

Proficiency Testing Samples in Radioactivity Analysis:
What they are and how to choose the
appropriate ones.

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Why PT?

PT provides an objective evidence of the competence of the participant. It can be used to:

- Identify measurement problems;
- Compare measurement procedures;
- Compare operator capabilities;
- Compare analytical systems;
- Evaluate measurement uncertainty;
- Determine measurement precision and/or trueness;
- Improve performance;
- Educate staff;
- Instill confidence in staff, management, and external users of laboratory services;
- Satisfy regulatory and accreditation bodies.

What kind of PT? Develop Strategy.

Factors to consider:

- Identify area of technical competence;
- Formulate Data Quality Objectives and Measurement Quality Objectives for specific tasks;
- Select Appropriate Method/Instruments and develop/adopt Analytical/Measurement procedures;
- Evaluate and select existing PT scheme or design a new one;
- Define Level and Frequency of participation;
- Verify the Competence of PT provider (ISO 17043);
- Verify that PT scheme is independent of any manufacturing or marketing interests in equipment, reagents or calibrators in its field of operation.

PT strategy

Perform Risk assessment by considering, for example:

- Limitations in methodology, e.g. instrument instability or interferences from matrix components;
- Quality and availability of Certified Reference Materials (CRMs);
- Complexity of the test procedure and changes in requirements, e.g. lower compliance limits;
- Number of tests/calibrations/measurements undertaken between PT rounds;
- Experience, knowledge and turnover of technical staff;
- How the results will be used, e.g. forensic science and environmental control, and the consequences of a wrong result being reported to a customer.

PT Sample

Proficiency test item (PT item) - Sample, product, artefact, **reference material**, piece of equipment, measurement standard, data set or other information used for proficiency testing *

In Radioactivity analysis, PT Sample most likely is a radioactive source prepared as a CRM fit for certain use in the analytical/measurement process. Manufacturing of these sources is driven by five major parameters of the final products:

- Uncertainty of the Activity Value;
- Homogeneity;
- Stability;
- Quantity prepared;
- Commutability.

* B. Brookman and I. Mann (eds.) Eurachem Guide: Selection, Use and Interpretation of Proficiency Testing (PT) Schemes (3rd ed. 2021).

Commutability - There are various definitions expressing this concept:

VIM. 5.15 - commutability of a reference material - property of a **reference material**, demonstrated by the closeness of agreement between the relation among the **measurement results** for a stated **quantity** in this material, obtained according to two given **measurement procedures**, and the relation obtained among the measurement results for other specified materials

Clinical Laboratory Standards Institute Guideline EP30-A recommends the use of the following definition for the term *commutability*: "The equivalence of the mathematical relationships among the results of different measurement procedures for an RM and for representative samples of the type intended to be measured."

Preparation and Certification of Large-Sized Dried (LSD) Spike IRMM-1027o, JRC 2013. - The degree of equivalence in the analytical behavior of real samples and a CRM with respect to various measurement procedures (methods) is summarized in a concept called 'commutability of a reference material'

Commutability

All sources used in the measurement process, PT Samples included, should be as much commutable (fit for purpose) as possible.

The commutability of a CRM is a crucial characteristic and must be established *a priori*. When commutability of a CRM is not established, the results from routinely used methods cannot be legitimately compared with the certified value of CRM.

Producer of CRM (PT Samples) can establish commutability for some materials and applications (as ISO 17034 suggests), but it is up to the laboratory to verify or establish the commutability of PT Samples. If it is not possible, laboratory should estimate potential problems of using not totally commutable CRM-PT Sample.

Laboratory can have great impact in designing commutable PT Samples with predefine parameters for specific application.

PT sample – “used as a whole” option

One of the reasons for the shortage of CRMs on the market is the cost of manufacturing. To drive costs down, EZA have adopted “use as a whole” concept for some of PT Sample preparation (no subsampling or division). Sample(s) is prepared per customer specs regarding isotopic composition, activity level, matrix and quantity usually by spiking of chosen matrix. User (and provider) of PT samples must ensure that spikes are not absorbed by container materials and the sample is transferred from the container quantitatively for further analysis. This option provides exceptional commutability and significant manufacturing cost reduction, allowing:

- Less restrictive (if not at all, in case of radiochemical analysis) homogeneity requirements;
- Flexibility in matrix choice, including perishable materials;
- Flexibility in spike choice (including refractory Pu and U, specific complexes etc.);
- Overall lower uncertainty for the activity value of isotopes used for spiked;
- Manufacturing of PT Samples within regulatory requirements (ISO 17025, ISO 17043 etc.).

PT samples for Determination of tritium in water

1. DI water spiked with tritiated water in flame sealed glass ampule – usually used directly for Liquid Scintillation Analysis
2. DI water spiked with with tritiated water, and some dissolved and not dissolved solid matter added. Not suitable for direct analysis and will require distillation
3. DI water spiked with tritiated water, some dissolved and not dissolved solid matter and some organics labeled with C-14 are added. Not suitable for direct analysis and will require oxidation of the organics and distillation.

PT samples for Determination of Plutonium in Soil.

- Analytical practice for plutonium determination in soil utilizes methods based on analysis of leachable plutonium and total sample dissolution methods. EZA approach for providing PT Samples for plutonium analysis covers both. Pu-239 is used in leachable form and Pu-238 in refractory form, or vice versa. Analysis of refractory form will require total dissolution of the sample.
- PT Samples are used as a whole, no division or subsampling, quantitative transfer from the shipping container for further analysis.
- Customer has a choice of getting soil PT Sample spiked with leachable form of plutonium, refractory form or both in the same sample.

PT Samples for Gamma Spec Analysis

- Organization have decided to establish network of laboratories capable of performing gamma spec analysis in emergency situations. All participating laboratories have the capabilities to perform gamma spec analysis, however it should not be assumed that results from all the laboratories will be comparable. Each laboratory's results must be assessed and verified.
- It has been decided that the participating laboratories will be provided with PT Samples spiked with fresh U-235 fission mixture isotopes. It was correctly assumed, that if a laboratory can analyze this complex mixture (including parent-daughter relations, decay correction to certain time, etc.) and provide correct results, it is fit for gamma spec analysis.
- 500 ml liquid PT Samples were provided to each laboratory, and it took several runs before labs were provided comparable results and were approved to perform analysis.

Participating in Proficiency Testing is expensive, and laboratories should pay close attention to PT process. Working together with PT provider laboratory should be able to select/design a PT scheme and develop commutable PT Samples to satisfy its needs.

After all,– “... it is the responsibility of the participants themselves to select the appropriate proficiency testing scheme and to evaluate their results correctly”*.

*Annex C, ISO 17043

Thank you