The IAEA-TERC-2023-01 world wide proficiency test on the determination of anthropogenic and natural radionuclides in water, soil and contaminated surface samples

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IAEA-TERC-2023-01 World Wide Proficiency Test Exercise
Individual Evaluation Report for Labcode 37

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Abstract

The IAEA Terrestrial Environmental Radiochemistry Laboratory (TERC) provided a Proficiency Test (PT) on determination of anthropogenic and natural radionuclides in water, soil and simulated contaminated surface samples. This year's PT was designed to monitor and demonstrate the performance and analytical capability of the participating laboratories and to identify gaps and problem areas where further development is needed. The sample set was formulated to support these goals.

1 Description of Samples

- Sample 1: Spiked Water, anthropogenic and natural radionuclides, gross alpha and beta (GAB)
- Sample 2: Spiked Water, anthropogenic and natural radionuclides and GAB (environmental levels)
- Sample 3: Spiked Water, quality control sample, no data reporting and evaluation
- Sample 4: Japanese Soil, anthropogenic and natural radionuclides
- Sample 5: Simulated contaminated surface sample, gamma and beta emitting radionuclides
- Sample 6: Simulated contaminated surface sample, gamma emitting radionuclide (Cs-134)
- Sample 7: Simulated contaminated surface sample, beta emitting radionuclide (Sr-90)

1.1 Sample 1, 2, Spiked Water

Matrix origin:

Drinking water sourced from Seibersdorf, Austria.

Sample preparation:

The raw water was gravimetrically spiked with known amounts of a prepared standard solution, containing a mixture of certified radionuclides and acidified to < pH 2 (0.05M HNO3) for stability. The stability of the samples has been tested and guaranteed during the PT reporting period only. Sample 01 and Sample 02 were spiked with several radionuclides. This included H-3 and U isotopes for Sample 01 and Sr-90, Ra-226 and progeny for Sample 02. The identification of gamma emitting radionuclides is one of the tasks of this proficiency test, therefore they are not specified in advance.

The activity concentration and uncertainty were assigned by formulation, and control measurements by gamma-ray spectrometry, alpha spectrometry and liquid scintillation counting have been performed prior to shipment.

1.2 Sample 4, Japanese Soil

The soil was collected on 25 September 2020 in Japan by the University of Tsukuba. It was milled and sieved, and the grain size fraction used for this exercise was 90-250 µm. The bottled samples were sterilised (25 kGy gamma dose) and the related certificate is available on the PT website. It contains caesium and naturally occurring radionuclides typically found in soils.

Approximately 100 g has been provided for gamma-ray spectrometry measurement.

The determination of target values and associated uncertainties of the anthropogenic and natural radionuclides has been carried-out by measurements in one laboratory and confirmed by measurements in another laboratory. The samples were characterized for gamma emitting radionuclides only.

1.3 Samples 5,6 and 7, Simulated contaminated surface samples

Three circles for surface contamination monitoring have been included in this proficiency test (Figure 1). They were prepared using our in-house printing technique. One blank sample of the same size, outlined with black ink (not-spiked) was also added for correction (Sample 08).

The blue ink was spiked with a gamma-emitting radionuclide, Cs-134. The exercise asked for it's identification and activity to be reported (Sample 06).

The magenta ink was spiked with Sr-90 and the activity should be reported (Sample 07), with correction for Y-90 activity.

The mixed ink sample (blue and magenta) has been printed at a lower activity compared to the single ink circles for both radionuclides (Sample 05). Both Cs-134 and Sr-90 activity should be reported for this sample.

The reference date 2023-01-01 should be used for decay correction.

2 Evaluation of results

Results reported by participants were evaluated using the following stepwise approach.

The evaluation follows the methodology applied for the annual IAEA proficiency testing schemes in the areas of radionuclide measurements and trace element analysis.

2.1 Relative bias (Samples 1, 2, 4, 5, 6 and 7)

The relative bias is the relative difference between the reported and the target value (the best estimate of the true value)

$$Bias_{relative} = \frac{Value_{reported} - Value_{target}}{Value_{target}} * 100\%$$

The relative bias is compared to the Maximum Acceptable Relative Bias (MARB) which has been determined for each property value, considering the analytical methods, the analyte level in the sample and the complexity of the analysis.

When $|Bias_{relative}| \leq MARB$, the result will be rated "Accepted (A)" for trueness.

2.2 Evaluation approach for radionuclide activity concentration values (Samples 1, 2 and 4) and massic activity values (Samples 5, 6 and 7)

Based on fit-for-purpose and good laboratory practice principles, the expanded relative uncertainty should cover the relative bias (as defined in 2.1):

The P value corresponds to the relative combined uncertainty of the relative bias

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

The relative bias is then compared to the expanded uncertainty of the relative bias:

$$|Bias_{relative}| \leq k * P$$

where k is the coverage factor: $k = 2.58$ for a level of confidence of 99%.

When the above criterion is fulfilled, the reported result is not significantly different from the target values considering the uncertainties associated with both values. The reported uncertainty of measurement is large enough to cover the bias of the method.

In addition, the P value is compared to the MARB.

$$P \leq MARB$$

When this criterion is fulfilled, the measurement uncertainty is not overestimated and fit-for-purpose in relation to the MARB criterion of this PT exercise. When both criteria related to the measurement uncertainty are fulfilled, the reported result is rated "accepted (A)" for precision (measurement uncertainty). The result is rated "Not Accepted (N)" for precision if either of the two conditions are not fulfilled.

The final score is assigned according to the detailed evaluation described above. The possible scores are listed below:

- "Accepted (A)" when both, trueness and precision were rated "Accepted"
- "Not Accepted (N)" when the trueness rating is "Not Accepted"
- "Warning (W)" when the trueness rating is “Accepted” but the precision rating is "Not Accepted"

2.3 Evaluation parameters calculated for intercomparison analyte values

2.3.1 Z-Score

The z score provides information on the trueness of the reported value in relation to the standard deviation for proficiency assessment σ_{PT}, which is assigned based on fitness for purpose.

$$z = \frac{Value_{reported} - Value_{target}}{\sigma_{PT}}$$

The following criteria apply for performance ratings based on obtained z scores

- $|z| \leq 2$ ... accepted (A)
- $2 < |z| < 3$ ... questionable (Q)
- $|z| \geq 3$ ... not accepted (N)

2.3.2 Zeta-Score

The ζ (zeta) score allows a combined assessment of the reported value and the reported uncertainty of measurement, and thus of the accuracy of the reported result.

$$\zeta = \frac{Value_{reported} - Value_{target}}{\sqrt{(u_{reported})^2 + (u_{target})^2}}$$

The following criteria apply for performance ratings based on obtained zeta scores

- $|\zeta| \leq 2$ ... accepted (A)
- $2 < |\zeta| < 3$ ... questionable (Q)
- $|\zeta| \geq 3$ ... not accepted (N)

3 Data Evaluation Tables

Target Values for activity concentration of radionuclides in Sample 1

TABLE 1. Target values

Sample	Analyte	Technique	Target Value	Uncertainty ($k = 1$)	Unit	MARB [%]
1	H-3	beta	29.0	1.5	Bq/kg	30.00
1	Co-60	gamma	14.7	0.7	Bq/kg	20.00
1	U-234	alpha	5.44	0.27	Bq/kg	30.00
1	U-235	alpha	0.26	0.01	Bq/kg	30.00
1	U-238	gamma	5.60	0.28	Bq/kg	30.00
1	U-238	alpha	5.60	0.28	Bq/kg	30.00
1	Am-241	gamma	22.3	1.1	Bq/kg	20.00

Evaluation Table for Sample 1

TABLE 2. Evaluation Results for Sample 1

Analyte	Technique	Reported value [Bq/kg]	Reported uncertainty ($k = 1$) [Bq/kg]	Relative bias [%]	P-Test [%]	Trueness evaluation	Precision evaluation	Final Score
Co-60	gamma	14.89	0.97	1.3	8.07	A	A	A
Am-241	gamma	22.7	1.3	1.8	7.56	A	A	A

Target Values for activity concentration of radionuclides in Sample 2

TABLE 3. Target values

Sample	Analyte	Technique	Target Value	Uncertainty ($k = 1$)	Unit	MARB [%]
2	Sr-90	beta	14.2	0.7	Bq/kg	30.00
2	Sb-125	gamma	73.0	3.6	Bq/kg	20.00
2	Cs-134	gamma	40.0	2.0	Bq/kg	20.00
2	Cs-137	gamma	44.1	2.2	Bq/kg	20.00
2	Pb-210	gamma	5.23	0.26	Bq/kg	30.00
2	Pb-210	beta	5.23	0.26	Bq/kg	30.00
2	Po-210	alpha	5.21	0.26	Bq/kg	30.00
2	Ra-226	alpha	6.32	0.32	Bq/kg	30.00
2	Ra-226	gamma	6.32	0.32	Bq/kg	30.00

Evaluation Table for Sample 2

TABLE 4. Evaluation Results for Sample 2

Analyte	Technique	Reported value [Bq/kg]	Reported uncertainty ($k = 1$) [Bq/kg]	Relative bias [%]	P-Test [%]	Trueness evaluation	Precision evaluation	Final Score
Sb-125	gamma	66.1	3.1	-9.5	6.81	A	A	A
Cs-134	gamma	38.5	2.1	-3.8	7.40	A	A	A
Cs-137	gamma	44.6	3.3	1.1	8.92	A	A	A
Ra-226	gamma	6.82	0.88	7.9	13.86	A	A	A
Ra-226	alpha	6.65	0.62	5.2	10.61	A	A	A

Target values for massic activity of radionuclides in Samples 5, 6 and 7 (surface samples)

TABLE 5. Target values

Sample	Analyte	Technique	Target Value	Uncertainty ($k = 1$)	Unit	MARB [%]
5	Cs-134	gamma	5.40	0.21	Bq/sample	30
5	Sr-90	beta	2.25	0.10	Bq/sample	30
6	Cs-134	gamma	7.84	0.31	Bq/sample	30
7	Sr-90	beta	4.52	0.22	Bq/sample	30

Evaluation Table for Samples 5, 6 and 7 (surface samples)

TABLE 6. Evaluation Results for surface samples

Sample	Analyte	Technique	Reported value [Bq/sample]	Reported uncertainty ($k = 1$) [Bq/sample]	Relative bias [%]	P-Test [%]	Trueness evaluation	Precision evaluation	Final Score
5	Cs-134	gamma	5.59	0.64	3.5	12.09	A	A	A
5	Sr-90	beta	2.60	0.37	15.6	14.91	A	A	A
6	Cs-134	gamma	8.12	0.92	3.6	12.00	A	A	A
7	Sr-90	beta	5.10	0.7	12.8	14.56	A	A	A

Intercomparison parameters for activity concentration of radionuclides in Sample 1

TABLE 7. Robust mean and standard deviation

Sample	Analyte	Robust mean	Robust standard deviation	Unit	MARB [%]
1	gross alpha	27.8	6.4	Bq/kg	40
1	gross beta	17.3	5.8	Bq/kg	40

Evaluation Table for Sample 1

TABLE 8. Evaluation Results for Sample 1

Analyte	Reported value [Bq/kg]	Reported uncertainty ($k = 1$) [Bq/kg]	Relative bias [%]	Trueness evaluation	z score	z score evaluation	zeta score	zeta score evaluation
gross alpha	28.6	2.6	2.9	A	0.13	A	0.12	A
gross beta	18.3	3.4	5.8	A	0.17	A	0.15	A

Intercomparison parameters for activity concentration of radionuclides in Sample 2

TABLE 9. Robust mean and standard deviation

Sample	Analyte	Robust mean	Robust standard deviation	Unit	MARB [%]
2	gross alpha	13.2	6.1	Bq/kg	40
2	gross beta	135	42	Bq/kg	40

Evaluation Table for Sample 2

TABLE 10. Evaluation Results for Sample 2

Analyte	Reported value [Bq/kg]	Reported uncertainty ($k = 1$) [Bq/kg]	Relative bias [%]	Trueness evaluation	z score	z score evaluation	zeta score	zeta score evaluation
gross alpha	17.8	2.9	34.8	A	0.75	A	0.68	A
gross beta	154	21	14.1	A	0.45	A	0.40	A

4 Contributors to evaluation and report

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References

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