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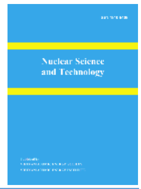

# Study on the repeatability and reproducibility of low-level radioactivity measurements using the analysis of variance tool

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**Abstract:** The repeatability and reproducibility need to be investigated to evaluate the stability of low-level radioactivity measurements with respect to an analysis method, as well as their dependence on measuring parameters. In this study, the procedures for gross alpha/beta and gamma-ray activity measurements were investigated in the Department of Radiation and Environment Safety at the Center for Nuclear Technologies (CNT). Moreover, an interlaboratory comparison was organized with other institutions, i.e. Dalat Nuclear Research Institute (DNRI), Institute for Nuclear Science and Technology (INST), and Institute of Public Health in HCM (IPH-HCM). To evaluate the repeatability and reproducibility of measures undertaken by the low-level radioactivity measurements, the certified reference materials (CRM), i.e. IAEA-CRM-385 (Natural and artificial radionuclides in sediment from the Irish Sea), NIST-SRM-4322c (Americium-241 Radioactivity Standard), and IAEA-CRM-RGK-1, together with 6 collected real samples were analyzed for $^{238}$U, $^{232}$Th, $^{226}$Ra, $^{228}$Ra, $^{210}$Pb and $^{40}$K. The analysis of variance (ANOVA) tool with respect to the average, standard deviation, *F*, *p*-value, and *F*-crit parameters was applied for the evaluation. The obtained results in the study revealed that the repeatability and reproducibility were stable for the low-level radioactivity measurements at the CNT which meets the laboratory's quality management requirements.

**Keywords**: *Low-level radioactivity, repeatability, reproducibility, ANOVA, NIST-4322c, IAEA-385, IAEA-RGK-1.*

## I. INTRODUCTION

Implementing a quality management system based on the requirements specified in ISO/IEC 17025 standards at low-level radioactivity measurement laboratories is challenging, mainly due to the fact that these laboratories provide the environmental samples testing servise together with the implementation of research and development activities [1, 2].

Analysis of radionuclides in environmental samples is carried out daily in many laboratories. These data are used for a variety of purposes including environmental surveys and dose assessment in man and the environment, in decision making on economic and health aspects. It is important to ensure the accuracy and precision of the analysis results to guarantee that decisions are based on reliable results. The increasing need for reliable data creates a concomitant need for a quality management system to support the acquisition of precise, accurate data. A vital element of data defensibility, objective evidence of precision, and accuracy is essential to the accomplishment of any environmental program

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that depends on analytical data. For that purpose, effective quality management is necessary to maintain high-quality results. Documentation of the laboratory is planned, described and performed systematically, recorded, and reviewed [3, 4, 5].

Measurement of the total activity of the calibration source is used to check the efficiency calibration and general operating parameters of the system. The detector-shield background, detector efficiency, peak shape and, peak drift are measured and verified if they are within the acceptance limits [6].

Also, an external control can be implemented in the form of various interlaboratory proficiency tests and intercomparisons. The analysis of results achieved in the proficiency tests and control charts of the laboratory z-score give participants the possibility of a long-term follow-up of their performance promoting the improvement of the quality management system [7, 8, 9, 10].

A quality management system should address an organization's unique needs, however, the elements all systems have in common include: The organization’s quality policy, and quality objectives; Quality manual; Procedures, instructions, and records. Moreover, it is necessary to add the selection and validation of analytical methodology; the resources used for the analysis; and the laboratory operations for sample handling [11, 12, 13].

All of these proposed measures were implemented in the Department of Radiation and Environment Safety, Center for Nuclear Technologies (CNT). Since the operation of this department includes daily measurements of a large number of samples, it is essential to have a stable and accurate measuring system, so that the results are accurate, precise and repeatable. The test needs to be recognized as conforming to the ISO17025 system so that the analysis results issued by CNT's testing laboratory to the user will be recognized by other testing laboratories. The procedures are implemented for gross alpha/beta and gamma-ray activity measurements. The Series 5 XLB Automatic Low Background Alpha/Beta Counting System (Canberra, USA) and the gamma-ray spectrometer using an HP Germanium detector GC-5519 (Canberra, USA) are readily controlled in terms of accuracy, precision, and repeatability of the results, as well as the stability of the instruments. Also, the laboratory organized an interlaboratory with other institutions, i.e. the Dalat Nuclear Research Institute (DNRI), the Institute of Nuclear Science and Technology (INST), and the Institute of Public Health in HCMC (IPH-HCM).

The evaluation of repeatability and reproducibility aim to prove that the capacity of the testing laboratory for low-level radioactivity measurements at CNT has been in accordance with requirements of the ISO/IEC-17025:2017.

## II. METHODOLOGY

Statistical process control is a collection of methodologies for measuring the quality of goods and services, as well as the measures [14]. A variety of statistical methodologies may be used to examine repeatability and reproducibility [15]. The difference in measurements is obtained while measuring the same object repeatedly is known as repeatability. The variability of the measuring system caused by variances in operator behavior is referred to as reproducibility [16].

To evaluate the correlation between two data sets, the *F*-test and *t*-test can be utilized. The *F*-test (*F*-Fisher standard) is used to examine the repeatability of two data sets, whereas the *t*-test (*T*-Student standard) is used to compare the average values of two data sets. Simply defined and similar results must be obtained when measurements are performed on the same sample using the same or different methodologies [17].

The equation for calculation of the experimental *F* value:

$$F = \frac{S_1^2}{S_2^2}$$

In which, the equation for calculation of the variance on each set of data ($S^2_1$, $S^2_2$):

$$S_x^2 = \frac{1}{n}\left[n_1(x_1-\overline{x})^2 + n_2(x_2-\overline{x})^2 + \cdots + n_k(x_k-\overline{x})^2\right]$$

Where:

*F*: Experimental *F* value.

$S^2_1$ , $S^2_2$: Variations of two sets of data

If: $F \le F$-crit ($\alpha$, $k_1$, $k_2$): The two sets of data have the same repeatability (precision).

*F*-crit ($\alpha$, $k_1$, $k_2$): search through the *F*-crit value table.

$k_1$, $k_2$: degree of freedom ($k_1$ = $n_1$-1; $k_2$ = $n_2$-1)

$n_1$, $n_2$: numbers of experimental runs for two data sets

$\alpha$: significance level,

It is important to compare two variances (Fisher function) prior to comparing two average values. The standard deviation and *t*-stat value (*t*-experimental) were calculated by the following formula when the two variances are consistent ($F \le F$-crit), then the results were compared with the $t_c$ value (as it can be seen in the table):

$$S_c^2 = \frac{(n_1-1)S_1^2 + (n_2-1)S_2^2}{n_1+n_2-2}$$

$$t - Stat = \frac{|\overline{x_1} - \overline{x_2}|}{S_c^2\left(\frac{1}{n_1}+\frac{1}{n_2}\right)}$$

In which,

*t*-stat: *t*-experimental value;

$t_c$ ($\alpha$, $k$): value *t* looks at the table of significance level $\alpha$, degrees of freedom $k$, $k = n_1 + n_2 - 2$;

$n_1$, $n_2$: numbers of experimental runs for two data sets;

$S^2_1$ , $S^2_2$ : variations of two sets of data;

$\bar{x}_1, \bar{x}_2$: average value of two sets of data;

If *t*-stat $\le t_c$ ($\alpha$, $k$): there is no difference in the results of the two sets of data.

If *t*-stat $> t_c$ ($\alpha$, $k$): there is a difference in the results of the two sets of data.

Nevertheless, the *t*-test should not be used in this situation when there are more than two sets of data to compare since it produced more “false positive” findings and increase the total number of comparison pairs that must be generated. The analysis of variance (ANOVA) method is the most precise way to evaluating reproducibility and repeatability in this case. Microsoft Excel is the most basic ANOVA analysis tool. The ANOVA table [16] is shown in Table I.

**Table I.** The ANOVA table

| **Source of Variation Appraiser** | **Sum of Squares *SS*** | **Degrees of Freedom *df*** | **Mean Square MS** | ***F*** | **Significance *p*-value** | ***F*-crit** |
|---|---|---|---|---|---|---|
| Between Groups | SSA | *k*-1 | $MSA = \frac{SSA}{k-1}$ | $F = \frac{MSA}{MSE}$ | FDIST(F,d1,d2) | *F*-table value |
| Within Groups | SSE | *n*-*k* | $MSE = \frac{SSA}{n-k}$ | | | |
| Total | SST | *n*-1 | | | | |

$$SSA = \sum_{j=1}^{k} n_j (\overline{x_j} - \overline{x})^2$$

$$SSE = \sum_{j=1}^{k}\sum_{i=1}^{n_j} (x_{ij} - \overline{x}_j)^2$$

$$SST = \sum_{j=1}^{k} n_j (\overline{x}_j - \overline{x})^2 + \sum_{j=1}^{k}\sum_{i=1}^{n_j} (x_{ij} - \overline{x}_j)^2 = SSA + SSE$$

$k$ = number of appraisers;

$n$ = the number of trials.

The purpose of the analysis of variance is to verify if the population means are equal. The answer for hypothesis testing will be based on the *F*-value and the *p*-value compared to the value of *F*, obtained from statistical tables or software. If $F \leq F$-crit, the two sets of data have the same repeatability (precision). In other words, the null hypothesis means that the variance and mean are equal. Conversely, if $F > F$-crit, there is a difference. When $F \leq F$-crit we compare the *p*-value with the significance level $\alpha = 0.05$, and we can decide whether to accept or reject the null hypothesis. Specifically, we accept the null hypothesis if the *p*-value is greater than the significance level $\alpha$ [17]. The *p*-value can be found by using the function FDIST (*F*-statistic,d1,d2) in Microsoft Excel. The evaluation is done by comparing the *F* and *F*-crit, and the *p*-value as described above. If the criteria are met, the testing results will be "accepted".

## III. EXPERIMENTAL

In this work, the accuracy, repeatability, and reproducibility of low-level radioactivity measurements at the Center for Nuclear Technologies were evaluated by analyzing several certified reference materials (CRMs) and some collected samples. All of these proposed measures were performed on the gamma-ray spectrometer using an HP Germanium detector GC-5519 (Canberra, USA) and on the Series 5 XLB Automatic Low Background Alpha/Beta Counting System (Canberra, USA). In addition, some samples were measured at DNRI, INST, and IPH-HCM for interlaboratory comparison.

For the determination of gamma-ray emitting radioactive in soil [18], TCVN 10758-3 (ISO 18589-3) was applied. The samples were pre-treated on-site, which included the removal of large stones and organic materials. After being dried at 40°C to a consistent dry weight, the soil samples were crushed and homogenized with a sieved (1 mm in diameter). The homogenized soil samples were sealed in containers for 21 days to achieve radioactive equilibrium and be ready for gamma-ray measurements. Each sample was measured 24 hours in Marinelli geometry on the HPGe GC-5519 gamma-ray spectrometer, which features a relative efficiency of 55% and the detector energy resolution of 1.9 keV at the 1332 keV of Co-60 gamma-ray peak. The analysis was performed using Genie 2000 software.

Gross alpha/beta radioactive activity is measured using the standard methods TCVN 6053 (ISO 9696) and TCVN 6219 (ISO 9697). The two primary processes in the alpha-beta measurement procedure are drying out the water and measuring the alpha and beta in the residue that is collected using a Gas Flow Proportional Counters. To be more precise, the total dissolved solids (TDS) concentration should be ascertained following the collection of a 1-liter water sample. To obtain the appropriate amount of residue, the necessary amount of CaSO4 should be added if the TDS is less than 100 mg/L. Evaporate the sample on the electric stove until 50mL of water remains. After that, the sample was sulfated and burnt to ash. The radioactivity of ash samples was tested by using the Alpha and Beta counting system. Gross Alpha, Beta Measurement System Model: S5XLBPF of Canberra Company, USA, serial number 13000650 was used for the measurements. The system employs Gas Flow Proportional Counters with an 80 g/cm$^2$ detector window thickness, beta background of 0.7-0.9 cpm, alpha background of 0.05 – 0.1 cpm, beta counting efficiency of 37% for Sr-90/Y-90 sources, and alpha counting efficiency of 29% for Am-241 sources. The device has an automated measuring mode that allows for the storage of 50 measurement samples.

To evaluate the accuracy and the reproducibility of the radioactivity measurement of gamma-ray and gross alpha/beta, the IAEA-CRM-385 (Natural and artificial radionuclides in sediment from the Irish sea) and liquid reference samples were measured on gamma-ray and gross alpha/beta spectrometer of the Center for Nuclear Technologies (CNT). NIST-SRM-4322c (Americium-241 Radioactivity Standard) was added to 20 liters of tap water to create the gross alpha measurement sample, the sample was coded as TA-01. A total of 19 liters of tap water dissolved with IAEA-CRM-RGK-1 (Potassium sulfate) made up the beta measurement sample labeled as TB-01. These samples were subsequently dispatched to the following affiliated laboratories: DNRI, INST, and IPH-HCM.

To evaluate the repeatability for gamma-ray measurement, approximately 2-4 kg of surface soil samples were collected in Dak Lak province To evaluate the Repeatability for gross alpha/beta, the water sample collection includes the followings: surface water at Saigon River; groundwater in Tay Ninh and Ho Chi Minh City; bottled mineral water; domestic wastewater at Tan Quy Dong wastewater treatment plant, District 7, Ho Chi Minh City; and tap water samples taken at the Center for Nuclear Technologies. At different periods, the technicians analyzed collected samples by using the same method and equipment. ANOVA was used to determine the variability among technicians.

To evaluate the repeatability, real samples (a soil sample for gamma-ray measurement, 6 water samples for total alpha-beta activity measurement) were analyzed by the CNT laboratory. Additionally, to evaluate the repeatability, standard samples were coded and sent to other laboratories for analysis to compare with the CNT laboratory results.

## IV. RESULTS AND DISCUSSION

A quality management system and procedures were established for carrying out standard quality examinations, intra-laboratory comparisons, and inter-laboratory comparisons, which were demonstrated below.

To evaluate the repeatability, the experimental samples were prepared as described in the previous section, and two employees carried out the experiment according to the established procedure, each of which consisted of six examinations, then y undertook six additional examinations and the results were recorded by the principle examiner. Comparative evaluation of similarity using the ANOVA method of analysis of variance was done by using Excel. The Between Groups line in the Excel ANOVA table was examined to determine the *F* and *F*-crit values from which the results are reported in Tables II and III.

**Table II.** Results of the real sample analysis by using the gamma-ray spectrometer and the evaluation of the result for the sample among analysts

| **Radionuclides** | **Analysis results** | | **Criteria parameter** | | | **Evaluation (*)** |
|---|---|---|---|---|---|---|
| | *Average* | *S.D.* | *F* | *p*-value | *F*-crit | |
| $^{238}U$ | 22.14 | 0.36 | 0.00 | 0.96 | 4.96 | Accepted |
| $^{232}Th$ | 35.82 | 0.41 | 0.05 | 0.82 | 4.96 | Accepted |
| $^{226}Ra$ | 18.72 | 0.35 | 0.35 | 0.57 | 4.96 | Accepted |
| $^{228}Ra$ | 42.49 | 0.36 | 0.32 | 0.58 | 4.96 | Accepted |
| $^{210}Pb$ | 24.86 | 0.24 | 0.17 | 0.69 | 4.96 | Accepted |
| $^{40}K$ | 53.14 | 0.26 | 0.15 | 0.70 | 4.96 | Accepted |

**Table III.** Result of the real sample analysis using the gross alpha/beta radioactivity and the evaluation of the result of the sample analysis among analysts

| **Gross beta/alpha radioactivity (Bq/L)** | **Samples** | **Analysis results** | | **Criteria parameter** | | | **Evaluation*** |
|---|---|---|---|---|---|---|---|
| | | *Average* | *S.D.* | *F* | *p*-value | *F*-crit | |
| Gross beta | Ground water 1 | 0.167 | 0.008 | 0.018 | 3.682 | 0.982 | Accepted |
| | Waste water | 0.415 | 0.013 | 0.500 | 3.682 | 0.616 | Accepted |
| | Bottled water | 0.190 | 0.011 | 0.044 | 3.682 | 0.957 | Accepted |
| | Tap water | 0.057 | 0.005 | 0.026 | 3.682 | 0.974 | Accepted |
| | Raw water | 0.174 | 0.007 | 0.058 | 3.682 | 0.944 | Accepted |
| Gross alpha | Ground water 2 | 0.083 | 0,003 | 0.188 | 3.682 | 0.831 | Accepted |

*Accepted is evaluated by comparing the *F* and *F*-crit, and the *p*-value as described in the above mentioned section.

Considering the confidence interval of 95%, it means α = 0.05. Observing the two data tables above, we can see that all *F* values were smaller than *F*-crit and *p*-value was greater than α. In this instance, we can therefore conclude that the null hypothesis - there was no discernible difference between the performed tests - was reasonable to accept. In other words, at the 95% confidence level, the evaluation results showed no difference in repeatability between the two examiners.

The TA-01 and TB-01 samples were delivered to other laboratories for analysis. The data were divided into two categories: the interlaboratory and internal, and the similarity of the results between the two groups was evaluated using the *F*-test and the *t*-test. The *F*-test (*F*-Fisher standard) was used to assess the reproducibility of two sets of data, whereas the *t*-test (*t*-Student standard) was used to compare two data sets' mean values. The following tables provide results and evaluations.

The measurement results for evaluation of the accuracy and reproducibility of the radioactivity measurements of gamma-ray and gross alpha/betas are displayed in Tables IV and V.

**Table IV.** The results of gamma-ray measurements on the IAEA-385 CRM sample of testing laboratories and the evaluations

| **Laboratories*** | **Inter-Laboratory results** (Bq/kg) | | | |
|---|---|---|---|---|
| | $^{40}K$ | $^{137}Cs$ | $^{238}U$ | $^{232}Th$ |
| DNRI | 674.40 | 20.14 | 28.63 | 32.70 |
| | 644.00 | 19.64 | 28.41 | 35.03 |
| INST | 609.90 | 18.40 | 26.69 | 34.10 |
| | 608.30 | 19.10 | 26.76 | 33.60 |
| IPH-HCM | 573.19 | 17.73 | 25.80 | 31.83 |
| CNT | 617.72 | 19.44 | 28.63 | 33.60 |
| | 606.80 | 19.57 | 28.41 | 32.51 |
| | 602.19 | 19.42 | 26.69 | 31.62 |
| | 608.00 | 19.60 | 26.76 | 32.30 |
| | 588.00 | 19.30 | 25.80 | 31.90 |
| *F* | 4.71 | 2.57 | 3.16 | 3.05 |
| *F*-Critical | 6.39 | 6.39 | 6.39 | 6.39 |
| *t*-Stat | 0.53 | 0.95 | -2.45 | 1.64 |
| *t*-Critical | 2.31 | 2.31 | 2.31 | 2.31 |
| Evaluation | accepted | accepted | accepted | accepted |

**Table V.** The results of gross alpha/beta activity in water samples TA-01 and TB-01 analyzed by different laboratories

| **Laboratories*** | **Inter-laboratory results** | |
|---|---|---|
| | *Gross beta radioactivity in TB-01 (Bq/L)* | *Gross alpha radioactivity in TA-01 (Bq/L)* |
| DNRI | 2.53 | 0.20 |
| | 2.41 | 0.19 |
| INST | 2.59 | 0.22 |
| | 2.82 | 0.23 |
| IPH-HCM | 2.56 | 0.20 |
| | 2.42 | 0.21 |
| CNT | 2.64 | 0.20 |
| | 2.35 | 0.19 |
| | 2.47 | 0.22 |
| | 2.54 | 0.20 |
| | 2.49 | 0.21 |
| | 2.48 | 0.19 |
| *F* | 2.18 | 1.45 |
| *F*-Critical one-tail | 5.05 | 5.05 |
| *t*-Stat | 0.81 | 0.94 |
| *t*-Critical | 2.23 | 2.22 |
| Evaluation | accepted | accepted |

*DNRI: Dalat Nuclear Research Institute; INST: Institute for Nuclear Science and Technology; IPH-HCM: Institute of Public Health in Ho Chi Minh City.

According to the evaluated results displayed in Tables 4 and 5, all data show that: $F < F$-crit and $t$-Stat < $t$-Critical, consequently the results of the two sets of data were comparable. At 95% confidence, the results of this study reveal that that there was no significant difference in the testing capacity of registered criteria between internal data and data from external laboratories.

The results show that the considerable stability of the measuring system with accuracy, precision, and repeatability of the measurement results was acceptable [19]. In the situations where the measured value exceeded the acceptability limits, an adequate correction procedure was implemented.

## V. CONCLUSIONS

The obtained results in the study revealed that the repeatability and reproducibility were stable for the low-level radioactivity measurements at the CNT which meets the laboratory's quality management requirements. The laboratory has organized an interlaboratory comparison program. The evaluated results were acceptable which serves as a confirmation of the reliability of the measurements conducted in this laboratory. The evaluation results of repeatability and reproducibility have proven that the capacity of the testing laboratory at CNT has been in accordance with requirements of the ISO/IEC-17025:2017.

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