermalized NAA#1 irradiation hole of the HANARO research reactor was reported in a previous work.[9,10]

In this study, a comparative analysis of an air monitoring sample is implemented to develop a user k_0-calculation program and to evaluate the accuracy as well as the convenience and simplicity of the analysis through a comparison of the results obtained from the individual software prepared by each country as a regional collaborative research program.

2. Experimental

2.1. Sampling and sample preparation

Airborne particulate matter (APM) for the fine particle (<2.5 μm EAD) and coarse particle (2.5-10 μm EAD) fractions were collected using the Gent SFU sampler (low volume air sampler) with two kinds of polycarbonate filters (ϕ 47 mm, 0.4 and 8 μm pore size, Nuclepore) at an urban site in Daejeon city according to the recommended method by the IAEA.[11] Atmospheric conditions such as temperature, humidity, wind direction and wind velocity were recorded regularly during the collection of the samples. The flow rate was adjusted to 18 L/min at the beginning of the sampling and collected for 24 hours twice a week in 2002. During this period, 50 samples for two particle fractions were collected at two sites individually. The collected samples were pre-stored for 24 hours in a controlled atmosphere (20 ℃, RH 50%) and put into polyethylene vials and then weighed in the same conditions. Electrostatic charges were controlled by a ^{210}Po radioactive source. One set of the APM is arbitrarily extracted for the comparative analysis. Three kinds of certified reference materials, CRM (NIST SRM 1648, urban particulate matter; NIST SRM 2783, urban dust on the filter media; NIES CRM, No.8, urban dust) were used for the analytical quality control and the proficiency test.

2.2. Gamma-ray spectrometric method

The prepared sample was irradiated with thermal neutrons using the Pneumatic Transfer System (PTS, Φ_t = 2.80 x 10^{13} n/cm^2·s, R_{cd} ~ 205) at the HANARO research reactor at the Korea Atomic Energy Research Institute. Applied analytical conditions, irradiation time(T_i), cooling time(T_d) and counting time(T_c) were as follows ; (1) for the short nuclides : T_i = 2 m, T_d = 10～30 m and T_c = 300～600 s, (2) for the medium nuclides : Ti = 10 m, T_d = 1～2 d and T_c = 2,000 s, (3) and for the long nuclides : T_i = 4 h, T_d = 1～2 w and T_c = 4,000～8,000 s. Acquisition of the gamma-ray spectrum was carried out using a high purity Ge detector (a 25% relative efficiency and 1.85 keV resolution (FWHM) at 1332 keV of ^{60}Co and the peak to Compton ratio was 45:1) coupled to a personal computer and an 8k-multichannel analyzer (919A MCB, EG&G ORTEC, USA). Energy and efficiency calibrations were done using the multi-nuclide reference sources (Isotope Products Lab., ML 7500 series, 0.118" active diameter, disc type) traceable to NIST. Gamma Vision software (EG&G ORTEC, USA) for the energy and efficiency calibrations, and the acquisition of the gamma spectra and peak analysis was used.[12] Concentration of the elements was calculated using the in-house calculation program (Windows PC-code, Labview software) of KAERI with a well established nuclear data library[13,14], which was developed at this laboratory for a rapid and simple data treatment with the gamma-ray spectrum obtained at preset detection conditions. The detection limits for the elements can be calculated by

Currie's quantitative definition with a 10% allowable uncertainty.[15] The assessment of the measurement uncertainty was evaluated as a combined uncertainty including most of the sources of the standard uncertainty to be considered in the INAA. All of the analytical data were treated statistically.

2.3. Determination of the k_0-parameters

The Cd-ratio method was applied to determine the α parameter for the deviation of the epithermal neutron distribution form 1/E-law, approximated by the $1/E^{1+\alpha}$ dependence and the *f* parameters, as well as the thermal to epithermal flux ratio of the NAA #1 irradiation hole used. Two monitors with the three nuclides of ^{197}Au, ^{94}Zr and ^{96}Zr, so called a triple-monitor, were used to assure the optimal selectivity for both the reliability of the result and experimental simplicity.[10] Relevant nuclear data for the calculation of the k_0-parameters are available in the literature.[4] Four sets of monitors, consisting of pieces of Zr sheet (99.7329%, thickness 0.125 mm), Au-Al wire (Au 0.1124%, diameter 0.508 mm) and a pure cadmium cover (thickness 1.0 mm) were applied. Each monitor set was irradiated for 10 minutes.

2.4. Calculation of elemental contents by individual k_0-software

For the comparative analysis of the air particulate matter, three k_0-software programs were used as follows; a commercial and/or home-made program; KAERI-NAA (Korea) based on KAYZERO/SOLCOI (DMS, Belgium), ADVNAA (CIAE, China) and k_0-DALAT (DNRI, Vietnam). The content of the elements for one set of the air filter samples and NIST SRM 2783 were calculated by each k_0-software using the gamma-ray spectrum data files with a sample description in detail, a table of the detection efficiency, the k_0-parameters of the irradiation hole and the analytical conditions provided by Korea. However, there is no consideration for the type and size of the sample. Also, a peak search and analysis for each nuclide in the same gamma-ray spectrum was done according to their reference and the measurement system.

3.1. k_0-parameters (α, *f*) for irradiation hole

The calibration of the reactor neutron spectrum parameters related to k_0-NAA at the NAA #1 irradiation hole in the HANARO research reactor was carried out by the "Cd-ratio for the triple-monitor" method.[3] Both values of the α and f parameters to be used for the calculation of the elemental concentration are 0.127±0.005 (R=-0.98) and 1,140±70, respectively.[10] Because the neutrons of the NAA #1 irradiation hole are thermalized well as shown by their high *f* value, there is only a small fraction of the epithermal neutrons in comparison to the thermal neutrons, which is the value of the cadmium ratio, $R_{Cd,Au}$ which is measured to be about 82.

3.2. Quality control for analytical method

The analytical results of two kinds of the certified reference materials (NIST SRM 1648, urban particulate matter; NIST SRM 2783, urban dust on the filter media) for the analytical quality control and the proficiency test in our laboratory are presented in *Table* 1 and the other results for the concentrations of 25 elements in the certified reference material, NIES CRM No-8, urban dust for an analytical quality control are presented in *Table* 2. Only 20 elements could be compared with the certified and recommended values. The relative errors (RE) were less than 10% except for As, Mg, Sc, Se, Sr in the three CRM. The relative standard deviation (RSD) were in the range of 1 to 10% except for Ca, Hg, I in the case of SRM 1648 and Eu, Fe, K, Ta, Ti, Yb in the case of CRM No-8. From the proficiency test, the Z-score[16, 17] calculated from the certified values, measured values and their standard deviation or uncertainty, where, $Z = (\text{Value}_{\text{certi.value}} - \text{Value}_{\text{Lab}}) / \{(\text{Unc.}_{\text{certi.value}})^2 + \text{Unc.}_{\text{Lab}}\}^2\}^{1/2}$, are within ±2 except for Cd (more than 3) using the certified reference material, NIST SRM 2783 and they are presented in *Fig*. 1. The analytical work can be confirmed as a good performance for most of the elements.

Table 1. Analytical results of the certified reference material NIST SRM 2783(urban dust on filter media) and NIST SRM 1648(urban particulate matter) by INAA

Element	NIST SRM 2783				NIST SRM 1648			
	Certified Value (ng/filter)	Experimental value (ng/filter)	RE (%)	RSD (%)	Certified Value (mg/kg)	Experimental value (mg/kg)	RE (%)	RSD (%)
Al	23210±530	21790 ± 500	-6.1	2.3	34200±1100	32700 ± 1240	-4.4	3.8
As	11.8±1.2	10.1 ± 0.4	-14.1	3.9	115±10	82.1 ± 3.2	-28.6	3.9
Ba	335±50	363 ± 17	8.2	4.7	(737)	710 ± 53	-3.8	7.5
Br		242 ± 4		1.7	(500)	480 ± 16	-4.0	3.4
Ca	13200±1700	13170 ± 1100	-0.2	8.3	-	7080 ± 950	-	13.4
Ce	23.4±3.5	22.0 ± 0.7	-5.8	3.2	(55)	45.0 ± 2.1	-18.1	4.7
Cl		1200 ± 110		9.1	(4500)	4720 ± 73	4.9	1.5
Co	7.7±1.2	7.5 ± 0.78	-2.5	10.4	(18)	18.8 ± 0.19	4.4	1.0
Cr	135±25	148 ± 12	9.8	8.1	403±12	385 ± 24	-4.5	6.3
Cu	404±42	365 ± 38	-9.7	10.4	609±27	523 ± 19	-14.1	3.6
Fe	26500±1600	28870 ± 1670	8.9	5.8	39100±1000	35420 ± 1070	-9.4	3.0
Hg	-	-	-	-	-	3.67 ± 0.63	-	17.2
I	-	-	-	-	(20)	23.3 ± 3	16.5	12.2
In	-	-	-	-	(1.0)	0.96 ± 0.08	-4.0	8.3
K	5280±520	5210 ± 130	-1.3	2.5	10500±100	10430 ± 570	-0.7	5.4
La		11.9 ± 0.8		6.7	(42)	33.9 ± 1.5	-19.3	4.4
Mn	320±12	330 ± 3.4	2.8	1.0	(860)	894 ± 61	4.0	6.8
Na	1860±100	1840 ± 18	-1.1	1.0	4250±20	4180 ± 110	-1.6	2.6
Sb	71.8±2.6	75 ± 7	4.1	9.4	(45)	38.0 ± 0.96	-15.7	2.5
Sc	3.54±0.28	3.9 ± 0.2	10.9	5.1	(7)	5.65 ± 0.22	-19.3	3.9
Se					27±1	31.7 ± 1.31	17.5	4.1
Sm	2.04±0.15	1.95 ± 0.05	-4.4	2.6	(4.4)	3.14 ± 0.12	-28.6	3.8
Ti	1490±240	1460 ± 48	-2.0	3.3	(4000)	3980 ± 220	-0.5	5.6
V	48.5±6.0	49.4 ± 1.3	1.8	2.6	140±3	136 ± 4	-2.7	2.6
Zn	1790±130	1730 ± 190	-3.4	10.8	4760±140	4360 ± 170	-8.4	3.8

3.3. Comparison of the result by three k_0-calculations

The analytical results of the NIST SRM 2783 and the sets of fine and coarse particles of the APM obtained by the three kinds of k_0-calculation are summarized in *Table* 3. Elemental contents of the samples were converted by the amount of the air filter size analyzed for a relative comparison. For a comparison of the difference between each values, the measured values from each country were divided by the certified value as shown *Fig*. 2. The difference of the calculated mean values by the three *ko*-calculation methods are in the range of 1 to 20% except Mg, Ca, Rb, and the values of Vietnam are higher than those of Korea and China relative to the certified values. It is assumed that there is no consideration of the sample geometric effect and a systematic error by individual peak analysis. The short-lived nuclides such as Cu, Cl, Mg, I and the long-lived nuclides of a low count rate such as Ce, Co, and Sb showed a large difference. It is suspected that these discrepancies were caused by factors such as the efficiency calculation method and the neutron flux

Table 2. Analytical results of the certified reference material, NIES CRM No-8, Urban dust by INAA

Element	Nuclid (Energy, keV)	Experimental						Certi. Value			RE(%)
		Value Range			Mean	±	SD				
Al	Al-28(1779)	3098	~	3365	3230	±	119	3300	±	200	-2.1
As	As-76(559)	2.68	~	3.27	3.01	±	0.30	2.6	±	0.2	15.7
Ba	Ba-139(165)	110	~	126	119	±	6				
Br	Br-82(554)	57.7	~	60.5	58.7	±	1.5		56		4.8
Ca	Ca-49(3084)	4776	~	5154	4912	±	156	5300	±	200	-7.3
Ce	Ce-141(145)	3.36	~	3.59	3.48	±	0.11		3.1		12.2
Cl	Cl-38(1642)	862	~	1018	908	±	63				
Co	Co-60(1173)	3.41	~	3.64	3.51	±	0.12	3.3	±	0.3	6.5
Cr	Cr-51(320)	26.2	~	30.4	27.8	±	2.3	25.5	±	1.5	8.9
Eu	Eu-152(1408)	0.047	~	0.063	0.057	±	0.009		0.05		-14.7
Fe	Fe-59(1099)	1099	~	6228	5311	±	799				
Hf	Hf-181(482)	0.18	~	0.21	0.19	±	0.02				
Hg	Hg-203(279)	0.21	~	0.23	0.22	±	0.01				
K	K-42(1524)	1089	~	1539	1236	±	179	1150	±	80	7.5
La	La-140(1596)	1.28	~	1.35	1.31	±	0.04	1.2	±		9.2
Mg	Mg-27(1014)	11.42	~	1308	1220	±	59	1010	±	50	20.8
Mn	Mn-56(1810)	69.72	~	71.55	70.3	±	0.7				
Na	Na-24(1368)	1369	~	1976	1958	±	20	1920	±	80	2.0
Sb	Sb-122(564)	5.77	~	5.88	5.81	±	0.07	6.0	±	0.4	-3.2
Sc	Sc-46(889)	0.54	~	0.57	0.55	±	0.02		0.55		0.4
Se	Se-75(264)	1.59	~	1.89	1.79	±	0.17		1.3		37.4
Sm	Sm-153(103)	0.19	~	0.22	0.20	±	0.02		0.20		-0.1
Sr	Sr-87m(388)	92	~	105	101	±	5	89	±	3	13.9
Ta	Ta-182(1221)	0.10	~	0.14	0.11	±	0.02				
Th	Th-233(311)	0.36	~	0.42	0.39	±	0.03		0.35		11.4
Ti	Ti-51(320)	281	~	364	318	±	36				
V	V-52(1434)	14.89	~	16.12	15.50	±	0.46	17	±	2	-8.8
W	W-187(685)	5.64	~	6.48	6.08	±	0.42				
Yb	Yb-169(198)	0.09	~	0.12	0.10	±	0.02				
Zn	Zn-65(1115)	1116	~	1191	1143	±	42	1040	±	50	9.9

parameters, since the peak analysis for some nuclides is not identical. The results of the comparative analysis of the fine and coarse particles in the APM are presented in *Fig*. 3 and *Fig*. 4, respectively. The ratio of the concentration refers to s obtained relative to the values by the non k_0-method of Korea as an arbitrary reference for the comparison between the calculation methods. The difference in the measured values are similar in the both of results of the CRM and APM. The error of the counting statistics for each reported values of the long half-life nuclide generally ranged from 10 to 30% and these values are higher than those of the short half-life nuclide. Also, the deviation between the China and Vietnam results was due mainly to a difference in the

Table 3. Comparison of the results (ng) for the CRM and APM from the three k_0-calculation and the non k_0-calculation of KAERI.

Nuclide	ADVNAA			k_o-DALAT			KAERI k_o-method			KAERI non k_o-method		
	SRM	Coarse	Fine	SRM	Coarse	Fine	SRM	Coarse	Fine	SRM	Coarse	Fine
Al-28	8180	6140	1080	8200	6240	1120	6790	5320	939	7080	5260	923
As-76	6.57	10.8	21.4	7.63	11.4	24.7	6.27	11.5	21.2	5.36	9.92	18.1
Ba-139	107	165	46.4	104	201	51.0	79.0	162	40.0	85.5	171	42.3
Br-82	146	70.2	207	161	74.2	226	133	72.0	190	134	73.9	191
Ca-49	4990			9240			4340			4610		
Ce-141	9.69	14.4	2.36	12.7	15.9	3.35	11.3	12.7	3.45	11.4	12.4	3.41
Cl-38	454	1950	529	538	2110	588	397	1790	472	417	1830	480
Co-60	5.04	4.96	5.20	6.36	5.63	4.03	5.56	5.98	4.42	5.62	6.07	4.37
Cr-51	153	93.2	84.4	154	89.0	84.5	154	92.8	83.1	155	91.5	81.9
Cs-134		1.29	2.38		2.20	1070		1.35	1.65		1.46	1.74
Cu-66	138	137	69.7	147	139	74.0	92.5	108	67.7	89.4	101	62.6
Dy-165	0.52			0.59			0.54			0.68		
Fe-59	18200	7710	6290	20800	8650	5360	16700	8730	5010	16300	8520	4770
Hf-181	0.96	1.55	0.62		1.60		0.94	1.49	0.63	0.94	1.46	0.61
I-128		6.89	17.3		9.5	18.3		6.38	15.9		7.15	17.8
In-116m		0.24	0.28		0.27	0.30		0.21	0.25		0.19	0.22
K-42	1960	4840	4430	1940	5060	5040	2300	5820	4300	2280	5830	41730
Lu-177		0.06	0.04		0.07						0.11	0.04
Mg-27	2890	1580	285	34.1	2120		1440	1370	334	1550	1420	348
Mn-56	118	125	84.5	120	131	91.0	105	113	76.5	104	110	74.6
Na-24	677	2070	625	671	2150	644	600	1840	565	587	1760	540
Rb-86	31.6	24.6	28.7		24.3		67.8	34.0	26.8	70.6	35.5	27.3
Sb-124	41.9	28.9	39.8	52.6	28.3	52.7	42.8	29.5	41.6	42.1	29.3	40.0
Sc-46	2.41	1.64	0.81	2.71	1.74	0.87	2.20	1.73	0.75	2.21	1.73	0.73
Sm-153	9.13	1.55	0.20	9.84	1.59	0.21	9.48	1.46	0.19	9.38	1.58	0.19
Th-233	2.06	2.47	0.53	1.70	5.83		2.06	2.50	0.50	2.04	2.43	0.48
Ti-51	553	389	77.0	553	399	92.7	481	340	73.0	481	326	70.0
V-52	18.0	10.0	6.44	18.3	9.63	6.57	15.5	7.84	5.82	15.9	7.66	5.66
Yb-175		0.53	0.45					0.69	0.32		0.66	0.31
Zn-65	1260	611	1010	1260	644		1200	573	863	1200	579	849

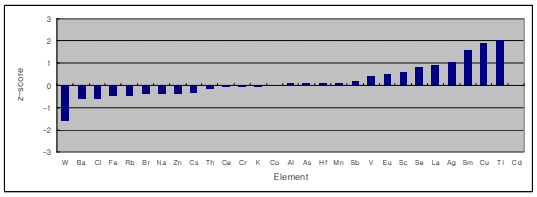

Fig. 1. Results of the proficiency test (z-score) of the urban dust sample, NIES.

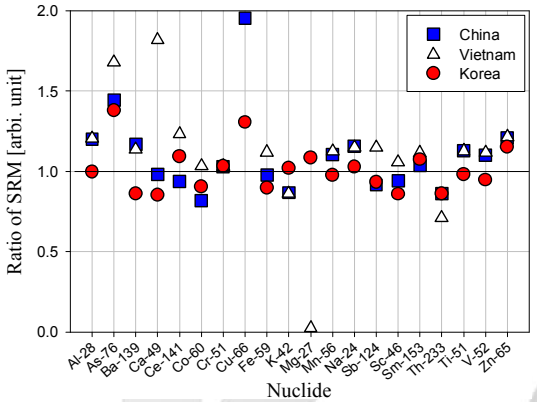

Fig. 2. Comparison of the ratios between the certified value and the measured value for the certified reference material NIST SRM 2783(urban dust on filter media) from the three k_0-calculation.

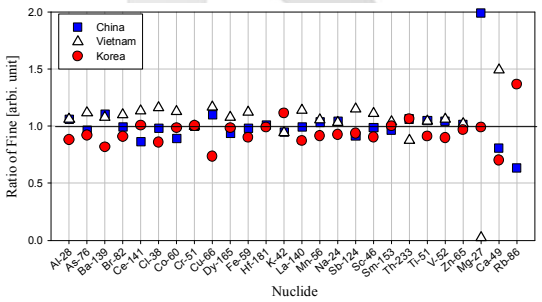

Fig. 3. Comparison of the ratios between the certified value and the measured value for the coarse particle of APM from the three k_0-calculation.

correction of the peak area. However, the results of the comparative analysis for the APM using the k_0-NAA methods that were obtained from the three participating countries (China, Korea and Vietnam) showed a difference in the use of each k_0-software and demonstrated a similar accuracy for the concentration of the elements.

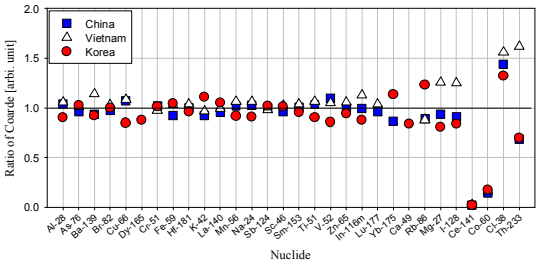

Fig. 4. Comparison of the ratios between the certified value and the measured value for the fine particle of APM from the three k_0-calculation.

4. Conclusions

The analytical quality control of the NAA was implemented using three certified reference materials and a quantitative comparative analysis of the airborne particulate matter was carried out by the well known k_0-standardization method. The results of the comparison between these k_0-softwares show that they have both drawbacks and advantages in their performances and characteristics. The accuracy of the elemental concentration dependence on the contents of simplified and modified software were founded to be 20%, due to a systematic error involving the detection efficiency, gamma peak analysis and geometric effect of the sample. The considerations of the geometric effect by the type and size of the sample, a peak analysis and the corresponding corrections are illustrated as important factors in the application of the k_0-NAA method.

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