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Research Article

GROSS ALPHA/BETA ACTIVITY DETERMINATION IN DRINKING WATER PROFICIENCY TESTS

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Abstract
Water intended for drinking purposes has to be analyzed first for gross alpha/beta activity according to national and international standards and recommendations. According to Albanian legislation, Article 6, the gross alpha/beta radioactivity concentration in water, should be below the level of 0.1 Bq/L and 1 Bq/L respectively for human consumption. Our laboratory participated in an interlaboratory comparison organized by IAEA Terrestrial Environmental Laboratory under suggestion of ALMERA members. Proficiency Test among environmental radioactivity monitoring laboratories for the determination of gross alpha/beta activity concentration in drinking water and contaminated surfaces (2018, 2020). Independent standard methods were used for the reference value determination. Each sample was pretreated on site with nitric acid until reaching a pH level below 2. By creating relatively non-polar surfaces, this procedure avoids a loss of radionuclide fractions due to absorption into the walls of the containers. The Gross alpha/beta activity measurements were done using gas-flow proportional counter (GPC) method. The total dissolved solids should not exceed a surface density of 5 mg/cm2 for gross alpha determination and 10 mg/cm2 for gross beta determination The performance of participating laboratories was evaluated with respect to the reference values using relative deviations. The results presented from our laboratory in both inter comparisons (IAEA-TEL-2018-03) and (IAEA-TEL-2020-03) were all acceptable. The laboratory for the measurement of gross alpha/beta radioactivity and our Institute as a part of Tirana University is in the process of accreditation, so far the participation in the inter comparisons exercise is very important.

Keywords: Drinking Water, Environmental Radioactivity, Interlaboratory Comparison

INTRODUCTION

The main processes contributing to the internal exposure of the human body to ionizing radiation are represented by air inhalation and by water and food ingestion. UNSCEAR (United Nations Scientific Committee on the Effects of Atomic Radiation) estimates the contribution of natural sources to the population's effective dose at 2.4 mSv/year, this dose comprising the amount of 0.29 mSv/year due to water and food consumption (UNSCEAR 2016 Report; Violeta et. al., 2020). The radioactivity of drinking water is an environmental factor which contributes to the population exposure to ionizing radiations and the activity of monitoring the water radioactive content is the responsibility of the national public health systems by ensuring the maintaining of the effective dose by ingestion in the provided limits (TECDOC-1788, 2016; TECDOC1401, 2004; Violeta et. al., 2020; Caridi, et. al., 2021; Kupe, et. al. IJITIS, 2023).

Water intended for drinking purposes has to be analyzed first for gross alpha/beta activity according to national and international standards and recommendations (Council Directive 2013/51; Decision No. 957, 2015; M. Gomez, et. al., CREST, 2023; WHO, 2018; WHO, 2022). According to Albanian legislation, Article 6, the gross alpha/beta radioactivity concentration in water, should be below the level of 0.1 Bq/L and 1 Bq/L respectively for human consumption (Decision No. 957, 2015). Gross alpha/beta activity measurements are widely applied as a screening technique on environmental monitoring not only for drinking water but also in other fields (e.g., industrial applications, etc.) (Sun & Semkow, 1998; Semkow & Parekh, 2001; Desideri et. Al., 2007; Jia, Torri, & Magro, 2009; Altıkulac, Turhan, & Gumus, 2015; Cfarku, et. al., 2020; Ho et. al., 2020; Kusuma, 2020; Suwarni, 2021; Zurhaimi & Mufit, 2022; Herawati, Khairinal, & Idrus, 2023).

Since gross alpha/beta measurements are very important for the country, our laboratory's main goal is to provide reliable and accurate results to assure the public that the dose received from water consumption is below the level determined by the national law (Decision No. 957, 2015). For this reason, in order to assess our standing on a European level compared to other laboratories, participation in intercomparisons with them is essential to evaluate ourselves and our measurements. The new EU drinking water directive (Council Directive 2013/51; Jobba´gy, Watjen, & Meresova, 2010), which includes gross alpha/beta activity screening levels, and IAEA Terrestrial Environmental Laboratory organize periodically interlaboratory comparisons on the determination of anthropogenic and natural radionuclides in water and soil samples with the suggestion of the ALMERA members to check the fitness for purpose of this method and the performance of monitoring laboratories in the world-wide countries (IAEA Almera Proficiency Tests; Osvath et. Al., 2015; Yohanie et al., 2023; Fitriana & Waswa, 2024; Zakiyah, Boonma, & Collado, 2024).

The Environmental Laboratory of the Department of Radiometry and Radiochemistry (Institute of Applied Nuclear Physics) took part in the 2018 and 2020 interlaboratory comparison, the IAEA-TEL-2018-03, the IAEA-TEL-2020-03 proficiency test exercise.

In the interlaboratory comparison 2018 participated a total of 267 laboratories from 66 different countries, which successfully reported data in the frame of the IAEA-TEL-2018-03 proficiency test exercise. For each of the analyses in scope the target value, the target uncertainty and the MARB are evaluated (IAEA-TEL-2018-03 WWOPT). In this World-Wide Proficiency Test a set of prepared samples were distributed to all the participated laboratories: the sample 01 drinking water spiked with anthropogenic gamma emitting isotopes and ^{89}Sr, ^{90}Sr, (in equilibrium with ^{90}Y), ^{210}Pb (^{210}Po), sample volume approx. 500g, sample 02, drinking water spiked with anthropogenic gamma and beta emitters, sample volume approx. 500g and sample 03, drinking water as QC sample, with known radionuclide content (isotope and activity with associated uncertainty), sample volume approx. 500 g; contaminated surface samples: sample 05, sample 06 and sample 07 contaminated surfaces for surface contamination measurements..

In the Proficiency Test Interlaboratory comparison 2020 participated a total of 215 laboratories from 58 different countries, which successfully reported data in the frame of the IAEA-TEL-2020-03 World Wide Open Proficiency Test Exercise. In this World-Wide Proficiency Test a set of prepared samples were distributed to all the participated laboratories: the samples 01 Water spiked with anthropogenic and natural radionuclides, sample volume approx. 500 g; sample 02, Water spiked with anthropogenic gamma and beta emitters, sample volume approx. 500 g and sample 03, Water as QC sample, with known radionuclide content (isotope and activity with associated uncertainty), sample volume approx. 500 g, sample 05, sample 06 and sample 07 printed disks samples for gross alpha and beta measurement, ^{90}Sr (^{90}Y), ^{244}Cm and mixed respectively.

The data is evaluated by the Terrestrial Environment Laboratory (TEL) of the NA Environment Laboratories using its standard approach for proficiency test evaluations. The main objectives of this Proficiency Test were determination of anthropogenic and natural radionuclide in water and evaluation of gross alpha/beta surface contamination by all the participated laboratories. In this study we will evaluate gross alpha and gross beta radioactivity in the unknown samples as well as the uncertainties that come from the measurements.

RESEARCH METHOD

IAEA-TEL-2018-03 World Wide Proficiency Test Exercise. Matrix origin of the samples distributed from the IAEA used for the sample preparation was tap water from Seibersdorf, Austria. The water was gravimetrically spiked with known amounts of standard solution containing a mixture of certified radionuclides and acidified with 0.05M HNO_3 for stability. Sample 01 was spiked with anthropogenic gamma emitter radionuclides, ^{210}Pb (^{210}Po) and two beta emitters: ^{89}Sr and ^{90}Sr. Sample 02 was spiked with a primary coolant of the nuclear power reactor.

All these surfaces have been prepared by printing technique. The uniformity of the printed surfaces has been tested by measurement of emitted particles in counts/cm^2/s on every tenth surface from the production order. Sample 05 beta contaminated $^{90}Sr/^{90}Y$. Sample 06 alpha contaminated with ^{214}Am. Sample 07 beta contaminated with $^{90}Sr/^{90}Y$ + ^{214}Am.

IAEA-TEL-2020-03 World Wide Open Proficiency Test Exercise. Matrix origin of the samples used for the sample preparation was tap water from Seibersdorf, Austria. The water was gravimetrically spiked with known amounts of standard solution containing a mixture of certified radionuclides and acidified with 0.05M HNO_3 for stability. The Sample 01, were spiked with anthropogenic and natural gamma emitter radionuclides and ^{90}Sr. Sample volume approx. 500 g. Sample 02, were spiked with broken chains of the ^{232}Th - ^{228}Ra alpha emitters progenies and ^{228}Th - ^{224}Ra. Sample 03, were spiked water as QC sample, with known radionuclide content (isotope and activity with associated uncertainty), sample volume approx. 500 g. All these surfaces were prepared by printing technique. The uniformity of the printed surfaces has been tested by measurement of emitted particles in counts/cm^2/s on every tenth surface from the production order. They are contaminated with gamma emitters and beta.

After the arrival of the samples prepared by the Seibersdorf laboratory, the treatment began in our laboratory for their preparation for gross alpha/beta analysis. Each sample was pretreated on site with nitric acid until reaching a pH level below 2. By creating relatively non-polar surfaces, this procedure avoids a loss of radionuclide fractions due to absorption into the walls of the containers. Furthermore, the procedure prevents the formation of precipitates and any biological activity resulting in loss of analytes. The Gross alpha/beta activity measurements were done using gas-flow proportional counter (GPC) method. The total dissolved solids should not exceed a surface density of 5 mg/cm^2 for gross alpha determination and 10 mg/cm^2 for gross beta determination (A. Mauring et. al., IAEA-TEL-2018-03 ALMERA Proficiency Test; A. Mauring et. al., IAEA-TEL-2020-03 ALMERA Proficiency Test). Accordingly, an aliquot of 100 mL of each sample was evaporated to dryness at, and the final concentration of the total dissolved solids (mg/L) was determined. Afterwards, based on the concentration of the total dissolved solids, an appropriate test aliquot of each sample was chosen and was slowly evaporated in a sand bath to avoid spattering. The total dissolved solids were then transferred to a 2-in.-diameter stainless steel planchet (measurement geometry), using 8 M NHO3 to carefully wash the beaker. The planchets were then oven-dried at 80^{0}C for 24 hours and then weighted and stored in desiccators before analysis. The samples were measured after 30 min to prevent water uptake from the possible presence of nitrates (EPA, Doc. No: PB 80-224744).

The surface contaminated samples and simulated aerosol filter samples were measured directly with the SAB-100 probe for measurement of surface contamination designed to be used with any CSP survey meter (Radiagem 2000) (F. Cfarku et. al, AIP Conf. Proceed. 2075, 2019; EPA 402-R-12-005; IAEA-TECDOC-1092).

The samples were measured for 36000 s using an ultra-low background gas-flow proportional counter model MPC-9604 (Protean Instrument Corporation) equipped with an ultra-thin window gas flow proportional detector using P-10 (Argon/Metan) gas mixture. Also, the measuring system has passive (lead) and active (guard detector) shielding. The software Vista 2000 serves as a rider of the data measuring. Calibration of the proportional counter for measuring gross alpha and beta activities involves several steps, such as selecting the appropriate values of the bias, plateau, and discriminates. The operating high voltage for simultaneous alpha/beta measurement with the MPC-9604 detector was set at 1455 V.

The MDS also has improved inter-detector shielding about 5.1 cm of shielding between adjacent detectors, virtually eliminating counts from hotter-than-expected samples in nearby detectors.

Self-absorption of the alpha and beta particles in the dry solids of the evaporated salt was corrected based on measurements using a standardized ^{239}Pu and ^{90}Sr solution. Efficiency calibration was made using ^{239}Pu and ^{90}Sr (^{90}Y) standards of known activity (F. Cfarku, et. al., JRNC, 2014). Detection limits were calculated as three times the standard deviation of blank values and give values of 0.01 Bq/l for gross alpha and 0.03 Bq/l for gross beta radioactivity. The uncertainty given is the expanded uncertainty (k=2). In the uncertainty budget is taken into account the relative uncertainty of: tracer activity, counting, efficiency, blank and background measurements (IAEA, TECDOC1401, 2004).

The measurement of contaminated samples and simulated aerosol filter samples was performed in the fixed distance 5mm foam material with AB-100 probe. After 20 readings for each sample, the Alpha and Beta efficiency was calculated.

RESULTS AND DISCUSSION

The data is evaluated according to the following steps: The relative bias between the reported and the target value (the best estimation of the true value) is exressed by the following equation:

$$\mathrm{Bias}_{\mathrm{relative}} = \frac{\mathrm{Value}_{\mathrm{reported}} - \mathrm{Value}_{\mathrm{target}}}{\mathrm{Value}_{\mathrm{target}}} \times 100\% \qquad (1)$$

The relative bias is compared to the Maximum Acceptable Relative Bias (MARB) which has been determined for each measured considering the physical background of radio-analytical methods including the level of radioactivity and the complexity of the task. If the $\mathrm{Bias}_{\mathrm{relative}} \leq \mathrm{MARB\ Value}$ the result will be "Accepted" or "A" for accuracy. Based on fit for purpose and the good laboratory practice principles, the expanded relative combined uncertainty should cover the relative bias:

$$P = \sqrt{\left(\frac{u_{\mathrm{target}}}{A_{\mathrm{target}}}\right)^2 + \left(\frac{u_{\mathrm{reported}}}{A_{\mathrm{reported}}}\right)^2} \times 100 \qquad (2)$$

Where k is the coverage factor, for the 99% confidence level, k = 2.58. If the result is between the ± M ARB values, but it is not overlapping with the target value within their uncertainties, this equation helps to decide whether they are significantly different or not. The P value is compared to the MARB also (ISO/IEC 17043:2010; ISO 13528:2015) if both the:

$$P \leq \mathrm{MARB} \qquad (3)$$

And

$$\mathrm{Bias}_{\mathrm{relative}} \leq k * P \qquad (4)$$

Fulfilled, the reported results will be "Accepted" for the precision. If one of them is insufficient, the result will be assigned the "Not accepted" or "N" status for precision. The final score according to the above detailed evaluation:

• “Accepted” when both, accuracy and precision achieved “Accepted” status
• “Not Accepted” when the accuracy is “Not accepted”
• “Warning” or "W" when accuracy is “Accepted” but the precision is “Not accepted”

Also, for each reported value were calculated the Z-Score:

$$z = \left|\frac{\mathrm{Value}_{\mathrm{reported}} - \mathrm{Value}_{\mathrm{target}}}{\mathrm{robustsd}}\right| \qquad (5)$$

In tables 1 and 2 were presented our results for the World-Wide Proficiency Test IAEA-TEL-2018 and IAEA-TEL-2020-03 respectively. In the uncertainty budged is taken into account the relative uncertainty of: tracer activity, counting, efficiency, blank and background measurements.

Table 1. Reported and evaluated results for IAEA-TEL-2018-03

Sample No.	Robust Mean	Robust SD	Reported Value	Reported Unc.	Z-Score	Z-Score Evaluation
Sample 01 gross alpha	104.5	24.1	90.23	9.47	0.59	A
Sample 01 gross beta	242	76	212	19.56	0.39	A
Sample 05 gross beta	0.62	0.3	0.47	0.03	0.5	A
Sample 06 gross alpha	0.3	0.2	0.52	0.07	1.1	A
Sample 07gross alpha	0.2	0.1	0.36	0.04	1.6	A
Sample 07 gross beta	2	0.9	0.88	0.08	1.24	A

Table 2. Reported and evaluated results for IAEA-TEL-2020-03

Sample No.	Robust Mean	Robust SD	Reported Value	Reported Unc.	Z-Score	Z-Score Evaluation
Sample 01 gross beta	170	27	140.2	12.35	1.32	A
Sample 02 gross alpha	40	14	31.52	3.13	0.6	A
Sample 02 gross beta	38	7	36.85	3.23	0.16	A
Sample 05 gross beta	12.4	3.5	11.77	1.09	0.18	A
Sample 06 gross beta	8.6	2.5	8.39	0.32	0.08	A
Sample 07 gross beta	7.6	2.5	7.74	0.61	0.06	A

As can be seen from the tables 1 and 2, the results of our laboratory for all measured values gross alpha activity and gross beta activity are all Acceptable (A). In the table 1 the results for gross alpha/beta measurements in the sample 02 was not requested. The results for gross alpha activity concentration in the 2018 and 2020 intercomparisons in drinking water samples have the Z-score 0.59 and 0.6, respectively. This demonstrates the stability of the entire alpha measurement method of the laboratory and the ongoing accuracy of the alpha measurements. The results for gross alpha activity concentration in the 2018 intercomparison for surface contaminated samples 06 and 07 have Z-score 1.1 and 1.6, respectively.

The results for gross beta activity concentration in the 2018 and 2020 intercomparision in drinking water samples with the same composition have the Z-score 0.39 and 0.16, respectively. The improvement in the results for gross beta measurement in 2020 can be explained by the use of the sample set from previous intercomparisons as an additional factor in the construction of the calibration curve for the measurement system. The results for gross beta activity concentrations in the 2018 intercomparision for surface contaminated samples 05 and 07 have Z-score 0.5 and 1.24, respectively. The results for gross beta activity concentrations in the 2020 intercomparision for surface contaminated samples 05, 06 and 07 have Z-score 0.18, 0.08 and 0.06, respectively. We see an improvement in the results for gross beta activity concentration in 2020 for contaminated surfaces, which can be explained by different factors such as new calibration factor, different geometry, etc.

In the intercomparison IAEA-TEL-2018-03 from 267 participating laboratories from 66 different countries, the results were reported by 86 laboratories. The results for gross alpha and beta activity concentration in drinking water sample 01 from reported values were 73 % Acceptable and Warning and 27 % Not acceptable for gross alpha activity concentration, while for gross beta activity concentration the reported results were 66 % Acceptable and Warning and 34% Not acceptable. Regarding the results of gross beta activity concentration in the surface contaminated samples 05, 06 and 07 from the reported values were 20 %, 15 % and 29 % Not acceptable, respectively.

In the intercomparison IAEA-TEL-2020-03 from 215 participating laboratories from 58 different countries, the results were reported by 109 laboratories. The results for gross beta activity concentration in drinking water sample 01 from the reported values were 18 % Not acceptable. The results for gross alpha and beta activity concentration in drinking water sample 02, from the reported values were 44% and 30 % Not acceptable, respectively. Regarding the results of gross beta activity concentration in the surface contaminated samples 05, 06 and 07 from the reported values were 17 %, 25 % and 17 % Not acceptable, respectively.

All the values reported by our laboratory were acceptable in all samples. In both intercomparisons exercises it is observed that the values of gross alpha activity concentration have a better Z-score than those of gross beta activity concentration. This fact suggests more attention in the future gross beta activity determination.

CONCLUSION

Measured values and associated standards uncertainties (U) of the proficiency test samples for gross alpha and gross beta activity concentration in the interlaboratory comparisons in both cases 2018 year and 2020 year were all acceptable. The biggest deviation observed in the results of the gross beta activity concentration for drinking water samples is 18 % between the measured value and reference value, observed in the 2020 intercomparision.

The biggest deviation observed in the results of the gross beta activity concentration for surface contaminated samples is 56 % between the measured value and reference value of the gross beta activity concentration, observed in the 2018 intercomparision. The pure Z-score for surface contamination for gross alpha activity concentrations was observed in the 2018 intercomparision. The best performance of our results was in the determination of the gross alpha activity concentration for drinking water samples. The reported results by our laboratory for determining gross alpha and beta activity concentration in drinking water and surface contaminated samples were reliable.

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AUTHOR CONTRIBUTIONS

F. Cfarku and I. Bërdufi have written the main manuscript text. F. Cfarku has made the calculations and I. Bërdufi has prepared tables and enters the formulas. Both authors reviewed the manuscript and discussed the results and conclusions.

CONFLICTS OF INTEREST

The author(s) declare no conflict of interest.

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