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Method Article

Performance testing and comparative study of natural radioactivity in soil samples using high purity germanium (HPGe) detector[☆]

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A B S T R A C T

High Purity germanium (HPGe) detectors are found to be suitable for nuclear techniques for measuring radionuclides with very good energy resolution. Inter-comparison exercise is an important tool for external quality control that enables determination of the accuracy and uncertainty of detector measurement system. In this work, a comparative study of natural radioactivity in soil samples was conducted between the laboratory of Autorité nationale de Radioprotection et de Sûreté Nucléaire (ARSN), Burkina Faso and the laboratory of Radiation Protection Institute of Ghana Atomic Energy Commission (RPI-GAEC), Ghana to ascertain the reliability and accuracy of measurements made in Burkina Faso. For this purpose:

- Some replicate soil samples, assumed as proficiency test samples, were analyzed on both the High Purity Germanium detector of ARSN and RPI.
- The statistical performance indicators of z-score, precision, trueness and relative bias were used for the evaluation. The limit for acceptable precision and the maximum acceptable bias for all the radionuclides were set to 20% and 15% respectively.

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A R T I C L E I N F O

Method name: Comparative analysis of environmental samples for performance testing*Keywords:* NORM, Indicators, RPI, ARSN, Laboratory*Article history:* Received 5 April 2021; Accepted 24 May 2021; Available online 25 May 2021

[☆] The performance of the gamma spectrometry system of ARSN laboratory was found to be satisfactory when compared to the result of RPI and its measurement results accurate and reliable.

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Specifications table

Subject Area:	Environmental Science
More specific subject area:	Radiological monitoring of the environment
Method name:	Comparative analysis of environmental samples for performance testing
Name and reference of original method:	[1] Guembou, J. S. C., Samafou, P., Moyo, M. N., Gregoire, C., Mekontso, E. J. N., Ebongue, A. N., Ousmanou, M., & David, S. (2017). Precision measurement of radioactivity in gamma-rays spectrometry using two HPGe detectors (BEGe-6530 and GC0818-7600SL models) comparison techniques: Application to the soil measurement. <i>MethodsX</i> , 4, 42–54. https://doi.org/10.1016/j.mex.2016.12.003
	[2] El-gamal, H., Negm, H., Hasabelnaby, M., & El-gamal, H. (2019). Detection efficiency of NaI (TI) detector based on the fabricated calibration of HPGe detector. <i>Journal of Radiation Research and Applied Sciences</i> , 12(1), 360–366. https://doi.org/10.1080/16878507.2019.1672313
Resource availability:	Not applicable

Method details

The inter-laboratory comparison exercise is one of the most important tools used to ascertain the accuracy and reliability of measurement results from any laboratory [3]. In the evaluation reports of inter-laboratory comparison exercises, the organizer guides the participants on appropriate measurement techniques to use, and detects the non-conformities [3]. The resolution of the non-conformities found will improve the methods employed by the participants as well as the performance of their systems [3].

The samples used in an inter-comparison should have roughly the same composition and concentration as the samples generally analyzed [4]. The agreement of the results obtained by a particular laboratory with the target value is the measure of the accuracy for these measurement results [4].

The laboratory of ARSN-Burkina Faso was established in 2018 for the purpose of environmental radiation monitoring. The laboratory provides some services such as monitoring of mining areas for the assessment of the level of NORM radionuclides. The ARSN laboratory intends to start the monitoring of imported food and other items from developed countries for the assessment of natural and artificial radionuclides. The objective is to ensure the protection and safety of the population and the environment associated with the effect of radiological hazards to the populace.

Since the establishment of the ARSN laboratory there has not been any comparison study with other laboratories in or outside the African region, to check the accuracy and reliability of measurements made in the country in order to determine the sensitivity or performance of the HPGe. A comparative study of natural radioactivity in soil samples will therefore help to determine the reliability and accuracy associated with the measured results for the measurements made with both HPGe detector from ARSN (Burkina Faso) and RPI-GAEC (Ghana).

In this study, some replicate soil samples, used as proficiency test samples (spiked samples), were distributed among the two laboratories of ARSN and RPI for NORM measurement. The assumption made will enable the use of the performance criteria or indicators commonly used by IAEA in proficiency test and inter-laboratory comparison exercises as tools for evaluation the performance of ARSN and the credibility of its measurement results.

Material and methods

Characteristics of the study area

Villy lies on the latitude 12°16'46" N and the longitude 2°09'53" W. It is a village located in the Boulkiemde province at about 90 km from Ouagadougou, the capital.

The Boulkiemde province belongs mainly to the Precambrian D formations and to a lesser extent the Precambrian C [5]. Magmatites, granites and a few greenstone intrusions make up the bulk of the geological formations of the area [5]. The province has a tropical climate characterized by a long dry

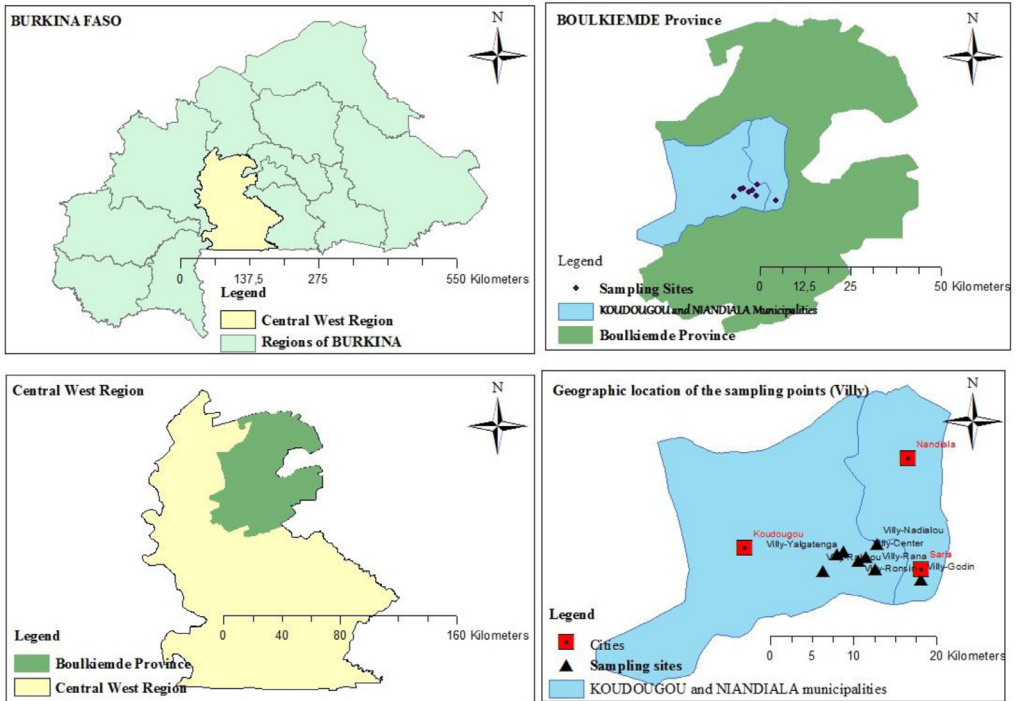


Fig. 1. Geographic locations of the sampling points.

season and a wet season of about four (4) months. The precipitation varies between 600 mm and 1000 mm [6].

The study area, Villy, is a set of eight entities: Villy-Centre, Villy-Ronsin, Villy-Godin, Villy-Nadioulou, Villy-Rana, Villy-Siguivoussé, Villy-Yalgatenga and Villy-Ralmou. Fig. 1 shows the geographic locations of the sampling points in the study area.

Samples collection and preparation

Twenty four (24) soil samples were collected in eight (8) locations within farmlands of Villy, a village located in the Bouleiemde province, in the central west region of Burkina Faso as shown in Fig. 1. At each selected farmland, three soil samples were randomly collected into labeled polyethylene bags using a coring tool of 20 cm [7,8]. At each sampling point, the vegetation and roots were removed before the usage of the coring tool for the sampling.

The soil samples were air-dried for one week under laboratory conditions to remove the moisture. Each sample was then dried in a temperature controlled furnace at 105 °C until a constant mass (about 6 h) to ensure that the moisture is completely removed [4]. Each of the dried samples was ground in a mortar and sieved through one millimeter mesh screen to obtain fine particle textured version. Each homogenate sample was divided into two replicate samples. One replicate was transferred into a Marinelli beaker, closed and tightly sealed with cellotape to avoid the escape of radon gas [9,10], and kept for roughly one month for secular equilibrium to occur before the analysis at ARSN. The other replicate sample was placed in labeled hermetically-closed polyethylene bag and transported to Ghana for analysis at RPI. Altogether, twenty four soil samples were sent to laboratories of ARSN, Burkina Faso and RPI, GAEC, Ghana respectively.

At RPI, the soil samples were placed in 50 mL geometries and stored in the storage room for roughly one month for the secular equilibrium to attain before the analysis. The weight of the sample

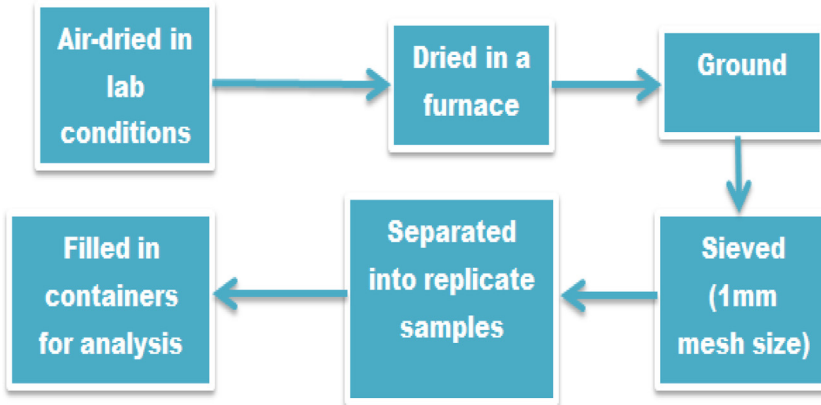


Fig. 2. Sample preparation process.

Table 1
HPGe system's characteristics.

Specifications	ARSN Lab	RPI lab
Detector model	GC4018	GX4020
• Geometry	Coaxial one open end, closed end facing window	Coaxial one open end, closed end facing window
• Diameter (mm)	60.9	60.5
• Length (mm)	63.3	61.5
• Distance from window (mm)	6	6
• Relative efficiency (%)	40	40
• Resolution (keV at 1.33 MeV of Co)	1.8	2
Cryostat model	7500SL/S	7500SL
Preamplifier model	2002CSL	2002CSL
Shielding	Lead lined with copper	Lead lined with copper, cadmium and plexiglass sheets
Software	Genie 2000	Genie 2000

is the difference between the weight of the filled container and the weight of the empty container. Fig. 2 shows the sample preparation process.

Sample analysis

Analysis of samples was conducted in two laboratories for the measurement of NORM radionuclides: ARSN (*Autorité nationale de Radioprotection et de Sûreté Nucléaire*) laboratory, Burkina Faso and RPI (Radiation Protection Institute) laboratory, GAEC, Ghana. The gamma spectrometry systems used for this study have the specifications shown in Table 1.

The gamma spectrometry system of ARSN laboratory was calibrated for energy and efficiency using a Europium point source and mixed radionuclides standard. Its measurement traceability is provided by Czech Metrology Institute (CMI). The gamma spectrometry system of RPI was calibrated for energy and efficiency using a mixed radionuclides standard from Deutschen Kalibrierdienst (DKD) and some IAEA reference materials: IAEA-RGK-1 (K-ore), IAEA-RGU-1 (U-ore) and IAEA-RGTh-1 (Th-ore). All the efficiency calibrations above mentioned used standards with known activities filled in the same geometries as the samples to be analyzed.

The activity concentration of U-238 in a soil sample was estimated as the average activity concentrations of its daughter radionuclides Bi-214 (295.21 keV; 351.92 keV) and Pb-214 (609.31 keV; 1764.49 keV) [9,10,11]. In the same manner, the activity concentration of Th-232 was obtained from the average activity concentrations of Pb-212 (238.63 keV), Tl-208 (583.19 keV; 2614.53 keV) and

Ac-228 (911.21 keV). The activity concentration of K-40 was calculated at its only energy line of 1460.83 keV.

The activity concentration of a radionuclide in a sample was calculated using the following equation:

$$A_{sp} = \frac{N_{sp}}{\varepsilon P_{\gamma} TM} \quad (1)$$

where;

N_{sp} is the net count of the gamma energy peak emitted by that radionuclide from the sample;

ε is the efficiency obtained at the gamma energy of interest;

P_{γ} is the gamma emission probability;

T is the sample counting time

M is the weight (kilogram) of the sample.

The uncertainty associated to the activity concentration of a radionuclide (ΔA) was calculated based on the following equation [1]:

$$\frac{\Delta A}{A} = \sqrt{\left(\frac{\Delta N}{N}\right)^2 + \left(\frac{\Delta P_{\gamma}}{P_{\gamma}}\right)^2 + \left(\frac{\Delta \varepsilon}{\varepsilon}\right)^2 + \left(\frac{\Delta M}{M}\right)^2} \quad (2)$$

where

ΔN is the uncertainty of the count rate;

ΔP_{γ} is the uncertainty of emission probability;

$\Delta \varepsilon$ is the uncertainty of efficiency and ΔM is the uncertainty of the weight [1].

Statistical performance indicators

Based on the approach adopted by IAEA in inter-comparison exercises and proficiency tests [3], the performance of ARSN laboratory in comparison with RPI laboratory will be assessed using the following rating parameters: relative bias, z-score, precision and trueness [2]. The replicate samples analyzed in each laboratory were assumed to have the same properties as proficiency test samples. The results obtained from the gamma spectrometry system of RPI laboratory were considered as target values because this laboratory serves as a regional designated center of radiation protection for IAEA member states in sub-Sahara and beyond. The HPGe detector at RPI has been used for scientific activities and as a laboratory for students project work and thesis for the past two decades and some of the articles and thesis carried were published in reference journals [9,10,12–15]. In view of this, the reliability of the measurement results provided by ARSN can be ascertained with the laboratory at RPI, GAEC.

- Relative bias

To estimate the bias between the measurement results from ARSN laboratory and RPI laboratory, the relative bias is expressed as follows:

$$RB = \frac{Value_{ARSN} - Value_{RPI}}{Value_{RPI}} \times 100 \quad (3)$$

- The z-score value

The z-score is calculated from the ARSN and RPI laboratories' measurement results and the standard deviation using the following equation:

$$z_{Score} = \frac{Value_{ARSN} - Value_{RPI}}{\sigma} \quad (4)$$

The standard deviation (σ) is assumed to be: $0.1 \times Value_{RPI}$

If $|z_{Score}| \leq 2$ the ARSN laboratory performance is satisfactory

If $2 < |z_{Score}| < 3$ the ARSN laboratory performance is questionable

And if $|z_{Score}| \geq 3$ the ARSN laboratory performance is unsatisfactory

Table 2

Activity concentration of U-238, Th-232 and K-40 for soil samples measured using the HPGe gamma spectrometry systems of ARSN and RPI.

Sample code	Activity concentration, Bq/kg					
	U-238		Th-232		K-40	
	Value _{ARSN}	Value _{RPI}	Value _{ARSN}	Value _{RPI}	Value _{ARSN}	Value _{RPI}
VIL02S	24.42±1.31	24.69±0.54	18.61±1.05	21.96±0.86	118.59±6.38	122.77±4.64
VIL03S	25.60±1.37	26.95±0.57	20.58±1.16	22.61±0.86	124.20±6.66	116.32±4.39
VIL05S	24.16±1.31	23.29±1.52	17.40±0.99	18.47±1.93	176.65±9.35	190.52±12.18
VIL06S	32.12±1.69	34.68±1.60	40.41±2.23	44.65±2.56	143.42±7.64	131.56±9.74
VIL07S	33.34±1.75	32.87±1.65	40.43±2.23	44.34±2.66	149.25±7.92	148.98±11.38
VIL08S	25.85±1.41	31.58±1.74	18.46±1.05	22.05±2.04	177.47±9.39	172.49±11.62
VIL09S	50.60±2.61	47.68±1.84	40.47±2.23	43.95±2.59	84.43±4.67	85.48±8.60
VIL10S	58.17±2.99	53.81±2.00	42.96±2.37	48.36±2.81	76.39±4.27	85.35±8.73
Range	24.16-58.17	23.29-53.81	17.40-42.96	18.47-48.36	76.39-177.47	85.35-190.52
Average	34.28±1.80	35.07±1.43	29.91±1.66	33.30±2.04	131.30±7.03	131.68±8.91

- The precision

To evaluate the precision of ARSN laboratory, the evaluation parameter P is calculated using this formula:

$$P = \sqrt{\left(\frac{u_{RPI}}{Value_{RPI}}\right)^2 + \left(\frac{u_{ARSN}}{Value_{ARSN}}\right)^2} \times 100\% \quad (5)$$

where u_{RPI} and u_{ARSN} are respectively the total uncertainty associated with the measurement result $value_{RPI}$ and $value_{ARSN}$.

If $P \leq LAP$ then the ARSN laboratory result is acceptable for precision. Where LAP is the Limit of Acceptable Precision and is defined in advance by the organizer.

- Trueness

To evaluate the trueness, two values $A1 = |Value_{RPI} - Value_{ARSN}|$ and $A2 = 2.58 \times \sqrt{u_{RPI}^2 + u_{ARSN}^2}$ are compared.

If $A1 \leq A2$, the ARSN laboratory's result is acceptable for trueness.

In the final evaluation, the performance indicators of precision and trueness are combined.

- If a measurement result is "acceptable" for both precision and trueness, the final score is "acceptable".
- If a measurement result is "not acceptable" for both precision and trueness, the final score is "not acceptable".
- If a measurement result is "not acceptable" for one of the two, the score for relative bias (RB) is compared with a defined value called Maximum Acceptable Bias (MAB). If $RB \leq MAB$ the measurement result will be "warning"; if not the measurement result will be "not acceptable".

The limit for acceptable precision and the maximum acceptable bias for all the radionuclides were set to 20% and 15% respectively. This choice was in line with the studies conducted by Omar EL Samad and Rana Baydoun [16].

Results and discussion

Activity concentrations of radionuclides

The activity concentrations of U-238, Th-232 and K-40 were determined for eight replicate samples using the two gamma spectrometry systems from ARSN and RPI laboratories and the results are presented in Table 2.

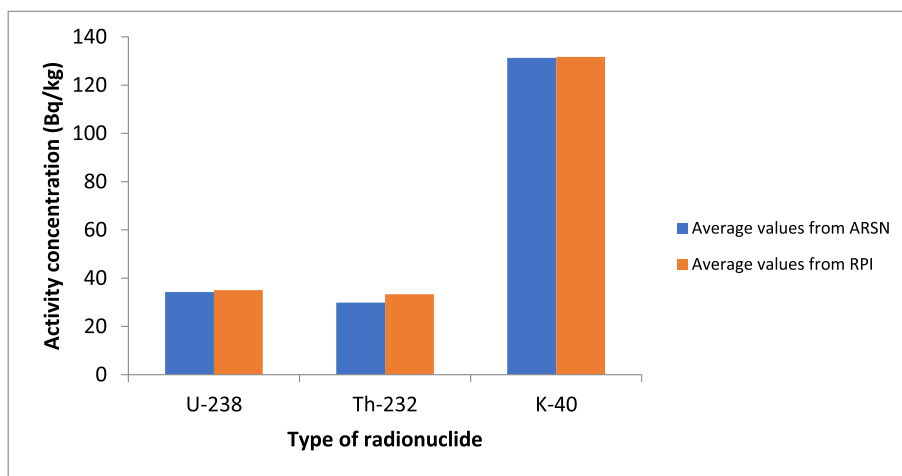


Fig. 3. Average activity concentrations for U-238, Th-232 and K-40 in soil samples from both laboratories of ARSN and RPI.

The average activity concentrations for U-238, Th-232 and K-40 in the soil samples analyzed at ARSN laboratory were found in the ranges of 24.16–58.17 Bq/kg, 17.40–42.96 Bq/kg and 76.39–177.47 Bq/kg respectively with the respective average values of 34.28 ± 1.80 Bq/kg, 29.91 ± 1.66 Bq/kg and 131.30 ± 7.03 Bq/kg. At RPI laboratory, the calculated average activity concentration for U-238, Th-232 and K-40 were 35.07 ± 1.43 Bq/kg, 33.30 ± 2.04 Bq/kg and 131.68 ± 8.91 Bq/kg and that of their respective values range from 23.29 to 53.81, 18.47 to 48.36 and 85.35 to 190.52 Bq/kg. The evaluation of the relative bias in Table 3 indicated that the activity concentrations for U-238, Th-232 and K-40 in each soil sample measured from both laboratories were almost the same with exception of RB(Th-232) for VIL02S and VIL08S and RB(U-238) for VIL08S, all the calculated values of relative bias were lower than the maximum acceptable bias. The samples which recorded the values of relative bias greater than the maximum acceptable bias, their difference was found to be negligible as shown in Table 3. Fig. 3 shows the chart of average activity concentrations for U-238, Th-232 and K-40 obtained in soil samples using both the HPGe from laboratories of ARSN and RPI. It shows no significant difference between the results obtained from both laboratories. Fig. 4 presents the chart of the average statistical uncertainties for U-238, Th-232 and K-40 obtained from the two laboratories. It indicates a none negligible difference between the average uncertainty values for each radionuclide measured with both laboratories. This may be attributed to the fact that (i) some replicates samples were used for the study instead of proficiency test samples, (ii) the sensitivity of the detectors are different. The relative uncertainties for U-238, Th-232 and K-40 were also calculated for the results obtained from both laboratories and used to plot the graphs presented in Fig. 5. The system for which the relative uncertainties for the studied radionuclide are low is considered as the most suitable for the analysis of that radionuclide [1]. It follows that the ARSN's system is suitable for the measurement of high and medium gamma energies and the RPI's system is suitable for the low gamma energies.

Comparison between the performance of the ARSN laboratory and RPI

The statistical performance indicators (z-score, relative bias, precision and trueness) were used to assess the performance of the ARSN gamma spectrometry system and the proficiency test for trueness and precision of the measurement results.

Table 3 gives the performance evaluation results obtained using the statistical performance indicators.

The results $|z_{score}| \leq 2$ were obtained for all the radionuclides. So, the performance of the ARSN laboratory gamma spectrometry system is satisfactory.

Table 3
Performance evaluation results.

Sample code	Relative Bias			Z-score values			Precision			Trueness			Evaluation of results		
	RB(U)	RB(Th)	RB(K)	z-score(U)	z-score (Th)	z-score(K)	P(U)	P(Th)	P(K)	T(U)	T(Th)	T(K)			
VIL02S	1.09	15.26	3.40	0.11	1.53	0.34	5.79	6.87	6.57	A	A	A	A	A	A
VIL03S	5.01	8.98	6.77	0.50	0.90	0.68	5.75	6.80	6.56	A	A	A	A	A	A
VIL05S	3.74	5.79	7.28	0.37	0.58	0.73	8.48	11.90	8.30	A	A	A	A	A	A
VIL06S	7.38	9.50	9.01	0.74	0.95	0.90	6.93	7.96	9.12	A	A	A	A	A	A
VIL07S	1.43	8.82	0.18	0.14	0.88	0.02	7.26	8.15	9.30	A	A	A	A	A	A
VIL08S	18.14	16.28	2.89	1.81	1.63	0.29	7.75	10.86	8.57	A	A	A	A	A	A
VIL09S	6.12	7.92	1.23	0.61	0.79	0.12	6.44	8.07	11.48	A	A	A	A	A	A
VIL10S	8.10	11.17	10.50	0.81	1.12	1.05	6.34	8.01	11.66	A	A	A	A	A	A

A: Acceptable

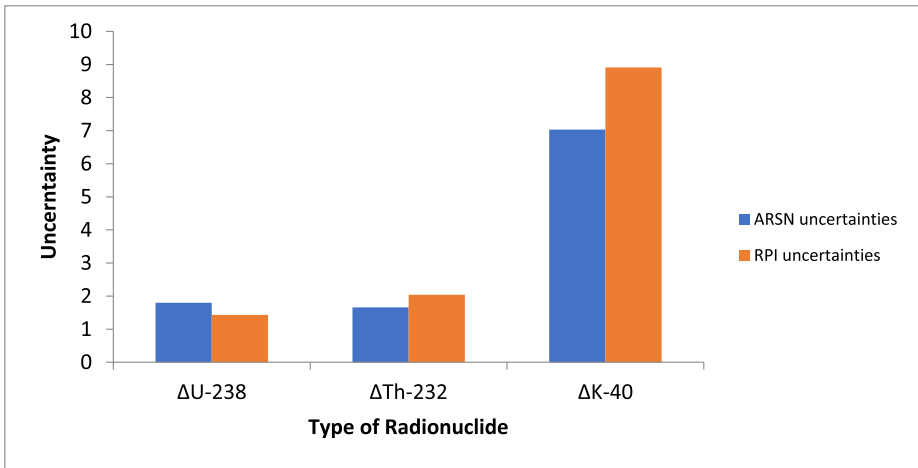


Fig. 4. Average statistical uncertainties for U-238, Th-232 and K-40 obtained from the two laboratories.

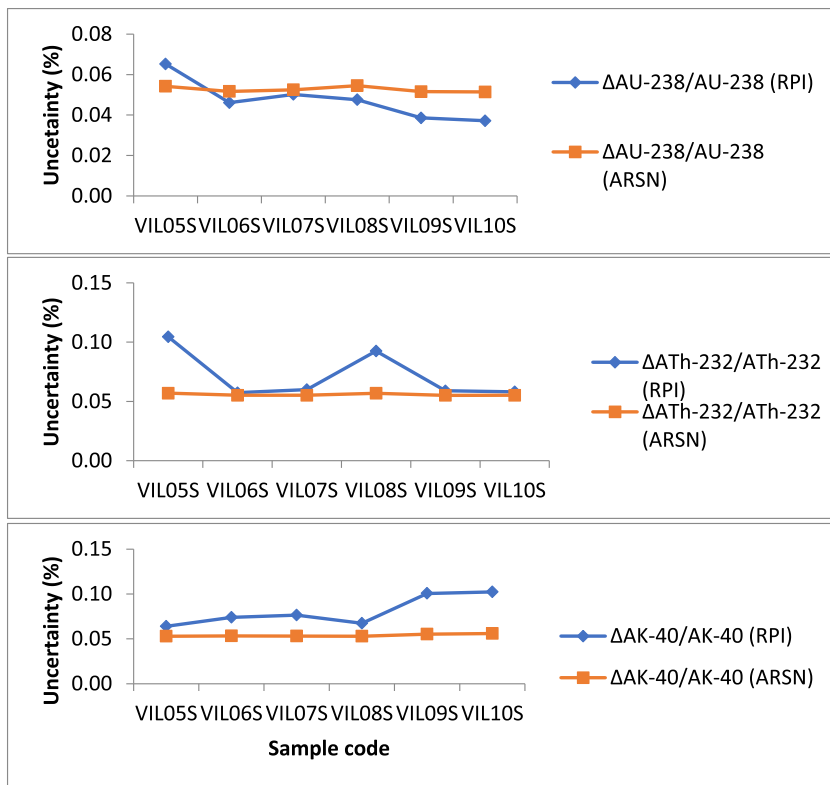
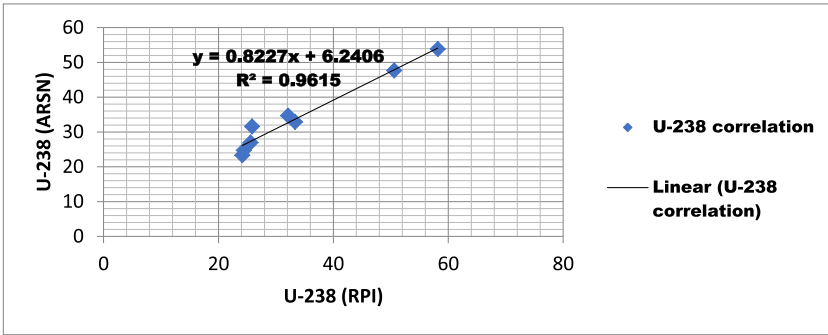
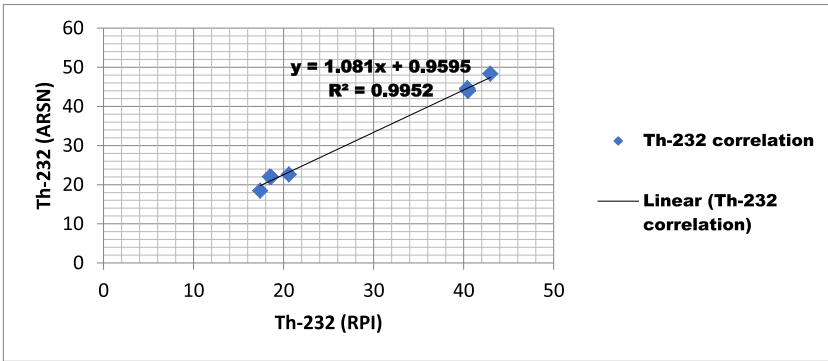


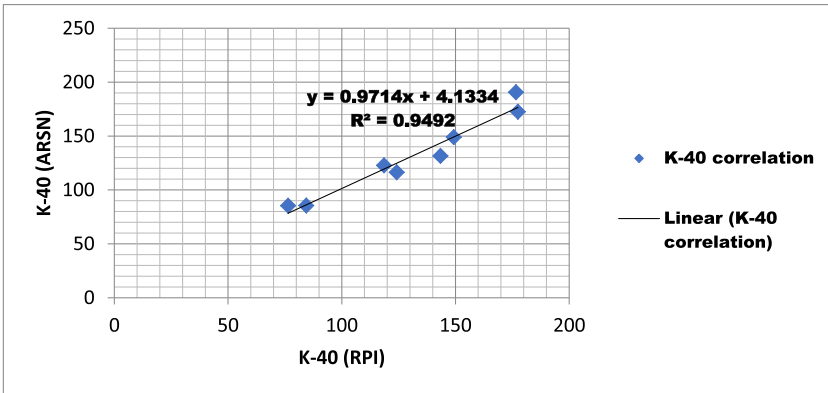
Fig. 5. Standard deviations between a couples of measurements using the detectors of two systems.



(a) Correlation between the activity concentrations of U-238



(b) Correlation between the activity concentrations of Th-232



(c) Correlation between the activity concentrations of K-40

Fig. 6. Correlation between the activity concentrations of U-238, Th-232 and K-40 using two gamma spectrometry systems of ARSN and RPI.

The results $A1 \leq A2$ and $P \leq LAP$ were obtained for all the radionuclides. So, the proficiency test for trueness and precision was acceptable for all the studied radionuclides. This enables the conclusion that the measurements results provided by the ARSN laboratory are accurate and reliable.

Fig. 6 (a-c) presents the relation between the measured activity concentrations of the respective radionuclides U-238, Th-232 and K-40 with the HPGe gamma spectrometry system of ARSN laboratory and the HPGe gamma spectrometry system of Environmental Radiation Protection laboratory of RPI. The Pearson's correlation coefficients (r) for U-238, Th-232 and K-40 were found to be 0.9806, 0.9976 and 0.9742 respectively which are nearly 1. The result indicates a strong, positive correlation between the measurements from both laboratories.

Conclusion

A comparative study of natural radioactivity in soil samples using HPGe from ARSN and RPI has been carried out. The activity concentration and uncertainty associated with the radionuclides in the soil were used to assess the performance of the HPGe system of ARSN and ascertain the reliability and accuracy of its measurement results. By using the proficiency test procedure, commonly applied by ALMERA (Analytical Laboratories for the Measurement of Environmental Radioactivity) network in inter-comparison exercises and proficiency tests, the evaluation results were acceptable for all the radionuclides. The conclusion from this is that the ARSN laboratory shows high performance and the measurements results obtained with its gamma spectrometry system are accurate and reliable.

To confirm this result, a proficiency test organized by ALMERA or a project of regional inter-comparison exercise needs to be carried out with appropriate proficiency test samples.

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Declaration of Competing Interest

Financial support for this study was provided by the International Atomic Energy Agency (IAEA) and Autorité nationale de Radioprotection et de Sûreté Nucléaire (ARSN). There are no conflicts of interest.

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