A METHOD VALIDATION FOR DETERMINATION OF GROSS ALPHA AND GROSS BETA IN WATER SAMPLE USING LOW BACKGROUND GROSS ALPHA/BETA COUNTING SYSTEM

PENENTUSAHAN KAEDAH PENENTUAN GROS ALFA DAN GROS BETA DALAM SAMPEL AIR MENGGUNAKAN SISTEM PEMBILANG GROS ALFA/BETA BERLATARBELAKANG RENDAH

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Abstract

Method validation (MV) for the measurement of gross alpha and gross beta activity in water (drinking, mineral and environmental) samples using Low Background Gross Alpha/Beta Counting System was performed to characterize precision, accuracy and reliable results. The main objective of this assignment is to ensure that both the instrument and method always good performed and resulting accuracy and reliable results. Generally, almost the results of estimated RSD, z-score and U_{score} *were reliable which are recorded as ≤ 30%, less than 2 and less than 1.5, respectively. Minimum Detected Activity (MDA) was estimated based on the counting time of 100 minutes and present background counting value of gross alpha (0.01 – 0.35 cpm) and gross beta (0.50 - 2.18 cpm). Estimated Detection Limit (DL) was 0.1 Bq/L for gross alpha and 0.2 Bq/L for gross beta and expended uncertainty was relatively small of 9.77% for gross alpha and 10.57 % for gross beta. Align with that, background counting for gross alpha and gross beta was ranged of 0.01 – 0.35 cpm and 0.50 - 2.18 cpm, respectively. While, sample volume was set at minimum of 500 mL and maximum of 2000 mL. These proven the accuracy and precision result that are generated from developed method/technique is satisfactory and method is recommended to be used. Therefore, it can be concluded that the MV found no doubtful on the ability of the developed method. The test result showed the method is suitable for all types of water samples which are contained several radionuclides and elements as well as any impurities that interfere the measurement analysis of gross alpha and gross beta.*

Abstrak

Penentusahan kaedah (MV) bagi pengukuran keaktifan gros alfa dan gros beta dalam sampel air (minuman, mineral dan alam sekitar) menggunakan Sistem Pembilang Gros Alfa/Beta Berlatarbelakang Rendah telah dilaksanakan dengan pencirian ketepatan, kejituan dan keputusan yang dipercayai. Objektif utama tugasan ini ialah untuk memastikan kedua-dua peralatan dan kaedah sentiasa berprestasi baik dan menghasilkan keputusan yang tepat dan dipercayai. Secara umumnya, keseluruhan keputusan yang dianggarkan bagi RSD, z-score dan U_{score} *adalah dipercayai masing-masing mencatatkan sebagai ≤ 30%, < 2 dan < 1.5. Aktiviti Minima Dikesan (MDA) yang dianggarkan berdasarkan masa pembilangan 100 minit dan nilai pembilangan latarbelakang gros alfa (0.01 – 0.35 cpm) dan gros beta (0.50 - 2.18 cpm). Penganggaran Had Pengesanan (DL) adalah 0.1 Bq/L untuk gros alfa dan 0.2 Bq/L untuk gros beta; dan "expended uncertainty" adalah kecil secara relatifnya dengan 9.77% untuk gros alfa dan 10.57% untuk gros beta. Selari dengan itu, pembilangan latarbelakan gros alfa dan gros beta masing-masing dalam julat 0.01 – 0.35 cpm dan 0.50 - 2.18 cpm. Manakala, isipadu sampel telah ditetapkan pada kadar minima ialah 500 mL dan maksima ialah 2000 mL. Ini telah membuktikan ketepatan dan kejituan keputusan yang dihasilkan daripada pembangunan kaedah/teknik adalah berpuashati dan disarankan untuk digunakan. Dengan itu, boleh disimpulkan bahawa MV tiada keraguan ke atas keupayaan teknik yang telah dibangunkan.. Keputusan ujian juga didapati sesuai untuk semua jenis sampel air yang mempunyai beberapa radionuklid dan unsur serta bahan asing yang mengganggu analisis pengukuran gros alfa dan gros beta.*

Keywords: Accuracy, gross alpha, gross beta, method validation, reliable

INTRODUCTION

Radioactive contamination of the environment can be defined as any increase in the natural background radiation arising out of human activities involving the use of naturally occurring or artificially produced radioactive substances (Patel 1980). It is typically the result of a loss of control of radioactive materials during the production or use of radioisotopes. Such contamination could be occasional, accidental (unplanned), planned or continuous. The presence of radioactivity in the environment is caused by naturally occurring radionuclides and cosmic radiation but also by artificial radionuclides, which have been incorporated due to fallout from nuclear accidents and nuclear weapons testing (EPA 2000).

The presence of radionuclides in water poses a number of health hazards, especially when these radionuclides are deposited in the human body through drinking. Dissolved radionuclides in water emit particles (alpha and beta) and photons (gamma) which gradually expose living tissues (Alam et al. 1999; Gruber et al. 2009). Human and animal studies show that radiation exposure at low to moderate doses may increase the long-term incidence of cancer (Amrani and Cherouati 1999; Collman et al. 1991; Gofman 1990). The potential adverse effect from ingestion of radionuclides, through drinking water, requires a standard to be set in order to protect the members of public from radiation exposure above permissible levels. For the purpose to characterize precision, accuracy and reliable results for measurement of gross alpha and beta activity in water, the method validation (MV) should be implemented to verify test methods compliance with consumer requirement.

Method validation is a key requirement to implement a laboratory measurement or analysis of technical development to ensure that both the instrument and method always good performed and resulting accuracy and reliable results. Furthermore, it is important for the purpose of laboratory accreditation in compliance with MS ISO/IEC 17025 standards or national and international guidelines and also to convince the clients. To complement the activities of method validations, several parameters need to be tested. The parameters are specificity, precision/repeatability, bias/accuracy, linearity/and working range, detection limit, robustness/sensitivity, ruggedness/reproducibility.

In order to ensure the methods/techniques of the measurement can produce accuracy and reliable results, the method should be verified using some internal standard or certified reference material (SRM or CRM) which has certified value and traceability. CRM is used in most analytical measurement to reduce systematic errors that may arise during the preparation of complex samples. On the other hand, in-house prepared standard solution and standard solution series, also known as quality control samples were also used to verify test methods for the above mentioned parameters. Methods of verification procedures carried out in accordance with ISO guidelines and accreditation bodies such as ISO, INAB, NATA. Background measurements are carried out to ensure no excessive background activity measured by counting equipment that will affect the outcome of the analysis. Align with that, the main objective for the current work is to ensure both the instrument and method always good performed and resulting accuracy and reliable results in measuring activity of gross alpha and gross beta in water (drinking, mineral and environmental) samples using Low Background Gross Alpha/Beta Counting System.

EXPERIMENTAL AND METHODOLOGY

The method used is the benchmark method for gross alpha and gross beta radioactivity determinations. This method involved evaporating the sample until to a thin layer of solid residue in a stainless-steel planchet. Then, it was analyzing alpha and beta particles emitted from the source using gas-flow proportional counting namely Low Background Gross Alpha/Beta Counting System. The benefit of this method is its rapidity and relatively low-cost in comparison to performing radionuclide specific analysis.

Due to RAS's laboratory was not have a CRM/SRM in solution form, the in-house standard solution which is known its activity was prepared and was used in all method validation parameters test. The solution was contained several gross alpha radionuclides such as ^{210}Po, ^{226}Ra and ^{232}Th; and gross beta radionuclides such as ^{40}K, ^{90}Sr and ^{3}H. The actual specific activity of gross alpha and gross beta in the in-house prepared standard solution are 1.2055 Bq/g and 2.8236 Bq/g, respectively. The in-house prepared standard solution was spiked in 8 glass beakers and added 500 mL of distilled water. The solution was evaporated on a hot plate at temperature less than water boiling point until the volume of water reach to 10 mL. The solution was transferred drop by drop using plastic dropper into planchet and dried under IR lamp until residue was almost dried. Then, planchet which are contained the residue was counted using Low Background Gross Alpha/Beta Counting System for 100 minutes. All the measured data were calculated for the activity of gross alpha and gross beta and statistical analyses such as mean, standard deviation (SD), relative standard deviation (RSD), U_{score}, z-score etc. Then, all parameters of the method validation were tested based on their criteria.

RESULTS AND DISCUSSION

Specificity

Specificity test have proven that such method can measure the activity of gross alpha and gross beta despite of in-house prepared standard solution contains various radionuclides emitting beta, alpha and their daughter of emitting gamma. Generally, the results of the measured activity of gross alpha and gross beta in seven replicates of in-house prepared standard solution were different of 15.4% for gross alpha and 28.8% different for gross beta compared to their actual activities (average gross alpha = 0.13 ± 0.01 Bq/L and average gross beta = 0.28 ± 0.04 Bq/L) (Table 1).

Table 1: Actual and measured activity of gross alpha and gross beta for specificity test

No. of measurement	Actual activity (Bq/L)		Measured activity (Bq/L)	
	Gross alpha	Gross beta	Gross alpha	Gross beta
Measurement 1	0.13 ± 0.01	0.33 ± 0.04	0.12 ± 0.01	0.19 ± 0.02
Measurement 2	0.13 ± 0.01	0.33 ± 0.04	0.10 ± 0.01	0.21 ± 0.02
Measurement 3	0.13 ± 0.01	0.33 ± 0.04	0.12 ± 0.01	0.21 ± 0.02
Measurement 4	0.15 ± 0.02	0.39 ± 0.04	0.11 ± 0.01	0.20 ± 0.02
Measurement 5	0.13 ± 0.01	0.33 ± 0.04	0.08 ± 0.01	0.18 ± 0.02
Measurement 6	0.15 ± 0.02	0.39 ± 0.04	0.10 ± 0.01	0.22 ± 0.02
Measurement 7	0.11 ± 0.01	0.28 ± 0.03	0.13 ± 0.01	0.21 ± 0.02
Mean	0.13 ± 0.01	0.28 ± 0.04	0.11 ± 0.01	0.20 ± 0.02

The results of gross alpha and gross beta activities seem to be less uniformity, indicating these are probably due to some factors:

(i) cross-contaminated during evaporating and drying the samples,
(ii) high background counting,
(iii) high content of residue in sample may be affected to counting efficiency value and activity calculation,
(iv) effect of growth radioactivity from some daughter of the radionuclides,
(v) volatilized of some radionuclide such as Po-210 during evaporating and drying the samples.

However, the gross alpha and gross beta activities still can be measured easily and rapidly using established method in RAS.

Precision/Repeatability

The precision of a measurement analysis can be assessed from some criteria such as the relative standard deviation (RSD), repeatability limit (RL), precision index (%) and acceptance criteria (Table 2). The estimated RSD were 8.93% for gross alpha and 4.23% for gross beta. Refer to those values, the results showed an analysis of the activity of gross alpha and gross beta were less than 10%, thus it can be concluded the precision/ repeatability testing were in a good agreement.

Meanwhile, the repeatability limits for measurement of gross alpha and gross beta were 0.11. However, the precision indexes (PI) showed as a warning limit ($10 < PI < 15$) for both standard solution, this indicating the method still can be used to measure the activity of gross alpha/beta in various types of samples. Furthermore, from the assessment of the acceptance criteria indicated that the method can be accepted to use, whereas the value of absolute mean bias (AMB) for both quality control samples (gross alpha and gross beta) was less than the limit of repeatability (2.8xSD) (Table 2).

Acceptance of the methods can also be determined on the distribution of the measurement values in control chart. Refer to the Figure 1 indicated that 100% of gross alpha and gross beta were distributed within the limits of acceptance (1SD). Thus, to further enhance the precision / repeatability of the gross alpha/beta measurement and their distribution are always within in acceptance zone, control charts should be regularly monitored and improvement action is needed.

Table 2: Precision index, repeatability limit and acceptance criteria for precision/repeatability test in measuring of gross alpha and gross beta activity

No. of measurement	Actual activity (Bq/L)		Measured activity (Bq/L)	
	Gross alpha	Gross beta	Gross alpha	Gross beta
Measurement 1	0.57 ± 0.06	1.45 ± 0.15	0.47 ± 0.05	0.97 ± 0.10
Measurement 2	0.55 ± 0.05	1.39 ± 0.15	0.41 ± 0.04	0.94 ± 0.10
Measurement 3	0.59 ± 0.06	1.51 ± 0.16	0.49 ± 0.05	0.96 ± 0.10
Measurement 4	0.57 ± 0.06	1.45 ± 0.15	0.48 ± 0.05	1.01 ± 0.11
Measurement 5	0.55 ± 0.05	1.39 ± 0.15	0.48 ± 0.05	0.94 ± 0.10
Measurement 6	0.50 ± 0.05	1.28 ± 0.14	0.38 ± 0.04	0.93 ± 0.10
Measurement 7	0.50 ± 0.05	1.28 ± 0.14	0.43 ± 0.04	0.87 ± 0.10
Mean			0.45 ± 0.05	0.95 ± 0.10
$\lvert$Absolute mean bias, AMB$\rvert$			0.10	0.45
Standard deviation (SD)			0.04	0.04
RSD (%)			8.93	4.23
Repeatability limit, RL			0.11	0.11
Precision index, PI (%)			13.82	14.95
Acceptance criteria			AMB < RL	AMB < RL

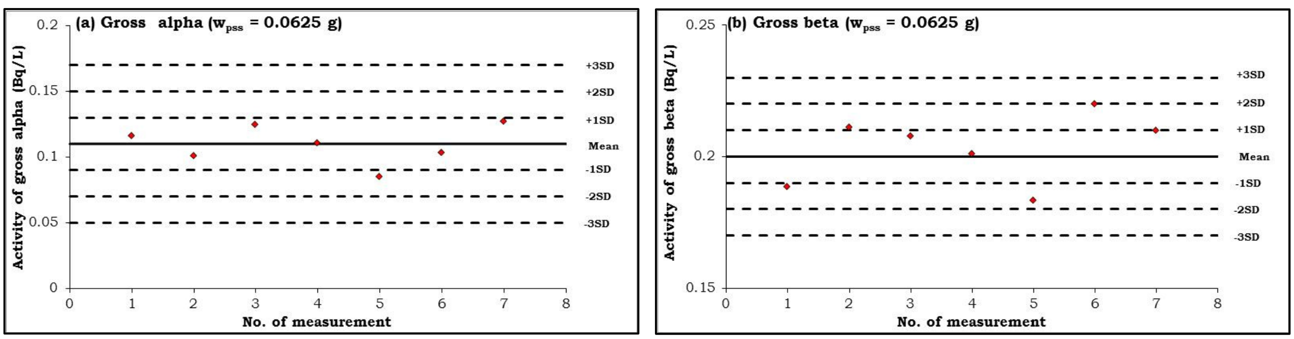

Figure 1: Control chart for radioactivity measurement of gross alpha (a) and gross beta (b) in in-house prepared standard solution

Bias/Accuracy

Bias and accuracy of measurement can be determined via U_{scores} and z-score calculation. According to IAEA (2002) the report result does not significantly differ from the expected or certified value if U_{score} is less than 1.64. Thus, the estimated U_{score} value < 1.64 for gross alpha (U_{score} = 1.43) indicated this method/technique used was not biased and the results generated were not significantly different from the expected or certified value (Table 3). Meanwhile, such result for gross beta (U_{score} = 2.52) was due to some factors that affect the measurement as discussed earlier. However, the accuracy for all measurements which was assessed using z-score analysis found less than 2 (z-score < 2) (Table 3). This can be concluded that the quality measurement of gross alpha and gross beta was reliable (satisfactory) and can be used for any standard solutions.

Table 3: Estimation of U_{score} and z-score for bias/accuracy test in radioactivity measurement of gross alpha and gross beta

No. of measurement	Actual activity (Bq/L)		Measured activity (Bq/L)	
	Gross alpha	Gross beta	Gross alpha	Gross beta
Measurement 1	0.57 ± 0.06	1.45 ± 0.15	0.47 ± 0.05	0.97 ± 0.10
Measurement 2	0.55 ± 0.05	1.39 ± 0.15	0.41 ± 0.04	0.94 ± 0.10
Measurement 3	0.59 ± 0.06	1.51 ± 0.16	0.49 ± 0.05	0.96 ± 0.10
Measurement 4	0.57 ± 0.06	1.45 ± 0.15	0.48 ± 0.05	1.01 ± 0.11
Measurement 5	0.55 ± 0.05	1.39 ± 0.15	0.48 ± 0.05	0.94 ± 0.10
Measurement 6	0.50 ± 0.05	1.28 ± 0.14	0.38 ± 0.04	0.93 ± 0.10
Measurement 7	0.50 ± 0.05	1.28 ± 0.14	0.43 ± 0.04	0.87 ± 0.10
Mean	0.55 ± 0.05	1.39 ± 0.15	0.45 ± 0.05	0.95 ± 0.10
Standard deviation (SD)	0.03	0.08	0.04	0.04
U_{score}			1.43	2.52
z-score			0.88	0.76

Linearity and Working Range

The results of linearity test showed a strong linear correlation between the amount standard solution added and the measured activity for gross alpha and gross beta (Figure 2). The Pearson coefficient, r for gross alpha and gross beta was found to be 0.9951 and 0.9984, respectively. Since the line indicating a high linearity, the line can be extrapolates towards minimum and maximum to extend the working range. Slight deviation for the linearity of gross alpha (0 – 0.2 Bq) and gross beta (0 – 0.5 Bq) could be due to some factors as discussed in specificity test. Thus, this working range at the point 0.2 Bq (gross alpha) and 0.5 Bq (gross beta) for this method will be set as a detection limit if calculations of DL using Curie limit equation is not reasonable. Meanwhile, the values more than 0.2 Bq (gross alpha) and 0.5 Bq (gross beta) were fit as a working range for radioactivity measurement of gross alpha and gross beta. For the preliminary conclusion, this method is very suitable to measure a large range from the detection limit to higher activity which is more than environmental levels.

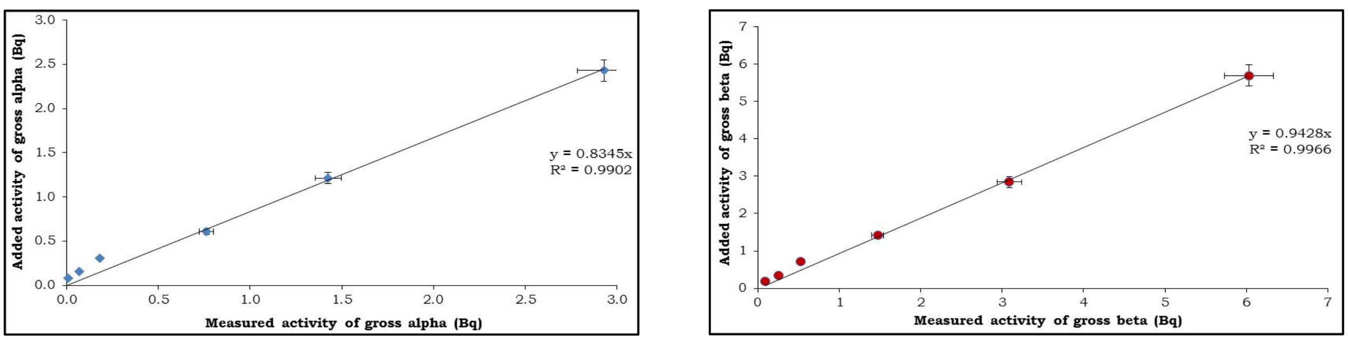

Figure 2: Linearity test curve for gross alpha (left) and gross beta (right) activity

Detection Limit (DL)

Generally, the DL estimated for activity measurement of gross alpha and gross beta in standard solution in respectively was 0. 1 Bq/L and 0.2 Bq/L with the background counting of 0.01 – 0.35 cpm (gross alpha) and 0.5 – 2.00 (gross beta) and counting time of 100 minutes. In order to get a lower MDA, counting time for sample should be extended at-least 2 hours. However, the counting time of 60 minutes has been sufficient for this method to measure quantitatively the activity of gross alpha and gross beta in water samples.

Robustness/Sensitivity

The results of the measurement for robustness/sensitivity test were summarized in Table 4 (different instruments) and Table 5 (different operators).

i) Different instrument

Table 4 showed the mean value for the measurement between actual activity and measured activity was quite close for each instrument, indicated that both instrument are good performance and efficient to measure variation activity of gross alpha and gross beta. The RSDs for both instruments were far below than 30% (set for gross alpha and gross beta laboratory). Refer to the z-score analysis, they were found less than 2 (z-score < 2) resulting that the quality of measurement for gross alpha and gross beta was reliable (satisfactory) using any instruments. Since there are different values resulting from both measurements, this probably related to some factors such as background counting, counting efficiency, etc. Thus, this mean that the slight different among the measurement results found still can be tolerance.

Table 4: Characterization of robustness test for measuring the activity of gross alpha and gross beta using different instrument

No. of measurement	Actual activity (Bq/L)		Measured activity (Bq/L)			
			Instrument 1		Instrument 2	
	Gross alpha	Gross beta	Gross alpha	Gross beta	Gross alpha	Gross beta
Measurement 1	0.68 ± 0.07	1.73 ± 0.18	0.55 ± 0.05	1.21 ± 0.13	0.50 ± 0.05	1.31 ± 0.13
Measurement 2	0.57 ± 0.06	1.45 ± 0.15	0.47 ± 0.05	0.97 ± 0.10	0.38 ± 0.03	1.07 ± 0.10
Measurement 3	0.55 ± 0.05	1.39 ± 0.14	0.41 ± 0.04	0.94 ± 0.10	0.35 ± 0.03	1.05 ± 0.10
Measurement 4	0.57 ± 0.06	1.45 ± 0.15	0.53 ± 0.05	1.02 ± 0.11	0.38 ± 0.03	1.14 ± 0.11
Measurement 5	0.59 ± 0.06	1.51 ± 0.16	0.49 ± 0.05	0.96 ± 0.10	0.43 ± 0.04	1.09 ± 0.11
Measurement 6	0.57 ± 0.06	1.45 ± 0.15	0.48 ± 0.05	1.01 ± 0.11	0.35 ± 0.03	1.18 ± 0.12
Measurement 7	0.68 ± 0.07	1.73 ± 0.18	0.48 ± 0.05	1.16 ± 0.12	0.41 ± 0.04	1.30 ± 0.13
Measurement 8	0.55 ± 0.05	1.39 ± 0.14	0.48 ± 0.05	0.94 ± 0.10	0.47 ± 0.05	1.11 ± 0.11
Mean	0.59 ± 0.06	1.51 ± 0.16	0.49 ± 0.04	1.03 ± 0.11	0.41 ± 0.04	1.16 ± 0.11
Standard deviation (SD)			0.04	0.10	0.05	0.09
RSD (%)			8.16	9.71	9.75	7.76
z-score			0.77	0.81	0.91	0.89

ii) Different operator

The mean value for the measurement carried out by different operator was quite close between respective actual activity and measured activity (Table 5). This indicating the mean value from the measurement for both operators was still close to their actual activity. While, the RSDs for both operators were below than 30%, this proven the precision degree of measurement by both operators is good. All the z-score values were less than 2, displayed the quality of measurement for gross alpha/beta activities carried out by both operator was acceptable (satisfactory) and reliable. Align with that, operators were competence and well trained. Generally, this can be concluded the different characterization of instrument and operator consider being in a good agreement with robustness test.

Table 5: Characterization of robustness test for measuring the activity of gross alpha and gross beta by different operators

No. of measurement	Operator 1				Operator 2			
	Actual activity (Bq/L)		Measured activity (Bq/L)		Actual activity (Bq/L)		Measured activity (Bq/L)	
	Gross alpha	Gross beta	Gross alpha	Gross beta	Gross alpha	Gross beta	Gross alpha	Gross beta
Measurement 1	0.68 ± 0.07	1.73 ± 0.18	0.55 ± 0.05	1.21 ± 0.13	0.50 ± 0.07	1.28 ± 0.13	0.38 ± 0.04	0.93 ± 0.09
Measurement 2	0.57 ± 0.06	1.45 ± 0.15	0.47 ± 0.05	0.97 ± 0.10	0.48 ± 0.06	1.23 ± 0.13	0.43 ± 0.04	0.77 ± 0.07
Measurement 3	0.55 ± 0.05	1.39 ± 0.14	0.41 ± 0.04	0.94 ± 0.10	0.50 ± 0.07	1.28 ± 0.13	0.43 ± 0.04	0.87 ± 0.08
Measurement 4	0.57 ± 0.06	1.45 ± 0.15	0.53 ± 0.05	1.02 ± 0.11	0.48 ± 0.06	1.23 ± 0.13	0.36 ± 0.03	0.96 ± 0.09
Measurement 5	0.59 ± 0.06	1.51 ± 0.16	0.49 ± 0.05	0.96 ± 0.10	0.48 ± 0.06	1.23 ± 0.13	0.28 ± 0.03	0.64 ± 0.06
Measurement 6	0.57 ± 0.06	1.45 ± 0.15	0.48 ± 0.05	1.01 ± 0.11	0.48 ± 0.06	1.23 ± 0.13	0.36 ± 0.03	0.81 ± 0.08
Measurement 7	0.68 ± 0.07	1.73 ± 0.18	0.48 ± 0.05	1.16 ± 0.12	0.52 ± 0.07	1.34 ± 0.14	0.37 ± 0.04	0.76 ± 0.07
Measurement 8	0.55 ± 0.05	1.39 ± 0.14	0.48 ± 0.05	0.94 ± 0.10	0.48 ± 0.06	1.23 ± 0.13	0.31 ± 0.03	0.76 ± 0.07
Mean	0.59 ± 0.06	1.51 ± 0.16	0.49 ± 0.04	1.03 ± 0.11	0.49 ± 0.06	1.26 ± 0.13	0.36 ± 0.04	0.81 ± 0.08
Standard deviation, SD			0.04	0.10			0.05	0.10
RSD (%)			8.16	9.71			13.88	12.35
z-score			0.77	0.81			0.61	0.79

Ruggedness / Reproducibility

The results of the ruggedness/reproducibility test at different counting time and sample volume are summarized in Table 6 (gross alpha) and Table 7 (gross beta). Generally, the mean value of gross alpha and gross beta activity by changing some parameters such as counting time and sample volume showed reliable results and quite closed to the mean value of actual condition (100 minutes and 500 mL). Furthermore, the RSDs for all analysis were far below than 30% (RSD < 10%), this indicating the results found to be precise and consistence. Thus, a method/technique developed was found to be rugged/strength, efficient and can be reproducibility as well as ability to measure the gross alpha and gross beta from various radionuclides include their daughter even though there has some changes of parameter during analysis.

Table 6: variation of parameters to test the ruggedness/reproducibility for measuring the activity of gross alpha

No. of Measurement	Activity of gross alpha (Bq/L)					
	Counting time (minutes)			Sample volume (mL)		
	95	100	105	495	500	505
Measurement 1	0.15 ± 0.01	0.42 ± 0.04	0.25 ± 0.02	0.43 ± 0.04	0.42 ± 0.04	0.16 ± 0.01
Measurement 2	0.22 ± 0.02	0.45 ± 0.04	0.32 ± 0.03	0.26 ± 0.02	0.45 ± 0.04	0.28 ± 0.03
Measurement 3	0.16 ± 0.01	0.42 ± 0.04	0.28 ± 0.02	0.33 ± 0.03	0.42 ± 0.04	0.33 ± 0.03
Measurement 4	0.20 ± 0.02	0.45 ± 0.04	0.36 ± 0.03	0.24 ± 0.02	0.45 ± 0.04	0.26 ± 0.02
Measurement 5	0.21 ± 0.01	0.43 ± 0.04	0.32 ± 0.03	0.22 ± 0.03	0.43 ± 0.04	0.27 ± 0.02
Mean	0.19 ± 0.01	0.43 ± 0.04	0.31 ± 0.03	0.30 ± 0.03	0.43 ± 0.04	0.26 ± 0.02
Standard deviation (SD)	0.03	0.01	0.04	0.08	0.01	0.06
RSD (%)	15.79	2.33	12.90	26.67	2.33	23.08

Table 7: variation of parameters to test the ruggedness/reproducibility for measuring the activity of gross beta

No. of Measurement	Activity of gross beta (Bq/L)					
	Counting time (minutes)			Sample volume (mL)		
	95	100	105	495	500	505
Measurement 1	1.45 ± 0.14	1.09 ± 0.10	1.10 ± 0.11	1.06 ± 0.10	1.09 ± 0.10	0.91 ± 0.09
Measurement 2	1.48 ± 0.14	1.05 ± 0.10	1.10 ± 0.11	0.83 ± 0.08	1.05 ± 0.10	0.80 ± 0.08
Measurement 3	1.37 ± 0.13	1.09 ± 0.10	1.12 ± 0.11	0.89 ± 0.08	1.09 ± 0.10	0.93 ± 0.09
Measurement 4	1.50 ± 0.14	1.17 ± 0.11	1.31 ± 0.13	0.85 ± 0.08	1.17 ± 0.11	0.82 ± 0.08
Measurement 5	1.40 ± 0.13	1.03 ± 0.10	1.06 ± 0.10	0.92 ± 0.09	1.03 ± 0.10	0.82 ± 0.08
Mean	1.44 ± 0.04	1.09 ± 0.10	1.14 ± 0.11	0.91 ± 0.09	1.09 ± 0.10	0.86 ± 0.08
Standard deviation (SD)	0.05	0.05	0.09	0.08	0.05	0.05
RSD (%)	3.47	4.59	7.90	8.79	4.59	5.81

CONCLUSION

Developed method/technique for the measurement of gross alpha/beta activity in bottled drinking water, mineral water and environmental samples using Low Background Gross Alpha/Beta Counting System was compliance with the instrument always good performed and characteristics of precision, accuracy and reliable results. Generally, results of the method validation found no doubtful on the ability of the developed method. It has met all the test parameters that were carried out. The test result showed the method was suitable for all types of water samples which are contained several radionuclides and elements as well as any impurities or substances that interfere the measurement analysis of gross alpha/beta.

Almost the values of estimated RSD, z-score and U_{scores} were accuracy which is recorded as $\leq$ 30%, less than 2 and less than 1.5, respectively. This proven the accuracy and precision result that are generated from developed method/technique is satisfactory and method is recommended to be used. Minimum detection activity (MDA) was estimated based on the counting time of 100 minutes and present background counting value of gross alpha (0.01 – 0.35 cpm) and gross beta (0.50 - 2.18 cpm). Estimated detection limit (DL) was 0.1 Bq/L for gross alpha and 0.2 Bq/L for gross beta. Align with that, background counting for gross alpha and gross beta was ranged of 0.01 – 0.35 cpm and 0.50 – 2.18 cpm, respectively. While, sample volume was set at minimum of 500 mL and maximum of 2000 mL.

Generally, in order to optimize the suitability of the methods/techniques for generating more accurate, precision and reliable results, it is necessary to perform an analysis according to work instruction based on some important parameters such as counting time, sample weight/volume etc.

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