Performance Test of K_0-NAA and Relative Method for Analysis of Al, Mg, K Nuclides in SRM Lake Sediment Sample

Sunardi and Darsono

Center for Accelerator Science and Technology, National Nuclear Energy Agency, Jl. Babarsari P.O. Box 6101 Yogyakarta

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ABSTRACT

A performance test has been conducted of k_0-NAA and relative methods for analysis of Al, Mg, K nuclides in SRM lake sediment sample (SRM SL-3). The performance test included validation, accuracy and precision tests, as well as t and F test. This test is done in order to know the performance of the both methods. Test results on validation test of both the k_0-NAA and relative methods obtained were still in good performance with z-score values within the range of $-2 \leq x \leq 2$, in the accuracy and precision test showed that the two methods used were still reliable or passed the test for elemental analysis. The results of the evaluation t-test were at 95% confidence level showed that both methods were no significant differences in the test results, and the evaluating the F test showed that there are no differences in the accuracy between the two methods. The performance test data are expected to be used by practitioners and users of neutron activation analysis services as the reference option of analysis method to optimize the objectives to be achieved.

Keywords: *performance test; NAA; relative method; k_0-NAA; SRM SL-3*

ABSTRAK

Telah dilakukan uji performa metode k_0-AAN (Analisis Aktivasi Neutron) dan metode relatif untuk analisis nuklida dalam bahan standar SRM SL-3. Uji performa meliputi uji performa validasi, uji akurasi dan presisi, uji t dan uji F dengan tujuan untuk mengetahui unjuk kerja kedua metode tersebut. Hasil uji performa validasi baik metode k_0-AAN maupun metode relatif diperoleh nilai z-score masih dalam rentang $-2 \leq x \leq 2$, sehingga kedua metode masih dalam kategori memuaskan, pada uji akurasi dan presisi menunjukkan kedua metode yang digunakan masih layak atau lolos uji untuk analisis unsur. Hasil evaluasi uji t pada derajat kepercayaan 95% menunjukkan kedua metode tidak ada perbedaan yang nyata dalam hasil ujinya sedang evaluasi uji F menunjukkan tidak ada perbedaan kecermatan antar kedua metode. Dengan data uji performa ini diharapkan dapat digunakan para pelaku dan pengguna jasa analisis aktivasi neutron sebagai referensi pilihan untuk mengoptimalkan tujuan yang akan dicapai

Kata Kunci: *uji performa; AAN; metode relative; metode k_0-AAN; SRM SL-3*

INTRODUCTION

Technique of neutron activation analysis (NAA) is a multi-element analysis technique, both qualitative and quantitative utilizing neutron beam that is generated by a nuclear reactor or neutron generator accelerator. NAA technique has advantages because the test results have high sensitivity, conducted simultaneously, and does not damage the sample. The NAA is well suited to study homogeneity of small samples because of its dynamic range of elemental sensitivity. A high potential of NAA for accuracy measurement compared to other analytical techniques is especially valuable for certification of element contents and for checking accuracy of other trace element analytical techniques [1-2]

The NAA can widely be applied in several scientific fields, namely in environmental control and monitoring, geo- and cosmochemical research, in the life sciences (*e.g.*, determination of essential and toxic trace elements in organisms), archaeological research, in material research, and in quality assurance of other trace element analytical techniques (control analyses, preparation, and certification of reference materials of chemical composition). Environmental pollution control and monitoring, especially air pollution monitoring is a typical example of application of NAA. Air pollution monitoring concerns the determination of the incidence, elemental composition, and size of aerosol particles in the ambient or indoor air, or studying the above parameters in combustion aerosols, which are the main source of air pollution [3]. Also NAA is used to determine of aluminium, silicon and magnesium in geological matrices [4] and rare earth elements in soil samples [5].

* Corresponding author. Tel: +62-85728738074
Email address: sunardip3tm@batan.go.id

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The method used to determine of element contents can use relative, absolute and k_0-NAA methods. Hence the k_0-standardization method transforms NAA to a highly effective, manageable and competitive determination method. For this reason, k_0 standardization in NAA is increasingly being used in many laboratories worldwide [6-7]. This method consists of sample preparation, irradiation of samples, gamma ray spectra analysis, calculation of elemental content and gamma-ray spectrometry measurement of the activity of the radionuclides formed without any chemical treatment. It is the simplest way of performing NAA using relative and k_0-NAA methods. Detection efficiency calibration was carried out using ^{152}Eu standard gamma-ray source.

The results of a laboratory test can be acknowledge for the truth if the laboratory has possessed a certificate of test result assessment or has been accredited. It ensures to users of laboratory services that the test results produced have a good value in accuracy and precision. The accuracy of test results is basically supported by calibrated laboratory facilities and infrastructure, and the method being used is validated.

In order to support R & D results of a laboratory for these activities generate output and outcome to industry/environment and formally trustworthy, the laboratory must be accredited and the equipments must meet the quality system according to the guidelines of KNAPPP 02-2007 from the ministry of Research and Technology of Indonesia. Testing method used is the standard method or developed method, but it has been validated by firmed standards using Standard Reference Material (SRM), thus the quality system of R & D should be prepared and applied in accordance with the guidelines of the ISO-9000 series of standard and its equivalents or ISO/IEC Guide 25 and its derived national standard.

In order to support the quality assurance program to generate the desired test data consistently, continuously, planned, controlled and efficiently ensuring the quality improvement of laboratory operations, it is necessary for the inter-comparison test on the method used in the laboratory. Besides that, the comparison test aims to convince users of laboratory services that the appliance is working properly and the evaluation result meets the applicable regulation.

Each test method has a different character in testing. This distinction is that will be compared, so that these activities are not looking for a good or bad method. Anyhow, the comparative test data is expected to be used by practitioners and users of such neutron activation analysis (NAA) methods services as reference options to optimize the objectives to be achieved. Comparative test methods used in order to fulfill one of the clauses contained in ISO/IEC 17025-2008.

The purpose of this test is to assist the performance of testing laboratories and assesses their performance in elemental analysis, particularly for those parameters that are applied in the proficiency test. The result of this proficiency testing is very important in the assessment of the overall performance of a laboratory and constitutes a considering matter by the national accreditation committee (NAC) in the provision and maintenance of accreditation status.

NAA analysis technique is based on the reaction of neutron to nucleus, where samples should be analyzed with neutron irradiation. The core elements are atoms in the sample that will capture a neutron and turn into radionuclides by emitting radioactive γ-ray. γ-ray emitted energy generally has a very characteristic for each radionuclide, so it can be identified using gamma spectroscopy technique. Radioactivity, formed or count per second (cps), can be known like equation [8].

$$\text{cps} = \frac{P_A}{LT} = \left(\frac{N_A \theta m}{M}\right)(\sigma\phi)\,\text{S.D.C.}\varepsilon\gamma \qquad (1)$$

where, P_A is the photo peak area, LT is calculation life time, N_A is the Avogadro number, σ is the cross section (barns) (cm^2), Φ is the neutron flux ($cm^{-2}.s^{-1}$), λ is the decay provision (s^{-1}), C is the correction factor during enumeration, $C = (CL\text{-}e^{-\lambda CL})/\lambda LT$, CL is the clock time, θ is the isotope abundances, m is the element mass, M is the isotope mass, ε is the detection efficiency, γ is the gamma yield. Concentrations are usually carried out by relative or k_0-NAA.

Relative Method

In this method, a standard and sample are irradiated together. Meanwhile, the activities of both sample and the standard are measured in identical geometry with respect to the detector. By using the mass of the element in the standard ($m_{x,std}$) and count rates of the standard ($cpc_{x,std}$) and sample ($cps_{x,sample}$), the mass of the element in the sample ($m_{x,sample}$) is determined by the equation.

$$m_{x,sample} = m_{x,std} \cdot \frac{cps_{x,sample}}{cps_{x,std}} \cdot \frac{D_{std}}{D_{sample}} \qquad (2)$$

where $D = e^{-\lambda t}$ is the decay factor, t is the decay time

Single Comparator or k_0-NAA Method

Sample and single standard are irradiated together by comparing the activity of ratio between the sample and the single standard that can be used to determine the concentration of the footage being

analyzed. The concentration of the elements contained in the sample can be calculated by equation [8-9].

$$C(\mu g.g^{-1}) = \frac{\frac{P_A / LT}{S.D.C.W}}{\frac{P_A / LT}{S.D.C.w}} \cdot \frac{1}{k_0} \cdot \frac{\left(f + Q_0(\alpha)^*\right)}{\left(f + Q_0(\alpha)\right)} \cdot \frac{\varepsilon^*}{\varepsilon} \quad (3)$$

where W and w = period footage and a single comparator, f = neutron flux ratio of epithermal to thermal, α = neutron flux distribution parameter, $Q_0(\alpha)$ = $(I_0(\alpha)/\sigma_0)$ = per-comparison between the integral resonance to thermal neutron cross section, I_0 = integral resonance for epithermal spectrum, σ_0 = cross section of the thermal neutron, ε = detector efficiency, k_0 is expressed by the equation:

$$k_0 = \frac{M^*\theta\sigma_0\gamma}{M\theta^*\sigma_0^*\gamma^*} \quad (4)$$

the symbol '*' indicates a parameter of comparison

EXPERIMENTAL SECTION

Materials

The materials used in this study are standard radioactive sources, such as, Co-60, Cs-137 and Eu-152 to calibrate the energy and efficiency of detectors, the IAEA standard reference material sediment lake (SRM SL-3).

Sample Preparation and Instrumentation

SRM SL-3 element was weighed between 7-10 mg and wrapped with polypropylene and placed in the polypropylene capsule. The element was then irradiated in a Dhruva reactor (India) with neutron flux of 5.10^{13} n/cm^2.s for 1 min. After irradiation, the element was counted using gamma spectrometry by HPGe detector.

Procedure

Performance validation

Performance validation of the test method used, whether satisfactory or not, is determined by the value of Z-score. Z-score is almost the same as the standard normal distribution, where standard deviation is only determined by the standard deviation of Horwitz. Value Z-Score [10-11] that can be determined by the equation (5)

$$Z - Score = \frac{x_i - X}{s} \quad (5)$$

where x_i is the value of the analysis, X is a reference value, and (s) is standard deviation value which can be obtained by the equation $s = 0.02 \times X^{0.8495}$ (standard deviation according to Horwitz)

Accuracy and precision test

This activity is aimed to determine the accuracy and the precision of the NAA method corresponds to the standards provided by the IAEA [11]. Accuracy can be accepted if the results of the analysis can meet the equation (6) while precision is acceptable if the results of the calculation according to the equation (7).

$$|N_s - N_a| \le 1{,}95 \times \sqrt{U_s^2 + U_a^2} \quad (6)$$

$$\left[\sqrt{\left(\frac{U_S}{N_S}\right)^2 + \left(\frac{U_a}{N_a}\right)^2} \times 100\right] \le \left[\sqrt{\left(\frac{U_S}{N_S}\right)^2 + (\sigma_H)^2} \times 100\right] \quad (7)$$

where: U_s, U_a is uncertainty from the certificate and calculation result; N_s, N_a is average value from the certificate and calculation result; σ_H is Horwitz constant ($0.02\ X^{0.8496}$)

F and t test

The t-test (t_0) is one of the statistical tests where it is used to test the truth or falsity hypothesis which states that between the two mean values (mean) of samples taken at random from the same population, there was no significant difference, while F-test evaluation aims to determine the precision of the method used, to show how large or small the variance of repeated measurements, smaller the variance of measurement data more closely results on the method used [12].

The value t_0, can be calculated [9-10] by the equation:

$$t_0 = \frac{\left|\bar{x}_1 - \bar{x}_2\right|}{\sqrt{\frac{s_1^2}{n_1} + \frac{s_2^2}{n_2}}} \quad (8)$$

where, $\bar{x}_1, \bar{x}_2$ is average of method test for k_0-AAN and relative method; s_1, s_2 is standard deviation of k_0-NAA method and relative method; n is the number of test repetition

While the F-test (F_0) can be determined by the equation [10-11]:

$$F_0 = \frac{S^2_{k_0 - AAN}}{S^2_{relative}} \quad (9)$$

where, S is standard deviation

RESULT AND DISCUSSION

The result of the analysis of the concentration levels of elements Al, Mg, K in SRM SL-3 determined by using equation (2) for relative methods and equation (3) for k_0-NAA method was shown in Table 1.

Table 1. Al, Mg, K element concentration in SRM SL-3 analyzed using k_0-NAA and relative method [13]

No	Element concentration of Al (mg/g)		Element concentration of Mg (mg/g)		Element concentration of K (mg/g)	
	k_0-NAA	Relative	k_0-NAA	Relative	k_0-NAA	Relative
1	24.15 ± 1.74	25.05 ± 1.23	27.29 ± 1.96	27.58 ± 1.68	9.07 ± 1.18	8.31 ± 0.87
2	25.52 ± 1.34	25.69 ± 1.34	27.84 ± 1.87	26.93 ± 1.71	8.59 ± 0.99	8.77 ± 0.68
3	24.87 ± 1.48	24.97 ± 1.39	26.88 ± 1.91	27.11 ± 1.55	8.12 ± 0.98	8.87 ± 0.98
4	25.31 ± 1.56	25.56 ± 1.43	27.32 ± 1.85	27.44 ± 1.88	8.84 ± 1.05	9.06 ± 1.10
$\bar{x}$	24.96 ± 1.53	25.32 ± 1.35	27.33 ± 1.90	27.26 ± 1.71	8.65 ± 1.05	8.75 ± 0.91
	SL-3: 24.50 ± 1.30		SL-3: 27.00 ± 2.43		SL-3: 8.74 ± 0.787	

Table 2. Calculation result of *Z-score* of k_0-AAN and relative method

Element	Standard value (mg/g)	Average of analysis result (mg/g)		Z-score	
		k_0-AAN	Relative	k_0-AAN	Relative
Al	24.50 ± 1.30	24.96 ± 1.53	25.32 ± 1.35	1.49	2.71
Mg	27.00 ± 2.43	27.33 ± 1.90	27.26 ± 1.71	1.00	0.79
K	8.74 ± 0.79	8.65 ± 1.05	8.75 ± 0.91	-0.71	0.08

Description: $-2 \leq x \leq 2$ is satisfying category; $+2 < x < +3$ and $-2 < x < -3$ is warned category; $-3 \leq x \leq 3$ is outlier category

Table 3. The result of the calculation accuracy of k_0-AAN and relative methods

Element	Accuracy calculation of k_0-NAA		Accuracy calculation of relative method		Precision calculation of k_0-NAA		Precision calculation of relative method	
	Left side	Right side	Left side	Right side	Left side	Right side	Left side	Right side
Al	0.46	3.91	0.82	3.65	0.81	3.12	0.75	3.07
Mg	0.33	6.01	0.26	1.95	1.14	3.41	1.09	3.41
K	0.09	5.79	0.01	2.35	1.91	2.40	1.38	1.55

Performance Validation Test

Data processing with this technique requires a value that becomes a reference for assessing the performance of a laboratory and a standard deviation of the target. In this case the standard deviation is according to Horwitz [10-11]. Performance validation of the test method used, whether satisfactory or not, is determined by the value of Z-score. Z-score is almost the same standard as normal distribution. Standard deviation is only determined by the standard deviation Horwitz. Using data in Table 1, Z-Score of k_0-NAA and relative method can be determined by the equation (5) and it can be shown in Table 2.

Seen in Table 2, a Z-score k_0-NAA method and relative methods of three elements, namely Al, Mg, K, are respectively 1.49, 1.00, -0.71 and 2.71, 0.79 , 0.08. In general, the performance validation test results of both methods give satisfactory results still in the satisfying category, as a Z-score is still within the range of $-2 \leq x \leq 2$, except on Al elemental analysis using relative method, the value of Z-score of 2.71 is categorized warned. This is probably due to uneven neutron exposure or uneven counting geometry of the sample. According to previous study [14] who has performed an analysis using the k_0-NAA with the same sample as we done it was obtained a Z-score value ± 1. With reference to the result it can be said that in this study the performance test of both k_0-NAA and relative methods is still useful and proper to be used.

Accuracy and Precision Test

From the results of the validation methods that was done, it can be determined whether the accuracy and precision of k_0-NAA method and the relative method were relatively acceptable or pass the test. Rated accuracy is the closeness of the result of the analysis of the average true values or the deviation value of the test result data to the true value, while precision is the suitability of the results of analyzes of some repeatability of measurements in the same way expressed in value relative standard deviation [11]. To determine the accuracy and the precision of the NAA method in accordance with the standards provided by the IAEA, the experimental results in Table 1 should be tested by using a procedure in reference [10]

According to reference [15], it is obtained that accuracy and precision on Mg element measured by using SRM 8704 sample, are 95.53% and 94.88% respectively. In this research, anyhow, in measurement of accuracy and precision, whether it corresponds to the requirement or not, it is tested by using the equation (6) and (7) according to IAEA standard [11]. The result of the accuracy calculation and the precision in this work is shown in Table 3.

From the results of the calculations in Table 3, the accuracy and precision values of k_0-NAA and relative methods for the elements Al, Mg, K, their values between the left side and the right side obtained show that the left side is smaller than the right side. It proves

Table 4. Absolute deviation data, the deviation of the squared deviations relative to the k_0-NAA method and the method of relative elements of Al

No	Average elemental test of Al (mg/g)		deviation (x- $\bar{x}$)	
	k_0-NAA	Relative method	k_0-NAA	Relative method
1	24.15	25.05	-0.81	-0.27
2	25.52	25.69	0.56	0.37
3	24.87	24.97	-0.09	-0.35
4	25.31	25.56	0.35	0.24
	$\sum$ = 99.85	$\sum$ =101.27		
	$\bar{x}$ = 24.96	$\bar{x}$ = 25.32		
	Absolute deviation (s)		1.81	1.23
	s^2		3.28	1.51
	s relative (s/ $\bar{x}$) (%)		7.25	4.80

Table 5. The result of three sediment samples by k_0-NAA and relative method

No	Concentration of Al (mg/g)		Concentration of Mg (mg/g)		Concentration of K (mg/g)	
	k_0-NAA	Relative	k_0-NAA	Relative	k_0-NAA	Relative
1	44.22 ± 1.31	45.11 ± 1.43	25.27 ± 0.72	26.51 ± 0.92	10.80 ± 1.06	10.61 ± 0.99
2	37.86 ± 1.13	38.62 ± 1.27	17.45 ± 0.96	16.83 ± 0.87	11.51± 1.03	12.71 ± 1.04
3	55.36 ± 1.36	54.56 ± 1.21	23.67 ± 0.87	24.88 ± 0.92	9.19 ± 0.88	9.72 ± 0.91

Description: 1 = sample 1; 2 = sample 2; 3 = sample 3

that the data of accuracy test result meets the equation (8) and precision test meets the equation (9), hence the methods used both k_0-NAA and relative methods may be feasible or pass the test to be used in elemental analysis.

T-test Evaluation

The t-test (t_0) is one of the statistical tests where is used to test the truth or falsity null hypothesis which states that between the two mean values (mean) of samples taken at random from the same population, there was no significant difference. In this case the goal is to determine whether there is a difference between the average levels of the element k_0-NAA method and relative method. To determine the mean difference by means of the t test, the data in Table 1 may be determined average value analysis, absolute deviation and relative deviation of the results is shown in Table 5. Then the value t_0, can be calculated [10] by the equation (8).

This statistical test is to compare the value t_0 premises to the t-value calculation results in a table or reference. By using a significance level of 5% or 95% confidence level, the value of t_{table} is of 2.28 [10-11], the other reference stated t_{table} is 2.228 [16], and stated that if value t_0 < t-table it can be said there is no difference in the mean test results and vice versa if the value t_0 > t-table it means there is a difference in meaning.

By using equation (8), the t_0 value of elements in SRM investigated can be determined, for Al element the t_0 value was 0.330. In the same way for Mg and K elements, the t_0 values were respectively 0.099 and 0.135. This calculation result of t_0 values of the elements Al, Mg and K obtained in this research was below 2.28, so it can be said that there are no difference the test results between k_0-NAA method or relative methods because the t-test of both methods yielded t_0 below t_{table}.

Evaluation of F-test

F-test evaluation aims to determine the precision of the method used, to show how large or small the variance of repeated measurements, smaller the variance of measurement data more closely results on the method used. F-test (F_0) can be determined by the equation (9). By using the data in Table 4, so the value F_0 can be calculated by equation (8). For elements of Al the F_0 value was 2.12, while the elements of Mg and K the F_0 values respectively were 1.06 and 1.82. If a significance level of 5% is used, the value of F_{table} is 5.05 and it is said that if the value of F_0 (calculate) < F-table (5%) then it can be regarded as no difference in the precision of the k_0-NAA and relative methods to the significance level of 5%, and vice versa if the value of F_0 (calculate) > F-table so there are differences in precision between both methods

From the calculation of the F-test it can be said that there is no difference in the accuracy in testing the elements Al, Mg and K for k_0-NAA method or methods relative, because the value of $F_{(calculate)}$ is under F_{table} value, thus meeting the requirements for materials analysis.

Application of the Method Tested by Performance

The method tested by performance is then applied to test Al, Mg, and K in the sample of three sediments. Element concentration for relative method and k_0-NAA are determined by equation (2) and (3). The result of three sediment samples by k_0-NAA and its comparison to relative method of NAA is shown in Table 5.

CONCLUSION

From the performance test results it can be concluded that the validation of both methods are satisfactory shown by z-score values obtained are still within the range of $-2 \leq x \leq 2$. The accuracy and precision tests showed that both methods pass the test for elemental analysis. The results of the evaluation t-test at 95% confidence level showed that both methods no significant differences in the test results more ever evaluating the F test showed no difference in the accuracy between the two methods. With the performance test data is expected to be used by practitioners and users of neutron activation analysis services as the reference option of analysis method to optimize the objectives to be achieved.

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