STATISTICAL PLAN 2027

The French version is the reference version

1. OBJECTIVES OF THE PROFICIENCY TESTING PROGRAM

The main objectives of the proficiency testing program are described in the campaign plan.
The objectives described in the statistical plan are the statistical objectives. They allow for the evaluation of the following performance:

- Performance evaluated by comparison to the criteria described in ISO 28218, NF ISO 12790, and ANSI N13-30 2011. This performance evaluation is particularly relevant to radiotoxicology laboratories.
- Performance evaluated by comparison to other participants.
- Performance evaluated by comparison to the measurement uncertainty of the assigned sample value.

2. FREQUENCY OF PROFITABILITY TESTING

The program is conducted during a single annual campaign of various interlaboratory comparisons.

3. STATISTICAL PLAN PARAMETERS

Data type: Quantitative and qualitative data are possible.

Minimum number of participants: 3

Target number of participants: Depending on the matrix and the radionuclides being measured, the target number of participants varies from 6 to 60.

Number of entities to be measured: Depending on the matrix and the radionuclide, the number of entities to be measured varies from 1 to 5. No repeat measurements are required: each participating laboratory provides only one result per radionuclide for a given sample.

4. METHOD USED BY PARTICIPANTS

No method is mandated. Laboratories must use their routine methods, as indicated in the campaign plan.

Laboratories may use a method under development.

5. STATISTICAL ASSUMPTIONS USED

The statistical assumptions are based on:

- The data set follows a normal distribution.

▪ Elimination of the influence of outliers using a Grubbs test when the number of data points is ≤ 12, or when the data point has a z-score > 5 when the number of data points is > 12. Eliminating outliers allows us to verify the first assumption.

6. STATISTICAL METHODS PLANNED

Descriptive statistics: number, minimum value, maximum value, median, scaled median absolute deviation, arithmetic mean, and arithmetic standard deviation. The scaled median absolute deviation, arithmetic mean, and arithmetic standard deviation will be used for data sets ≤ 12.

Complex statistical models: outlier detection using the Grubbs test for data sets ≤ 12, and algorithm A from the NF ISO 13528 standard for robust mean and robust standard deviation.

Graphs:
▪ Bias diagrams (ascending order of results with uncertainties, ascending order of relative biases)
▪ Bar charts of z'scores in ascending order
▪ Bar charts of zeta-scores in ascending order
▪ Diagrams combining the z'score (x-axis) and the zeta-score (y-axis) : in development

7. DETERMINATION OF ASSIGNED VALUES

PROCORAD determines assigned values using three methods:
▪ The assigned value is calculated from the masses of the properties used in the case of samples prepared by formulation.
▪ The assigned value is the robust mean calculated according to algorithm A of standard NF ISO 13528.
▪ The assigned value is the arithmetic mean.

The different situations for determining the assigned value will be described below: natural or artificial radionuclides, number of data points ≤ 12, assigned value obtained by formulation not validated according to section 7.8.1 of standard NF ISO 13528.

Metrological traceability is ensured for artificial radionuclides. There is no metrological traceability for natural radionuclides and for certain artificial radionuclides in the following interlaboratory comparisons:
▪ Actinides in fecal ash (Th-228, Th-230, Th-232, U-234, U-235, and U-238),
▪ Gamma/X-ray emitters for potassium-40,
▪ DTPA actinides in urine (Pu-238, Pu-239, Am-241, and Cm-244),
▪ Tritium in urine for the sample from an exposed worker,
▪ Mass uranium in urine when the natural concentration cannot be negligible compared to the overload,
▪ Organically bound tritium in wheat or natural matrix.

PROCORAD does not have criteria for target uncertainties. The uncertainties of standards with metrological traceability are usually less than 5%.

8. DATA PROCESSING

Results are expressed in scientific notation with 3 significant figures in the format X,XX E±YY.

Participant results are not analyzed by peer group. The different methods can be visualized on the bias graphs provided to participants.

Participants must provide the uncertainties for their results (at k=2), and these are taken into account for evaluation. Outlier handling differs depending on the number of data points.
- If the number of data points is ≤12, a Grubbs test is performed once.
- If the number of data points is >12, the robust mean and robust standard deviation are calculated according to Algorithm A of the NF ISO 13528 standard to calculate the z-score for each participant's result. A z-score > 5 for a result is considered an outlier.

Outliers are excluded from the calculations.

There is no procedure for evaluating values excluded from the analysis: the participant is notified that their result is an outlier.

The standard deviation for proficiency assessment is determined for each round of interlaboratory comparisons with more than 12 results.

The standard deviation for proficiency assessment σPT is the robust standard deviation s* calculated according to algorithm A of the NF ISO 13528 standard.

For interlaboratory comparisons with fewer than 12 results, no standard deviation for proficiency assessment is determined; in this case, only the bias and the zeta-score are used to assessment.

9. STATISTICAL ANALYSIS OF RESULTS

The analysis of results and performance evaluations are carried out in accordance with the NF ISO 13528 standard, "Statistical methods used in proficiency testing by interlaboratory comparisons."

9.1 Descriptive statistics

Number of participating laboratories: p

Minimum value (min): corresponds to the smallest result in the series

Maximum value (max): corresponds to the highest result in the series

Median (med):

p elements (x1, x2, …, xp) $\text{med}(x) = x_{\{(p+1)/2\}}$ if p odd

$$\text{med}(x) = \frac{x_{\{p/2\}} + x_{\{1+p/2\}}}{2} \quad \text{if p even}$$

scaled median absolute deviation : $\text{MAD}_e(x) = 1{,}483 \times \text{med}(|x_i - \text{med}(x)|)$

arithmetic mean : $\bar{x} = \frac{\sum_{i=1}^{p} x_i}{p}$

The arithmetic standard deviation s $s = \sqrt{\frac{\sum_{i=1}^{p}(x_i - \bar{X})^2}{p-1}}$

9.2 Statistics for performance evaluation

9.2.1 Outlier Handling

If the number of data points is ≤ 12, a Grubbs test is performed once.

Calculation of G_{min} and G_{max} and comparison to G_{tab}

$G_{min} = \frac{|x_{min} - \bar{x}|}{s}$ and $G_{max} = \frac{|x_{max} - \bar{x}|}{s}$

Critical values G_{tab} Grubbs test risk α 5%									
3	4	5	6	7	8	9	10	11	12
1,155	1,481	1,715	1,887	2,020	2,196	2,215	2,290	2,355	2,412

If G_{min} and/or G_{max} > G_{tab}, then the value x_{min} and/or x_{max} is/are excluded.

The descriptive statistics parameters are recalculated: these are truncated descriptive parameters.

▪ If the number of data points is > 12, the robust mean X* and robust standard deviation s* are calculated according to algorithm A of the NF ISO 13528 standard to calculate the z-scores for each participant's result. A z-score > 5 obtained for a result is considered an outlier and will be excluded. 1) Initial Phase

The initial values of X* and s* are calculated as follows:

X* = median of xi (i = 1, 2, 3, ..., p) xi: result from participating laboratory
s* = MADe(x)

2) Iterative Phase

The new values of X* and s* are updated:

Calculation of δ = 1.5 s*

For each value of xi, calculation of xi*

xi* = X* - δ if xi < X* - δ

xi* = X* + δ if xi > X* + δ

xi* = xi otherwise

$X^* = \frac{\sum x_i}{p}$ and $s^* = 1.134 \times \sqrt{\frac{(x_i - X^*)^2}{p-1}}$

The estimates of X* and s* are derived iteratively by updating the data until the process converges: typically 5 or 6 iterations.

Once X* and s* are defined, the z-score is calculated for each participant's result.

$$z - \text{score} = \frac{x_i - X^*}{s^*}$$

All z-scores > 5 are excluded.

A new robust mean and a new robust standard deviation are calculated without the excluded values. These are the truncated robust mean and the truncated robust standard deviation.

9.2.2 Choix de la valeur assignée

This corresponds to:

- The certified value X_{ref} calculated during sample preparation in the case of a radioactive standard or a dilution of a radioactive standard used as a surcharge. The value is known before the comparison. This is called the reference assigned value.
- Or to the robust mean X* calculated from the participants' results, for example, in the case of natural radioelements. This is called the consensus assigned value.
- Or to the arithmetic mean $\bar{X}$ in the case of a number of results ≤ 12.

The certified value X_{ref} is validated by comparison to the robust mean X* if the following equation is satisfied :

$$|x_{ref} - X^*| \leq 2 \times \sqrt{u_{(ref)}^2 + \left(1{,}25 \times \frac{s^*}{\sqrt{p}}\right)^2}$$

If this inequality is not respected, the expert chooses the robust mean as the assigned value.
The expert may also, when the inequality is not respected, choose the certified value as the assigned value. In this case, the expert must conduct investigations to validate this choice.

9.2.3 Estimation of the Uncertainty of the Assigned Value

The expansion factor is k=2 for the expanded uncertainty.
▪ If the assigned value is a reference value: $U_{(Xref)}$. The uncertainty of the assigned value is calculated using the GUM method based on the main sources of uncertainty encountered during the preparation of the radioactive standard or the preparation of the radioactive standard dilution.
▪ If the assigned value is derived as a robust mean calculated from the participants' results, the uncertainty $U_{(X^*)}$ is calculated as follows:

$$U_{(X^*)} = 2 \times \frac{1.25 s^*}{\sqrt{p}}$$

▪ If the assigned value is deduced from the arithmetic mean (in the case of a number of results ≤ 12), the uncertainty $U_{(arith)}$ is:

$$U_{(arith)} = 2 \times \frac{s}{\sqrt{p}}$$

The uncertainty $U_{(Xref)}$ will be increased to account for any inhomogeneity and/or instability according to a method described by the expert laboratory (according to standard NF ISO 13528, based on the inter-sample standard deviation) during the annual meeting and in the final report.

In the case of $U_{(X^*)}$ and $U_{(ass)}$the variability related to inhomogeneity and/or instability is included in the standard deviation of the results provided by the participants.

9.2.4 Standard deviation for the proficiency assessment

The standard deviation for the proficiency assessment σPT is set by PROCORAD as being equal to the robust standard deviation s*.

9.3 Performance Criteria

The NF ISO 13528:2022 standard (§5.3 Considerations on the statistical distribution of results) states that "it is important to verify, at least visually, the approximate symmetry." Therefore, in certain cases (asymmetrical distribution, high coefficient of variation, fewer than 7 results, etc.), the expert will specify that the assigned value and its uncertainty are provided for informational purposes only, to warn you against using these values to evaluate your performance.

9.3.1 Bias

The bias value is given by the following relationship:

$$\text{Bias } \% = \frac{x_i - X}{X} \times 100$$

X is the assigned value determined, depending on the situation, as the certified value Xref, the robust mean X*, or the arithmetic mean $\overline{X}$.
The performance criterion for the bias percentage is that of ISO 28218 and NF ISO 12790-1:

-25% < Bias% < 50% satisfactory result

9.3.2 The z'-score

The z'-score is used instead of the z-score because the aptitude standard deviation σPT is the robust standard deviation s* calculated from the participants' results (see section 9.5.1 of NF ISO 13528), and the number of results can vary from 6 to 50 depending on the interlaboratory comparisons. This ensures consistent interpretation, independent of the number of participants.

The z'-score of a result, denoted z'_i, is given by the following relationship:

$$z'_i = \frac{x_i - X}{\sqrt{(s^*)^2 + u^2(X)}}$$

With $u^2(X)$, the standard uncertainty of the assigned value determined, depending on the situation, as the standard uncertainty of the certified value X_{ref}, the standard uncertainty of the robust mean X*, or the standard uncertainty of the arithmetic mean $\overline{X}$, the calculations of which are described in section 9.2.3 of this document.

The interpretation of the z'-score is as follows:

$|$z'-score$|$ ≤ 2: satisfactory result

2 < $|$z'-score$|$ ≤ 3: questionable result, requires monitoring

$|$z'-score$|$ > 3: discordant result requiring action.

9.3.3 The zeta-score (ζ-score)

To assess a participant's ability to provide results close to the assigned value within the stated uncertainty limits, PROCORAD provides the ζ-score as a performance criterion.

The ζ-score of a result, denoted ζ_i, is given by the following relationship:

$$\zeta_i = \frac{x_i - X}{\sqrt{(u_{x_i})^2 + u^2(X)}}$$

With u_{xi} being the standard uncertainty of the result provided by the participating laboratory and $u^2_{(X)}$ being the standard uncertainty of the assigned value determined, depending on the situation, as the standard uncertainty of the certified value $X_{réf}$ the standard uncertainty of the robust mean X*, or the standard uncertainty of the arithmetic mean $\overline{X}$, the calculations of which are described in section 9.2.3 of this document.

The interpretation of the κ-score is as follows:

ζ-score ≤ 2: satisfactory result

2 < ζ-score ≤ 3: questionable result, requires monitoring

ζ-score > 3: discordant result requiring action.

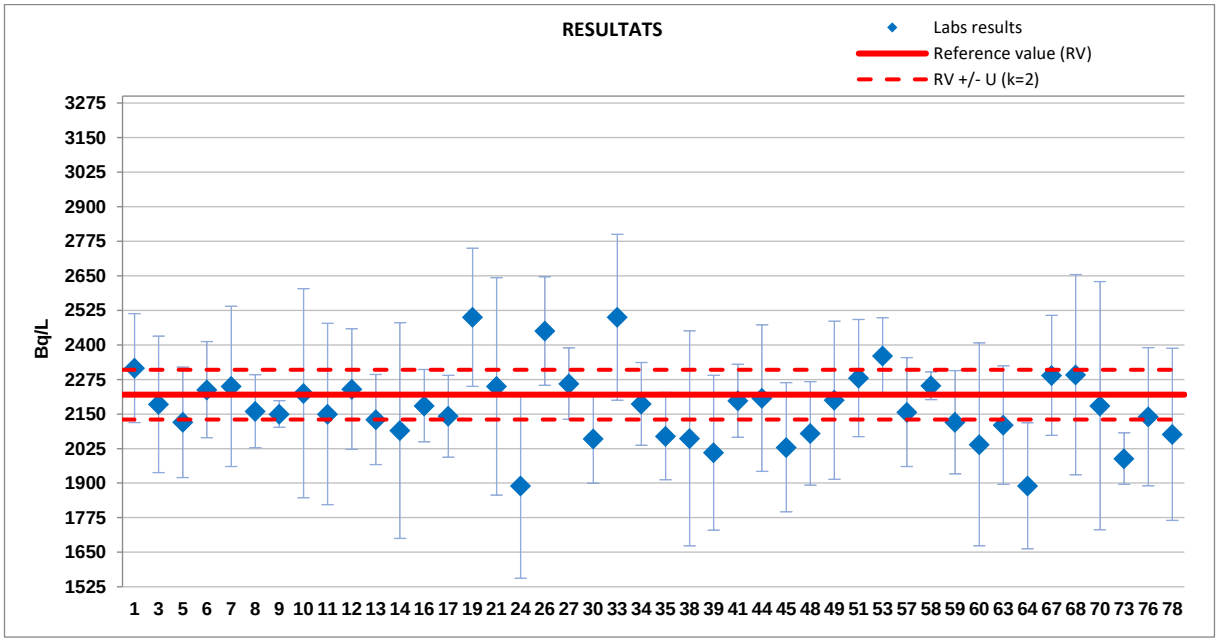
9.4 Other information provided

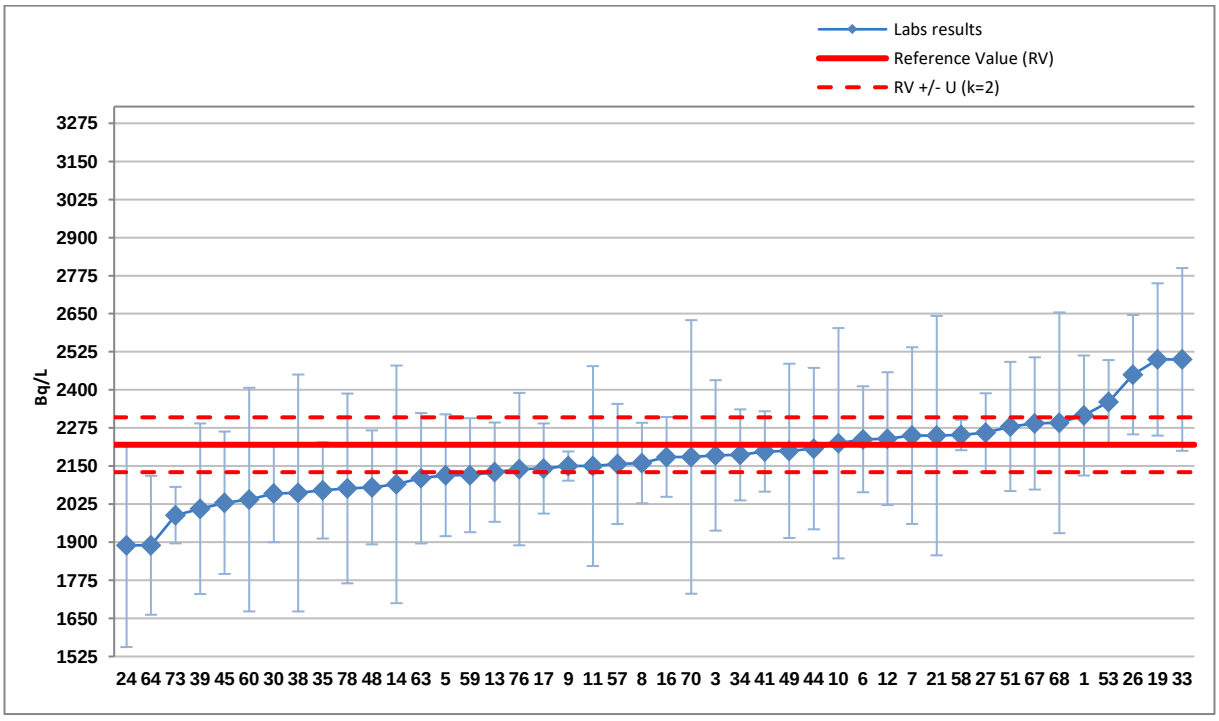
u_{min}: the lower limit relative uncertainty, calculated from the standard uncertainty of the assigned value according to section 9.8.3 of standard NF ISO 13528

u_{max}: the upper limit relative uncertainty, calculated from the standard uncertainty of the robust mean according to section 9.8.4 of standard NF ISO 13528

10. DIAGRAMS

10.1 Positioning and bias diagrams





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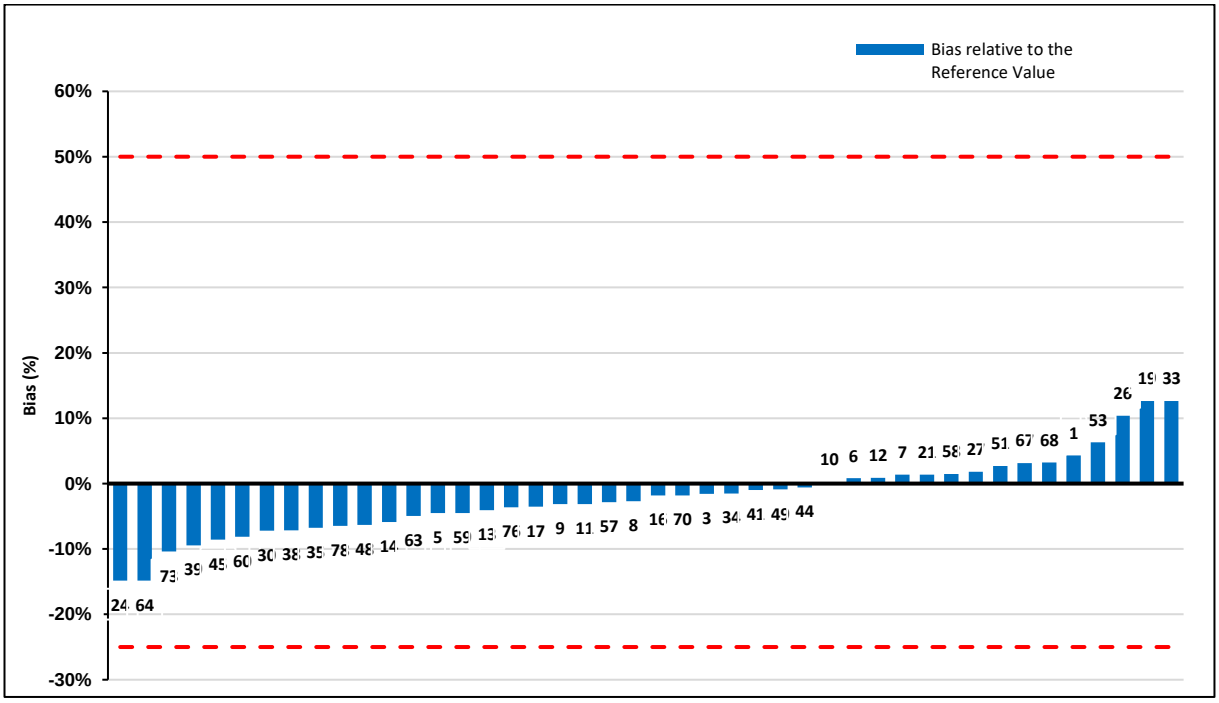
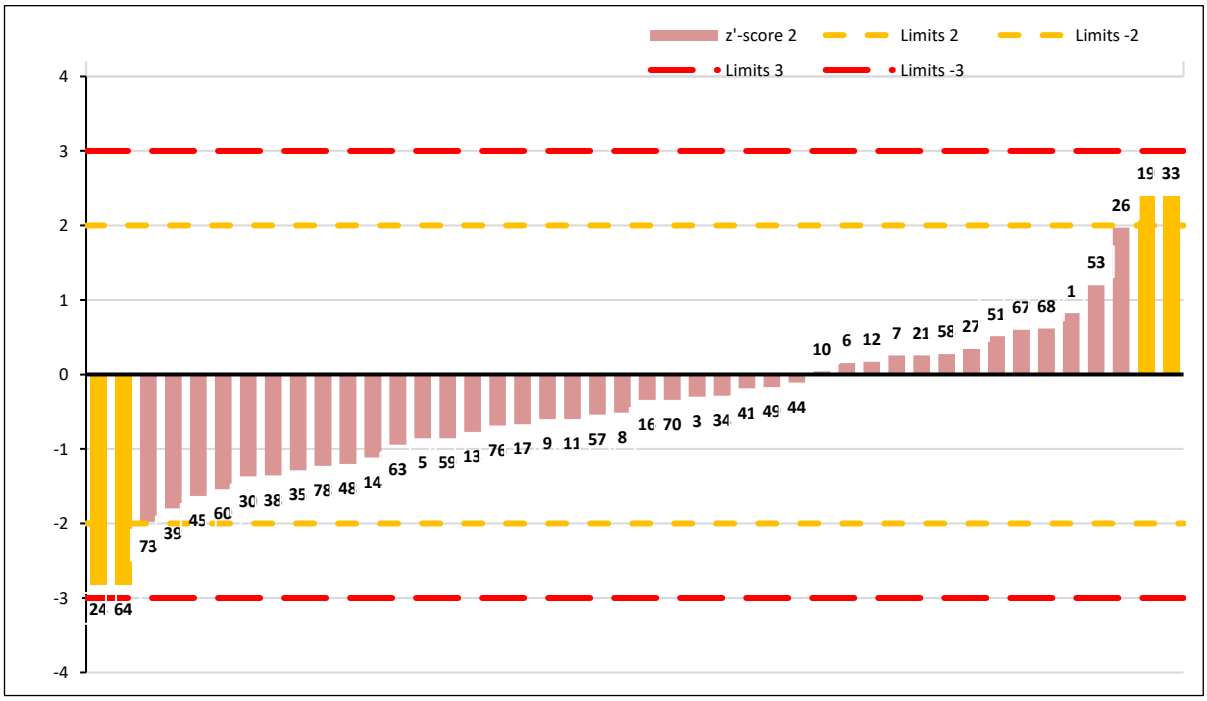
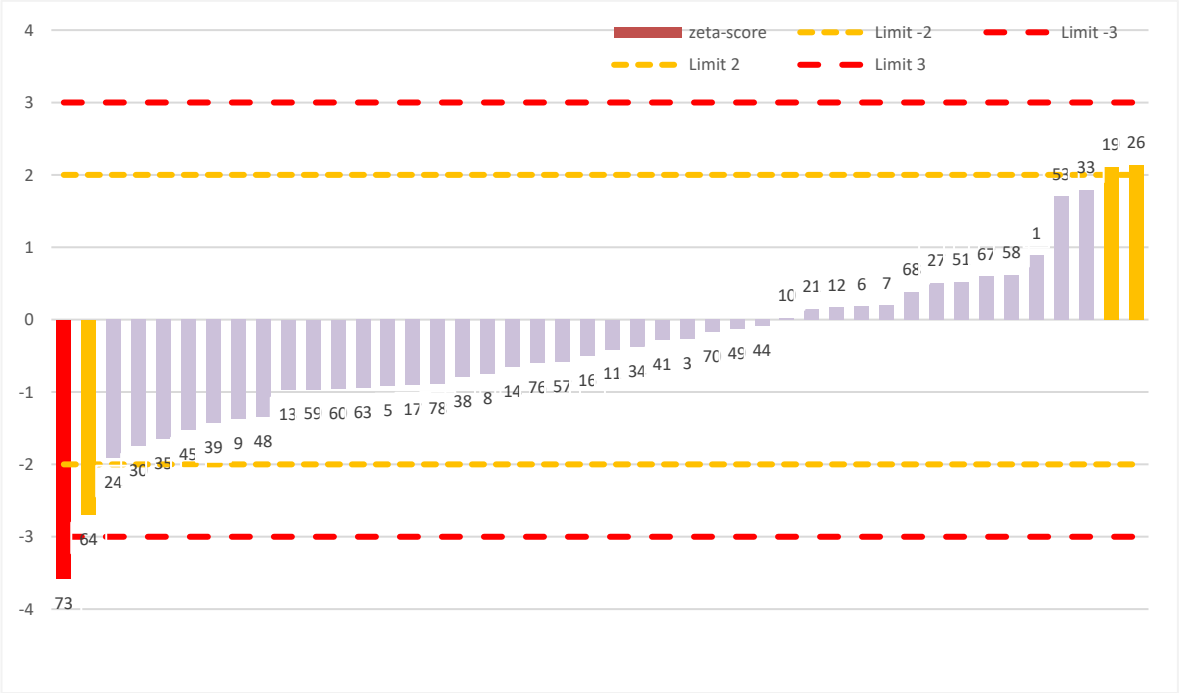


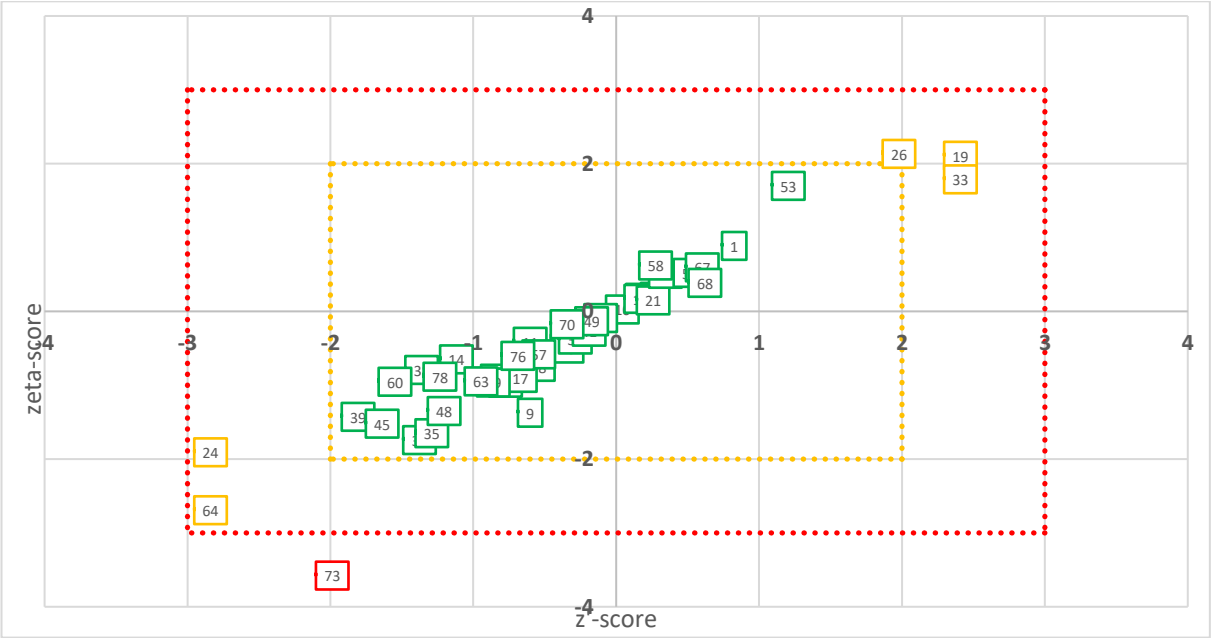
10.2 Z'-score diagrams



10.3 ζ-scores diagrams

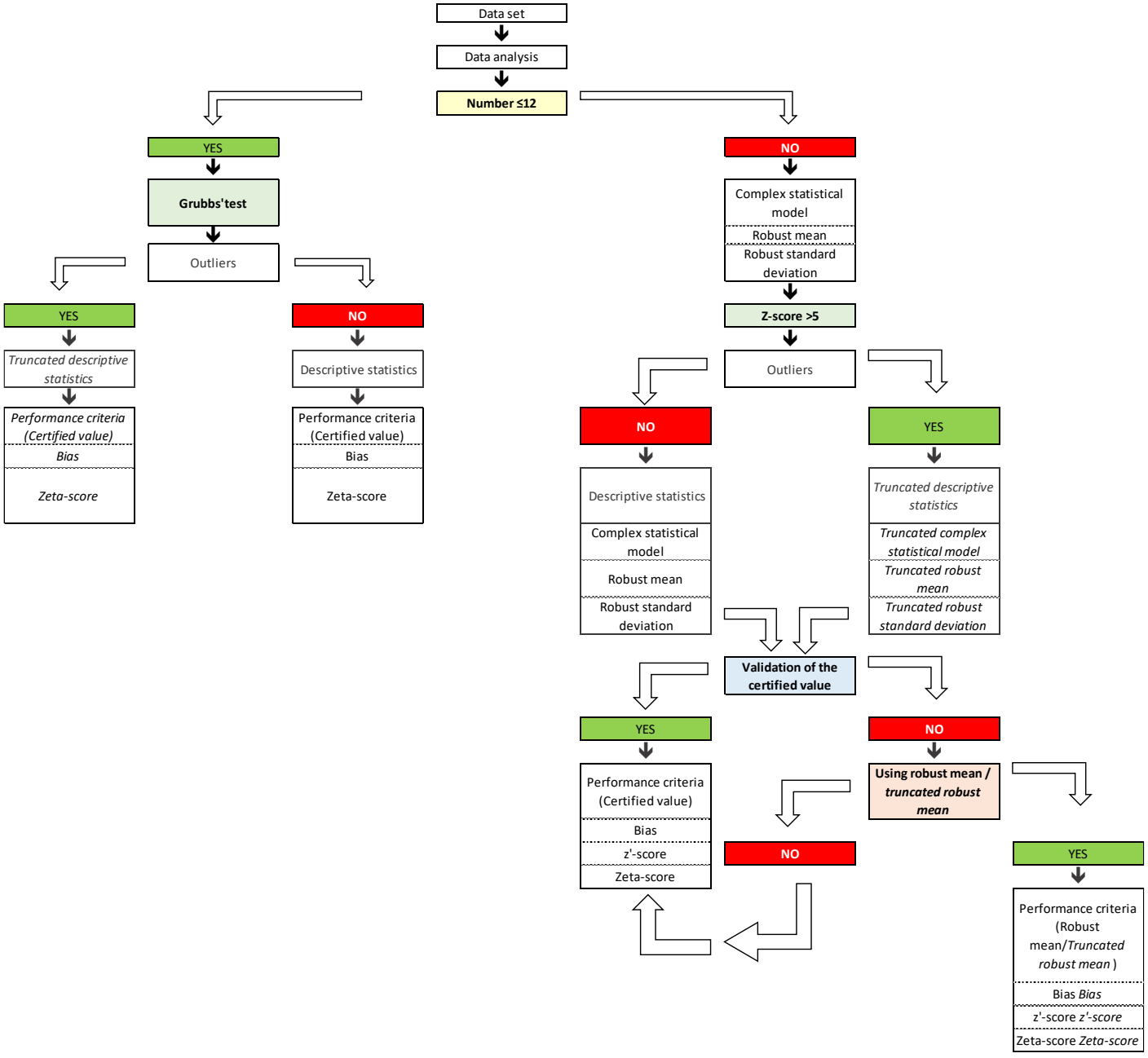


10.4 Diagrams combining the z'-score and the ζ-score



11. DATA PROCESSING FLOWCHARTS

11.1 Artificial Radionuclides



11.2 Natural Radionuclides

